

**Department of Chemistry**

**PROJECT WORK**

**Marks List and Attendance**

Of

**VI SEMESTER**



# ರಾಣಿ ಜನ್ಮಾಂಗು



## ଶିଶୁଶିଳ୍ପାଳୟ



ஸாங்கிளிம். பார்வையு டெல்டா- 04, திருச்சிராம்பம், சிராம - 591158  
(ஸ்ரீ வித்திவீரப்பு - 2020)

# RANI CHANNAMMA UNIVERSITY

Vidyasangama, National Highway - 04, Bhootaramanahatti, Belagavi - 591156

(NAAC Accredited with B+ Grade - 2021)

## ಕುಲಸಚಿವರಕಾಯಾಂಲಯ

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## Office of the Registrar

ಪ.ಸಂ.: ರಾಜ್ಯ/ಬೆಳ್ಗಾವಿ/ಸ್ನಾತಕ ವಿಭಾಗ/2024-25/ ೬೬೯

ದಿನಾಂಕ: 20.05.2024

## ಸುತ್ತೊಂಡಿ

ವಿಷಯ: ಸ್ನಾತಕ ಕೋರ್ಸನ 6ನೇ ಸಮಿಸ್ತರಾನಲ್ಲಿ NEP ಪರ್ಯಾಕ್ರಮದಲ್ಲಿ INTERNSHIP/PROJECT  
ವಿಷಯದ ಕುರಿತು.

ಉಲ್ಲೇಖ: ಮಾನ್ಯ ಕುಲಪತಿಗಳ ಅನುಮೋದನೆ ದಿನಾಂಕ: 15.05.2024.

ಮೇಲ್ಮೈಸಿದ ವಿಷಯ ಹಾಗೂ ಉಲ್ಲೇಖಿಸಿದನ್ನು, ಈ ಮೂಲಕ ಎಲ್ಲ ಸ್ವಾತಕ ಮಹಾವಿದ್ಯಾಲಯಗಳ ರುಗಳಿಗೆ ತಿಳಿಸುವುದೇನೆಂದರೆ, ಸ್ವಾತಕ ಕೋರ್ಸನ ೬ನೇ ಸೆಮಿಸ್ಪರ್ಣನ NEP ಪಠ್ಯತ್ರಮದಲ್ಲಿ HIP/PROJECT ಎಂಬ ವಿಷಯವು ಇರುತ್ತದೆ. ಸದರಿ ಪಠ್ಯತ್ರಮಗಳನ್ನು INTERNSHIP/PROJECT COM, B.S.W, B.B.A, B.SC, B.C.A, B.SC(ST) ಕೋರ್ಸಗಳ ವಿವರವಾದ ನಿಯಮಾವಳಿಗಳನ್ನು ಈ ಮೊಂದಿಗೆ ಕಳುಹಿಸಿಕೊಡಲಾಗಿದೆ. ಸದರಿ ಪಠ್ಯತ್ರಮವನ್ನು ಹಾಗೂ ಇತರೆ ಅಂಶಗಳನ್ನು ೬ನೇ ಸೆಮಿಸ್ಪರ್ಣನಲ್ಲಿ ಕಾಳ್ಜಿಲ್ಲಿ ಈ ಮೂಲಕ ತಿಳಿಸಲಾಗಿದೆ.

Def W 215 from  
ಲುಪ್ಪಕುಲಸಚಿವರು

## ಉಪಕುಲಸಚಿವದ್ವಾ

ಉಪಕುಲಸಚಿವರು  
ರಾಣ ಚನ್ನನ್ನು ವಿಶ್ವಾಲಯ  
ಬೆಳ್ಗಾವಿ

ଲଗତୁ:

#### \***ఎల్ల కోసఫెరాల్ కు INTERNSHIP/PROJECT న నియమావళిగభు**

ಇವರಿಗೆ,

1. ಪ್ರಾಚಾರ್ಯರು, ಸಂಗೀಳ್ಳ ರಾಯ್ ಪ್ರಥಮದಬೇರ್ ಘಟಕ ಮಹಾವಿದ್ಯಾಲಯ. ಬೆಳಗಾವಿ.
2. ಪ್ರಾಚಾರ್ಯರು, ಬೆಳಗಾವಿ, ವಿಜಯಪುರ ಮತ್ತು ಬಾಗಲಕೋಟಿ ಜಿಲ್ಲೆಗಳ ಸಂಯೋಜಿತ ಸ್ನಾತಕ ಮಹಾವಿದ್ಯಾಲಯಗಳು.
3. ರಕ್ಷಾ ಪ್ರತಿ.

COURSE		BSC/BCA/BSC-ST						
Type of Course	Theory/ Practical	Credits	Instruction hour/week	Total No. of Lectures/ Hours / Semester	Duration of Exam	Formative Assessment Marks	Summative Assessment Marks	Total Marks
INTERNSHIP/ PROJECT	Practical	02	--	--	--	50	0	50

**Course Outcomes (COs): At the end of the Course students will be able to:**

- CO1:** Conduct the field visit based on objectives of the internship
- CO2:** Participation in a professional activity and gain practical work experience,
- CO3:** Learning the behavioural approach and fascinate in communication
- CO4:** Interact with the different personalities of local agencies.
- CO5:** Preparation of the report with advanced techniques/technology

**Note:** 1. Whenever an internship is not feasible, the students can choose the Project work  
 2. The internship/project work load can be shown in the time table for each faculty

**Project Work:** Short-term work in the college/other Institutions: The project work may include the work carried out in Educational Institutions /R & D organizations/review of current literature/ theoretical methods/ Mathematical applications.

Practical work may involve the execution of programs/ studies on properties/characterizations/ applications/activities for reported/unreported research or any suitable combination thereof. In the case of the students who would like to work outside the campus, the Supervising staff member may visit him/her/them.

Formative Assessment for Internship	
Assessment	Distribution of Marks
Case study/Field activity	20
Project report preparation & discussion	20
Viva-Voce	10
<b>Total</b>	<b>50 Marks</b>

**B.L.D.E.ASSOCIATION'S**  
**S. B. ARTS AND K. C. P. SCIENCE COLLEGE VIJAYAPUR**  
**DEPARTMENT OF CHEMISTRY**

**Students Project Details -2023-24**

**Class : B.Sc VI Semester**

**Course Title : INTI ( PROJECT WORK)**

**No. of Credits : 02**

**Course code :21BSC6INT1L**

**Project Incharge : Dr. S.D.Lamani**

**Project Title : INVESTIGATION OF NICOTINE IN TOBACCO PRODUCTS**

<b>Sl.No</b>	<b>Student Id No</b>	<b>Name of the Student</b>
1	U15KM21S0314	ANUSHA
2	U15KM21S0318	MEGHA BABURAO BIRADAR
3	U15KM21S0322	SOMANATH PATIL
4	U15KM21S0323	RAJASHREE REVANASIDDA UPPAR
5	U15KM21S0330	AISHWARYA SEELIN
6	U15KM21S0343	POOJA GIRAMALLAPPA TELI
7	U15KM21S0345	MAHANTESH NINGAPPA DESAI
8	U15KM21S0346	POOJA BAGALI
9	U15KM21S0353	BHAVYA MUDAKAPPA NAYAK

**Project Incharge : Dr. S. N. Unki**

**Project Title : Estimation of Oxalic acid in Tea leaves**

<b>Sl.No</b>	<b>Student Id No</b>	<b>Name of the Student</b>
1.	U15KM21S0450	MUBARAK MAIBOOBSAB MULLA
2.	U15KM21S0460	PRIYANKA KSHIRASAGAR
3.	U15KM21S0474	KAVYA SHANTAPPA HEBBAL
4.	U15KM21S0509	BHAGYASHREE NINGAPPA JATTI
5.	U15KM21S0514	MEGHA NATIKAR
6.	U15KM21S0553	NINGARAJ HONNAPPAGOUDA PATIL
7.	U15KM21S0557	AKSHATA JUMMANAVAR
8.	U15KM21S0578	NETRA KAKASAB PATIL
9.	U15KM21S0295	ABHISHEK KOLAKAR

**Project Incharge : Smt. Malati Chanagond**

**Project Title : Separation and Identification of Plant pigments in Spinach and Amaranth leaves using paper Chromatography.**

<b>Sl.No</b>	<b>Student Id No</b>	<b>Name of the Student</b>
1.	U15KM21S0307	VISHAL MAHADEV HONNALLI
2.	U15KM21S0316	BIAGYASHREE MALIPATIL
3.	U15KM21S0317	YAMANAPPA ILAGAR
4.	U15KM21S0319	DANESHWARI YALLAPPA BHOVI
5.	U15KM21S0373	CHANNABASAVARAJ SANNAPPA
6.	U15KM21S0379	REKHA
7.	U15KM21S0381	BASAVARAJ SIDDAPPA HANDRAL

**Project Incharge : Mr. Anilkumar Patil**

**Project Title : Acidity in tea leaves**

<b>Sl.No</b>	<b>Student Id No</b>	<b>Name of the Student</b>
1.	U15KM21S0382	METRE AISHWARYA SIDDARAM
2.	U15KM21S0397	PATIL SURAJ SAHEBGOUDA
3.	U15KM21S0408	SOUMYA VENKANAGOUDA PATIL
4.	U15KM21S0411	SHASHIKALA RAMESH
5.	U15KM21S0423	MALLIKARJUN HIREMATH
6.	U15KM21S0424	HIREMATH POOJA DUNDAYYA
7.	U15KM21S0433	NIRMALA RAMANNA NAIKODI
8.	U15KM21S0442	SHRUSHTI SHARANU
9.	U15KM21S0454	KAVYA SHEKHAR GARASANGI

**Project Incharge : Mr. Shivaraj Patil**

**Project Title : Analysis of honey**

<b>Sl.No</b>	<b>Student Id No</b>	<b>Name of the Student</b>
1.	U15KM21S0465	CHINNAMMA JAGADEV
2.	U15KM21S0471	MADHU MALLAPPA BARATAGI
3.	U15KM21S0476	POOJA MALLADI
4.	U15KM21S0481	SIDDARAM TILLIHAL
5.	U15KM21S0510	VAISHNAVI PADATARE
6.	U15KM21S0516	KEERTHI BASAVARAJ RATHOD
7.	U15KM21S0554	SUSHMITA CHANDRASHEKHAR
8.	U15KM21S0562	TEJASHWINI PANDIT
9.	U15KM21S0570	KALAVATI GAJAKOSH

**Project Incharge : Mr. Vinod Devannavar**

**Project Title : Estimation of Ingredients present in Chocolate.**

Sl.No	Student Id No	Name of the Student
1.	U15KM21S0571	ROOPA
2.	U15MY21S0086	POOJA NANDABASAPPA
3.	U15MY21S0050	Anushree Biradar
4.	U15MY21S0010	RADIKA SP
5.	U15MY21S0269	AISHWARYA HONNAD
6.	U15MY21S0347	LAXMI HOSUR
7.	U15MY21S0352	PRAGATI GODIHAL
8.	U15MY21S0118	MEGHA KUDAGI
9.	U15MY21S0103	SUMA VADDAR

**Project Incharge : Miss. Vidya Jabagoudar**

**Project Title : Extraction of Caffeine from Tea powder.**

Sl.No	Student Id No	Name of the Student
1.	U15MY21S0044	ANKITA VOGMORE
2.	U15MY21S0248	BHAGYSHREE K
3.	U15MY21S0457	SUKANYA B
4.	U15MY21S0296	KAVERI UPPAR
5.	U15MY21S0533	AISHWARYA C
6.	U15MY21S0493	AISHWARYA S
7.	U15MY21S0267	SAVITRI JAMABAGI
8.	U15MY21S0252	MEGHA SAYGAVI
9.	U15MY21S0109	SONIKA KADAM

**Project Incharge : Miss. Laxmi Savalage**

**Project Title : Foaming capacity of Soap**

Sl.No	Student Id No	Name of the Student
1.	U15KM21S0332	ARPITA SOLAPUR
2.	U15KM21S0338	ANJANA BIRADAR
3.	U15KM21S0292	SANJANA PAWATE
4.	U15KM21S0386	SIDDANAGOUDA BIRADAR
5.	U15KM21S0432	LINGARAJ M HATTI
6.	U15KM21S0507	RAHUL S BAGALI
7.	U15KM21S0513	PRAJWAL YATHNOOR
8.	U15KM21S0332	ARPITA SOLAPUR
9.	U15KM21S0338	ANJANA BIRADAR

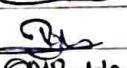
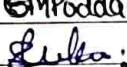
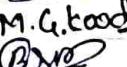
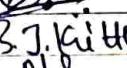
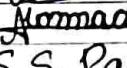
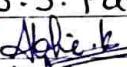
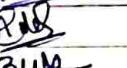
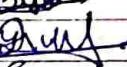
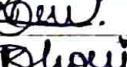
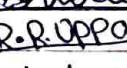
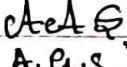
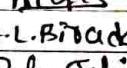
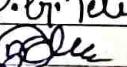
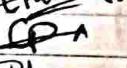
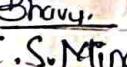
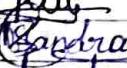
BLDEA's  
S B Arts and K C P Science College, Vijayapur  
Department of Chemistry

Attendance Sheet of Project work Viva-voce Aug- 2023-24

Class : B.Sc VI Semester

Subject code : 21BSC6INT1L

Date : 14/8/2024

SL NO	UUCMS NO.	NAME OF THE STUDENT	Signature
1	U10AD21S0010	RADHIKA PACHAPURE	
2	U15KM21S0044	ANKITA MADEV VAGAMORE	
3	U15KM21S0050	ANUSHREE BIRADAR	
4	U15KM21S0092	IRANNA KUMBAR	
5	U15KM21S0103	SUMA PODDAR	
6	U15KM21S0109	SANIKA KADAM	
7	U15KM21S0118	MEGHA KOODAGI	
8	U15KM21S0144	BHAGYSHRI SOMANAGOUDE MUDNUR	
9	U15KM21S0248	BHAGYASHREE KITTUR	
10	U15KM21S0252	MEGHA S SAYAGAVI	
11	U15KM21S0267	SAVITRI SIDDAPPA JAMBAGI	
12	U15KM21S0269	AISHWARYA MAHADEV HONNAD	
13	U15KM21S0292	SANJANA SHIVANANDA PAWATE	
14	U15KM21S0295	ABHISHEK KOLAKAR	
15	U15KM21S0296	KAVERI DHULAPA UPPAR	
16	U15KM21S0307	VISHAL MAHADEV HONNALLI	
17	U15KM21S0314	ANUSHA	
18	U15KM21S0316	BHAGYASHREE MALIPATIL	
19	U15KM21S0317	YAMANAPPA ILAGER	
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21	U15KM21S0319	DANESHWARI BHOVI	
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28	U15KM21S0346	POOJA MALAPPA BAGALI	
29	U15KM21S0347	LAKSHMI RAVI HOSUR	
30	U15KM21S0352	PRAGATI SIDDAPPA GODIHALA	
31	U15KM21S0353	BHAVYA MUDAKAPPA NAYAK	
32	U15KM21S0373	CHANNABASAVARAJ SANNAPPA MINAJAGI	
33	U15KM21S0378	BHUMIKA	
34	U15KM21S0379	REKHA	
35	U15KM21S0381	BASAVARAJ HANDRALA	
36	U15KM21S0386	SIDDANAGOULD SIDDALINGAPPA BIRADAR	
37	U15KM21S0394	SACHIN MUTTAPPA CHOURE	

SL NO	UUCMS NO.	NAME OF THE STUDENT	Signature
38	U15KM21S0397	SURAJ SAHEBAGOURA PATIL	<i>S Patil Bleu.</i>
39	U15KM21S0408	SOUMYA VENKANAGOURA PATIL	
40	U15KM21S0411	SHASHIKALA RAMESH HUGAR	
41	U15KM21S0417	SURAJ SURESH KUPPI	<i>S De Kuppi</i>
42	U15KM21S0422	DANESHWARI MADAPPA GUGGARI	
43	U15KM21S0423	MALLIKARJUN HIREMATH	
44	U15KM21S0424	HIREMATH POOJA DUNDAYYA	
45	U15KM21S0430	LAXMI BASAPPA BIRADAR	<i>Laxmi B. Biradar</i>
46	U15KM21S0432	LINGARAJ MALINGARAYA HATTI	
47	U15KM21S0433	NIRMALA RAMANNA NAIKODI	
48	U15KM21S0436	POORNIMA	<i>Poornima</i>
49	U15KM21S0439	AISHWARYA	
50	U15KM21S0442	SHRUSHTI SHARANU PATTANASHETTI	
51	U15KM21S0443	VEERESH ANGADI	<i>VA NA</i>
52	U15KM21S0444	SHIVANI BALASAB GHADGE	<i>Sh Ghadge</i>
53	U15KM21S0450	MUBARAK MAIBOOSAB MULLA	
54	U15KM21S0454	KAVYA SHEKIIAR GARASANGI	<i>Kavya</i>
55	U15KM21S0457	SUKANYA SHARANAPPA BIRAKABBI	<i>S.S. Birakabbi</i>
56	U15KM21S0460	PRIYANKA A KSHIRASAGAR	<i>P.K.S.</i>
57	U15KM21S0465	CHINNAMMA J SHIRASHYAD	<i>Chinna Shirashyad</i>
58	U15KM21S0471	MADHU MALLAPPA BARATAGI	<i>M. M. B. Baratagi</i>
59	U15KM21S0474	KAVYA SHANTAPPA HEBBAL	
60	U15KM21S0476	POOJA MALLADI	<i>Pooja Malladi</i>
61	U15KM21S0479	SURESH SINGH H RAJAPUT	<i>R Rajaput</i>
62	U15KM21S0481	SIDDARAM TILLIHAL	<i>Siddaram Tillihal</i>
63	U15KM21S0493	AISHWARYA VIJAYAKUMAR SATAPUTE	<i>Aishwarya Satapute</i>
64	U15KM21S0505	NINGARAJ BHIMANNA YATHANUR	<i>NB. Yathanur</i>
65	U15KM21S0507	RAHUL SANGANAGoud BAGALI	<i>Rahul Bagali</i>
66	U15KM21S0509	BHAGYASHREE NINGAPPA JATTI	<i>B. N. Jatti</i>
67	U15KM21S0510	VAISHNAVI V PADATARE	<i>V.N. Padatare</i>
68	U15KM21S0513	PRAJWAL YATANOOR	<i>Prajwal Yatanoor</i>
69	U15KM21S0514	MEGHA NATIKAR	<i>Megha Natikar</i>
70	U15KM21S0516	KEERTHI BASAVARAJ RATHOD	<i>Keerti Rathod</i>
71	U15KM21S0533	AISHWARYA TAMMARAY CHADACHAN	<i>Aishwarya Chadachan</i>
72	U15KM21S0553	NINGARAJ HONAPPAGoud PATIL	<i>N.R. Patil</i>
73	U15KM21S0554	SUSHMITA CHANDRASHEKHAR BIRADAR	<i>S.C.B.</i>
74	U15KM21S0557	AKSHATA	<i>Akshata</i>
75	U15KM21S0562	TEJASHWINI PANDIT MALASIDDANAVAR	<i>Tejaswini Pandit</i>
76	U15KM21S0564	SAHANA PATTAR	<i>Sahana Pattar</i>
77	U15KM21S0570	KALAVATI RUDRAPPA GAJAKOSH	<i>Kalavati Rudrappa Gajakosh</i>
78	U15KM21S0571	ROOPA	<i>Roopa</i>
79	U15KM21S0578	NETRA KAKASAB PATIL	<i>Netra Kakasab Patil</i>
80	U15MY21S0086	POOJA N BABALESHWAR	<i>RBS</i>



**Head**

Department of Chemistry  
S.B.Arts & K.C.P.Science College,  
Vijayapur.



**Principal**

S.B.Arts & K.C.P.Science College,  
VIJAYAPUR.

Department of Chemistry

Marks list of Project work Aug- 2023-24

Class : B.Sc VI Semester

No. of Credits : 02

Course Title : INTI ( PROJECT WORK)

Course code :21BSC6INT1L

SL NO	UTCMS NO.	NAME OF THE STUDENT	PROJECT REPORT (OUT OF 20)	PRESENTATION (OUT OF 20)	VIVA-VOCE (OUT OF 10)	TOTAL (50)
1	U10AD21S0010	RADHIKA PACHAPURE	19	20	09	48
2	U15KM21S0044	ANKITA MADEV VAGAMORE	18	18	9	44
3	U15KM21S0050	ANUSHREE BIRADAR	19	20	09	48
4	U15KM21S0092	IRANNA KUMBAR	16	18	08	42
5	U15KM21S0103	SUMA PODDAR	20	19	09	48
6	U15KM21S0109	SANIKA KADAM	20	20	10	50
7	U15KM21S0118	MEGHASHODAGI	18	17	08	43
8	U15KM21S0144	BHAGYSHRI SOMANAGOUDA MUDNUR	19	20	09	48
9	U15KM21S0248	BHAGYASHREE KITTUR	20	19	09	48
10	U15KM21S0252	MEGHASHAYAGAVI	19	19	09	47
11	U15KM21S0267	SAVITRI SIDDAPPA JAMBAGI	20	20	10	50
12	U15KM21S0269	AISHWARYA MAHADEV HONNAD	20	19	10	49

SL NO	UTUCMS NO.	NAME OF THE STUDENT	PROJECT REPORT (OUT OF 20)	PRESIDENTATION (OUT OF 20)	VIVA - VOCE (OUT OF 10)	TOTAL (50)
13	U15KM21S0292	SANJANA SHIVANANDA PAWATE	19	20	10	49
14	U15KM21S0295	ABHISHEK KOLAKAR	16	17	08	41
15	U15KM21S0296	KAVERI DHULAPA UPPAR	19	20	10	49
16	U15KM21S0307	VISHAL MAHADEV HONNALLI	20	20	09	49
17	U15KM21S0314	ANUSHYA	19	19	09	47
18	U15KM21S0316	BHAGYASHREE MALIPATIL	20	20	10	50
19	U15KM21S0317	YAMANAPPA ILAGER	20	19	08	47
20	U15KM21S0318	MEGHA BABURAO BIRADAR				
21	U15KM21S0319	DANESHWARI BHOVI	20	20	10	50
22	U15KM21S0323	RAJASHREE REVANASIDDA UPPAR	17	19	09	45
23	U15KM21S0330	AISHWARYA SEELIN	18	19	09	46
24	U15KM21S0332	ARPITA SOLAPUR	19	20	09	48
25	U15KM21S0338	ANJANA L BIRADAR	19	20	09	48
26	U15KM21S0343	POOJA GIRAMALLAPPA TELI	19	19	09	47
27	U15KM21S0345	MAHANTESH NINGAPPA DESAI	17	17	07	41
28	U15KM21S0346	POOJA MALAPPA BAGALI	19	19	09	47
29	U15KM21S0347	LAKSHMI RAVI HOSUR	20	20	09	49
30	U15KM21S0352	PRAGATI SIDDAPPA GODIHALA	19	20	09	48
31	U15KM21S0353	BHAVYA MUDAKAPPA NAYAK	19	20	10	49
32	U15KM21S0373	CHANNABA SAVARAJ SANNAPPA MINAJAGI	20	19	09	48
33	U15KM21S0378	BHUMIKA	20	20	10	50
34	U15KM21S0379	REKHA	20	19	10	49
35	U15KM21S0381	BASAVARAJ HANDRALA	19	19	08	46
36	U15KM21S0386	SIDDANAGOUD SIDDALINGAPPA BIRADAR	20	20	10	50
37	U15KM21S0394	SACHIN MUTTAPPA CHOURI	20	20	9	49

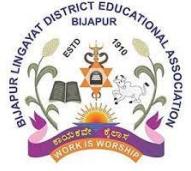
SL NO	UUCMS NO.	NAME OF THE STUDENT	PROJECT REPORT (OUT OF 20)	PRESIDENT ON (OUT OF 20)	VIVA-VOCE (OUT OF 10)	TOTAL (50)
38	U15KM21S0397	SURAJ SAHEBAGOUDA PATIL	20	20	08	48
39	U15KM21S0408	SOUMYA VENKANAGOUDA PATIL	20	20	10	50
40	U15KM21S0411	SHASHIKALA RAMESH HUGAR	20	20	09	49
41	U15KM21S0417	SURAJ SURESH KUPPI	20	20	09	49
42	U15KM21S0422	DANESHWARI MADAPPA GUGGARI	20	20	10	50
43	U15KM21S0423	MALLIKARJUN HIREMATH	20	20	08	48
44	U15KM21S0424	HIREMATH POOJA DUNDAYYA	20	20	08	48
45	U15KM21S0430	LAXMI BASAPPA BIRADAR	20	20	10	50
46	U15KM21S0432	LINGARAJ MALINGARAYA HATTI	19	20	10	49
47	U15KM21S0433	NIRMALA RAMANNA NAIKODI	20	20	08	48
48	U15KM21S0436	POORNIMA	20	20	10	50
49	U15KM21S0439	AISHWARYA	20	20	10	50
50	U15KM21S0442	SHRUSHTI SHARANU PATTANASHETTI	20	20	08	48
51	U15KM21S0443	VEERESH ANGADI	20	20	09	49
52	U15KM21S0444	SHIVANI BALASAB GHADGE	20	20	10	50
53	U15KM21S0450	MUBARAK MAIBOOBSAB MULLA	18	20	10	48
54	U15KM21S0454	KAVYA SHEKHAR GARASANGI	20	20	09	49
55	U15KM21S0457	SUKANYA SHARANAPPA BIRAKABBI	19	19	10	48
56	U15KM21S0460	PRIYANKA A KSHIRASAGAR	20	18	10	48
57	U15KM21S0465	CHINNAMMA J SHIRASHYAD	20	20	09	49
58	U15KM21S0471	MADHU MALLAPPA BARATAGI	20	20	10	50
59	U15KM21S0474	KAVYA SHANTAPPA HEBBAL	20	18	10	48
60	U15KM21S0476	POOJA MALLADI	20	19	10	49
61	U15KM21S0479	SURESH SINGH H RAJAPUT	16	11	7	44
62	U15KM21S0481	SIDDARAM TILLIHAL	20	20	10	50
63	U15KM21S0493	AISHWARYA VIJAYAKUMAR SATAPUTE	20	20	10	50
64	U15KM21S0505	NINGARAJ BHIMANNA YATHANUR	20	16	07	43
65	U15KM21S0507	RAHUL SANGANAGOUD BAGALI	20	20	10	50
66	U15KM21S0509	BHAGYASHREE NINGAPPA JATTI	18	20	10	48

SL NO	UUCMS NO.	NAME OF THE STUDENT	PROJECT REPORT (OUT OF 20)	PRESENTATION (OUT OF 20)	VIVA-VOCE (OUT OF 10)	TOTAL (50)
67	U15KM21S0510	VAISHNAVI V PADATARE	20	20	09	49
68	U15KM21S0513	PRAJWAL YATANOOR	17	17	07	41
69	U15KM21S0514	MEGHNA NATIKAR	20	20	08	48
70	U15KM21S0516	KEERTHI BASAVARAJ RATHOD	20	20	10	50
71	U15KM21S0533	AISHWARYA TAMMARAY CHADACHAN	20	20	10	50
72	U15KM21S0553	NINGARAJ HONAPPAGOUD PATIL	10	16	07	33
73	U15KM21S0554	SUSHMITA CHANDRASHEKHAR BIRADAR	20	20	10	50
74	U15KM21S0557	AKSHATA	20	18	10	48
75	U15KM21S0562	TEJASHWINI PANDIT MALASIDDANAVAR	20	20	10	50
76	U15KM21S0564	SAHANA PATTAR	20	19	10	49
77	U15KM21S0570	KALAVATI RUDRAPPA GAJAKOSH	20	20	09	49
78	U15KM21S0571	ROOPA	19	20	09	48
79	U15KM21S0578	NETRA KAKASAB PATIL	18	20	10	48
80	U15MY21S0086	POOJA N BABALESHWAR	20	20	10	50

**Head**

Department of Chemistry  
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VIJAYAPUR.



**B. L. D. E. Association's**  
**S. B. Arts and K. C. P. Science College,**  
**Vijayapur-586103**

**DEPARTMENT OF CHEMISTRY**

**Project on**

**Synthesis and Characterization of Schiff  
Bases Derived from 3-Acetyl Coumarins  
and 2,4-Dinitrophenylhydrazine**

**By**

**Name: AISHWARYA**

**RCU No: U15KM21S0439**

**Submitted To**

**Dr. K. Mahesh Kumar**

**2023-24**

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## Acknowledgement

*I would like to express my special thanks of gratitude to my subject teacher Dr. K. Mahesh Kumar sir to give his guidance to make the successful completion of this project.*

*I also want to give special thanks to our Head of the Department Dr. S. D. Lamani sir and principal Dr. R. M. Mirdhe madam who gave me this golden opportunity to do this wonderful project on the topic "Synthesis and Characterization of Schiff Bases Derived from 3-Acetyl Coumarins and 2,4-Dinitrophenylhydrazine", so that I will get to know about detailed information for the same.*

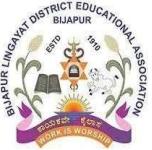
*Secondly I would like to thank my parents and classmates who helped me to complete this project within the given time frame.*

**AISHWARYA**



## **TITLE OF THE PROJECT**

**SYNTHESIS AND CHARACTERIZATION OF SCHIFF  
BASES DERIVED FROM 3-ACETYL COUMARINS  
AND 2,4-DINITROPHENYLHYDRAZINE**



**B. L. D. E. ASSOCIATION'S**  
**S. B. ARTS AND K. C. P. SCIENCE COLLEGE, VIJAYAPUR**  
**DEPARTMENT OF CHEMISTRY**



**Date:**

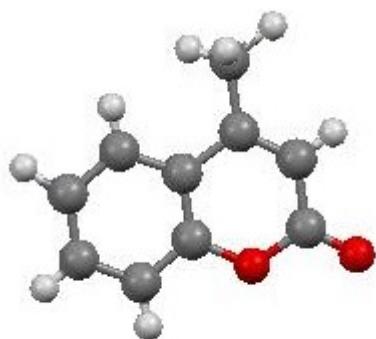
## **CERTIFICATE**

This is to certify that, Miss AISHWARYA (U15KM21S0439) studying in B. Sc. VI semester during the year 2023-24, has completed the project entitled **“Synthesis and Characterization of Schiff Bases Derived from 3-Acetyl Coumarins and 2,4-Dinitrophenylhydrazine”**. This Project work is in partial fulfillment for the award of degree of Bachelor of Science. The project work satisfies the requirements prescribed in the curriculum of **“Rani Channamma University, Belagavi”**.

**Guide of the Project**

**Head of the Department**

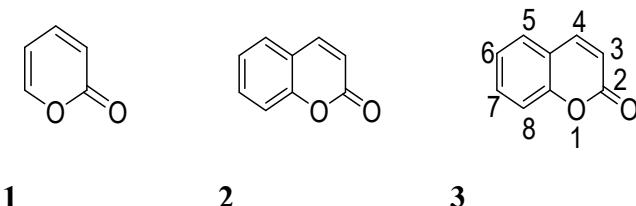
# Introduction to the Chemistry of Coumarins



## 1. CHEMISTRY OF COUMARINS

### 1.1 INTRODUCTION

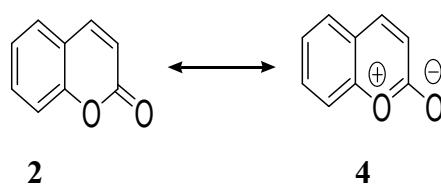
Coumarins are an important class of oxygen heterocycles, which are widespread in plant kingdom and have been extensively reported on. Their chemical structure can be looked upon as arising out of the fusion of a benzene ring to pyran-2-one **1**, across the 5 and 6 positions in skeleton.



The parent coumarin **2** was first isolated by Vogel in 19<sup>th</sup> century from Tonka beans<sup>1</sup> and even to this date finds itself still in use as perfumery and flavoring agent. Figure **3** represent the numbering system used in coumarin skeleton<sup>2</sup>.

### Structure and reactivity

Aromatic nature of heterocyclic ring of coumarin is disputable, because coumarin shows some reactions of aliphatic compounds and other characteristics of aromatic compounds. The complete aromaticity in coumarin can be only realized if O-CO function contributes two electrons to form 10 $\pi$  electron system. This means that coumarin should be a resonance hybrid, to which contribution from canonical form **4** is significant. However, no evidence is found in the spectra of coumarin to suggest that contribution from betaine form **4** is considerable

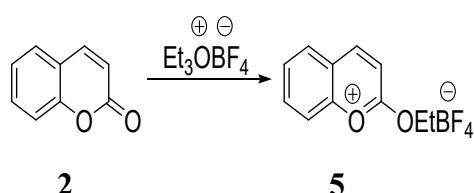


The infrared absorption spectrum of coumarin shows an absorption band at 1710  $\text{cm}^{-1}$  which is attributed to lactone carbonyl group but not a betain from. In the  $^1\text{H}$  NMR spectrum of coumarin<sup>3</sup>, the signal due to protons of C3 and C4 appears in the region of 6.45  $\delta$  ppm and 7.80  $\delta$  ppm with coupling constants of 9.8Hz. These values are typical of *cis* alkene rather than an aryl ring<sup>4</sup>. Finally the  $^{13}\text{C}$  NMR spectra of coumarins<sup>5</sup> are consistant with an essentially aliphatic heterocyclic ring. The chemical shifts of C2, C3 and C4 in coumarin remarkably close to the values for the corresponding carbons in  $\alpha$ -pyrone and are given below

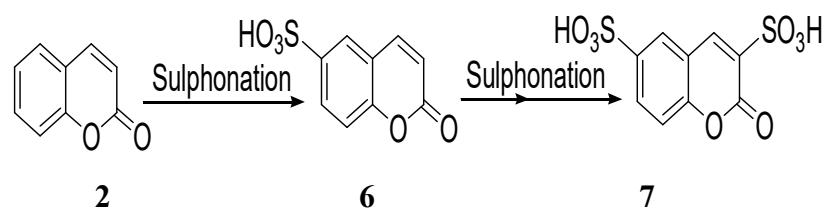
Compound	C2	C3	C4
$\alpha$ -Pyrone	162.0	116.7	144.3
Coumarin	160.4	116.4	143.4

But coumarin does show aromatic character in its pattern of reactivity, e.g.,

The carbonyl oxygen can be alkylated<sup>6</sup> by powerful agents to give stable pyrillium salts **5**.

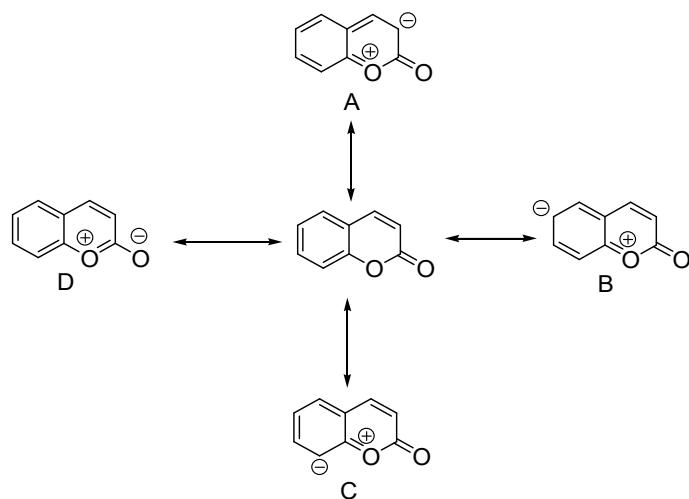


Coumarin nucleus is susceptible to electrophilic substitution<sup>6</sup>. Sulphonation takes place initially in the carbocyclic ring at C6, to give **6**, but under more forcing conditions one more  $-\text{SO}_3\text{H}$  group can be introduced at C3, to obtain coumarin-3, 6-disulphonic acid **7**.

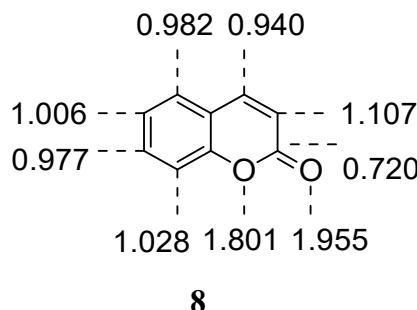


As in case of simple pyrones the properties of heterocyclic ring of coumarin are greatly influenced by the presence of substituents.

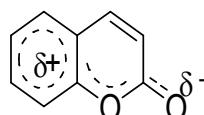
Anantatakrishnan<sup>7</sup> discussed the “Mills-Nixon effect” in which the reactivity of coumarin was rationalized based on the comparative studies of bromination and nitration of coumarin, naphthalene and benzene. By considering the possible electron movements in coumarin molecule, Thakur and Shah<sup>8</sup> predicted that C6 and C8 as the most reactive centres. The electron movements are as shown below.



Greater electron densities can be seen on C6 and C8 from the resonating structures B and C. Out of these two, C6 seems to be more reactive because of its proximity to the oxygen atom, similar to the reactivity of para position of phenol. Structure A though imparts more electron density to the C3 position, the electrophilic substitution at C3 is less, probable due to its closeness to the electron withdrawing carbonyl group. Infact the  $\pi$  electron densities calculated by Song and Gorden<sup>9</sup> are quite close to the resonance picture of the molecule. The structure **8** represents the  $\pi$  electron densities for the ground state of coumarin.



By considering the structure's B, C and D Bassingnan and Cogrossi<sup>10</sup> have proposed structure **9** which is according to them represents the hybrid or resonating state of molecule.



However the contributing structure of the type (D) does not have strong spectral evidences, the position of the carbonyl frequency in the IR spectrum ( $1710\text{ cm}^{-1}$ ) is more in favor of an enol lactone<sup>11</sup>. Hence the contribution from such type of structures is negligible and the resonating state **9** appears to be less probable.

Coumarin has been used as a powerful model in elucidating the electronic structures and photo reactivity of psoralenes. The configurational analysis of coumarin by Song et al.<sup>12</sup> in the ground state indicates some charge transfer delocalization extending to the ethylenic region. The dipole movements of coumarin ( $4.82 \times 10^{-8}\text{ e.s.u}$ ) determined earlier by Rao<sup>13</sup> also indicates the similar delocalization.

## Spectral studies

### UV-Spectra:

The UV spectra of coumarins and their methyl derivatives were reported by Ganguly and Bagchi.<sup>14</sup> The introduction of methyl group in various positions does not change the nature of the spectrum to a greater extant. The  $\lambda_{\text{max}}$  and  $\epsilon$  values of coumarins are 273 nm (40,368) & 309 nm (37,449).

### **IR-Spectra:**

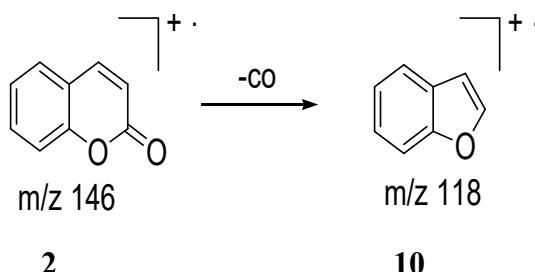
The IR spectrum of coumarin was reported by Murthi and Sheshadri.<sup>15</sup> The parent coumarin shows lactone carbonyl at  $1705\text{ cm}^{-1}$ ,  $\nu_{\text{C}=\text{C}}$  at  $1608\text{ cm}^{-1}$ ,  $1450\text{ cm}^{-1}$  and  $\nu_{\text{C}-\text{O}-\text{C}}$  at  $1254\text{ cm}^{-1}$ .

### **PMR-Spectra:**

The PMR spectrum of coumarins was reported by Dharmatti et al.<sup>16</sup> The C3- H of coumarin resonates at  $6.45\delta$  ppm and C4-H at  $7.80\delta$  ppm.

### **Mass spectra:**

The electron impact on coumarins has been studied by Baenes et al.<sup>17</sup> The molecular ion peak and fragmentation shows transient formation of Benz furan **10**.

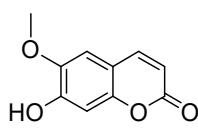


### **Crystal structure:**

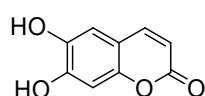
The Crystal structure of coumarin was first reported by S.Ramswamy<sup>18</sup> in 1941. Coumarin crystals are in orthorhombic system, it has space group  $\text{Pca}^z$  with  $Z=4$ . The structure consists of nearly planar molecules held together by Vander Waals forces, x-ray crystallographic data<sup>19</sup> of some coumarins are tabulated below.

<b>Coumarin</b>	<b>Space group. No of molecules Unit cell</b>	<b>Unit cell parameters (<math>\text{\AA}</math>) (<math>^\circ</math>)</b>
Coumarin <sup>20</sup>	Orthorhombic $\text{Pca}^z$ ; $Z=4$	$a=15.46, b=5.67, c=7.91$ $\alpha=\beta=\gamma=90$
4-Hydroxy Coumarin <sup>21</sup>	Orthorhombic $\text{P}2_1\text{2}_1\text{2}_1$ ; $Z=4$	$a=10.11, b=12.18, c=6.95$ $\alpha=\beta=\gamma=90$
7-Hydroxy-4-methyl coumarin <sup>22</sup>	Orthorhombic $\text{P}2_1\text{2}_1\text{2}_1$ ; $Z=4$	$a=10.18, b=12.02, c=6.15$ $\alpha=\beta=\gamma=90$
4-[(4-Fluoro) arylaminomethyl coumarin <sup>23</sup>	Orthorhombic $\text{P}2_1\text{2}_1\text{2}_1$ ; $Z=4$	$a= 5.7973, b=13.9415,$ $c=17.9166$ $\alpha=\beta=\gamma=90$

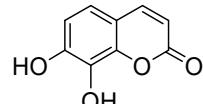
**List of some of the biologically active and naturally occurring coumarins**



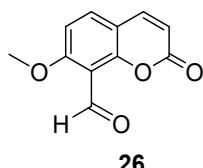
**23**  
common name: **Scopoletin**<sup>24</sup>  
occurrence: barks of wild cherry atropa



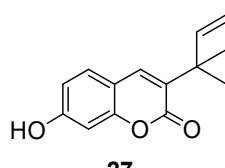
**24**  
common name: **Esculetin**<sup>25</sup>  
occurrence: barks of horse chestnut



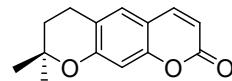
**25**  
common name: **Daphxetin**<sup>26</sup>  
occurrence: extracted from malaphodium divaricatum



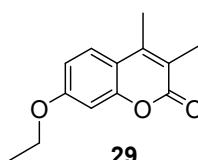
**26**  
common name: **Paniculat**<sup>27</sup>  
occurrence: leaves of M. Exotica and M. Paniculata



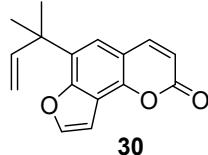
**27**  
common name: **Angustifolin**<sup>28</sup>  
occurrence: extracted from Rata Angustifolia



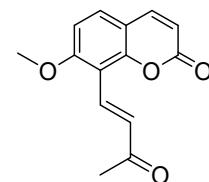
common name:  
**Dyhdroxanthyltin**<sup>29</sup>  
occurrence: extracted from acrial parts of seseli tortuosum



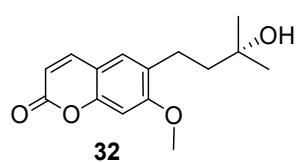
common name:  
**7-Ethoxy-3,4-dimethyl coumarin**<sup>30</sup>  
occurrence: extracted from Edgeworthia Gerdanari



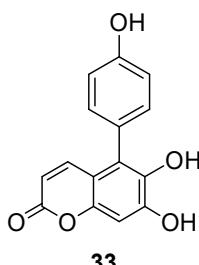
**30**  
common name: **Glycocoumarin**<sup>31</sup>  
occurrence: extracted from the roots of Glycyrhiza Uralensis



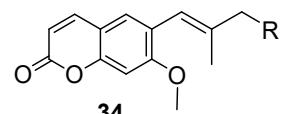
**31**  
common name: **Ostheno**<sup>32</sup>  
occurrence: extracted from M. Exotica



common name: **Dihydrosuberinol**<sup>33</sup>  
occurrence: extracted from the roots of Limonica Acidissima



**33**  
common name: **Seretin**<sup>34</sup>  
occurrence: extracted from the roots of Haplophyllum daurium



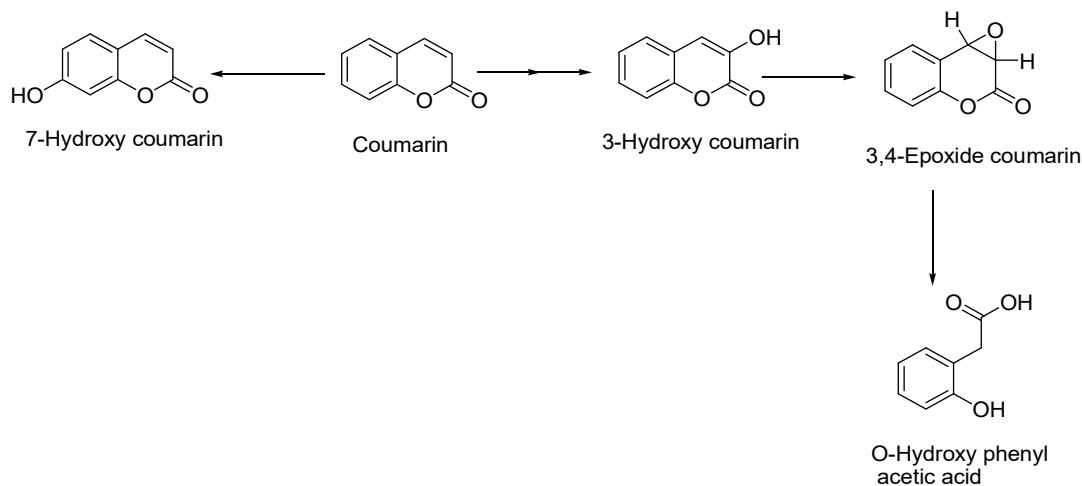
**34**  
common name: **Piloselloidam**<sup>35</sup>  
occurrence: extracted from the roots of Mutisia Spinosa

## Metabolism of coumarin

There are two major pathways involved in coumarin metabolism (Scheme 1). In human body, coumarin is metabolized to 7-hydroxy coumarin via aromatic hydroxylation by cytochrome P450 2A6 gene, which is then excreted as the glucuronide and sulphate-conjugates<sup>36</sup>. In the case of rodents like rats and mice, coumarin undergoes C-3 hydroxylation in the pyran ring and ultimately metabolized to o-hydroxy phenyl acetic acid<sup>37</sup>, via the reactive intermediate of 3, 4-epoxide<sup>38</sup>, which is predicted to be responsible for the

hepatotoxicity caused by coumarin. Thus, the hepatotoxicity of coumarin is dependent upon its species-specific metabolism<sup>39</sup>.

Biochemical studies in mice have shown that coumarin at dose of 100 mg kg<sup>-1</sup> caused a 2 to 15-fold increase in plasma aminotransferases and also subcapsular and centrilobular necrosis in histopathological studies<sup>40</sup>. It has also been observed that coumarin at the dose of 200 mg kg<sup>-1</sup> caused selective clara cell injury in mouse lung<sup>41</sup>, whereas 3,4-dihydro coumarin did not cause any injury at higher dosage (800 mg kg<sup>-1</sup>). These results support the hypothesis that the existence of 3, 4-epoxide intermediate contributes to the observed toxicity.



**Scheme-1**

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# Present Work

## Synthesis and Characterization of Schiff Bases Derived from 3-Acetyl Coumarins and 2,4-Dinitrophenylhydrazine

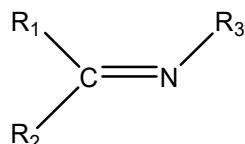


## 2. PRESENT WORK

### 2.1 INTRODUCTION

Heterocyclic compounds have garnered considerable interest due to their extensive potential applications. Coumarins constitute an important class of oxygen-containing heterocycles found in various plant sources in the form of benzopyrone derivatives. This naturally occurring lactone group was first isolated from tonka beans in 1820. They contain the coumarin nucleus ( $2H$ -1-benzopyran-2-one) and are important in natural products and synthetic organic chemistry [1,2]. Compounds containing the coumarin moiety and its derivatives have been associated with various biological applications such as antibacterial, antimicrobial, anti-inflammatory, antiviral, antioxidant, antimutagenic, anticancer, antibiotic, anti-HIV, etc. [3,4]. It has been found that naturally occurring antibiotics such as novobiocin and clorobiocin contain the 3-aminocoumarin moiety [5,6]. Because of their low toxicity, exceptional photostability, good solubility, ease of preparation and high fluorescence quantum yield, coumarin and its derivatives are also extensively used in fluorescent probes, laser dyes, optical materials and other fields [7,8].

Schiff bases, named after Hugo Schiff [9], are formed when any primary amine reacts with an aldehyde or a ketone under specific conditions. Structurally, a Schiff base (also known as imine or azomethine) (**Fig. 1**) is a nitrogen analogue of an aldehyde or ketone in which the carbonyl group (C,O) has been replaced by an imine or azomethine group.



R<sub>1</sub>, R<sub>2</sub>, and/or R<sub>3</sub> = alkyl or aryl

**Fig. 1** General structure of a Schiff base

Schiff bases are some of the most widely used organic compounds. They are used as pigments and dyes, catalysts, intermediates in organic synthesis, and as polymer stabilisers [10]. Schiff bases have also been shown to exhibit a broad range of biological activities, including antifungal, antibacterial, antimalarial, antiproliferative, anti-inflammatory, antiviral, and antipyretic properties [10,11]. Imine or azomethine groups are present in various natural, natural-derived, and non-natural compounds. The imine group present in such compounds has been shown to be critical to their biological activities [12-14].

### Synthesis of Schiff bases

The first preparation of imines was reported in the 19th century by Schiff (1864).

Since then a variety of methods for the synthesis of imines have been described [15]. The classical synthesis reported by Schiff involves the condensation of a carbonyl compound with an amine under azeotropic distillation [16]. Molecular sieves are then used to completely remove water formed in the system [17]. In the 1990s an in situ method for water elimination was developed, using dehydrating solvents such as tetramethyl orthosilicate or trimethyl orthoformate [18,19]. In 2004, Chakraborti et al. [20] demonstrated that the efficiency of these methods is dependent on the use of highly electrophilic carbonyl compounds and strongly nucleophilic amines. They proposed as an alternative the use of substances that function as Brønsted-Lowry or Lewis acids to activate the carbonyl group of aldehydes, catalyze the nucleophilic attack by amines, and dehydrate the system, eliminating water as the final step [20]. Examples of Brønsted-Lowry or Lewis acids used for the synthesis of Schiff bases include  $ZnCl_2$ ,  $TiCl_4$ ,  $MgSO_4$ -PPTS,  $Ti(OR)_4$ , alumina,  $H_2SO_4$ ,  $NaHCO_3$ ,  $MgSO_4$ ,  $Mg(ClO_4)_2$ ,  $H_3CCOOH$ ,  $Er(OTf)_3$ ,  $P_2O_5/Al_2O_3$ ,  $HCl$  [20–32]. In the past 12 years a number of innovations and new techniques have been reported, including solvent-free/clay/microwave irradiation, solid-state synthesis, K-10/microwave, water suspension medium,  $[bmim]BF_4$ /molecular sieves, infrared irradiation/no solvent,  $NaHSO_4.SiO_2$ /microwave/solventfree, solvent-free/ $CaO$ /microwave, and silica/ultrasound irradiation.

The work to be described in this chapter involves the synthesis of various Schiff Bases Derived from 3-Acetyl Coumarins and 2,4-Dinitrophenylhydrazine. Coumarin analogues such as 3-acetylcoumarin display improved biological properties [33] is a key pharmacophore in many pharmaceutical agents.[34] It is also used for the synthesis of a variety of natural products.[35] Linkage of various alkyl or aryl moieties at any position of coumarins has resulted in novel molecular matrices which were associated with anti-microbial and anti-inflammatory agents has been reported.

Scientists have recently directed their efforts into using it as a starting material for the synthesis of heterocyclic scaffolds such as oxazole; pyrazole; thiophene; thiazole; pyridine; diazepine; benzoxocin; benzoxepin; and pyrimidine derivatives. The coumarin-Schiff bases are synthesized via different synthetic pathways and also applied as a starting material for preparation more complex structures with variety of biological effect.

In view of this, a brief account of literature on the structure and biological activities of coumarinyl ethers is presented below.

3-Acetylcoumarin has been employed as a starting material in the synthesis of numerous scaffolds. The 3-acetylcoumarin moiety is involved (36) (either as a reagent,

product or substructure) are experiencing a huge upward trend.

The present investigation was aimed at synthesizing the conjugates of various 3-acetyl coumarins with 2,4-dinitrophenyl hydrazine's in the form of Schiff bases.

## 2.2. Methods

### 2.2.1. Materials and tools

The ingredients in this research are salicylaldehyde, ethylacetacetate, piperidine, Chlorobenzene, Hydrazine Hydrate, Glacial acetic acid, Methanol, n-hexane p.a, ethyl acetate p.a, technical nhexane, technical ethylacetate,. The laboratory equipment in this research are reflux devices, glassware, Whatman filter paper, evaporator, analytical balance, thermometer, thin layer chromatography (TLC) plates, Fourier Transform Infrared (FTIR).

### 2.2.2. Present work

Synthesis of 3-acetylcoumarins (**3a-3c**) were brought about by the knoevenagel condensation of salicylaldehydes with ethylacetacetate (Scheme 1). 2,4-Dinitrophenylhydrazine (**6**) was synthesized as per our B. Sc. 6<sup>th</sup> semester organic lab manual (Scheme 2) by using Chlorobenzene (**4**). The substituted Schiff Bases (**7a-7c**) Derived from 3-Acetyl Coumarins and 2,4-Dinitrophenylhydrazine were synthesized on water bath by refluxing the reaction mixture of substituted 3-acetylcoumarins (**3a-3c**) and 2,4-Dinitrophenylhydrazine (**6**) in the presence of catalytic amount of glacial acetic acid in methanol (Scheme 3).

All the products gave satisfactory analytical and spectroscopic data, which are in full accordance with their assigned structures.

### 2.2.3. Experimental

This section deals with the preparation of following compounds.

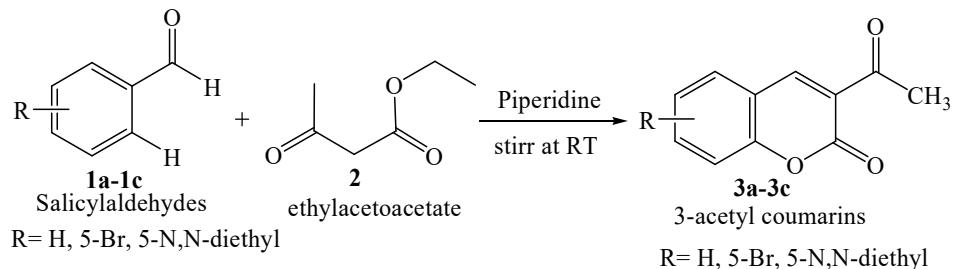
1. Preparation of 3-acetyl coumarins (**3a-3c**).
2. Preparation of 2,4-Dinitrophenylhydrazine (**6**).
3. Preparation of substituted Schiff Bases (**7a-7c**) Derived from 3-Acetyl Coumarins and 2,4-Dinitrophenylhydrazine

#### 1. Preparation of 3-acetyl coumarins (**3a-3c**):

Synthesis of 3-acetylcoumarins was done by reacting (0.08 mol, 8.73 mL) salicylaldehydes, (0.09 mol, 12.52 mL) ethylacetacetate, and (0.5 mL) piperidine and stirred at room temperature for 30 minutes. Then, the reaction was monitored using thin layer chromatography plates (TLC plates) with the solvent system (ethyl acetate: n-hexane 1:3). Then, the mixture was extracted with ethyl acetate. The ethyl acetate fraction was added with

anhydrous  $\text{Na}_2\text{SO}_4$  then evaporated until the solvents run out [37, 38].

When the reaction was monitored, a dominant spot was formed on the TLC plate. Product from this reaction has a yellow solid in about 14.78 g and 95.97% yield. It has a melting point range about 112- 115°C which is an agreement with the literature value.



**Scheme-1:** Synthesis of substituted 3-acetyl coumarins (3a-3c)

## 2. Preparation of 2,4-Dinitrophenylhydrazine (6):

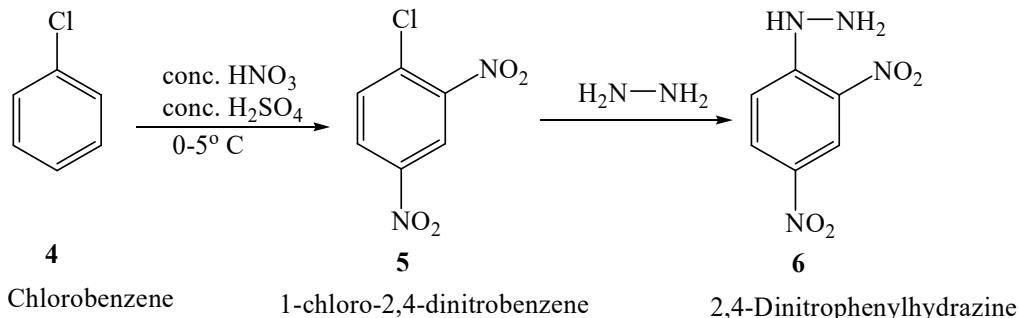
### Step-I: Preparation of 1-chloro-2,4-dinitrobenzene(5):

4.0 g of chlorobenzene are added drop by drop to a mixture of 6.4 g of nitric acid (d=1.50 g/ml) and 13.6 g of sulfuric acid (d=1.84 g/ml) while the mixture is stirred mechanically. The temperature rises because of the heat of the reaction, but is not allowed to go above 50-55° C. After all the chlorobenzene has been added, the temperature is raised slowly to 95° C and is kept there for 2 hours longer while the stirring is continued. The upper layer of light yellow liquid solidifies when cold. It is removed, broken up under water, and rinsed. The spent acid, on dilution with water, precipitates an additional quantity of 1-chloro2,4-dinitrobenzene. All the product is brought together, washed with cold water, then several times with hot water while it is melted, and finally once more with coldwater under which it is crushed. Then it is drained and allowed to dry at ordinary temperature. The product, melting at about 50° C, consists largely of 1-chloro-2,4-dinitrobenzene, m.p. 53.4° C, along with a small quantity of the 2,6-dinitro compound, m.p. 87-88° C.

### Step-II: Preparation of 2, 4-Dinitrophenylhydrazine (6):

3.5 g. of hydrazine sulfate is suspended in 15 cc. of hot water in a 400-cc. beaker and stirred by hand during the addition of 8.5 g. of potassium/Sodium acetate. The mixture is boiled five minutes and then cooled to about 70°; 7.5 cc. of alcohol is added, and the solid is filtered with suction and washed with 10 cc. of hot alcohol. The filtered hydrazine solution is saved for the next step. In a 1-l. flask fitted with a stirrer and reflux condenser, 5.05 g. of technical 2,4-dinitrochlorobenzene is dissolved in 15 cc. of alcohol; the hydrazine solution is added, and the mixture is refluxed with stirring for an hour. Most of the product separates during the first ten minutes; it is cooled well, filtered, and washed, once with 5 cc. of warm

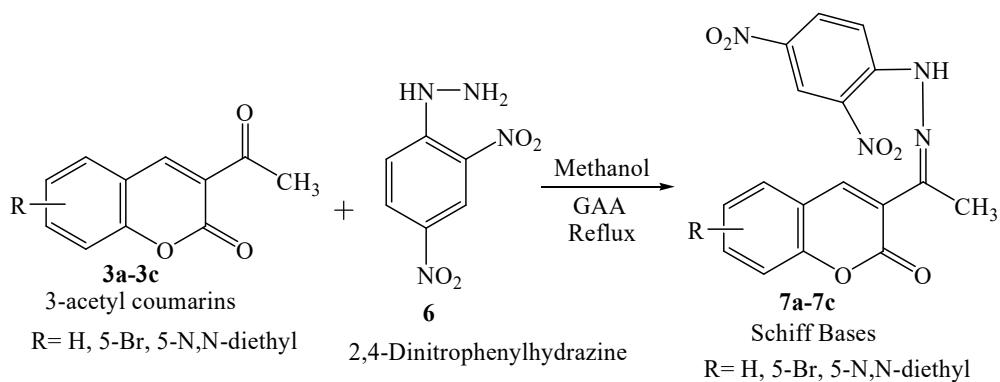
alcohol (60°) to remove unchanged halide and then with 10 cc. of hot water. The solid weighs 3 g. and melts at 190–192° with evolution of gas; it is pure enough for most purposes. By distilling half the alcohol from the filtrate a less pure second crop is obtained; this is recrystallized from n-butyl alcohol (10 cc. per g.).



**Scheme-2:** Synthesis of 2,4-Dinitrophenylhydrazine (6)

### 3. Preparation of substituted Schiff Bases (7a-7c) Derived from 3-Acetyl Coumarins and 2,4-Dinitrophenylhydrazine:

The synthesis of the Schiff base of 3-acetyl coumarins (7a-7c) were started by adding 5 mmol of 2,4-Dinitrophenylhydrazine (6) to the solution of 3-acetyl coumarins (3a-3c) (5 mmol) and methanol (10 mL) by adding catalytic amount of glacial acetic acid. The mixture was continuously stirred and then refluxed for 4-12 h at 75– 80 °C. The colored precipitates were filtered, dried, and recrystallized from DMF. The preparation of this compound was carried out lined in Scheme (3) and its physical properties as shown in table (1). Including melting point (powder) and around 75 % yield were obtained and these compound were identified by FT-IR Spectroscopy.

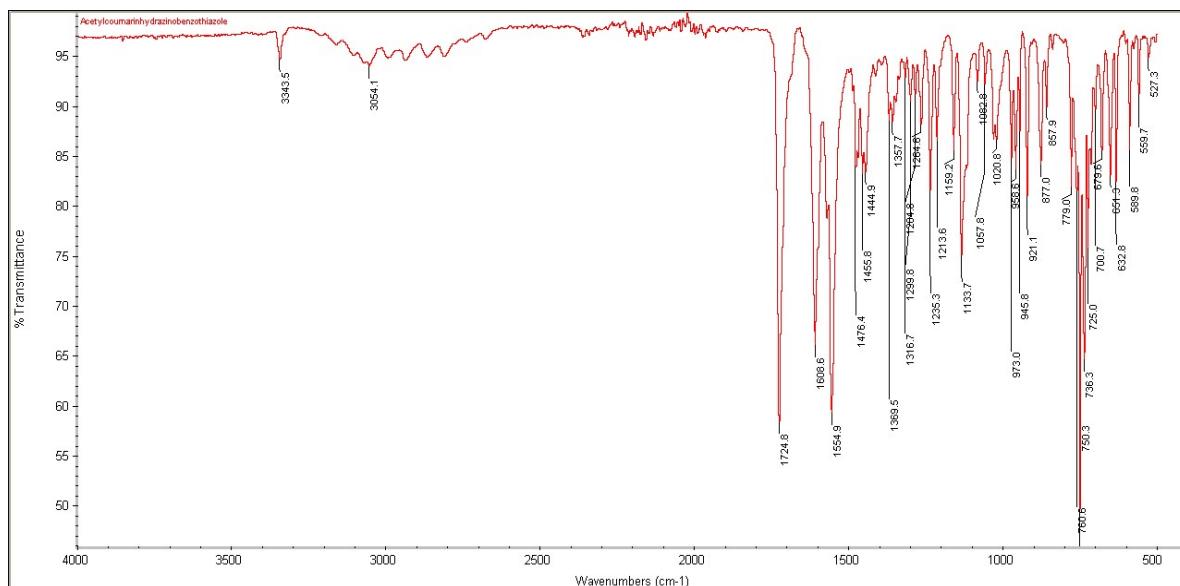


**Scheme-3:** Synthesis of substituted Schiff Bases (**7a-7c**) Derived from 3-Acetyl Coumarins and 2,4-Dinitrophenylhydrazine

## 2.2.4. Results and discussion

### Infrared spectral Studies

The IR spectrum of Schiff Base **7a** [Spectrum No.1] exhibited the lactone carbonyl stretching band around  $1724\text{ cm}^{-1}$  and amino group showed the band around  $3343\text{ cm}^{-1}$ . The C=N stretching and C-O-C stretching frequency bands were observed around  $1555\text{ cm}^{-1}$  and  $1235\text{ cm}^{-1}$  respectively. The IR spectral data of all the compounds are given below.



**Spectrum-1:** FTIR spectrum of 3-acetylcoumarin Schiff base **7a**

The physical constants and analytical data of synthesized compounds (**7a-7c**) have been given below.

#### i. Schiff Base **7a**:

**Molecular Formula :**  $\text{C}_{17}\text{H}_{12}\text{N}_4\text{O}_6$

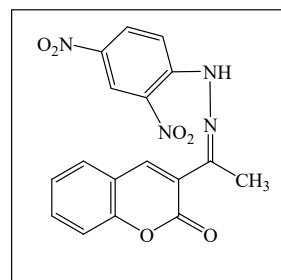
**Physical State** : Yellow coloured solid

**mp[°C]** : 214-216

**GC-MS (*m/z*)** : 368

**IR (KBr, cm<sup>-1</sup>)** :  $(1724)\text{cm}^{-1}, (3343)\text{cm}^{-1}, (1555)\text{cm}^{-1}, (1235)\text{cm}^{-1}$ , due to

$\nu(\text{C}=\text{O})\text{str}$ ,  $\nu(\text{NH}_2)$ ,  $\nu(\text{C}=\text{N})$  aliphatic,  $\nu(\text{COC})$



**Elemental analysis for  $\text{C}_{17}\text{H}_{12}\text{N}_4\text{O}_6$  :** Calcd for - C, 55.44; H, 3.28; N, 15.21; O, 26.06 ;

**found** – C, 55.38; H, 3.25; N, 15.16; O, 25.98.

**ii. Schiff Base 7b:**

**Molecular Formula :** C<sub>17</sub>H<sub>11</sub>BrN<sub>4</sub>O<sub>6</sub>

**Physical State** : Orange colored solid

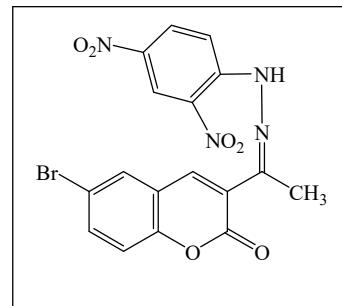
**mp[°C]** : 170-172

**GC-MS (m/z)** : 446

**IR (KBr, cm<sup>-1</sup>)** : (1720)cm<sup>-1</sup>, (3348)cm<sup>-1</sup>, (1548)cm<sup>-1</sup>, (1230)cm<sup>-1</sup>, due to

$\nu$ (C=O)str,  $\nu$ (NH2),  $\nu$ (C=N) aliphatic,  $\nu$ (COC)

**Elemental analysis for C<sub>17</sub>H<sub>11</sub>BrN<sub>4</sub>O<sub>6</sub> :** Calcd for - C, 45.66; H, 2.48; Br, 17.87; N, 12.53; O, 21.47 ; **found** – C, 45.62; H, 2.41; Br, 17.83; N, 12.48; O, 21.42.



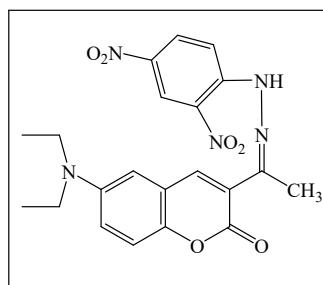
**iii. Schiff Base 7c:**

**Molecular Formula :** C<sub>21</sub>H<sub>21</sub>N<sub>5</sub>O<sub>6</sub>

**Physical State** : Brown colored solid

**mp[°C]** : 200-202

**GC-MS (m/z)** : 439



**IR (KBr, cm<sup>-1</sup>)** : (1718)cm<sup>-1</sup>, (3338)cm<sup>-1</sup>, (1550)cm<sup>-1</sup>, (1239)cm<sup>-1</sup>, due to

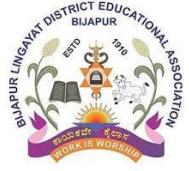
$\nu$ (C=O)str,  $\nu$ (NH2),  $\nu$ (C=N) aliphatic,  $\nu$ (COC)

**Elemental analysis for C<sub>21</sub>H<sub>21</sub>N<sub>5</sub>O<sub>6</sub> :** Calcd for - C, 55.44; H, 3.28; N, 15.21; O, 26.06 ; **found** – C, 55.32; H, 3.20; N, 15.11; O, 25.94.

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**B. L. D. E. Association's**  
**S. B. Arts and K. C. P. Science College,**  
**Vijayapur-586103**

**DEPARTMENT OF CHEMISTRY**

**Project on**  
**Synthesis and Characterization of Ethers**  
**Derived from 2-bromoacetyl Benzenes and**  
**7-hydroxy-4-methyl Coumarin**

**By**

**Name: DANESHWARI M GUGGARI**  
**RCU No: U15KM21S0422**

**Submitted To**  
**Dr. K. Mahesh Kumar**  
**2023-24**

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*Secondly I would like to thank my parents and classmates who helped me to complete this project within the given time frame.*

**DANESHWARI**



## **TITLE OF THE PROJECT**

**Synthesis and Characterization of Ethers Derived  
from 2-bromoacetyl Benzenes and 7-hydroxy-4-  
methyl Coumarin**



**B. L. D. E. ASSOCIATION'S**  
**S. B. ARTS AND K. C. P. SCIENCE COLLEGE, VIJAYAPUR**  
**DEPARTMENT OF CHEMISTRY**



Date:

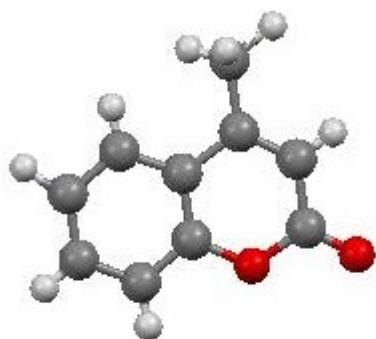
## **CERTIFICATE**

This is to certify that, **Miss Daneshwari M Guggari (U15KM21S0422)**  
studying in B. Sc. VI semester during the year 2023-24, has completed the  
project entitled "**Synthesis and Characterization of Ethers Derived from 2-bromoacetyl Benzenes and 7-hydroxy-4-methyl Coumarin**". This Project  
work is in partial fulfillment for the award of degree of Bachelor of Science.  
The project work satisfies the requirements prescribed in the curriculum of "  
**Rani Channamma University, Belagavi**".

**Guide of the Project**

**Head of the Department**

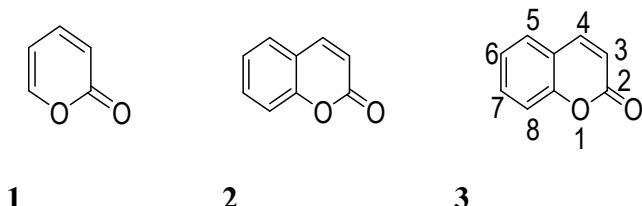
# Introduction to the Chemistry of Coumarins



## 1. CHEMISTRY OF COUMARINS

### 1.1 INTRODUCTION

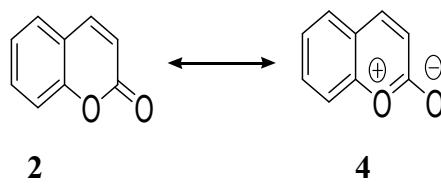
Coumarins are an important class of oxygen heterocycles, which are widespread in plant kingdom and have been extensively reported on. Their chemical structure can be looked upon as arising out of the fusion of a benzene ring to pyran-2-one **1**, across the 5 and 6 positions in skeleton.



The parent coumarin **2** was first isolated by Vogel in 19<sup>th</sup> century from Tonka beans<sup>1</sup> and even to this date finds itself still in use as perfumery and flavoring agent. Figure **3** represent the numbering system used in coumarin skeleton<sup>2</sup>.

### Structure and reactivity

Aromatic nature of heterocyclic ring of coumarin is disputable, because coumarin shows some reactions of aliphatic compounds and other characteristics of aromatic compounds. The complete aromaticity in coumarin can be only realized if O-CO function contributes two electrons to form 10 $\pi$  electron system. This means that coumarin should be a resonance hybrid, to which contribution from canonical form **4** is significant. However, no evidence is found in the spectra of coumarin to suggest that contribution from betaine form **4** is considerable

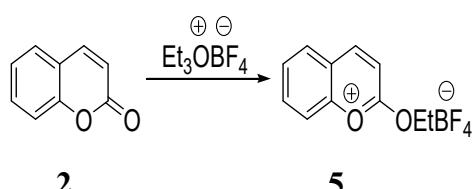


The infrared absorption spectrum of coumarin shows an absorption band at 1710  $\text{cm}^{-1}$  which is attributed to lactone carbonyl group but not a betain from. In the  $^1\text{H}$  NMR spectrum of coumarin<sup>3</sup>, the signal due to protons of C3 and C4 appears in the region of 6.45  $\delta$  ppm and 7.80  $\delta$  ppm with coupling constants of 9.8Hz. These values are typical of *cis* alkene rather than an aryl ring<sup>4</sup>. Finally the  $^{13}\text{C}$  NMR spectra of coumarins<sup>5</sup> are consistant with an essentially aliphatic heterocyclic ring. The chemical shifts of C2, C3 and C4 in coumarin remarkably close to the values for the corresponding carbons in  $\alpha$ -pyrone and are given below

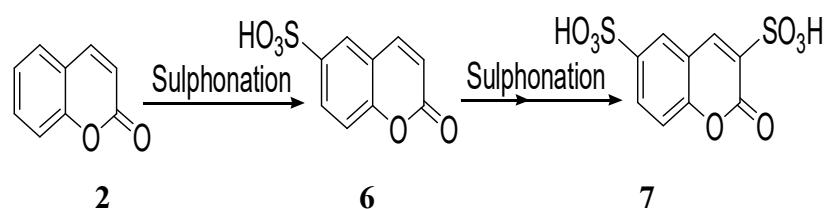
Compound	C2	C3	C4
$\alpha$ -Pyrone	162.0	116.7	144.3
Coumarin	160.4	116.4	143.4

But coumarin does show aromatic character in its pattern of reactivity, e.g.,

The carbonyl oxygen can be alkylated<sup>6</sup> by powerful agents to give stable pyrillium salts **5**.

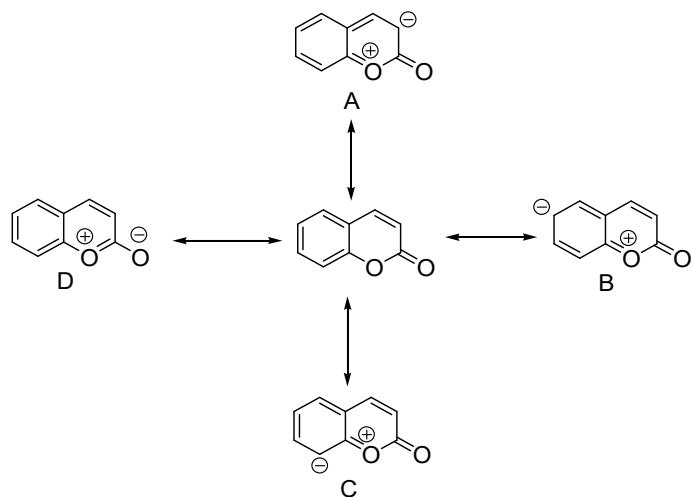


Coumarin nucleus is susceptible to electrophilic substitution<sup>6</sup>. Sulphonation takes place initially in the carbocyclic ring at C6, to give **6**, but under more forcing conditions one more  $-\text{SO}_3\text{H}$  group can be introduced at C3, to obtain coumarin-3, 6-disulphonic acid **7**.

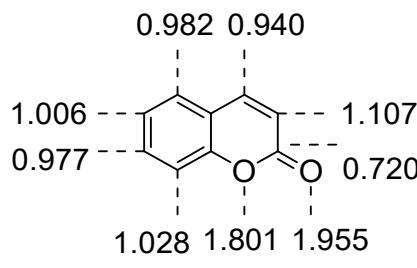


As in case of simple pyrones the properties of heterocyclic ring of coumarin are greatly influenced by the presence of substituents.

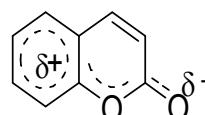
Anantatakrishnan<sup>7</sup> discussed the “Mills-Nixon effect” in which the reactivity of coumarin was rationalized based on the comparative studies of bromination and nitration of coumarin, naphthalene and benzene. By considering the possible electron movements in coumarin molecule, Thakur and Shah<sup>8</sup> predicted that C6 and C8 as the most reactive centres. The electron movements are as shown below.



Greater electron densities can be seen on C6 and C8 from the resonating structures B and C. Out of these two, C6 seems to be more reactive because of its proximity to the oxygen atom, similar to the reactivity of para position of phenol. Structure A though imparts more electron density to the C3 position, the electrophilic substitution at C3 is less, probable due to its closeness to the electron withdrawing carbonyl group. Infact the  $\pi$  electron densities calculated by Song and Gorden<sup>9</sup> are quite close to the resonance picture of the molecule. The structure **8** represents the  $\pi$  electron densities for the ground state of coumarin.



By considering the structure's B, C and D Bassingnan and Cogrossi<sup>10</sup> have proposed structure **9** which is according to them represents the hybrid or resonating state of molecule.



However the contributing structure of the type (D) does not have strong spectral evidences, the position of the carbonyl frequency in the IR spectrum ( $1710\text{ cm}^{-1}$ ) is more in favor of an enol lactone<sup>11</sup>. Hence the contribution from such type of structures is negligible and the resonating state **9** appears to be less probable.

Coumarin has been used as a powerful model in elucidating the electronic structures and photo reactivity of psoralenes. The configurational analysis of coumarin by Song et al.<sup>12</sup> in the ground state indicates some charge transfer delocalization extending to the ethylenic region. The dipole movements of coumarin ( $4.82 \times 10^{-8}\text{ e.s.u}$ ) determined earlier by Rao<sup>13</sup> also indicates the similar delocalization.

## Spectral studies

### UV-Spectra:

The UV spectra of coumarins and their methyl derivatives were reported by Ganguly and Bagchi.<sup>14</sup> The introduction of methyl group in various positions does not change the nature of the spectrum to a greater extant. The  $\lambda_{\text{max}}$  and  $\epsilon$  values of coumarins are 273 nm (40,368) & 309 nm (37,449).

### IR-Spectra:

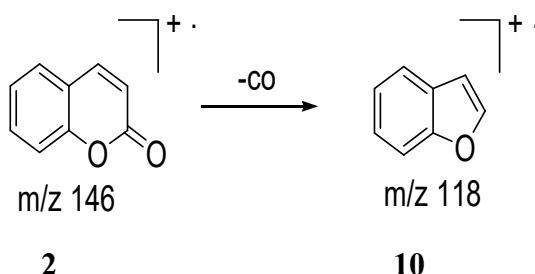
The IR spectrum of coumarin was reported by Murthi and Sheshadri.<sup>15</sup> The parent coumarin shows lactone carbonyl at  $1705\text{ cm}^{-1}$ ,  $\nu_{\text{C}=\text{C}}$  at  $1608\text{ cm}^{-1}$ ,  $1450\text{ cm}^{-1}$  and  $\nu_{\text{C}-\text{O}-\text{C}}$  at  $1254\text{ cm}^{-1}$ .

### PMR-Spectra:

The PMR spectrum of coumarins was reported by Dharmatti et al.<sup>16</sup> The C3- H of coumarin resonates at  $6.45\delta$  ppm and C4-H at  $7.80\delta$  ppm.

### Mass spectra:

The electron impact on coumarins has been studied by Baenes et al.<sup>17</sup> The molecular ion peak and fragmentation shows transient formation of Benz furan **10**.

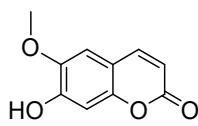


### Crystal structure:

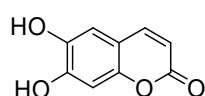
The Crystal structure of coumarin was first reported by S.Ramswamy<sup>18</sup> in 1941. Coumarin crystals are in orthorhombic system, it has space group  $\text{Pca}^z$  with  $Z=4$ . The structure consists of nearly planar molecules held together by Vander Waals forces, x-ray crystallographic data<sup>19</sup> of some coumarins are tabulated below.

Coumarin	Space group. No of molecules Unit cell	Unit cell parameters ( $\text{\AA}$ ) ( $^\circ$ )
Coumarin <sup>20</sup>	Orthorhombic $\text{Pca}^z$ ; $Z=4$	$a=15.46, b=5.67, c=7.91$ $\alpha=\beta=\gamma=90$
4-Hydroxy Coumarin <sup>21</sup>	Orthorhombic $\text{P}2_1\text{2}_1\text{2}_1$ ; $Z=4$	$a=10.11, b=12.18, c=6.95$ $\alpha=\beta=\gamma=90$
7-Hydroxy-4-methyl coumarin <sup>22</sup>	Orthorhombic $\text{P}2_1\text{2}_1\text{2}_1$ ; $Z=4$	$a=10.18, b=12.02, c=6.15$ $\alpha=\beta=\gamma=90$
4-[(4-Fluoro) arylaminomethyl coumarin <sup>23</sup>	Orthorhombic $\text{P}2_1\text{2}_1\text{2}_1$ ; $Z=4$	$a=5.7973, b=13.9415,$ $c=17.9166$ $\alpha=\beta=\gamma=90$

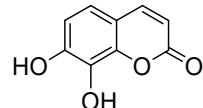
**List of some of the biologically active and naturally occurring coumarins**



**23**  
common name: **Scopoletin**<sup>24</sup>  
occurrence: barks of wild cherry atropa



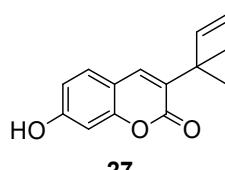
**24**  
common name: **Esculetin**<sup>25</sup>  
occurrence: barks of horse chestnut



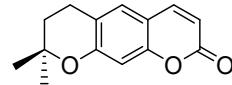
**25**  
common name: **Daphxetin**<sup>26</sup>  
occurrence: extracted from malaphodium divaricatum



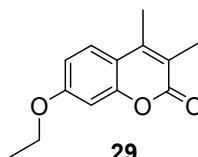
**26**  
common name: **Paniculat**<sup>27</sup>  
occurrence: leaves of M. Exotica and M. Paniculata



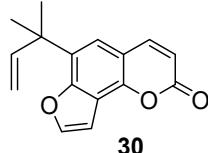
**27**  
common name: **Angustifolin**<sup>28</sup>  
occurrence: extracted from Rata Angustifolia



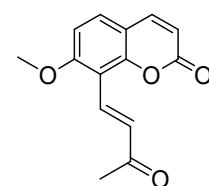
**28**  
common name: **Dyhdroxanthyltin**<sup>29</sup>  
occurrence: extracted from acrial parts of seseli tortuosum



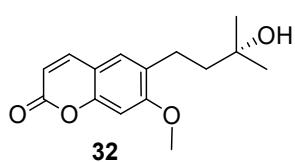
**29**  
common name:  
**7-Ethoxy-3,4-dimethyl coumarin**<sup>30</sup>  
occurrence: extracted from Edgeworthia Gerdanari



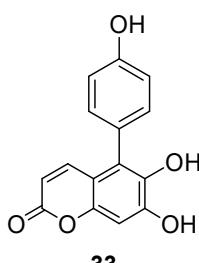
**30**  
common name: **Glycocooumarin**<sup>31</sup>  
occurrence: extracted from the roots of Glycyrhiza Uralensis



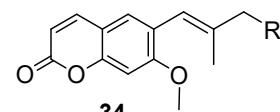
**31**  
common name: **Ostheno**<sup>32</sup>  
occurrence: extracted from M. Exotica



**32**  
common name: **Dihydrosuberinol**<sup>33</sup>  
occurrence: extracted from the roots of Limonica Aciddissima



**33**  
common name: **Seretin**<sup>34</sup>  
occurrence: extracted from the roots of Haplophyllum daurium



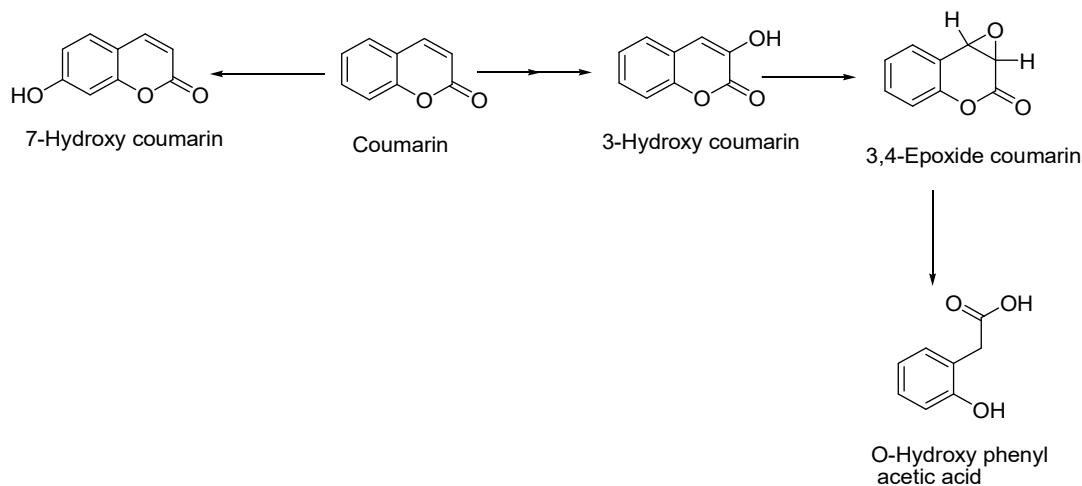
**34**  
common name: **Piloselloidam**<sup>35</sup>  
occurrence: extracted from the roots of Mutisia Spinosa

## Metabolism of coumarin

There are two major pathways involved in coumarin metabolism (Scheme 1). In human body, coumarin is metabolized to 7-hydroxy coumarin via aromatic hydroxylation by cytochrome P450 2A6 gene, which is then excreted as the glucuronide and sulphate-conjugates<sup>36</sup>. In the case of rodents like rats and mice, coumarin undergoes C-3 hydroxylation in the pyran ring and ultimately metabolized to o-hydroxy phenyl acetic acid<sup>37</sup>, via the reactive intermediate of 3, 4-epoxide<sup>38</sup>, which is predicted to be responsible for the

hepatotoxicity caused by coumarin. Thus, the hepatotoxicity of coumarin is dependent upon its species-specific metabolism<sup>39</sup>.

Biochemical studies in mice have shown that coumarin at dose of 100 mg kg<sup>-1</sup> caused a 2 to 15-fold increase in plasma aminotransferases and also subcapsular and centrilobular necrosis in histopathological studies<sup>40</sup>. It has also been observed that coumarin at the dose of 200 mg kg<sup>-1</sup> caused selective clara cell injury in mouse lung<sup>41</sup>, whereas 3,4-dihydro coumarin did not cause any injury at higher dosage (800 mg kg<sup>-1</sup>). These results support the hypothesis that the existence of 3, 4-epoxide intermediate contributes to the observed toxicity.



**Scheme-1**

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# Present Work

## Synthesis and Characterization of Ethers Derived from 2-bromoacetyl Benzenes and 7-hydroxy-4-methyl Coumarins

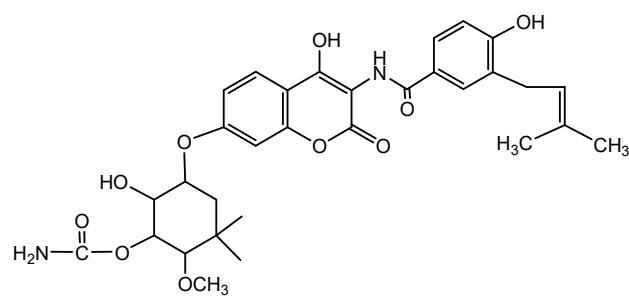


## 2. PRESENT WORK

### 2.1 INTRODUCTION

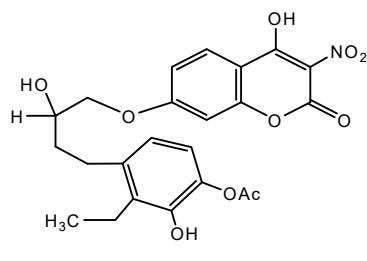
Heterocyclic compounds have garnered considerable interest due to their extensive potential applications. Coumarins constitute an important class of oxygen-containing heterocycles found in various plant sources in the form of benzopyrone derivatives. This naturally occurring lactone group was first isolated from tonka beans in 1820. They contain the coumarin nucleus ( $2H$ -1-benzopyran-2-one) and are important in natural products and synthetic organic chemistry [1,2]. Compounds containing the coumarin moiety and its derivatives have been associated with various biological applications such as antibacterial, antimicrobial, anti-inflammatory, antiviral, antioxidant, antimutagenic, anticancer, antibiotic, anti-HIV, etc. [3,4]. It has been found that naturally occurring antibiotics such as novobiocin and clorobiocin contain the 3-aminocoumarin moiety [5,6]. Because of their low toxicity, exceptional photostability, good solubility, ease of preparation and high fluorescence quantum yield, coumarin and its derivatives are also extensively used in fluorescent probes, laser dyes, optical materials and other fields [7,8].

The discovery of Novobiocin<sup>1</sup> **1** [9] with O-glycosidic linkage enhanced the importance of coumarin in the field of antibiotics.



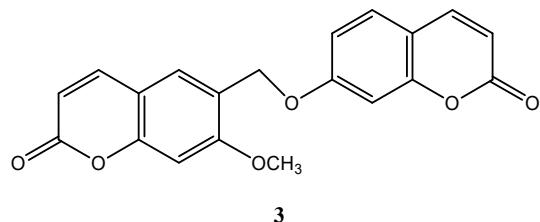
**1**

Coumarin derivatives containing aryloxy groups are found to be anti-histamines and also antagonize the effects of slow reacting substances of anaphylaxis. The most active compound in the series **2** is reported by Buckle *et.al*<sup>2</sup>[10].

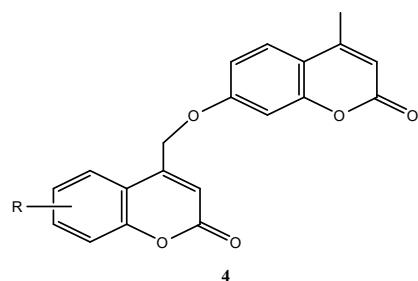


**2**

Bis-coumarinyl ether **3** has been isolated from *Lasiocphon eriocephalus*.<sup>8</sup>[11]

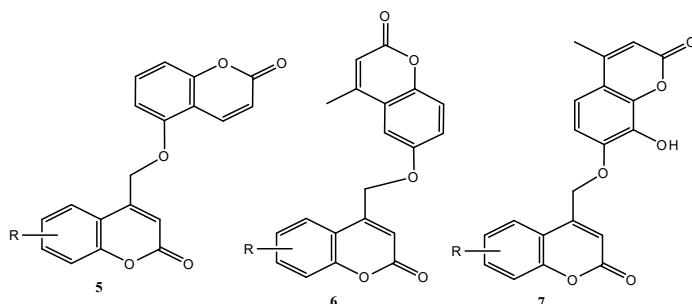


Several new bis-coumarinyl ethers which are higher homologues and regioisomers of Lasioccephalin **4** have been synthesised and their anti-microbial activity against five microorganisms has been reported<sup>15</sup>[12].



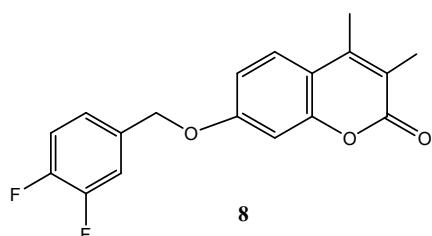
$R = H, 6-CH_3, 7-CH_3, 7-OCH_3, 5,6\text{-benzo}, 7,8\text{-benzo}$

Sheelavantar *et. al*<sup>17</sup>[13] synthesized some new bis-coumarinyl ethers **5**, **6** and **7** as analogues of Lasioccephalin and their anti-microbial activity was reported.

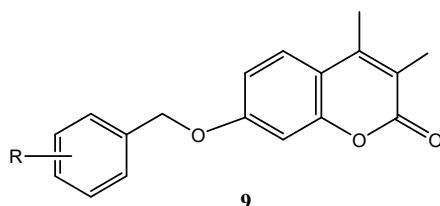


$R = 6-CH_3, 7-CH_3, 6-OCH_3, 5,6\text{-benzo}, 7,8\text{-benzo}, 6-Cl$

A large series of coumarin derivatives compounds were tested for their monoamine oxidase A and B (MAO-A and MAO-B) inhibitory activity. Most of the compounds acted preferentially on MAO-B with  $IC_{50}$  values in the micromolar to low-nanomolar range; high inhibitory activities towards MAO-A were also measured for sulfonic acid esters. The most active compound was 7-[(3,4-difluorobenzyl)oxy]-3,4-dimethylcoumarin **8**, with an  $IC_{50}$  value toward MAO-B OF 1.14 Nm. CoMFA was also performed on two data sets of MAO-A and MAO-B inhibitors [14].<sup>24</sup>

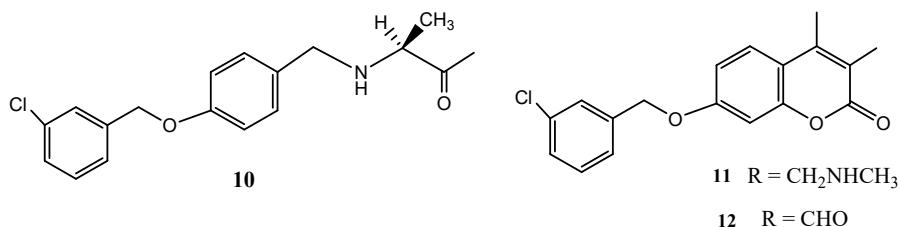


Novaroli *et al.*<sup>25</sup>[15] studied the impact of species-dependent differences between human and rat MAO-B on inhibitor screening was evidenced for coumarin derivatives **9**. All examined compounds have shown greater inhibitor potency toward human MAO-B than toward rat MAO-B. Moreover, no correlation was found between human and rat PIC<sub>50</sub> values.

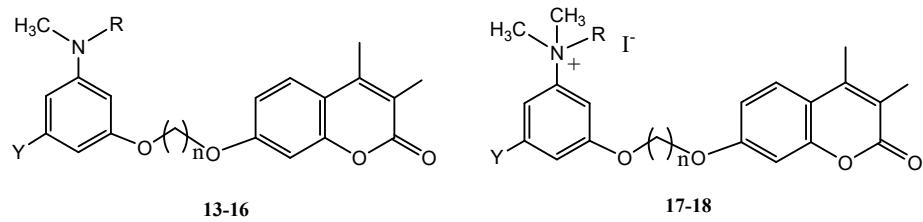


R = H, CN, CH<sub>3</sub>, OCH<sub>3</sub>, OCF<sub>3</sub>, NHCOCH<sub>3</sub>, F, Cl, CF<sub>3</sub>, NO<sub>2</sub>, Pentafluoro

Structures of human mono amine oxidase B (MAO-B) in complex with safinamide **10** and two coumarin derivatives **11** and **12**, all sharing common benzyloxy substituent, were determined by X-ray crystallography. These compounds competitively inhibit MAO-B occupying both the entrance and the substrate cavities and showing a similarly oriented benzyloxy substituent.<sup>26</sup>[16]



A number of coumarin ethers **13-16** and their salts **17-18** have been reported as highly potent, dual binding site AChE inhibitors<sup>27</sup>.[17]



13, n = 3, R = CH<sub>3</sub>

14, n = 4, R = CH<sub>3</sub>

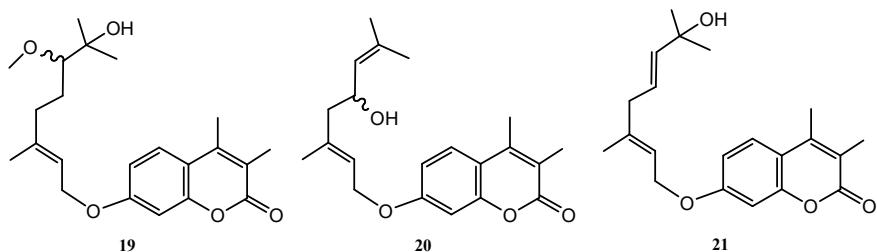
$$15, \quad n = 3, R = Bn$$

$$16, \quad n = 4, R = Bn$$

17, n = 3, R = CH<sub>3</sub>

**18**, n = 4, R = CH<sub>3</sub>

Ma et.al<sup>28</sup>[18] reported the isolation and characterization of new coumarin derivatives (**19-21**) from *Notopterygium forbessi* and evaluated for binding affinities to the opioid and dopamine receptors.



In view of the hitherto discussion on the importance of aryl ether linkage at various positions in the coumarin moiety, it was contemplated to utilize unsubstituted, nitro and bromo substituted phenols to generate a molecular library of resulted 4-aryloxymethylcoumarins and comparative study of their antimicrobial activities.

## Synthesis of Coumarin Ethers

The work to be described in this chapter involves the synthesis of various Coumarin Ethers Derived from 7-hydroxy-4-methyl coumarin and 2-bromoacetyl benzenes.

Scientists have recently directed their efforts into using it as a starting material for the synthesis of heterocyclic scaffolds such as oxazole; pyrazole; thiophene; thiazole; pyridine; diazepine; benzoxocin; benzoxepin; and pyrimidine derivatives. The coumarin-ethers are synthesized via different synthetic pathways with variety of biological effect.

The present investigation was aimed at synthesizing the conjugates of various 7-hydroxy-4-methyl coumarin and 2-bromoacetyl benzenes in the form of ethers. 7-hydroxy-4-methyl coumarin and 2-bromoacetyl benzenes have been employed as a starting materials in the synthesis of numerous scaffolds.

## 2.2. Methods

### 2.2.1. Materials and tools

The ingredients in this research are salicylaldehyde, ethylacetacetate, piperidine, Chlorobenzene, Hydrazine Hydrate, Glacial acetic acid, Methanol, n-hexane p.a, ethyl acetate p.a, technical nhexane, technical ethylacetate,. The laboratory equipment in this research are reflux devices, glassware, Whatman filter paper, evaporator, analytical balance, thermometer, thin layer chromatography (TLC) plates, Fourier Transform Infrared (FTIR).

## 2.2.2. Present work

Synthesis of 7-hydroxy-4-methyl coumarin (**3**) was brought about by the pechmann condensation of resorcinol (**1**) with ethylacetoacetate (**2**) as per our B. Sc. 5<sup>th</sup> semester organic lab manual (Scheme 1) by using concentrated sulphuric acid as cycling agent. The substituted Ethers (**7a-7c**) Derived from 7-hydroxy-4-methyl coumarin and 2-bromoacetyl benzenes were synthesized on water bath by refluxing the reaction in the presence of Anhydrous.  $K_2CO_3$  in dry ethanol (Scheme 2).

All the products gave satisfactory analytical and spectroscopic data, which are in full accordance with their assigned structures.

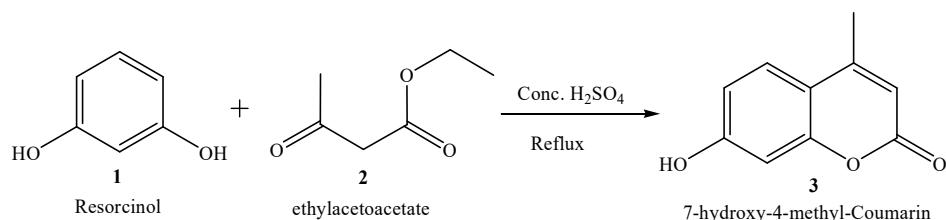
## 2.2.3. Experimental

This section deals with the preparation of following compounds.

1. Preparation of 7-hydroxy-4-methyl Coumarin (**3**).
2. Preparation of Ethers (**5a-5c**) Derived from 2-bromoacetyl Benzenes (**4a-4c**) and 7-hydroxy-4-methyl Coumarin (**3**)

### 1. Preparation of 7-hydroxy-4-methyl Coumarin (**3**):

A solution of 10.1 gm of resorcinol (**1**) and 10.3 gm of ethylacetoacetate (**2**) was added drop wise with stirring to 100 ml of conc.  $H_2SO_4$ . So that the temperature of reaction mixture did not raise above the 100 °C the reaction on complete addition mixture was kept at ambient temperature for 18 hr and then poured with vigorous stirring to the mixture of ice and water. The precipitate was filter off and washed with cold water then dried under reduced pressure to afford the crude solid mass. On recrystallized from aqueous alcohol gives final compound (**3**).

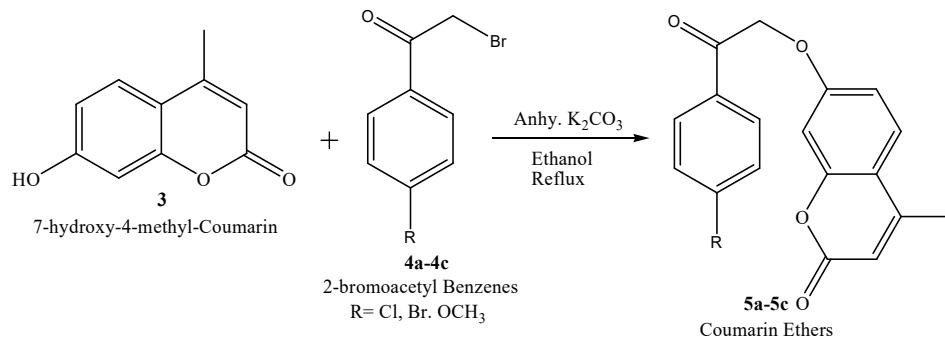


**Scheme-1:** Synthesis of substituted 7-hydroxy-4-methyl Coumarin (**3**)

### 2. Preparation of Ethers (**5a-5c**) Derived from 2-bromoacetyl Benzenes and 7-hydroxy-4-methyl Coumarin:

The synthesis of 7-hydroxy-4-methyl coumarin ethers (**5a-5c**) were started by adding 1 mmol of 7-hydroxy-4-methyl coumarin (**3**) and 2-bromoacetyl benzenes (**4a-4c**) (1 mmol) in anhydrous ethanol (10 mL) by adding anhydrous  $K_2CO_3$  as a base. The mixture was

continuously refluxed for 16-24 h at 80 – 100 °C. After cooling the reaction mixture poured on crushed ice, filtered, dried, and recrystallized from ethanol & 1,4-Dioxane mixture. The preparation of this compounds were carried out lined in Scheme -2 and its physical properties as shown below, including melting points (powder) and around 75 % yield were obtained and these compound were identified by FT-IR Spectroscopy.

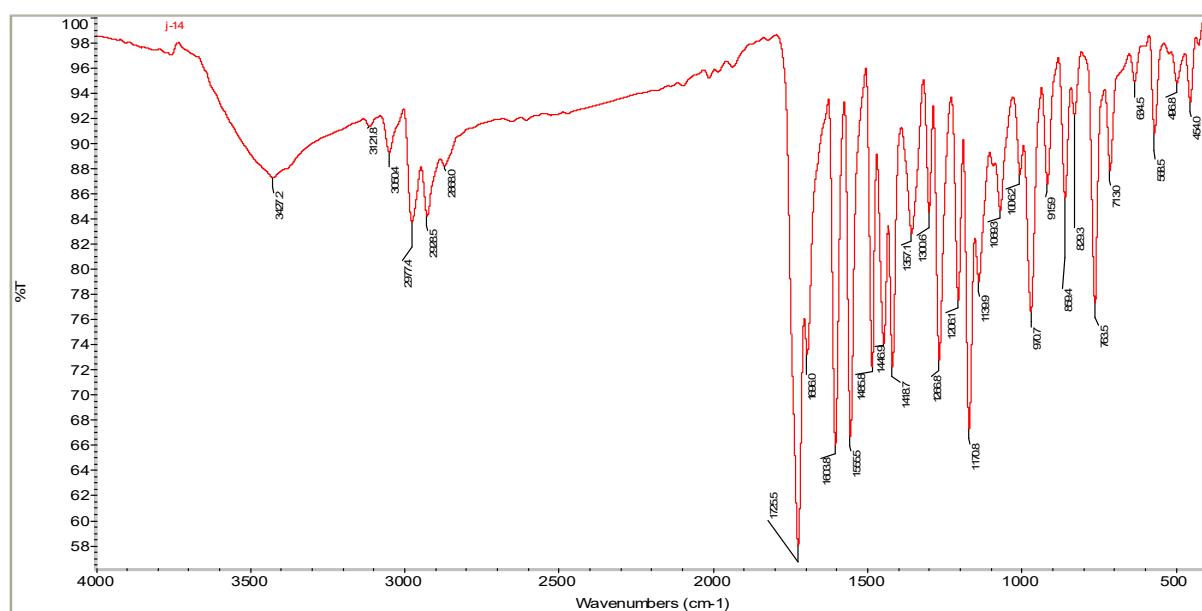


**Scheme-2:** Synthesis and Characterization of Ethers (**5a-5c**) Derived from 2-bromoacetyl Benzenes and 7-hydroxy-4-methyl Coumarin

## 2.2.4. Results and discussion

### Infrared spectral Studies

The IR spectrum of Schiff Base **5a** [Spectrum No.1] exhibited the lactone carbonyl stretching band around 1725 cm<sup>-1</sup> and another keto group showed the band around 1696 cm<sup>-1</sup>. The C-O-C stretching frequency band was observed around 1170 cm<sup>-1</sup> respectively. The IR spectral data of all the compounds are given below.



**Spectrum-1:** FTIR spectrum of Coumarin Ether 5a

The physical constants and analytical data of synthesized compounds (**5a-5c**) have been given below.

**i. Coumarin Ether **5a**:**

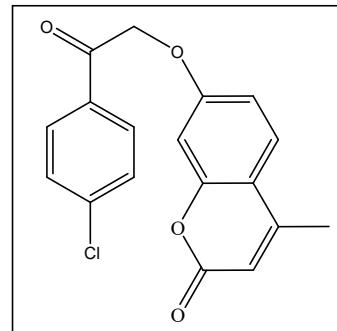
**Molecular Formula :**  $C_{18}H_{13}ClO_4$

**Physical State** : light yellowish colour solid

**mp[°C]** : 124-126

**GC-MS (m/z)** : 328

**IR (KBr, cm<sup>-1</sup>)** : (1725)cm<sup>-1</sup>, (1696)cm<sup>-1</sup>, (1170)cm<sup>-1</sup>, due to  $\nu$ (Cou C=O)str,  $\nu$ (C=O)str,  $\nu$ (COC)



**Elemental analysis for  $C_{18}H_{13}ClO_4$  : Calcd for - C, 65.76; H, 3.99; Cl, 10.78; O, 19.47 ; found - C, 65.71; H, 3.95; Cl, 10.73; O, 19.41.**

**ii. Coumarin Ether **5b**:**

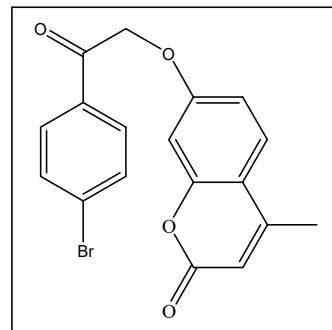
**Molecular Formula :**  $C_{18}H_{13}BrO_4$

**Physical State** : yellowish colour solid

**mp[°C]** : 138-140

**GC-MS (m/z)** : 372

**IR (KBr, cm<sup>-1</sup>)** : (1728)cm<sup>-1</sup>, (1692)cm<sup>-1</sup>, (1174)cm<sup>-1</sup>, due to  $\nu$ (Cou C=O)str,  $\nu$ (C=O)str,  $\nu$ (COC)



**Elemental analysis for  $C_{18}H_{13}BrO_4$  : Calcd for - C, 57.93; H, 3.51; Br, 21.41; O, 17.15 ; found - C, 57.88; H, 3.47; Br, 21.35; O, 17.11.**

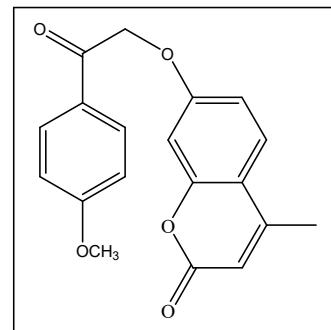
**iii. Coumarin Ether **5c**:**

**Molecular Formula :**  $C_{19}H_{16}O_5$

**Physical State** : White color solid

**mp[°C]** : 154-156

**GC-MS (m/z)** : 324



**IR (KBr, cm<sup>-1</sup>)** : (1730)cm<sup>-1</sup>, (1686)cm<sup>-1</sup>, (1164)cm<sup>-1</sup>, due to  $\nu$ (Cou C=O)str,  $\nu$ (C=O)str,  $\nu$ (COC)

**Elemental analysis for C<sub>21</sub>H<sub>21</sub>N<sub>5</sub>O<sub>6</sub> : Calcd for - C, 70.36; H, 4.97; O, 24.67 ; found – C, 70.30; H, 4.92; O, 24.61.**

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