



**Marathwada Shikshan Prasarak Mandal's**

## **Sunderrao Solanke Mahavidyalaya, Majalgaon**



### **INTERNAL QUALITY ASSURANCE CELL**

#### **CRITERION-6: GOVERNANCE, LEADERSHIP AND MANAGEMENT**

##### **6.3 Faculty Empowerment Strategies**

**6.3.1 The institution has effective welfare measures and Performance Appraisal System for teaching and non-teaching staff**

**Sample PBAS 2019-2020**



**DR. BABASAHEB AMBEDKAR MARATHWADA UNIVERSITY,  
AURANGABAD**

**Name of College: Sunderrao Solanke, Mahavidyalaya, Majalgaon, Dist. Beed**  
Self-Assessment-Cum-Performance Appraisal forms API-PBAS Proforma  
CAS Promotion for Assistant Professor/Associate Professor/Professor and Direct  
Recruitment of Associate Professor/Professor in Universities and Colleges.  
**For Faculty of Humanities/Science and Technology/Commerce and  
Management/Interdisciplinary**

**Reference:-**

- i) The Gazette of India: Extraordinary, part III Section 4 dated 18<sup>th</sup> July, 2018.
- ii) Government of Maharashtra Misc-2018 CR56/18/UNI date 8<sup>th</sup> March 2019.

**(ACADEMIC YEAR 2019-2020)**

**PART A: GENERAL INFORMATION AND ACADEMIC BACKGROUND**

1. Name ( in Block Letters): **Dr. IPPER SHANKAR NAGNATH**
2. Department : **CHEMISTRY**  
Current Designation & Grade Pay: **Assistant Professor, (AL 12<sup>th</sup>)**
3. Date of last promotion: **20/07/2018**
4. Which position and grade pay are you applicant under CAS? : **Associate Professor,  
Stage 4 (AL 13 A)**
5. Date of eligibility for promotion: **20/07/2021**
6. Address (With PIN code) : **Sunderrao Solanke Mahavidyalaya, Majalgaon Dist –  
Beed**

**Pin – 431131. (Maharashtra)**

**Telephone No. : 9763287724**

**Email: isn13@rediffmail.com**

7. Academic Qualification (From SSC till Post Graduation)

Examinations	Name of Board/University	Year of Passing	Percentage of marks obtained	Division/Classes/Grade		Special/Optional Subject
S.S.C.	Aurangabad	March 1996	64.13 %	First Class		Math/Sci/ Soc
H.S.C.	Aurangabad	March 1998	67.33 %	First Class		Phy/Chem/Bio/Soc
Bachelor of Science	S.R.T.M.U.Nanded	M/A 2002	68.00%	First Class with Distinction		Botany, Zoology, Chemistry
Master of Science	SRTMU, Nanded	M/A2004	65.00%	First Class		Chemistry
SET	Pune University	August 2006	Qualified	Qualified		Chemical Sciences

8. Research Degree(s)

Degree	Title	Date of award	University
Ph.D.	<i>"SYNTHESIS, CHARACTERIZATION AND ANTIMICROBIAL STUDIES OF METAL COMPLEXES OF SOME BIOLOGICALLY ACTIVE LIGANDS"</i>	05 Oct. 2019	Dr.Babasaheb Ambedkar Marathwada University, Aurangabad

10. Appointments held prior to joining this institution

Designation	Name of the Employer	Date of		Salary with Grade	Reason of leaving
		Joining	Leaving		

11. Posts held after appointment at this institution:-

Designation	Department	Period of working	
		From	To
Assistant Professor	Department of Chemistry ACS College Kille-Dharur	20/07/2007	28/06/2013
Assistant Professor	Dept. of Chemistry, Sunderrao Solanke Mahavidyalaya, Majalgaon.	29/06/2013	Till Today

12. Period of Teaching Experience :-

P.G. Class (in Years) : 04 Year

U.G. Class (in Years) : 13 Years

13. Research Experience excluding years spent for M. Phil / Ph.D. (in years): 6 Yrs

14. Fields of Specialization under the Subject / Discipline: - Organic Chemistry

15. Academic Staff College Orientation / Refresher Course/ ISTE - AICTE Sponsored STTP/SBP attended so far.

Name of the Course / Summer School	Place	Duration	Sponsoring Agency
Short Term Course MOOCs, E-Content Development and Open Educational Resources	UGC-HRDC, M.D.S. University, AJMER	2 March to 7 <sup>th</sup> 2020	UGC-HRDC



**PART-B ACADEMIC PERFORMANCE INDICATRS (API) :**

**Table-I**

**Assessment Criteria and Methodology for University / College Teachers**

Category	Name of Activity	Unit of Calculation		Self-Appraisal Grading for Assistant Professor / Associate Professor / Professor	Verified API Grading by Committee
		Actual Class spent per year	% of Teaching		
	[1]	[2]	[3]	[4]	
1.	Teaching : (Number of Classes taught / total classes assigned) X 100% (Classes taught includes sessions on tutorial, lab and other teaching related activities)			i) Good : 80% & above ii) Satisfactory : Below 80 % but 70 % & above iii) Not satisfactory Less than 70 %	
	Teaching Black Board			<b>GOOD</b>	
	B. Sc.-T. Y. Paper-XIV	41			
	B. Sc.-T. Y. Paper-XVI	42			
	Teaching ICT based.	08			
	Practical / Laboratory				
	B. Sc.-T. Y. lab course XV+XVIII	172			
	B. Sc.- S. Y. lab course IX+XII	168			
	B. Sc.-F. Y. lab Course III+IV	80			
	Tutorials / assignments / Project	10			
	Field Work				
	Group discussion	04			
	Seminars	04			
remedial Teaching	---				
Clarifying doubts within and outside the class hours	02				
Additional teaching to support counseling mentoring	02				
Total Actual hours spent	<b>533/540</b>	<b>98.70</b>			

2. Insolvent in the University / College students related activities / research activities

2.	Activities	Specify Actual participation in year	Total Days Spent per year	Self-Appraisal Grading for Assistant Professor / Professor	Verified API Grading By Committee
	Involvement in the University / College students related activities / research activities			i) <b>Good:</b> Involved in at least 3 activities. ii) <b>Satisfactory:</b> 2 activities. iii) <b>Not-satisfactory:</b> Not involved / undertaken any of the activities. <b>Note :</b> Number of activities can be within or across the broad categories of activities,	
	<b>(a) Administrative responsibilities</b> such as Head/ Chairperson / Dean / Director / Coordinator, Warden etc.	Administrative responsibility as a Head of the Department	Throughout year	<b>GOOD</b>	
	<b>(b) Examination and evaluation</b> duties assigned by the college / university or attending the examination paper evaluation.				
	(i) Question Paper Setting			<b>GOOD</b>	
	(ii) Invigilation / Supervision	University Examination Nov. 2019	15 Days		
	(iii) Flying Squad				
	(iv) CS / ACS / Custodian				
	(v) CAP / Director Assistant director				
	(vi) Unfair Menace Committee				
	(vii) Grievance committee				
	(viii) Internal Assessment	Test, Project Evaluation	10 Days		

	(ix) External Assessment	University Evaluation	10 Days		
	(x) Revolution				
	(xi) Result Preparation( College Level for Internal Assessment)	Internal Assessment	03 Days		
	(xii) M.Phil. Ph.D. Thesis evaluation / any other				
	<b>(c) student related co-curricular extension and field based activities</b> such as student clubs, carrier counseling, study visits, student seminars and other events, Cultural, sports, NCC, NSS and community services.	i) Coordinator, NSS	Throughout year	<b>GOOD</b>	
		ii) Member of Training and placement cell	Throughout year		
		iii) Chairman of Admission Committee	30 Days		
		iv) Member of Library Committee.	Throughout year		
		v) Seminars of B. Sc. III year students & study Visit	04 days 01 days		
		vi) Carrier Counseling	Throughout year		
	<b>(d) Organizing seminars / conferences/ Workshops, etc and other college / university activities</b>	National Conference on Recent Trends in Physics, Chemistry & Mathematics (RTPCM – 2020).	01 Days	<b>GOOD</b>	
	(e) Evidence of actively involved in guiding PhD students. i) No. of Registered candidate. ii) No. of Awarded Candidates.				
	( f ) Conducting Minor or major Research Project sponsored by national				

or inter-national agencies i) Above 10 laces ii) Below 10 laces,				
(g) At least one single or joint publication in peer reviewed or UGC list of journals i) No of Papers Published ( Single author ): ii) No of published (joint author)	No of Papers Published - <b>08</b>	180 Days	<b>GOOD</b>	
	Single author <b>01</b>			
	Joint author <b>07</b>			
<b>Overall grading (attached List)</b> <b>Good :</b> Good in teaching and satisfactory or good in activity at No 2 Or <b>Satisfactory :</b> Satisfactory in teaching and good or satisfactory in activity at Sr.No. 2 <b>Not Satisfactory :</b> If neither good nor satisfactory in overall grading.			<b>GOOD</b>	

**Table 2**

**Methodology for University and College Teachers for calculating Academic / Research Score**

**CATEGOEY- III: RESEARCH AND ACADEMIC CONTRIBUTIONS**

**1. Research Papers in Peer Reviewed or UGC listed Journals (08 points per publication)**

Sl. No	Title of research article/Paper Published	Name of journal Volume, number, & page Number	ISBN/ISSN No	Whether peer Reviewed Impact Factor, If any	Whether you are the main author	Self-Appraisal Score	API Score Verified	Page No. of relevant document
1	Synthesis, Spectral study, Characterization and Antimicrobial Activity of Zinc(II) Complex of Chalcone of Pyridine-2-Carbaldehyde	International Journal for Research in Applied Science & Engineering Technology, Vol. 7, Issue IX, Sep-2019	ISSN: 2321-9653	Peer Reviewed IF – 7.177	Yes	9.1 (8+5)* 0.7		
2	Synthesis, spectral study, characterization of Cobalt (II) and Copper(II) complexes of chalcones	Current Pharma Research 25 Sep-2019,416	ISSN: 2230-7842	Peer Reviewed IF –0-1	Yes	9.1 (8+5)* 0.7		
3	Synthesis, Spectral study, Characterization and Antimicrobial Activity of Copper (II) Complex of Chalcone of 5-Nitrofurfural	International Journal of Scientific Research, Vol. 8, Issue 10, Oct-2019	ISSN: 2277-8179	Peer Reviewed IF – 5.71 SJIF	Yes	9.1 (8+5)* 0.7		
4	Synthesis, Characterization and Antimicrobial Activity of 5-hydroxy-2-(5-methylfuran-2-yl)-4H-chromen-4-one	Journal of The Gujarat Research Society, Volume 21 issue 14, Nov-2019	ISSN: 0374-8588	UGC listed Journal IF 4.3	Yes	9.1 (8+5)* 0.7		



5	Synthesis, spectral Characterization and Antimicrobial Activity of 2-(furan-2-yl)-5-hydroxy-4H-chromen-4-one	Journal of The Gujarat Research Society, Volume 21 issue 13, Dec-2019	ISSN: 0374-8588	<b>UGC listed Journal</b> <b>IF 4.3</b>	Yes	13 (8+5)* 1.0		
6	Synthesis, Spectral Study, Properties and Antimicrobial activity of (E)-1-(2,6-dihydroxyphenyl)-3-(5-nitrofuranyl)prop-2-en-1-one	Journal of The Gujarat Research Society, Volume 21 issue 13, Dec-2019	ISSN: 0374-8588	<b>UGC listed Journal</b> <b>IF 4.3</b>	Yes	9.1 (8+5)* 0.7		
7	Synthesis, Characterization and Antimicrobial study of Nickel(II) Complex of (E)-1-(2,6-dihydroxyphenyl)-3-(5-methylfuran-2-yl)prop-2-en-1-one	Our Heritage Journal Volume 68, Special issue-12 Feb-2020	ISSN: 0474-9030	<b>UGC listed Journal</b> <b>IF 6.8</b>	Yes	9.1 (8+5)* 0.7		
8	Aliquat 366: An Efficient Catalyst For Knoevenagel Condensation Under Solvent-Free Condition	Our Heritage Journal Volume 68, Special issue-12 Feb-2020	ISSN: 0474-9030	<b>UGC listed Journal</b> <b>IF 6.8</b>	No	9.1 (8+5)* 0.7		
<b>Total API</b>						<b>76.7</b>		



**(2) (a) Publications ( other than Research papers) (Books, Chapters in Books )**

(i) Books published with ISSN / ISBN / number

International publisher: 12 points per Book for Single Author

National Publisher: 10 points per Books for Single Author.

Sr. No.	Title of Book with no. of Papers	publishers name with ISSN / ISBN /No.	International / National publisher	No. of Co. Author	whether Principal Author / Co. Author	Self appraisal Score	API Score Verified	Page No. of relevant Documents
TOTAL (2) (a) (i) :						NIL		

(2) (a) (ii) Chapter in Edited Book with ISSN / ISBN (5 points per chapter)

Sr. No.	Title of Chapter with page Nos.	Name of Book	Publisher name & ISSN / ISBN NO	No. of Co. Author	whether Principal Author / Co. Author	Self appraisal Score	API Score Verified	Page No. Relevant Documents
TOTAL (2) (a) (ii) :								

(2) (a) (iii) Editor of Book with ISSN / ISBN

Editor of Book by International publisher : 10 points per Book for Single author

Editor of Book by National publisher : 8 points per Book single author

Sr. No.	Title of Chapter with page Nos.	Publisher name & ISSN / ISBN NO	International Publisher	No. of Co. Author	whether Principal Author / Co. Author	Self appraisal Score	API Score Verified	Page No. Relevant Documents
TOTAL (2)(a) (iii) :						NIL		

**(2) (b) Translation works in Indian and Foreign Languages by qualified faculties (3 points per Chapter or Research paper) (8 points per Book)**

Sr. No.	Title of Chapter with page Nos.	Publisher name & ISSN / ISBN NO	International Publisher	No. of Co. Author	whether Principal Author / Co. Author	Self appraisal Score	API Score Verified	Page No. Relevant Documents
TOTAL (2)(b) :						NIL		
Total (2) : (2) (a) (i) + (2) (a) (ii) + (2)(a)(iii)+(2) (b)								

**(3) Creation of ICT mediated Teaching Learning pedagogy and content and development of new and innovative courses and curricula**

**(3) (a) Development of Innovative pedagogy : ( 5 points per Innovative pedagogy)**

Sr. No.	Title of Innovative pedagogy	Types of Teaching Learning Environments Face-to-Face / Networked / Open and distance / Virtual / if any	specify ICTs resources web link : You Tube videos Audios / smart Classroom / simulation game / Blogging / Online discussion forums / virtual Laboratories / telecast / picture / Model / charts if any	Date of approval from authority	Date of Implementation	self-appraisal score	API Score Verified	Page No. Relevant Documents
Sub Total (3) (a)						NIL		

**(3) (b) design of new curricula and courses : (02 points per curricula / course )**

Sr. No.	Name of Programme where curricula introduced	title of new curricula and courses	specify ICTs resources web link : You Tube link / audio / video / telecast Picture Model / Charts if any	Date of approval from authority	Date of Implementation	self-appraisal score	API Score Verified	Page No. Relevant Documents
Sub Total (3) (a)						NIL		

**(3) (c) MODCs :**

**(3) (c) (i) Development of complete MOOCs in 4 quadrants (4 credit course ) (20 per curricula / course )(In case of MOOCs of Lesser credits 05 marks / Credit )**

Sr. No.	Name of Programme where curricula introduced	Course Credits	Title of new MOOC curricula	specify ICTs resources : web link / You tube link : audio / video / Telecast picture Model / charts if any	Date of approval from authority if any	Date of Implementation	self-appraisal Score	API Score Verified	Page No. Relevant Documents
Sub Total (3) (C) (i)						NIL			

**(1) (c) (ii) MOOCs ( Developed in 4 quadrants) per module / lecture (5 point per module / Lecture )**

Sr. No.	Name of Programme where curricula introduced	Course Credits	Title of new MOOC curricula	specify ICTs resources : web link / You tube link : audio / video / Telecast picture Model / charts if any	Date of approval from authority if any	Date of Implementation	self-appraisal Score	API Score Verified	Page No. Relevant Documents
Sub Total (3) (C) (ii)						NIL			

**(2) (C) (iii) content writer / subject matter expert for each module of MOOCs ( at least one quadrant) ( 2 points per curricula / course )**

Sr. No.	Name of Programme where curricula introduced	Course Credits	Title of new MOOC curricula	specify ICTs resources : web link / You tube link : audio / video / Telecast picture Model / charts if any	Date of approval from authority if any	Date of Implementation	self-appraisal Score	API Score Verified	Page No. Relevant Documents
Sub Total (3) (C) (iii)						NIL			

**(3) (c) (iv) Course Coordinator for MOOCs (4 credit course ) (8 Points per curricula / Course ) (In case MOOCs of lesser credits 02 marks / credit )**

Sr. No.	Name of Programme & course	Course Credits	Title of new MOOC curricula	specify ICTs resources : web link / You tube link : audio / video / Telecast picture Model / charts if any	Date of approval from authority if any	Date of Implementation	self-appraisal Score	API Score Verified	Page No. Relevant Documents
Sub Total (3) (c) (iv)						NIL			
Total : (3) (c) (i) + (3) (c) (ii)+ (3) (c) (iii)+ (3) (c) (iv)						NIL			

**(3) (d) e- Content**

**(3) (d) (i) development of e-content in 4 quadrants a complete course / e-book  
( 12 Points per curricula / Course )**

Sr. No.	title of e-content course / e-book with no. of pages. ISSN / ISBN NO of any	name of Programme & Course to which introduced	specify ICTs resources : web link	whether peer reviewed	No. of co-Authors	whether principal author / co-authors	Self approval Score	API Score Verified	Page No. Relevant Documents
Sub Total (3) (d) ( i )							NIL		

**(3) (d) (ii) e-content ( development in 4 quadrant ) per Module ( 5 points per module / course)**

Sr. No.	title of e-content course / e-book with no. of pages. ISSN / ISBN NO of any	name of Programme & Course to which introduced	specify ICTs resources : web link	whether peer reviewed	No. of co-Authors	whether principal author / co-authors	Self approval Score	API Score Verified	Page No. Relevant Documents
Sub Total (3) (d) ( ii )							NIL		

**(3) (d) (iii) Contribution to development of e-content module in complete course /paper /e- book (at least one quadrant) (2 POINTS PER MODULE/COURSE )**

Sr. No.	title of e-content course / e-book with no. of pages. ISSN / ISBN NO of any	name of Programme & Course to which introduced	specify ICTs resources : web link	whether peer reviewed	No. of co-Authors	whether principal author / co-authors	Self approval Score	API Score Verified	Page No. Relevant Documents
Sub Total (3) (d) ( iii )							NIL		



**(3) (d) (iv) Editor of e- content for complete course / paper / e- book ( 10 points per course / paper )**

Sr. No.	Title of Content course / e Book with no. of pages ISSN/ISBN NO if any	Name of Programme & Course to which introduced	specify ICT's resource's web link	Whether Peer reviewed	No. of Co-Author's	Whether Principal Author/ Co-Author	Self-appraisal Score	API Score Verified	Page No of Relevant Document
Subtotal(3) (d)(iv)							NIL		
total(3)(d):(3)(d)(i)+(3)(d)(ii)-(3)(d) (iii)+(3)(d)(iv)							NIL		
total(3):(3)(a)+(3)(b)+(3)(c)+(3)(d)							NIL		

**(4) Research Score**

**(4) (a) Research guidance :**

**(Ph.D.: 10 points per degree awarded & 5 per thesis submitted)**

**(M.Phil./ PG dissertation :2 point per degree awarded)**

Sr. No	Sr. No	Number of candidate Enrolled	No .of thesis Submitted with dates	No. of Candidate Degree Awarded with dates	Self appraisal Score	API Score Verified	Page No. Relevant Documents
	M.Phil/ P G Dissertation						
	Ph.D.						
Sub Total:(4)(a)					NIL		

**(4)(b) Research Projects Completed:**

**A:More than 10 lakhs (10 points per Project)**

**B: Less than 10 Lakhs ( 5 point per Project)**

Sr. No	Type of Project A/B	Title of Project	Sponsored agency	Date of Completion	Whether Co-PI	Grant Received	Self Appraisal Score	API Score Verified	Page no of Relevant Document
Sub Total(4)(b)							NIL		

**(4)(C) Research Projects Ongoing :**  
**A : More then 10 Lakhs (5 Point per Project)**  
**B: Less than 10 Lakhs (2 Points per Project)**

Sr. No	Type of Project A/B	Title of Project	Sponsored agency	Date of Completion	Whether Co-PI	Grant Received	Self Appraisal Score	API Score Verified	Page no of Relevant Document
Sub Total(4)(c)							NIL		

**(4)(d) Consultancy:**

Sr. No	Title of Consultancy Project	Sponsored agency	date of starting	Amount Mobilized (Rs.Lakh)	Self appraisal Score	API Score Verified	Page No. Relevant Documents
sub Total:(4)(d)					NIL		
Total (4):(4)(a)+(4)(b)+(4)(C)+(4)(d)					NIL		

**(5)(a) Patents: (10 Points per international Patent and 7 points per national patent)**

Sr. No	Title Of Patent Project	Patent Number	Sponsored Agency if any	Date of Award	International National	Self Appraisal Score	API Score Verified	Page No. Relevant Document
sub Total:(5)(a)						NIL		

**(5)(b)\* Policy Document (Submitted to an International Body/ Organization like UNO/UNESCO/World Bank /international monetary Fund etc. Central Government of State Government)**

**A: International (10 points per Policy Document)**

**B: National (07 Points Per Policy Document)**

**C: State (05 Points Per Policy Document)**

Sr. No	Title Of policy Document	Name Of Submitted Agency	International/national/state	Policy Document no	Date of Acceptant	Self Appraisal Score	API Score Verified	Page No. Relevant Document
sub Total:(5)(b)						NIL		



**(5) (C) \* Awards / Fellowship :A : International (07 Points per Policy Document)  
B : National (05 Point per Policy Document )**

Sr. No.	Name of Award / Fellowship	Date of Received	International / national	Name of Awardees Academic Body / Association	Self Appraisal Score	API Score Verified	Page No. Relevant Document
01	<b>Best Researcher Award</b>	18 & 19 Jan-2020	International	<b>International Best Scientist Awards, VDGGOOD Professional Association, New Delhi</b>	07		
sub Total:(5)(C)					<b>07</b>		
Total : (5) (a)+(5) (b)+(5)(c)					<b>07</b>		

**(6) Invited Lectures / Resource Person / Paper Presentation in Seminars / Conferences / Full Paper in Conference Proceedings ( Paper presented in Seminars / conferences and also Published as full paper in conference Proceedings will be counted only once)**

International (Abroad) : (07 points per seminars : Conferences ) International (within country) : (05 Points per seminars / conferences ) National: ( 3 Points per Seminars / conferences ) State / University Level: ( 2 points per Seminars / Conferences )

Sr. no	Title of Presentation in Academic Session	Title of Conference Seminar	Mode of presentation invited Lectures/ Resource Person / paper presentation	Name Of Organizer	whether International (Abroad) International (within Country) National / State / University Level	Self Appraisal Score	API Score Verified	Page No. Relevant Document
1)	<b>Synthesis, Properties and Antimicrobial activity of (E)-1-(2,6-dihydroxyphenyl)-3-(5-nitrofuranyl)prop-2-en-1-one</b>	<b>Emerging Trends and Challenges in Biosciences (ETCB-2019)</b>	<b>R. B. Attal Arts, Science &amp; Commerce College, Georai Dist. Beed (MS)</b>		<b>National Conference</b>	<b>03</b>		

2)	Resource Person	Human Resource Enrichment Programme (HREP 2019-20)	Sunderrao Solanke Mahavidyalaya, Majalgaon	Workshop	02		
Total (6)					05		

**Note: The Research score for research papers would be augmented as follows:**

Peer Reviewed or UGC-Listed Journals (Impact factor to be determined as per Thomson Reuters list) : i) Paper in refereed journals without impact factor – 5 Points ii) Paper with impact factor less than 1-10 Points iii) Paper with impact factor between 1 and 2-15 Points iv) Paper with impact factor between 2 and 5-20 Points v) Paper with impact factor between 5 and 10-25 Points vi) Paper with impact factor 10-30 Points

- Two authors : 70 % of total value of publication for each author :
- More than two authors : 70 % of total value of publication for the First / principal / Corresponding author and 30 % of total value of publication for each of the joint authors.

**Joint Projects :** Principal Investigator and Co-Investigator would get 50 % each.

**Note :**

- Paper presented if part of edited book or proceeding then it can be claimed only once.
- For joint supervision of research students. the formula shall be 70 % of the total score for Supervisor and co-supervisor, both shall get 7 marks each.
- For the purpose of calculating research score of the teacher, the combined research score form the category of 5 (b) . Policy Document and 6 Invited Lectures / Resources person / paper presentation shall have an uppers capping of thirty percent of the total research score of the teacher concerned.
- There research score shall be from the minimum of three categories out of six categories.

**Summary (Category III)**  
**Academic / Research Score**

<b>Details of Academic &amp; Research Activities</b>	<b>Self Appraisal Score</b>	<b>API Score Verified by Committee</b>	<b>Remarks</b>
Research Papers in Peer Reviewed or UGC Listed Journals	76.7		
Publications (Other than Research Papers)	00		
Creation of ICT Mediated Teaching learning pedagogy and content development of new and innovative courses and curricula	00		
Research guidance / Projects Completed / Projects ongoing / Consultancy	00		
Patents / Policy Documents / Awards / Fellowships	07		
Invited lectures / Resource person / Papers presented in Seminars, Conferences / Full paper in Conference Proceeding	05		
<b>Grand Total</b>	<b>88.7</b>		


**IV. SUMMARY OF SCORES**

<b>Category</b>	<b>Criteria</b>	<b>Total – API Score for Assessment period</b>
II	Activities: Over Grading 1. Teaching, 2. Innovations in the University / College student related activities / research activities	Good Good
III	Academic / Research Score	<b>88.7</b>

## UNDERTAKING

I **Dr. Shankar Nagnath Ipper** undertake that the information provided is correct as per records submitted by me to College / Institute and / or documents enclosed along with the duly filled PBAS Performa.

Date:  
Place: Majalgaon.

  
Signature of the Faculty with Designation  
Dr. S. N. Ipper  
Assistant Professor


Date:  
Place: Majalgaon

  
Signature  
Head of the Department

Date:  
Place: Majalgaon

  
Signature of Director IQAC  
Internal Quality Assurance Cell (IQAC)  
Sunderrao Solanke Mahavidyalaya,  
Majalgaon, Dist. Beed (MS)

Date:  
Place: Majalgaon

  
Signature of Principal  
PRINCIPAL  
Sunderrao Solanke Mahavidyalaya  
Majalgaon Dist. Beed (M.S.)

**N. B.:** The individual PBAS Performa duly filled along with all enclosures, submitted for CAS promotions will be duly verified by the college/Institute/university as necessary and placed before the Screening cum Evaluation Committee or Selection Committee for assessment / verification.



Dr. Babasaheb Ambedkar  
Marathwada University  
Aurangabad-431004 Maharashtra (India)  
NAAC Re-accredited A



डॉ. बाबासाहेब आंबेडकर  
मराठवाडा विद्यापीठ

औरंगाबाद-४३१००४, महाराष्ट्र(भारत)  
नेक समितीतर्फे 'अ' दर्जा प्राप्त

Telephone  
Office PBX : (0240) 2403399/400  
C.O.E. : (0240) 2403200  
Fax : (0240) 2403199/188  
Website : [www.bamu.ac.in](http://www.bamu.ac.in)  
<http://bamua.digitaluniversity.ac>  
E-Mail : [coe@bamu.ac.in](mailto:coe@bamu.ac.in)

Office of the Director,  
Board of Examinations & Evaluation  
कार्यालय, संचालक,  
परीक्षा व मूल्यमापन मंडळ

Ref. No. Lx/D/CAS/UG/CAS/OCT-NOV-2019-1800

Date : 11.10.2019

To,  
PROF. SHANKAR NAGNATHIPPIER  
SUNDERRAO SOLANKE MAHAVIDYALAYA, MAJALGAON,  
TO, MAJALGAON, DIST- BEED

Cell No.: 9763287724  
Email : [isn13@rediffmail.com](mailto:isn13@rediffmail.com)

Subject: Assessment of Answer-Books Science Faculty Examination October/November 2019

Sir/Madam,

I am directed by the Hon'ble Vice-Chancellor to invite you to assess the Answer Book of Under Graduate of Faculty Examination held in October-November 2019 in the subject of CHEMISTRY

The assessment work in all the subject will Commence has commenced from MRS KESHARBAI SONAJIRAO KSHIRSAGAR ALIAS KAKU ARTS SCIENCE COMMERCE COLLEGE, BEED at the D CAS Centre

May I request you to make it convenient to attend the assessment of Answer Books of your subject on CHEMISTRY Semester I to VI

The remuneration T.A. D.A. Local conveyance will be paid on spot as per the University rules.

I am further directed by the Hon'ble Vice-Chancellor to invite your attention to the following provision of Maharashtra Public Universities Act, 2016 in respect of Examination work. The Section 48 of Act is reproduced as under:-

*"48(4) It shall be obligatory on every teacher and on the non-teaching employee of the University, affiliated, conducted colleges, community colleges or recognized institutions to render necessary assistance and service in respect of examinations of the university and evaluation of students as prescribed by statutes. If any teacher or non-teaching employee fails to comply with the order of the university or college or institution, in this respect, it shall be treated as misconduct and the employee shall be liable for disciplinary action. In case of failure on the part of the teacher or nonteaching employee of any affiliated college, conducted college, community college or recognized institution, to comply with the order of the university in this respect, the Vice-Chancellor shall have power to take an appropriate action against them, which may include imposing penalties including suspension of approval to the appointment of a teacher, as may be prescribed by the Statutes."*

Your co-operation in such a great venture is earnestly solicited.

Thanking you

Yours sincerely,

Director  
Board of Examinations & Evaluation

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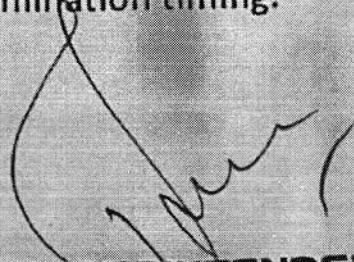
M.S.P.Mandal's  
SUNDERRAO SOLANKE MAHAVIDYALAYA MAJALGAON,  
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Invigilation Programme  
Senior college Exam -Oct./Nov.- 2019

Name of Invigilator: ---Mr. S. N. Ipper

Sr.No.	Date	Morning Session	Evening Session
01	10-10-2019 to	-11-	-11-
02	24-10-2019		
03			
04			

Note: 1) Invigilation Programme is Mandatory.

2) Invigilator must be Present at Exam venue before half an hour of the examination timing.

  
**CHIEF SUPERINTENDENT**  
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Marathwada Shikshan Prasarak Mandal's



# R. B. Attal Arts, Science & Commerce College

Georai Tq. Georai Dist. Beed

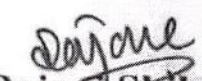
## National Conference on Emerging Trends and Challenges in Biosciences

11<sup>th</sup> August, 2019

### CERTIFICATE

This is to certify that Dr./Mr./Mrs. S. N. Inper has Chaired the session / participated / invited lecture / presented / entitled Synthesis, Spectral study . . . . . in One Day National Conference on "Emerging Trends and Challenges in Biosciences" organized by R. B. Attal Arts, Science and Commerce College, Georai, Dist. Beed on 11<sup>th</sup> August, 2019.

  
**Dr. Pangrikar P. P.**  
Convener

  
**Dr. Rajani Shikhare**  
Principal & Organizer



Marathwada Shikshan Prasarak Mandal's

# Sunderrao Solanke Mahavidyalaya, Majalgaon

Dist. Beed (M.S.) 431 131

(NAAC Re-Accredited 'A' Grade with CGPA 3.21)

National Conference on

"Recent Trends in Physics, Chemistry & Mathematics (RTPCM – 2020)"

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This is to certify that, Dr./Mr./Mrs./Prof. *S. N. Ipper*

from *Sunderrao Solanke Mahavidyalaya*

has actively participated as

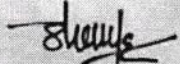
a Delegate/ Resource Person/ Chair Person during National Conference on "Recent Trends in Physics, Chemistry &


Mathematics (RTPCM-2020)" held on 4<sup>th</sup> February 2020 Organized by Department of Physics, Chemistry & Mathematics,

Sunderrao Solanke Mahavidyalaya, Majalgaon.

He/ She has presented paper entitled, "*Synthesis, Characterization and Antimicrobial Study of Cadmium(II) complex.*"

  
Mr. G. K. Sanap  
Convener

  
Dr. S. N. Ipper  
Organizing Secretary

  
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Mandal's Sunderrao Solanke Mahavidyalaya, Majalgaon on 18 August 2019.

Venue : Swatantrya Senani Ramrao Awargaonkar Law College, Beed.

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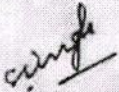
*Assistant Professor, Chemistry*

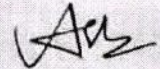
*Sunderrao Solanke Mahavidyalaya, Majalgaon, Beed Maharashtra  
affiliated to Dr. Babasaheb Ambedkar Marathwada University, Aurangabad  
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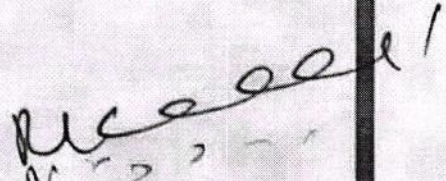
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*and Obtained Grade (A) .*

  
(Prof. Shiv Dayal Singh)  
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# Synthesis, Spectral Study, Characterization and Antimicrobial Activity of Zinc (II) Complex of Chalcone of Pyridine-2-Carbaldehyde

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**Abstract:** Metal complex of Zn(II) has been synthesized with newly prepared biologically active ligand. This ligand was prepared by the Claisen-Schmidt condensation method of 2,6-dihydroxy acetophenone and pyridine-2-Carbaldehyde. The structure of the complex has been proposed by the analytical data, conductivity measurement, magnetic moment, IR spectrum, Electronic absorption spectrum, thermal studies and XRD analysis. Analytical data confirmed 1:2 stoichiometry and the electronic spectral data, IR, magnetic moment, TG-DTA suggests that Zn(II) complex has square planar geometry. Absence of coordinated water molecules in Zn(II) complex is confirmed by TG-DTA studies. The conductivity data show that the complex is non electrolyte. Antimicrobial activities of complex with selected bacterial strain and fungal strain carried out and the results have been compared with commercial standards. In this paper we prepare chalcone of pyridine-2-Carbaldehyde by Claisen-Schmidt condensation method and synthesize Zn(II) metal complex and characterize them by Infrared, Electronic absorption spectra, magnetic susceptibility, CHO analysis, solution conductivity, XRD study, TG-DTA and antimicrobial activity.

**Keywords:** Electronic absorption spectrum, Infrared spectrum, TG-DTA, Elemental analysis, Antimicrobial activities, Physico-chemical property, Magnetic susceptibility and Conductivity.

## I. INTRODUCTION

Chalcones constitute an important group of natural products, chemically they consist of open chain flavonoids in which the two aromatic rings are joined by  $\alpha$ ,  $\beta$  unsaturated carbonyl system. The name chalcone is given by Kostanecki and Tambar [1]. Many complexes of chalcones are synthesized and studied in the literature [2-3]. It is believed that the ( $>CO-C=C<$ ), moiety imparts biological characteristics to this class of compounds. Such  $\alpha$ ,  $\beta$ -unsaturated carbonyl compounds and their metal complexes possess interesting biochemical properties, such as antitumour, antioxidant, anti-fungal and antimicrobial activities [4]. The electronic absorption spectrum, infrared spectrum, magnetic moment, TG-DTA supports the square planar geometry of the metal complex of chalcone. All crystals of a substance possess the same elements of symmetry. The computer program, used for indexing data was powder-X. Furthermore, biological activities of complex with selected bacterial strain and fungal strain carried out and the results have been compared with commercial standard [5]. The X-ray powder diffractogram of the metal complexes was used for the structural characterization and determination of lattice dimensions.

## II. MATERIALS AND METHODS

### A. Synthesis of chalcone of Pyridine-2-Carbaldehyde

The reagents used for preparation of chalcone of pyridine-2-carbaldehyde are of A.R. grade. 2,6-dihydroxy acetophenone (0.01mol) and pyridine-2-carbaldehyde (0.01mol) are dissolved in ethyl alcohol (25 mL) and then potassium hydroxide 10 mL (40%) were added to it. The reaction mixture was heated for 3 hours till yellow-brown color was obtained. The progress of reaction was monitored by TLC, after completion of reaction the content was poured into ice cold water and then acidified by dil. HCl the solid obtained was filtered and the crude product was recrystallized from ethyl alcohol to give pyridine chalcone[6].

### B. Synthesis of metal complex

The solution of 0.02 mole of chalcone of pyridine-2-carbaldehyde was taken in round bottom flask containing 30 ml of anhydrous methanolic solution and boiled for 10 minutes. A hot solution of 0.01 mole, of Zinc Nitrate in 20 ml of methanol was added drop wise to the solution of the chalcone of pyridine-2-carbaldehyde To this reaction mixture, 10% alcoholic ammonia was added up to slightly alkaline pH. The complex was precipitated at 9 pH range. The pH 8-10 range was definite for these complexes [7]. The



contents were stirred on magnetic stirrer for one hour. The solid metal complex separated out and washed with methanol three to four times. Dried in vacuum desiccators over anhydrous granular calcium chloride. The melting point/decomposition a temperature of the complex was determined by Thiele's melting apparatus. The reactions of formation of Zn(II) complex is shown in figure (1).

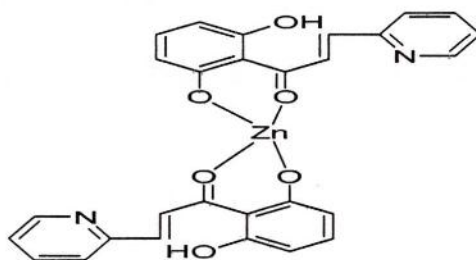


Fig.(1): Metal complexes of Zinc (II) with chalcone of pyridine-2-carbaldehyde

### III. RESULTS AND DISCUSSION

#### A. Infra Red Spectrum

1) *Infrared spectrum of Zn(II) complex:* The infrared spectra of ligands and Zn(II) complex of chalcone of pyridine-2-carbaldehyde was recorded on a Perkin-Elmer Spectrum RX-IFTIR Spectrophotometer over the range 4000-400  $\text{cm}^{-1}$  using KBr pellet at CIL, Chandigarh, Punjab.

Seema Habib assigned [8] the ligand which shows a weak broad band around 3047-3029  $\text{cm}^{-1}$ . In Zn complexes do not show any absorbance for -OH of the coordinated water molecule. In the IR spectra of all ligands, an intense band appearing around 1656  $\text{cm}^{-1}$  is attributed to C=O group. The medium intensity band appearing around 1530  $\text{cm}^{-1}$  in the ligand and the complexes are assigned to C=O aromatic. The M-O band for Zn(II) was observed at 500  $\text{cm}^{-1}$ .

Chiara Sulpizio assigned that, [9]  $\text{Zn}^{+2}$  do not show any significant shift compared to the free ligand. While the  $\text{Zn}^{+2}$  complexes show a shift of C=O stretching mode from 1630 to 1610  $\text{cm}^{-1}$  indicating coordination of carbonyl oxygen to a metal ion.

In the chalcone of pyridine-2-carbaldehyde a sharp strong band observed at 1597  $\text{cm}^{-1}$  is attributed to (C=C) stretching mode in the IR spectra of ligand. This band observed at 1437  $\text{cm}^{-1}$  in Zn(II) complexes. The slight shift in the band of (C=C) stretching frequency is due to change in electron distribution across (C=C) bond in the metal complexes[10].

The presence of phenolic -OH is confirmed by peaks at 3055  $\text{cm}^{-1}$  in the ligand, In the spectra of Zn(II) complexes, there is the complete disappearance of the peak at 3055  $\text{cm}^{-1}$  in chalcone which suggests absence of phenolic group -OH indicates its coordination. (C-O-C) is shifted to a lower wave number compared with a free ligand. In the IR spectra of ligand, the strong bands appeared in the region 1623 & 1666  $\text{cm}^{-1}$  are assigned to  $\nu$  C=O) of stretching frequency [11]. It is shifted towards lower frequency than corresponding ligands and appeared at 1599  $\text{cm}^{-1}$  in metal complexes. Such a lowering in stretching vibration of  $\nu$  (C=O) in chalcone indicates the participation of chalcone carbonyl in complexation. In the IR spectra of Zn(II) complexes, In Zn(II) complexes new band is observed at 507  $\text{cm}^{-1}$  due to the (M-O) bond.

Table no. (1): Infrared absorption frequencies ( $\text{cm}^{-1}$ ) of chalcone of pyridine-2-carbaldehyde Zn(II) complex.

Ligand/ Metal complexes	$\nu(\text{OH})$ $\text{cm}^{-1}$	$\nu(\text{H}_2\text{O})$ $\text{cm}^{-1}$	$\nu$ ((-CO- CH=CH-)) $\text{cm}^{-1}$	$\nu$ ((C-O- C) $\text{cm}^{-1}$			
chalcone of pyridine-2-carbaldehyde	3055		1666	1099	$\nu$ (C=C) $\text{cm}^{-1}$	Aromatic ring (C=C) $\text{cm}^{-1}$	$\nu(\text{M-O})\text{cm}^{-1}$
[Zn (chalcone of pyridine-2carbaldehyde) <sub>2</sub> ]	3068	-	1599	1052	1437	1369	507

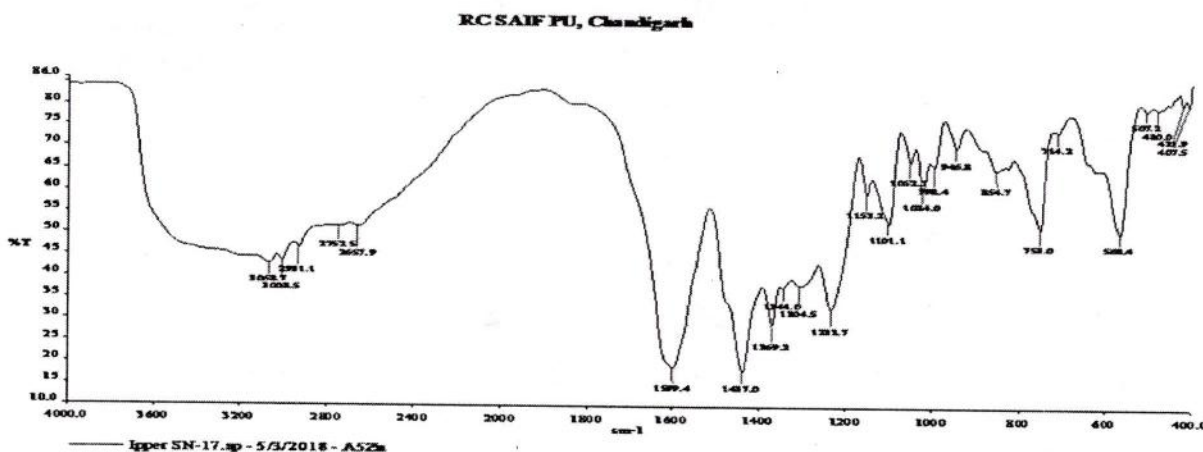


Fig. (2): Infrared spectra of Zn metal complex.

**B. Physical Parameters**

Table no.(2): Physical parameters of Zinc (II) complex.

Metal complex	Ligand	p <sup>H</sup> range ppt	Color	M.P. °C
Zinc(II) complex	chalcone of pyridine-2-carbaldehyde	7.0-7.5	Reddish brown	280

**C. CHO Analysis**

The carbon, hydrogen, oxygen, cobalt and copper metals percentage in Zn(II) complex of chalcone measured at SAIF Cochin, Kerala. The calculated and measured values of CHO analysis are matching and are given in the table no.(3).

Table no. (3): CHO analysis

Metal complexes	Chemical formula	Mol. Wt.	Elemental analysis : % found (calculated)						
			C	H	N	O	S	X(Br)	M
Zn (II) complex	[C <sub>28</sub> H <sub>20</sub> O <sub>6</sub> N <sub>2</sub> Zn]	545	61.61 (57.80)	3.69 (4.16)	5.13 (4.81)	17.58 (21.99)	-	-	11.90 (11.23)

**D. Magnetic Susceptibility, Solution Conductivity And Electronic Absorption Spectral Data**

- Magnetic Susceptibility:** The  $\mu_{\text{eff}}$  (B.M.) values at room temperature for Zn(II) complex is dimagnetic these values agree with Square planar geometry of the metal complex [12-13].
- Solution Conductivity:** The solution conductivities of  $10^{-3}$  M solution of metal complex in DMSO were measured on EQUIPTRONICS digital conductivity meter EQ - 660 with  $20 \mu\Omega$  to  $200 \mu\Omega$  at 298K temperature. In the present investigation Zn(II) complex is reddish brown in color, stable to air and moisture. Decomposes at high temperature rather than showing sharp melting points. They are insoluble in water and soluble in DMSO, DMF. The low conductivity values in DMSO solution ( $10^{-3}$  M) are given in table no.(4) indicates non-electrolytic nature.
- Electronic Absorption Spectral Study:** Electronic absorption spectrum was measured on SL159, single beam UV-VIS spectrophotometer.

The electronic spectrum of Zn(II) complex studied in the present investigation exhibit absorption band at  $27247(367 \text{ nm}) \text{ cm}^{-1}$  which are assigned to charge transfer band.



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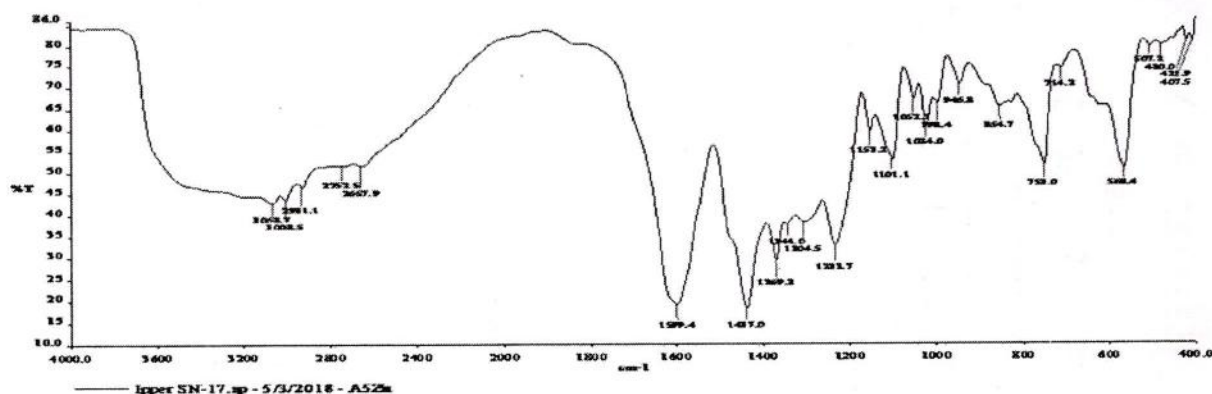


Fig. (2): Infrared spectra of Zn metal complex.

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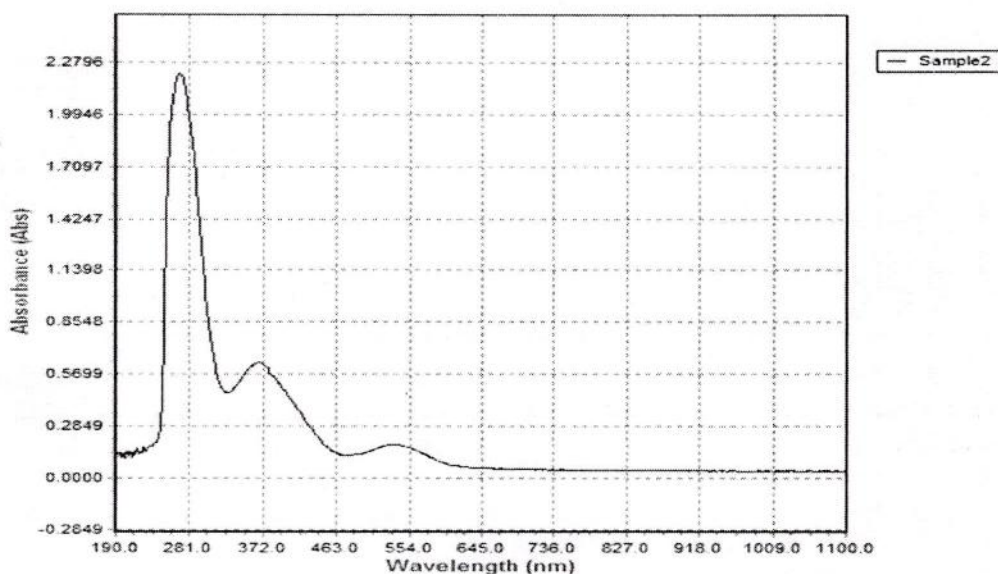


Fig. (3): Electronic absorption spectra of Zn (II) complex of chalcone of pyridine-2-carbaldehyde

Table no. (4): Solution conductivity, magnetic and electronic absorption spectral data of Zn(II) complex.

Zn(II) Complex	Molar Conductance $\text{Ohm}^{-1}\text{cm}^2\text{mol}^{-1}$	$\mu_{\text{eff}}$ (B.M.)	Absorption Maxima (LMCT) $\text{cm}^{-1}$ Charge transfer.
Chalcone of pyridine-2-carbaldehyde	8.04	Diamagnetic	27247(367)

#### E. Thermal Analysis Zn(II) Complex Chalcone of Pyridine-2-Carbaldehyde

The simultaneous thermogravimetric, differential thermal analysis of Zn(II) complex chalcone of pyridine-2-carbaldehyde was performed in an inert nitrogen atmosphere on Perkin Elmer STA 6000 at SAIF, Cochin, Kerala. The heating rate was  $10^\circ/\text{min}$  and flow rate of nitrogen 50 ml/min. The reference substance used was  $\alpha\text{-Al}_2\text{O}_3$  in platinum crucible and sample weighed in the range of 4-12 mg. The thermogram of Zn(II) complex of chalcone of pyridine-2-carbaldehyde is presented in figure (4). This curve reveals that there is absence of lattice as well as coordinated water in the complex.

The TG-DTA curve of Zn(II) complex chalcone of pyridine-2-carbaldehyde reveals that the complex is thermally stable decomposes above  $250^\circ\text{C}$  and there is no weight loss up to  $250^\circ\text{C}$  indicating the absence of lattice water as well as coordinated water. The first step shows rapid decomposition within a temperature range  $250\text{-}330^\circ\text{C}$  with a weight loss of 22.67% (calc. wt. loss 23.79%) which may be due to loss of non-coordinated part two pyridine ring fragments of the ligand. This is confirmed by an endothermic peak at  $293.00^\circ\text{C}$  in DTA. The second step decomposition with a weight loss of 36.05% in the range  $340\text{-}480^\circ\text{C}$ , corresponds to the decomposition of coordinated part of the complex. This is confirmed by an endothermic peak at  $414.05^\circ\text{C}$  in DTA. The third step decomposition with a weight loss of 28.98% in the range  $490\text{-}600^\circ\text{C}$ , corresponds to the decomposition of remaining coordinated part of the complex. This is confirmed by an endothermic peak at  $557.36^\circ\text{C}$  in DTA. The compound finally decomposes above  $600^\circ\text{C}$  with a weight loss 14.92% and form ZnO as final product.

The thermal behavior of zinc metal complexes in the present study indicates high thermal stability. Decomposition of all the complexes is started at a relatively higher temperature ( $\sim 250^\circ\text{C}$ ), finally giving a metal oxide residue. Thermograms of all the Zn complexes indicate the absence of lattice water as well as coordinated water molecules and it exhibit higher thermal stability.



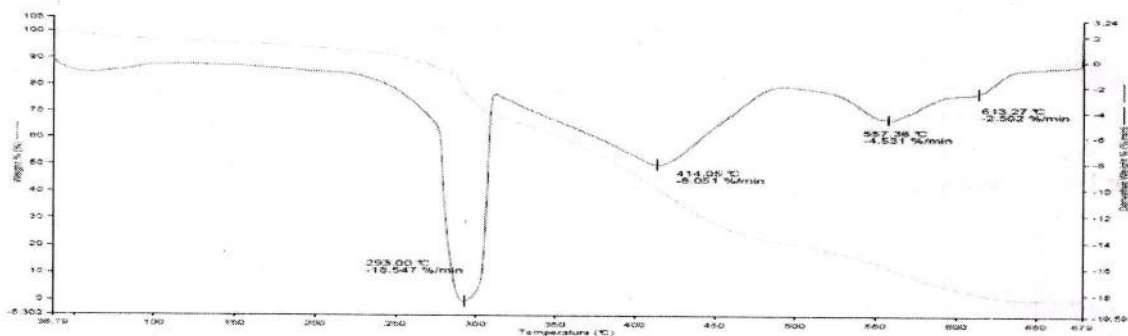


Fig. (4): TG-DTA curve of Zn(II) complex of chalcone of pyridine-2-carbaldehyde

F. X-Ray Diffraction Spectral Studies Of Metal Complex Of Zn(II) Complex Of Chalcone Of Pyridine-2-Carbaldehyde

The XRD spectral study has been done at SAIF, Cochin, Kerala. The X-ray diffraction patterns of Zn (II) complex is shown in (Fig .5). The standard deviation observed is within the permissible range. [14] The observed and calculated densities of Zn(II) complex of chalcone of pyridine-2-carbaldehyde are  $1.909 \text{ g cm}^{-3}$  and  $1.816 \text{ g cm}^{-3}$  respectively. Chalcone of pyridine-2-carbaldehyde was of Zn(II) complex is found to be tetragonal lattice type with space group P2/m and lattice parameters are  $a (\text{Å}) = 4.9162$   $b (\text{Å}) = 4.9162$   $c (\text{Å}) = 5.4089$   $\alpha = 90^\circ$   $\beta = 90^\circ$   $\gamma = 90^\circ$  unit cell volume (V) =  $328.46 \text{ Å}^3$

1) Unit cell data and crystal lattice parameters for Co(II)

Unit cell data and crystal lattice parameters

$a (\text{Å}) = 4.9162$   $b (\text{Å}) = 4.9162$   $c (\text{Å}) = 5.4089$   $\alpha = 90^\circ$   $\beta = 90^\circ$   $\gamma = 90^\circ$  Volume (V) =  $328.46 \text{ Å}^3$

Density (obs.) =  $1.2196 \text{ g cm}^{-3}$  Density (cal.) =  $1.1265 \text{ g cm}^{-3}$  Z = 4 Crystal system = Tetragonal

Space group = P2/m Standard deviation (%) = 0.027

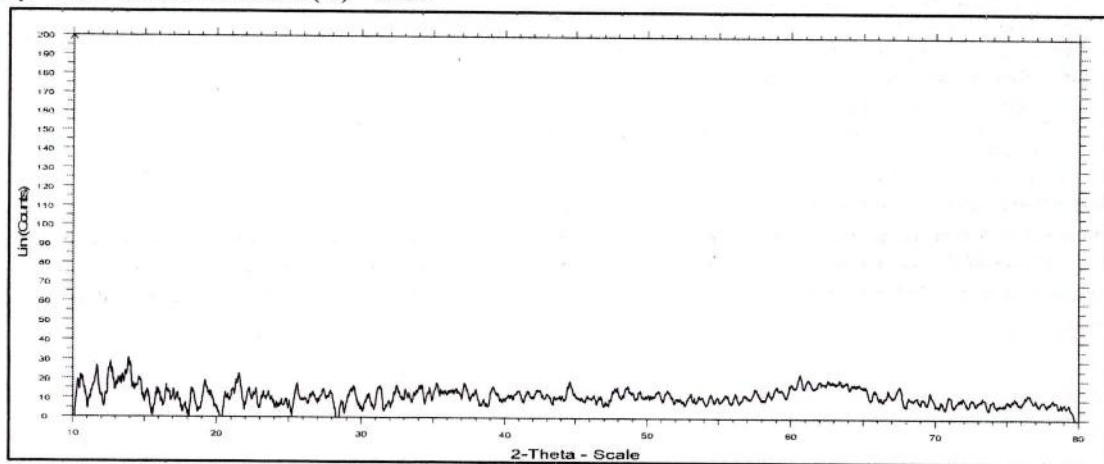


Fig. (5): X-ray diffractogram of Zn(II) complex of chalcone of pyridine-2-carbaldehyde

G. Antimicrobial Activity

Antimicrobial activity was assayed by cup plate agar diffusion method [15] by measuring inhibition zones in mm. In vitro antimicrobial activity of all synthesized compounds and standard have been evaluated against strains of The fungal toxicity of Zn(II) complex was studied in vitro against *Aspergillus niger* ATCC 16404, *Saccharomyces cerevisiae* ATCC 9763, *Candida albicans* ATCC10231 fungal pathogens at fixed 1% concentration. The antibacterial activity of Zn(II) complex was studied, for evaluating antibacterial activity Gram positive and Gram negative bacterial pathogens were used. *Staphylococcus aureus* ATCC 6538, *Bacillus megaterium* ATCC 2326, *Bacillus subtilis* ATCC 6633 were Gram positive pathogens used in this study. *Escherichia coli* ATCC8739, *Salmonella typhi* ATCC9207, *Shigella boydii* ATCC 12034, *Enterobacter aerogenes* ATCC13048, *Pseudomonas aerogenosa* ATCC9027, *Salmonella abony* NCTC6017 were the Gram-negative pathogens used in this stud. From the results of antimicrobial activity of ligands and complexe it is clear that the Zn(II) complex shows enhanced activity than ligand. The increase in antimicrobial activity is due to faster diffusion of metal complex as a whole through the cell membrane or due to the combined activity of the metal and ligand.





#### IV. CONCLUSION

The Zn (II) complex was colored, insoluble in most of the organic solvent but soluble in organic solvent. The stoichiometry ratio of the metal complexes obtained has been found to be 1:2. Solution conductivity of this metal complex reveals nonelectrolytic nature. The infrared spectral data indicate that the ligand act as mononegative bidentate species towards Zn(II) complex. The electronic spectral data, IR spectrum, magnetic moment, TG-DTA suggests that Zn(II) has Square planar geometry. The CHO analysis gives C, H, and O percentage in the metal complex. The XRD parameters shows that the structure of Zn (II) is tetragonal and has space group = P2/m.

#### V. ACKNOWLEDGEMENT

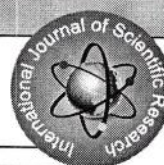
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#### REFERENCES

- [1] S.V.Kostanecki, and Tambar, J.Chem.Ber., 32:1921(1899).
- [2] N.V.Damme, V.Zalisky, A.J.Lough, M.T. Lemaire, Polyhedron, 89, (2015) 155-159
- [3] M.Layek, M. Ghos, S.Sain, M. Fleck, P.T. Pmuthiah, S.J. Jenniefer, J. Ribas, D.Banadyopadhyay, Journal of Molecular Structure, 1036, (2013) 422-426.
- [4] V. N. Patange, B. R. Arbad, Synthesis, spectral, thermal and biological studies of transition metal complexes of 4-hydroxy-3-[3-(4-hydroxyphenyl)-acryloyl]-6-methyl-2H-pyran-2-one, J. Serb. Chem. Soc. 76 (9) (2011) 1237-1246.
- [5] A. J. Deshmukh, R. M. Patil, R. S. Lokhande and D. H. Ner, Synthesis, characterization and antimicrobial activity of copper (II) complexes of some 2-hydroxy-4, 5-dimethyl substituted chalcones, International Journal of Advanced Research and Review, 3(1), 2017, 27-31.
- [6] Habib, S.I., Kulkarni P.A. (2013). Synthesis and antimicrobial activity of some new chalcones of pyridine/pyrrole carboxaldehyde, Scholars Research Library, Der Pharmacia letter, 5(2),101-104
- [7] Habib, S.I., Kulkarni, P.A.(2015). Synthesis and antimicrobial activity of chalcone derivatives of Ni(II) and Co(II) complexes-an in vitro evaluation, ICAIJ,10(4), [152-156].
- [8] Habib, S.I. (2017). Synthetic and structural studies of Fe (II) and Zn(II) complexes of 2'-hydroxy chalcones derived from pyridine-2-carboxaldehyde, pyrrole-2-carboxaldehyde. Der Pharma Chemica, 9(10):80-83.
- [9] Chiara Sulpizio, Simon T.R., Muller, Qi Zhang, Lothar Brecker, Annette Rompel. (2016). Synthesis, characterization, and antioxidant activity of Zn<sup>+2</sup> and Cu<sup>+2</sup> coordinated polyhydroxy complexes, Monatsh Chem. 147:1871-1881; DOI 10.1007/s00706-016-1822-7.
- [10] R.A. Shastri, Synthesis and antimicrobial activity of some novel pyrazoline and flavones derivatives derived from furan chalcone; World Journal of pharmaceutical Research, 4 June 2015, vol 4, issue 7, 642-651.
- [11] S.N. Ipper, N.K. Raut and G.K. Kakde, Physico-chemical properties of furan chalcone. Ind. J. Appl. Res. 2017; 7(6):683-684.
- [12] Sirsat, S.B., Halikar, N.K. Pund, M.M. and Vartale, S.P. (April/June 2012). Synthesis and biological screening of some novel hetero-aryl-chalcone and their Complexes. Research Journal of Pharmaceutical Biological and Chemical Sciences. Vol.3 issue 2, 240-248.
- [13] P.S.Mane, S.G.Shirodkar, and T.K.Chondhekar, J. Indian Chem. Soc., 79(A), 154 (2002).
- [14] Salima Tabti, A. Djedouani, D. Aggoun, I. Warad, S. Rahmouni, S. Romdhane, H. Fouzi; New Cu(II), Co(II) and Ni(II) complexes of chalcone derivatives: Synthesis X-ray crystal structure, Electrochemical properties and DFT computational studies; Journal of Molecular Structure, 1155(2018) 11-20.
- [15] A. L. Barry, Procedure and theoretical consideration for testing antimicrobial agents in agar media, 5<sup>th</sup> Edition, William Wilkins Baltimore (1991).



## SYNTHESIS, SPECTRAL STUDY, CHARACTERIZATION AND ANTIMICROBIAL ACTIVITY OF COPPER (II) COMPLEX OF CHALCONE OF 5-NITROFURFURAL



## Chemistry

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## ABSTRACT

A metal complex of Cu(II) has been synthesized with newly prepared biologically active ligand. This ligand was prepared by the Claisen-Schmidt condensation method of 2,6-dihydroxy acetophenone and 5-Nitrofurfural. The structure of the complex has been proposed by the analytical data, conductivity measurement, magnetic moment, electronic absorption spectrum, thermal studies and XRD analysis. Analytical data confirmed 1:2 stoichiometry and the magnetic moment, TG-DTA suggests that Cu(II) complex has octahedral geometry. Presence of coordinated water molecules in Cu(II) complex is confirmed by TG-DTA studies. The conductivity data show that the complex was non electrolyte. Antimicrobial activities of complex with selected bacterial strain and fungal strain carried out and the results have been compared with commercial standards. The metal complex exhibit impressive potent biocidal activity than ligand.

## KEYWORDS

TG-DTA, XRD study, Antimicrobial activities, Physico-chemical property, Magnetic susceptibility and Conductivity.

## 1. INTRODUCTION

Chalcones constitute an important group of natural products, chemically they consist of open chain flavonoid in which the two aromatic rings are joined by  $\alpha, \beta$  unsaturated carbonyl system. The name chalcone is given by Kostanecki and Tambar [1]. Many complexes of chalcones are synthesized and studied in the literature [2]. It is believed that the ( $>CO-C=C<$ ), moiety imparts biological characteristics to this class of compounds. Such  $\alpha, \beta$ -unsaturated carbonyl compounds and their metal complexes possess interesting biochemical properties, such as antitumor, antioxidant, anti-fungal and antimicrobial activities [3]. The magnetic moment, TG-DTA supports the octahedral geometry of the metal complex of chalcone. All crystals of a substance possess the same elements of symmetry. The computer program, used for indexing data was powder-X. In this program all the essential features of X-ray program are presented and in addition it calculates the deviation in lattice parameter a, b, c in Å and  $\alpha, \beta$  and  $\gamma$  in degree and minutes with better combination of h, k, l values until the final deviation is within the permissible limit of 2%. The refined values also give the volume of unit cell. [4]. The X-ray powder diffractogram of the metal complexes were used for the structural characterization and determination of lattice dimensions.

## 2. MATERIALS AND METHODS

## 2.1 Synthesis of chalcone of 5-Nitrofurfural

The reagents used for preparation of chalcone of 5-Nitrofurfural are of A.R. grade. Concentrated Sulphuric acid (1.4 mL) was added dropwise to 5-Nitrofurfural (0.01) dissolved in glacial acetic acid (30mL) then 2,6-dihydroxy acetophenone (0.01 mol) was added and the reaction mixture was stirred for 10 hours at room temperature. The progress of the reaction was monitored by TL, ice cold water (100 mL) was added and the precipitate was collected by filtration and washed carefully with water and cold ethanol. The pure product was obtained by recrystallization in anhydrous ethanol [5].

## 2.2 Synthesis of metal complex

The solution of 0.02 mole of chalcone of 5-Nitrofurfural was taken in round bottom flask containing 30 ml of anhydrous methanolic solution

Table no. (1): CHO analysis

Metal complexes	Chemical formula	Mol. Wt.	Elemental analysis : % found (calculated)						
			C	H	N	O	S	X(Br)	M
Cu (II) complex	$[C_{26}H_{20}O_{14}N_2Cu]$	647	48.23 (48.19)	3.14 (3.11)	4.37(4.32)	34.60 (34.56)	-	-	10.00 (9.80)

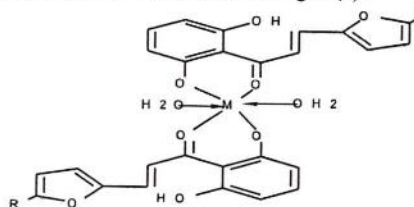
## 3.3 Magnetic susceptibility, solution conductivity and electronic absorption spectral data

## Magnetic susceptibility

The observed magnetic moment values of octahedral Cu(II) complexes fall in the range 1.8 to 2.1 B.M. These values are in good agreement with the moment reported for mononuclear high spin octahedral Cu(II) complexes by earlier workers [8].

and boiled for 10 minutes. A hot solution of 0.01 mole, of Copper Sulphate in 20ml of methanol was added drop wise to the solution of the chalcone of 5-Nitrofurfural. To this reaction mixture, 10% alcoholic ammonia was added up to slightly alkaline pH. The complex was precipitated at 8 pH range. The pH 8-10 range was definite for these complexes [6].

The content was stirred on magnetic stirrer for one hour. The solid metal complex separated out and washed with methanol three to four times. Dried in vacuum desiccators over anhydrous granular calcium chloride. The melting point/decomposition temperature of the complex was determined by Thiele's melting apparatus. The reactions of formation of Cu(II) complex is shown in figure (1).

Fig.(1): Metal complex of Copper (II) with chalcone of 5-Nitrofurfural R = -NO<sub>2</sub>, M = Cu(II)

## 3. RESULTS AND DISCUSSION

## 3.1 Physical parameters

Metal complex of Copper(II) with chalcone of 5-Nitrofurfural was reddish brown in color. The complex was precipitated at 8 pH range, having Melting point 290°C. The complex is insoluble in water and soluble in DMSO, DMF [7].

## 3.2 CHO analysis

The carbon, hydrogen, oxygen, and copper metals percentage in Cu(II) complex of chalcone measured at SAIF Cochin, Kerala. The calculated and measured values of CHO analysis are matching and are given in the table no.(1).

## Solution conductivity and electronic absorption spectral data

The solution conductivities of  $10^{-3}$  M solution of metal complex in DMSO was measured on EQUIPTRONICS digital conductivity meter EQ-660 with  $20 \mu\Omega$  to  $200 \mu\Omega$  at 298K temperature. The complex was insoluble in water and soluble in DMSO, DMF. The low conductivity values in DMSO solution ( $10^{-3}$  M) which indicates non-electrolytic nature.



The Solution conductivity Cu(II) complex was (4.81), The magnetic moment of Cu(II) complex was(1.93 BM) and The electronic absorption spectra of Cu(II) complex was showed two bands at 25,906(386)  $^3B_{1g} \rightarrow ^2E_g$  and 38759 (258)  $cm^{-1}(nm)$  Charge Transfer band.

In the present investigation, Cu(II) show UV transition band in the range 24154 to 26560  $cm^{-1}$  which is attributed to  $^3B_{1g} \rightarrow ^2E_g$  and charge transfer band observed in the range 35026-39370  $cm^{-1}$  indicating distorted octahedral geometry around the Cu(II) ion[9-10].

### 3.4 Thermal analysis Cu(II) complex chalcone of 5-Nitrofurural:

The simultaneous thermogravimetric, differential thermal analysis of Cu(II) complex chalcone of 5-Nitrofurural was performed in an inert nitrogen atmosphere on Perkin Elmer STA 6000 at SAIF, Cochin, Kerala. The heating rate was 10 $^{\circ}$ /min and flow rate of nitrogen 50 ml/min. The reference substance used was  $\alpha$  Al<sub>2</sub>O<sub>3</sub> in platinum crucible and sample weighed in the range of 4-12 mg. The thermogram of Cu(II) complex chalcone of 5-Nitrofurural is presented in figure(2). This curve reveals that there is presence of lattice as well as coordinated water in the complex.

The thermogravimetric analysis shows that, the first weight loss at 89.23 $^{\circ}$ C indicating the presence of lattice water with weight reduction 7.69 % (calc. wt. loss 6.50%). The second loss due to coordinated water molecule in the complex takes place at 136.93 $^{\circ}$ C. In the third step loss of 10.51% (calc. wt. loss 9.00%) to the temperature in the range 200-300 $^{\circ}$ C. This is supported by an endothermic peak in DTA at 246.98 $^{\circ}$ C due to the two -NO<sub>2</sub> groups in the ligand. The fourth step covering the reaction interval 350 to 370 $^{\circ}$ C with a considerable weight loss of 26.35% (calc. wt. loss 26%) supported by a sharp endothermic peak at 355.41 $^{\circ}$ C in DTA plot may be due to the decomposition of coordinated part of the complex. The fifth step covering the reaction interval 360-420 $^{\circ}$ C with 10% mass reduction corresponds to a loss of coordinated part of the complex. Beyond that residue attained almost constant weight loss of 12.29% corresponding to formation of CuO as a residue, final product.

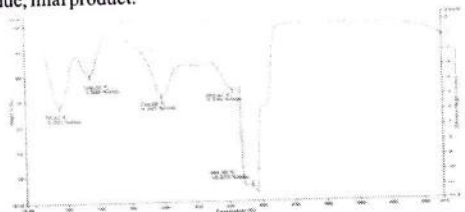


Fig. (2): TG-DTA curve of Cu(II) complex of chalcone of 5-Nitrofurural

### 3.5 X-ray diffraction spectral studies of metal complex of Cu(II) complex of chalcone of 5-Nitrofurural

The XRD spectral study has been done at SAIF, Cochin, Kerala. The standard deviation observed was within the permissible range. The observed density was 1.2919  $gcm^{-3}$  and calculated densities was 1.3567  $gcm^{-3}$  respectively. The Cu(II) complex of was triclinic lattice type P. For these complex lattice parameters are and  $a=7.9168 \text{ \AA}$ ,  $b=8.9168 \text{ \AA}$ ,  $c=10.8432 \text{ \AA}$ ,  $\alpha=82^{\circ}$ ,  $\beta=79^{\circ}$ ,  $\gamma=89^{\circ}$ ,  $V=738.78 \text{ \AA}^3$  respectively, and Standard deviation was found to be 0.14%.

#### Unit cell data and crystal lattice parameters for Cu(II):

Unit cell data and crystal lattice parameters  $a (\text{ \AA}) = 7.9168$   $b (\text{ \AA}) = 8.9168$   $c (\text{ \AA}) = 10.8432$   $\alpha = 82^{\circ}$   $\beta = 79^{\circ}$   $\gamma = 89^{\circ}$  Standard deviation (%) = 0.14 Volume ( $V$ ) = 738.78  $\text{ \AA}^3$  Density (obs.) = 1.2919  $gcm^{-3}$  Density (cal.) = 1.3567  $gcm^{-3}$   $Z = 2$  Crystal system= Triclinic Space group = P2/m

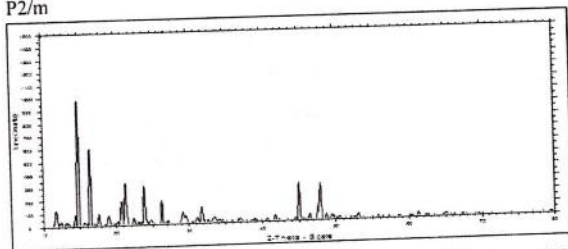


Fig. (3): X-ray diffractogram of Cu(II) complex of chalcone of 5-Nitrofurural

### 3.6 Antimicrobial activity

Antimicrobial activity was assayed by cup plate agar diffusion method by measuring inhibition zones in mm [11]. In vitro antimicrobial activity of all synthesized compounds and standard have been evaluated against strains of the fungal toxicity of Cu(II) complex was studied *in vitro* against *Aspergillus niger* ATCC16404, *Saccharomyces cerevisiae* ATCC9763, *Candida albicans* ATCC10231 fungal pathogens at fixed 1% concentration.

The antibacterial activity of Cu(II) complex was studied, for evaluating antibacterial activity Gram positive and Gram negative bacterial pathogens were used. *Staphylococcus aureus* ATCC 6538, *Bacillus megaterium* ATCC 2326, *Bacillus subtilis* ATCC 6633 were Gram positive pathogens used in this study. *Escherichia coli* ATCC8739, *Salmonella typhi* ATCC9207, *Shigella boydii* ATCC 12034, *Enterobacter aerogenes* ATCC13048, *Pseudomonas aerogenosa* ATCC9027, *Salmonella abony* NCTC6017 were the Gram-negative pathogens used in this study.

From the results of antimicrobial activity of ligands and complex it is clear that the complex shows enhanced activity than ligand[12]. The increase in antimicrobial activity is due to faster diffusion of metal complex as a whole through the cell membrane or due to the combined activity of the metal and ligand[13].

### CONCLUSION

The Cu(II) complex was reddish brown in color, insoluble in most of the organic solvent but soluble in organic solvent. The stoichiometry ratio of the metal complex obtained has been found to be 1:2. Solution conductivity of this metal complex reveals nonelectrolytic nature. The electronic spectral data, magnetic moment, TG-DTA suggests that Cu(II) has Octahedral geometry. The CHO analysis gives C, H, and O percentage in the metal complex. The XRD parameters shows that the structure of Cu (II) is triclinic and has space group = P2/m.

### Acknowledgement

Authors are thankful to Principal of Sunderrao Solanke Mahavidyalaya, Majalgaon Dist. Beed (India) for providing laboratory facilities.

### REFERENCES

- [1] S.V.Kostaneccki, and Tambar, J.Chem.Ber., 32:1921(1899).
- [2] N.V.Damme, V.Zalisky, A.J.Lough, M.T. Lemaire, Polyhedron, 89, (2015) 155-159.
- [3] V. N. Patange, B. R. Arbad, Synthesis, spectral, thermal and biological studies of transition metal complexes of 4-hydroxy-3-[3-(4-hydroxyphenyl)-acryloyl]-6-methyl-2H-pyran-2-one, J. Serb. Chem. Soc. 76 (9) (2011) 1237-1246.
- [4] A. J. Deshmukh, R. M. Patil, R. S. Lokhande and D. H. Ner, Synthesis, characterization and antimicrobial activity of copper (II) complexes of some 2-hydroxy-4, 5-dimethyl substituted chalcones, International Journal of Advanced Research and Review, 3(1), 2017, 27-31.
- [5] Hong jin, Yucong Geng, Zhiyi Y.C, Ke Jao, Jaiping Hou. (2009). Pesticide Biochemistry and Physiology, 93133-137.
- [6] Habib, S.I., Kulkarni, P.A.(2015). Synthesis and antimicrobial activity of chalcone derivatives of Ni(II) and Co(II) complexes-an in vitro evaluation, ICAU,10(4), [152-156].
- [7] S.N. Ipper, N.K. Raut and G.K. Kakde, Physico-chemical properties of furan chalcone. Indian Journal of Applied Research. 2017; 7(6):683-684.
- [8] Sirsat, S.B., Halikar, N.K. Pund, M.M. and Vartale, S.P. (April/June 2012). Synthesis and biological screening of some novel hetero-aryl-chalcone and their Complexes. Research Journal of Pharmaceutical Biological and Chemical Sciences. Vol.3 issue 2, 240-248.
- [9] Hong-Yun, Z., Dong-Li, C., Pei-Kun, C., De-Ji, C., Guang-Xia, C., Hong-Quan, Z., (1992). Polyhedron, 11, 2313.
- [10] Reddy, Vidyavati, Patil, N. and Angadi S.D. (2008 July). Synthesis, characterization and antimicrobial activity of Cu(II), Co(II) and Ni(II) complexes with O,N and S donor ligands; E-Journal of Chemistry Vol. 5, No.3, 577-583.
- [11] A. L. Barry, Procedure and theoretical consideration for testing antimicrobial agents in agar media, 5th Edition, William Wilkins Baltimore (1991).
- [12] S. I. Habib, S. G. Konda, M. A. Basser, P. A. Kulkarni, Asian J. Research Chem., Oct-Dec, 2(4), (2009).
- [13] Sengupta, S. K., Pandey, B. K., Shrivastava, B. K., and Sharma, V. K., (1998). Transition Metal Chem., 23, 4, 349-353.



# Synthesis, spectral Characterization and Antimicrobial Activity of 2-(furan-2-yl)-5-hydroxy-4H-chromen-4-one

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## Abstract

Flavonoids are also known as plant pigments or co-pigments. Flavonoids are the largest group of naturally occurring phenolic compounds which occur in different plant parts both in a free state and as glycosides. 2-(furan-2-yl)-5-hydroxy-4H-chromen-4-one was synthesized by Claisen-Schmidt condensation method from 2,6-dihydroxy acetophenone and 2-furaldehyde to gives chalcone and which on oxidation with DMSO/I<sub>2</sub>. Spectroscopic characterization using UV-visible, IR, <sup>1</sup>H NMR, Mass spectra, properties and antimicrobial activity has been investigated for 2-(furan-2-yl)-5-hydroxy-4H-chromen-4-one. The IR bands for Carbonyl group (-C=O in pyron ring) for 2-(furan-2-yl)-5-hydroxy-4H-chromen-4-one appear at 1605 per cm which agrees with the general range of flavones. The 2-(furan-2-yl)-5-hydroxy-4H-chromen-4-one has two matching bands at 245 nm and other at 360 nm which lies in the range of flavone. In the mass spectrum of 2-(furan-2-yl)-5-hydroxy-4H-chromen-4-one molecular ion peak is observed m/z and calculated m/z corresponding to [M<sup>+</sup>] peaks is in good agreement with their structure. The flavone shows moderate to good Antibacterial and Antifungal activity.

**Keywords:** 2-(furan-2-yl)-5-hydroxy-4H-chromen-4-one, UV-visible, IR, <sup>1</sup>H NMR, Mass spectrum, CHO analysis and Antimicrobial Activity.

## 1. Introduction

The flavonoids or bioflavonoids are a ubiquitous group of polyphenolic substances which are present in most plants. They also occur as glycosides. Chemically, flavonoids show a fifteen-carbon skeleton (C6-C3-C6) which consists of two phenyl rings connected by a three carbon bridge. Flavonoids are found to occur in different parts of the plant roots, barks, wood, leaves, flowers, fruits, and seeds. Flavonoids are synthesized in all parts of the plants. They play role in providing color, fragrance and taste to the fruits, flowers and seeds, which makes them



attractants for insects, birds or mammals, which aid in pollen or seed transmission.<sup>1</sup> Flavonoids lead to potent antioxidant activity, the most important function of flavonoids to scavenge hydroxyl radicals, superoxide anions and lipid peroxy radicals. Multiple combinations of hydroxyl groups, sugar, oxygen and methyl groups attached to these structures create the various classes of flavonoids, flavonols, flavonones, flavones, flavon-3-ols (catechins), anthocynins and isoflavones.<sup>2</sup> Chalcone is an inter-mediate compound in the biosynthesis of flavonoids, which are the substances widespread in plants and with an array of biological activities. Several therapeutically interesting biological activities of certain flavonoids have been reported including antibacterial,<sup>3</sup> antiviral, anti-inflammatory, antiallergic, antithrombotic,<sup>4</sup> antimutagenic, antineoplastic, neuroprotective properties,<sup>5</sup> and antioxidant properties, etc.

## 2. Materials and Methods

The chalcone of 2-furaldehyde was synthesized by Claisen-Schmidt condensation of 2,6-dihydroxyacetophenone (0.01 mol) and 2-furaldehyde (0.01 mol). Which on oxidation with DMSO/I<sub>2</sub> it gives flavones. The chemicals used for this synthesis are of AR grade.

### Synthesis of Chalcone:

A mixture of 2,6-dihydroxy acetophenone (0.01 mol) and 2-furaldehyde (0.01 mol) are dissolved in ethanol (20 mL) and then solution of potassium hydroxide 10 mL (15%) were added to it. The mixture was stirred for overnight. The progress of the reaction was monitored by TLC. It was then poured on ice cold water and acidified with dilute HCl. The coffee brown solid was precipitates, filtered and washed with water and recrystallized from ethanol.<sup>6</sup>

### Synthesis of Flavone:

The chalcone of 2-furaldehyde was dissolved in 20 ml DMSO, to this catalytic quantity of iodine was added. Contents were refluxed for one hour, the progress of the reaction was monitored by TLC and the reaction mixture was left overnight. It was then poured on ice cold water, the separated solid was filtered washed with cold water followed by a dilute sodium-thiosulphate solution. The product was crystallized from ethanol.<sup>7-8</sup>

The melting point of the flavone was determined by an open capillary tube and is unconfirmed. Infra red spectrum was measured using FT-IR spectrophotometer, UV-visible spectrum measured on SL 159 single beam UV-VIS spectrophotometer, <sup>1</sup>H NMR measured on Bruker AVANCE II 400 MHz Spectrophotometer and Mass spectrum was measured on Mass spectrophotometer. The purity the compound was checked by TLC plate which were precoated with silica gel using solvent ethyl acetate and petroleum ether (3:7). The reaction for formation of flavone is given figure (1).

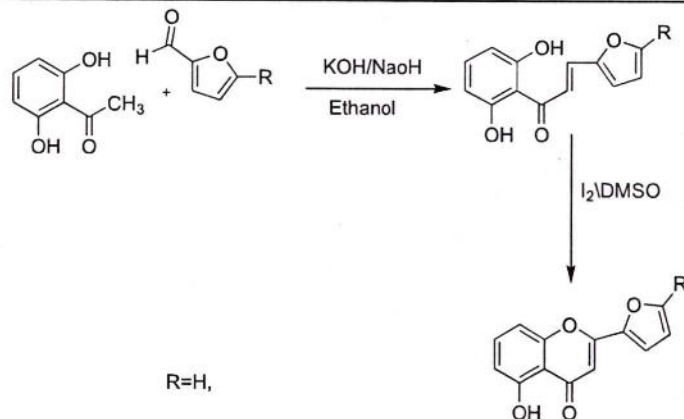


Fig.(1): Reaction of formation of Flavone.

### 3. Results and discussion

#### 3.1 Properties

The flavone having IUPAC name 2-(furan-2-yl)-5-hydroxy-4H-chromen-4-one was synthesized by Claisen-Schmidt condensation method and its structure is stable at room temperature, insoluble in water and is soluble in organic solvent (ethyl alcohol). The physico-chemical properties of flavone and CHO analysis by calculation method are given in table no. (1). The reaction of formation of flavone is shown in figure (1). Elemental analysis showed that the percentage of the Oxygen, hydrogen and carbon found experimentally is equivalent to the calculated values for this compound. The chemical structure of  $C_{13}H_8O_4$  is shown in figure (2).

Table no. (1): The properties of flavone and Elemental analysis data.

Mol. Formula	Color	Mol. Wt.	M.P °c	Found (Calculated) %			
				C	H	O	N
$[C_{13}H_8O_4]$	Coffee brown	228	272	68.40 (68.42)	3.49 (3.53)	28.00 (28.04)	---



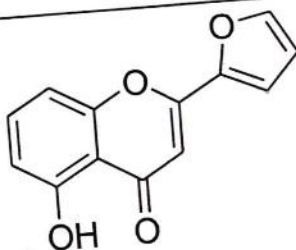


Fig.(2): Chemical structure of  $C_{13}H_8O_4$

### 3.2 Infra red spectrum

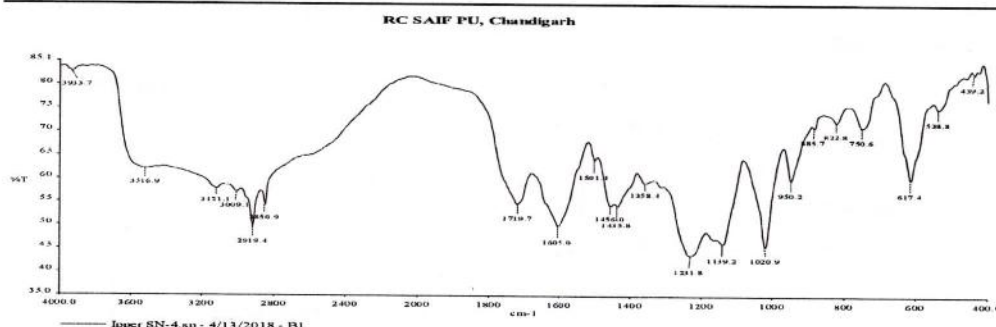
The IR spectrum of  $\alpha, \beta$ -unsaturated carbonyl group has characteristic bands of chalcone at prominent bands between 1625 to 1650 per cm.

The characteristic peaks in infra red spectrum give the presence of particular functional group.<sup>9</sup> The region at which other absorption bands appear depends on the type of aromatic / hetero-aromatic rings as well as the substituent present on these rings. The infrared spectrum of chalcone of flavone was recorded on a Perkin- Elmer Spectrum RX-IFTIR Spectrophotometer in the range 4000-400  $cm^{-1}$  using potassium bromide pellet at CIL, Chandigarh, Punjab. The IR bands for 2-(furan-2-yl)-5-hydroxy-4H-chromen-4-one appears at 1605 per cm which agrees with the general range of flavone. The change in the position of a band is observed due to change in stretching vibration mode of bond involving coordinated atom. The stretching frequency of flavone is represented in table number (2) and the IR spectrum in figure (3).

Table no. (2): The stretching frequency of flavone

Molecule	$\nu(OH)$ Enolic	(-CO-CH=C H-) $\alpha, \beta$ -unsaturated carbonyl group	Carbonyl group (-C=O in pyron ring)	(C-O-C) Stretching Frequency	(C=C) Stretching Frequency	Aromatic Ring (C=C) Stretching Frequency	Ar-H Stretching Frequency	-NO <sub>2</sub> stretching frequency
[ $C_{13}H_8O_4$	3516	-	1605	1232	1502	1456	3121	-

Fig.(3): IR spectrum of 2-(furan-2-yl)-5-hydroxy-4H-chromen-4-one



### 3.3 UV-visible spectrum

Generally, the flavones and flavonols exhibit absorption in 320-380 nm regions (band I) and 240-270 nm regions (band II). The UV-visible spectrum of 2-(furan-2-yl)-5-hydroxy-4H-chromen-4-one measured on SL 159 single beam UV-VIS spectrophotometer. The UV-visible spectrum of flavone is given in figure (4). In 2-(furan-2-yl)-5-hydroxy-4H-chromen-4-one has two matching bands at 245 nm and other at 360 nm which lies in the range of flavone.

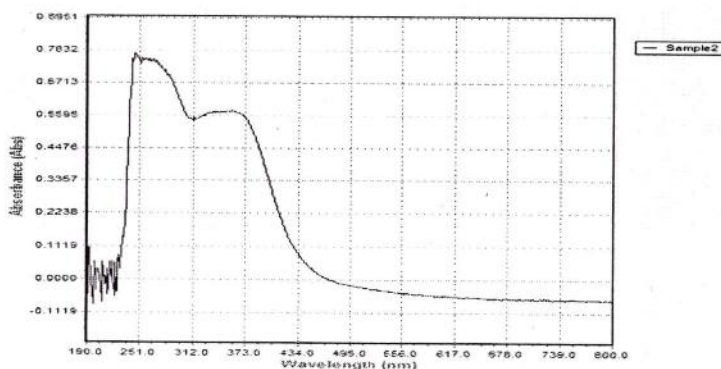


Fig.(4): UV-visible spectrum of 2-(furan-2-yl)-5-hydroxy-4H-chromen-4-one

### 3.4 <sup>1</sup>H NMR spectral study

In the analysis of organic molecule, <sup>1</sup>H NMR spectra plays very important role. It is the most valuable technique in structural investigation.<sup>10</sup>

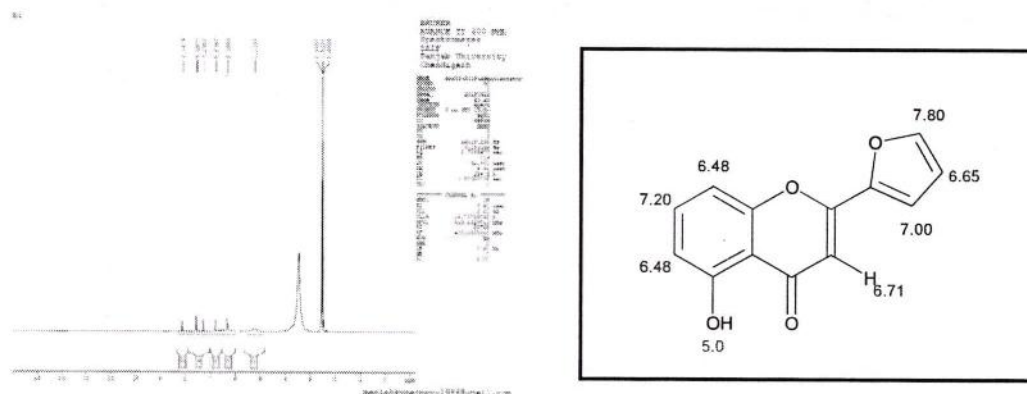
The <sup>1</sup>H NMR spectrum of chalcone of 5-nitro-furfural is recorded on Bruker AVANCE II 400 MHz Spectrophotometer in DMSO solvent using TMS as an internal standard at SAIF,



Chandigarh, Punjab are shown in figure (5), and spectral data in table no. (3) and Chemical shift ( $\delta$ ) ppm in figure (5)

Table no. (3):  $^1\text{H}$  Nuclear magnetic resonance spectral data of chalcone of 5-nitro-furfural

Chemical Shift ( $\delta$ ) ppm	Number of Protons	Multiplicity (Splitting)	Assignment
6.48-7.20	3H	m	Aromatic protons
5.0	1H	s	-OH group present on the aromatic benzene ring
6.71	1H	S	$\alpha$ -H on-unsaturated carbonyl system
6.65-7.80	3H	m	Protons on furan ring



**Fig.(5):** <sup>1</sup>H Nuclear Magnetic Resonance Spectrum of 2-(furan-2-yl)-5-hydroxy-4H-chromen-4-one and Chemical shift ( $\delta$ ) ppm of protons in PMR spectrum.

### 3.5 Mass spectrum

Mass spectroscopy is the most accurate technique for the determination of molecular weight of compound. In this technique matter is bombarded with highly energetic electrons. Then matter absorbs or ejects electrons, from it. When it ejects electrons charged species are formed. Anionic and cationic radicals are detected by detector. Detector never detects radical and neutral species. The mass spectrum is a plot representing the  $m/e$  values of various ions against their relative percent intensity. The highest intense peak in the spectrum is called base peak. The intensity of other peaks is shown relative to the base peak. The peak at extreme right corresponds to the molecular weight of the original molecule. The molecular ion is called parent ion and usually denoted as  $[M]^+$  ion. In mass spectrum peaks are also noticed. Mass spectroscopy also separates the isotopes. The mass spectrum 2-(furan-2-yl)-5-hydroxy-4H-chromen-4-one was recorded on Waters, Q-TOF Micro Mass (LC-MS).at SAIF, Chandigarh, Punjab.

The mass spectrum of chalcone of 2-(furan-2-yl)-5-hydroxy-4H-chromen-4-one is represented in figure (6) and molecular mass. In the mass spectrum of 2-(furan-2-yl)-5-hydroxy-4H-chromen-4-one, molecular ion peak is observed  $m/z$  and calculated  $m/z$  corresponding to  $[M]^+$  peak are in good agreement with their structure.



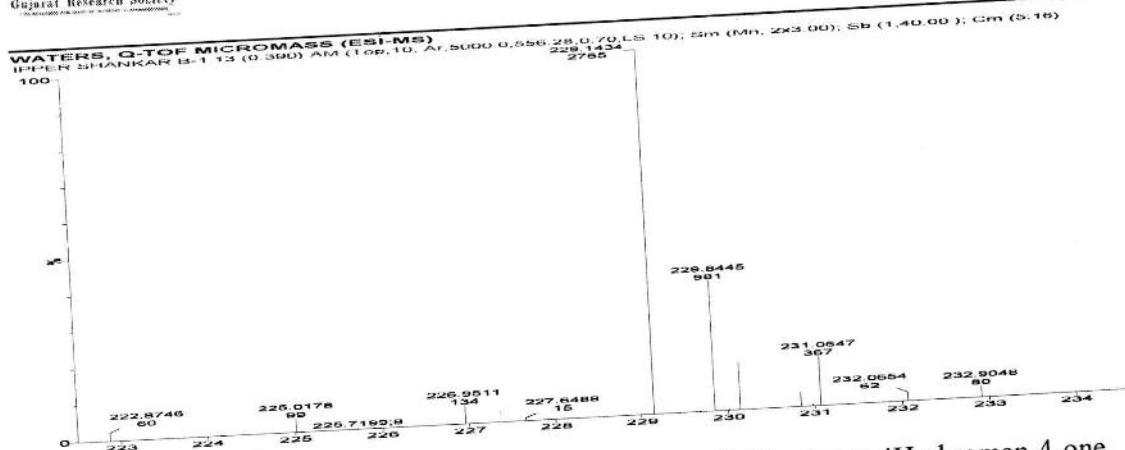


Fig.(6): Mass spectrum of chalcone of 2-(furan-2-yl)-5-hydroxy-4H-chromen-4-one.

Table no. (4): Antifungal Activity of 2-(furan-2-yl)-5-hydroxy-4H-chromen-4-one

Ligands	Antifungal Growth		
	<i>Aspergillus niger</i> ATCC 16404	<i>Saccharomyces cerevisiae</i> ATCC 9763	<i>Candida albicans</i> ATCC10231
	1%	1%	1%
Flavone	12	13	12
Standard	26	24	34

Table no. (5): Antibacterial Activity of 2-(furan-2-yl)-5-hydroxy-4H-chromen-4-one

Ligands	Antibacterial Growth Diameter of inhibition zone (mm)									
	<i>S.typhi</i> ATCC 9207	<i>E.aerogenes</i> ATCC1 3048	<i>B.subtilis</i> ATC C 6633	<i>P.aerogenosa</i> ATCC9 027	<i>S.abony</i> NCTC 6017	<i>B.megaterium</i> ATCC 2326	<i>E.Coli</i> ATCC 8739	<i>S.aureus</i> ATC C 6538	<i>S.boydii</i> ATC C 1203 4	<i>B.cereus</i> ATC C 1457 9
	1%	1%	1%	1%	1%	1%	1%	1%	1%	1%
Flavone	08	12	10	14	13	14	15	14	13	14
Std	27	33	34	33	30	32	29	25	27	33

The flavone was screened *in vitro* for antifungal activity. The fungal toxicity of flavone was studied *in vitro* against *Aspergillus niger* ATCC 16404, *Saccharomyces cerevisiae* ATCC 9763, *Candida albicans* ATCC10231 fungal pathogens at fixed 1% concentration.

The flavone and was screened *in vitro* for antibacterial activity. The antibacterial activity of flavone was studied, for evaluating antibacterial activity Gram positive and Gram negative bacterial pathogens were used. *Staphylococcus aureus* ATCC 6538, *Bacillus megaterium* ATCC 2326, *Bacillus subtilis* ATCC 6633 were Gram positive pathogens used in this study. *Escherichia coli* ATCC8739, *Salmonella typhi* ATCC9207, *Shigella boydii* ATCC 12034, *Enterobacter aerogenes* ATCC13048, *Pseudomonas aerogenosa* ATCC9027, *Salmonella abony* NCTC6017 were the Gram-negative pathogens used in this study.<sup>11-13</sup>

#### 4. Conclusion

The chalcone of 2-(furan-2-yl)-5-hydroxy-4H-chromen-4-one was synthesized by Claisen-Schmidt condensation method. The IUPAC name of this compound is 2-(furan-2-yl)-5-hydroxy-4H-chromen-4-one. The Chemical shift ( $\delta$ ) ppm from <sup>1</sup>H NMR spectral study is between 5.0 - 7.86. The mass spectrum of this chalcone shows molecular ion peak observed m/z and calculated m/z for [M<sup>+</sup>], peak is in good agreement. The elemental analysis gives percentage of CHO in chalcone of 5-nitro-furfural.



**Acknowledgement** Authors are thankful to Principal, Sunderrao Solanke Mahavidyalaya, Majalgaon, Dist. Beed (India) for providing laboratory facilities.

### References

- [1] Koes, R.E. Quattrocchio, F. (1994). The flavonoid biosynthetic pathway in plants Function and Evolution BioEssays, 16, 123-132.
- [2] Gupta, S. Kumar, N. Kumar S. Sharma, P.K. int. jou. Therapeutic application, Vol 7, 1012, 1-8.
- [3] Jadhav, S.B. Bagul, K.R. Bagul, P.R. Gaikwad, K.V. (2008) Oriental Journal of Chemistry, 24, 583-588.
- [4] Ozdemir, A. Zitouni, G.T. Reveal, K.G. and Guven, K. (2007). "Synthesis and antimicrobial activity of 1-(4-aryl-2-thiazolyl)-3-(2-thienyl)-5-aryl-2-pyrazoline derivatives," Eur. J. of Med. Chem, vol.42, issue 3, 403-409.
- [5] Anderson, A.G. (2001). Journal of the American College of Nutrition, 20, 327- 336.
- [6] S.N.Ipper, N.K.Raut, P.P.Dixit and G.K.Kakade, Synthesis, spectral study and properties of (E)-3-(furan-2-yl)-1-(2,6-dihydroxy)prop-2-en-1-one, International Journal of Basic and Applied Research, September 2018, Vol.8, No.9, pp.961-968.
- [7] Shastri, R.A. (2015 4<sup>th</sup> June). Synthesis and antimicrobial activity of some novel pyrazoline and flavones derivatives derived from furan chalcone; World Journal of Pharmaceutical Research, Vol.4, issue7, 642-651.
- [8] E.Sushanti VH, S.Matsjeh, Mustafa and T.D.Wahyuningsih, Improver synthesis of 2,6-dihydroxy-3,4,-Dimethoxy Chalcone by grinding to synthesize 5-hydroxy -3', 4'-Dimethoxy flavones, Indo.J.Chem., 2014, (2), 174-178.
- [9] Y.B.Vibhute, M.A.Baseer, Synthesis and activity of a new series of chalcones as antibacterial agents, Indian Journal of Chemistry, Vol.42B, January 2003, pp.202-205.
- [10] S.N.Ipper, N.K.Raut, P.P.Dixit and G.K.Kakade, Synthesis, spectral study and properties of Pyridine chalcone, IJAIEEM, Volume 7, Issue 8, August-2018, pp.20-24.
- [11] A.G.Rivera, H.A.Mariscal, N.R.Ceronio, L.F.Roa-de la Fuente and C.E.Loboto-Garcia, Synthesis and antiinflammatory activity of three nitro chalcones, Bioorganic and Medicinal Chemistry Letters, 23(2013)5519-5522.

[12] Y.B. Vibhute, M.A. Baseer, Synthesis and activity of a new series of chalcones as antibacterial agents, Indian Journal of Chemistry, Vol.42B, January 2003, pp.202-205.

[13] K.P. Kakde, S.P. Kakde and S.Y. Deshmukh, Synthesis and characterization of some Bromo substituted chalcones by green synthesis way and Aurones 2-Benzylidene -1-Benzofuran-3-one by cyclization method, Vol.4, Issue 1, December 2014, 1591-1597.



# SYNTHESIS, SPECTRAL STUDY, PROPERTIES AND ANTIMICROBIAL ACTIVITY OF (E)-1-(2,6- DIHYDROXYPHENYL)-3-(5- NITROFURAN-2-YL)PROP-2-EN-1-ONE

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## Abstract

The chalcone is an important group of natural product, as the starting materials in the synthesis of various heterocyclic compounds. (E)-1-(2,6-dihydroxyphenyl)-3-(5-nitrofuran-2-yl)prop-2-en-1-one chalcone was synthesized by Claisen-Schmidt condensation method from aromatic aldehyde and ketone. Spectroscopic characterization using UV-visible, IR, <sup>1</sup>H NMR, Mass spectra, properties and chemical tests has been investigated for chalcone of 5-nitro-furfural. From the reaction mechanism it is observed that condensation of aldehyde group of chalcone of 5-nitro-furfural and ketone group of 2,6-dihydroxy acetophenone takes place in the presence of alkali catalyst. The IR bands for chalcone of 5-nitro-furfural appear at 1623 per cm which agrees with the general range of chalcones. The chalcone of 5-nitro-furfural has two matching UV bands at 231nm and other at 311nm which lies in the range of chalcone. In the mass spectrum of chalcone of 5-nitro-furfural, molecular ion peak is observed m/z and calculated m/z corresponding to [M<sup>+</sup>], [M<sup>+</sup>+1] and [M<sup>+</sup>+2] peaks are in good agreement with their structure. The chalcone of 5-nitro-furfural shows moderate to good Antibacterial and Antifungal activity.

**Keywords:** Chalcone of 5-nitro-furfural, UV-visible, IR, <sup>1</sup>H NMR, Mass spectrum, CHO analysis Antimicrobial activity.

## 1. Introduction

Chalcones and Flavones have been used extensively in the synthesis of metal complexes and contributed to greater extent for development of coordination chemistry. Chalcones are a class of  $\alpha$ ,  $\beta$ - unsaturated carbonyl compounds that form the central core for a variety of naturally occurring biological activities. Chalcones attracted interest due to its pharmacological activities.

It showed activity as antioxidant, antibacterial, antifungal, anticancer and antidepressant [1-2]. Chalcones are known as phenyl styryl ketones, benzyl-acetophenones, benzylidene acetophenones or alternatively called  $\beta$ -phenyl acrylophenone [3-4]. Chalcones have many applications as artificial sweeteners, polymerization catalysts, fluorescent agent, brightening agent, heat stabilizer, Vis light, UV light and aging [5-6]. Chalcones are colored compounds because of the presence of the chromophore and auxochromes [7]. There are number of methods for the synthesis of chalcones including the Claisen-Schmidt, Wittig reaction and Friedel-Crafts acylation [8]. In this paper synthesis, properties, chemical tests, Spectroscopic characterization using UV-visible, IR,  $^1\text{H}$  NMR, Mass spectra has been investigated for chalcone of 5-nitro-furfural.

## 2. Materials and Methods

The chalcone of 5-nitro-furfural was synthesized by Claisen-Schmidt condensation of 2,6-dihydroxy acetophenone (0.01 mol) and 5-nitro-2-furaldehyde (0.01 mol). The chemicals used for this synthesis are of AR grade. To synthesize this compound concentrated Sulphuric acid (1.4ml) was added drop wise to 5-nitro-furfural (0.01) dissolved in glacial acetic acid (30ml) then 2,6-dihydroxy acetophenone (0.01 mol) was added and the reaction mixture was stirred for 10 hours at room temperature, ice cold water (100ml) was added, the precipitate was collected by filtration and washed carefully with water and cold ethanol. The pure product was obtained by recrystallization in anhydrous ethanol [9]. The melting point of the chalcone of 5-nitro-furfural was determined by an open capillary tube and is unconfirmed. Infra red spectrum was measured using FT-IR spectrophotometer, UV-visible spectrum measured on SL 159 single beam UV-VIS spectrophotometer,  $^1\text{H}$  NMR measured on Bruker AVANCE II 400 MHz Spectrophotometer and Mass spectrum was measured on Mass spectrophotometer. The purity the compound was checked by TLC plate which were precoated with silica gel using solvent ethyl acetate and petroleum ether (3:7). The reaction mechanism of formation of chalcone of 5-nitro-furfural is given figure (1).

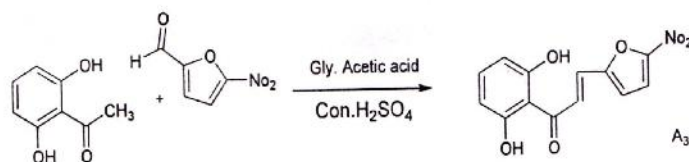


Fig.(1): Reaction of chalcone of 5-nitro-furfural



### 3. Results and discussion

#### 3.1 Properties

The chalcone of 5-nitro-furfural having IUPAC name (E)-1-(2,6-dihydroxyphenyl)-3-(5-nitrofur-2-yl)prop-2-en-1-one was synthesized by Claisen-Schmidt condensation method and its structure is stable at room temperature, insoluble in water and is soluble in organic solvent (ethyl alcohol). The stoichiometry of the compound represented as 1:1, 5-nitro-2-furaldehyde and 2,6-dihydroxy acetophenone ratio.[10] The physico-chemical properties of chalcone of 5-nitro-furfural and CHO analysis by calculation method are given in table no.(1). The completion of reaction was checked by thin layer chromatography, Wilson's test, FeCl<sub>3</sub> test and KMnO<sub>4</sub> test. The reaction between 5-nitro-2-furaldehyde and 2,6-dihydroxy acetophenone is shown in figure (1). From the reaction mechanism it is observed that condensation of aldehyde group of chalcone of 5-nitro-furfural and ketone group of 2,6-dihydroxy acetophenone takes place in the presence of alkali catalyst. Elemental analysis showed that the percentage of the nitrogen, hydrogen and carbon found experimentally is equivalent to the calculated values for this compound. The chemical structure of C<sub>13</sub>H<sub>9</sub>NO<sub>6</sub> is shown in figure (2).

Table no. (1): The properties of chalcone of 5-nitro-furfural and Elemental analysis data.

Mol. Formula	Color	Mol. Wt.	M.P °c	Found (Calculated) %			
				C	H	O	N
[C <sub>13</sub> H <sub>9</sub> NO <sub>6</sub> ]	Orange	244	247	56.71 (56.73)	3.33 (3.30)	34.90 (34.88)	5.09 (5.09)

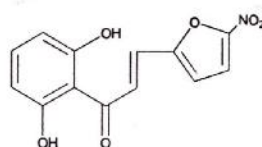


Fig.(2): Chemical structure of C<sub>13</sub>H<sub>9</sub>NO<sub>6</sub>

**3.2 Infra red spectrum** The IR spectrum of  $\alpha$ ,  $\beta$ -unsaturated carbonyl group has characteristic bands of chalcone at prominent bands between 1625 to 1650 per cm<sup>-1</sup>[11].

The characteristic peaks in infra red spectrum give the presence of particular functional group [12]. The region at which other absorption bands appear depends on the type of aromatic / hetero-aromatic rings as well as the substituent present on these rings. The infrared spectrum of chalcone of 5-nitro-furfural was recorded on a Perkin-Elmer Spectrum RX-IFTIR Spectrophotometer in the range 4000-400 cm<sup>-1</sup> using potassium bromide pellet at CIL,

Chandigarh, Punjab. The IR bands for chalcone of 5-nitro-furfural appear at 1623 per cm which agrees with the general range of chalcones. The change in the position of a band is observed due to change in stretching vibration mode of bond involving coordinated atom. The stretching frequency of chalcone of 5-nitro-furfural is represented in table number (2) and the IR spectrum in figure (3).

Molecule	$\nu(\text{OH})$ Enolic	(-CO-CH=CH-) $\alpha, \beta$ -unsaturated carbonyl group	Carbon yl group (-C=O in pyron ring)	(C-O-C) Stretching Frequency	(C=C) Stretching Frequency	Aromatic Ring (C=C) Stretching Frequency	Ar-H Stretching Frequency	-NO <sub>2</sub> stretching frequency
[C <sub>13</sub> H <sub>9</sub> NO <sub>6</sub> ]	3127 cm <sup>-1</sup>	1623 cm <sup>-1</sup>	-	1056 cm <sup>-1</sup>	1537 cm <sup>-1</sup>	1495 cm <sup>-1</sup>	2980 cm <sup>-1</sup>	1537 cm <sup>-1</sup>

Table no. (2): Stretching of chalcone of 5-nitro-furfural.

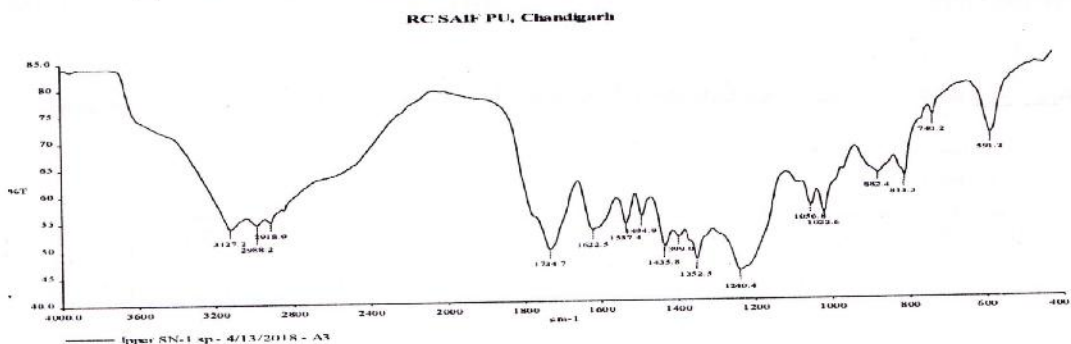


Fig.(3):IR spectrum of chalcone of 5-nitro-furfural.

### 3.3 UV-visible spectrum

The major absorption band in chalcones (Band I) usually occurs in the range 340-390 nm, although chalcones lacking B-ring oxygenation may have their Band First absorption at considerably shorter wavelengths and Band Second have a minor peak in the 220-270 nm region. The UV-visible spectrum of chalcone of 5-nitro-furfural measured on SL 159 single beam UV-VIS spectrophotometer. The UV-visible spectrum of chalcone of 5-nitro-furfural is given in



figure (4) and it's spectral data in table no. (3).In chalcone of 5-nitro-furfural has two matching bands at 231nm and other at 311nm which lies in the range of chalcone.

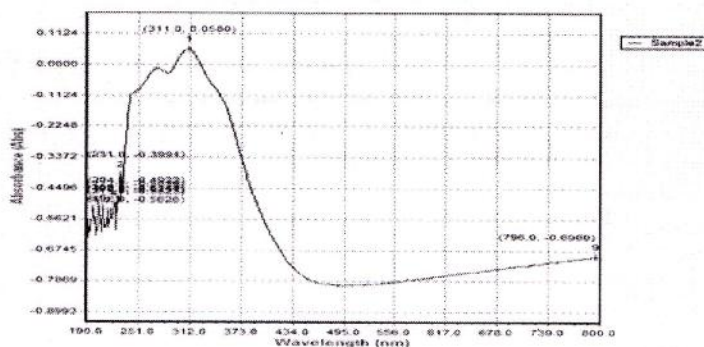


Fig.(4): UV-visible spectrum of chalcone of 5-nitro-furfural

Absorption spectrum	Wavelength
absorbance	$\lambda$ (nm)
0.3991	231.0
0.0580	311.0
0.6960	796.0

Table no. (3): UV-visible spectral data of chalcone of 5-nitro-furfural

### 3.4 $^1\text{H}$ NMR spectral study

In the structure determination of organic molecule,  $^1\text{H}$  NMR spectra have important role. It is the most valuable technique in structural investigation [13].

The  $^1\text{H}$  NMR spectrum of chalcone of 5-nitro-furfural is recorded on Bruker AVANCE II 400 MHz Spectrophotometer in DMSO solvent using TMS as an internal standard at SAIF, Chandigarh, Punjab are shown in figure (5), and spectral data in table no. (4) and Chemical shift ( $\delta$ ) ppm in figure (6).

Table no. (4):  $^1\text{H}$  Nuclear magnetic resonance spectral data of chalcone of 5-nitro-furfural

Chemical Shift ( $\delta$ ) ppm.	Number of Protons	Multiplicity (Splitting)	Assignment
6.48-7.20	3H	m	Aromatic protons
5.0	2H	S	-OH group present on aromatic benzene ring.
7.56	1H	d	$\alpha$ -H on-unsaturated carbonyl system
7.90	1H	d	$\beta$ -H on-unsaturated carbonyl system
7.55-7.86	2H	d	Protons on furan ring.

Fig.(6): Chemical shift ( $\delta$ ) ppm of protons in PMR spectrum.

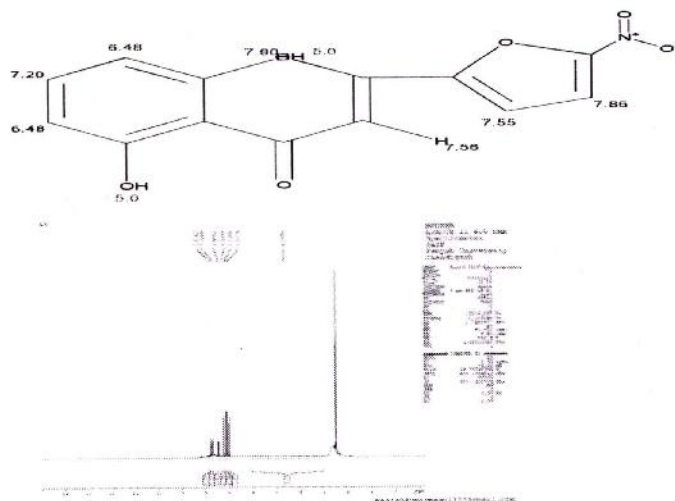
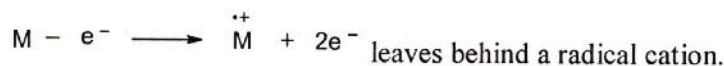


Fig.(5): <sup>1</sup>H Nuclear Magnetic Resonance Spectrum of chalcone of 5-nitro-furfural



### 3.5 Mass spectrum

Mass spectroscopy is the most accurate technique for the determination of molecular weight of compound. In this technique matter is bombarded with highly energetic electrons. Then matter absorbs or ejects electrons, from it. When it ejects electrons charged species are formed.



Anionic and cationic radicals are detected by detector. Detector never detects radical and neutral species. The mass spectrum is a plot representing the m/e values of various ions against their relative percent intensity. The highest intense peak in the spectrum is called base peak. [14] The intensity of other peaks is shown relative to the base peak. The peak at extreme right corresponds to the molecular weight of the original molecule. The molecular ion is called parent ion and usually denoted as  $[M]^+$  ion. In mass spectrum  $[M^+ + 1]$  and  $[M^+ + 2]$  peaks are also noticed. Mass spectroscopy also separates the isotopes. The mass spectrum chalcone of 5-nitro-furfural was recorded on Waters, Q-TOF Micro Mass (LC-MS).at SAIF, Chandigarh, Punjab.

The mass spectrum of chalcone of 5-nitro-furfural is represented in figure (7) and molecular mass in table no.(5). In the mass spectrum of chalcone of 5-nitro-furfural, molecular ion peak is observed m/z and calculated m/z corresponding to  $[M^+]$ ,  $[M^+ + 1]$  and  $[M^+ + 2]$  peaks are in good agreement with their structure.

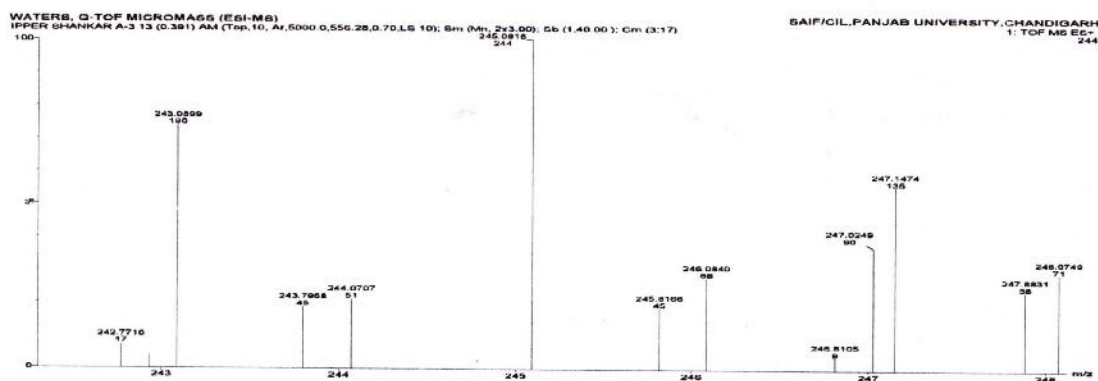


Fig.(7): Mass spectrum of chalcone of 5-nitro-furfural.

Table no. (5): Molecular mass from mass spectral data for chalcone of 5-nitro-furfural.

Molecule	$[M^+ + 2]$	$[M^+ + 1]$	$[M^+]$ Molecular mass found (calculated)
$C_{13}H_9NO_6$	247	246	245

### 3.6 Antimicrobial Activity

Table no. (6): Antimicrobial Activity of chalcone of 5-nitro-furfural

Ligands	Antifungal Growth		
	<i>Aspergillus niger</i>	<i>Saccharomyces cerevisiae</i>	<i>Candida albicans</i>
	ATCC 16404	ATCC 9763	ATCC10231
	1%	1%	1%
chalcone of 5-nitro-furfural	14	--	15
Standard	26	24	34

Ligands	Antibacterial Growth Diameter of inhibition zone (mm)									
	<i>S.typhi</i>	<i>E.aerogenes</i>	<i>B.subtilis</i>	<i>P.aerogenosa</i>	<i>S.abony</i>	<i>B.megaterium</i>	<i>E.Coli</i>	<i>S.aureus</i>	<i>S.boydii</i>	<i>B.cereus</i>
	ATCC 9207	ATCC 3048	ATCC 6633	ATCC 9027	NCTC 6017	ATCC 2326	ATCC 8739	ATCC 6538	ATCC 12034	ATCC 14579
chalcone of 5-nitro-furfural	15	15	12	10	--	14	12	--	11	14
Std	27	33	34	33	30	32	29	25	27	33



The chalcone of 5-nitro-furfural was screened *in vitro* for antifungal activity. The fungal toxicity of chalcone of 5-nitro-furfural was studied *in vitro* against *Aspergillus niger* ATCC 16404, *Saccharomyces cerevisiae* ATCC 9763, *Candida albicans* ATCC10231 fungal pathogens at fixed 1% concentration.

The chalcone of 5-nitro-furfural and was screened *in vitro* for antibacterial activity. The antibacterial activity of chalcone of 5-nitro-furfural was studied, for evaluating antibacterial activity Gram positive and Gram negative bacterial pathogens were used. *Staphylococcus aureus* ATCC 6538, *Bacillus megaterium* ATCC 2326, *Bacillus subtilis* ATCC 6633 were Gram positive pathogens used in this study. *Escherichia coli* ATCC8739, *Salmonella typhi* ATCC9207, *Shigella boydii* ATCC 12034, *Enterobacter aerogenes* ATCC13048, *Pseudomonas aerogenosa* ATCC9027, *Salmonella abony* NCTC6017 were the Gram-negative pathogens used in this study. [11-12] The Antimicrobial activity of chalcone of 5-nitro-furfural in Table no 6 shows that the chalcone of 5-nitro-furfural shows moderate to good Antibacterial and Antifungal activity [15].

#### 4. Conclusion

The chalcone of 5-nitro-furfural was synthesized by Claisen-Schmidt condensation method. The IUPAC name of this compound is (E)-1-(2,6-dihydroxyphenyl)-3-(5-nitrofuranyl)prop-2-en-1-one. The Chemical shift ( $\delta$ ) ppm from  $^1\text{H}$  NMR spectral study is between 5.0 -7.86. The mass spectrum of this chalcone shows molecular ion peaks observed  $m/z$  and calculated  $m/z$  for  $[\text{M}^+]$ ,  $[\text{M}^++1]$  and  $[\text{M}^++2]$  peaks are in good agreement. The elemental analysis gives percentage of CHO in chalcone of 5-nitro-furfural. The chalcone of 5-nitro-furfural have moderate to good Antibacterial and Antifungal activity.

#### Acknowledgement

Authors are thankful to Principal, Sunderrao Solanke Mahavidyalaya, Majalgaon, Dist. Beed (India) for providing laboratory facilities.

#### References

- [ 1 ] D.Tyagi, International Research Journal of Engineering and Technology, Volume 04, Issue 12, December -2017, pp.741-747.
- [2] S.N.Ipper,N.K.Raut,P.P.Dixit and G.K.Kakade, Synthesis,spectral study and properties of (E)-3-(furan-2-yl)-1-(2,6-dihydroxy)prop-2-en-1-one,International Journal of Basic and Applied Research ,September 2018,Vol.8,No.9,pp.961-968.

- [3] D.I.Ugwu and et al., Synthesis and pharmacological applications of chalcones: A Review, *Int.J.Chem.Sci.* : 13(1); 2015, 459-500.
- [4] A.M.Abdulla, Synthesis, characterization and antibacterial activity of (E)-chalcone derivatives, *European Journal of Chemistry*, 4(3) (2013), 207-210.
- [5] A.B. Linke and D.E. Eveleigh, *Z. Naturforsch*, 1975,308,340.
- [6] D.A.Akhmedzade,V.D.Yasnopol'skii, Y.I.Golovanova and *AzerbkhimZh*,117,1968, *Chem. Abstr.*1968, 69,107333n.
- [7] D.N. Dhar, *The chemistry of chalcones and related compounds*, Wiley Interscience, New York (1981).
- [8] P.K.P.Gaitry Chopra, Chalcones: A brief Review, *International Journal of Research in Engineering and Applied Sciences*, Vol.6, Issue 5(May- 2016) pp.173-185.
- [9] H.Jin,Y.Geng,Z.Yu,K.Tao and T.Hou, Lead optimization and anti-plant pathogenic fungi activities of Daphneolone analogous from *stellera chamaejasme L.*,*Pesticide Biochemistry and Physiology* ,93(2009)133-137.
- [10] R.A.Shastri, Synthesis and antimicrobial activity of some novel pyrozone and flavones derivatives derived from furan chalcones, *World Journal of Pharmaceutical Research*, Vol.4, Issue7, 642-651, June - 2015.
- [11] A.G.Rivera,H.A.Mariscal,N.R.Ceronio,L.F.Roa-dela Fuente and C.E.Loboto-Garcia, Synthesis and anti-inflammatory activity of three nitro chalcones, *Bioorganic and Medicinal Chemistry Letters*,23(2013)5519-5522.
- [12] Y.B.Vibhute, M.A.Baseer, Synthesis and activity of a new series of chalcones as antibacterial agents, *Indian Journal of Chemistry*, Vol.42B, January 2003, pp.202-205.
- [13] S.N.Ipper, N.K.Raut, P.P.Dixit and G.K.Kakade, Synthesis, spectral study and properties of Pyridine chalcone,*IJAEM*,Volume 7,Issue 8,August-2018,pp.20-24.
- [14] K.P.Kakde,S.P.Kakde and S.Y.Deshmukh, Synthesis and characterization of some Bromo substituted chalcones by green synthesis way and Aurones 2-Benzylidene -1-Benzofuran-3-one by cyclization method,*Vol.4,Issue 1,December 2014,1591-1597.*



---

[15] E.Sushanti V.H., S.Matsjeh, Mustafa and T.D.Wahyuningsih, Improver synthesis of 2,6-dihydroxy-3,4-Dimethoxy Chalcone by grinding to synthesize 5-hydroxy-3',4' Dimethoxy flavones, Indo.J.Chem., 2014, (2), 174-178.

# Synthesis, Characterization and Antimicrobial Activity of 5-hydroxy-2-(5-methylfuran-2-yl)-4H-chromen-4-one

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## Abstract

There has been increasing in the research on flavonoids from plant sources because of their versatile health benefits reported in various epidemiological studies. Fruits and vegetables are the main dietary sources of flavonoids for human, along with tea and wine. 5-hydroxy-2-(5-methylfuran-2-yl)-4H-chromen-4-one was synthesized by Claisen-Schmidt condensation method from 2,6-dihydroxy acetophenone and 5-methyl-Furaldehyde to gives chalcone and which on oxidation with DMSO/I<sub>2</sub>. Spectroscopic characterization using UV-visible, IR, <sup>1</sup>H NMR, Mass spectra, properties and antimicrobial activity has been investigated for 5-hydroxy-2-(5-methylfuran-2-yl)-4H-chromen-4-one. The IR bands for Carbonyl group (-C=O in pyron ring) for 5-hydroxy-2-(5-methylfuran-2-yl)-4H-chromen-4-one appear at 1606 per cm which agrees with the general range of flavones. The 5-hydroxy-2-(5-methylfuran-2-yl)-4H-chromen-4-one has two UV matching bands at 258 nm and other at 339 nm which lies in the range of flavone. In the mass spectrum of 5-hydroxy-2-(5-methylfuran-2-yl)-4H-chromen-4-one molecular ion peak corresponding to [M<sup>+</sup>] peak is in good agreement with their structure. The flavone shows moderate to good Antibacterial and Antifungal activity.

**Keywords:** 5-hydroxy-2-(5-methylfuran-2-yl)-4H-chromen-4-one, UV-visible, IR, <sup>1</sup>H NMR, Mass spectrum, CHO analysis and Antimicrobial Activity.

## 1. Introduction

The flavonoids consist of a large group of polyphenolic compounds having a benzo-pyrone structure and are ubiquitously present in plants. The term flavonoids are generally used to describe a broad collection of natural products that includes a C<sub>6</sub>-C<sub>3</sub>-C<sub>6</sub> Carbon frame work. Flavones are medicinal natural products. Prevention and cure of disease using flavonoids as a photochemical are known due to its wide spectrum activities it attracts researchers of the world. Hence cheap easy lab feasible synthetic methodology is the demand of pharmaceutical



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## Abstract

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## 1. Introduction

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industries. In that point of view, we optimized the Claisen-Schmidt method for the synthesis of flavones. Different molar ratios, solvent, temperature and catalysts will be examined during experiments. Also the effect of structure and substituent on the product formation and effect of groups and other on starting material will be considered. Oxidation is one of the most important processes, it produces free radicals. In turn, these radicals start chain reactions that damage cells and cause oxidative stress. This oxidative stress in cells results in the development of a wide range of diseases like Alzheimer's disease,<sup>1-2</sup> Parkinson's disease,<sup>3</sup> the pathologies caused by diabetes<sup>4-5</sup> rheumatoid arthritis and neuro-degeneration in motor neuron.<sup>6</sup> Antioxidants flavones have been studied specifically as they terminate the chain reactions by removing free radical intermediates and inhibit other oxidation reactions. Flavones occupy a special place in the realm of natural and synthetic organic chemistry owing to their useful biological activities such as anti-oxidant, anxiolytic, anticancer, analgesic, and antimicrobial. During the past few years various methods have been reported.

## 2. Materials and Methods

The chalcone of 5-methyl-Furaldehyde was synthesized by Claisen-Schmidt condensation of 2,6, di-hydroxyacetophenone (0.01 mol) and 5-methyl-Furaldehyde (0.01 mol). Which on oxidation with DMSO/I<sub>2</sub> it gives flavones. The chemicals used for this synthesis are of AR grade.

### Synthesis of Chalcone:

The mixture of 2,6-dihydroxy acetophenone (0.01 mol) and 5-methyl-Furaldehyde (0.01 mol) is dissolved in ethanol (20 mL) and then sodium hydroxide 10 mL (40%) was added to it. The mixture was stirred for overnight till brown color ppt was observed. The progress of the reaction was monitored by TLC, from thin layer chromatography the completion of the reaction is observed. After completion of the reaction, the contents were poured into ice cold water and then acidified by dil.HCl. The solid obtained was filtered and crude product was recrystallized from ethanol to give the chalcone.<sup>7</sup>

### Synthesis of Flavone:

The chalcone of 5-methyl-Furaldehyde was dissolved in 20 ml DMSO, to this catalytic quantity of iodine was added. Contents were refluxed for one hour, the progress of the reaction was monitored by TLC and the reaction mixture was left overnight. It was then poured on ice cold water, the separated solid was filtered washed with cold water followed by a dilute sodium-thiosulphate solution. The product was crystallized from ethanol.<sup>8-9</sup> The melting point of the flavone was determined by an open capillary tube and is unconfirmed. Infra red spectrum was measured using FT-IR spectrophotometer, UV-visible spectrum measured on SL 159 single beam UV-VIS spectrophotometer, <sup>1</sup>H NMR measured on Bruker AVANCE II 400 MHz Spectrophotometer and Mass spectrum was measured on Mass spectrophotometer. The purity the compound was checked by TLC plate which was precoated with silica gel using solvent ethyl acetate and petroleum ether (3:7). The reaction for formation of flavone is given figure (1).



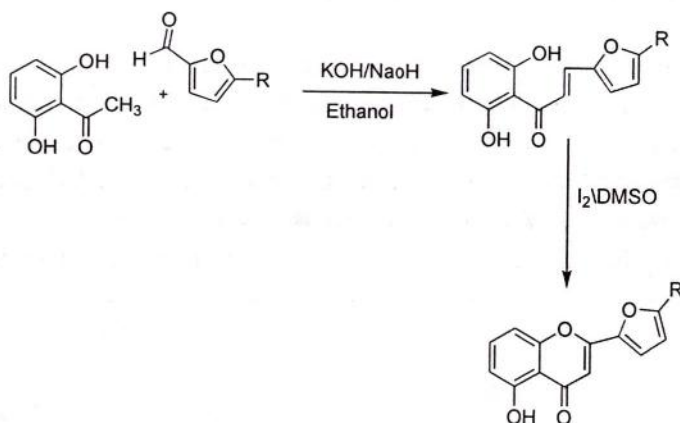


Fig.(1): Reaction of formation of Flavone.  $R=CH_3$

### 3. Results and discussion

#### 3.1 Properties

The flavone having IUPAC name 5-hydroxy-2-(5-methylfuran-2-yl)-4H-chromen-4-one, was synthesized by Claisen-Schmidt condensation method and its structure is stable at room temperature, insoluble in water and is soluble in organic solvent (ethyl alcohol)<sup>10</sup>. The physico-chemical properties of flavone and CHO analysis by calculation method are given in table no. (1). The reaction of formation of flavone is shown in figure (1). Elemental analysis showed that the percentage of the Oxygen, hydrogen and carbon found experimentally is equivalent to the calculated values for this compound. The chemical structure of  $[C_{14}H_{10}O_4]$  is shown in figure (2).

Table no. (1): The properties of flavone and Elemental analysis data.

Mol. Formula	Color	Mo l. Wt.	M.P °c	Found (Calculated) %			
				C	H	O	N
$[C_{14}H_{10}O_4]$	Yellowish brown	242	220	69.46 (69.42)	4.19 (4.16)	26.47 (26.42)	---

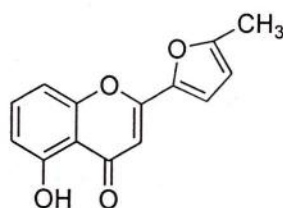


Fig.(2): Chemical structure of C<sub>14</sub>H<sub>10</sub>O<sub>4</sub>

### 3.2 Infra red spectrum

The IR spectrum of pyron ring carbonyl group has characteristic bands of flavone at prominent bands between 1600 to 1625 per cm. The characteristic peaks in infra red spectrum give the presence of particular functional group.<sup>11</sup> The region at which other absorption bands appear depends on the type of aromatic / hetero-aromatic rings as well as the substituent present on these rings. The infrared spectrum of chalcone of flavone was recorded on a Perkin-Elmer Spectrum RX-IFTIR Spectrophotometer in the range 4000-400 cm<sup>-1</sup> using potassium bromide

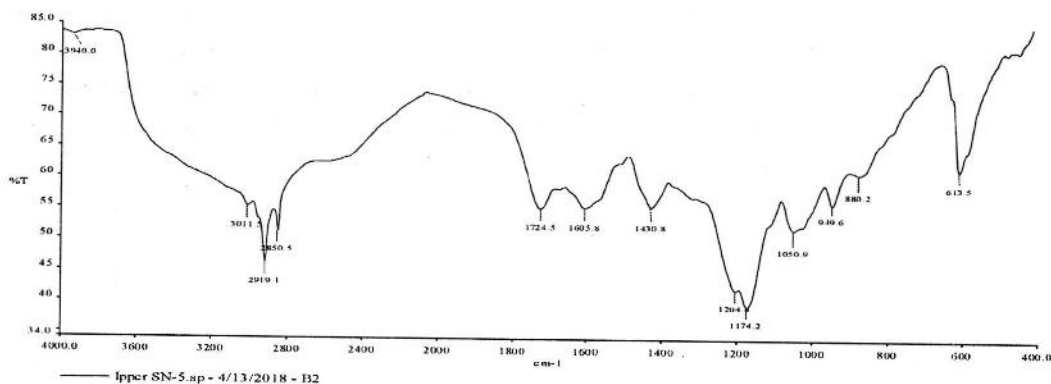
Molecule	$\nu(\text{OH})$ Enolic	(-CO-CH=CH-) $\alpha,\beta$ -unsaturated carbonyl group	Carbonyl group (-C=O in pyron ring)	(C-O-C) Stretching Frequency	(C=C) Stretching Frequency	Aromatic Ring (C=C) Stretching Frequency	Ar-H Stretching Frequency	stretching frequency
Flavone	3011	-	1606	1204	1550	1430	2919	-

pellet at CIL, Chandigarh, Punjab. The IR bands for 5-hydroxy-2-(5-methylfuran-2-yl)-4H-chromen-4-one, appears at 1605 per cm which agrees with the general range of flavone. The change in the position of a band is observed due to change in stretching vibration mode of bond involving coordinated atom. The stretching frequency of flavone is represented in table number (2) and the IR spectrum in figure (3).

Table no. (2): The stretching frequency of flavone

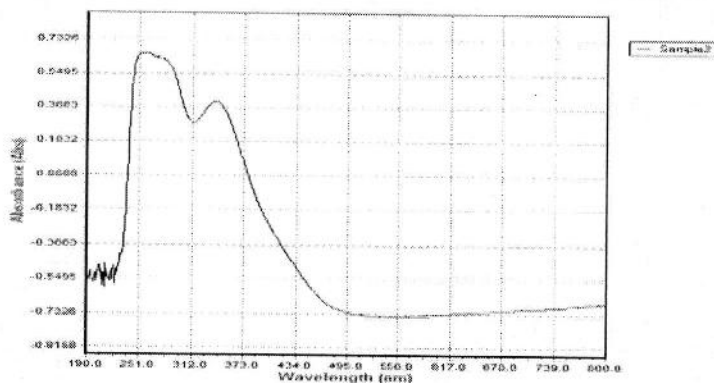


**Fig.(3):** IR spectrum of 5-hydroxy-2-(5-methylfuran-2-yl)-4H-chromen-4-one,  
 RC SAIF PU, Chandigarh



### 3.3 UV-visible spectrum

Generally, the flavones and flavones exhibit absorption in 320-380 nm regions (band I) and 240-270 nm regions (band II). The UV-visible spectrum 5-hydroxy-2-(5-methylfuran-2-yl)-4H-chromen-4-one measured on SL 159 single beam UV-VIS spectrophotometer. The UV-visible spectrum of flavone is given in figure (4). In 5-hydroxy-2-(5-methylfuran-2-yl)-4H-chromen-4-one, has two matching bands at 258 nm and other at 339 nm which lies in the range of flavone<sup>12</sup>.



**Fig.(4):** UV-visible spectrum of 5-hydroxy-2-(5-methylfuran-2-yl)-4H-chromen-4-one.

### 3.4 $^1\text{H}$ NMR spectral study

In the analysis of organic molecule,  $^1\text{H}$  NMR spectra plays very important role. It is the most valuable technique in structural investigation.<sup>10</sup>

The  $^1\text{H}$  NMR spectrum of chalcone of 5-hydroxy-2-(5-methylfuran-2-yl)-4H-chromen-4-one is recorded on Bruker AVANCE II 400 MHz Spectrophotometer in DMSO solvent using TMS as an internal standard at SAIF, Chandigarh, Punjab are shown in figure (5), and spectral data in table no. (3) and Chemical shift ( $\delta$ ) ppm in figure (5)

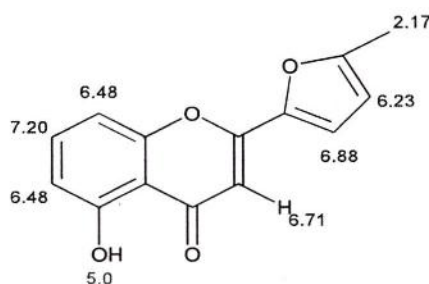
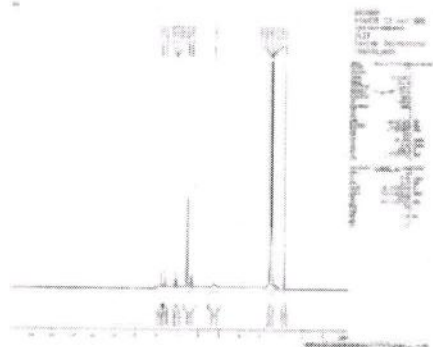


Table no. (3):  $^1\text{H}$  Nuclear magnetic resonance spectral data of 5-hydroxy-2-(5-methylfuran-2-yl)-4H-chromen-4-one

Chemical Shift ( $\delta$ ) ppm	Number of Protons	Multiplicity (Splitting)	Assignment
6.48-7.20	3H	m	Aromatic protons
5.0	1H	s	-OH group present on the aromatic benzene ring
6.71	1H	s	$\alpha$ -H on-unsaturated carbonyl system
6.23-6.88	2H	d	Protons on furan ring



2.17	3H	m	Methyl protons attached to furan ring
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**Fig.(5):**  $^1\text{H}$  Nuclear Magnetic Resonance Spectrum of 5-hydroxy-2-(5-methylfuran-2-yl)-4H-chromen-4-one and Chemical shift ( $\delta$ ) ppm of protons in PMR spectrum.

### 3.5 Mass spectrum

The highest intense peak in the spectrum is called base peak. The intensity of other peaks is shown relative to the base peak. The peak at extreme right corresponds to the molecular weight of the original molecule. The molecular ion is called parent ion and usually denoted as  $[\text{M}]^+$  ion. The mass spectrum of 5-hydroxy-2-(5-methylfuran-2-yl)-4H-chromen-4-one was recorded on Waters, Q-TOF Micro Mass (LC-MS) at SAIF, Chandigarh, Punjab. The mass spectrum of chalcone of 5-hydroxy-2-(5-methylfuran-2-yl)-4H-chromen-4-one is represented in figure (6) and molecular mass. In the mass spectrum of 5-hydroxy-2-(5-methylfuran-2-yl)-4H-chromen-4-one molecular ion peak is observed  $m/z$  and calculated  $m/z$  corresponding to  $[\text{M}]^+$  peak are in good agreement with their structure.<sup>13</sup>

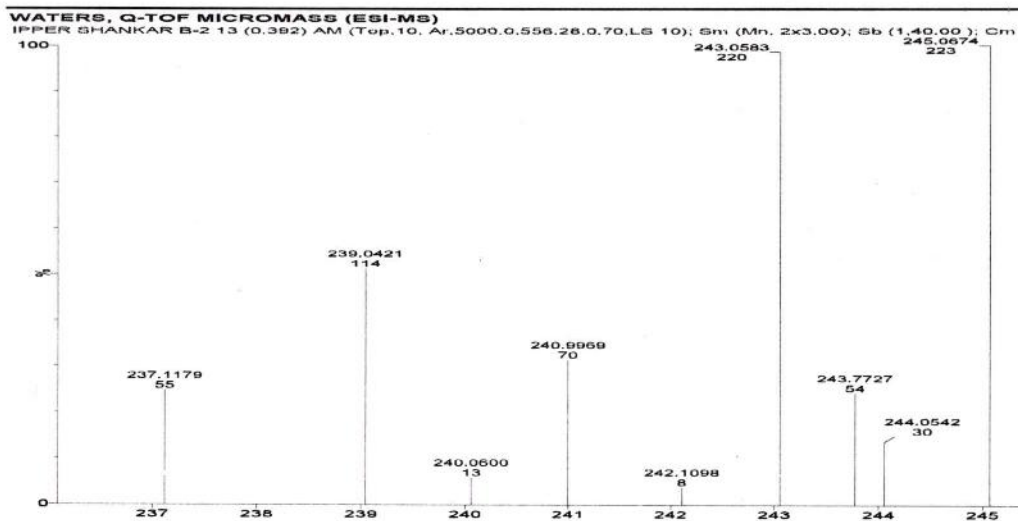


Fig.(6): Mass spectrum of 5-hydroxy-2-(5-methylfuran-2-yl)-4H-chromen-4-one

Table no. (4): Antifungal Activity of 5-hydroxy-2-(5-methylfuran-2-yl)-4H-chromen-4-one.

Ligands	Antifungal Growth		
	<i>Aspergillus niger</i>	<i>Saccharomyces cerevisiae</i> ATCC 9763	<i>Candida albicans</i> ATCC10231
	ATCC 16404		
	1%	1%	1%
Flavone	10	11	08
Standard	26	24	34

Table no. (5): Antimicrobial Activity of 5-hydroxy-2-(5-methylfuran-2-yl)-4H-chromen-4-one.



Ligands	Antibacterial Growth Diameter of inhibition zone (mm)									
	<i>S.typhi</i> ATCC 9207	<i>E.aerog</i> <i>enes</i> ATCC1 3048	<i>B.sub</i> <i>tilis</i> ATC C 6633	<i>P.aerog</i> <i>enosa</i> ATCC9 027	<i>S.abon</i> <i>y</i> NCTC 6017	<i>B.megat</i> <i>erium</i> ATCC 2326	<i>E.Coli</i> ATCC 8739	<i>S.aur</i> <i>eus</i> ATC C 6538	<i>S.bo</i> <i>ydi</i> ATC C 1203 4	<i>B.cer</i> <i>eus</i> ATC C 1457 9
	1%	1%	1%	1%	1%	1%	1%	1%	1%	1%
Flavone	09	08	15	13	11	12	12	10	15	13
Std	27	33	34	33	30	32	29	25	27	33

In the treatment of diseases, inhibitory chemicals employed to kill micro-organisms or prevent their growth, are called the anti-microbial activity. The biotic component of the world includes a vast variety of living organism. The biotic component which includes all the living organisms belonging to five kingdoms.

The flavone was screened *in vitro* for antifungal activity. The fungal toxicity of flavone was studied *in vitro* against *Aspergillus niger* ATCC 16404, *Saccharomyces cerevisiae* ATCC 9763, *Candida albicans* ATCC10231 fungal pathogens at fixed 1% concentration.

The flavone and was screened *in vitro* for antibacterial activity. The antibacterial activity of flavone was studied, for evaluating antibacterial activity Gram positive and Gram negative bacterial pathogens were used. *Staphylococcus aureus* ATCC 6538, *Bacillus megaterium* ATCC 2326, *Bacillus subtilis* ATCC 6633 were Gram positive pathogens used in this study. *Escherichia coli* ATCC8739, *Salmonella typhi* ATCC9207, *Shigella boydii* ATCC 12034, *Enterobacter aerogenes* ATCC13048, *Pseudomonas aerogenosa* ATCC9027, *Salmonella abony* NCTC6017 were the Gram-negative pathogens used in this study.<sup>14-15</sup>

#### 4. Conclusion

The 5-hydroxy-2-(5-methylfuran-2-yl)-4H-chromen-4-one, was synthesized by Claisen-Schmidt condensation method. The IUPAC name of this compound is 5-hydroxy-2-(5-methylfuran-2-yl)-4H-chromen-4-one. In flavones, the <sup>1</sup>H NMR spectrum appearance of a signal at δ-6.71 singlets (1H of pyran ring) supported the flavones structures. The mass spectrum of this flavone shows molecular ion peak observed m/z and calculated m/z for [M<sup>+</sup>], peak is in good agreement. The elemental analysis gives percentage of CHO in 5-hydroxy-2-(5-methylfuran-2-yl)-4H-chromen-4-one.

**Acknowledgement** Authors are thankful to Principal, Sunderrao Solanke Mahavidyalaya, Majalgaon, Dist. Beed (India) for providing laboratory facilities.

### References

- [1] Christen, Y. (2000) Journal of the American College of Nutrition, 71, 621–629.
- [2] Nunomura, A.R. Castellani, X.Zhu, P. Moreira, G. Perry, M. S. (2006) Journal of Neuropathology & Experimental Neurology, 65, 631–641.
- [3] Wood-Kaczmar, A.S. Gandhi, N. (2006) Molecular Medicine, 12, 521–528.
- [4] Davi G.A., Falco, C. (2005) Antioxid Redox Signal, 7, 256–268.
- [5] Hitchon, C. El-Gabalawy H. (2004) Arthritis.
- [6] Miller, A.L. (1996) Alternative Medicine Review, 1, 103-111.
- [7] Omar. H. Al-Obaidi, (2014). Synthesis, spectral characterization, theoretical evaluation of new Co(II) and Mn(II) complexes of flavones., Journal of Applicable Chemistry, 3(1):157-163.
- [8] Shastri, R.A. (2015 4<sup>th</sup> June). Synthesis and antimicrobial activity of some novel pyrazoline and flavones derivatives derived from furan chalcone; World Journal of Pharmaceutical Research, Vol.4, issue7, 642-651.
- [9] E.Sushanti VH, S.Matsjeh, Mustafa and T.D.Wahyuningsih, Improver synthesis of 2,6-dihydroxy-3,4,-Dimethoxy Chalcone by grinding to synthesize 5-hydroxy -3' ,4'-Dimethoxy flavones, Indo.J.Chem., 2014,(2), 174-178.
- [10] S.N. Ipper, N.K. Raut and G.K. Kakde, Physico-chemical properties of furan chalcone. Ind. J. Appl. Res. 2017; 7(6):683-684.
- [11] S. N. Ipper, N. K. Raut, P. P. Dixit and G. K. Kakade, Synthesis, spectral study and properties of (E)-3-(5-bromofuran-2-yl)-1-(2,6-dihydroxyphenyl)prop-2-en-1-one, Journal of Pharma Research (World Inventia Publishers) Vol.7, Issue 10, 2018, pp.220-223.
- [12] Y.B.Vibhute, M.A.Baseer, Synthesis and activity of a new series of chalcones as antibacterial agents, Indian Journal of Chemistry, Vol.42B, January 2003, pp.202-205.
- [13] S.N.Ipper, N.K.Raut, P.P.Dixit and G.K.Kakade, Synthesis, spectral study and properties of (E)-3-(furan-2-yl)-1-(2,6-dihydroxy)prop-2-en-1-one, International Journal of Basic and Applied Research, September 2018, Vol.8, No.9, pp.961-968.
- [14] A.G.Rivera, H.A.Mariscal, N.R.Ceronio, L.F.Roa-de la Fuente and C.E.Loboto-Garcia, Synthesis and antiinflammatory activity of three nitro chalcones, Bioorganic and Medicinal Chemistry Letters, 23(2013)5519-5522.
- [15] Sirsat, S.B., Halikar, N.K. Pund, M.M. and Vartale, S.P. (2012 April/June). Synthesis and biological screening of some novel hetero-aryl-chalcone and their Complexes. Research Journal of Pharmaceutical Biological and Chemical Sciences. Vol.3 issue 2, 240-248.





## Synthesis, Characterization And Antimicrobial Study Of Nickel(II) Complex Of (E)-1-(2,6-Dihydroxyphenyl)-3-(5-Methylfuran-2-Yl)Prop-2-En-1-One

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### Abstract:

The synthesis of Nickel (II) metal complex has been synthesized by using novel (E)-1-(2,6-dihydroxyphenyl)-3-(5-methylfuran-2-yl)prop-2-en-1-one ligand. The ligand was prepared by the Claisen-Schmidt condensation method of 2,6-dihydroxy acetophenone and 5-methylfurfural. The structure of the complex has been characterized by the analytical data, conductivity measurement, magnetic moment, UV-Vis spectra, thermal studies and XRD analysis. Analytical data shows 1:2 stoichiometry and the magnetic moment, TG-DTA suggests that Ni(II) complex has octahedral geometry. The presence of coordinated water molecules in Ni (II) complex is confirmed by thermal studies. The conductivity data reveals that the complex is non electrolyte. Antimicrobial study of complex with selected bacterial strain and fungal strain carried out and the results have been compared with commercial standards. The Ni (II) complex shows moderate to good Antibacterial and Antifungal activity.

**Keywords:** Antimicrobial activities, TG-DTA, XRD study, Physico-chemical property, Magnetic Susceptibility and Conductivity.

### 1. Introduction:

Chalcones constitute an important group of natural products, which has two aromatic rings joined by  $\alpha$ ,  $\beta$  unsaturated carbonyl system. The name chalcone is given by Kostanecki and Tambar [1]. The  $>CO-C=C<$  moiety imparts biological characteristics to the Chalcones. Such  $\alpha$ ,  $\beta$ -unsaturated carbonyl group in chalcone is found to be responsible for their antimicrobial activity [2]. The metal complexes possess interesting biochemical properties, such as antitumor, antioxidant, and antimalarial, anti-fungal and antimicrobial activities [3]. The magnetic moment, TG-DTA supports the octahedral



geometry of the metal complex of chalcone. All crystals of a substance possess the same elements of symmetry. The computer program, used for indexing data was powder-X [4]. The X-ray powder diffractogram of the metal complex was used for the structural characterization and determination of lattice dimensions.

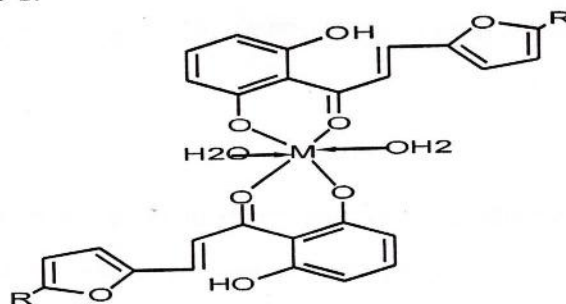
## 2. Materials and Methods:

### 2.1 Synthesis of (E)-1-(2,6-dihydroxyphenyl)-3-(5-methylfuran-2-yl)prop-2-en-1-one ligand:

The reagents used for preparation of (E)-1-(2,6-dihydroxyphenyl)-3-(5-methylfuran-2-yl)prop-2-en-1-one are of A.R. grade. The mixture of 2,6-dihydroxy acetophenone (0.01 mol) and 5-methylfuraldehyde (0.01 mol) is dissolved in ethanol (20 mL) and then sodium hydroxide 10 mL (40%) was added to it. The mixture was stirred for overnight till brown color ppt was observed. The progress of the reaction was monitored by TLC, from thin layer chromatography the completion of the reaction is observed. After completion of the reaction, the contents were poured into ice cold water and then acidified by dil. HCl. The solid obtained was filtered and crude product was recrystallized from ethanol to give the chalcone [5].

### 2.2 Synthesis of Metal Complex:

The solution of 0.02 mole of (E)-1-(2,6-dihydroxyphenyl)-3-(5-methylfuran-2-yl)prop-2-en-1-one was taken in round bottom flask containing 30 ml of anhydrous methanolic solution and boiled for 10 minutes. A hot solution of 0.01 mole, of Nickel Nitrate in 20 ml of methanol was added drop wise to the solution of the chalcone of 5-methylfurfural to this reaction mixture, 10% alcoholic ammonia was added up to slightly alkaline pH. The complex was precipitated at 8 pH range. The pH 8-10 range was definite for these complexes [6]. The content was stirred on magnetic stirrer for one hour. The solid metal complex separated out and washed with methanol three to four times. The melting point of the complex was determined by Thiele's melting apparatus. The reactions of formation of Ni(II) complex is shown in **Figure-1**.



**Figure-1:** Metal complex of Nickel (II) with (E)-1-(2,6-dihydroxyphenyl)-3-(5-methylfuran-2-yl)prop-2-en-1-one







### 3. Results and Discussion:

#### 3.1 Physical parameters:

Metal complex of Nickel (II) with (E)-1-(2,6-dihydroxyphenyl)-3-(5-methylfuran-2-yl)prop-2-en-1-one was reddish brown in color. The complex was precipitated at 8 pH range, having Melting point 280<sup>0</sup>C. The complex is insoluble in water and soluble in DMSO, DMF [7].

#### 3.2 CHO analysis:

The carbon, hydrogen, oxygen, Nickel metal percentage in Ni (II) complex of chalcone measured at SAIF Cochin, Kerala. The calculated and measured values of CHO analysis are matching and are given in the Table-1.

Table-1: Study CHO analysis synthesized Ni (II) complex

Metal complex	Chemical formula	Mol. Wt.	Elemental analysis : % found (calculated)						
			C	H	N	O	S	X(Br)	M
Nickel (II)	[C <sub>28</sub> H <sub>26</sub> O <sub>10</sub> Ni]	581	57.86 (65.02)	4.50 (5.06)	-	27.52 (18.56)	-	-	10.09 (11.34)

#### 3.3 Magnetic susceptibility, solution conductivity and electronic absorption spectral data

##### Magnetic susceptibility:

The magnetic moment values of octahedral Ni(II) complex fall in the range 2.9 to 3.4 B.M. These values of magnetic moment are slightly higher than expected from spin only values of 2.83 B.M. due to spin-orbit coupling and contribution from the <sup>3</sup>A<sub>2g</sub> And <sup>3</sup>T<sub>2g</sub> state. These values are in good agreement with the moment reported for octahedral Ni (II) complex by earlier workers [8].



### Solution conductivity and electronic absorption spectral data:

The solution conductivities of  $10^{-3}$  M solution of metal complex in DMSO were measured on EQUIPTRONICS digital conductivity meter EQ - 660 with  $20 \mu\Omega$  to  $200 \mu\Omega$  at 298K temperature. They are insoluble in water and soluble in DMSO, DMF. The low conductivity values in DMSO solution ( $10^{-3}$  M) are indicates non-electrolytic nature. The Solution conductivity **Ni (II) complex** was **(6.00)**, **Figure-2**.

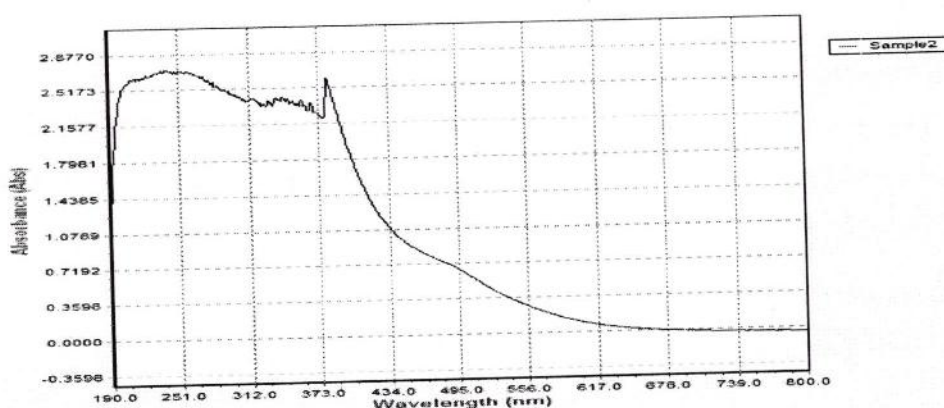


Figure-2: Electronic absorption spectrum

The magnetic moment of Ni(II) complex was **(2.92 BM)** and The electronic absorption spectra of Ni(II) complex was showed two bands at **26315 (380)  $^3A_{2g}$  (F)  $\rightarrow$   $^3T_{1g}$  (P)** and **29761(336)  $cm^{-1}$  (nm) Charge Transfer band** indicating distorted octahedral geometry around the Ni(II) ion [9-10].

### 3.4 Infra red spectrum:

The IR spectrum of  $\alpha, \beta$ -unsaturated carbonyl group has characteristic bands of chalcone at prominent bands between 1625 to 1650 per cm. The characteristic peaks in infra red spectrum give the presence of particular functional group. The region at which other absorption bands appear depends on the type of aromatic / hetero-aromatic rings as well as the substituent present on these rings. The infrared spectrum of metal complex of Nickel (II) with (E)-1-(2,6-dihydroxyphenyl)-3-(5-methylfuran-2-yl)prop-





2-en-1-one was recorded on a Perkin- Elmer Spectrum RX-IFTIR Spectrophotometer in the range 4000-400  $\text{cm}^{-1}$  (Table-2) using potassium bromide pellet at CIL, Chandigarh, Punjab. The stretching frequency of metal complex of Nickel (II) with (E)-1-(2,6-dihydroxyphenyl)-3-(5-methylfuran-2-yl)prop-2-en-1-one is represented in table number (2) and the IR spectrum in Figure-3.

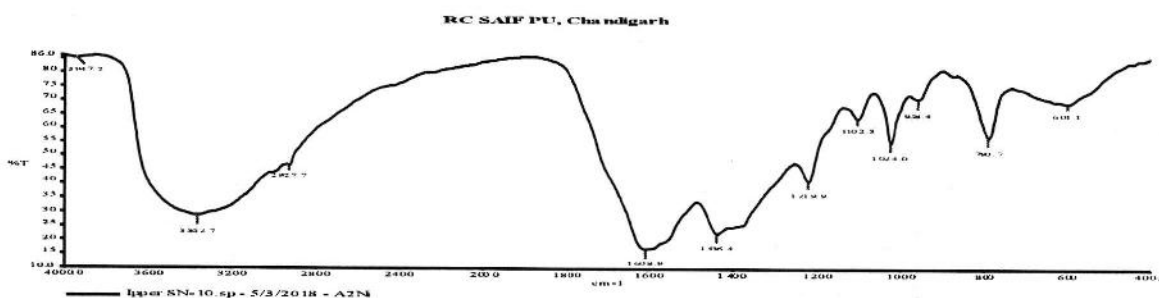


Figure-3: IR spectrum of metal complex of Nickel (II) with (E)-1-(2,6-dihydroxyphenyl)-3-(5-methylfuran-2-yl)prop-2-en-1-one

Table-2: IR spectral data Ni(II) complex of (E)-1-(2,6-dihydroxyphenyl)-3-(5-methylfuran-2-yl)prop-2-en-1-one:

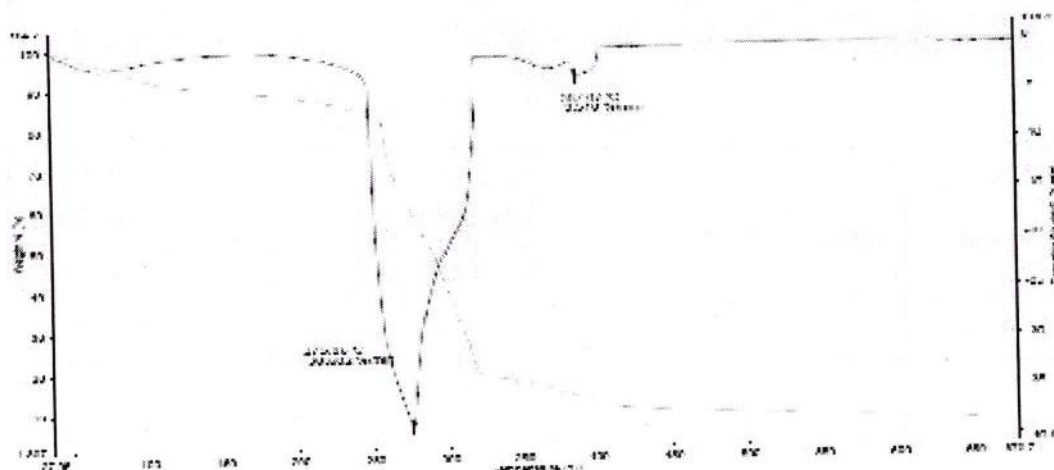
Ligand/ Metal complexes	$\nu$ (OH) $\text{cm}^{-1}$	$\nu$ (H <sub>2</sub> O) $\text{cm}^{-1}$	$\nu$ (-CO- CH=CH-) $\text{cm}^{-1}$	$\nu$ (-C=O in pyron ring) $\text{cm}^{-1}$	$\nu$ (C- O-C) $\text{cm}^{-1}$	$\nu$ (C=C) $\text{cm}^{-1}$	Aromat ic Ring (C=C) $\text{cm}^{-1}$	$\nu$ (M-O) $\text{cm}^{-1}$
[Ni(A <sub>2</sub> ) <sub>2</sub> ]	2927	3362	1608	-	1024	1436	1219	601

3.5 Thermal analysis Ni(II) complex of (E)-1-(2,6-dihydroxyphenyl)-3-(5-methylfuran-2-yl)prop-2-en-1-one:



The simultaneous thermo gravimetric, differential thermal analysis of Ni(II) complex (E)-1-(2,6-dihydroxyphenyl)-3-(5-methylfuran-2-yl)prop-2-en-1-one was performed in an inert nitrogen atmosphere on Perkin Elmer STA 6000 at SAIF, Cochin, Kerala. The heating rate was  $10^{\circ}/\text{min}$  and flow rate of nitrogen 50 ml/min. The reference substance used was  $\alpha \text{ Al}_2\text{O}_3$  in platinum crucible and sample weighed in the range of 4-12 mg. The thermogram of Ni(II) complex (E)-1-(2,6-dihydroxyphenyl)-3-(5-methylfuran-2-yl)prop-2-en-1-one is presented in figure-2. This curve reveals that there is presence of lattice as well as coordinated water in the complex.

The thermogram of Ni(II) complex of (E)-1-(2,6-dihydroxyphenyl)-3-(5-methylfuran-2-yl)prop-2-en-1-one (**Figure-4**) shows high thermal stability and two-step decomposition. The first step within temperature range  $240\text{-}330^{\circ}\text{C}$  with mass loss of 40.36% (calc. wt. loss 41.03%) due to two fragments of  $\text{C}_6\text{H}_5\text{O}_2$  take place. This is confirmed by an endothermic peak at  $275.86^{\circ}\text{C}$  in DTA curve. The second step decomposition observed within  $380\text{-}420^{\circ}\text{C}$  with a weight loss of 46.32%. This may be due to the loss of two fragment of 5-methyl-furan and enone part of chalcone of metal complex. Beyond that TG curve attains a constant weight loss 14.44% level corresponding to their metal oxide that is NiO.



**Figure-4:** TG-DTA curve of Ni(II) complex of (E)-1-(2,6-dihydroxyphenyl)-3-(5-methylfuran-2-yl)prop-2-en-1-one

### Thermodynamic and Kinetic Parameters





Akahira [11], first introduced that decomposition and kinetic studies of thermal reactions are useful in determining thermodynamic and kinetic parameters like free energy, entropy change, activation energy, pre-exponential factor. Thermal decomposition studies of materials are useful in predicting thermal stability (Table-3).

The negative values of the entropy of activation ( $\Delta S$ ) indicate that the metal complex is thermally stable.  $\Delta G$  is positive for the complexes revealing that the free energy of the final residue is higher than that of the initial complex, and all decomposition steps are non-spontaneous processes. Also, the value of free energy of activation,  $\Delta G$  increases significantly for the subsequent decomposition stages of a given complex[12].

**Table-3: Thermodynamic and Kinetic Parameters of Ni(II) complex of (E)-1-(2,6-dihydroxyphenyl)-3-(5-methylfuran-2-yl)prop-2-en-1-one:**

Metal complex	Method	Step	Decomp. Temp.	Order of Reaction	Ea(KJ mol <sup>-1</sup> )	$\Delta S$ (KJ mol <sup>-1</sup> )	$\Delta G$ (KJ mol <sup>-1</sup> )	Z (S <sup>-1</sup> )	Correlation Coefficient (r)
A <sub>2</sub> Ni	H-M C-R	I	290	0.9	13.42	-161.70	24.89	47529.95	0.999
					70.96	-138.30	80.81	738243	0.998
A <sub>2</sub> Ni	H-M C-R	II	580	0.9	6.73	-171.09	18.93	14335.18	0.987
					8.69	-86.94	14.89	355917865.6	0.996

### 3.6 X-ray diffraction spectral studies of metal complex of Ni (II) complex of (E)-1-(2,6-dihydroxyphenyl)-3-(5-methylfuran-2-yl)prop-2-en-1-one:

The XRD spectral study has been done at SAIF, Cochin Kerala. The standard deviation observed for A<sub>2</sub>Ni is 0.023% which is within the permissible limit of 2%. The observed and calculated densities are 1.9583 gm<sup>-3</sup> and 1.7392 gm<sup>-3</sup> respectively. The volume is found to be 339.43 Å<sup>3</sup> and complex crystallizes in the monoclinic system with 1 atom per unit cell. The lattice parameters are a=13.345 Å, b=8.252 Å, c= 9.756 Å,  $\alpha=90^\circ$ ,  $\beta= 109.78^\circ$ ,  $\gamma=90^\circ$ .

#### Unit cell data and crystal lattice parameters for Ni (II):



Unit cell data and crystal lattice parameters

$a$  (Å) = 13.345,  $b$  (Å) = 8.252,  $c$  (Å) = 9.765,  $\alpha = 90^\circ$ ,  $\beta = 109.78^\circ$ ,  $\gamma = 90^\circ$ , Standard deviation (%) = 0.23, Volume (V) = 339.43 Å<sup>3</sup>, Density (obs.) = 1.9583 gcm<sup>-3</sup>, Density (cal.) = 1.7392 gcm<sup>-3</sup>,  $Z = 4$   
Crystal system= Monoclinic, Space group = P2/m. (Figure-5)

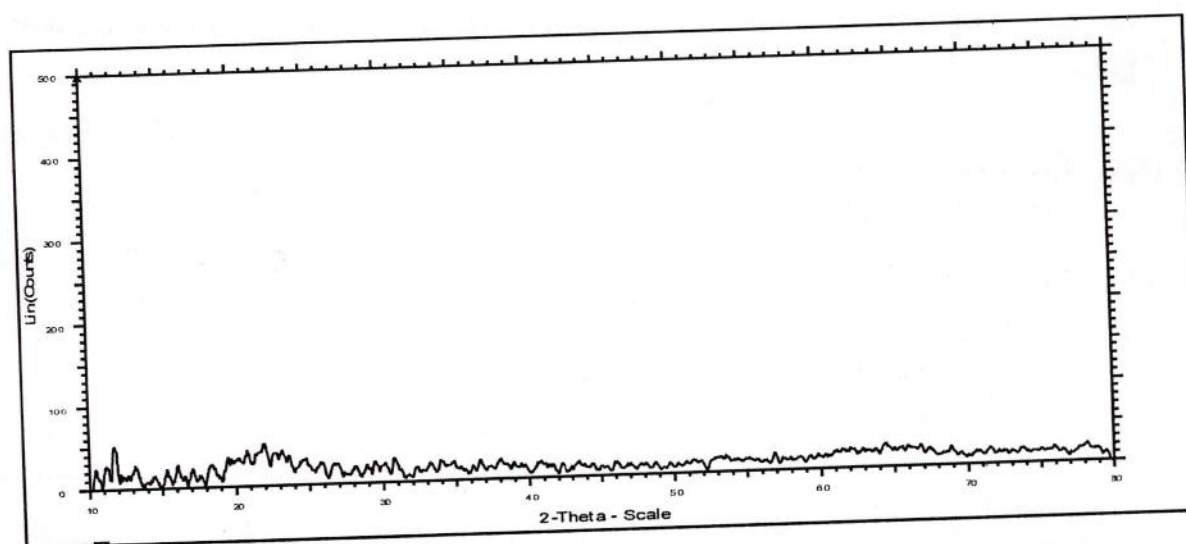


Figure-5: X-ray diffractogram of Ni (II) complex of (E)-1-(2,6-dihydroxyphenyl)-3-(5-methyl furan-2-yl)prop-2-en-1-one

### 3.7 Antimicrobial activity:

Antimicrobial activity was assayed by cup plate agar diffusion method by measuring inhibition zones in mm. In vitro antimicrobial activity of all synthesized compounds and standard have been evaluated against strains of The fungal toxicity of Ni(II) complex was studied *in vitro* against *Aspergillus niger* ATCC 16404, *Saccharomyces cerevisiae* ATCC 9763, *Candida albicans* ATCC10231 fungal pathogens at fixed 1% concentration.

The antibacterial activity of Ni(II) complex was studied, for evaluating antibacterial activity Gram positive and Gram negative bacterial pathogens were used. *Staphylococcus aureus* ATCC 6538, *Bacillus megaterium* ATCC 2326, *Bacillus subtilis* ATCC 6633 were Gram positive pathogens used in this study. *Escherichia coli* ATCC8739, *Salmonella typhi* ATCC9207, *Shigella boydii* ATCC 12034,





- [5] Omar. H. Al-Obaidi, Synthesis, spectral characterization, theoretical evaluation of new Co(II) and Mn(II) complexes of flavones., *Journal of Applicable Chemistry*, 3(1), 157-163 (2014)
- [6] S. N. Ipper, G. K. Sanap and G. K. Kakade, Synthesis, spectral Study, characterization and antimicrobial activity of Zinc(II) Complex of chalcone of pyridine-2-Carbaldehyde. *IJRASET*, 7(IX), 453-458 (2019)
- [7] S. N. Ipper, N. K. Raut and G. K. Kakde, Physico-chemical properties of furan chalcone. *Ind. J. Appl. Res.* 7(6), 683-684 (2017)
- [8] K.G. Karamunge, Y.B. Vibhute, Synthesis and structural studies of complexes of Cu(II), Ni(II) and Co(II) with 2-hydroxy chalcones; *Int. J. Chem. Sci.* 10(1), 490-500 (2012)
- [9] M.S. Suresh, V. Prakash, Preparation and characterization of Cr(III), Mn(II), Co(II), Ni(II), Cu(II), Zn(II) and Cd(II) chelates of schiff's bases derived from vanillin and 4-amino antipyrine. *Int. J. phy. Sci.*, 5(14), 2203-2211 (2010)
- [10] Muneerah M. Al-Mogren and Abdel-Nasser M.A. Alaghaz, Synthesis, spectral and quantum chemical calculations of mononuclear Nickel(II), Copper(II) and Cadmium(II) complexes of new Schiff-base ligand. *Int. J. Electrochem. Sci.*, 8, 8669-8685 (2013)
- [11] T. Akaharia, *Sci. Papers Inst. Phy. Chem. Res.*, 9, 165 (1928)
- [12] A. A. Frost, R. G. Pearson, "Kinetics and Mechanism", John Wiley, New York. (1961)
- [13] S. K. Sengupta, B. K. Pandey, B. K. Shrivastava and V. K. Sharma, *Transition Metal Chem.*, 23, 4, 349-353 (1998).



*Enterobacter aerogenes* ATCC13048, *Pseudomonas aerogenosa* ATCC9027, *Salmonella abony* NCTC6017 were the Gram-negative pathogens used in this study.

From the results of antimicrobial activity of ligands and complex it is clear that the complex shows enhanced activity than ligand. The increase in antimicrobial activity is due to faster diffusion of metal complexes as a whole through the cell membrane or due to the combined activity of the metal and ligands [13].

#### **Conclusion:**

The Ni (II) complex was colored, soluble in most of the organic solvent. The stoichiometry ratios of the metal complexes are obtained has been found to be 1:2. Solution conductivity of this metal complex reveals nonelectrolytic nature. The electronic spectral data, magnetic moment, TG-DTA suggests that Ni(II) has Octahedral geometry. The CHO analysis gives C, H, and O percentage in the metal complex. The XRD parameters shows that the structure of Ni(II) is Monoclinic and has space group = P2/m. From the antimicrobial activity of ligand and complex it is clear that the complex shows enhanced antimicrobial activity than ligand.

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#### **References:**

- [1] S. V. Kostanecki and Tambar, J. Chem. Ber., 32, 1921 (1899)
- [2] N. V. Damme, V. Zaliskyy, A. J. Lough, M. T. Lemaire, Polyhedron, 89, 155-159 (2015)
- [3] Omar. H. Al-Obaidi, Study of the cytotoxicity effect of new Co(II), Mn (II) Ni(II), and Cu(II) complexes of chalcone on cancer and antimicrobial activity, PCE, 1(2),14-18 (2014)
- [4] S. I. Habib, P. A. Kulkarni, XRD analysis of transition metal complexes of substituted heterocyclic chalcones. World Journal of Pharmaceutical Research, 6(7), 650-657 (2017)





## Aliquat 366: An Efficient Catalyst For Knoevenagel Condensation Under Solvent- Free Condition

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### Abstract:

Aliquat 366 phase transfer catalyst has been utilized as a novel and efficient catalyst for the Knoevenagel condensation of aldehydes with active methylene compounds such as ethyl cyanoacetate, cyanoacetamide and malononitrile to afford substituted olefins. The reaction proceeds smoothly under mild and solvent-free conditions at room temperature and the products are obtained in excellent yields.

**Keywords:** Aliquat 366, Knoevenagel condensation, Grinding.

### Introduction:

In recent years, solid-state organic reactions have caused great interests, which have many advantages such as high efficiency and selectivity, easy separation and purification, mild reaction conditions, and benefit to industry as well as environment. Organic synthesis in the absence of solvent is a powerful tool for the generation of structurally diverse molecules, due to their special selectivity, the ease of set-up and work-up, arousing great interest [1-3]. Moreover, solvent-free reactions sometimes are faster, taking just a few minutes rather than hours to complete, because the reactants are close contact with each other. This aspect, coupled with the lower overall costs of running a reaction without solvent and no specially needed equipment, could become a decisive factor.

Knoevenagel condensation reactions have been extensively studied as an important carbon-carbon bond forming reaction. This condensation, various catalyst are used such as Te(IV)Cl<sub>4</sub> [3], Ammonium acetate [4], ZnCl<sub>2</sub> [5], KSF-montmorillonite [6], KF-Al<sub>2</sub>O<sub>3</sub> [7], Ethylenediammonium diacetate [8], CaO [9], Mg-Al-*t*-Bu-Hydrocalcite [10], Phosphene [11],



ionic liquid [bmim][BF<sub>4</sub>] [12], PEG400 and anhydrous K<sub>2</sub>CO<sub>3</sub> [13]. In recent years, chemists paid more and more attention to the clean synthesis of alkenes by condensation reactions.

Therefore, the introduction of efficient and new methods based on green methodology is still in demand. In continuation of our ongoing research to develop newer, environmentally benign methods Aliquat 336 is used as efficient catalyst for the Knoevenagel condensation of aldehydes with active methylene compounds such as ethyl cyanoacetate, cyanoacetamide and malononitrile to afford substituted olefins. Aliquat 336 is a water insoluble quaternary ammonium salt made by the methylation of tri octyl/decyl amine, which is capable of forming oil soluble salts of an ionic species at neutral or slightly alkaline pH. Aliquat 336 is a versatile and affordable cation source for an entirely new family of hydrophobic ionic liquids [14]. It can form salts with anions over a wider pH range than primary, secondary or tertiary amines. For this reason Aliquat 336 finds application in environments from acid to slightly alkaline pH. It is used as a phase transfer catalyst [15].

### Results and Discussion:

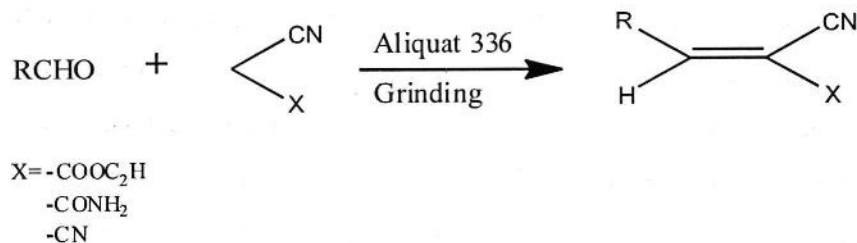
To investigate the role of catalyst and effect of grinding, first the reaction of p-chlorobenzaldehyde, ethylcyanoacetate and catalytic amount of Aliquat 336 are ground together in mortar and pestle at room temperature, the reaction starts immediately usually with gentle heat production, the reaction mixture turned into brownish viscous liquid then to thick brownish mass and finally to free flowing powder. Surprisingly, reaction mixture turned into the desired product within a short period.

On the other hand, indication of softening for some seconds followed by immediate hardening were visually observed only in syntheses with p-chlorobenzaldehyde with malononitrile (entry 3r). Subsequently, the scope of the Knoevenagel condensation of other aldehydes with various active methylene compounds catalyzed by Aliquat 336 scheme-1 in grinding was investigated. We found that the Knoevenagel condensation of aldehydes with malononitrile occurred easily in the presence of grinding to form corresponding products. Both electron-rich and electron-deficient aldehydes worked well to give the corresponding arylidene derivatives in excellent yields (3a-s) in Table-1. The results are summarized in table 1. Miscibility of Aliquat 336 with water makes the workup process quite easy as the





catalyst can be easily removed from product simply by washing the product with water.



Scheme-1: Knoevenagel condensation using Aliquat 336 catalyst

Table 1: Aliquat 336 catalyzed Knoevenagel condensation under solvent-free condition

Entry <sup>a</sup>	-R	-X	Time	Yield <sup>b</sup> (%)	Melting point (°C)
3a	C <sub>6</sub> H <sub>5</sub>	COOC <sub>2</sub> H <sub>5</sub>	3 min.	92	52
3b	4-ClC <sub>6</sub> H <sub>4</sub>	COOC <sub>2</sub> H <sub>5</sub>	2 min.	96	163
3b	4-ClC <sub>6</sub> H <sub>4</sub>	COOC <sub>2</sub> H <sub>5</sub>	10 min	89	163
3c	2-CH <sub>3</sub> OC <sub>6</sub> H <sub>4</sub>	COOC <sub>2</sub> H <sub>5</sub>	5 min.	92	69
3d	4-H <sub>3</sub> COC <sub>6</sub> H <sub>4</sub>	COOC <sub>2</sub> H <sub>5</sub>	3 min.	93	99
3e	4-OHC <sub>6</sub> H <sub>4</sub>	COOC <sub>2</sub> H <sub>5</sub>	5 min.	90	171
3f	4-NO <sub>2</sub> C <sub>6</sub> H <sub>4</sub>	COOC <sub>2</sub> H <sub>5</sub>	4 min.	95	171
3g	C <sub>6</sub> H <sub>5</sub> -CH=CH-	COOC <sub>2</sub> H <sub>5</sub>	3 min.	92	114
3h	Vanillin	COOC <sub>2</sub> H <sub>5</sub>	5 min.	94	112
3i	2-furyl	COOC <sub>2</sub> H <sub>5</sub>	3 min.	96	84
3j	C <sub>6</sub> H <sub>5</sub>	CONH <sub>2</sub>	2 min.	92	120
3k	4-ClC <sub>6</sub> H <sub>4</sub>	CONH <sub>2</sub>	2 min.	93	210
3l	3-CH <sub>3</sub> OC <sub>6</sub> H <sub>4</sub>	CONH <sub>2</sub>	3 min.	95	204
3m	4-CH <sub>3</sub> OC <sub>6</sub> H <sub>4</sub>	CONH <sub>2</sub>	3 min.	94	215
3n	Vanillin	CONH <sub>2</sub>	3 min.	93	206
3o	4-NO <sub>2</sub> C <sub>6</sub> H <sub>4</sub>	CN	3 min.	95	84
3p	4-CH <sub>3</sub> OC <sub>6</sub> H <sub>4</sub>	CN	2 min.	94	80
3q	3-CH <sub>3</sub> OC <sub>6</sub> H <sub>4</sub>	CN	1 min.	94	92
3r	4-ClC <sub>6</sub> H <sub>4</sub>	CN	2 min.	93	165
3s	2-furyl	CN	2 min.	95	64

<sup>a</sup>All the products are characterized by MP, IR and <sup>1</sup>H NMR

<sup>b</sup>isolated yields after recrystallized



### Experimental section:

Melting points were determined in open capillary tubes and are uncorrected. The purity of compounds was checked by Merck silica gel TLC plates, and the spots were detected by UV light absorption. The IR spectra were recorded in matrix of KBr with Perkin-Elmer spectrometer.  $^1\text{H}$  NMR spectra were recorded on Varian NMR Spectrometer, Model Mercury Plus (400MHz) and the chemical shifts ( $\delta$ ) are given in ppm relative to signal for TMS as an internal standard. All the products were identified by comparison of analytical data (melting point, IR and  $^1\text{H}$  NMR.) with those reported of authentic samples.

### General procedure:

An equimolar quantity of the aldehydes (1 mmol), and active methylene compounds (1 mmol) mixed thoroughly and then 0.1 mmol of Aliquat 336 was added. The reaction mixture was ground in the mortar and pestle for 30 sec. to 5 min. at appropriate time mentioned in table 1. The reaction was monitored by TLC, on completion of reaction; the reaction mixture was treated with cold water. The product was filtered, dried, and the crude compounds were recrystallized from a mixture of ethanol and water to get the desired compound in pure form (3a-s).

### Conclusion:

This new method is superior to the reported methods [5,9,10] with respect to the reaction time and simplicity of the procedure. The attractive features of this procedure are the mild and solvent-free reaction conditions, high conversions, cleaner reaction profiles and operational simplicity, all of which make it a useful and attractive strategy for the preparation of arylidene derivatives.

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**References:**

- [1] K. Tannaka and F. Toda, Chem. Rev. 100 (2000)1025.
- [2] D. Villemain and B. Martin, Synth. Commun. 25 (1995)3135.
- [3] R. H. Khan, R. K. Mathur and A. C. Ghosh, Synth. Commun. 26(4) (1996)683.
- [4] A. K. Mitra, A. De and N. Karchaudhuri, Synth. Commun. 29(16) (1999)2731.
- [5] R. P. Shanthan and R. V. Venkataratnam, Tetrahedron Lett. 32(41) (1991)5821.
- [6] D-Q. Shi, X-S. Wang, C-S. Yao and L. Mu, J. Chem. Res. 7 (2002)344.
- [7] S-X. Wang, J-T. Li, W-Z. Yang and T-S. Li, Ultrason Sonochem. 9(3) (2002)159.
- [8] Ce. Su, Z-C. Chen and Q-G. Zheng, Synthesis. 4 (2003)555.
- [9] (a) Y. Peng and G. Song, Ind. J. Chem. 42B (2003) 924. (b) Y. Lu, Z. Ren, W. Cao, W. Tong and M. Gao, Synth Commun. 34 (2004)2047.
- [10] B. M. Choudary, M. Lakshmi Kantam, B. Kavita, Ch. Venkat Reddy and F. Figueras, Tetrahedron. 56(47) (2000) 9357.
- [11] J. S. Yadav, B. V. Subba Reddy, A. K. Basak, Boddapati Visali, Akkerala Venkat Narsaiah and Kommu Nagaiah, Eur. J. Org. Chem. (2004)546.
- [12] X. Fan, X. Hu, X. Zhang and J. Wang, Aust. J. Chem. 57(11) (2004)1067.
- [13] Y-Q. Cao, Z. Dai, R. Zhang and B. Hua, Synth. Commun. 34(16) (2004)2965.
- [14] J. Mikkola; P. Virtanen; R. Sjöholm, Green Chem., 8, 250, (2006).
- [15] K. Sato; M. Aoki; Science, 281, 1646, (1998)

*Theme- New horizons in chemical sciences.*

*Guest Editor- R.P. Pawar*

**Synthesis, spectral study, characterization of cobalt (II) and copper (II) complexes of chalcones.**

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**ABSTRACT**

Metal complexes of Co(II) and Cu(II) have been synthesized with newly prepared biologically active ligands. These ligands were prepared by the Claisen-Schmidt condensation method of 2-6, dihydroxy acetophenone and 5-methyl-2-furaldehyde. The structures of the complexes have been proposed by the analytical data, conductivity measurement, magnetic moment, IR spectrum, Electron absorption spectrum, thermal studies, and XRD analysis. Analytical data confirmed 1:2 stoichiometry and the electronic spectral data, IR, magnetic moment, TG-DTA suggests that Co(II) and Cu(II) complexes have octahedral geometry. The presence of coordinated water molecules in Co(II) and Cu(II) complexes is confirmed by TG-DTA studies. The conductivity data show that all these complexes are non-electrolytes. Antimicrobial activities of complexes with selected bacterial strain and fungal strain carried out and the results have been compared with commercial standards. In this paper, we prepare chalcone of 5-methyl furfural by Claisen-Schmidt condensation method and synthesize Co(II) and Cu(II) metal complexes and characterize them by Infra-red, Electronic absorption spectra, magnetic susceptibility, CHO analysis, solution conductivity, XRD study, TG-DTA, and antimicrobial activity.

**KEYWORDS**

Transition metal complex, Electron absorption spectrum, Infrared spectrum, TG-DTA.



## 1. INTRODUCTION

Chalcones constitute an important group of natural products, chemically they consist of open chain flavonoids in which the two aromatic rings are joined by  $\alpha, \beta$  unsaturated carbonyl system. The name chalcone is given by Kostanecki and Tambar[1]. Cobalt(II) and Copper(II) complexes of chalcones are synthesized and studied in the literature [2-3]. It is believed that the ( $>CO-C=C<$ ), moiety imparts biological characteristics to this class of compounds. Such  $\alpha, \beta$ -unsaturated carbonyl compounds, and their metal complexes possess interesting biochemical properties, such as antitumor, antioxidant, anti-fungal and antimicrobial activities[4]. The electronic absorption spectrums, infrared spectrum, magnetic moment, TG-DTA support the octahedral geometry of the metal complexes of chalcones. All crystals of a substance possess the same elements of symmetry. The atoms in the plane are responsible for the scattering of X-rays. The spacing  $d$  depends on the dimensions of the unit cell. The relative intensities of orthorhombic, monoclinic, and triclinic are characterized by 3, 4 or 6 lattice constants. The powder XRD method is used in the present investigation for crystal structure determination. It gives information about inter atomic distance[5]. The pycnometric densities of each of the complexes were determined using a specific gravity bottle by the principle of Archimedes[6]. Depending on these values of axial length and axial angles the crystal is classified as cubic, tetragonal, orthorhombic, trigonal, hexagonal, monoclinic and triclinic structures. The computer program, used for indexing data was powder-X. In this program all the essential features of X-ray program are presented and in addition it calculates the deviation in lattice parameter  $a, b, c$  in Å and  $\alpha, \beta$  and  $\gamma$  in degree and minutes with better combination of  $h, k, l$  values until the final deviation is within the permissible limit of 2%. The refined values also give the volume of the unit cell. By using the literature value for a definite crystal system, the probable space group was assigned to each of the complexes. Furthermore, biological activities of complexes with selected bacterial strain and fungal strain carried out and the results have been compared with the commercial standard[7]. The X-ray powder diffractogram of the metal complexes was used for the structural characterization and determination of lattice dimensions. In this paper, we prepare chalcone of 5-methyl furfural by Claisen-Schmidt condensation method and synthesize Co(II) and Cu(II) metal complexes and characterize them by Infra-red, Electronic absorption spectra, magnetic susceptibility, CHO analysis, solution conductivity, XRD study, TG-DTA, and antimicrobial activity.

## 2. MATERIALS AND METHODS

### 2.1. Synthesis of chalcone of 5-methyl furfural

The reagents used for the preparation of furan chalcone are of A.R. grade. The mixture of 2,6-dihydroxy acetophenone (0.01 mol) and 5-methyl-furaldehyde (0.01 mol) are dissolved in ethanol (20ml) and then sodium hydroxide 10ml (40%) was added to it. The mixture was stirred for 3 hours till brown colored precipitate was observed. The progress of the reaction was monitored by TLC. After completion of the reaction, the contents were poured into ice water and

then acidified by dil. HCl. The solid obtained was filtered and the crude product was recrystallized from ethanol to give the chalcone [8-9].

### 2.2. Synthesis of metal complex

The solution of 0.02 mole of chalcone of 5-methyl furfural was taken in a round bottom flask containing 30 ml of anhydrous methanolic solution of chalcone of 5-methyl furfural was used and boiled for 10 minutes. A hot solution of 0.01 mole, of Cu(II) sulfate and Co(II) nitrates in 20 ml of methanol was added drop wise to the solution of the chalcone of 5-methyl furfural. To this reaction mixture, 10% alcoholic ammonia was added up to slightly alkaline pH. The complexes of different metals were precipitated at different pH range. The pH8-10 range was definite for these complexes.

The contents were stirred on a magnetic stirrer for one hour. The solid metal complex separated and washed with methanol three to four times. Dried in vacuum desiccators over anhydrous granular calcium chloride. The melting point/decomposition temperatures of the complexes were determined by Thiele's melting apparatus. The reactions of the formation of Co(II) and Cu(II) complexes are shown in figure (1). The IR spectrum was measured at CIL, Chandigarh, Punjab, Electron absorption spectrum at Sunderrao Solanke Mahavidyalaya Majalgaon and CHO analysis, TG-DTA and XRD measured at SAIF Cochin, Kerala.

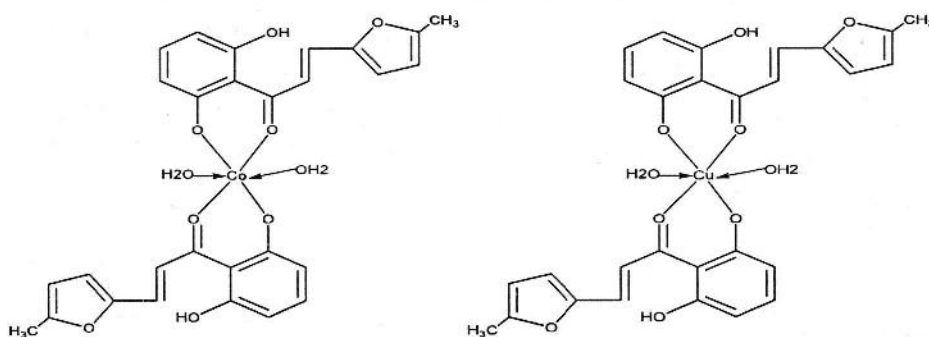


Fig.1. Metal complexes of cobalt (II) and copper (II) with chalcone of 5-methyl furfuraldehyde.

## 3. RESULTS AND DISCUSSION

### 3.1. Infra-red spectrum

#### The infrared spectrum of Co(II) complex

The infrared spectra of ligands and Co(II) complex of chalcone of 5-methyl furfural were recorded on a Perkin-Elmer Spectrum RX-IFTIR Spectrophotometer over the range 4000-400  $\text{cm}^{-1}$  using KBr pellet at CIL, Chandigarh, Punjab. The number in the spectrum of the complex indicates coordination through the oxygen of (C=O) group. The M-O bond for Co(II) complex is observed at 510-600 $\text{cm}^{-1}$ [10]. The presence of phenolic -OH is confirmed by peaks at 3420 $\text{cm}^{-1}$  and 3037  $\text{cm}^{-1}$  in the ligand, In the spectrum of Co(II) complex, there is complete disappearance of peaks at 3420 $\text{cm}^{-1}$  and 3037  $\text{cm}^{-1}$  in chalcone suggesting absence of phenolic group -OH indicates its co-ordination,(C-O-C) is shifted to a lower wave number comparing with free ligand. In the IR spectrum of Co(II) complex, the strong bands that appeared in the region 1623



$\text{cm}^{-1}$  are assigned to  $\nu$  (C=O) of stretching frequency. It is shifted towards lower frequency than the corresponding ligand and appeared at 1590 and 1611  $\text{cm}^{-1}$  in metal complexes. Such a lowering in stretching vibration of  $\nu$  (C=O) in chalcone indicates the participation of chalcone carbonyl in complexation. In the IR spectrum of Co(II) complex: the new bands are observed at 3263  $\text{cm}^{-1}$  and 3162  $\text{cm}^{-1}$  which are due to the -OH intense broadband of co-ordinate water molecules. In Co(II) complex new band is observed at 510-600  $\text{cm}^{-1}$  due to the (M-O) bond. The IR spectrum of Cu(II) complex of chalcone of 5-methyl furfural is presented in figure (1) and their group absorption frequencies are given in Table 1.

Chiara Sulpizio assigned that, IR spectra of  $\text{Cu}^{2+}$ , the intensity of -OH vibration at 3300  $\text{cm}^{-1}$  reduced or broadened for copper complexes indicating that hydroxyl group would be interacting with metal. There is a shift of the carbonyl stretch from 1640 to 1610  $\text{cm}^{-1}$  for ligands in the presence of copper meaning that the bond strength of the C=O bond is weakened this is a strong indication for the participation of the carbonyl moiety in complexation process. Additionally, a new bond is observed at 500  $\text{cm}^{-1}$  in the spectra of metal complexes[11].

The presence of phenolic-OH in chalcone of 5-methyl furfural is confirmed by peaks at 3420 $\text{cm}^{-1}$  in the ligand. In the spectrum of Cu(II) complex, the appearance of a peak at 3223 $\text{cm}^{-1}$  in chalcone suggests the absence of phenolic group -OH indicates its co-ordination,(C-O-C) is shifted to a lower wave number comparing with free ligand [12]. In the IR spectrum of ligand, the strong band appeared in the region 1623  $\text{cm}^{-1}$  is assigned to  $\nu$  C=O) of stretching frequency. It is shifted towards lower frequency than corresponding ligands and appeared at 1613 $\text{cm}^{-1}$  in metal complexes. Such a lowering in stretching vibration of  $\nu$  (C=O) in chalcone indicates the participation of chalcone carbonyl in complexation. In Cu(II) a complex new band is observed at 480  $\text{cm}^{-1}$  due to the (M-O) bond.

**Table 1.** Infrared absorption frequencies ( $\text{cm}^{-1}$ ) of chalcone of 5-methyl furfural Co(II) and Cu(II) complexes.

Ligand/ Metal complexes	$\nu(\text{OH})$ $\text{cm}^{-1}$	$\nu(\text{H}_2\text{O})$ $\text{cm}^{-1}$	$\nu$ ((-CO- CH=CH- ) $\text{cm}^{-1}$	$\nu$ ((C- O-C) $\text{cm}^{-1}$	$\nu$ (C=C) $\text{cm}^{-1}$	Aromatic ring (C=C) $\text{cm}^{-1}$	$\nu(\text{M-O})\text{cm}^{-1}$
chalcone of 5-methyl furfural	3420	-	1623	1095	1558	1439	-
[Co(chalcone of 5-methyl furfural ) <sub>2</sub> ]	3003	3263	1590	1024	1383	1220	580
[Cu(chalcone of 5-methyl furfural ) <sub>2</sub> ]	3223	3223	1613	1024	1582	1435	480

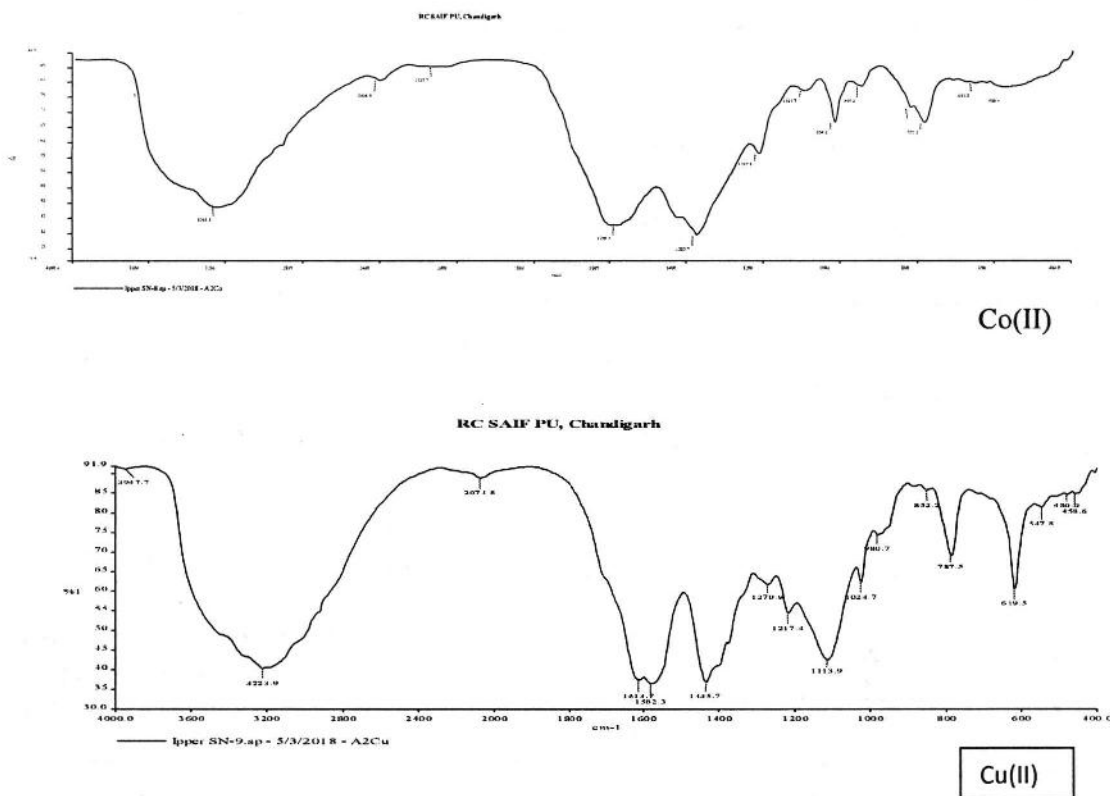


Fig. 2. Infrared spectra of Co(II) and Cu(II) metal complexes.

Table 2. Physical parameters of Cobalt (II) and Copper (II) complexes.

Metal complex	Ligand	p <sup>H</sup> range ppt.	Color	M.P. <sup>0</sup> C
cobalt(II) complex	chalcone of 5-methyl furfural	7.5 -8.5	coffee	>300
copper(II) complex	chalcone of 5-methyl furfural	7.0 - 7.5	brown	>300

**CHO analysis**

The carbon, hydrogen, oxygen, cobalt and copper metals percentage in Co(II) and Cu(II) complexes of chalcone measured at SAIF Cochin, Kerala. The calculated and measured values of CHO analysis are matching and are given in table no.(3).



Table 3. CHO analysis data.

Metal complexes	Chemical formula	Mol. Wt.	Elemental analysis: % found (calculated)						
			C	H	N	O	S	X(Br)	M
Co (II) complex	[C <sub>28</sub> H <sub>26</sub> O <sub>6</sub> Co]	517	64.89 (64.99)	5.04 (5.06)	-	18.59 (18.55)	-	-	11.29 (11.39)
Cu (II) complex	[C <sub>28</sub> H <sub>26</sub> O <sub>6</sub> Cu]	522	64.38 (64.42)	4.98 (5.01)	-	18.29 (18.38)	-	-	11.99 (12.17)

3.2. Magnetic susceptibility, solution conductivity, and electronic absorption spectral data  
Magnetic susceptibility

The  $\mu_{\text{eff}}$  (B.M.) values at room temperature for Co(II) complex is 4.57 B.M. and Cu(II) 1.85 B.M., these values agree with octahedral geometry of the metal complexes [13-14].

3.2.1. Solution conductivity

The solution conductivities of  $10^{-3}$  M solution of the metal complex in DMSO were measured on Equipronics digital conductivity meter EQ - 660 with  $20 \mu\Omega$  to  $200 \mu\Omega$  at 298K temperature. In the present investigation Co(II) and Cu(II) complexes are coffee and brown, stable to air and moisture. Decomposes at high temperatures rather than showing sharp melting points. They are insoluble in water and soluble in DMSO, DMF. The low conductivity values in the DMSO solution ( $10^{-3}$  M) are given in Table 4 indicates non-electrolytic nature.

3.2.2. Electronic absorption spectral study

The electronic absorption spectrum was measured on SL159, single beam UV-VIS spectrophotometer.

The electronic spectrum of Co(II) complex studied in the present investigation exhibit absorption bands at  $25641(390 \text{ nm})\text{cm}^{-1}$  and  $29411(340 \text{ nm})\text{cm}^{-1}$  which are assigned to  ${}^4T_{1g(F)} \rightarrow {}^4T_{1g(P)}$  and charge transfer bands respectively.

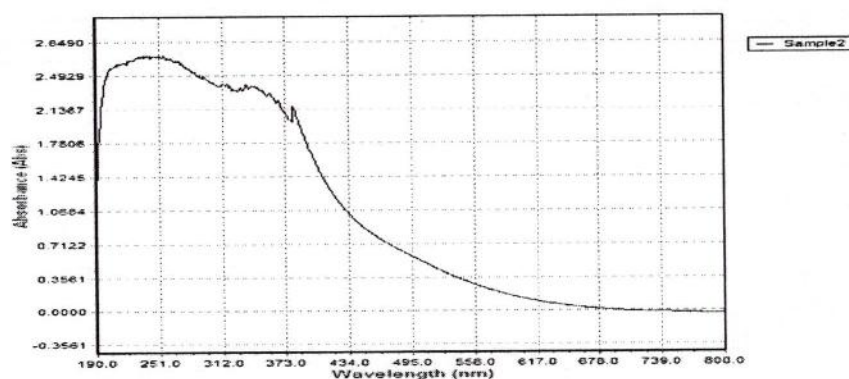


Fig. 3. Electronic absorption spectra of Co (II) complex of chalcone of 5-methyl furfural.

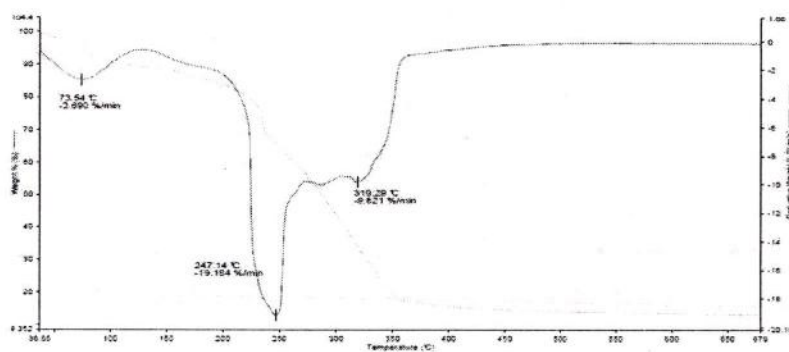
**Table 4.** Solution conductivity, magnetic and electronic absorption spectral data of Co(II) and Cu(II) complexes.

Ligand	Metal complex	Molar Conductance $\text{Ohm}^{-1} \text{cm}^2 \text{mol}^{-1}$	$\mu_{\text{eff}}$ (BM.)	Absorption maxima $\text{cm}^{-1}$ (nm)	
				${}^4\text{T}_{1\text{g}}(\text{F}) \rightarrow {}^4\text{T}_{1\text{g}}(\text{P}),$ ${}^2\text{B}_{1\text{g}} \rightarrow {}^2\text{E}_{\text{g}}$	Charge transfer
chalcone of 5-methyl furfural	Co(II) complex	4.37	4.57	25641(390)	29411(340)
chalcone of 5-methyl furfural	Cu(II) complex	3.0	1.85	25906(386)	34246 (292)

**3.2.3. Thermal analysis Co(II) complex chalcone of 5-methyl furfural**

The simultaneous thermogravimetric, differential thermal analysis of Co(II) complex chalcone of 5-methyl furfural was performed in an inert nitrogen atmosphere on Perkin Elmer STA 6000 at SAIF, Cochin, Kerala. The heating rate was  $10^\circ/\text{min}$  and the flow rate of nitrogen 50 ml/min. The reference substance used was  $\alpha \text{Al}_2\text{O}_3$  in a platinum crucible and the sample weighed in the range of 4-12 mg. The thermogram of Co(II) complex of chalcone of 5-methyl furfural is presented in figure (3). This curve reveals that there is a presence of lattice as well as coordinated water in the complex.

The TG curve of Co(II) complex of chalcone of 5-methyl furfural(Fig.4) shows first weight loss at  $73.54^\circ\text{C}$  indicating the presence of lattice water with weight loss 5.54% (calc. wt. loss 6.02%). The second loss due to a coordinated water molecule in the complex takes place at  $150^\circ\text{C}$ . In the third step loss of non-coordinated two fragments of 5-methyl-furan part of ligand takes place. In the fourth step weight loss of 31.74% (calc. wt. loss 31.03%) within the temperature range  $300-350^\circ\text{C}$ . which is authenticated by broad endotherm in the DTA curve at  $319.29^\circ\text{C}$ . The last stage decomposition occurs with a mass loss of 24.66% (calc. wt. loss 24.13%) in the range  $360-510^\circ\text{C}$ , due to the loss of two enone part of chalcones fragment. This is authenticated by broad endotherm in the DTA curve. Above  $550^\circ\text{C}$ , the TG curve attains a constant level corresponding to its metal oxide (CoO).



**Fig. 4.** TG-DTA curve of Co(II) complex of chalcone of 5-methyl furfural.



### 3.2.4. X-ray diffraction spectral studies of metal complexes

#### Cobalt (II) complex

The XRD spectral study has been done at SAIF, Cochin, Kerala. The X-ray diffraction patterns of Co(II) complex is shown in (Fig. 4). The standard deviation observed is within the permissible range. The observed and calculated densities of Co(II) complex of chalcone of 5-methyl furfural are  $1.909 \text{ gcm}^{-3}$  and  $1.816 \text{ gcm}^{-3}$  respectively. Chalcone of 5-methyl furfural of Co(II) complex is found to be monoclinic lattice type with space group P2/m and lattice parameters are  $a=8.9169 \text{ \AA}$ ,  $b=7.6183 \text{ \AA}$ ,  $c=5.4089 \text{ \AA}$ ,  $\alpha=90^\circ$ ,  $\beta=98^\circ$ ,  $\gamma=90^\circ$  satisfies the condition  $\alpha=\gamma=90^\circ$ ,  $\beta\neq 90^\circ$  unit cell volume  $V=231.24 \text{ \AA}^3$ .

#### Copper (II) complex

The Cu(II) complex of chalcone of 5-methyl furfural was studied by X-ray powder diffraction (Fig.6). The standard deviation observed was within the permissible range. The observed and calculated densities are  $1.7795 \text{ gcm}^{-3}$  and  $1.9753 \text{ gcm}^{-3}$  respectively. The Cu(II) complex of chalcone of 5-methyl furfural is triclinic lattice-type P. For this complex lattice parameters are  $a=11.23 \text{ \AA}$ ,  $b=13.12 \text{ \AA}$ ,  $c=17.00 \text{ \AA}$ ,  $\alpha=85^\circ$ ,  $\beta=81^\circ$ ,  $\gamma=82^\circ$ ,  $V=753 \text{ \AA}^3$ . These values match with the study of Cu(II) complexes of chalcone derivatives were synthesized by Salima Tabti and et al.[15]. The values of unit cell data and crystal lattice parameters for Co(II) and Cu(II) complexes calculated from indexed X-ray diffraction data are as given below.

#### Unit cell data and crystal lattice parameters for Co(II)

$a (\text{ \AA}) = 8.9169$ ,  $b (\text{ \AA}) = 7.6183$ ,  $c (\text{ \AA}) = 5.4089$ ,  $\alpha = 90^\circ$ ,  $\beta = 98^\circ$ ,  $\gamma = 90^\circ$ , Standard deviation (%) = 0.18, Volume (V) =  $231.24 \text{ \AA}^3$ , Density (obs.) =  $1.909 \text{ gcm}^{-3}$ , Density (cal.) =  $1.816 \text{ gcm}^{-3}$ , Z = 1, Crystal system = Monoclinic and Space group = P2/m.

#### Cu(II)

$a (\text{ \AA}) = 11.23$ ,  $b (\text{ \AA}) = 13.12$ ,  $c (\text{ \AA}) = 17.0$ ,  $\alpha = 85^\circ$ ,  $\beta = 81^\circ$ ,  $\gamma = 82^\circ$ , Standard deviation (%) = 0.17, Volume (V) =  $753.05 \text{ \AA}^3$ , Density (obs.) =  $1.7795 \text{ gcm}^{-3}$ , Density (cal.) =  $1.9753 \text{ gcm}^{-3}$ , Z = 2, Crystal system = triclinic and Space group = P2/m.

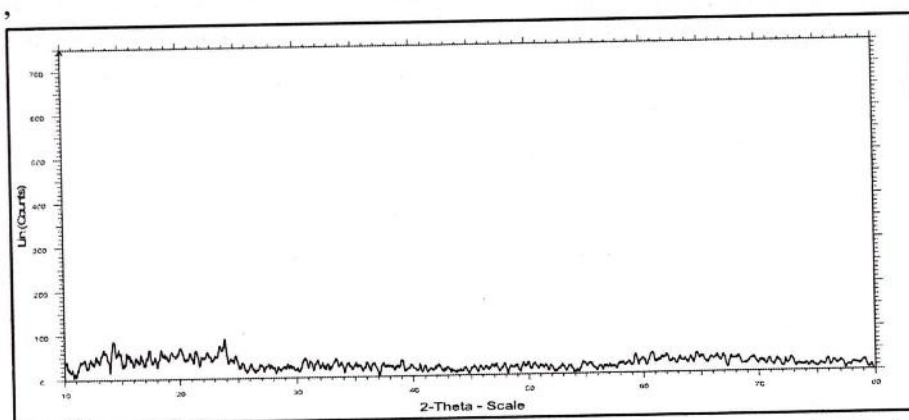


Fig. 5. X-ray diffractogram of Co(II) complex of chalcone of 5-methyl furfural.

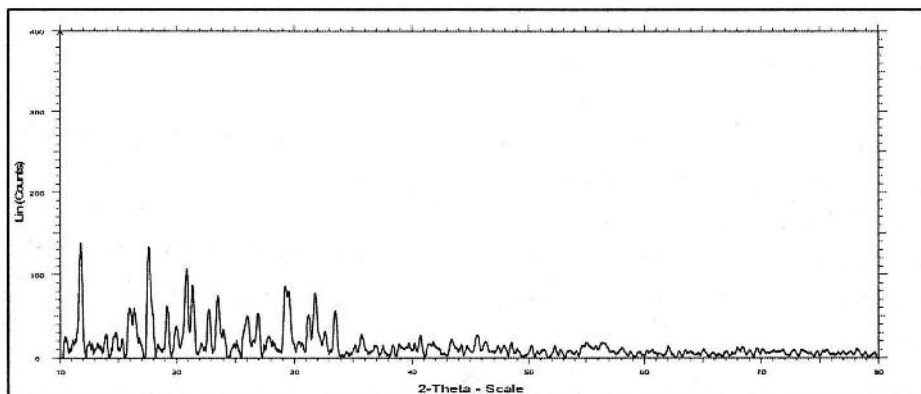


Fig. 6. X-ray diffractogram of Cu(II) complex of chalcone of 5-methyl furfural.

### 3.3. Antimicrobial activity

Antimicrobial activity was assayed by the cup plate agar diffusion method [15] by measuring inhibition zones in mm. In vitro antimicrobial activity of all synthesized compounds and standards have been evaluated against strains of bacteria *Bacillus cereus*, *Staphylococcus aureus* and strains of fungi *Aspergillus niger*, *Candida albicans* the standard used was penicillin and Griseofulvin. From the results of antimicrobial activity of ligands and complexes it is clear that the complexes show enhanced activity than ligands. The increase in antimicrobial activity is due to faster diffusion of metal complexes as a whole through the cell membrane or due to the combined activity of the metal and ligands.

### 4. CONCLUSION

The Co(II) and Cu(II) complexes are colored, insoluble in most of the organic solvent but soluble in an organic solvent. The stoichiometry ratio of the metal complexes obtained is 1:2. The solution conductivity of these metal complexes reveals a non-electrolytic nature. The infrared spectral data indicate that the ligand act as mono negative bidentate species towards Co(II) and Cu(II) complexes. The electronic spectral data, IR spectrum, magnetic moment, TG-DTA suggests that Co(II) and Cu(II) has octahedral geometry. The CHO analysis gives C, H, and O percentages in the metal complexes. The XRD parameters show that the structure of Co(II) is monoclinic with space group = P2/m and that of Cu(II) is triclinic and has space group = P2/m.

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## 6. REFERENCES

1. Kostanecki, S.V. (1899). *J.Chem.Ber.*,32, 1921.
2. Van Damme, N., Zalisky, V., Lough, A. J., &Lemaire, M. T. (2015). Structure and magnetic properties of a cobalt (III) complex with redox-active 1-(2-pyridylazo)-2-phenanthrol (papl). *Polyhedron*, *89*, 155-159.
3. Layek, M., Ghosh, M., Sain, S., Fleck, M., Muthiah, P. T., Jenniefer, S. J.,& Bandyopadhyay, D. (2013). Synthesis, crystal structure and magnetic properties of nickel (II) and cobalt (III) complexes of a pentadentate Schiff base. *Journal of Molecular Structure*, *1036*, 422-426.
4. Patange, V. N., & Arbad, B. R. (2011). Synthesis, spectral, thermal and biological studies of transition metal complexes of 4-hydroxy-3-[3-(4-hydroxyphenyl)-acryloyl]-6-methyl-2H-pyran-2-one. *J. Serb. Chem. Soc*, *76*(9), 1237-1246.
5. Shah, S.S., Goswami, K. (2014). Synthesis, characterization and biological activity of some novel chalcone compounds and their Cobalt and Nickel complexes having benzyloxy dibromo acetophenone moiety, *Der Pharmacia letter*, *6*(3):304-312.
6. Kubaisi, A. A., & Ismail, K. Z. (1994). Nickel (II) and palladium (II) chelates of dehydroacetic acid Schiff bases derived from thiosemicarbazide and hydrazinecarbodithioate. *Canadian journal of chemistry*, *72*(8), 1785-1788.
7. Deshmukh AJ, Patil RM, LokhandeRS, Ner DH, (2017) Synthesis, characterization and antimicrobial activity of copper (II) complexes of some 2-hydroxy-4, 5-dimethyl substituted chalcones, *International Journal of Advanced Research and Review*, *3*(1), 27-31.
8. Shastri, R. A. (2015). Synthesis and Antimicrobial Activity of Some Novel Pyrazoline and Flavone Derivatives Derived From Furan Chalcones. *World Journal of Pharmaceutical Research*, *4*, 642-651.
9. Pouralimardan, O., Chamayou, A. C., Janiak, C., & Hosseini-Monfared, H. (2007). Hydrazone Schiff base-manganese (II) complexes: Synthesis, crystal structure, and catalytic reactivity. *Inorganica Chimica Acta*, *360*(5), 1599-1608.
10. Chiara, Sulpizio., Simon, T.R., Muller, Zhang Qi., Brecker, Lothar., Rampal, Annte. (2016). Synthesis, Characterization and Antioxidant activity of Zn<sup>2+</sup> and Cu<sup>2+</sup> coordinated polyhydroxychalcone complexes, *Montash Chem.* *147*:1871-1881.
11. Ipper, S.N., Raut, N.K., Dixit, P.P., Kakade, G.K., (2018). Synthesis, spectral study and properties of (E)-3-(5-bromofuran-2-yl)-1-(2,6-dihydroxyphenyl)prop-2-en-1-one, *Journal of Pharma Research*, *7*(10), 220-223.
12. Mane, P.S., Shirodkar, S.G., Chondhekar, T.K. (2002). *J. Indian Chem. Soc.*, *79*(A), 154.
13. Habib, S. I., Shah, S., Aseer, N. N. M. A., & Kulkarni, P. A. (2011). Synthesis and characterization of cobalt (ii), nickel (ii), and copper (ii) complexes of some 2'-hydroxy chalcones. *J. Chem. Pharm. Res*, *3*, 788-792.
14. Tabti, S., Djedouani, A., Aggoun, D., Warad, I., Rahmouni, S., Romdhane, S., & Fouzi, H. (2018). New Cu (II), Co (II) and Ni (II) complexes of chalcone derivatives: Synthesis,


*Curr. Pharm. Res.* **2019**, 416, 123-133

X-ray crystal structure, electrochemical properties, and DFT computational studies.  
*Journal of Molecular Structure*, 1155, 11-20.

15. Barry, A. L. (1991). Procedures and Theoretical Considerations for testing Antimicrobial Agents in agar media. *Antibiotics in laboratory medicine*.

  
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