Ph.D Thesis entitled

CATALYSIS & NANOCATALYSIS FOR GREEN ORGANIC TRANSFORMATIONS

Submitted by

YOGITA MADAN

2009RCY103

Department of Chemistry

MALAVIYA NATIONAL INSTITUTE OF TECHNOLOGY JAIPUR

July, 2014

Ph.D Thesis entitled

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SUBMITTED IN PARTIAL FULFILMENT OF THE REQUIREMENTS
FOR THE AWARD OF THE DEGREE OF

DOCTOR OF PHILOSOPHY
IN
CHEMISTRY

SUBMITTED BY

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July, 2014

Supervisors' Certificate

This is to certify that the work reported in this thesis entitled **"CATALYSIS & NANOCATALYSIS FOR GREEN ORGANIC TRANSFORMATIONS"** has been carried out by **Ms. YOGITA MADAN** under my supervision for the degree of **DOCTOR OF PHILOSOPHY** at Malaviya National Institute of Technology Jaipur. The thesis embodies the original work done by her and has not been earlier carried out anywhere to the best of my knowledge and belief.

(Dr.Ragini Gupta)
Associate Professor
Department of Chemistry
MNIT, Jaipur
Jaipur-302017
India

(Dr.Meenakshi Jain)
Associate Professor
Department of Chemistry
University of Rajasthan
Jaipur-302004
India

DECLARATION

I hereby certify that the work which is being presented in this thesis entitled **"Catalysis &Nanocatalysis for Green Organic Transformations"** in fulfilment of the requirement of Doctor of Philosophy and submitted to the Malaviya National Institute of Technology Jaipur is an authentic record of my own work carried out at Department of Chemistry under the supervision of Dr.Ragini Gupta, Associate Professor, Department of Chemistry, Malaviya National Institute of Chemistry Jaipur and Dr.Meenakshi Jain, Associate Professor, Department of Chemistry, University of Rajasthan, Jaipur. The results contained in this thesis have not been submitted in part or full, to any other University or Institute for the award of any degree. The content of the thesis has been checked using software 'Plagiarism Detector'.

Yogita Madan
ID 2009RCY103
Department of Chemistry
MNIT Jaipur
India

Date:

MALAVIYA NATIONAL INSTITUTE OF TECHNOLOGY, JAIPUR

CANDIDATE'S DECLARATION

I hereby certify that the work which is being presented in the thesis entitled "Catalysis and Nanocatalysis for Green Organic Transformations" in partial fulfilment of the requirements for the award of the degree of Doctor of Philosophy and submitted in the Department of Chemistry, Malaviya National Institute of Technology, Jaipur is an authentic record of my own work carried out at Department of Chemistry during the period from July, 2009 to July, 2014 under the supervision of Dr.Ragini Gupta, Associate Professor, Department of Chemistry, Malaviya National Institute of Technology, Jaipur and Dr.Meenakshi Jain, Associate Professor, Department of Chemistry, University of Rajasthan, Jaipur.

Yogita Madan

Date: ID 2009RCY103

This is to certify that the above statement made by the candidate is true to the best of my knowledge.

(Dr.Ragini Gupta)
Associate Professor
Department of Chemistry
MNIT, Jaipur
Jaipur-302017
India

(Dr.Meenakshi Jain)
Associate Professor
Department of Chemistry
University of Rajasthan
Jaipur-302004
India

(Supervisors)

Ph.D Viva-voce examination of Ms. Yogita Madan, Research Scholar, has been held on ……

Signature of Supervisors Signature of External Examiner

"One of the greatest titles in the world is parent, and one of the biggest blessings in the world is to have parents to call Ma and Pa".

Dedicated to My Parents……..

(Krishan Kumar Madan & Usha Madan)

"Without you, I'm nothing. With you, I'm something. Together, we're everything".

Dedicated to My Husband……..
(VineetNangia)

Acknowledgement

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At this moment of accomplishment, first of all I pay homage to my guide, ***DrRagini Gupta****. Associate Professor, Department of Chemistry, Malaviya National Institute of Technology Jaipur. This work would not have been possible without her guidance,scholarly inputs, support and encouragement. Under her guidance I successfully overcame many difficulties and learned a lot. She gave her valuable suggestions. Her unflinching courage and conviction will always inspire me. I will forever be thankful for supporting me during these past five years. I am also very grateful to her for scientific advice, knowledge and many insightful discussions. Thank you to Madam, for being a good guide who encouraged and expected us to think more independently about our experiments and results.*

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I am fortunate enough to have ***Mr. VineetNangia*** *as a life partner who is always by my side whenever I needed him for a moral support. He gave me strength to overcome the adverse circumstances that comes in the path of research work. The words seem to be small enough to express the cooperation rendered by him.*

I take this opportunity to sincerely acknowledge ***Council of Scientific and Industrial Research, New Delhi****, for providing financial assistance in the form of Senior Research Fellowship which buttressed me to perform my work comfortably.*

It would not have been possible to write my doctoral thesis without the help and support of the kind people around me, to only some of whom it is possible to give particular mention here.

At the end I thank the almighty for giving me enough courage to cope with adversities in an ideal manner.

Dated: 17.07.2014 ***(Yogita Madan)***

ABSTRACT

Introduction

Synthesis of organic molecules is a **'Science'** which fulfills critical living needs such as medicines, polymers, fibres, fuels, paints, lubricants and a myriad of other value added materials essential for present and future needs of mankind. However, this science for the synthesis of various chemical products is highly inefficient, generates a lot of chemical waste, hazardous to the environment and human beings. To overcome these drawbacks, new synthetic procedures incorporating Green Chemistry Principles and catalysis germinated. Catalysis lies at the heart of countless chemical protocols which effectively, creatively and economically transfers materials developed in academic research laboratories to the chemical industry. With the advent of nanocatalysis, this **'State of the Art Green Organic Transformations'** have been superimposedover a matrix of various exciting new manufacturing procedures that warrants the success of sustainability in synthesis.

The thesis entitled **'Catalysis and Nanocatalysis for Green Organic Transformations'** is divided into seven chapters covering the catalyzed/nanocatalyzed green organic transformations of various bioactive heterocycles, their characterization and bioactivity.

Chapter 1: Prefatory note

This chapter gives an introduction of catalysts and nanocatalysts and enumerates their importance in various green organic transformations for the synthesis of heterocyclic compounds. It also briefly describes the work undertaken during the present endeavour.

Chapter 2: A 'Mini-Review' on catalysis and nanocatalysis for green organic transformations

This chapter gives a bird's eye view of the utility of various catalysts and nanocatalysts employed in heterocyclic chemistry *via* green chemical techniques such as Microwave irradiation, Ultrasonication and Mechanochemistry (Grinding). Particular emphasis is given on metal oxide nanoparticles as catalyst.

Chapter 3: Synthesis of 2-phenyl-3-(1-(2-phenyl-1*H*-indol-3-yl) vinyl)-1*H*-indole derivatives using ZnOnanocatalyst

Chapter 4: Synthesis of 4',5'-dihydrofuro(4,5-a)-1,3-dione)spiro(indolin-3,2'-quinolin)-2-one derivatives and their evaluation for anti-microbial and analgesic activity

Chapter 5: Synthesis of 3-methyl-1-phenyl-4-(2-phenyl-1*H*-indol-3-yl)-4, 5-dihydro-1*H*-pyrazol[3,4-d]pyrimidin-6-(3a*H*)-one derivatives using ZnOnanocatalyst and their evaluation for anti-microbial and anti-inflammatory activity

Chapter 6: Synthesis of N-((3-methyl-5-oxo-1-phenyl-4,5-dihydro-1*H*-pyrazol-4-yl)(2-phenyl-1*H*-indol-3-yl)-methyl)-acetamide derivatives using CuO nanoparticles

Chapter 7: ZnO nanocatalyzed synthesis of the library of Knoevengel condensed products of indole-3-carbaldehyde and various active methylene groups

Chapter 8: Future Scope and Directions

CONTENTS

ANNEXURE-I

LIST OF ABBREVIATIONS

S. No.	Abbreviation	Expanded Form
1.	AI	Activity Index
2.	$CDCl_3$	Deuterated chloroform
3.	CDRI	Central drug research institute
4.	CMC	Carboxymethylcellulose
5.	CPCSEA	Committee designed for the purpose of control and supervision of experiments
6.	CuOnano	Copper oxide nanoparticles
7.	DMSO	Dimethyl sulfoxide
8.	EDDA	Ethylene diamine di acetate
9.	ESI	Electron spray ionisation
10.	FAB	Fast atomic bombardment
11.	HDA	Hetero diels alder reaction
12.	HRMS	High resolution mass spectroscopy
13.	IEC	Institutional ethic committee
14.	$InCl_3$	Indium trichloride
15.	IR	Infra-red
16.	IZ	Zone of inhibition
17.	MCR	Multicomponent reaction
18.	MIC	Minimum inhibitory concentration
19.	MNPs	Magnetic nanoparticles
20.	M.P	Melting point
21.	NMR	Nuclear magnetic resonance
22.	NPs	Nano particles
23.	PEG 400	Poly ethylene glycol 400
24.	δ-Ppm	Chemical shift in parts per million
25.	PTF	Protein farnesyl transferase
26.	Q-TOF	Quadrupole time of flight
27.	SEM	Scanning electron microscope
28.	TLC	Thin layer chromatography
29.	TMS	Tetra methyl silane
30.	XRD	X-ray diffraction
31.	ZnOnano	Zinc oxide nanoparticles
32.	ν cm^{-1}	Frequency in cm^{-1}

ANNEXURE-II

LIST OF FIGURES

14.	5.3	^{13}C NMR spectra of 3-methyl-1-phenyl-4-(2-phenyl-1*H*-indol-3-yl)-4, 5-dihydro-1*H*-pyrazol[3,4-d]pyrimidin-6-(3a*H*)-one
15.	5.4	ESI mass spectra of 3-methyl-1-phenyl-4-(2-phenyl-1H-indol-3-yl)-4, 5-dihydro-1*H*-pyrazol[3,4-d]pyrimidin-6-(3a*H*)-one
16.	5.5-5.9	Zone of inhibition against *A. niger*, *S. aureus*, *S. flexneri*, *P. vulgaris* and *T. rubrum* at 10^{-2} mg/ml, 10^{-3} mg/ml, 10^{-2} mg/ml, 10^{-2} mg/ml and 10^{-2} mg/ml, respectively
17.	5.10	Graphical Representation for MIC of antimicrobial activity
18.	6.1	IR spectra of N-((3-methyl-5-oxo-1-phenyl-4,5-dihydro-1*H*-pyrazol-4-yl)(2-phenyl-1*H*-indol-3-yl)-methyl)-acetamide
19.	6.2	^{1}H NMR spectra of N-((3-methyl-5-oxo-1-phenyl-4,5-dihydro-1*H*-pyrazol-4-yl)(2-phenyl-1*H*-indol-3-yl)-methyl)-acetamide
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21.	7.1	IR spectra of *(Z)*-3-methyl-1-phenyl-4-((2-phenyl-1*H*-indol-3-yl)-methylene)-1*H*-pyrazol-5(4*H*)-one
22.	7.2	IR spectra of 2-((2-phenyl-1*H*-indol-3-yl)-methylene)-cyclohexane-1,3-dione
23.	7.3	IR spectra of *(Z)*-3-methyl-4-((2-phenyl-1H-indol-3-yl)-methylen)-isoxazol-5(4*H*)-one
24.	7.4	^{1}H NMR spectra of *(Z)*-3-methyl-1-phenyl-4-((2-phenyl-1H-indol-3-yl)-methylene)-1*H*-pyrazol-5(4*H*)-one
25.	7.5	^{1}H NMR spectra of 2-((2-phenyl-1H-indol-3-yl)-methylene)-cyclohexane-1,3-dione
26.	7.6	^{1}H NMR spectra of *(Z)*-3-methyl-4-((2-phenyl-1*H*-indol-3-yl)-methylen)-isoxazol-5(4*H*)-one
27.	7.7	ESI spectra of *(Z)*-3-methyl-1-phenyl-4-((2-phenyl-1H-indol-3-yl)-methylene)-1*H*-pyrazol-5(4*H*)-one
28.	7.8	ESI spectra of 2-((2-phenyl-1H-indol-3-yl)-methylene)-cyclohexane-1,3-dione
29.	7.9	ESI spectra of *(Z)*-3-methyl-4-((2-phenyl-1H-indol-3-yl)-methylen)-isoxazol-5(4*H*)-one

ANNEXURE-III

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21.	6.1	Synthesis of N-((3-methyl-5-oxo-1-phenyl-4, 5-dihydro-1H-pyrazol-4-yl)(2-phenyl-1H-indol-3-yl)methyl)acetamides
22.	7.1	Synthesis of (Z)-3-methyl-1-phenyl-4-((2-phenyl-1H-indol-3-yl)methylene)-1H-pyrazol-5(4H)-one derivatives, 2-((2-phenyl-1H-indol-3-yl)methylene)cyclohexane-1,3-dione derivatives and (Z)-3-methyl-4-((2-phenyl-1H-indol-3 yl)methylene)isoxazol-5(4H)-one derivatives
23.	7.2	Keto-enol tautomerism in (Z)-3-Methyl-1-Phenyl-4-((2-Phenyl-1H-Indol-3-yl)methylene)-1H-pyrazol-5(4H)-one derivatives

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20.	2.30-2.31	Copper ferrite nanoparticles used for aldol condensation reaction
21.	2.31	$MgAl_2O_4$ nanocatalyzed synthesis of quinolone derivatives
22.	2.32	Nanocrystalline sulfated zirconia for green orgain transformation
23.	2.33	Nano organocatalyzed green organic transformations
24.	3.1	Synthesis of 2-phenyl-3-(1-(2-phenyl-1H-indol-3-yl) vinyl)-1H-indoles
25.	3.2	Plausible mechanism of the synthesis of 2-phenyl-3-(1-(2-phenyl-1H-indol-3-yl) vinyl)-1*H*-indoles
26.	4.1	Synthesis of 4',5'-dihydrofuro(4,5-a)-1,3-dione)-spiro(indolin-3,2'-quinolin)-2-one
27.	5.1	Synthesis of 3-methyl-1-phenyl-4-(2-phenyl-1H-indol-3-yl)-4,5-dihydro-1*H*-pyrazol[3,4-d]pyrimidin-6(3a*H*)-ones
28.	6.1	Synthesis of N-((3-methyl-5-oxo-1-phenyl-4, 5-dihydro-1*H*-pyrazol-4-yl)(2-phenyl-1*H*-indol-3-yl)-methyl)-acetamides
29.	7.1	Synthesis of *(Z)*-3-methyl-1-phenyl-4-((2-phenyl-1*H*-indol-3-

		yl)methylene)-1*H*-pyrazol-5(4*H*)-one derivatives, 2-((2-phenyl-1H-indol-3-yl)-methylene)-cyclohexane-1,3-dione derivatives and *(Z)*-3-methyl-4-((2-phenyl-1*H*-indol-3 yl)-methylen)-isoxazol-5(4*H*)-one derivatives
30.	7.2	Keto-enol tautomerism in *(Z)*-3-Methyl-1-Phenyl-4-((2-Phenyl-1*H*-Indol-3-yl)methylene)-1*H*-pyrazol-5(4*H*)-one derivatives

ANNEXURE-IV

LIST OF TABLES

APPENDIX-I

LIST OF NEW SYNTHESIZED COMPOUNDS

Chapter-3		
S. No.	**Compound No.**	**Name of the Synthesized Compounds**
1.	3a	2-Phenyl-3-(1-(2-phenyl-1*H*-indol-3-yl)-vinyl)-1*H*-indole
2.	3b	2-(4-Chlorophenyl)-3-(1-(2-(4-chlorophenyl)-1*H*-indol-3-yl)-vinyl)-1*H*-indole
3.	3c	2-(4-Bromophenyl)-3-(1-(2-(4-bromophenyl)-1*H*-indol-3-yl)-vinyl)-1*H*-indole
4.	3d	2-p-Tolyl-3-(1-(2-p-tolyl-1*H*-indol-3-yl)-vinyl)-1*H*-indole
5.	3e	2-(4-Fluorophenyl)-3-(1-(2-(4-fluorophenyl)-1*H*-indol-3-yl)-vinyl)-1*H*-indole
Chapter-4		
6.	4a	4’,5’-dihydrofuro(4,5-a)-1,3-dione)spiro(indolin-3,2’-quinolin)-2-one
7.	4b	4’,5’-dihydrofuro(4,5-a)-1,3-dione)spiro(5-fluoroindolin-3,2’-quinolin)-2-one
8.	4c	4’,5’-dihydrofuro(4,5-a)-1,3-dione)spiro(5-chloroindolin-3,2’-quinolin)-2-one
9.	4d	4’,5’-dihydrofuro(4,5-a)-1,3-dione)spiro(5-bromoindolin-3,2’-quinolin)-2-one
10.	4e	4’,5’-dihydrofuro(4,5-a)-1,3-dione)spiro(5-aminoindolin-3,2’-quinolin)-2-one
11.	4f	4’,5’-dihydrofuro(4,5-a)-1,3-dione)spiro(5-methylindolin-3,2’-quinolin)-2-one
12.	4g	4’,5’-dihydrofuro(4,5-a)-1,3-dione)spiro(indolin-3,6’-fluoro-2’-quinolin)-2-one
13.	4h	4’,5’-dihydrofuro(4,5-a)-1,3-dione)spiro(5-fluor-indolin-3,6’-fluoro-2’-quinolin)-2-one

Chapter-5		
14.	2a	3-Methyl-1-phenyl-4-(2-phenyl-1*H*-indol-3-yl)-4,5-dihydro-1*H*-pyrazol[3,4-d]pyrimidin-6(3a*H*)-one
15.	2b	4-(2-(4-Fluorophenyl)-1*H*-indol-3-yl)-3-methyl-1-phenyl-4,5-dihydro-1*H*-pyrazol[3,4-d]pyrimidin-6(3a*H*)-one
16.	2c	4-(2-(4-Chlorophenyl)-1*H*-indol-3-yl)-3-methyl-1-phenyl-4,5-dihydro-1*H*-pyrazol[3,4-d]pyrimidin-6(3a*H*)-one
17.	2d	4-(2-(4-Bromophenyl)-1*H*-indol-3-yl)-3-methyl-1-phenyl-4,5-dihydro-1*H*-pyrazol[3,4-d]pyrimidin-6(3a*H*)-one
18.	2e	3-Methyl-1-phenyl-4-(2-p-tolyl-1*H*-indol-3-yl)-4,5-dihydro-1*H*-pyrazol[3,4-d]pyrimidin-6(3a*H*)-one
19.	2f	4-(2-(4-Dichlorophenyl)-1*H*-indol-3-yl)-3-methyl-1-phenyl-4,5-dihydro-1*H*-pyrazol[3,4-d]pyrimidin-6(3a*H*)-one
Chapter-6		
20.	3a	N-((3-Methyl-5-oxo-1-phenyl-4,5-dihydro-1*H*-pyrazol-4-yl)(2-phenyl-1*H*-indol-3-yl)-methyl)-acetamide
21.	3b	N-((2-(4-Fluorophenyl)-1*H*-indol-3-yl)(3-methyl-5-oxo-1-phenyl-4,5-dihydro-1*H*-pyrazol-4-yl)-methyl)-acetamide
22.	3c	N-((2-(4-Chlorophenyl)-1*H*-indol-3-yl)(3-methyl-5-oxo-1-phenyl-4,5-dihydro-1*H*-pyrazol-4-yl)-methyl)-acetamide
23.	3d	N-((2-(4-Bromophenyl)-1*H*-indol-3-yl)(3-methyl-5-oxo-1-phenyl-4,5-dihydro-1*H*-pyrazol-4-yl)-methyl)-acetamide
24.	3e	N-((3-Methyl-5-oxo-1-phenyl-4,5-dihydro-1*H*-pyrazol-4-yl)(2-p-tolyl-1*H*-indol-3-yl)-methyl)-acetamide
25.	3f	N-((2-(4-Aminophenyl)-1*H*-indol-3-yl)(3-methyl-5-oxo-1-phenyl-4,5-dihydro-1*H*-pyrazol-4-yl)-methyl)-acetamide

Chapter-7		
26.	2a	*(Z)*-3-Methyl-1-phenyl-4-((2-phenyl-1*H*-indol-3-yl)-methylene)-1*H*-pyrazol-5(4*H*)-one
27	2b	*(Z)*-4-((2-(4-Fluorophenyl)-1*H*-indol-3-yl)-methylene)-3-methyl-1-phenyl-1*H*-pyrazol-5(4*H*)-one
28	2c	*(Z)*-4-((2-(4-Chlorophenyl)-1*H*-indol-3-yl)-methylene)-3-methyl-1-phenyl-1*H*-pyrazol-5(4*H*)-one
29	2d	*(Z)*-4-((2-(4-Bromophenyl)-1*H*-indol-3-yl)-methylene)-3-methyl-1-phenyl-1*H*-pyrazol-5(4*H*)-one
30	2e	*(Z)*-4-((2-(4-Aminophenyl)-1*H*-indol-3-yl)-methylene)-3-methyl-1-phenyl-1*H*-pyrazol-5(4*H*)-one
31	3a	2-((2-Phenyl-1*H*-indol-3-yl)-methylene)-cyclohexane-1,3-dione
32	3b	2-((2-(4-Fluorophenyl)-1*H*-indol-3-yl)-methylene)-cyclohexane-1,3-dione
33.	3c	2-((2-(4-Chlorophenyl)-1*H*-indol-3-yl)-methylene)-cyclohexane-1,3-dione
34.	3d	2-((2-(4-Bromophenyl)-1*H*-indol-3-yl)-methylene)-cyclohexane-1,3-dione
35.	3e	2-((2-(4-Aminophenyl)-1*H*-indol-3-yl)-methylene)-cyclohexane-1,3-dione
36.	4a	*(Z)*-3-Methyl-4-((2-phenyl-1*H*-indol-3-yl)-methylen)-isoxazol-5(4*H*)-one
37.	4b	*(Z)*-4-((2-(4-Fluorophenyl)-1*H*-indol-3-yl)-methylen)-3-methylisoxazol-5(4*H*)-one
38.	4c	*(Z)*-4-((2-(4-Chlorophenyl)-1*H*-indol-3-yl)-methylen)-3-methylisoxazol-5(4*H*)-one
39.	4d	*(Z)*-4-((2-(4-Bromophenyl)-1*H*-indol-3-yl)-methylen)-3-methylisoxazol-5(4*H*)-one

APPENDIX-II

LIST OF PUBLICATIONS/ABSTRACT

PAPERS

S. No.	Title	Author	Journal Name	Page No.
1.	Indium trichloride catalyzed Diels-Alder Reaction: Synthesis of Novel 5-Butyl-11a-aryl-4a,5,11,11a-tetrahydro-11bH-indolo[3,2-c]quinolone-1,4-diones	Ragini Gupta, Anshu Jain, **Yogita Madan**	Journal of Heterocyclic Chemistry	2013, 50, 6, 1342-1345
2.	A 'one pot', environmentally friendly, multicomponent synthesis of 2-amino-5-cyano-4-[(2-aryl)-1*H*-indol-3-yl]-6-hydroxypyrimidines and their antimicrobial activity	Ragini Gupta, **Yogita Madan,**Anshu Jain	Journal of Heterocyclic Chemistry	2014, 51, 5, 1395-1403
3.	Microwave assisted ZnO nanocatalyzed Biginelli synthesis of 3-methyl-1-phenyl-4-(2-phenyl-1H-indol-3-yl)-4, 5-dihydro-1H-pyrazolo[3,4-d]pyrimidin-6(3aH)-one derivatives derivatives and evaluation of their bioactivity	Ragini Gupta, **Yogita Madan**, EktaMenghani	Journal of Applicable Chemistry	2014, 3, 5, 1955-1966
4.	An efficient approach for the synthesis of 2-phenyl-3-(1-(2-phenyl-1H-indol-3-yl) vinyl)-1H-indole derivatives using ZnOnanocatalyst	Ragini Gupta, **Yogita Madan**	International Journal of Nanotechnology	Communicated

5.	Ultrasound Ppromotedimino Diels-Alder reaction of ketamine-isatin for the generation of spiro[indoline-3,2-quinoline]-2-ones using PEG as a green solvent and evaluation of their anti-microbial and analgesic activity	Ragini Gupta, **Yogita Madan**, EktaMenghani	International Journal of Research in Chemistry and Environment	2015, 5, 1. 106-117
6.	CuO nanoparticles catalyzed selective synthesis of N-((3-methyl-5-oxo-1-phenyl-4,5-dihydro-1H-pyrazol-4-yl)-2-phenyl-1H-indol-3-yl)methyl-acetamide derivatives	Ragini Gupta, Yogita Madan	Catalysis Communication	Communicated
7.	An exploration to synthesize (Z)-3-methyl-1-phenyl-4-((2-phenyl-1H-indol-3-yl)methylene)-1H-pyrazol-5(4H)-one derivatives, 2-((2-phenyl-1H-indol-3-yl)methylene)cyclohexane-1,3-dione derivatives and (Z)-3-methyl-4-((2-phenyl-1H-indol-3 yl)methylene)isoxazol-5(4H)-one derivatives	Ragini Gupta, Yogita Madan	Chemical Science Reviews and Letters	Communicated

ABSTRACT

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