JANTA SHIKSHAN SANSTH'S

KISAN VEER MAHAVIDYALAYA ,WAI

Department of Chemistry

Number of students undertaking project work 2023-24

Core Course Practical in Chemistry

M.Sc. II Sem IV

Sr.No.	Roll No.	Name of Students.	Name of project.
1	06	BHOSALE TRUPTI KRISHNAT	Hydrothermally synthesized Manganese carbonate for supercapacitors
2	27	PISAL SNEHAL RAJENDRA	Glycerol as a green solvent for efficient one pot & catalyst free synthesis of 2,4,5 triaryl & 1,2,4,5 tetraimidazole derivative
3	23	SHINDE KAJAL RAJENDRA	Synthesis of Benzodiazepine derivatives
4	07	BHOSALE PRATIKSHA PRADIP	Synthesis & characterization of $NiCoPO_4$ for super-capacitor
5	25	TARATE VAISHNAVI ANIL	Synthesis of Benzodiazepine derivatives.
6	16	SHINDE MAYUR SANJAY	Synthesis & characterization of azo dyes based on nitroanilines
7		PISAL AKASH RAMDAS	AB.
8	28	KHANDE SIDDHI SANJAY	Glycerol as green solvent for efficient one pot & catalyst free synthesis of 2,4,5-triaryl & 1,2,4,5 tetraimidazole derivatives
9	37	NANAWARE ABHISHEK RAMCHANDRA	Synthesis of Paracetamol tablet
10	35	JADHAV ANMOL ANIL	Synthesis of Barbituric acid & their derivatives
11	21	SALUNKHE ANIKET SANJAY	Emblica officinalis catalysed Knoevenagel Condensation Reaction.
12	18	PISAL KETAN KISHOR	Synthesis of α,β-unsaturated carbonyl comp'd by using Knoevenagel condensation.
13	09	DHAGE SHAMBHURAJ NANDKUMAR	Synthesis of Green copper Nanoparticles using Medicinal plant Hygenia abyssinica Leaf extract.

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14	11	GHARGE ANKITA RAJENDRA	Synthesis & characterization of N & O substituted anhydride derivatives
15	19	SANAS ABHIJIT MOHAN	Synthesis of α,β-unsat. by using Knoevengel condensation
16	33	SHELAR SWAPNALI SUBHASH	Synthesis of Benzodiazepine derivatives.
17	01	SANAP SOMINATH PARMESHWAR	Synthesis & characterization of Azo dyes based on Nitroanilines.
18	03	CHAVAN GANESH JAYWANT	Hydrothermally synthesized Manganese carbonate for supercapacitor
19	22	PAWAR GANESH BALASO	Synthesis of Green copper Nanoparticles using Medicinal plant Hagenia abyssinica-Leaf extract
20	12	SALUNKHE ASHUTOSH SATISH	Synthesis & characterization of $NiCoPO_4$ for supercapacitor.
21	34	PISAL PRATIBHA ZAKAS	Synthesis of Barbituric acid & their derivatives.
22	32	MULLA RASHIDA SIRAJAHAMAD	Synthesis of Barbituric acid & their derivatives
23	24	GHADAGE YASHASHRI PRAMOD	Synthesis & characterization of N & O substituted anhydride derivatives
24	14	MULIK GANESH KIRAN	Synthesis & characterization of some heterocyclic compounds by using schiff bases.
25	13	RAJE SHUBHAM RAJENDRA	Synthesis & characterization of some heterocyclic compounds by using Schiff bases
26	36.	PISAL PRATIK SURYAKANT	Emblica officinalis catalysed Knoevenagel Condensation Reaction.
27	20	WAGH AGAMYA SHANKAR	Synthesis & Characterization of Azo dye formacacia catechu.
28	29	ITHAPE RUTIK SUDHAKAR	Synthesis & characterization of Bare Ru & RuO_2 nanoparticles.
29	17	KACHARE RAHUL SUBHASH	synthesis & characterization of azo dyes Formacacia Catechu
30	31	SURYAWANSHI PRIYANKA CHINCHOLPPA	Water extract of Piper Nigrum seed.
31	08	VAIRAT PRIYANKA ANANDA	Synthesis & characterization of $NiCoPO_4$ for super capacitor

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32	26	JAMDADE SNEHAL SANJAY	Water extract of Piper Nigrum seed
33	10	NANAWARE HIMANSHU AVINASH	Synthesis of Paracetamol tablet
34	05	BAGAL AISHWARYA SOMESHWAR	Synthesis & characterza of $NiCoPO_4$ for supercapacitor
35	04	KAMBLE MAYURI MEGHNATH	Synthesis & characterization of $NiCoPO_4$ for supercapacitor
36	02	DABHADE SHARVARI MILIND	Synthesis & characterization of $NiCoPO_4$ for supercapacitor
37	30	SANAS SANKET ANIL	Synthesis & characterization of Bare Ru & RuO_2 nano-particles.

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CERTIFICATE

This is to certify that **Mr. Abhishek Nanaware & Mr. Himanshu Nanaware** has successfully completed the project work on **"SYNTHESIS OF PARACETAMOL 500 MG TABLET "** for year 2023-2024. This project is submitted the degree of M.Sc. in Organic chemistry of Shivaji University , Kolhapur .

Examiner **Project Guide** **Head**

(Department of Chemistry)

Head
Department of Chemistry
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WAI
Dist. Satara

JANATA SHIKSHAN SANSTHA'S
KISAN VEER MAHAVIDYALAYA, WAI

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DEPARTMENT OF CHEMISTRY

This is to certify that. Miss. Mulla Rashida S., Miss. Pisal Pratibha Z., Mr. Jadhav Anmol A. has successfully completed the project work on "SYNTHESIS OF BARBITURIC ACID & THEIR DERIVATIVES" which is being submitted here with as partial fulfillment for the award of Degree of Master of Science Department of Chemistry, Shivaji University Kolhapur.

This project is the result of data information collected from the respective information media and we have successfully verified the result obtained.

All the resluting aspects are found to be correct and appropriate in the view of this project and the best of our knowledge.

Date :- / /2024

Place : - Wai

Mrs. D. S. Patil
Head of Chemistry Department
Kisan Veer Mahavidyalaya, Wai

Prof. Dr. D. N. Zambare
Project Guide
Kisan Veer Mahavidyalaya,
Department of Chemistry

External Examiner
Shivaji University Kolhapur

JANATA SHIKSHAN SANSTHA'S

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This is to Certify that, the Project report entitled "Glycerol as a green solvent for efficient, one-pot and catalyst free synthesis of 2,4,5-triaryl and 1,2,4,5-tetraaryl imidazole derivatives." Submitted by Miss. Snehal R. Pisal, Miss. Siddhi S. Khande in fulfillment of project work, prescribed by Shivaji University, Kolhapur for M.Sc II Course in Organic Chemistry have been completed satisfactorily under my guidance during the academic year 2023-24 the conclusions drawn are based on the experimental work carried out by them.

To the best of my knowledge & belif, the matter presented here is original & has not been submitted earlier.

Date :-

Place :- Wai

Miss. D.S.Patil
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This is to Certify that, following candidates ***Mr. Shubham Rajendra Raje and Mr. Ganesh Kiran Mulik*** of M.Sc.-II (Org. Chem.) has successfully completed the project work entitled ***"Synthesis and characterization of some heterocyclic compounds by using Schiff bases"*** in practical fulfillment of the award of Master of Chemistry as laid down by the **Shivaji University, Kolhapur** during the academic year 2023-2024.

Date :- 26/03/2024

Place :- Wai.

Guide

(Mrs. D. S.Patil)

Examiner

Head

Department of chemistry

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This is to certify that, **Miss. Shinde Kajal Rajendra, Miss. Tarate Vaishnavi Anil,Miss. Shelar Swapnali Subhash,** has successfully completed the project work on **"Synthesis of Benzodiazepine Derivatives"** which is being submitted here with as partial fulfillment for the award of **Degree of Master of Science Department of Chemistry,** ShivajiUniversity Kolhapur.

This project is the result of data information collected from the respective information media and we have successfully verified the result obtained.

All the resulting aspects are found to be correct and appropriate in the view of This project and the best of our knowledge.

Date:-26/03/ 2024

Place:- Wai

Prof. Dr. D. N. Zambare
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This is to Certify that, following candidate **Mr. Ganesh Balaso Pawar** and of M.Sc.-II has successfully completed the project work entitled "Synthesis of Green Copper Nanoparticles Using Medicinal Plant"

in practical fulfillment of the award of Bachelor of Chemistry as laid down by the Shivaji University, Kolhapur during the academic year 2023-2024.

Date :

Place:- Wai.

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(Mrs. D.S.Patil)

Head

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Examiner

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This is to Certify that, following candidates **Miss. Ghadage Yashashri Pramod Miss. Gharge Ankita Rajendra** of M.Sc.-II (Org. Chem.) has successfully completed the project work entitled ***"SYNTHESIS AND CHARACTERIZATION OF N AND O SUBSTITUTED ANHYDRIDE DERIVATIVES"*** in practical fulfillment of the award of Master of Chemistry as laid down by the **Shivaji University, Kolhapur** during the academic year 2023-2024.

Date :- 26/03/24

Place :- Wai.

Guide	Examiner	Head
(Mrs. D.S.Patil)		Department of chemistry

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Certificate

Janata Shikshan Sanstha's

Kisan Veer Mahavidyalaya Wai

This is to certify that the work incorporated in the project entitled

"Synthesis And charecterisation of Bare Ru And RuO_2 Nanoparticle "

Submitted by **Rutik Sudhakar Ithape. And Sanket Anil Sanas.** MSc. -II, Organic chemistry, was carried out by the candidate under our supervision during academic year 2023-2024.

Prof. (Dr). D..N..Zambare.
Head
Department Of Chemistry

External Examiner

Mrs. Patil D. S.
Project Guide

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This is to Certify that, following candidate **Mr. Ketan Kishor Pisal** and **Mr. Abhijit Mohan Sanas** of M.sc II has successfully completed the project work in "Synthesis of α, β – unsaturated carbonyl compund by using knoevenagal condensation "practical fulfillment of the award of Bachelor of Chemistry as laid down by the Shivaji University, Kolhapur during the academic year 2023-2024.

Date :27/02/2024

Place:- Wai.

Guide

(Mrs. D.S.Patil)

Head

Department of Chemistry

Examiner

Shivaji University Kolhapur

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Department Of Chemistry
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JANATA SHIKSHAN SANSTHA'S

Kisan Veer Mahavidyalaya, Wai

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This is to Certify that, the Project report entitled Water Extract of *Piper Nigrum* Seed Submitted by Miss, Snehal S, Jamdade And Miss. Priyanka C. Suryawanshi in fulfillment of project work, prescribed by Shivaji University, Kolhapur for M.Sc II Course inOrganic Chemistry have been completed satisfactorily under my guidance during the academic year 2023-24 the conclusions drawn are based on the experimental work carried out by them.

To the best of my knowledge & belif, the matter presented here is original & has not been submitted earlier.

Date :- 26/03/2024

Place :- Wai

Miss. D.S.Patil
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Dr. D.N. Zambare
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Examiner
Kisanveer Mahavidyalaya,
Wai

J. S. SANSTHA'S KISAN VEER MAHAVIDYALAYA Wai Dist. Satara

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KISAN VEER MAHAVIDYALAYA, WAI

DEPARTMENT OF CHEMISTRY

Certificate

This is to certify that the project report entitled "SYNTHESIS AND CHARACTRISATION OF NiCoWO4 FOR SUPERCAPCITOR" submitted by Pratiksha Pradip Bhosale, Sharvari Milind Dabhade , Ashutosh Satish Salunkhe. in fulfilment of the project work, prescribed by SHIVAJI UNIVERSITY, KOLHAPUR for M.Sc. course in organic chemistry have been completed satisfactorily under my guidance year 2023-2024.

Place: Wai

Date:

Dr. S. B. Wategaonkar
(Project Guide)

Prof. (Dr). D. N. Zambare
HOD

External Examiner
Shivaji University, Kolhapur

JANATA SHIKSHAN SANSTHA'S
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DEPARTMENT OF CHEMISTRY

Certificate

This is to certify that the project report entitled "SYNTHESIS AND CHRACTERISATION OF $NiCoPO_4$ FOR SUPERCAPACITOR" submitted by **Miss.Kamble Mayuri Meghnath, Miss.Vairat Priyanka Ananda, Miss.Bagal Aishwary Someshwar** in fulfilment of the project work, prescribed by SHIVAJI UNIVERSITY, KOLHAPUR for M.Sc. course in organic chemistry have been completed satisfactorily under my guidance year 2023-2024.

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Date:

Dr. S.B. Wategaonkar
(Project Guide)

Prof. (Dr). D.N.Zambare
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Shivaji University, Kolhapur

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DEPARTMENT OF CHEMISTRY

Certificate

This is to certify that the project report entitled **"SYNTHESIS AND CHARACTERISATION OF AZO DYES BASED ON NITROANILINES"** submitted by **Mr. SHINDE MAYUR SANJAY, SANAP SOMINATH PARMESHWAR** in fulfilment ofthe project work, prescribed by SHIVAJI UNIVERSITY, KOLAHAPUR for M.Sc. course in Organic Chemistry have been completed satisfactorily under my guidance during the academic year 2023-2024.

Place : Wai

Date :

Miss. P.S. Jaigude
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KISAN VEER MAHAVIDYALAYA,WAI

DEPARTMENT OF CHEMISTRY

Certificate

This is to certify that the project report entitled **"SYNTHESIS AND CHARACTERISATION OF AZO DYE FROM ACACIA CATECHU"** submitted by ***Mr. WAGH AGAMYA SHANKAR, Mr. KACHARE RAHUL SUBHASH*** in fulfilment ofthe project work, prescribed bySHIVAJI UNIVERSITY, KOLAHAPUR forM.Sc. course in Organic Chemistry have been completed satisfactorily undermyguidance duringtheacademicyear 2023-2024.

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Date:

23/3/24

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(ProjectGuide)

Prof(Dr.)D.N.Zambare

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CERTIFICATE

This is to certify the dissertation entitled "**Hydrothermally Synthesized Manganese Carbonate for Supercapacitors**" is the work of **Miss. Bhosale Trupti Krishnat & Mr.Ganesh Jaywant Chavan** for the partial fulfillment of the requirement for the degree of M.Sc. in Chemistry of Shivaji University Kolhapur. They have completed their work under my guidance. Their work is original, and it has not been submitted for this or any other degree or diploma of this or any other institution.

They have worked in Department of Chemistry, **Kisan Veer Mahavidyalaya, Wai** for sufficient time for the dissertation.

Project Guide

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Janata Shikshan Sanstha's
Kisan Veer Mahavidyalaya, Wai

A

PROJECT REPORT

ON

"Synthesis of Benzodiazepine Derivatives"

SUBMITTED TO

SHIVAJI UNIVERSITY OF KOLHAPUR

FOR THE DEGREE OF

MASTER OF SCIENCE

IN

ORGANIC CHEMISTRY

By

Miss. Shinde Kajal Rajendra
Miss. Tarate Vaishnavi Anil
Miss. Shelar Swapnali Subhash

UNDER THE GUIDANCE OF

Mrs. Patil D. S. (M.Sc. NET, SET)
Assistant Professor,
Department of Chemistry, Kisan Veer Mahavidyalaya, Wai

JANATA SHIKSHAN SANTHA'S
KISAN VEER MAHAVIDYALAYA, WAI

CERTIFICATE
DEPARTMENT OF CHEMISTRY

This is to certify that, **Miss. Shinde Kajal Rajendra, Miss. Tarate Vaishnavi Anil,Miss. Shelar Swapnali Subhash,** has successfully completed the project work on **"Synthesis of Benzodiazepine Derivatives"** which is being submitted here with as partial fulfillment for the award of **Degree of Master of Science Department of Chemistry,** ShivajiUniversity Kolhapur.

This project is the result of data information collected from the respective information media and we have successfully verified the result obtained.

All the resulting aspects are found to be correct and appropriate in the view of This project and the best of our knowledge.

Date:-26/03/ 2024

Place:- Wai

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Shivaji University, Kolhapur

J. S. SANSTHA'S KISAN VEER MAHAVIDYALAYA Wai Dist. Satara

Acknowledgment

I would like to acknowledge my approbation and humility to my esteemed guide Mrs.D. S. Patil Assistant Professor, Department of Chemistry, Kisan veer Mahavidyalaya, Wai for his constant counseling and proper guidance throughout my project work.

I would like to extend my gratitude to Prof. Dr.D.N. Zambre, Head, Department of Chemistry, Kisan veer Mahavidyalaya, Wai for their constant encouragement and support.

I am grateful to Hon. Prin. Dr. G.J. Fagare for providing me essential and possible facilities. Without his support this work can't be complete to fulfill the expectations.

I may be failing in my duties if I do not thank to Mrs.D.S.Patil and other teaching and non-teaching staff of department of chemistry for their constant encouragement and support. Due to their support only I can fulfill the experimental work.

I would more than ever like to thank to all classmates for being a great mates ever in my life. Without my all friend I can't complete any work, so due their positive support I can complete my research project work.

Last but not the list I bow my head before my mother and father for their supreme sacrifice and internal benediction in evolving my personality. Their ocean like bowl of care, shower of love and affection as well as inspiration have made these great success.

Miss. Shinde Kajal Rajendra
Miss. Tarate Vaishnavi Anil
Miss. Shelar Swapnali Subhash

DECLARATION

I hereby declare that the Project Report entitled, **"Synthesis of Benzodiazepine Derivatives"** completed and written by me has not previously formed the basis for the award of any Degree or Diploma or other similar title of this or any other University or examining body.

Place:Wai

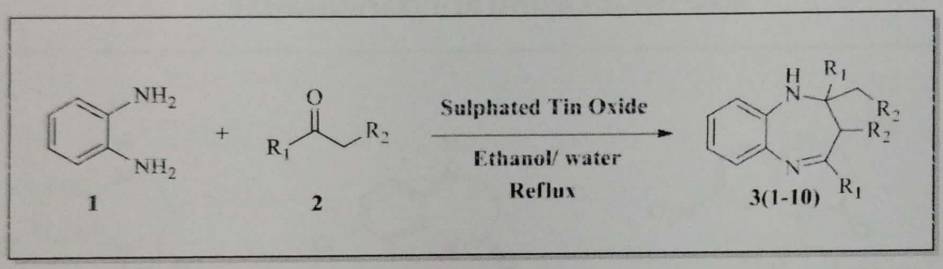
Date:26/3/2024

Sr.No	Roll No	Name	Signature
1	23	Shinde Kajal Rajendra	K.R.Shinde
2	25	Tarate Vaishnavi Anil	Tarate
3	33	Shelar Swapnali Subhash	Shelar

INDEX

1. ABSTRACT

A competent protocol for synthesis of benzodiazepine derivatives has been developed by condensation of o-phenylenediamine and various ketones using sulfated tin oxide as heterogeneous solid super acid catalyst in ethanol water (1:1-v/v) at reflux condition. The synthesized catalyst was validated by Infrared spectra, X-ray powder diffraction, Scanning electron microscopic images and EDS maps. The optimization of reaction was carried for different solvents and loading of catalyst. The Synthesized compounds were confirmed by spectral analysis. The method is advantageous in accordance with environmentally benign procedure, short reaction time, easy work up, reusable catalyst and high yields.

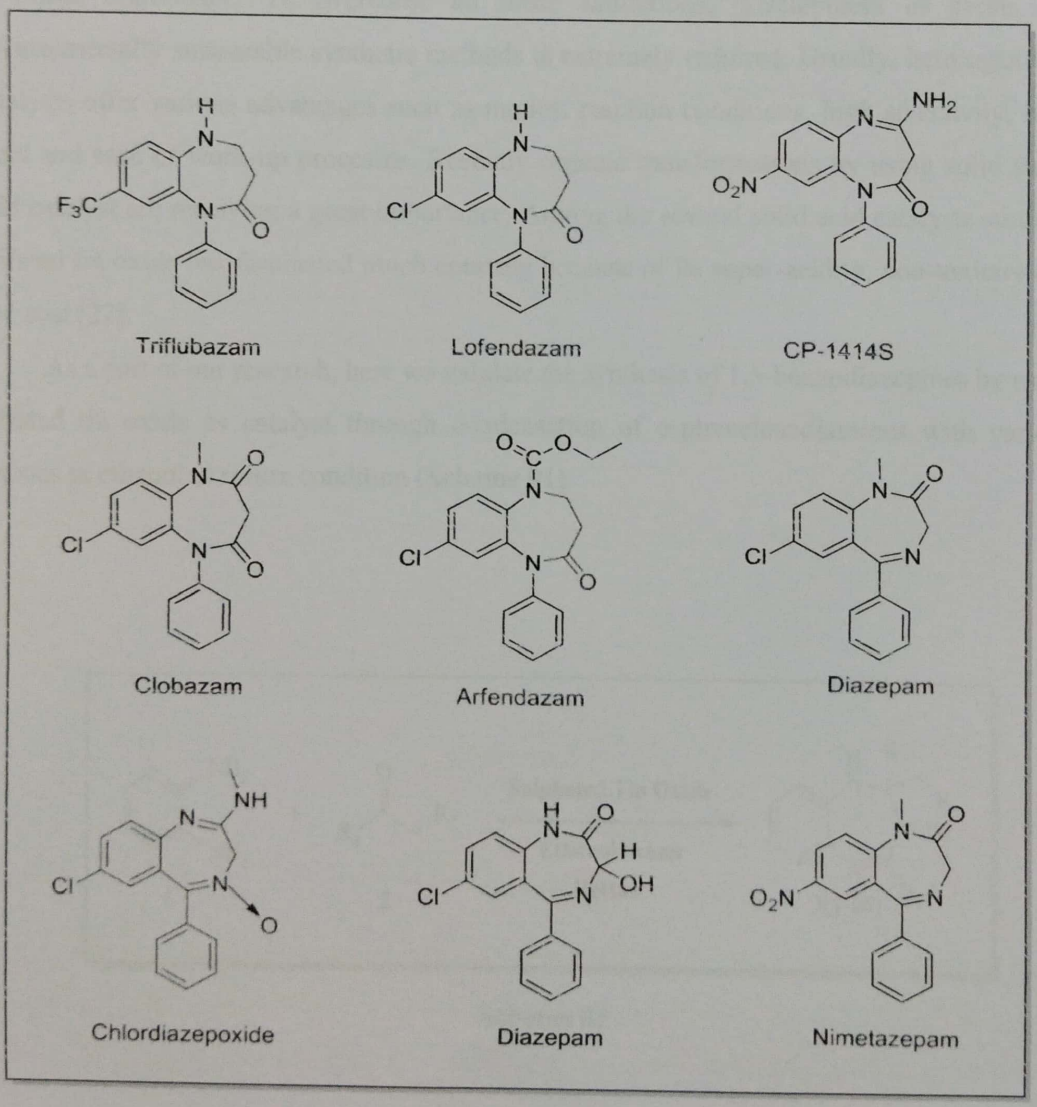


Key words : sulfated tin oxide, o-phenylenediamine, ketones, reflux

2. INTRODUCTION

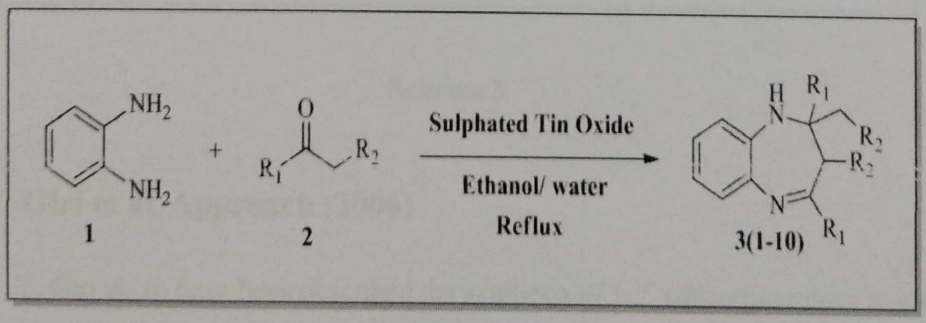
Benzodiazepines are crucial nitrogen containing heterocyclic compounds that possess a variedarray of therapeutic and pharmacological properties. It's substantial central nervous system (CNS) depressant characteristic make benzodiazepines highly used psychotropic [1].They are broadly used as antianxiety, sedative, anticonvulsant, analgesic, hypnotic agents, anti-depressive andanti-inflammatory agents [2].The 1, 5-dibenzodiazepines have been narrated to reveal inhibitory activities towards HIV-1 protease[3-4].Fused ring systems such as triazole, oxazino, furanobenzodiazepines can be prepared from 1,5-benzodiazepines synthons [5]. The derivatives of 1,5 benzodiazepines are also used as dyes for acrylic fibers in Photography[6].

BENZODIAZEPINE DRUGS SCAFFOLD



Owing to their extensive applications, numerous approaches for the synthesis of benzodiazepines have been reported by reaction between o-phenylenediamines (OPDAs) and enones, ketones, or β-halo ketones using several homogenous catalysts such as BF_3:etherate[7], $NaBH_4$[8], polyphosphoric acid [9], solvent free under microwave irradiation[10], $ZnCl_2$[11], $Yb(OTf)_3$[12], ionic liquids [13].Along with that various solid acid as well as solid supported catalysts have been used for the synthesis of 1,5-benzodiazepines that includes sulfate zirconia[14], amberlyst-15[15], stannic oxide NPs[16], polymer-supported $FeCl_3$[17], Al_2O_3/P_2O_5[18], Zeolite[19], H-MCM-22[20], and $Hg(OTf)_3$[21]. However, all of these procedures have difficulties, which include expensive reagents, drastic reaction conditions, comparatively long reaction time, low yields, generation of side products and difficulty in retrieval and reuse of the catalysts. Consequently, developing a novel approach for the synthesis of 1,5-benzodiazepines in terms of being eco-friendly, simple and economically feasible is still of prime importance. To overcome all those limitations, development of green and environmentally sustainable synthetic methods is extremely required. Usually, heterogeneous catalysts offer various advantages such as modest reaction conditions, high selectivity, high yield and ease of work-up processes. Recently organic transformations by using solid super acid catalyst are receiving a great importance. Among the several solid acid catalysts studied, sulfated tin oxide has fascinated much courtesy because of its super-acidity, non-toxicity and low cost [22].

As a part of our research, here we validate the synthesis of 1,5-benzodiazepines by using sulfated tin oxide as catalyst through condensation of o-phynelenediamines with various ketones in ethanol at reflux condition **(Scheme 01)**.



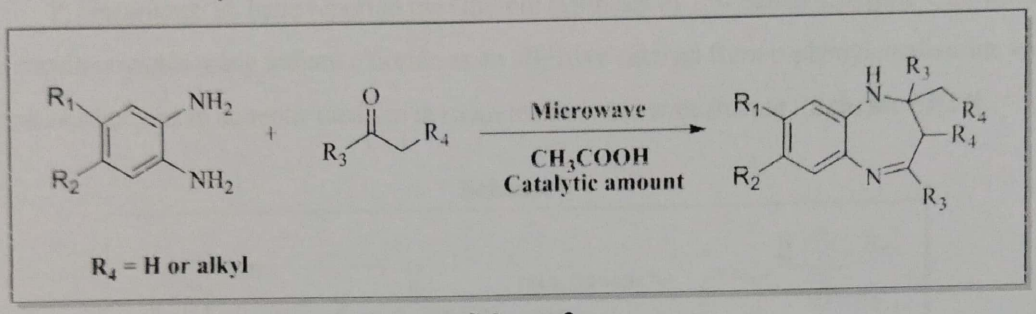
Scheme 01

3. REVIEW OF LITERATURE:

The crucial research practices dealing with synthesis of 1, 5-benzodiazepines using diverse routes are mentioned herewith.

3.1 M. Pozarentzi et al. Approach (2002)

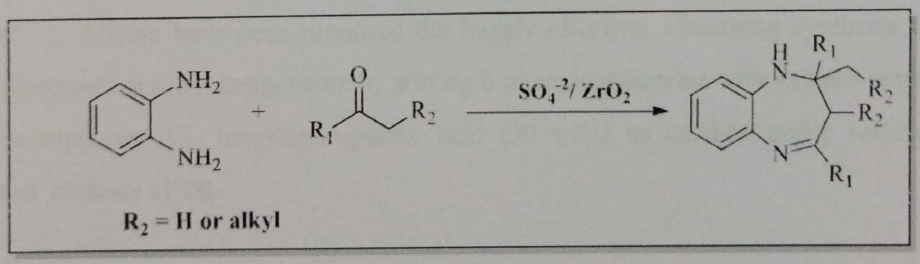
M. Pozarentzi et.al. have synthesized 2,3-Dihydro-1*H*-1,5-benzodiazepine from the condensation reaction between *o*-Phenylenediamine and various ketones, in the presence of acetic acid as a catalyst under solvent free condition. **(Scheme 2).[24]**



Scheme 2

3.2 Benjaram M. Reddy et al. Approach(2003)

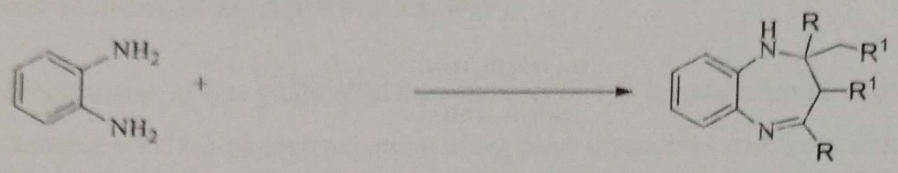
Benjaram M. Reddy et. al. reported 2,3-Dihydro-1*H*-1,5-benzodiazepine synthesis via condensation of between *o*-Phenylenediamine and various ketones under solvent free condition using versatile superacid catalyst sulfated zirconia **(Scheme 3)[25].**



Scheme 3

3.3 B. Y. Giri et al. Approach (2006)

B. Y. Giri et. al. have been described the synthesis of 1, 5-benzodiazepines in great yields using 5%wt Monoammonium salt of 12-tungstophosphoric acid [$(NH_4)H_2PW_{12}O_{40}$] as catalyst via condensation of o-phenylenediamine with several aldehydes in dichloroethane at reflux condition with stirring. **(Scheme 4)[26].**

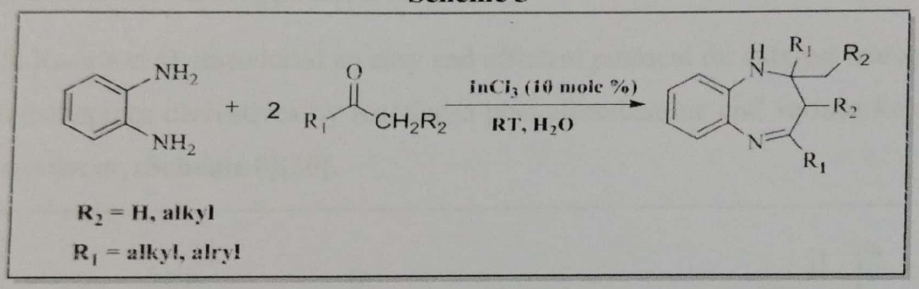


Scheme 4

3.4 P. Hazarika et al. Approach(2007)

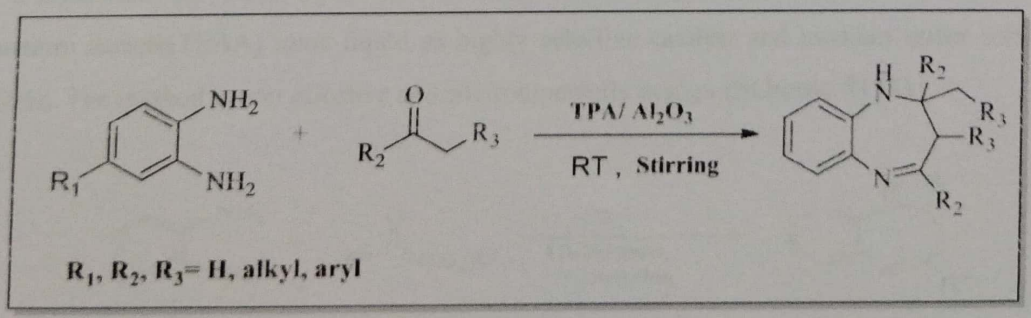
P. Hazarikaet. al. have reported the One-pot synthesis of 1,5- benzodiazepine derivatives in excellent yields using indium chloride as an effective catalyst from o-phenylenediamine and a various ketones in aqueous medium at room temperature with stirring. **(Scheme 5)[27]**

Scheme 5



3.5 M. A. Alibeik et al. Approach (2008)

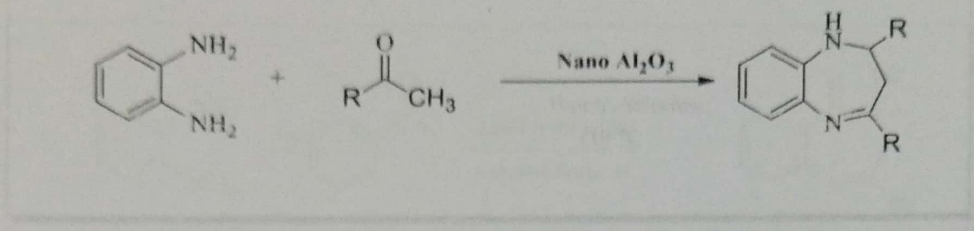
M. A. Alibeik have been proposed the hugely effective, discerning synthesis of 1,5-benzodiazepines at room temperature by stirring o-phenylenediamine with ketones employing alumina supported 12- tungstophosphoric acid (30 wt%) as catalyst under solvent free condition **(Scheme 6)[28].**



Scheme 6

3.6 R. Hekmatshoar et al. Approach (2009)

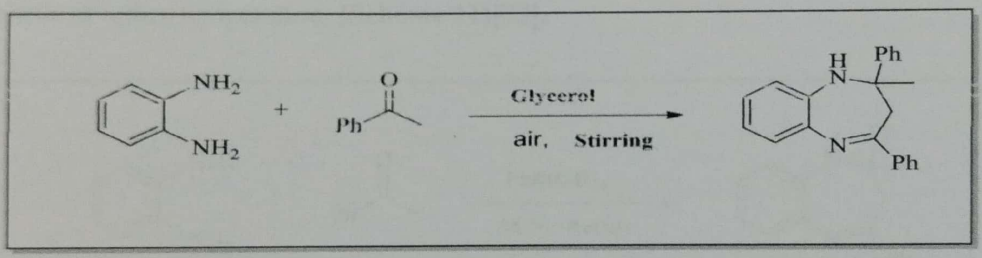
R. Hekmatshoaret. al was established the synthesis of 1,5- benzodiazepine derivatives by condensing ketones with 1,2-phenylendiamine using nano crystalline Al_2O_3 as catalyst at 110 °C with stirring in aqueous media . **(Scheme 7)[29].**



Scheme 7

3.7 C. S. Radatz et al. Approach (2011)

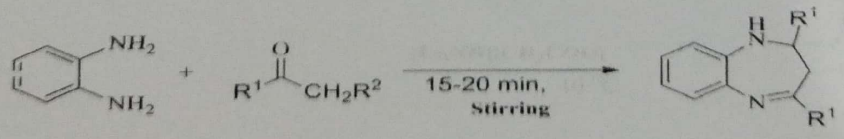
C. S. Radatz et al. introduced an easy and efficient protocol for catalyst free synthesis of 1,5- benzodiazepines derivatives by reacting o-phenylenediamine and various ketones using glycerol as solvent. **(Scheme 8)[30].**



Scheme 8

3.8 P. Attri Approach(2012)

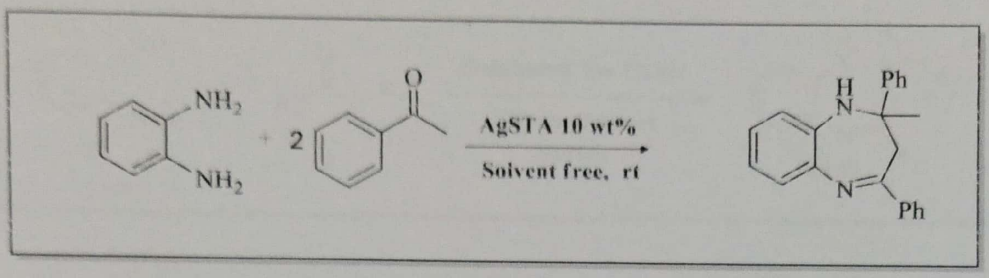
P. Attriet. al. have synthesized 1,5- benzodiazepine derivatives by using triethyl ammonium acetate(TEAA) ionic liquid as highly selective catalyst and medium under solvent free condition. The method is cost effective and environmentally benign.**(Scheme 9) [31].**



Scheme 9

3.9 A.H. Jadhav et al. Approach (2013)

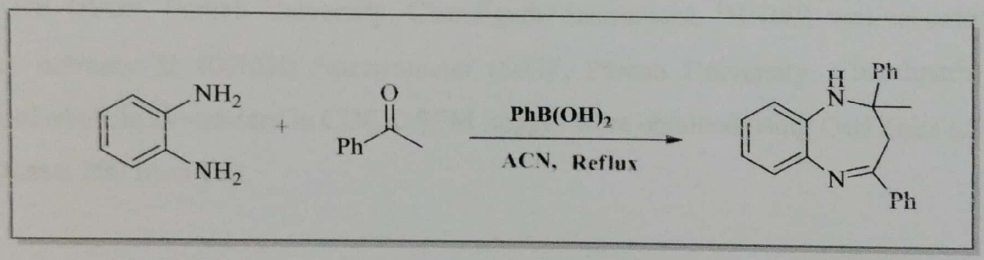
A.H. Jadhav et al. developed a simple and efficient process for functionalized 1,5-benzodizepines synthesis by catalytic condensation of benzene-1,2-diamines with various ketones using silicotungstic acid catalyst with stirring at room temperature. **(Scheme 10)[32].**



Scheme 10

3.10 S. V. Goswami et al. Approach (2017)

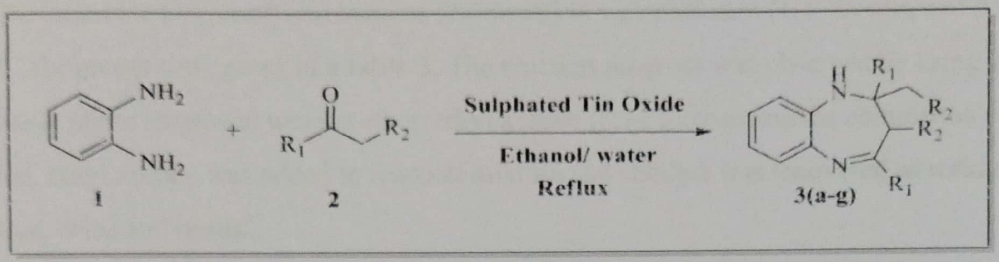
S. V. Goswami et al. have done the synthesis of 1,5- benzodiazepine derivatives using phenyl boronic acid as an efficient catalyst by condensing various ketones and 1,2- phenyl diamine in acetonitrile at reflux temperature. **(Scheme 11)[33].**



Scheme 11

4. PRESENT WORK

In the present work, we have developed synthesis of 1,5-benzodiazepine derivatives using sulfated tin oxide as solid super acid catalyst at reflux condition in ethanol.



4.1 Materials and Method

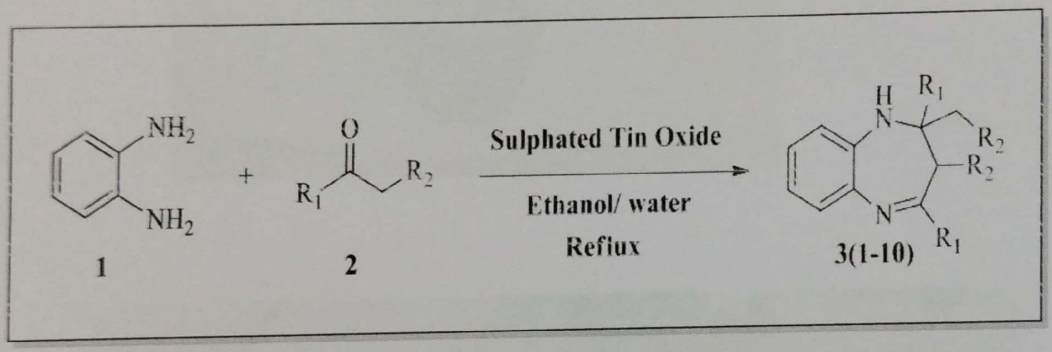
All the reagents used were brought from Sigma Aldrich, SD Fine. Solvents utilized for chromatography were distilled to make them pure. The reactions were reviewed with TLC using aluminum coated with silica gel. Melting points of produced derivatives were assessed on Fisher John's apparatus. The synthesized derivatives were examined by IR 1H NMR spectroscopy and Mass Spectrometry. IR spectra were invaded from a Perkin Elmer Spectrum RX FTIR (SAIF, Punjab University, Chandigarh) instrument. ^{1}HNMR was recorded on a Bruker Advance II 400MHz Spectrometer (SAIF, Punjab University, Chandigarh) using tetramethylsilane as standard in $CDCl_3$. SEM images were obtained using Carl Zeiss LSM 710 and Quanta 200 from FEI.

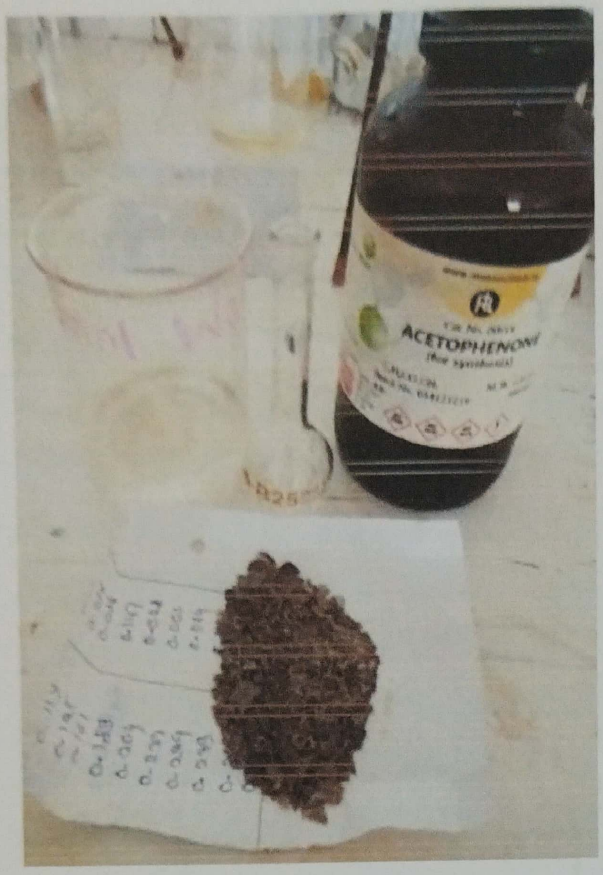
4.2 Synthesis of Sulfated Tin Oxide Catalyst

Sulfated tin oxide was produced ensuing literature process [23]. 50 g of stannous chloride was dissolved in 150 ml water. The pH of solution was adjusted to 8 with addition of 20% ammonia drop wise with continuous stirring. The precipitate developed then appended in 200 ml cold ammonium acetate solution (1 to 5%). The solid generated was salvaged by filtration and dried at 100 ^{0}C for 24-30 h. 20 ml concentrated H_2SO_4 was added slowly to obtained tin oxide and allowed to stand for 1 h. The solid obtained was filtered and dried at 100 ^{0}C, then further heated at 500° C for 4 h and potted in a closed sample bottle.

4.3 General Procedure for Synthesis of Substituted Benzodiazepine Derivatives

Sulfated tin oxide catalyst (25wt %) was added to a mixture containing o-phenylenediamine (10mmol) and ketones (25 mmol) in water/ethanol (1:1, 25 ml), and heated at 80°C for proper time given in a **table 3**. The reaction progress was observed by using TLC, the mobile phase employed was pet ether: ethyl acetate (8:2). Succeeding the completion of the reaction, ethyl acetate was added to reaction mixture and catalyst was recovered as residue by filtration, dried and reused.





5. RESULT AND DISCUSSION

5.1 Characterization of Synthesized Sulfated Tin Oxide

The synthesized sulfated tin oxide was inveterate by FTIR [Fig. 1]. The peaks at 985.45, 1074.16, 1193.72, 1170.58, and1355.71 cm^{-1}clearly indicates the presence of sulfate groups attached to tin.Super acidity of STO is due to the attached sulfate groups

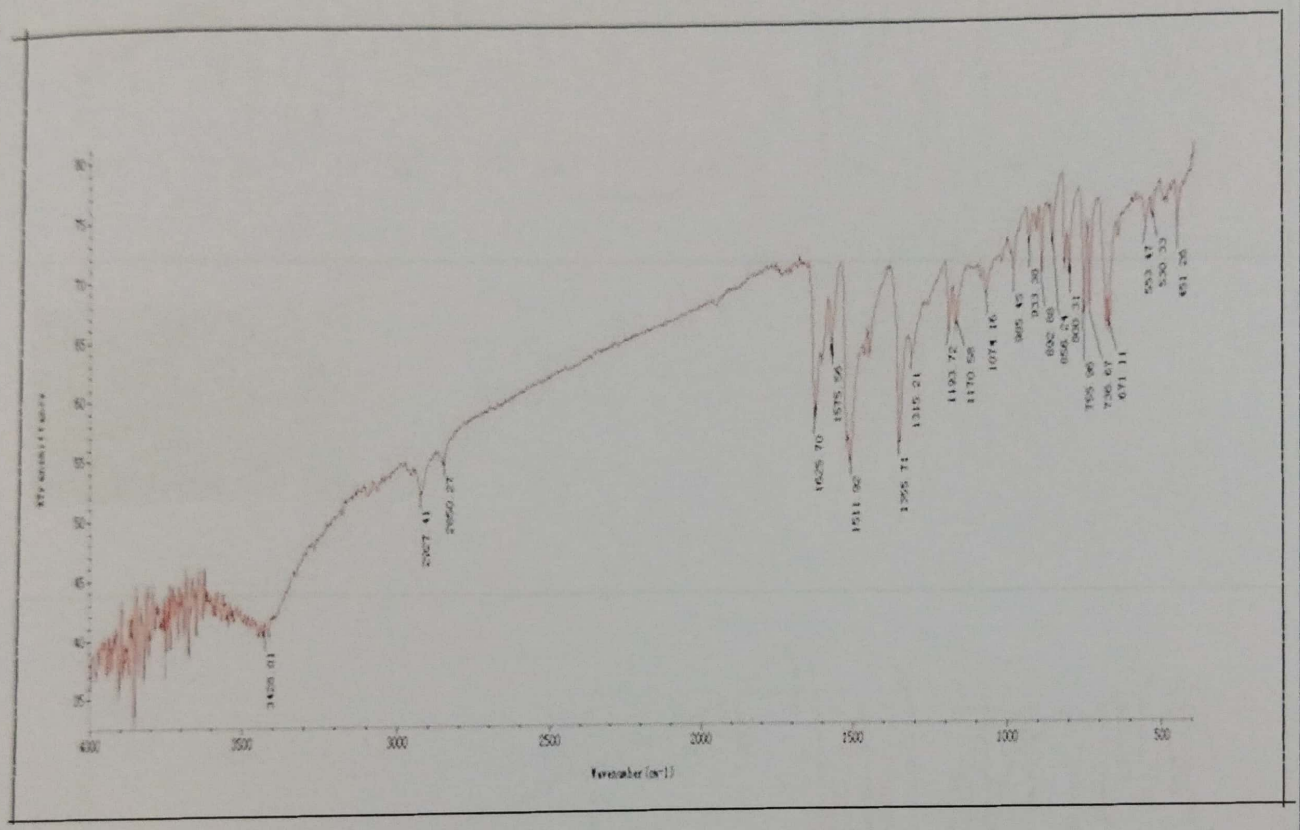
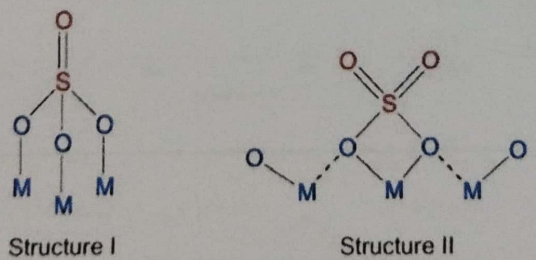
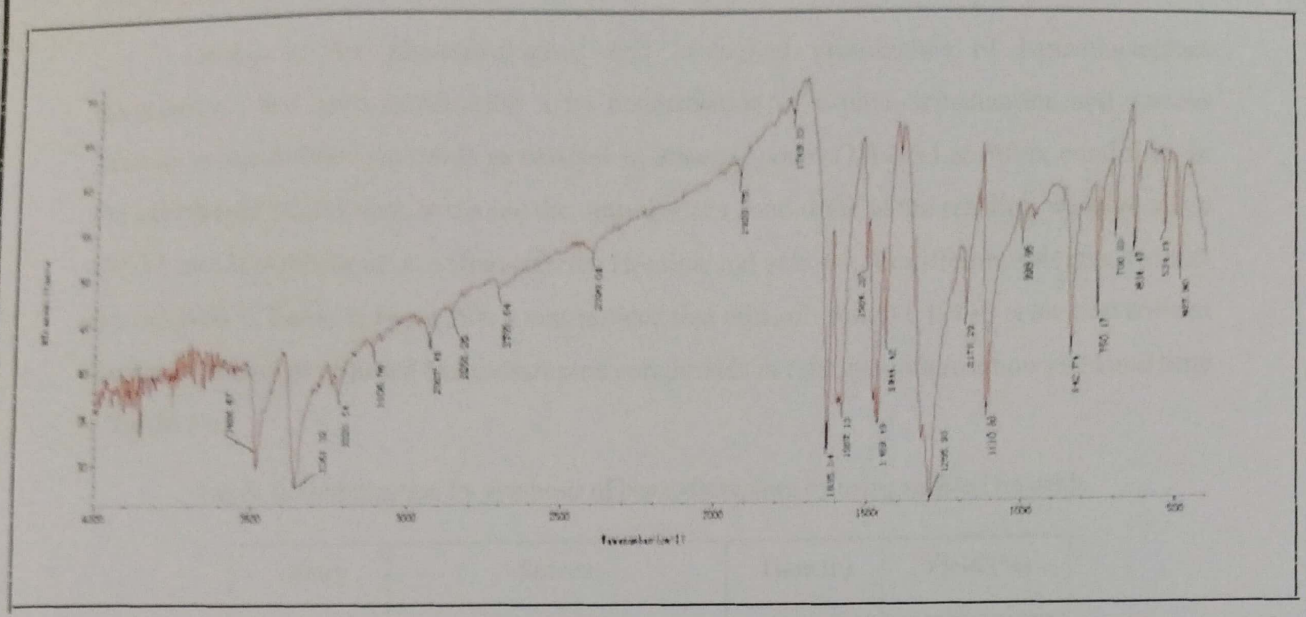


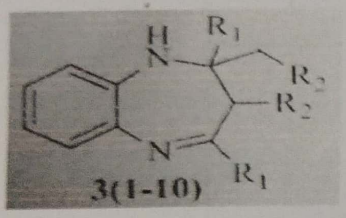
Figure 1 : Infra-Red Spectrum of Silfated Tin Oxide [STO]



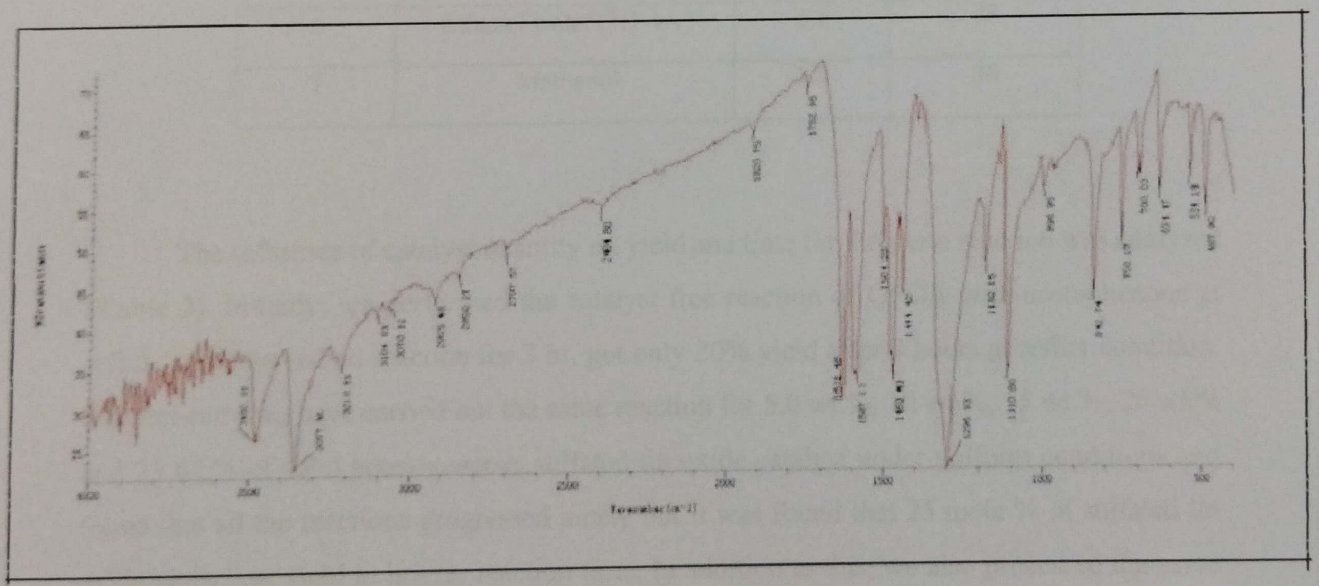
Structure I Structure II

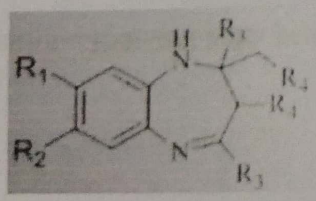
M = Sn





3(1-10)





5.2 Optimization of reaction conditions

Owing to the pharmacological and biological prominence of benzodiazepines derivatives , We have synthesized it by condensation of o-phynelenediamine and various ketones using sulfated tin oxide as catalyst in ethanol: water (1:1-v/v) at reflux condition. In the calibration experiment, to decide the optimization conditions of the reaction we have taken OPDA and acetophenone as a characteristic reaction and vetted it for different solvents. Results are outlined in **Table 1**. From this, it was noticed that ethanol: water(1:1-v/v) is the best solvent for the synthesis of required benzodiazepine compounds in relation with reaction yield and time (**Table 1**).

Table 1: Optimization for synthesis of benzodiazepines by using sulfated tin oxide

Entry	Solvent	Time (h)	Yield[a](%)
1	Toluene	3.8	72
2	Acetonitrile	4.0	75
3	DMF	4.2	78
4	Chloroform	3.8	80
5	Ethanol	2.2	88
6	Ethanol water (1:1-v/v)	2.0	88
7	Methanol	2.4	80

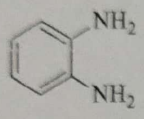
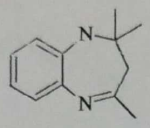
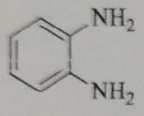
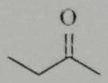
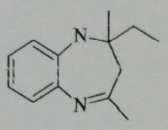
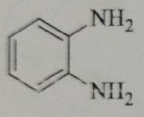
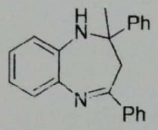
The influence of catalyst quantity on yield and time for the same reaction was analyzed (**Table 2**). Initially, we performed the catalyst free reaction of OPDA with acetophenone at reflux, we observed no reaction for 3 hr, got only 20% yield after 5 hours at reflux condition. Subsequently we have carried out the same reaction for 5.0 wt.%, 10 wt.%, 15 wt.%, 20 wt.% and 25 wt.% of solid heterogeneous sulfated tin oxide catalyst under uniform conditions and found that all the reactions progressed nicely but it was found that 25 mole % of sulfated tin oxide gives best yield in briefer reaction time. In addition to this, we also processed the same reaction applying other heterogeneous catalyst providing less yields. (**Table 2**).

Table 2: Optimization of catalyst for synthesis of benzodiazepines by using sulfated tin oxide

Entry	Catalyst amount in wt%	Time (hr)	Yield[a](%)
1	No catalyst	5	20
1	05	3.4	70
2	10	3.0	76
3	15	2.6	81
4	20	2.3	84
5	25	2.0	88

Owing to promising reactivity of sulfated tin oxide, it has been exercised in varied organic transformations. We have developed efficient and green approach for synthesis of biologically important benzodiazepines by employing sulfated tin oxide in ethanol: water. To study the synthetic dimensions and effectiveness of the protocol, a series of symmetrical and unsymmetrical ketones were reacted with di-amino-arenes and substituted diaminoarenes under the optimized reaction conditions. The outcomes are précised in **Table 3**.

Table 3: Synthesis of substituted benzodiazepine derivatives by using Sulfated tin oxide as catalyst at reflux condition in ethanol: water media.

Entry	Diamine	Ketone	Product	Yield%	Time (hr)	M.P. ^{0}C (Obs)
1				90	2.0	136-138
2				88	2.1	138-140
3				88	2.0	150-152

7. TLC ANALYSIS

After completion of the reaction, the formed products were analyzed by thin layer chromatography (TLC).

Fig. TLC of Sulphated Tin Oxide [Catalyst]

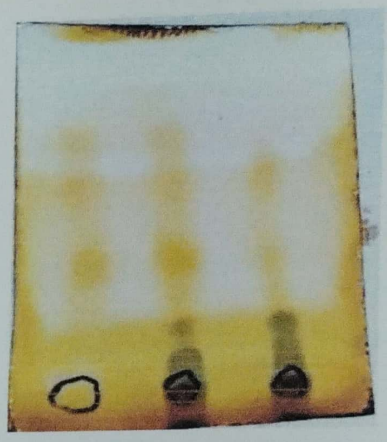


Fig. TLC of Derivative

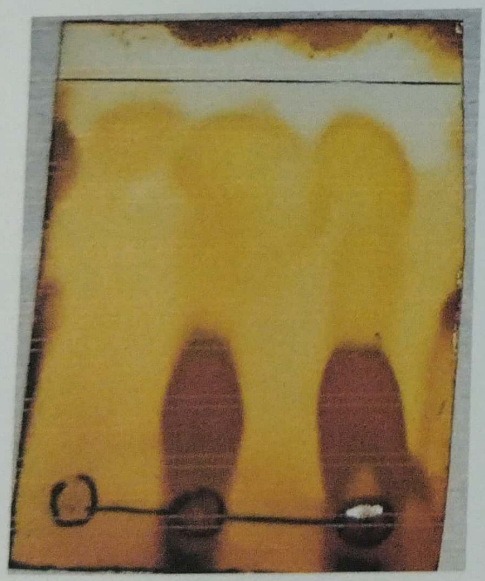
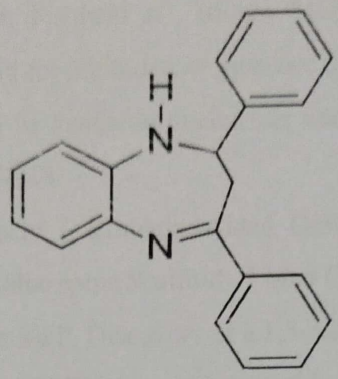


Fig. TLC of Derivative

8. CONCLUSION

A solid acid heterogeneous catalyst of sulfated tin oxide was prepared and wasexercised in the catalytic synthesis of 1,5-benzodiazepines in ethonolic aqueous medium underreflux condition. The offered catalytic schemerevealed90 % yield of 1,5-benzodiazepines at reflux temperature using 25 mole % of sulfated tin oxide catalyst. The sulfated tin oxide catalystcan be recycled by a simple filtration process after completion of the reaction and reprocessedfor five cycles with no significant loss of catalytic activity and selectivity. The results reveal that sulfated tin oxide is an exceptional and environmentally benign solid acid catalyst for the synthesis of 1,5-benzodiazepine under mild, clean, high yielding and environmentally affable reaction conditions.

9. SPECTRAL ANALYSIS



IUPAC NAME= 2,4-dihenyl-2,3-dihydro-1H-1,5 benzodiazepine

	IR VALUE	
1.	3431 per cm	N-H streching
2.	3018 per cm	Aromatic C-H streching
3.	1644 per cm	C-N streching
4.	1307-1470 per cm	Aromatic C-C streching
5.	1216 per cm	C-N bending
6.		

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