

# Synthesis and spectral characterization of carboxylate ligand derived from 2, 4-dnph

---



BY

Abubakar Abdur-Rehman

ID: 13004067009

SUPERVISOR:

Hamid Raza

---

DEPARTMENT OF CHEMISTRY  
SCHOOL OF SCIENCE  
UNIVERSITY OF MANAGEMENT AND TECHNOLOGY,  
LAHORE, PAKISTAN  
2017

# Synthesis and spectral characterization of carboxylate ligand derived from 2, 4-dnph

Submitted to University of Management and Technology Lahore

In partial fulfillment of the requirements

For the award of degree of

## **BS IN CHEMISTRY**

BY

Abubakar Abdur-Rehman

ID

1	3	0	0	4	0	6	7	0	0	9
---	---	---	---	---	---	---	---	---	---	---

SESSION: 2013-2017

DEPARTMENT OF CHEMISTRY  
SCHOOL OF SCIENCE  
UNIVERSITY OF MANAGEMENT AND TECHNOLOGY,  
LAHORE, PAKISTAN

بِسْمِ اللَّهِ الرَّحْمَنِ الرَّحِيمِ

*In the name of  
Allah,  
The most Compassionate,  
The most merciful*

The Prophet (Peace be upon him) said,  
“Allah makes the way to Jannah easy for him who treads the path in  
search of knowledge.”

(Al-Muslim)

University of Management and Technology, Lahore

**Similarity Report**

Turnitin Originality Report

SYNTHESIS AND SPECTRAL CHARACTERIZATION OF CARBOXYLATE LIGAND,  
DERIVED FROM 2,4 DINITROPHENYLHYDRAZINE by Abubakar Abdur-rehman  
From Reference Desk (Nov) (LRC (Info))

- Processed on 01-Aug-2017 15:53 PKT
- ID: 834479392
- Word Count: 8298

Similarity Index

4%

Similarity by Source

Internet Sources:

1%

Publications:

3%

Student Papers:

1%

**sources:**

< 1% match (publications)

Susan Richardson, "Application of DNPH Derivatization with LC/MS to the Identification of Polar Carbonyl Disinfection Byproducts in Drinking Water", Ozone Science & Engineering, 12/01/2000

< 1% match (publications)

Sundaraganesan, N., "FTIR, FT-Raman spectra and ab initio, DFT vibrational analysis of 2,4-dinitrophenylhydrazine", Spectrochimica Acta Part A: Molecular and Biomolecular Spectroscopy, 200701



Checked by



Verified by CLO

**Note:**

- Sometimes the overall similarity index may be a smaller than the repository percentages combined. This would be due to overlapping text within the repositories.
- It is a system generated report.



---

## **DECLARATION**

**I ABUBAKAR S/O ABDUR-REHMAN      ID: 13004067009**

Session **2013-2017** hereby declare that the matter printed in the thesis titled ***“SYNTHESIS AND SPECTRAL CHARACTERIZATION OF CARBOXYLATE LIGAND DERIVED FROM 2,4-DNPH”*** is my own work and has not been printed, published and submitted as research work, thesis or publication in any form in any University, Research institution etc. in Pakistan or Abroad.

*Dated:* \_\_\_\_\_

\_\_\_\_\_

***(Abu\_Bakar)***



---

## **RESEARCH COMPLETION CERTIFICATE**

Certified that the research work contained in this thesis titled, “SYNTHESIS AND SPECTRAL CHARACTERIZATION OF CARBOXYLATE LIGAND DERIVED FROM 2, 4-DNPH” has been carried out and completed by **ABU-BAKAR ABDURREHMAN**, ID: **13004067009**. The quantum and the quality of the work contained in this thesis is adequate for the award of Degree of BS in Chemistry.

---

**Supervisor**

---

**Dr. Sammia Shahid**  
**Chairperson,**  
Department of Chemistry,  
UMT, Lahore.

## **Dedication**

DEDICATE THIS REPORT TO HOLY PROPHET MOHAMMAD  
(PEACE BE UPON HIM)

THE GREAT TEACHER AND REFORMER.

MY FAMILY WHOSE COURAGE AND PRAYERS ENABLE ME TO  
COMPLETE THIS WORK.

**ABU-BAKAR ABDURREHMAN**

## **ACKNOWLEDGEMENT**

I am really thankful to **ALLAH Almighty**, Who is one and only creator of everything, He knows all the concealed and evident things of universe, who enable me to perform and complete this work. Countless salutations are upon the **Holy Prophet Mohammad** (peace be upon him) who is the best reformer and teacher of the world, who gave us the sense of acquiring knowledge from everywhere.

I would like to express my profound gratitude to my supervisor **Dr. Hamid Raza**, Assistance Professor Department of Chemistry University of Management and Technology Lahore, whose expertise, understanding and patience, added considerably to my graduate experience. I really appreciate his devoted, loveable behavior for me during my work.

I am thankful to honourable Dean of School of Science University of Management and Technology **Dr. Azhar Iqbal** for his guidance during my course studies.

I am thankful to **Dr. Sammia Shahid**, Chairperson of Chemistry Department, University of Management and Technology and to **Dr. Khurrum Shahzad**, for their guidance and supporting attitude during my research work.

I am also thankful to **Mr Mehrouz** research officer at Pakistan Council of Scientific and Industrial Research (PCSIR), Lahore kind help in FT-IR during my research work.

I am thankful to all my colleagues and friends for their unforgettable company, corporation and help during my entire course study.

At the end I am very much thankful to all my family members, my parents and especially my sisters whose encouragement and prayers enable me to reach my destination.

**May Allah bless all of them.**

## **ABSTRACT**

Most of the ligands have been synthesized from different aldehydes and amines with the continuous magnetic stirrer method. The ligand in this study is synthesized from the reaction of Maleic anhydride with 2, 4-Dinitrophenylhydrazine, which gives the yellow solid product of 2,4DNPH-M-P, with a sharp melting point that is insoluble in most of organic solvents.

The spectroscopic study helps to predict the structure of the product 2,4DNPH-M-P and confirm the results using the Fourier transform infrared spectroscopy (FTIR) technique which identifies the different bonding present in it.

The pH and conductivity of the synthesized carboxylate ligand 2,4DNPH-M-P is checked in different solvents. It has different pH and conductivity in different solvents. Methanol and Ethanol has high conductivity compared to other solvents, and the pH of the solvents changes from basic to acidic medium. The synthesized carboxylate ligand 2,4DNPH-M-P has many important applications in biomedical, complexes formation, and Metal Organic Frameworks (MOFs).



# CONTENTS

Acknowledgement	v
Abstract	vi
List of table's	x
List of figures	xi

## **CHAPTER 01: INTRODUCTION 01-13**

1.1	Physical properties of Maleic anhydride	02
1.2	Chemical Properties of Maleic anhydride	03
1.2.1	Reactivity	03
1.2.2	Flammability	03
1.3	Synthesis & spectral characterization of Maleic anhydride	03
1.4	Applications of Maleic anhydride	04
1.4.1	Bio-adhesives	04
1.4.2	Polyester Resin	04
1.4.3	Additives in lubricants	04
1.4.4	Food & beverages	05
1.5	Health Hazards	05
1.6	History of 2,4-DNPH	06
1.7	Physical Properties of 2,4-DNPH	06
1.8	Synthesis of 2,4-DNPH	07
1.8.1	Bromobenzene	07
1.8.2	2,4-Dinitrobromination	07
1.8.3	2,4-Dinitrophenylhydrazine	08
1.9	Structure & Spectral Characterization of 2,4-DNPH	08



---

1.9.1	FTIR Spectroscopy Analysis	08
1.9.2	NMR Spectroscopy	08
1.9.3	Fluorescence Study	09
1.9.4	Optical Transmittance	09
1.10	DNPH Test for Carbonyl Compounds	09
1.11	Applications of 2, 4-Dinitrophenylhydrazine	10
1.11.1	Determine Formaldehyde in air	10
1.11.2	Determine microgram quantity of Prednisone	10
1.11.3	Identification of Polar Carbonyl Compounds in Drinking Water	10
1.11.4	For Vitamin C & Blood Analysis	10
1.11.5	Identification of Carbonyl Compounds	11
1.12	Source and Potential Exposure of 2, 4-DNPH	11
1.12.1	Air	11
1.12.2	Water	11
1.12.3	Soil	11
1.13	Potential Health Hazards & Toxicity	12
1.13.1	Acute Health Effects	12
1.13.2	Chronic Health Hazards	12
1.14	Control and Protection	13
1.14.1	Clothing	13
1.14.2	Eye protection	13
1.14.3	Respiratory protection	13
1.15	Aims & Objectives	13



---

<b>CHAPTER 2: LITERATURE REVIEW</b>	<b>14-23</b>
<b>CHAPTER 3: EXPERIMENTAL WORK</b>	<b>24-33</b>
3.0 Experimental work	25
3.1 Materials	25
3.2 Apparatus and Instruments	25
3.3 Analytical and Separation techniques	26
3.4 Solubility	26
3.5 Synthesis of Carboxylate Ligand	27
3.6 Procedure	28
3.7 Reaction and Percentage Yield	29
3.8 Melting Points	29
3.9 Conductivity & pH of 2, 4-DNPH-M-P in Different Solvents	30
3.10 Effect of pH in different solvents in the presence of 2, 4-DNPH-M-P	31
3.11 Conductivity effect in different solvents in the presence of 2, 4-DNPH-M-P	32
<b>CHAPTER 4: RESULTS AND DISCUSSIONS</b>	<b>34-41</b>
Summary	40
Conclusion	40
<b>CHAPTER 5: REFERENCES</b>	<b>42-45</b>



---

## **LIST OF TABLES**

3.4.1	Solubility of 2, 4-DNPH in Different Solvents	26
3.4.2	Solubility of Maleic anhydride in Different Solvents	27
3.8	Melting Points of 2, 4-DNPH-M-P in Different Solvents	30
3.9	Effect of Carboxylate Ligand on pH and Conductivity	31

---

## **LIST OF FIGURES**

4.1	Fourier Transform Infrared Spectroscopy (FT-IR)	35
4.2	FTIR Spectrum of 2, 4-Dinitrophenylhydrazine	37
4.3	FTIR Spectrum of Maleic Anhydride	38
4.4	FTIR Spectrum of synthesized Carboxylate Ligand 2, 4-DNPH-M-P	39

---

## Introduction

### MALEIC ANHYDRIDE

Maleic anhydride with its formula  $C_2H_2(CO)_2O$ , is a needle like, colorless crystalline. It is a versatile compound that it contains many applications itself. There are three active sites present in the maleic anhydride. It acts as an intermediate in the fine chemical industry, especially in the agriculture chemical synthesis and oil additive lubricants. Maleic anhydride is a classic Diels-Alder reagent. It has been used mostly in resin formulation.

#### 1.1 Physical properties of Maleic anhydride

- I. The chemical formula of Maleic anhydride is  $C_4H_2O_3$  and has the molecular weight of 98.06 g/mol.
- II. Maleic anhydride decomposes in water and forms colorless or white solid crystals.
- III. The boiling point of maleic anhydride is  $202^\circ\text{C}$  and its melting point is  $52.8^\circ\text{C}$ .
- IV. Maleic anhydride has the specific gravity 1.48 and its conversion factor is;  
 $1 \text{ ppm} = 4.0 \text{ mg/m}^3$ .
- V. Maleic anhydride has a pungent smell with the 0.32 ppm threshold odor.
- VI. Vapor pressure of Maleic anhydride has been observed between 0.1\_0.2 mm Hg at  $25^\circ\text{C}$ . (*Jiang, Feng et al. 2014*)