Republic of Iraq

Ministry of Higher Education and Scientific Research

Diyala University/ College of Science

Department of Chemistry

Preparation of some organic compounds and study it's biological activity

Submitted to the Council of the Chemistry Department-College of science- Diyala University

to complete the requirements of obtaining bachelor's degree

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Supervised by:

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بسم الله الرحمن الرحيم

(قَالُوا سُبْحَانَكَ لَا عِلْمَ لَنَا إِلَّا مَا عَلَّمْتَنَا إِنَّكَ أَنْتَ الْعَلِيمُ الْحَكِيمُ)

صدق الله العظيم

البقرة(32)

Gifting

I dedicate all my efforts to:

The leader of my life…….Prophet Mohammed

(Allah peace be upon him)

To my life love……My parents

To the candles on my way.....My brothers and My sisters

To everyone helped me to do this work….I introduce my work with respect.

Appreciation and Thanks

As seniors, we inevitably are pacing our approximately the last steps in our university life. We should have a halt remembering our past years in the university with our honourable instructors who exerted their effort on raising the future generation so as to raise the nation again.

Before we go on, we present our great thanks, love and appreciation to those who carry the holy message in life; those who smoothed the knowledge for us; to our virtuous instructors.

"Be a scientist, if you could not, a learner, if you could not like scientist, if you could not then do not dislike them"

I especially thank and appreciate:Division head the chemistry. dr. wassanBaqir Ali

We would like to tell him the rejoiceful Hadith of the messenger of Allah (peace be upon him and his family and chosen companions):

"The whale in the sea, and the birds in the sky bless who teaches people the good."

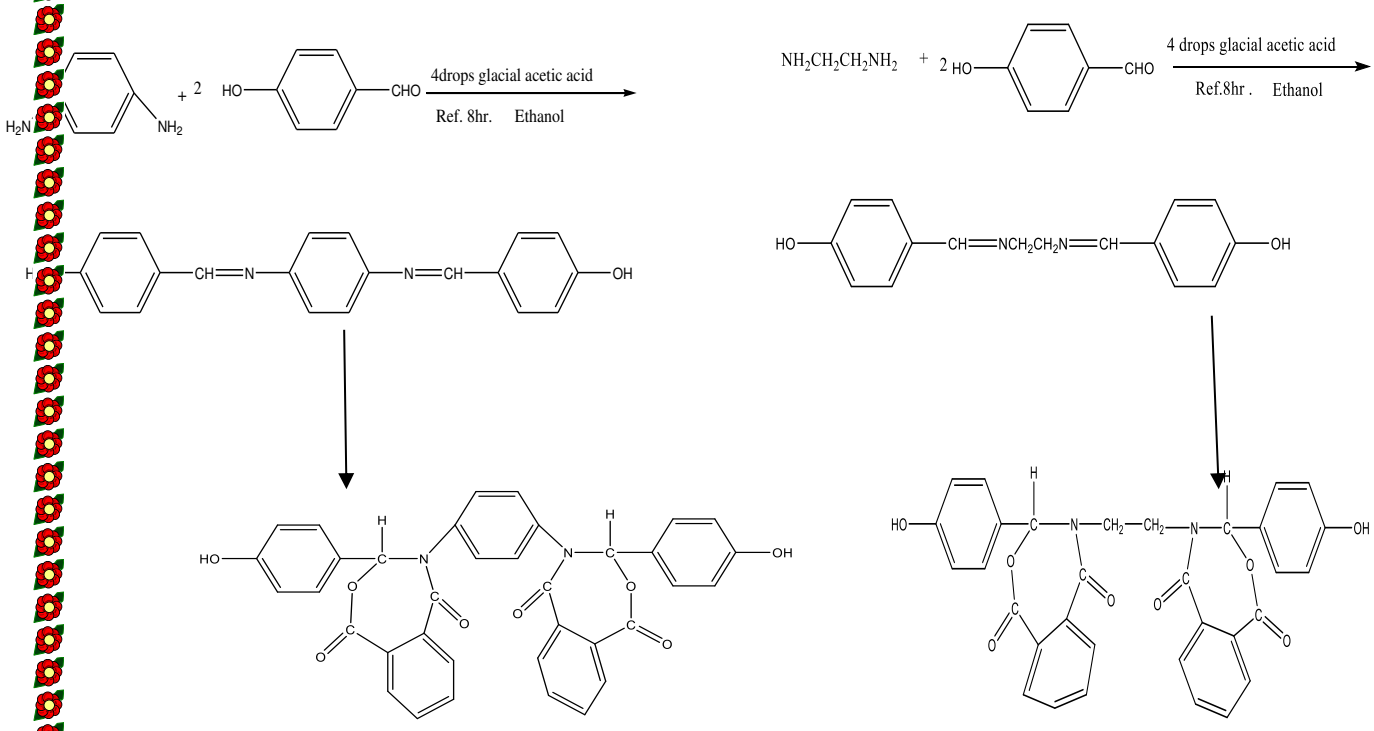
We also think everyone helped us to complete this research, cooperated gave us the hand of help and who provided us with the required information. To whom who made us optimistic on our way and provided us with help, facilities, thoughts, and information, bat may be they do not feel their role of that so we thank them a lot.

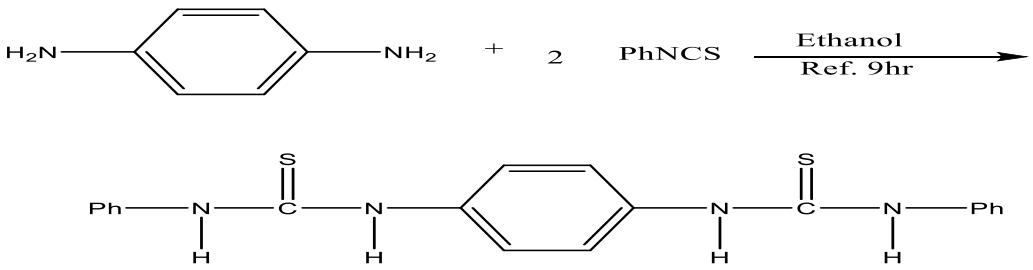
As for the special thank, we absolutely give it to those who didn't help us, hindered our research process a little, and planted thorn on our research way; otherwise we can not help feeling enjoyed our research, nor the positive contest, and therefore, we could not reach what we have reached, so many thanks. and Allah is the granter of success.

List of contents

SUMMARY

The schiff bases are very important class of compounds used in biological and industrial fields. In this research we prepared five compounds according to the scheme below. The synthesized compounds have been characterized by some spectroscopic methods such as (FT-IR) and (^{1}H-NMR) and determination of their physical properties such as melting points.





Chapter one

Chapter one

introduction

1.1. General introduction of heterocyclic compounds

A cyclic organic compound containing all carbon atoms in ring formation is referred to as a carbocyclic compound. If at least one atom other than carbon, forms a part of the ring system then it is designated as a heterocyclic compound. Nitrogen, oxygen and sulfur are the most common heteroatom but heterocyclicrings containing other hetero atom are also widely known . an enormous number of heterocyclic compounds are known and this number is increasing rapidly. Accordingly the literature on the subject is vary vast. Heterocyclic compound may be classified into aliphatic and aromatic . the aliphatic heterocyclic are the cyclic analogues of amines, ethers, thioethers, amides, etc. Their properties are particularly influenced by the presence of strain in the ring.[(1)] These compounds generally consist of small(3-and4-membered) and common (5 to 7 member) ring systems.The aromatic heterocyclic compound, in contrast, are those which have a heteroatom in the ring and behave in a manner similar to benzene in some of their properties. Furthermore, these compounds also comply with the general rule proposed by Höckel. This rule states that aromaticity is obtained in cyclic conjugated and planar systems containing(4n+2)π electrons. The conjugated cyclic rings contain six π-electrons as

in benzene, and this forms a conjugated molecularorbital system which is thermodynamically more stable than the non-cyclically conjugated system. This extra stabilization results in a diminished tendency of the molecule to react by addition but a larger tendency to react by substitution in which the aromatic ring remains intact.

A heterocyclic ring may comprise of three or more atoms which may by saturated or unsaturated. Also the ring may contain more than one hetero atom which may be similar or dissimilar.[(2)]

1.2.p-phenylenediamine

p-phenylenediamine (PPD) is an organic compound with the formula $C_6H_4(NH_2)_2$.This derivative of aniline is a white solid, but samples can darken due to air oxidation.It is mainly used as a component of engineering polymers and composites. It is also an ingredient in hair dyes. It's produced via two routes. Most commonly , 4-nitrochlorobenzene is treated with ammonia and the resulting 4-nitroaniline is then hydrogenated:

$$ClC_6H_4NO_2 + 2NH_3 \rightarrow H_2NC_6H_4NO_2 + NH_4Cl$$

$$H_2NC_6H_4NO_2 + 3H_2 \rightarrow H_2NC_6H_4NH_2 + 2H_2O$$

In the second rout, aniline is converted to diphenyltriazine, which is converted by acid-catalysis to 4-aminoazobenzene.

Hydrogenation of the latter affords PPD. It's a precursor to aramid plastics and fibers such as Kevlar. These application exploit PPD'S difunctionality, i.e. the presence of two anmines which allow the molecules to be strung together. This polymer arises from the reaction of PPD and terephthaloyl chloride. The reaction of PPD with phosgene gives the diisocyanate, a precursor to urethane polymers.

This compound is a common hair dye. It's use is being supplanted by other aniline analogues and derivatives such as 2,5-diaminotoluene. Other popular derivatives include tetraaminopyrimidine and indoanilines and indophenols. Derivatives of diaminopyrazole give red and violatcolours. In these applications, the nearly colourless dye precursor oxidizes to the dye. It ś easily oxidizes, and for this reason derivatives of PPD are used as antiozonants in production of rubber products. The substituent's, naphthyl, isopropyl etc. affect the effectiveness of their antioxidant roles as well as their properties as skin irritants.[3]

1-3. Schiff bases-

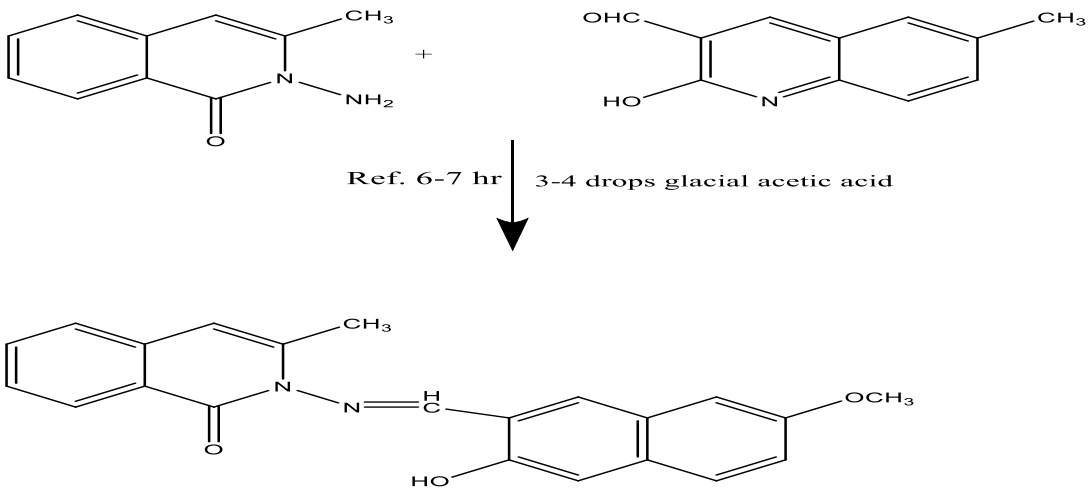
A Schiff bases the compounds that containing imines group or called (Azomethen) and it's called Schiff bases due to the Schiff scientist[4] which preparation this type of the compounds in (1764) of condensation of an aldehydes or ketones (aliphatic or

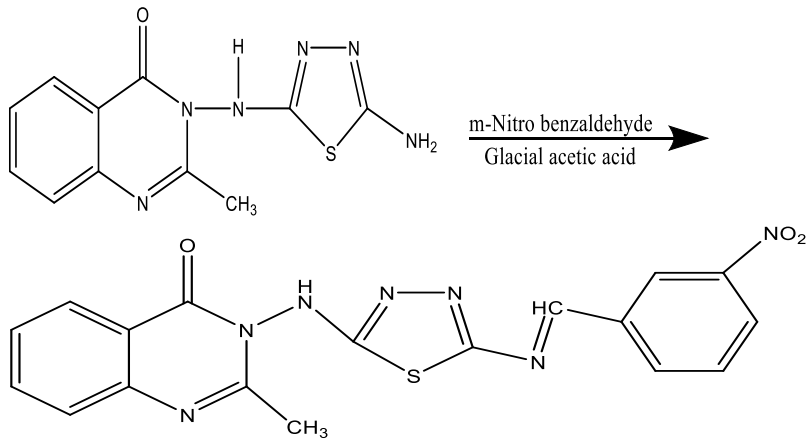
aromatic).with aprimary amine or with amino acids. As well as the base that derived from amine and ketone is called (ketimine) and the base that derived from amine and aldehyde is called (aldimine).

the Schiff bases could be known as a hydrazone that produced form the hydrazone reaction the appropriate acid with ketones or aldehydes in solvent suitable.

1.3.1. Synthesis methods of Schiff bases

In general the Schiff bases prepared from the reaction of equal moles of aldehydes or ketones with the primary amines in appropriate solvent for aperoid of time and some times add some drops of the glacial acetic acid or hydrochloricacid or drops of pyridine as auxiliaries[5,6] as it show in the following equations:-

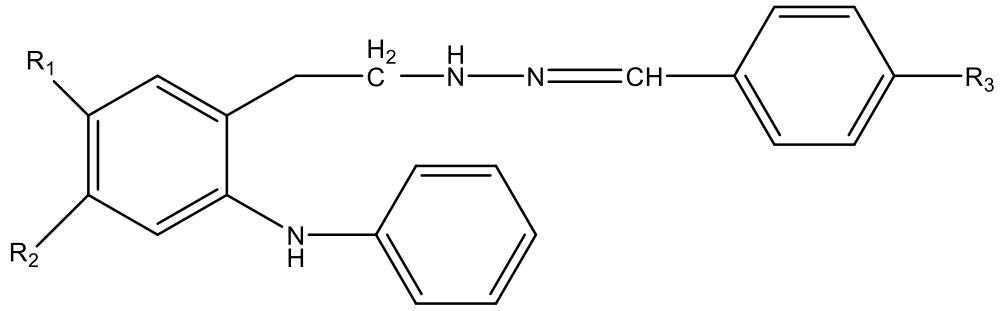




1.3.2. Applications of Schiff bases

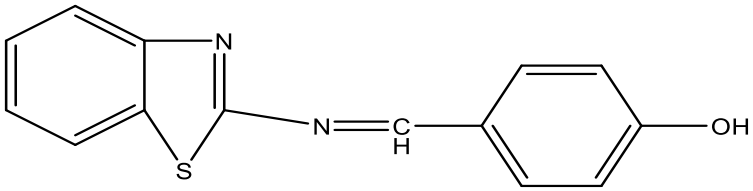
The Schiff bases are considered of important compounds because they have biological activity[7] which they are used in preparing some pharmaceuticals[8] that prevent the growth of germs and anti-insects[9] and anti-fungal[10] and anti-bacterial[11].

The Schiff bases own this biological activity due to the izomethine group which are found in their compound so it gives the Schiff bases this biological activity .it has been found that the Schiff bases show activity to the bacilli that the cause tuberculosis disease[12] as in the compound follow:

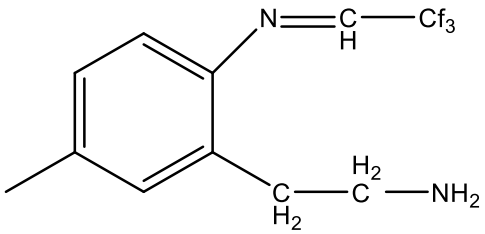


R^1= -OCH_3, CH_3 R^2= -CH_2OCH_3 ,R^3= -Cl

Some other compounds also showed anti bacterial activity [13] and other anti fungal[14,15] as in the compound follow:-

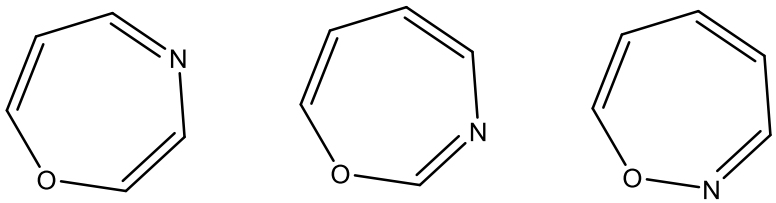


the Schiff bases were used as anti anxiolytics[16] as in the follow:-



1.4.The oxazepine compounds

The oxazepineis septuple annular unsaturated compound annular composition contain non-homogeneous atoms and there are three isomers represent the oxazepin compounds are 1،2- 1،3- 1،4 the oxazepin and this puuctuation depends on the location of two atoms nitrogen and oxygen as following(17)



1,4-oxazepin 1,3-oxazepin 1,2-oxazepin

Which the septuple annular uneven when comparison with the benzene ring hexagonal ring and that because of the increased size of

the ring and as a result that the ring take form resembled the boat due to the spatial distribution of atoms which make them more stable, as well as the non-prescription equation is the reason that make this compounds form the non aromatic and because of it's importance in medical

and psychological field the resear chars were interested in number preparation of this compounds[18] as well as new derivatives preparation where formed in (1965) which are used for the treatment of diseases of stress and nervous tension[19].

Chapter two

Chapter Two

Experimental part

2.1: The devices used at work

***Use the following devices for the preparation of compounds and spectral measurements and physical properties of* compounds prepared.**

1-Electric balance

2-Digital Melting point measurement

3-Oven apparatus

4-Hotplat stirrer

5-Reflux apparatus

6- The infrared measurement using the device **PERKIN ELMER SPEACTUM-65** within the range [5000-400] using KBR disc in the Chemistry lab / Diyala University Department.

2.2: Chemicals

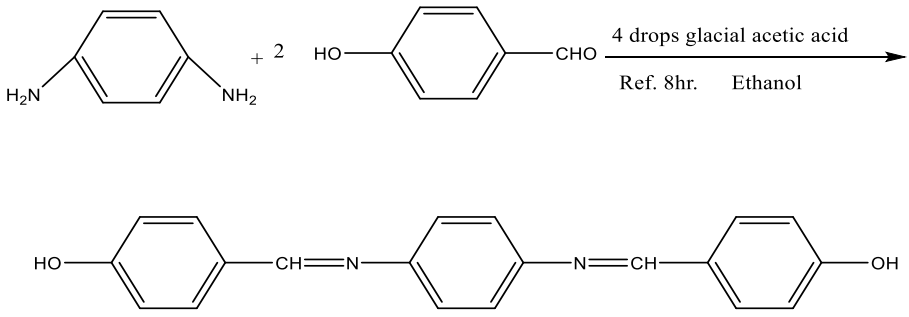
The following utilized chemicals were used listed with formula .

NO	*Substances purity%*	*Company*	*Formula*
1	*P-phenylenediamine 97%*	*BDH*	*C6H4N2*
2	*P-hydroxybenzyldehyde 99.9%*	*Merk*	$C_6H_6O_2$
3	*Ethelenediamine 97%*	*Merk*	$C_2H_8N_2$
4	*Phthalic anhydride 99%*	*Merk*	$C_8H_4O_3$
5	*Ethanol absolute 99.5%*	*GCC*	C_2H_5OH
6	*Glacial acetic acid 99.9%*	*BDH*	$C_2H_4O_2$
7	*Phenyl Isothiocyanate 98%*	*Merk*	C_7H_5NS
8	*Dimethyl sulfoxide*	*CDH*	C_2H_6OS

2.3:The preparation of the proposed compound

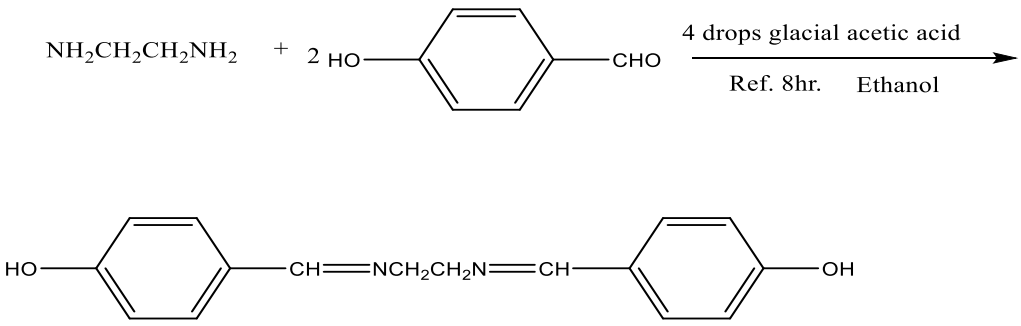
2.3.1: preparation of Compound C_1:($C_{20}H_{16}N_2O_2$)

In100 ml round bottom flask equipped was placed a mixture of(0.008 ml, 0.97 gm) of P-hydroxybenzyldeheyde and (0.004 ml, o.43 gm) of P-phenylenediamine in 25 ml of ethanol absolute with (4)drops of glacial acetic acid. The reaction mixture was refluxed in water bath at 78°c for 8hr,the solvent was then removed and the resulting solid was recrystallized from ethanol,dried and measured percentage.



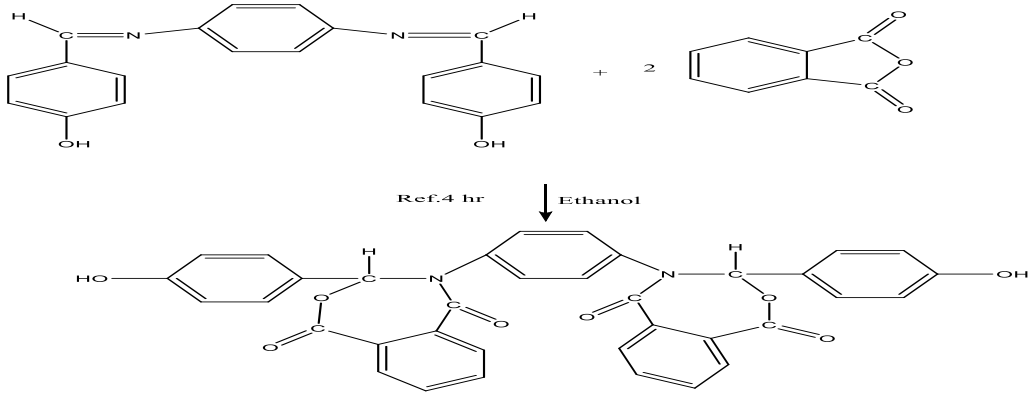
2.3.2: preparation of Compound C_2:($C_{16}H_{16}N_2O_2$)

Take in a 100 ml round bottom flask equipped was placed a mixture of (0.008 ml,0.97 gm) P-hydroxybenzyldehyde and (0.004 ml, 0.24) of Ethelenediamine in 25 ml of ethanol absolute with (4) drops of glacial acetic acid. The reaction mixture was refluxed in water bath at 78c for 8hr, the solvent was then removed and the resulting solid was recrystallized from ethanol, dried and measured percentage.



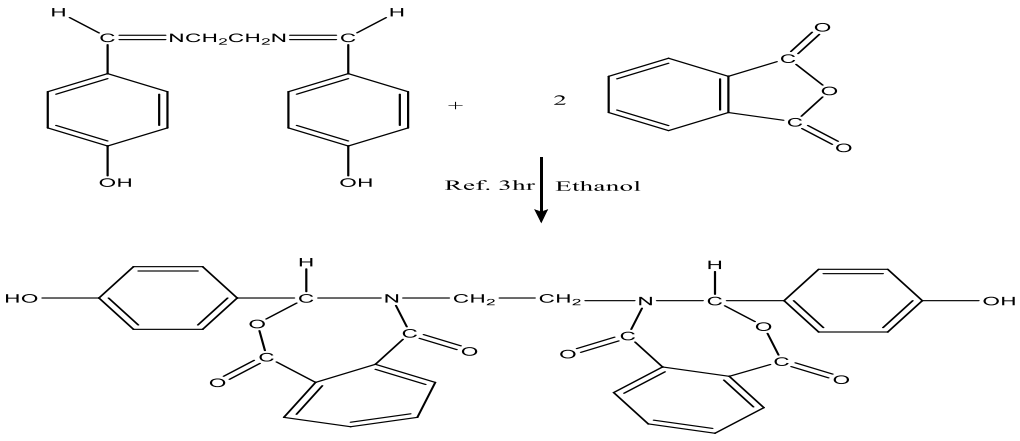
2.3.3: preparation of Compound C_3:($C_{36}H_{24}N_2O_8$)

Take in a 100 ml round bottom flask equipped was placed a mixture of(0.001ml, 0.51gm) of the C_1 compound and(0.002ml, 0.29gm) of phthalic anhydride in 25 ml of ethanol absolute. The reaction mixture was refluxed in water bath at 90 °c for 3hr, the solvent was then removed and the resulting solid was recrystallized form ethanol, dried and measured percentage.



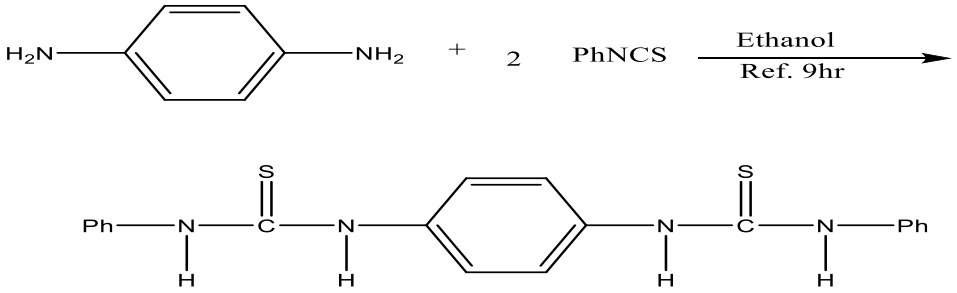
2.3.4: preparation of Compound C_4:($C_{30}H_{24}N_2O_8$)

Take in 100 ml in round bottom flask equipped was placed a mixture of (0.002ml,0.54gm) of the C_2 Compound and(0.004ml,0.59gm) of phthalic anhydride in 25 ml of ethanol absolute. The reaction mixture was refluxed in water bath at 90°c for 3hr, the solvent was then removed and the resulting solid was recrystallized from ethanol, dried and measured percentage.



2.3.5: preparation of Compound C_5:($C_{20}H_{18}N_4S_2$)

Take in 100 ml round bottom flask equipped was placed a mixture of (0.01 ml, 1.35gm) of phenyl isothiocyanate and (0.005 ml, o.54 gm) of P-phenylenediamie in 25 ml of ethanol absolute. The reaction mixture was refluxed in water bath at 90 ^{o} c for 9hr, the solvent was then removed and the resulting solid was recrystallized from anhydrous and filtered, dried, and measured percentage.



2.4: Biological activity of synthesized compounds

To study the microbiological effects of some prepared compounds correspond to the wells assay, there were two species of bacterial, Escherichia coli (gram negative) and staphylococcus aurous (gram positive).

2.4.1: Wells plate assay

The solution of the prepared compounds in a suitable solvents and were applied to the selected agar medium that has been inoculated with suitable test culture. The antimicrobial agent diffuses in an over-colony circle around the wells of application; the radial growth of the colony was recorded on the completion of incubation and mean diameters of the zone of inhibition were recorded to represent the degree of the antimicrobial agent.

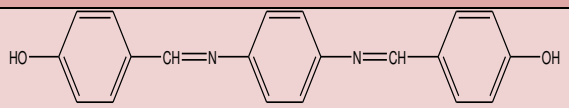
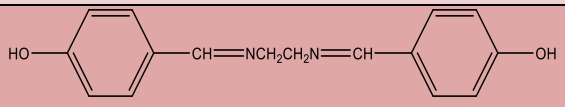
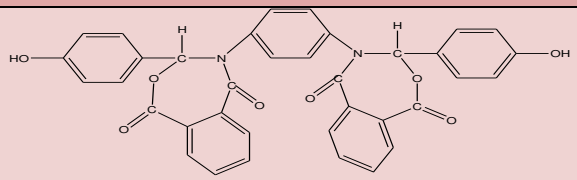
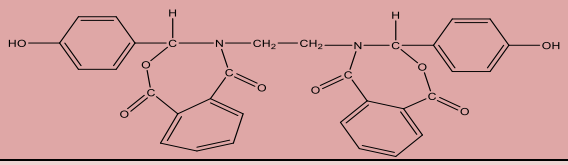
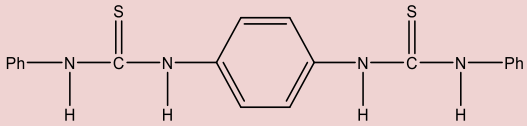
2.4.2: Test of the biological activity

The synthesized compounds [C_1],[C_2],[C_3],[C_4],[C_5] were dissolved in dimethylsulfoxide(DMSO) in two concentration (5000, 4000)ppm and tested against two types of bacteria Escherichia coli and staphylococcus aurous the experiment was conducted by using nutrient agar plates. The plates were incubated at 37^{o} C for 24 hours. The inhibition zones caused by the various compounds were examined.

2.5: Table shows the physical properties for the prepared compounds:

NO.	*M.F*	*M.Wt(gm/ml)*	*M.P(^{o}C)*	*Yield%*	*Colour*
C_1	$C_{20}H_{16}N_2O_2$	*318.148*	*223-225*	*93%*	*Yellow*
C_2	$C_{16}H_{16}N_2O_2$	*269.748*	*253-255*	*94%*	*Dark brown*
C_3	$C_{36}H_{24}N_2O_8$	*612.572*	*249-251*	*14%*	*Straw*
C_4	$C_{30}H_{24}N_2O_8$	*540.512*	*204-206*	*5%*	*Gray*
C_5	$C_{20}H_{18}N_4S_2$	*378.524*	*247-249*	*92%*	*white*

2.6: Table shows the chemical properties and the scientific name for the prepared compounds:

Com. No	*Comp. Structure*	*Comp. Name*
C_1		4,4'-{benzene-1,4-diylbis[nitrilo(*E*)methylylidene]}diphenol
C_2		4,4'-{ethane-1,2-diylbis[nitrilo(*E*)methylylidene]}diphenol
C_3		--
C_4		--
C_5		1,1'-benzene-1,4-diylbis[3-phenyl(thiourea)]

Chapter Three

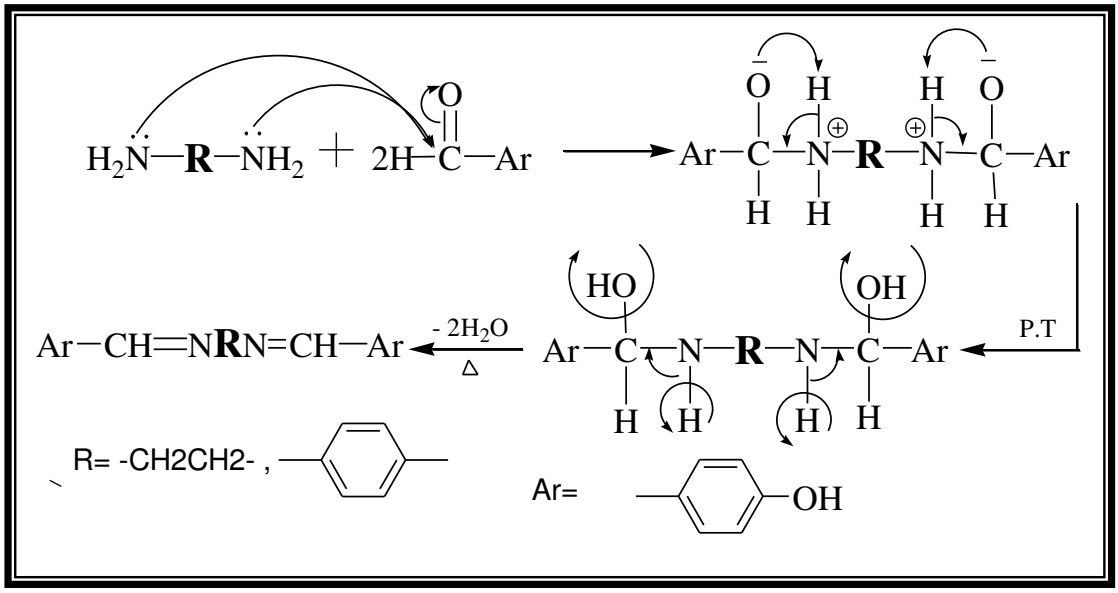
Chapter Three

Result and discution

3.1: Preparation of Schiff's bases compounds [C_1-C_2]

Schiff's bases have been widely reported to be biologically versatile compounds, having antifungal and herbicidal properties .The Schiff's bases [C_1, C_2] have been synthesized by condensation of one mole of 1,4-phenylenediamine or Ethylenediamine with two mole of p-hydroxybenzaldehydes in absolute ethanol as a solvent and few drops of glacial acetic acid (GAA).

The mechanism of the reaction may be outlined as follows :



Scheme 1

The reaction proceeds via nucleophilic attack of the amine on the carbonyl carbon of the aldehyde with the loss of a water

molecule. The structure of the synthesized compounds has been characterized and identified by FTIR spectrum.

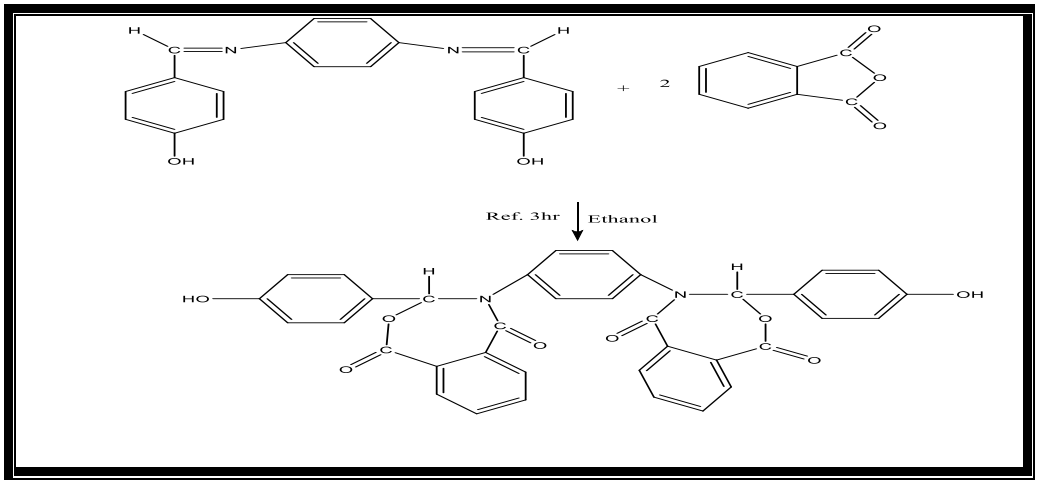
The FTIR spectrum (Fig.3) of compound [C_1] shows an absorption band at (3090 cm^{-1}) due to (C-H) aromatic stretching vibration, (2881cm^{-1}) due to stretching vibration of (C-H) aliphatic , (1599 cm^{-1}) due to stretching vibration of (C=N), and (1515-1443 cm^{-1})due to stretching vibration of (C=C) aromatic.

The ^{1}H-NMR spectrum of compound [C_1], (Fig. 1), shows the following data : 6.7-7.8(dd, 8H, Ar-H), 8.46(S,2H, N=CH) and 10.02(S,2H,OH).

The FTIR spectrum (Fig.4) of compound [C_2] shows an absorption band at (3009 cm^{-1}) due to (C-H) aromatic stretching vibration, (2919 cm^{-1}) due to stretching vibration of (C-H) aliphatic , (1639 cm^{-1}) due to stretching vibration of (C=N), and (1515-1607 cm^{-1})due to stretching vibration of (C=C) aromatic.

3.2: Preparation of compounds [C_3-C_4]

The compounds [C_3, C_4] have been synthesized by condensation of one mole of Schiff's bases [C_1, C_2] with two mole of phthalic anhydride in absolute ethanol as a solvent.



Scheme 2

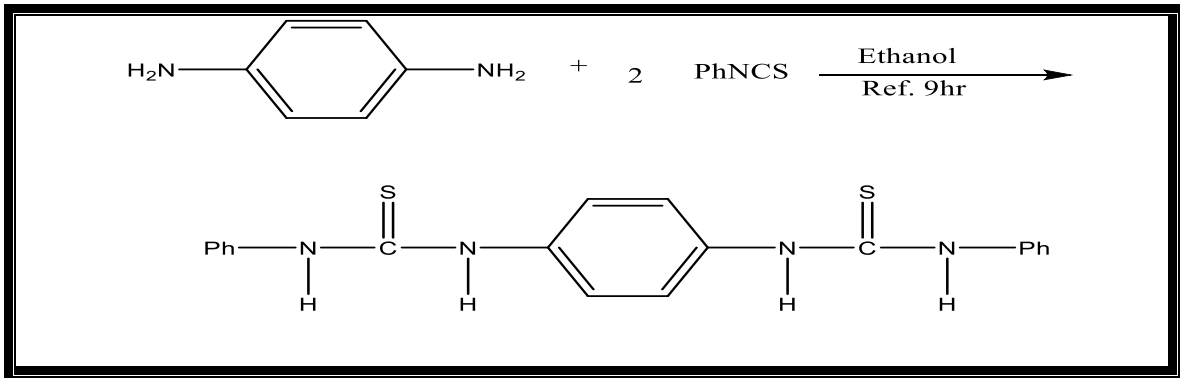
The FTIR spectrum (Fig.5) of compound [C_3] shows an absorption band at (3002 cm^{-1}) due to (C-H) aromatic stretching vibration, (2878 cm^{-1}) due to stretching vibration of (C-H) aliphatic , (1583-1595 cm^{-1})due to stretching vibration of (C=C) aromatic , (1657 cm^{-1}) due to stretching vibration of (C=O) amide ,(1698 cm^{-1}) due to stretching vibration of (C=O) lactone , (3321 cm^{-1}) due to stretching vibration of (O -H) ,(1320-1291 cm^{-1}) due to stretching vibration of (C-O).

The FTIR spectrum (Fig.6) of compound [C_4] shows an absorption band at (3002 cm^{-1}) due to (C-H) aromatic stretching vibration, (2878 cm^{-1}) due to stretching vibration of (C-H) aliphatic , (1562-1474 cm^{-1})due to stretching vibration of (C=C) aromatic , (1630 cm^{-1}) due to stretching vibration of (C=O)amide , (1689 cm^{-1}) due to stretching vibration of (C=O) lactone, (1689 cm^{-1}) due to

stretching vibration of (O -H) ,(1323 cm^{-1}) due to stretching vibration of (C -O).

3.3: Preparation of compounds [C_5]

The compound [C_5] have been synthesized by condensation of one mole of 1,4-phenylenediamine with two mole of phenylisothiacyanato in absolute ethanol as a solvent .



Scheme 3

The FTIR spectrum (Fig.7) of compound [C_5] shows an absorption band at (3032 cm^{-1}) due to (C-H) aromatic stretching vibration, (2801 cm^{-1}) due to stretching vibration of (C-H) aliphatic , (1595 cm^{-1}) due to stretching vibration of (C=N), and (1548-1450 cm^{-1})due to stretching vibration of (C=C) aromatic , (1630 cm^{-1}) due to stretching vibration of (C=O)amide , (1689 cm^{-1}) due to stretching

vibration of (C=O) lactone, (1689 cm^{-1}) due to stretching vibration of (O -H) ,(1323 cm^{-1}) due to stretching vibration of (C - O).

3.4: Biological activity:

Microorganism causes different kind of diseases to humans and animals. Discovery of chemotherapeutic agents played a very important role in controlling and preventing such diseases.

Chemotherapeutic agents are isolated either from living organism known as antibiotics or they are chemical compounds prepared by chemist such as the sulfa drugs etc.

The use of organic compounds with active known antimicrobial properties, can be of great significance in therapeutic treatments.

The most essential feature of good chemotherapeutic agent is that, it must show a high degree of selective toxicity towards a microorganism, so that, it can be given in sufficient doses to inhibit or kill the microorganism throughout the body without harming the body cell. Heterocyclic rings constitute an important class of compounds having a wide spectrum of biological activity.

The Biological activity of compound (C1,C3,C4) aginst (E-Coli)

Figures

Referenses

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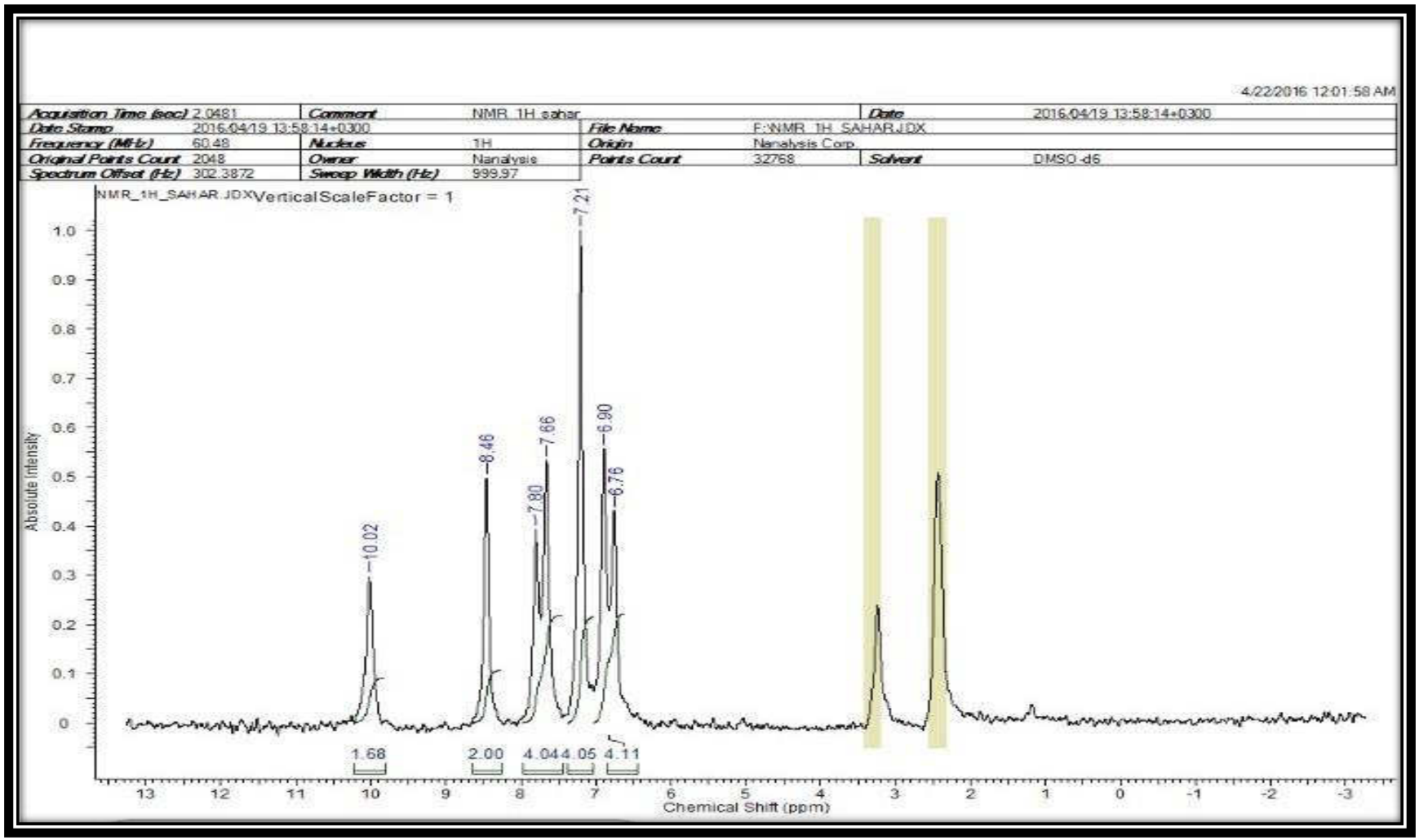

Fig.(1): ^{1}H NMR Spectrum of Compound (C_1)

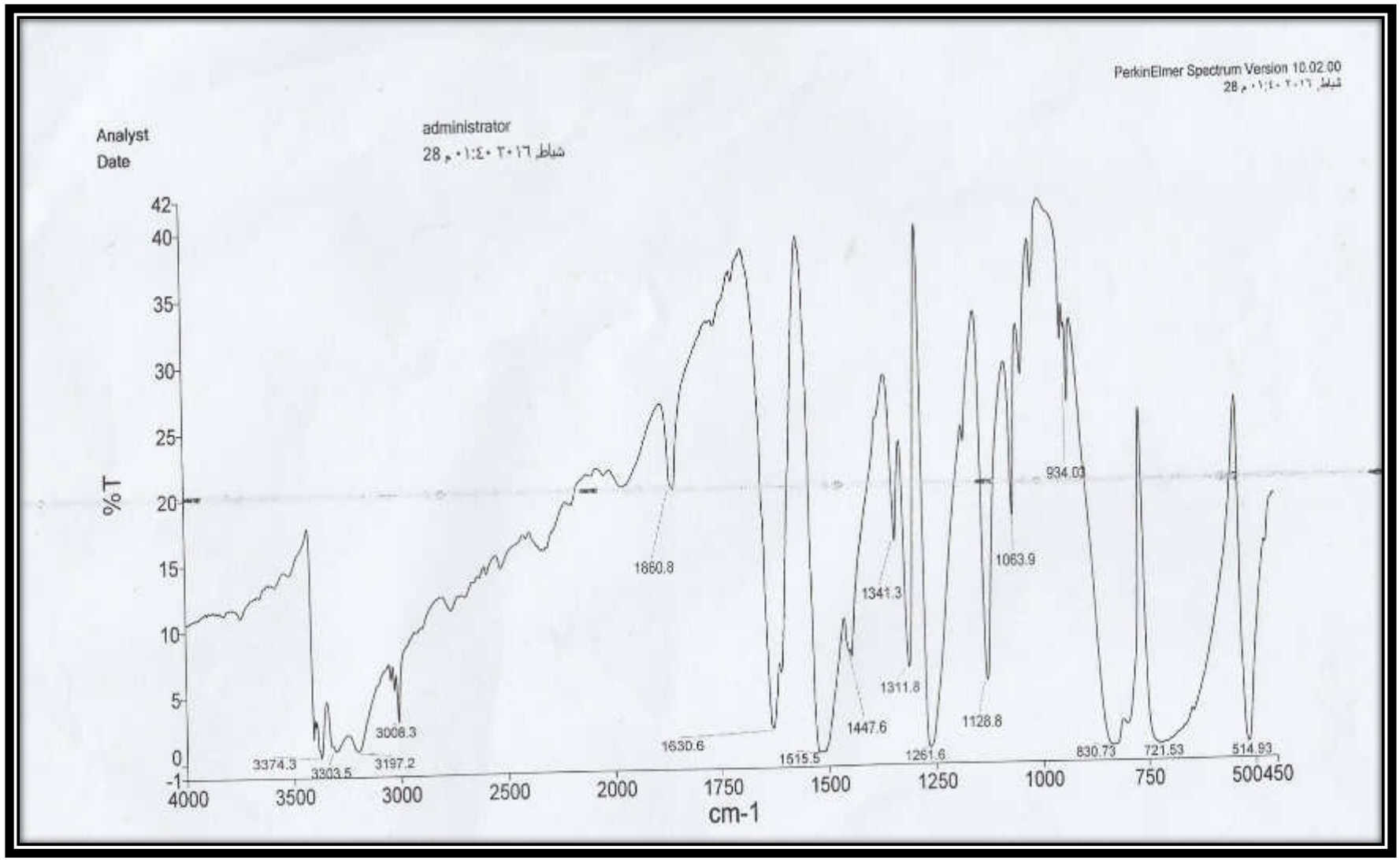

Fig.(2): FTIR Spectrum of 1,4-phenylenediamine Compound

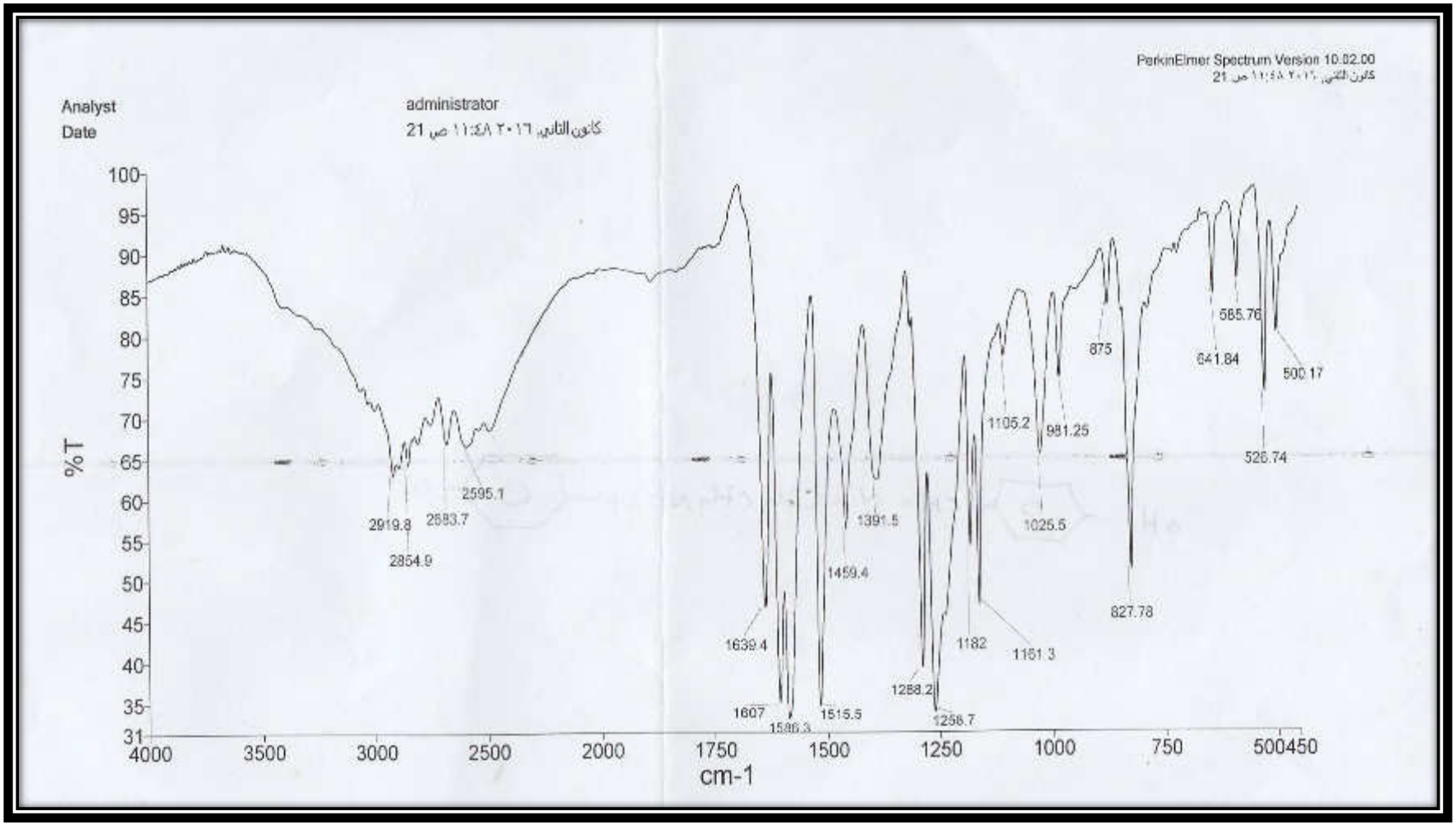

Fig.(3): FTIR Spectrum of Compound (C_1)

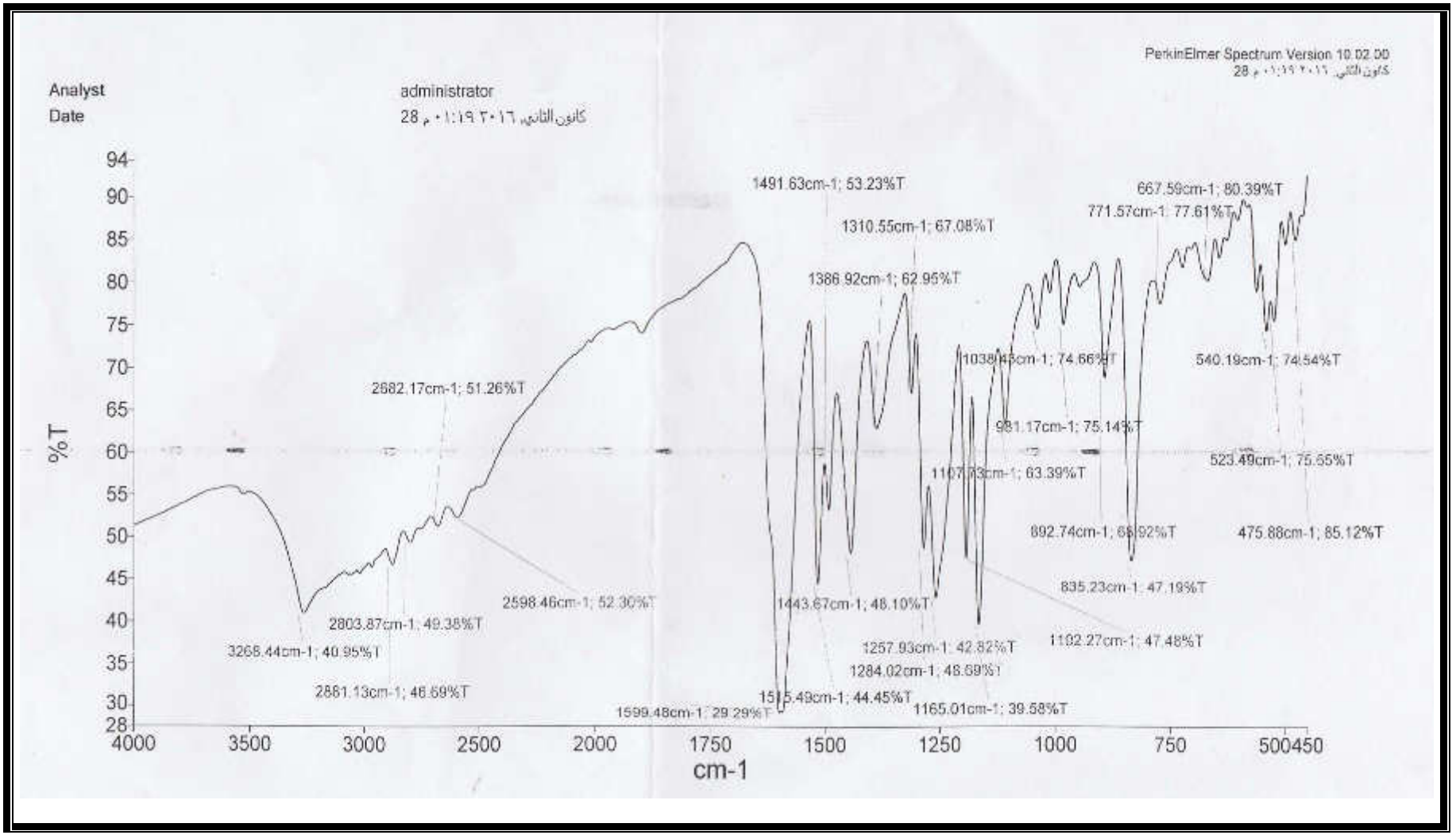

Fig.(4): FTIR Spectrum of Compound $(C)_2$

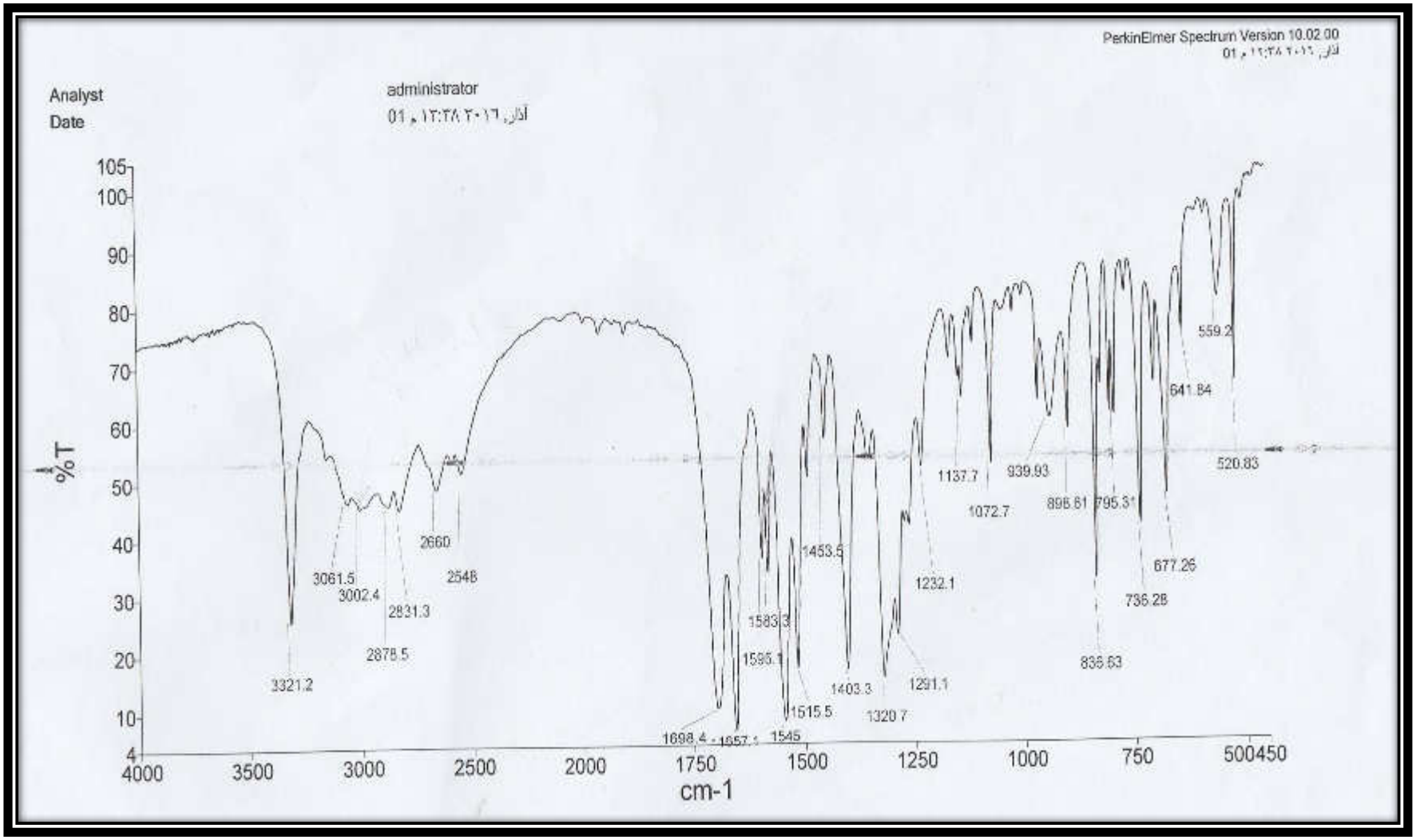

Fig.(5): FTIR Spectrum of Compound (C_3)

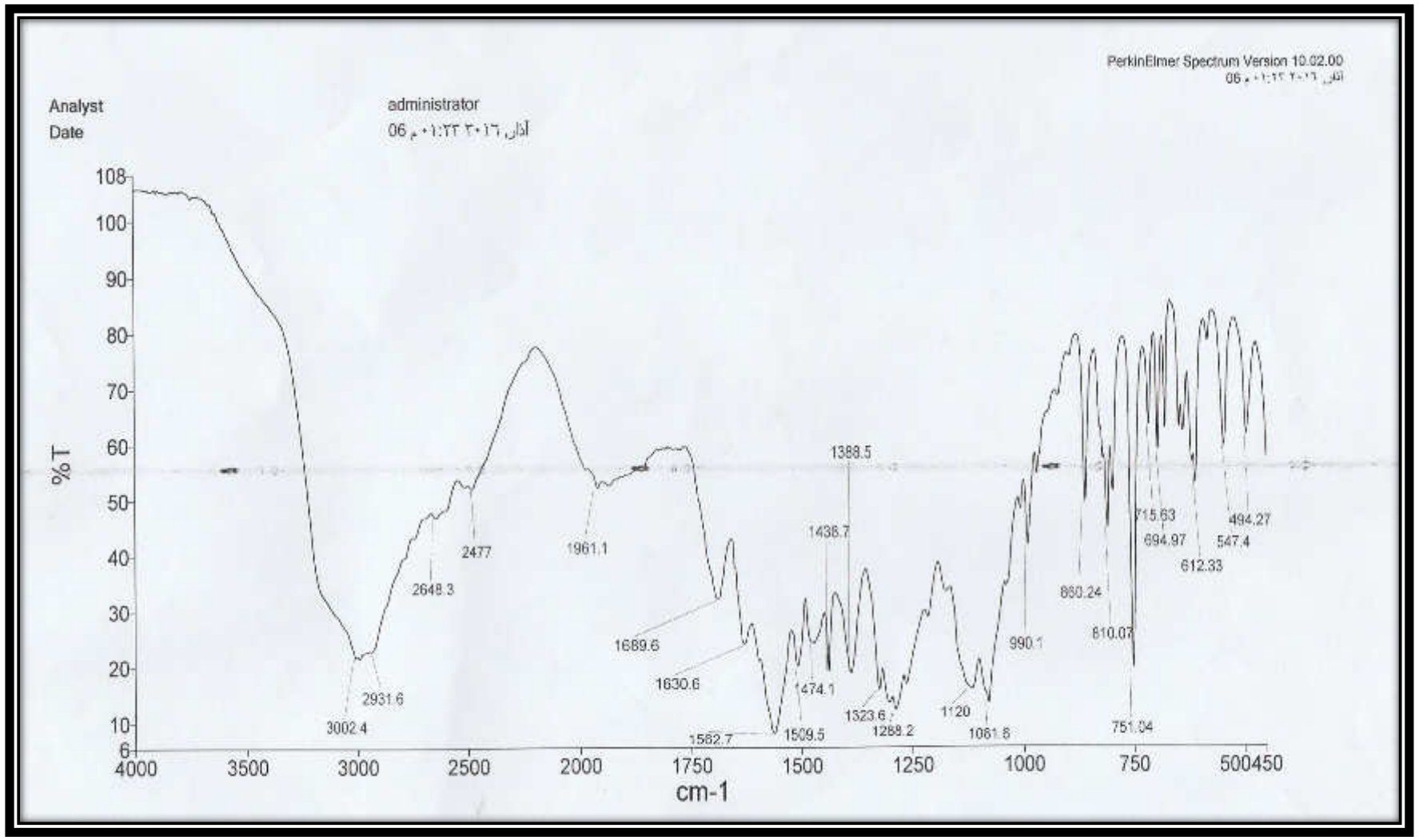

Fig.(6): FTIR Spectrum of Compound (C_4)

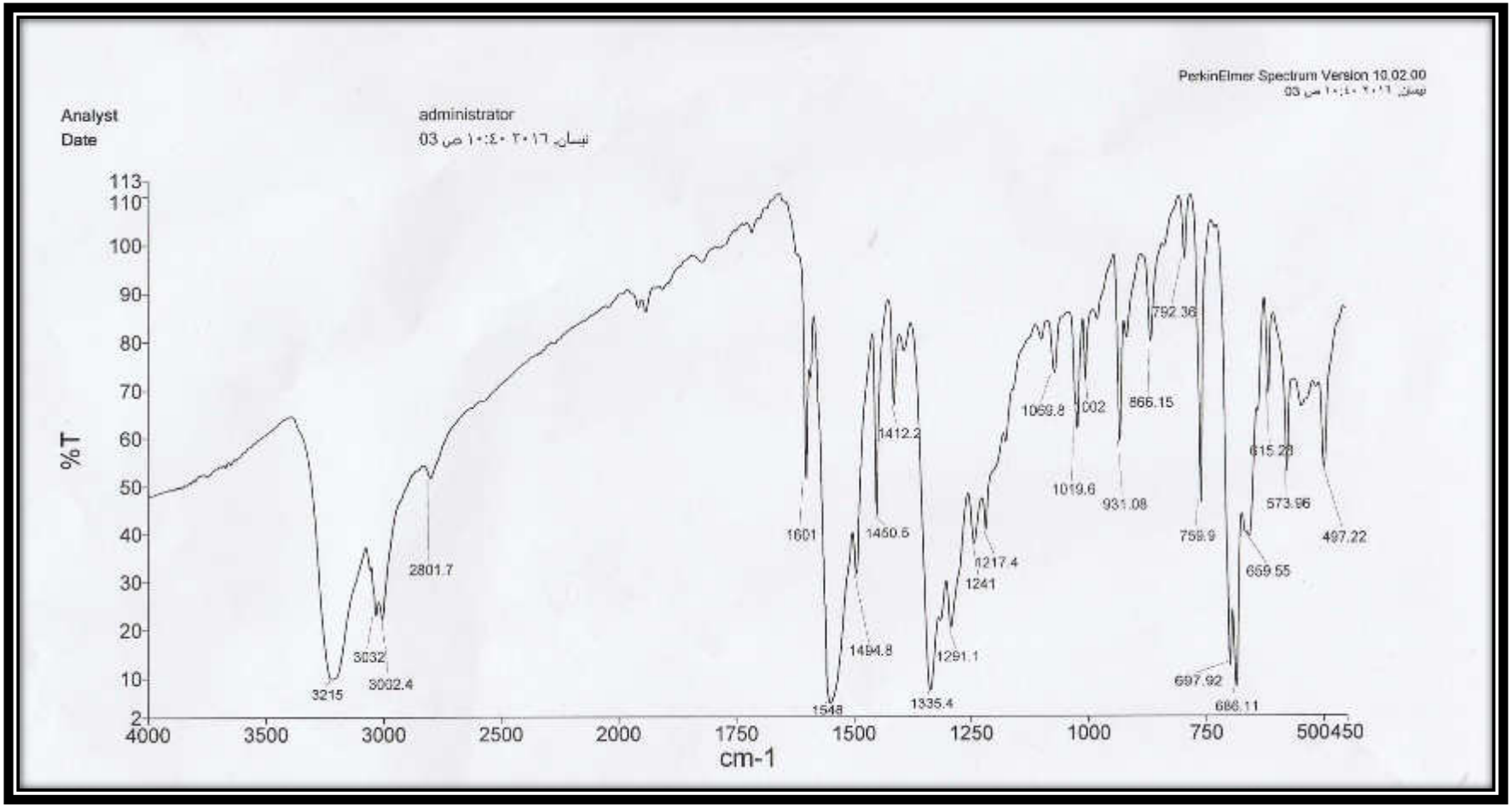

Fig.(7): FTIR Spectrum of Compound (C_5)