ISSN: 0975-8585

Research Journal of Pharmaceutical, Biological and Chemical Sciences

(Synthetic, Spectral, Chemo, Thermo) Studying of New Oxadiazole Derivatives.

Mohammed Alaa Abdulzahra*.

Chemistry Department, College of Education for Pure sciences., university of kerbala, Iraq.

ABSTRACT

Many studying carried out on new oxadiazole derivatives in this paper such as organic , spectral , thermal ,studying .Oxadiazole derivatives have been prepared by reaction between diamine aromatic compound with aldehyde derivative and oxadizole derivative to yield five compounds from 4,4-diamino phenyl benzene. The structure of the newly prepared compounds (five compounds) were characterized by (TLC) and many techniques((UV-Spectral ,FT.IR ,^{1}H.NMR ,DSC-Measurement)),melting points then studying of (thermal measurements for stability of compounds , studying of physical characterization)

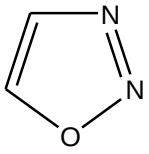
Keywords: arom , oxadizole, di amine, carbonyl .

Corresponding author

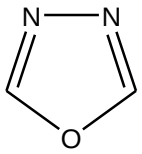
ISSN: 0975-8585

INTRODUCTION

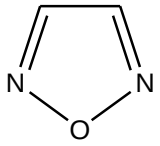
Oxadiazole ring is a heterocyclic aromatic compound ,its formula ($C_2H_2N_2O$), has five membered ring consisting of two nitrogen atoms (2 N) with (2 C)carbon atoms, and (1 oxygen atom) .,it have four isomers of its structure ,shown in the figures[1-3]:



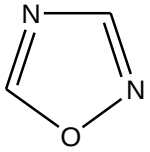
1,2,3-oxadiazole
(Diazo-oxides)



1,3,4-oxadiazole
(Biazole)



1,2,5-oxadiazole



1,2,4-oxadiazole
(Azoxime)

Oxadiazole ring has potential bioactive properties. The oxadiazole compounds have been found to exhibit diverse pharmaceutical activities represented as antimicrobial, anti-HIV , anti tubercular ,anti malarial ,analgesic ,anti-inflammatory , anticonvulsant , hypoglycemic and other biological properties represented as lipid peroxidation inhibitor[4-8] .

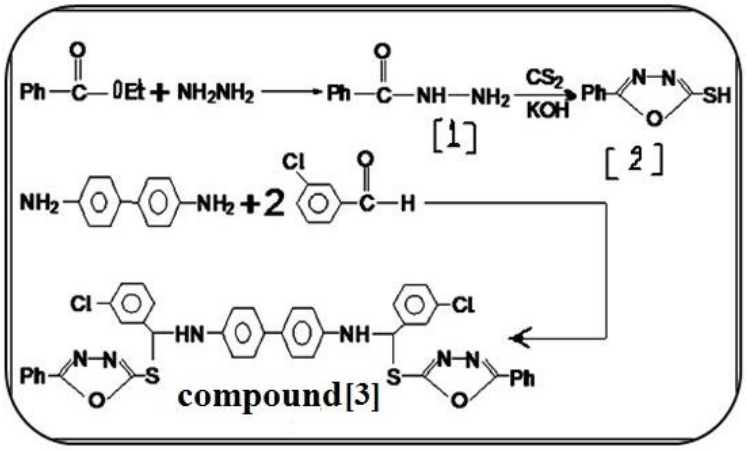
A large number of oxadiazole derivatives have been prepared and many of these compounds have shown a wide spectrum of antimicrobial activity. The observation that some oxadiazoles with different substituents have antimicrobial activity at various position on the heterocyclic ring resulted in antibacterial agents of several potencies. Large numbers from antimicrobial agents available for treatment of microbial infections, emergence of many drugs resistant organism has posed a great challenge to the scientists[9-15]**.**

Experimental Part:

All measurement were carried out by : **Uv-Visible –**Spectra ,**FT.IR**- spectra ,KBr –disc ,shimadzu 8300 .,**^{1}H.NMR** –spectra in DMSO–solvent in Canada for all compounds .,**Thermal analysis** (**DSC- Analysis**) in Canada for most compounds.

Preparation of Compounds [1 – 3] :

Ethyl benzoate (0.05 mole) and (0.05 mole) of hydrazine were reacted with refluxing for (4 hrs) , the resulting is compound [1] (0.04 mole) reacted with carbon disulfide and solution of potassium hydroxide in ethanol ,after (20 hrs) ,the precipitate dried to give compound [2] , which (0.08 mole) heated with (0.04 mole) of 4,4-diamino phenyl benzene with (0.08 mole) of meta- chloro benzaldehyde in presence of absolute ethanol (8hrs) to produce (80%) of compound [3] according to procedure [3, 4] .



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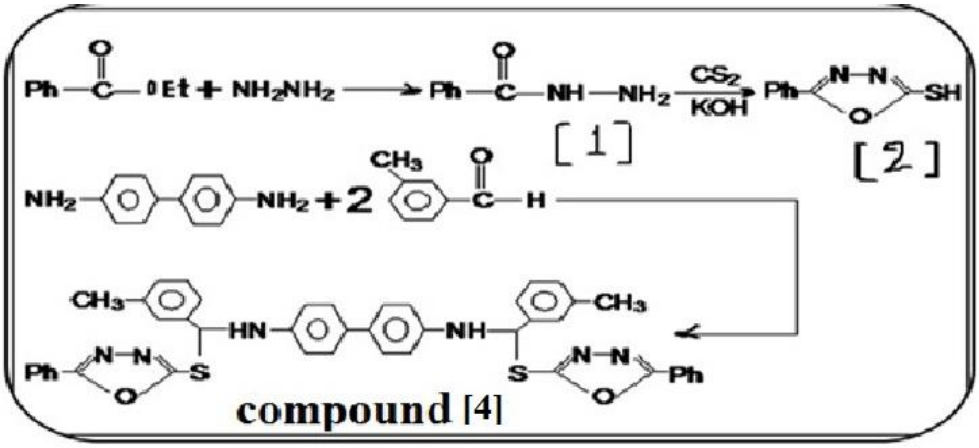
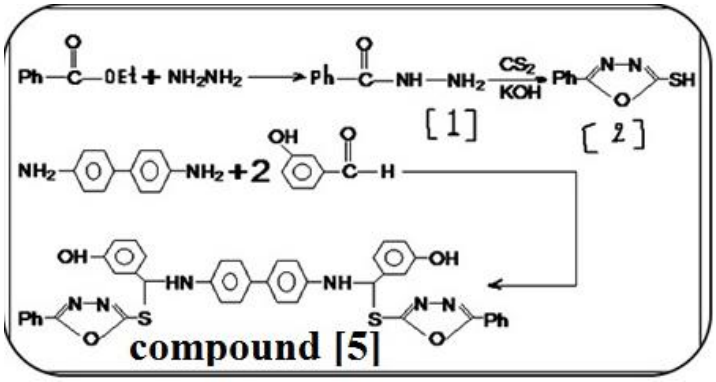
Preparation of Compound [4] :

(0.08 mole) Of compound [2] heated with (0.04 mole) of 4,4-diamino phenyl benzene with (0.08 mole) of meta- methyl benzaldehyde in presence of absolute ethanol (7hrs) to produce (84%) of compound [4] according to procedure[4, 9]., after that filtered and dried , then re crystallization from absolute ethanol.



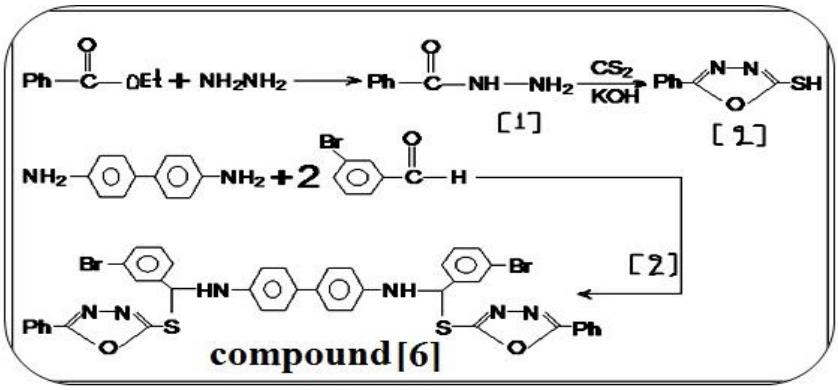
Preparation of Compound [5] :

(0.08 mole) Of compound [2] heated with (0.04 mole) of 4,4-diamino phenyl benzene with (0.08 mole) of meta- hydroxy benzaldehyde in presence of absolut ethanol (9 hrs) to produce (82%) of compound [5] according to procedure[4,10]., after that filtered and dried , then re crystallization from absolute ethanol.



Preparation of Compound [6] :

(0.08 mole) Of compound [2] heated with (0.04 mole) of 4,4-diamino phenyl benzene with (0.08 mole) of meta-bromo benzaldehyde in presence of absolut ethanol (8hrs) to produce (78%) of compound [6] according to procedure[4, 9].,after that filtered and dried ,then re crystallization from absolute ethanol.



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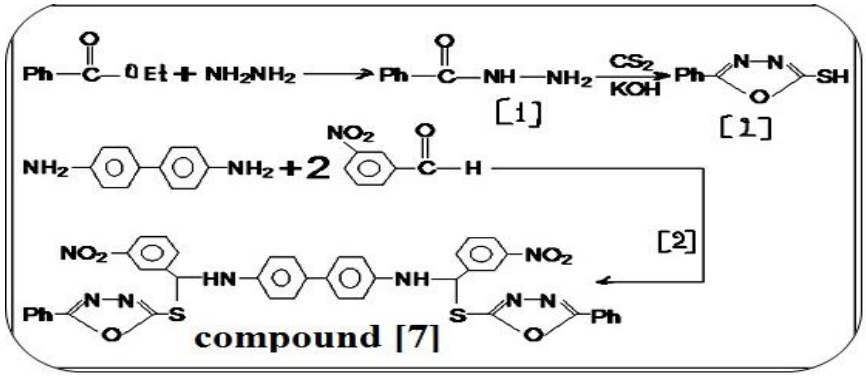
Preparation of Compound [7] :

(0.08 mole) Of compound [2] heated with (0.04 mole) of 4,4-diamino phenyl benzene with (0.08 mole) of meta- nitro benzaldehyde in presence of absolut ethanol (11hrs) to produce (80%) of compound [7] according to procedure[4, 10]., after that filtered and dried , then re crystallization from absolute ethanol.



RESULTS AND DISCUSSION

The formatted compounds from oxadiazole derivatives in this work prepared by reaction between di amine compound with aldehydes derivatives with phenyl mercapto oxadiazole . several studying carried out in many techniques (spectral methods , thermal , Analytical , physical measurements)

UV-Visible – Scanning and Physical properties :

All prepared compounds [1-7] in this work were scanned for maximum wave length by electronic spectroscopy technique in absolute ethanol as a solvent for salvation of compounds., all data of (spectrum , products %) shown in Table (1) and some figures (1- 3).

Table (1) : λmax and products % of Compounds .

Compounds	λmax (nm)	Product %
Compound [1]	260	80
Compound [2]	292	82
Compound [3]	376	80
Compound [4]	398	84
Compound [5]	430	82
Compound [6]	408	78
Compound [7]	400	80

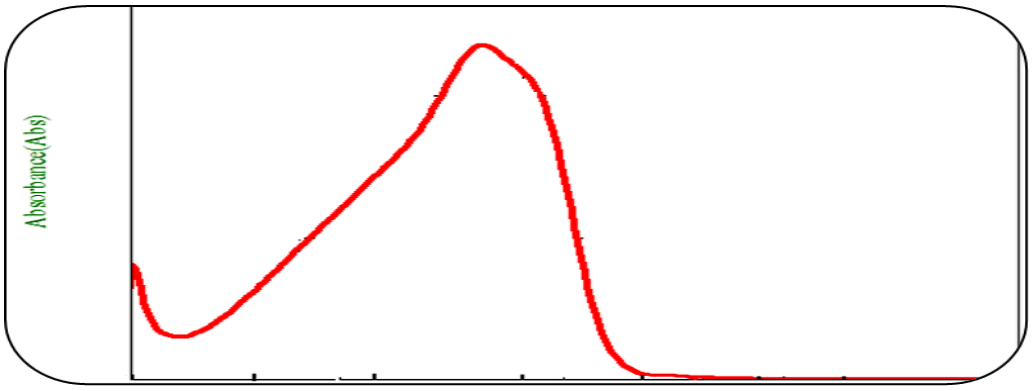


Fig (1) : UV-Visible of Compound [5]

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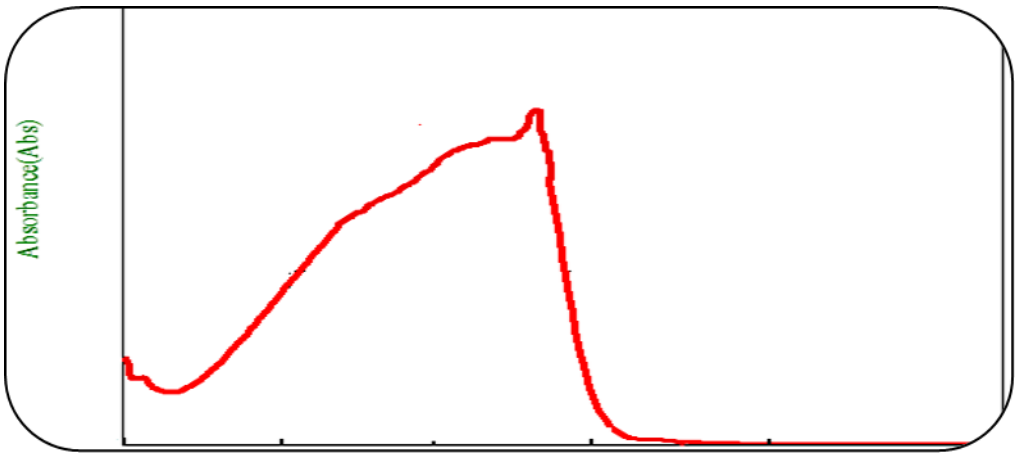

Fig (2) : UV-Visible of Compound [6]

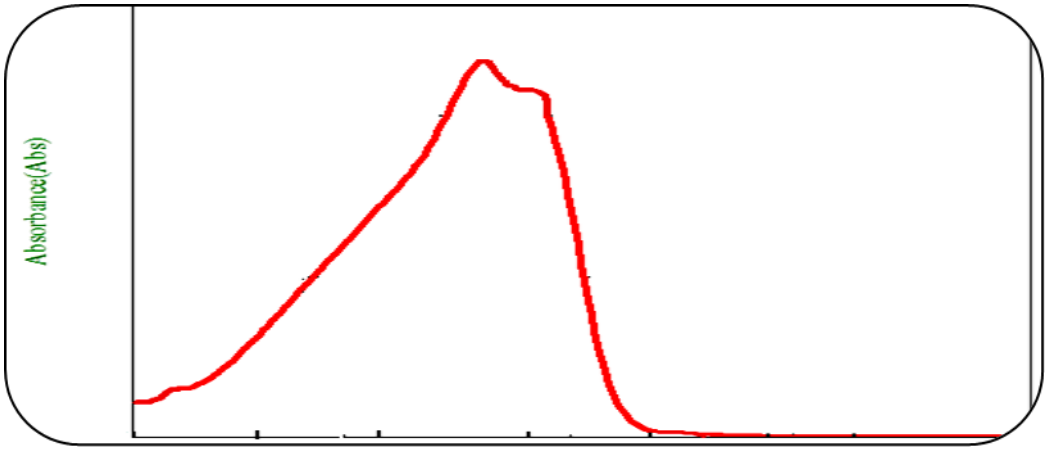

Fig (3) : UV-Visible of Compound [7]

I.R–spectra : showed frequency at [(1692)cm^{-1} due to (-CO-N-) amide , (3412, 3378)cm^{-1} due to (NH_2-) amine group] in compound [1] , which disappeared and other frequencies appeared in formatted compounds such as [(1631 - 1651)cm^{-1} due to (C=N) endocycle and (3300 - 3340)cm^{-1} due to (NH-) in compounds [3 ,4, 5 , 6, 7] ., band at (787)cm^{-1} to (C-Cl)] in compound [3]., frequency (CH) aliphatic : (2924) in compound [4]., frequency at (3441)cm^{-1} to ,(OH) hydroxyl group in compound [5]., frequency at (692)cm^{-1} to (C-Br) in compound [6] ., frequency at (1312 , 1506)cm^{-1} to (NO_2) in compound [7] and other results of functional groups in Table (2) and some of figures (4-8).

Table (2): I.R Spectra (cm^{-1}) of Compounds .

Compounds	Only Important Groups
Compound [1]	(CO-NH)carbonyl of amide:1676 , (NH_2): (3412 , 3378) , (NH) of amid : 3110 .
Compound [2]	(C=N)endocycle :1645 , (SH): 2432 .
Compound [3]	(NH) :3340 , (C=N)endocycle :1655 , (C-Cl) : 787 .
Compound [4]	(NH) :3310 ,(C=N)endocycle :1632 ,(CH) aliphatic :2924 .
Compound [5]	(NH) :3314 ,(C=N)endocycle :1647 ,(OH) hydroxyl group:3441 .
Compound [6]	(NH) :3300 ,(C=N)endocycle :1631 ,(C-Br): 692 .
Compound [7]	(NH) :3313 ,(C=N)endocycle :1651 ,(NO_2)group: (1312, 1506) .

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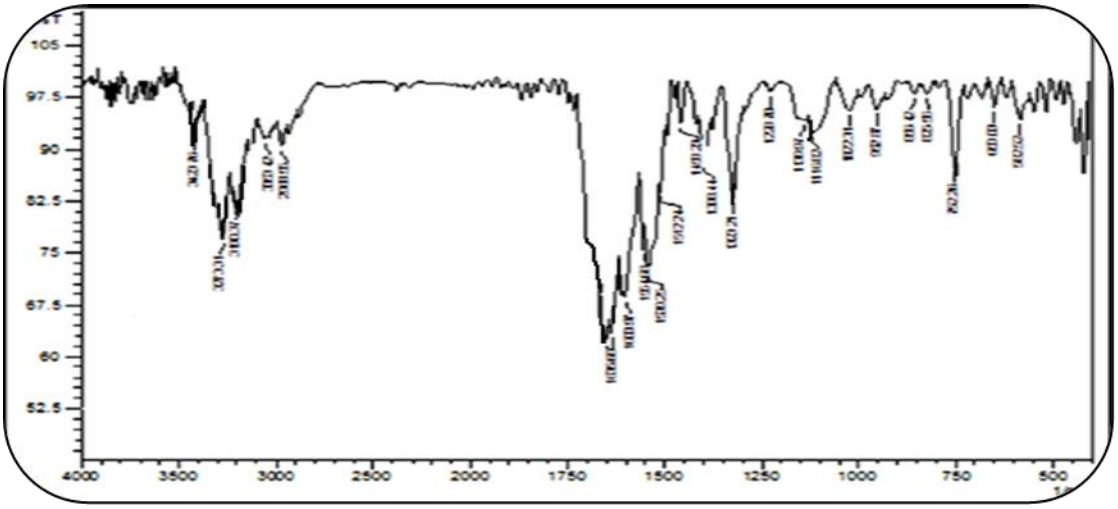

Fig (4): I.R spectra of Compound [1]

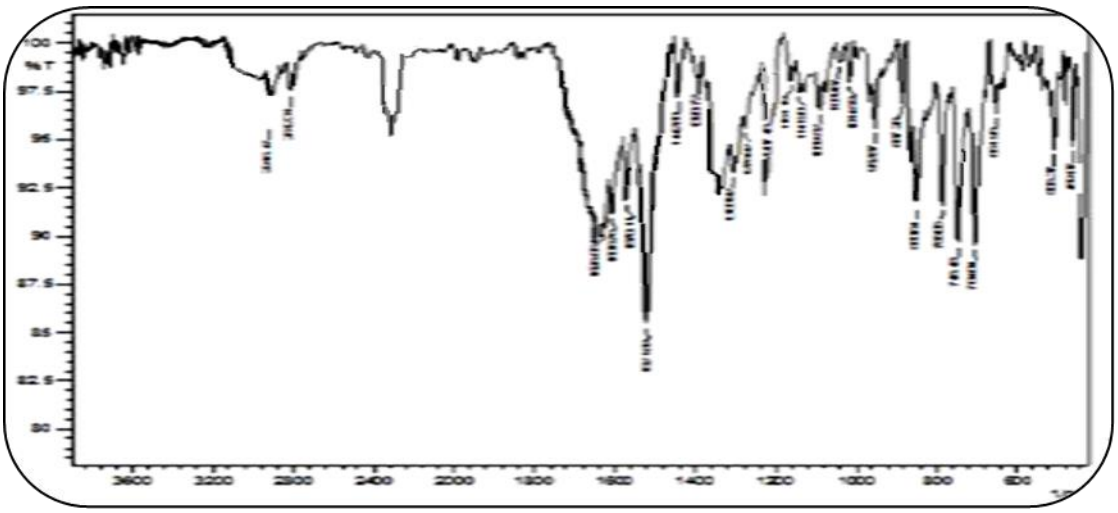

Fig (5): I.R spectra of Compound [2]

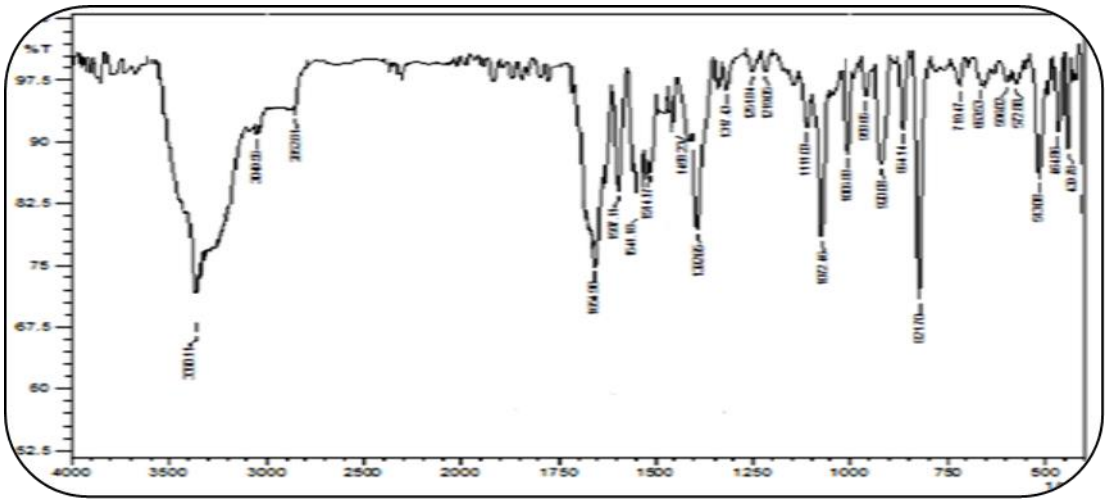

Fig (6): I.R spectra of Compound [3]

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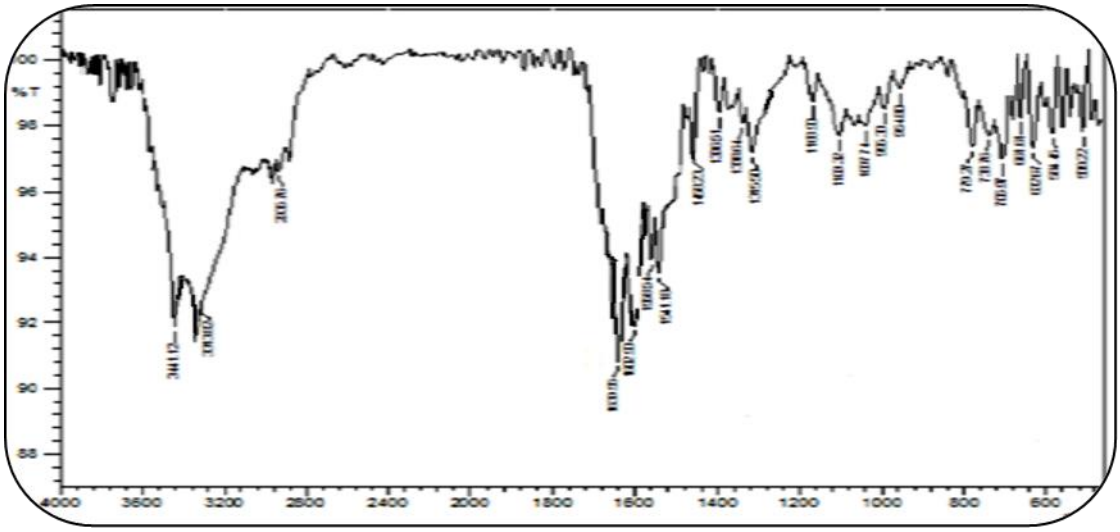

Fig (7): I.R spectra of Compound [5]

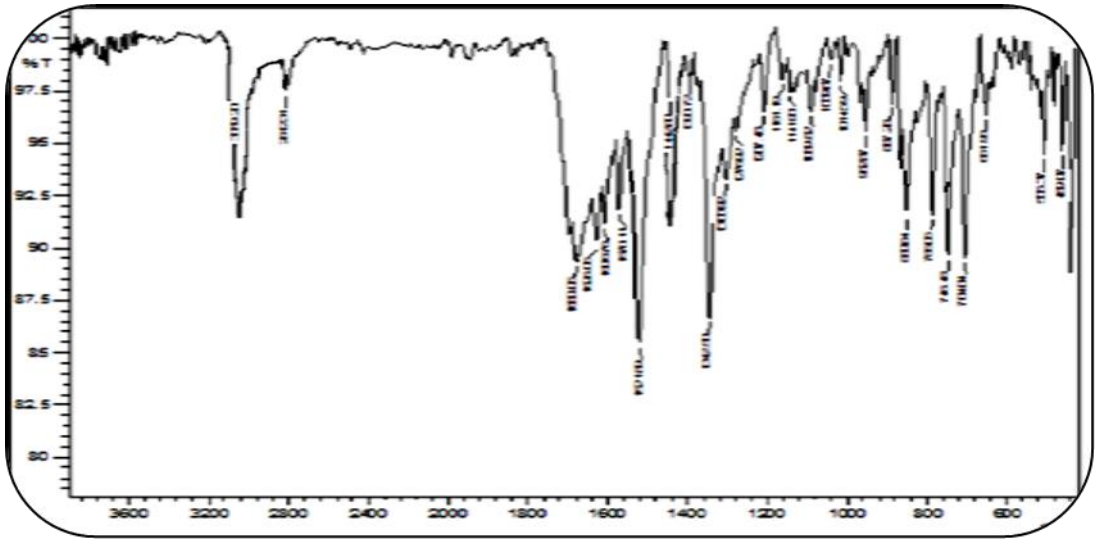

Fig (8): I.R spectra of Compound [6]

^{1}H.NMR – spectra : resonance spectra appeared signals for all functional groups in each formatted compounds ., shown peaks at Б 5.47 (NH_2) amine group , 9.06 (NH-CO) amide in compound [1]., while compound [2] appeared peaks at 11.10 (SH) thiol group . Signals at 3.05 (S-CH) , 5.35 (NH) amine group in compound [3] , peaks at 3.00 (S-CH) , 5.22 (NH) amine group , 0.97(CH_3) in compound [4] . Peaks at 3.28 (S-CH) , 5.36 (NH) amine group , 10.76 (OH) phenol in compound [5] ., but in compound [6] appeared signals at 3.28 (S-CH) , 5.08 (NH) amine group , and in compound [7] at 3.00 (S-CH) , 5.21 (NH) amine group .,other data in Table (3) and figures (9-13) .

Table (3):^{1}H.NMR Spectra in (δ ppm) of Compounds .

Compounds	Important peaks
Compound [1]	5.47 (NH_2) amine group , 9.06 (NH-CO) amide .
Compound [2]	11.10 (SH) thiol group.
Compound [3]	3.05 (S-CH) , 5.35 (NH) amine group.
Compound [4]	3.00 (S-CH) , 5.22 (NH) amine group , 0.97(CH_3).
Compound [5]	3.28 (S-CH) , 5.36 (NH) amine group , 10.76 (OH) phenol.
Compound [6]	3.28 (S-CH) , 5.08 (NH) amine group.
Compound [7]	3.00 (S-CH) , 5.21 (NH) amine group.

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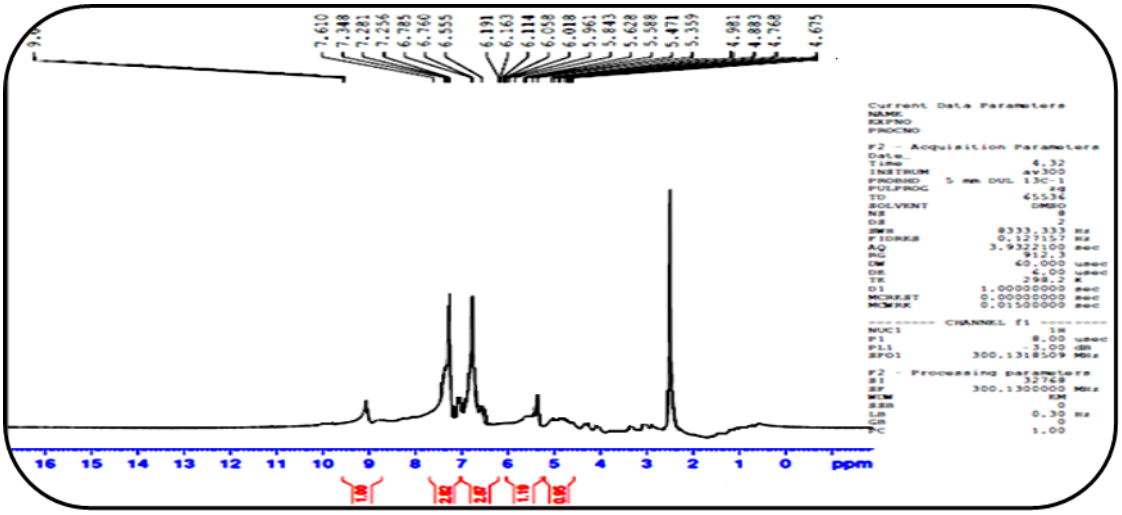

Fig (9): ^{1}H.NMR spectra of Compound [1]

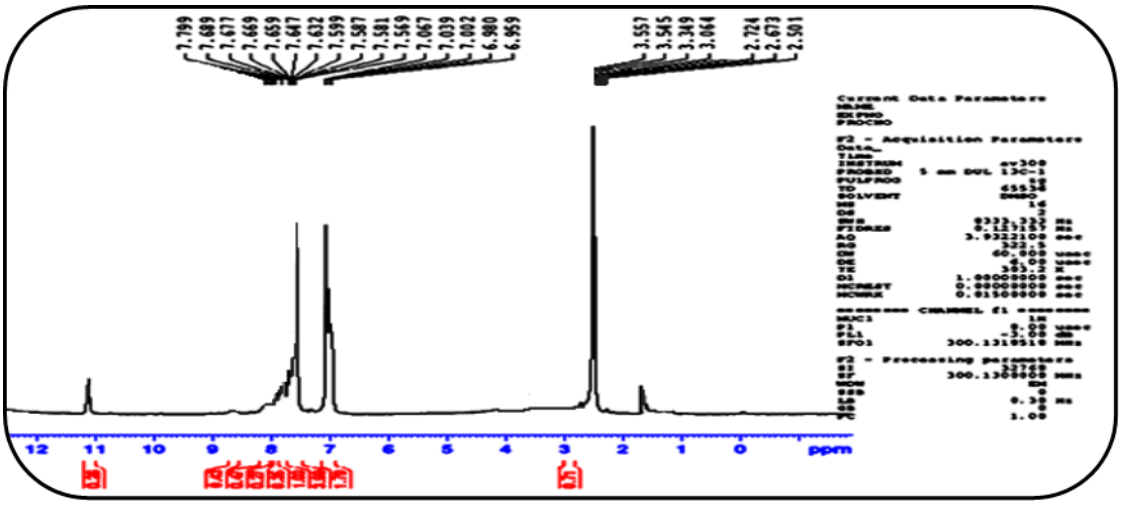

Fig (10): ^{1}H.NMR spectra of Compound [2]

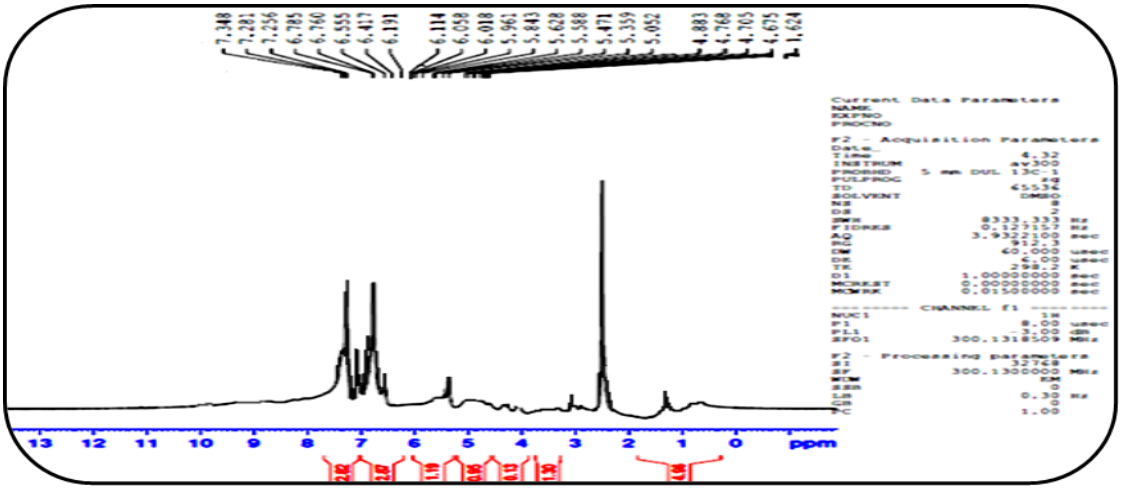

Fig (11): ^{1}H.NMR spectra of Compound [3]

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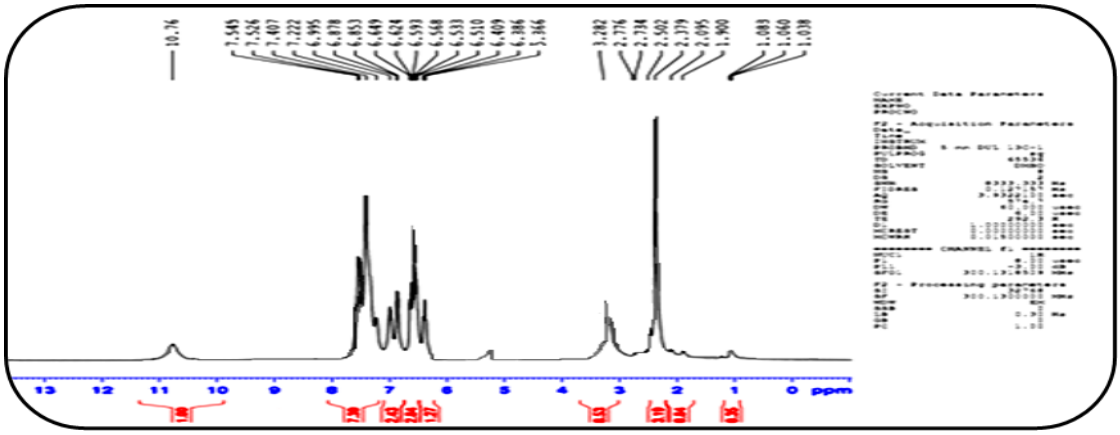

Fig (12): ^{1}H.NMR spectra of Compound [5]

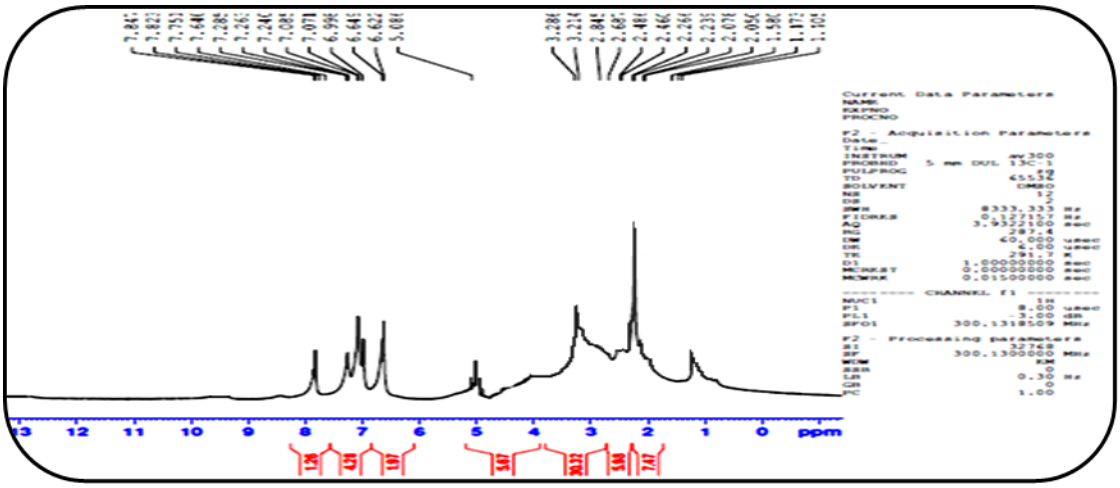

Fig (13): ^{1}H.NMR spectra of Compound [6]

Thermal Studying (DSC- Curves) :

DSC–Thermal analysis carried out for most compounds according to procedures of studying[14], all results in figures(14-18) ,DSC- measurements of selected compounds appeared high stability toward high temperature in most of analysis :

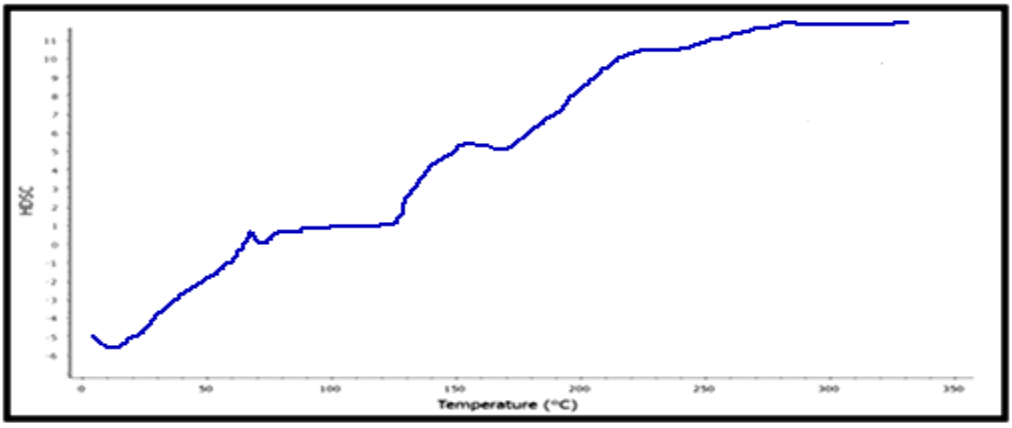

Fig (14) : DSC of Compound [3]

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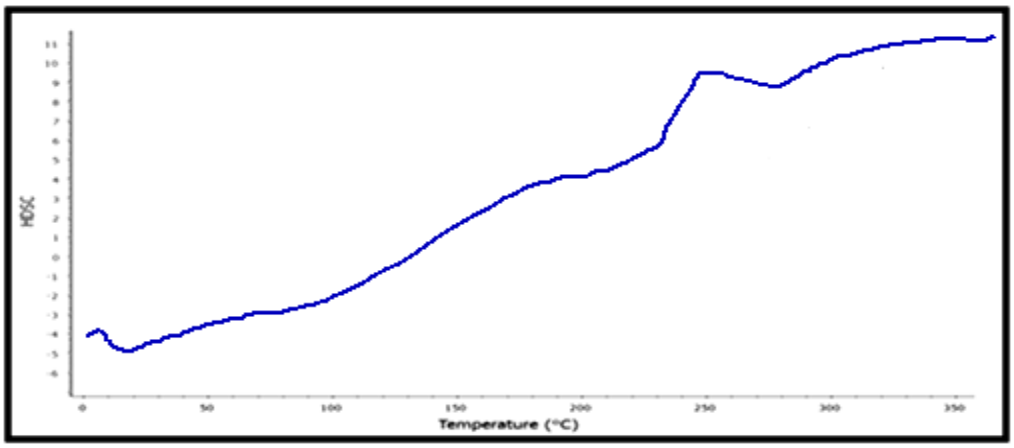

Fig (15) : DSC of Compound [4]

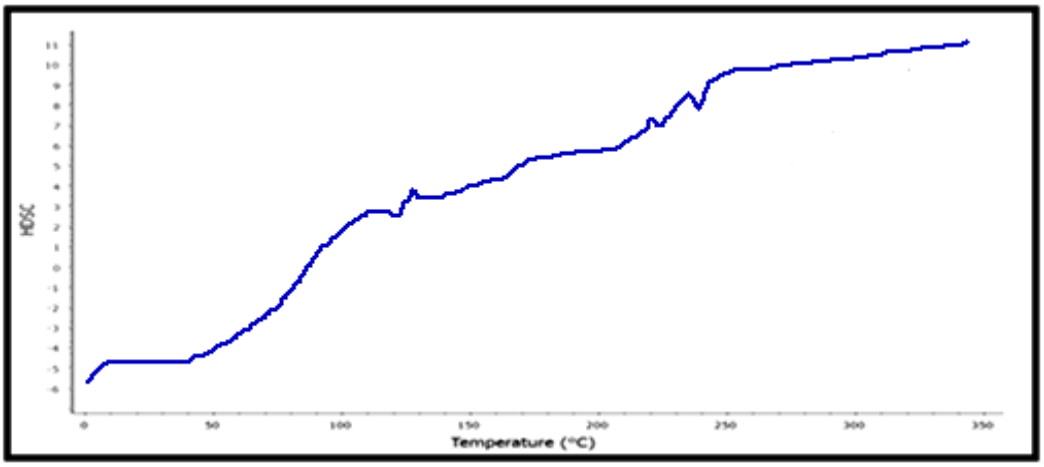

Fig (16) : DSC of Compound [5]

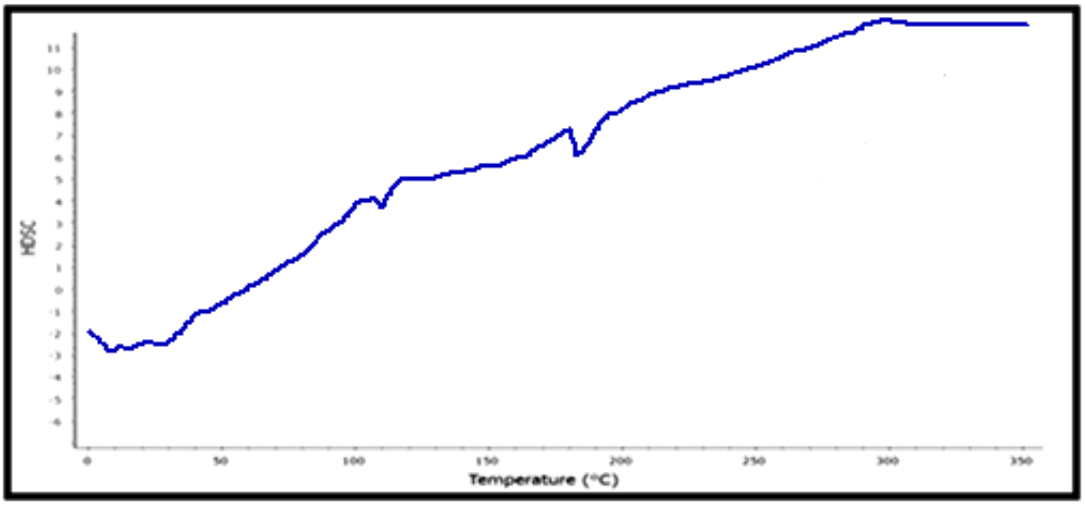

Fig (17) : DSC of Compound [6]

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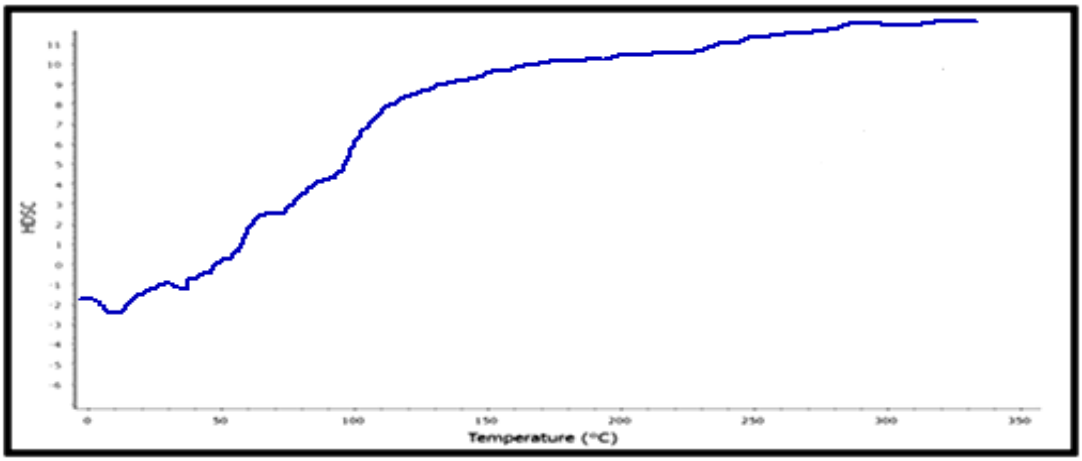

Fig (18) : DSC of Compound [7]

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