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RESEARCH ARTICLE

Synthesis of New Organic Compounds Via Three Components Reaction with Studying of (Identification ,Thermal Behavior, Bioactivity on Bacteria of Teeth)

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Abstract

All compounds in this work prepared via mannich reaction through various starting materials like (furan , thiophene , benzothiazole derivatives) with functional groups in terminals of compounds like (hydroxyl group , carboxyl group) ., our method involved reaction between diamine compounds with aromatic aldehyde. then identification of synthesized compounds via identification techniques (FT.IR , H.NMR , Mass) – spectrophotometric , studying of thermal behavior , studying of bioactivity behavior for our compounds toward types of bacteria (bacteria of Mouth like (Streptococcus Mutans , Streptococcus Salivarius) .

Keywords: *three components, bacteria, activity.*

Introduction

A small effects were noted with the use of various starting materials and reactants in any reaction promoted the formation of new series organic compounds which involved two or three components in reaction .

Some of the first multi component reactions [1-9] to be reported function through carbonyl compounds into more reactive intermediates, which can react further with a nucleophile like:

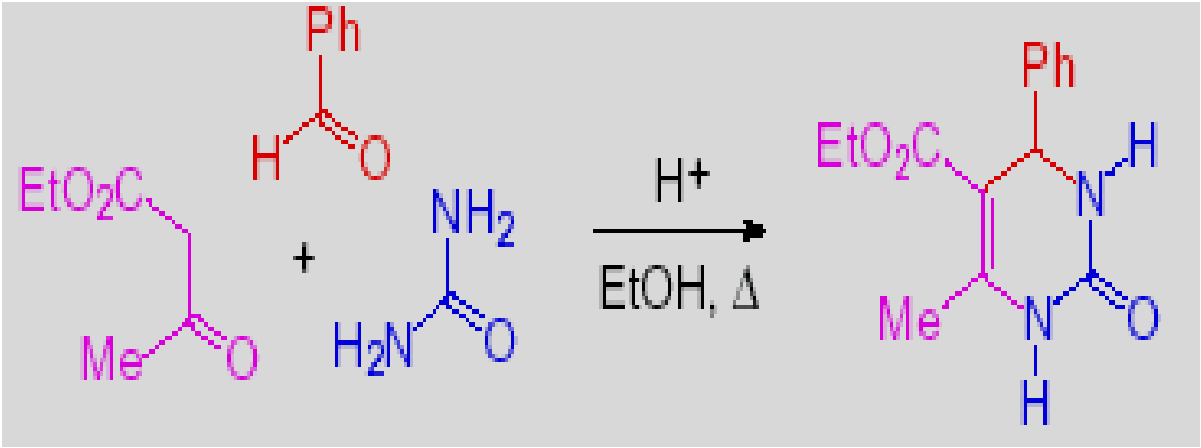


Fig .1: Reaction of Three Components

Organic reactions occur between organic molecules to produce new organic compounds which depend on type of reaction, conditions of reaction, concentrations of reactants and have different physical and chemical properties which have various applications[10-34] and uses .

Experimental Materials

The bio materials supplied from ((Sigma , chemicals company)) ,biological studying carried out in Bio – lab in biological department., Chemical Studying and preparation of concentrations carried out in chemistry department .

Experimental Part

STEP.1: Synthesis of Compound (1)

P-Hydroxy aniline (0.01 mole) reacted with (0.01mole) of ammonium thiocyanate in presence of bromine with glacial acetic acid (drop by drop) and rotation in ice path according to literature[21],to give precipitation which filtered and dried then re crystallized to yield amine compounds , which (0.02 mole) reacted with (0.01 mole) of benzaldehyde to give compound (1).

STEP .2: Synthesis of Compound (2)

P- Amino benzoic acid (0.01 mole) reacted with (0.01mole) of ammonium thiocyanate in presence of bromine with glacial acetic acid (drop by drop) and rotation in ice path according to literature[21] ,to give precipitation which filtered and dried then re crystallized to produce amine compounds ,which (0.02 mole) reacted with (0.01 mole) of benzaldehyde to give compound (2).

STEP .3: Synthesis of Compound (3)

P- Amino benzoic acid (0.01 mole) reacted with (0.01mole) of ammonium thiocyanate in presence of bromine with glacial acetic acid (drop by drop) and rotation in ice path according to literature[21] ,to give precipitation which filtered and dried then re crystallized to yield amine compounds , which (0.02 mole) reacted with (0.01 mole) of furfural to give compound (3).

STEP .4: Synthesis of Compound (4)

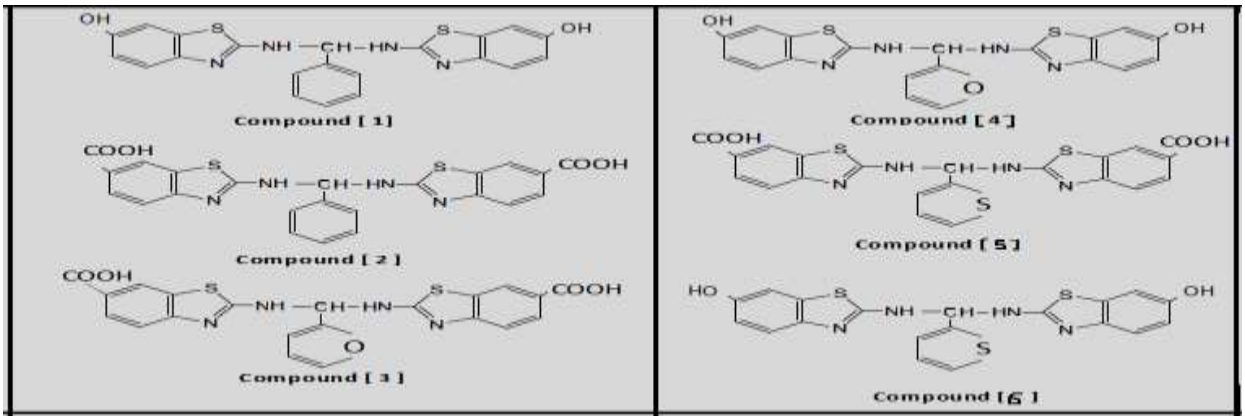
P- Hydroxy aniline (0.01 mole) reacted with (0.01mole) of ammonium thiocyanate in presence of bromine with glacial acetic acid (drop by drop) and rotation in ice path according to literature[21] ,to give precipitation which filtered and dried then re crystallized to yield amine compounds ,which (0.02 mole) reacted with (0.01 mole) of furfural to give compound (4).

STEP .5: Synthesis of Compound (5)

P- Amino benzoic acid (0.01 mole) reacted with (0.01mole) of ammonium thiocyanate in presence of bromine with glacial acetic acid (drop by drop) and rotation in ice path according to literatures[17 ,21] ,to give precipitation which filtered and dried then re crystallized to yield amine compounds , which (0.02 mole) reacted with (0.01 mole) of 2-formal thiophene to give compound (5).

STEP .6: Synthesis of Compound (6)

P- Hydroxy aniline (0.01 mole) reacted with (0.01mole) of ammonium thiocyanate in presence of bromine with glacial acetic acid (drop by drop) and rotation in ice path according to literature[21] ,to give precipitation which filtered and dried then re crystallized to yield amine compounds , which (0.02 mole) reacted with (0.01 mole) of 2-formal thiophene to give compound (6).



Scheme .1: Preparation of Compounds [1-6]

Results and Discussion

Our compounds studied by chemical techniques and Biological Activity against two types of bacteria. **Organic Identifications with Many Techniques The FT.IR- Spectra :** absorption bands appeared at (NH-) Amine : 3194 ., (C=N) Endocycle: 1622 (OH) Phenol: 3389 in compound(1) , bands are appeared at (NH-) Amine : 3187 ., (C=N) Endocycle: 1629 , (CO-O) :1706 in compounds (2) ,while other bands appeared at (NH-) Amine : 3212 ., (C=N) Endocycle: 1625

.,(CO-O) : 1718 in compound (3) ., bands (NH-) Amine : 3175 ., (C=N) Endocycle: 1620 ., (OH) Phenol: 3397 in compound (4) , bands at (NH-) Amine : 3199 ., (C=N) Endocycle: 1630 ., (CO-O) : 1712 in compound (5) ., while compound [6] gave bands at (NH-) Amine : 3202 ., (C=N) Endocycle: 1621 ., (OH) Phenol: 3362, all bands summarized in Table (1) .

Table 1: FT.IR- data (cm^{-1}) of Compounds (1-6)

Comp	Other Groups
(1)	(NH-) Amine : 3194 ., (C=N) Endocycle: 1622 ., (OH) Phenol: 3389
(2)	(NH-) Amine : 3187 ., (C=N) Endocycle: 1629 ., (CO-O) : 1706
(3)	(NH-) Amine : 3212 ., (C=N) Endocycle: 1625 ., (CO-O) : 1718
(4)	(NH-) Amine : 3175 ., (C=N) Endocycle: 1620 ., (OH) Phenol: 3397
(5)	(NH-) Amine : 3199 ., (C=N) Endocycle: 1630 ., (CO-O) : 1712
(6)	(NH-) Amine : 3202 ., (C=N) Endocycle: 1621 ., (OH) Phenol: 3362

The ^{1}H.NMR- Spectra: showed peaks at δ DMSO-d6(solvent): 2.50 ., (NH) Proton of amine: 5.61 ., (OH) Phenol : 10.93 ., Protons of Phenyl ring: (6.71-7.64) in compound (1) .While compound (2) showed signals at DMSO-d6(solvent): 2.50 ., (NH) Proton of amine: 5.34 ., (CO-OH) Proton of carboxyl group: 13.03 ., Protons of Phenyl ring: (6.91-7.80). ., compound(3) appeared peak at DMSO-d6(solvent): 2.50 ., (NH) Proton of amine: 5.82 ., (CO-OH) Proton of carboxyl group: 13.17 ., Protons of Phenyl ring: (6.96 -7.39) . But compounds (4) showed signals at DMSO-d6(solvent): 2.50 ., (NH) Proton of amine: 5.74 ., (OH) Phenol : 10.67 ., Protons of Phenyl ring: (6.98-7.52) ., While compound (5) showed signals at DMSO-d6(solvent): 2.50 ., (NH) Proton of amine: 5.21 ., (CO-OH) Proton of carboxyl group: 13.03 ., Protons of Phenyl ring: (6.96 -7.62)., but compound [6] appears DMSO-d6(solvent): 2.50 ., (NH) Proton of amine: 5.61 ., (OH) Phenol : 10.93 ., Protons of Phenyl ring: (6.82-7.69) and other signals in Table (2) .

Table 2: H.NMR-data (δ - ppm) of Compounds (1-6)

Comp	Other groups
(1)	DMSO-d6(solvent): 2.50 ., (NH) Proton of amine: 5.61 ., (OH) Phenol : 10.93 ., Protons of Phenyl ring: (6.71-7.64).
(2)	DMSO-d6(solvent): 2.50 ., (NH) Proton of amine: 5.34 ., (CO-OH) Proton of carboxyl group: 13.03 ., Protons of Phenyl ring: (6.91-7.80).
(3)	DMSO-d6(solvent): 2.50 ., (NH) Proton of amine: 5.82 ., (CO-OH) Proton of carboxyl group: 13.17 ., Protons of Phenyl ring: (6.96 -7.39).
(4)	DMSO-d6(solvent): 2.50 ., (NH) Proton of amine: 5.74 ., (OH) Phenol : 10.67 ., Protons of Phenyl ring: (6.98-7.52).
(5)	DMSO-d6(solvent): 2.50 ., (NH) Proton of amine: 5.21 ., (CO-OH) Proton of carboxyl group: 13.03 ., Protons of Phenyl ring: (6.96 -7.62).
(6)	DMSO-d6(solvent): 2.50 ., (NH) Proton of amine: 5.61 ., (OH) Phenol : 10.93 ., Protons of Phenyl ring: (6.82-7.69).

The Mass Spectra : the spectra gave good evidences for formation of our compounds through fragments in Figures (2-4):

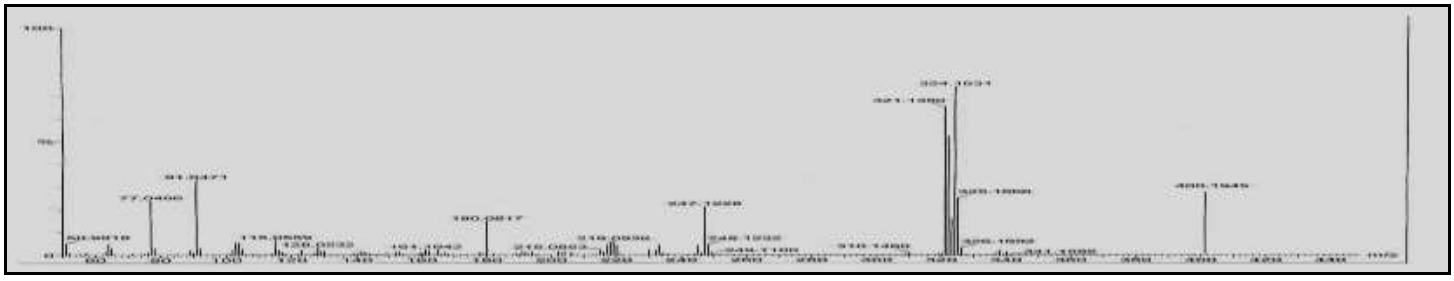


Fig 2: Mass Spectra of Compound (2)

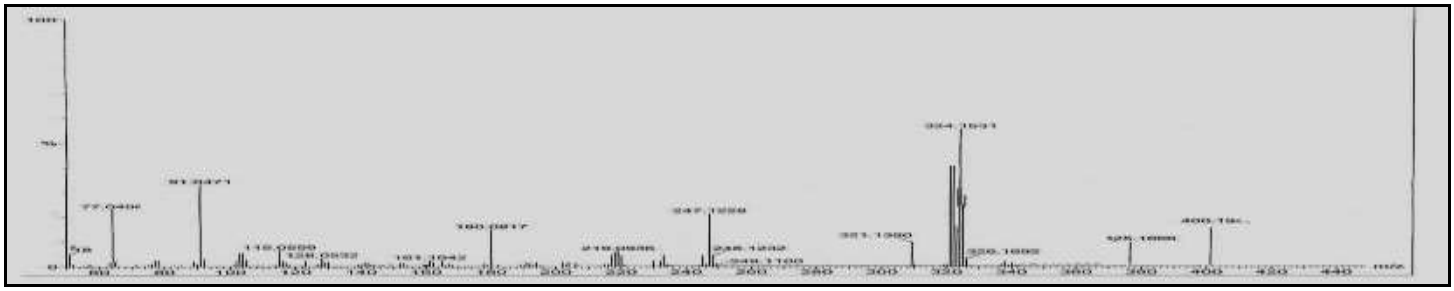


Fig 3: Mass Spectra of Compound (4)

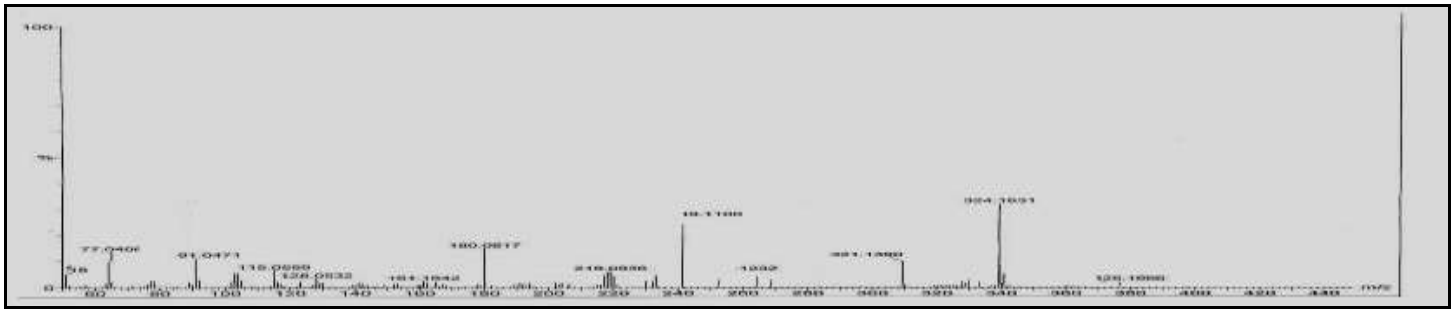


Fig 4: Mass Spectra of Compound (6)

Samples of Bacteria and Microbial Effect

According to studying[22 , 28] ,the activity for compounds was tested on two types of bacteria which collected from mouth of patients in hospital , The antimicrobial results are abstracted in Table (3). Our results of antibacterial studies appeared good activity against all types of bacteria at concentrations (5 , 15 , 25 mg.ml-1)were summarized in Table (3).

The two types of bacteria (from mouth) which tested (Streptococcus Salivarius , Streptococcus Mutans)

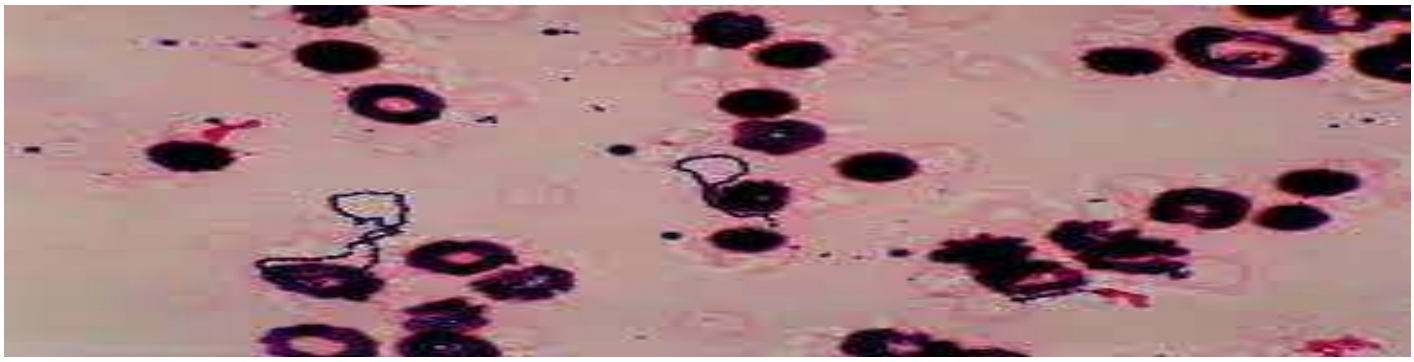


Fig. 5: Streptococcus Salivarius

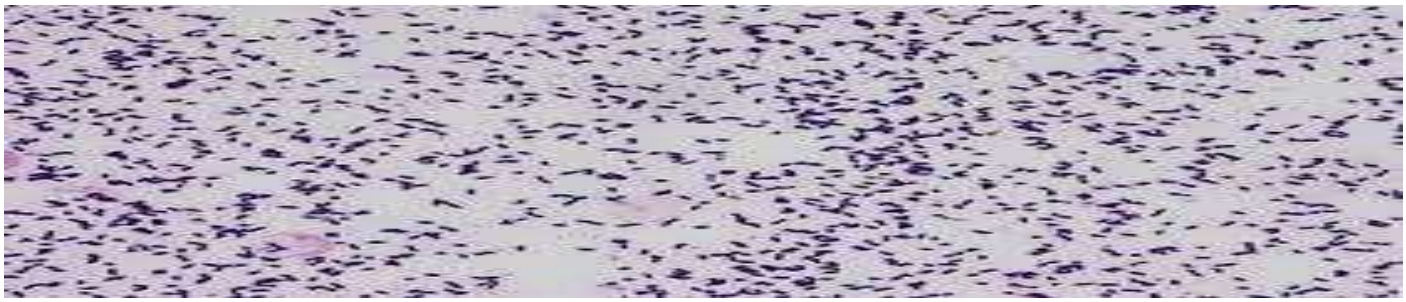


Fig. 6: Streptococcus Mutans

Fig. 7: Bacteria of Mouth

Effect of Synthesized Compounds on Bacteria (Mouth)

The formatted compounds [1-6] were tested according to their action against bacteria are described Table (3). The presence of thiophene ring gave high antibacterial effect may enhance or increase the biological activity of the sulfur atom in compounds[5,6]. The antimicrobial results are listed at Table (3). From results of antibacterial studies it was found to be potentially activity against towards two types of bacteria ,which gave good indicators from the results that the biological activity of all compounds have high biological activity which inhibit the growth of bacteria .The prepared compounds [5 , 6] have higher activity than other compounds which due to presence of sulfur atoms in their structures(35-44) in thiophene ,the mechanism of

action for this compounds involved formation of hydrogen bonding with the active centers of the cell constituents resulting in the interference with the normal cell process.

Table3: Antibacterial Activity of Compounds (Inhibition Zone in (mm)) as average of three Concentrations (5, 15, 25 mg.ml-1)

Compounds	(average of three Measurements) Streptococcus Salivarius	(average of three Measurements) Streptococcus Mutans
[1]	4	4<
[2]	4	4
[3]	8	6
[4]	8	10
[5]	14	12
[6]	14	10

Fig .8: Antibacterial activity – Streptococcus Salivarius

Fig .9: Antibacterial activity – Streptococcus Mutans

Thermal Behavior of Compounds

DSC–Thermal curves carried out for all compounds according to work[9], the results in Figures(10-12) ,DSC- curves of prepared compounds appeared high stability toward high temperature in all curves

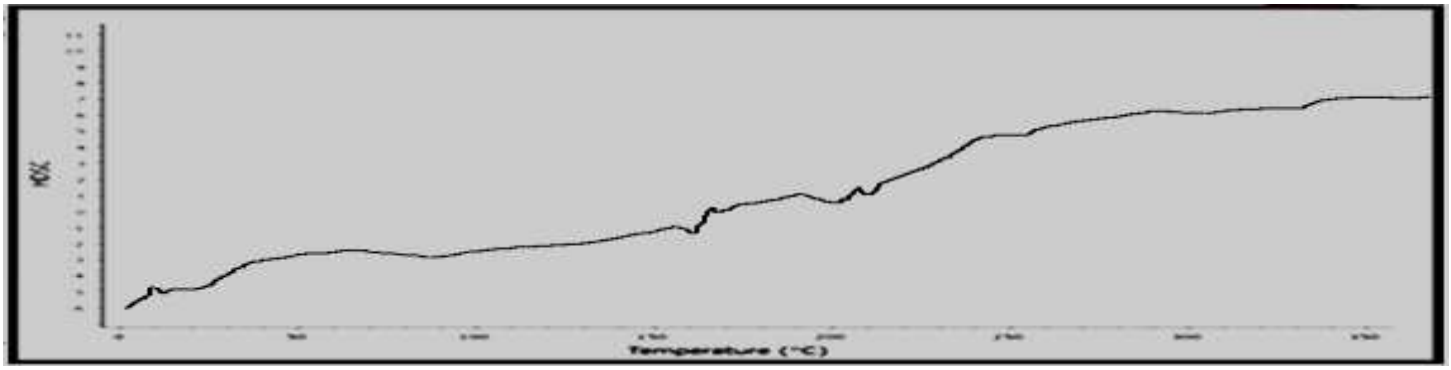


Fig .10: Thermal Curve of Compound [4]=

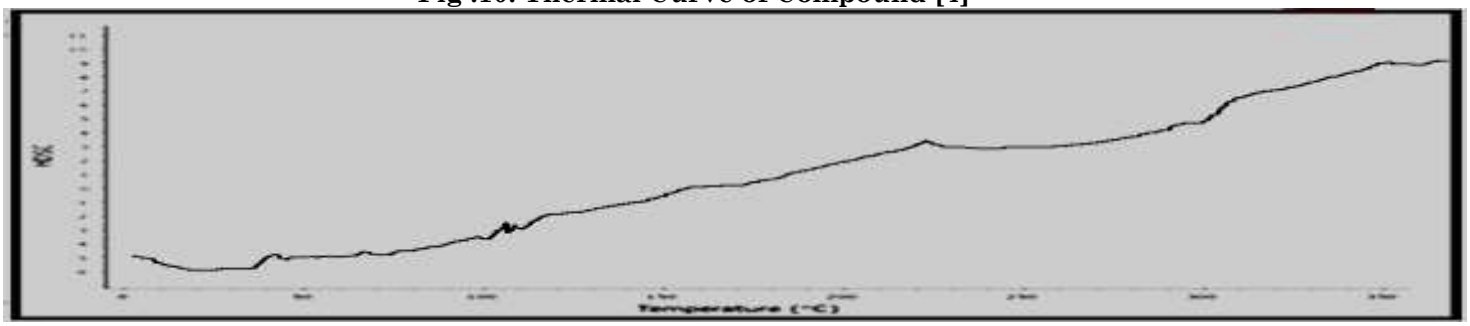


Fig .11: Thermal Curve of Compound [5]

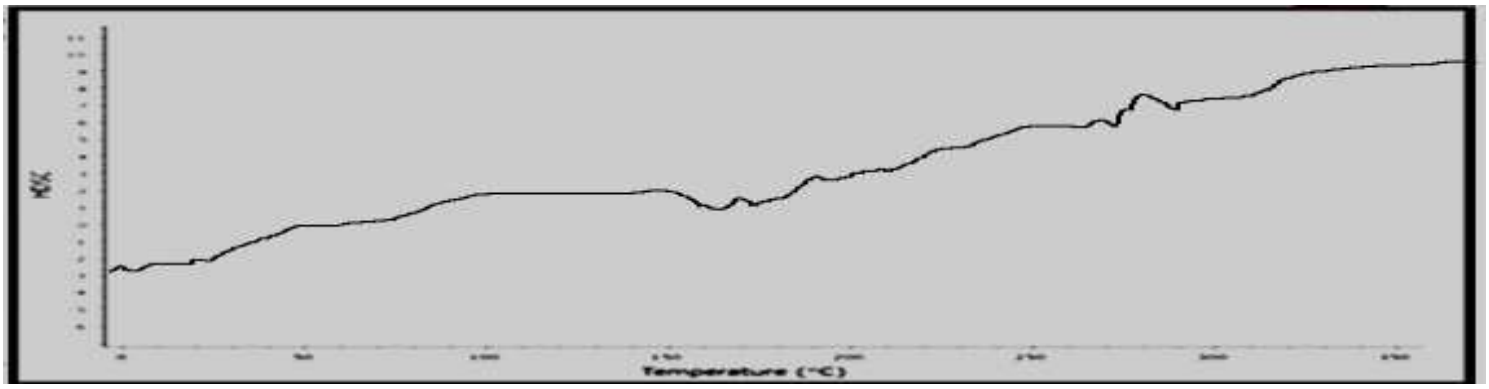


Fig .12: Thermal Curve of Compound [6]

Conclusion

The results gave strong evidence of stability of our compounds through thermal curves and good biological activity through biological assay .

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