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RESEARCH ARTICLE

Synthesis of New Reagents and Studying of (Spectral, Chromatography, Thermal, Solubility) Behavior

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Abstract

New reagents were prepared from cephalexine drug as macro reagents via several steps like (esterification , substitution , condensation, insertion) reactions to produce new analytical reagents . The first reagent involved two imine group , while the second reagent included two amide groups ,and third reagent involved two amide as a cycle by insertion reaction. Our reagents investigated by chemical techniques like (FT.IR , H.NMR , Mass)- Spectra and studied through (chromatographic curves , thermo curves) ., which indicated our results and gave evidences for formatted reagents.

Keywords: *Reagent, Ester, Amide, Insertion, Cephalexine.*

Introduction

Cephalexin is a class of drugs which belongs to a class of drugs named cephalosporin's (antibiotics) , it used to treat bacterial infections , viruses like the common cold [1-7].

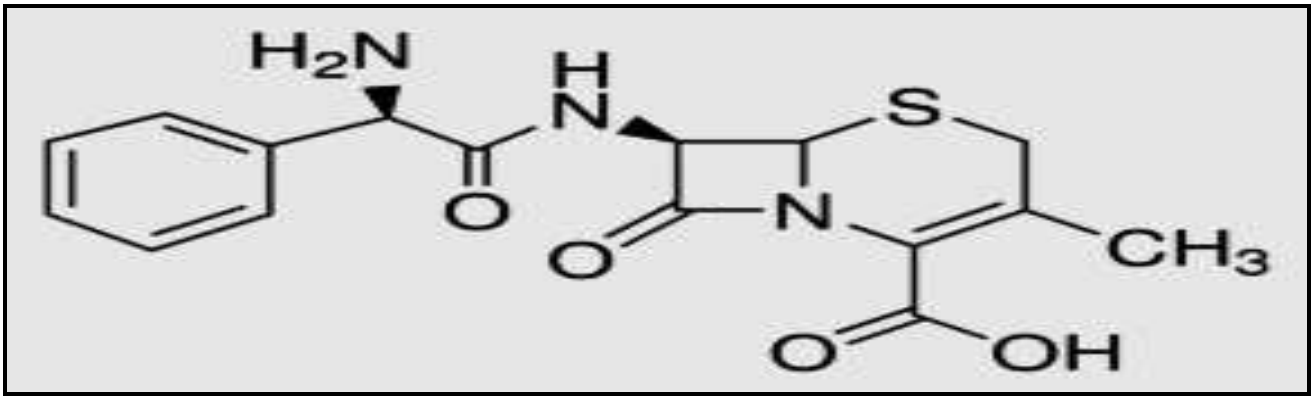


Fig .1: Cephalexin

All drugs produce via many steps of chemical reactions like condensation , for this reason the drugs are a part of organic compounds [8-15] .

The microbial action of Cephalexin is due to the inhibition of cell wall in bacteria by inhibition the final Trans peptidation step [16-25]. All drugs reacted as chemical compounds to produce other drugs have many applications in several fields [26-46].

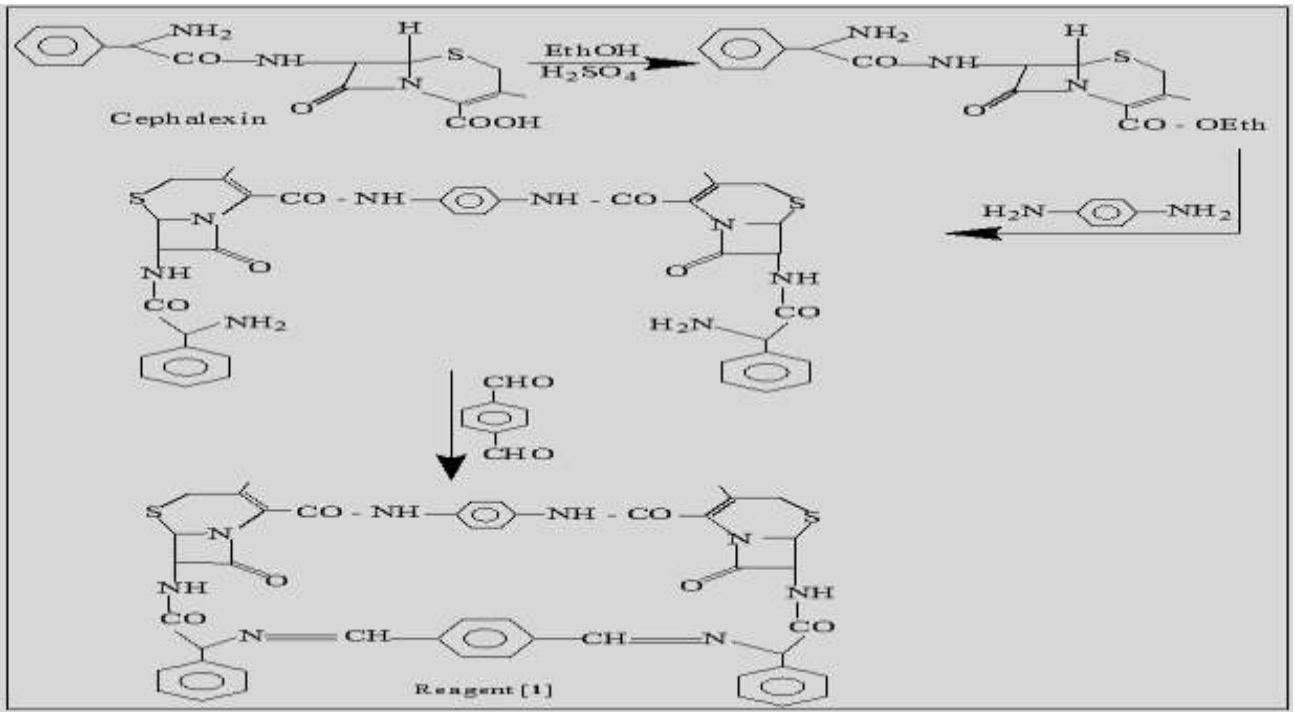
Procedures

Cephalexin powder and other compounds supplied from Sigma Company in high purity.

Experimental Part

Synthesis of Reagent

Cephalexin- ester (0.02 mole) refluxed with 1,4-phenyl diamine (0.01mole) for (3hrs) according to literature[21] ,to yield precipitation which filtered then dried and re crystallized to produce amide compounds , which (0.01 mole) reacted with (0.01 mole) of 1,4-formal benzaldehyde with drops of glacial acetic acid to obtain reagent [1].



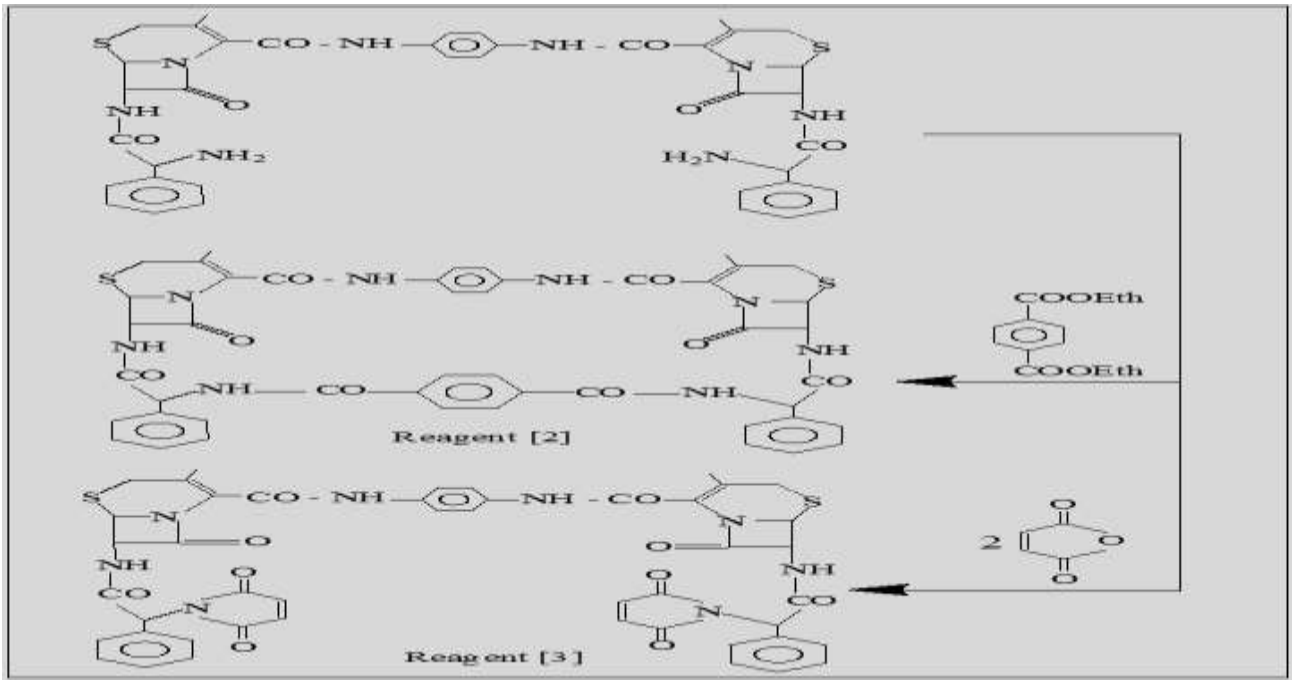
Scheme .1: Preparation of Reagent [1]

Synthesis of Reagent

Cephalexin- ester (0.02 mole) refluxed with 1,4-phenyl diamine (0.01mole) for (3hrs) according to literature[21] ,to yield precipitation which filtered then dried and re crystallized to produce amide compounds ,which(0.01 mole) reacted with (0.01 mole) of diethyltiphthalate to obtain reagent[2].

Synthesis of Reagent

Cephalexin- ester (0.02 mole) refluxed with 1,4-phenyl diamine (0.01mole) for (3hrs) according to literature [21] ,to yield precipitation which filtered then dried and re crystallized to produce amide compounds ,which(0.01 mole) reacted with (0.02 mole) of maliec anhydride with acetone as a solvent to obtain reagent [3].



Scheme .2: Preparation of Reagents [2, 3]

Results and Discussion

Organic Spectra

The FT.IR- Technique

The spectra appeared many bands at(NH-) Amide: 3205., (CH=N) Imine group: 1619., (CO-N)Amide : 1692 , (CO-NH)Amide: 1687 in compound(1) , but other bands appeared at (NH-) Amide: 3221., (CO-N)Amide: 1696, (CO-NH)Amide : (1689, 1681) in compounds (2) ,while other bands appeared at (NH-) Amide: 3215., (CO-N) Amide: 1692 , (CO-NH)Amide: (1687, 1676) ., (CH=CH) Alkene in maleimide cycle: 3090 in compound (3)., all bands in details summarized in Table 1.

Table 1: FT.IR- data (cm^{-1}) of Reagents (1-3)

Reagents	Functional Groups
(1)	(NH-) Amide : 3205 ., (CH=N) Imine group: 1619 ., (CO-N)Amide : 1692 , (CO-NH)Amide : 1687 .
(2)	(NH-) Amide: 3221 .,(CO-N)Amide : 1696 , (CO-NH)Amide : (1689 , 1681).
(3)	(NH-) Amide: 3215 .,(CO-N)Amide : 1692 , (CO-NH)Amide : (1687 , 1676) ., (CH=CH) Alkene in maleimide cycle : 3090

The ^{1}H.NMR- Technique

Which indicated to peaks at ƃ DMSO-d6 (solvent): 2.50., (NH) Proton of amide: (10.01, 10.19) Protons of Aromatic ring: (6.83 -7.71), (CH=N)proton of Imine group : 8.17 in compound (1) ,but compound (2) gave peaks at (NH) Proton of amide: (10.12, 10.03 , 10.0), Protons of Aromatic ring : (6.97 -7.80)., Compound (3) indicated to peaks at (NH) Proton of amide: (10.18, 10.11) .,Protons of Aromatic ring : (7.06-7.89)., (CH=CH) Alkene in maleimide cycle: (6.05 , 6.22)., and other peaks appeared in Table 2.

Table 2: H.NMR-data (ƃ - ppm) of Reagents (1-3)

Reagents	Functional Groups
(1)	DMSO-d6(solvent): 2.50 ., (NH) Proton of amide :(10.01, 10.19) .,Protons of Aromatic ring : (6.83 -7.71) , (CH=N)proton of Imine group : 8.17 .
(2)	DMSO-d6(solvent): 2.50 ., (NH) Proton of amide :(10.12, 10.03 , 10.0) .,Protons of Aromatic ring : (6.97 - 7.80) .
(3)	DMSO-d6(solvent): 2.50 ., (NH) Proton of amide :(10.18 , 10.11) .,Protons of Aromatic ring : (7.06 -7.89) ., (CH=CH) Alkene in maleimide cycle: (6.05 , 6.22).

The Mass - Technique

Which gave fragments for prepared reagents in our work , which appeared in Figures 1, 2:

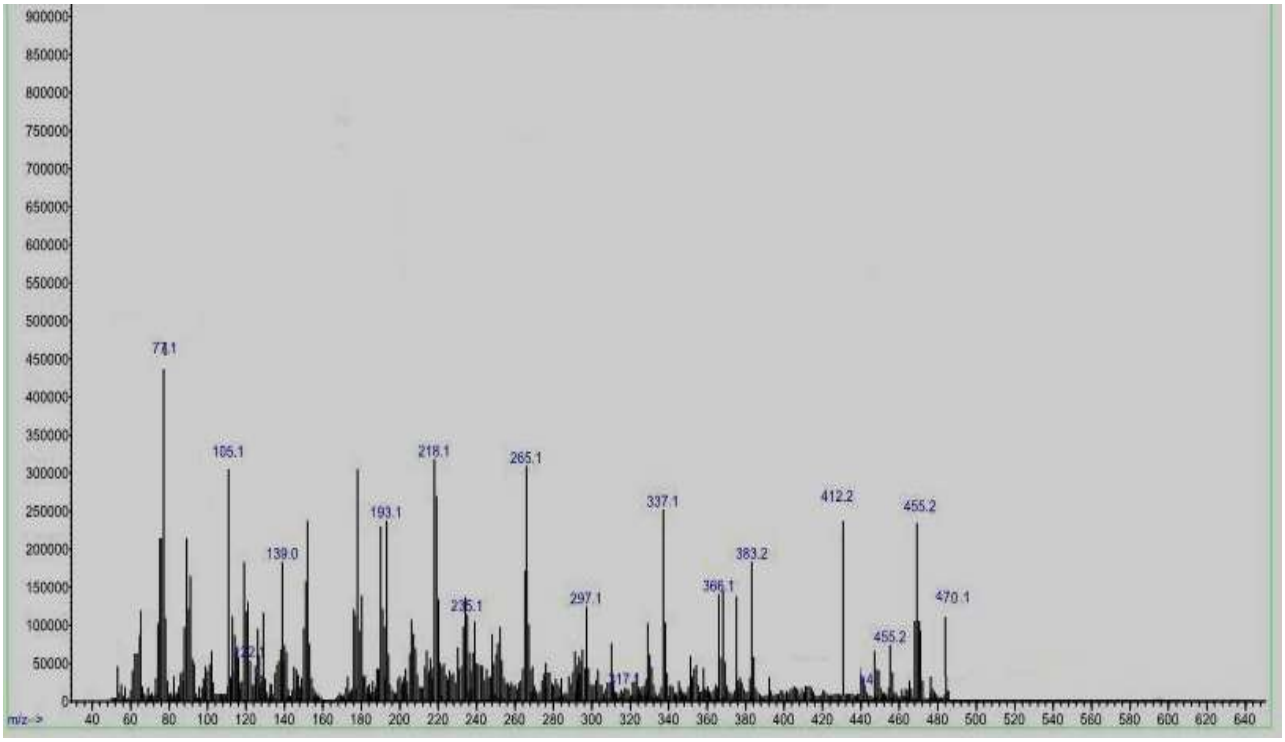


Fig 1: Mass Spectra of Reagent [1]

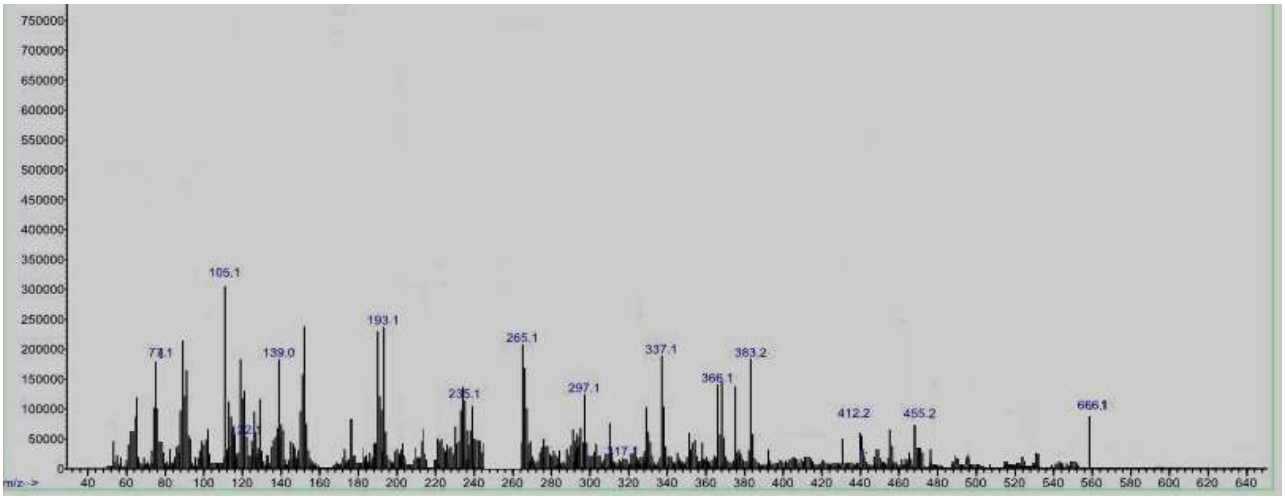


Fig2: Mass Spectra of Reagent [3]

Chromatographic Studies of Reagents

Three reagents solutions were diluted to low concentration (1 ppm), then injected with Hamilton Syringe via Nitrogen gas .

The reagents separated in this technique according to polarity and interaction of compounds, we found from our results that the first reagent [1] separated at the first time , then reagent [3] , and the last separation for reagent [2] , Figures 3-5.

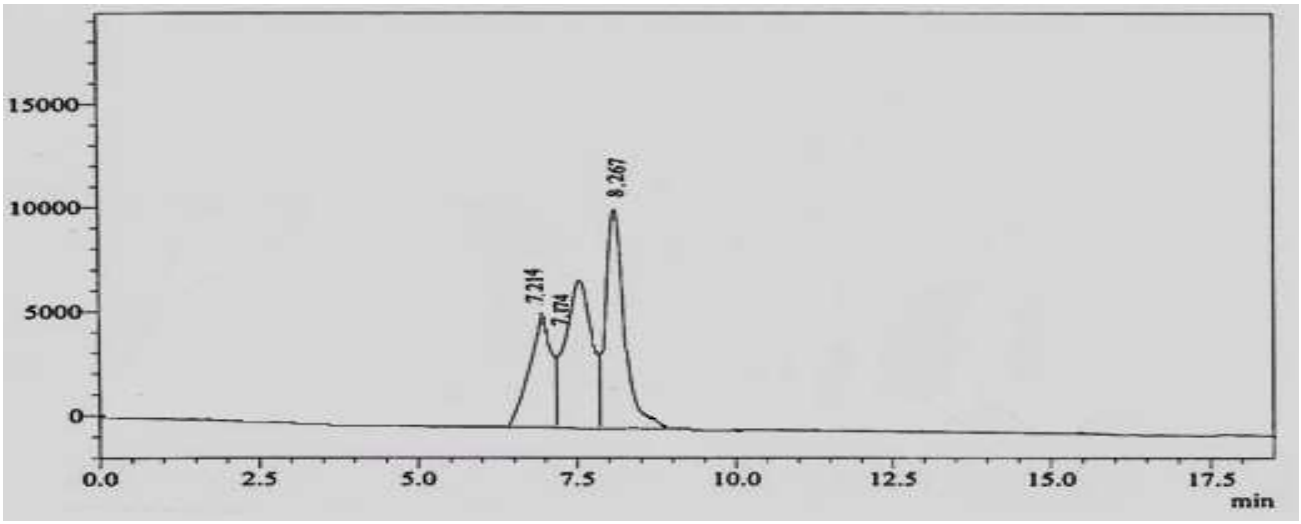


Fig 3: Chromotogram of Reagent [1]

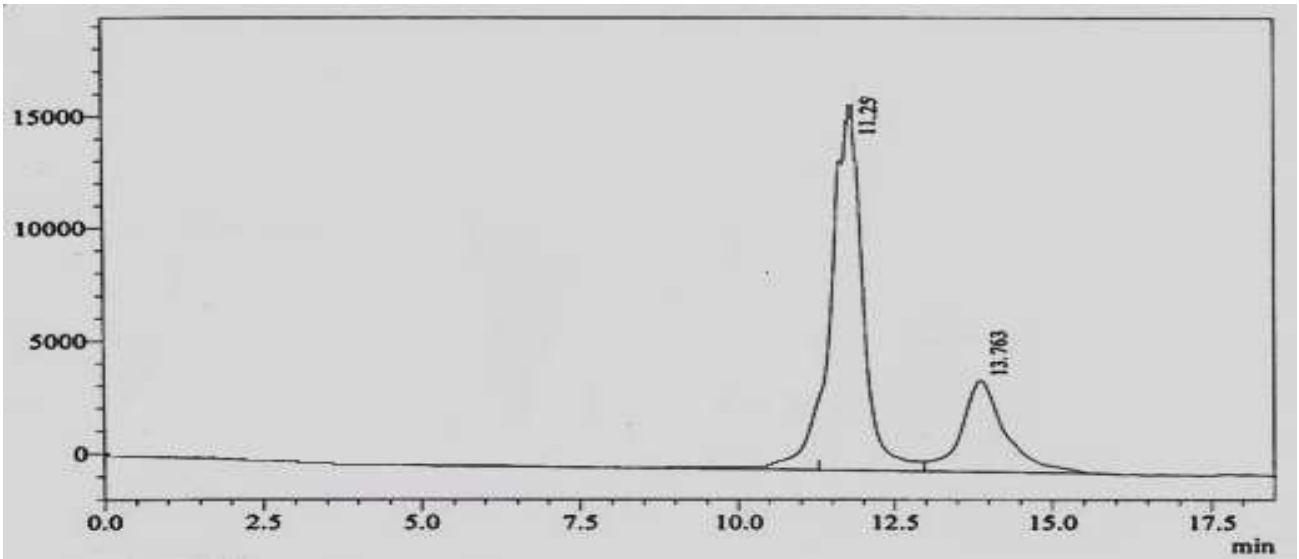


Fig 4: Chromotogram of Reagent[2]

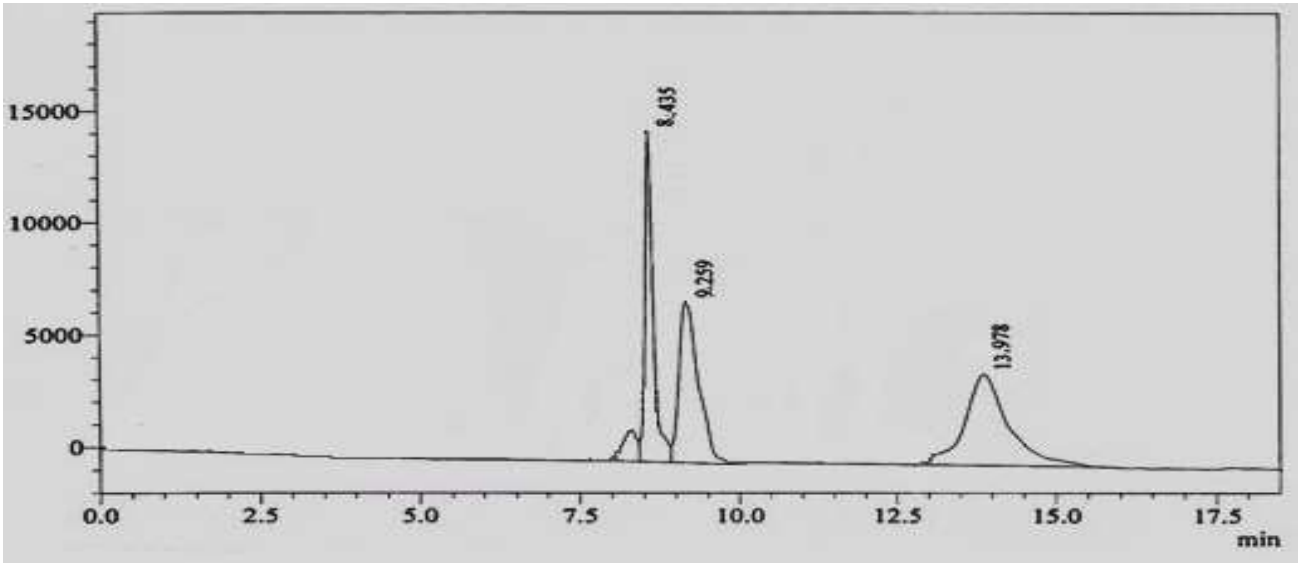


Fig 5: Chromotogram of Reagent [3]

Thermal Measurements of Reagents

Thermal Measurements of our three reagents carried out according to procedures [9] ,all results in Figures 6-8, Thermal measurements of three reagents indicated to stability of compounds toward high temperatures in all thermal curves :

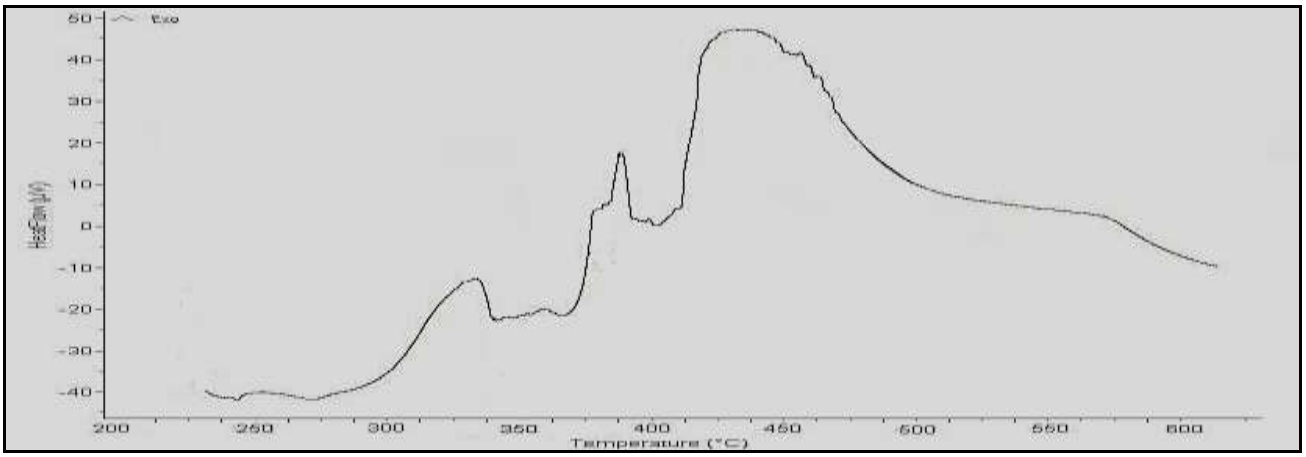


Fig 6: Thermal Curve of Reagent [1]

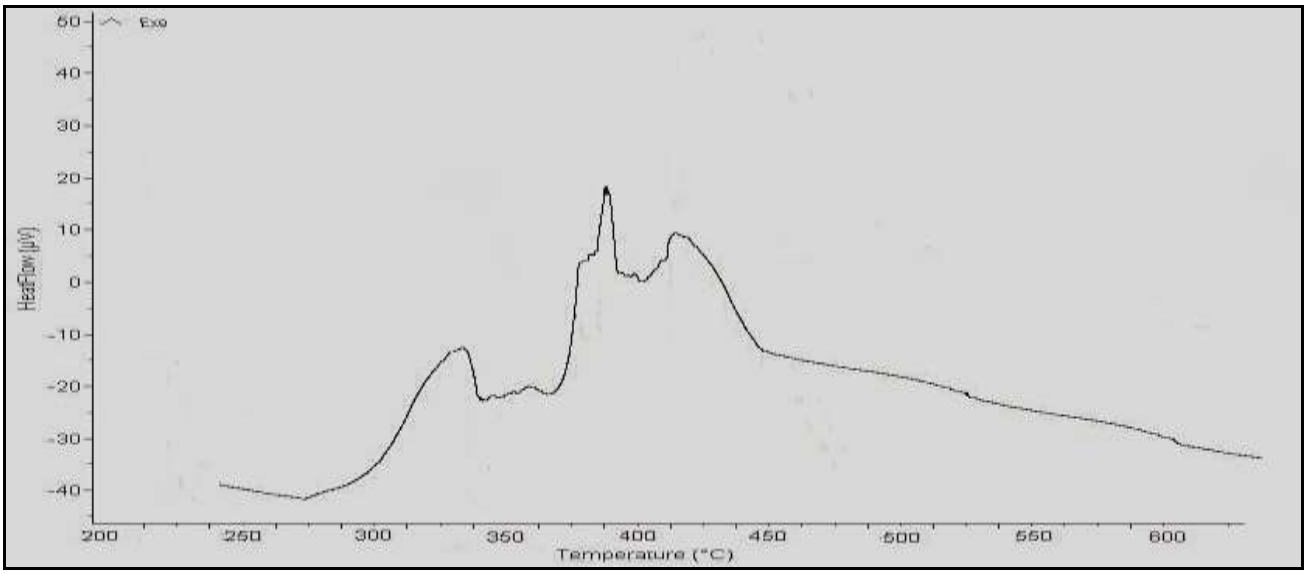


Fig 7: Thermal Curve of Reagent [2]

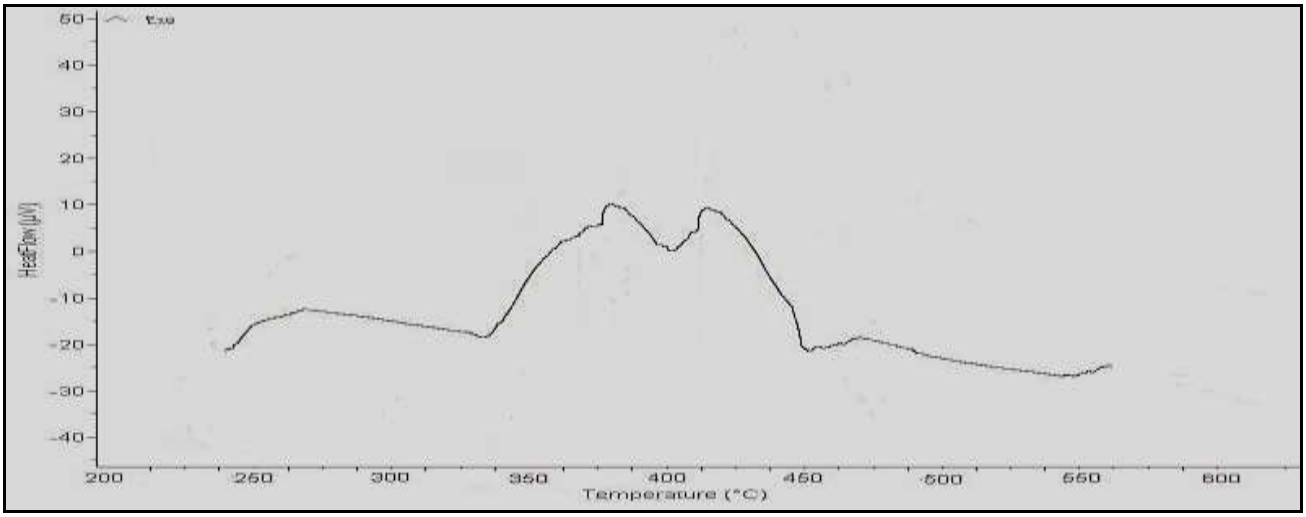


Fig 8: Thermal Curve of Reagent [3]

Behavior of Reagents in Many Solvents

All reagents which prepared in this work screened with series of solvents according to (nature , polarity of solvents , activity of functional groups) [23] in our reagents in this work , we found the results indicate that the reagents compactable with the fact (like dissolve like), the results abstracted in Table 3.

Table 3: Behavior of Reagents in Many Solvents

Reagents	Solvents					
	C_2H_5OH	DMSO	Methanol	CCl_4	$CHCl_3$	Dioxan
(1)	+	+	+	-	-	-
(2)	+	+	+	-	-	-
(3)	+	+	+	-	-	-

Conclusion

The reagents separated according to (nature, polarity, activity of functional groups , molecular weight) of reagents , all reagents gave high stability in high temperatures

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