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Research paper

Formation of New Ligands Imine- (Oxazole, Thiazole, Thiophene) And Study of (Chemical Investigation and Chromatographic Applications)

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Abstract

(Oxazole, Thiazole, Thiophene) derivatives- Imine ligands were synthesized in this paper by using many reactions such as cyclization reaction, chalcone reaction in basic medium, condensation reaction to format four new ligands included heterocycles represented by oxazole, thiophene, thiazole derivatives with imine group in same compounds. The four ligands have been identified by several methods such as spectra of (UV-Visible investigation, 1H NMR, FT.IR) and study of chromatographic applications for all ligands.
Keywords: *React, derivative, form.*

1. Introduction

Thiazole, Oxazole and thiophene are an important cycles in many bio-molecules Because of their activity in most bio-systems and their spectrum in medicinal fields, in pharmaceutical uses, in synthetic chemistry in more than field[(1-11)]:

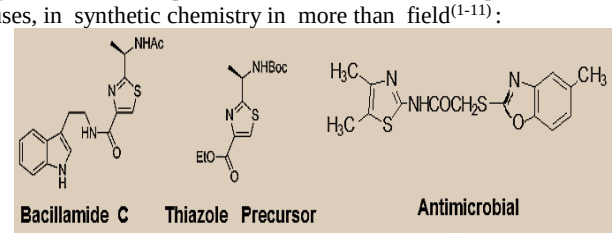


Fig.1: Thiazole, Oxazole in Medical field

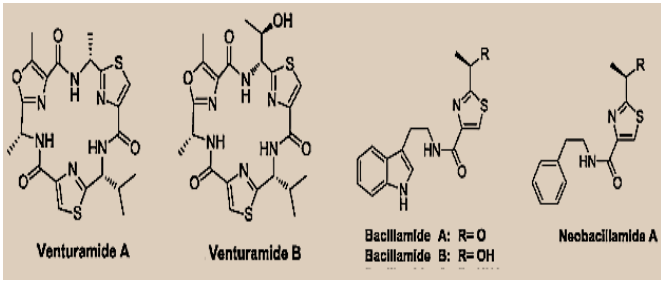


Fig. 2: Thiazole, Oxazole inBiomolecules and drugs

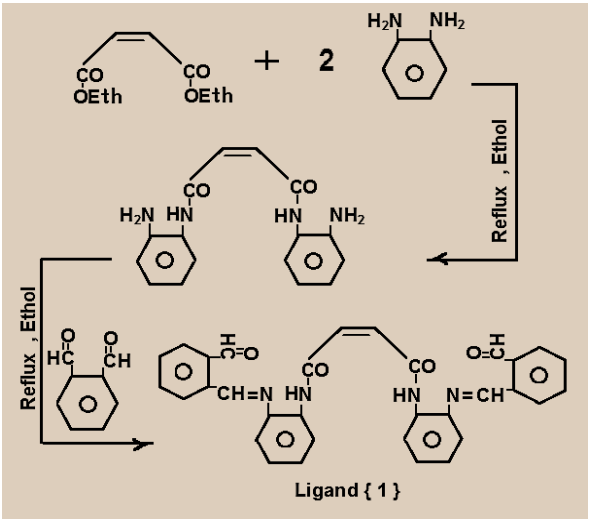
Imine compounds are of the most important class of chemical compounds[(12-24)], which were formatted firstly by Hugo Schiff in year 1864 which prepared by condensation[(25-40)] reaction between carbonyl compounds such as aldehydes or ketones and primary aromatic amine compounds.

1.1. Experimental part

All starting materials in high purity, the characterization carried out with spectrophotometric techniques: Uv-Vis spectra., FT.IR spectra were recorded on a Perkin Elmer-spectrum with(KBr)disc. H.NMR spectra were recorded by using (DMSO-d_6) as a solvent and chromatography technique.

1.2. Synthesis of Ligand[1]

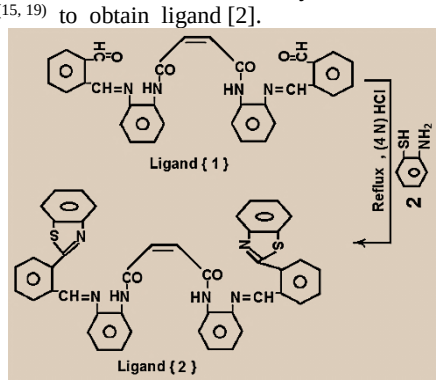
Diethyl maliete (0.1 mole) was reacted in condensation reaction with (0.2mole) of ortho-phenyldiamine with presence of absolute ethanol for (3hrs), the resulting compound (0.01 mole) refluxed with(0.02 mole) ortho-formal benzaldehyde with drops of glacial acetic acid ,the resulting compound was filtered, dried and recrystallized according to literatures[(11, 19,20)] to yield ligand [1].



Scheme 1: Preparation of Ligand [1]

1.3. Synthesis of ligand [2]

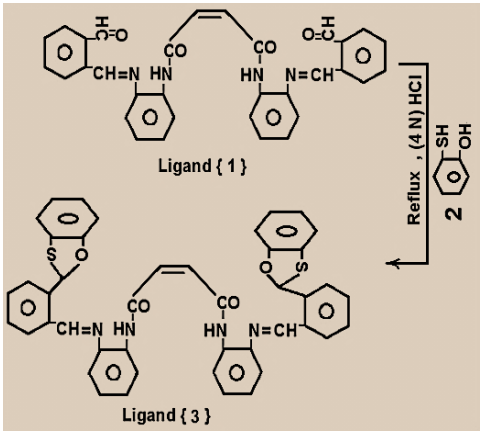
Ligand {1 } (0.1 mole) was refluxed with (0.2mole) of ortho-thiolaniline with (4N) of hydrochloric acid, the resulting compound was filtered, dried and recrystallized according to literatures[15, 19] to obtain ligand [2].



Scheme 2: Preparation ofligand [2]

1.4. Synthesis of ligand [3]

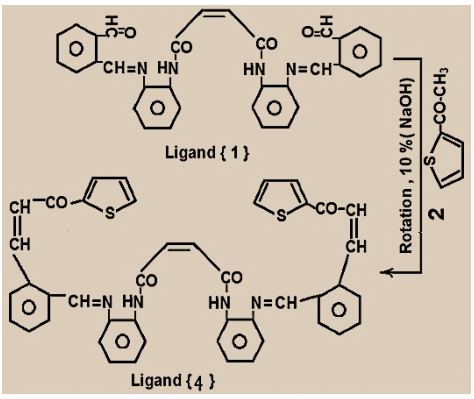
Ligand {1} (0.1 mole) was refluxed with (0.2mole) of ortho-thiolphenolwith (4N) of hydrochloric acid, the resulting compound was filtered, dried and recrystallized according to literatures[15, 33] to obtain ligand [3].



Scheme 3: Preparation of ligand [3]

1.5.Synthesis of ligand [4]

The ligand {1} (0.01 mol) was reacted with 2-acetothiophene (0.02mole) with (10 % of sodium hydroxide) solution in room temperature, the resulting compound filtered, dried, and recrystallized with ethanol according to literatures[15, 19] to give ligand [4].



Scheme 4: Preparation ofligand [4]

2. Results and discussion

Oxazole and thiazole have a wide spectrum of applications and uses in most chemistry fields ., in this work ,we synthesized four new ligands linked with imine group in same compounds., then chemical and spectral identification, study of chromatographic applications:

2.1. Characterization methods

UV-Visible Identification: It found by scanning of several solutions of ligands for maximum wave length of four ligands by using Uv-Visible spectrophotometer:

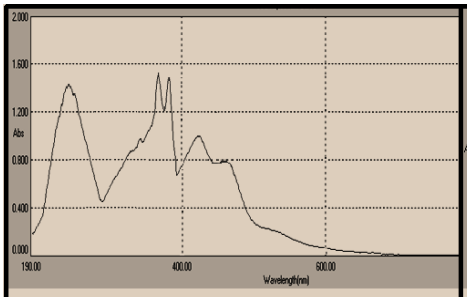


Fig (3): Uv-Visible of Ligand [1]

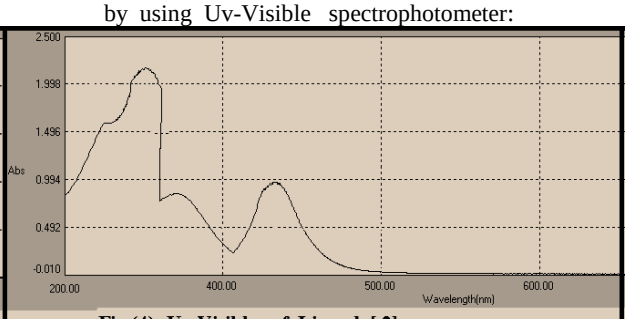


Fig (4): Uv-Visible of Ligand [2]

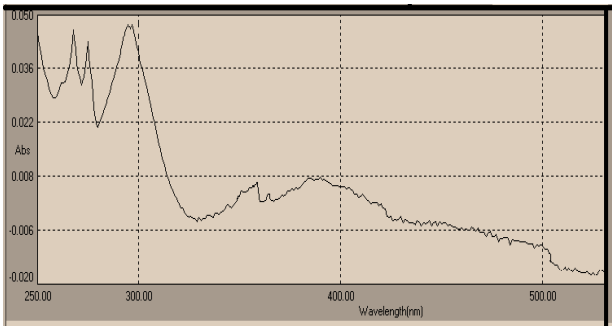


Fig (5): Uv-Visible of Ligand [3]

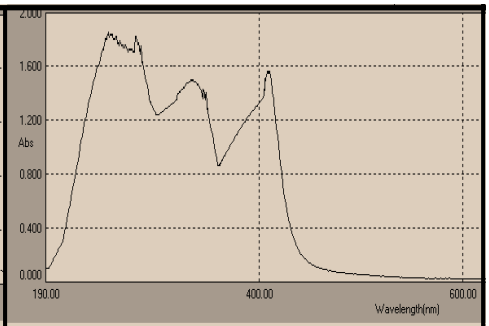


Fig (6): Uv-Visible of Ligand [4]

The FT.IR- Identification : It showed absorption bands at (-CH=N-)Imine group:(1606)., (CO-H-)carbonyl of aldehyde: 1700,(NH-CO-)amine of amide: 3250 ,(CO-N-) carbonyl of amide : 1688 , (CH=CH)alkene: 3064 in ligand(1), but other bands (-CH=N-)Imine group: (1622) ,(NH-CO-)amine of amide: 3260 ,(CO-N-) carbonyl of amide: 1690 , (CH=CH)alkene: 3067.,(C=N)endocycle of thiazole: 1640 in ligand(2)., other bands (-CH=N-)Imine group: (1618) ,(NH-CO-)amine of amide: 3254 ,(CO-N-) carbonyl of amide : 1686 , (CH=CH)alkene: 3074 .,(C-O) endocycle of oxazole: 1246in ligand(3) ., but in Ligand (4) showed bands (-CH=N-) Imine group: (1613) ,(NH-CO-) amine of amide: 3241 ,(CO-N-) carbonyl of amide : 1678, (CH=CH)alkene: 3081., (CO-CH=CH-) cabonyl of chalcone : 1694in ligand(4), other functional bands are seen in figures(7, 8).

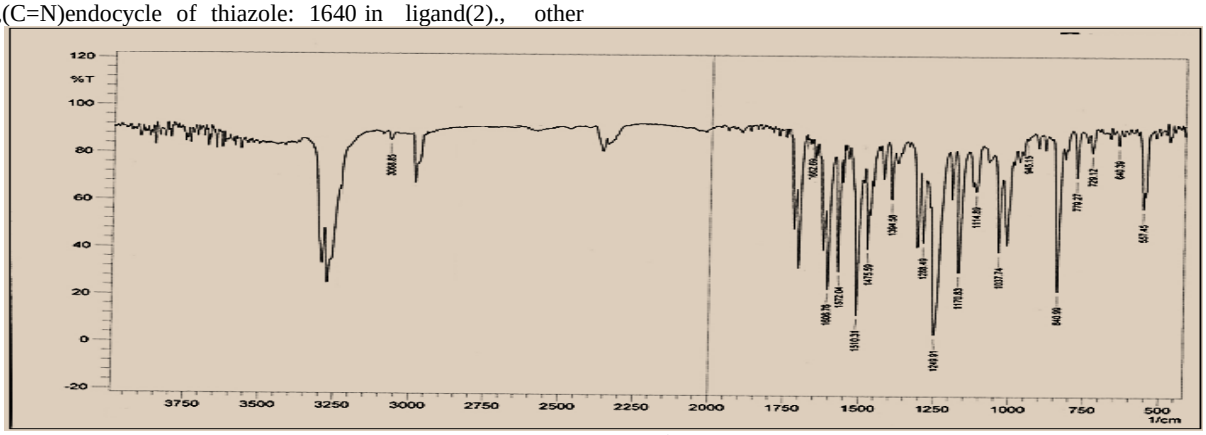


Fig. 7: FT.IR- Spectrum of Ligand [1].

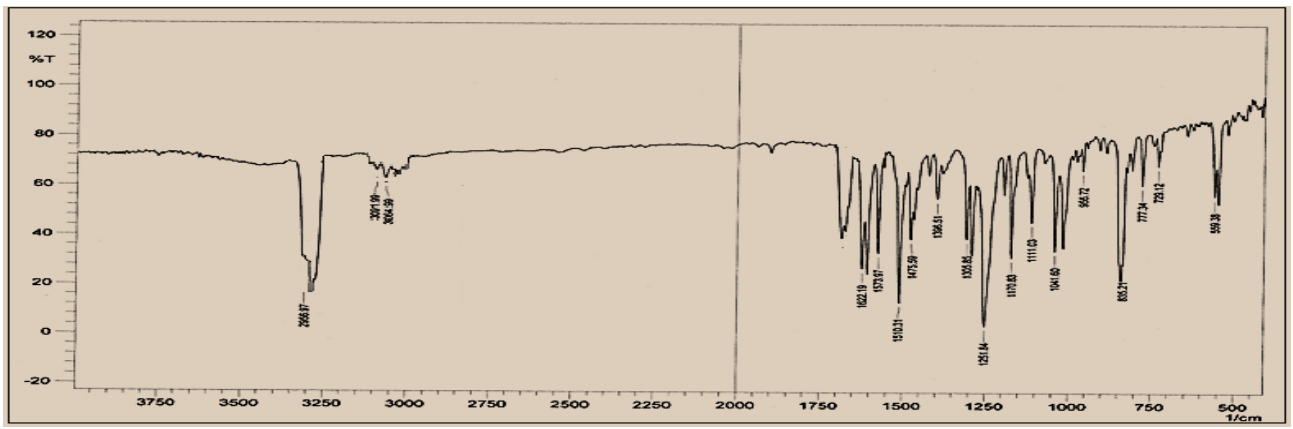


Fig. 8: FT.IR - Spectrum of Ligand [2]

The ^{1}H.NMR- Identification: It was foundmany signals at ϭ DMSO-d6(solvent): 2.50., proton of aldehyde (CO-H) : 11.24 , (NH-CO-)amide : 9.46, Protons of Aromatic ring : (7.25-7.52) , (CH=N)Imine group: 8.37, (CH=CH-) protons: (5.30, 5.71)in ligand (1), (NH-CO-)amide : 9.32, Protons of Aromatic ring : (7.11-7.69), (CH=N) Imine group: 8.21, (CH=CH-)protons : (5.27 , 5.60)in ligand (2) ,also it gave (NH-CO-)amide : 9.84 , Protons of Aromatic ring : (7.16-7.49) , (CH=N) Imine group: 8.56 , (CH=CH-)protons : (5.27, 5.60)in ligand (3), the spectrum gave (NH-CO-)amide : 9.22, Protons of Aromatic ring : (6.99-7.51), (CH=N) Imine group: 8.25, (CH=CH-CO)protons of chalcone : (5.73, 5.93)in ligand (4)., and functional signals are shown in figures (9 , 10,11).

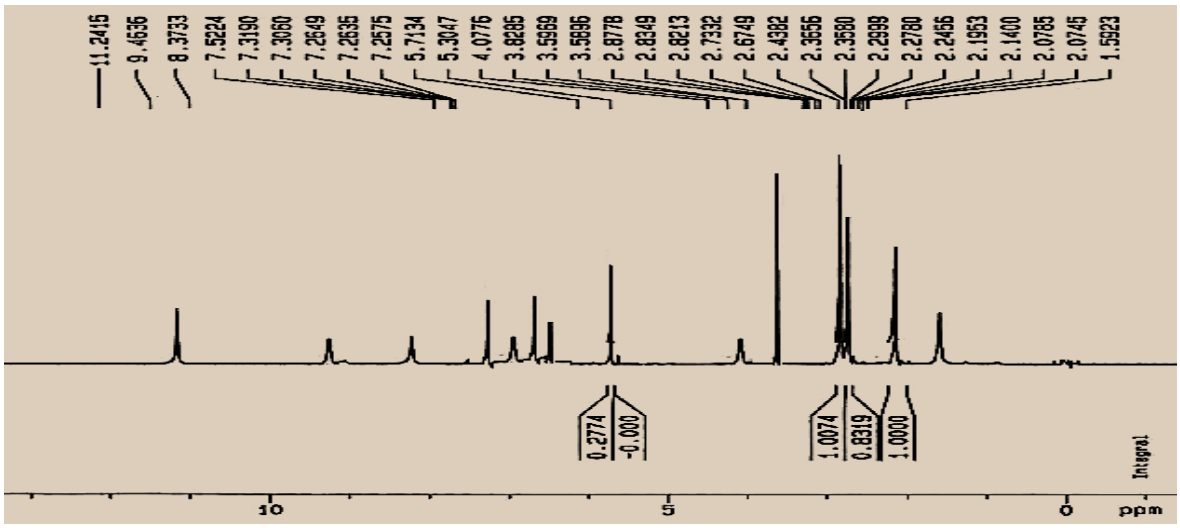


Fig. 9: H.NMR – Spectrum of Ligand [1]

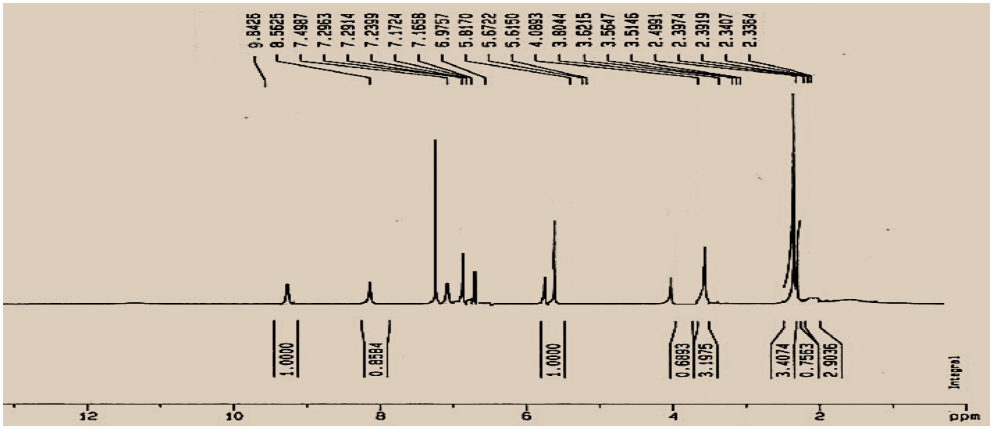


Fig. 10: H.NMR- Spectrum of Ligand [3]

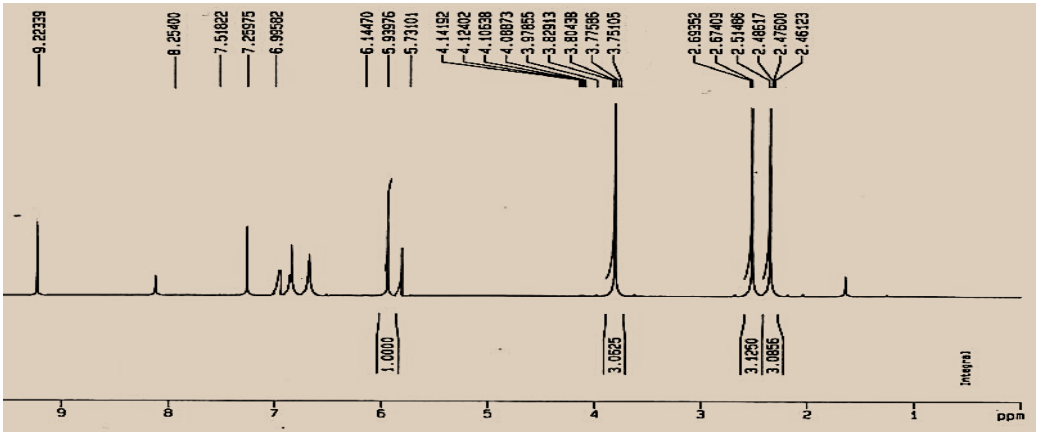


Fig.11: H.NMR- Spectrum of Ligand [4]

2.2. Chromatographic study[11] of ligands

Many diluted concentration (1 ppm) from our ligands were prepared , after that injected with using a syringe (Hamilton) in capacity (10ml) by using carrier[Nitrogen (gas flow 25 ml/min)]. The ligands separated according to their polarity and nature of functional groups and their molecular weight .,for this reason compound [4] separated at the last time[11], figures (12-15).

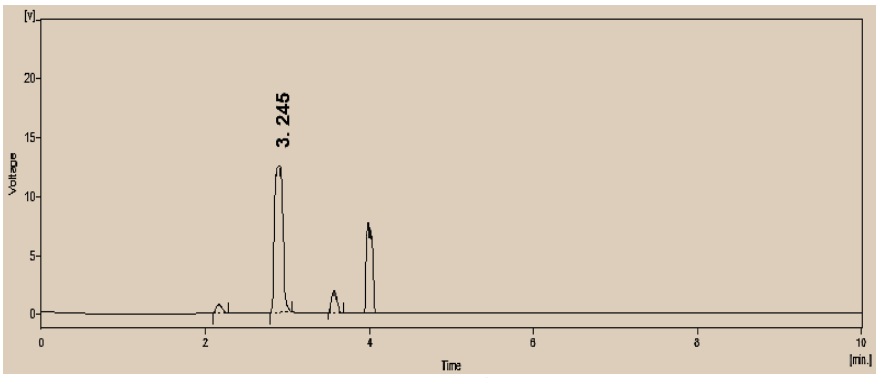


Fig. 12: Chromotogram of Compound [1]

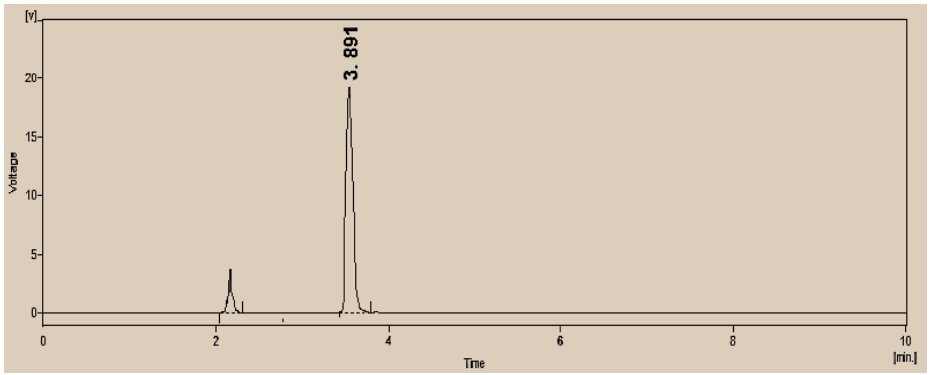


Fig. 13: Chromotogram of Compound [2]

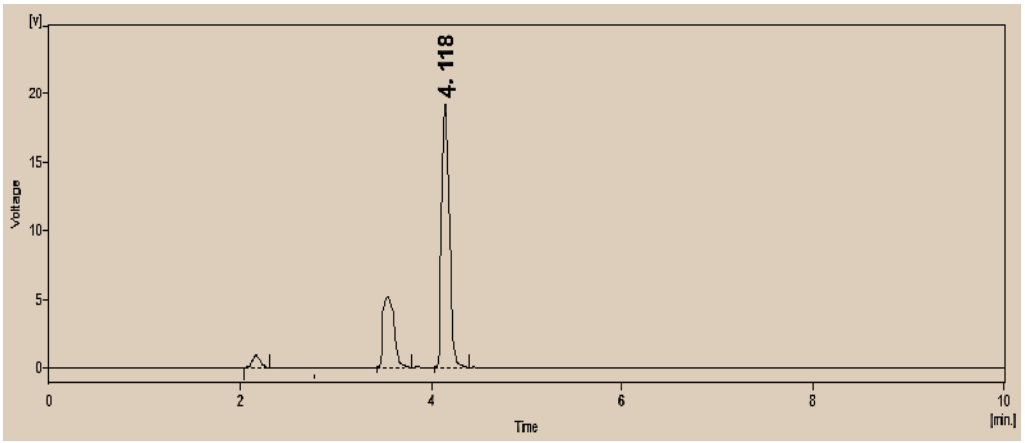


Fig. 14: Chromotogram of Compound [3]

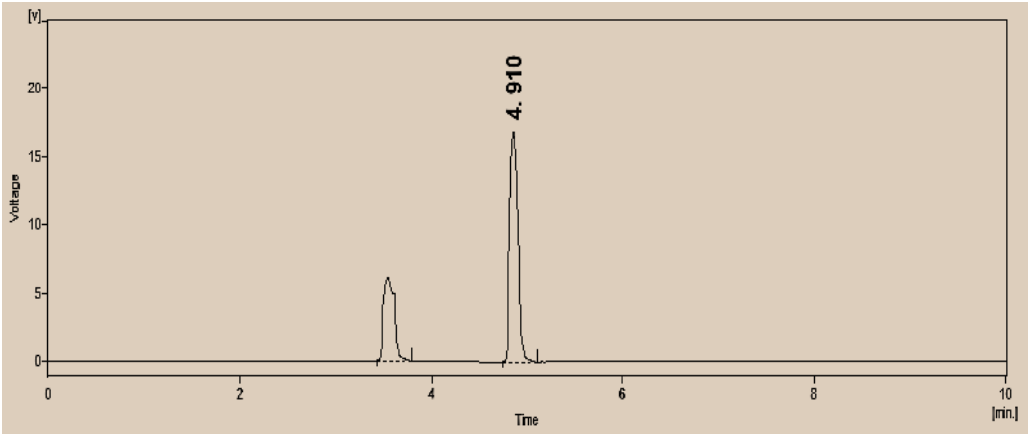


Fig. 15: Chromotogram of Compound [4]

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