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RESEARCH ARTICLE

Synthesis and Identification Some of the Seven Memberd Ring Derivatives from Carboxylic Acid

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Abstract

The research was involved preparation of cyclic compounds which are containing of more than one hetero Atoms in their structures like(N)-nitrogen or(O) oxygen, in these work the first reacting 2-(4-isobutylphenyl) propanoic acid to change to the ethyl 2-(4-isobutylphenyl) propanoate the second reacting to change to the 2-(4-isobutylphenyl) propanehydrazide.Atom.Aseven-member ringcompounds were prepared By condensation of N-Ndi methyl amino benzaldehyde with(M2)To give(A1)) and condensation of 4-chloro salicylaldehyde with(M2) to /give(A2). These compound were found to react with maleic anhydride and phthalic anhydride and succinct anhydrideTo give 7-memberd ring (N1), (N2) and (Y1), (Y2) and (Y3). All these synthesized compound have been characterized by melting point elemental analysis FTIR and HMNR spectroscopy by their un corrected melting points,elemental analysis and FT-IR spectra

Introduction

Ibuprofen Is a medicine for non steroid anti-inflammatory[2]and is used analgesic –antiinflammatory[3] and is used to relieve the symptoms of arthritis, dysmenorrhea and in the treatment of prevention of Alzheimer's disease[4] and reduce the risk of developing Parkinson's disease belongs to medication non steroid[5] working on inhibiting an enzyme (Cyclo oxygenase) and symbolizes his (COX)inhibit[6] manufacturing acids prostaglandin fatty works on the inhibition of platelet clumping[7][8] , but when you use for long periods it will produce many have side effects such as gastrointestinal disease , peptic ulcer nausea, indigestion, and because it contains a set of (COOH) effective make us can add Aldehyde ether Vehicles such as the formation of compounds Schiffs bases and compounds , including attending annular sevenring importance pharmaceutical and biological.

Expermentl

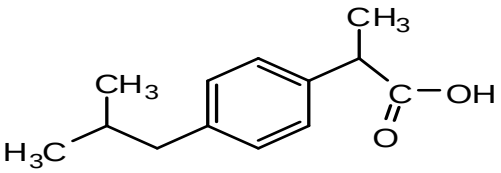
All chemicals materials were supplied from Merck and BDH-chemical company(uk). All measurements were carried out by:-

Melting points :Electrothemal 9300,melting point Engineering LTD,U,K FT-IRspectra :Fourrier transforminfrared shimadzu (8300)(FI-IR),Kbr disc was performed by co.S.Q.Iraq.

Elemental Analysis (C.H.N):EA-017mth in Lab of Babylon University. HNMR spectra: in center Lab of kashan university in Iran.

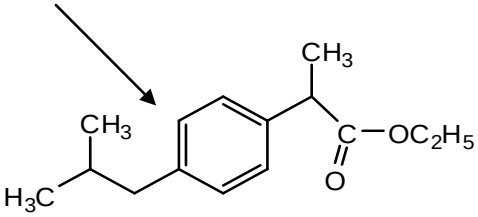
Synthesis methods Preparation of the compound ethyl 2 -(4-isobutylphenyl) propanoate.

Been taking(10) gm of (Ibuprofen)2-(4-iso butylphenyl)propanoic acid. is dissolved in(40) ml of ethanol absolute. After the completion of solvent is added 4 drop of H2SO4concenter. Escalation for 16h. Follow-up interaction by (TLC). After the completion of the reaction we instillation the solvent .Then Added cold water. We conclude by CCl4. wash water distilled water article Na_2CO_3 5% and then with distilled water until neutral drain the over Na2SO4.



2-(4-isobutylphenyl)propanoic acid

i



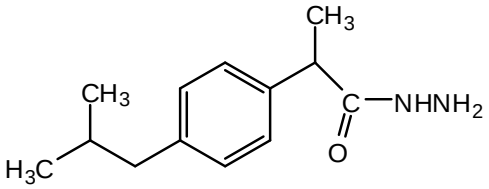
ethyl 2-(4-isobutylphenyl)propanoate

i=ETOH abs,H,ref

M1

preparation the compound 2 - (4-isobutylphenyl) propanehydrazide

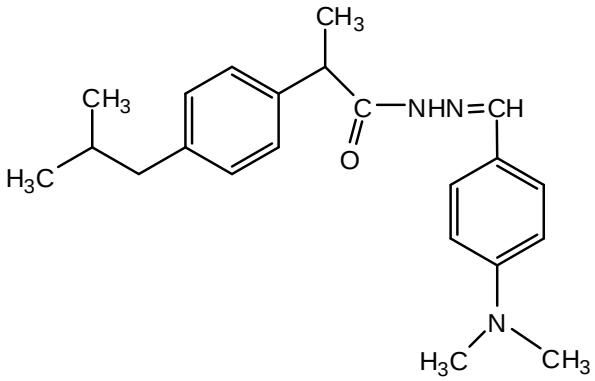
Taking the amount of (M1) 6gm ethyl 2 - (4-isobutylphenyl) propanoate and dissolved in 30ml ethanol absolute and then add 9ml of NH2NH2-H2O80% gradually while stirring and then escalation for 16h and follow-up interaction mediated (TLC) and after the completion of the reaction we distilling the solvent and then we add cold water and let the mixture for a full day after that we filtration, washing with distilled water and let dry material then we re-crystallization Kdhsalna then we drain the material in the form of pure white crystals melting point 74ċ (M2).



2-(4-isobutylphenyl)propanehydrazide

M2

Preparation of compound N'-(4 - (dimethylamino) benzylidene) -2 - (4-isobutylphenyl) propanehydrazide.
Taken(0.5) gm of the compound (M2) and melted in (30) ml of ethanol absolute and add(0.33) gm (dimethylamino) benzaldehyde 4 - then add 3drop of GAA and escalation for 8h are monitored interact mediated (TLC) produse86% (A1) .

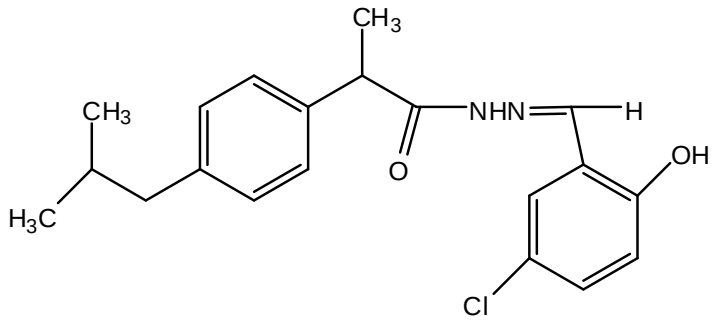


N'-(4-(dimethylamino)benzylidene)-2-(4-isobutylphenyl)propanehydrazide

A1

Synthesisof N'-(5-chloro-2-hydroxybenzylidene) - 2- (4-isobutylphenyl) propanehydrazide

0.5 mg of (M2 was taken and dissolved in (30)ml of ethanol absolute and -5-chloro-2-hydroxybenzaldehyde added and 3drop frame G-A-A than refluxed for 12 hrs was performed. And Followed up the interaction by(TLC) After cooling precipitated it re-crystallized from ethanol and gave Consists Article of white color MP125ċ(A2)

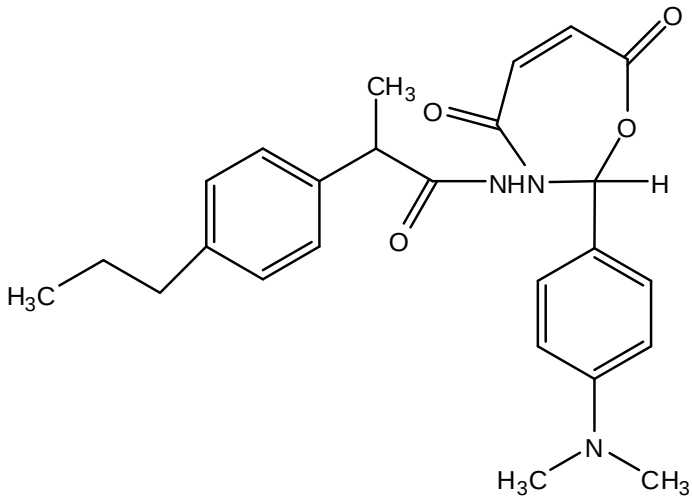


N'-(5-chloro-2-hydroxybenzylidene)-2-(4-isobutylphenyl)propanehydrazide

A2

Synthesis of(Z)-N-(2-(4-(dimethylamino)phenyl)-4,7-dioxo-1,3-oxazepin-3(2H,4H,7H)-yl)-2-(4-isobutylphenyl)propanamide

Amixture of equimolar amounts (0.5)gm of(A1)and (0.13)mol of maleic anhydride were dissolved in (30)ml of dry benzene and refluxed for (8hrs)After cooling aprecipitate it re crystallized from ethanol Consists Articleof orange color MP125ċ

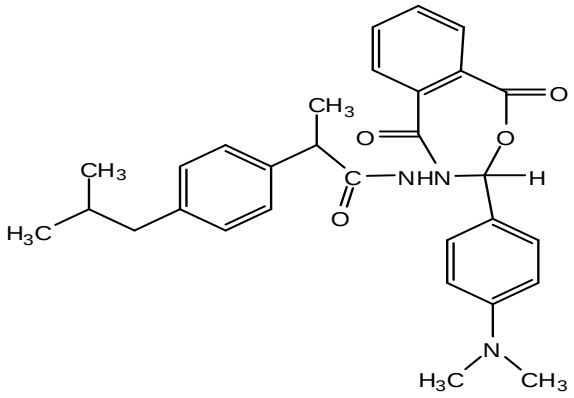


(*Z*)-*N*-(2-(4-(dimethylamino)phenyl)-4,7-dioxo-1,3-oxazepin-3(2*H*,4*H*,7*H*)-yl)-2-(4-isobutylphenyl)propanamide

N1

SynthesisofN-(3-(4-(dimethylamino)phenyl)-1,5-dioxobenzo[e] [1,3]oxazepin-4(1H,3H,5H)-yl)-2-(4-isobutylphenyl)propanamide

Amixture of equimolar amounts (0.5)gm of(A1)and (0.22)mol of phthalic anhydride were dissolved in (30)ml of dry benzene and refluxed for (8hrs)After cooling aprecipitate it re crystallized from ethanol Consists Articleof read color MP75ċ

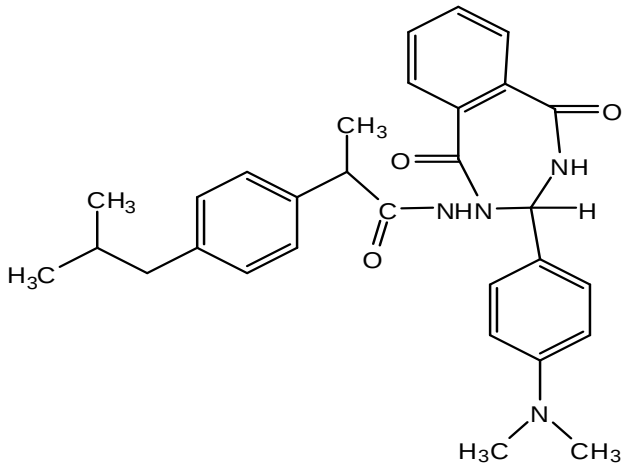


N-(3-(4-(dimethylamino)phenyl)-1,5-dioxobenzo[*e*][1,3]oxazepin-4(1*H*,3*H*,5*H*)-yl)-2-(4-isobutylphenyl)propanamide

N2

Synthesis of N-(3-(4-(dimethylamino)phenyl)-1,5-dioxo-4,5-dihydro-1H-benzo[e][1,3]diazepin-2(3H)-yl)-2-(4-isobutylphenyl)propanamide

Take(0.15)from N-(3-(4-(dimethylamino)phenyl)-1,5-dioxobenzo[e][1,3]oxazepin-4(1H,3H,5H)-yl)-2-(4-isobutylphenyl)propanamide(N2) dissolved in (30)ml of dry benzene and added (0.15) from vinyl hydrazine and rfalaks (12)hrs in 65ċ After cooling aprecipitate it re crystallized from ethanol Consists Articleof grren yiow Mp144ċ

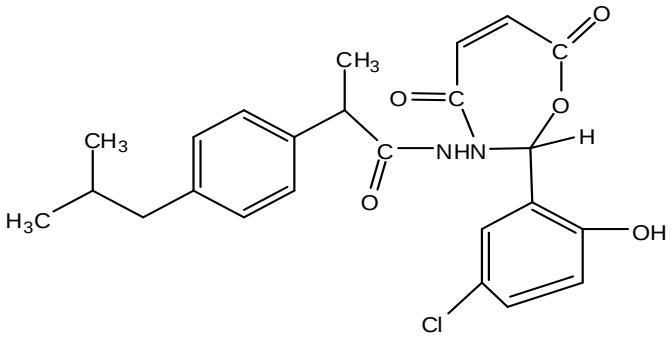


N-(3-(4-(dimethylamino)phenyl)-1,5-dioxo-4,5-dihydro-1*H*-benzo[*e*][1,3]diazepin-2(3*H*)-yl)-2-(4-isobutylphenyl)propanamide

N2η

Synthesis of (Z)-N-(2-(5-chloro-2-hydroxyphenyl)-4,7-diox o-1,3-oxazepin-3(2H,4H,7H)-yl)-2-(4-isobutylphenyl)propanamide

Amixture of equimolar amounts (0.5)gm of(A2)and (0.13)mol of maleic anhydride were dissolved in (30)ml of dry benzene and refluxed for (8hrs)After cooling aprecipitate it re crystallized from ethanol Consists Articleof white color MP125ċ

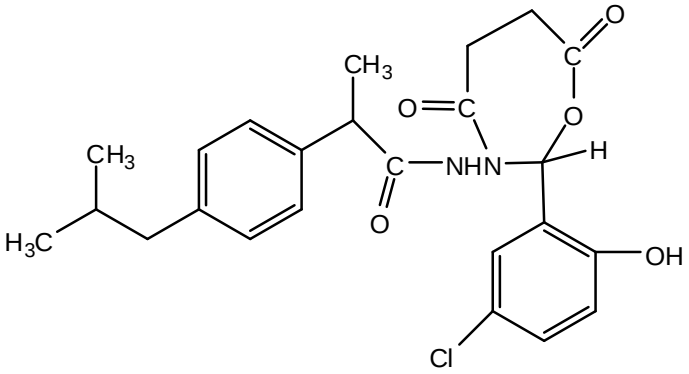


(*Z*)-*N*-(2-(5-chloro-2-hydroxyphenyl)-4,7-dioxo-1,3-oxazepin-3(2*H*,4*H*,7*H*)-yl)-2-(4-isobutylphenyl)propanamide

B1

Synthesis N-(2-(5-chloro-2-hydroxyphenyl)-4,7-dioxo-1,3-oxazepan-3-yl)-2-(4-isobutylphenyl)propanamide

Amixture of equimolar amounts (0.5)gm of(A2)and (0.13)mol of succinc anhydride were dissolved in (30)ml of dry benzene and refluxed for (13hrs)After cooling aprecipitate it re crystallized from ethanol Consists Articleof white color MP125ċ

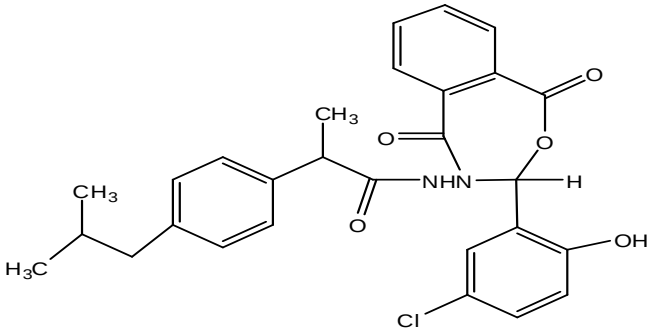


N-(2-(5-chloro-2-hydroxyphenyl)-4,7-dioxo-1,3-oxazepan-3-yl)-2-(4-isobutylphenyl)propanamide

B2

SynthesisofN-(3-(5-chloro-2-hydroxyphenyl)-1,5dioxobenzo [e] [1,3]oxazepin-4(1H,3H,5H)-yl)-2-(4-isobutylphenyl)propanamide

Amixture of equimolar amounts (0.5)gm of(A2)and (0.22)mol of phthalic anhydride were dissolved in (30)ml of dry benzene and refluxed for (8hrs)After cooling aprecipitate it re crystallized from ethanol Consists Articleof Pale yellow color MP93ċ

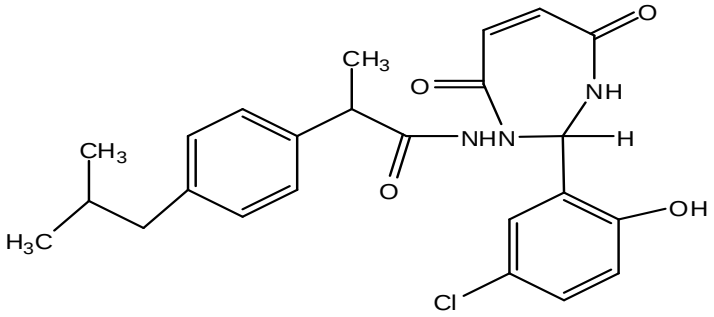


N-(3-(5-chloro-2-hydroxyphenyl)-1,5-dioxobenzo[*e*][1,3]oxazepin-4(1*H*,3*H*,5*H*)-yl)-2-(4-isobutylphenyl)propanamide

B3

Synthesis of Z)-N-(2-(5-chloro-2-hydroxyphenyl)-4,7-dioxo-2,3,4,7-tetrahydro-1H-1,3-diazepin-1-yl)-2-(4-isobutylphenyl)propanamide

Take(0.15)from in(B2) N-(2-(5-chloro-2-hydroxyphenyl)-4,7-dioxo-1,3-oxazepan-3-yl)-2-(4-isobutylphenyl)propanamide dissolved (30)ml of dry benzene and added (0.02) from vinyl hydrazine and rfalaks (12)hrs in 65ċ After cooling aprecipitate it re crystallized from ethanol Consists Articleof grren yiow Mp144ċ



(*Z*)-*N*-(2-(5-chloro-2-hydroxyphenyl)-4,7-dioxo-2,3,4,7-tetrahydro-1*H*-1,3-diazepin-1-yl)-2-(4-isobutylphenyl)propanamide

B2η

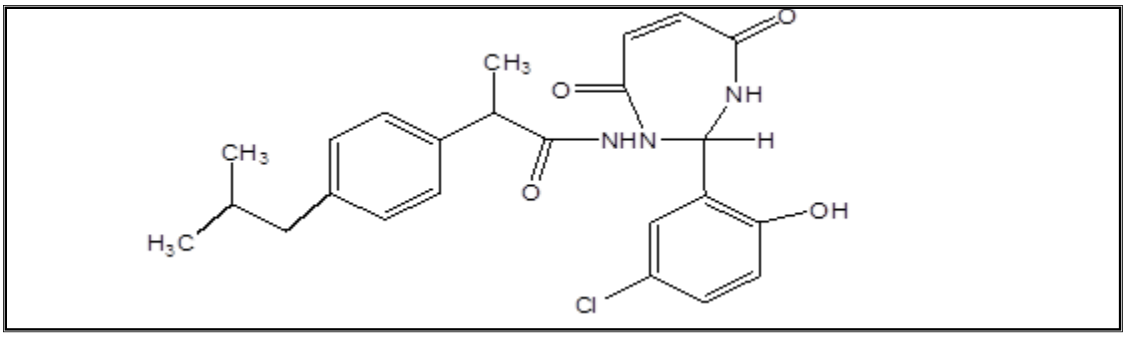
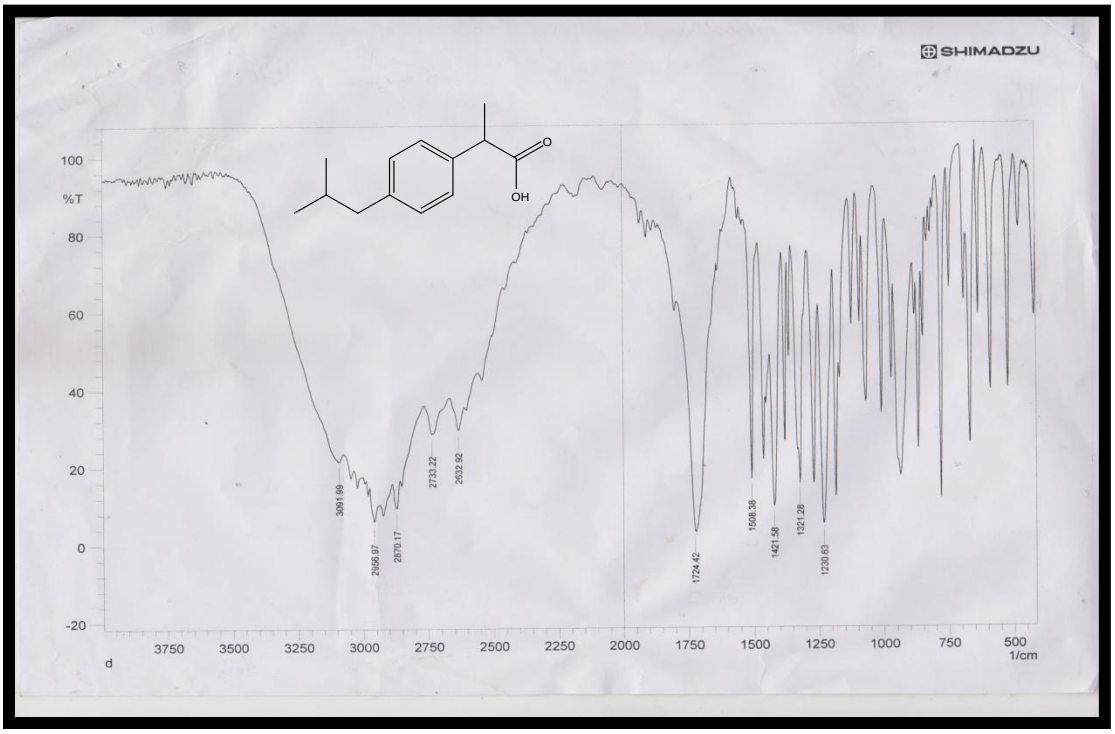


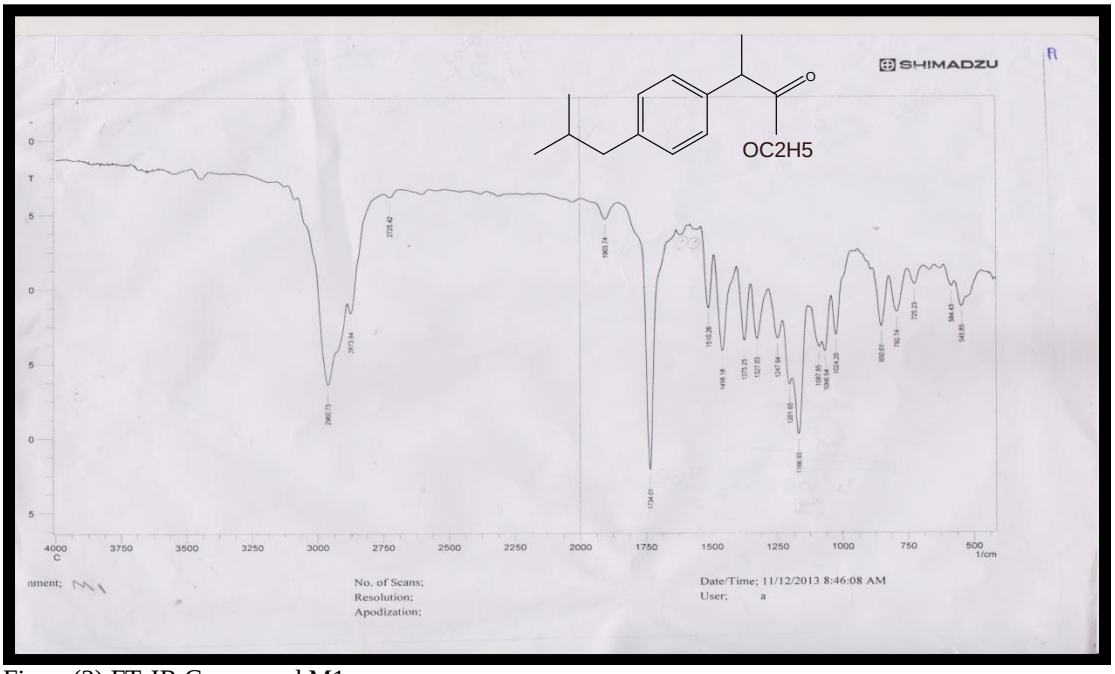
Figure 8.(z)-N(2-5-chloro-2-hy

TABLE1.Phyical properties of chemical compound.

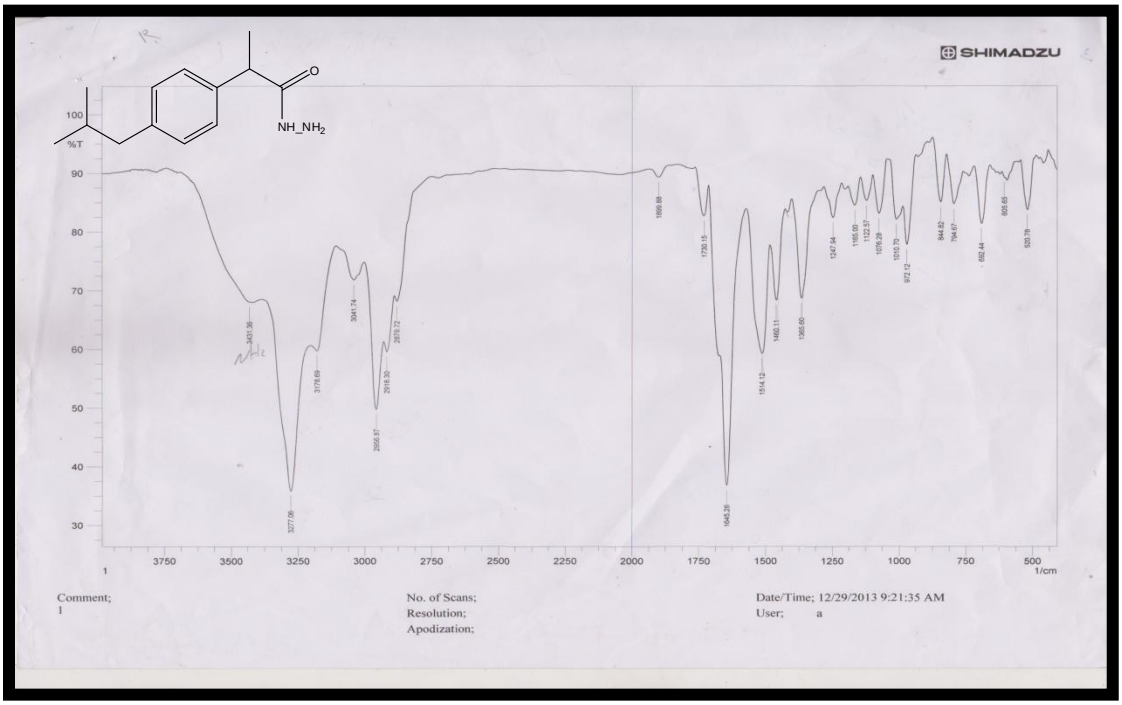
Compd code	Mol.Formula	Mol.wt	Rf valuc by TLC	Yield %	M.P(˚C)
M	C13H18O2	206.28	0.86	76	76 ˚C
M1	C15H22O2	234.33	0.6	78	Ligut
M2	C13H20N2O	220.31	0.6	87.5	78 ˚C
A1	C23H31N3O	365.51	0.5	81	145˚C
A2	C21H25ClN2O2	372.89	0.53	67	140 ˚C
N1	C26H31N3O4	449.54	0.6	79	120 ˚C
N2	C30H33N3O4	499.60	0.5	82	75 ˚C
N2η	C30H34N4O3	498.62	0.6	61	127-129C
B1	C24H25ClN2O5	456.92	0.51	80	90-92 ˚C
B2	C24H27ClN2O5	458.93	0.57	85	87 ˚C
B3	C28H27ClN2O5	506.98	0.5	73	89-93 ˚C
B3η	C24H28ClN3O4	457.95	0.45	85	110 ˚C



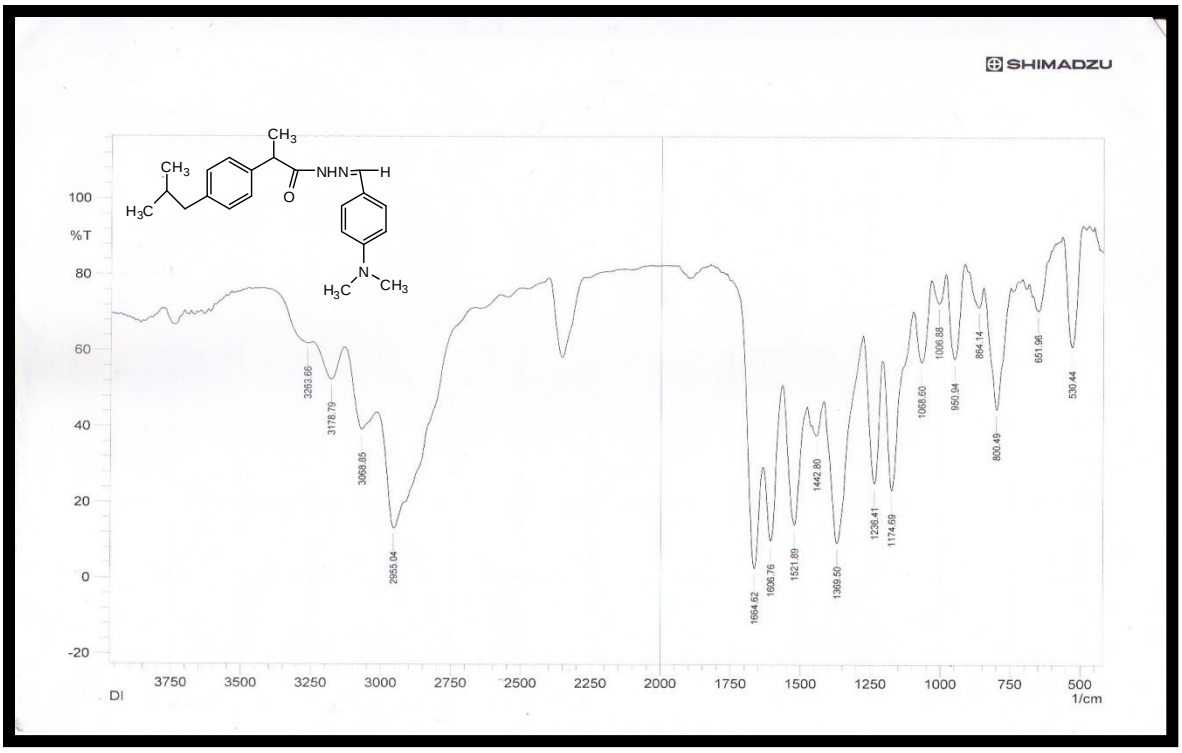
Figure(1) FT-IR Compound M



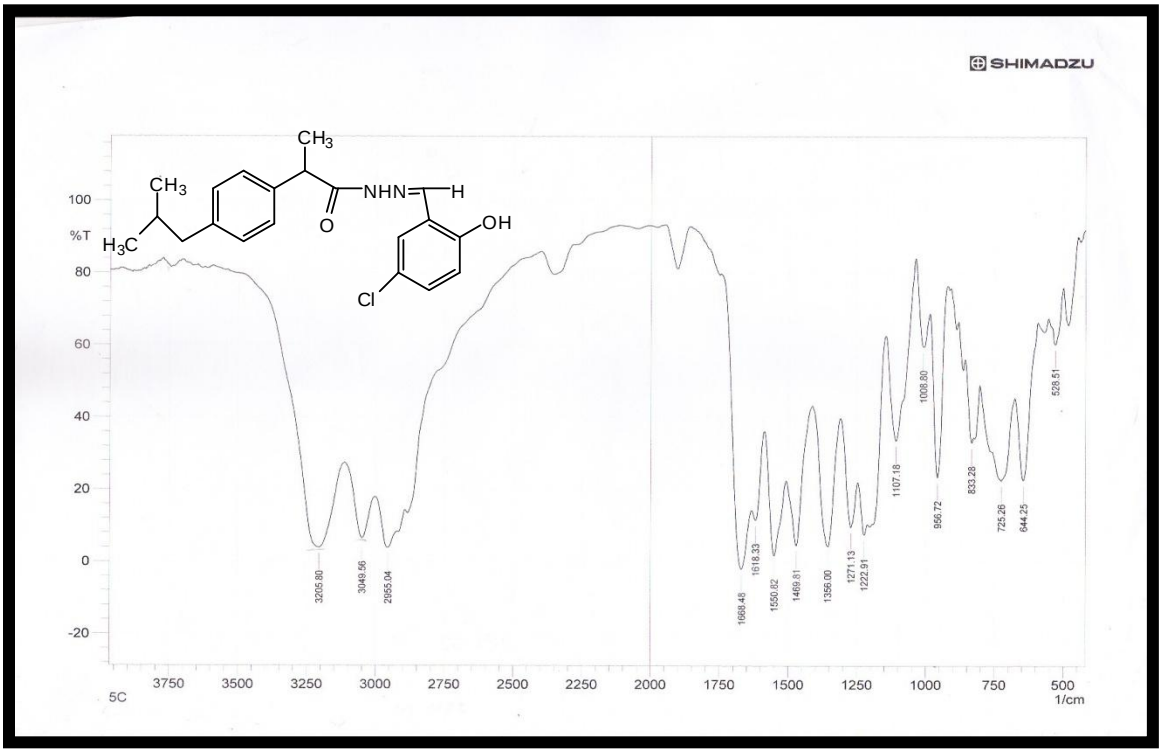
Figure(2) FT-IR Compound M1



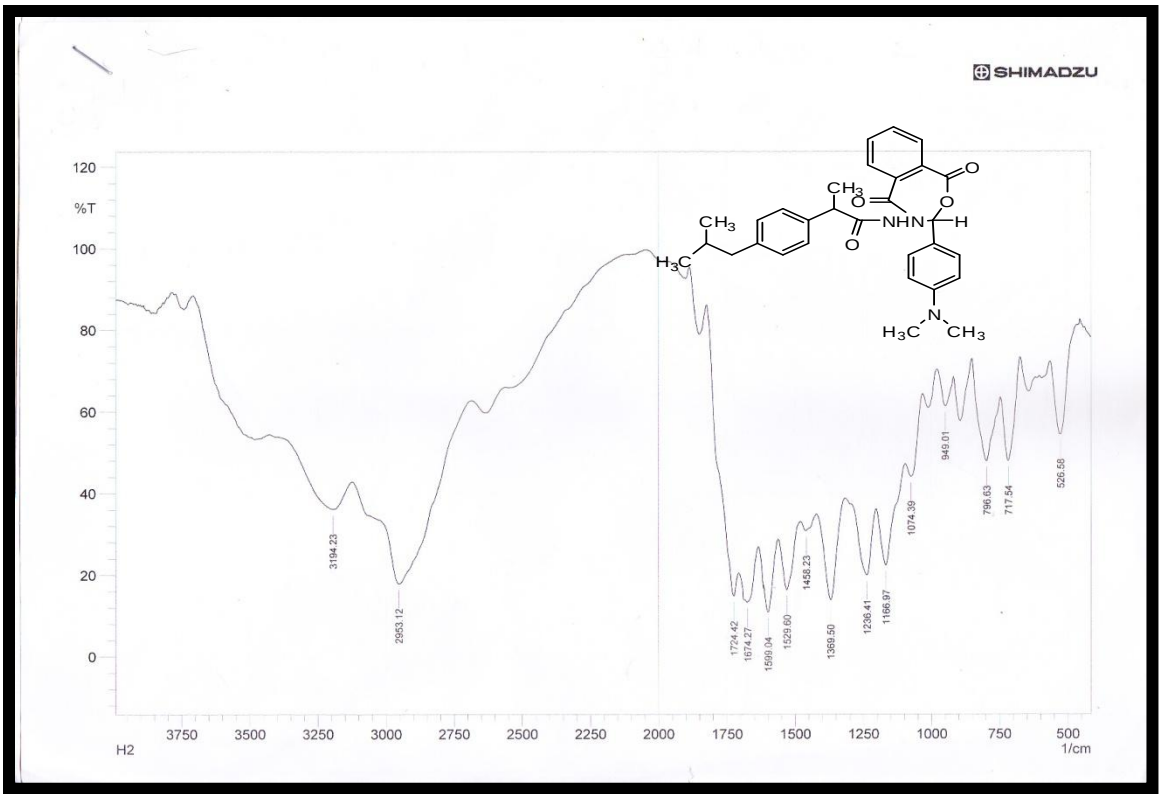
Figure(3) FT-IR Compound M2



Figure(4) FT-IR Compound A1



Figure(4) FT-IR Compound A2



Figure(5) FT-IR Compound N2

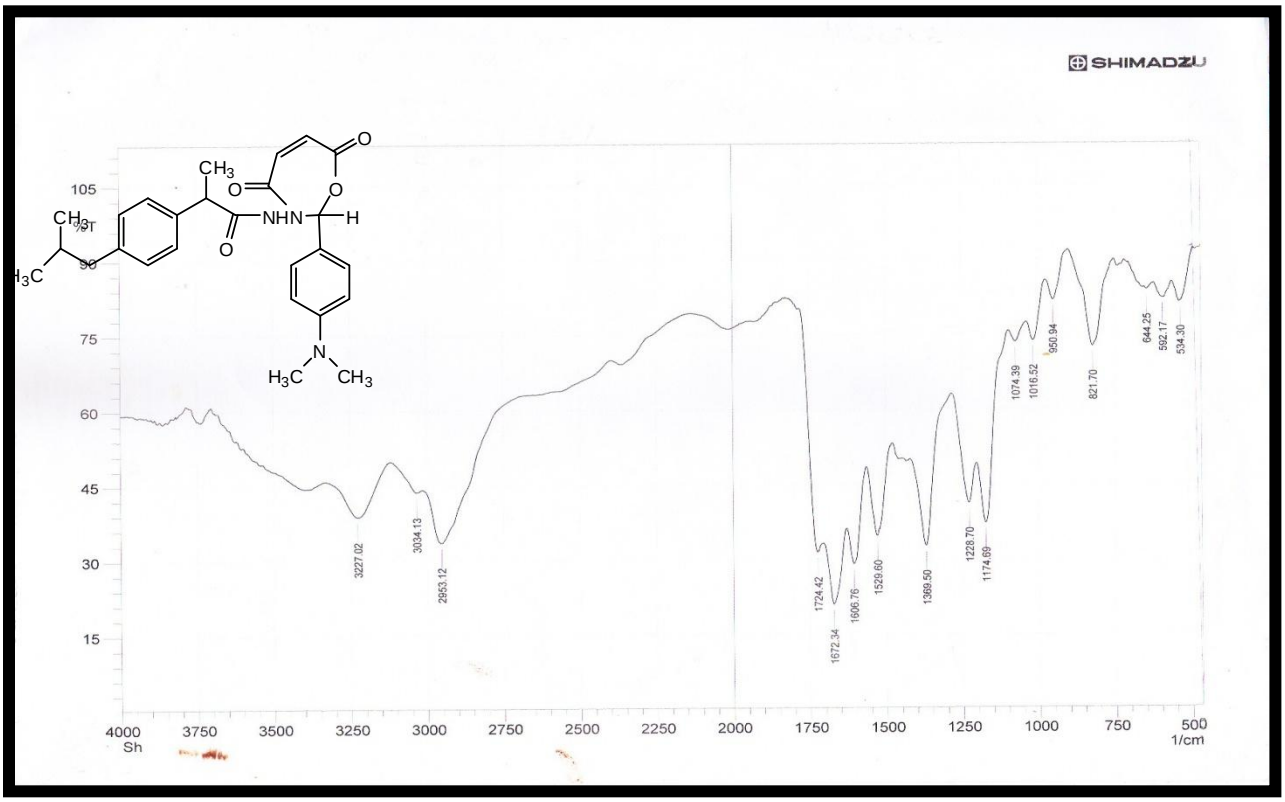


Figure (6) FT-IR Compound N1

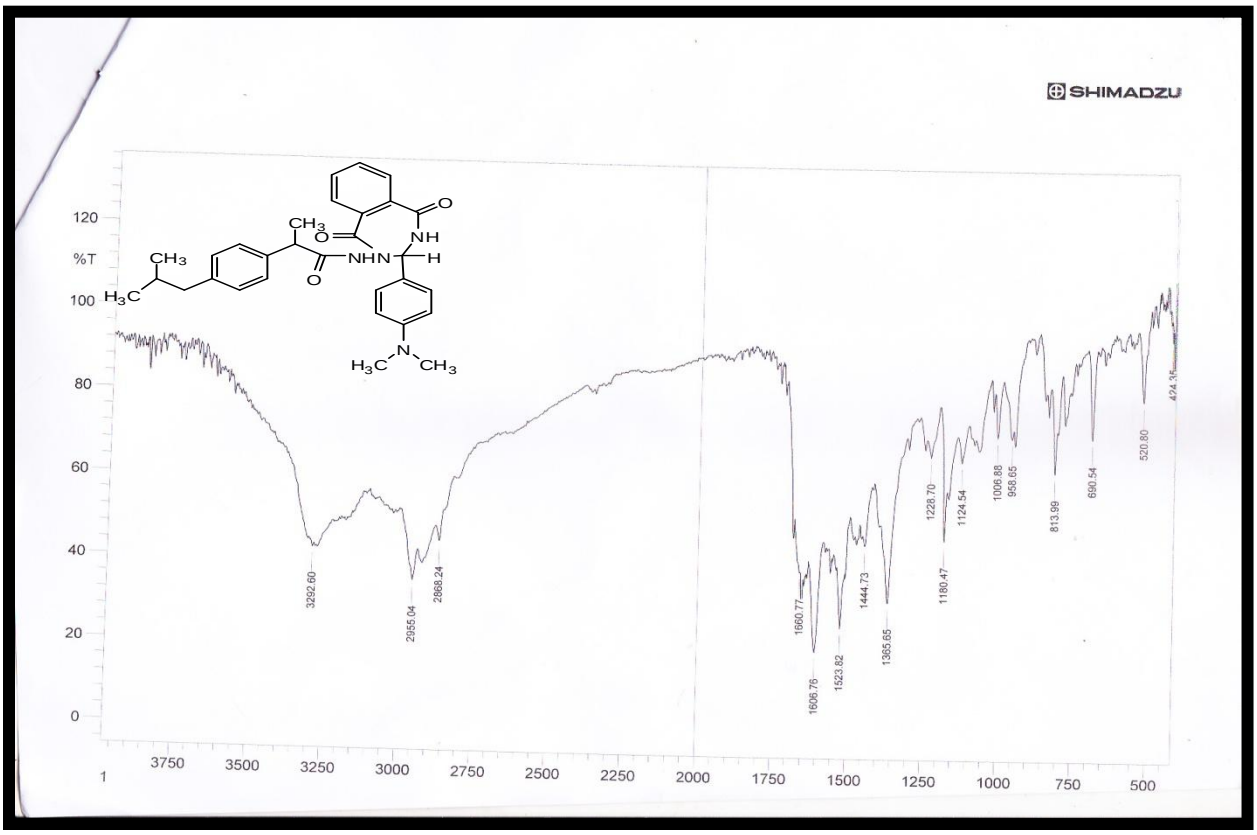


Figure (7) FT-IR Compound N2η (diazebin)

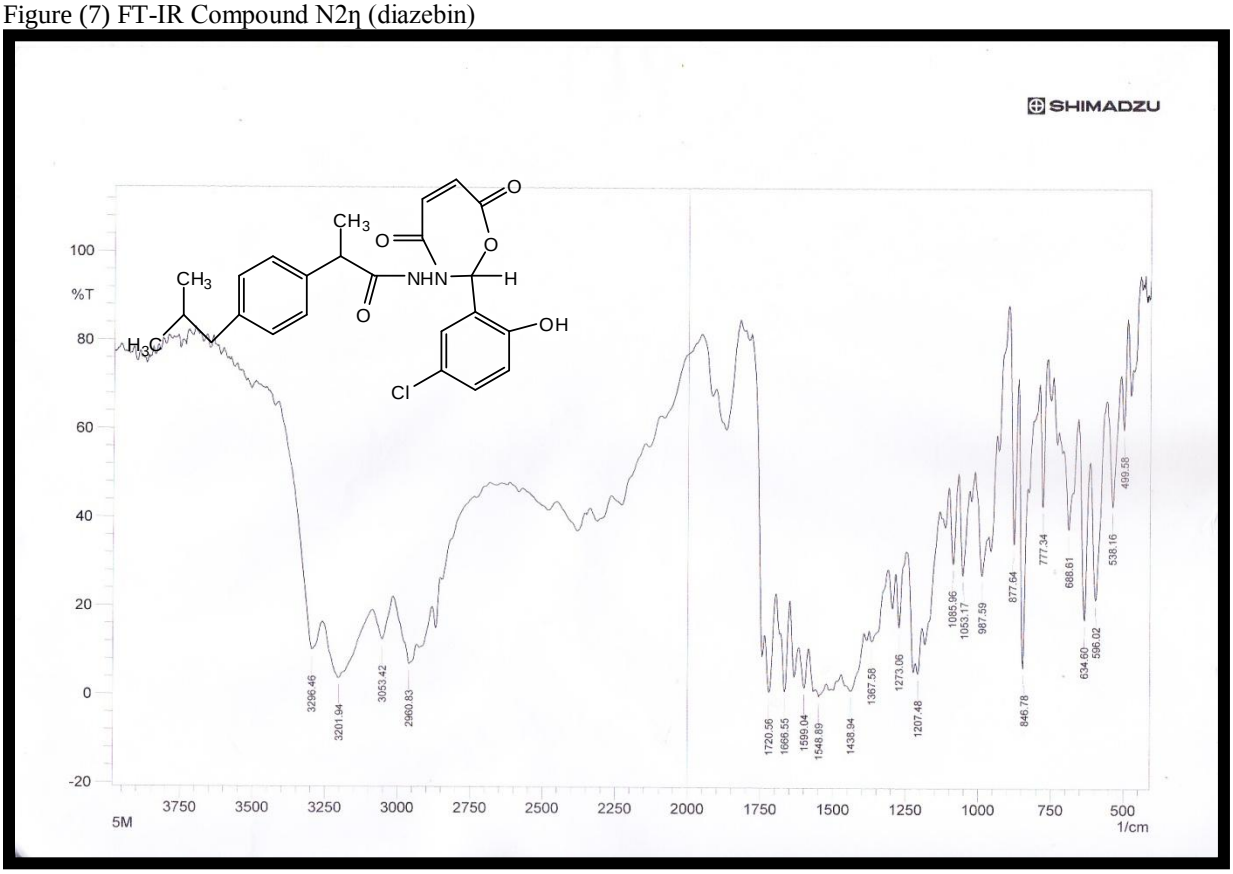


Figure (8) FT-IR Compound B1

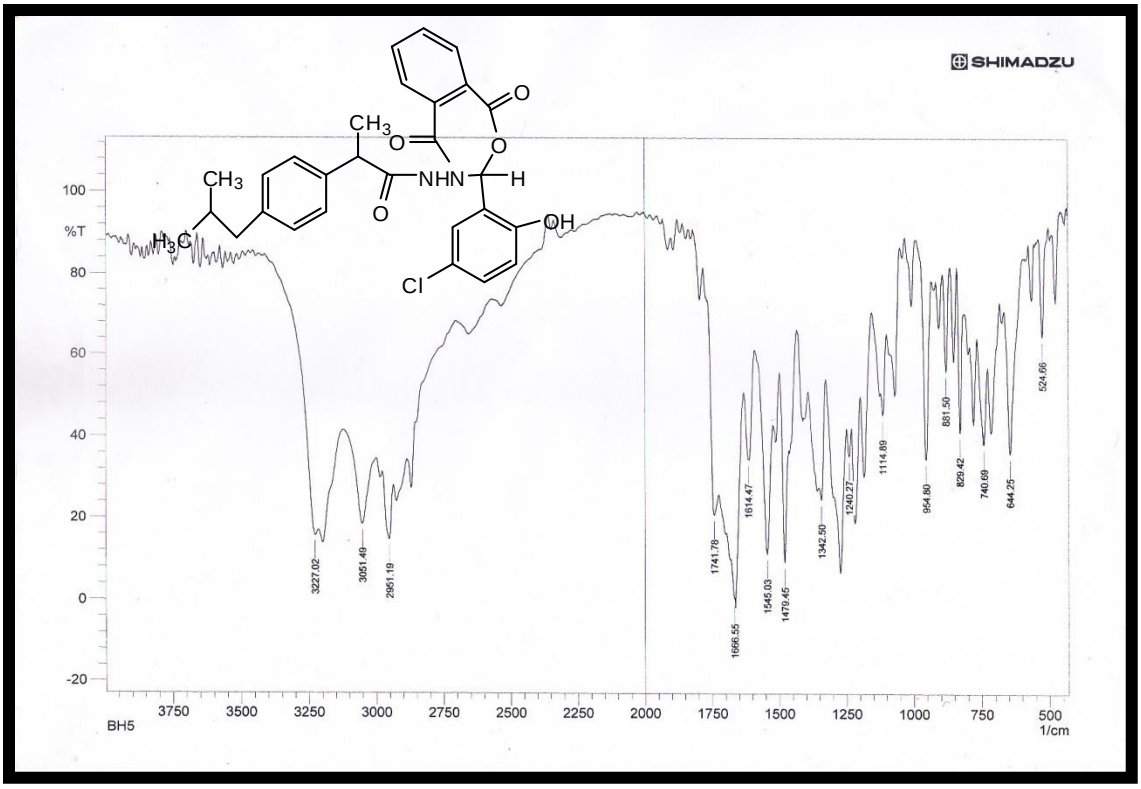
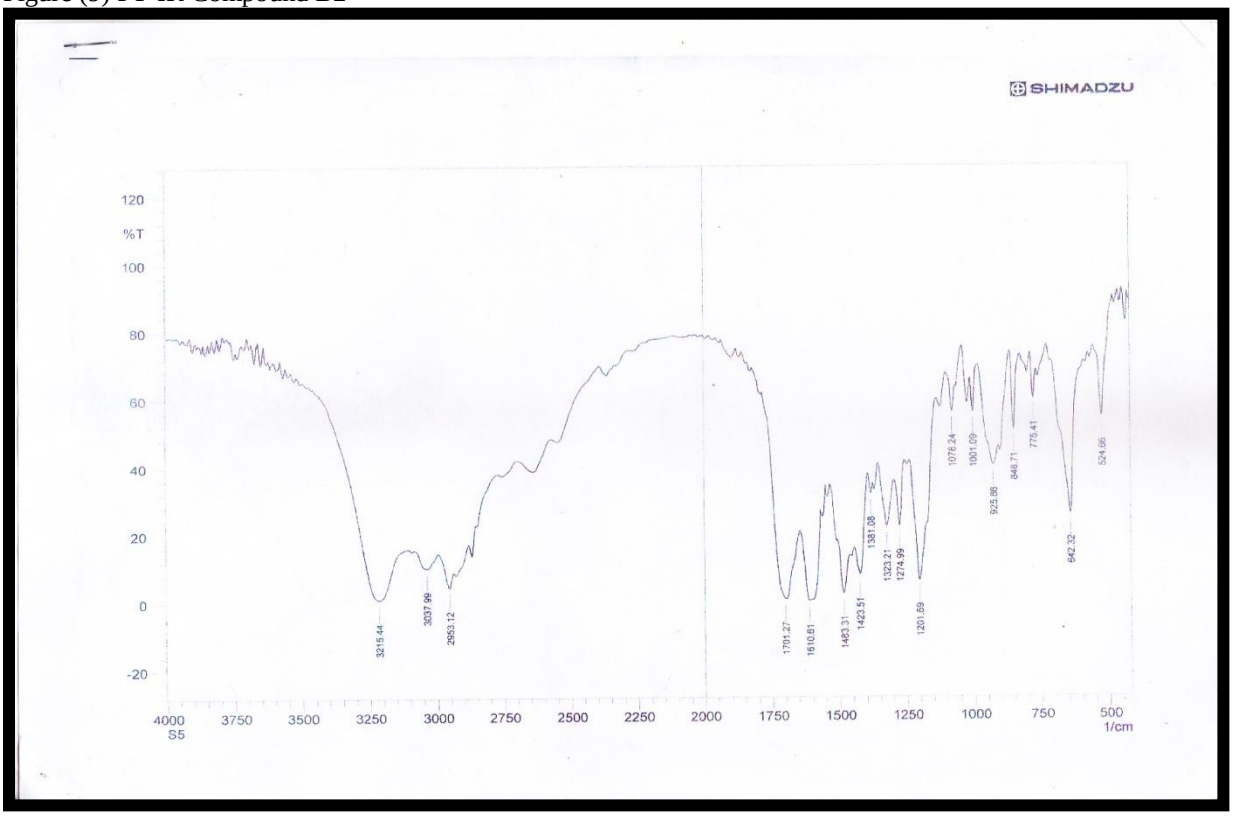


Figure (9) FT-IR Compound B2



Figure(10) FT-IR Compound B3

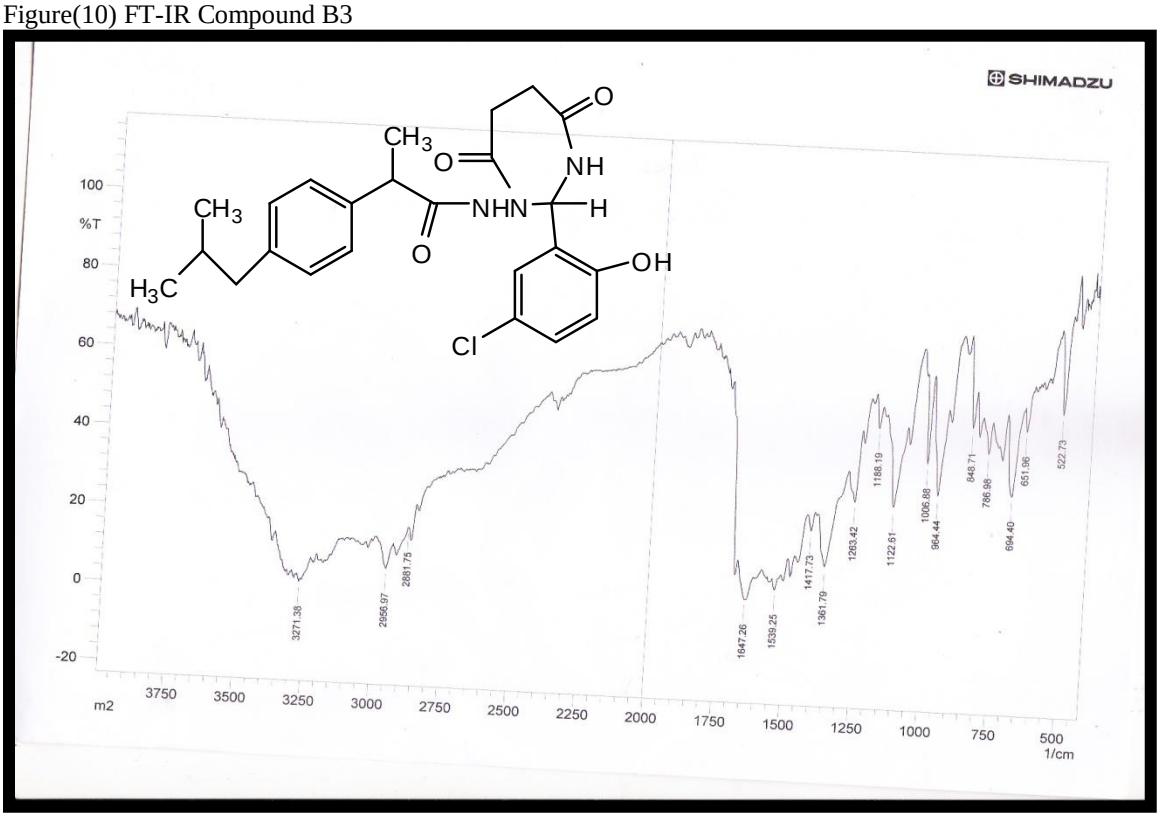


Figure (11) FT-IR Compound B2η

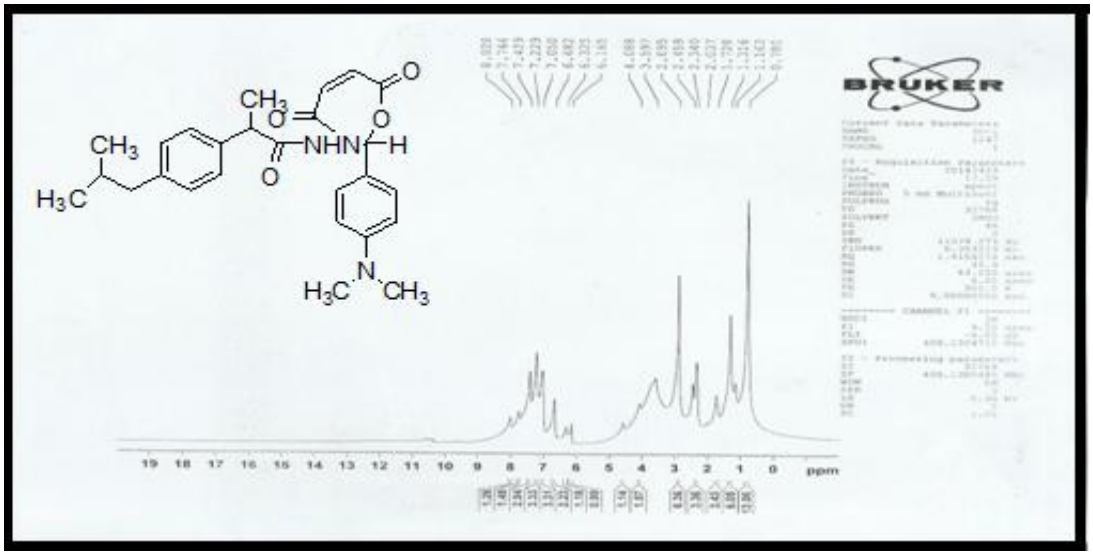


Figure (12) ¹H-NMR Compound N1

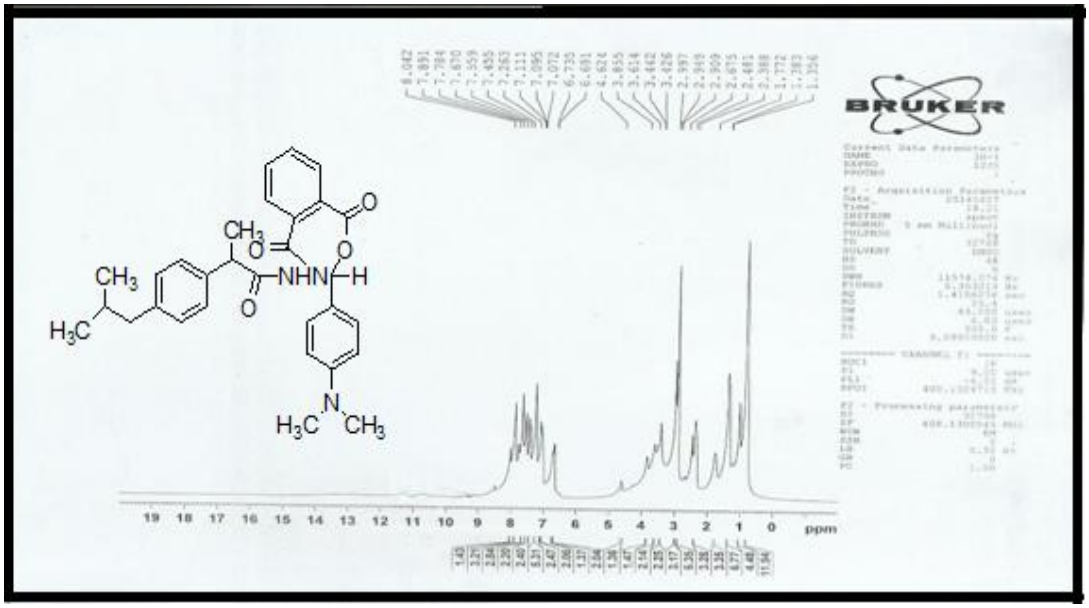


Figure (12) ¹H-NMR Compound N2

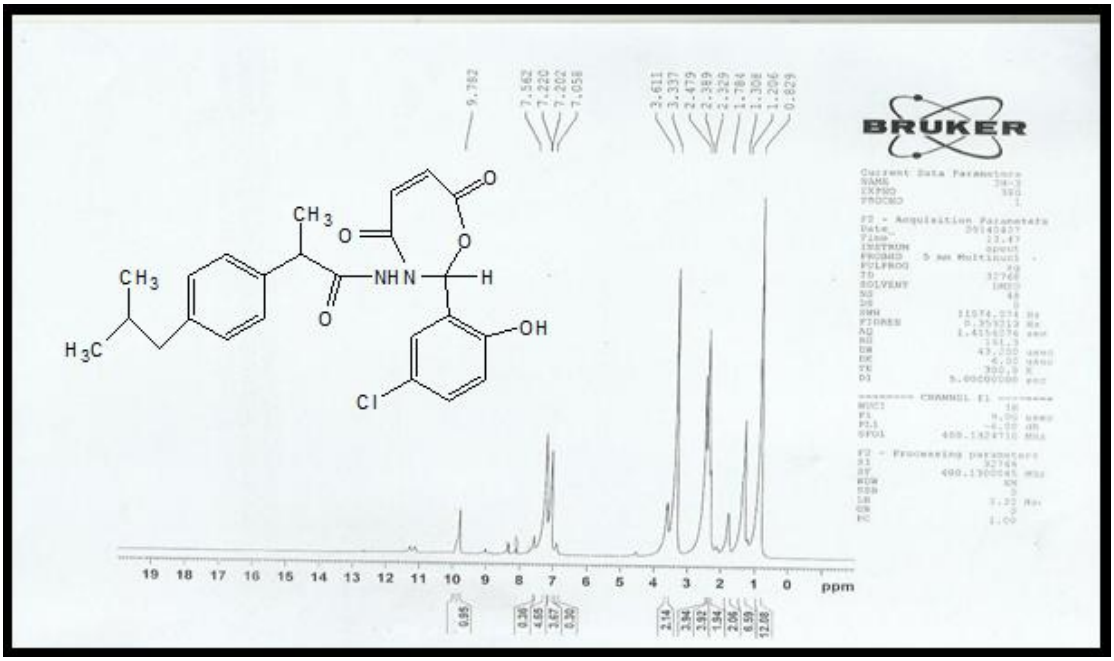


Figure (13) ¹H-NMR Compound B1

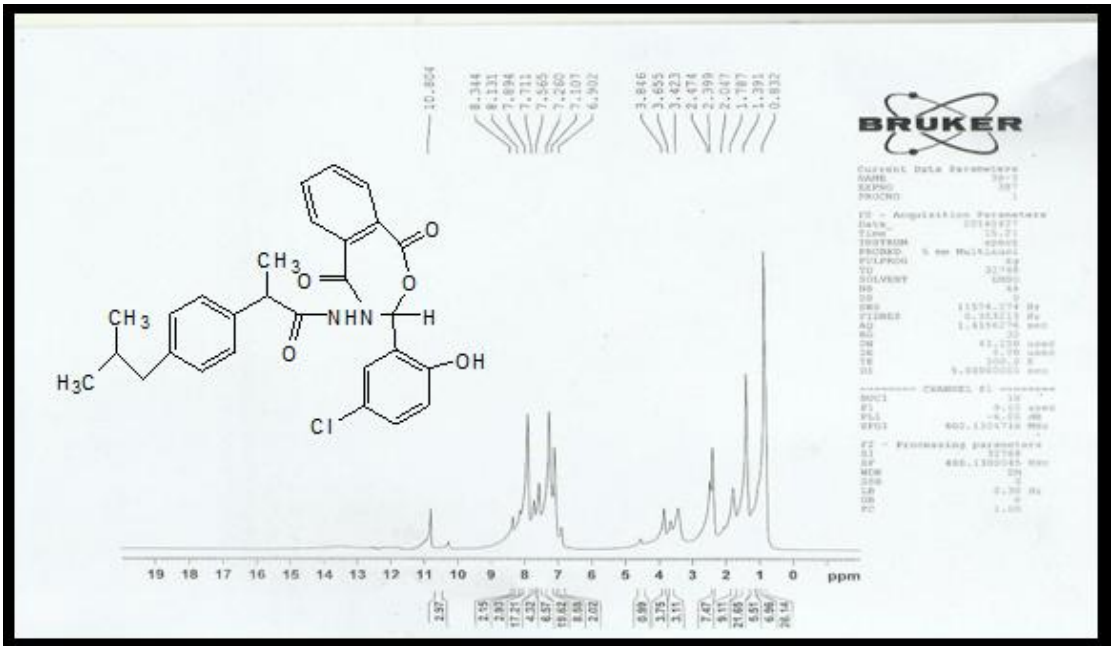


Figure (14) ¹H-NMR Compound B2

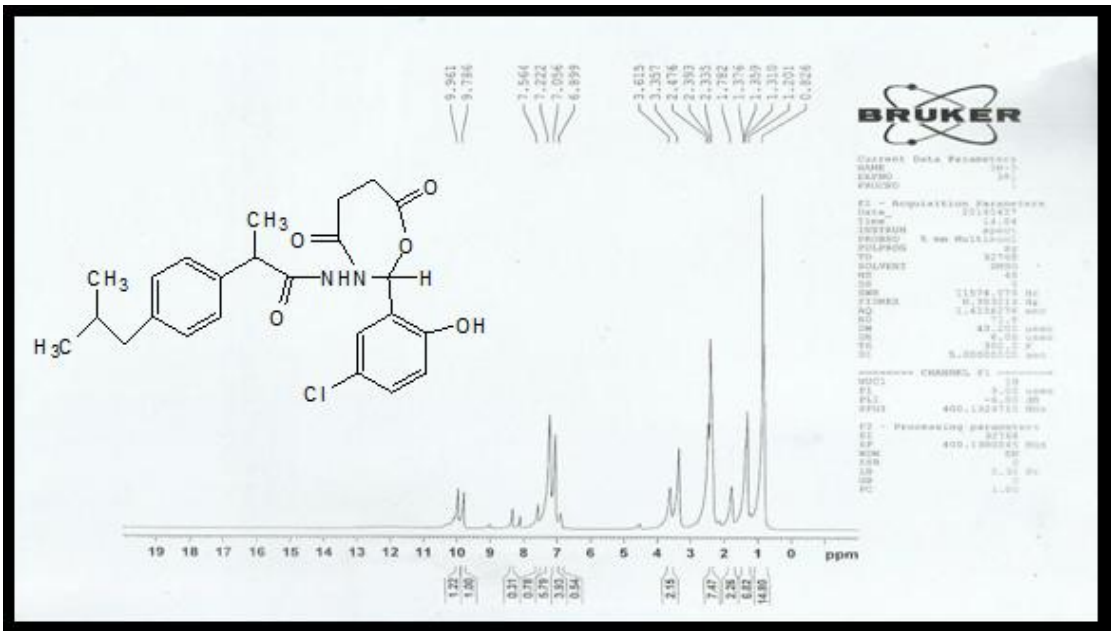


Figure (15) ¹H-NMR Compound B3

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