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*Synthesis , characterization 4,5- di phenyl imidazoles

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ABSTRACT:

A seven- member hetero cyclic compound were prepared by reaction 4,5- di phenyl imidazoles with P-amino aceto phenone give azo compound which after with reaction derivitives amines (3-nitro aniline, 3-amino phenol , 2- Bromo aniline 4- Nitro aniline , 4-Methoxey aniline , 2,4- di chloro aniline) which after with reaction phathalic anhydride to give 7-membered heterocyclic(7-12). And has follow–up of the interaction by thin layar chromatography , melting point, FT-IR ,C.H.N anylasis.

Introduction:

Imidazole was planar, five membered hetero aromatic molecular having two nitrogen named first as gluoxaline (first synthesis with glyoxal and ammonia) amphoteric nature , susceptible to electrophilic and nucleophilic attack.high stability to thermal, acid , base, oxidation and reduction conditions extensive intramolecular hydrogen bonding [(1)] various substitutes 4,5-di phenyl imidazoles derivatives have also been found to posses important activities such as anti helmintic[(2)] anti-inflammatory[(3)],antioxidants[(4)] ,antibacterial [(5)], antimicrobial[(6)], anticancer[(7)], antifungal[(8)], andanalgesic[(9)].

Schiff bases are reported to show avarity of interesting biological activity including[(10)],antimicrobial [(11)],antioxidant[(12)] ,antibacterial[(13)] ,anti – inflammatory[(14)]

Oxazapine aseven-memberd ring systems have reviewed cyclo addition reaction of alkenes , the cyclic transation state must correspound to one arrangement of the participating orpitals that can maintain a bonding interaction between the reaction components [(15)]

Experimental

- All chemicals used were supplied from merk and BDH- chemical company
- All measurement were carried out by:
- Melting point: Electrothermal , melting point 9300-U.K
- FT-IR spectra: tests can Shimadzu(FT-IR 8000 series, japan)
- Elemental analysis Eurorectro , EA 3000, Italy

The Research is apart of on MSC. Thesis in the case of the First researcher

Synthesis of Schiff bases (1-6)

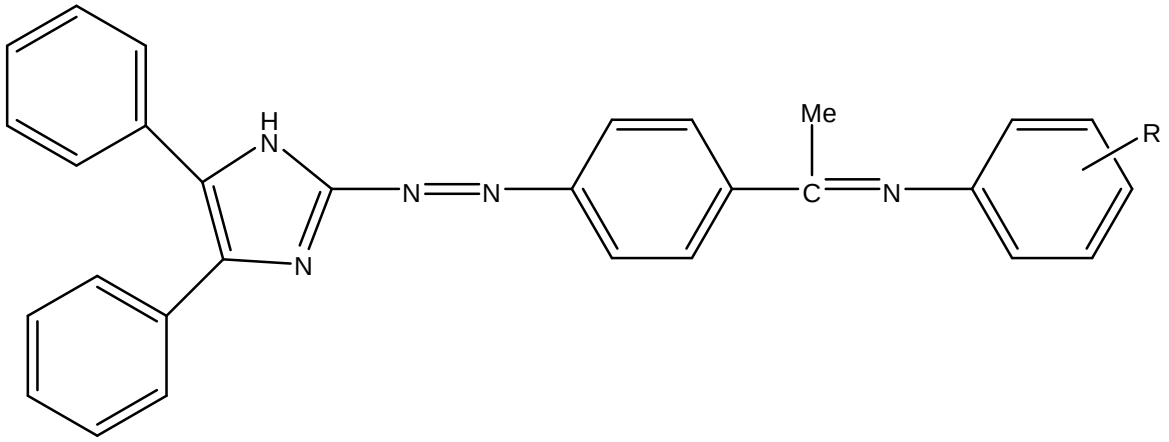
Aseries of Schiff bases were prepared from reaction (0.01 mol) of (0.5 gm) of azo 4,5-di phenyl imidazole and aniline derivatives (3-Nitro aniline,3-Hydroxy anilin, 2-Bromo anilin,4 - Nitro aniline,4-Methoxy aniline,2,4-di Chloro aniline) (o.o1 mol) (0.2gm) in 25 ml of absolute Ethanol and (2-3) drops glycial acetic acid the resulting mixture were refluxed for (6-8)hr , the solid that separated on cooling were filtered off and dried.

Synthesis of oxazipine compounds (7-12)

Amixture of Schiff bases(1-6)(0.01 mol) with phathalic inhydride (0.01 mol) were heated under reflux for (14-16) hr (50Ċ) with dry Benzene (15ml) , the solid product precipite that separated up on cooling were filtered off and re crystallized from ethanol.

Result and disscassion:

The Schiff bases were prepared from azo 4,5- di phenyl imidazole and aniline derivatives at70Ċ in the presen absolute ethanol and the reactions were by TLC (MeOH: C_6 H_6)(1:4)



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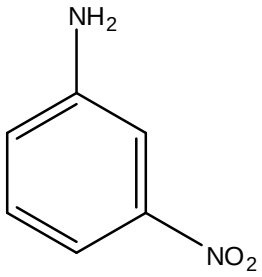
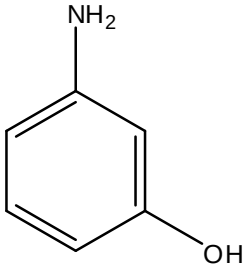
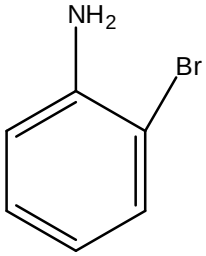


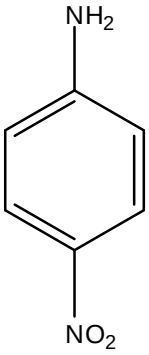
H_1



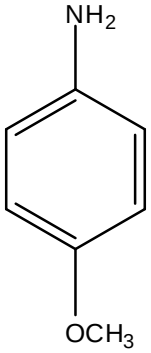
H_2



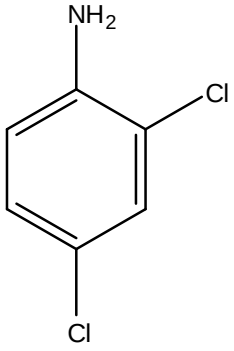
H_3



H_4



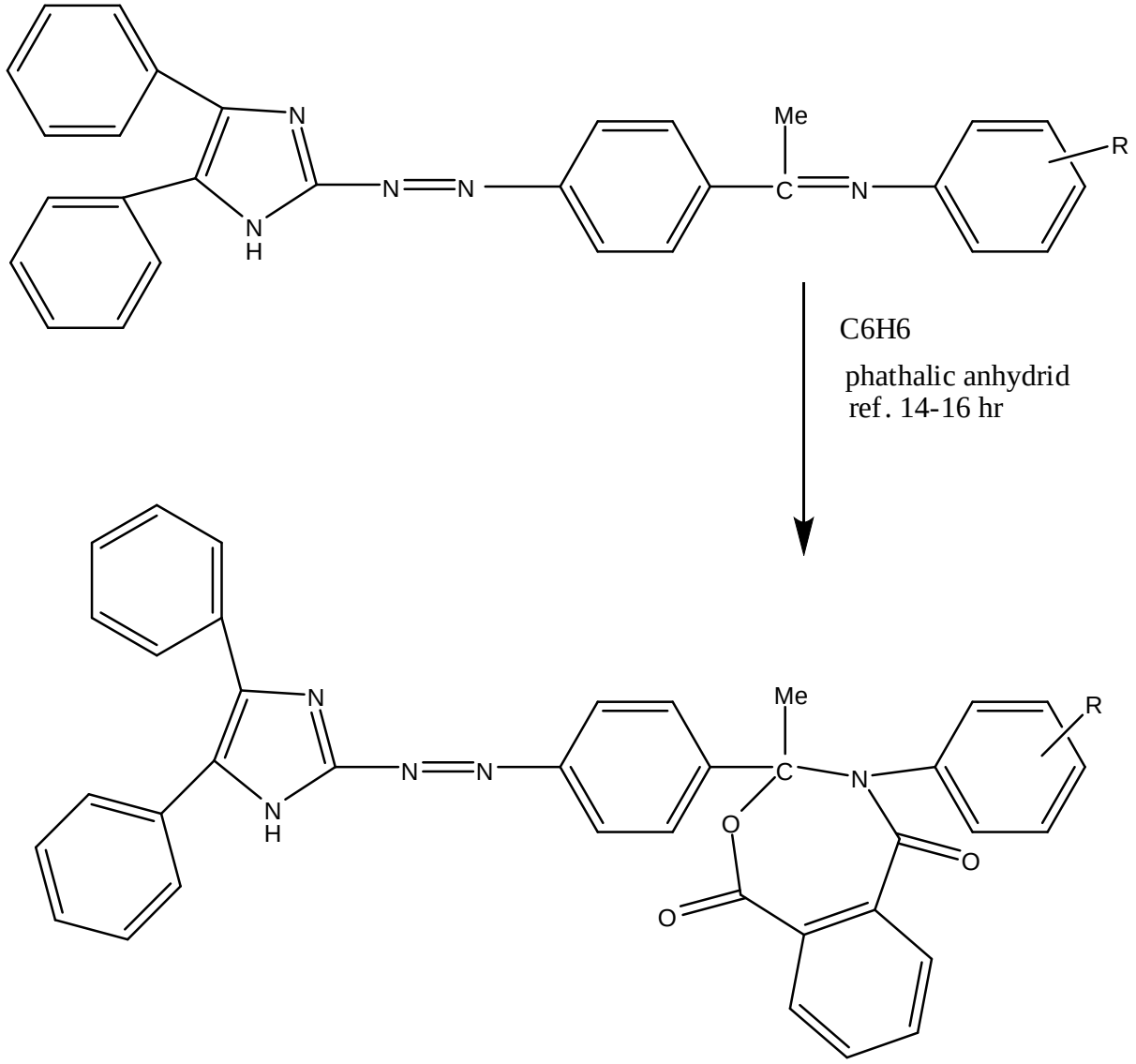
H_5



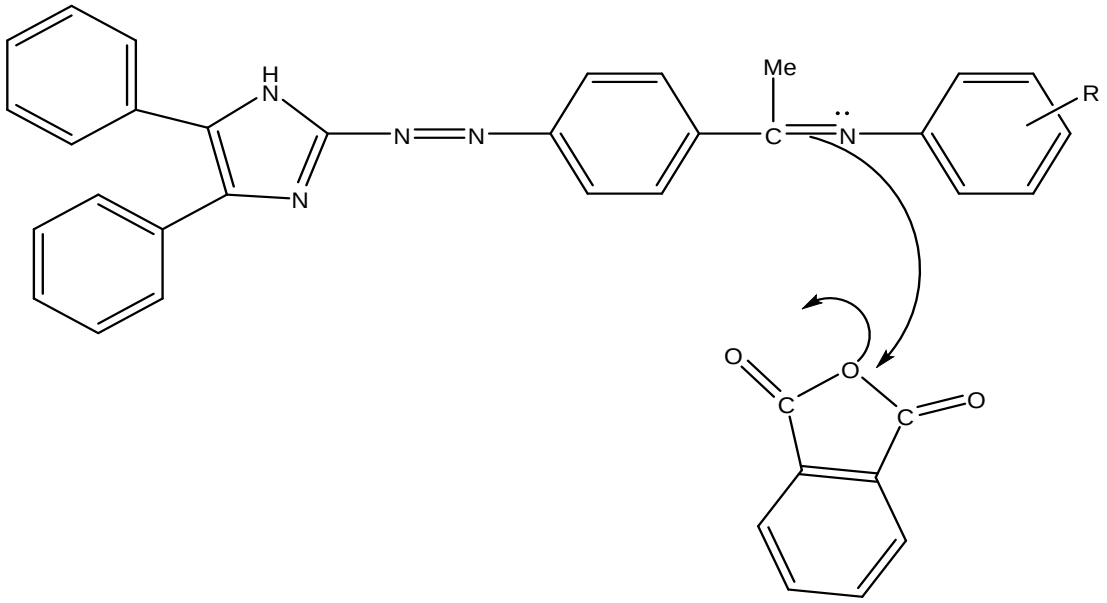
H_6

The oxazepine were prepared from Schiff bases and phathalic inhydride at 50Ċ in the dry

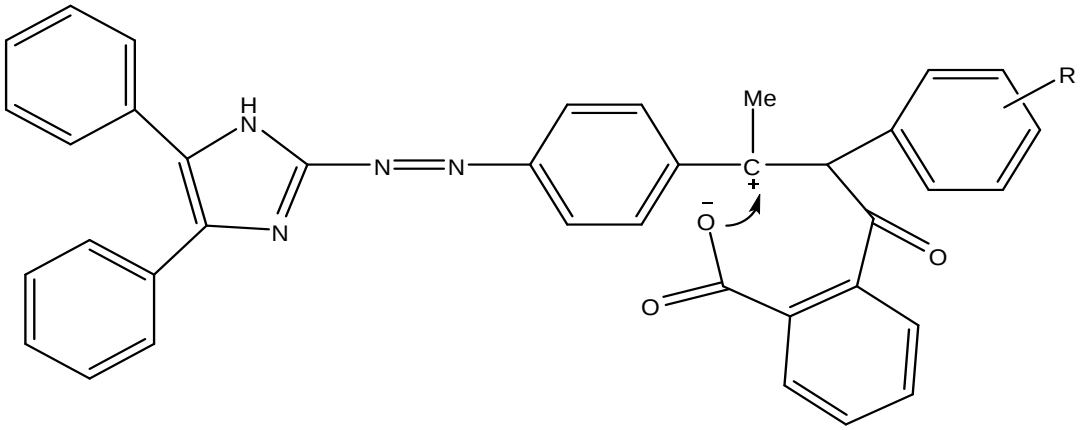
Benzen and the reaction were by TLC (MeOH: C6H6) (1:4)

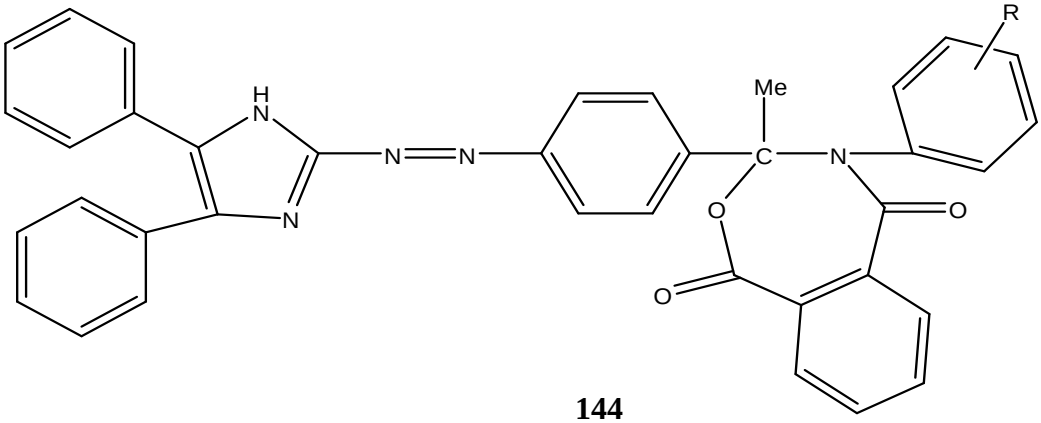


The mecanisim of oxazipine[16] :



Phath. inhyd.





FT-IR Spectra of schiff bases for compounds showed clear absorption bands at(3400-3200)Cm-1 to N-H from imidazole, (3060) cm-1 for C-H aromatic ,(1676-1680) cm-1 for(C=N) While this bands is disappear and two bands are appear at (1676-1722) cm-1 due to (lacton/ lactam) group of Oxazipine compounds are appear on band at (1681) cm-1 due to lactam group.

Tabel(1)- FT-IR data (cm-1)in KBr of compounds [1-12]

Comp. No.	Molecular Formula	(C-H) Str. Aromatic	(C-H) Str. aliphatic	(C=N) Imine group	(C=O) Str. Lactone/ Lactam	(N-H) imidazole	(AZO)	(C=C) aromatic	Other band appear refer to(R) group
1	m.$C_{29}H_{22}N_6O_2$	3059 m	2856 w	1680s	-	3381m	1510m	1597s	1359-1555m
2	$C_{29}H_{23}N_5O$	3057m	2850w	1680s	-	3383m	1508m	1597s	3383m
3	$C_{29}H_{22}N_5Br$	3059m	2880w	1680s	-	3360m	1485m	1599s	696s
4	P.$C_{29}H_{22}N_6O_2$	3060m	2900w	1676m	-	3326m	1631m	1662m	1398-1577m
5	$C_{30}H_{25}N_5O$	3059m	2935w	1680s	-	3400m	1504m	1597s	1074m
6	$C_{29}H_{21}N_5Cl_2$	3059m	2900w	1680s	-	3150m	1485m	1597s	700s
7	m-$C_{37}H_{26}N_6O_5$	3066m	2860w	-	1708m 1670m	3302m	1606m	1653m	1354-1558m
8	$C_{37}H_{27}N_5O_4$	3063m	2870w	-	1716m 1681m	3250m	1600m	1651m	3250m
9	$C_{37}H_{26}N_5BrO_3$	3061m	2900w	-	1718s 1670s	3389w	1516m	1595s	698s
10	P-$C_{37}H_{26}N_6O_5$	3070m	2900w	-	1710m 1660s	3250m	1600m	1620w	1360-1550w
11	$C_{38}H_{29}N_5O_4$	3070m	2972w	-	1718s 1680s	3408s	1597s	1651m	1109-1388m
12	$C_{37}H_{25}N_5Cl_2O_3$	3061m	2900w	-	1722s 1681s	3425w	1581w	1597m	765m

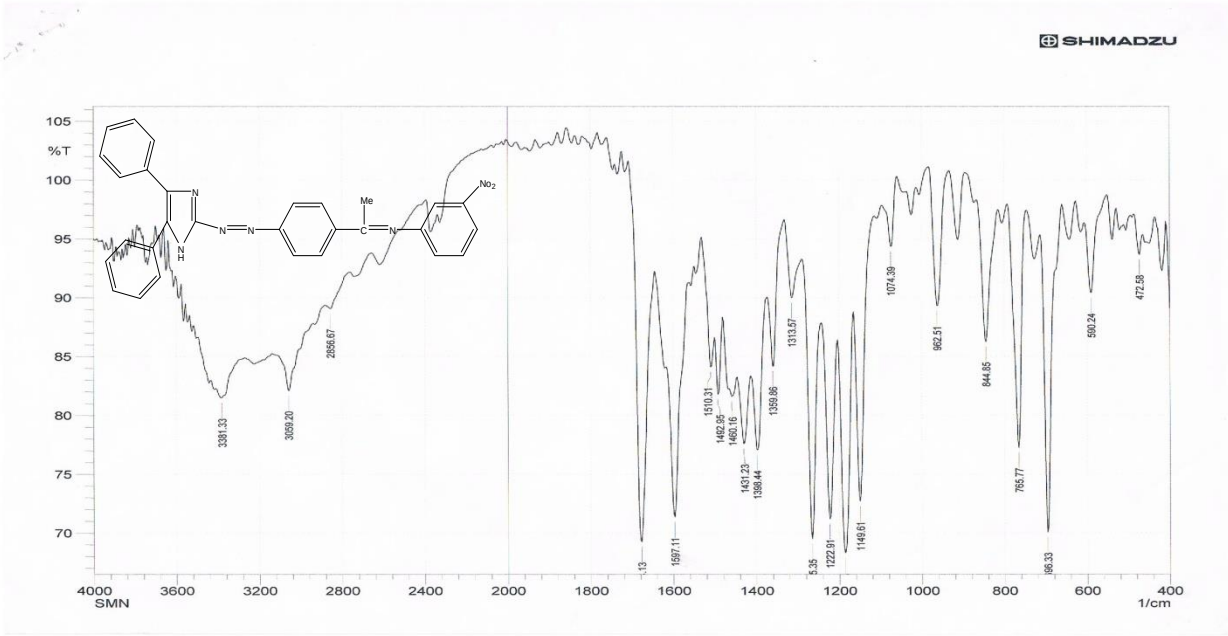
w=weak m=medium s=strong

Tabel (2). physical properties of compounds (1-12)

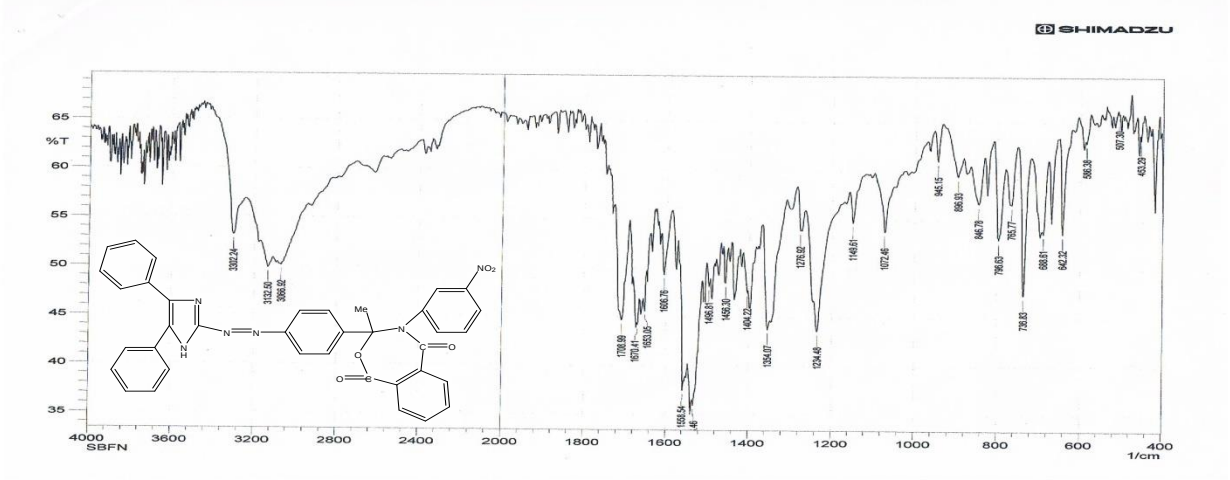
Comp. No.	Molecular formula	Mp Ċ	Colour	Purfication Solevent	Yield%
1	m.C_{29} H_{22} N_6 O_2	192-194	Brown	Ethanol	94.94
2	C_{29} H_{23} N_5 O	81decomp.	Dark-brown	-	88.46
3	C_{29} H_{22} N_5 Br	61-63	Dark-red	-	66.9
4	P.C_{29} H_{22} N_6 O_2	77-75	Dark- brown	-	91.44
5	C_{30} H_{25} N_5 O	80-82	Dark-red	-	82.97
6	C_{29} H_{21} N_5 Cl_2	66-68	brown	-	77.375
7	m-C_{37} H_{26} N_6 O_5	228-230	yellow	-	56.45
8	C_{37} H_{27} N_5 O_4	109-111	Dark-brown	-	87.19
9	C_{37} H_{26} N_5 BrO_3	78decomp	Dark-red	-	84.36
10	P-C_{37} H_{26} N_6 O_5	102-104	brown	-	81.27
11	C_{38} H_{29} N_5 O_4	130-132	Dark red	-	76.49
12	C_{37} H_{25} N_5 Cl_2 O_3	91-93	Dark red	-	90.269

Table (3).Elemental Analysis of compound

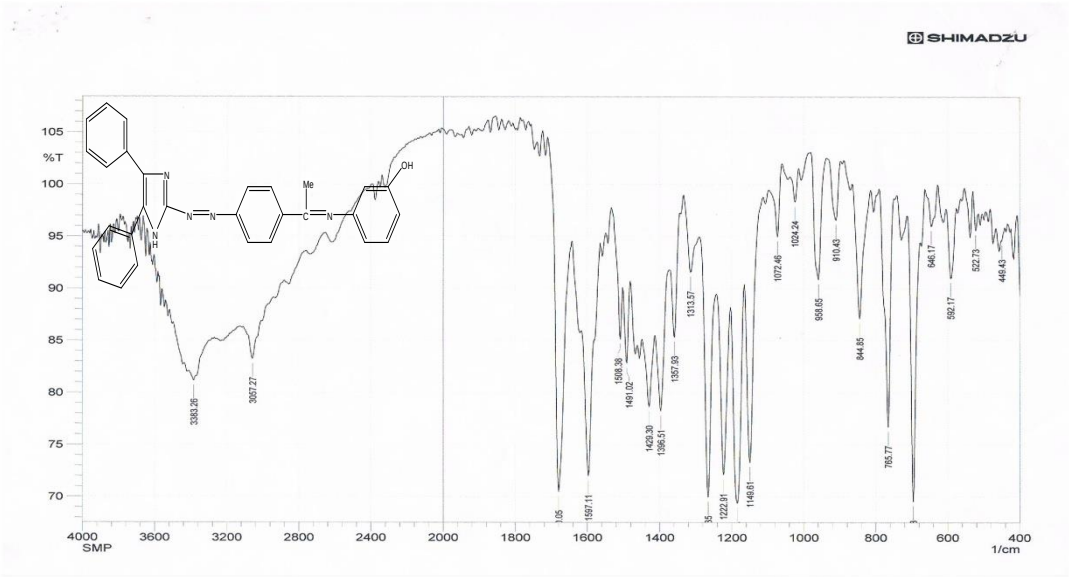
Comp. NO	M.F	Cal./Found C%	H%	N%
1	$C_{29}H_{22}N_6O_2$	71.604 71.428	4.526 4.501	17.283 17.252
2	$C_{29}H_{23}N_5O$	76.148 76.497	5.032 5.093	15.317 15.407
8	$C_{37}H_{27}N_5O_4$	73.388 73.371	4.462 4.381	11.570 11.465
10	$C_{37}H_{26}N_6O_5$	70.031 70.054	4.100 4.068	13.249 13.189
12	$C_{37}H_{25}N_5Cl_2O_3$	67.477 67.135	3.799 3.712	10.638 10.546



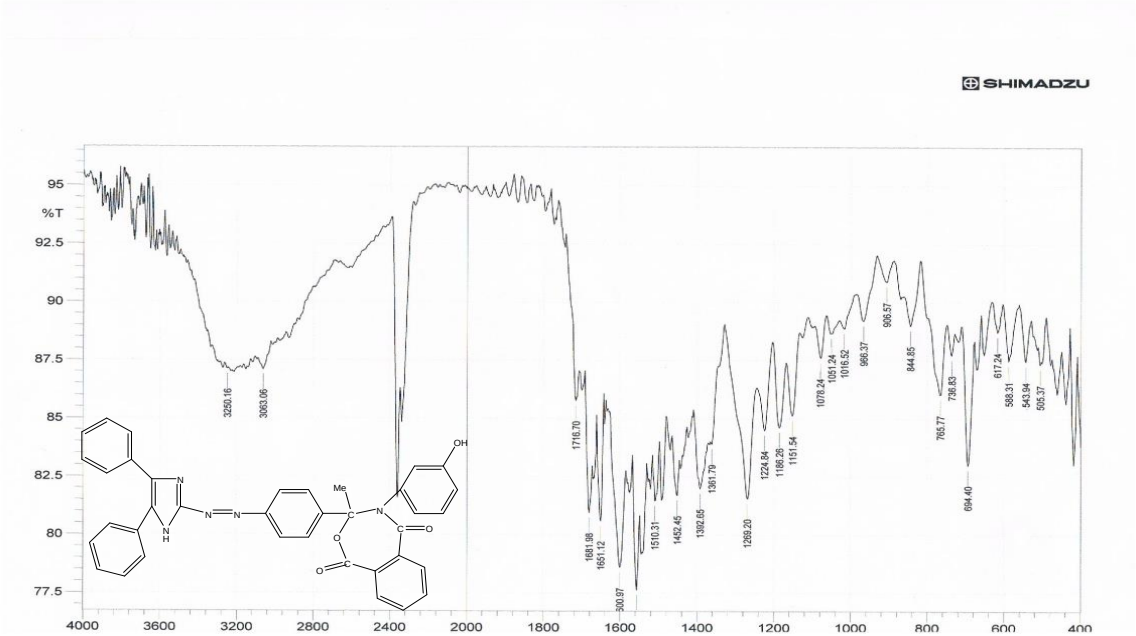
FT-IR Spctra of compound(1)



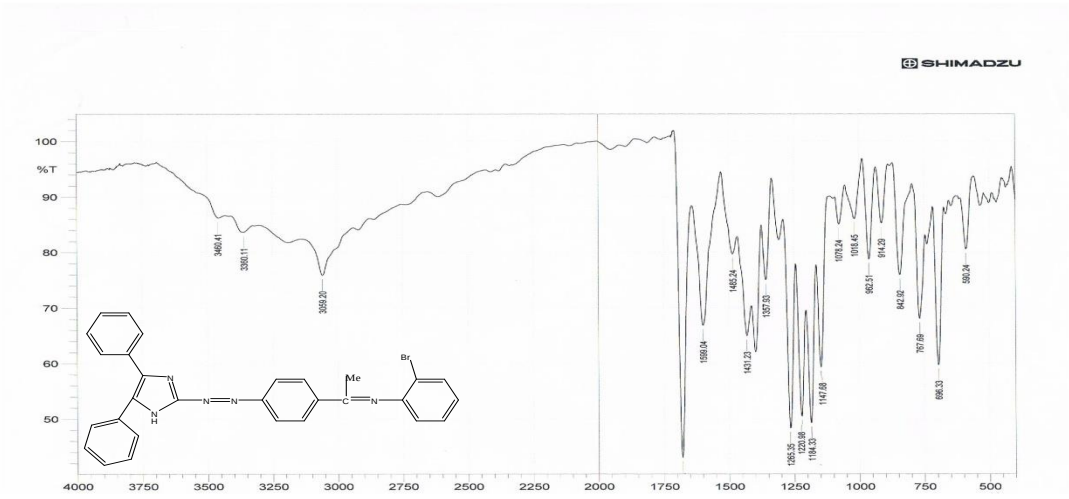
FT-IR Spctra of compound(7)



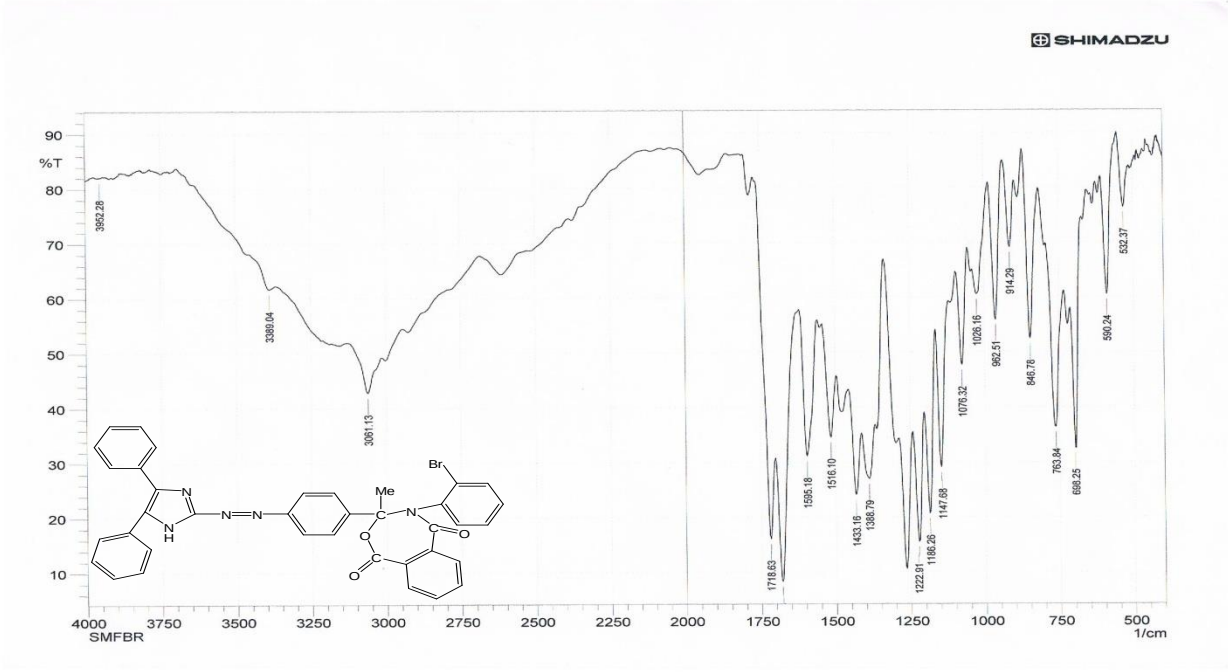
FT-IR Spctra of compound(2)



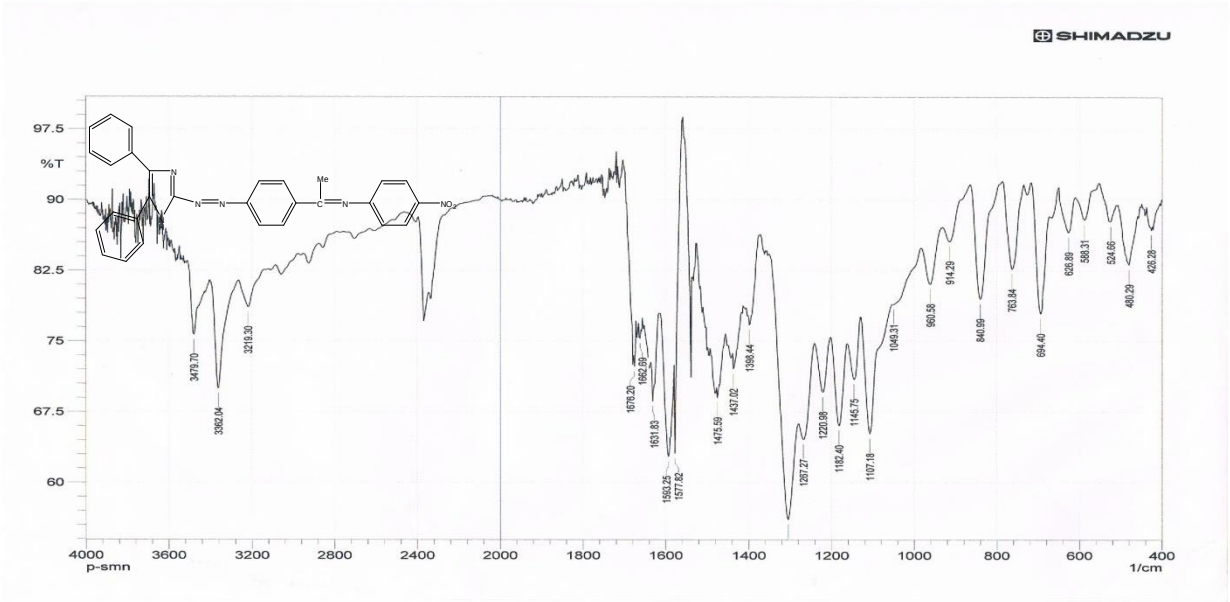
FT-IR Spctra of compound(8)



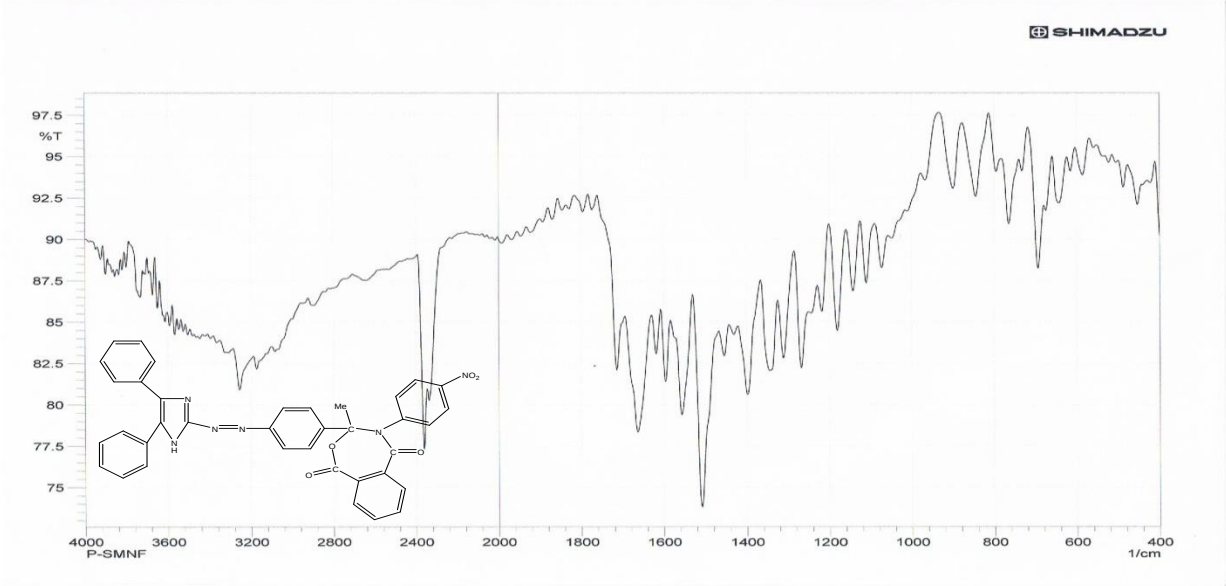
FT-IR Spctra of compound(3)



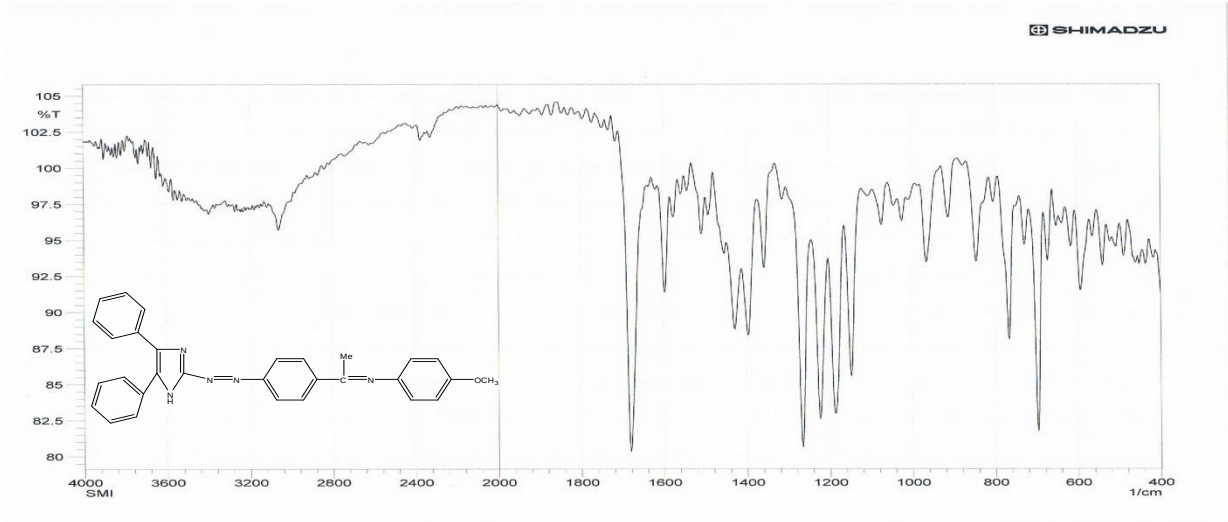
FT-IR Spctra of compound(9)



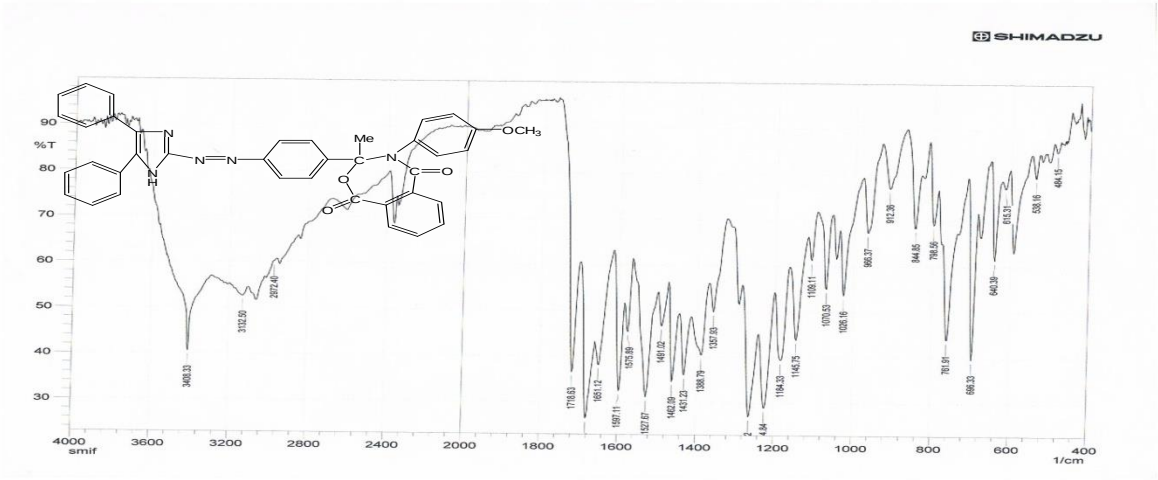
FT-IR Spctra of compound(4)



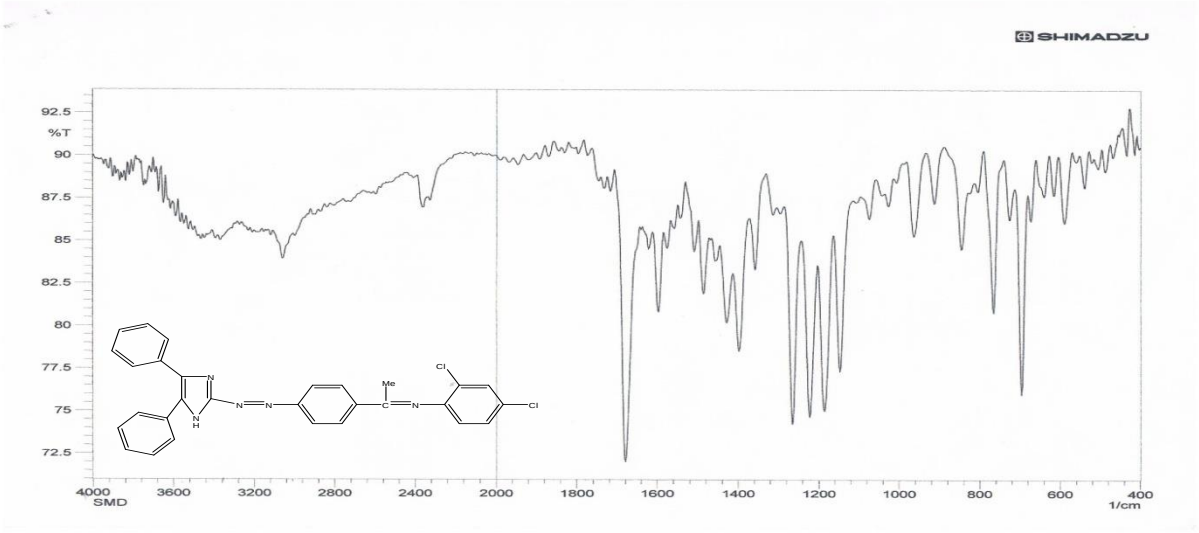
FT-IR Spctra of compound(10)



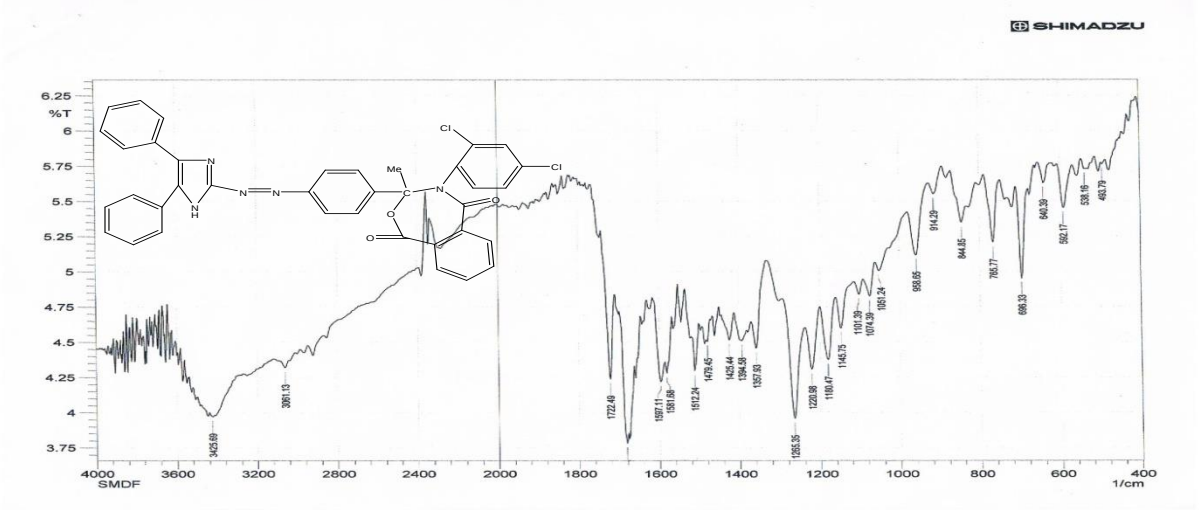
FT-IR Spctra of compound(5)



FT-IR Spctra of compound(11)



FT-IR Spctra of compound(6)



FT-IR Spctra of compound(12)

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*تحضير مركبات سباعية الحلقة 4,5 داي فينيل ايميدازول

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تاريخ الاستلام:9\6\2013 **تاريخ القبول:4\7\2013**

الخلاصة

تم تحضير مركبات سباعية الحلقة غير متجانسة وذلك من خلال تفاعل 4,5-داي فنيل ايميدازول مع البارا- امينو اسيتوفينون اعطى مركب الازو ومن ثم فوعل مع امينات (3-نايتروانلين ، 3-امينو فينول ، 2-بروموانلين ، 4-نايترو انلين ،4-ميثوكسي انلين ،4,2-داي كلورو انلين) بعد ذلك فوعل مع انهيدريد الفثالك فاعطى مركبات سباعية الحلقة غير متجانسة (7-12) وتمت متابعة سير التفاعل بوساطة تقنية كروموتوغرافيا الطبقة الرقيقة ثم شخصت هذة المركبات بوساطة درجات الانصهار وطيف الاشعة تحت الحمراء وطيف الرنين النووي المغناطيسي وكذلك التحليل الدقيق للعناصر.

***البحث مستل من رسالة ماجستير للباحث الاول .**