Kirkuk University Journal /Scientific Studies (KUJSS)
Volume 11, Issue 3, September 2016 , p.p(254-262)
ISSN 1992 – 0849

Synthesis of some new Pyrazoline derivatives derived from Ibuprofen

Khalid A. Al-badrany[1] , Ali- I. Al-jobury[2] , Amar .S. Mohammed[3]

[1]chemistry Department /college of education for pure Science / Tikrit university

khalidaziz382@gmail.com[1]

[٢،٣]biology Department / college of education for pure Science / Kirkuk university

ali ibrahem5261@yahoo.com[2] , aamrr 1970@yahoo.com[3]

Received date : 18 / 1 / 2016 **Accepted date : 22 / 8 / 2016**

ABSTRACT

Ethyl 2-(4-isobutylphenyl) propanoate (A_1), was synthesized from 2-(4-isobutylphenyl)propanoic acid and the reaction of compound (A1) with hydrazine hydrate in ethanol gave 2-(isobutylphenyl) propanhydrazide (A2) the second step in this work were synthesized 2-aryliden eindan-1,3-dione(A3-8) From the condensation of aromatic aldehyde with eindandione to gain six novel compounds from the α-β-unsaturated compound Then refluxed with the acid hydrazide (A2) to gave the pyrazoline derivative (A9-13). The structure of the synthesized compounds are confirmed by CHN analyses . I.R, ^{1}H-NMR & ^{13}C-NMR spectra and Some chemical physical data.

Keywords: *pyrazolin/galkons/eindandione*

Kirkuk University Journal /Scientific Studies (KUJSS)
Volume 11, Issue 3, September 2016 , p.p(254-262)
ISSN 1992 – 0849

تحضير بعض مشتقات البايرازولين الجديدة المشتقة من الايبوبروفين

خالد عبدالعزيز عطية البدراني[١] ، علي ابراهيم مصطفى الجبوري[٢] ، عمار سعدالدين محمد[٣]

[١]قسم الكيمياء / كلية التربية للعلوم الصرفة / جامعة تكريت

khalidaziz382@gmail.com[1]

[2,3]قسم علوم الحياة / كلية التربية للعلوم الصرفة / جامعة كركوك

ali ibrahem5261@yahoo.com[2] , aamrr 1970@yahoo.com[3]

تاريخ استلام البحث: ١٨ / ١ / ٢٠١٦ **تاريخ قبول البحث: ٢٢ / ٨ / ٢٠١٦**

الملخص

تم تحضير اثيل -٢-(٤-ايزوبروبيل فنيل) بروبانويت (١) من تفاعل حامض -٢-(٤-ايزوبروبيل فنيل) بروبانويك مع الايثانول بوجود حامض الكبريتيك المركز . تفاعل المركب (١) المحضر مع الهيدرازين المائي في الايثانول يعطي حامض -٢-(٤-ايزوبروبيل فنيل) بروبان هايدرازايد. الخطوة الثانية تحضير ٢-اريلدين اندان ١و٣-داي -اون (٣-٧) من تكاثف الالديهايدات الاروماتيه مع الاندان داي اون في وسط قاعدي . ثم تصعد حراريا مع الهيدرازايد(٢) لتعطي مشتقات البايرازولين . شخصت المركبات بالطرق الفيزياوية والطيفية لاثبات صحة التراكيب المحضرة.

الكلمات الدالة : البايرازولين / جالكونات / الاندان داي اون .

1. INTRODUCTION

The pyrazoline derivatives have various pharmacological activities such as antibacteria [1], antifungal [2], antioxdint [3] and anticancer [4] and antipyretic activity [5].by the way, it was found that α-β-unsaturated compound bases had antimicrobial[6,7] antiinflamnary [8,9,10,11,12],analgesic [13,14] antitumor activity [15] besides various other activities. In our previous work, we synthesized some

Kirkuk University Journal /Scientific Studies (KUJSS)
Volume 11, Issue 3, September 2016 , p.p(254-262)
ISSN 1992 – 0849

pyrazoline derivatives from the reaction of α-β-unsaturated compound with acid hydrazide.

2.Experimental part

Melting points were determined on Electrothermal. melting point Apparatus are uncorrected, and the IR absorption spectra were recorded by FTIR model 84005 Shimadzu Japan. Infrared spectrophotometer as KBr disk. ^{1}H-NMR & ^{13}C-NMR spectra were recorded by Ultra shield 300 MHz. Bruker 2003.al-albaet university Jordan .

2.1. preparation of ethyl 2-(4-isobutylphenyl) propanoate (A1):

2-(4-isobutylphenyl) propanoic acid (0.01 mole ,2.06gm) dissolved in 50 ml abs. ethanol in presence of conc. H_2SO_4 . the reaction mixture was refluxed for 6-7 hr after cooling the crud neutralized with dilute $NaHCO_3$. the oily layer extracted with diethyl ether the ester dried with $MgSO_4$, the product was recrystalized from ethanol to obtain yellow crystal m.p (50-51c^{o}) yield 75 %.

2.2. preparation of 2-(4-isobutylphenyl) propane acid hydrazyd (A2):

A mixture of ethyl 2-(4-isobutylphenyl)isopropanoate (A1) (0.001mol , 0.234gm) and (0.006 mol) from hydrazine hydrate 85% in 50 ml ethanol was refluxed for 24 hr . After cooling the product was precipitated as solid Then it was collected by vacuum suction filtration and the product was recrystalized from ethanol to obtain yellow crystal m.p (70-71c^{o}) yield 86 % .

2.3. preparation of 2-arylideneindan-1,3-dione(A3-8):

To a mixture of indan-1,3-dione (0.001 mole , 0.146gm)and benzyldehyd derivatives (0.001mole) in 20 ml abs. ethanol was added an alcoholic solution of NaOH (1%,10ml) the reaction mixture was stirred for (4-5hr) at room temperature , then it was neutralized with dilute HCl , the mixture was kept in refrigerator overnight. The product was precipitated as solid. Then it was collected by vacuum suction filtration and washed with warm water.The product was recrystalized from ethanol and dried at room temperature to obtain compounds (A3-8). The physical properties and CHN *anly*. Show in Table (1).

Kirkuk University Journal /Scientific Studies (KUJSS)
Volume 11, Issue 3, September 2016 , p.p(254-262)
ISSN 1992 – 0849

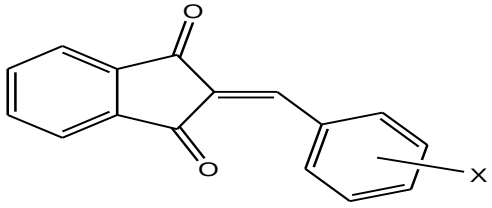


Table (1): physical properties of Compounds(A3-8)

No.	X	M.P C°	Yield %	Color	Molecular formula	CHN analysis % Calc / found		
						C%	H%	N%
A_3	2,4-Cl_2	194-195	80	Yellow	$C_{16}H_8Cl_2O_2$	63.36 63.24	2.64 2.56	33.99 34.20
A_4	4-$N(CH_3)_2$	205-206	79	Red	$C_{18}H_{15}NO_2$			
A_5	4-NO_2	229-231	84	Green	$C_{16}H_9NO_4$			
A_6	4-F	160-161	81	Green	$C_{16}H_9FO_2$			
A_7	4-Cl	176-177	70	Yellow	$C_{16}H_9ClO_2$			
A_8	H	150-151	60	Green	$C_{16}H_{10}O_2$	82.05 81.92	4.27 4.21	13.67 13.87

4. Preparation of 2-(4-isobutylphenyl)isopropan-3-Aryl-3-4dihydroindeno[3,2-C]pyrazol-4-one:-(A9-14)

A mixture of compound (A2) (0.001 mol) , and (0.001 mol) of substituted chalcon in AcOH 15 ml was reflexed for (4hr) . After cooling 60 ml of cold water was added in portions to the crud mixture reaction . The product was precipitated as solid,then collected by vacuum suction filtration and washed with warm water . The product was recrystalized from suitable solvent and dried at room temperature to offered compounds (A9-14) . the physical properties Show in Table (2).

Kirkuk University Journal /Scientific Studies (KUJSS)
Volume 11, Issue 3, September 2016 , p.p(254-262)
ISSN 1992 – 0849

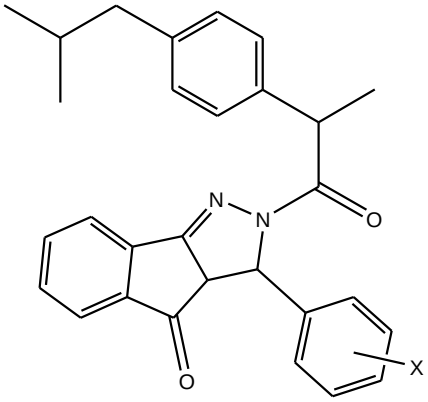


Table (2): physical properties of Compounds(A3-8)

No.	X	M.P C°	Yield %	Color	Molecular formula
A_9	2,4-Cl_2	179-180	30	Orange	$C_{29}H_{26}Cl_2N_2O_2$
A_{10}	4-$N(CH_3)_2$	188d	50	Green	$C_{31}H_{33}N_3O_2$
A_{11}	4-NO_2	201-202	34	Yellow	$C_{29}H_{27}N_3O_4$
A_{12}	4-F	199-201	35	Brawn	$C_{29}H_{27}FN_2O_2$
A_{13}	4-Cl	160-161	48	Yellow	$C_{29}H_{27}ClN_2O_2$
A_{14}	H	220d	55	Yellow	$C_{29}H_{28}N_2O_2$

5. Result and discussion

Etheyl 2-(4-isobutylphenyl) propanoate (A_1) was synthesized by reaction of 2-(4-isobutylphenyl) propanoic acid with ethanole in presence of conic . H_2SO_4.The IR spectra showed band at (1750cm-1) due to stretching (c=o) group. 2-(4-isobutyl phenyl) propane acid hydrazyd (A_2) was synthesized by the reaction of (A_1) with acid hydrazid in presence of ethanol . the IR spectrum of (A_2) showed band in (1340cm^{-1}) due to starching (N-H) group band at (1680cm^{-1}) for (c=o) group . the H^1-NMR spectrum ($DMSO_6$) of compound (A_2) showed signal at (9.1ppm) for (H,NH) , signal at (3.9ppm) for (2H,NH2), signal at (1.2,3.1) for (3H,CH3,H,CH) and signal at (7.02-7.6) for (4H,phenyl) .the synthesized of compound 2-arylidenindan-1-3-dione (A_{3-8}) were confirmed by there melting point and .IR, H^1-NMR, the spectra carctrazation data are given in Tables (3,4) , 2-(4-isobutylphenyl)isopropan-3-aryl-3-

Kirkuk University Journal /Scientific Studies (KUJSS)
Volume 11, Issue 3, September 2016 , p.p(254-262)
ISSN 1992 – 0849

4—dihydroindeno{3,2-c}pyrazole -4-one (A_{9-14}), were synthesized by the reaction of compound (A_{3-8}) with acid hydrazyd (A_2) , the prepared compounds were confirmed by there melting point and .IR, H^1-NMR, the spectra carctrazation data are given in Tables (5,6) .

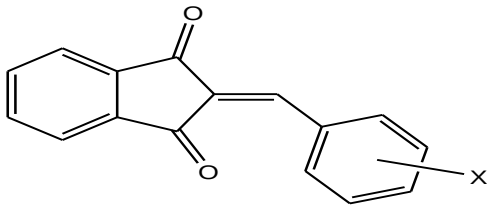


Table (3): IR spectrum data compound (A_{3-8})

Compound No.	R	ν cm-1 (KBr disk)			
		C=O	C=C	Ar C=C	others
A_3	2,4-Cl_2	1656	1602	1540	756 C-Cl
A_4	4-$N(CH_3)_2$	1665	1594	1520	2980 CH_3
A_5	4-NO_2	1666	1604	1560	1346 NO_2(sym) 1508(asmy)
A_6	4-F	1654	1595	1520	
A_7	4-Cl	1667	1608	1575	756 C-Cl
A_8	H	1654	1606	1540	

Table (4): H-NMR spectral data compound (A_{7-8})

Compound No.	H-NMR spectral data (δ ppm)
A_7	8.75 (H,CH) 7.6-7.9 (13H. phenyl)
A_8	8.75 (H,CH) 7.6-7.9 (13H. phenyl)

Kirkuk University Journal /Scientific Studies (KUJSS)
Volume 11, Issue 3, September 2016 , p.p(254-262)
ISSN 1992 – 0849

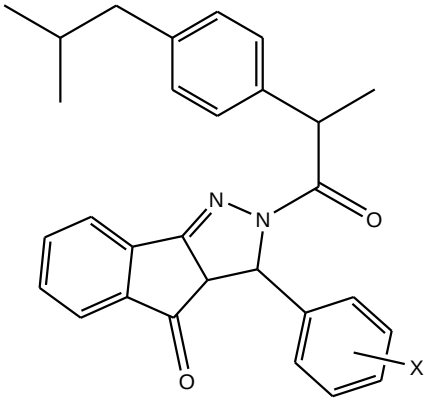


Table (5): IR spectrum data compound (A_{9-14})

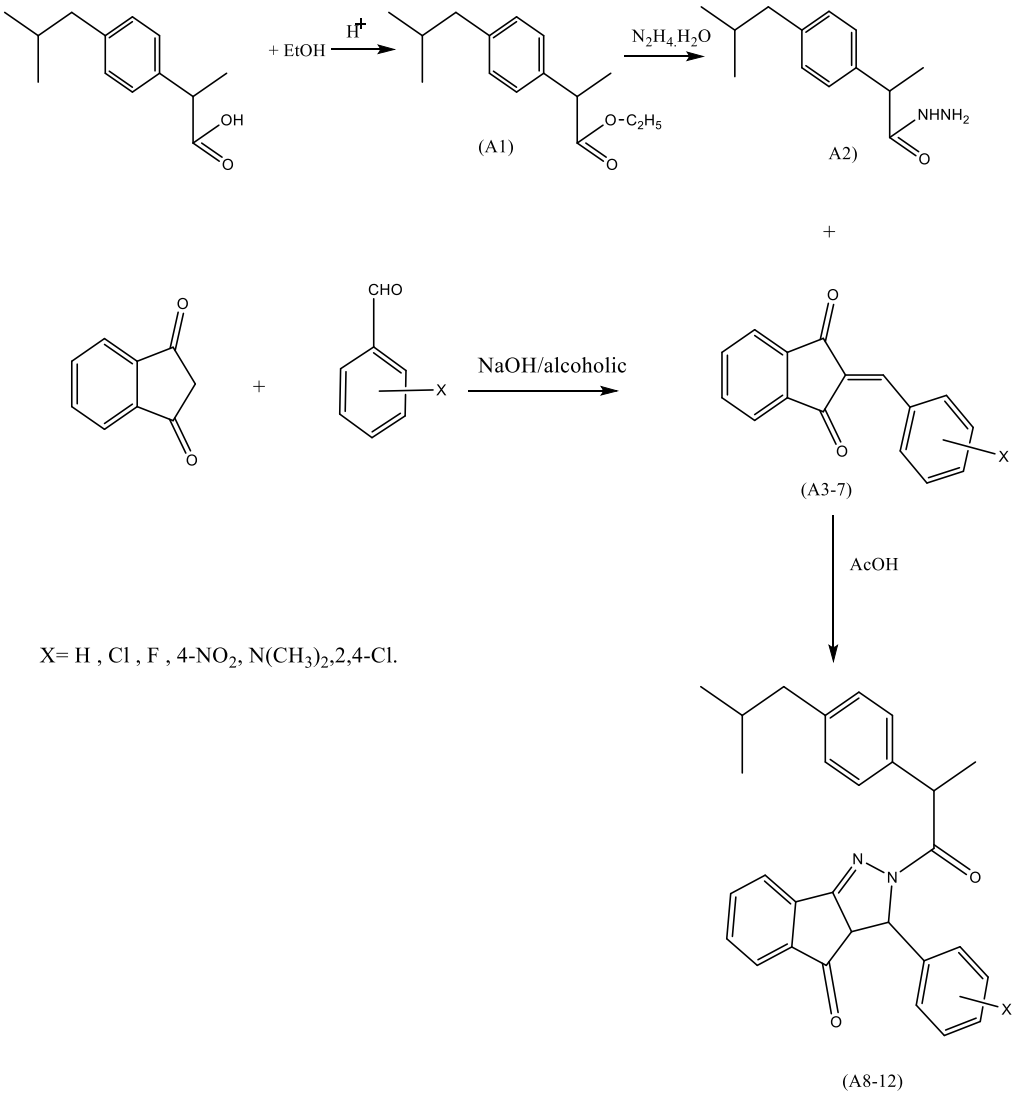
Compound No.	R	ν cm-1 (KBr disk)			
		C=O	N-C=O	C=N	others
A_9	2,4-Cl_2	1710	1670	1604	756 C-Cl
A_{10}	4-$N(CH_3)_2$	1709	1710	1590	2980 CH_3
A_{11}	4-NO_2	1705	1690	1606	1346 NO_2(sym) 1508(asmy)
A_{12}	4-F	1709	1680	1606	
A_{13}	4-Cl	1704	1689	1580	756 C-Cl
A_{14}	H	1708	1670	1604	

Table (6): H-NMR spectral data compound (A_{13-14})

Compound No.	H-NMR spectral data (δ ppm)
A_{13}	3.5 for (C4 in pyrazolin) 4.9 for (C5 in pyrazolin) 1.1 (3H, CH3) 1.5 (2H,CH2) 1.9 (H,CH) 7.1-8.3 (13H. phenyl)
A_{14}	3.9 for (C4 in pyrazolin) 4.9 for (C5 in pyrazolin) 3.4 (H.C2 acetyl) 1.25 (3H, CH3) 2.5 (2H,CH2) 2.5 (H,CH) 7.1-8.3 (13H. phenyl)

Kirkuk University Journal /Scientific Studies (KUJSS)
Volume 11, Issue 3, September 2016 , p.p(254-262)
ISSN 1992 – 0849

Scheme: the rout synthesis of compounds.



Reference

[1] A.,S.Hamad Elgazwy,Ekhlass Nassar & Myssoune Y. Zaki,Organic **Chem.Curr.Res.,1,5,(2012).**

[2] S. H.,Deepka K.& Mukta S.,**Amercan-Eurasian. J. of Sci. Res.5(4),257-263(2010).**

[3] R. S.,M.Bhagavan, S.Kavimani,p.venkappayya,R.Kumar & Ch.Sridevi,C.

Rathamani,K.Suganthi ,**J of Chem.1,1,39-45(2008).**

[4] M.nutana,Dr.v.Hurinadhababu,**IJIPSR,2,7,1311-2154.(2014).**

[5] A. A.F. Allouche,H. Fitoni & F.Chabchoub, **Mediterranean J. of Chem.3,2,864-876.(2014).**

[6] M.A.S.A.Javed,M. Zaheen Hassan.**MedChem.Res.21,1261-1270.(2012).**

[7]S.G.Khan,P.B.Mohit,R.Bh.Pandhave,S.A.Raju.Advance,**Pharm.Bulletin,4,2,105-112,(2014).**

[8] A.B.Pandya,D.G.Prajapati & S.S.Pandya,**J. of App.pharm.Sci.,2,8,226-232(2012).**

[9] S.S.Sondhi,S.Rajvanshi,N.Singh,Sh.Jain,A.M.Lanoti,**CEJC,2,1,141-187,(2004).**

[10] D.Bardalai,P.P.Selvam,Int.Res.**J.pharmAAp Sci.,2,3,1-8(2012).**

[11] R.J.Nevagi,**Der pharm Lett.,6,5,274-284(2014).**

[12] P.N.Balaji,L.Kanaka,K.mohen, & muni,**Der.Pharm.Sinica.,3(6)685-689(2012).**

[13] M.Amir & Sh.kumar.,**Ind.J.of Chem.44B,2532-2537(2005).**

[14] S. Rollas & G. kucukguzel.,**Molecules,12,1910-1939(2007).**

[15] S.Sraphar & Y.Rajender.**,E-J.of Chem.,9(4)1810-1815,(2012).**