Rafidain Journal of Science
Vol. 29, No.3, pp. 28-36, 2020
DOI : 10.33899/rjs.2020.166309
https://rsci.mosuljournals.com

ISSN: 1608-9391
e-ISSN: 2664-2786

Received: 15 /3/2020
Accepted: 18/5/ 2020

Synthesis of some New Spiro 1,3,4-Oxadiazolines Derived from Azlactone

Mohamad R. Mohamad **Shaymaa K. Younis***
Department of Chemistry/ College of Science/ University of Mosul
***E-mail: khazaalyounis@yahoo.com**

ABSTRACT

In this work a mixture of hippuric acid (N-benzoyl glycine) (1) and substituted benzaldehyde undergone Erlenmeyer and Perkin reactions respectively to afford the 4-aryl methylidene-2-phenyl oxazole-5-one (2a-d). This reaction proceeded under acidic media using acetic anhydride and sodium acetate. The azlactones (2a-d) used as starting material to prepare the imidazolone derivatives. New 4-aryl methylidene-5-oxo-2-phenyl-N'-(substituted cyclohexylidene)-4,5-dihydro imidazole-1-carbohydrazide(3a-j) were prepared from reactions the azlactones (2a-d) with Schiff's bases which prepared through condensation reaction of semicarbazide with substituted cyclo hexanone. Finally, the preparation of new 1-(4-acetyl-5-spiro substituted cyclohexyl-1,3,4-oxadiazol-2-yl)-4-aryl methylidene-2-phenyl-1H-imidazol-5-one (4a-j) via intracyclization reaction of imidazolones (3a-j) in the presence of acetic anhydride as suitable cyclizing agent.

The structure of the synthesized compounds were illustrated by the available physical and spectral analysis (M.P, T.L.C, U.V, FT-IR and some of them by ^{1}H-NMR) .

Keywords: Spiro 1,3,4-oxadiazolines, oxazolones, imidazolones, hippuric acid

INTRODUCTION

It's well known that all useful and novel heterocyclic compounds were derived from a very active and starting materials to ensure that all products involving activity in field of applications (Nami *et al.*, 2017; Gomha *et al.*, 2020). In this research azlactones (oxazolones) are five membered heterocyclic compounds with two hetero atoms nitrogen and oxygen was used as good starting material to prepare different types of heterocyclic compounds [imidazolons (3a-j) and spiro -1,3,4-oxadiazoles(4a-j)]. Actually, oxazolones perform an important role as anticancer (Jat *et al.*, 2012), anti-obesity (Conway *et al.*, 2009), anti-diabetic (Taile *et al.*, 2009), neuroleptic (Cascio *etal*,1989), anti-inflammatory (Crespo, 2000) and seductive (Mesaik *et al.*, 2004). Moreover, these compounds play a good role as starting material on organic chemistry (Terada *et al.*, 2011; El-Araby *et al.*, 2012; Moorkoth *et al.*, 2013).

In this research and because of the advantages mentioned above, it is planned to prepare a new series of spiro -1,3,4-oxadiazoles represented by compounds 1-(4-acetyl-5-spiro (substituted cyclo hexylidene)-1,3,4-oxadiazo-2yl)-4-anylidene-2-phenyl-1H-imidazol-5-one (4a-j). Firstly substituted Z-4-benzylidene oxazol -4,5-dihydro -5-one (2a-d) was reacted with schiff bases (a, b & c) to afford the 4-aryl methylidene-5-oxo-2-phenyl-N'-(substituted cyclohexylidene)-4,5-dihydro imidazole-1-carbohydrazide (3a-j) and the latter will be used as key intermediate to synthesize spiro -1,3,4-oxadiazoles (4a-j).

EXPERIMENTAL

Melting points (M.P.) were measured on Stuart SMP10. Melting Point apparatus and are uncorrected. Proton-Nuclear Magnetic Resonance (^{1}H-NMR) spectra were recorded using, Bruker DMX-500 NMR Spectrophotometer (400 MHz by using TMS as internal standard using DMSO-d_6 as a solvent, in University of Gaziantap, Turkey. [(s) singlet; (d) doublet; (t) tertiary; (q) quintet; (m) multiple]. Infrared (FT-IR) spectra were recorded as (KBr) disc using a Thermo Mattson 300 Infrared Spectrophotometer. Ultraviolet (UV) spectra were performed on Shimadzu UV-160 Ultraviolet–Visible Spectrophotometer, using DMF as a solvent. Thin layer chromatography (TLC) were carried out on Eastman chromatogram sheet (20x20) cm, 13181 silica gel with fluorescent indicator (No. 6060).

1- Synthesis of substituted Z-4-benzylidene oxazol -4,5-dihydro -5-one(2a-d) (Idriss and Saeed, 2016):

The mixture of, (0.224g/0.0125mole) hippuric acid (1), (0.0125mole) substituted benzaldehyde and (0.25mol) of sodium acetate grinds with (2-3 drops) of acetic anhydride was added with stirring followed by free solvent reflux for (3hrs.), cooling with adding (20ml) of methanol slowly and the contents allowed to stand overnight. The forming precipitate was filtered off and washed thoroughly with cold water to remove to excess of acid, followed by crystallization from ethanol. The purity of compounds was observed by T.L.C (8:2; benzene: methanol). (Table 1).

Table 1: The physical properties for oxazolones (2a-d)

Comp. No.	X	M.P (°C)	(%) Yield	T.L.C. Benzene Methanol (8:2)
2a	H	166-167*	58	0.600
2b	p-Cl	145-147*	63	0.588
2c	p-NO_2	211-212*	89	0.560
2d		171-173	78	0.583

* recorded (Fahmy *et al.*, 2016)

2- Synthesis of 1-(substituted cyclohexylidene) semicarbazone(a-c) (Tao *et al.*, 2006; Younis, 2010):

A solution (0.01mole) of semi carbazide (20 ml) of ethanol was added to an ethanolic solution (20ml) of (0.01 mole) substituted cyclo hexanone in presence of (5-7drops) glacial acetic acid and the reaction mixture was stirring for (10 min) followed by reflux for (30min). Cooling and calculate the forming product by filtration then recrystallization from absolute ethanol and the purity were controlled by TLC technique using solvent system (benzene:methanol) in (8:2) ratio to yield compounds: **(a)** by using 2-methyl cyclo hexanone to produce white solid product in yield (92%), m.p.(196 -167°C) , R_f :(0.701), UV-visable ($\lambda_{max\ (nm)}$): 238, FT-IR υ (cm^{-1}): NH_2 (3452), NH (3191), C=O lactam (1672), C=N (1575) and C-N (1500). **(b)**: by using 3,5,5-trimethyl cyclo 2-hexenone to produce yellow solid product in yield (92%), m.p. (234-235°C), R_f : (0.550), UV-visable ($\lambda_{max\ (nm)}$): 266, FT-IR υ (cm^{-1}): NH_2 (3463),NH (3198),C=O lactam(1699), C=N(1570), C=C (1530) and C-N(1480). **(c)**: by using dimedone to produce white solid product in yield (86%), m.p. (198- 199°C), R_f :(0.510), UV-visible ($\lambda_{max\ (nm)}$): 280, FT-IR υ (cm^{-1}): NH_2 (3247), NH (3177), C=O lactam (1671) ,C=O ketone (1612), C=N (1525) and C-N (1480).

3- Synthesis of 4-aryl methylidene-5-oxo-2-phenyl-N'-(substituted cyclohexylidene)-4,5-dihydro imidazole-1-carbohydrazide (3a-j) (Younis, 2010; Moorkoth *et al.*, 2013):

A mixture of (0.0125mole) Schiff bases (a-c) and (0.0125mole) substituted oxazolones (2a-d) in dioxane (40ml) with (5-10drops) glacial acetic acid , was stirred for (30min) followed by reflux for (3hrs.).The reaction mixture was then cooled and the forming precipitate calculated by filtration, washed and recrystallized from ethanol . (Table 2).

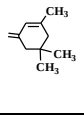
Table 2: The physical properties for imidazolones (3a-j)

Comp. No.	X		Molecular Formula	M.P (°C)	Yield (%)	T.L.C. Benzene Methanol (8:2)	Colour
3a	H		$C_{26}H_{26}N_4O_2$	163-166	88	.730 0	pale yellow
3b	p-Cl		$C_{26}H_{25}ClN_4O_2$	191-192	58	0.714	pale yellow
3c	p-NO_2		$C_{26}H_{25}N_5O_4$	172-174	72	.700 0	yellow
3d			$C_{28}H_{28}N_4O_3$	197-198	69	0.800	yellowish green
3e	H		$C_{25}H_{24}N_4O_3$	100-101	64	0.472	brown
3f	p-Cl		$C_{25}H_{23}ClN_4O_3$	112-115	94	0.520	yellow
3g			$C_{27}H_{28}N_4O_5$	82-83	82	0.720	yellow
3h	H		$C_{24}H_{23}N_4O_2$	115-117	69	o.550	pale yellow
3i	p-Cl		$C_{24}H_{23}ClN_4O_2$	138-140	79	0.590	yellow
3j			$C_{26}H_{26}N_4O_3$	112-113	86	0.462	yellowish green

4- Synthesis of 1-(4-acetyl-5-spiro (substituted cyclo hexylidene)-1,3,4-oxadiazo-2yl)-4-anylidene-2-phenyl-1H-imidazol-5-one (4a-j) (Tao *et al.*, 2006):

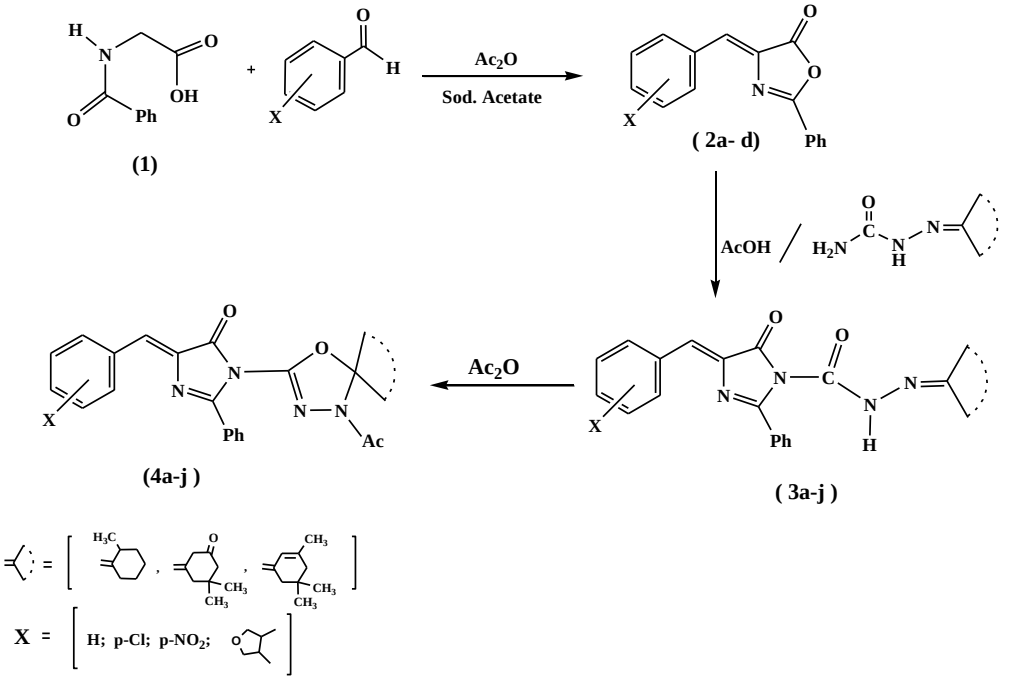
The Compounds (3a-j) were stirred for (30 min.) with (7ml) acetic anhydride and the mixture was refluxed for (3hrs.), cooling then poured into crashed ice, stirring, filtration and washed thoroughly with petroleum ether (60-80). Table (3).

Table 3: The physical properties for Spiro 1,3,4-oxadiazolidines (4a-j)

Comp. No.	X		Molecular Formula	M.P (°C)	Yield (%)	T.L.C. Benzene Methanol (8:2)	Colour
4a	H		$C_{28}H_{28}N_4O_3$	140-141	85	0.510	yellow
4b	p-Cl		$C_{28}H_{27}ClN_4O_3$	178-179	89	0.540	yellow
4c	p-NO_2		$C_{28}H_{29}N_5O_4$	242-243	95	0.640	yellow
4d			$C_{30}H_{30}N_4O_4$	189-190	96	0.900	yellowish green
4e	H		$C_{26}H_{26}N_4O_4$	161-162	88	0.440	pale brown
4f	p-Cl		$C_{26}H_{25}ClN_4O_4$	147-148	87	0.789	brown
4g			$C_{28}H_{28}N_4O_5$	185-186	96	0.800	yellowish brown
4h	H		$C_{25}H_{24}N_4O_3$	144-147	76	0.722	reddish brown
4i	p-Cl		$C_{25}H_{24}O_3ClN_4$	183-184	77	0.775	brown
4j			$C_{27}H_{27}N_4O_4$	170-172	76	0.576	yellowish green

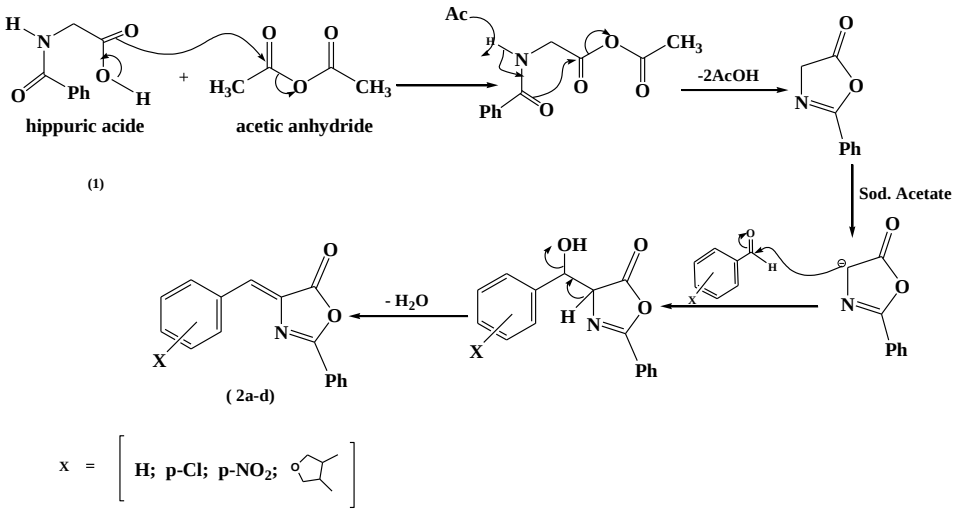
RESULT AND DISCUSSION

This presentation could be summarized through the following general Scheme (1):



Scheme (1)

Erlenmeyer and Perkin were used to prepare azlactons represented by substituted oxazolones in acidic media as shown in Scheme (2) (Fozooni *et al.*, 2017; Al-Hazam and Mahood, 2019).



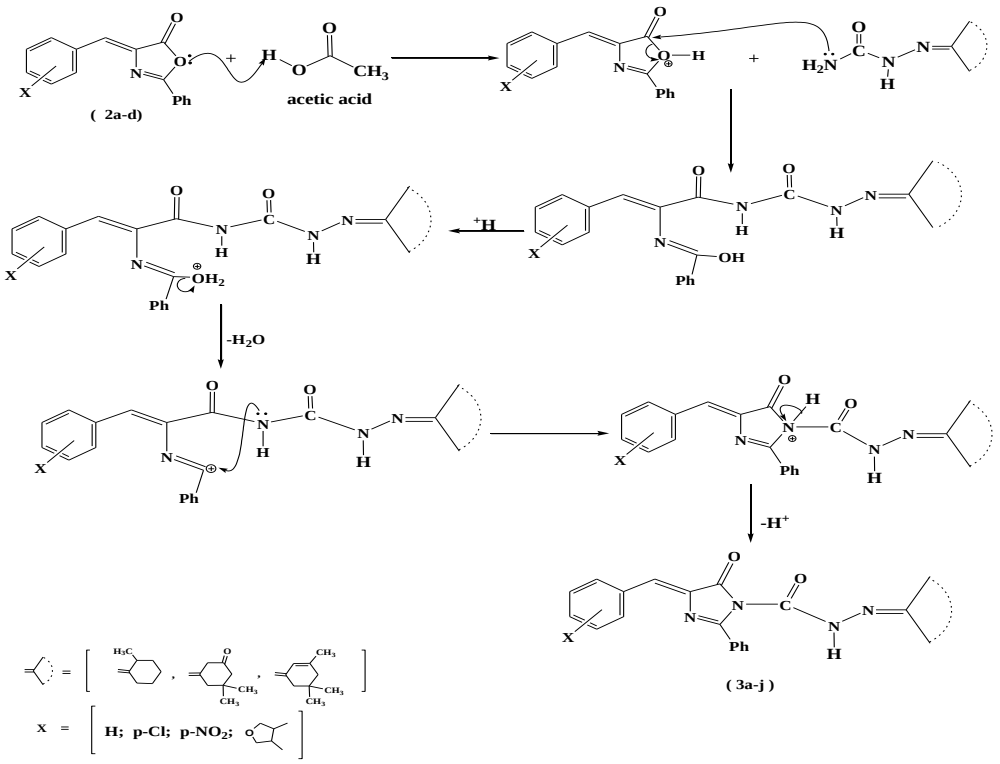
Scheme (2)

Synthesis of the oxazolones (2a-d) were achieved by reaction of hippuric acid (1) with substituted benzaldehyde in presence of acetic anhydride in dry conditions via Perkin reaction. The structure of compound (2a-d) were confirmed by spectral data results, FT-IR spectra showed the following absorption band at (1765-1794cm^{-1}) and (1641-1652cm^{-1}) refer to (C=O lactone) and (C=C) functional groups respectively ,Table(4) showed all absorption bands which support the formation structure . Its purity was determined via thin layer chromatography (T.L.C) using solvent system (benzene: Methanol) in ratio (8:2), (Table 1).

Table 4: The spectral data for oxazolones (2a-d)

Comp. No.	X	FT-IR (KBr), υ (cm^{-1})				*U.V DMf* λ_{max}*(nm)*
		C =O lactone	C=C	C=N cyclic	others	
2a	H	1787	1650	1549	—	384
2b	p-Cl	1783	1642	1581	Cl 769	255
2c	p-NO_2	1794	1652	1597	NO_2 asym 1516 sym 1333	253
2d		1765	1641	1600	C-O-C asym 1262 sym 1153	272

The Schiff bases namely 1-(substituted cyclo hexylidene)simecarbazide (a, b and c) [were synthesized through direct condensation reaction between semicarbazide and substituted cyclo hexanone], when treatment with oxazolones (2a-d) in acidic media will afford a series of 4-aryl methylidene-5-oxo-2-phenyl-N'-(substituted cyclohexylidene)-4,5-dihydro imidazole-1-carbohydrazide (3a-j) as shown in the following Scheme (3),(Idriss and Saeed, 2016).



Scheme (3)

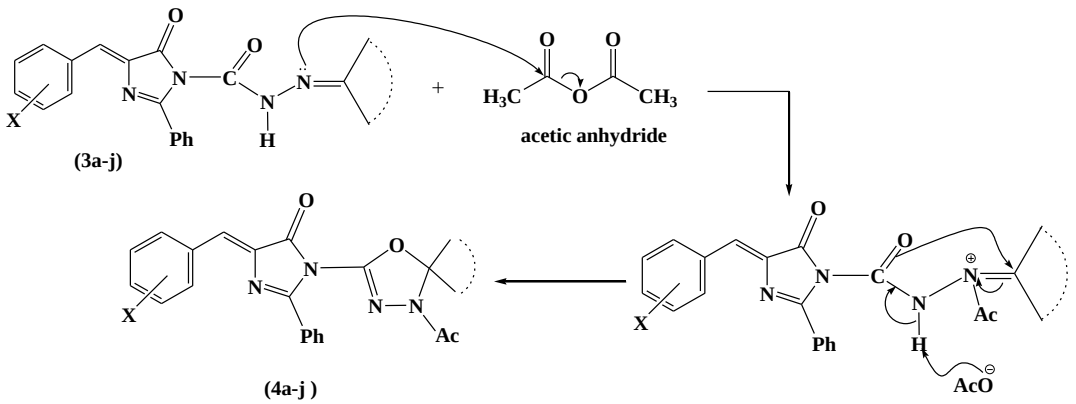
The imidazolones′ (3a-j) purity was identified by T.L.C technology while structural assignment of these products is strongly supported, thus in FT-IR showed two carbonyl groups one of them is for amid and the other for lactam respectively, (Table 5).

Table 5: The spectral data for imidazolones (3a-j)

Comp. No.	X		FT-IR (KBr) , υ (cm^{-1})							U.V DMf λ_{max} (nm)
			NH	C=O lactam	C=O amide	C=C	N=C cyclic	N=C acyclic	Other	
3a	H		3442	1783	1730	1645	1592	1547	C=C 1500	359
3b	p-Cl		3452	1789	1651	1583	1550	1484	Cl 774 C=C 1510	305
3c	p-NO_2		3326	1794	1735	1660	1597	1558	C=C 1520 NO_2 asym 1513 sym 1337	323
3d			3196	1768	1642	1601	1530	1487	C=C 1507 C-O-C asym 1261 sym 1157	333
3e	H		3271	1790	1680	1575	1525	1490	C=O 1630	359
3f	p-Cl		3333	1790	1689	1606	1528	1478	Cl 699 C=O 1632	308
3g			3188	1786	1720	1602	1485	1444	C=O 1636 C-O-C asym 1248 sym 1125	310
3h	H		3394	1761	1700	1646	1589	1523	—	399
3i	p-Cl		3472	1791	1740	1658	1593	1534	Cl 741	324
3j			3228	1779	1720	1645	1550	1487	C-O-C asym 1258 sym 1167	324

The ^{1}H-NMR spectra for compounds (3c, 3e and 3j) showed selective peaks which came in agreement with the suggested structures of imidazoles. (Table 7).

Finally, the spiro compounds (4a-j) were formed via intracyclization reaction in presence of acetic anhydride which act as a good acidic cyclization agent. Scheme (4) (Martins *et al.*, 2000).



Scheme (4)

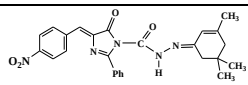
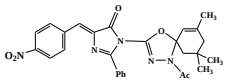
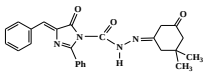
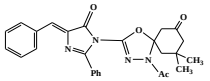
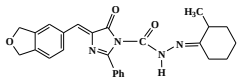
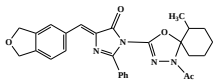
The present of acetyl group in FT-IR spectra and the absence of (C=O) amide gave good evidence that the spiro-1,3,4-oxadiazolines were formed, (Table 6).

Table 6: The spectral data for Spiro 1,3,4-oxadiazolidines (4a-j)

Comp. No.	X		FT-IR (KBr), υ (cm^{-1})					*U.V DMf* λ_{max} *(nm)*
			C=O Lactam	C=O amide	C=C	N=C cyclic	Other	
4a	H		1783	1716	1630	1525	C=C 1606	304
4b	p-Cl		1786	1718	1646	1582	C=C 1609 Cl 775	239
4c	p-NO_2		1793	1720	1650	1597	C=C 1615 NO_2 asym 1516 sym 1332	240
4d			1766	1701	1641	1575	C=C 1600 C-O-C asym 1262 sym 1156	269
4e	H		1782	1705	1591	1545	C=O 1644	224
4f	p-Cl		1785	1702	1581	1520	C=O 1645 Cl 774	266
4g			1762	1690	1606	1530	C=O 1666 C-O-C asym 1259 sym 1158	204
4h	H		1765	1640	1599	1510	—	275
4i	p-Cl		1766	1647	1585	1551	Cl 772	242
4j			1767	1642	1595	1520	C-O-C asym 1257 sym 1159	238

On the other hand, the ^{1}H-NMR spectra for compounds (4c, 4e and 4j) singlet peak, at (1.98, 2.00 and 3.25) respectively due to the acetyl group with disappearing of NH absorption band as shown in (Table 7).

Table 7: The ^{1}H-NMR spectral data for compounds (3c&4c, 3e&4e, 3j&4j)

Comp. No.	Structure	^{1}H-NMR, DMSO-d6, δ (ppm)
3_c		cyclo hexene(**CH$_2$**-C,2H,s,1.41; **CH$_2$**-C=C,2H,s,1.82; **CH**=C,1H,s,2.60) ; 2CH$_3$(6H; s,1.23); CH$_3$(3H; s,1.78); NH(1H; s, 7.49); **HC**=C(1H, s, 7.66); Ph(5H; m, 7.70-8.19); Ar-H(4H; doublet of doublet, 8.36-8.57, AB system)
4_c		cyclo hexene(**CH$_2$**-C(CH$_3$)$_2$,2H,s,2.41; **CH$_2$**-C=C,2H,s,2.60; **CH**=C,1H,s, 2.61) ; 2CH$_3$(6H; s,1.23); CH$_3$(3H; s,1.91); acetyl(3H; s, 3.25); **HC**=C-Ar (1H, s, 7.48); Ph(5H; m, 7.66-7.80); Ar-H(4H; doublet of doublet , 8.18-8.57, AB system)
3_e		cyclo hexanone (**CH$_2$**-C(CH$_3$)$_2$,2H, s,1.61; **CH$_2$**-C=O,2H s,3.48; N=C-**CH$_2$**-C=O, 2H, s,3.65) ; 2CH$_3$(6H; s,1.38; **HC**=C-Ar(1H, s, 6.34); Ph-C=N(5H; m, 7.28-7.62); Ph-C=C, 5H, m, 7.94-8.00) ;NH(1H; s, 10.37)
4_e		cyclo hexanone (**CH$_2$**-C(CH$_3$)$_2$,2H,s,2.41; **CH$_2$**-C=O,2H,s, 2.60; O-C-**CH$_2$**-C=O,2H, s,3.50) ; 2CH$_3$(6H; s,1.80; acetyl(3H; s, 2.30); **HC**=C-Ar(1H, s,7.38); Ph-C=N(5H; m, 7.66-7.77); Ph-C=C, 5H, m, 8.14-8.34)
3_j		cyclo hexane (CH(CH$_3$)-**CH$_2$**-CH$_2$,2H, b,1.32; CH$_2$-**CH$_2$**-CH$_2$,2H,b,1.44; N=C-CH$_2$-**CH$_2$** ,2H, b,1.54; N=C-**CH$_2$**-CH$_2$, 2H, b, 1.60; C-**CH**(CH$_3$)-CH$_2$,1H,b, 1.70) ;CH$_3$(3H; b,1.29); **HC**=C-Ar(1H, s, 6.52); piperonal-CH$_2$(4H; s,6.03); Ph-H(5H; m, 7.71-7.94); Ar-H(3H; m, 8.00-8.13); NH(1H; s, 10.3)
4_j		cyclo hexane(CH(CH$_3$)-**CH$_2$**-CH$_2$,2H, q ,1.13; CH$_2$-**CH$_2$**-CH$_2$,2H,p ,1.24; O-C-CH$_2$-**CH$_2$** ,2H,b ,1.44; O-C-**CH$_2$**-CH$_2$, 2H, t ,1.87; C-**CH**(CH$_3$)-CH$_2$,1H,m ,2.00) ; CH$_3$(3H; d,0.91); acetyl(3H; s, 1.98); **HC**=C-Ar(1H, s, 6.52); piperonal-CH$_2$(4H; s,6.17); Ph-H(5H; m, 7.10-7.94); Ar-H(3H; m, 8.07-8.13)

REFERENCES

Al-Hazam, H.A.; Mahood A.M. (2019). Synthesis, characterization and insilco study of new 1,3-oxazolone-5-(4-Cl)-one and-oxazolone-5-(4-NO2)-one Derivatives, *IOP Conf. Ser.: Mater. Sci. Eng.*, **571**, 012083.

Cascio, G.; Manghisi, E.; Fregnan, G. (1989). 5-Piperazinylalkyl-2(*3H*)-oxazolones with neuroleptic activity. *J. Med. Chem.*, **32**(10), 2241-2247.

Conway, P. A.; Devine, K.; Paradisi. F. (2009). A simple and efficient method for the synthesis of erlenmeyer azlactones. *Tetrahedron*, **65**, 2935-1938.

Crespo, M. I. (2000). Synthesis and biological evaluation of 3,4- diaryl oxazolonnes: a new class of orally active cyclooxygenase-2 inhibitors. *J. Med. Chem.*, **43**(2), 214-223.

El-Araby, M.; Omar, A.; Hassanein, H.H.; El-Helby, A.H.; Abdel-Rahman, A.A. (2012). Design, synthesis and in vivo anti-inflammatory activities of 2,4-diaryl-5-4H-imidazolone derivatives. *Molecules*, **17**, 12262–12275.

Fahmy, A.F.M.; El-Sayed, A.A.; Hemdan, M. (2016). Multicomponent synthesis of 4- arylidene-2-phenyl-5(4H)-oxazolones (azlactones) using a mechanochemical approach. *Chem. Central J.*, **10**(59), 1-7.

Fozooni, S.; Khoshdast1, H.; Hassani, H.; Hamidian, H. (2017). Synthesis of oxazolone and imidazolone derivatives in presence of H_2O_2 promoted fly ash as a novel and efficient catalyst. *J. Sci., Islamic Republic of Iran*, **28**(3), 221 - 230.

Gomha, S.M.; Abdel-aziz, H.M. ; Abolibda, T.Z.; Hassan, S.A.; Abdalla M.M. (2020). Green synthesis, molecular docking and pharmacological evaluation of new triazolo-thiadiazepinylcoumarine derivatives as sedative-hypnotic scaffold. *J. Heterocyclic Chem.*, **57**(3),1034-1043.

Idriss, I.KH.; Saeed, A.E.M. (2016). Synthesis and characterization of some imidazole derivatives. *Int. J. Innovative Sci., Engineer. and Technol.*, **3(**8), 34-42.

Islam, Md.R.; Mohsin M. (2007). Synthesis of isatin, 5-chloroisatin and their Δ^2-1,3,4-oxadiazoline derivatives for comparative cytotoxicity study on brin shrimp. *Bangladesh J. Pharmacol.*, **2**, 7-12.

Jat, L.R.; Mishra, R.; Pathak, D. (2012). Synthesis and anticancer activity of 4-benzylidine-2-phenyl oxazol-5(4*H*)-one derivatives. *Int. J. Pharm. Sci.*, **4**(1), 378- 380.

Martins, A.M.A.; Moglioni, A.G.; Brousse, B.; Moltrasio, G.Y.; D'Accorso, N. B. (2000). Synthesis and characterization of 2,2-disubstituted thiadiazolines. *ARKIVOC*, **iv**, 627-640.

Mesaik, A.; Rahat, S.; Khan, M.; Ullah, Z.; Choudary, M.I.; Murad, S.; Ismail, Z.; Rahman, A.; Ahmad, A. (2004). Synthesis and immunodilatory properties of selected oxazolone derivatives. *Bioorganic and Medicinal Chem.*, **12**, 2049-2057.

Moorkoth, S.; Josephb, A.; Srinivasan, K. Kb.; Gopalan, K. Nc. (2013). Synthesis and evaluation of biological activity of imidazolidinone analogues of 2- aminochromone. *Inter. J. Pharma Bioscience and Technol.*, **1**(3), 130-141

Nami N.; Zareyee, D.; Ghasemi, M.; Forouzani, M.; Mirzad, S.; Hashemi, S.M. (2017). An efficient method for synthesis of some heterocyclic compounds containing 3-iminoisatin and 1,2,4-triazole using Fe_3O_4 magnetic nanoparticles. *J. Sulfur Chem.*, **38**(3), 279-290.

Taile, V.; Hatzade, K.; Gaidhane, P.; Ingle, V. (2009). Synthesis and biological activity of 4-(4-hydroxybenzylidene)-2-(substituted styryl) oxazol-5-ones and their o-glucosides. *Turk. J. Chem.*, **33**, 295-305.

Tao, J.; Cao, L.; Wang, C.; Wang, D. (2006). Synthesis of 1,3,4-oxadiazoles and 1,3-thiazolidinones containing 1,4,5,6-tetrahydro-6-pyridazinone. *J. Chinese Chem. Soc.*, **53**, 1193-1197.

Terada, M.; Moriya, K.; Kanomata, K.; Sorimachi, K. (2011). Chiral broensted acid catalyzed stereoselective addition of azlactones to 3-vinylindoles for facile access to enantioenriched tryptophan derivatives. *Angew. Chem., Int. Ed.*, **50** (52), 12586-12590.

Younis, S.K. (2010). Synthesis of some new 1,2,4,5-tetrazine derivatives via Diels-Alder reaction. *J. Raf. Sci.*, **2**(1), 65- 76.

Younis, S.K. (2010). Microwave Assisted Synthesis of some New Chromene Derivatives. *J. Raf. Sci.*, **21**(1), 44- 52.

تحضير عدد من مركبات سبايرو 1، 3، 4- اوكسادايازولين الجديدة والمشتقة من الآزالاكتون

الملخص

في هذا العمل يخضع مزيج مؤلف من كل من حامض الهيبيوريك (N-بنزويل كلايسين) (1) والبنزالديهايد المعوض لتفاعل إرلينماير وبيركن على التوالي لاعطاء مركبات 4 -ارايل مثيليدين-2-فنيل اوكسازول-5-اون (2a-d)، يتم هذا التفاعل تحت ظروف حامضية بوجود انهيدريد حامض الخليك وخلات الصوديوم. تستخدم مركبات الآزالاكتون (2a-d) كمادة اولية في تحضير مشتقات الايميدازولون، حيث تحضر مركبات 4- ارايل مثيليدين-5-اوكسو-2-فنيل-N'(معوض الهكسايليدين الحلقي) -5,4- ثنائي هيدرو ايميدازول-1-كاربوهيدرازايد (3a-j) الجديدة من خلال تفاعل الآزالاكتون (2a-d) مع عدد من قواعد شيف التي تحضر من تفاعل السيمي كاربازايد مع معوضات الهكسانون الحلقي. وأخيرا تحضر مركبات 1- (4-اسيتيل-5-سبايرو معوض الهكسيل الحلقي-4,3,1- اوكسادايازول-2-يل) -4- ارايل مثيليدين-2-فنيل-1 هيدرو-ايميدازول-5-اون الجديدة (4a-j) عبر تفاعل الغلق الضمني لمركبات الايميدازولون (3a-j) بوجود انهيدريد حامض الخليك كعامل غلق مناسب.

الصيغ التركيبية للمركبات المحضرة تم تشخيصها بالطرائق الفيزيائية والطيفية المتوفرة (FT-IR, U.V, T.L.C, M.P , ولبعض منها تم اجراء قياس طيف ^{1}H-NMR).

الكلمات الدالة: سبايرو-4,3,1-اوكسادايازول، اوكسازولون، ايميدازولون، حامض الهيبيوريك.