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Synthesis and characterization of Various Amides Via Oxazepine Compounds

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Abstract:

N-Furfuralidene-(1-phenyl-2.3-dimethyl pyrazolone amine [1] was prepared by condensation of furfuraldehyde with pyrazolone amine . The compound [1] was found to react with maleic anhydride to produce 2-furfuryl-3-(1-phenyl-2,3-dimethyl pyrazolone)-2,3-dihydro [1,3]-oxazepine-4,7-dione [2] which was reacted with different secondary amine (morpholine , piperidine , diethyl amine) to give amide direvatives of maleic acid [3-5] .

Introduction :

These compounds are one class of heterocyclic compound that have a wide spectrum of uses like preparation of various compounds[1-3], antimicrobial[4] analgesic [5] agents, antibacterial[6,7] other uses[8,9] .These compounds are considered an important branch compounds due to their implication in drugs and industrial fields which have one or more of the heteroatoms such as (nitrogen , oxygen ….)[1-3,10] .
There are found as construction units in many of biological molecules[10] .

Experimental :

All chemical used were supplied from BDH-Chemical company and Fluka-AG .
-Melting point were recorded using : Electro thermal 9300 , melting point engineering , LTD , U. K .
-Infrared spectra were recorded using Fourier transform infrared shimadzu (8300) (FT.IR) infrared spectrophotometer , KBr disc was performed by co.s.q.c.Iraq .
-Elemental analysis (C.H.N) were carried out by EA-O17 mth in center Lab-Instiute. Of earth and environ mental science , Al-Albyat university , Jordan .
-UV-visible spectra were recorded in : shimadzu-1700, double beam with computerized , Japan.

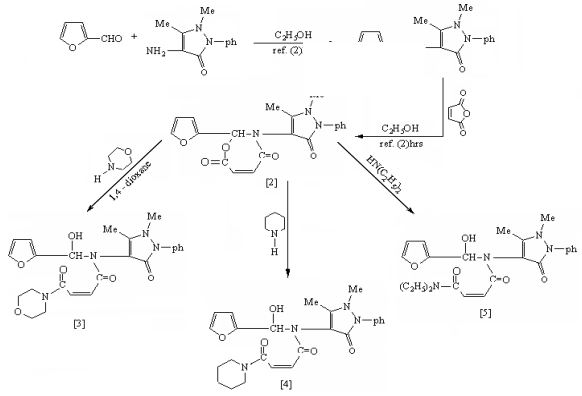
Experimental Work :

N-furfuralidene –(1-phenyl-2,3-dimethyl pryazolone amine [1][1,11-5] A mixture of equimolar amount (0.07 mole) of furfuraldehyde and 1-phenyl-2,3-dimethyl-4-amine-pyrazole were reacted by condensation in ethanol for (2) hrs and recrystallized from ethanol to give colored crystal of Schiff's base [1].
2-furfuryl-3-(1-phenyl-2,3-dimethyl pyrazolone)-2,3-di hydro [1,3]-oxazepine-4,7-dione [2][1,3] A mixture of (0.05 mole) of compound [1] with maleic anhydride was

refluxed for (3) hrs in dry benzene , The solvent was removed and recrystallized from dry 1,4-dioxane to give colored crystalline solid of 1,3-oxazepine compound [2] .
2-furfuryl-3-(1-phenyl-2,3-dimethyl pyrazolone)-2,3-dihydro [1,3]-oxazepine-4,7-dione with 4-(morpholine or piperidine or diethyl amine) [3-5][16]Dissolve (0.005 mole) of 2-furfuryl -3-(1-phenyl-2,3-dimethyl pyrazolone)-2,3-dihydro [1,3]-oxazepine-4,7-dione [2] in dry 1,4-dioxane, (0.02 mole) of dry (morpholine or piperidine or diethyl amine) was added dropwise with stirring, the mixture was heated to (80 C̊) in water bath for (30 min) , the separated crystalline solid was filtered and recrystallized from 1,4-dioxane to give amide derivatives of maleic acid [3-5] .

Results and discussion :

All reactions in this work are presented in scheme (1):



Scheme (1)

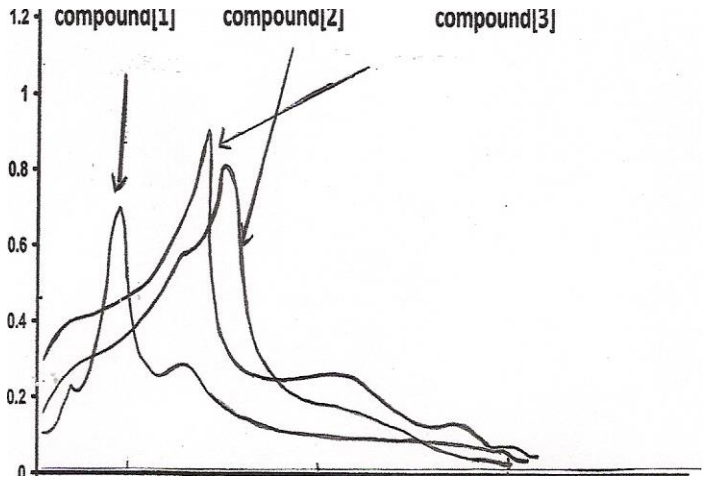
All synthesized compounds [1-5] have been characterization by melting point and spectroscopic methods (Uv-Vis , FT. IR) and C. H. N-analysis .FT. IR-showed appearance band at (1615)cm $^{-1}$ [1,3] due to azomethine (C=N) group of compound [1] , while this band is disappear and other band is appear at (1690)cm^{-1} [1] due to (lactone/lactame) group of oxazepine compound [2] .The formation of compounds [3-5] are followed by disappearance of (lactone) absorption band at (1690)cm $^{-1}$, and appearance of two bands : at (3450)cm $^{-1}$ due to (-OH) group[17] and other band at (1650)cm $^{-1}$ [16] due to () of synthesized amides [3-5] , this strong evidence to formation of compounds [1-5] . Other date of functional groups shown in the following Table (1), (C. H. N)-analysis and melting points of compounds [1-5] shown in the Table [2] .
The Uv-spectra of compound [2] shown absorption maxima[1,16] at (395)nm due to charge transfer of the furfuryl group and the oxazepine cyclic , while the absorption is decrease in compounds [3-5] to (378-360)nm due to break of the oxazepine cyclic[1] and formation of amides [3-5] , Table [3]

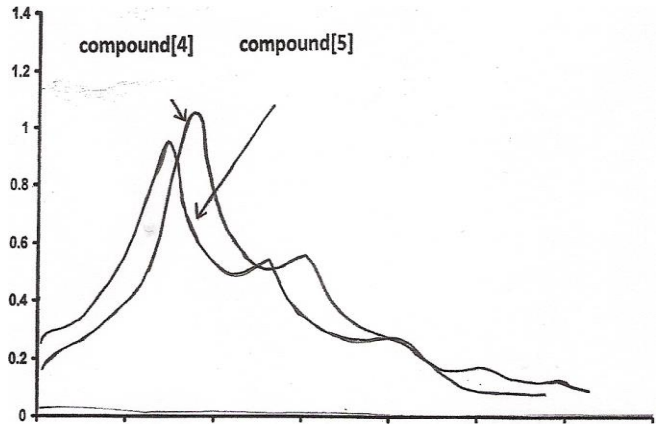
Table (3) – UV- Visible absorption maxima of compounds [1-5]

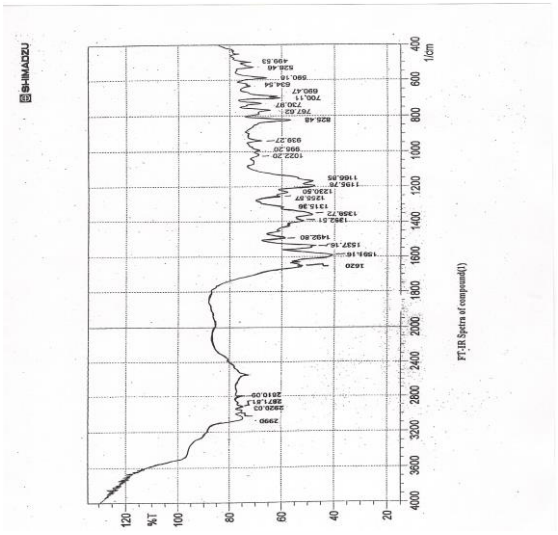
Com. No.	λ_{max} (nm)
1	290
2	395
3	378
4	360
5	345

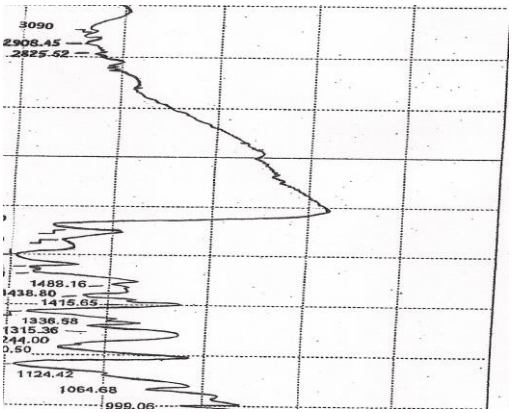
Acknowlegement :

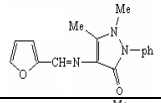
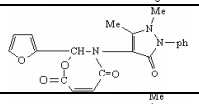
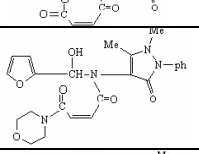
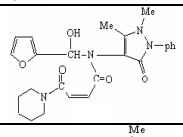
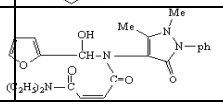
I would like to express my thanks to Mr. Muhanad abu Al-soaud in center lab.Institute of Earth and Environmental science-AL-byat University H. J. K. in Jordon for providing C. H. N. element analytical and some material .






SHIMADZU
FT-IR Spectra of compound(1)
%T
1/cm


3090
2908.45
2825.52
1488.16
1438.80
1415.65
1336.58
1315.36
1124.42
1064.68

Comp. No.	Structural Formula	υ (C-H) str. Aromatic Alipatic	υ (C=N) Imine Group	(C=O) str. Lactone ,Lactame	(C-O) str. Lactone	(-OH) str.	(C=O) of amide
1		3080 2920,2990	1615	–	–	–	1620
2		3090 2908,2825	–	1690 1680	1240	–	1610
3		3040 2908,2819	–	– 1675	–	3450	1640
4		3045 2935,2908	–	– 1680	–	3480	1655
5		3091 2935,2891	–	– 1695	–	3455	1685

References :

1- Nagham. M. Al-Jamali , Ph.D. Thesis, University of Baghdad (2008) .
2- Xin.P ,Zhang.M and Fang.W.,J.Chinese Chem.Soci.,47,535-539 (2002).
3- Birgul.O and Cesur.Z., Molecules,9 ,894-901 (2004) .
4- N. F. Eweiss and A. A. Bahajaj, J. Heterocyclic. Chem. 24, 1173 (1987) .
5- Vakula. T. Radha and V. Range, J. Pract. Chem., 315, 185 (1973) .
6-Yang. Z ., Ren-Z. Qiao, peng-Feixu, Zi-Yi. Zhany and kai-Bei. Yn, J. of the Chinese Chem.. Soc., 49, 369 (2002) .
7- Hansong. C. Zengmingli and Y. Han, J. Agric. Food. Chem., 48, p.5312-5315 (2000) .
8- Hellen.K and Balasubramaniar.N., Ind.Journal of Chemical Technology ,9,363-372 (2002) .

9- Feas. A. Hussain, ph. D. Thesis, University of Baghdad (2005) .
10- F. A. Carey "Organic Chemistry" Mc Graw-Hill, Inc. Americal, 3, p. 453-454 (1996).
11- Reevs.R .,J.Am.Chem.Soc.,84 ,3332 (1962) .
12- Nagham. M. Al-Jamali , Journal of Al-Qadisiah for pure Science.,. 10,1, p. 131-138 (2005) .
13- Nagham. M. Al-Jamali and Sami . W. Radhi ; Al- Mustansiria Journal of Science., . 15,. 3, p. 56-66 (2004) .
14- Nagham. M. Al-Jamali , Journal of Babylone ., 11,.3, p. 452-458 (2006) .
15- Nagham. M. Al-Jamali , Journal of Al-Qadisiah for pure Science., 9,. 4, p. 257-267 (2004) .
16- Obaid .H. Abid., National Journal of Chemistry , 3 , p. 480-492 (2001) .
17- Nagham. M. Al-Jamali .,J.Al-Qadisiah for pure Science.,3 ,12 (2007) .

Table (1) FT.IR date (Cm^{-1}) of compounds [1-5]
Table (2)-physical data of compounds [1-5]

Comp. No	M.F	m. p/ C °	Calc. / Found	C %	H %	N %
1	$C_{16}H_{15}N_3O_2$	198	86.32 86.19		5.33 5.39	14.94 14.70
2	$C_{20}H_{17}N_3O_5$	150	64.00 64.09		4.53 4.60	11.20 11.08
3	$C_{24}H_{26}N_4O_6$	181	60.75 60.81		5.48 5.32	11.81 11.95
4	$C_{25}H_{28}N_4O_5$	165	64.65 64.74		6.03 6.14	12.06 12.18
5	$C_{24}H_{28}N_4O_5$	173	63.71 63.34		6.19 6.08	12.38 12.22

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تحضير ودراسة خصائص أمايدات مختلفةعن طريق مركبات الأوكسازبين

نغم محمود الجمالي
قسم الكيمياء-كلية التربية-جامعة الكوفة

الخلاصة :

تم تحضير المركب N- فورفورالدين – (1- فنيل – 3,2 – ثنائي مثيل بايرازولون أمين [1] بتكاثف فورفورالديهايد مع امين البايرازولون . ثم تم مفاعلة المركب الاخير مع انهيدريد الماليك فأعطى 2- فورفوريل -3-(1-فنيل -3,2- ثنائي مثيل بايرازولون) – 3,2 – ثنائي هايدرو [3,1] - اوكسازبين – 7,4 – دايون [2] والذي تم مفاعلته مع امينات ثانوية مختلفة منها (المورفولين ، البايبيريدين ، ثنائي اثيل امين) فاعطى مشتقات الامايد لحامض الماليك [3-5] .