Synthesis of Some new 2-(4-Aryliminophenoxy)N-Arylacetamide Via p-hydroxy benzaldehyde.

*Ruwa M. Mauf** *Asmaa H. Sultan*** *AmeeraM.Farag****

Received 20, December, 2012
Accepted 11, March, 2014

Abstract

Chloroacetamide derivatives (2a-g) have been prepared through reaction of chloroacetyl chloride(1) (which prepared by the reaction of chloroacetic acid with thionyl chloride) with primary aromatic amines and sulfa compounds to afford compounds (2a-g) which then reacted with p-hydroxy benzaldehyde via Williamson reaction to obtaine the new compounds 2-(4-formyl phenoxy)-N-aryl acetamide (3a-g). Finally , compounds (3a-g) will be use as a good synthon to prepare the Schiff bases represented by compounds 2-(4-aryliminophenoxy)-N-arylacetamide (4a-g). through , reaction with some primary aromatic amine. All the prepared compounds were investigated by the available physical and spectroscopic methods.

Key words: Schiff base, amides, ether.

Introduction

Amide and similar compounds are well known as useful building blocks for the synthesis of variety of drug, it is well known in literature that nitrogen and sulfur containing compounds are essentially used in medicinal purpose for the treatment of different kinds of fungal, bacterial infection along with treatment of gastric, and anticancer [(1)], also they have been as sayed invitro for their biological activity against E-coli. Amide had been reported to posses antibactrial activity [(2)] substituted amide are acting as inhibitors of HIV proteans [(3)], hypertension as analgesic [(4)]. Amide linkages are not only the key chemical connection of proteins but they are also the basis for some of the most varsatik and widely used synthetic polymer [(5)], on the other hand ethers compounds showed bio logical activity such as amide which are used as antipyretic agent [(6)], anti inflammatory [(7)], herbcidic [(8)], antimicrobial activity antimaterial activity [(9,10)], whereas, the biological activity of Schiff base compounds were appeared as anticancer [(11)] herbicidal activities [(12)], antifungal [(13)], antibacterial agent [(14)]. Infact the Schiff bases are important not only medical chemistry, but also in organic synthetic chemistry.

Material and Methods:

Melting point were determined on an electro thermal 9300 melting point apparatus and were uncorrected. The IR spectra were determined as potassium bromide plat on pye Unicom SP. 2000 FTIR spectrophotometer. The UV spectra were recorded on shimadzu (UV-160) UV-Visible spectrophotometer using $CHCl_3$ as a solvent

Preparation of α-chloroacetamides (2a-g) [(15)]

*Department of Chemistry College of education for Girels University of Mosul
**Department of Chemistry College of Science University of Mosul

A mixture of chloro acetic acid (0.03 mole, 1.4 g) and thionyl chloride (0.04 mole , 2ml) via calcium chloride tube, the reaction mixture was stirred and heated at 70-75 ºC for 30 mins. The excess of thionyl chloride was evaporated.

The mixture was coolded to 0ºC in ice bath then dry benzene (25 ml) and amine (0.033 mole) were added drop wise with shaking . The mixture was refluxed for 30 mins, washed with cold water, and the solution was made alkaline with 10% sodium hydroxide. The precipitate was filtered, wash with water and recrystalized from methanol-water to obtain the desired product as shown in Table 1.

Table (1): the physical properties and spectroscopic data of compounds (2a-g)

Comp No	m.p ^{0}c	Yield %	IR νcm^{-1} (KBr)				UV max $CHCl_3$
			υC=O	υN-H	υS=O	υS=O*	
2a	132-134	72	1685	3290	-	-	255
2b	119-121	63	1675	3290	-	-	254
2c	118-120	-	1680	3280	-	-	263
2d	160-162	53	1680	3280	-	-	254
2e	218-220	55	1680	3300	1340	1185	263
2f	223-225	85	1670	3300	1336	1166	265
2g	111-113	84	1680	3275	-	-	264

Note: compound (2a-g) showed ester (υC=O) at 1720 cm^{-1}.

Preparation of 2-(4-formyl phenoxy)-N-aryl acetamide (3a-g) [16].

Sodium (0.4 g) was dissolved in absolute ethanol (25 ml). The solution is cooled and hydroxybenzaldehyde compound (0.033 mole) and (0.048 mole) chloro acetamide were added slowly. The reaction mixture was stirred and refluxed for 3 hours and an ice bath. The formed precipitate was filtered, washed and recrystallized from methanol, to obtain the desired product as shown in Table 2.

Table (2): the physical properties and spectroscopic data of compounds (3a-g)

Comp No	m.p ^{0}c	Yield %	IR νcm^{-1} (KBr)					UV max
			υAr-O-c	υC-H	υN-H	υS=O	υS=O*	$CHCl_3$
3a	171-173	65	1254	2850	3431	-	-	252
3b	163-165	70	1244	2235	3288	-	-	253
3c	203-205	80	1261	2850	3475	-	-	257
3d	152-154	60	1252	2851	3452	-	-	258
3e	dec200	75	1284	2854	3417	1320	1200	270
3f	160-163	60	1259	2852	3467	1310	1160	263
3g	dec250	75	1238	2871	3282	-	-	267

Note: the compound (3g) showed esteric (υC=O) at 1714cm^{-1}.

Preparation of 2-(4-aryliminophenoxy)-N-aryl acetamide (4a-g) [17].

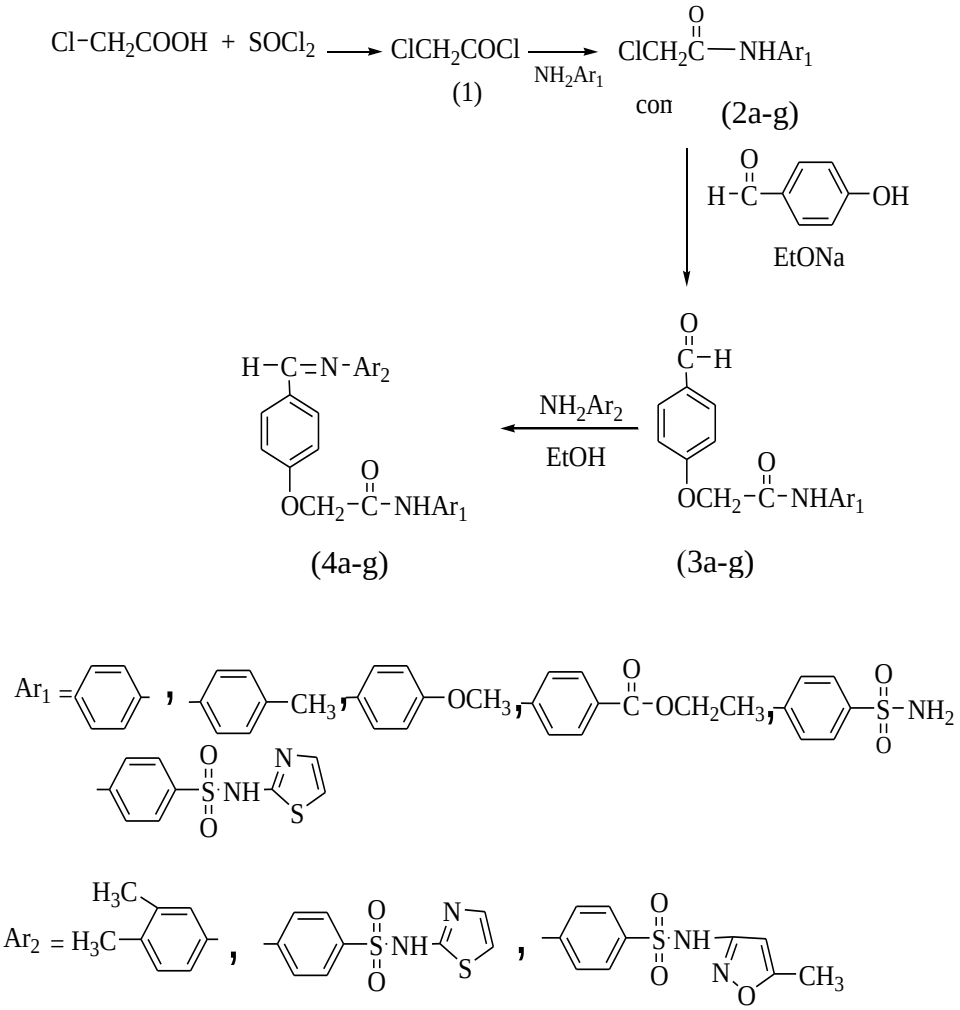
Equilmolar (0.033 mole) of aromatic amine or sulfa compounds with (4a-g) compounds, each one dissolvent in absolute ethanol (25ml) was heated to be homogenous solutions. The solution of compound (3a-g) was added gradually to the above solution of amines or sulfa compounds. The mixture was refluxed for 30 mins with stirring. After cooling the precipitate was filtered and recrystallized from ethanol to obtain the products (4a-g) as shown in (Table 3).

Table (3): the physical properties and spectroscopic data of compounds (4a-g)

Comp No	m.p ^{0}c	Yield %	IR νcm^{-1} (KBr)					UV max
			υC=N	υC-H	υN-H	υS=O	υS=O*	$CHCl_3$
4a	dec 201	53	-	-	-	-	-	254
4b	202-204	70	1601	1269	-	-	-	254
4c	dec 200	73	1599	1284	-	-	-	253
4d	182-184	55	1597	1269	-	-	-	254
4e	104-106	42	1599	1282	1340	1138	1138	273
4f	dec 103	70	1604	1331	1324	1162	1162	268
4g	163-165	81	1597	1263	-	-	-	263

Results and Discussion:

Concerning results and discussion, it has been found that many other compounds and Schiff base were formed to be pharmaceutical active compounds. Clarifying this more, scheme (1) shows that the compounds (4a-g) were synthesized by four stage, the first stage is the formation of chloroacetylchloride (1).while the second stage is the formation of chloroacetamide derivatives (2a-g). The third stage is the formation of new compounds 2-(4-formyl phenoxy-N-arylacetamide (3a-g) (which prepared by the reaction of compounds (2a-g) with p-hydroxy benzaldehyde via Williamson reaction). the fourth stage, compounds (3a-g) used to prepare Schiff base (4a-g) through , reaction with some primary aromatic amine



Scheme 1

The IR spectra of compounds (2a-g) , indicated in Table (1),showed strong stretching absorption band for amid υC=O at low frequencies (1685-1670) cm^{-1}, This is due to the resonance effect[18] broad band for υN-H at (3300-3275) cm^{-1}, for asymmetric and symmetric (υS=O) at (1340-1336) cm^{-1} and (1185-1166) cm^{-1} respectively. UV spectrum of compounds (2a-g) showed λmax (264-254) nm due to the electronic transition (n ⟶ π*) , shown in Table (1).

The IR spectrum of compounds (3a-g) , indicated in Table (2), showed strong absorption band υC-O-Ar at (1284-1238)cm^{-1}, υH-C=O at (2871-2835) cm^{-1} for aldehyde

The UV spectrum of compounds (3a-g) showed bath chromic shift in λmax (272-252) nm due to the electronic transition (n ⟶ π*).

The IR spectrum of compounds (4a-g) , indicated in Table (3), showed strong character sting band at (1604-1597) cm^{-1} due to the υC=N (imine group) in these compounds.

The UV spectrum (4a-g) showed wavelength of the absorption band at (273-253) nm due to the electronic transition (n⟶ π*) which occurred on the imine group of these compounds , shown in Table (3).

References:

1. R.K Singh, Singh A., Mishra R.S, Thakur, J.Int.Pharm., 2009, 1, 1.

2. Wilson and S.Gisvold, Text Book of organic medicinal and pharmaceutical chemistry, Lippin cott Company, Phildelphia, New York, London, 165(1998).

3. A.F . Charles, J.K . Dale, E.W.Norman, J.Bio.Org.Med., 5, 2725-2728,1995.

4. K.C Chalurara, B.K. Ishwar (2010), J.Org.Chem., 2, 1368-1371.

5. J.W. Bode, and R.R.Vijaya, J.Nat.Sci., 48, 471-479, 2001.

6. M.A. Sheat, S.A. Ali , J.Edu.Sci,23, 64, 2005.

7. R.H. Longan, "Synthesis of ethers" , Dalls county community college district north lake college, 5, 2, 1997.

8. H.Hort, O.Hart, L.E. Crain, "Organic chemistry ashort course", 9th Ed, Houghtom Mifflin company, Toronto, 204, 344,1995.

9. 10. A.Satyanaray, V.Maddi, S.N. Mamledesai, S.Swamy, J. Ind. Pharm.Sci, 69, 847-849,2007.

11. M.Arshinaqvi, V.Ariktla, Z.Shahnawaa, D.Rao, metho. J.Chem., 6, 75-78, 2009.

12. A.Chohan, H.Sajjad, H.Zahid, J.Am.Sci, 25, 599-607, 2010.

13. Y., Zhaoqi, S.Pimhua, J.molban K., 214, 2006.

14. A.M Hafez, A.E . Taggi, H.Wack , B.Young , J.Am.Chem.Sci., 124, 6626, 2002.

15. A.Vogel, "Text Book of Partical Organic chemistry", 4th Edn., Longman, London , 423, 1972.

16. M.A. Sheat and A.Alyai., J.Edu. Sci., 28, 36, 2004.

17. A.Vogel, "Text Book of Partical Organic chemistry", 4th Edn., Longman, London , 502, 1972.

18. V.M. Parikh, "Absorption spectroscopy of organic molecules", 189, 528, 530, 1985.

تحضير عدد من مركبات 2-(4- اريل ايمينوفينوكس)-N اريل استياميد من بارا-هيدروكسي بنزلديهايد

رؤى مزاحم الشماع* اسماء حاجم سلطان** اميرة محمد فرج **

*قسم الكيمياء كلية التربية للبنات جامعة الموصل

**قسم الكيمياء كلية العلوم جامعة الموصل

الخلاصة:

حضرعدد من اميدات كلوريد حامض الخليك (2a-g) من تفاعل كلوريد كلورو الاسيتيل (1) (والمحضر سلفا من تفاعل كلوريد حامض الخليك مع كلوريد الثايونيل). مع بعض من الامينات الاولية الاروماتية المختلفة وعدد من مركبات السلفا كخطوة اولية لتحضيرعدد من مشتقات اميدات كلوريد حامض الخليك (2a-g) . ومن ثم مفاعله مركبات (2a-g) مع مركب البارا-هيدروكسي بنزالدهايد باستخدام تفاعل وليامسون للحصول على 2-(4-فورميل فينوكسي)-N-اريل اسيتاميد (3a-g) ، واخيرا استخدمت المركبات (3a-g) كمواد اولية مناسبة لتحضير عدد من قواعد شيف 2-(4-اريل ايمينوفينوكسي)-N-اريل اسيتاميد (4a-g) من خلال تفاعلها مع بعض من الامينات الاولية الاروماتية وعدد من مركبات السلفا وتم تشخيص المركبات المحضرة باستخدام الطرائق الفيزياوية والطيفية المتوفرة.