Asian Journal of Chemistry Vol. 19, No. 6 (2007), 4939-4941

NOTE

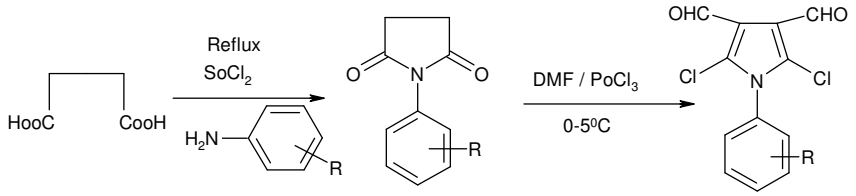
Preparation and Antimicrobial Activity study of 2,5-Dichloro-3,4-diformyl-(N-substituted phenyl)pyrroles

A.P. RAJPUT† and S.S. RAJPUT*
Department of Chemistry, S.V.S. Arts & Science College, Dondaicha-425 408, India
E-mail: ssrajput65@rediffmail.com

Various dichloro, diformyl derivatives of pyrrole were prepared on formylation using Vilsmeier Haack reagent from various succinamide derivatives. These compounds were characterized on the basis of spectral analysis. All these newly synthesized compounds were tested for their antimicrobial activity.

Key Words: Vilsmeier Haack reagent, Antimicrobial activity, N-substituted phenyl-pyrroles.

In continuation of our studies in Vilsmeier-Haack reaction, its synthetic utility and biological activity[1-6], 2,5-dichloro 3,4-diformyl(N-substituted phenyl)pyrroles (**IIa-f**) is synthesized by formylation of various succinamides (**Ia-f**) using Vilsmeier-Haack reagent ($DMF/POCl_3$). The succinimides (**Ia-f**) were prepared by known procedures[7] in which succinic acid was refluxed with thionyl chloride and different aromatic amines (**Scheme-I**). The compounds **IIa-f** were characterized on the basis of elemental analysis (Table-1) and spectral data (Table-2).



Ia-f **IIa-f**

Scheme-I

Synthesis of succinamide derivative I(a-f): A mixture of succinic acid (11.8 g, 0.1 m) and thionyl chloride (26.18 g, 2.2 m) taken and refluxed for 0.5 h. Dissolved different aromatic amines (0.1 m) in 5 mL of benzene. The aromatic amines were added in above reaction mixture and reflux the reaction mixture till HCl gas is evolved. The product was recrystallized from alcohol.

†Department of Chemistry, Jai Hind College, Dhule-424 002, India.

TABLE-1
PHYSICAL DATA

Compd.	R	m.p. (ºC)	Yield (%)	m.f.
Ia	-H	140	82.85	$C_{10}H_9NO_2$
Ib	$-CH_3$	142	82.00	$C_{11}H_{11}NO_2$
Ic	$-OCH_3$	154	74.14	$C_{11}H_{11}NO_3$
Id	*o*-Cl	102	70.82	$C_{10}H_8NO_2Cl$
Ie	*m*-Cl	104	76.38	$C_{10}H_8NO_2Cl$
If	*p*-Cl	150	74.00	$C_{10}H_8NO_2Cl$
IIa	-H	150	15.30	$C_{12}H_7NO_2Cl_2$
IIb	$-CH_3$	110	35.47	$C_{13}H_9NO_2Cl_2$
IIc	$-OCH_3$	97	26.85	$C_{13}H_9NO_2Cl_2$
IId	*o*-Cl	115	25.35	$C_{12}H_6NO_2Cl_3$
IIe	*m*-Cl	85	32.33	$C_{12}H_6NO_2Cl_3$
IIf	*p*-Cl	156	68.25	$C_{12}H_6NO_2Cl_3$

TABLE-2
SPECTRAL DATA OF COMPOUND (**IIa-f**)

Compd.	IR (KBr) (cm^{-1})	^1H NMR ($CDCl_3$) (δ ppm)
IIa	2839, (Ald-H), 1702 (>C = O) 714 (-C- Cl), 1372 (-C-N)	10.29 (1H, S, -CHO) 7.6 (5H, M, Ar-H)
IIb	2855 (Ald- H), 1682 (>C = O) 725 (-C-Cl), 1377 (-C-N)	10.29 (1H, S, -CHO) 7.26 (5H, M, Ar-H) 2.40 (3H, S, $-CH_3$)
IIc	2855 (Ald-H), 1705 (>C = O) 721 (- C- Cl), 1375 (-C-N)	10.26 (1H, S, CHO) 7.14 (4H, M, Ar-H) 3.89 (3H, S, $-OCH_3$)
IId	2855 (Ald-H), 1711 (>C = O) 762 (- C-Cl), 1392 (-C-N)	10.29 (1H, S,-CHO) 7.38 (4H, M, Ar-H)
IIe	2855 (Ald-H), 1711 (>C = O) 762 (- C- Cl), 1392 (-C-N)	10.29 (1H, S, CHO) 7.28 (4H, M, Ar-H)
IIf	2855 (C -H), 1705 (>C = O) 721 (- C- Cl), 1377 (-C-N)	10.29 (1H, S,-CHO) 7.39 (4H, M, Ar-H)

Synthesis of 2,5-dichloro 3,4-diformyl(N-substituted phenyl) pyrroles (II a-f): In DMF (0.24 mol), freshly distilled $POCl_3$ (0.12 mol) was added dropwise with constant stirring. The succinamides **Ia-f** (0.02 mol) were then added to it in small aliquots with stirring. The reaction mixture

was heated at 60-70ºC for 6 h. It was kept over night and was then slowly added to crushed ice with vigorous stirring. Stirring was continued for another 0.5 h. The resulting clear solution was then neutralized with NaOH. This solution was then heated in water bath at 50-60ºC for 0.5 h. After cooling, brown coloured solid compound **IIa-f** were obtained. These were then recrystallized using suitable solvents.

Antimicrobial activity: The compounds **IIa-f** were screened for their *in vitro* antimicrobial activity against *E. coli*, *P. vulgaris, B. subtitles, A. niger* and *S. aureous*. The cup plate agar diffusion method was used. The compounds were tested at the concentration of 1 mg/mL in polyethylene glycol. The results were compared with streptomycin at the same concentration. All these compounds showed moderate to good activity. All these compounds found inactive against *S. aureous* (Table-3).

TABLE-3
ANTIMICROBIAL ACTIVITY

Compd.	*E. coli*		*P. vilgaris*		*S. aureous*		*B. subtilis*		*A. nigar*	
	ZI	INH	ZI	INH	ZI	INH	ZI	INH	ZI	INH
IIa	19	57.58	27	81.81	-	-	26	68.42	20	47.61
IIb	28	84.00	24	72.72	-	-	30	78.75	19	45.23
IIc	-	-	27	81.81	-	-	19	57.58	25	59.52
IId	30	90.00	28	84.84	-	-	31	81.58	-	-
IIe	25	75.76	24	72.72	-	-	28	73.68	27	64.29
IIf	31	93.93	-	-	-	-	20	52.63	22	52.36
Std	33	100.00	33	100.00	-	-	38	100.00	42	100.00

ZI = Zone of inhibition (1 mm), INH = Inhibition (%)

ACKNOWLEDGEMENTS

Thanks are due to Dr. K.B. Patil, Principal, Jai Hind College, Dhule and Mr. D.A. Patil, G.M.R & D, M/s. Universal Starch Chem Allied Ltd. Starch Chem Allied Ltd. Dondaicha, for providing laboratory facilities.

REFERENCES

1. R.A. Pawar and S.M. Jain, *Indian J. Chem.*, **13**, 304 (1975).
2. R.A. Pawar, A.I. Kohak and V.N. Gogte, *Indian J. Chem*., **14B**, 375 (1986).
3. R. Hayes and O. Meth-Cohn, *Tetrahedron Lett.*, **23**, 1613 (1982).
4. H.I. Bell, M. McGurie and G.A. Freeman, *J. Hetrocycl. Chem*., **20**, 41 (1983).
5. R.A. Pawar and A.P. Rajput, *Indian J. Chem*., **28B**, 866 (1989).
6. M.L. Maddox and J.M. Muchowski, *J. Org. Chem.*, **55**, 5793 (1990).
7. K. Halwe and S.K. Srivastava, *J. Indian Chem. Soc.*, **72**, 59 (1995).

(*Received*: 17 June 2006; *Accepted*: 4 May 2007) AJC-5652