Asian Journal of Chemistry Vol. 14. No. 1 (2002), 181–184

Synthesis of 2-Thiophenyl-3-Substituted Phenyl-4-Oxo-5-Substituted Benzylidine Thiazolidine

MOHD. IDREES MOHD. SIDDIQUE*, A.G. DOSHI and A.W. RAUT
Postgraduate Department of Chemistry,
Shri Shivaji Science College, Morshi Road, Amravati-444 603, India

4-Thiazolidinones are known for their antibacterial, antifungal, antithyroid and amoebicidal properties. 2-Thiophenyl-3-substituted phenyl-4-oxo-thiazolidines were condensed with aromatic aldehyde (benzaldehyde, anisaldehyde, salicylaldehyde and 2-nitrobenzaldehyde) to yield 2-thiophenyl-3-subsituted phenyl-4-oxo-5-substituted benzylidine-thiazolidine. The structures of these compounds were confirmed by spectral and chemical data.

Key words: Thiazolidinones, synthesis, characterization.

INTRODUCTION

Literature survey reveals that various thiazolidinones[1, 2] have attracted considerable attention as they are also endowed with wide range of pharmaceutical activities. 4-oxo-thiazolidine and their 5-aryllidine derivatives possess therapeutic activity[3–5]. Thiazolidine-4-ones[6] are known to possess various biological activities, *viz.*, amoebicidal[7], hypnotic[8], anticonvulsant[9] and mosquito repellent[10].

It has also been shown to have deep impact on antithyroid activities[11] with variation in structure. The presence of N—C—S linkage in the compound has been shown to have antiradiation[12], nematocidal[13] and antifungal activity[14]. The derivatives of thiazolidine-4-one are synthesised by various workers[15].

Thus, it is worthwhile to synthesise some new members of this class.

EXPERIMENTAL

Preparation of 2-thiophenyl-3-(4′-methyl phenyl)-4-oxo-5-(2′′-hydroxy benzylidine) thiazolidine

A mixture of 2-thiophenyl-3-(4′-methyl phenyl)-4-oxo-thiazolidine(0.01 M, 2.75 g.) and salicylaldehyde (0.01 M, 1.22 mL) was dissolved in 25 mL glacial acetic acid. To this mixture anhydrous sodium acetate (0.01 M, 0.82 g) was added. This mixture was refluxed for 5 h with constant stirring. The solid separated out. washed with water twice or thrice.The resulting solid was crystallised from ethanol to get compound **(5a)**, m.p. 98°C, yield 78%.

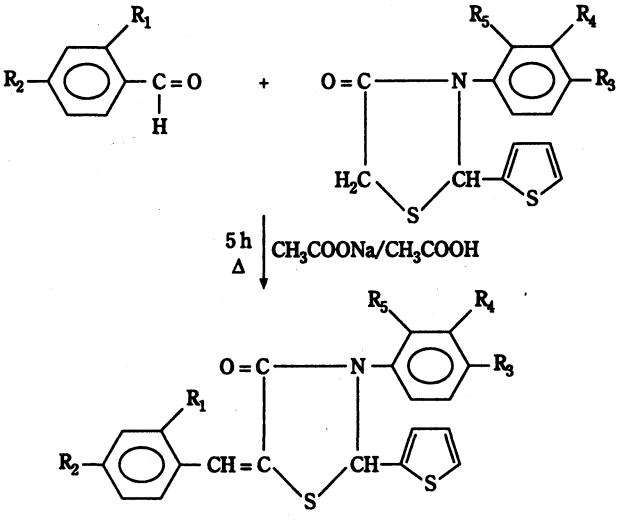
Properties of Compound 5a: It is a pale cream crystalline solid compound, m.p. 98°C. From analytical data its m.f. was found to be $C_{21}H_{17}NO_2S_2$, the molecular weight being 379. UV-Vis spectrum was recorded in methanol. λ_{max} values are 339 nm and 232 nm. These values correspond to $n \rightarrow \pi^*$ and $\pi \rightarrow \pi^*$ transition in 2-thiophenyl-3-substituted phenyl-4-oxo-5-substituted benzylidine-

Address for correspondence: c/o Dr. A.W. Raut, 25, Keshav Colony, Camp, Amravati-444 602 (M.S.), India.

thiazolidine. The large value of λ_{max} indicated extended conjugation. IR spectrum was recorded in Nujol. 2926 (C—H stretching in CH_3); 2723 (C—H stretching); 1661 (—C=O stretching); 1622 (azomethine stretching); 1240 (C—S—C stretching in thiophene); 1188 (C—N stretching); 841, 785 (C—H bending in —CH=C); 716 cm^{-1} (C—S—C in thiazolidinone). PMR spectrum was recorded in $CDCl_3$, with TMS as internal standard 1.6 δ (s, 3H, CH_3); 2.3 δ (s, 1H, N—CH); 3.8 δ (s, 1H, —CH=C); 6.2 to 7.3 δ (m, 11H, Ar—H); 9.8δ (s, 1H, OH).

From these spectral and chemical data the compound **5a** is 2-thiophenyl-3-(4′-methyl phenyl)-4-oxo-5-(2″-hydroxy benzylidine)-thiazolidine.

Reaction:



Similarly other 2-thiophenyl-3-(2′,4′-dinitro phenyl hydrazone)-4-oxo-5-substituted benzylidine-thiazolidine were also prepared. These are listed in Table-2.

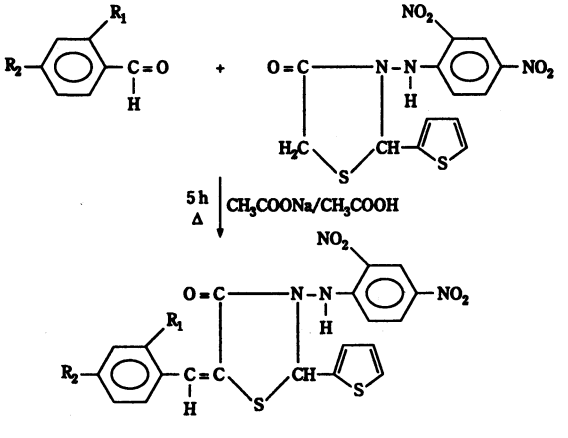


TABLE-1
SYNTHESIS, m.p., YIELD AND COLOUR OF 2-THIOPHENYL-3-(SUBSTITUTED PHENYL)-4-OXO-5-(SUBSTITUTED BENZYLIDINE)-THIAZOLIDINE.

Compound	R_1	R_2	R_3	R_4	R_5	m.p. (°C)	Yield %	m.f.	Colour
5a	OH	H	CH_3	H	H	98	78	$C_{21}H_{17}NO_2S_2$	Pale cream
5b	H	H	CH_3	H	H	119	75	$C_{21}H_{17}NOS_2$	Raw Silk
5c	H	OCH_3	CH_3	H	H	121	72	$C_{22}H_{19}NO_2S_2$	Broken white
5d	NO_2	H	CH_3	H	H	117	74	$C_{21}H_{17}N_2O_3S_2$	Broken white
6a	OH	H	Cl	H	H	80	69	$C_{20}H_{14}NO_2S_2Cl$	Raw silk
6b	H	H	Cl	H	H	89	65	$C_{20}H_{14}NOS_2Cl$	Broken wihte
6c	H	OCH_3	Cl	H	H	91	68	$C_{21}H_{16}NO_2S_2Cl$	Raw silk
6d	NO_2	H	Cl	H	H	77	67	$C_{20}H_{13}N_2O_3S_2Cl$	Pale cream
7a	OH	H	COOH	H	H	206	60	$C_{21}H_{15}NO_4S_2$	Pale cream
7b	H	H	COOH	H	H	196	59	$C_{21}H_{15}NO_3S_2$	Pale cream
7c	H	OCH_3	COOH	H	H	207	57	$C_{22}H_{17}NO_4S_2$	Pale cream
7d	NO_2	H	COOH	H	H	184	62	$C_{21}H_{14}N_2O_5S_2$	Pale cream
8a	OH	H	NO_2	H	H	130	68	$C_{20}H_{14}N_2O_4S_2$	Golden yellow
8b	H	H	NO_2	H	H	165	65	$C_{20}H_{14}N_2O_3S_2$	Pale cream
8c	H	OCH_3	NO_2	H	H	175	63	$C_{21}H_{16}N_2O_4S_2$	Pale cream
8d	NO_2	H	NO_2	H	H	127	61	$C_{20}H_{13}N_3O_5S_2$	Palewater green
9a	OH	H	H	H	NO_2	63	67	$C_{20}H_{14}N_2O_4S_2$	Golden brown
9b	H	H	H	H	NO_2	69	65	$C_{20}H_{14}N_2O_3S_2$	Golden brown
9c	H	OCH_3	H	H	NO_2	71	62	$C_{21}H_{16}N_2O_4S_2$	Golden brown
9d	NO_2	H	H	H	NO_2	81	59	$C_{20}H_{13}N_3O_5S_2$	Golden brown
10a	OH	H	H	NO_2	H	69	70	$C_{20}H_{14}N_2O_4S_2$	Pale cream
10d	NO_2	H	H	NO_2	H	89	68	$C_{20}H_{13}N_3O_5S_2$	Sandstone
11a	OH	H	H			146	68	$C_{24}H_{17}NO_2S_2$	Raw silk
11b	H	H	H			167	69	$C_{24}H_{17}NOS_2$	Broken white
11c	H	OCH_3	H			165	63	$C_{25}H_{19}NO_2S_2$	Pale cream
11d	NO_2	H	H			148	66	$C_{24}H_{16}N_2O_3S_2$	Pale cream
12a	OH	H	OCH_3	H	H	76	78	$C_{21}H_{17}NO_3S_2$	Pale cream
12b	H	H	OCH_3	H	H	99	73	$C_{21}H_{17}NO_2S_2$	Broken white
12c	H	OCH_3	OCH_3	H	H	105	75	$C_{22}H_{19}NO_3S_2$	Broken white
12d	NO_2	H	OCH_3	H	H	86	70	$C_{21}H_{16}N_2O_4S_2$	Golden brown

TABLE-2
SYNTHESIS, m.p., YIELD AND COLOUR OF 2-THIOPHENYL-3(2′,4′-DINITRO PHENYL HYDRAZONE)-4-OXO-5-SUBSTITUTED BENZYLIDINE-THIAZOLIDINE

Compound	R_1	R_2	m.p. (°C)	Yield (%)	m.f.	Colour.
13a	OH	H	205	70	$C_{20}H_{14}N_4O_6S_2$	Signal red
13b	H	H	188	72	$C_{20}H_{14}N_4O_5S_2$	Deep orange
13c	H	OCH_3	189	79	$C_{21}H_{16}N_4O_6S_2$	Orange
13d	NO_2	H	196	74	$C_{20}H_{13}N_5O_7S_2$	Golden brown

REFERENCES

1. A.M. Fahmy, K.M. Hassan, A.A. Khalif and K. Shankar, *Indian J. Chem.*, **26B,** 884 (1987).
2. O.S. Grass, A. Chimirri, P. Monoforte, G. Fenech, M. Zappalar and A.M. Monoforte, *Farmaco Ed. Sci.*, **43,** 851 (1988), *Chem Abstr.*, **110,** 50734c (1989).
3. P.B. Trivedy, N.K. Undavia, A.M. Dave, K.N. Bhatt and N.C. Desai, *Indian J. Chem.*, **32B,** 760 (1993).
4. J.J. Bhatt, B.R. Shah, P.B. Trivedi, N.K. Undana and N.K. Desai, *Indian J. Chem.*, **33B,** 1189 (1994).
5. R.S. Ludthi and S.D. Shrivastava, *Indian J. Chem.*, **36A,** 947 (1997).
6. B. Dash, M. Patra and P.K. Mahapatra, *J. Indian Chem. Soc.*, **62,** 460 (1995).
7. A.R. Surrey and R.D. Cutier, *J. Am. Chem. Soc.*, **76,** 578 (1958).
8. J. Doran and H.A. Sonie, *J. Org. Chem. Soc.*, **3,** 193 (1938).
9. H.D. Troutman and M.M. Long, *J. Am. Chem. Soc.*, **70,** 3436 (1948).
10. W.D. Skinner, H.H.C. Tony, G. Bordy and T.E. Edulard, *Chem. Abstr. (Biochem. Section)*, **83,** 18931t (1975).
11. L.P. Damkiv and M.S. Avagusino Vichzb, *Nauk. L. Prakis Vivak Med. Inst.*, **24,** 58 (1963); *Chem Abstr.*, **62,** 16596c (1965).
12. G.R. Hendricks, *J. Med. Pharm. Chem.*, **8,** 762 (1962).
13. J.R. Geisy, French Patent, 1,395,669 (9 April 1965).
14. J. Kimugawa and K. Nojare, French Patent, 8562 (4 May, 1965); *Chem Abstr.*, **63,** 5653h (1965).
15. Anjani Solonkee, Kishore Kapadia, Pankaj Mistry and Patel Jayesh, *Asian J. Chem.*, **10,** 840 (1998); *Orient J. Chem*, **15,** 143 (1999); **16,** 421 (2000).

(*Received*: 26 July 2001; *Accepted:* 1 October 2001) AJC-2473