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Synthesis of New 5-Arylazo-3-Benzyl-2-Arylimino-4-Thiazolidinones

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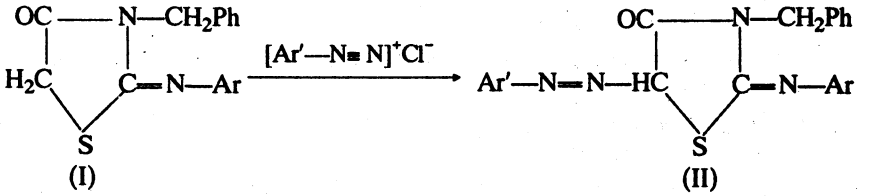
A number of 5-arylazo-3-benzyl-2-arylimino-4-thiazolidinones have been synthesised by coupling 3-benzyl-2-arylimino-4-thiazolidinones with aryldiazonium chlorides. Structure of these compounds was supported by spectral studies.

INTRODUCTION

The 4-thiazolidinone nucleus has drawn the attention of various workers due to significant biological properties such as antibacterial[1,2], anticonvulsant[3] and antifungal[4,5]. It has been reported that introduction of azo group into 4-thiazolidinones considerably enhances their antibacterial and fungicidal activities[6]. Further sulphonamides and their derivatives possess antibacterial activity and introduction of this group into 4-thiazolidinones also augments their antibacterial activity[7,8].

In view of these findings, the author synthesised two series of 5-arylazo-3-benzyl-2-arylimino-4-thiazolidinones(IIa and IIb) possessing the structural requirements essential for the significant activity from 3-benzyl-2-arylimino-4-thiazolidinones(I)[9]. The compounds of the type IIa were synthesised by coupling I with phenyl diazonium chloride and that of type of IIb by coulping I with *p*-sulphonamidophenyl diazonium chloride.

The structure of these compounds was supported with help of I.R. seectral data. The compounds are described in Table 1.



(I) (II)

(II_a), $(Ar' = C_6H_5—)$ (II_b) $(Ar' = H_2NO_2S—pC_6H_4—)$

Preparation of 5-Phenylazo-3-Benzyl-2-Benzylimino-4-Thiazolidinone

To an ice-cold solution of freshly distilled aniline (2 g; 0.22 mole) in hydrochloric acid (5 ml) was added a pre-cooled solution of sodium nitrite (1.2 g; 0.017 mole). The mixture was stirred well and a few crystals of sodium acetate were added to it. The resulting diazo solution was poured slowly with stirring

TABLE 1
5-ARYLAZO-3-BENZYL-2-ARYLIMINO-4-THIAZOLIDINONES (II)

Compd.	Ar	Ar′	m. pt. (°C)	Yield %	Molecular Formula	N(%)*	
						Found	Required
1.	Phenyl	Phenyl	53	86	$C_{22}H_{18}N_4OS$	14.37	14.51
2.	*o*-Tolyl	Phenyl	78	59	$C_{23}H_{20}N_4OS$	14.09	14.00
3.	*p*-Tolyl	Phenyl	76	62	$C_{23}H_{20}N_4OS$	14.12	14.00
4.	*p*-Anisyl	Phenyl	76	60	$C_{23}H_{20}N_4O_2S$	13.54	13.46
5.	*p*-Phenetyl	Phenyl	82	76	$C_{24}H_{22}N_4O_2S$	13.09	13.02
6.	*p*-Fluorophenyl	Phenyl	90	52	$C_{22}H_{17}FN_4OS$	14.17	13.86
7.	*o*-Chlorophenyl	Phenyl	90	68	$C_{22}H_{17}ClN_4OS$	13.02	13.32
8.	*p*-Chlorophenyl	Phenyl	73	60	$C_{22}H_{17}ClN_4OS$	13.28	13.32
9.	*p*-Iodophenyl	Phenyl	117	72	$C_{22}H_{17}IN_4OS$	11.16	11.14
10.	*o*-Hydroxyphenyl	Phenyl	105	59	$C_{22}H_{18}N_4O_2S$	13.86	13.93
11.	*m*-Hydroxyphenyl	Phenyl	145	56	$C_{22}H_{18}N_4O_2S$	14.15	13.93
12.	1-Naphthyl	Phenyl	110	61	$C_{26}H_{20}N_4OS$	13.06	12.84
13.	2-Pyridyl	Phenyl	115	66	$C_{21}H_{17}N_5OS$	17.81	18.09
14.	Cyclohexyl	Phenyl	71	73	$C_{22}H_{24}N_4OS$	14.17	14.28
15.	*p*-Bromophenyl	*p*-Sulphonamidophenyl	125	73	$C_{22}H_{18}BrN_5O_3S_2$	12.98	12.87
16.	*p*-Iodophenyl	*p*-Sulphonamidophenyl	165[d]	69	$C_{22}H_{19}IN_5O_3S_2$	11.78	11.84
17.	*o*-Hydroxyphenyl	*p*-Sulphonamidophenyl	103	62	$C_{22}H_{19}N_5O_4S_2$	14.64	14.55
18.	*m*-Hydroxyphenyl	*p*-Sulphonamidophenyl	143	56	$C_{22}H_{19}N_5O_4S_2$	14.46	14.55
19.	*p*-Hydroxyphenyl	*p*-Sulphonamidophenyl	140[d]	62	$C_{22}H_{19}N_5O_4S_2$	14.24	14.55
20.	4-Hydroxy-2-methylphenyl	*p*-Sulphonamidophenyl	147	73	$C_{23}H_{21}N_5O_4S_2$	14.08	14.14
21.	5-Chloro-2-hydroxyphenyl	*p*-Sulphonamidophenyl	145	57	$C_{22}H_{18}ClN_5O_4S_2$	13.75	13.58

*All the compounds were also analysed for C, H and satisfactory results were obtained.

to an ice-cold solution of 3-benzyl-2-benzylimino-4-thiazolidinones (2g; 0.007 mole) in acetone (25 ml) and glacial acetic acid (25 ml) and the stirring was continued for half an hour. The reaction mixture was diluted with water when a coloured-precipitate was obtained. The precipitate was washed with water, dried and recrystallised from ethanol, m.pt. 78°C, yield 67% (found: C 69.27, H 5.13 and N 13.73%; calcd. for $C_{23}H_{20}N_4OS$: C 69.00, H 5.00 and N 14.00%).

I.R. spectrum of the compound showed characteristic absorption peaks at (cm^{-1}): 2960, 2885 (CH_2); 1725 (C═O); 1645 (C═N); 1610, 1460 (—N═N—) and 745 (C—S).

Other 5-phenylazo derivatives synthesised by this method are listed in Table 1 along with their relevant data.

Preparation of 5-p-Sulphonamidophenylazo-3-Benzyl-2-Arylimino-4-Thiazolidinone

A solution of sulphanilamide (1.2 g, 0.007 mole) dissolved in concentrated hydrochloric acid (5 ml) and water (10 ml) was cooled in ice-bath and diazotised with sodium nitrite (1.2 g, 0.017 mole) in water (5 ml), the *p*-sulphonamidophenyl diazonium chloride solution thus obtained was added slowly with stirring to a previously cooled solution of 3-benzyl-2-*o*-tolylimino-4-thiazolidinone (2 g, 0.007 mole) in ethanol (30 ml) and acetone (15 ml) containing crystals of sodium acetate (3 g, 0.022 mole). The stirring was continued for about 1 hr. Excess of water was added to the reaction mixture. After several hrs, a coloured precipitate was obtained which was collected, washed with water, dried and recrystallised from ethanol, m.pt. 86°C, yield 62% (found: C 57.87, H 4.31 and N 14.46%; calcd. for $C_{23}H_{21}N_5O_3S_2$: C 57.62, H 4.38 and N 14.61%).

I.R. spectrum of the compound showed the characteristic absorption peaks at (cm^{-1}: 3450, 3375 (NH); 2960, 2870 (CH_2); 1740 (C═O), 1650 (C═N); 1610, 1470 (—N═N—), 1350, 1150 (SO_2) and 745 (C—S).

Similarly other 5-p-sulphonamidophenylazo derivatives were prepared and are listed in Table 1 along with their relevant data.

Biological screenings for different activities of the compounds are in progress. The results will be communicated in another paper.

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