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SYNTHESIS OF SOME NEW 3-BENZYL-2-ARYLIMINO-4-THIAZOLIDINONE-1,1-DIOXIDES

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Twenty new 3-benzyl-2-arylimino-4-thiazolidinone-1,1-dioxides (2) were synthesized by oxidising 3-benzyl-2-arylimino-4-thiazolidinones (1) with potassium permanganate in glacial acetic acid. The homogeneity and purity was confirmed by TLC and some of the dioxides were screened for their insecticidal, acaricidal and herbicidal activities.

A number of 3-substituted-2-arylimino-4-thiazolidinones were synthesized earlier¹⁻⁴ and some of them showed interesting biological properties.^{4,5} In view of these findings, the author synthesized twenty new 1,1-dioxides (2) from 3-benzyl-2-arylimino-4-thiazolidinones (1)⁴ in order to find out what effect conversion of the cyclic —S— group to an SO₂ group will have upon the biological properties. These compounds were obtained by oxidizing 3-benzyl-2-arylimino-4-thiazolidinones dissolved in glacial acetic acid with potassium permanganate. The purity and homogeneity were tested by TLC.

EXPERIMENTAL

Melting points were taken in open capillary tubes and are uncorrected. uv spectra were taken on a Perkin-Elmer 202 automatic recording spectrometer using methanol as a solvent. ir spectra were taken on a Perkin-Elmer 621 spectrophotometer in the form of KBr discs. nmr spectra were recorded on a Varian A-60D machine in CDCl₂ with TMS as an internal standard.

3-Benzyl-2-arylimino-4-thiazolidinones were synthesized by condensing 1-benzyl-3-aryl-2-thioureas with monochloroacetic acid in the presence of anhydrous sodium acetate by the method described earlier.⁴

3-Benzyl-2-o-ethoxyphenylimino-4-thiazolidinone-1,1-dioxide

To a solution of 3-benzyl-2-o-ethoxyphenylimino-4-thiazolidinone (3.26 g, 0.01 mole) in glacial acetic acid (30 ml) was added 3% aqueous solution of potassium permanganate (75 ml) drop by drop with constant stirring for about half an hour, and the stirring was continued for another half an hour. At the end of the reaction, sulfur dioxide was passed through the mixture (to remove the excess of potassium permanganate) till a color-less solution was obtained. Excess of water was added to the reaction mixture and it was allowed to stand overnight; a semi-solid precipitate was separated. It was collected, washed thoroughly with water and recrystallized from absolute ethanol, m.p. 102; yield 2.94 g (82%).

The uv spectrum of the compound showed adsorption maxima at 217 and 282 m μ . On comparing this with the uv spectrum of 3-benzyl-2-o-ethoxyphenylimino-4-thiazolidinone it can be seen that conversion of 4-thiazolidinone into 1,1-dioxide brings about bathochromic shifts.

The ir spectrum showed peaks at: 2940, 2880 cm $^{-1}$ (CH₂ stretching), 1725 cm $^{-1}$ (C=O stretching), 1650 cm $^{-1}$ (C=N stretching), 1350, 1140 cm $^{-1}$ (SO₂ stretching) and 750 cm $^{-1}$ (C-S stretching).

The nmr spectrum (CDCl₃) showed different signals of chemical shifts at δ : 1.40 (t, 3H; C₆H₅-O-CH₂-CH₃-o, J=7.5 cps), 4.12 (q, 2H; C₆H₅-O- CH_2 CH₃-o. J=7.5 cps), 3.88 (S, 2H; cyclic $-CH_2$ CO), 5.18 (S, 2H; due to C₆H₃ CH_2) and 7.38 (complex multiplet, 9H; aromatic protons).

All other 3-benzyl-2-arylimino-4-thiazolidinone-1,1-dioxides were prepared by the above method. Their physical constants along with the analytical data are given in Table I.

BIOLOGICAL TESTING

Compound Nos. 7 and 18 were subjected to nematicidal, insecticidal, acaricidal and herbicidal testings but none of these compounds showed any activity.

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TABLE I
3-Benzyl-2-arylimino-4-thiazolidinone-1,1-dioxides (2)

S. No.	Ar]	Molecular formula	Yield %	M.p.			Analyses	
S. 190				°Ć		%C	%Н	%N
1.	Phenyl	C ₁₆ H ₁₄ N ₂ O ₃ S	72	72	Found	61.24	4.30	9.08
2.	o Tobul	CHNOS	63	90	Calcd.	61.15	4.46	8.92
2.	o-Tolyl	$C_{17}H_{16}N_2O_3S$	62	80	Found Calcd.	62.05 62.19	4.81 4.88	8.73 8.54
3.	p-Tolyl	$C_{17}H_{16}N_2O_3S$	68	87	Found	62.37	4.86	8.36
٥,	p roiji	0171116112035	00	0,	Calcd.	62.19	4.88	8.54
4.	o-Ethoxyphenyl	$C_{18}H_{18}N_2O_4S$	82	102	Found	60.18	5.22	7.95
		18-18-2-4-	-		Calcd.	60.31	5.03	7.82
5.	p-Fluorophenyl	$C_{16}H_{13}FN_2O_3S$	54	100	Found	57.76	4.10	8.51
					Calcd.	57.83	3.91	8.43
6.	o-Chlorophenyl	$C_{16}H_{13}CIN_2O_3S$	50	80	Found	55.21	3.86	8.19
					Calcd.	55.09	3.73	8.03
7.	p-Chlorophenyl	$C_{16}H_{13}ClN_2O_3S$	51	70	Found	55.18	3.88	8.24
					Calcd.	55.09	3.73	8.03
8.	o-Bromophenyl	$C_{16}H_{13}BrN_2O_3S$	53	124	Found	48.69	3.42	7.25
					Calcd.	48.85	3.31	7.12
9.	p-Bromophenyl	$C_{16}H_{13}BrN_2O_3S$	55	119	Found	48.97	3.52	7.24
10		C II DIOC		1.55	Calcd.	48.85	3.31	7.12
10.	p-Iodophenyl	$C_{16}H_{13}IN_2O_3S$	57	155	Found	43.76	2.84	6.48
11	2.5 Diables subsess	I C II CINOR	03	100	Calcd.	43.64	2.95	6.36
11.	2,5-Dichloropheny	$C_{16}H_{12}Cl_2N_2O_3S$	82	100	Found Calcd.	50.25 50.13	3.02 3.13	7.52 7.31
12.	o-Hydroxyphenyl	$C_{16}H_{14}N_2O_4S$	63	210	Found	58.29	4.12	8.62
	o-rrydroxyphenyr	C ₁₆ 11 ₁₄ 1 4 ₂ O ₄ 5	0.5	(decompn.)	Calcd.	58.18	4.12	8.48
13. 14.	m-Hydroxyphenyl	$C_{16}H_{14}N_2O_4S$	68	220	Found	58.02	4.30	8.52
	m riydroxyphonyr	0161114112045	00	(decompn.)		58.18	4.24	8.48
	p-Hydroxyphenyl	$C_{16}H_{14}N_2O_4S$	68	223–25	Found	58.22	4.36	8.30
	h h h h h h h h h h h h h h h h h h	0 1622142 120 40		(decompn.)	Calcd.	58.18	4.24	8.48
15.	4-Hydroxy-2-	$C_{17}H_{16}N_2O_4S$	63	220	Found	59.16	4.78	8.32
	methylphenyl	17 10 2 4		(decompn.)	Calcd.	59.30	4.65	8.14
16.	5-Chloro-2-	$C_{16}H_{13}ClN_2O_4S$	61	>250	Found	52.79	3.33	7.56
	hydroxy- methylphenyl	1V 1V 2 4			Calcd.	52.67	3.57	7.68
17.	1-Naphthyl	$C_{20}H_{16}N_2O_3S$	64	117	Found	65.81	4.48	7.78
		20 10 2 3			Calcd.	65.93	*4.39	7.69
18.	2-Pyridyl	$C_{15}H_{13}N_3O_3S$	63	130	Found	57.31	4.00	13.1
	•				Calcd.	57.14	4.13	13.33
19.	Benzyl	$C_{17}H_{16}N_2O_3S$	54	76	Found	61.97	4.67	8.63
					Calcd.	62.19	4.88	8.54
20.	Cyclohexyl	$C_{16}H_{20}N_2O_3S$	54	83	Found	60.15	6.40	8.86
					Calcd.	60.00	6.25	8.75

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