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Synthesis and antifungal activity of some naphthalene

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Abstract
This paper present the naphthalene-1,5-dioxymethylene bis-(2-arylamino-3-yl-thiazolidinones) and bis-(1-aryl-3-yl-2-thiohydantoins) were prepared from Naphthalene-1,5-dioxymethylene bis-(4-aryl-3-thiocarbamides) and screened for their antifungal activity against Penicillium expansum, *Nigrospora* sp. and Trichothecium sp.

Keywords: Bis-thiazolidinones, thiohydantoins, antifungal activity

Introduction
Various bis-thiazolidinones exhibit antitubercular [1], hypnotic [2] and antifungal activity. The presence of N-C-S linkage has been postulated to account for the antifungal activity of 4. Thiozolidinones [3-7]. Thionydantoins are also associated with a broad biocidal spectrum [8, 9]. Hence, it was thought interesting to convert 1,5-dihydroxynaphthalene to Naphthalene-1,5-dioxymethylene bis-(4-aryl-3-thiocarbamides) (3), which on cyclisation with monochloroacetic acid/sodium acetate and monochloroacetic acid/pyridine were converted to bis-(2-arylimino-3-yl-thioazolidin4-ones) (4) and bis (1-aryl-3-yl-2-thiohydantoins) (5) respectively. The steps involved in the synthesis are shown in scheme 1. The antifungal activity of all these compound 3,4,5 has also been studied.

Experimental
Melting points were determined in open capillary tubes and are uncorrected. The IR spectra were recorded in KBr pellets on a Nicolet 400 D spectrophotometer and ^{1}H NMR spectra in CDCI, on Hitachi R-1500, 60 MHz spectrometer using TMS as an internal standard. All chemicals used were of laboratory grade.

Table 1(a): Analytical and spectral data of naphthalene-1,5-dioxymethylene bis-(4-aryl-3-thiocarbamide) (3)

Compound*	R	Mol. Formula	M.P.	Yield %	Analysis (%) Found (Calcd.)	
					%N	%S
3a	C_6H_5	$C_{28}H_{24}N_4O_4S_2$	170	45	10.11	11.65
					(10.29)	(11.76)
3b	p-Cl-C_6H_4	$C_{28}H_{22}Cl_2N_4O_4S_2$	125	42	8.95	10.35
					(9.15)	(10.45)
3c	p-Br-C_6H_4	$C_{28}H_{22}Br_2N_4O_4S_2$	156	61	7.85	9.01
					(7.97)	(9.11)
3d	p-OCH_3-C_6H_4	$C_{30}H_{28}N_4O_6S_2$	190	56	9.12	10.45
					(9.27)	(10.59)
3e	p-CH_3-C_6H_4	$C_{30}H_{28}N_4O_4S_2$	185	67	9.75	11.10
					(9.79)	(11.18)
3f	p-NO_2-CH_4	$C_{28}H_{22}N_6O_8S_2$	176	56	13.10	9.95
					(13.24)	(10.09)
3g	o-Cl-C_6H_4	$C_{28}H_{22}Cl_2N_4O_4S_2$	210	36	8.80	10.15
					(9.15)	(10.45)
3h	m-Cl-C_6H_4	$C_{28}H_{22}Cl_2N_4O_4S_2$	230	45	8.95	10.35
					(9.15)	(19.45)

IR (KBr): 3320 (N-H, Stretch). 1650 (-CONH-), 1610 (C=C aromatic). 1225 (-NHCSNH-)

Table 1(b): Antifungal activity of naphthalene-1,5-dioxymethylene bis-(4-aryl-3-thiocarbamide) (3)

Compound	Zone of inhibition at 1000 ppm (%)		Trichothecium sp.
	Penicillium expansum	Nitrospora sp.	
3a	100	79	100
3b	100	100	100
3c	75	81	78
3d	55	100	100
3e	100	100	42
3f	68	72	85
3g	82	100	100
3h	100	62	35

Table 2(a): Analytical and spectral data of naphthalene-1,5-dioxymethylene bis-(2-aryl-imino-3-yl-thiazolidin-4-ones)(4)

Compound*	R	Mol. Formula	M.P.	Yield %	Analysis (%) found (Calcd.)	
					%N	%S
4a	C_6H_5	$C_{32}H_{24}N_4O_6S_2$	154	45	8.85	10.15
					(8.97)	(10.24)
4b	p-Cl-C_6H_4	$C_{32}H_{22}Cl_2N_4O_6S_2$	134	42	7.95	9.25
					(8.09)	(9.24)
4c	p-Br-C_6H_4	$C_{32}H_{22}Br_2N_4O_6S_2$	156	62	7.01	8.01
					(7.16)	(8.18)
4d	p-OCH_3-C_6H_4	$C_{34}H_{28}N_4O_6S_2$	143	54	8.10	9.24
					(8.18)	(9.35)
4e	p-CH_3-C_6H_4	$C_{34}H_{28}N_4O_6S_2$	224	61	8.45	9.75
					(8.58)	(9.81)
4f	p-NO_2-CH_4	$C_{32}H_{22}N_6O_{10}S_2$	156	46	11.65	8.85
					(11.76)	(8.96)
4g	o-Cl-C_6H_4	$C_{32}H_{22}Cl_2N_4O_6S_2$	216	66	8.01	9.15
					(8.09)	(9.24)
4h	m-Cl-C_6H_4	$C_{32}H_{22}Cl_2N_4O_6S_2$	121	35	7.95	9.10
					(8.09)	(9.24)

IR (KB): 3010 (CH). 1650 (C=0, exocyclic), 1640 (C=0, endocyclic), 1600 (C=C aromatic), 1230 (C-S-C thiazolidinone)

Table-2(b): Antifungal activity of naphthalene-1,5-dioxymethylene bis-(2-aryl-imino-3-yl-thiazolidin-4-ones) (4)

Compound	Zone of inhibition at 1000 ppm (%)		Trichothesium sp.
	Penicillium expansum	Nitrospora sp.	
4a	100	75	100
4b	100	100	100
4c	85	57	88
4d	56	100	100
4e	100	100	45
4f	58	78	65
4g	89	100	85
4h	68	60	35

Naphthalene-1,5-dioxymethylene bis-(4-aryl-3-thiocarbamides) (3)

A mixture of ammonium thiocyanate (0.1 mol) and acetone (50 ml) was placed in a flask and solution of diacetyl chloride of 1,5,dihydroxynaphthalene (0.05 mol) in acetone (50 ml) was added through a dropping funnel with stirring. Aryl amine (0.1 mol) in acetone (50 ml) was then added to the reaction mixture in small portions. When the addition was over, the reaction mixture was refluxed for 3h, cooled and poured into ice water. The resulting precipitate was filtered and washed with water and crystallized from ethanol. Various Naphthalene-1,5-dioxymethylene bis-(4-aryl-3-thiocarbamides) thus prepared are recorded in Table 1.

Table 3(a): Analytical and spectral data of naphthalene-1,5-dioxymethylene bis-(1-aryl-3-yl-2-thiohydantoins) (5)

Compound*	R	Mol. Formula	M.P.	Yield %	Analysis (%) found (Calcd.)	
					%N	%S
5a	C_6H_5	$C_{32}H_{24}N_4O_6S_2$	154	45	8.79	10.20
					(8.97)	(10.24)
5b	p-Cl-C_6H_4	$C_{32}H_{22}Cl_2N_4O_6S_2$	134	42	7.85	9.15
					(8.09)	(9.24)
5c	p-Br-C_6H_4	$C_{32}H_{22}Br_2N_4O_6S_2$	156	62	7.08	8.12
					(7.16)	(8.18)
5d	p-OCH_3-C_6H_4	$C_{34}H_{28}N_4O_6S_2$	143	54	7.95	9.14
					(8.18)	(9.35)

5e	p-CH_3-C_6H_4	$C_{34}H_{28}N_4O_6S_2$	224	61	8.50	9.70
					(8.58)	(9.81)
5f	p-NO_2-CH_4	$C_{32}H_{22}N_6O_{10}S_2$	156	46	11.70	8.75
					(11.76)	(8.96)
5g	o-Cl-C_6H_4	$C_{32}H_{22}Cl_2N_4O_6S_2$	216	66	7.85	9.01
					(8.09)	(9.24)
5h	m-Cl-C_6H_4	$C_{32}H_{22}Cl_2N_4O_6S_2$	121	35	8.01	9.15
					(8.09)	(9.24)

IR (KBr): 1670 (C=0, exocyclic), 1660 (C=O, endocyclic), 1580 (C=C aromatic), 1320 (C=S)

Table 3(b): Antifungal activity of naphthalene-1,5-dioxymethylene bis-(1-aryl-3-yl-thiohydantions) (5)

Compound	Zone of inhibition at 1000 ppm (%)		Trichothesium sp.
	Penicillium expansum	Nitrospora sp.	
5a	100	90	100
5b	100	100	65
5c	100	57	85
5d	45	100	100
5e	100	100	75
5f	68	85	43
5g	85	100	80
5h	100	80	100

Naphthalene-1,5-dioxymethylene bis-(2arylimino-3-yl-thiazolidin-4-ones) (4)

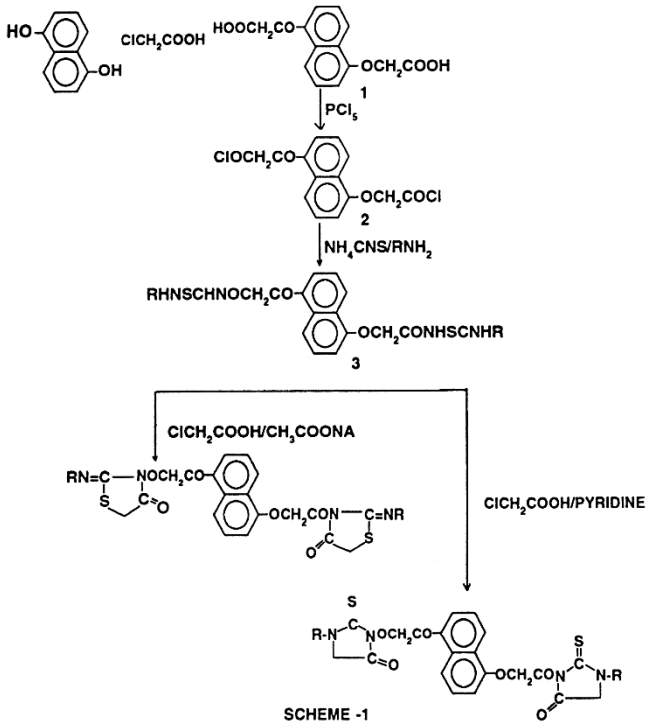
A mixture of Naphthalene-1,5-dioxymethylene bis-(4-aryl-3-thiocarbamides) (0.01 mol), monochloroacetic acid (0.02 mol) and anhydrous sodium acetate (0.02 mol) was refluxed in dimethyl formamide (50 ml) for 4-5 h. The reaction mixture was cooled, poured into ice-cold water and kept overnight. The precipitate thus obtained was filtered, dried and crystallized from ethanol. The compounds (Table 2) were characterized by their melting points and analytical data along with spectral analysis.

Naphthalene-1,5-dioxymethylene bis-(1-aryl-3-yb2-thiohydantoins) (5)

Naphthalene: 1,5dioxymethylene bis-(4-aryl-3-thiocarbamides) (0.0025 mol) was dissolved in minimum amount of pyridine. To this mixture alcohol and dioxane (1:1) was added. This mixture was refluxed for 10 h. On cooling, it was poured into ice cold water. Solid mass that separated out was filtered, dried and crystallized from ethanol. Analytical and spectral data of these compound are recorded in Table 3.

Antifungal activity

Compounds 3,4,5 were screened for their antifungal activity against Penicillium expansum, *Nigrospora* sp. and Trichothecium sp., as the test fungi by paper-disc plate method at concentration levels of 2.0 and 0.2% (w/v) in DMSO. Standard PDA medium was used. Filter paper discs of diameter 12 mm were used and the diameters of zones of inhibition formed around each disc after incubating for a period of 48h at 25-30 °C were recorded. Results were compared with reference to fungicides, Dithane-Z78 and Thiram-75W. The compound 4 and 5 were found to be less fungicidal then their precursor 3. On comparison with reference to fungicides, they were found to be less effective.



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