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Synthesis of Some Sulphonamido and Amino Alkanes and Their Antifungal Activity

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A series of new substituted sulphonamido alkanes and amino alkanes have been prepared. The structures of these compounds were confirmed by spectral and analytical data. These compounds were screened for their antifungal activity

INTRODUCTION

The oxygen, nitrogen and sulphur containing heterocyclic compounds have been used in various medicinal fields. The sulphonamide derivatives were associated with various physiological and biological properties.[1, 2] Also the sulphur containing heterocyclic compounds possess anticancer and antifungal properties[3–5]. The amino alkane compounds also possess various biological properties[6, 7]. The above findings and observations regarding less attention on the synthesis of sulphonamido and aminoalkanes have prompted us to undertake synthesis of substituted sulphonamido alkanes and substituted amino alkanes.

The 1-(substituted heteroxy)-2/3-substituted sulphonamido alkanes (1–28) and 1-(substituted heteroxy)-2/3-alkyl amino alkanes (29–42) (scheme 1) were synthesised by treating substituted heteroxybromo alkanes[8] with sulphonamides and alkyl amines respectively.

EXPERIMENTAL

The melting points of all the compounds were determined in open capillaries using paraffin bath and are uncorrected. IR spectra are recorded on Perkin-Elmer 1420 spectrophotometer in nujol and PMR spectra on FT 80A PMR spectrophotometer using $CDCl_3$ as solvent and TMS as internal standard. The completion of reaction and purity of synthesised compounds were checked by TLC.

1-(4′-Methyl coumarin-7′-oxy)-2-(3′,4′-dichloro sulphonamido) ethane (5)

To the solution of 3,4-dichloro sulphonamide (0.01 mole 2.25 g) in dry pyridine, 1-(4′-methyl coumarin-7′-oxy)-2-ethyl bromide (0.01 mole, 2.83 g) was added with stirring. The reaction mixture was refluxed in water bath for 2 h. Then the reaction mixture was neutralized with acetic acid. The solid separated was extracted with ether. The ether layer was evaporated to get the title product.

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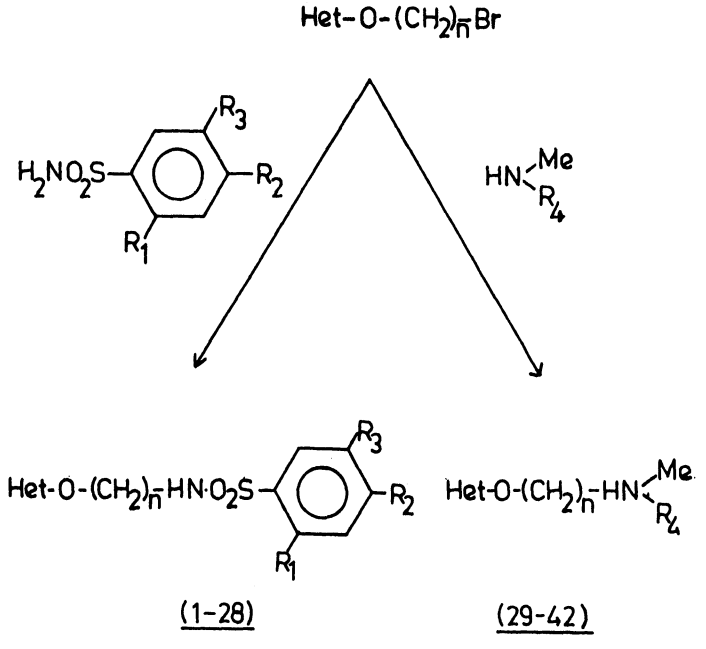
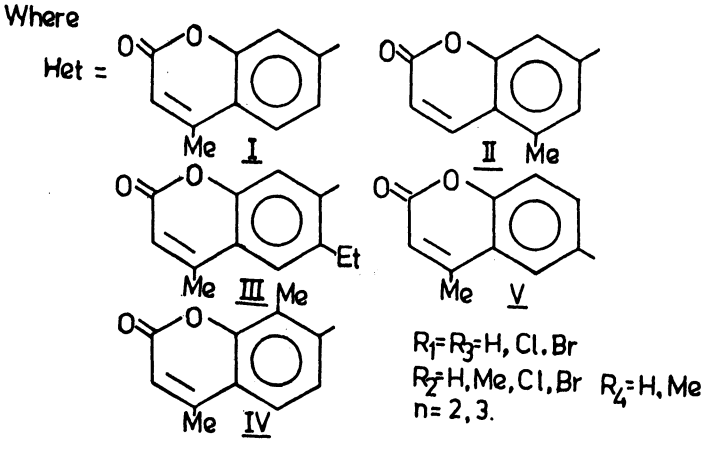
It was recrystallised from aq. alcohol. Yield 65%, m.p. 101°C; IR: 3360 (—NH stretch), 1720 (>C=O), 1610 (C=C) and 1250 cm^{-1} (C—O—C—); PMR: δ





Scheme-I

2.2 (s, 3H, CH_3), 3.5–3.7 (t, 2H, —CH_2—CH_2—O—), 4.3-4.5 (t, 2H, —OCH_2), 6.1 (s, 1H, >C=C—H), 6.6 (s, 1H, NH) and 6.8–7.3 (m, 6H, Ar—H).

All other compounds of this series were prepared by above procedure. Their m.p., yields and analytical data are given in Table-1.

TABLE-1
CHARACTERISATION DATA OF 1-(SUBSTITUTED HETEROXY)02/3-SUBSTITUTED SULPHONAMIDO ALKANES (1–28)

Compd. No.	Het	R_2	R_3	R_1	n	Yield (%)	m.p. (°C)	Molecular formula	%N	
									Found	Calcd.
1.	I	H	Cl	Cl	2	67	161	$C_{18}H_{15}O_5NSCl_2$	3.33	3.27
2.	I	H	Br	Br	2	62	157	$C_{18}H_{15}O_5NSBr_2$	2.81	2.70
3.	I	Br	H	H	2	60	170	$C_{18}H_{16}O_5NSBr$	3.09	3.19
4.	I	Cl	H	H	2	70	170	$C_{18}H_{16}O_5NSCl$	3.72	3.56
5.	I	Cl	Cl	H	2	65	101	$C_{18}H_{15}O_5NSCl_2$	3.33	3.27
6.	I	Me	H	H	2	70	140	$C_{19}H_{19}O_5NS$	3.56	3.75
7.	II	Cl	H	H	2	65	150	$C_{18}H_{16}O_5NSCl$	3.70	3.56
8.	II	Cl	Cl	H	2	60	175	$C_{18}H_{15}O_4NSCl_2$	3.50	3.27
9.	III	H	Br	Br	2	66	170	$C_{20}H_{19}O_5NSBr_2$	2.67	2.56
10.	III	H	Cl	Cl	2	65	172	$C_{20}H_{19}O_5NSCl_2$	3.21	3.07
11.	III	Cl	H	H	2	68	205	$C_{20}H_{20}NSCl$	4.50	4.10
12.	III	Cl	Cl	H	2	60	185	$C_{20}H_{19}O_5NSCl_2$	3.21	3.07
13.	III	Br	H	H	2	69	170	$C_{20}H_{20}O_5NSBr$	3,20	3.00
14.	III	Me	H	H	2	70	178	$C_{21}H_{23}O_5NS$	3.56	3.49
15.	IV	H	Br	Br	2	60	178	$C_{19}H_{17}O_5NSBr_2$	2.53	2.63
16.	IV	H	Cl	Cl	2	72	160	$C_{19}H_{17}O_5NSCl_2$	3.00	3.16
17.	IV	Br	H	H	2	70	175	$C_{19}H_{10}O_5NSBr$	3.11	3.09
18.	IV	Br	H	H	3	70	120	$C_{20}H_{20}O_5NSBr$	3.20	3.00
19.	IV	Cl	H	H	2	65	151	$C_{19}H_{18}O_5NSCl$	3.50	3.43
20.	IV	Cl	H	H	3	67	130	$C_{20}H_{20}O_5NSCl$	3.41	3.32
21.	IV	Cl	Cl	H	2	59	180	$C_{19}H_{17}O_5NSCl_2$	3.42	3.16
22.	IV	Cl	Cl	H	3	65	155	$C_{20}H_{18}O_5NSCl_2$	3.23	3.09
23.	IV	Me	H	H	2	66	135	$C_{20}H_{21}O_5NS$	3.51	3.61
24.	V	H	Br	Br	2	54	190	$C_{18}H_{15}O_5NSBr_2$	2.60	2.70
25.	V	H	Cl	Cl	2	60	180	$C_{18}H_{15}O_5NSCl_2$	3.50	3.27
26.	V	Br	H	H	2	65	175	$C_{18}H_{16}O_5NSBr$	3.25	3.19
27.	V	Cl	H	H	2	70	152	$C_{18}H_{16}O_5NSCl$	3.41	3.56
28.	V	Cl	Cl	H	2	75	173	$C_{18}H_{15}O_5NSCl_2$	3.39	3.27

All compounds were crystallised from aq. alcohol.

1-(4′-Methyl coumarin-7′-oxy)-2-(methyl amino) ethance (29)

To a solution of 1-(4′-methyl coumarin-7′-oxy)-2-ethyl bromide (0.01 mole, 2.83 g) in dry pyridine, methyl amine (0.05 mole, 1.55 g) was added. The reaction mixture was refluxed for 2 h and then it was neautralised by acetic acid. The solid thus obtained was extracted with ether. The ethereal layer was evaporated to get the title product. It was crystallised from aq. alcohol. Yield 65%, m.p. 170°C; IR: 3400 (NH stretch), 1740 (C=O), 1610 (C=C) and 1260 cm^{-1} (C—O—C); PMR: δ 2.3 (s, 3H, CH_3), 2.4 (s, 3H, NH—CH_3), 3.6–3.8 (t, 2H, —CH_2—CH_2—O), 4.2–4.4 (t, 2H, OCH_2), 6.2 (1H, C=C—H), 6.3 (s, 1H, NH) and 6.9–7.2 (m, 3H, Ar—H).

All other compunds of this series were prepared by above procedure. Their m.pts. yields and analytical data are given in Table-2.

TABLE-2
CHARACTERISATION DATA OF 1-(SUBSTITUTED HETEROXY)-2/3-ALKYL AMINO ALKANES (29–42)

Compd No.	Het	R_4	n	Yield (%)	m.p. (°C)	Molecular formula	% N	
							Found	Calcd.
29.	I	H	2	65	170	$C_{13}H_{15}O_3N$	6.21	6.00
30.	I	Me	2	60	178	$C_{14}H_{17}O_3N$	5.49	5.66
31.	I	H	3	66	80	$C_{14}H_{17}O_3N$	5.51	5.66
32.	I	Me	3	60	83	$C_{15}H_{19}O_3N$	5.44	5.36
33.	II	H	2	65	127	$C_{13}H_{15}O_3N$	6.21	6.00
34.	II	Me	2	65	235	$C_{14}H_{17}O_3N$	5.50	5.66
35.	II	H	3	75	194	$C_{14}H_{17}O_3N$	5.49	5.66
36.	II	Me	3	60	87	$C_{15}H_{19}O_3N$	5.21	5.36
37.	III	H	2	60	154	$C_{15}H_{19}O_3N$	5.40	5.36
38.	III	Me	2	60	207	$C_{16}H_{21}O_3N$	5.19	5.09
39.	IV	H	2	57	209	$C_{15}H_{19}O_3N$	5.43	5.36
40.	IV	Me	2	69	240	$C_{15}H_{19}O_3N$	5.30	5.36
41.	V	H	2	70	240	$C_{13}H_{15}O_3N$	6.10	6.00
42.	V	Me	2	65	242	$C_{14}H_{17}O_3N$	5.49	5.66

All compounds were crystallised from aq. alcohol.

RESULTS AND DISCVSSION.

The synthesised substituted sulphonyl alkanes were screened for their antifungal activity against *Alternaria brassicicola* and *Fusarium udam* and some of them found to possess good antifungal activity while all the amino alkanes were found to be neutral to both *Alternaria brassicicola* and *Fusarium udam*. The activity data of these compounds is tabulated in Table-3. During this screening carbendazim was used as reference antifungal compound.

TABLE-3
THE ANTIFUNGAL ACTIVITY SCREENING DATA OF 1-(SUBSTITUTED HETEROXY)-2/3-SUBSTITUTED SULPHONAMIDO ALKANE (1–28)

Compd. No.	*Alternaria brassicicola*		*Fusarium udam*	
	Antagonists	Neutral	Antagonists	Neutral
1.	–	+	+9	–
2.	–	+	–	+
3.	+19	–	–	+
4.	–	+	+7	–
5.	+17	–	+16	–
6.	+7	–	+3	–
7.	–	+	–	+
8.	+11	–	+14	–
9.	+9	+	–	+
10.	+16	–	+16	–
11.	–	–	+18	–
12.	+17	–	+11	–
13.	+6	–	+9	–
14.	+15	–	+12	–
15.	–	+	–	+
16.	–	+	+7	–
17.	–	+	+9	–
18.	–	+	–	+
19.	–	+	+6	–
20.	+7	–	+6	–
21.	+9	–	–	+
22.	–	+	+10	–
23.	–	+	–	+
24.	–	+	+9	–
25.	+9	–	+12	–
26.	+11	–	–	+
27.	–	+	+7	–
28.	+7	–	+11	–
Bavistan (Carbendazim)	+15	–	+13	–

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