

Synthesis of Bis(4-arylthiosemicarbazido)-,  
Bis(2-arylamino-1,3,4-thiadiazol-5-yl) and  
Bis(4-aryl-1,2,4-triazolin-3-thione-5-yl)pentanes and Related Compounds.

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Synthesis of several bis(4-arylthiosemicarbazido- **1**, bis(2-arylamino-1,3,4-thiadiazol-5-yl)- **5** and bis(4-aryl-1,2,4-triazolin-3-thione-5-yl) **2** pentanes is described. Sulphides **3** and sulphones **4** are also prepared from **2** by alkylation and subsequent oxidation with hydrogen peroxide respectively.

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Among azoles, triazoles and thiadiazoles are known to have antifungal [1,3], antibacterial [4,6] and insecticidal [7] properties. The antimicrobial properties of azoles mainly depend upon the nature of the functional group and their point of attachment. Dobosz [8] has recently reported the antitubercular activity of 1,2,4-triazoles in which the 4-position of the triazole ring is substituted either with a thiosemicarbazido function or bridged with a methylene group at the 5-position of another triazole ring.

Recently it has been reported [9] that sulphides and

sulphones derived from 1,2,4-triazoline-5-thione possess remarkable pesticidal activity. Taking these structural features into consideration it was thought worthwhile to synthesize bis(4-aryl-1,2,4-triazolin-3-thione-5-yl)pentane **2**, their sulphides **3** and sulphones **4** as well as bis(2-arylamino-1,3,4-thiadiazol-5-yl)pentanes **5** to study their antimicrobial activities.

Compound **1** obtained [10,11] by the reaction of pivaloyl hydrazide with arylisothiocyanate was cyclized with alkali and orthophosphoric acid to give bis(4-aryl-

Scheme - 1

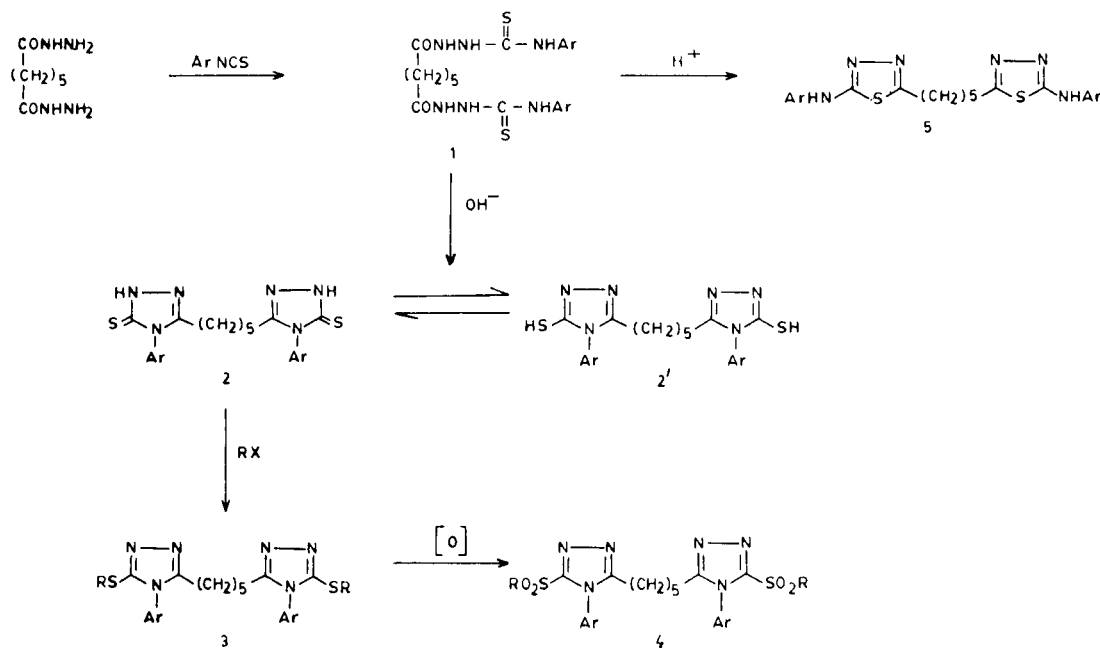


Table I

4, 4'-Diarylpimaloylthiosemicarbazides **1** and Bis(4-aryl-1,2,4-triazoline-3-thione-5-yl)pentane **2**

Compound No.	Ar	MP °C	Molecular Formula	M <sup>+</sup>	Elemental analysis (%)					
					Calcd.			Found		
					C	H	N	C	H	N
<b>1 b</b>	<i>o</i> -Tolyl	135	C <sub>23</sub> H <sub>30</sub> N <sub>6</sub> O <sub>2</sub> S <sub>2</sub>	486	56.17	6.21	17.27	56.33	6.29	17.44
<b>1 c</b>	<i>m</i> -Toyl	174	C <sub>23</sub> H <sub>30</sub> N <sub>6</sub> O <sub>2</sub> S <sub>2</sub>	486	56.17	6.21	17.27	56.42	6.14	17.32
<b>1 d</b>	<i>m</i> -Fluorophenyl	177	C <sub>21</sub> H <sub>24</sub> N <sub>6</sub> F <sub>2</sub> O <sub>2</sub> S <sub>2</sub>	494	51.00	4.89	16.99	51.37	4.77	16.86
<b>2 b</b>	<i>o</i> -Tolyl	307-308	C <sub>23</sub> H <sub>26</sub> N <sub>6</sub> S <sub>2</sub>	450	61.30	5.82	18.65	61.42	5.88	18.87
<b>2 d</b>	<i>m</i> -Fluorophenyl	232	C <sub>21</sub> H <sub>20</sub> N <sub>6</sub> F <sub>2</sub> S <sub>2</sub>	458	55.01	4.40	18.33	55.27	4.66	18.47

Table II

Bis(4-aryl-3-alkylthio-1,2,4-triazol-5-yl)pentane **3** and Bis (4-aryl-3-alkylsulphonyl-1,2,4-triazol-5-yl) pentane **4**

Compound No.	Ar	MP °C	Molecular Formula	M <sup>+</sup>	Elemental analysis (%)					
					Calcd.			Found		
					C	H	N	C	H	N
Ar = Phenyl										
<b>3 d</b>	Allyl	89-100	C <sub>27</sub> H <sub>30</sub> N <sub>6</sub> S <sub>2</sub>	502	64.51	6.02	16.72	64.74	6.11	16.49
<b>3 e</b>	Benzyl	86-87	C <sub>35</sub> H <sub>34</sub> N <sub>6</sub> S <sub>2</sub>	602	69.74	5.69	13.94	69.98	5.71	13.77
<b>3 f</b>	<i>p</i> -Chlorobenzyl	63	C <sub>35</sub> H <sub>32</sub> Cl <sub>2</sub> N <sub>6</sub> S <sub>2</sub>	671	62.58	4.80	12.51	62.64	4.94	12.61
<b>3 g</b>	<i>p</i> -Nitrobenzyl	96	C <sub>35</sub> H <sub>32</sub> N <sub>8</sub> O <sub>4</sub> S <sub>2</sub>	692	60.68	4.66	16.17	60.49	4.39	16.02
<b>3 h</b>	2-Nitro-4-tri-fluoromethylphenyl	164	C <sub>35</sub> H <sub>26</sub> F <sub>6</sub> N <sub>8</sub> S <sub>2</sub> O <sub>4</sub>	800	52.50	3.27	13.99	52.31	3.44	13.79
<b>3 i</b>	2,4-Dinitrophenyl	221	C <sub>33</sub> H <sub>26</sub> N <sub>10</sub> S <sub>2</sub> O <sub>8</sub>	754	52.52	3.47	18.56	52.62	3.49	18.71
Ar = <i>o</i> -Toyl										
<b>3 j</b>	Methyl	58	C <sub>25</sub> H <sub>30</sub> N <sub>6</sub> S <sub>2</sub>	478	62.73	6.32	17.59	62.75	6.34	17.59
Ar = <i>m</i> -Toyl										
<b>3 k</b>	Methyl	119-120	C <sub>25</sub> H <sub>30</sub> N <sub>6</sub> S <sub>2</sub>	478	62.73	6.32	17.56	62.71	6.31	17.59
<b>3 l</b>	Ethyl	98-100	C <sub>27</sub> H <sub>34</sub> N <sub>6</sub> S <sub>2</sub>	506	64.00	6.76	16.58	64.39	6.84	16.44
<b>3 m</b>	<i>n</i> -Butyl	74	C <sub>31</sub> H <sub>42</sub> N <sub>6</sub> S <sub>2</sub>	562	66.15	7.52	14.93	66.33	7.59	14.63
<b>3 n</b>	Benzyl	83-84	C <sub>37</sub> H <sub>38</sub> N <sub>6</sub> S <sub>2</sub>	630	70.44	6.07	13.32	70.22	6.21	13.44
<b>3 o</b>	<i>p</i> -Chlorobenzyl	110-111	C <sub>37</sub> H <sub>36</sub> Cl <sub>2</sub> N <sub>6</sub> S <sub>2</sub>	699	63.51	5.19	12.01	63.71	5.31	12.08

Table II (continued)

Compound No.	Ar	MP °C	Molecular Formula	M <sup>+</sup>	Elemental analysis (%)					
					C	Calcd. H	N	Found C	Found H	N
Ar = Fluoro-phenyl										
3 p	<i>n</i> -Butyl	113-114	C <sub>29</sub> H <sub>36</sub> F <sub>2</sub> N <sub>6</sub> S <sub>2</sub>	570	61.03	6.36	14.72	61.40	6.57	14.55
3 q	Benzyl	126-127	C <sub>35</sub> H <sub>32</sub> F <sub>2</sub> N <sub>6</sub> S <sub>2</sub>	638	65.81	5.05	13.16	65.71	5.14	13.13
3 r	<i>p</i> -Nitrobenzyl	120	C <sub>35</sub> H <sub>30</sub> F <sub>2</sub> N <sub>8</sub> O <sub>4</sub> S <sub>2</sub>	728	57.68	4.15	15.37	57.44	4.22	15.48
3 s	2-Nitro-4-trifluoro-methylphenyl	164	C <sub>35</sub> H <sub>24</sub> F <sub>8</sub> N <sub>8</sub> O <sub>4</sub> S <sub>2</sub>	836	50.25	2.89	13.99	50.35	2.91	13.71
3 t	<i>p</i> -Nitrophenyl	83-84	C <sub>33</sub> H <sub>26</sub> F <sub>2</sub> N <sub>8</sub> O <sub>4</sub> S <sub>2</sub>	700	56.56	3.74	15.99	56.44	3.78	15.74
3 u	2,4-Dinitrophenyl	229	C <sub>33</sub> H <sub>24</sub> F <sub>2</sub> N <sub>10</sub> O <sub>8</sub> S <sub>2</sub>	790	50.13	3.06	17.71	50.39	3.14	17.89
Ar = Phenyl										
4 b	Ethyl	154	C <sub>25</sub> H <sub>30</sub> N <sub>6</sub> O <sub>4</sub> S <sub>2</sub>	542	55.33	5.57	15.49	55.48	5.69	15.86
4 c	<i>n</i> -Butyl	125-126	C <sub>29</sub> H <sub>38</sub> N <sub>6</sub> O <sub>4</sub> S <sub>2</sub>	598	58.17	6.40	14.03	58.43	6.64	14.21
4 d	Benzyl	169-170	C <sub>35</sub> H <sub>34</sub> N <sub>6</sub> O <sub>4</sub> S <sub>2</sub>	666	63.04	5.14	12.60	63.14	5.19	12.64
4 e	<i>p</i> -Nitrobenzyl	75	C <sub>35</sub> H <sub>32</sub> N <sub>8</sub> O <sub>4</sub> S <sub>2</sub>	756	55.55	4.26	14.81	55.77	4.23	14.93
4 f	<i>p</i> -Chlorobenzyl	110	C <sub>35</sub> H <sub>32</sub> Cl <sub>2</sub> N <sub>6</sub> O <sub>4</sub> S <sub>2</sub>	735	57.14	4.38	11.42	57.01	4.39	11.49
4 g	2-Nitro-4-trifluo-romethylphenyl	239	C <sub>35</sub> H <sub>26</sub> F <sub>6</sub> N <sub>8</sub> O <sub>8</sub> S <sub>2</sub>	864	48.61	3.03	12.96	48.44	3.13	12.87
4 h	2,4-Dinitrophenyl	241	C <sub>33</sub> H <sub>26</sub> N <sub>10</sub> O <sub>12</sub> S <sub>2</sub>	818	48.41	3.20	17.11	48.66	3.39	17.44
Ar = Fluoro-phenyl										
4 i	<i>n</i> -Butyl	136	C <sub>29</sub> H <sub>36</sub> F <sub>2</sub> N <sub>6</sub> O <sub>4</sub> S <sub>2</sub>	634	54.87	5.72	13.24	54.49	5.61	13.29
4 j	Benzyl	190	C <sub>35</sub> H <sub>32</sub> F <sub>2</sub> N <sub>6</sub> O <sub>4</sub> S <sub>2</sub>	702	59.82	4.59	11.96	59.67	4.33	11.84
4 k	<i>p</i> -Nitrobenzyl	65	C <sub>35</sub> H <sub>30</sub> F <sub>2</sub> N <sub>8</sub> O <sub>8</sub> S <sub>2</sub>	792	53.03	3.81	14.13	53.14	3.77	14.22
4 l	2-Nitro-4-trifluo-romethylphenyl	85	C <sub>35</sub> H <sub>24</sub> F <sub>8</sub> N <sub>8</sub> O <sub>8</sub> S <sub>2</sub>	900	46.67	2.69	12.44	46.41	2.77	12.39
4 m	2,4-Dinitrophenyl	120	C <sub>33</sub> H <sub>24</sub> F <sub>2</sub> N <sub>10</sub> O <sub>12</sub> S <sub>2</sub>	854	46.37	2.83	16.39	46.71	2.81	16.44
Ar = <i>m</i> -Tolyl										
4 n	Benzyl	121-122	C <sub>37</sub> H <sub>38</sub> N <sub>6</sub> O <sub>4</sub> S <sub>2</sub>	694	63.96	5.51	12.09	63.87	5.90	12.14
4 o	<i>p</i> -Chlorobenzyl	173	C <sub>37</sub> H <sub>36</sub> Cl <sub>2</sub> N <sub>6</sub> O <sub>2</sub> S <sub>2</sub>	763	58.19	4.75	11.00	58.27	4.81	11.24

1,2,4-triazoline-3-thione-5-yl)pentane **2** and bis(2-aryl-amino-1,3,4-thiadiazol-5-yl)pentane **5** respectively. The thiones **2** were transformed into sulphides **3** by reaction with alkyl and aralkyl halides in alcoholic sodium hydroxide or DMF/anhydrous sodium carbonate. The resulting

sulphides **3** were oxidized [11] to sulphones **4** with 3% acidic potassium permanganate or hydrogen peroxide. The latter oxidizing agent was found superior to acidic potassium permanganate in respect to purity and yield of the sulphones.

The ir spectra of bis-thiosemicarbazides **1** showed three characteristic peaks at 3250-3150, 2980-2900 and 1700-1650  $\text{cm}^{-1}$  due to -NH, -OH and  $>\text{C}=\text{O}$  stretching frequency respectively. Complete disappearance of carbonyl frequency was noticed in **2** and **5** after the base and acid cyclization of **1**. Absence of peaks in the region 2600-2550  $\text{cm}^{-1}$  in **2** excluded the possibility of the thiol form **2'** in the solid state.

All the compounds were screened for antifungal activity against *Candida albicans*, *Cryptococcus neoformans*, *Saporotrichum schenckii*, *Trichophyton mentagrophytes* and *Aspergillus fumigatus* at the concentration of 100  $\mu\text{g/ml}$  and antibacterial activity against *Streptococcus faecalis*, *Klebsiella pneumoniae*, *Escherichia coli*, *Pseudomonas aeruginosa* and *Staphylococcus aureus* at 100  $\mu\text{g/ml}$  but none of the compounds exhibited worth mentioning activity.

## EXPERIMENTAL

Melting points were determined in an open capillary on a Thomas Hoover apparatus and are uncorrected. The ir spectra were recorded on a Perkin-Elmer spectrometer 137E. The  $^1\text{H}$ -nmr spectra of the compounds were obtained from a Perkin-Elmer R-32 spectrometer using TMS as internal standard and the  $^{13}\text{C}$  spectra of some compounds were recorded on a Brücker WM-400. Mass spectra were recorded on a Jeol JMSD-300 spectrometer.

### 4,4'-Diphenylpimaloylthiosemicarbazide (**1a**).

A solution of pimaloylhydrazide (1.88 g, 10 mmoles) in 20 ml of ethanol was refluxed with phenylisothiocyanate (2.8 g, 21 mmoles) for 2 hours. During this period, the precipitate was filtered after cooling. The crude product was washed with ethanol and finally crystallized from DMF:water, yield 3.6 g (79%), mp 173°; ms:  $m/z$  458 ( $\text{M}^+$ )  $^{13}\text{C}$  nmr (DMSO- $d_6$ ):  $\delta$  181.15 (s,  $\text{C}=\text{O}$ ), 171.79 (s,  $\text{C}=\text{S}$ ), 127.96 (s,  $\text{C}_3$  and  $\text{C}_5$  aryl), 139.08 (s,  $\text{C}_1$ -aryl), 124.8 (s,  $\text{C}_2$ ,  $\text{C}_4$  and  $\text{C}_6$ -aryl), 33.12 (s,  $\text{C}_1$  and  $\text{C}_5$ -pentyl), 28.24 (s,  $\text{C}_2$  and  $\text{C}_4$ -pentyl), 24.23 (s,  $\text{C}_3$ -pentyl).

Anal. Calcd. for  $\text{C}_{21}\text{H}_{22}\text{N}_6\text{O}_2\text{S}_2$ : C, 55.00; H, 5.71; N, 18.32. Found: C, 55.22; H, 5.81; N, 18.45.

Other compounds prepared in this series are listed in Table 1, **1b-d**, along with their relevant data.

### Bis(4-phenyl-1,2,4-triazoline-3-thione-5-yl)pentane (**2a**).

A solution of 4,4'-diphenylpimaloylthiosemicarbazide (**1a**, 2.0 g) in 5N aqueous sodium hydroxide (25 ml) was refluxed for 3 hours and filtered. The filtrate on neutralization with dilute hydrochloric acid provided a white precipitate which was filtered, washed with water and crystallized from ethanol, yield 75%, mp 225°; ms:  $m/z$  422 ( $\text{M}^+$ ); ir (potassium bromide): 3050 ( $\gamma$  NH), 2900 ( $\gamma$  CH), 1330, 1290 and 1100  $\text{cm}^{-1}$  ( $\text{C}=\text{S}$ );  $^{13}\text{C}$  nmr (DMSO- $d_6$ ):  $\delta$  67.7 (s,  $\text{C}_3$ -triazol ring), 151.89 (s,  $\text{C}_5$ -triazole ring), 133.69 (s,  $\text{C}_1$ -aryl), 129.34 (s,  $\text{C}_3$  and  $\text{C}_5$ -aryl), 128.09 (s,  $\text{C}_2$ ,  $\text{C}_4$  and  $\text{C}_6$ -aryl), 27.21 (s,  $\text{C}_1$  and  $\text{C}_5$ -pentyl), 24.87 (s,  $\text{C}_2$  and  $\text{C}_4$ -pentyl), 24.5 (s,  $\text{C}_3$ -pentyl).

Anal. Calcd. for  $\text{C}_{21}\text{H}_{22}\text{N}_6\text{S}_2$ : C, 59.69; H, 5.25; N, 19.89. Found: C, 59.63; H, 5.19; N, 19.91.

### Bis(4-*m*-tolyl-1,2,4-triazoline-3-thione-5-yl)pentane (**2c**).

This compound was prepared from **1c** (2.0 g) as described in the preceding experiment, yield 70%, mp 278-280°; ms:  $m/z$  450 ( $\text{M}^+$ );  $^1\text{H}$  nmr (DMSO- $d_6$ ):  $\delta$  1.1-1.5 (m, 6H,  $3\text{CH}_2$ ), 2.13-2.43 (m, 10H,  $2\text{CH}_3$  and  $2\text{CH}_2$ ), 6.96-7.4 (m, 8H, ArH).

Anal. Calcd. for  $\text{C}_{23}\text{H}_{26}\text{N}_6\text{S}_2$ : C, 61.33; H, 5.78; N, 18.67. Found: C, 60.92; H, 5.83; N, 18.45.

Two other compounds, **2b,d**, thus prepared are listed in Table 1.

### Bis(2-phenylamino-1,3,4-thiadiazol-5-yl)pentane (**5a**).

A solution of 4,4'-diphenylpimaloylthiosemicarbazide (**1a**, 2.25 g) in orthophosphoric acid (15 ml) was warmed to 120-135° for an hour under stirring. The reaction content after cooling was poured on crushed ice and the white precipitate thus obtained was filtered, washed with water and isolated as phosphoric acid salt, yield 60%, mp 233-235°; ir (potassium bromide): 3250 ( $\gamma$  NH), 2950 ( $\gamma$  CH), 1610, 1560 ( $\gamma$   $\text{C}=\text{C}$ ,  $\delta$  NH), 2950, 1060 ( $\gamma$   $\text{P}=\text{O}$ ).

Anal. Calcd. for  $\text{C}_{21}\text{H}_{22}\text{N}_6\text{S}_2 \cdot 2\text{H}_3\text{PO}_4 \cdot \text{H}_2\text{O}$  (636): C, 39.62; H, 4.72; N, 13.21. Found: C, 39.95; H, 4.54; N, 13.63.

### Bis(2-*o*-tolylamino-1,3,4-thiadiazol-5-yl)pentane (**5b**).

The title compound was obtained in 50% yield from the acid cyclization of 4,4'-di(*o*)tolylpimaloylthiosemicarbazide (**1b**) following the preceding procedure, mp 256°; ir (potassium bromide): 3250 ( $\gamma$  NH), 2950 ( $\gamma$  CH), 1610, 1560 ( $\gamma$   $\text{C}=\text{C}$ ,  $\delta$  NH), 1040  $\text{cm}^{-1}$  ( $\gamma$   $\text{P}=\text{O}$ ).

Anal. Calcd. for  $\text{C}_{23}\text{H}_{26}\text{N}_6\text{S}_2 \cdot 2\text{H}_3\text{PO}_4 \cdot \text{H}_2\text{O}$  (664): C, 41.56; H, 5.12; N, 12.65. Found: C, 41.56; H, 5.23; N, 12.33.

### Bis(2-*m*-tolylamino-1,3,4-thiadiazol-5-yl)pentane (**5c**).

The compound was obtained in 55% yield from the cyclization of **1c** in orthophosphoric acid as described above, mp 208-210°; ir (potassium bromide): 3450 ( $\gamma$  NH), 2970 ( $\gamma$  CH), 1610, 1560 ( $\gamma$   $\text{C}=\text{C}$ ,  $\delta$  NH), 1060  $\text{cm}^{-1}$  ( $\gamma$   $\text{P}=\text{O}$ ).

Anal. Calcd. for  $\text{C}_{23}\text{H}_{26}\text{N}_6\text{S}_2 \cdot 2\text{H}_3\text{PO}_4 \cdot 2.5\text{H}_2\text{O}$  (691): C, 39.94; H, 5.36; N, 12.16. Found: C, 39.70; H, 5.04; N, 11.82.

### Bis(4-phenyl-3-methylthio-1,2,4-triazol-5-yl)pentane (**3a**).

A solution of **2a** (1.0 g) in alcoholic sodium hydroxide (10 ml, 8%) was refluxed with methyl iodide (0.8 g) for 3 hours, cooled, poured into ice cold water and left overnight. The precipitate thus obtained was filtered, washed with water and finally crystallized from ethanol-water, yield 60%, mp 165°; ms:  $m/z$  450 ( $\text{M}^+$ );  $^{13}\text{C}$  nmr (DMSO- $d_6$ ):  $\delta$  155.22 (s,  $\text{C}_3$ -triazole), 151.89 (s,  $\text{C}_5$ -triazole), 133.69 (s,  $\text{C}_1$ -aryl), 129.34 (s,  $\text{C}_3$  and  $\text{C}_5$ -aryl), 128.09 (s,  $\text{C}_2$ ,  $\text{C}_4$  and  $\text{C}_6$ -aryl), 27.21 (s,  $\text{C}_1$  and  $\text{C}_5$ -pentyl), 24.87 (s,  $\text{C}_2$  and  $\text{C}_4$ -pentyl), 24.5 (s,  $\text{C}_3$ -pentyl), 14.1 (s, 2,  $\text{SCH}_3$ ).

Anal. Calcd. for  $\text{C}_{23}\text{H}_{26}\text{N}_6\text{S}_2$ : C, 61.30; H, 5.82; N, 18.65. Found: C, 61.57; H, 5.77; N, 18.73.

### Bis(4-phenyl-3-ethylthio-1,2,4-triazol-5-yl)pentane (**3b**).

The title compound was prepared from the reaction of **2a** (1.0 g) and ethyl iodide as described in the preceding experiment. It was isolated as usual and crystallized from water-ethanol, yield 50%, mp 142-143°; ms:  $m/z$  478 ( $\text{M}^+$ );  $^1\text{H}$  nmr (deuteriochloroform):  $\delta$  1.1-1.4 (m, 12H,  $2\text{CH}_3$  +  $3\text{CH}_2$ ), 2.63 (t, 4H,  $2\text{CH}_2$ ), 3.1 (q, 4H,  $\text{S-CH}_2$ ), 7.0-7.6 (m, 10H, ArH).

Anal. Calcd. for  $\text{C}_{25}\text{H}_{30}\text{N}_6\text{S}_2$ : C, 62.76; H, 6.28; N, 17.57. Found: C, 63.00; H, 6.52; N, 17.73.

### Bis(4-phenyl-3-*n*-butylthio-1,2,4-triazol-5-yl)pentane (**3c**).

A solution of **2a** (2.11 g) and anhydrous sodium carbonate (1.06 g) in DMF (15 ml) was treated with *n*-butyl bromide (1.37 g) and stirred at room temperature for 4 hours. The reaction content was poured into ice cold water and left overnight in a refrigerator. The oily product separated was triturated with water and finally crystallized from DMF-water, yield 75%, mp 110°; ms: *m/z* 534 (*M*<sup>+</sup>); <sup>1</sup>H nmr (DMSO-*d*<sub>6</sub>): δ 0.83 (t, 6H, 2CH<sub>3</sub>), 1.16-1.60 (m, 14H, 7CH<sub>2</sub>), 2.43 (t, 4H, 2CH<sub>2</sub>), 2.9 (t, 4H, SCH<sub>2</sub>), 7.2-7.6 (m, 10H, ArH).

*Anal.* Calcd. for C<sub>29</sub>H<sub>38</sub>N<sub>6</sub>S<sub>2</sub>: C, 65.17; H, 7.12; N, 15.73. Found: C, 65.52; H, 7.11; N, 15.41.

Other compounds, **3d-u**, were prepared similarly and are listed in Table 2 along with their relevant data.

#### Bis(4-phenyl-3-methylsulphonyl-1,2,4-triazol-5-yl)pentane (**4a**).

A solution of **3a** (1.15 g) in a minimum quantity of glacial acetic acid (8 ml) was oxidized by dropwise addition of aqueous potassium permanganate solution (3%) until the violet colour persisted. The mixture was filtered, washed several times with water and finally crystallized with dilute acetic acid, yield 56%, mp 110-112°; ms: *m/z* 514 (*M*<sup>+</sup>); ir (potassium bromide): 2930, 2860 (γ CH), 1600, 1500 (γ C=C, δ CH), 1160 cm<sup>-1</sup> (γ S=O).

*Anal.* Calcd. for C<sub>23</sub>H<sub>26</sub>N<sub>6</sub>S<sub>2</sub>O<sub>4</sub>: C, 53.69; H, 5.05; N, 16.34. Found: C, 54.04; H, 5.30; N, 16.51.

Other compounds, **4b-o**, thus prepared are listed in Table 2 along with their relevant data.

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