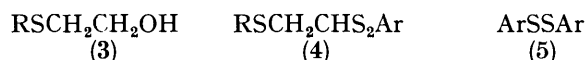
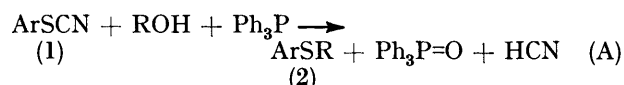


## Interaction of Aryl Thiocyanates and Ethylene Glycol under the Influence of Triphenylphosphine: Formation of 1,2-Bis(arylthio)ethanes and of Aryl 2-Aryloxyethyl Sulphides

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The interaction of aryl thiocyanates and 2-(arylthio)ethanols (3: R = Ar) in the presence of triphenylphosphine appears to be a general route to 1,2-bis(arylthio)ethanes (4: R = Ar) in which the two aryl groups are not necessarily the same: likewise 2-(aryloxy)ethanols (6: R = Ar) afford a range of novel aryl 2-aryloxyethyl sulphides (7: R = Ar). Under conditions where phenyl thiocyanate with ethylene glycol and triphenylphosphine provides 2-(phenylthio)ethanol (3a). *p*-nitrophenyl thiocyanate gives 1,2-bis(*p*-nitrophenylthio)ethane (4a) directly without the intermediacy of the 2-(arylthio)ethanol (3b): *p*-methoxyphenyl thiocyanate with ethylene glycol gives none of the desired products. These reactions are rationalised in terms of the different modes of decomposition of the intermediate thiophosphorane ether (8).

We have recently shown that, in the presence of triphenylphosphine, aryl thiocyanates (1) react with



primary alcohols to give high yields of alkyl aryl sulphides (2) according to equation (A); in addition we reported that phenyl thiocyanate gives, with a large

Since the glycol reaction offers a potentially useful and perhaps improved route to 2-(arylthio)ethanols (3; R = Ar) and to 1,2-bis(arylthio)ethanes (4; R = Ar), we have now investigated its application to other aryl thiocyanates. It was soon found that whereas the nature of any substituent on the aryl group has little effect on the reaction of aryl thiocyanates with primary monohydric alcohols,<sup>1</sup> there are significant differences in the reactions with ethylene glycol, and we therefore report first on the second stage of the glycol reaction, *i.e.* (3)→(4).†

TABLE 1

Reactions between RSCH<sub>2</sub>CH<sub>2</sub>OH (3) and ArSCN (1) to give RSCH<sub>2</sub>CH<sub>2</sub>SAr (4): <sup>a</sup> the percentage yield <sup>a</sup> of ArSSAr (5) is given in parentheses

	PhSCN (1a)	<i>p</i> -NO <sub>2</sub> C <sub>6</sub> H <sub>4</sub> SCN (1b)	<i>o</i> -NO <sub>2</sub> C <sub>6</sub> H <sub>4</sub> SCN (1c)	<i>p</i> -MeOC <sub>6</sub> H <sub>4</sub> SCN (1d)
PhSCH <sub>2</sub> CH <sub>2</sub> OH (3a)	(4a) 78 (10)	(4b) 65 (30)		
<i>p</i> -NO <sub>2</sub> C <sub>6</sub> H <sub>4</sub> SCH <sub>2</sub> CH <sub>2</sub> OH (3b)	(4b) 73 (15)	(4c) 66 (29)		(4d) 35 (30)
<i>o</i> -NO <sub>2</sub> C <sub>6</sub> H <sub>4</sub> SCH <sub>2</sub> CH <sub>2</sub> OH (3c)		(4e) 78 (15)	(4f) 72 (10)	
<i>p</i> -MeOC <sub>6</sub> H <sub>4</sub> SCH <sub>2</sub> CH <sub>2</sub> OH (3d)		0 (38) <sup>b</sup>		0 (65) <sup>c</sup>
MeSCH <sub>2</sub> CH <sub>2</sub> OH (3e)		(4g) 86 (10)		0 (10) <sup>d</sup>

<sup>a</sup> Based on initial ArSCN (1). <sup>b</sup> 58% recovery of ArSCN (1). <sup>c</sup> 10% recovery of ArSCN (1). <sup>d</sup> 70% yield of ArSR.

TABLE 2

Reactions between ROCH<sub>2</sub>CH<sub>2</sub>OH (6) and ArSCN (1) to give ROCH<sub>2</sub>CH<sub>2</sub>SAr (7).<sup>\*</sup> The percentage yield of (5) is given in parentheses \*

	<i>p</i> -NO <sub>2</sub> C <sub>6</sub> H <sub>4</sub> SCN (1b)	<i>o</i> -NO <sub>2</sub> C <sub>6</sub> H <sub>4</sub> SCN (1c)	<i>p</i> -MeOC <sub>6</sub> H <sub>4</sub> SCN (1d)	2,4,6-Me <sub>3</sub> C <sub>6</sub> H <sub>2</sub> SCN (1e)
MeOCH <sub>2</sub> CH <sub>2</sub> OH (6a)	(7d) 81 (0)	(7c) 85 (0)	(7a) 78 (0)	(7b) 63 (2)
PhOCH <sub>2</sub> CH <sub>2</sub> OH (6b)	(7f) 75 (15)		(7e) 81 (0)	
<i>p</i> -NO <sub>2</sub> C <sub>6</sub> H <sub>4</sub> OCH <sub>2</sub> CH <sub>2</sub> OH (6c)	(7g) 83 (5)			

\* Based on ArSCN (1).

molar excess of ethylene glycol, 2-(phenylthio)ethanol (3a; R = Ph) which may, in a separate reaction, be converted by the same process into 1,2-bis(phenylthio)ethane (4a; R = Ph).<sup>1</sup>

† Following submission of this paper, but before it had appeared, K. A. M. Walker (*Tetrahedron Letters*, 1977, 4475) reported that a number of *N*-arylthiosuccinimides react with primary and secondary alcohols in the presence of tri-*n*-butylphosphine to give high yields of alkyl aryl sulphides. Since the phosphine oxide and succinimide are both soluble in water, isolation of the sulphide is readily achieved. Preliminary experiments carried out in these laboratories indicate that similar advantages attend the replacement of triphenylphosphine by tri-*n*-butylphosphine in the preparation of alkyl aryl sulphides from aryl thiocyanates and alcohols.

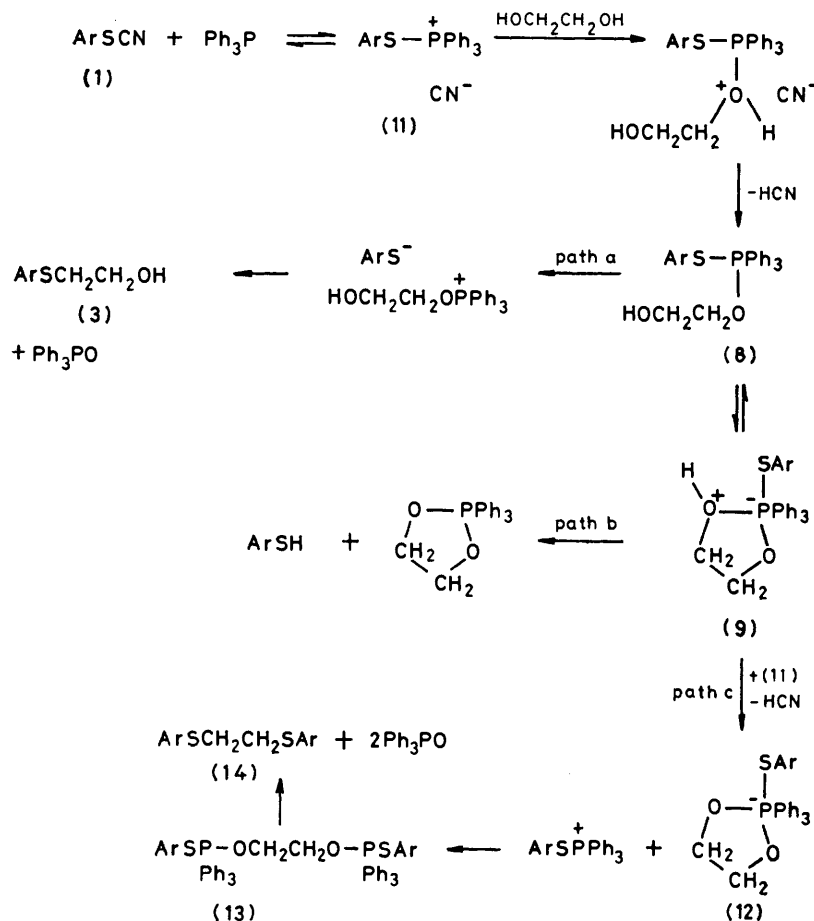
## RESULTS

With the exception of 2-(*p*-methoxyphenylthio)ethanol (3d), which was reduced to *p*-methoxythiophenol, all the 2-(arylthio)ethanols (3a—c) investigated behaved like simple primary alcohols to give acceptable yields of the bis-sulphides (4a—f); additionally although 2-(methylthio)ethanol (3e) reacted with *p*-nitrophenyl thiocyanate (1b) to give an 86% yield of 1-(*p*-nitrophenylthio)-2-(methylthio)ethane (4g), with *p*-methoxyphenyl thiocyanate (1d) there resulted methyl *p*-methoxyphenyl sulphide (Table 1). In all reactions there was some (10—30%) disulphide (5) formation.

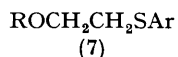
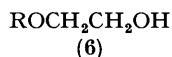
Likewise, 2-methoxy-, 2-phenoxy-, and 2-(*p*-nitro-

phenoxy)-ethanols (6a—c, respectively) reacted with a range of aryl thiocyanates (1b—e) to give the appropriate novel 2-methoxy- or 2-aryloxy-ethyl aryl sulphides (7a—g) in high yield, in this case with less (0—26%) disulphide (5) formation (Table 2). These procedures for the preparation

and the 2-(aryltio)ethanol; furthermore it was shown that the former reaction was not retarded nor was the yield of the bis-sulphide (4c) reduced by the addition to the reaction mixture of (3b) which at the end was recovered. Thus, in the case of the reaction of ethylene glycol and *p*-nitrophenyl



of compounds (4) and (7) are more convenient than most others available.



The results of greater mechanistic significance were obtained in the first stage of the glycol reaction, *i.e.* (1)→(3; R = Ar). *o*-Nitrophenyl thiocyanate (1c) behaved analogously to phenyl thiocyanate (1a) to give the 2-(aryltio)ethanol (3c); *p*-methoxyphenyl thiocyanate (1d) gave none of the expected (3d), but was largely converted to the thiol and thence to the disulphide (5; Ar = *p*-MeOC<sub>6</sub>H<sub>4</sub>). However, when equimolar quantities of *p*-nitrophenyl thiocyanate (1b) and triphenylphosphine were heated at 100 °C in a large molar excess of ethylene glycol, the bis-sulphide (4c) and triphenylphosphine oxide were obtained in high yield; none of the expected 2-(aryltio)ethanol (3b) could be detected. It was noted that the formation of the bis-sulphide (4c) directly from the thiocyanate (1b) and the glycol was quicker, under similar reaction conditions, than the formation of (4c) from (1b)

thiocyanate (1b) it would appear that the bis-sulphide (4c) is obtained without the intermediacy of the 2-(aryltio)ethanol (3b).

It was then found that other divalent sulphur species, ArSY, participated in phosphine-alcohol reactions to give alkyl aryl sulphides; both *N*-(phenylthio)phthalimide and diphenyl disulphide (and their *p*-nitro-derivatives) react

TABLE 3

Reaction of ArSY with alcohols, ROH, to give ArSR [yield (%) ArSR<sup>a</sup>/yield (%) ArSSAr (5)<sup>a</sup>/yield (%) ArSH<sup>b</sup>]

Ar	Y	R = Me	Pr <sup>n</sup>
Ph	Phthalimido	86/0/0	90/0/0
<i>p</i> -NO <sub>2</sub> C <sub>6</sub> H <sub>4</sub>	Phthalimido	92/0/0	85/0/0
Ph	SPh	91/0/90	92/0/90
<i>p</i> -NO <sub>2</sub> C <sub>6</sub> H <sub>4</sub>	SC <sub>6</sub> H <sub>4</sub> NO <sub>2</sub> - <i>p</i>	85/0/90	96/0/95

<sup>a</sup> Based on ArSY (1). <sup>b</sup> Based on ArSSAr.

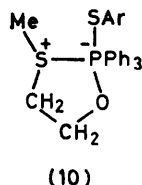
both with methanol and with *n*-propanol to give the desired sulphides in good yield (Table 3). When the above

*p*-nitro-derivatives were separately reacted with ethylene glycol in the presence of triphenylphosphine, 2-(*p*-nitrophenylthio)ethanol (3b) was obtained in 68 and 78% yields respectively with none of the bis-sulphide (4c) being detected; these reagents therefore behave analogously to phenyl thiocyanate (1a) and not to *p*-nitrophenyl thiocyanate (1b). *p*-Nitrophenyl thiocyanate reacted with propane-1,3- and butane-1,4-diols to give the corresponding  $\omega$ -(*p*-nitrophenylthio)alkanols in 75 and 50% yields, respectively; no bis-sulphides were detected.

#### DISCUSSION

The mechanism suggested for the formation of alkyl aryl sulphides from aryl thiocyanates, triphenylphosphine, and monohydric alcohols<sup>1</sup> may be applied to the conversion of phenyl thiocyanate and ethylene glycol into 2-(phenylthio)ethanol (3a) (Scheme, path a).

That *p*-methoxyphenyl thiocyanate does not follow the same course may be associated with the fact that *p*-MeOC<sub>6</sub>H<sub>4</sub>S<sup>-</sup> is a much poorer leaving group than is PhS<sup>-</sup>; if it had the opportunity to leave the thiophosphorane ether (8; Ar = *p*-MeOC<sub>6</sub>H<sub>4</sub>), then doubtless its high nucleophilicity would lead to rapid formation of the 2-(aryltio)ethanol (3; Ar = *p*-MeOC<sub>6</sub>H<sub>4</sub>). However, association of the lone pair of electrons of the hydroxy group in (8; Ar = *p*-MeOC<sub>6</sub>H<sub>4</sub>) with the phosphorus atom would lead, in the extreme, to the dipolar structure (9; Ar = *p*-MeOC<sub>6</sub>H<sub>4</sub>), from which the observed thiol could be eliminated much more easily than could its anion from (8; Ar = *p*-MeOC<sub>6</sub>H<sub>4</sub>) (Scheme, path b). Support for the intermediacy of (9; Ar = *p*-MeOC<sub>6</sub>H<sub>4</sub>) in the above reaction comes both from the isolation of related hexaco-ordinate spirophosphate(-1) ions,<sup>2,3</sup> and from the formation of alkyl aryl sulphide in the reaction of *p*-methoxyphenyl thiocyanate (1d) and 2-(methylthio)ethanol (3e), which could involve the analogous formation of the sulphonium intermediate (10; Ar = *p*-MeOC<sub>6</sub>H<sub>4</sub>) and its subsequent demethylation; when *p*-nitrophenyl thiocyanate (1b) is used in the reaction with (3e), the aryl group is apparently not sufficiently nucleophilic to effect demethylation of (10; Ar = *p*-NO<sub>2</sub>C<sub>6</sub>H<sub>4</sub>) and the normal course of reaction (Scheme, path a) is followed to afford the bis-sulphide (4g).



Certainly the free hydroxy group in (8) has some bearing on the reaction, since the glycol monomethyl and monophenyl ethers (6a and b, respectively) react with *p*-methoxyphenyl thiocyanate in the same manner as do primary monohydric alcohols (Scheme, path a).

The nitro-substituent in the intermediate (10; Ar = *p*-NO<sub>2</sub>C<sub>6</sub>H<sub>4</sub>) formed in the reaction of *p*-nitrophenyl thiocyanate and ethylene glycol serves to stabilise the

negative charge on the phosphorus atom and thus provide the opportunity for deprotonation of the oxonium centre by the cyanide ion of the phosphonium cyanide (11; Ar = *p*-NO<sub>2</sub>C<sub>6</sub>H<sub>4</sub>) (Scheme, path c); the phosphonium ion then reacts with the negatively-charged ion (12; Ar = *p*-NO<sub>2</sub>C<sub>6</sub>H<sub>4</sub>) to give the bis(thiophosphorane ether) (13; Ar = *p*-NO<sub>2</sub>C<sub>6</sub>H<sub>4</sub>) and hence the bis-sulphide (14; Ar = *p*-NO<sub>2</sub>C<sub>6</sub>H<sub>4</sub>) and two molecules of triphenylphosphine oxide.

When the nitro-substituent is in the *ortho*-position, formation of the dipolar intermediate (9; Ar = *o*-NO<sub>2</sub>C<sub>6</sub>H<sub>4</sub>) is impeded by competition from the oxygen of the nitro-group, and hence the normal course (Scheme, path a) of the reaction is followed. Similarly, with propane-1,3-diol and butane-1,4-diol, cyclisation of the thiophosphorane ether is much less likely on account of the ring size involved and again, only one of the hydroxy groups is replaced by an arylthio-substituent.

The formation of bis-sulphide (14) directly from ethylene glycol (Scheme, path c) requires two deprotonations. The failure of the nitrophenylphthalimide and of the nitrophenyl disulphide to follow this same course may be attributed to the fact that not only are the phthalimide and the thiolate ions much less basic than the cyanide ion, but also that their respective conjugate acids are involatile, hence making the deprotonations much less likely and permitting path a (Scheme) to be followed.

#### EXPERIMENTAL

I.r. spectra were recorded on a Perkin-Elmer 257 instrument, <sup>1</sup>H n.m.r. spectra on a Perkin-Elmer R20A or Varian HA100 spectrometer, and mass spectra on an A.E.I. MS 902 spectrometer. All the compounds gave satisfactory analytical and spectroscopic data.

*Glycols, Glycol Monoethers, and Monothioethers.*—Ethylene glycol and its monomethyl ether, propane-1,3-diol, and butane-1,4-diol were AnalaR or good reagent grade, and were carefully dried \* by conventional procedures prior to fractional distillation. 2-(Phenoxy)ethanol and its *p*-nitro-derivative were obtained by condensation of the corresponding phenols and ethylene carbonate in the presence of lithium hydride.<sup>4</sup> All the  $\omega$ -alkylthio- and  $\omega$ -aryltio-alkanols were prepared from  $\omega$ -chloroalkanols and the appropriate sodium thiolate following the general procedure of Bennett and Berry.<sup>5</sup>

*Interaction of Aryl Thiocyanates and Glycols, and their Monoethers and Monothioethers, in the Presence of Triphenylphosphine.*—In a nitrogen atmosphere, the aryl thiocyanate (25 mmol), triphenylphosphine (25 mmol), and the hydroxy compound (25 mmol) were heated under reflux in dry dioxan (100 ml); the progress of the reaction was monitored as previously reported.

In some reactions, dioxan was replaced as solvent by the glycol or ethylene glycol monomethyl ether being used as reactants; no significant variation in yields was observed. At the end of the reaction, the solvent was removed *in vacuo* and the products separated by column chromatography on silica gel (80–200 mesh; 80 × 3 cm). Elution was

\* Reaction yields were significantly lowered by trace amounts of water.

TABLE 4  
 Physical data for 1,2-bis(arylthio)ethanes, RSCH<sub>2</sub>CH<sub>2</sub>SAr (4)

	R	Ar	M.p. (°C)	<sup>1</sup> H n.m.r. (τ)		M <sup>+</sup> (%) <sup>a</sup>	Formula	Analysis, Found (required) (%)			
				1-CH <sub>2</sub>	2-CH <sub>2</sub>			C	H	N	S
(4a)	Ph	Ph	66—68 <sup>b</sup>	7.0	7.0	246	C <sub>14</sub> H <sub>14</sub> S <sub>2</sub>				
(4b)	Ph	<i>p</i> -NO <sub>2</sub> C <sub>6</sub> H <sub>4</sub>	126—128 <sup>c</sup>	6.8	6.8	291 (29)	C <sub>14</sub> H <sub>13</sub> NO <sub>2</sub> S <sub>2</sub>	57.6 (57.7)	4.6 (4.5)	4.5 (4.8)	21.9 (22.0)
(4c)	<i>p</i> -NO <sub>2</sub> C <sub>6</sub> H <sub>4</sub>	<i>p</i> -NO <sub>2</sub> C <sub>6</sub> H <sub>4</sub>	136 <sup>d</sup>	6.65	6.65	336 (26)	C <sub>14</sub> H <sub>12</sub> N <sub>2</sub> O <sub>4</sub> S <sub>2</sub>				
(4d)	<i>p</i> -NO <sub>2</sub> C <sub>6</sub> H <sub>4</sub>	<i>p</i> -MeOC <sub>6</sub> H <sub>4</sub>	68—70	6.8	7.0	321 (31)	C <sub>15</sub> H <sub>15</sub> NO <sub>3</sub> S <sub>2</sub>	56.0 (56.1)	4.7 (4.7)	4.0 (4.4)	19.7 (19.9)
(4e)	<i>o</i> -NO <sub>2</sub> C <sub>6</sub> H <sub>4</sub>	<i>p</i> -NO <sub>2</sub> C <sub>6</sub> H <sub>4</sub>	127—128	6.55	6.55	336 (26)	C <sub>14</sub> H <sub>12</sub> N <sub>2</sub> O <sub>4</sub> S <sub>2</sub>	50.3 (50.0)	3.7 (3.6)	8.2 (8.3)	18.8 (19.0)
(4f)	<i>o</i> -NO <sub>2</sub> C <sub>6</sub> H <sub>4</sub>	<i>o</i> -NO <sub>2</sub> C <sub>6</sub> H <sub>4</sub>	208 <sup>e</sup>	6.7	6.7	336 (16)	C <sub>14</sub> H <sub>12</sub> N <sub>2</sub> O <sub>4</sub> S <sub>2</sub>				
(4g) <sup>f</sup>	Me	<i>p</i> -NO <sub>2</sub> C <sub>6</sub> H <sub>4</sub>	96—98	7.2	7.2	229 (61)	C <sub>9</sub> H <sub>11</sub> NO <sub>2</sub> S <sub>2</sub>	47.1 (47.2)	4.7 (4.8)	6.3 (6.1)	27.5 (27.9)

<sup>a</sup> By mass spectrometry. <sup>b</sup> Lit.,<sup>6</sup> m.p. 69 °C. <sup>c</sup> Lit.,<sup>7</sup> m.p. 136 °C. <sup>d</sup> Lit.,<sup>8</sup> m.p. 136 °C. <sup>e</sup> Lit.,<sup>9</sup> m.p. 205 °C. <sup>f</sup> Compound (IVg) is a 1-(alkylthio)-2-(arylthio)ethane.

 TABLE 5  
 Physical data for 1-(aryl- or alkyl-oxy)-2-(arylthio) ethanes, ROCH<sub>2</sub>CH<sub>2</sub>SAr (7)

	R	Ar	M.p. (B.p./mm) °C	<sup>1</sup> H n.m.r. (τ)		M <sup>+</sup> (%) <sup>*</sup>	Formula	Analysis, Found (required) (%)			
				1-CH <sub>2</sub>	2-CH <sub>2</sub>			C	H	N	S
(7a)	Me	<i>p</i> -MeOC <sub>6</sub> H <sub>4</sub>	115—117(0.5)	6.65	7.35	198 (100)	C <sub>10</sub> H <sub>14</sub> O <sub>2</sub> S				
(7b)	Me	2,4,6-Me <sub>3</sub> C <sub>6</sub> H <sub>2</sub>	(120—122/0.1)	6.65	7.3	210	C <sub>12</sub> H <sub>16</sub> OS	68.8 (68.6)	8.8 (8.6)		15.0 (15.2)
(7c)	Me	<i>o</i> -NO <sub>2</sub> C <sub>6</sub> H <sub>4</sub>	55—56	6.35	6.85	213 (3)	C <sub>9</sub> H <sub>11</sub> NO <sub>3</sub> S				
(7d)	Me	<i>p</i> -NO <sub>2</sub> C <sub>6</sub> H <sub>4</sub>	41—42	6.4	6.9	213 (27)	C <sub>9</sub> H <sub>11</sub> NO <sub>3</sub> S	50.7 (50.7)	5.0 (5.2)	6.7 (6.6)	15.2 (15.0)
(7e)	Ph	<i>p</i> -MeOC <sub>6</sub> H <sub>4</sub>	50—52	5.8	6.75	260 (40)	C <sub>15</sub> H <sub>16</sub> O <sub>2</sub> S	69.4 (69.2)	6.1 (6.2)		12.2 (12.3)
(7f)	Ph	<i>p</i> -NO <sub>2</sub> C <sub>6</sub> H <sub>4</sub>	96—98	5.7	6.5	275 (4)	C <sub>14</sub> H <sub>13</sub> NO <sub>3</sub> S	61.1 (61.0)	5.0 (4.7)	4.9 (5.1)	11.3 (11.6)
(7g)	<i>p</i> -NO <sub>2</sub> C <sub>6</sub> H <sub>4</sub>	<i>p</i> -NO <sub>2</sub> C <sub>6</sub> H <sub>4</sub>	124—125	5.7	6.55	320	C <sub>14</sub> H <sub>12</sub> N <sub>2</sub> O <sub>6</sub> S	52.8 (52.5)	3.8 (3.8)	8.5 (8.8)	10.1 (10.0)

\* Mass spectrometry.

started with light petroleum (b.p. 60—80 °C) and expedited by the gradual addition of toluene until a 1 : 1 mixture of the two solvents was reached. Elution occurred in the order: thiophenols, 1,2-bis(arylthio)ethanes, diaryl disulphides, and 2-(arylthio)ethanols. Finally, triphenylphosphine oxide was eluted with ethanol.

*Interaction of N-(Arylthio)phthalimides and Diaryl Disulphides with Alcohols and Ethylene Glycol in the Presence of Triphenylphosphine.*—The reactions with methanol and with n-propanol were carried out and the products isolated as previously reported for the corresponding reactions of the alcohols and arylthiocyanates. Methyl and n-propyl phenyl sulphides were separated by distillation *in vacuo*. The reaction with ethylene glycol was carried out in dioxan as solvent as described above; the bulk of the resultant phthalimide separated from the cooled (0 °C) reaction mixture and was removed by filtration prior to chromatography of the other products.

Physical, analytical, and spectroscopic data for the 1,2-bis(arylthio)ethanes and the 2-(aryloxy)ethanols obtained are shown in Tables 4 and 5, respectively; further

spectroscopic data are available in Supplementary Publication No. SUP 22543 (3 pp.).\*

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\* For details see Notice to Authors No. 7, *J.C.S. Perkin I*, Index issue, 1978.

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