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Nucleophilic reactions of 5-(aryl)thianthrenium bromides with sodium aryl oxides

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epoc ABSTRACT: Reactions between 5-(aryl)thianthrenium bromides (**1a–e**) with aryl (Ar) groups phenyl (**a**), *p*-tolyl (**b**), *p*-anisyl (**c**), *p*-chlorophenyl (**d**) and *p*-bromophenyl (**e**) and sodium aryl oxides (**7a–c**, Ar′ONa, Ar′ = phenyl, *p*-tolyl and *p*-chlorophenyl) were carried out in acetonitrile at 80 °C. Results are compared with those of earlier reactions of **1a–e** with corresponding sodium aryl thiolates (Ar′SNa). In contrast with the thiolate reactions, those with **7a–c** were too slow at room temperature to be useful. Reactions of **1a–c** at 80 °C gave small amounts of benzene and toluene from **1a** and **b**, small amounts of diaryl ethers ArOAr′ (**9**) and thianthrene (Th) and large amounts (90–97%) of 2-(ArS)-2′-(Ar′O)diphenyl sulfide (**10**). Compared with reactions of **1a–c**, those of **1d** and **e** gave larger amounts (10–17%) of **9**, lesser amounts (25–30%) of **10** and substantial amounts (42–55%) of 2-[4-(Ar′O)-ArS]-2′-(Ar′O)diphenyl sulfide (**12**). Small amounts of 1,4-di(Ar′O)benzene (**11**) were also obtained from reactions of **1d** and **e**. Formation of **11** and **12** is preceded by replacement of halogen by Ar′O$^-$. Structures of products **10** and **12** were deduced by comparison with earlier products from thiolate reactions, elemental analyses, mass spectrometry molecular masses and, in the case of **10**, the crystal structure of **10d**. It is proposed that products **9–12** are formed by nucleophilic reactions of Ar′O$^-$ at *ipso* positions in **1**. Copyright © 2003 John Wiley & Sons, Ltd.
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KEYWORDS: 5-(aryl)thianthrenium; nucleophilic reactions; sodium aryl oxides

INTRODUCTION

Reactions of 5-(aryl)thianthrenium bromides (**1a–e**) with aryl thiolates (Ar′S$^-$) were reported recently from these laboratories.[1] Diaryl sulfides (**2**), thianthrene (Th) and tetraaryltrisulfides (**3**), Scheme 1, were the main products from reactions of **1a–c**. From **1a** and **b** small amounts of benzene and toluene were also formed. Reactions of **1d** and **e** gave smaller amounts of **2** and **3**, but considerable amounts of 1,4-(diarylthio)benzenes (**4**) and pentaaryltetrasulfides (**5**). It was deduced that the products **2–5** and Th were formed by ligand coupling routes within sulfurane intermediates (**6**). We have now studied for comparison the reactions of **1a–e** with analogous sodium aryl oxides, NaOAr′ (**7a–c**), Ar′ = phenyl (**a**), *p*-tolyl (**b**) and *p*-chlorophenyl (**c**).

RESULTS AND DISCUSSION

The structures of reactants **7** and of products **8–12** are given in Scheme 2. Structures of **10** and **12** were deduced from analogous reactions with aryl thiolates,[1] analytical data, satisfactory ^{1}H NMR spectroscopy and, in the case of **10**, the crystal structure of **10d**. In contrast with reactions of **1a–e** with aryl thiolates, which occurred readily at room temperature, reactions with the aryl oxides at room temperature were too slow to follow to completion. These reactions, therefore, were carried out at 80 °C. As in the earlier work, the tabulated results are divided into two groups, those of **1a–c** reactions in Table 1 and those of **1d** and **e** reactions in Table 2. The main product (91–97%) from reactions of **1a–c** was a 2-(arylthio)-2′-(aryloxy)diphenyl sulfide (**10**). Small amounts of diaryl ether (**9**) were formed, the yields of those from **1a** being notably larger than from **1b** and **c**. Small amounts of benzene (**8a**) and toluene (**8b**) were obtained from **1a** and **b**. In the reactions of **1d** and **e** substantial amounts (10–17%) of diaryl ether (**9**) were formed, a contrast, particularly, with the amounts of **9** (~1%) that were obtained from **1b** and **c**. Opening of the thianthrenium ring of **1d** and **e**, giving products **10** and **12**, was again the dominant

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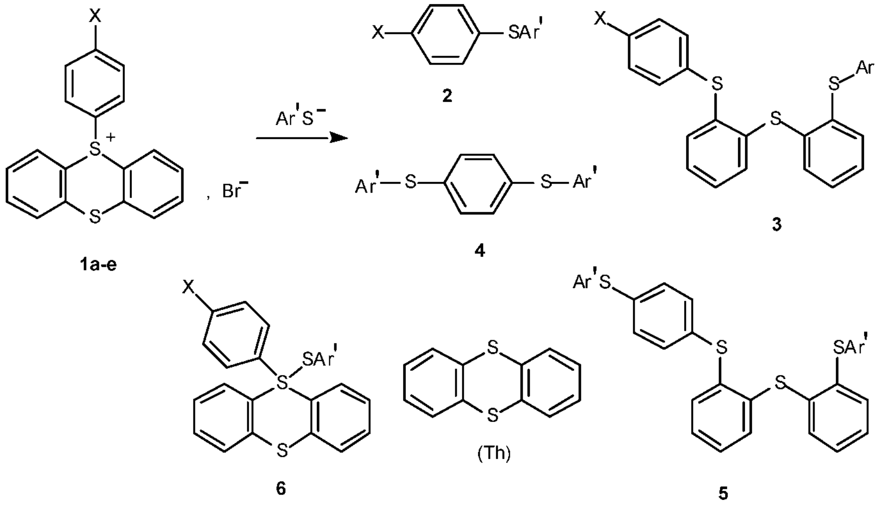
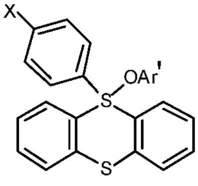


1, X = **a**, H; **b**, Me; **c**, MeO; **d**, Cl; **e**, Br

Scheme 1

reaction. In these reactions, the formation of **12**, representing 60–70% of ring-opening reaction, had to be preceded by replacement of the halogen atom in **1**. Surprisingly, very little 1,4-(diaryloxy)benzene (**11**) was formed, in contrast with the reactions of **1d** and **e** with thiolates,[1] in which substantial amounts (33–70%) of the analogous 1,4-(diarylthio)benzene were obtained. The last column in each table is an account of the fate of the thianthrene ring in **1**, amounting to 93–101% in Table 1 and 92–101% in Table 2. In principle, the sum of yields of **8** and **9** in Table 1 and of **9** and **11** in Table 2 should equal the yield of Th, an accounting that is met only approximately.

In the reactions of **1a**–**e** with aryl thiolates, we have argued the case that the formation of diaryl sulfides (**2**) and opening of the thianthrenium ring occurred by ligand coupling (LC) within sulfurane intermediates (**6**, X = H, Me, MeO, Cl, Br, Ar′S). Developing such a case for the reactions of **1a**–**e** with **7a**–**c** is not so compelling. There are differences in the relative amounts of products in the thiolate and aryl oxide reactions that prompt this conclusion. Most striking is the paucity in the amounts (0–3.4%) of 1,4-di(aryloxy)benzenes (**11a**–**c**) as compared with the amounts (33–70%) of 1,4-(diarylthio)benzenes (**4**).[1] Formation of **11**, as with formation of **4**, requires first the replacement of halogen in **1** by the nucleophile, Ar′O$^-$ in the present case. It is evident from the yields of **12a**–**c** (42–55%) that this is a facile reaction. Once replacement had occurred (**13**, Scheme 3), however, continuation to formation of **11** and thianthrene (Th) was apparently inhibited. Instead, **13** was carried on mostly to **12** by further reaction with Ar′O$^-$. Inhibition of formation of **11** could result from the formation of a sulfurane (**14**, Scheme 4) in which the aryloxyaryl and aryloxy groups were both apical, allowing LC to give **12** but incapable of leading to **11**. Apical alkyloxy groups (R_FO) in a stable sulfurane of thianthrene have been reported by Perozzi and Martin.[2] If this were the case with **11**, we would have to abandon it, however, for example, for **9i**–**k**, which were obtained in significant amounts (11–17%). That is, analogously oriented apical groups in the sulfurane **15** would not allow for the formation of **9**. A more consistent interpretation of formation of **9** and **11**, then, is *ipso* nucleophilic attack (Scheme 5). The trend in yields of **9** (Tables 1 and 2), in the order X = Br >Cl >H >Me ≥MeO, suits the electronic effects of X on nucleophilic attack at the position in XC_6H_4 *ipso* to the sulfonium sulfur atom, with Hammett $\sigma_p = 0.232$ (Br), 0.227 (Cl), −0.170 (Me) and − 0.268 (MeO).[3] Inhibition of forming **11** by nucleophilic attack of Ar′O$^-$ on the *ipso* position in **13** is similarly understandable, with, for example, σ_p (PhO) = −0.320.[3]



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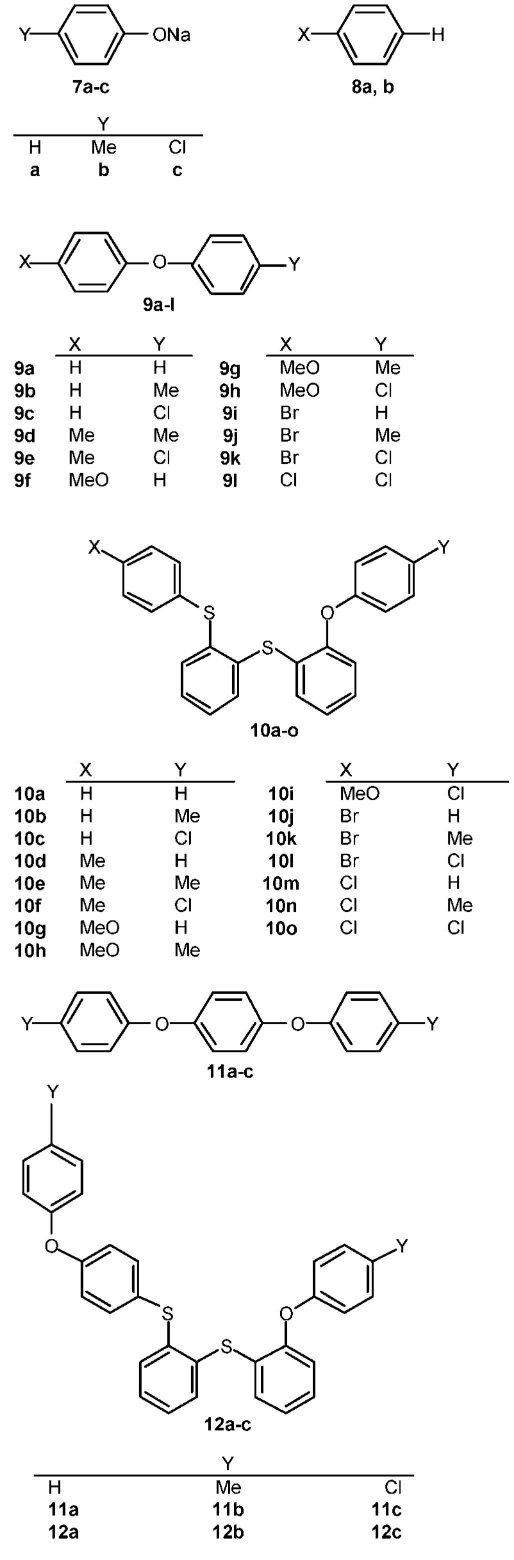
The formation of small amounts of benzene from **1a** and of toluene from **1b** no doubt requires the reaction of **7a**–**c** at the sulfonium sulfur atom of **1a** and **b**. Nevertheless, the thiophilicity of the aryl thiolates appears not to be shared by the aryl oxides, so that the formation of the other products of these reactions is better attributable to aromatic nucleophilic substitution rather than to LC reactions, as is shown in Scheme 5. In this Scheme, path a, which leads to **9** or **11**, is inhibited by electron-donating groups (Me, MeO, Ar′O), causing path b, which leads to **10** or **12**, to be dominant.



	Y	
H	Me	Cl
a	b	c

	X	Y		X	Y
9a	H	H	**9g**	MeO	Me
9b	H	Me	**9h**	MeO	Cl
9c	H	Cl	**9i**	Br	H
9d	Me	Me	**9j**	Br	Me
9e	Me	Cl	**9k**	Br	Cl
9f	MeO	H	**9l**	Cl	Cl

	X	Y		X	Y
10a	H	H	**10i**	MeO	Cl
10b	H	Me	**10j**	Br	H
10c	H	Cl	**10k**	Br	Me
10d	Me	H	**10l**	Br	Cl
10e	Me	Me	**10m**	Cl	H
10f	Me	Cl	**10n**	Cl	Me
10g	MeO	H	**10o**	Cl	Cl
10h	MeO	Me			

	Y	
H	Me	Cl
11a	**11b**	**11c**
12a	**12b**	**12c**

Scheme 2

EXPERIMENTAL

The method of drying solvent MeCN and the columns A and B for gas chromatography (GC) have been described earlier.[4] Mass spectrometric data were supplied by Dr T. Marriott, Rice University, and elemental analyses were carried out by Desert Analytics (Tucson, AZ, USA). Some diaryl ethers and phenols for preparing sodium aryl oxides were obtained from commercial sources. The preparation of 5-(aryl)thianthrenium bromides (**1a**–**e**) has been described earlier.[1]

*Sodium aryl oxides (**7a**–**c**).* An example is given with sodium phenoxide (**7a**). A solution of 43 g (0.46 mmol) of phenol in 50 ml of dry diethyl ether was added to a protected, stirred suspension of 5.5 g (0.24 mmol) of sodium in 100 ml of diethyl ether. Stirring was continued until sodium could no longer be seen. The white solid product was filtered, washed with hexane to remove phenol and dried under vacuum, to give 24 g (0.21 mmol, 87%) of **7a**.

*Diaryl ethers (**9**) and 1,4-(diaryloxy)benzenes (**11**).* These were prepared essentially as described.[5] A stirred mixture of the appropriate phenol (48 mmol) and KOH (30 mmol) was heated to 140 °C until water no longer distilled off. Next, the appropriate aryl halide (20 mmol) and 240 mg of electrolytic grade Cu powder were added, and heating was continued at 170–180 °C for 14–18 h. Aqueous NaOH was added to the mixture, which was then extracted with diethyl ether. The residue from evaporation of the dried ether solution was either distilled under reduced pressure or passed through a column of silica gel before crystallization from hot ethanol.

9b, from phenol and 4-bromotoluene, 64%, b.p. 160–162 °C/70 mmHg. Lit.[6] b.p. 227–228 °C.

9e, from *p*-cresol and 4-bromochlorobenzene, 75%, m.p. 54–55 °C. Lit.[7] m.p. 55–56 °C.

9f, from phenol and 4-bromoanisole, 83%, b.p. 176–178 °C/20 mmHg. Lit.[8] b.p. 168–170 °C/15 mmHg.

9g, from *p*-cresol and 4-bromoanisole, 82%, m.p. 47–47.5 °C. Lit.[9] m.p. 45–46 °C.

9h, from 4-chlorophenol and 4-bromoanisole, 83%, m.p. 54–55 °C. Lit.[10] oil.

9j, from *p*-cresol and 1,4-dibromobenzene, 35%, m.p. 68.5–69 °C. Lit.[5] m.p. 67 °C.

9k, from 4-chlorophenol and 1,4-dibromobenzene, 37%, m.p. 40–41 °C. Lit.[5] m.p. 36 °C.

9l, from 4-chlorophenol and 4-bromochlorobenzene, 61%, m.p. 28–29 °C. Lit.[11] m.p. 30 °C.

11a, from phenol and 1,4-dibromobenzene, 38%, m.p. 74.5–75.5 °C. Lit.[12] m.p. 76 °C.

11b, from *p*-cresol and 1,4-dibromobenzene, separated chromatographically from **9j**, 40%, m.p. 104.5–105.5 °C. Lit.[5] m.p. 106 °C.

11c, from 4-chlorophenol and 1,4-dibromobenzene, separated chromatographically from **9k**, 36%, m.p. 99.5–100.5 °C. Lit.[5] m.p. 100 °C.

*Reactions of 5-(aryl)-thianthrenium bromides (**1a**–**e**) with sodium aryl oxides (**7a**–**c**) at 80 °C.* Reactions at room temperature were found to be too slow to be useful

Table 1. Products of reactions of **1a–c** with **7a–c**[a]

	Reactants		Products (%)[b]				
Run	**1**	**7**	**8**[c]	**9**[c]	**10**[d]	Th[c]	Sum (%) (Th + **10**)
1	**a**	**a**	**a**, 2.9	**a**, 3.8	**a**, 90	6.7	97
2	**a**	**b**		**b**, 6.4	**b**, 97	4.1	101
3	**a**	**c**		**c**, 8.7	**c**, 91	7.7	99
4	**b**	**a**	**b**, 0.8	**b**, 1.9	**d**, 92	3.6	96
5	**b**	**b**		**d**, 0	**e**, 96	0	96
6	**b**	**c**		**e**, 0.9	**f**, 95	2.3	97
7	**c**	**a**		**f**, 0.1	**g**, 91	2.9	94
8	**c**	**b**		**g**, 1.2	**h**, 92	1.2	93
9	**c**	**c**		**h**, 0.2	**i**, 93	1.3	94

[a] In MeCN at 80 °C.
[b] Yields are based on the amount of **1**.
[c] GC yield.
[d] Isolated yield.
[e] Average of two runs.

Table 2. Products of reactions of **1d** and **e** with **7a–c**[a]

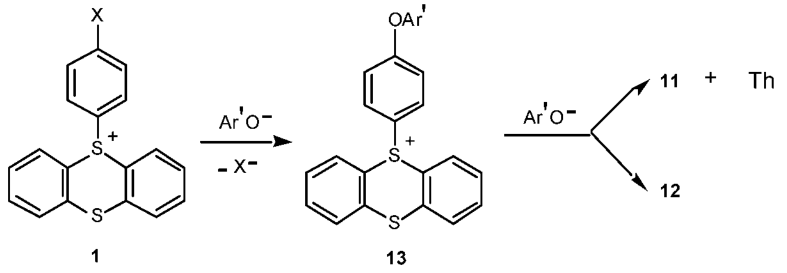
	Reactants		Products (%)[b]					
Run	**1**	**7**	**9**[c]	**10**[d]	**11**[c]	**12**[d]	Th[c]	Sum (%) (Th + **10** + **12**)
1	**d**	**a**	**c**, 10	**m**, 25	**a**, 1.8	**a**, 55	14	94
2	**d**	**b**	**e**, 11	**n**, 28	**b**, 0.3	**b**, 49	15	92
3	**d**	**c**	**l**, 11	**o**, 25	**c**, 3.4	**c**, 49	18	92
4	**e**	**a**	**i**, 17	**j**, 30	**a**, 0	**a**, 42	22	94
5	**e**	**b**	**j**, 16	**k**, 31	**b**, 0.4	**b**, 42	24	97
6	**e**	**c**	**k**, 17	**l**, 23	**c**, 3.2	**c**, 55	23	101

[a] In MeCN at 80 °C.
[b] Based on the amount of **1**.
[c] GC yield.
[d] Isolated yield.

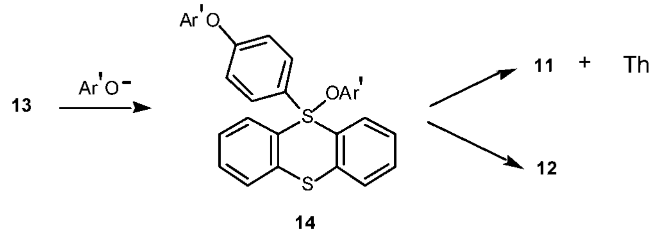
and were therefore carried out at 80 °C in either a septum-capped flask or in a sealed ampule. Examples are given with **1a** and **1e**.

Reaction of **1a** *with* **7a**. A mixture of 279 mg (0.746 mmol) of **1a**, 354 mg (3.05 mmol) of **7a** and naphthalene as GC standard in 20 ml of MeCN was heated in a septum-capped flask at 80 °C for 2 days. After adding butanone to the cooled solution as a second GC standard, assay for **9a** and thianthrene was carried out on column A and of benzene (**8a**) on column B. The solution was then poured into 50 ml of water, which was followed by three extractions with diethyl ether. Evaporation of the dried ether solution gave a residue which was placed on a silica gel column. Naphthalene, Th and **9a** were washed from the column with petroleum ether (PE). Elution with PE–diethyl ether (20:1) gave 260 mg (0.673 mmol, 90%) of 2-phenoxy-2′-(phenylthio)diphenyl sulfide (**10a**), m.p. 75–77 °C.

Reaction of **1e** *with* **7a**. The same procedure was used with 289 mg (0.640 mmol) of **1e** and 372 mg (3.20 mmol) of **7a**, but with the omission of butanone. Elution with PE–diethyl ether (20:1) gave 50.2 mg of **10j**, 94.6 mg of



Scheme 3



Scheme 4

12a and a mixture of the two. The mixture was rechromatographed to give 39.5 mg of **10j** and 35.1 mg of **12a**. Thus the yield of **10j** (0.193 mmol) was 30% and that of **12a** (0.271 mmol) was 42%. **10j** was crystalliized from hot ethanol and had m.p. 75–75.5 °C, but **12a** remained as an oil. All other reactions were carried out similarly.

*Attempted preparation of 1,4-bis(phenoxy)benzene (**11a**) by reaction of 4-bromodiphenyl ether (**9i**) with **7a** at 80 °C.* A mixture of 168 mg (0.674 mmol) of **9i** and 287 mg (2.48 mmol) of **7a** in 20 ml of MeCN was stirred for 3 days at 80 °C. GC analysis of the cooled solution failed to show the presence of **11a**.

Elemental analyses. Analyses were obtained for all crystalline products **10**.

10a, calcd for $C_{24}H_{18}S_2O$: C, 74.6; H, 4.66; S, 16.6. Found: C, 74.5; H, 4.68; S, 16.3%.

10b, calcd for $C_{25}H_{20}S_2O$: C, 75.0; H, 5.03; S, 16.0. Found: C, 74.9; H, 4.83; S, 15.7%.

10d, calcd for $C_{25}H_{20}S_2O$: C, 75.0; H, 5.03; S, 16.0. Found: C, 74.7; H, 5.07; S, 16.2.

10e, calcd for $C_{26}H_{22}S_2O$: C, 75.3; H, 5.35; S, 15.5. Found: C, 75.0; H, 5.22; S, 15.4%.

10f, calcd for $C_{25}H_{19}S_2OCl$: C, 69.0; H, 4.40; S, 14.7; Cl, 8.15. Found: C, 69.2; H, 4.21; S, 14.5; Cl, 8.17%.

10h, calcd for $C_{26}H_{22}S_2O_2$: C, 72.5; H, 5.15; S, 14.9. Found: C, 72.8; H, 5.11; S, 14.5%.

10i, calcd for $C_{25}H_{19}S_2O_2Cl$: C, 66.6; H, 4.25; S, 14.2; Cl, 7.86. Found: C, 66.5; H, 4.28; S, 14.1; Cl, 7.47%.

10j, calcd for $C_{24}H_{17}S_2OBr$: C, 61.9; H, 3.68; S, 13.8; Br, 17.2. Found: C, 62.0; H, 3.49; S, 14.0; Br, 16.8%.

10k, calcd for $C_{25}H_{19}S_2OBr$: C, 62.6; H, 3.99; S, 13.4; Br, 16.7. Found: C, 62.5; H, 3.94; S, 13.8; Br, 16.6%.

10n, calcd for $C_{25}H_{19}S_2OCl$: C, 69.0; H, 4.40; S, 14.7; Cl, 8.15. Found: C, 69.1; H, 4.29; S, 15.0; Cl, 7.84%.

Mass spectrometry. Molecular masses were measured on all products **10** and **12** that did not crystallize. The error in ppm is given in parentheses.

10c, calcd for $C_{24}H_{17}S_2OCl$: 420.040939. Found: 420.040370 (1.4).

10g, calcd for $C_{25}H_{20}S_2O_2$: 416.090475. Found: 416.090354 (0.29).

10l, calcd for $C_{24}H_{16}S_2OBrCl$: 497.951461. Found: 497.951497 (0.072).

10m, calcd for $C_{24}H_{17}S_2OCl$: 420.040939. Found: 420.040821 (0.28).

10o, calcd for $C_{24}H_{16}S_2OCl_2$: 454.001966. Found: 454.001535 (0.95).

12a, calcd for $C_{30}H_{22}S_2O_2$: 478.106125. Found: 478.106030 (0.20).

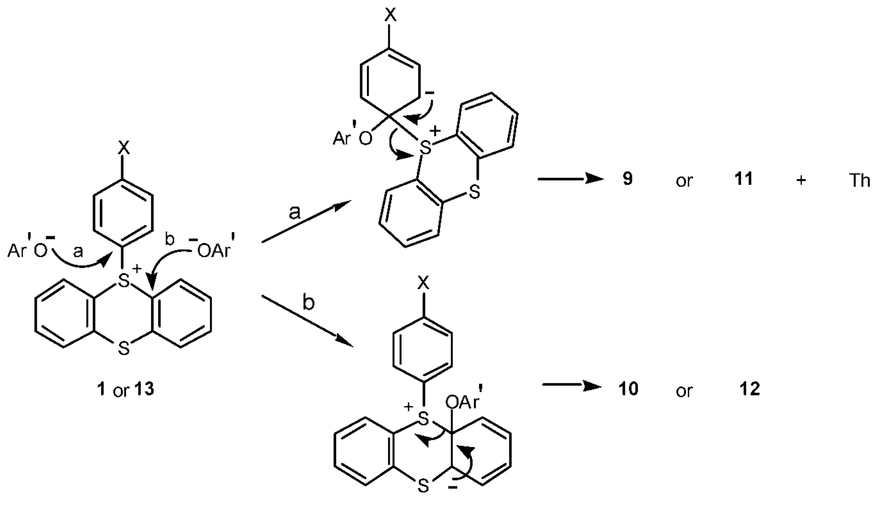
12b, calcd for $C_{32}H_{26}S_2O_2$: 506.137425. Found: 506.137682 (0.51).

12c, calcd for $C_{30}H_{20}S_2O_2Cl_2$: 546.028181. Found: 546.027669 (0.94).

*NMR spectroscopy of **10** and **12**.* ^{1}H NMR spectra were recorded for most of these compounds in $CDCl_3$ at 300 or 500 MHz; δ in ppm, J in Hz.

10b, 300, δ: 7.366–7.078, m, 13H; 7.047–6.996, m, 1H; 6.862, dd, 3H, $J = 6.8$, 1.7 (av.); 2.322, s, 3H.

10d, 300, δ: 7.336–7.258, m, 5H; 7.142–6.930, two m, 12H; 2.342, s, 3H.



Scheme 5

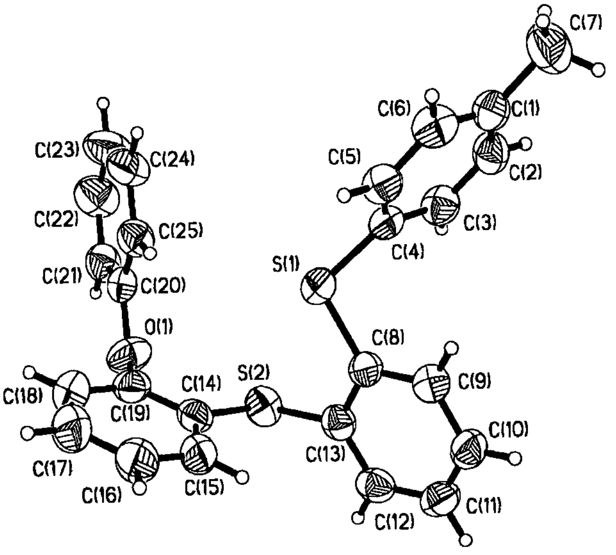


Figure 1. Ortep diagram of 2-(*p*-tolylthio)-2′-(phenyloxy)diphenyl sulfide (**10d**)

10e, 500, δ: 7.307–7.284, m, 3H; 7.190–7.156, m, 1H; 7.134–7.088, m, 7H; 7.028–6.972, m 2H; 6.892–6.857, m, 3H; 2.342, s, 3H; 2.321, s, 3H.

10f, 300, δ: 7.296–7.234, m, 6H; 7.151–7.090, m, 6H; 6.936–6.876, m, 4H; 2.353, s, 3H.

10g, 300, δ: 7.398–7.289, m, 5H; 7.200–6.980, m, 8H; 6.930–6.829, m, 4H; 3.815, s, 3H.

10h, 300, δ: 7.406–7.327, m, 3H; 7.187–6.999, m, 7H; 6.915–6.836, m, 6H; 3.820, s, 3H; 2.324, s, 3H.

10i, 300, δ: 7.392–7.245, m, 3H; 7.106–6.939, m, 7H; 6.928–6.841, m, 6H; 3.829, s, 3H.

10k, 500, δ: 7.326–7.309, m, 2H; 7.260–7.098, m, 10H; 7.036, td, 1H, $J = 7.7$ (av.), 1.3 (av.); 6.865, dd, 1H, $J = 8.0$, 1.0; 6.813, dd, 1H, $J = 8.5$, 2.0; 2.325, s, 3H.

10n, 500, δ: 7.252–7.202, m, 5H; 7.191–7.136, m, 5H; 7.103, d, 2H; 7.030, td, 1H, $J = 7.8$ (av.), 1.3 (ave); 6.864, dd, 1H, $J = 8.75$, 1.0; 6.820, dd, 2H, $J = 7.0$, 2.0; 2.321, s, 3H.

12b, 500, δ: 7.357, dd, 2H, $J = 7.0$, 2.0; 7.314, dd, 1H, $J = 7.0$, 2.0; 7.200–7.152, m, 4H; 7.140–7.102, m, 4H; 7.038–6.993, m, 2H; 6.950, dd, 2H, $J = 6.5$, 2.0; 6.923–6.870, m, 5H; 2.359, s, 3H; 2.322, s, 3H.

X-ray crystallographic data. Data for **10d** were collected as described earlier[1] for the Ortep diagram in Fig. 1. Attempts to grow single crystals of other products **10** were not made.

Supplementary material

Five tables giving crystal data and structural refinements, atomic coordinates and equivalent isotropic displacement parameters, bond lengths and angles, anisotropic displacement parameters, and hydrogen coordinates and equivalent isotropic displacement parameters for 2-(*p*-tolylthio)-2′-(phenoxy)diphenyl sulfide, $C_{25}H_{20}OS_2$, are available at the epoc website at http://www.wiley.com/epoc. The crystal structure has been deposited at the Cambridge Crystallographic Data Centre (deposition number: 193527).

Acknowledgements

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