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Nucleophilic Substitution on Dialkoxy Disulfides. Reactions with Mercaptans or Amines

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Dialkoxy disulfides (1) readily reacted with mercaptans or secondary amines to give alkoxyalkyl trisulfides (4) or alkoxyamino disulfides (5) with elimination of alcohol. These alkoxy sulfides (4 or 5) further reacted with mercaptans or secondary amines to give unsymmetrical dialkyl tetrasulfides (6), alkylamino trisulfides (7), and unsymmetrical diamino disulfide (8). However, reaction of 1 with *N,N*-dimethyl-*p*-phenylenediamine gave *p*-dimethylamino-*N*-thiosulfinylaniline (10). Reaction of 1 and benzylamine or furfurylamine afforded dibenzylideneamino tetrasulfide (11a) or difurfurylideneamino tetrasulfide (11b), whereas 1 and β-phenylethylamine or DL-α-phenylethylamine gave thioamides, PhC(=O)C(=S)NHR (13). Treatment of 1 with thiobenzamide afforded benzonitrile, sulfur, and alcohol.

Dialkoxy disulfides (1) were initially prepared by the reaction of sodium alcoholates with sulfur monochloride[1] with two structures, **1** and **2**, proposed for the products. Raman spectra[2] and dipole-moment data[3] favored the structure **1**, but **2** could not be rigorously excluded. In recent years, Thompson et al. reported an excellent method for the preparation of **1** by the reaction of alcohols and sulfur monochloride in the presence of triethylamine (eq 1) and proved that these compounds have the disulfide structure **1** by NMR and x-ray analysis.[4] Little attention has been paid to reactions of **1**. Previous investigations were not extended beyond investigation of reactions with sodium alcoholate,[1c,5] alkyllithium,[4] and β-diketone.[4] It is seen that the products in these reactions are formed by attack of nucleophiles such as OR^-, R^-, RCO^-CHCOR on sulfur with cleavage of the sulfur–sulfur or sulfur–oxygen bond. Recently, we have also found that[6] equimolar thiocarboxylic acids readily displace an alcohol moiety and afford acylalkoxy trisulfides (**3**). We have now studied reactions of **1** with other nucleophiles.

ROSSOR (**1**) ROSOR (**2**)

$$2ROH + SCl_2 + 2Et_3N \longrightarrow 1 + 2Et_3NHCl \quad (1)$$

$$1 + R'C(=O)SH \longrightarrow ROSSSC(=O)R' + ROH \quad (2)$$

(**3**)

Results and Discussion

Dialkoxy disulfides (1) react readily with equimolar amounts of mercaptan in carbon tetrachloride. The alcohol is eliminated gradually, and monosubstituted products, alkoxyalkyl trisulfides (**4**), are obtained in 20–50% yields along with disubstituted products, symmetrical dialkyl tetrasulfides. Elimination of alcohol was confirmed by infrared spectra and gas chromatography. Results are shown in Table I. The IR spectra of **4** showed absorptions similar to those of **1** in –SO– (660–725 cm^{-1}) and >CO– (880–1020 cm^{-1}) stretching bands (Table III, Supplementary Material). The NMR spectra of **4** showed simple absorptions in its protons of methylene adjacent to an oxygen atom, RCH_2O– (Table III, Supplementary Material), with no apparent magnetic nonequivalence.[7]

Secondary amines were less reactive than mercaptans and their reaction with **1** required refluxing in CCl_4 for 4–8 h. Alkoxyamino disulfides (**5**) (Scheme I) were obtained in 19–74%

Scheme I

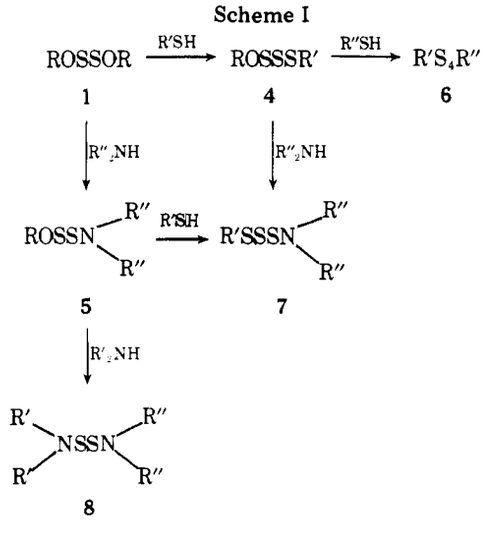

Table I. Monosubstituted Products of Dialkoxy Disulfides[a]

ROSSOR + XH → ROSSX + ROH

Compd	R	X	Bp, °C (mm)	Yield, %
4a	C_2H_5	C_2H_5S	72.5 (3.2)	45
4b	C_2H_5	n-C_3H_7S	66 (0.9)	43
4c	C_2H_5	i-C_3H_7S	72 (1.4)	50
4d	C_2H_5	t-C_4H_9S	53 (0.6)	40
4e	CH_3	t-C_4H_9S	51 (1.0)	22
5a	C_2H_5	$(C_2H_5)_2N$	58 (2.1)	74
5b	C_2H_5	$(CH_2)_5N$	85 (1.0)	41
5c	CH_3	$(C_2H_5)_2N$	63 (7)	54
5d	CH_3	$(CH_2)_4N$	53 (1.1)	19
5e	CH_3	$(i$-$C_3H_7)_2N$	53 (0.5)	22

[a] Satisfactory analytical data (±0.2% for C, H, S, and N) were reported for all compounds in the table.

yields as shown in Table I and Table III (Supplementary Material). The remaining alkoxy group in **4** and **5** could be further displaced with mercaptans or secondary amines to give unsymmetrical dialkyl tetrasulfides (**6**) and alkylamino trisulfides (**7**)[8] in good yields (Scheme I). These results are shown in Table II and Table IV (Supplementary Material). Unsymmetrical diamino disulfide (**8**) was obtained by the reaction of **5** with the other secondary amine, but the yield was lower than those of the other disubstituted products, **6** or **7**. Displacement of alkoxy groups as outlined in Scheme I by –SR or $-NR_2$ groups gives unsymmetrical polysulfides not readily prepared directly from sulfur halides.

Reactions of **1** with primary amines[9] gave a variety of products as follows. When *N*,*N*-dimethyl-*p*-phenylenediamine and equimolar amounts of diethoxy disulfide (**1a**) were refluxed in benzene, the color of the solution gradually turned to deep violet with elimination of ethanol. *p*-Dimethylamino-*N*-thiosulfinylaniline (**10**)[10] was obtained by column chromatography of the reaction mixture. Presumably **10** is generated by elimination of ethanol from the intermediate ethoxyamino disulfide (**9**) (eq 3 and 4).

$$p\text{-}Me_2NC_6H_4NH_2 + EtOSSOEt \xrightarrow{\mathbf{1a}} [p\text{-}Me_2NC_6H_4NHSSOEt]\ (\mathbf{9}) + EtOH \quad (3)$$

$$\mathbf{9} \longrightarrow p\text{-}Me_2NC_6H_4N{=}S{=}S\ (\mathbf{10}) + EtOH \quad (4)$$

Benzylamine and **1a** in benzene afforded dibenzylideneamino tetrasulfide (**11a**),[11] sulfur, and ethanol (eq 5). Considering the formation of **10** from **1a** and Me_2NPhNH_2, it seems reasonable to assume that this tetrasulfide (**11a**) would be formed also via the thiosulfinyl compound in the following way. Namely, benzylamine and **1a** initially afford the thiosulfinyl compound which isomerizes to benzylideneamino hydrogen disulfide (**12**) with proton transfer. Two molecules of **12** then attack **1a** to form the hexasulfide which decomposes to give **11a** with loss of sulfur (eq 6 and 7). According to this assumption, 2 mol of benzylamine should react with 3 mol of **1a**. This was indirectly supported by the fact that the yield of **11a** increased from 40 to 60% by varying the molar ratio of **1a** to benzylamine from 1 to 1.5. Similarly, furfurylamine reacted with **1a** to give difurfurylideneamino tetrasulfide (**11b**) (eq 8).

$$2PhCH_2NH_2 + 3EtOSSOEt \xrightarrow{\mathbf{1a}} PhC(H){=}NS_4N{=}C(H)Ph\ (\mathbf{11a}) + 6EtOH + 2S \quad (5)$$

Table II. Disubstituted Products of Dialkoxy Disulfides[a]

XSSOR + YH → XSSY + ROH (R = ethyl)

Compd	X	Y	Bp, °C (mm)	Yield, %
6a	C_2H_5S	n-C_3H_7S	79 (0.19)	60
6b	C_2H_5S	i-C_3H_7S	75 (0.07)	63
7a	C_2H_5S	$(C_2H_5)_2N$	72 (0.08)	60
7a	$(C_2H_5)_2N$	C_2H_5S		67
7b	i-C_3H_7S	$(C_2H_5)_2N$	80 (0.12)	73
7c	C_2H_5S	$(CH_2)_4N$	56 (0.23)	56
8a	$(C_2H_5)_2N$	$(CH_2)_4N$	86 (0.07)	37

[a] See footnote *a*, Table I.

$$PhCH_2NH_2 \xrightarrow[-2EtOH]{\mathbf{1a}} [PhCH_2N{=}S{=}S] \longrightarrow [PhC(H){=}NSSH]\ (\mathbf{12}) \quad (6)$$

$$2\,[\mathbf{12}] + \mathbf{1a} \xrightarrow[-2EtOH]{} [PhC(H){=}NS_6N{=}C(H)Ph] \longrightarrow \mathbf{11a} + 2S \quad (7)$$

$$2 \text{ (furfurylamine)} + 3\,\mathbf{1a} \longrightarrow \text{(bis-furfurylideneamino tetrasulfide)}\ (\mathbf{11b}) + 6EtOH + 2S \quad (8)$$

The reaction of **1a** with β-phenylethylamine or DL-α-phenylethylamine differed from that observed with aniline or benzylamine. Although a thioamide (**13**) was separated by column chromatography on silica gel, the IR spectra of the crude products before being chromatographed showed no ν_{NH} or $\nu_{C=O}$ bands. This suggests that **13** is formed by the decomposition of unidentified intermediates during the chromatography. The exact mechanism of the reaction is still not elucidated. The structure of thioamides (**13**) was confirmed by NMR, IR, and mass spectra as described in the Experimental Section.

$$RNH_2 \xrightarrow[-EtOH,-S]{EtOSSOEt\ (\mathbf{1a})} PhC(=O)C(=S)NHR\ (\mathbf{13}) \quad (9)$$

(R = $PhCH_2CH_2$-, PhCH(CH$_3$)-)

13a **13b**

Diethoxy disulfide (**1a**) did not react with benzamide, but it did so with thiobenzamide to give benzonitrile, sulfur, and ethanol (eq 10). The reaction probably proceeded again via thiobenzoyl-*N*-thiosulfinylamine followed by elimination of sulfur (eq 11).

$$PhC(=S)NH_2 + EtOSSOEt \xrightarrow{\mathbf{1a}} PhC{\equiv}N + 3S + 2EtOH \quad (10)$$

$$PhC(=S)NH_2 \xrightarrow[-EtOH]{\mathbf{1a}} [PhC(=S)NSSOEt] \xrightarrow{-EtOH} [PhC(=S)N{=}S] \longrightarrow [Ph{-}C{\equiv}N\ (S_2\text{ ring})] \xrightarrow{-S} PhC{\equiv}N \quad (11)$$

Experimental Section

IR spectra were measured with a Hitachi EPI-G2 spectrometer. NMR spectra were determined in CCl_4 or $CDCl_3$ solution with a Varian A-60 or JEOL JNM-PMX-60 (60 MHz) spectrometer. Mass spectra were obtained on Hitachi double-focusing mass spectrometer RMU-7M at 70 eV. Dialkoxy disulfides were prepared by the method of the literature.[4] All other reagents were obtained commercially.

Alkoxyalkyl Trisulfides (4). A solution of 4.34 g (0.07 mol) of ethyl mercaptan in 20 mL of CCl_4 was added to a stirred solution of 10.74 g (0.07 mol) of diethoxy disulfide (**1a**) in 30 mL of CCl_4 at room temperature, and then the temperature of the mixture was gradually raised to 50 °C and the stirring was continued for an additional 3 h. The reaction mixture was evaporated and EtOH was removed as its CCl_4 azeotrope. The residual liquid was distilled under reduced pressure to give 5.40 g of ethoxyethyl trisulfide (**4a**), bp 72.5 °C (3.2 mm). The other compounds (**4b–e**) were obtained in a similar way.

Alkoxyamino Disulfides (5). A solution of 9.42 g (0.06 mol) of **1a** and 4.38 g (0.06 mol) of diethylamine in 75 mL of CCl_4 was refluxed for 4 h. The solvent and EtOH were removed by evaporation, and the residue was distilled to give 8.04 g of ethoxydiethylamino disulfide (**5a**), bp 58 °C (2.1 mm). The other compounds (**5b–e**) were obtained in a similar way.

Unsymmetrical Dialkyl Tetrasulfides (6). A solution of 3.80 g (0.05 mol) of *n*-propylmercaptan in 20 mL of CCl_4 was added to a stirred solution of 8.50 g (0.05 mol) of **4a** in 30 mL of CCl_4 at room temperature, and the stirring was continued for an additional 1.5 h. Finally, the reaction mixture was refluxed for 2 h. Ethanol and CCl_4 were removed by evaporation and the residue was distilled to give 6.03 g of ethyl-*n*-propyl tetrasulfide (**6a**), bp 79 °C (0.19 mm). Ethylisopropyl tetrasulfide (**6b**) was obtained in a similar way.

Alkylamino Trisulfides (7). A solution of 6.80 g (0.04 mol) of **4a** and 2.92 g (0.04 mol) of diethylamine in 50 mL of CCl_4 was refluxed for 7 h. Ethanol and CCl_4 were removed, and the residue was distilled to give 4.73 g of ethyldiethylamino trisulfide (**7a**), bp 72 °C (0.08 mm). The other compounds, **7b** and **7c**, were obtained in a similar way. Ethyldiethylamino trisulfide (**7a**) was also obtained by refluxing **5a** and ethyl mercaptan in CCl_4 for 7 h.

Unsymmetrical Diamino Disulfide (8). A solution of 11.0 g (0.061 mol) of **5a** and 4.31 g (0.061 mol) of pyrrolidine in 60 mL of CCl_4 was refluxed for 5 h, CCl_4 and EtOH were removed, and the residue was distilled to give 4.64 g of diethylaminopyrrolidyl disulfide (**8a**), bp 86 °C (0.07 mm).

Reaction of 1a with *N,N*-Dimethyl-*p*-phenylenediamine. A solution of *N,N*-dimethyl-*p*-phenylenediamine (4.08 g, 0.03 mol) and **1a** (4.62 g, 0.03 mol) in 50 mL of benzene was refluxed for 6 h, the solvent was removed, and the residue was chromatographed on silica gel using dry benzene to give *p*-dimethylamino-*N*-thiosulfinylaniline (**10**). Recrystallization from *n*-hexane gave 0.2 g of deep violet needles, identified by melting point and IR spectra:[10] mp 112–113 °C dec (lit. 113–115 °C); IR (KBr) 1605, 1535, 1315, 1290, 1180, 830, and 680 cm^{-1}.

Reaction of 1a with Benzylamine or Furfurylamine. A solution of 4.62 g (0.03 mol) of **1a** and 2.14 g (0.02 mol) of benzylamine in 75 mL of benzene was refluxed for 16 h. Then EtOH was removed as its benzene azeotrope by evaporation. The residue was chromatographed on silica gel using *n*-hexane as eluent to give 0.81 g of sulfur and 2.01 g (60%) of dibenzylideneamino tetrasulfide (**11a**). Recrystallization from dry MeOH gave yellow needles, identified by elementary analysis, melting point, and spectral data in the literature:[11] mp 100–101 °C (lit. 100.5–102 °C); NMR (CCl_4) δ 7.88 (s, 2 H), 7.04–7.47 (phenyl, 10 H); IR (KBr) $\nu_{C=N}$ 1600 cm^{-1}. Anal. Calcd for $C_{14}H_{12}N_2S_4$: N, 8.32; S, 38.11. Found: N, 8.28; S, 37.98. Difurfurylideneamino tetrasulfide (**11b**) was obtained from **1a** and furfurylamine in a similar way, yield 55%, as yellow needles from a mixture of *n*-hexane and benzene (4:1): mp 95–95.5 °C; NMR ($CDCl_3$) δ 7.96 (s, 2 H), 7.44 (d, 2 H), 6.88 (d, 2 H), 6.40 (q, 2 H); IR (KBr) $\nu_{C=N}$ 1600 cm^{-1}. Anal. Calcd for $C_{10}H_8N_2O_2S_4$: C, 37.96; H, 2.55; N, 8.85; S, 40.53. Found: C, 37.87; H, 2.58; N, 8.68; S, 40.60.

Reaction of 1a with β-Phenylethylamine or DL-α-Phenylethylamine. A solution of 4.62 g (0.03 mol) of **1a** and 3.63 g (0.03 mol) of β-phenylethylamine in 75 mL of benzene was refluxed for 24 h, and the color of the solution turned to dark red. Benzene and EtOH were removed by evaporation, and the residue was chromatographed on silica gel. Sulfur (1.45 g) was first separated by elution with *n*-hexane. Further elution with benzene gave 1.23 g of benzoyl-*N*-(2-phenylethyl)thioformamide (**13a**), mp 86–90 °C, which was recrystallized from *n*-hexane to give light yellow needles, mp 91–92 °C; IR (KBr) $\nu_{C=O}$ 1675 cm^{-1}, ν_{NH} 3180 cm^{-1}; NMR ($CDCl_3$) δ 8.56–8.10 (br, 1 H), 8.04–7.24 (phenyl, 10 H), 4.08 (q, 2 H), 3.08 (t, 2 H). Anal. Calcd for $C_{16}H_{15}NOS$: C, 71.34; H, 5.61; N, 5.20; S, 11.90. Found: C, 71.35; H, 5.62; N, 5.19; S, 11.86. The mass spectrum exhibited peaks at *m/e* 269 (M^+), 178 ($M^+ - PhCH_2$), 169 ($M^+ - PhC_2H_4$), 149 ($M^+ - PhC_2H_4NH$), 120 ($M^+ - PhCOCS$), 105 ($PhCO^+$), 104, 103, 91. Similarly, DL-α-phenylethylamine (3.63 g) reacted with **1a** (4.62 g) to give sulfur (0.9 g) and benzoyl-*N*-(1-phenylethyl)thioformamide (**13b**) (1.62 g) as a reddish-orange liquid by chromatography using benzene–hexane (1:2) as eluent: IR of **13b** $\nu_{C=O}$ 1660 cm^{-1}, ν_{NH} 3250 cm^{-1}; NMR δ (CCl_4) 9.16 (br d, 1 H) 8.07–6.93 (phenyl, 10 H) 5.72 (m, 1 H) 1.57 (d, 3 H). Anal. Calcd for $C_{16}H_{15}NOS$: C, 71.34; H, 5.61; N, 5.20; S, 11.90. Found: C, 71.42; H, 5.68; N, 5.00; S, 11.83. Mass spectrum *m/e* 269 (M^+), 236, 164, 149, 120, 105, 104, 103.

Reaction of 1a with Thiobenzamide. Thiobenzamide (2.74 g, 0.02 mol) suspended in 30 mL of CCl_4 and **1a** (3.08 g, 0.02 mol) was refluxed for 2 h. Thiobenzamide gradually dissolved and sulfur began to precipitate. After the reaction was over, EtOH and CCl_4 were removed by evaporation, the sulfur was filtered, and the filtrate was distilled to give 1.6 g (78%) of benzonitrile, bp 90.5 °C (33 mm) [lit. 69 °C (10 mm)], which was identified by the IR spectrum.

Registry No.—**1a,** 28752-22-9; **1b,** 28752-21-8; **4a,** 63833-15-8; **4b,** 63833-16-9; **4c,** 63833-17-0; **4d,** 63833-18-1; **4e,** 63833-19-2; **5a,** 63833-20-5; **5b,** 63833-21-6; **5c,** 63833-22-7; **5d,** 63833-23-8; **5e,** 63833-24-9; **6a,** 63833-25-0; **6b,** 63833-26-1; **7a,** 63833-27-2; **7b,** 63833-28-3; **7c,** 63833-29-4; **8a,** 63833-30-7; **10,** 53692-08-3; **11a,** 25829-04-3; **11b,** 63833-31-8; **13a,** 63833-32-9; **13b,** 63833-33-0; C_3H_7SH, 107-03-9; *i*-C_3H_7SH, 75-33-2; *t*-C_4H_9SH, 75-66-1; $(CH_2)_5NH$, 110-89-4; $(CH_2)_4NH$, 123-75-1; $(i\text{-}C_3H_7)_2NH$, 108-18-9; ethyl mercaptan, 75-08-1; diethylamine, 109-89-7; *N,N*-dimethyl-*p*-phenylenediamine, 99-98-9; benzylamine, 100-46-9; furfurylamine, 617-89-0; β-phenylethylamine, 64-04-0; DL-α-phenylethylamine, 618-36-0.

Supplementary Material Available: Table III containing IR and NMR spectral data of **1, 4,** and **5** and Table IV containing NMR spectral data of **6, 7,** and **8** (2 pages). Ordering information is given on any current masthead page.

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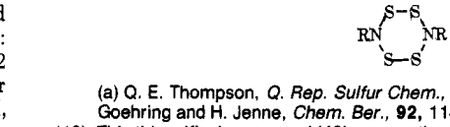

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