on a Perkin-Elmer Model 137 spectrophotometer. Chlorocarbonylsulfenyl chloride[5] and 5-*p*-chlorophenyl-1,3,4-oxathiazol-2-one (1), mp 129–131 °C (lit.[5a] mp 127.5 °C; lit.[6] mp 127–130 °C) were prepared by literature methods. GC yields were determined with internal standards (usually chlorobenzene) and calibration mixtures.

Ethyl 2-Oxo-1,3,4-oxathiazole-5-carboxylate (4). A mixture of 99.7 g (0.85 mol) of ethyl oxamate and 552 g (4.23 mol) of chlorocarbonylsulfenyl chloride in toluene was held at reflux for 5.25 h and then was concentrated under vacuum. Benzene was added to the residue, and the solution was extracted twice with water, twice with 5% $NaHCO_3$, again with water, and was dried ($CaSO_4$) and concentrated under vacuum. The residual oil was filtered to remove sulfur and was crystallized twice from methylcyclohexane to give 95.61 g (64%) of white solid, mp 49–50.5 °C.

Anal. Calcd for $C_5H_5NO_4S$: C, 34.29; H, 2.88. Found: C, 34.28; H, 2.81.

Ethyl 3-(*p*-Chlorophenyl)-1,2,4-thiadiazole-5-carboxylate (3). A solution of 4.27 g (0.020 mol) of 5-(*p*-chlorophenyl)-1,3,4-oxathiazol-2-one and 7.93 g (0.080 mol) of ethyl cyanoformate in 40 mL of dodecane was held at reflux (145–160 °C) for 21 h, cooled, and filtered to give 3.32 g of needles, mp 82–84 °C. Another 0.75 g of product, mp 82–84 °C, was obtained from the filtrate (total yield 76%).

Anal. Calcd for $C_{11}H_9ClN_2O_2S$: C, 49.17; H, 3.38; N, 10.42. Found: C, 48.94; H, 3.19; N, 10.43.

Ethyl 5-Phenyl-1,2,4-thiadiazole-3-carboxylate (6a). A solution of 8.76 g (0.050 mol) of ethyl 2-oxo-1,3,4-oxathiazole-5-carboxylate (4) in 180 g (35 equiv, 1.75 mol) of redistilled benzonitrile was held at reflux for 72 h (after 25 h, most of 4 was gone, and ethyl cyanoformate was present in ca. 7% yield). The solution, which contained the product in 62% yield (GC assay), was concentrated under vacuum to 90 °C (0.5 Torr). The residue was heated with 200 mL of hexane at reflux; the supernatant was decanted, treated with charcoal, filtered, and concentrated under vacuum to 6.1 g of oil and solid. The mixture was chromatographed on 200 g of silica gel (Woelm, for dry column chromatography) with benzene. The first 500 mL of eluate contained 0.8 g of product and impurities. The next 1200 mL of eluate gave 3.82 g (33%) of pure liquid product (GC assay), which crystallized after several months: mp 32–35 °C; $n^{25}D$ 1.5937; mass spectrum *m/e* (rel intensity, fragment) 234 (18, M^+), 206 (3, $M^+ - C_2H_4$), 189 (10, $M^+ - OEt$), 135 (100, $M^+ - EtO_2CCN$), 103 (14, $C_6H_5CN^+$). The infrared spectrum of this material showed considerable differences from that of the isomeric ethyl 3-phenyl-1,2,4-thiadiazole-5-carboxylate:[1] IR (film) 5.75 (s), 8.17 (m), 8.40 (s), 8.62 μ (m); only very weak absorptions were present at 8.94; 9.00, and 14.05 μ where the 5-carboxylate absorbs strongly.

Anal. Calcd for $C_{11}H_{10}N_2O_2S$: C, 56.40; H, 4.30. Found: C, 56.38; H, 4.40.

Ethyl 5-(*p*-Chlorophenyl)-1,2,4-thiadiazole-3-carboxylate (6b). A sample of *p*-chlorobenzonitrile (Eastman) was distilled and then crystallized twice from methylcyclohexane to remove impurities that interfered in the following reaction.

A solution of 8.76 g (0.050 mol) of ethyl 2-oxo-1,3,4-oxathiazole-5-carboxylate and 68.8 g (0.50 mol, 10 equiv) of purified *p*-chlorobenzonitrile was held at 190 °C for 72 h. The solution, which contained the product in 69% yield, was concentrated under vacuum to 150 °C (ca. 2 Torr). The residue was crystallized from heptane (charcoal) to give 8.25 g (61%) of gold crystals, mp 98–99.5 °C. A small sample was crystallized twice from heptane (charcoal) to give a white solid, mp 98–99.5 °C.

Anal. Calcd for $C_{11}H_9ClN_2O_2S$: C, 49.17; H, 3.38. Found: C, 49.21; H, 3.29.

Ethyl 5-(4-Ethoxycarbonylphenyl)-1,2,4-thiadiazole-3-carboxylate (6c). A solution of 1.75 g (0.010 mol) of oxathiazolone 4 and 17.5 g (0.10 mol) of ethyl *p*-cyanobenzoate was stirred at 190 °C for 72 h, cooled, and dissolved in acetone. GC analysis of the solution revealed that the product had formed in 53% yield. Concentration of the solution to 90 °C (0.1 Torr) and two crystallizations of the residue from ethanol (charcoal) at −20 °C gave 0.72 g (23.5%) of beige solid, mp 66–67.5 °C.

Anal. Calcd for $C_{14}H_{14}N_2O_4S$: C, 54.89; H, 4.61. Found: C, 54.89; H, 4.60.

Ethyl 5-*p*-Tolyl-1,2,4-thiadiazole-3-carboxylate (6d). A solution of 7.48 g (0.0427 mol) of oxathiazolone 4 and 50.0 g (0.427 mol) of *p*-tolunitrile (redistilled) was held at 190 °C for 72 h. The reaction mixture, which contained the product in 16% yield, was concentrated under vacuum to 90 °C (0.5 Torr) to give 4.4 g of black residue. Elution chromatography of this material on 200 g of silica gel (Woelm, for dry column chromatography) with benzene gave 1.7 g of product. Crystallization of this material from hexane (charcoal) gave 0.72 g (7%) of solid, mp 65–66.5 °C.

Anal. Calcd for $C_{12}H_{12}N_2O_2S$: C, 58.05; H, 4.87; N, 11.28. Found: C, 58.32; H, 4.91; N, 11.29.

Thermolysis of Ethyl 2-Oxo-1,3,4-oxathiazole-5-carboxylate (4). A 2.00-g (0.01142 mol) sample of 4 was heated under a slow N_2 stream in a 25-mL flask, fitted with a distillation head, in an oil bath at 235–290 °C. The gas stream was bubbled through excess aqueous barium hydroxide. Barium carbonate formed in 94% yield. The liquid distillate consisted of 0.54 g (48%) of pure ethyl cyanoformate (IR, GC analyses). The pot residue, 0.35 g (96%), consisted solely of sulfur (blank IR spectrum).

Control Experiment. A solution of 0.21 g of ethyl cyanoformate in 18.0 g of distilled benzonitrile was held at reflux for 86 h. GC analyses of the solution before and after the heating revealed greater than 99% disappearance of the ethyl cyanoformate in the 86-h period.

Registry No.—1, 17452-79-8; 3, 61689-39-2; 4, 61689-40-5; ethyl oxamate, 617-36-7; chlorocarbonylsulfenyl chloride, 2757-23-5; ethyl cyanoformate, 623-49-4.

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A Convenient Preparation of Unsymmetrical Disulfides. Synthesis of 11,12-Dithiatetradecyl and 11,12-Dithiatridecyl Acetates[1a,b]

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In conjunction with our studies regarding the stereochemical and electronic requirements of the sex pheromone receptors of the European corn borer (*Ostrinia nubilalis*, Hubner[2]) and the red-banded leaf-roller (*Argyrotaenia velutinana*, Walker[3]) moths[4] we required an efficient synthesis of 11,12-dithiatetradecyl acetate (1) and 11,12-dithiatridecyl acetate (2). Literature procedures[5] gave poor yields of 1 and 2 and large amounts of the corresponding symmetrical disulfides. We wish to report a convenient synthesis of unsymmetrical disulfides by a simple procedure that affords disulfides 1 and 2 in good yields.

$CH_3CO_2(CH_2)_{10}SSCH_2CH_3$ (1) $\quad$ $CH_3CO_2(CH_2)_{10}SSCH_3$ (2)

Unsymmetrical disulfides have been prepared by reaction of thiols with sulfenimides,[6] sulfenate esters,[7] sulfenylated thiocarbonates,[8] sulfoxides,[9] and sulfenyl halides[10] and by the reaction of sulfenyl halides with symmetrical disulfides.[11] All of these methods suffer from side reactions (eq 2–4)

$$RS^- + R'S{-}Y \rightleftarrows R{-}S{-}S{-}R' + Y^- \quad (1)$$

$$Y^- + R{-}S{-}S{-}R' \rightleftarrows R'S^- + R{-}S{-}Y \quad (2)$$

$$RS^- + R{-}S{-}S{-}R' \rightleftarrows R{-}S{-}S{-}R + R'S^- \quad (3)$$

$$R'S^- + R{-}S{-}S{-}R' \rightleftarrows R'{-}S{-}S{-}R' + RS^- \quad (4)$$

which have the effect of randomizing the products between

the two symmetrical disulfides and the desired unsymmetrical disulfide.

Proper choice of Y in eq 1 could make the initial nucleophilic attack essentially irreversible. Ideally, Y^- should be a very weak nucleophile. The alkyl thiosulfates ($Y = SO_3^-$) seemed particularly appropriate since sulfite ion is a poor nucleophile, but the literature[12] indicated that treatment of alkyl thiosulfates with mercaptide ions gave a nonstatistical product mixture in which the symmetrical disulfides were the major products. In this system, it is clear that the symmetrical disulfides are formed via the processes summarized in eq 3 and 4. We reasoned that randomization by these processes would be minimized by running the reaction in the presence of an excess of the disulfide corresponding to the mercaptide ion used. In practice this has proved to be the case. The method is illustrated by the synthesis of **1** and **2**.

Treatment of 10-bromo-1-decyl acetate (**3**) with sodium thiosulfate in aqueous methanol gave the alkyl thiosulfate (**4**), eq 5.[13] Addition of the alkyl thiosulfate (**4**) to a stirred mixture of the appropriate disulfide and mercaptide ion (5:0.9 equiv) gave the desired unsymmetrical disulfide (**5**) in good overall yield from **3** ($R = CH_3$, 89%; $R = C_2H_5$, 93%), eq 6. Excess disulfide (**6**) derived from the mercaptide ion reagent (**7**) is included in the reaction mixture so that any undesired mercaptide ion (**8**) formed via eq 7 will be reconverted to the desired product (**5**) via eq 9 rather than to the waste product, symmetrical disulfide (**9**) via eq 10.

$$\underset{\mathbf{3}}{CH_3CO_2(CH_2)_{10}Br} \xrightarrow{Na_2S_2O_3} \underset{\mathbf{4}}{CH_3CO_2(CH_2)_{10}SSO_3^-Na^+} \quad (5)$$

$$\underset{\mathbf{4}}{CH_3CO_2(CH_2)_{10}SSO_3^-Na^+} + RS^- \xrightarrow[RSSR]{} Na_2SO_3 + \underset{\mathbf{5}}{CH_3CO_2(CH_2)_{10}S{-}SR} \quad (6)$$

$$\underset{\mathbf{7}}{CH_3CO_2(CH_2)_{10}S{-}SR} + RS^- \rightleftarrows \underset{\mathbf{8}}{CH_3CO_2(CH_2)_{10}S^-} + \underset{\mathbf{6}}{RS{-}SR} \quad (7)$$

$$\underset{\mathbf{5}}{CH_3CO_2(CH_2)_{10}S{-}SR} + RS^- \rightleftarrows RS^- + \underset{\mathbf{5}}{CH_3CO_2(CH_2)_{10}S{-}SR} \quad (8)$$

$$\underset{\mathbf{8}}{CH_3CO_2(CH_2)_{10}S^-} + \underset{\mathbf{6}}{RS{-}SR} \rightleftarrows RS^- + \underset{\mathbf{5}}{CH_3CO_2(CH_2)_{10}S{-}SR} \quad (9)$$

$$\underset{\mathbf{8}}{CH_3CO_2(CH_2)_{10}S^-} + CH_3CO_2(CH_2)_{10}S{-}SR \rightleftarrows RS^- + \underset{\mathbf{9}}{CH_3CO_2(CH_2)_{10}S{-}S(CH_2)_{10}O_2CCH_3} \quad (10)$$

The method is particularly useful for synthesis of unsymmetrical disulfides in which one component of the disulfide is quite dear and the other is easily available.

Biological activity of **1** and **2** will be described in a separate publication.

Experimental Section

10-Bromodecyl Acetate. Conversion of 1,10-decanediol to 10-bromodecanol was accomplished by a standard literature procedure[14] (52% yield). Acetylation (Ac_2O/pyridine) followed by aqueous workup and short-path distillation yielded analytically pure material (85%): bp 109–111 °C (0.1 Torr); IR (CCl_4) 2940 (s), 2860 (s), 1745 (s, C=O), 1460 (m), 1365 (m), 1250 (s), 1040 cm^{-1} (m); NMR (CCl_4) δ 1.10–1.85 [m, 16, $(CH_2)_8$], 1.93 (s, 3, CH_3CO_2), 3.30 (t, 2, CH_2Br, $J = 6$ Hz), 3.92 (t, 2, CH_2O, $J = 6$ Hz); mass spectrum (70 eV) *m/e* (rel intensity) 220 (18) $(M^+ + 2) - HOAc$, 218 (18) $M^+ - HOAc$, 192 (10), 190 (10), 150 (32), 148 (32), 97 (35), 83 (40), 69 (52), 55 (62), 43 (100). The synthesis of 10-bromodecyl acetate has been reported,[15] but no physical data for the compounds were reported.

Anal. Calcd for $C_{12}H_{23}O_2Br$: C, 51.62; H, 8.30; Br, 28.62. Found: C, 51.48; H, 8.25; Br, 28.70.

Preparation of Sodium Mercaptides.[16] The disulfide, toluene (2 mL/mmol disulfide), and freshly cut sodium metal (2 equiv/equiv of disulfide) were combined in a flask and refluxed until all the sodium metal was gone (ca. 18 h). The reaction mixture was cooled to room temperature, and the sodium mercaptide salt was filtered off and dried in vacuo in a desiccator.

Preparation of Dithia Compounds. The alkyl halide was dissolved in 1:1 methanol–water (2 mL/mmol halide), sodium thiosulfate (1 equiv) was added, and the solution was refluxed for 1 h.[13a] The reaction mixture was cooled to room temperature and slowly pipetted into a stirred mixture of alkyl disulfide–sodium mercaptide (5:0.9 equiv). When the addition was complete, the solution was stirred for 5 min. Ether–pentane (1:1; 2 mL/equiv of halide) was added, and the mixture was stirred overnight.

The solution was vacuum filtered into a separatory funnel and the layers were separated. The organic layer was washed with water and brine and dried (Na_2SO_4), and the solvent was removed in vacuo (aspirator). The residual liquid was subjected to high vacuum to remove residual disulfide. The residue was chromatographed on silica gel (10 g of SiO_2/g compound). Elution with hexane removed minor impurities. Elution with methylene chloride–hexane (1:9 and 2:8) gave TLC pure product. Distillation via a Hickmann still gave analytically pure disulfide.

11,12-Dithiatridecyl Acetate. Using the procedure described above, 10-bromodecyl acetate (2.8 g, 10 mmol) and sodium methyl mercaptide gave 2.6 g (89%) of 11,12-dithiatridecyl acetate. Chromatography yielded 1.4 g (50%, no attempt to optimize recovery) of TLC pure material. Distillation of 1 g yielded 0.9 g of analytically pure material: bp ca. 130 °C (0.05 Torr); IR (CCl_4) 2940 (s), 2860 (s), 1748 (s, C=O), 1470 (m), 1370 (m), 1245 (s), 1045 cm^{-1} (m); NMR (CCl_4) δ 1.15–1.90 [m, 16, $(CH_2)_8$], 1.97 (s, 3, CH_3CO_2), 2.36 (s, 3, CH_3SS), 2.5–2.85 (m, 2, CH_2SS), 3.95 (t, $J = 6$ Hz, 2, CH_2O); mass spectrum (70 eV) *m/e* (rel intensity) 278 (62) M^+, 96 (45), 83 (100), 69 (78), 55 (98), 43 (97), 32 (100).

Anal. Calcd for $C_{13}H_{26}O_2S_2$: C, 56.07; H, 9.41; S, 23.03. Found: C, 55.94; H, 9.32; S, 22.89.

11,12-Dithiatetradecyl Acetate. Using the procedure described above, 10-bromodecyl acetate (2.8 g, 10 mmol) and sodium ethyl mercaptide gave 2.7 g (93%) of 11,12-dithiatetradecyl acetate. Chromatography and distillation [Hickmann still, bp 120 °C (0.15 Torr)] yielded analytically pure material: IR (CCl_4) 2930 (s), 2860 (s), 1748 (s, C=O), 1465 (m), 1370 (m), 1245 (s), 1045 cm^{-1} (w); NMR (ccl_4) δ 1.1–1.85 [m, 19, $(CH_2)_8 + CH_3CH_2S$], 1.94 (s, 3, CH_3CO_2), 2.40–2.85 (m, 4, CH_2SSCH_2), 3.95 (t, $J = 6$ Hz, 2, CH_2O); mass spectrum (70 eV) *m/e* (rel intensity) 292 (48) M^+, 97 (35), 83 (79), 69 (62), 55 (84), 43 (100).

Anal. Calcd for $C_{14}H_{28}O_2S_2$: C, 57.49; H, 9.65; S, 21.92. Found: C, 57.29; H, 9.44; S, 22.13.

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Registry No.—**1**, 61689-33-6; **2**, 61689-34-7; **3**, 33925-77-8; 10-bromodecanol, 53463-68-6; sodium methyl mercaptide, 5188-07-8; sodium ethyl mercaptide, 811-51-8.

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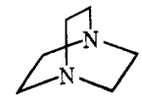
Communications

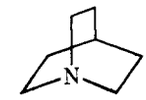
Halogen–Amine Complexes in Chemical Synthesis. 1. The Oxidation of Alcohols by 1,4-Diazabicyclo[2.2.2]octane·2Br_2 Complex

Summary: 1,4-Diazabicyclo[2.2.2]octane·2Br_2 complex employed with additional amine shows promise as a new reagent to oxidize benzyl alcohol to benzaldehyde in high yields, and potential to oxidize secondary alcohols to ketones in the presence of primary alcohols.

Sirs: We are initiating a study of the properties of halogen–amine complexes as reagents in chemical synthesis. In particular, we are examining the Br_2 complexes of 1,4-diazabicyclo[2.2.2]octane (**1**, commonly called Dabco) and similar bicyclic tertiary amines in the quinuclidine (**2**) system.[1]



1



2

The Dabco·2Br_2 complex first reported by Herrick[2a] is readily prepared by combining carbon tetrachloride solutions of the amine and bromine.[2b] This complex is a nonhygroscopic yellow solid that is very stable "on the shelf". It decomposes at 155–160 °C. Unlike the well-known pyridine perbromide complex described by Williams[3] as "a red crystalline solid, decomposing slowly in moist air and smelling strongly of bromine", the Dabco·2Br_2 complex is not affected by ordinary exposure to light, air, or water, and has no offensive odor of bromine or amine. Thus, Dabco·2Br_2 is a source of active

Table I. Alcohol Oxidation

	Reactants, mmol			Conditions			Products, mmol				
Entry	Dabco·2Br_2 complex	Dabco	Alcohol	Solvent[a]	Reaction temp, °C	Reaction time, h	Recovered alcohol	Product, aldehyde or ketone	Product + recovered alcohol	Material balance	% yield
						Benzyl Alcohol					
1	0.499	1.505	2.00	CH_3CN	25	52	1.00	0.81	1.81	91	81
2	0.501	1.502	1.98	CH_3CN	50	6.5	1.35	0.86	2.21	112	86
3	0.498	1.512	1.04	CH_3CN	50	89	0.41	0.56	0.97	93	56
4	0.478	1.523	1.98	CH_2Cl_2	Reflux	11	1.07	0.93	2.00	101	97
5	0.501	1.512	2.04	CH_2Cl_2	Reflux	18	1.16	0.96	2.12	104	96
						Cyclohexanol					
6	0.508	0.529	2.07	CH_3CN	50	3.0 (15.5)[b]	1.31 (1.26)	0.72 (.71)	2.03 (1.97)	98 (95)	71 (70)
7	0.557	1.539	2.19	CH_3CN	50	2.7	1.59	0.71	2.30	105	64
8	0.500	1.615	2.07	CH_3CN	25	69.5	1.83	0.17	2.00	97	17
9	0.498	0	2.02	CH_3CN	50	3.0	1.60	0	1.60	79	0
						Cyclopentanol					
10	0.499	0.476	2.09	CH_3CN	50	3.0	1.55	.52	2.07	100	52
						2-Pentanol					
11	0.513	1.630	2.01	CH_3CN	50	16.0	1.35	0.26	1.61	80	26
12	0.504	0.589	2.20	CH_3CN	50	3.0	1.76	0.36	2.12	96	36
						1-Heptanol					
13	0.504	0.506	2.02	CH_3CN	50	3.2	1.54	0.09	1.63	81	10
						1-Butanol					
14	0.499	0.557	1.99	CH_3CN	50	3.0	1.43	0.11	1.54	77	11
15	0.505	1.526	2.05	CH_3CN	50	19.3	1.22	0	1.22	59	0

[a] The volume of solvent was 2 mL for all reactions except for the benzyl alcohol runs where 4 mL was used. [b] Values in parentheses refer to the same reaction mixture after a longer reaction time.