IR spectra were measured as films or Nujol mulls on a Perkin-Elmer 377 grating spectrophotometer using NaCl cells. GC data were obtained on a Hewlett-Packard Model 5850 A gas chromatograph using a 3% SE-30 Chromosorb column; conversions were corrected for detector response. TLC was performed on silica gel plates from Merck.

Materials. Sodium borohydride and solvents, commercially available grade reagents, were used as purchased. Catalysts, halides, and sulfonate esters were prepared by standard procedures or were obtained commercially.

General Procedure for Reduction under PTC. A solution of $NaBH_4$ in water (2.6 mL/g) was added at the indicated temperature in 30 min with magnetic stirring to a solution of substrate and catalyst in a suitable solvent; the stirring was maintained for the appropriate time (see Tables I and II). The layers were separated, and the product was obtained by distillation of the organic phase. From the distillation residue, the catalyst was recovered in nearly quantitative yields; it may be reused as such.

Registry No. 1-Iodohexadecane, 544-77-4; 1-bromohexadecane, 112-82-3; 1-chlorohexadecane, 4860-03-1; 1-fluorohexadecane, 408-38-8; 1-bromododecane, 143-15-7; 1-bromooctane, 111-83-1; 2-bromohexadecane, 74036-96-7; 1,10-dibromoundecane, 74036-98-9; 1,10-dibromodecane, 4101-68-2; 1-dodecyl methanesulfonate, 51323-71-8; 2-octyl methanesulfonate, 924-80-1; 1-dodecyl toluenesulfonate, 10157-76-3; 11-bromoundecan-1-ol, 1611-56-9; methyl 11-bromoundecanoate, 6287-90-7; 11-bromoundecanoic acid *N,N*-dimethylamide, 2732-31-2; bromocyclodecane, 2749-64-6; bromocyclohexane, 108-85-0; 1-phenyl-1-bromoethane, 585-71-7; 1-phenyl-1-chloroethane, 672-65-1; β-bromostyrene, 103-64-0; 3-phenyl-3-bromoprop-1-ene, 70032-14-3; 2,4-dinitrochlorobenzene, 97-00-7; 2-chloronitrobenzene, 88-73-3; 4-chloronitrobenzene, 100-00-5; diphenylchloromethane, 41376-15-2; benzyl bromide, 100-39-0; benzyl chloride, 100-44-7; 4-nitrobenzyl bromide, 100-11-8; 4-nitrobenzyl chloride, 100-14-1; 4-bromobenzyl bromide, 589-15-1; 4-chlorobenzyl bromide, 622-95-7; 4-fluorobenzyl bromide, 459-46-1; 4-cyanobenzyl bromide, 17201-43-3; 1,4-dibromomethylbenzene, 623-24-5; hexadecane, 544-76-3; dodecane, 112-40-3; octane, 111-65-9; 2-bromoundecane, 39563-54-7; decane, 124-18-5; 1-bromodecane, 112-29-8; octene, 62777-59-7; undecan-1-ol, 112-42-5; methyl undecanoate, 1731-86-8; undecanoic acid *N,N*-dimethylamide, 6225-09-8; cyclododecane, 294-62-2; cyclododecene, 1501-82-2; cyclohexane, 110-82-7; cyclohexene, 110-83-8; phenylethane, 100-41-4; 1-phenyl-1-deuterioethane, 1861-02-5; styrene, 100-42-5; phenylacetylene, 536-74-3; 3-phenylprop-1-ene, 300-57-2; 1,3-dinitrobenzene, 99-65-0; nitrobenzene, 98-95-3; diphenylmethane, 101-81-5; toluene, 108-88-3; 4-nitrotoluene, 99-99-0; 4-bromotoluene, 106-38-7; 4-chlorotoluene, 106-43-4; 4-fluorotoluene, 352-32-9; 4-cyanotoluene, 104-85-8; *p*-xylene, 106-42-3; sodium borohydride, 16940-66-2.

Ketenes. 17. Reaction of Thiophosgene with Dimethylketene,[1] a Stable Thio Acid Chloride

James C. Martin,* P. Glenn Gott, Ronald H. Meen, and Peter W. Raynolds

Research Laboratories, Tennessee Eastman Company, Eastman Chemicals Division, Eastman Kodak Company, Kingsport, Tennessee 37662

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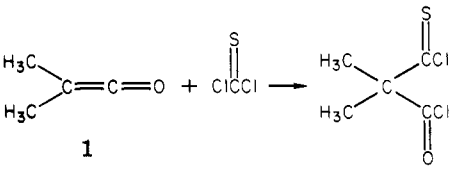
Staudinger reported that thiophosgene did not react with diphenylketene.[2] However, we found that thiophosgene reacted exothermically with dimethylketene (**1**) [2-methyl-1-propen-1-one] to give a high yield of 3-chloro-2,2-dimethyl-3-thioxopropanoyl chloride (**2**, eq 1).

The properties of aliphatic thio acid chlorides have not been reported. Mayer and Scheithauer[3] reported that these compounds cannot be prepared by the methods used

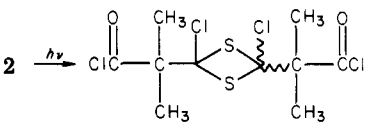


(1)

1 **2**

to prepare aromatic thio acid chlorides and suggested that the aliphatic compounds are unstable. Thus the stability of **2**, which could be distilled at temperatures as high as 100 °C, is unusual.

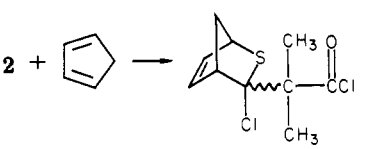
Compound **2** readily dimerized under the influence of sunlight or ultraviolet light to the solid dithietane **3** (eq 2) in a manner similar to the dimerization of thiophosgene.[4] A sample of **2** stored in the dark for several months did not dimerize.



(2)

3

Another reaction involving the carbon–sulfur bond of **2**, analogous to the reaction of thiophosgene with cyclopentadiene,[5] was the thermal [4 + 2] cycloaddition of cyclopentadiene to **2** to give the bicyclic adduct **4** of undetermined stereochemistry (eq 3).

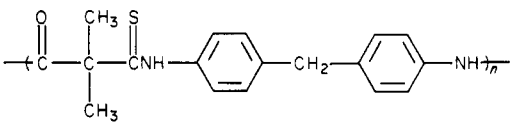


(3)

4

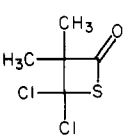
Other reactions of **2** in some ways parallel the reactions of dimethylmalonyl dichloride,[6] although **2** is generally less reactive. Compound **2** reacted readily with active hydrogen compounds, e.g., with phenol to give the diphenyl ester **5**, with methanol to give the dimethyl ester **6**, and with aniline to give the dianilide **7** (Scheme I). Methanethiol (1 equiv) and 1 equiv of sodium hydroxide reacted with **2** to give the ester **8**, and 2 equiv of each afforded the diester **9**. Compound **2** and 1,3-dimethylurea gave the thiobarbituric acid **10** in good yield.

Compound **2** reacted with 4,4′-methylenedianiline under interfacial polymerization conditions to give the polyamide–thioamide **11**.



11

When assigning the structure **2** to the adduct of thiophosgene and **1**, it was necessary to consider an alternate structure, **12**. This 2-thietanone could arise from the



12

addition of a ketene across a carbon–sulfur double bond,[7]

(1) Paper 16 in this series: R. D. Burpitt, K. C. Brannock, R. G. Nations, and J. C. Martin, *J. Org. Chem.*, **36**, 2222 (1971).
(2) H. Staudinger, O. Göhring, and M. Schöller, *Chem. Ber.*, **47**, 40 (1914).
(3) R. Mayer and S., Scheithauer, *Chem. Ber.*, **98**, 829 (1965).
(4) A. Schönberg and A. Stephenson, *Chem. Ber.*, **66**, 567 (1933).
(5) M. S. Raasch, *J. Org. Chem.*, **40**, 161–172 (1975).
(6) J. C. Martin, K. C. Brannock, and R. H. Meen, *J. Org. Chem.*, **31**, 2966 (1966).

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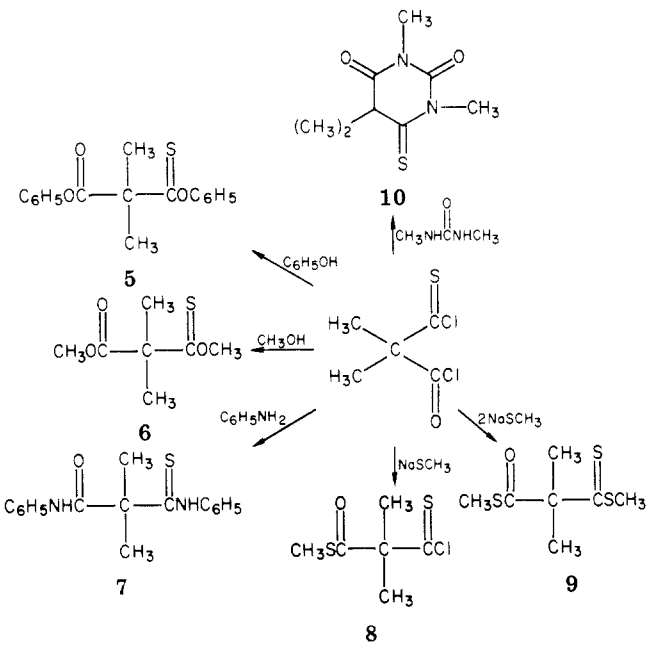
Scheme I



would show the same behavior as **2** in the nucleophilic displacement reactions described, and would show a carbonyl absorption at the same position in the infrared spectrum as that for **2**.[8]

Structure **2** was assigned on the basis of spectral and chemical evidence, as well as by analogy to the product of a known reaction. The ^{13}C spectrum showed two low-field resonances at δ 172.6 and 185.8, and the IR spectrum had strong absorptions at 5.55–5.65 and 8.95 μm, consistent with the presence of an acid chloride and a thio acid chloride,[9] respectively. The Diels–Alder reaction of a carbon–sulfur double bond is known,[5] while compound **12** would not be expected to react with cyclopentadiene. Finally, the proposed structure, **2**, has precedent in the reaction of **1** and phosgene, yielding dimethylmalonyl dichloride.[10]

The mechanism for the formation of **2**, and likewise for the formation of dimethylmalonyl dichloride from **1** and phosgene, probably involves direct insertion into the carbon–chlorine bond. Ketene is known to insert into carbon–oxygen single bonds (with Lewis acid catalysis), into carbon–halogen bonds (both with and without catalysis), and into nitrogen–halogen and sulfur–halogen bonds without catalysis.[11,12] We do not believe that **12** is an intermediate in the formation of **2** because **12** would not be expected to rearrange to **2** under the reaction conditions, and 4,4-dichloro-3,3-difluorothietan-2-one is a known, stable compound.[13]

Experimental Section

3-Chloro-2,2-dimethyl-3-thioxopropanoyl Chloride (2). Dimethylketene (61 g, 0.87 mol) was added to a stirred solution of 100 g (0.87 mol) of thiophosgene in 500 mL of carbon tetrachloride over a period of 15 min. The reaction temperature was controlled at 25–45 °C by intermittent use of a cooling bath. The reaction solution was stirred for 12 h at room temperature and then distilled through a 12-in. packed column to give 143 g (89%) of **2** as a red, waxy solid: mp 32–34 °C; bp 62–62.5 °C (8.4 mm); n^{20}_D 1.5171; IR (neat) 5.55, 5.65, 7.20, 7.33, 8.95 μm; NMR ($CDCl_3$) δ 1.74; mass spectrum, *m/e* 184, 156 (–CO), 121 (–CO, Cl); ^{13}C NMR ($CDCl_3$) δ 185.8, 172.6, 73.5, 26.6; UV max 257 nm (ϵ 3980), 430 (11).

Anal. Calcd for $C_5H_6Cl_2OS$: C, 32.5; H, 3.3; Cl, 38.3; S, 17.3. Found: C, 32.7; H, 3.3; Cl, 38.6; S, 17.2.

2-Butyl-3-chloro-2-ethyl-3-thioxopropanol Chloride. In a manner similar to the preparation of **2**, butylethylketene and thiophosgene afforded 86% of 2-butyl-3-chloro-2-ethyl-3-thioxopropanoyl chloride, bp 66–70 °C (0.3 mm).

Anal. Calcd for $C_9H_{14}Cl_2OS$: C, 44.8; H, 5.9; Cl, 29.4; S, 13.3. Found: C, 44.7; H, 6.0; Cl, 29.7; S, 13.1.

2,4-Dichloro-$\alpha,\alpha,\alpha',\alpha'$-tetramethyl-1,3-dithietane-2,4-bis(acetyl chloride) (3). A 14.5-g sample of **2**, contained under nitrogen in a glass vial, was irradiated for 7 days with a Hanovia 54A-10 medium-pressure mercury arc lamp. The resulting mixture was filtered to remove 6.6 g (45%) of **3**, mp 136.5–145 °C. Recrystallization from benzene gave **3** as a colorless solid: mp 139–143 °C; IR (KBr) 5.68 μm; NMR ($CDCl_3$) δ 1.75 (s, 12).

Anal. Calcd for $C_{10}H_{12}Cl_4O_2S_2$: C, 32.5; H, 3.3; Cl, 38.3; S, 17.3; mol wt 370.1. Found: C, 32.6; H, 3.3; Cl, 38.7; S, 17.5; mol wt (ebullioscopic in benzene) 372.6.

3-Chloro-α,α-dimethyl-2-thiabicyclo[2.2.1]hept-5-ene-3-acetyl Chloride (4). A solution of 5 g (0.075 mol) of cyclopentadiene in 10 mL of hexane was maintained at –20 °C while a solution of 12.9 g (0.07 mol) of **2** in 5 mL of hexane was added during a 5-min period. The resulting solution was warmed to 25 °C for 1 h and recooled to –15 °C to give 12.5 g (71%) of **4**, mp 34.5–37 °C. The crystals were stored at –70 °C but degraded to a dark oil after 1 day at room temperature: IR (CCl_4) 5.62 μm; NMR ($CDCl_3$) δ 6.8–6.4 (m, 1 H), 6.3–6.0 (m, 1 H), 4.24 (br s, 1 H), 3.93 (br s, 1 H), 2.60 (d, J = 9, 1 H), 2.2–1.8 (m, 1 H), 1.58 (s, 3 H), 1.46 (s, 3 H); mass spectrum, *m/e* 250.

Anal. Calcd for $C_{10}H_{12}Cl_2OS$: C, 47.8; H, 4.8; Cl, 28.2; S, 12.8. Found: C, 48.0; H, 4.6; Cl, 28.3; S, 12.7.

***O,O*-Diphenyl Dimethylthiomalonate (5).** A solution of 22 g (0.12 mol) of **2** and 27 g (0.28 mol) of phenol in 100 mL of xylene was refluxed for 18 h. The solvent was removed in vacuo, and the residue was recrystallized once from ethyl ether and once from hexane to give 17 g (47%) of **5**: mp 94.5–96.5 °C; NMR (CCl_4) δ 7.5–6.9 (m, 10 H), 1.75 (s, 6 H); UV max 242 nm (ϵ 3930).

Anal. Calcd for $C_{17}H_{16}O_3S$: C, 68.0; H, 5.4; S, 10.7. Found: C, 68.1; H, 5.4; S, 10.4.

***O,O*-Dimethyl Dimethylthiomalonate (6).** A solution of 15 g (0.08 mol) of **2** in 60 mL of methanol was refluxed for 6 h. The excess methanol was removed in vacuo, and the residue was distilled through a short packed column to give 11.4 g (80%) of **6**: bp 80–82 °C (18 mm); n^{20}_D 1.4725; IR (KBr) 5.72 μm; UV max (cyclohexane) 239 nm (log ϵ 3.85), 376 (1.03); NMR (neat) δ 1.42 (s, 6 H), 3.60 (s, 3 H), 4.05 (s, 3 H).

Anal. Calcd for $C_7H_{12}O_3S$: C, 47.7; H, 6.9; S, 18.2. Found: C, 47.8; H, 7.0; S, 18.2.

2,2-Dimethyl-1-thiomalonanilide (7). A solution of 3.7 g (0.02 mol) of **2** in 5 mL of toluene was added during a 15-min period to a stirred solution of 4.2 g (0.045 mol) of aniline and 6 g (0.06 mol) of triethylamine in 10 mL of toluene. The temperature rose from 25 to 55 °C and then was maintained at 80 °C for 45 min. The solvent was evaporated under a stream of nitrogen, and the residue was diluted with water to give 2.8 g (47%) of **7**, mp 123–126 °C. Recrystallization from ethanol gave **5** as yellow crystals: mp 126–127 °C; IR (KBr) 6.02 μm; NMR ($CDCl_3$) δ 1.73 (s, 6), 7.10–7.80 (m, 10 H), 8.30 (s, 1 H), 10.42 (s, 1 H).

Anal. Calcd for $C_{17}H_{18}N_2OS$: C, 68.4; H, 6.1; N, 9.4; S, 10.7. Found: C, 68.3; H, 6.2; N, 9.3; S, 10.8.

***S*-Methyl 3-Chloro-2,2-dimethyl-3-thioxothiopropionate (8).** A partially frozen mixture of 6.7 g (0.14 mol) of methanethiol, 4 g (0.1 mol) of NaOH, and 100 mL of water was stirred in a Waring blender while a solution of 18.5 g (0.1 mol) of **2** in 50 mL of methylene chloride was added rapidly. After 8 min at 25 °C, the organic phase was separated, dried, and distilled through a

(7) H. Staudinger and H. Freudenberger, *Chem. Ber.*, **61**, 1576–1583 (1928); H. Staudinger, "Die Ketene", Enkc, Stuttgart, 1912, referred to in ref 11, p 74.

(8) P. Y. Johnson and G. A. Berchtold, *J. Org. Chem.*, **35**, 584–592 (1970).

(9) Thiophosgene shows a ^{13}C NMR signal of δ 169.9 ($CDCl_3$) and absorbs at 8.95 μm in the infrared region (film).

(10) R. G. Nations and K. C. Brannock (Eastman Kodak Co.), U.S. Patent 3 220 935 (1965); *Chem. Abstr.*, **64**, 9599*g* (1966).

(11) H. Ulrich, "Cycloaddition Reactions of Heterocumulenes", Academic Press, New York, 1967.

(12) W. Steinkopf and M. Kühnel, *Chem. Ber.*, **75**, 1323–1340 (1942).

(13) A. C. Pierce, U.S. Patent 3 661 743 (1972); *Chem. Abstr.* **77**, 82167 (1972).

short Vigreux column to give 8.1 g (41%) of **8** as a red liquid: bp 50–57 °C (0.2 mm); IR (neat) 5.97 μm; NMR (CCl_4) δ 1.69 (s, 6), 2.35 (s, 3). GLC (silicone QF-1 on Chromosorb P) showed this sample of **8** to have about 8% of **9** as an impurity.

***S,S*-Dimethyl Dimethyl-3-thioxodithiomalonate (9).** A partially frozen mixture of 14 g (0.3 mol) of methanethiol, 8 g (0.2 mol) of NaOH, and 200 mL of water was stirred vigorously in a Waring blender while a solution of 18.5 g (0.1 mol) of **2** in 100 mL of methylene chloride was added rapidly. A temperature of 10–15 °C was maintained for 15 min by external cooling. The organic phase was separated, dried, and distilled through a short Vigreux column to give 11.9 g (57%) of **9**: bp 81 °C (0.2 mm); n^{20}_D 1.5982; IR (neat) 6.0 μm; UV max (cyclohexane) 215 (log ε 3.76), 311 (3.91); NMR ($CDCl_3$) δ 1.71 (s, 6 H), 2.25 (s, 3 H), 2.60 (s, 3 H).

Anal. Calcd for $C_7H_{12}OS_3$: C, 40.4; H, 5.8; S, 46.2. Found: C, 40.6; H, 5.9; S, 46.0.

1,3,5,5-Tetramethyl-4-thiobarbituric Acid (10). A mixture of 9.25 g (0.05 mol) of **2**, 4.4 g (0.05 mol) of 1,3-dimethylurea, and 30 mL of ethylene dichloride was refluxed for 15 h. Vacuum concentration and recrystallization at low temperatures from small volumes of toluene gave 6.6 g (66%) of **10**: mp 64–65 °C; IR (KBr) 5.82, 6.0 μm; NMR ($CHCl_3$) δ 1.72 (s, 6 H), 3.39 (s, 3 H), 3.82 (s, 3 H).

Anal. Calcd for $C_8H_{12}N_2O_2S$: C, 48.0; H, 6.0; N, 14.0; S, 16.0. Found: C, 48.0; H, 6.3; N, 13.6; S, 15.7.

Poly[1,4-phenylenemethylene-1,4-phenylene(2,2-dimethyl-1-thioxomalonamido)] (11). A mixture of 5.0 g (0.025 mol) of 4,4′-methylenedianiline, 5.3 g of Na_2CO_3, and 60 mL of water was stirred in a Waring blender while 4.63 g (0.025 mol) of **2** in 250 mL of chloroform was added rapidly. The mixture was stirred for 10 min and then poured into an evaporating dish where the chloroform was allowed to evaporate. The resulting polymer was washed with water and dried to give 7.0 g of **11**: softening from 155 to 190 °C; IR (KBr) 6.10, 6.32 μm; {η} (phenol/tetrachloroethane), 0.24.

Acknowledgment. We thank Mr. Art Spaugh for obtaining and interpreting the ^{13}C NMR of **2**, as well as the reviewers for many useful references and comments.

Registry No. **1**, 598-26-5; **2**, 29309-53-3; **3**, 78127-90-9; **4**, 78127-91-0; **5**, 78127-92-1; **6**, 78127-93-2; **7**, 29309-62-4; **8**, 78149-15-2; **9**, 78127-94-3; **10**, 78127-95-4; **11** polymer, 78127-54-5; **11** repeating unit, 78198-87-5; thiophosgene, 463-71-8; butylethylketene, 17139-73-0; 2-butyl-3-chloro-2-ethyl-3-thioxopropanoyl chloride, 29309-54-4; cyclopentadiene, 542-92-7; 1,3-dimethylurea, 96-31-1; 4,4′-methylenedianiline, 101-77-9.

Direct Substitution vs. Elimination–Addition in Substitution Reactions of *n*-Butyl 1-Butanesulfinyl Sulfone

John L. Kice* and Shi-Ming Wu

Department of Chemistry, Texas Tech University, Lubbock, Texas 79409

Received April 8, 1981

Both aryl α-disulfones (**1**) and sulfinyl sulfones (**2**) are very reactive toward nucleophiles, the reactions taking the course shown in eq 1 and 2, respectively.[1,2] In such reactions the sulfinyl sulfone is the more reactive, generally by a factor of 10^3–10^4.

$$\underset{\mathbf{1}}{ArSO_2SO_2Ar} + Nu^- \rightarrow ArSO_2Nu + ArSO_2^- \quad (1)$$

$$\underset{\mathbf{2}}{ArS(O)SO_2Ar} + Nu^- \rightarrow ArS(O)Nu + ArSO_2^- \quad (2)$$

Recently it was shown[3] that while alkyl α-disulfones, such as *n*-$BuSO_2SO_2Bu$-*n* (**3**), are also reactive toward nucleophiles, their substitution reactions with most nucleophiles proceed via an elimination–addition mechanism (eq 3) rather than by direct substitution at a sulfonyl group (as in eq 1). We were therefore curious whether alkyl

$$\underset{\mathbf{3}}{(n\text{-}PrCH_2SO_2)_2} + Nu^- \xrightarrow[\text{determining}]{\text{rate}} NuH + n\text{-}PrCH{=}SO_2 \xrightarrow{NuH} n\text{-}PrCH_2SO_2Nu + n\text{-}PrCH_2SO_2^- \quad (3)$$

sulfinyl sulfones when reacting with nucleophiles would be found to prefer a reaction pathway involving an initial elimination rather than the direct substitution at the sulfinyl group observed with their aryl counterparts.

A priori one can envisage three different possible courses for initial reaction of a nucleophile with an alkyl sulfinyl sulfone, such as *n*-$BuS(O)SO_2$-Bu-*n*. These are direct substitution at the sulfinyl group (eq 4a), elimination to form a sulfine (eq 4b), and elimination to form a sulfene (eq 4c). Based on the behavior of alkyl α-disulfones,[3] one

$$\underset{\mathbf{4}}{n\text{-}PrCH_2S(O)SCH_2Pr\text{-}n} + Nu^- \xrightarrow{k_{ds}^{SO}} n\text{-}PrCH_2S(O)Nu + n\text{-}PrCH_2SO_2^- \quad (4a)$$

$$\xrightarrow{k_{se}^{SO}} NuH + n\text{-}PrCH{=}S{=}O + n\text{-}PrCH_2SO_2^- \quad (4b)$$

$$\xrightarrow{k_{se}^{SO_2}} n\text{-}PrCH_2SO^- + n\text{-}PrCH{=}SO_2 + NuH \quad (4c)$$

would expect that elimination processes should have the best chance to predominate when the attacking nucleophile is one that is strongly basic, such as an alkoxide ion or a highly basic amine. For this reason we elected to examine first the reactions of (a) CH_3O^- and (b) piperidine with *n*-butyl 1-butanesulfinyl sulfone, *n*-$BuS(O)SO_2Bu$-*n* (**4**), since if these do not show evidence of elimination (eq 4b or 4c) being strongly preferred over direct substitution (eq 4a), it is unlikely that elimination ever competes successfully with direct substitution in reactions of common nucleophiles with alkyl sulfinyl sulfones.

To determine the possible importance of elimination vs. direct substitution in these reactions of **4** we have used two probes: (1) Is the substitution product (*n*-$PrCH_2S(O)OCH_3$ or *n*-$PrCH_2S(O)NC_5H_{10}$) expected for direct substitution formed in significant yield? (2) If it is, is it formed in a deuterated medium with no incorporation of deuterium at the α carbon to the sulfinyl group, or is much or all of the product *n*-PrCHDS(O)Nu, as would be the case if its origin was via addition of NuH to the sulfine (*n*-PrCH=S=O) formed by eq 4b, i.e.

$$n\text{-}PrCH_2S(O)SO_2CH_2Pr\text{-}n \xrightarrow[\text{eq 4b}]{Nu^-} n\text{-}PrCH{=}S{=}O \xrightarrow{NuD} n\text{-}PrCHDS(O)Nu$$

Reaction of sulfinyl sulfone **4** (dissolved in methylene chloride) with a solution of sodium methoxide in methanol led to the formation of methyl 1-butanesulfinate,[4] *n*-

(1) Kice, J. L.; Legan, E. *J. Am. Chem. Soc.* **1973**, *95*, 3912.
(2) Kice, J. L.; Mullan, L. F. *J. Am. Chem. Soc.* **1976**, *98*, 4259.
(3) Farng, L.-P. O.; Kice, J. L. *J. Am. Chem. Soc.* **1981**, *103*, 1137.
(4) Harpp, D. N.; Back, T. G. *J. Org. Chem.* **1973**, *38*, 4328.

0022-3263/81/1946-3913$01.25/0