

**Derivatives of 4-Chloro-3,5-dinitrobenzotrifluoride. 3.**  
**Synthesis of 1,6-Dinitro-3,8-bis(trifluoromethyl)thianthrene**  
**and Related Compounds<sup>1</sup>**

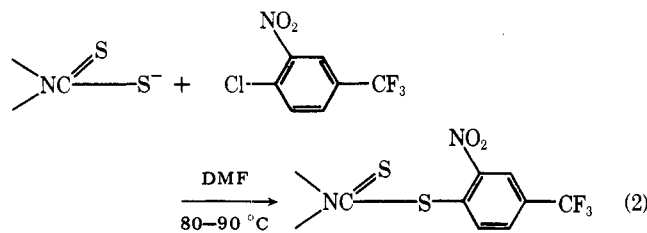
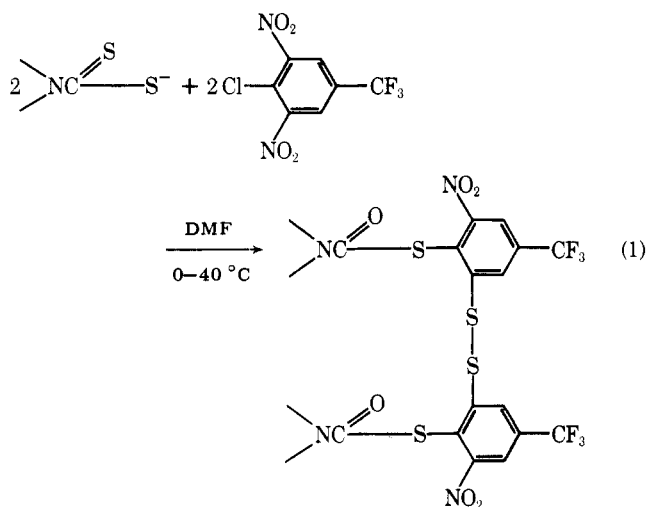
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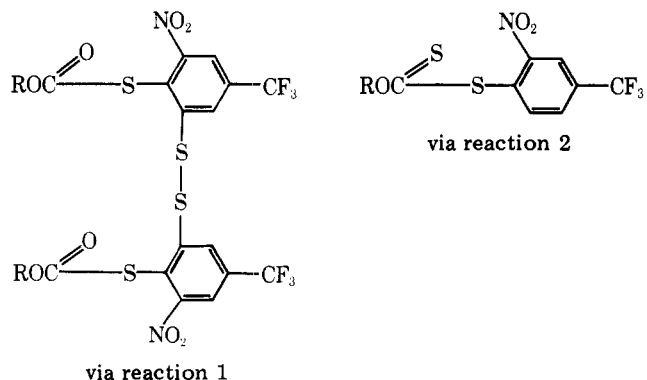
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The reaction of potassium ethyl or isopropyl dithiocarbonate with 4-chloro-3,5-dinitrobenzotrifluoride afforded 1,6-dinitro-3,8-bis(trifluoromethyl)thianthrene (1). Depending on reaction conditions, temperature, and solvent, the reaction of 4-chloro-3-nitrobenzotrifluoride with potassium ethyl or isopropyl dithiocarbonate furnished either bis(2-nitro-4-trifluoromethyl) sulfide (2) and dialkyl dithiocarbonate (4 or 5) or 2-nitro-4-trifluoromethylphenyl alkyl sulfide (6 or 7) or bis(2-nitro-4-trifluoromethyl) disulfide (3). Possible mechanisms and supporting NMR and mass spectral data are discussed. The assigned structure for 1 was verified by x-ray crystal structure analysis.

In a previous communication<sup>2</sup> we reported that the reaction of sodium or triethylamine salts of disubstituted dithiocarbamic acids with 4-chloro-3,5-dinitrobenzotrifluoride or 4-chloro-3-nitrobenzotrifluoride afforded the products as illustrated by reactions 1 and 2, respectively.

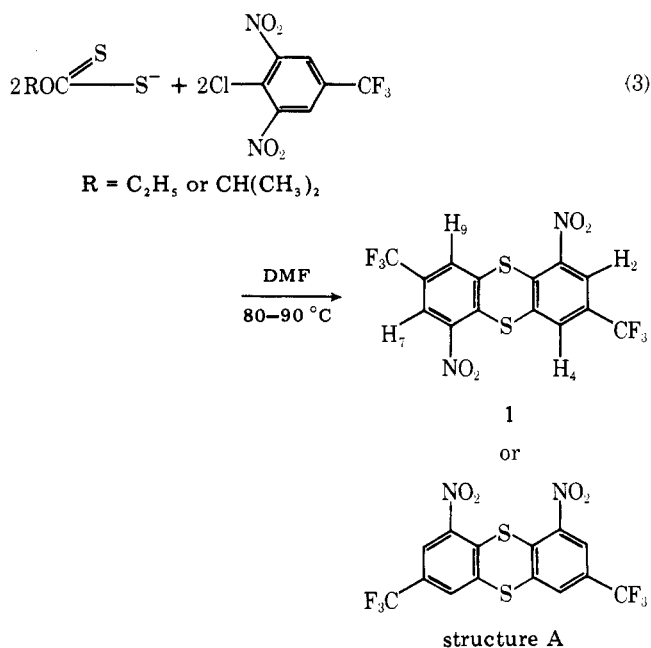


It was anticipated that replacing the sodium or triethylamine salts of disubstituted dithiocarbamic acids in the above reactions 1 and 2 with potassium alkyl dithiocarbonates would have furnished the following analogous products:



However, this was not the case, for the reaction of potassium ethyl or isopropyl dithiocarbonate with 4-chloro-3,5-dinitrobenzotrifluoride in DMF at 80–90 °C afforded 1,6-dinitro-3,8-bis(trifluoromethyl)thianthrene (1) in fair yields. However, based on elemental analysis, molecular weight, NMR, and mass spectral data structure A had to be considered.

Since the above spectral data could not distinguish between structure 1 and A, x-ray crystallographic study was under-

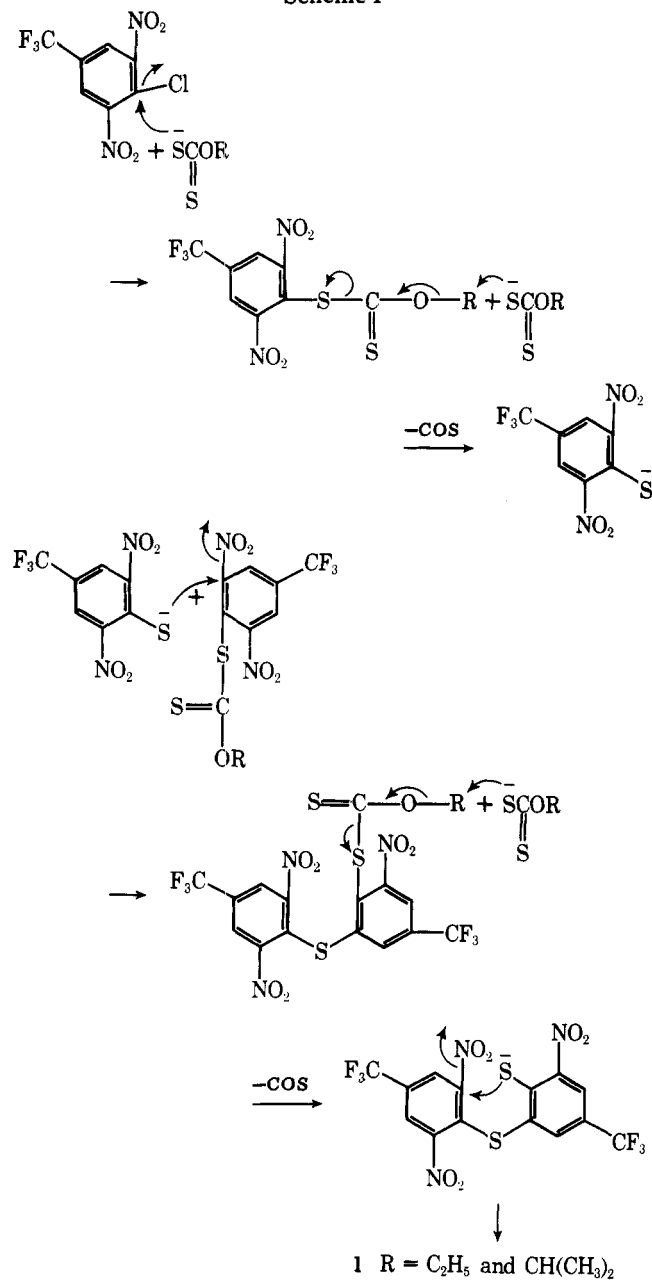


taken. As noted in Figure 1, this study furnished definitive proof that the compound isolated in reaction 3 possessed structure 1. The molecule 1 has two planar halves which form an angle of 137°. Each sulfur atom is in the plane of one benzene ring, to which it is slightly more closely bonded, and 0.13 (1) Å out of the planes of the rings to which they are attached by 32 (1)°. The S1 to O3 distance of 2.727 (9) Å is the same as the S2 to O1 distance of 2.722 (9) Å.

The proposed mechanism for reaction 3 is depicted in Scheme I. The possible mass spectral fragmentation route for 1 is shown in Scheme II (microfilm edition; see paragraph concerning supplementary material).<sup>3</sup>

Depending on reaction conditions, temperature, and solvent, the reaction of 4-chloro-3-nitrobenzotrifluoride with potassium ethyl or isopropyl dithiocarbonate afforded different products. Szmant and Lapinski<sup>4</sup> reported that the reaction of the above halogen compound with potassium ethyl dithiocarbonate in refluxing ethanol for 2 days furnished

Scheme I



bis(2-nitro-4-trifluoromethylphenyl) sulfide (2). Upon reinvestigation of this reaction, we obtained a crude product in 64% yield containing 93% of 2 and 7% of bis(2-nitro-4-trifluoromethylphenyl) disulfide (3). Moreover, a 44% yield of diethyl dithiocarbonate (4) was also isolated from this reaction mixture.

Repeating the above reaction except using potassium isopropyl dithiocarbonate and isopropyl alcohol furnished a crude product in 33% yield containing 45 and 55% of 2 and 3,

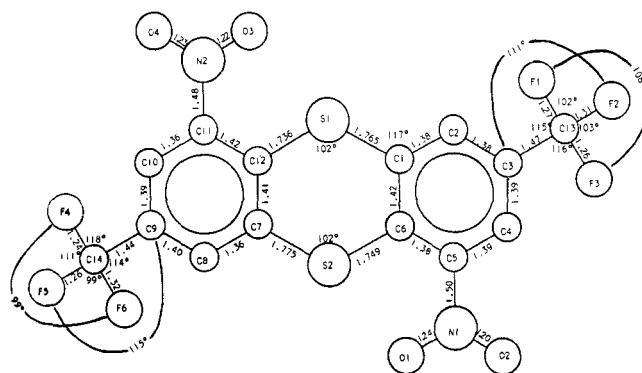
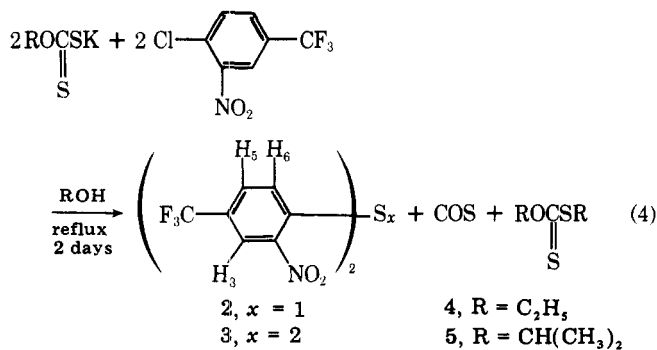
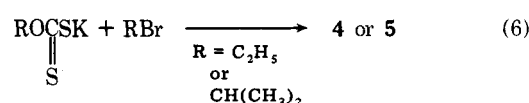
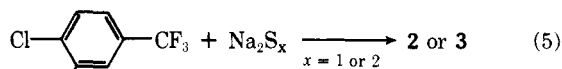


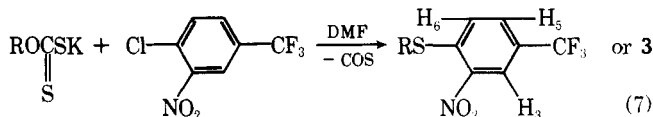
Figure 1. Bond distance and angles for 1.

respectively. In addition a 41% yield of diisopropyl dithiocarbonate (5) was obtained. The lower yield of crude 2 and the higher percentage of 3 contained in the crude product when potassium isopropyl dithiocarbonate was employed in reaction 4 could be explained on the basis of the steric hindrance effect of the bulky isopropyl group. The structures of 2, 3, 4, and 5

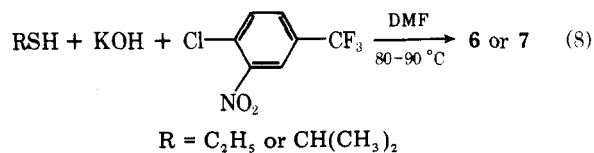


were elucidated by the conventional reactions 5 and 6. The proposed mechanism for reaction 4 is illustrated in Scheme III.

Employing the same reactants as specified in reaction 4 except replacing ethyl or isopropyl alcohol with DMF furnished a novel method for the synthesis of 2-nitro-4-trifluoromethylphenyl ethyl sulfide (6), the isopropyl sulfide (7), or 3 in yields as noted below. Proof of structure for 6 and 7 was

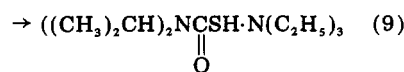


R	Temp, °C	Time, days	% yield		
			6	7	3
C <sub>2</sub> H <sub>5</sub>	75-80	2	96		
CH(CH <sub>3</sub> ) <sub>2</sub>	75-80	2		47	
C <sub>2</sub> H <sub>5</sub>	25-30	5	64		
CH(CH <sub>3</sub> ) <sub>2</sub>	25-30	5			50

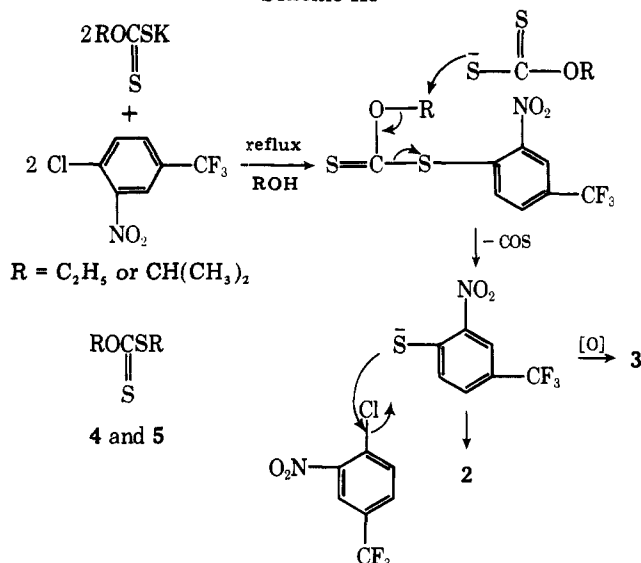


established by the conventional reaction 8. The proposed mechanism for reaction 7 is depicted in Scheme IV.

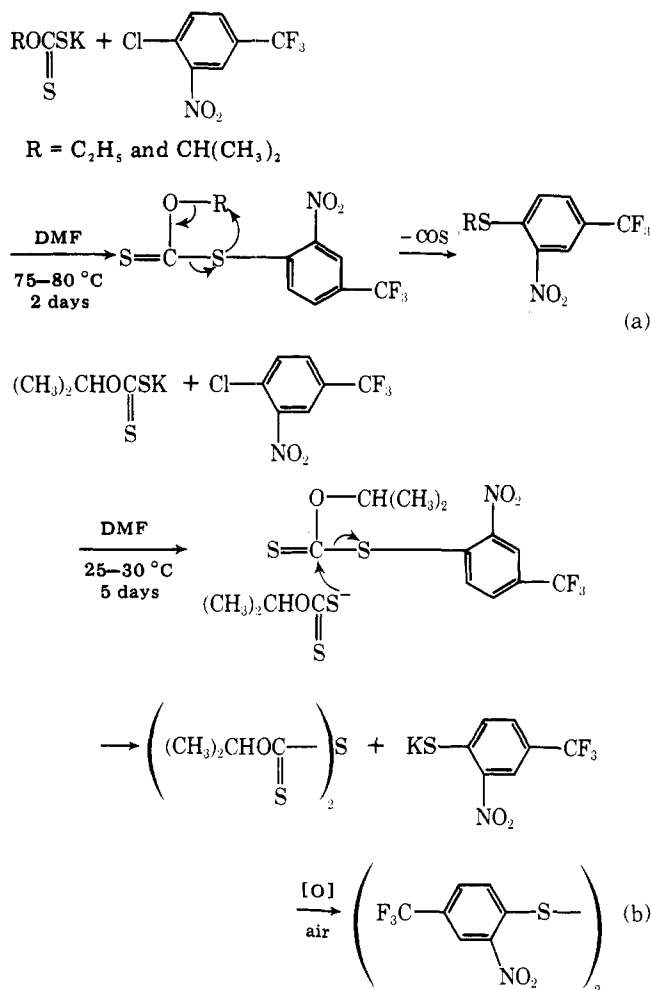
Evidence for the liberation of carbonyl sulfide in reactions 3, 4, and 7 was obtained by the formation of the triethylamine salt of diisopropylthiolcarbamic acid when the gas was allowed to bubble through a solution containing diisopropylamine and triethylamine at 0-10 °C. The identification of



Scheme III



Scheme IV



the liberated carbonyl sulfide lends support for the proposed mechanisms depicted in Schemes I, III, and IV. Moreover, the isolation and identification of 4 and 5 in reaction 4 afforded additional evidence for the proposed mechanism cited in Scheme III.

### Experimental Section

NMR spectra were obtained with a Varian A-60 NMR spectrometer. The chemical shifts are reported in  $\delta$ , using tetramethylsilane as reference. All melting points were taken upon a Fisher-Johns block

and are uncorrected. The mass spectra for 1, 3, 6, and 7 were determined with a Varian MAT CH-7 mass spectrometer operating at an ionizing potential of 70 eV using the direct insertion probe technique with a source temperature of  $250^\circ\text{C}$ . Vapor phase chromatographic analyses of 2 and 3 were performed with an F and M 720 gas chromatograph. A 0.125 in.  $\times$  1 ft stainless steel column packed with 10% OV-17 on Chromosorb W-HP 80-100 mesh was operated isothermally at  $203^\circ\text{C}$  with a He pressure of 40 psi. The peak areas were determined by disk integration. A disk integration factor of 1.216 was determined for baseline corrections. 2 and 3 were run to determine the elution times. Three authentic mixtures were made up containing known amounts of 2 and 3 for determination of area correction factors, 1.0269 for 2 and 0.9743 for 3. Tetrahydrofuran was the diluent for all samples. X ray for 1 was determined by using a Syntex P2<sub>1</sub> and XTL using a  $2\theta$ - $\theta$  scan with graphite monochromatized  $\text{Cu K}\alpha$  radiation.

**1,6-Dinitro-3,8-bis(trifluoromethyl)thianthrene (1).** To a stirred solution containing 0.22 mol of potassium ethyl or isopropyl dithiocarbamate dihydrate in 200 mL of dimethylformamide, 54 g (0.2 mol) of 4-chloro-3,5-dinitrobenzotrifluoride was added in one portion. An exothermic reaction set in causing a temperature rise from  $25$  to  $50^\circ\text{C}$  over a 1-min period. The stirred reaction mixture was heated at  $80-90^\circ\text{C}$  for 28 h and at  $25-30^\circ\text{C}$  for 18 h. During the heating period carbonyl sulfide was liberated. After cooling to  $0^\circ\text{C}$ , 800 g of ice water and 200 mL of ethyl ether were added and stirring continued at  $5-25^\circ\text{C}$  for 2 h. The oily solid was collected by filtration, washed successively with water until the washings were neutral to litmus, then with 200 mL of ethyl ether, and air dried at  $25-30^\circ\text{C}$ . The crude product, mp  $245-248^\circ\text{C}$ , was obtained in 34% yield. After two recrystallizations from ethyl acetate it melted at  $275-276^\circ\text{C}$ : NMR (THF)  $\delta$  7.90 (m, 2, H<sub>4</sub> and H<sub>9</sub>), 8.07 (m, 2, H<sub>2</sub> and H<sub>7</sub>); mass spectrum  $m/e$  (rel intensity) 442 (100), 396 (13.1), 364 (37.1), 350 (16.6), 338 (26.2), 331 (18.1), 318 (23.6), 306 (18.3), 281 (19.9), 269 (30.0), and 69 (16.1).

Anal. Calcd for  $\text{C}_{14}\text{H}_4\text{F}_6\text{N}_2\text{O}_4\text{S}_2$ : C, 38.02; H, 0.91; F, 25.77; N, 6.33; S, 14.50; mol wt, 442.3. Found: C, 37.98; H, 0.93; F, 25.90; N, 6.40; S, 14.41; mol wt, 450 ( $\text{CHCl}_3$ ).

The ethyl ether of the filtrate was removed in vacuo. The filtrate was extracted with three 450-mL portions of chloroform. The combined chloroform extracts were washed with water until neutral to litmus and dried over sodium sulfate and the chloroform was removed in vacuo on a rotary evaporator at a maximum temperature of  $70^\circ\text{C}$  at 1-2 mm. The residue (43 g) was dissolved in 200 mL of 60/40 mixture of cyclohexane/chloroform. One-half of this solution was chromatographed over 540 g of silica gel. Elution with 60/40 mixture of cyclohexane/chloroform gave four unidentifiable fractions.

**Bis(2-nitro-4-trifluoromethylphenyl) Sulfide (2), Diethyl Dithiocarbamate (4), and Diisopropyl Dithiocarbamate (5).** 2 and 4. A procedure reported by Szmant and Lapinski<sup>4</sup> was employed. To a stirred solution containing 158 g (0.7 mol) of 4-chloro-3-nitrobenzotrifluoride in 350 mL of ethanol, 120 g (0.75 mol) of purified potassium ethyl dithiocarbamate was added and heated to  $80^\circ\text{C}$  over a 1-h period. At reflux a very exothermic reaction set in causing a rapid liberation of carbonyl sulfide. After the initial vigorous reaction had subsided, the stirred reaction mixture was heated at reflux for 2 days. After cooling to  $0^\circ\text{C}$ , the precipitate was collected by filtration, slurried with 1 L of water in order to remove the potassium chloride, filtered, and air dried at  $50^\circ\text{C}$ . The crude product 2, mp  $123-130^\circ\text{C}$ , was obtained in 64% yield. The crude 2 was analyzed by VPC and found to contain 93% of 2 and 7% of bis(2-nitro-4-trifluoromethylphenyl) disulfide (3). After three recrystallizations from heptane-ethyl acetate (3:1), 2 melted at  $146-147^\circ\text{C}$ ; NMR ( $\text{Me}_2\text{SO}-d_6$ )  $\delta$  7.70 (d, 2, H<sub>6</sub>), 8.00 (d, 2, H<sub>5</sub>), 8.65 (s, 2, H<sub>3</sub>). A mixture melting point with authentic samples<sup>2,5</sup> was not depressed and their NMR spectra were identical.

Anal. Calcd for  $\text{C}_{14}\text{H}_6\text{F}_6\text{N}_2\text{O}_4\text{S}$ : C, 40.79; H, 1.47; N, 6.79; S, 7.78. Found: C, 40.72; H, 1.48; N, 6.75; S, 7.91.

The ethanol in the filtrate was removed in vacuo at maximum temperature of  $60^\circ\text{C}$  at 10-12 mm. The residue was filtered to remove a small amount of solids and distilled in vacuo. Diethyl dithiocarbamate (4), bp  $42^\circ\text{C}$  (0.25 mm) ( $n_D^{25}$  1.5337), was obtained in 44% yield: NMR ( $\text{CDCl}_3$ )  $\delta$  1.32 (t, 6,  $\text{SCH}_2\text{CH}_3$ ), 1.42 (t, 6,  $\text{OCH}_2\text{CH}_3$ ), 3.15 (q, 2,  $\text{SCH}_2$ ), 4.70 (q, 2,  $\text{OCH}_2$ ).

Anal. Calcd for  $\text{C}_5\text{H}_{10}\text{O}_2\text{S}_2$ : C, 39.97; H, 6.71; S, 42.68. Found: C, 39.95; H, 6.71; S, 42.58.

**2 and 5.** The same procedure described above was employed except that 0.75 mol of potassium isopropyl dithiocarbamate dihydrate and 500 mL of isopropyl alcohol were used. Crude 2, mp  $121-143^\circ\text{C}$ , was obtained in 33% yield. The crude 2 was analyzed by VPC and found to contain 45% of 2 and 55% of the disulfide (3). After three recrystallizations from heptane-ethyl acetate, the crude product contained

43% of 2 and 57% of 3 and no further purification was attempted. Diisopropyl dithiocarbonate 5, bp 57–58 °C (0.4 mm) ( $n_D^{25}$  1.5140), was obtained in 41% yield: NMR (CDCl<sub>3</sub>)  $\delta$  1.40 [2 d, 12, (CH<sub>3</sub>)<sub>2</sub>-CHOC(=S)SCH(CH<sub>3</sub>)<sub>2</sub>], 3.85 (h, 1, SCH), 5.88 (h, 1, OCH).

Anal. Calcd for C<sub>7</sub>H<sub>14</sub>O<sub>2</sub>S<sub>2</sub>: C, 47.15; H, 7.91; S, 35.96. Found: C, 47.27; H, 7.94; S, 35.86.

**4 and 5 (Conventional Method).**—A stirred slurry containing 0.42 mol of potassium ethyl or isopropyl dithiocarbonate and 0.4 mol of ethyl or isopropyl bromide in 200 mL of acetone was heated at reflux for 2 days. The potassium bromide was removed by filtration and the acetone removed in vacuo at maximum temperature of 60 °C at 10–12 mm. The residue was distilled in vacuo. 4 and 5 having the identical boiling point,  $n_D^{25}$ , and NMR spectra as above were obtained in 87 and 91% yield, respectively.

Anal. For 4. Calcd for C<sub>5</sub>H<sub>10</sub>O<sub>2</sub>S<sub>2</sub>: C, 39.17; H, 6.71; S, 42.68. Found: C, 40.17; H, 6.76; S, 42.67. For 5. Calcd for C<sub>7</sub>H<sub>14</sub>O<sub>2</sub>S<sub>2</sub>: C, 47.15; H, 7.91; S, 35.36. Found: C, 47.22; H, 7.93; S, 35.93.

**2-Nitro-4-trifluoromethylphenyl Ethyl Sulfide (6) and 2-Nitro-4-trifluoromethylphenyl Isopropyl Sulfide (7). Method I.** To a stirred solution containing 0.75 mol of purified potassium ethyl or isopropyl dithiocarbonate in 300 mL of DMF, 158 g (0.7 mol) of 4-chloro-3-nitrobenzotrifluoride in 200 mL of DMF was added dropwise in 1 h while not allowing the temperature to exceed 60 °C. After the exothermic reaction had subsided, the stirred reaction mixture was heated at 80–85 °C for 2 days. During the first several hours of the heating period carbonyl sulfide was liberated. After cooling to 0 °C, 1000 g of ice water was added and stirring continued at 0–10 °C for 1 h. The oily precipitate was collected by filtration, washed with cold water until neutral to litmus, and air dried at 25–30 °C. 6, mp 66–68 °C, and 7, mp 73–75 °C, were obtained in 96 and 47% yield, respectively. After recrystallization from heptane, 6 and 7 melted at 71–72 and 79–80 °C, respectively. 6: NMR (CDCl<sub>3</sub>)  $\delta$  1.45 (t, 3, CH<sub>3</sub>CH<sub>2</sub>S), 3.10 (q, 2, CH<sub>3</sub>CH<sub>2</sub>S), 7.65 (m, 2, H<sub>5</sub> and H<sub>6</sub>), 8.55 (s, 1, H<sub>3</sub>); mass spectrum  $m/e$  (rel intensity) 251 (6.5), 219 (2.3), 206 (13.5), 174 (4.5), 159 (6.8), 146 (3.3), 140 (3.3), 126 (3.2), 95 (2.1), 69 (4.7), 45 (4.2), and 18 (100).

Anal. Calcd for C<sub>9</sub>H<sub>9</sub>F<sub>3</sub>NO<sub>2</sub>S: C, 43.03; H, 3.21; N, 5.58; S, 12.76. Found: C, 43.01; H, 3.22; N, 5.65; S, 12.86.

7: NMR (CDCl<sub>3</sub>)  $\delta$  1.45 (d, 6, C(CH<sub>3</sub>)<sub>2</sub>), 3.65 (h, 1, CH), 7.65 (m, 2, H<sub>5</sub> and H<sub>6</sub>), 8.42 (s, 1, H<sub>3</sub>); mass spectrum  $m/e$  (rel intensity) 265 (3.8), 233 (19.6), 206 (19.8), 159 (23.6), 157 (8.1), 95 (8.5), 69 (8.1), 43 (100), 41 (45.7), 39 (15.1), and 27 (25.8).

Anal. Calcd for C<sub>10</sub>H<sub>10</sub>F<sub>3</sub>NO<sub>2</sub>S: C, 45.28; H, 3.80; F, 21.49; N, 5.28; S, 12.09. Found: C, 45.54; H, 3.73; F, 21.60; N, 5.40; S, 12.07.

**Method II. Conventional.** To a stirred slurry containing 0.2 mol of ethyl or isopropyl mercaptan, 13.2 g (0.2 mol) of 85% potassium hydroxide, and 200 mL of DMF, 45.2 g (0.2 mol) of 4-chloro-3-nitrobenzotrifluoride was added in one portion. An exothermic reaction set in causing a temperature rise from 23 to 70 °C. The stirred reaction mixture was heated at 80–90 °C for 18 h. The products 6 and 7 were isolated as described in method I. 6, mp 67–68 °C, and 7, mp 64–66 °C, were obtained in 94 and 85% yield, respectively. After recrystallization from heptane, 6 and 7 melted at 71–72 and 79–80 °C, respectively. The mixture melting point of 6 and 7 derived from methods I and II was not depressed and their NMR spectra were identical.

Anal. (6) Calcd for C<sub>9</sub>H<sub>9</sub>F<sub>3</sub>NO<sub>2</sub>S: C, 43.03; H, 3.21; F, 22.69; N, 5.58; S, 12.76. Found: C, 43.01; H, 3.24; F, 22.80; N, 5.61; S, 12.88.

Anal. (7) Calcd for C<sub>10</sub>H<sub>10</sub>F<sub>3</sub>NO<sub>2</sub>S: C, 45.28; H, 3.80; F, 21.49; N, 5.28; S, 12.09. Found: C, 45.16; H, 3.81; F, 21.25; N, 5.18; S, 12.21.

**Reaction of Potassium Ethyl or Isopropyl Dithiocarbonate with 4-Chloro-3-nitrobenzotrifluoride in DMF at 25–30 °C. 6 and Bis(2-nitro-4-trifluoromethylphenyl) Disulfide (3).** To a stirred slurry at –20 °C containing 0.22 mol of potassium ethyl or isopropyl dithiocarbonate in 200 mL of DMF, 45.1 g (0.20 mol) of 4-chloro-3-nitrobenzotrifluoride was added in one portion. The stirred reaction mixture was maintained at 25–30 °C for 5 days. After cooling to 0 °C, 800 g of ice water was added and stirring continued at 0–10 °C for 1 h. The oily precipitate was collected by filtration, washed with water until neutral to litmus, then with 300 mL of heptane, and air dried at 25–30 °C. 6, mp 71–72 °C, and 3, mp 140–146 °C, were obtained in 64 and 50% yield, respectively. After recrystallization, 6 from heptane and 3 from heptane–ethyl acetate (5:2), 6 and 3 melted at 72 and 163–164 °C, respectively. The mixture melting point of 6 derived from

methods I and II was not depressed and their NMR spectra were identical.

Anal. (6) Calcd for C<sub>9</sub>H<sub>9</sub>F<sub>3</sub>NO<sub>2</sub>S: C, 43.03; H, 3.21; F, 22.69; N, 5.58; S, 12.76. Found: C, 43.21; H, 3.11; F, 22.83; N, 5.56; S, 12.83.

3: NMR (Me<sub>2</sub>SO-*d*<sub>6</sub>)  $\delta$  8.05 (s, 4, H<sub>5</sub> and H<sub>6</sub>), 8.75 (s, 2, H<sub>3</sub>); mass spectrum  $m/e$  (rel intensity) 444 (1.1), 425 (1.8), 222 (11.1), 206 (7.8), 176 (7.3), 174 (33.5), 166 (100), 164 (74.3), 158 (8.3), 157 (10.6), 146 (19.8), 126 (17.3), 113 (15.9), and 95 (87.5).

Anal. Calcd for C<sub>14</sub>H<sub>6</sub>F<sub>6</sub>N<sub>2</sub>O<sub>4</sub>S<sub>2</sub>: C, 37.84; H, 1.36; F, 25.66; N, 6.30; S, 14.43. Found: C, 37.83; H, 1.40; F, 25.75; N, 6.32; S, 14.38.

**3. Conventional Method.** The procedure using 4-chloro-3-nitrobenzotrifluoride and sodium disulfide in ethanol was employed.<sup>6</sup> Crude 3, mp 151–153 °C, was obtained in 94% yield. After recrystallization from heptane–ethyl acetate (5:2), 3 melted at 163–164 °C. The mixture melting point with the product derived above was not depressed and their NMR spectra were identical.

Anal. Calcd for C<sub>14</sub>H<sub>6</sub>F<sub>6</sub>N<sub>2</sub>O<sub>4</sub>S<sub>2</sub>: C, 37.84; H, 1.36; F, 25.66; N, 6.30; S, 14.43. Found: C, 37.71; H, 1.32; F, 25.86; N, 6.32; S, 14.50.

**X-Ray Crystallography for 1.**<sup>7</sup>—Crystal Data. From single crystal diffractometry using Cu K $\alpha$  radiation with  $\lambda = 1.5418$  Å,  $a = 4.898$  (2) Å,  $b = 6.640$  (3) Å,  $c = 49.54$  (3) Å,  $\beta = 92.6$  (7)°,  $v = 1609$  (1) Å<sup>3</sup>;  $Z = 4$ ; space group  $P2_1/c$ ,  $D_x = 1.83$  g/cm<sup>3</sup>,  $D_m = 1.75$  g/cm<sup>3</sup>.

**Structure Determination.** The data collection and structure solution were carried out using a Syntex P2<sub>1</sub> diffractometer and XTL structure calculation package. Only the intensities of 1353 unique reflections judged to be significantly above background were used in refinement. The data were corrected for Lp but not for absorption. The structure was solved straightforwardly using direct methods. Twenty-four atoms were found on an  $E$  map calculated from 147  $E$ 's using the phase set having the highest figure of merit as determined by MULTAN.<sup>8</sup> The remaining four nonhydrogen atoms were found on a Fourier map. The F atoms had elongated and overlapping peaks characteristic of high thermal motion. The model refined isotropically to  $R = 0.15$  and anisotropically to  $R = 0.099$  with the F atoms having large temperature factors. A difference Fourier was calculated and showed a peak having electron density of 1.4 e/Å<sup>3</sup>. Four of the peaks were at positions calculated for the H atoms. All calculated H atoms were input with associated parameters held constant and refinement proceeded to  $R = 0.092$ . The interatomic distances and angles are depicted in Figure 1.

A conventional Fourier was calculated and the above mentioned large residual peak on the difference Fourier showed up again. This was attributed to our model of three ellipsoidal atoms being a poor approximation for the electron distribution in a nearly freely rotating CF<sub>3</sub> group. Since the objective of the work, to determine the molecular structure, had been accomplished, no adjustment to the model was made.

**Acknowledgment.** The assistance of R. W. Fuhrhop and F. L. May is kindly appreciated.

**Registry No.**—1, 62796-18-3; 2, 365-55-9; 3, 860-39-9; 4, 623-79-0; 5, 19615-06-6; 6, 22057-35-8; 7, 62796-19-4; 4-chloro-3,5-dinitrobenzotrifluoride, 393-75-9; potassium ethyl dithiocarbonate, 140-89-6; potassium isopropyl dithiocarbonate, 140-92-1; ethyl mercaptan, 75-08-1; isopropyl mercaptan, 75-33-2; potassium hydroxide, 1310-58-3; 4-chloro-3-nitrobenzotrifluoride, 121-17-5.

**Supplementary Material Available.** Mass fragmentation route for 1 (Scheme II) and atomic coordinate tables (4 pages). Ordering information is given on any current masthead page.

## References and Notes

- Presented at the 173rd National Meeting of the American Chemical Society, Organic Division, New Orleans, La., March 1977.
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