adecylamine, 143-27-1; octadecylamine, 124-30-1; *p*-chloroaniline, 106-47-8; *p*-nitroaniline, 100-01-6; *p*-cyanoaniline, 873-74-5; 2-aminopyridine, 504-29-0; aniline, 62-53-3; *p*-toluidine, 106-49-0; *p*-anisidine, 104-94-9; *p*-aminobenzamide, 2835-68-9; *p*-toluenesulfonamide, 70-55-3; benzenesulfenamide, 98-10-2; *p*-chlorobenzenesulfonamide, 98-64-6; NCS, 128-09-6; NCB, 128-08-5; methylamine, 74-89-5; ethylamine, 75-04-7; isopropylamine, 75-31-0; butylamine, 109-73-9; *tert*-butylamine, 75-64-9; 1-adamantylamine, 768-94-5; 4-aminomethylpyridine, 3731-53-1; 2-aminobenzamide, 88-68-9.

References and Notes

(1) (a) Parts of this paper have been presented at the 9th Middle Atlantic Regional Meeting of the American Chemical Society, Wilkes-Barre, Pa., April 1974; VIth International Symposium on Sulfur Chemistry, Bangor, Wales, July 1974; and 7th Central Regional Meeting of the American Chemical Society, Morgantown, W.Va., May 1975; taken from the Ph.D. Thesis of A. D. Dawson, Temple University, 1975. (b) This is part 16 in the Iminosulfuranes series. Part 15: A. K. Sharma, T. Ku, A. Dawson, and D. Swern, *J. Org. Chem.*, **40**, 2758 (1975).

(2) (a) R. Appel and W. Büchner, *Chem. Ber.*, **95**, 849 (1962); (b) F. Knoll, F. M. Mueller-Kalben, and R. Appel, *ibid.*, **104**, 3716 (1971).

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(8) Knoll, Mueller-Kalben, and Appel[2b] prepared three *N*-alkyl-*S*,*S*-dialkyliminosulfonium hexachloroantimonates in good yield from dimethyl (or methyl ethyl) chlorosulfonium hexachloroantimonates and isopropylamine or 1,2-ethylenediamine; melting points, NMR spectra, and elemental analyses were given. Appel and Büchner[2a] mentioned the preparation of *N*-cyanoethyl-*S*,*S*-diethyliminosulfurane (but not its salts) from diethylsulfilimine and acrylonitrile but without supporting details or characterization (W. Büchner, Dissertation, University of Heidelberg, 1960; not available to us). M. Haake and H. Benack, *Synthesis*, 308, 310 (1976), recently reported the preparation of alkyl(aryl)dialkylaminosuccinimidosulfonium salts from sulfenamides and NCS, and have also prepared ylides from the salts.

(9) After our study had been completed, P. Claus, W. Rieder, P. Hofbauer, and E. Vilsmaier, *Tetrahedron*, **31**, 505 (1975), reported the use of NCS for the efficient preparation of *N*-arylsulfilimines (*N*-aryliminosulfuranes) and their picrates from thioethers and arylamines. In the few cases where duplication of compounds exists, their results and ours agree. However, as we had reported earlier,[1a] the NCS and NCB pathways can also be employed with sulfonamides and carboxamides.

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(15) Dimethyl sulfide, diethyl sulfide, diphenyl sulfide, tetramethylene sulfide, and thiophene (Aldrich) were dried with freshly activated (450 °C, 12 h) Linde molecular sieves before use. β,β'-Dicyanodiethyl sulfide (Aldrich) was used as received. Triethylamine (Aldrich) was purified by fractional distillation from phenyl isocyanate. *N*-Chlorosuccinimide (Arapahoe or Aldrich) was recrystallized from hot water. *N*-Chlorobenzotriazole was prepared by the literature procedure.[5] Methylamine and ethylamine (MCB) were condensed from cylinders. Isopropylamine, butylamine, benzylamine, cyclohexylamine, α-methylbenzylamine, *o*- and *p*-nitroaniline, *p*-chloroaniline, *p*-toluenesulfonamide, *p*-chlorobenzenesulfonamide (Eastman), *d*- and *l*-α-methylbenzylamine, adamantylamine, *p*-aminobenzonitrile, *o*-aminobenzamide (Aldrich), *tert*-octylamine (Rohm and Haas), hexadecylamine, octadecylamine (Armour), and benzenesulfonamide (MCB) were used as received; in all cases purity was 97% or greater. All solvents were the purest and driest grades; they were purified when necessary. For IR, a Perkin-Elmer Infracord 137B or Pye Unicam SP 1000 were used. For NMR, a Varian A-60A or XL-100 with tetramethylsilane or 2,2-dimethyl-2-silapentane-5-sulfonate (Norell) as internal standards were used. Melting points (uncorrected) were taken on a Thomas-Hoover capillary apparatus. Elemental analyses were performed by Micro-Analysis, Inc., Wilmington, Del. For TLC, Eastman silica gel Chromagrams or Analtech prescored silica gel plates with fluorescent indicator were used. Spots were visualized under UV or by development in a closed chamber containing iodine crystals.

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Synthesis of 2,4,6-Trinitrobenzenesulfenyl Chloride and Derivatives[1a]

Gaku Yamamoto[1b] and Morton Raban*

Department of Chemistry, Wayne State University, Detroit, Michigan 48202

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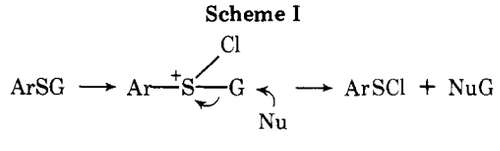
The synthesis of 2,4,6-trinitrobenzenesulfenyl chloride was accomplished by chlorinolysis of phenyl 2,4,6-trinitrophenyl disulfide which could be prepared by reaction of potassium thiopicrate with benzenesulfenyl chloride. The sulfenyl chloride reacted with alcohols, secondary amines, and the silver salts of N-alkylsulfonamides to afford, respectively, sulfenate esters, sulfenamides, and sulfenylsulfonamides.

Nitrobenzenesulfenyl chlorides are well known and the subjects of an extensive literature.[2] The *o*- and *p*-nitrobenzenesulfenyl chlorides are easily prepared and have received considerable attention. The synthesis of 2,4-dinitrobenzenesulfenyl chloride is the subject of an *Organic Syntheses* preparation[3] and is readily available from commercial sources. It has found application not only in synthetic and analytical chemistry, but also in natural product chemistry as a protecting group for the hydroxyl function and numerous derivatives have been characterized.[4] By contrast, 2,4,6-trinitrobenzenesulfenyl chloride (1) heretofore has been a completely unknown compound. In the course of our investigations of the dynamic stereochemistry of sulfenamides,[5] we became interested in 1 and have directed our efforts to its synthesis and the preparation of some of its derivatives.[6]

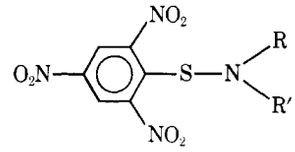
The mononitro- and dinitrobenzenesulfenyl chlorides are readily prepared by chlorinolysis of the corresponding thiols, symmetrical disulfides, or sulfides using chlorine gas or sulfuryl chloride as the chlorinating agent.[2,3] All of these reactions presumably involve electrophilic attack at sulfur followed by nucleophilic displacement of the sulfenyl chloride as a neutral leaving group (Scheme I).

Scheme I



This sequence requires that the sulfur atom act as a nucleophile in the initial step leading to the formation of the chlorosulfonium ion and the presence of an additional ortho nitro group suggests that this step should be less favorable for the synthesis of 1 as compared with its mono- and dinitro analogues. In accord with this expectation, Kharasch et al. were unable to prepare 1 by chlorinolysis of benzyl 2,4,6-trinitrophenyl sulfide (2) using sulfuryl chloride, although this

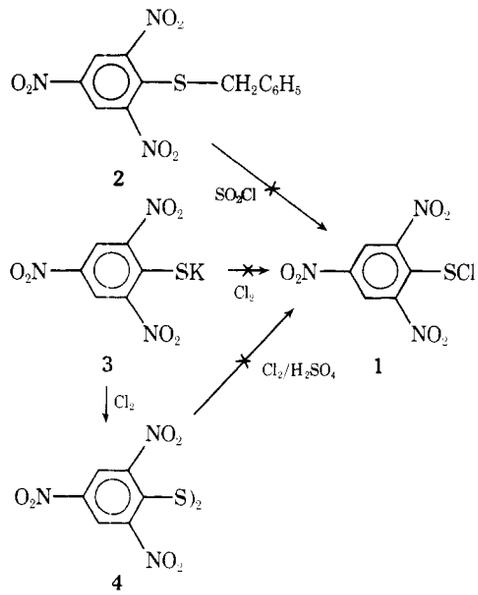
Table I. *N,N*-Dialkyl(aryl)-2,4,6-trinitrobenzenesulfenamides



Registry no.	R	R′	Yield, %	Mp, °C	Anal. Found (calcd) C	H	N	S
60882-81-7	$CHMe_2$	CH_2Ph	66	151–152	49.10 (48.98)	3.95 (4.11)	14.40 (14.28)	8.40 (8.17)
60882-82-8	$CHMe_2$	$CHMe_2$	72	108–109	41.87 (41.86)	4.66 (4.68)	16.19 (16.27)	9.61 (9.31)
60882-83-9	CH_2Ph	$2,4,6\text{-}Me_3C_6H_2$	19	164–165	56.33 (56.40)	4.48 (4.30)	12.00 (11.96	6.71 (6.84)

reaction readily affords 2,4-dinitrobenzenesulfenyl chloride from benzyl 2,4-dinitrophenyl sulfide[7] (Scheme II). Our initial

Scheme II

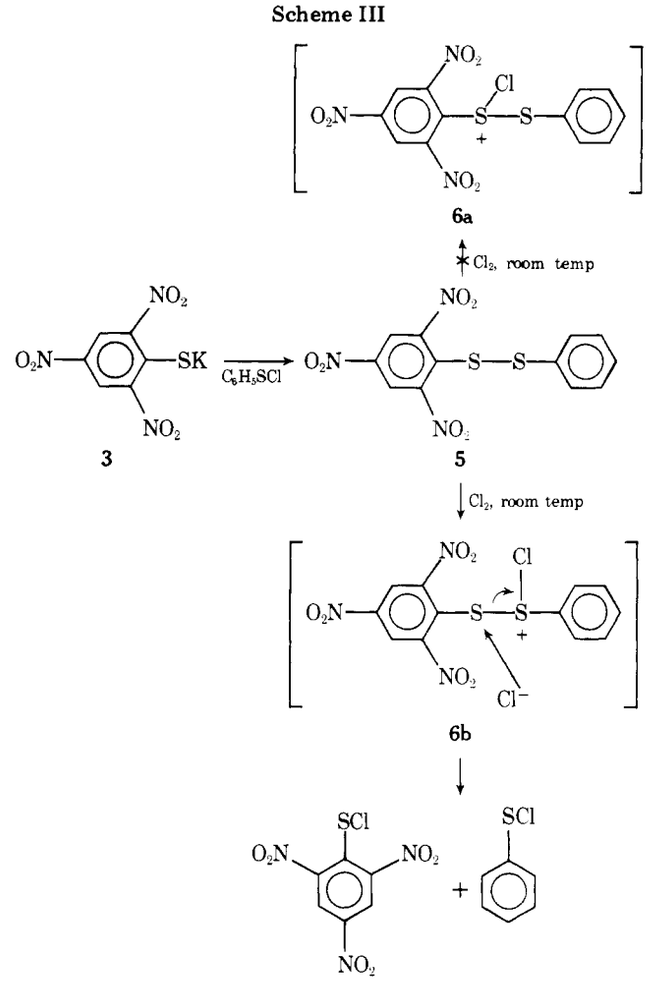


effort involved the chlorination of the potassium thiolate, **3**,[8] reasoning that the negatively charged thiolate anion would be sufficiently nucleophilic to undergo reaction. While reaction did occur, the product obtained was the symmetrical disulfide **4** rather than the desired **1**. This result is not surprising since the chlorination of thiols to yield sulfenyl chlorides is thought to proceed via the symmetrical disulfides as intermediates.[2] The disulfide, in this case, resisted further chlorinolysis even in the presence of oleum, which is recommended by Kharasch in the chlorinolysis of bis(2,4-dinitrophenyl) disulfide.[9]

Since we associated the refractory nature of **4** with the very low nucleophilicity of a sulfur attached to a trinitrophenyl ring, we reasoned that chlorination of a mixed disulfide, such as **5**, might provide a successful route to **1**. Although the formation of intermediate **6a** would be prevented by the trinitrophenyl ring, another more favorable chlorosulfonium ion, **6b**, would be possible which should undergo facile nucleophilic displacement (possibly aided by the electron-withdrawing nitro groups) (Scheme III).

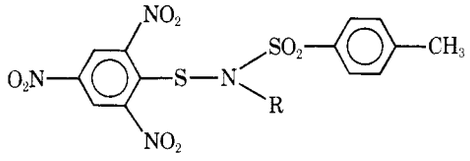
In the fact, this route proved successful. Thus, phenyl 2,4,6-trinitrophenyl disulfide (**4**), prepared by reaction of benzenesulfenyl chloride with **3**, reacted smoothly with chlorine gas at room temperature to afford a mixture of sulfenyl chlorides. The desired product could be isolated from this mixture by selective crystallization. 2,4,6-Trinitrobenzenesulfenyl chloride seems to be rather sensitive to moisture but can be stored for periods of more than 1 month at 5 °C. Longer storage can probably be accomplished by using a sulfenamide as a storage compound,[2] although this was not attempted. The regeneration of the sulfenyl chloride by treatment of a sulfenamide with HCl would not require electrophilic attack on sulfur and should take place readily.

Scheme III



The sulfenyl chloride **1** reacts with nucleophiles, alcohols and amines yielding stable derivatives, sulfenate esters and sulfenamides. Reaction with benzyl alcohol in the presence of pyridine afforded benzyl 2,4,6-trinitrobenzesulfenate. Upon treatment with secondary amines in the presence of a suitable base (pyridine, triethylamine, or an excess of the secondary amine) at room temperature, the corresponding sulfenamides (Table I) are formed from **1**.

Table II. *N*-*p*-Toluenesulfonyl-*N*-alkyl-2,4,6-trinitrobenzenesulfenamides



Registry no.	R	Yield, %	Mp, °C	Anal. Found (calcd) C	H	N	S
60882-84-0	CH_2Ph	48	152–153	47.61 (47.62)	3.23 (3.20)	11.22 (11.11)	12.47 (12.71)
60882-85-1	$CHMe_2$	42	144–146	42.10 (42.10)	3.54 (3.53)	12.44 (12.28)	13.96 (14.05)
60882-86-2	CHMePh	48	167–169	48.59 (48.64)	3.46 (3.50)	10.98 (10.81)	12.43 (12.37)
60882-87-3	CMe_2CH_2OMe	22	158–160	43.46 (43.20)	3.96 (4.03)	11.16 (11.19)	12.55 (12.81)

Synthesis of *N*-arenesulfonyl-*N*-alkyl-2,4,6-trinitrobenzenesulfenamides required a modification in our previous procedure used for obtaining mononitro and dinitro analogues. Previously, we had prepared sulfenylsulfonamides by reaction of the sulfenyl chloride with the lithium salt of the *N*-alkylsulfonamide, which had been prepared in situ by treatment of the sulfonamide with butyllithium.[5] This method, however, was not applicable in the present case; tar formation occurred and no desired product could be isolated. Possibly, electron transfer occurs from the sulfonamide anion to the highly electron-deficient trinitrobenzene ring leading to a radical cation and a radical anion which can undergo subsequent reaction. The use of the corresponding silver salt of the sulfonamide was considered since the greater covalent character of the Ag–N bond would hinder electron transfer. Addition of aqueous silver nitrate to an aqueous solution of the sodium salt of the sulfonamide resulted in the precipitation of the silver salt as a white to gray powder which appeared to be stable to air and moisture. Although further purification and definite characterization were not attempted, the infrared spectra exhibited no absorption which could be associated with the stretching of an N–H bond. Treatment of these silver salts with **1** in benzene at room temperature yielded the desired sulfenylsulfonamides in moderate yield (Table II).

Reaction of **1** with olefins, such as cyclohexene and stilbene, resulted in formation of addition products as indicated by NMR spectra of the reaction mixtures, but isolation of crystalline products has not yet been successful.

Experimental Section

Bis(2,4,6-trinitrophenyl) Disulfide (4). An unsuccessful attempt at preparing the sulfenyl chloride by direct action of chlorine gas on potassium 2,4,6-trinitrobenzenethiolate[8] was made. Thus, chlorine gas was introduced to a dark red suspension of potassium 2,4,6-trinitrobenzenethiolate in dichloromethane at room temperature. Reaction occurred instantaneously, the dark red solid disappeared, and a yellowish solid, probably potassium chloride, formed. After completion of the reaction, the solid was removed by filtration and the solution was evaporated, affording yellow crystals, mp 179–180 °C dec (from tetrahydrofuran–hexane). Elemental analysis and mass spectrometry indicated that the compound was the symmetrical disulfide; no M^+, base peak $M^+/2$. Anal. Calcd for $C_{12}H_4N_6O_{12}S_2$: C, 29.52; H, 0.82; N, 17.21; S, 13.13. Found: C, 29.92; H, 0.73; N, 17.50; S, 12.92.

The disulfide was rather unstable and prolonged heating in tetrahydrofuran afforded the corresponding monosulfide, mp 224–225 °C (lit. mp 228–228.5 °C[10]).

Chlorinolysis of the disulfide was tried using fuming sulfuric acid as a catalyst without success.[11]

Phenyl 2,4,6-Trinitrophenyl Disulfide (5). A solution of 14.5 g of freshly prepared benzenesulfenyl chloride in 20 ml of benzene was added dropwise during the course of 1.5 h to an ice-cold suspension of potassium 2,4,6-trinitrobenzenethiolate,[8] prepared from 24.8 g (0.1 mol) of picryl chloride and 22.0 g (0.11 mol) of freshly prepared potassium sulfide pentahydrate ($K_2S·5H_2O$),[13] in 100 ml of benzene. The reaction mixture was stirred at 5°C for 1 h. The solid formed was filtered off and washed with 30 ml of benzene. The combined benzene solution was concentrated to a volume of 50 ml using a rotary evaporator. After addition of 20 ml of hexane, the solution was kept overnight in a refrigerator, affording 24.2 g (69%) of yellow crystals, mp 87–88 °C (from tetrahydrofuran–hexane): NMR ($CDCl_3$) δ 7.30 (5 H, s), 8.79 (2 H, s).

Anal. Calcd for $C_{12}H_7N_3S_2O_6$: C, 40.79; H, 2.00; N, 11.89, S, 18.15. Found: C, 40,82; H, 1.90; N, 11.98; S, 18.31.

2,4,6-Trinitrobenzenesulfenyl Chloride (1). Chlorine gas was slowly introduced from a cylinder and through sulfuric acid to a magnetically stirred suspension of 10.6 g (30 mmol) of **5** in 200 ml of carbon tetrachloride. A slight evolution of heat was detected. Introduction of the gas was continued for 20 min after the solid disulfide had completely dissolved and a clear orange solution had been obtained. Completion of the reaction was determined by observing the NMR spectrum of the reaction mixture; signals of the starting disulfide [δ 7.14 (5 H, s) and 8.82 (2 H, s)] were completely replaced by the product signals [δ 7.3–8.0 (5 H, m) and 9.1 (2 H, s)]. The excess chlorine and a large part of the solvent were evaporated using a rotary evaporator, to a volume of ca. 30 ml. The red-orange oily solution was kept in a freezer at −20 °C overnight and afforded yellow crystals in a yield of 7.2 g (86%), mp 61–63 °C (from benzene–hexane), NMR (CCl_4) δ 8.95 (s).

Anal. Calcd for $C_6H_2N_3O_6SCl$: C, 25.77; H, 0.72; N, 15.03; S, 11.47; Cl, 12.68. Found: C, 25.83; H, 0.65; N, 14.90; S, 11.85; Cl, 13.25.

Benzyl 2,4,6-Trinitrobenzenesulfenate. A solution of 2,4,6-trinitrobenzenesulfenyl chloride (1.40 g, 5 mmol) in 25 ml of benzene was added dropwise to a solution of benzyl alcohol (0.54 g, 5 mmol) and pyridine (0.40 g, 5 mmol) in 50 ml of benzene at room temperature. Precipitation of a solid (pyridine hydrochloride) occurred immediately. The mixture was stirred for 30 min, filtered, and evaporated. The residual oil was chromatographed through a silica gel column with benzene as an eluent. Pale yellow crystals, mp 100 °C dec (from tetrahydrofuran–hexane), were obtained, which were sparingly soluble in chloroform but soluble in benzene and dimethyl sulfoxide: NMR (dimethyl sulfoxide) δ 4.82 (2 H, s), 7.39 (5 H, s), and 9.12 (2 H, s). Anal. Calcd for $C_{13}H_9N_3SO_7$: C, 44.45; H, 2.58; N, 11.96; S, 9.13. Found: C, 44.40; H, 2.3; N, 12.00; S, 9.39.

This compound decomposed rapidly in moist dimethyl sulfoxide but quite slowly in dry dimethyl sulfoxide.

***N,N*-Dialkyl-2,4,6-trinitrobenzenesulfenamides.** A solution of 10 mmol of 2,4,6-trinitrobenzenesulfenyl chloride in 20 ml of benzene was added dropwise to a solution of 20 mmol of a secondary amine in 50 ml of benzene. The mixture was stirred at room temperature for 5 h, the solid was filtered off, and the solution was evaporated. The residue was chromatographed on silica gel with hexane–benzene (9:1) as an eluent. The pure products were obtained by recrystallization from tetrahydrofuran–ethanol (Table I).

Preparation of Silver Salts of *N*-Alkyl-*p*-toluenesulfonamides. To an ice-cold solution of 10 mmol of an *N*-alkyl-*p*-toluenesulfonamide in 20 ml of methanol was added 10 ml of 1 N aqueous sodium hydroxide and the mixture was stirred for 2 h. A solution of 10 mmol of silver nitrate in 3 ml of water and 10 ml of methanol was added dropwise to the above solution. By the end of addition, white

to slightly gray powdery solid had formed. The mixture was stirred for a further 30 min and filtered. The solid was dried in vacuo in a desiccator ($CaCl_2$), yield 95–99%. Infrared spectra indicated the absence of the N–H group.

***N*-Alkyl-*N*-*p*-toluenesulfonyl-2,4,6-trinitrobenzenesulfenamides.** A solution of 3.5 mmol of 2,4,6-trinitrobenzenesulfenyl chloride in 10 ml of benzene was added to a magnetically stirred suspension of 4 mmol of a silver salt of *N*-alkyl-*p*-toluenesulfonamide in 30 ml of benzene and the mixture was stirred at room temperature for 48 h. After filtration of the solid, the solution was evaporated and the residue chromatographed on silica gel with benzene–hexane (2:1) as an eluent. The products were purified by recrystallization from tetrahydrofuran–hexane (Table II).

Registry No. —**1**, 60882-88-4; **3**, 16158-74-0; **4**, 60882-89-5; **5**, 60882-90-8; NHR_1R_2 (R_1 = $CHMe_2$; R_2 = CH_2Ph), 102-97-6; NHR_1R_2 (R_1 = $CHMe_2$; R_2 = $CHMe_2$), 108-18-9; NHR_1R_2 (R_1 = CH_2Ph; R_2 = 2,4,6-$Me_3C_6H_2$), 60882-91-9; *p*-$MeC_6H_4SO_2NHR$ (R = CH_2Ph)·Ag, 60882-92-0; *p*-$MeC_6H_4SO_2NHR$ (R = CH_2Me_2)·Ag, 60882-93-1; *p*-$MeC_6H_4SO_2NHR$ (R = CHMePh)·Ag, 60882-94-2; *p*-$MeC_6H_4SO_2NHR$ (R = CMe_2CH_2OMe)·Ag, 60882-95-3; benzenesulfenyl chloride, 931-59-0; benzyl 2,4,6-trinitrobenzenesulfenate, 60882-96-4; benzyl alcohol, 100-51-6.

References and Notes

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(11) The use of fuming sulfuric acid as a chlorinolysis catalyst was recommended by Kharaschin in the case of bis(2,4-dinitrophenyl) disulfide.[9,12]
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(13) W. P. Bloxam, *J. Chem. Soc.*, **79**, 756 (1900).

Derivatives of 4-Chloro-3,5-dinitrobenzotrifluoride. 2. Synthesis of 2-(Trifluoromethyl)-4-nitrobenzimidazo[2,1-*b*]benzothiazole and Related Compounds[1]

J. J. D'Amico,* C. C. Tung, and W. E. Dahl

Monsanto Agricultural Products Company, Research Department, St. Louis, Missouri 63166

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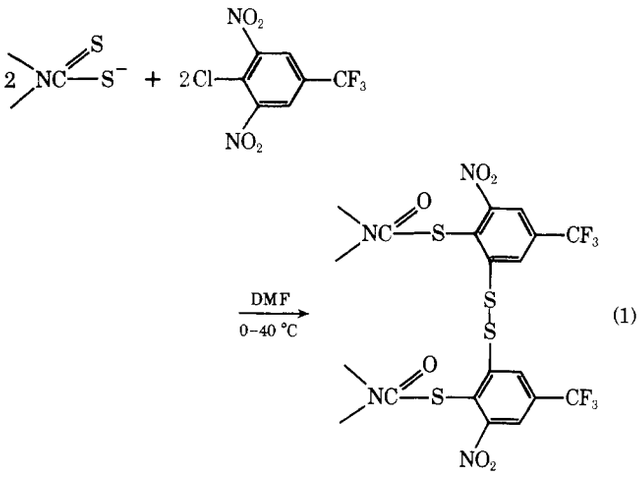
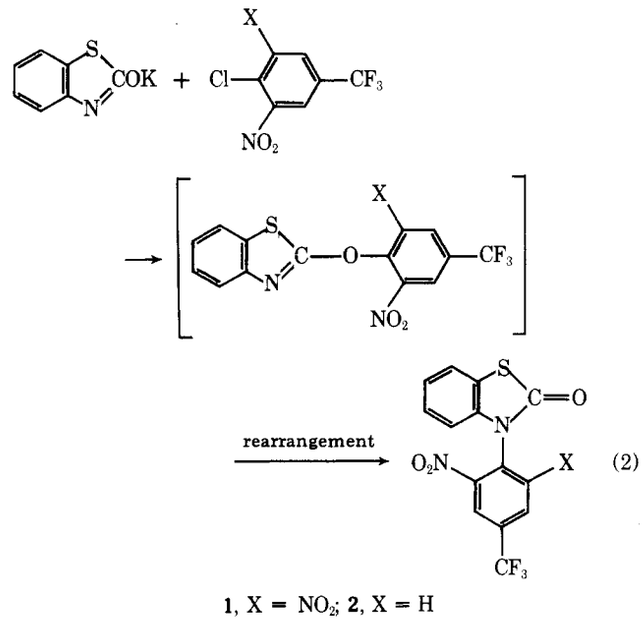
Depending on reaction temperatures the reaction of potassium 2-mercaptobenzimidazole with 4-chloro-3,5-dinitrobenzotrifluoride afforded either the expected 2-(2,6-dinitro-4-trifluoromethylphenylthio)benzimidazole (**3**) or the unexpected 4-nitrobenzimidazo[2,1-*b*]benzothiazole (**5**). Possible mechanism and supporting NMR, IR, and mass spectral data are discussed.

In a previous communication[2] we reported that the reaction of sodium or triethylamine salts of disubstituted dithiocarbamic acids with 4-chloro-3,5-dinitrobenzotrifluoride afforded the product as illustrated by reaction 1. Thus it appeared desirable to replace the above anion with other nucleophiles.



The reaction of potassium 2-benzothiazolol with 4-chloro-3,5-dinitrobenzotrifluoride or 4-chloro-3-nitrobenzotrifluoride in dimethylformamide at 90–100 °C afforded 3-(2,6-dinitro-4-trifluoromethylphenyl)-2-benzothiazolinone (**1**) and 3-(2-nitro-4-trifluoromethylphenyl)-2-benzothiazolinone (**2**),



1, X = NO_2; **2**, X = H

respectively (reaction 2). The NMR, IR, and mass spectral data for **1** and **2** were in agreement for the proposed structures.

Depending on reaction temperatures, the reaction of potassium 2-mercaptobenzimidazole with 4-chloro-3,5-dinitrobenzotrifluoride in dimethylformamide afforded either the