with the same solvent system and then with benzene (125 mL) gave unknown products (0.1 g).

Reaction of *N*-Nitroso-*N*-benzylformamide-d_1 with α-Naphthyllithium. An ethereal solution of α-naphthyllithium was prepared from the reaction of 1-bromonaphthalene (3.7 g, 18 mmol) with lithium metal (0.25 g) in anhydrous ether (80 mL) at reflux. The ethereal solution of α-naphthyllithium was added dropwise to a solution of *N*-nitroso-*N*-benzylformamide-d_1 (3.9 g, 24 mmol) in anhydrous ether (75 mL) at −20 to −30 °C. The resulting mixture was stirred at that temperature for an additional hour and then allowed to stir at ambient temperature overnight. Water (70 mL) was added and the organic layer was separated. The aqueous layer was extracted with ether once. The combined ethereal layer was quenched with ether once. The combined ethereal layer was quenched with acetic acid (10 mL). The ethereal layer was washed with water with water twice and then wiith saturated aqueous sodium bicarbonate. The ethereal layer was finally washed with water once and dried over Na_2SO_4. Evaporation of the ether under reduced pressure afforded an oil (4.9 g), which was deposited on a preparative column chromatography (silica gel, 32 g). Elution with hexanes (120 mL) gave a mixture (1.85 g) of naphthalene and benzyl bromide. GC–mass spectroscopic analysis of the mixture exhibited no deuterium incorporation in either naphthalene or benzyl bromide.

Reduction of 1 with $LiAlH_4$. A solution of *N*-nitroso-*N*-benzylformamide (0.983 g, 6 mmol) in anhydrous ether (25 mL) was added to a suspension of $LiAlH_4$ (86.7 mg, 2.3 mmol) in ether (25 mL) at −5 °C. The resulting mixture was stirred at −5 °C for an additional hour and then quenched by adding a solution of acetyl chloride (0.62 g, 7.9 mmol) in anhydrous ether (10 mL) at −5 °C. The mixture was stirred at ambient temperature overnight. The filtrate from removal of inorganic materials was concentrated in vacuo to leave an oil (0.632 g), which was chromatographed on silica gel (40 g). Elution with benzene (100 mL) afforded an oil (31 mg) whose NMR spectrum suggested it to be mainly a mixture of benzyl chloride and bibenzyl in a molar ratio of 5:2 by the NMR integration, along with impurities coming from silicon grease. Continued elution with benzene (100 mL) gave a mixture (0.173 g) of *N*-nitroso-*N*-benzylacetamide and benzyl formate in a molar ratio of 4:3 estimated by the NMR integration of the benzylic hydrogens. The identity of each of those components was established by comparison of the spectral data with those of authentic samples. The next fraction eluted with benzene (100 mL) yielded crude benzyl acetate (71 mg, 8%) whose IR and NMR spectra were in agreement with those of an authentic sample. Elution with a mixture of benzene and dichlormethane (1:1 to 1:2, v/v, 100 mL) gave an unknown product (50 mg). Elution with dichloromethane (100 mL) and ethanol (100 mL) afforded another unknown product (0.11 g).

Acknowledgment. We thank Dr. I. Shinkai of Merck, Sharpe and Dohme for the GC–MS data.

Registry No. 1-*d*, 85995-48-8; 1, 85995-49-9; benzylamine, 100-46-9; benzyl isocyanide, 10340-91-7; benzylformamide-*d*, 85995-47-7; phenylmagnesium bromide, 100-58-3; benzyl bromide, 100-39-0; diphenylmagnesium, 555-54-4; (*p*-methoxyphenyl)-magnesium bromide, 13139-86-1; phenyllithium, 591-51-5; phenyldiazomethane, 766-91-6; α-naphthyllithium, 14474-59-0.

Supplementary Material Available: Detailed experimental description of the following reactions : reaction of 1 with *two* equivalents of phenylmagnesium bromide, *one* and *two* equivalent(s) of (*p*-methoxyphenyl)magnesium bromide at −25 °C, with *two* equivalents of phenyllithium at −60 °C, with naphthyllithium. Control experiments of 1 with magnesium bromide and of (*p*-methoxyphenyl)magnesium bromide with benzyl bromide and the Meerwein–Pondorff–Verley reduction (12 pages). Ordering information is given on any current masthead page.

Diaryl Sulfide Cleavage by Sodium Sulfide in Dipolar Aprotic Solvents

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Received November 16, 1982

The interaction of sodium sulfide with diaryl sulfides that possess electron-withdrawing substituents (e.g., cyano or nitro) on the aromatic rings results in cleavage of the thioether and the formation of sodium aryl sulfides in dipolar aprotic solvents. Exchange reactions between these diaryl sulfides and sodium aryl sulfides are also observed in dipolar aprotic solvents. The cleavage and exchange reactions were employed to prepare mixed diaryl or alkyl aryl sulfides from symmetrical diaryl sulfides.

Although nucleophilic aromatic substitution reactions of aryl halides with sodium sulfide have been studied extensively,[1,2] reactions of sodium sulfide that involve leaving groups other than halides have not been examined in detail.[3] In this paper we describe a study of the interactions of sodium sulfide with diaryl sulfides that results in carbon–sulfur bond scission and the displacement of sodium aryl sulfides. We also describe the use of this reaction to carry out the conversion of symmetrical diaryl sulfides to alkyl aryl or mixed diaryl sulfides.

We uncovered the existence of a thioether cleavage process while examining the interaction of sodium sulfide with 4-chloronitrobenzene in a 1:1 molar ratio. We expected to obtain sodium 4-nitrophenyl sulfide, according to eq 1. However, when the reaction was monitored by

$$\mathrm{ArCl} + \mathrm{Na_2S} \rightarrow \mathrm{NaSAr} \quad (1)$$

high-pressure liquid chromatography, we were surprised to find that the formation of sodium 4-nitrophenyl sulfide was a complex process and involved the formation and subsequent cleavage of bis(4-nitrophenyl) sulfide (see Figure 1). Thus the reaction sequence for this system can be described more accurately by eq 2.

$$\mathrm{ArCl} + \mathrm{Na_2S} \rightarrow \mathrm{NaSAr} + \mathrm{ArCl} \rightarrow \mathrm{S(Ar)_2} + \mathrm{Na_2S} \rightarrow 2\mathrm{NaSAr} \quad (2)$$

It is suspected that for many activated aryl halides the formation of sodium aryl sulfides proceeds at least in part via the process shown in eq 2. The extent of diaryl sulfide formation would depend on the kinetics of formation of the diaryl sulfide from the reaction of the sodium aryl sulfide with the aryl halide, as compared with the rate of formation of the sodium aryl sulfide. Since the thioether cleavage reaction had not been described previously, it was

(1) For a general review of synthetic procedures to diaryl sulfides see: Oae, S., Ed. "Organic Chemistry of Sulfur"; Plenum Press: New York, 1977; p 231.
(2) Uhlenboeck, J. H. *Recl. Trav. Chim. Pays-Bas* **1961**, 80. Campbell, J. Robert, *J. Chem. Soc.* **1964**, 1830–33. Talen, H. W. *Recl Trav. Chim. Pays-Bas* **1928**, *47*, 782–95.
(3) Bunnett, J. F.; Bernasconi, Claude *J. Am. Chem. Soc.* **1965**, *87*, 5209–18.

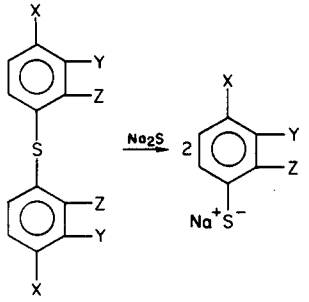
0022-3263/83/1948-2496$01.50/0

Table I. Experimental Details of Mixed Sulfide Syntheses

expt	compd[a]	synthesis route	purification method[b]	% yield	mp [bp] °C
1	8	7 + ethyl bromide	A	40	[123 (2.4 mm)]
2	9	12 + 4-chlorobenzonitrile	B	60	153-154
3	10	7 + methylene dibromide	C	75	152-153
4	14	12 + ethyl bromide	C	55	43-44
5	15[c]	13 + ethyl bromide	C	50	115-115.5

[a] Satisfactory analytical values (±0.3% for C, H, N, and S) were reported for 8-10 and 14. [b] Purification methods used were as follows: (A) distillation, (B) chromatography using methylene chloride/hexane (2:1), (C) recrystallization. [c] Lit.[12] mp 115-116.5 °C.

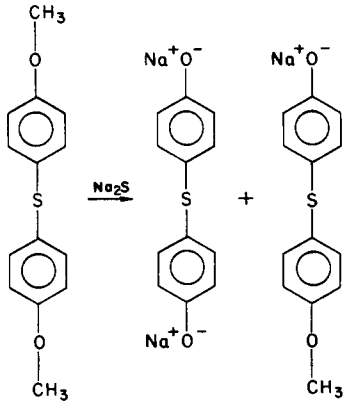
investigated further with the six diaryl sulfides indicated in eq 3.



(3)

1, X = NO_2; Y = Z = H
2, X = NO_2; Y = H; Z = NO_2
3, X = CN; Y = Z = H
4, X = H; Y = H; Z = CN
5, X = OCH_3; Y = Z = H
6, X = Y = Z = H

It was found that the activated compounds 1-4 undergo thioether cleavage in high yield to form the corresponding sodium aryl sulfides. The reactivity of the diaryl sulfides 1-4 to cleavage is 2 > 1 > 3 > 4. This is the same order of reactivity that is observed for the corresponding aryl halides during the preparation of the diaryl sulfides. Compound 5 underwent a demethylation reaction in preference to thiophenoxide displacement (eq 4).



(4)

5

The demethylation of substituted anisoles has been reported previously.[4] Compound 6 did not undergo reaction with sodium sulfide.

On the basis of these results the thiophenoxide displacement reaction appears to be limited to diaryl thioethers that possess strongly electron-withdrawing groups on the aromatic rings. This would be expected if the mechanism of cleavage involved nucleophilic aromatic substitution.[5] Thus the presence of two nitro groups

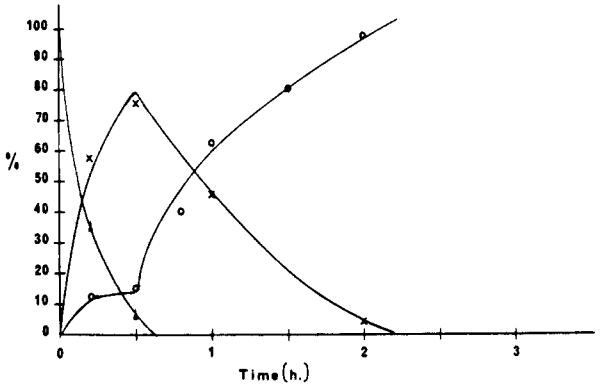
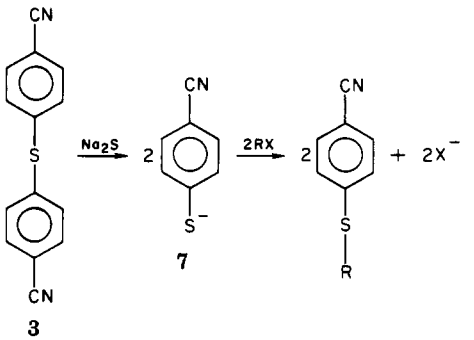


Figure 1. The reaction profile for the interaction of 4-chloronitrobenzene (▲) with sodium sulfide (in a 1:1.1 molar ratio) is shown. From the data it can be inferred that the thioether bis(4-nitrophenyl) sulfide (X) is formed in the process and is slowly consumed by sodium sulfide to yield sodium 4-nitrophenyl sulfide (O).

enhances the electrophilicity of the aromatic ring more than one nitro group, and a nitro group is more activating than a cyano group. The difference in reactivity between compounds 3 and 4 is likely a consequence of steric factors. During the synthesis of compounds 3 and 4 it was observed that in spite of the fact that the two thiophenoxides were formed at similar rates, the formation of 4 from sodium 2-cyanophenyl sulfide was substantially slower than the formation of 3 from sodium 4-cyanophenyl sulfide.

The reactivity of activated diaryl sulfides toward thioether cleavage allows for use of this process in the preparation of mixed-substituent aryl sulfides from symmetrical ones. This was accomplished by the initial cleavage of the diaryl sulfide followed by the addition of an aryl or alkyl halide to the reaction mixture. The process is illustrated in eq 5.



(5)

7

3

Derivatives were prepared from sodium 4-cyanophenyl sulfide as shown in Scheme I. Similar procedures were employed for compounds 1, 3, and 4 (see Table I). The sodium thiophenoxides derived from these cleavage reac-

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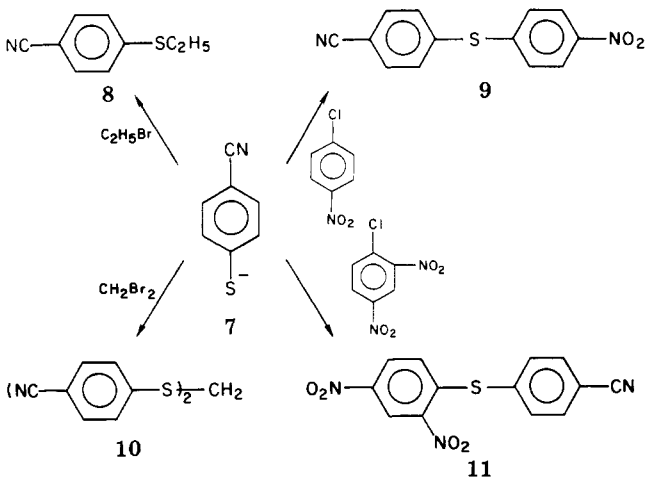
Scheme I



tions were very unreactive toward unactivated aryl halides such as chlorobenzene or *p*-dichlorobenzene even at reflux temperatures.

Finally it was found that the activated thioethers that undergo thiophenoxide displacement also undergo exchange (eq 6). The extent of exchange is governed by the

$$S(Ar) + NaSAr' \rightarrow S(Ar)(Ar') + NaSAr \quad (6)$$

stabilities of the thiophenoxides involved. For example, quantitative exchange was observed when bis(4-nitrophenyl) sulfide (**1**) was allowed to interact with sodium 4-cyanophenyl sulfide (a 1:3 reactant ratio was used). Furthermore, during this reaction the mixed thioether that was formed, 4-cyanophenyl 4-nitrophenyl sulfide, underwent further exchange to yield bis(4-cyanophenyl) sulfide (**3**) and sodium 4-nitrophenyl sulfide. However, when compound **3** was allowed to interact with sodium 4-nitrophenyl sulfide (a 1:3 reactant ratio was employed again), only 10% of exchange products were observed.

Experimental Section

Materials and Equipment. Dimethylformamide (Burdick and Jackson Laboratories, Inc.) was purified by distillation and stored over 3-Å molecular sieves. Anhydrous sodium sulfide (Alfa Products) was further dried by means of azeotropic distillation with toluene (calcium hydride was used as the drying agent in the Dean–Stark trap). 4-Chlorobenzonitrile (Aldrich) was purified by sublimation. The compounds 4-chloronitrobenzene, 2-chlorobenzonitrile, 2,4-dinitrochlorobenzene, diphenyl sulfide, and ethyl bromide (all obtained from Aldrich) were used as received. The derivative bis(4-methoxyphenyl) sulfide was prepared from the reaction of 4,4′-thiodiphenol (Aldrich) with dimethyl sulfate by the use of published methods.[6] The other thioethers were prepared by the reaction of sodium sulfide with the appropriate aryl halide. A general procedure is described in the next section.

High-pressure liquid chromatographic analysis was carried out by using a Waters Associates instrument on a C-18 reverse-phase column (Waters Associates). For typical HPLC conditions used in this study, see the Monitoring the Thioether Cleavage Process section.

General Procedure for Thioether Syntheses. The thioethers 1–4 described in this paper were prepared in dimethylformamide by using a 2:1 reactant ratio of aryl chloride to sodium sulfide. All manipulations were carried out in a nitrogen atmosphere to avoid the formation of disulfides. The preparation of bis(4-cyanophenyl) sulfide (**3**) is illustrative of the method employed. The compounds 4-chlorobenzonitrile (0.92 g, 0.0067 mol) and anhydrous sodium sulfide (0.27 g, 0.0035 mol) were combined with DMF (25 mL.) in a round-bottomed flask (50-mL capacity) fitted with a nitrogen inlet and a condenser, and the reaction mixture was allowed to reflux in a nitrogen atmosphere for 18 h. The mixture was allowed to cool, and ethyl bromide (1 mL) was added to ensure that all sulfides were inactivated. Then the mixture was poured into a large excess of water (250 mL) and the precipitate collected by filtration. The solid was washed with water several times and then dried in a vacuum oven at 100 °C. The solid was recrystallized from a 50% methanol/water mixture. The melting point was 135–136 °C.[7] The isolated yield was 65% (HPLC monitoring of this reaction indicated a quantitative yield). The thioethers **1**, **2**, and **4** were prepared in a similar manner and recrystallized from methanol/water (**1**) or from ethanol (**2**, **4**). The melting points were compared with literature values where possible and were as follows: **1**, 165 °C;[8] **2**, 195–196 °C;[9] **4**, 158–159 °C. The starting materials were pure by NMR and by microanalysis. Isolated yields were as follows: 60%, **1**; 45%, **2**; 65%, **4**.

Reaction of 4-Chloronitrobenzene with Sodium Sulfide Using a 1:1 Reactant Ratio. The compounds 4-chloronitrobenzene (0.3159 g, 0.002 005 mol), sodium sulfide (0.171 g, 0.002 19 mol), and biphenyl (0.0265 g, 0.000 172 mol) were combined with DMF (27.8 mL), and then the reaction mixture was heated rapidly to 120 °C by means of an oil bath. Samples were removed periodically with a syringe and added to a solution containing DMF and ethyl bromide. These were then examined by HPLC. The data are shown in Figure 1.

Monitoring the Thioether Cleavage Process. The cleavage reactions were monitored in the following way. The compounds sodium sulfide (0.1732 g, 0.001 759 mol), biphenyl (0.0266 g, 0.000 173 mol), and the appropriate diaryl sulfide (0.001 759 mol) were combined with DMF (25.0 mL) in a round-bottomed flask equipped with a condenser, a nitrogen inlet, and a syringe cap. The reaction mixture was placed in an oil bath maintained at 120 °C. The reaction was sampled periodically by means of a syringe. The sample was then rapidly added to a vial fitted with a syringe cap and containing ethyl bromide and DMF. It was found that the use of an acid such as acetic acid or hydrochloric acid as the quenching agent was not an effective workup procedure due to the partial oxidation of the organic hydrosulfides to disulfides during the quenching process. The reaction mixtures were then examined by means of HPLC [in general the solvent system employed was 50% by volume organic (composed of 90% acetonitrile and 10% methanol) and 50% water at a flow rate of 1.5 mL/min]. The amounts of diaryl sulfide and ethyl aryl sulfide were determined quantitatively by using a biphenyl internal reference. The rates of cleavage of the diaryl sulfides **1–4** were in the following order (percent diaryl sulfide remaining after 1 h of reaction in parentheses): **2** (0%) > **1** (14%) > **3** (26%) > **4** (83%). The rates of formation of compounds 1–4 from sodium sulfide and the aryl chloride were also measured (at a molar concentration of aryl halide to DMF of 0.152 mol/L at 120 °C) and were in the following order (percent diaryl sulfide formed after 2 h): **2** (99%) > **1** (70%) > **3** (47%) > **4** (10%). All of the experiments described above were quantitative (detection limit of the HPLC analysis method was 2% for side products). The interactions of the compounds **5** and **6** with sodium sulfide were also examined, but no diaryl sulfide cleavage was detected (i.e., less than 5% reaction).

Preparation of Aryl Alkyl and Mixed Diaryl Sulfides from Symmetrical Sulfides. The procedure for the preparation of sodium aryl sulfides for the synthesis of mixed diaryl sulfides from symmetrical ones was similar to the method described above except that no internal reference was employed, and the reaction scale involved the use of 2–5 g of sodium sulfide and the appropriate quantity of diaryl sulfide to obtain a 1:1 reactant ratio. Compounds **1–4** were employed in these reactions to yield, respectively, sodium 4-nitrophenyl sulfide, (**12**), sodium 2,4-dinitrophenyl sulfide (**13**), and sodium 4-cyanophenyl sulfide (**7**). The mixtures were stirred for 12–18 h at 130 °C prior to quenching with the alkyl or aryl halide. The reaction mixtures were examined by HPLC methods to ensure that the reaction was complete

(6) Shine, H. J.; Rahman, M.; Seeger, H.; Wu, G.-S. *J. Org. Chem.* **1967**, *32*, 1901.

(7) Ashley, J. N.; Barber, H. J.; Ewing, A. J.; Newbery, G.; Self, A. D. H. *J. Chem. Soc.* **1942**, 113.

(8) See ref 6, p 1908.

(9) Lukashevich, V. O.; Sergeeva, M. M. *Dokl. Akad. Nauk SSSR* **1949**, *67*, 1043.

(HPLC samples were quenched with ethyl bromide as above). The workup involved pouring the mixtures into an excess of water and collecting the solid products by filtration. The products were washed several times with water and then dried in a vacuum oven. Purification was effected by recrystallization or by liquid chromatography. The purity of the products was confirmed by HPLC, by ^{13}C NMR, and by elemental microanalysis (Galbraith Laboratories). Further characterization was obtained by field desorption mass spectrometry. Details of the purification, the percent isolated yields, and the melting points for the derivatives ethyl 4-cyanophenyl sulfide (**8**), ethyl 4-nitrophenyl sulfide (**14**), ethyl 2,4-dinitrophenyl sulfide (**15**),[10] bis[(4-cyanophenyl)thio]-methane (**10**), and 4-cyanophenyl 4-nitrophenyl sulfide (**9**) are given in Table I. The reactions of sodium 4-cyanophenyl sulfide (**7**) with *p*-dichlorobenzene or chlorobenzene was also examined, but it was found that even at reflux less than 10% of the sodium aryl sulfide underwent reaction with these aryl halides.

Thiophenoxide Exchange Reactions. A sample of 4-chlorobenzonitrile (1.00 g, 0.007 27 mol) was combined with sodium sulfide (0.57 g, 0.007 31 mol) and biphenyl (0.14 g, 0.000 908 mol) in dry DMF (20 mL). The reaction was allowed to proceed for 18 h at 130 °C. The mixture was sampled and examined by HPLC (ethyl bromide quench). Only sodium 4-cyanophenyl sulfide (**7**) was present. Then, bis(4-nitrophenyl) sulfide (**1**; 0.75 g, 0.002 71 mol) was added. Compound **1** was completely consumed within 1 h at 130 °C. It was replaced by the mixed thioether 4-cyanophenyl 4-nitrophenyl sulfide (**9**, 90% yield after 1 h). After 12 h compound **9** had also been consumed and replaced with bis-(4-cyanophenyl) sulfide (**3**; 92% yield based on the quantity of mixed thioether originally present).

The same procedure involving the initial formation of sodium 4-nitrophenyl sulfide (**12**) followed by addition of bis(4-cyanophenyl) sulfide (**3**) resulted in a small amount of exchange (less than 10% of compound **3** consumed over the course of 12 h).

(10) The synthesis of compound **11** from the interaction of sodium 4-cyanophenyl sulfide (**7**) with 2,4-dinitrochlorobenzene resulted in a product that contained the impurity bis[(4-cyanophenyl)thio]nitrobenzene as indicated by mass spectrometry. The impurity was less than 3% of the isolated product because no product other than compound **II** was indicated by HPLC. The structure of the bis-substituted impurity was not determined.

Acknowledgment. We thank M. M. Grade for the synthesis of several compounds used in this study and for the carrying out of some preliminary rate studies involving the synthesis of diaryl sulfides.

Registry No. **1**, 1223-31-0; **2**, 2253-67-0; **3**, 46836-99-1; **4**, 86047-00-9; **7**, 61628-44-2; **8**, 86047-01-0; **9**, 21969-10-8; **10**, 86047-02-1; **12**, 13113-79-6; **13**, 51256-42-9; **14**, 7205-60-9; **15**, 7343-55-7; C_2H_5Br, 74-96-4; CH_2Br_2, 74-95-3; Na_2S, 1313-82-2; 4-chlorobenzonitrile, 623-03-0; 1-chloro-4-nitrobenzene, 100-00-5; 2,4-dinitrobenzene, 97-00-7; 2-chlorobenzonitrile, 873-32-5.

Halogenated Ketenes. 38. Cycloaddition of α,β-Unsaturated Imines with Ketenes To Yield Both β- and δ-Lactams

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The cycloaddition of various types of α,β-unsaturated imines with diphenyl- and dichloroketenes yields both (2 + 2) and (4 + 2) cycloaddition products, i.e., β-lactams and δ-lactams, respectively. The cycloaddition products are dependent upon substitution in the imine and the ketene. The δ-lactams derived from dichloroketene are easily dehydrochlorinated to the corresponding 2-pyridones. All of the results are consistent with a two-step cycloaddition process involving a dipolar intermediate.

Several reports have appeared in the literature on the (2 + 2) cycloaddition of ketenes with imines to yield the biologically active β-lactams.[1-5] It has recently been established that these cycloadditions occur via a dipolar intermediate.[6] There are some scattered reports on the cycloaddition of α,β-unsaturated imines with ketenes, and some (4 + 2) cycloaddition products have been reported.[7,8] Since this reaction is occurring via a dipolar intermediate, α,β-unsaturated imines do, in fact, offer the possibility for ring closure to (4 + 2) cycloaddition products, and this could be a significant synthetic development for the preparation of δ-lactams and/or 2-pyridones. Therefore, this report describes a study of the reaction of diphenyl- and dichloroketenes with various α,β-unsaturated imines to determine the synthetic utility of this reaction for the preparation of β- and δ-lactams. During the course of this investigation, we learned of results on the cycloaddition of cyanoketenes with α,β-unsaturated imines.[9]

The cycloaddition of freshly distilled diphenylketene with α,β-unsaturated imines occurs readily in good yields at ambient temperatures to give the (2 + 2) cycloaddition products as shown in Scheme I. The reactions were complete within 1 h, and the solid products were easily isolated and purified by recrystallization. The structures of compounds **1a–k** are based primarily on the carbonyl band in the infrared at 1720–1742 cm^{-1}, proton and ^{13}C NMR, and elemental analysis. It is well established in the literature that the carbonyl absorption of β-lactams in the infrared occurs in the 1740 cm^{-1} range.[7,10] There was no evidence of any of the (4 + 2) cycloaddition products, the δ-lactams, in any of these cycloadditions. Some characteristic ^{13}C NMR data for compounds **1a–c,g** are shown in Table I.

The α,β-unsaturated imines were selected so that the steric requirement on both the N substituent and substitution on the carbon skelton would be varied. A wide

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