compounds were characterized by ^1H and ^{13}C NMR and mass spectral data.

17a. IR (neat, cm^{-1}): 3050, 2950, 1580, 1480, 1440, 1330, 1150, 1070, 1000, 910, 830, 680. ^1H NMR (200 MHz, $CDCl_3$): δ 7.45 (m, 4 H), 7.2 (m, 6 H), 4.3 (q, 2 H, J = 6.9 Hz), 3.05 (dd, 2 H, J = 5.8 and 12.1 Hz), 2.9 (dd, 2 H, J = 6.9 and 12.1 Hz), 2.3–2.09 (m, 2 H), 1.96 (t, 4 H, J = 7.5 Hz), 1.76–1.56 (m, 2 H). ^{13}C NMR (200 MHz, $CDCl_3$): δ 138.28, 129.97, 128.66, 126.50, 115.62, 77.00, 34.58, 32.77, 29.80. MS m/e (relative intensity): 468 [M^+, 20], 296 (20), 172 (20), 158 (20), 139 (40), 78 (100). HRMS m/e: M^+ calcd 468.0106, obsd 468.0113.

17b: IR (neat, cm^{-1}): 3050, 2950, 1580, 1480, 1440, 1330, 1150, 1070, 1000, 910, 830, 680. ^1H NMR (200 MHz, $CDCl_3$): δ 7.51 (m, 4 H), 7.25 (m, 6 H), 4.35 (q, 1 H, J = 6.8 Hz), 4.22 (q, 1 H, J = 6.9 Hz), 3.32–3.2 (dd, 1 H, J = 6.2 and 12.1 Hz), 3.1–2.9 (m, 3 H), 2.22–1.60 (m, 8 H). ^{13}C NMR (200 MHz, $CDCl_3$): δ 132.47, 130.06, 128.92, 126.70, 115.39, 79.04, 77.02, 35.85, 34.46, 32.98, 30.97, 29.98. MS m/e (relative intensity): 468 [M^+, 20], 296 (20), 172 (20), 158 (20), 139 (40), 78 (100). HRMS m/e: M^+ calcd 468.0106, obsd 468.0116.

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Registry No. 1, 821-41-0; 2, 821-09-0; 3, 3354-58-3; *cis*-4, 59981-81-6; *trans*-4, 24844-28-8; 5, 591-80-0; 6, 3675-31-8; 7, 4277-34-3; 8, 74912-33-7; 9, 3462-52-0; 10, 75526-73-7; 11, 65539-72-2; 12, 71098-92-5; 13 (isomer 1), 141508-53-4; 13 (isomer 2), 141508-54-5; 14, 65234-93-7; 15, 65291-16-9; 16a, 77552-08-0; 16b, 77552-07-9; 17a, 141553-80-2; 17b, 141553-81-3; 18a, 110840-63-6; 18b, 110902-54-0; DCN, 3029-30-9; PhSeSePh, 1666-13-3; *o*-cresol, 95-48-7; allyl bromide, 106-95-6; allyl *o*-tolyl ether, 936-72-1; cyclohexanone, 108-94-1; cyclohexanone morpholine enamine, 670-80-4; morpholine, 110-91-8; 2-allyl-1-cyclohexanone, 94-66-6; 3-bromocyclohexene, 1521-51-3; diethyl malonate, 105-53-3; diethyl (2-cyclohexenyl)malonate, 6305-63-1; ethyl 2-(2-cyclohexenyl)acetate, 21331-58-8; 1,5-cyclooctadiene, 111-78-4; 5,6-epoxycyclooctene, 637-90-1; 4-bromo-1-butene, 5162-44-7; ethyl formate, 109-94-4; 5-hydroxy-1,8-nonadiene, 94427-72-2; acetophenone, 98-86-2.

Supplementary Material Available: Experimental procedures for the preparation of starting materials 3, 4, 6, 7, 8, and 9 and spectral characterization of 3–4, 6–16, and 18 by IR, ^1H NMR, ^{13}C NMR, and mass spectra, Stern–Volmer plot for quenching of DCA and DCN by PhSeSePh (Figure 1), and double reciprocal plot of Φ_{disapp} vs PhSeSePh concentration [Φ^{-1} vs $[Q]^{-1}$] (Figure 2) (10 pages). Ordering information is given on any current masthead page.

The Synthesis of 11-Cyclopropyl-5,11-dihydro-4-(hydroxymethyl)-6*H*-dipyrido[3,2-*b*:2′,3′-*e*][1,4]diazepin-6-one, a Putative Metabolite of the HIV-1 Reverse Transcriptase Inhibitor Nevirapine

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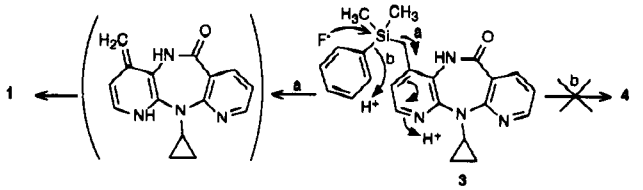
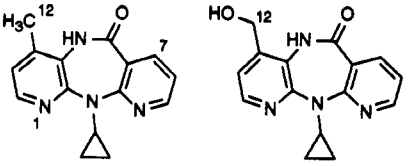
The dipyridodiazepinone 1 (nevirapine)[1,2] is a potent and selective noncompetitive inhibitor of HIV-1 reverse transcriptase and acts by a mechanism[3,4] distinct from that of nucleoside analogs such as AZT. It is currently undergoing clinical evaluation as a therapeutic agent against AIDS. We were interested in examining the metalation and subsequent functionalization of 1 in order to explore the chemistry of this novel tricyclic ring system, and one particular goal was the synthesis of the 12-hydroxy derivative 2 which, as a possible metabolite of 1, was required as a reference standard for metabolism studies. We expected that the 12-position of the diazepinone 1 would be susceptible to direct metalation,[5] and functionalization with a dimethylphenylsilyl group was chosen on the basis of its ready conversion to a hydroxyl group.[6]

Scheme I

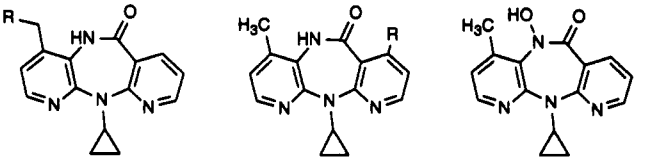


3



1 2

When the dianion of 1 was reacted with chlorodimethylphenylsilane either the 12-silyl derivative 3 or the 7-silyl derivative 5 could be obtained as the major product depending on the reaction conditions. The experimental results are presented in Table I and indicate that formation of the anion at the 7-position is kinetically favored whereas formation of the anion at the 12-position is thermodynamically favored. When all operations are carried out at <–65 °C quenching gives the 7-silyl derivative 5 in about 2:1 ratio over the 12-silyl derivative 3. At –35 °C, 5 is formed in only trace amounts, the major product being the 12-silyl derivative 3. Neither the use of a large excess of base (entries 2, 6) nor the addition of butyllithium with LDA (entry 3) or scale-up of the reaction (entry 4) noticeably affected the yields or product ratio.



3 R = $PhMe_2Si$-
4 R = FMe_2Si-
5 R = $PhMe_2Si$-
6 R = OH
7

(1) Merluzzi, V. J.; Hargrave, K. D.; Labadia, M.; Grozinger, K.; Skoog, M.; Wu, J. C.; Shih, C.-K.; Eckner, K.; Hattox, S.; Adams, J.; Rosenthal, A. S.; Faanes, R.; Eckner, R. J.; Koup, R. A.; Sullivan, J. L. *Science* **1990**, *250*, 1411.

(2) Hargrave, K. D.; Proudfoot, J. R.; Grozinger, K. G.; Cullen, E.; Kapadia, S. R.; Patel, U. R.; Fuchs, V. U.; Mauldin, S. C.; Vitous, J.; Behnke, M. L.; Klunder, J. M.; Pal, K.; Skiles, J. W.; McNeil, D. W.; Rose, J. M.; Chow, G. C.; Skoog, M. T.; Wu, J. C.; Schmidt, G.; Engel, W. W.; Eberlein, W. G.; Saboe, T. D.; Campbell, S. J.; Rosenthal, A. S.; Adams, J. *J. Med. Chem.* **1991**, *34*, 2231.

(3) Wu, J. C.; Warren, T. C.; Adams, J.; Proudfoot, J.; Skiles, J.; Raghavan, P.; Perry, C.; Potocki, I.; Farina, P. R.; Grob, P. *Biochemistry* **1991**, *30*, 2022.

(4) Cohen, K. A.; Hopkins, J.; Ingraham, R. H.; Pargellis, C.; Wu, J. C.; Palladino, D. E. H.; Kinkade, P.; Warren, T. C.; Rogers, S.; Adams, J.; Farina, P. R.; Grob, P. M. *J. Biol. Chem.* **1991**, *266*, 14670.

(5) Brandsma, L. *Preparative Polar Organometallic Chemistry 2*; Springer-Verlag: Berlin, 1990; p 136.

(6) (a) Fleming, I.; Henning, R.; Plaut, H. *J. Chem. Soc., Chem. Commun.* **1984**, 29. (b) Fleming, I.; Sanderson, P. E. J. *Tetrahedron Lett.* **1987**, *28*, 4229. (c) Colvin, E. W. *Silicon Reagents in Organic Synthesis*; Academic Press: San Diego, 1988; p 60.

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Table I. Reaction of the Dianion of 1 with Phenyldimethylchlorosilane

entry	1[a]	temp (°C)	base[a]	$PhMe_2SiCl$[a]	5[b] (%)	3[b] (%)	1[b] (%)
1	1.93	<-65	LDA (4.2)	2.7	34	13	39
2	1.95	<-65	LDA (10)	3.4	32	19	26
3	1.95	<-65	NaH (2.1), LDA (2.2), BuLi (2.0)	4.4	36	16	29
4	7.5	<-60	NaH (8.0), LDA (9.0), BuLi (8.8)	18.0	33	18	40
5	1.94	-35	LDA (4.2)	2.6	4	37	20
6	1.95	-35	LDA (10)	3.4	5	34	24

[a] Quantities are expressed in mmol. [b] Isolated yields.

Table II. Reaction of the Dianion of 1 with MoOPH

entry	1[a]	base[a]	MoOPH[a]	2 (%)
1	0.75	NaH (0.75)/LiHMDS (0.8)	0.75	0[b]
2	0.75	NaH (0.75)/KHMDS (0.8)	0.8	0[b]
3	19	NaH (19)/LDA (20)	20	12
4[c]	1.9	LDA (12)	1.8	35
5[d]	1.9	LDA (12)	2.2	47
6	1.9	LDA (9)	2.2	55

[a] Quantities are expressed in mmol. [b] Only starting material (1) was visible by TLC. [c] Reaction time 16 h, allowed to come to rt. [d] Reaction time 1 h at -30 °C.

The attempted oxidative desilylation of the 12-(phenyldimethylsilyl) compound 3 with tetrafluoroboric acid followed by peracetic acid[6c] gave none of the alcohol 2. Instead, only compound 1 was obtained. In retrospect, this result is not surprising for, while benzylic silyl groups have been oxidatively transformed into alcohols,[6c] protiodesilylation is also well documented in transformations of this type.[7,8] The treatment of 3 with tetrafluoroboric acid[6a] to form fluorosilane 4 can cause desilylation through protonation of the pyridine nitrogen (Scheme I, path a). The formation of 4 which is necessary for conversion to the alcohol[6a] requires a less likely protonation of the phenyl ring (Scheme I, path b). In contrast, oxidative desilylation of the 7-(phenyldimethylsilyl) derivative 5 successfully gave the 7-hydroxy compound 6, and we believe that this is the first such preparation of a hydroxypyridine.

Since the silylation/oxidative desilylation procedure did not give access to 2 we examined the direct oxidation of the dianion with oxodiperoxymolybdenum(pyridine)-hexamethylphosphoramide (MoOPH), and the results are presented in Table II. The alcohol 2 was only produced when LDA was used as the base, and the activating effect of excess LDA on the oxidizing ability of MoOPh is apparent from entries 5 and 6. This effect has been previously described.[9] The absence of the 7-hydroxy compound 6 from these reactions is consistent with the observation that, at the temperature necessary for the MoOPh oxidation (~-30 °C), the 12-anion is the major species in solution (Table I). The hydroxamic acid 7 was obtained as a side product in reaction 3 and was separately synthesized (10% yield) by reacting the monoanion of 1 with MoOPH.

Experimental Section

Melting points were determined on a Buchi 510 melting point apparatus and are uncorrected. ^1H NMR spectra were recorded on a Bruker WM-250 spectrometer operating at 250 MHz or a Bruker AC-270 spectrometer operating at 270 MHz with tetramethylsilane as the internal standard; J values are given in Hz. Mass spectra were recorded on a Finnegan 4023 GC/MS/DS spectrometer. Elemental analyses were determined by Midwest Laboratories, Indianapolis, IN. MoOPH was purchased from Aldrich Chemical Co. and used as received.

11-Cyclopropyl-5,11-dihydro-7-(dimethylphenylsilyl)-4-methyl-6*H*-dipyrido[3,2-*b*:2',3'-*e*][1,4]diazepin-6-one (5) and 11-Cyclopropyl-5,11-dihydro-4-[(dimethylphenylsilyl)-methyl]-6*H*-dipyrido[3,2-*b*:2',3'-*e*][1,4]diazepin-6-one (3). To a stirred suspension of 1 (2.0 g, 7.5 mmol) in dry THF (50 mL) under nitrogen was added NaH (50% in oil, 0.38 g, 8.0 mmol). The mixture was stirred at rt until hydrogen evolution ceased and was then cooled below -60 °C (internal temperature). LDA (1.5 M in cyclohexane, 6 mL, 9.0 mmol) was added dropwise followed, after 45 min, by butyllithium (1.6 M in hexane, 5.5 mL, 8.8 mmol). Stirring was continued for a further 15 min. Chlorodimethylphenylsilane (3.07 g, 18 mmol) was added via a syringe, and the mixture was stirred at -60 to -70 °C for 2 h and then allowed to come to rt. Water (3 mL) was added followed by ethyl acetate (100 mL), and the organic phase was worked up in the usual manner. Chromatography over silica gel (eluent 33% ethyl acetate/hexane to 100% ethyl acetate gradient) gave three fractions. Fraction 1 gave 5 (0.89 g, 2.45 mmol, 33%): mp 237-240 °C (ethyl acetate/hexane); ^1H NMR (DMSO-d_6) δ 9.88 (1 H, s, NH), 8.39 (1 H, d, J = 4.7, 9-H), 8.05 (1 H, d, J = 4.9, 2-H), 7.48-7.28 (5 H, m, Ph), 7.17 (1 H, d, J = 4.7, 8-H), 7.04 (1 H, d, J = 4.9, 3-H), 3.61 (1 H, m, 13-H), 2.11 (3 H, s, 4-Me), 0.86 (2 H, m, 14- or 15-H), 0.58 (3 H, s, SiMe), 0.54 (3 H, s, SiMe), 0.39-0.28 (2 H, m, 14- or 15-H); MS (CI) m/z 401 ($M + H^+$, 100). Anal. Calcd for $C_{23}H_{24}N_4OSi$: C, 68.97; H, 6.04; N, 13.99. Found: C, 68.93; H, 6.22; N, 13.83. Fraction 2 gave 3 (0.55 g, 1.38 mmol, 18%): mp 175-177 °C (ethyl acetate/hexane); ^1H NMR (DMSO-d_6) δ 9.91 (1 H, s, NH), 8.52 (1 H, dd, J = 2.0, 4.8, 7-H), 7.99 (1 H, d, J = 5.0, 2-H), 7.93 (1 H, dd, J = 2.0, 7.7, 9-H), 7.48-7.30 (5 H, m, Ph), 7.21 (1 H, dd, J = 4.8, 7.7, 8-H), 6.83 (1 H, d, J = 5.0, 3-H), 3.62 (1 H, m, 13-H), 3.04 (1 H, d, J = 13.1, 12-H), 2.30 (1 H, d, J = 13.1, 12-H), 0.88 (2 H, m, 14- or 15-H), 0.35 (2 H, m, 14- or 15-H), 0.15 (6 H, s, 2 × Si-Me); MS (CI) m/z 401 ($M + H^+$, 100). Anal. Calcd for $C_{23}H_{24}N_4OSi$: C, 68.97; H, 6.04; N, 13.99. Found: C, 69.14; H, 6.10; N, 14.09. Fraction 3 consisted of recovered 1 (0.79 g, 2.99 mmol, 40%).

11-Cyclopropyl-5,11-dihydro-7-hydroxy-4-methyl-6*H*-dipyrido[3,2-*b*:2',3'-*e*][1,4]diazepin-6-one (6). To a stirred solution of 5 (0.35 g, 0.88 mmol) in methylene chloride (2 mL) cooled to 0 °C was added tetrafluoroboric acid (0.19 g, 0.98 mmol). The mixture was stirred overnight at rt. The solvent was evaporated, and the crude fluorosilane was dissolved in 32% peracetic acid (2 mL) at 0 °C. Triethylamine (0.16 mL) was added, and the mixture was allowed to come to rt. After 4 h the mixture was neutralized with aqueous potassium hydroxide and extracted with ethyl acetate. The organic phase was dried, filtered, and evaporated. The residue crystallized from ethyl acetate/hexane to give 6 (0.16 g, 0.56 mmol, 63%); mp 224-226 °C; ^1H NMR (DMSO-d_6) δ (11.57, 1 H, br s, OH), 10.02 (1 H, br s, NH), 8.15 (1 H, br d, 9-H), 8.08 (1 H, d, J = 4.9, 2-H), 7.07 (1 H, d, J = 4.9, 3-H), 6.64 (1 H, br d, 8-H), 3.59 (1 H, m, 13-H), 2.33 (3 H, s, Me), 0.82 (2 H, m, 14- or 15-H), 0.30 (2 H, m, 14- or 15-H); MS (CI) m/z 283 ($M + H^+$, 100). Anal. Calcd for $C_{15}H_{14}N_4O_2$: C, 63.82; H, 5.00; N, 19.85. Found: C, 63.75; H, 5.00; N, 19.86.

11-Cyclopropyl-5,11-dihydro-5-hydroxy-4-methyl-6*H*-dipyrido[3,2-*b*:2',3'-*e*][1,4]diazepin-6-one (7). To a stirred suspension of 1 (0.50 g, 1.88 mmol) in dry THF was added NaH (50% in oil, 0.12 g, 2.5 mmol). The mixture was stirred at rt for 1 h and then cooled on ice. Powdered MoOPH (0.90 g, 2.0 mmol) was added in one portion. After warming to rt the mixture was stirred for a further 2 days. Water was added, and the solvent was then removed under reduced pressure. The residue was extracted with warm ethyl acetate, and the organic-soluble material was fractionated over silica gel to give the hydroxamic acid 7 (0.050 g, 0.18 mmol, 9.5%): mp 248-250 °C (ethyl acetate/

(7) Eaborn, C.; Jeffrey, J. C. J. Chem. Soc. 1957, 137.
(8) Tamao, K.; Ishida, N.; Kumada, M. J. Org. Chem. 1983, 48, 2120.
(9) Dean, B. D.; Truce, W. E. J. Org. Chem. 1981, 46, 3575.

ethanol); ^{1}H NMR (DMSO-d_6) δ 10.90 (1 H, s, OH), 8.49 (1 H, dd, J = 1.9, 4.8, 7-H), 8.17 (1 H, d, J = 4.9, 2-H), 8.03 (1 H, dd, J = 1.9, 7.6, 9-H), 7.24 (1 H, dd, J = 4.8, 7.6, 8-H), 7.13 (1 H, d, J = 4.9, 3-H), 3.63 (1 H, m, 13-H), 2.36 (3 H, s, 12-H), 0.91 (2 H, m, 14- or 15-H), 0.41 (2 H, m, 14- or 15-H); MS (CI) m/z 283 (M + H$^+$). Anal. Calcd for $C_{15}H_{14}N_4O_2 \cdot 0.25H_2O$: C, 62.83; H, 5.06; N, 19.54. Found: C, 63.07; H, 5.18; N, 19.45.

Optimized Procedure for Preparation of 2. To a stirred suspension of 1 (0.511 g, 1.92 mmol) in dry THF (20 mL) cooled on ice was added LDA (1.5 M in cyclohexane, 1.5 mL, 2.25 mmol) dropwise. After a clear solution was obtained, the mixture was cooled below −40 °C and a further quantity of LDA (4.5 mL, 6.75 mmol) was added over 1 min resulting in a deep red solution. After 5 min, MoOPH (0.95 g, 2.19 mmol) was added as a solid all at once and the reaction mixture was stirred at −30 to −40 °C for 75 min. Acetic acid (2 mL) and water (30 mL) were added, and the mixture was extracted with ethyl acetate (100 mL, 2 × 50 mL). The combined organic phase was dried over sodium sulfate, filtered, and evaporated. Fractionation of the residue over silica gel (eluent, chloroform/ethanol gradient) gave the alcohol 2 (0.299 g, 1.06 mmol, 55%): mp 243–245 °C (ethyl acetate/hexane); ^{1}H NMR (DMSO-d_6) δ 9.74 (1 H, br s, NH), 8.52 (1 H, dd, J = 1.9, 4.8, 7-H), 8.20 (1 H, d, J = 4.9, 2-H), 8.01 (1 H, dd, J = 1.9, 7.6, 9-H), 7.26 (1 H, d, J = 4.9, 3-H), 7.20 (1 H, dd, J = 4.8, 7.6, 8-H), 5.26 (1 H, br s, OH), 4.76 (1 H, d, J = 5.5, 12-H), 4.53 (1 H, d, J = 5.5, 12-H), 3.63 (1 H, m, 13-H), 0.88 (2 H, m, 14- or 15-H), 0.36 (2 H, m, 14- or 15-H); MS (CI), 283 (M + H$^+$, 100). Anal. Calcd for $C_{15}H_{14}N_4O_2$: C, 63.82; H, 5.00, N, 19.85. Found: C, 63.86; H, 5.15; N, 19.86.

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Registry No. 1, 129618-40-2; 2, 133627-24-4; 3, 141319-44-0; 5, 141319-45-1; 6, 133627-33-5; 7, 135794-73-9.

Arylthio Amidation, Etherification, and Lactonization of Alkenes Promoted by Oxidation of Bis(4-methoxyphenyl) Disulfide with Ammonium Peroxydisulfate

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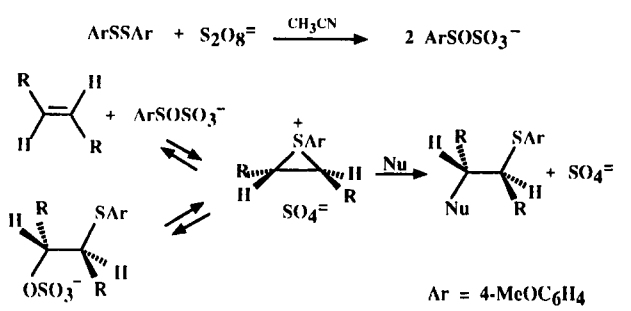
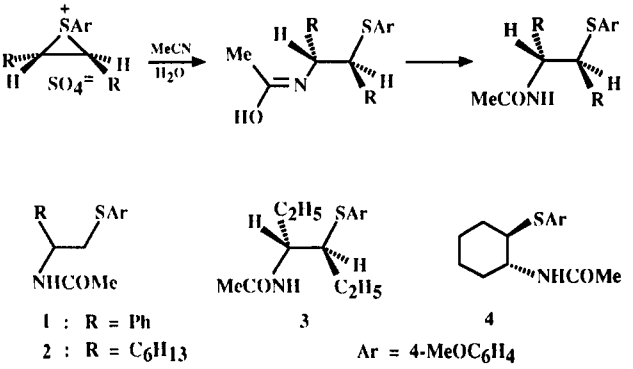
We have recently introduced the use of ammonium peroxydisulfate to effect the oxidation of diphenyl diselenide and to produce phenylselenenyl sulfate. Since the sulfate is a resonance-stabilized anion and a very poor nucleophile this reagent behaves as a very efficient electrophilic phenylselenenylating agent.[1,2] Ammonium peroxydisulfate also reacts with phenyl alkyl selenides to effect deselenenylation reactions from which phenylselenenyl sulfate is regenerated. Thus, several useful conversions of unsaturated compounds have been realized with a multistep, one-pot procedure which requires only catalytic amounts of diphenyl diselenide.[3]

We now report that ammonium peroxydisulfate also reacts with bis(4-methoxyphenyl) disulfide to produce an electrophilic sulfenylating agent which easily reacts with alkenes. Addition products are obtained when the reaction is carried out in the presence of either external or internal nucleophiles.

Scheme I



Scheme II



Results and Discussion

Preliminary experiments were carried out with several disulfides and unsaturated alcohols or acids in refluxing acetonitrile. With alkyl disulfides and with diphenyl disulfide no products of sulfur induced etherification or lactonization could be obtained; the starting disulfides were recovered, and ammonium peroxydisulfate effected the oxidation of the alkene. On the other hand, the same reactions carried out with bis(2,4-dimethoxyphenyl) disulfide gave exclusively rise to the oxidation of the disulfide to the corresponding thiosulfonate. Only with the bis(4-methoxyphenyl) disulfide the formation of the arylthio etherification or lactonization products could be observed, and therefore only this disulfide was employed to effect the reactions described below.

The reaction of ammonium peroxydisulfate with bis(4-methoxyphenyl) disulfide very likely proceeds in a way similar to that proposed for the corresponding reaction with diphenyl diselenide.[3] Thus, the interaction of the disulfide with the peroxydisulfate anions (either an electron transfer or an S_N2 process) is proposed to afford the (4-methoxyphenyl)sulfenyl sulfate. This is the reactive species which attacks the carbon carbon double bond to give a thiiranium intermediate which in the presence of a nucleophile affords the addition product (Scheme I).

Experiments, in which external oxygen nucleophiles were employed, were carried out by running the reactions in methanol or in acetonitrile and water. Unsatisfactory results were obtained in these cases, the products of arylthio methoxylation or hydroxylation of the various alkenes investigated being formed in poor yield. Better results were obtained in the reactions of arylthio amidation of alkenes which were carried out under experimental conditions identical to those already described for the phenylseleno amidation reactions,[4,5] i.e., in acetonitrile and

(1) Tiecco, M.; Testaferri, L.; Tingoli, M.; Chianelli, D.; Bartoli, D. *Tetrahedron Lett.* **1989**, *30*, 1417.
(2) Tiecco, M.; Testaferri, L.; Tingoli, M.; Bartoli, D.; Balducci, R. *J. Org. Chem.* **1990**, *55*, 429 and references cited therein.
(3) Tiecco, M.; Testaferri, L.; Tingoli, M.; Bartoli, D.; Marini, F. *J. Org. Chem.* **1991**, *56*, 5207 and references cited therein.

0022-3263/92/1957-4025$03.00/0