passed directly into a reaction flask containing a stirred toluene solution of the starting diazo compound kept at -78 °C. The operating pressure in the vacuum line was 0.02-0.05 Torr, but due to the N_2 evolution a rise in pressure to 1-2 Torr was observed. The stream of CS_2 vapor was, therefore, periodically interrupted (for ca. 1-2 min every 10 min) to allow reestablishment of the high vacuum. By the end of the reaction (after ca. 2 h for the experiments in this work) the mixture and the glass walls had turned black due to formation of polymeric carbon monosulfide. When the reaction was complete, the discharge was turned off and the stream of CS_2 vapor stopped. The vacuum line was filled with nitrogen, and the reaction mixture was allowed to warm to room temperature, followed by the workup procedures described below.

Di-*tert*-butylthioketene (5). A stirred solution of hydrazone **3**[12] (2.1 g, 14 mmol) in 70 mL of ether was treated with 8.0 g of nickel peroxide at room temperature Filtration and removal of the solvent in vacuo after 2 h gave crude **4**[10] as an orange oil which was dissolved in 130 mL of toluene and treated with CS generated from 5.0 mL (83 mmol) of CS_2 as described above. The black reaction mixture was filtered and distilled giving 0.6 g (30%) of **5**[5] as a violet oil (bp 69 °C (10 Torr)). All spectra (IR, ^1H and ^{13}C NMR, MS) were in agreement with the literature data.[5] The ^1H NMR spectrum showed a slight contamination (<5 mol%) of unreacted **4**.

(Tetrahydro-3,3,5,5-tetramethyl-4*H*-thiopyran-4-ylidene)methanethione (8). A stirred solution of hydrazone **6**[11] (2.3 g, 12 mmol) in 70 mL of ether was treated with 9.0 g of nickel peroxide. Filtration and evaporation of the solvent after 3 h gave **7**[11] as low-melting orange crystals which were dissolved in 170 mL of toluene and treated with CS generated from 15 mL (250 mmol) of CS_2 as described above. The resulting black reaction mixture was filtered and distilled to give 1.0 g (42%) of **8**[6] as a violet oil (bp 60-61 °C (0.05 Torr)). All spectra (IR, ^1H and ^{13}C NMR, MS) were in agreement with the literature data.[6]

Reduction of *N*-(Phenylsulfonyl)pyrroles with Sodium Cyanoborohydride in Trifluoroacetic Acid

Daniel M. Ketcha,* Kenneth P. Carpenter, and Qin Zhou

Department of Chemistry, Wright State University, Dayton, Ohio 45435

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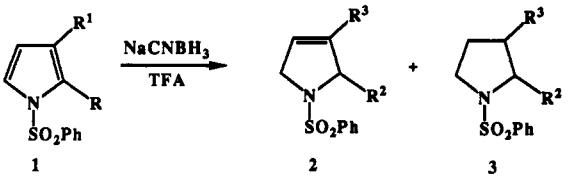
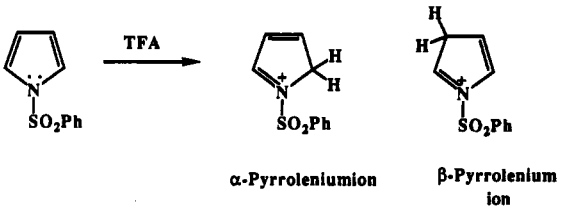
Due to the π-excessive character of the indole and pyrrole ring systems, these compounds are impervious to nucleophilic reducing species such as hydride reagents.[1,2] However, partial reduction of these heterocycles can be accomplished but only under acidic conditions via a sequence involving initial protonation (at C-3 for indoles and predominantly at C-2 for pyrroles), followed by reduction of the resultant iminium intermediates. Whereas indoles are susceptible to reduction by a variety of hydride sources under acidic conditions,[3] these methods have apparently not been successfully extended to pyrroles despite the fact that 3-pyrrolines (2,5-dihydropyrroles) represent useful synthetic intermediates.[4] To date, methods for the preparation of 3-pyrrolines by the partial reduction of pyrroles have been limited to the use of zinc in acidic media,[5] or in the case of some pyrrole-2-carboxylic acid derivatives, phosphonium iodide[6] or hypophosphorus acid[7] in hydroiodic acid. In light of our recent finding that sodium cyanoborohydride ($NaCNBH_3$) in trifluoroacetic acid (TFA)[8] represents a unique reducing system for the tandem reduction of the carbonyl group and the indole double bond of 2- and 3-acyl-1-(phenylsulfonyl)indoles to the corresponding alkyl-1-(phenylsulfonyl)indolines,[9] we have examined the analogous reaction upon *N*-(phenylsulfonyl)pyrroles and now report the first reduction of pyrroles by a hydride reducing agent (Scheme I).

Scheme I



Scheme II



The choice of the phenylsulfonyl protecting group in this study was predicated on the fact that 1-(phenylsulfonyl)pyrrole undergoes Friedel-Crafts acylation reactions with a remarkable degree of regiocontrol,[10] wherein acylation occurs at C-3 in the presence of aluminum chloride ($AlCl_3$) but predominantly at C-2 when catalyzed by boron trifluoride etherate ($BF_3 \cdot Et_2O$) or TFA.[11] Moreover, while the analogous Friedel-Crafts alkylation reactions upon the same substrate do not occur with the same degree of regioselectivity,[12] we have recently discovered that the selectively produced N-protected acylpyrroles can be reductively deoxygenated to the corresponding alkyl derivatives[13] without reduction of the pyrrole ring or loss of the protecting group using the borane-*tert*-butylamine complex ($^tBuNH_2$-BH_3)/$AlCl_3$ reducing system.[14] By these means then, a variety of 1-

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Table I. Reduction of 1-(Phenylsulfonyl)pyrroles

substrate	R	R^1	product	R^2	R^3	mp, °C	yield, %
1a	H	H	2a	H	H	120–121	75
1b	COMe	H	2b	Et	H	49–51.5	76
1c	COEt	H	2c	Pr	H	80–83	73
1d	H	COMe	2d	H	Et	oil	68
1e	H	COPh	2e	H	CH_2Ph	82–84	97
1f	Et	H	2b	Et	H	49–51.5	72[a]

[a] Product identical (IR, NMR) with that formed from 1b.

(phenylsulfonyl)pyrroles precursors could be readily prepared in anticipation of their conversion to selectively functionalized 3-pyrrolines.

As shown in Table I, when subjected to reduction with $NaCNBH_3$ in the presence of TFA, 1-(phenylsulfonyl)-pyrrole (**1a**)[12] is reduced to 1-(phenylsulfonyl)-3-pyrroline (**2a**),[15] while a sampling of 2- and 3-acyl-1-(phenylsulfonyl)pyrroles (**1b–e**)[11,13] undergoes tandem reduction of the carbonyl group and the pyrrole ring to provide the appropriately substituted alkyl-1-(phenylsulfonyl)-3-pyrrolines (**2b–e**) in good yield and without acid-catalyzed rearrangement of substituents.[16] Although for practical purposes this tandem reduction effectively precludes the need for prior reductive deoxygenation of the carbonyl groups, we have also carried out this reaction upon 2-ethyl-1-(phenylsulfonyl)pyrrole (**1f**),[12] thus demonstrating that an oxygen function adjacent to the ring is not a necessary requirement for reduction of the pyrrole ring.

As in the case of the reductions of pyrroles using zinc in hydrochloric acid,[5f] complete reduction to 1-(phenylsulfonyl)pyrrolidines (**3a–e**) can occur to some extent but usually less than 15%. Although these pyrrolidines were not characterized, they were observed (NMR and GC–MS) as contaminants of the crude reaction mixtures. As postulated for the reduction of N-unprotected pyrroles,[5f] it is believed that 3-pyrrolines (**2a–e**) are produced via initial (and predominant) protonation of the C-2 position[17] of the pyrrole ring followed by hydride reduction of the resultant α-pyrrolenium species (Scheme II). Formation of the fully reduced pyrrolidines (**3a–e**) can then be rationalized as resulting from an alternate pathway involving competing protonation at C-3. In this case, however, reduction of the β-pyrrolenium species presumably affords a 2-pyrroline, which as an enamine suffers further protonation and hydride reduction.

In light of this postulated mechanism, it is somewhat difficult to reconcile the success of the present method with the failure of other hydride or borane reagents to reduce the pyrrole ring under acidic conditions. Prior to this work, Maryanoff[18] and co-workers reported that in contrast to the facile reduction of indoles to the corresponding indolines with borane–tetrahydrofuran complex (BH_3–THF) in TFA, 1-methylpyrrole was recovered unchanged after treatment with the same reagents; conditions expectedly acidic enough to produce appreciable quantities of the protonated pyrrole. Moreover, in our hands, a number of reducing systems including BH_3–THF/TFA, $^tBuNH_2$–$BH_3/AlCl_3$, and $NaBH_4$ in the presence of TFA[3b,19] or $BF_3{\cdot}Et_2O$[20] all fail to reduce representative 1-(phenylsulfonyl)pyrroles. At present, it can only be surmised that the (acyloxy)boron species, generated in situ from $NaCNBH_3$ and TFA under the conditions of the reaction, represents a more efficient reductant than that derived from other reagents.[21] The nature of this (acyloxy)borohydride species is currently under investigation.

Experimental Section

Melting points were determined in open capillaries with an Electrothermal melting point apparatus and are uncorrected. Elemental analyses were performed by Midwest Microlab, Indianapolis, IN. Infrared spectra were recorded on a Perkin-Elmer 1330 instrument or using a Nicolet 5DX Fourier transform (FT) instrument. ^1H NMR spectra were obtained on a Varian EM 360 spectrometer and, in certain cases, ^1H and ^{13}C NMR were obtained on an IBM NR/100 FT NMR spectrometer at 100 MHz. Chemical shifts are reported in parts per million downfield from tetramethylsilane as an internal standard. Low-resolution mass spectra were determined using a Finnigan MAT INCOS 50 gas chromatograph–mass spectrometer.

Representative Procedure for the Reduction of 1-(Phenylsulfonyl)pyrroles Using $NaCNBH_3$/TFA. Preparation of 1-(Phenylsulfonyl)-3-pyrroline (2a). To magnetically stirred TFA (10 mL) at room temperature was added $NaCNBH_3$ (0.45 g, 7.25 mmol) slowly and in portions. (**Caution**: $NaCNBH_3$ reacts with TFA with vigorous evolution of hydrogen. Minor explosions are possible.) The resulting mixture was stirred for an additional 15 min, and **1a** (0.50 g, 2.41 mmol) was added slowly as a solid. After 1 h, additional $NaCNBH_3$ (0.45 g, 7.25 mmol) was added. The mixture was stirred overnight, quenched with water, and extracted with methylene chloride (CH_2Cl_2). The combined organic layers were washed with saturated sodium bicarbonate and water, dried with anhydrous sodium sulfate, filtered, and evaporated in vacuo. Chromatography on silica gel using hexanes–CH_2Cl_2 provided an oil (0.45 g), which slowly crystallized in vacuo. Recrystallization from methanol provided pure **2a** (0.38 g, 75%): mp 120–121 °C (lit.[15] mp 116–117 °C); IR (KBr) 3130, 2920, 1450, 1370, 1200, 1180, 1055, 765, 740, 690, 600, 620, 570 cm^{-1}; ^1H NMR ($CDCl_3$) δ 8.0–74 (m, 5 H), 5.6 (s, 2 H), 4.1 (s, 4 H); ^{13}C NMR ($CDCl_3$) δ 137.0, 132.6, 129.0, 127.2, 125.3, 54.7; mass spectrum, m/z 209 (M^+), 208, 141, 117, 104, 91, 77, 68 (100). Anal. Calcd for $C_{10}H_{11}NO_2S$: C, 57.39; H, 5.30; N, 6.70. Found: C, 57.54; H, 5.40; N, 6.70.

2-Ethyl-1-(phenylsulfonyl)-3-pyrroline (2b). The same procedure as described above but with **1b** gave **2b** (76%): mp 49–51.5 °C; IR (KBr) 3320, 2970, 2880, 1450, 1340, 1165, 1095, 760, 750, 690, 630, 585 cm^{-1}; ^1H NMR ($CDCl_3$) δ 8.3–7.4 (m, 5 H), 5.6 (s, 2 H), 4.5 (m, 1 H), 4.1 (s, 2 H), 1.8 (m, 2 H), 0.8 (t, 3 H, J = 7 Hz); ^{13}C NMR ($CDCl_3$) δ 137.8, 132.39, 129.2, 128.9, 127.1, 124.7, 68.1, 55.6, 28.5, 8.3; mass spectrum, m/z 237 (M^+), 236, 210, 209, 208 (100), 142, 141, 125, 77. Anal. Calcd for $C_{12}H_{15}NO_2S$: C, 60.73; H, 6.37; N, 5.90. Found: C, 60.72; H, 6.40; N, 6.01.

2-Propyl-1-(phenylsulfonyl)-3-pyrroline (2c). The same procedure as described above but with **1c** gave **2c** (73%): mp 78–83 °C; IR (KBr) 2950, 2880, 1445, 1335, 1155, 1090, 1055, 795,

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706, 605 cm^{-1}; ^{1}H NMR ($CDCl_3$) δ 8.7–7.4 (m, 5 H), 5.6 (s, 2 H), 4.5 (m, 1 H), 4.1 (s, 2 H), 2.5–1.3 (m, 4 H), 0.8 (t, 3 H, J = 7 Hz); mass spectrum, m/z 251 (M^+), 250, 208, 141, 125, 108, 91, 77 (100). Anal. Calcd for $C_{13}H_{17}NO_2S$: C, 62.12; H, 6.82; N, 5.57. Found: C, 62.10; H, 7.03; N, 5.63.

3-Ethyl-1-(phenylsulfonyl)-3-pyrroline (2d). The same procedure as described above but with **1d** gave **2d** (68%) as an oil: IR (KBr) 3040, 2970, 2880, 1460, 1370, 1170, 1120, 720, 600, 570 cm^{-1}; ^{1}H NMR ($CDCl_3$) δ 8.0–7.3 (m, 5 H), 5.3 (s, 1 H), 4.1 (s, 4 H), 1.8 (q, 2 H, J = 7 Hz), 0.9 (t, 3 H, J = 7 Hz); mass spectrum, m/z 237 (M^+), 236, 208, 141, 125, 96, 77 (100), 68. Anal. Calcd for $C_{12}H_{15}NO_2S$: C, 60.73; H, 6.37; N, 5.90. Found: C, 60.77; H, 6.40; N, 6.00.

3-Benzyl-1-(phenylsulfonyl)-3-pyrroline (2e). The same procedure as described above but with **1e** gave **2e** (97%): mp 82–84 °C; IR (KBr) 3080, 3050, 2970, 2930, 2880, 1615, 1460, 1340, 1165, 1095, 760, 750, 690, 630, 585 cm^{-1}; ^{1}H NMR ($CDCl_3$) δ 8.0–6.9 (m, 9 H), 5.2 (s, 1 H), 4.0 (s, 4 H), 3.2 (s, 2 H); mass spectrum, m/z 299 (M^+, 100), 208, 158, 141, 131, 115, 91, 77. Anal. Calcd for $C_{17}H_{17}NO_2S$: C, 68.20; H, 5.72; N, 4.68. Found: C, 68.00; H, 5.77; N, 4.61.

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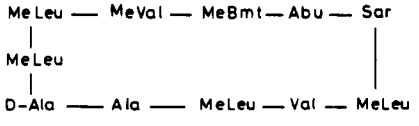
A Simple Route to (2*R*,3*R*,5*E*)-2-Hydroxy-3-methyl-5-heptenal: A Key Intermediate for MeBmt†

A. V. Rama Rao,* M. K. Gurjar, D. S. Bose, and R. Revathi Devi

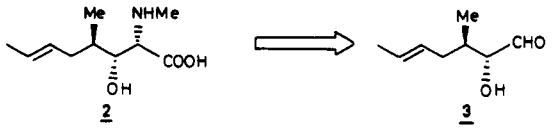
Indian Institute of Chemical Technology, Hyderabad 500 007, India

Received March 21, 1990

The efficacy of any synthetic protocol to the potent immunosuppressive agent, cyclosporine[1] (1), would largely depend on how efficiently large quantities of the unnatural β-hydroxy-α-amino acid, (4*R*)-4-[(*E*)-2-butenyl]-4,*N*-dimethyl-L-threonine (MeBmt) (2), present in the molecular structure of 1, are made available. Many synthetic routes to 2 have been reported.[2] The synthesis of 2 reported by Wenger,[2g] involving the highly stereocontrolled transformation of the key intermediate 3 into 2, is particularly interesting. However, the synthesis of 3 from diethyl (+)-tartrate required 18 steps including protecting–deprotecting of functional groups. Consequently we felt that an alternative but simple approach toward 3 could be derived, based on our[3a] findings that Sharpless epoxidation[3b] of 1-(benzyloxy)-3-buten-2-ol proceeds with a degree of efficiency.



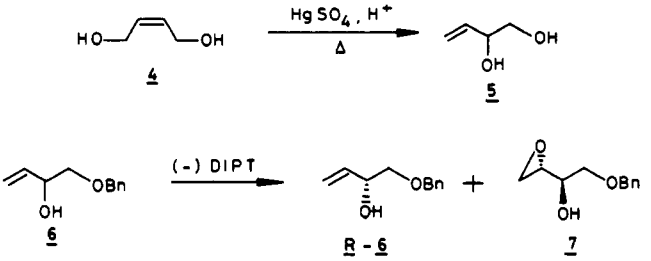
Cyclosporine 1



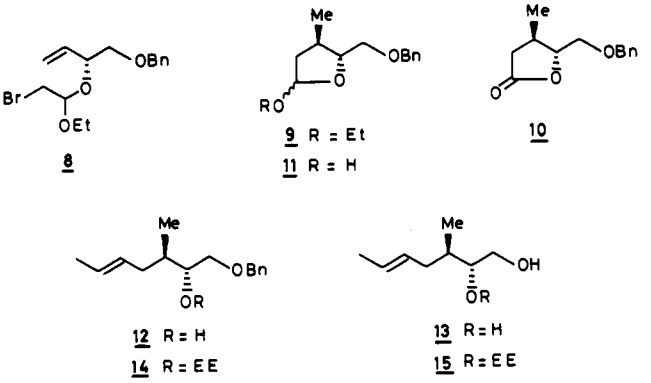
2 3

The starting material (5) was earlier prepared[3a] in our laboratory by one-step isomerization of *cis*-butene-1,4-diol (4) in the presence of catalytic amounts of mercuric sulfate and sulfuric acid in refluxing water for 1.5 h (Scheme I). Interestingly, now we observed that this isomerization could be more conveniently and rapidly effected in a microwave oven[4] in just 3 min (temperature of reaction mixture was ~50 °C), providing 5 in 60–70% yield. How the rate of the above rearrangement is enhanced by microwave irradiation could not reasoned with any proper explanation. However, we believe that due to high dielectric constant, water absorbs high microwave energy and being one of the reactants taking part in the reaction, concomitantly transfer this energy to the transition state to effect further reaction to occur rapidly.[5]

Scheme I



Scheme II



The kinetic resolution of the derived benzyl ether 6 (70%) under Sharpless condition[3,6] with (–)-diisopropyl tartrate (DIPT) as a chiral auxiliary led to the isolation of (*R*)-1-(benzyloxy)-3-buten-2-ol (*R*-6) (75% of theoretical yield, 95% ee)[3b] and (2*S*,3*S*)-1-(benzyloxy)-3,4-epoxybuten-2-ol (7).

Stork[7] and Ueno[8] have demonstrated that mixed bromo acetals derived from the allylic alcohols undergo stereoselective radical cyclization to form the *trans*-tetrahydrofuran derivative. Thus, compound *R*-6 was converted into the bromo acetal derivative 8 by the reaction with

† IICT Communication No. 2609.

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