(J = 2.7 Hz) between H-1 and H-4 also requires the geometry at C-1 to be the same as that in the saframycins. Although we were unable to obtain new samples of renieramycins A–D (**16–19**), we are now convinced that the stereochemistry of *all* renieramycins is the same as the stereochemistry of saframycin C, which was determined by X-ray analysis.[2c] There is good reason to believe that during the structural elucidation of renieramycins A–D[1b] the crucial NOEDS experiment, in which irradiation of H-14 resulted in enhancement of H-1, was performed with too high a power level so that both H-14 and H-22 were irradiated.[4] Professor T. Fukuyama has independently reached the same conclusion while studying the synthesis of saframycins[5] and renieramycins.[6]

The structure of renieramycin F (**15**) was deduced by comparison of spectra data with those of renieramycins E (**14**) and B (**17**).[1b] High-resolution mass measurement of the $(M + H - H_2O)^+$ peak at m/z 579.2285 and the presence of the $(M + H)^+$ peak at m/z 597 suggested a molecular formula of $C_{31}H_{36}N_2O_{10}$. The ^1H NMR spectrum contained a methoxyl signal at δ 3.53 (s, 3 H) and a signal at δ 3.76 (s, 1 H) that was assigned to an H-14 proton that is orthogonal to the H-13 proton. These data, together with the almost identical chemical shifts and coupling constants for the remaining signals, allowed the structure of renieramycin F (**15**) to be proposed.

During the entire period that this research was being conducted, renieramycins E (**14**) and F (**15**) were observed to be decomposing. The decomposition was fastest in chloroform solutions exposed to the air and during column chromatography and was significantly slower during HPLC and when the compounds were stored as dry powders under nitrogen. Among the observed degradation products were renierone (**1**) and mimosamycin (**2**), which together suggest that renieramycins E and F undergo an oxidative cleavage reaction. Considering that the ^1H NMR spectrum of the fresh crude extract of *Reniera* sp. appeared to be relatively clean and contained only those peaks later attributed to renieramycins E (**14**) and F (**15**) and contained no signals below δ 6.0, one must entertain the possibility that the "monomeric" products isolated previously are all artifacts of the isolation and chromatographic procedures employed.

Experimental Section

Collection and Isolation Procedures. The blue sponge *Reniera* sp. (59.1 g dry weight) was collected in shallow water (−2 m) at a small marine lake on Urukthapel Island, Palau, Western Caroline Islands. The sponge was frozen, and after <2 weeks, it was twice extracted for 2 days with methanol (2 × 1 L). The extracts were evaporated, and the aqueous residue (ca. 400 mL) was extracted with ethyl acetate (3 × 200 mL). The combined organic extracts were dried over sodium sulfate, and the solvent was evaporated to obtain a black tar-like oil (400 mg). Half of the oil was chromatographed on silica gel using a solvent gradient from hexane to ethyl acetate. The more polar fractions were combined, and the solvent was evaporated to yield a greenish-yellow powder (23 mg) that was separated by reversed-phase HPLC on C-18 Partisil using 9:1 methanol–water as eluant to obtain renierone (**1**, 3.5 mg, 0.006% dry weight), mimosamycin (**2**, 2.1 mg, 0.0035% dry weight), and renieramycin E (**14**, 5.0 mg, 0.0085% dry weight). The second portion of the black oil was chromatographed on Sephadex LH-20 using methanol as eluant. Those fractions that gave ^1H NMR spectra containing diagnostic methoxyl signals were combined to yield a green powder (25 mg) that was again subjected to reversed-phase HPLC separation to obtain renierone (**1**, 2.0 mg, 0.0035% dry weight) and a mixture of renieramycins (18.5 mg). The mixture was separated by HPLC on silica gel using 4:1 ethyl acetate–hexane as eluant to obtain renieramycin E (**14**, 10.8 mg, 0.018% dry weight) and renieramycin F (**15**, 4.5 mg, 0.008% dry weight).

Renieramycin E (14): amorphous yellow powder, unstable; UV (MeOH) 266 nm (ϵ 17 000); IR ($CHCl_3$) 3390, 1700, 1655, 1615 cm^{-1}; ^1H NMR ($CDCl_3$), see Table I; ^1H NMR (acetone-d_6) δ 1.35 (ddd, 1 H, J = 16.5, 11.2, 2.7 Hz), 1.56 (br s, 3 H), 1.74 (dq, 3 H, J = 7.2, 1.4 Hz), 1.85 (s, 3 H), 1.87 (s, 3 H), 2.27 (s, 3 H), 2.32 (d, 1 H, J = 21 Hz), 2.66 (dd, 1 H, J = 21, 7.2 Hz), 2.72 (dd, 1 H, J = 16.5, 2.5 Hz), 3.26 (m, 2 H), 3.89 (br d, 1 H, J = 1.8 Hz), 3.94 (s, 3 H), 3.99 (s, 3 H), 4.25 (dd, 1 H, J = 11.2, 1.8 Hz), 4.35 (d, OH, J = 6.5 Hz), 4.42 (m, 1 H), 4.45 (dd, 1 H, J = 11.2, 2.7 Hz), 4.53 (dd, 1 H, J = 6.5, 1.4 Hz), 5.99 (qq, 1 H, J = 7.2, 1.4 Hz); FABMS (m/z) 567 (2, M + H), 551 (44, M + 3H − H_2O), 549 (100, M + H − H_2O); HRMS m/z 549.2247, $C_{30}H_{33}N_2O_8$ requires 549.2237.

Renieramycin F (15): amorphous yellow powder, unstable; UV (MeOH) 265 nm (ϵ 12 000); IR ($CHCl_3$) 3300, 1710, 1660, 1615 cm^{-1}; ^1H NMR ($CDCl_3$), see Table I; ^1H NMR (acetone-d_6) δ 1.24 (ddd, 1 H, J = 17, 11.5, 2.5 Hz), 1.56 (br s, 3 H), 1.75 (dq, 3 H, J = 7.2, 1.4 Hz), 1.83 (s, 3 H), 1.90 (s, 3 H), 2.43 (s, 3 H), 2.68 (dd, 1 H, J = 17, 2.5 Hz), 3.20 (dt, 1 H, J = 11.5, 2.5 Hz), 3.31 (br d, 1 H, J = 2.2 Hz), 3.50 (s, 3 H), 3.87 (s, 1 H), 3.95 (s, 3 H), 3.97 (m, 1 H), 3.98 (s, 3 H), 4.25 (dd, 1 H, J = 11.2, 3 Hz), 4.31 (dd, 1 H, J = 11.2, 2.5 Hz), 4.40 (d, OH, J = 10.3 Hz), 4.42 (m, 1 H), 4.71 (dd, 1 H, J = 10.3, 2.5 Hz), 5.97 (qq, 1 H, J = 7.2, 1.4 Hz); FABMS (m/z) 597 (4, M + H), 581 (100, M + 3H − H_2O), 579 (52, M + H − H_2O); HRMS m/z 579.2285, $C_{31}H_{35}N_2O_9$ requires 579.2342.

Acknowledgment. We thank the Government and people of the Republic of Palau for facilitating the field research. This research was supported by a grant from the National Institutes of Health (CA49084-15) and a student Fellowship from Smith, Kline and French.

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Synthesis of 1,2,5-Thiadiazolidin-3-one 1,1-Dioxides: X-ray Structure Determination of 4,4-Diphenyl-1,2,5-thiadiazolidin-3-one 1,1-Dioxide

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Hydantoins, 1,3-imidazolidine-2,4-diones (**1**), are well known for their anticonvulsant activity.[1,2] In particular, 5,5-diphenylhydantoin (DPH, **1**, R = C_6H_5), is widely prescribed for control of generalized tonic–clonic (grand mal) epileptic seizures. The exact nature of the molecular mechanism is not known, but one theory relates activity to the hydrogen bonding ability of the drug to the receptor site (e.g. adenine of nucleic acids).[3]

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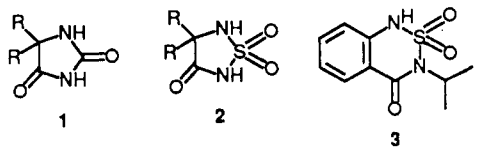
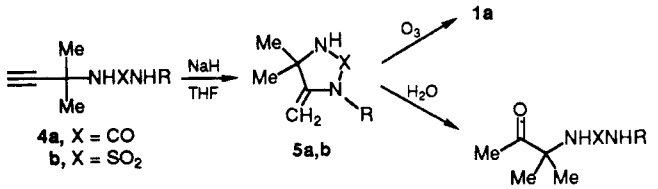
We have been interested in preparing 1,2,5-thiadiazolidin-3-one 1,1-dioxides (**2**) for both their potential anticonvulsant activity and because of their structural relationship to known herbicides (e.g. bentazon, **3**).[4,5] While a number of thiadiazolidine derivatives have been reported,[6] to our knowledge, only two accounts of the 1,2,5-thiadiazolidin-3-one 1,1-dioxide system have been reported.[7,8]



1 **2** **3**

In earlier work we showed that propargylic ureas (**4a**) could be cyclized to methylene imidazolidinones (**5a**), which upon ozonolysis gave hydantoins (**1a**).[9] However, similar attempts with propargylsulfamides (**4b**) were not successful owing to ease of hydrolysis of the thiadiazolidine intermediates (**5b** to **6b**) and because of other subsequent complex rearrangements.[9,10]



4a, X = CO
b, X = SO_2

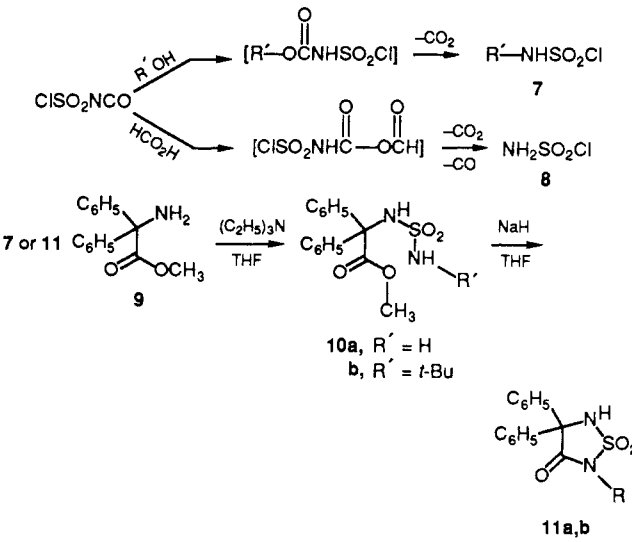
5a,b

6b

We now report an alternative route to thiadiazolidin-3-ones that involves treatment of 1,1-disubstituted glycine esters (**9**) with sulfamoyl chlorides (**7**, **8**). The intermediate sulfamides (**10**) are then cyclized to the 1,2,5-thiadiazolidin-3-one 1,1-dioxides (**11**) with sodium hydride in THF.

This reaction sequence thus provides access to 1,2,5-thiadiazolidin-3-one 1,1-dioxides; the success of which depends only upon availability of amino acid ester precursors (see the Experimental Section) and sulfamoyl chlorides prepared as shown above.

4,4-Diphenyl-1,2,5-thiadiazolidin-3-one 1,1-dioxide crystallizes in the monoclinic space group $C2/c$. As with diphenylhydantoin,[11,12] the two phenyl groups are approximately perpendicular to each other (88.32 (7)°). However, while the hydantoin ring is planar the thiadiazolidine ring is puckered. The N1, N2, C1, and C2 atoms are essentially planar but the S1 atom is 0.28 Å out of this plane, undoubtedly the result of the much longer N1–S1 (1.620 and 1.648 Å) bonds compared to the N–C bonds in DPH of 1.343 and 1.406 Å. The structure also indicates



7

8

9

10a, R′ = H
b, R′ = *t*-Bu

11a,b

dimeric intermolecular N–H···O hydrogen bonding between the N2 hydrogen and O3 of one with the same on an adjacent molecule (2.862 Å).

Experimental Section

1,1-Diphenylglycine,[13] 1,1-diphenylglycine methyl ester,[13] and (methoxycarbonyl)sulfamoyl chloride[14] were prepared according to the literature in 90, 45, and 96% yields, respectively.

***N*-[1,1-Diphenyl-1-(methoxycarbonyl)methyl]sulfamide (10a).** A 250-mL, three-necked, round-bottomed flask fitted with a reflux condenser, dropping funnel, nitrogen atmosphere, and thermometer was charged with 2.83 g (20.0 mmol) of chlorosulfonyl isocyanate. The temperature was lowered to 5 °C and 0.92 g (20.0 mmol) of anhydrous formic acid was added dropwise. The mixture was stirred at room temperature until gas evolution ceased. To the resulting sulfamoyl chloride mixture[15] was added dropwise a solution of 4.82 g (20.0 mmol) of diphenylglycine methyl ester and 2.01 g (20.0 mmol) of triethylamine in 75 mL of THF. The mixture was stirred overnight at room temperature, filtered and the solid washed with chloroform. The combined organics were washed with water and dried ($MgSO_4$). Removal of the solvent in vacuo and recrystallization from hexane–chloroform gave 2.65 g (41%) of **10a**. An analytical sample was prepared by a second recrystallization: mp 141.5–142 °C; ^{1}H NMR ($CDCl_3$) δ 3.68 (s, 2 H), 3.75 (s, 3 H), 6.35 (s, 1 H), 7.45 (m, 10 H); ^{13}C NMR (acetone-d_6) δ 53.44, 72.17, 128.42, 128.62, 129.79, 140.39, 172.77. Anal. Calcd for $C_{15}H_{16}N_2O_4S$: C, 56.23; H, 5.04; N, 8.75. Found: C, 55.83; H, 4.95; N, 8.68.

4,4-Diphenyl-1,2,5-thiadiazolidin-3-one 1,1-Dioxide (11a). To a 250-mL, three-necked, round-bottomed flask containing a stirred mixture of 0.45 g (18.8 mmol) of NaH and 10 mL of THF was added dropwise under nitrogen a solution of 1.10 g (3.43 mmol) of **10a** in 100 mL of THF. The mixture was stirred at reflux for 1 h and cooled to room temperature, and a small amount of water was added. The mixture was acidified with 10% HCl and extracted with ether. The organic extracts were dried ($MgSO_4$), and the solvent was removed in vacuo to give a semisolid which crystallized on standing. The product was recrystallized twice from hexane–chloroform to give 0.78 g (79%) of **11a**: mp 233–234 °C (lit.[7,12] mp 229 °C); ^{1}H NMR ($CDCl_3$) δ 7.1–7.8 (m, 10 H); ^{13}C NMR (acetone-d_6) δ 75.81, 128.16, 128.81, 129.01, 139.67, 170.72. Anal. Calcd for $C_{14}H_{12}N_2O_3S$: C, 58.31; H, 4.20; N, 9.72. Found: C, 58.08; H, 4.10; N, 9.69.

***N*-*tert*-Butyl-*N*′-[1,1-diphenyl-1-(methoxycarbonyl)methyl]sulfamide (10b).** A 250-mL, three-necked, round-bottomed flask equipped with a reflux condenser and dropping funnel and under a nitrogen atmosphere was charged with 1.56 g (21.0

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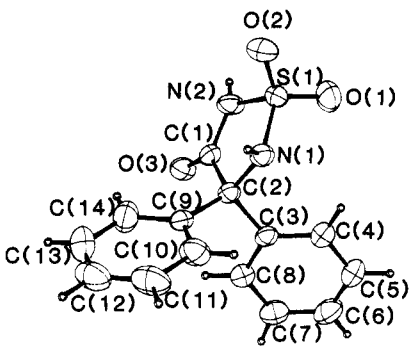
mmol) of *tert*-butyl alcohol and 50 mL of hexane. Chlorosulfonyl isocyanate (3.00 g, 21.0 mmol) in 25 mL of hexane was added dropwise at such a rate that the mixture gently refluxed. Following the addition the mixture was heated at reflux for 45 min and then cooled to room temperature. To the reaction mixture containing *tert*-butylsulfamoyl chloride was added dropwise a solution containing 5.00 g (21.0 mmol) diphenylglycine methyl ester and 3.00 g (30.0 mmol) of triethylamine in 75 mL of THF. The mixture was stirred for 2 days at room temperature. Workup as described above for **10a** gave 2.64 g (39%) of **10b**: mp 179–180 °C; ^{1}H NMR ($CDCl_3$) δ 1.1 (s, 9 H), 3.75 (s, 3 H), 6.1 (s, 1 H), 7.4 (m, 10 H); ^{13}C NMR ($CDCl_3$) δ 29.33, 53.59, 53.78, 71.15, 127.98, 128.31, 129.42, 138.45, 172.50. Anal. Calcd for $C_{19}H_{24}N_2O_4S$: C, 60.61; H, 6.44; N, 7.54. Found: C, 60.58; H, 6.29; N, 7.43.

4,4-Diphenyl-2-*tert*-butyl-1,2,5-thiadiazolidin-3-one 1,1-Dioxide (11b). Via the general procedure for the preparation of **11a**, a solution of 0.70 g (1.8 mmol) of **10b** in 50 mL of THF was added dropwise to a stirred mixture of 0.13 g (56 mmol) of NaH and 10 mL of THF. The mixture was heated at reflux for 75 min. Workup afforded 0.47 g (76%, from hexane–chloroform) of **11b**: mp 136–138 °C; ^{1}H NMR ($CDCl_3$) δ 1.65 (s, 9 H), 7.4 (s, 10 H); ^{13}C NMR ($CDCl_3$) δ 26.99, 61.52, 72.84, 127.53, 128.76, 128.89, 138.52, 169.67. Anal. Calcd for $C_{18}H_{20}N_2O_3S$: C, 62.77; H, 5.85; N, 8.13. Found: C, 62.73; H, 5.65; N, 8.02.

X-ray Determination of 4,4-Diphenyl-1,2,5-thiadiazolidin-3-one 1,1-Dioxide. Compound $C_{14}H_{12}O_3N_2S$ (288 g/mol) crystallizes in the monoclinic space group $C2/c$ with a = 16.467 (5) Å, b = 13.451 (4) Å, c = 13.386 (5) Å, β = 115.47 (3)°, and z = 8. The calculated crystal density is 1.43 g/cm^3. Data were



collected at room temperature on an Enraf-Nonius CAD-4 diffractometer with Mo Kα (λ = 0.7173 Å) radiation with an incident beam graphite monochromator. The structure was solved by direct methods and refined by full-matrix least-squares methods. Refinement of non-hydrogen atoms anisotropically and hydrogen atoms with isotropic temperature factors by using 1974 observed reflections ($I > 3\sigma(I)$) from a set of 2274 unique reflections gave a final R = 0.043. There are no significant features in the final difference Fourier map. All computer programs used were from the SDP package.[16]

Note Added in Proof. Recent work by H. Kohn further illustrates the utility of cyclization reactions like those shown for our conversion of **10a,b** to **11a,b**. Furthermore, their X-ray structure parameters of the first-determined 3-oxo-1,2,5-thiadiazolidine 1,1-dioxide agree with ours.[17]

Acknowledgment. Portions of this research were sponsored by The Research Corporation (J.W.T.) and The National Institutes of Health (E.D.S.), and their support is gratefully acknowledged.

Supplementary Material Available: Tables of atomic coordinates and bond lengths and angles are available from the Cambridge Crystallographic Data Base: Crystallographic Data Centre, Cambridge University, University Chemical Lab, Cambridge, CB2 1EW, England.

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Chiral Purity Determination by ^{1}H NMR Spectroscopy. Novel Use of 1,1′-Binaphthyl-2,2′-diylphosphoric Acid

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The ability to determine the chiral purity of a substrate is a highly investigated area of research in NMR, partly due to the increasing availability and utility of enantioselective organic reagents. In order to use NMR as a tool for such determinations the substrates must be made diastereomeric. This is accomplished by converting the enantiomers to diastereomers by the use of a chiral auxiliary. These auxiliaries come in three varieties: chiral lanthanide shift reagents[1] (CLSR), chiral "solvents" or complexing agents,[2] and chemical derivatizing reagents like Mosher's[3] or Anderson–Shapiro[4] reagent.

During the course of analyzing potential therapeutic agents, we became involved with the determination of the chiral purity of secondary and tertiary amines. We tried to use chiral lanthanide shift reagents and chiral complexing agents but often found them unacceptable for various reasons. These unsatisfactory results led us to explore the possibility of finding a "new" reagent. Of particular interest was to obtain a reagent that was easy to use and would have an NMR spectrum devoid of signals in the important 1–6 ppm region of the proton NMR spectrum. In this respect, we have examined the utility of (*R* or *S*)-1,1′-binaphthyl-2,2′-diylphosphoric acid (BNPPA) as a NMR chiral complexing agent. BNPPA is well known as a reagent for the resolution of amines.[6]

Further support for using BNPPA as a chiral reagent stems from a report showing the utility of α-methoxy-α-(trifluoromethyl)phenylacetic acid (MTPA) in the determination of amine chiral purity by proton NMR.[5] Because of its greater acidity it was felt that BNPPA would be a better complexing agent than MTPA and also would give rise to larger shift nonequivalence due to its more extensive ring current.

Results

Given in Table I are the data obtained for amine substrates using BNPPA as well as comparisons with MTPA data from the literature. For each compound there were several resonances that showed chemical shift nonequivalences between enantiomers, but not all were suitable for accurate integration and thus quantitation of composition. It can be seen that the $\Delta\delta$ values[7] for BNPPA are generally larger than those observed for MTPA, and in at least one instance this difference is substantial, e.g. **7**. The spectral data for **7** is shown in Figure 1. When there is good interaction between BNPPA and the substrate, as shown

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0022-3263/89/1954-5826$01.50/0