1 H, CHOH), 4.61 (d, J = 6, 1 H, OCHHO), 4.69 (d, J = 6, 1 H, OCHHO); $[\alpha]^{RT}_D$ −49.6° (c 0.70, $CHCl_3$). Anal. Calcd for $C_{11}H_{22}O_5$: C, 56.39; H, 9.46. Found: C, 56.42; H, 9.50.

Acknowledgment. We thank the Schweizerischer Nationalfonds zur Förderung der wissenschaftlichen Forschung and the Sandoz AG (Basel) for generous financial support of this work, W. Manser for the determination of elemental composition, B. Brandenberg for measuring 300-MHz ^1H NMR spectra, and C. Schregenberger and R. Hässig for measuring 20-MHz ^{13}C NMR spectra.

Registry No. (±)-**1**, 87518-81-8; (±)-**2**, 87518-82-9; (±)-**3**, 87518-83-0; (±)-**4**, 87518-84-1; (+)-**5**, 87518-85-2; (+)-**6**, 87518-86-3; (+)-**7**, 87518-87-4; (±)-*l*-**8**, 87518-88-5; (±)-*u*-**8**, 87518-89-6; (±)-*l*-**9**, 87518-90-9; (±)-*l*-**9**, 87518-91-0; (±)-*u*,*u*-**10**, 87518-92-1; (±)-*l*,*u*-**10**, 87583-28-6; (±)-*u*,*l*-**11**, 87583-29-7; (±)-*l*,*l*-**11**, 87583-30-0; (−)-**12**, 87518-93-2; (+)-**13**, 87518-94-3; (−)-**14**, 87518-95-4; (±)-*u*-**15**, 87518-96-5; (±)-*l*-**15**, 87518-97-6; (±)-*u*,*u*-**16**, 87518-98-7; (±)-*l*,*u*-**16**, 87583-31-1; (±)-*u*,*l*-**17**, 87583-32-2; (±)-*l*,*l*-**17**, 87583-33-3; (±)-*u*-**18**, 87518-99-8; (±)-*l*-**18**, 87519-00-4; (±)-*u*,*u*-**19**, 87519-01-5; (±)-*l*,*u*-**19**, 87583-34-4; (±)-*u*,*l*-**20**, 87583-35-5; (±)-*l*,*l*-**20**, 87583-36-6; (−)-**21**, 87519-02-6; **22**, 87519-03-7; chloromethyl methyl ether, 107-30-2; ethyl vinyl ether, 109-92-2; (2*S*,3*R*)-(−)-2-ethyl-3-methoxymethoxy-1-butanol, 87519-04-8; ethyl (2*R*,3*R*)-(−)-2-ethyl-3-hydroxybutanoate, 87519-05-9; ethyl (2*R*,3*R*)-2-ethyl-3-(methoxymethoxy)butanoate, 87519-06-0; 3-(1-ethoxyethoxy)-2-ethyl-1-butanol, 87519-07-1; ethyl 3-(1-ethoxyethoxy)-2-ethylbutanoate, 87519-08-2; (2*S*,3*R*)-(−)-3-benzyloxymethoxy-2-ethyl-1-butanol, 87519-09-3; chloromethyl benzyl ether, 3587-60-8; ethyl (2*R*,3*R*)-3-benzyloxymethoxy-2-ethylbutanoate, 87519-10-6; (±)-3-benzyloxymethoxy-1-butanol, 87519-11-7; (±)-3-benzyloxymethoxy-1-butanal, 87519-12-8; triphenylcarbomethoxymethylenephosphorane, 2605-67-6; (±)-3-benzyloxymethoxy-2-methyl-1-propanol, 87519-13-9; (±)-3-benzyloxymethoxy-2-methyl-1-propanal, 79027-30-8; (±)-*l*-3-benzyloxymethoxy-2-methyl-1-butanol, 87519-14-0; (±)-*u*-3-benzyloxymethoxy-2-methyl-1-butanol, 87519-15-1; (±)-*u*-3-benzyloxymethoxy-2-methyl-1-butanal, 87519-16-2; (±)-*l*-3-benzyloxymethoxy-2-methyl-1-butanal, 87519-17-3; (2*R*,3*R*)-2-ethyl-3-methoxymethyl-1-butanal, 87519-18-4; 3-(1-ethoxyethoxy)-2-ethyl-1-butanal, 87519-19-5; (2*R*,3*R*)-3-benzyloxymethoxy-2-ethyl-1-butanal, 87519-20-8; dimethyl (2-oxobutyl)phosphonate, 41162-15-6; benzyl alcohol, 100-51-6; benzoyl chloride, 98-88-4; 2,2-dimethoxypropane, 77-76-9.

The Denticulatins, Two Polypropionate Metabolites from the Pulmonate *Siphonaria denticulata*

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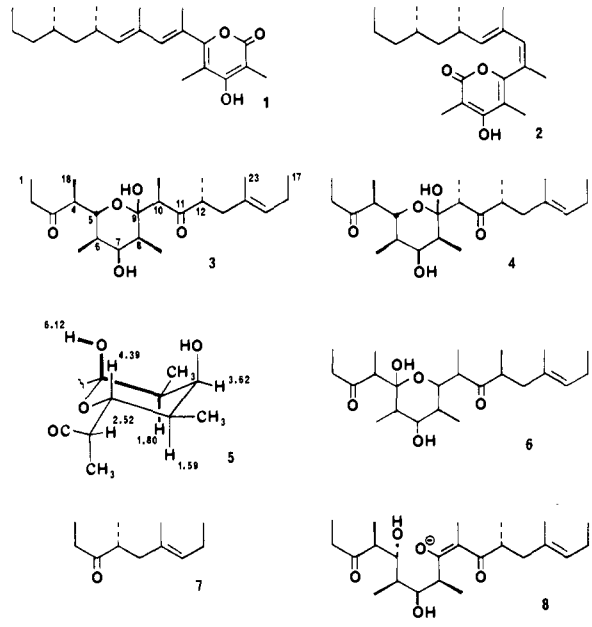
Received May 2, 1983

Abstract: Two isomeric polypropionate metabolites, denticulatin A (**3**) and denticulatin B (**4**), were isolated from the marine pulmonate mollusk *Siphonaria denticulata*. The structures were elucidated by interpretation of spectral data and a single-crystal X-ray diffraction study. Denticulatin A was ichthyotoxic at 10 μg/mL while denticulatin B was toxic at 30 μg/mL.

Siphonaria denticulata Quoy and Gaimard, 1833,[1] is an air-breathing marine mollusk of the subclass Pulmonata. This pulmonate mollusk is commonly found in the intertidal zone along the coast of New South Wales, Australia. The siphonariids, commonly known as false limpets, resemble limpets in both appearance and behavior. When submerged, siphonariids remain firmly clamped in crevices on rocks. Shortly after they are exposed by the retreating tide they move about the rocks to feed on encrusting algae and microorganisms, returning to their crevices when threatened by the heat of the sun or the incoming tide.[2] Thus, siphonariids are exposed to both marine and terrestrial predators and may employ chemical defense mechanisms. We have recently reported the isolation of two antimicrobial pyrones, diemenesin A (**1**) and diemenensin B (**2**) from *Siphonaria diemenensis*.[3] In this paper we describe the structural elucidation of denticulatin A (**3**) and denticulatin B (**4**), further examples of *Siphonaria* metabolites having a polypropionate carbon skelton.

Specimens of *S. denticulata* were collected at Coledale and Eden, New South Wales, Australia, in March 1982 and stored in acetone until needed. The ethyl acetate soluble material from the acetone extracts was chromatographed by HPLC on Partisil using ether–hexane (1:1) as the eluant to obtain denticulatin A (**3**, 0.06–0.12 mg/animal) and denticulatin B (**4**, 0.04–0.10 mg/animal). Initial examination of spectral data revealed that **3** and **4** were isomeric, had the same carbon skeleton, and probably differed in stereochemistry at a single center.

Both denticulatin A (**3**), $[\alpha]_D$ −30.7°, and denticulatin B (**4**), $[\alpha]_D$ −26.4°, had the molecular formula $C_{28}H_{40}O_5$. The high-resolution mass spectra indicated a molecular formula of $C_{28}H_{38}O_4$ for the highest mass peak but the ^{13}C NMR spectra required five oxygen atoms, two of which were incorporated into a hemiketal group that would be expected to eliminate water in the mass spectrometer. Since the compounds are so similar, we will describe



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0002-7863/83/1505-7413$01.50/0

Table I. ^{1}H NMR Data for Denticulatin A (3) and Denticulatin B (4)

H at C no.	3	multiplicity	4
1	1.03	t, 3 H, J = 7.5 Hz	1.06
2	2.47	m, 1 H	2.46
2	2.53	m, 1 H	2.54
4	2.52	m, 1 H	2.53
5	4.39	dd, 1 H, J = 10.5, 3 Hz	4.41
6	1.59	m, 1 H	1.62
7	3.62	m, 1 H, J = 9, 2.5, 2.5 Hz	3.56
7-OH	3.38	d, 1 H, J = 9	3.09
8	1.80	m, 1 H	1.69
9-OH	6.12	br s, 1 H	5.34
10	2.75	q, 1 H, J = 7 Hz	2.95
12	2.95	m, 1 H	2.67
13	2.19	dd, 1 H, J = 14, 3.5	2.30
13	1.73	dd, 1 H, J = 14, 9.5	1.72
15	5.14	t, 1 H, J = 7 Hz	5.19
16	2.00	m, 2 H, J = 7 Hz	2.01
17	0.94	t, 3 H, J = 7 Hz	0.96
18	1.04	d, 3 H, J = 7 Hz	1.02
19	0.97	d, 3 H, J = 7 Hz	0.96
20	1.10	d, 3 H, J = 7 Hz	1.21
21	1.20	d, 3 H, J = 7 Hz	1.16
22	0.93	d, 3 H, J = 7 Hz	0.93
23	1.58	br s, 3 H	1.62

only the interpretation of spectral data for denticulatin A (**3**), the single-crystal X-ray diffraction study on denticulatin B (**4**), and the data that established the relationship between the two metabolites.

The infrared spectrum of denticulatin A (**3**) contained two carbonyl bands at 1700 and 1685 cm^{-1} and hydrogen-bonded hydroxyl bands at 3440 and 3320 cm^{-1}. The ^{13}C NMR spectrum contained signals at δ 218.6 (s) and 212.0 (s) that confirmed the presence of two ketone carbonyl groups, at 131.7 (s) and 129.5 (d) assigned to a trisubstituted olefin, and at 102.9 (s) due to the hemiketal carbon. The ^{1}H NMR spectrum of denticulatin A (Table I) consisted of five isolated spin systems that incorporated signals for eight methyl groups, two of which were adjacent to methylene groups, the remaining six being adjacent to methine carbons. These data implied that the structure of denticulatin A (**3**) was based on a polypropionate carbon skeleton.

^{1}H NMR signals at δ 1.03 (t, 3 H, J = 7.5 Hz), 2.47 (dq, 1 H, J = 18, 7.5 Hz), and 2.53 (dq, 1 H, J = 18, 7.5 Hz) indicated the presence of an ethyl ketone at one end of the molecule while the other terminus consisted of an ethyl group attached to the trisubstituted olefin giving rise to signals at 0.94 (t, 3 H, J = 7 Hz), 2.00 (m, 2 H, J = 7 Hz), and 5.14 (t, 1 H, J = 7 Hz) with an olefinic methyl signal at 1.58 (brs, 3 H). The signals at δ 1.73 (dd, 1 H, J = 14, 9.5 Hz), 2.19 (dd, 1 H, J = 14, 3.5 Hz), 2.95 (m, 1 H), and 0.93 (d, 3 H, J = 7 Hz) were assigned to an olefinic methylene group adjacent to a methine bearing both a methyl group and a ketone. The isolated spin system at δ 2.75 (1 H, q, J = 7 Hz) and 1.20 (3 H, d, J = 7 Hz) must be due to a CH_3–CH group flanked by the hemiketal carbon and one of the ketone carbonyls. The remaining signals were assigned to the cyclic hemiketal residue **5**, with signals assigned as shown. The stereochemistry about the tetrahydropyran ring was defined by decoupling studies that indicated an axial–axial–equatorial relationship between the protons at δ 4.39, 1.59, and 3.62 respectively: the ~1-Hz W coupling between the hydroxyl proton at 6.12 and the methine proton at 1.80 required both groups to be axial.

Since denticulatin A contained two isolated ketones, interpretation of the ^{1}H NMR data did not distinguish between structures **3** and **6**. However, treatment of denticulatin A with DBU gave the ketone **7**, $[\alpha]_D$ −3.8°. Examination of a number of literature examples[4] indicated that the levorotatory enantiomer of ketone **7** must have the 4*R* stereochemistry. Assuming that the ketone **7** resulted from a retro-aldol reaction, denticulatin A must have the structure **3**. We could not, however, define the relative stereochemistry at carbons 4, 10, and 12. The geometry about the olefinic bond was *E* as indicated by the ^{13}C NMR data [δ 15.9 (q, C-23)].

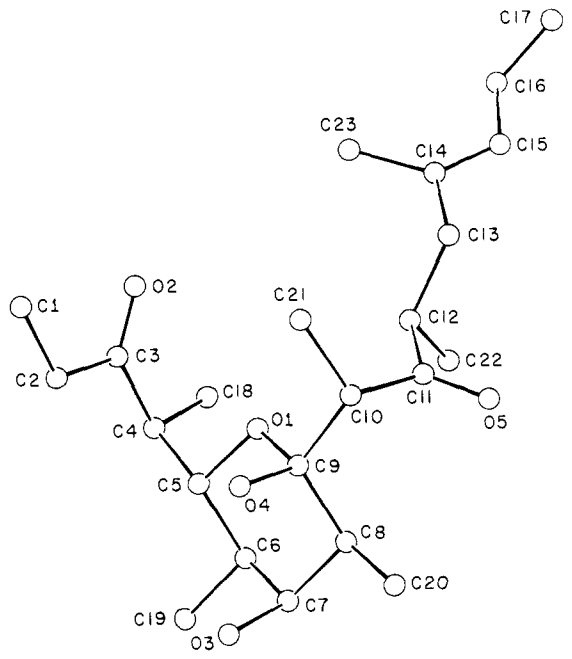

Figure 1. A computer-generated perspective drawing of denticulatin B (**4**). Hydrogens are omitted for clarity.

Comparison of the ^{1}H NMR spectra of denticulatin A and B (Table I) indicated only minor differences between the two molecules. A single-crystal X-ray diffraction analysis was performed on denticulatin B (**4**), mp 137–141 °C, in order to confirm the structural assignments and elucidate the elusive stereochemical features. A computer-generated perspective drawing of the final X-ray model less hydrogens of denticulatin B (**4**) is shown in Figure 1. The X-ray experiment determined only the relative configuration, and the absolute stereostructure shown was indicated by the optical rotation of the ketone **7** obtained by reaction of denticulatin B with DBU. In general, bond distances and angles agree well with accepted values although there is substantial thermal motion and consequent bond shortening at both ends of the molecule. There appears to be a hydrogen bond from OH-3 to O-4 with a distance of 2.71 Å. The pyran ring is in the chair conformation with all carbons substituents in equatorial positions. The hydroxyl groups are in axial orientations.

Comparison of the ^{1}H NMR spectra of denticulatin A (**3**) and deteniculatin B (**4**) revealed that significant chemical shift differences were associated with only the signals due to protons at carbons 8, 10, 12, 13, 20, and 21. Although we were unable to assign the methyl carbon signals with any confidence, the remaining signals in the ^{13}C NMR spectra were assigned by using J_R values, literature data,[5] and calculated chemical shift data.[6] These assignments suggested that the major differences in the ^{13}C NMR spectra of the denticulatins were found in the C-8 to C-11 region. The observation of a W coupling between the C-9 hydroxyl proton and the C-8 proton established the identical stereochemistry at carbons 8 and 9 in both molecules. Treatment of both denticulatin A and denticulatin B with DBU gave the same levorotatory ketone **7**. We therefore propose that denticulatins A (**3**) and B (**4**) differ in stereochemistry at C-10 only. This proposal was supported by the observation that treatment of denticulatin B (**4**) with DBU for a short period of time gave a 1:1 mixture of denticulatin A (**3**) and denticulatin B (**4**). We propose that this isomerization could most readily occur by way of the intermediate **8**.

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(6) Wehrli, F. W.; Wirthlin, T. "Interpretation of carbon-13 NMR spectra"; Heyden: London, 1976; pp 36–46.

Denticulatin B (**4**) was ichthyotoxic toward goldfish at 10 μg/mL (1 h) whereas denticulatin A (**3**) was toxic at 30 μg/μL (1 h) but caused a reversible loss of equilibrium at 10 μg/mL. It is therefore tempting to suggest that the denticulatins serve to inhibit predation. Despite the resemblance of the denticulatins to portions of several polyether antibiotics, we found no activity against common test organisms.

Experimental Section

Collection, Extraction, and Chromatography. (a) Coledale Sample. One hundred forty individuals of *Siphonaria denticulata* (average dry weight of animal, 26 mg) were collected by hand in the mid-intertidal zone in Coledale, Australia, in Dec 1981. Whole animals were placed immediately into acetone (75 mL) and allowed to steep at <5 °C for 7 months. The acetone was decanted and evaporated to leave an aqueous suspension that was diluted with distilled water to 100 mL and then extracted with ethyl acetate (4 × 70 mL). The combined ethyl acetate layers were dried over sodium sulfate and evaporated to leave a green oil (240 mg). The oil was filtered through a silica gel plug in ether and then separated by HPLC on a Whatman M-9 Partisil column using 2:1 ether–hexane as the eluant to yield, in addition to several fats, denticulatin A (**3**, 16.1 mg, 0.12 mg/animal) and denticulatin B (**4**, 13.4 mg, 0.10 mg/animal).

(b) Eden Sample. One hundred ninety individuals of *S. denticulata* (average dry weight of animal, 34 mg) were collected by hand in the mid-intertidal zone in Eden, Australia, in Dec of 1981. The animals were extracted according to the procedure above to obtain a crude ethyl acetate soluble fraction (212 mg) that was separated by HPLC to obtain fats, denticulatin A (**3**, 12.3 mg, 0.06 mg/animal), and denticulatin B (**4**, 7.9 mg, 0.04 mg/animal).

Denticulatin A (3): oil; $[\alpha]_D$ −30.7° (*c* 1.49, $CHCl_3$); IR (CCl_4) 3440, 3320, 1700, 1685 cm^{-1}; ^1H NMR ($CDCl_3$) see Table I; ^{13}C NMR (C_6D_6) δ 218.6 (s), 212.0 (s), 131.7 (s), 129.5 (d), 102.9 (s), 75.6 (d, J_R = 38.4 Hz), 69.8 (d, J_R = 40.4 Hz), 50.8 (d, J_R = 29.3 Hz), 47.3 (d, J_R = 26.7 Hz), 43.2 (d, J_R = 31.5 Hz), 42.8 (t), 38.8 (d, J_R = 22.3 Hz), 37.7 (d, J_R = 21.3 Hz), 32.8 (t), 21.7 (t), 15.9 (q), 15.7 (q), 14.5 (q), 13.6 (q, q), 12.0 (q), 8.1 (q), 8.0 (q); mass spectrum *m/z* (rel intensity), 378 (4), 296 (11), 291 (17), 267 (16), 240 (17), 211 (21), 167 (33), 166 (17), 57 (100); high-resolution mass measurement, obsd *m/z* 378.2768, $C_{23}H_{38}O_4$ (M − H_2O) requires *m/z* 378.2770.

Denticulatin B (4): mp 137–141 °C; $[\alpha]_D$ −26.4° (*c* 0.39, $CHCl_3$); IR ($CHCl_3$) 3390, 1710, 1695 cm^{-1}, ^1H NMR ($CDCl_3$) see Table I; ^{13}C NMR (C_6D_6) δ 218.4 (s), 209.3 (s), 132.0 (s), 129.5 (d), 101.9 (s), 76.3 (d, J_R = 36.6 Hz), 69.3 (d, J_R = 41.7 Hz), 52.3 (d, J_R = 31.5 Hz), 47.0 (d, J_R = 26.9 Hz), 43.0 (d, J_R = 30.2 Hz), 42.9 (t), 41.7 (d, J_R = 24.7 Hz), 37.7 (d, J_R = 22.0 Hz), 32.5 (t), 21.7 (t), 15.5 (q), 15.2 (q), 14.7 (q), 14.5 (q), 13.3 (q), 12.2 (q), 8.1 (q), 7.7 (q); mass spectrum *m/z* (rel intensity) 378 (18), 296 (26), 291 (33), 267 (28), 240 (28), 211 (28), 167 (35), 166 (17), 57 (100); high-resolution mass measurement, obsd *m/z* 378.2780, $C_{23}H_{38}O_4$ (M − H_2O) requires *m/z* 378.2770.

Base-Catalyzed Cleavage of the Denticulatins. (a) Denticulatin A. 1,8-Diazabicyclo[5.4.0]undec-7-ene (DBU) (5 drops) was added to a stirred solution of denticulatin A (**3**, 8 mg, 0.02 mmol) in dry benzene (10 mL) under a dry nitrogen atmosphere. After 30 min, the benzene was evaporated in vacuo, the residue was redissolved in ether, and the solution passed through a Sep-PAK of silica gel. Chromatography of the residue by HPLC on a Whatman M-9 Partisil column using 1:1 ether–hexane as the eluant gave (4*R*,6*E*)-4,6-dimethyl-6-nonen-3-one (**7**, 0.7 mg, 20% theoretical), $[\alpha]_D$ −3.8° (*c* 0.07, $CHCl_3$).

(b) Denticulatin B. DBU (5 drops) was added to a solution of denticulatin B (9 mg, 0.023 mmol) in dry benzene (10 mL). The reaction was repeated by using the procedure above to obtain (4*R*,6*E*)-4,6-dimethyl-6-nonen-3-one (**7**, 0.7 mg, 18% theoretical) $[\alpha]_D$ −3° (*c* 0.07, $CHCl_3$).

(4*R*,6*E*)-4,6-Dimethyl-6-nonen-3-one (7): oil; ^1H NMR ($CDCl_3$) δ 0.94 (t, 3 H, *J* = 7.6 Hz), 1.05 (t, 3 H, *J* = 7.2 Hz), 1.13 (d, 3 H, *J* = 6.8 Hz), 1.62 (s, 3 H), 2.42 (m, 1 H), 2.66 (t, 2 H, *J* = 7.6 Hz), 5.19 (t, 1 H, *J* = 7 Hz).

Base-Catalyzed Isomerization. DBU (5 drops) was added to a solution of denticulatin B (**4**, 11 mg, 0.028 mmol) in dry benzene (10 mL) and the mixture was stirred under an atmosphere of dry nitrogen for 5 min. The reaction mixture was poured onto 5% aqueous hydrochloric acid (25 mL) and extracted with ether (50 mL). The organic phase was washed with 5% aqueous hydrochloric acid (2 × 25 mL) followed by distilled water (3 × 25 mL) and then dried over anhydrous sodium sulfate. The solvent was evaporated to obtain a clear oil that was chromatographed by HPLC on a Whatman M-9 silica gel column in ether to obtain denticulatin A (**3**, 3 mg, 27% yield) and denticulatin B (**4**, 3 mg, 27% recovery).

Single-Crystal X-ray Diffraction Analysis of Denticulatin B (4). A crystal (0.7 × 0.6 × 0.5 mm) was selected for analysis. Preliminary X-ray photographs displayed orthorhombic symmetry and lattice constants of *a* = 16.200 (3), *b* = 16.303 (3), and *c* = 9.088 (2) Å were determined by a least-squares fit of 15 diffractometer measured 2θ values in the range 35.0 ≤ 2θ ≤ 45.0. Systematic extinctions, crystal density (~1.1 g/cm^3), and the presence of chirality were uniquely accommodated by space group $P2_12_12_1$ with one molecule of composition $C_{23}H_{40}O_5$ forming the asymmetric unit. All unique diffraction maxima with 2θ ≤114° were collected by using a variable-speed, 1° ω-scan and graphite monochromated Cu Kα radiation (1.54178 Å). After correction for Lorentz, polarization, and background effects, 1825 (96%) intensities were judged observed and used in later calculations. A phasing model was achieved by using the MULTAN series of programs[7] and after tangent formula recycling of a plausible 18-atom fragment, all 28 nonhydrogen atoms were located. Most hydrogens were located in a difference synthesis following partial refinement and the remainder were included at calculated positions. Block-diagonal least-squares refinement with anisotropic nonhydrogen atoms and isotropic hydrogens have converged to a current crystallographic residual of 0.087 for the observed reflections. Additional crystallographic data are described in the supplementary material (see paragraph at end of paper regarding supplementary material).

Acknowledgment. The animals were identified by Bruce Jenkins of the Australian Museum, Sydney. High-resolution mass measurements were provided by the Bio-organic, Biomedical Mass Spectrometry Resource (A. L. Burlingame, Director) supported by National Institutes of Health Grant RR00719. This study was funded by the National Science Foundation (CHE81-21471), the National Institutes of Health CA-24487), and New York State Sea Grant. D.J.F. thanks the Australian Department of Science and Technology for the award of a Senior Queen's Fellowship and the University of New South Wales for assistance in starting this research project.

Supplementary Material Available: Tables of fractional coordinates, thermal parameters, bond distances, and bond angles and ^1H NMR spectra of denticulatin A (**3**) and denticulatin B (**4**) (6 pages). Ordering information is given on any current masthead page.

(7) All crystallographic calculations were done on a PRIME 400 computer operated by the Materials Science Center and the Department of Chemistry, Cornell University. The principal programs used were REDUCE and UNIQUE, data reduction programs (Leonowicz, M. E., Cornell University, 1978), BLS78A, anisotropic block-diagonal least-squares refinement (Hirotsu, K.; Arnold, E., Cornell University, 1983), XRAY76, the X-ray System of Crystallographic Programs (Stewart, J. M., Ed., University of Maryland, Technical Report TR-445, March 1976), ORTEP, crystallographic illustration program (Johnson, C. K., Oak Ridge, ORNL-3794), BOND, molecular metrics program (Hirotsu, K., Cornell University, 1978), and MULTAN-78 ("A System of Computer Programs for the Automatic Solution of crystal Structures from X-ray Diffraction Data"; University of York; England; principal author P. Main; for a literature description of MULTAN see Germain, G.; Main, P.; Woolson, M. M. *Acta Crystallogr., Sect B* **1970**, *B26*, 274–285. Woolfson, M. M. *Acta* Crystallogr., *Sect A* **1977**, *A33*, 219–225).