1450, 1370, 1310, 1260, 1210, 1140, 1080, 1040, 1025 $cm^{-1}$.

Formate salt **64** (51 mg, 0.040 mmol) in pyridine (1 mL) and acetic anhydride (1 mL) was stirred at room temperature for 2 h. Purification by extraction [EtOAc/1 N HCl(aq), $NaHCO_3$(sat, aq) and NaCl(sat, aq) washes] and silica gel column chromatography (7:93 EtOH-$CH_2Cl_2$) afforded *N*-acetyl trimer **65** (37 mg, 72%) as a white solid: $^1H$ NMR (250 MHz, $CDCl_3$) 8.70 (d, 1 H), 8.49 (d, 2 H), 6.9-7.7 (m, 41 H), 6.11 (t, 1 H), 5.10 (m, 12 H), 4.79 (m, 1 H), 4.44 (m, 1 H), 4.31 (m, 1 H), 3.63 (s, 3 H), 3.2-3.6 (m, 6 H), 1.47 (s, 3 H); IR ($CHCl_3$) 3340, 3060, 3030, 2930, 2870, 1735, 1650, 1570, 1510, 1450, 1370, 1310, 1260, 1210 $cm^{-1}$.

A suspension of trimer **65** (37 mg, 0.0289 mmol) and 10% Pd on carbon (8 mg) in 1:1 $CH_2Cl_2$-MeOH (4 mL) was flushed with hydrogen, and 2.38 N HCl in MeOH (18 μL, 0.043 mmol) was added. The mixture was stirred at room temperature under an atmosphere of hydrogen for 12 h, and the catalyst was filtered (Celite) and washed with EtOAc. The combined filtrates were washed with 1 N HCl(aq) and 0.2 M aqueous citrate buffer (pH 5.5). The organic phase was dried ($Na_2SO_4$), evaporated under reduced pressure, and recrystallized from EtOAc/hexane to afford linear trimer **66** (16 mg, 75%) as a white powder: mp 155-157 °C (precipitated from MeOH with $H_2O$ and lyophilized); $^1H$ NMR (250 MHz, $CD_3OD$) 7.18-7.27 (m, 3 H), 6.88-6.94 (m, 3 H), 6.64-6.74 (m, 3 H), 4.75 (m, 1 H), 4.60 (m, 2 H), 3.86 (m, 1 H), 3.72 (s, 3 H), 3.50-3.65 (m, 5 H), 1.87 (s, 3 H); IR (KBr) 3700-2500 (br), 1730, 1635, 1580, 1540, 1525, 1450, 1360, 1330, 1260, 1170 $cm^{-1}$; mass spectrum (field desorption), *m/e* 740 ($M^+$).

**Δ-Cis Fe(III) Complex 67.** Linear trimer **66** (0.9 mg, 1.22 μmol) in MeOH (3.0 mL) was mixed with $4 \times 10^{-4}$ M $FeCl_3$ in MeOH (3.0 mL). The resulting violet solution was diluted 1:1 with 0.1 M aqueous phosphate buffer (pH 7.2) to afford a wine-red solution of ferric complex **67**: visible spectrum (3:1 pH 7.2 aqueous phosphate buffer-MeOH) $\lambda_{max}$ 495 nm (ε 4400); CD spectrum, vide Figure 1.

Equal volumes of ferric complex **67** ($1 \times 10^{-4}$ M) in 50% aqueous MeOH and ethylenediaminetetraacetic acid (EDTA) disodium salt in 0.1 M aqueous phosphate buffer (pH 7.2) were mixed, and the absorption at 495 nm was measured after equilibration of chelated Fe(III) was observed (constant optical density at 495 nm). The absorbance at 495 nm was 4%, 9%, and 86% diminished at $10^{-3}$, $10^{-2}$, and $10^{-1}$ M EDTA, respectively. By use of these data and a $pK_a$ of 10.1 ± 0.2 for linear analogue **66**, the formation constant was calculated; $K_f = 10^{46.5 \pm 1.2}$ ($K_f$ for Fe(III)·EDTA complex = $10^{25}$).[46]

**Acknowledgment** is made to the National Institutes of Health (Grant No. AM 20452 and GM 28828), Eli Lilly and Co., and Firmenich and Co. for their generous financial support. We thank Professor J. B. Neilands (Berkeley) for biological testing and for his continued collaboration, Mr. Curtis Adams, Ms. Barbara Beiber, and Mr. Mitchell Weitz for technical assistance, and Dr. Catherine Costello for field-desorption and exact mass spectra. We also thank Professors D. Kemp and S. Masamune for valuable discussions.

**Registry No.** **5**, 4726-96-9; **6**, 69146-58-3; **7**, 78088-92-3; **8**, 40351-09-5; **9**, 78088-93-4; **10**, 78088-94-5; **11**, 7724-78-9; **12**, 78088-95-6; **13**, 78148-29-5; **14**, 30414-15-4; **15**, 78148-30-8; **16**, 3262-72-4; **17**, 78088-96-7; **18**, 23680-31-1; **19**, 78088-97-8; **20**, 78088-98-9; **20** dicyclohexylammonium salt, 78088-99-0; **24**, 78089-00-6; **25**, 78089-01-7; **26**, 78089-02-8; **27** (isomer 1), 78089-03-9; **27** (isomer 2), 78089-04-0; **28** (isomer 1), 78089-05-1; **28** (isomer 2), 78089-06-2; **29**, 78089-07-3; **30**, 78089-08-4; **31**, 78089-09-5; **33**, 6081-61-4; **34**, 75299-15-9; **34** THP ether, 78089-10-8; **3** (isomer 1), 78148-31-9; **35** (isomer 2), 78148-32-0; **36**, 75299-17-1; **36** THP ether, 78089-11-9; **37**, 75299-18-2; **37** THP ether, 78089-12-0; **38**, 75299-19-3; **39**, 75363-09-6; **40**, 75363-10-9; **41**, 75299-20-6; **42**, 75363-11-0; **44**, 16947-86-7; **45**, 78089-13-1; **45** dicyclohexylammonium salt, 78089-14-2; **46**, 78089-15-3; **47**, 78089-16-4; **48**, 78089-17-5; **49**, 78148-33-1; **50**, 16947-84-5; **51**, 78089-18-6; **52**, 78089-20-0; **53**, 78089-21-1; **54**, 78089-23-3; **55**, 78089-24-4; **56**, 78089-25-5; **57**, 78089-26-6; **59**, 78089-27-7; **60**, 10491-78-8; **61**, 78089-28-8; **62**, 78089-30-2; **63**, 78089-31-3; **64**, 78089-33-5; **65**, 78089-34-6; **66**, 78089-35-7; **67**, 78090-01-4; 2,3-dihydroxybenzaldehyde, 24677-78-9; benzyl chloride, 100-44-7; 2,3-bis(benzyloxy)benzaldehyde, 5779-91-9; 2,3-bis(benzyloxy)benzoic acid, 74272-78-9; oxalyl chloride, 79-37-8; *p*-bromophenacyl bromide, 99-73-0; 2-(bromomethyl)anthraquinone, 7598-10-9; *p*-bromobenzyl bromide, 589-15-1.

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# Convenient Synthesis of the Pseudoguaiane Ring System

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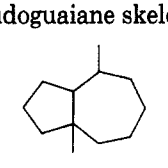
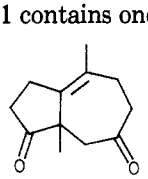
Ketone 8 was conveniently synthesized from acyclic precursors in a four-step procedure. Condensation of *cis*-1,4-dichloro-2-butene with diethyl ketone gave 2,7-dimethyl-4-cycloheptenone, which was alkylated with propargyl bromide. The triple bond was hydrated with mercuric ion impregnated Dowex 50 and the compound cyclized with potassium *tert*-butoxide to yield ketone 8.

The pseudoguaiane skeleton 1 contains one of the more



1



2

common ring systems found in hydroazulenic sesquiterpenoids.[1] Many early syntheses of the hydroazulenic ring system involved "special" reactions such as rearrangements, ring expansions and photolyses. Over the past decade, however, many syntheses using "conventional" procedures have appeared.[2] Recently, Lansbury[3] has used the different reactivities of the carbonyls in diketone 2,

(1) See: Devon, T. K; Scott, A. I. "Handbook of Naturally Occurring Compounds"; Academic Press: New York, 1972; Vol. II, pp 61-72.

(2) See, for example: Marshall, J. A.; Partridge, J. J. *Tetrahedron* 1969, *25*, 2159. Kato, M.; Kosugi, H.; Yoshikoshi, A. *Chem. Commun.* 1970, 185. Hendrickson, J. B.; Boeckman, R. K., Jr. *J. Am. Chem. Soc.* 1971, *93*, 1307. Heathcock, C. H.; Ratcliffe, R.; Van, J. *J. Org. Chem.* 1972, *37*, 1976. Marshall, J. A.; Ruden, R. A. *Ibid.* 1971, *36*, 2569. Marshall, J. A.; Green, A. E. *Ibid.* 1972, *37*, 982. Anderson, N. H.; Uh, H.-S. *Synthe. Commun.* 1973, *3*, 115. Anderson, N. H.; Uh, H.-S. *Tetradron Lett.* 1973, 2079. Buchanan, G. L.; Young, G. A. R. *J. Chem. Soc., Perkin Trans. 1*, 1974, 2404. Marshall, J. A.; Ruth, J. A. *J. Org. Chem.* 1974, *39*, 1971. De Clercq, P.; Vandewalle, M. *Ibid.* 1977, *42*, 3447.

(3) Lansbury, P. T.; Serelis, A. K. *Tetrahedron Lett.* 1978, 1909.

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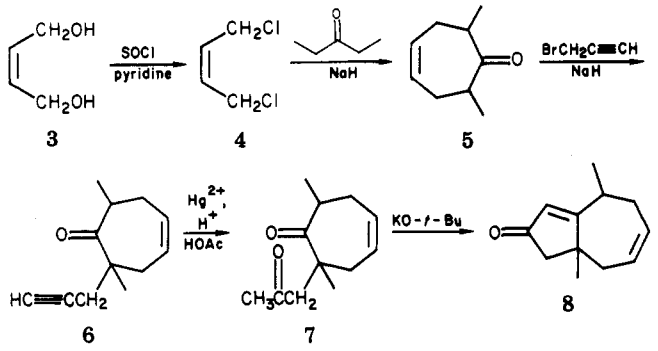
Scheme I




readily available from 2-methyl-1,3-cyclopentanedione, to provide an expeditious route to almost any pseudoguaiane.

In connection with studies of the properties of five- and seven-membered rings[4] and particularly the ring system of the hydroazulenic sesquiterpenoids, we have developed a convenient five-step synthesis of the fused five–seven ring system of the pseudoguaianes starting from readily available acyclic precursors.

*cis*-2-Butene-1,4-diol (**3**) was converted[5a] by thionyl chloride and pyridine to *cis*-1,4-dichloro-2-butene (**4**). 3-Pentanone was treated with 2 equiv of sodium hydride and was successfully reacted with **4** to yield[5b] 2,7-dimethyl-4-cycloheptenone (**5**), the identity of which was confirmed by hydrogenation to the known[6] 2,7-dimethylcycloheptanone. We assume that the predominant isomer in each case is the cis[6] since both methyl groups are epimerizable. Alkylation of the cycloheptenone **5** with sodium hydride and propargyl bromide gave 2,7-dimethyl-2-(2-propynyl)-4-cycloheptenone (**6**), the triple bond of which was hydrated with mercuric ion impregnated Dowex 50 and acetic acid[7] to give 2,7-dimethyl-2-oxopropyl-4-cycloheptenone (**7**), again presumably a mixture of diastereoisomers, although the predominant one was isolated by distillation in essentially pure form. Cyclization was achieved by using potassium *tert*-butoxide in *tert*-butyl alcohol to yield ketone **8**.

The reactivity of 2,7-dimethylcycloheptenones toward alkylation had been predicted to be low by analogy with 2,6-dimethylcyclohexanone. Indeed, we found that some procedures we attempted would work with 2,6-dimethylcyclohexanone but not with our cycloheptenone **5**. Thus we were able to form the enamine from 2,6-dimethylcyclohexanone and dimethylamine using the procedure of White and Weingarten[8] with $SnCl_4$ in place of $TiCl_4$ but not the enamine from **5** and dimethylamine. All our efforts to form the pyrrolidine or morpholine enamines of 2,6-dimethylcyclohexanone failed. Attempts to react the anion of **5** (formed by using lithium diisopropylamide) with propylene oxide to form a 2-hydroxypropyl side chain failed, even though they appeared to give a low yield with 2,6-dimethycyclohexanone. Attempts to form the same side chain by using the tetrahydropyranyl ether of 1-bromo-2-propanol[9] with the ethylmagnesium bromide complex of an *N*-*n*-butyl imine[10] of 2,6-dimethylcyclohexanone failed, as did attempts to react the ethylene ketal of bromoacetone[11] directly with the anion from **5**.

The utility of ketone **8** as a precursor for hydroazulene synthesis is, of course, limited by the difficulty of using the seven-membered-ring double bond in **6**, **7**, or **8** to selectively introduce a substituent in the 7-position of the hydroazulene. Nevertheless, we feel that the synthesis we have developed has potential application in other areas when a fused five–seven ring system has to be prepared.

## Experimental Section

NMR spectra were measured in $CDCl_3$ solution at 90 MHz on a Varian EM-390 spectrometer, IR spectra on a Beckman IR-3300 or Perkin-Elmer 337 spectrophotometer, and mass spectra on a Varian EM-600 spectrometer at a 70-eV ionizing voltage. GLC analyses of reaction mixture were performed on a Varian 90 gas chromatograph (thermal-conductivity detector) using 10–15 ft × 0.25 in. aluminum columns packed with 10% Carbowax 20M on 60–80 Chromosorb G at temperatures between 120 and 160 °C. Analyses were performed by Galbraith Laboratories.

**2,7-Dimethyl-4-cycloheptenone (5).** A 50% dispersion of sodium hydride in oil (4.8 g, 100 mmol of NaH) was washed with *n*-pentane to remove the oil and suspended in 70 mL of dry benzene at 0 °C under an $N_2$ atmosphere. A mixture of 4.3 g (50 mmol) of 3-pentanone and 3.2 g (25 mmol) of *cis*-1,4-dichloro-2-butene (prepared from *cis*-2-butene-1,4-diol (**3**) by a published procedure[5]) in 20 mL of benzene was added over 1 h to the stirred NaH suspension at 0 °C. Stirring was continued for a further 1.5 h at 0 °C before the reaction was allowed to warm to ambient temperature and then refluxed for 3 h. Excess NaH was destroyed by addition of 95% ethanol, and the mixture was poured into ice and acidified to pH 2 with cold 10% HCl. The mixture was saturated with NaCl and extracted with ether, and the ether extract was washed with $NaHCO_3$ solution and dried over $MgSO_4$. Evaporation of the solvent yielded 7.2 g of pale yellow oil. Fractionation at atmospheric pressure with a Vigreux column removed the excess 3-pentanone (bp 85–90 °C), leaving 3.45 g (69%) of relatively pure 2,7-dimethyl-4-cycloheptenone (**5**). This was used without further purification in the subsequent step, although a small sample was further purified by distillation: bp 84 °C (28 mm) (strong peppermint odor); NMR $\delta$ 1.01 (d, $J$ = 7.5 Hz with further 1-Hz splitting, collapsing to two signals 1 Hz apart on irradiation at $\delta$ 2.7; total 6 H), 1.7–3.0 (6 H), 5.7 (m, 2 H); IR 3000 (s), 1700 (s), 1450 (m), 1365 (m), 1185 (w), 1015 (w) $cm^{-1}$; mass spectrum, $m/e$ (relative intensity) 130 ($M^+$, 74), 123 (11), 110 (32), 109 (26), 96 (26), 95 (32), 92 (32), 91 (50), 82 (68), 81 (100).

**Hydrogenation of 2,7-Dimethyl-4-cycloheptenone.** 2,7-Dimethyl-4-cycloheptenone (**5**, 2.36 g) in 50 mL of 95% ethanol was hydrogenated in the presence of 0.3 g of 10% Pd/C for 3 h. Upon filtration and evaporation, an essentially quantitative yield of 2,7-dimethylcycloheptane was obtained. A small sample was

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(5) (a) Bobbitt, J. M.; Amundsen, L. H.; Steiner, R. I. *J. Org. Chem.* **1961**, *25*, 2230. (b) A referee has suggested that, assuming that the cyclization occurs by sequential monoanion generation and alkylation rather than via the dianion, there should be some concomitant formation of the five-membered-ring product i. We have carefully examined the

i

NMR spectra of the crude **5** and can find no evidence of significant quantitities of i. Crude **5** contains both cis and trans isomers and exhibits two doublets at about $\delta$ 1.0. Early fractions collected in the distillation of **5** contained only one of the isomers, presumably the cis.[6] The final fraction collected contained both isomers, but none of the fractions showed any clear evidence of either the ethyl or methyl group of i signals which would be expected to be readily detectable. The residue from the distillation was, unfortunately, of such a nature that meaningful spectra were not obtained. While we do not rule out the possibility of up to about 15% of i in the crude **5**, it appears that the conditions are such as to favor formation of the kinetic enolate of the initial alkylation product, resulting in the seven-membered-ring product being favored.

(6) Bhanot, O. S.; Dutta, P. C. *J. Chem. Soc. C* **1968**, 2589.

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(9) Stewart, C. A.; VanderWerf, C. A. *J. Am. Chem. Soc.* **1954**, *76*, 1259.

(10) House, H. O. "Modern Synthetic Reactions", 2nd ed; W. A. Benjamin: Menlo Park, CA, 1972; pp 580–582 and references cited therein.

(11) Field, N. D. *J. Am. Chem. Soc.* **1961**, *83*, 3504.

purified by preparative GLC on a 15-ft Carbowax 20M column at 160 °C: NMR δ 1.01 (d, $J$ = 7 Hz, 6 H), absence of signal at δ 5.7; IR similar to that of **5**.

**2,7-Dimethyl-2-(2-propynyl)-4-cycloheptenone (6).** To 7.02 g (50.9 mmol) of 2,7-dimethyl-4-cycloheptenone (**5**) in 40 mL of DMF was added 2.9 g (60.4 mmol) of 50% NaH dispersion. The mixture was stirred until the slow bubbling stopped (ca. 2 h). Propargyl bromide (7.6 g, 63.8 mmol) was added slowly with external cooling (vigorous reaction), and the mixture was stirred overnight at room temperature. The reaction mixture was poured into ice–water and extracted with ether, and the ethereal solution was washed with $NaHCO_3$ solution and NaCl solution, dried over $MgSO_4$, and evaporated under reduced pressure. The crude oil was fractionally distilled to give unchanged **5** [2.8 g; bp 47–52 °C (2.5 mm)] and **6** (0.94 g; bp 83–90 °C (2.5 mm); 18%, 30% with allowance for recovered **5**]: NMR δ 1.00 (d, $J$ = 7 Hz, each further split by 1 Hz, 3 H), 1.11 (s, 3 H), 1.8–2.8 (including C≡CH and H α to carbonyl at ca. 2.4), 3.5 (m, 1 H), 5.62 (m, 2 H). There was evidence of a mixture of isomers in some later fractions: IR 3225 (s), 2990 (sh), 2970 (s), 2950 (s), 2095 (w), 1690 (s), 1440 (m), 1180 (w), 1140 (w), 990 (m) $cm^{-1}$.

**2,7-Dimethyl-2-(2-oxopropyl)cyclohept-4-enone (7).** Propargyl ketone **6** (9.99 g, 5.56 mmol) in 8 mL of glacial acetic acid, 0.8 mL of water, and 0.44 g of Dowex 50 impregnated with $Hg^{2+}$ ion[7] was refluxed for 45 min. The solution was decanted, and the Dowex 50 was washed with ether (3 × 30 mL), which was added to the decanted solution. Remaining acid was neutralized by addition of $NaHCO_3$. The ether extract was dried over anhydrous $Na_2SO_4$ and evaporated under reduced pressure. The pale yellow oil was distilled to give 0.61 g (56%) of **7**: bp 104–106 °C (2 mm); NMR δ 1.07 (d, $J$ = 7 Hz, 3 H), 1.15 (s, 3 H), 1.8–3.0 (incld δ 2.00 (s) and δ 2.06 (s)), 5–6 (m, 2 H); IR 2950 (s), 1690 (s) with 1730 (sh), 1435 (m), and 1345 (m), 1145 (m), 1110 (m), 990 (m) $cm^{-1}$; mass spectrum, $m/e$ 196 ($M^+$).

**Cyclization of 2,7-dimethyl-2-(2-oxopropyl)cyclohept-4-enone (7).** Diketone **7** (9.76 g, 3.88 mmol) in 5 mL of *tert*-butyl alcohol was added dropwise to 0.76 g (6.78 mmol) of potassium *tert*-butoxide in 30 mL of *tert*-butyl alcohol, and the mixture was stirred under nitrogen at room temperature for 2 h. The mixture was then poured into 60 mL of ice-cold 10% $H_2SO_4$ and extracted with 100 mL of dichloromethane. The dichloromethane solution was washed with $NaHCO_3$ and then with water, dried over $Na_2SO_4$, and evaporated under reduced pressure. GLC analysis showed one major and three minor components in the crude residue. Distillation yielded 0.57 g (87%) of **8**: bp 122–128 °C (4 mm); NMR δ 1.21 (d, $J$ = 9 Hz, 3 H), 1.20 (s, 3 H), 1.8–2.8 (incld δ 2.2 (s), 6 H), 5.7 (d, $J$ = 1 Hz, 1 H), 5.82 (m, 2 H); IR 3015 (sh), 2980 (s), 2965 (s), 1700 (s with sh at 1735), 1600 (m), 1450 (w), 1370 (w), 1250 (w) $cm^{-1}$. The semicarbazone of **8** was prepared and recrystallized from alcohol; mp 185–187 °C. Anal. Calcd for $C_{13}H_{19}N_3O_3$: C, 66.92; H, 8.21; N, 18.01. Found: C, 66.55; H, 8.18; N, 17.58.

**Acknowledgment.** We thank the Robert A. Welch Foundation for generous financial support.

**Registry No.** *cis*-**4**, 1476-11-5; **5**, 31333-42-3; **6**, 78109-59-8; **7**, 78109-60-1; **8**, 78109-61-2; **8** semicarbazone, 78109-62-3; 3-pentanone, 96-22-0; 2,7-dimethylcycloheptanone, 7272-19-7; propargyl bromide, 106-96-7.

# Further Cembranoid Derivatives from the Red Sea Soft Corals *Alcyonium flaccidum* and *Lobophytum crassum*

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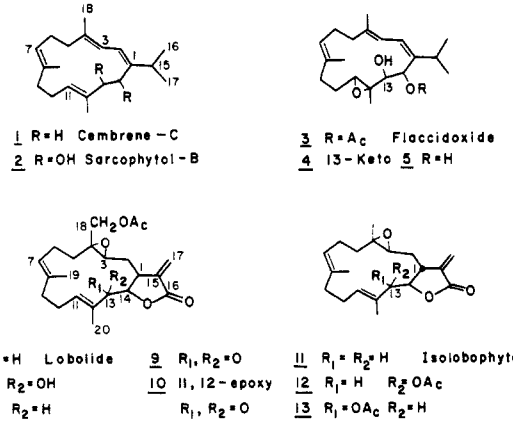
The cembranoid content of two soft corals has been investigated. Three compounds were isolated from *Alcyonium flaccidum* and were found to be cembrene-C (**1**), sarcophytol-B (**2**), and the as yet unreported 11,12-epoxy-13-hydroxy-14-acetoxycembrene-C (**3**), which was named flaccidoxide. From *Lobophytum crassum*, another soft coral whose chemical content was found to change remarkably with place of collection, were isolated two new 13-hydroxylobolides (**7** and **8**) in addition to lobolide (**6**), and their structures were characterized. The $^{13}C$ NMR spectra of all the above compounds were assigned.

Among the soft coral diterpenoids, the cembrane-type diterpenes form a large group of compounds which are widely distributed within the alcyonarian.[1,2] Of special interest are the observed, although not yet well understood, variations within a specific soft coral in the content of cembranes and other terpenoids.[2]

The case of *Sarcophyton glaucum* can serve as a good example for such variations as a function of location. Thus *S. glaucum* from the Red Sea yielded several cembranoids, the most prominent of which were the fish toxins sarcophine and sarcophytoxide (16-deoxysarcophine).[3] Tursch and co-workers,[4] on the other hand, found no sarcophine in *S. glaucum* specimens collected at Laing Island on the north coast of New Guinea but instead obtained sarcoglaucol, another new cembranoid fish toxin.[5] Most recently, Kobayashi and co-workers[6] reported isolation of

Chart I

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(2) For an up to date review, see B. Tursch, J. C. Braekman, D. Daloze, and M. Kaisin in "Marine Natural Products", Vol. 2, P. Scheuer Ed., Academic Press, New York, 1978, p 247.

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(4) M. Alberieci, J. C. Braekman, D. Daloze, and B. Tursch, *Bull. Soc. Chim. Belg.* **87**, 487 (1978).

(5) Sarcoglaucol has been isolated recently by us also from *S. auritum* (unpublished results).

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