Mycaperoxides F and G and a Related Norterpene Ketone from Southern Australian Marine Sponges, *Mycale* Species

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Received October 29, 1997

Investigation of two southern Australian marine sponges, *Mycale* spp., resulted in isolation of the known norsesterterpene mycaperoxide F methyl ester (**5**) together with a new norsesterterpene mycaperoxide G methyl ester (**10**) and a new norterpene ketone **11**. All structures were secured by spectroscopic analysis and chemical derivatization. The absolute stereochemistry previously assigned to **5** by application of the Horeau procedure has been revised by application of the Mosher procedure.

Among the array of novel bioactive metabolites reported from marine sponges are the norsesterterpene cyclic peroxides. To date such metabolites are restricted to only three genera, *Sigmosceptrella*, *Latrunculia*, and *Mycale*. In this regard Australian *Mycale* spp. have been particularly productive, yielding the norsesterterpene cyclic peroxides **1**−**3** from *Mycale ancorina*,[1,2] **4** and **5**[3] from *Mycale (Carmia)* cf. *spongiosa*,[4] and **1**−**3**, **6**, and **7** from an unidentified *Mycale* sp.[5] A Thai *Mycale* sp. returned the closely related compounds **8** and **9** (Chart 1).[6] In this report we revisit the known norsesterterpene **5**, to which we assign[3] the trivial name mycaperoxide F methyl ester, as well as describe the isolation and structure elucidation of the new cyclic peroxide mycaperoxide G methyl ester (**10**). We also take this opportunity to describe a new norterpene ketone **11** closely related to **5**.

A *Mycale* sp. (F77045) collected by hand (scuba) from Durras on the mid-south coast of New South Wales, Australia, yielded the known norsesterterpene **5** and the new norterpene ketone **11**. A second *Mycale* sp. (F77046) collected by trawling in the Great Australian Bight, several thousand kilometers to the west, yielded the new cyclic peroxide **10**. The crude ethanol extracts of both sponges were decanted, concentrated under reduced pressure, and methylated with CH_2N_2 prior to chromatographic fractionation. Because the ^{1}H NMR spectra of the crude *Mycale* sp. extracts did not indicate methyl esters, the natural products **5** and **10** are carboxylic acids. The characterization and structure elucidation of **5**, **10**, and **11** are described below.

Mycaperoxide F methyl ester (**5**) was isolated as a stable, colorless, optically active oil, that possessed spectroscopic data identical to that of an authentic sample.[4] Although the original report of **5** assigned absolute stereochemistry to the C-1−C-6 cyclic peroxide terminus, it did not comment on the absolute or even relative configuration of the Decalin ring system. Though our re-isolation of **5** provided an opportunity to reexamine this problem, repeated attempts to secure crystals of **5** for single crystal X-ray analysis were unsuccessful. Furthermore, 2D NMR analysis (NOESY, COSY, HMBC, HMQC) did not succeed in unambiguously defining the relative configuration of the Decalin ring system. Mosher analysis[7] of **5** did, however, overturn the earlier *tentative* 2*R*,3*R*,6*S* assignment of absolute stereochemistry.[4] To this end **5** was hydrogenated to yield the diol **12**, which was in turn converted to the (*R*)-MTPA ester **13** and the (*S*)-MTPA ester **14**. Diagnostic NMR chemical shift differences between these MTPA diastereomers [$\Delta\ \delta S$-δR; 2-CH_3 (+40 Hz), 2-H (+8 Hz), 6-CH_3 (−28 Hz)] confirmed a 3*S* and hence 2*S*,3*S*,6*R* absolute stereochemistry. That the earlier *tentative* assignment of absolute stereochemistry to **5** was incorrect is not altogether surprising given the very low optical yield reported in the Horeau analysis. This reversal of assignment serves to highlight the need to both calculate and take heed of the optical yield when applying the Horeau procedure.[4]

The ketone **11** possessed a molecular formula ($C_{18}H_{32}O_2$, Δ mmu −0.3) requiring three double bond equivalents. Examination of the ^{1}H NMR spectrum of **11** revealed a methyl ketone resonance (δ 2.15) reminiscent of the known norketones **15** and **16**, previously isolated as co-metabolites with **1**−**3**, **6**, and **7**.[5] Comparison of the NMR data (see Table 1) between **5** and **11** confirmed a common bicyclic moiety. The norterpene ketone **11** would appear to be related to the norsesterterpene cyclic peroxide **5** in the same way that the norterpene ketones **15** and **16** are related to the norsesterterpene cyclic peroxides **1** and **2**, respectively.

Mycaperoxide G methyl ester (**10**) possessed a molecular formula (M^+−2H, Δ mmu 0.4) requiring five double-bond equivalents. A comparison of NMR data between **5** and **10** (Tables 1 and 2) revealed a common cyclic peroxide moiety, but different bicyclic subunits. The molecular formula of **10** suggested the absence of the tertiary alcohol functionality. Furthermore, the C-10 secondary methyl (δ 0.80) in **5** was replaced by an olefinic methyl (δ 1.54) in **10**, which, along with the lack of a C-11 olefinic methine, suggested a $\Delta^{9,10}$ double bond. The ^{13}C NMR data for the bicyclic portion of **10** correlates well with that of the known marine natural

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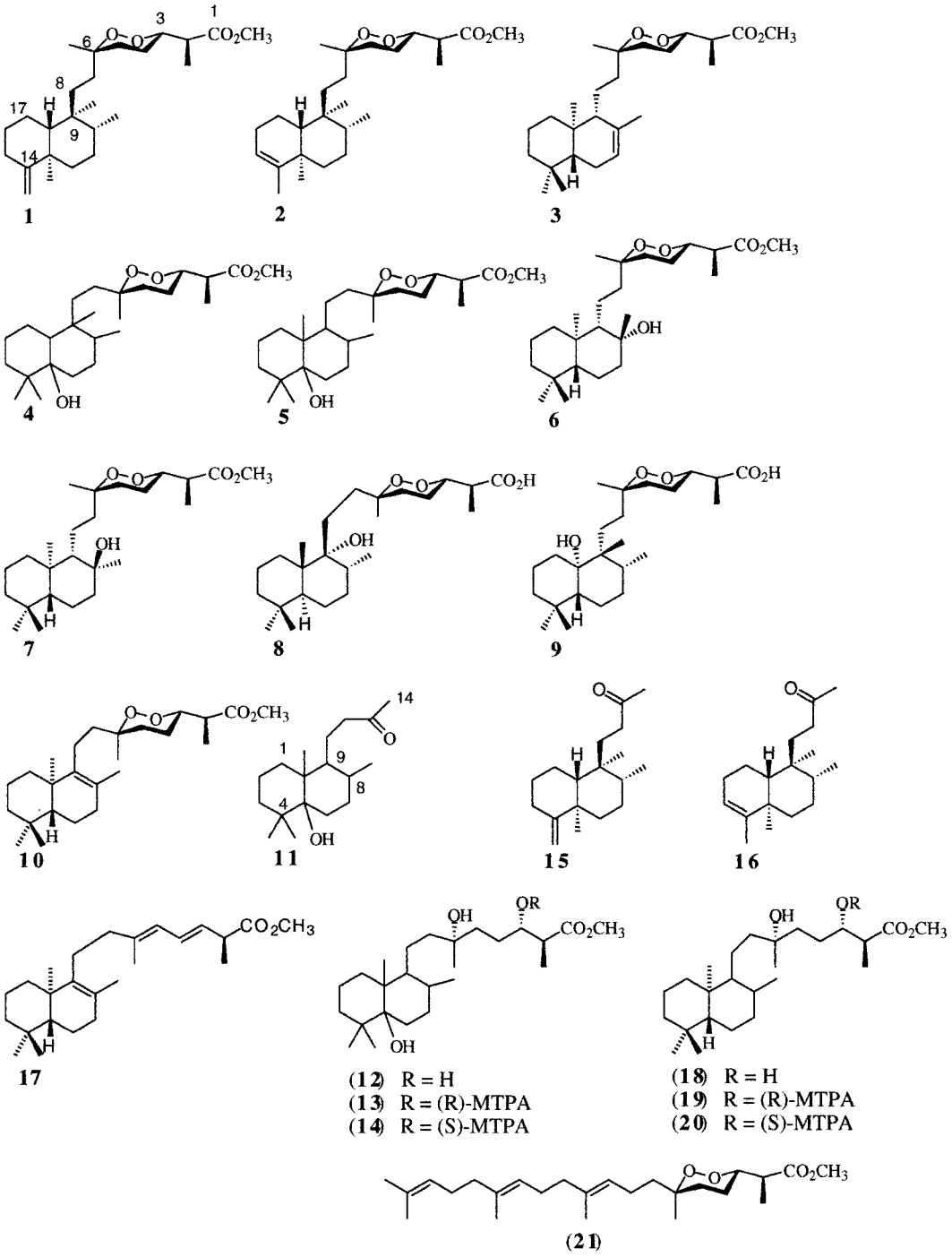
S0163-3864(97)00484-9 CCC: $15.00
Published on Web 04/08/1998

Chart 1



product **17** (Table 2) also isolated from an Australian sponge,[8] effectively defining the relative stereochemistry about this substructural unit. Hence, the gross structure for **10** can be assigned as shown.

The absolute stereochemistry about C-3 in **10** was experimentally established by application of the advanced Mosher procedure. To this end, **10** was hydrogenated to yield the diol **18**, which was in turn converted to the (*R*)-MTPA ester **19** and the (*S*)-MTPA ester **20**. Diagnostic NMR chemical shift differences ($\Delta\,\delta S - \delta R$; 2-$CH_3$ (+48 Hz), 2-H (+8 Hz), 6-CH_3 (−12 Hz)) confirmed a 3*S* and hence 2*S*,3*S*,6*R* absolute stereochemistry.

The experimentally measured $[\Phi]_D$ for **10** is −223. Literature analysis[9] has established the molar rotation contributed by the bicyclic unit to be either +191 (13*S*,18*S*) or −191 (13*R*,18*R*). Although no literature reports exist that define the molar rotation contribution due to the cyclic peroxide terminus in **10**, we are aware[10] of the new norsesterterpene cyclic peroxide **21**, which possesses this structural feature as the only chiral unit. Compound **21** was isolated as a minor metabolite along with several analogues from a southern Australian *Sigmosceptrella* sp. The $[\Phi]_D$ for **21** was measured at −66. This being the case, the *most likely* absolute stereochemistry for **10** is that derived from a 13*R*,18*R* bicyclic unit (−191) connected to the 2*S*,3*S*,6*R* cyclic peroxide unit (−66).

Compounds **1**−**10** are most likely derived from a familiar cyclization event on a common acyclic precur-

Table 1. ^{13}C NMR (100 MHz, $CDCl_3$) Data for **5**, **11**,[a] **10** and Selected Resonances for **17**[a]

carbon	5	11	10	17
1	174.5		174.5	
2	42.6		42.7	
3	81.7		81.7	
4	22.4		22.8	
5	32.1		32.2	
6	81.1	211.6	80.4	
7	38.1	39.6	40.4	
8	21.9	22.4	53.4	
9	41.5	41.7	139.7	140.1
10	36.1	35.9	126.0	126.0
11	25.5[b]	25.4[b]	33.6	33.6
12	27.2[b]	27.2[b]	19.0	19.1
13	77.2	77.4	51.9	51.9
14	37.9[c]	38.0[c]	33.0	33.3
15	27.2[b]	27.1[b]	41.7	41.8
16	27.7[b]	28.4[b]	19.0	19.1
17	34.0[b]	37.9[b]	36.9	37.0
18	39.2[c]	39.3[c]	39.1	39.0
CO_2CH_3	51.7		51.9	
2-CH_3	12.8		12.8	
6-CH_3	20.4	29.9	20.1	
14-CH_2				
9-CH_3				
10-CH_3	27.2[d]	27.2[d]	19.4	19.5
13-CH_3				
14-CH_3	22.0[d]	21.8[d]	21.7	21.7
	24.7[d]	24.7[d]	33.0	33.3
18-CH_3	15.7	15.7	21.1	20.1

[a] For comparison purposes the numbering for **11** in this table are as for **5** *a*, *b*, *c*, shifts may be exchanged within a column. Assignments supported by DEPT 135° and 90°, COSY and HMQC NMR experiments.

Table 2. Selected ^1H NMR (400 MHz, $CDCl_3$) Data for **5** and **10**

proton	5	10
2-H	2.66	2.57
3-H	4.22	4.23
CO_2CH_3	3.70	3.70
2-CH_3	1.14	1.15
6-CH_3	1.26	1.34

sor, with the variety of bicyclic units arising from the quenching of a range of intermediate carbocations, either through addition of H_2O or loss of a proton. Although such bicyclic units are common to a large array of terrestrial labdane and clerodane diterpenes, it is noteworthy that the hydroxylated bicyclic subunit in **4**, and those in **5** and **11**, are unprecedented. It would appear that a synthetic approach may be required to define unambiguously the stereochemistry about this unusual system.

Experimental Section

General Methods. See Rochfort and Capon.[11] ESIMS were acquired on a Micromass Quattro II mass spectrometer at varying cone voltages using a 50% MeCN−H_2O matrix. HRESIMS were run on a Bruker BioApex 47E FT mass spectrometer using a 50% MeCN−H_2O matrix.

Collection, Extraction, and Isolation. A *Mycale* sp. (307 g dry wt, Museum of Victoria registry no. F77045) was collected by scuba (−20 m) off Durras on the mid-south coast of NSW, Australia, and was immersed in EtOH and stored at −20 °C. [This specimen was massive lobate in growth form; beige in EtOH; highly compressible in texture and easily torn, with mucus present; oscules were not obvious; the surface was membranous and transparent with choanosomal fibers protruding; spicules included subtylostyles (fusiform, straight, length 220−246−260 μm); anisochelae (length 20−25−27.5 μm); sigmas (*c*-shaped, fusiform, length 80−95 μm); the ectosome was a contiuous palisade of pulmose bundles of subtylostyles; the choanosome consisted of meandering multispicular tracts of 10−20 subtylostyles becoming whispy and pulmose in the subectosomal region; sections of very thick fibers (2-mm diameter) cored by megascleres and stained red-brown are seen deep in the choanosome; interstitial collagen was abundant, granular, and filled with scattered megascleres and microscleres.] The decanted extract was concentrated under reduced pressure, methylated with ethereal diazomethane, and partitioned into CH_2Cl_2-soluble and CH_2Cl_2-insoluble material, the former being subjected to rapid silica filtration (petroleum spirits to EtOAc; 10% stepwise gradient). Fractions of interest were identified and further purified by HPLC (2 mL min^{-1} MeOH through a Phenomenex 5μ C_{18} 250 × 10 mm column) to yield the cyclic peroxide methyl ester **5** (80 mg, 0.026%) and the ketone **11** (8 mg, 0.003%).

A *Mycale* sp. (41 g dry wt, Museum of Victoria registry no. F77046) was collected by epibenthic sled at a depth of approximately 45 m from the Great Australian Bight, Australia, and was frozen and then transported to the laboratory where it was immersed in EtOH and stored at −20 °C. [This specimen was a massive, erect, thickly flabelliform-lamellate growth form; sandy orange in life, beige in EtOH; texture compressible, firm, fibrous; oscules inconspicuous, sunken; surface transparent detachable skin-like covering; spicules include subtylostyles (fusiform, straight, length 173−178−185 μm); anisochelae (length 22.5−25.5−27.5 μm); the ectosome is a detachable layer of bundles of subtylostyles forming a circular mesh in section; the choanosome consists of an irregular reticulation of thick spongin fibers (approximately 150−200 μm diameter) cored by loose tracts of megascleres, algal strands, and occasional fragments of detritus; interstitial collagen is abundant, granular, and filled with scattered megascleres and microscleres.] The decanted extract was concentrated under reduced pressure, methylated with ethereal diazomethane, and partitioned into CH_2Cl_2-soluble and CH_2Cl_2-insoluble material, the former being subjected to rapid silica filtration (petroleum spirits to EtOAc; 20% stepwise gradient). Fractions of interest were identified and further purified by HPLC (2 mL min^{-1} MeOH through a Phenomenex 5μ C_{18} 250 × 10 mm column) to yield the cyclic peroxide methyl ester **10** (12 mg, 0.029%).

Mycaperoxide F methyl ester (5): spectroscopic characteristics identical with an authentic sample.[3]

Mycaperoxide G methyl ester (10): stable colorless oil; $[\alpha]_D$ −55.5° (*c* 0.23, $CHCl_3$); IR ν_{max} ($CHCl_3$) 1730 cm^{-1}; ^1H NMR ($CDCl_3$, 400 MHz) δ 0.81, 0.86, 0.91 (3s, 14_a-CH_3, 14_b-CH_3, and 18-CH_3), 1.15 (d, J = 6.2 Hz, 2-CH_3), 1.34 (s, 6-CH_3), 1.54 (s, 10-CH_3), 2.57 (m, 2-H), 3.70 (s, CO_2CH_3), 4.23 (m, 3-H); ^{13}C NMR ($CDCl_3$, 100 MHz), see Table 1; EIMS (70 eV) *m/z* 404 (M^+−2H, 0.4), 318 (6), 205 (9), 191 (86), 181 (55), 149 (100), 121 (26), 109 (20), 95 (42), 83 (21), 69 (40), 56 (21); HREIMS *m/z* 404.2930 (calcd for $C_{25}H_{44}O_5$, 404.2926).

Ketone 11: colorless oil; $[\alpha]_D$ −19° (*c* 1.1, $CHCl_3$); ^1H NMR ($CDCl_3$, 400 MHz) δ 0.82, 0.82, 1.01 (3s, 4_a-CH_3, 4_b-CH_3, and 10-CH_3), 0.82 (d, J = 5.9, 8-CH_3), 2.15 (s, 14-H_3), 2.25 (ddd, J = 4.4, 11.7, 15.7 Hz, 1H), 2.46 (ddd, J = 5.1, 11.7, 11.7 Hz, 12-H_a), 2.60 (ddd, J = 5.1, 11.0, 15.7 Hz, 12-H_b); ^{13}C NMR ($CDCl_3$, 100 MHz), see Table 1; EIMS (70 eV) *m/z* 280 (M^+,1), 262 (1), 245 (2), 209 (100), 191 (20), 165 (2), 135 (8), 125 (37), 109 (17), 95 (27), 83 (89), 69 (41), 54 (89); HREIMS found 280.2399 ($C_{18}H_{32}O_2$ requires 280.2402). The sample of **11** decomposed before an IR spectrum could be acquired.

Hydrogenation of Mycaperoxide F Methyl Ester (5). A solution of **5** (14.4 mg) in ether (10 mL) was treated with 10% palladium on carbon (10 mg) and subjected to an atmosphere of H_2 gas for 12 h. The reaction mixture was filtered through Celite to yield the diol **12** (11.1 mg, 77%) as a stable yellow oil: IR ν_{max} ($CHCl_3$) 3680, 1710 cm^{-1}; ^1H NMR ($CDCl_3$, 300 MHz); δ 0.81 (3 s, 10-CH_3, 14_a-CH_3, and 14_b-CH_3), 1.00 (s, 18-CH_3), 1.10−1.80 (m, 4-CH_2, 5-CH_2, 7-CH_2, 8-CH_2, 9-CH, 10-CH, 11-CH_2, 12-CH_2, 15-CH_2, 16-CH_2, 17-CH_2), 1.13 (s, 6-CH_3), 1.19 (d, J = 7.1 Hz, 2-CH_3), 2.57 (dq, J = 7.1, 7.1 Hz, 2-H), 3.71 (m, 3-H and $-CO_2CH_3$); EIMS (70 eV) *m/z* 426 (M^+, 0.3%), 374 (10), 303 (3), 245 (11), 209 (42), 191 (75); HRESIMS *m/z* 449.3239 (calcd for $C_{25}H_{46}O_5Na$, 449.3243).

Hydrogenation of Mycaperoxide G Methyl Ester (10). Treatment of a sample of **10** (11.7 mg) in the same way as for **5** above, afforded the saturated diol **18** (9 mg, 75%) as a stable yellow oil: IR ν_{max} ($CHCl_3$) 3670, 3630, 1730 cm^{-1}; ^1H NMR ($CDCl_3$, 300 MHz); δ 0.82, 0.87, 0.94 (3s, 14_a-CH_3, 14_b-CH_3, and 18-CH_3), 0.70−2.40 (m, 4-CH_2, 5-CH_2, 7-CH_2, 8-CH_2, 9-CH, 10-CH, 11-CH_2, 12-CH_2, 13-CH, 15-CH_2, 16-CH_2, 17-CH_2, 10-CH_3), 1.19 (d, J = 7.1 Hz, 2-CH_3), 1.20 (s, 6-CH_3), 2.56 (dq, J = 7.1, 7.1 Hz, 2-H), 3.70 (m, 6-H), 3.71 (s, $-CO_2CH_3$); EIMS (30 eV) *m/z* 392 (M^+-H_2O %), 388 (4), 375 (4), 303 (4), 282 (5), 245 (9), 204 (69); HRESIMS *m/z* 433.3268 (calcd for $C_{25}H_{46}O_4Na$, 433.3297).

Reaction of Diol 12 with (*R*)-MTPA Acid. To a solution of diol **12** (4.5 mg) in dry CH_2Cl_2 (5 mL) was added DMAP (5 mg), DCC (13 mg), and (*R*)-MTPA acid (12 mg) and the mixture allowed to stir for 15 h. The mixture was then chromatographed on a normal-phase Sep-Pak (20% gradient elution from petroleum ether to EtOAc) and subjected to normal-phase HPLC (2.0 mL/min 20% EtOAc/petroleum ether, Phenomenex 5μ silica 250 × 10 mm column) to yield the (*R*)-MTPA ester **13** (0.6 mg, 9%) as a stable clear oil: $[\alpha]_D$ −14.7° (*c* 0.23 in $CHCl_3$); IR ν_{max} ($CHCl_3$) 3690, 1730 cm^{-1}; ^1H NMR ($CDCl_3$, 400 MHz); δ 0.80 (3s, 10-CH_3, 14_a-CH_3, and 14_b-CH_3), 0.98 (s, 18-CH_3), 1.09 (s, 6-CH_3), 1.10−1.80 (m, 4-CH_2, 5-CH_2, 7-CH_2, 8-CH_2, 9-CH, 10-CH, 11-CH_2, 12-CH_2, 15-CH_2, 16-CH_2, 17-CH_2), 1.11 (d, J = 7.3 Hz, 2-CH_3), 2.85 (dq, J = 7.3, 7.3 Hz, 2-H), 3.53 (s, MTPA $-CO_2CH_3$), 3.57 (s, $-CO_2CH_3$), 5.38 (m, 3-H), 7.40 (m, H-2′, H-4′, H-6′), 7.54 (m, H-3′, H-5′); ESIMS (cone voltage 25kV) *m/z* 681 (M + K, 28%), 665 (M + Na, 100%), 660 (M + NH_4, 40%), 643 (M + H, 8%).

Reaction of Diol 12 with (*S*)-MTPA Acid. To a solution of diol **12** (5.5 mg) in dry CH_2Cl_2 (5 mL) was added DMAP (5 mg), DCC (13 mg), and (*S*)-MTPA acid (12 mg) and the mixture allowed to stir for 15 h. The mixture was then chromatographed as described above for **13** to yield the (*S*)-MTPA ester **14** (1.8 mg, 22%) as a stable clear oil: $[\alpha]_D$ −22.2° (*c* 0.33 in $CHCl_3$); IR ν_{max} ($CHCl_3$) 3690, 1740 cm^{-1}; ^1H NMR ($CDCl_3$, 400 MHz); δ 0.80 (3s, 10-CH_3, 14_a-CH_3, and 14_b-CH_3), 0.97 (s, 18-CH_3), 1.02 (s, 6-CH_3), 1.10−1.80 (m, 4-CH_2, 5-CH_2, 7-CH_2, 8-CH_2, 9-CH, 10-CH, 11-CH_2, 12-CH_2, 15-CH_2, 16-CH_2, 17-CH_2), 1.19 (d, J = 7.2 Hz, 2-CH_3), 2.87 (dq, J = 7.2,7.2 Hz, 2-H), 3.53 (s, MTPA $-CO_2CH_3$), 3.64 (s, $-CO_2CH_3$), 5.38 (m, 3-H), 7.40 (m, H-2′, H-4′, H-6′), 7.54 (m, H-3′, H-5′); ESIMS (cone voltage 25kV) *m/z* 665 (M + Na, 100%), 660 (M + NH_4, 85%), 643 (M + H, 55%).

Reaction of Diol 18 with (*R*)-MTPA Acid. A solution of diol **18** (4.5 mg) in dry CH_2Cl_2 was treated the same way as for **12** to yield the (*R*)-MTPA ester **19** (0.9 mg, 13%); $[\alpha]_D$ −16.0° (*c* 0.08 in $CHCl_3$); IR ν_{max} ($CHCl_3$) 3690, 1730 cm^{-1}; ^1H NMR ($CDCl_3$, 400 MHz) δ 0.60−2.00 (m, 4-CH_2, 5-CH_2, 7-CH_2, 8-CH_2, 9-CH, 10-CH, 11-CH_2, 12-CH_2, 13-CH, 15-CH_2, 16-CH_2, 17-CH_2), 1.07 (d, J = 7.4 Hz, 2-CH_3), 1.11 (s, 6-CH_3), 2.85 (m, 2-H), 3.54 (s, MTPA $-CO_2CH_3$), 3.59 (s, $-CO_2CH_3$), 5.30 (m, 3-H), 7.40 (m, H-2′, H-4′, H-6′), 7.54 (m, H-3′, H-5′); EIMS (30 eV) *m/z* 608 (M^+-H_2O, 1%), 533 (3), 498 (13), 368 (8), 264 (7), 245 (17); ESIMS (cone voltage 25kV) *m/z* 649 (M + Na, 10%).

Reaction of Diol 18 with (*S*)-MTPA Acid. A solution of diol **18** (4.5 mg) in dry CH_2Cl_2 was treated the same way as for **12** to yield the (*S*)-MTPA ester **20** (2.1 mg, 30%): $[\alpha]_D$ −39.3° (*c* 0.21 in $CHCl_3$), IR ν_{max} ($CHCl_3$) 3690, 1740 cm^{-1}; ^1H NMR ($CDCl_3$, 400 MHz) δ 0.60−2.00 (m, 4-CH_2, 5-CH_2, 7-CH_2, 8-CH_2, 9-CH, 10-CH, 11-CH_2, 12-CH_2, 13-CH, 15-CH_2, 16-CH_2, 17-CH_2), 1.08 (s, 6-CH_3), 1.19 (d, J = 7.6 Hz, 2-CH_3), 2.87 (m, 2-H), 3.54 (s, MTPA $-CO_2CH_3$), 3.64 (s, $-CO_2CH_3$), 5.30 (m, 3-H), 7.40 (m, H-2′, H-4′, H-6′), 7.54 (m, H-3′, H-5′); ESIMS (cone voltage 25 kV) *m/z* 665 (M + K, 30%), 649 (M + Na, 10%), 644 (M + NH_4, 100%), 627 (M + H, 9%).

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NP970484L