Polyisoprenylated Benzoylphloroglucinol Derivatives from *Hypericum sampsonii*

Zhi Yong Xiao,[†] Qing Mu,*[,†] Winnie Ka Po Shiu,[‡] Yi Han Zeng,[†] and Simon Gibbons[‡]

School of Pharmacy, Fudan University, Shanghai 200032, People's Republic of China, and Centre for Pharmacognosy and Phytotherapy, The School of Pharmacy, University of London, London, United Kingdom

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Bioassay-directed fractionation using multidrug-resistant (MDR) *Staphylococcus aureus* resulted in the isolation of four new polyprenylated benzophloroglucinol derivatives, sampsoniones N–Q (**1–4**), and four known compounds, 7-epiclusianone (**5**) and sampsoniones B, L, and R, from the roots of *Hypericum sampsonii*. The structures of these compounds were established by analysis of spectroscopic data, and the structures of **4** and **5** were determined by single-crystal X-ray diffraction crystallography. In the bioassay, 7-epiclusianone (**5**) showed promising activity with a minimum inhibitory concentration (MIC) of 7.3 μM against the NorA overexpressing MDR *S. aureus* strain SA-1199B; the positive control antibiotic norfloxacin showed activity at MIC 100 μM.

Multidrug-resistant *Staphylococcus aureus* infections, particularly those caused by methicillin-resistant *S. aureus* (MRSA), have been a major threat to public health in hospitals and the community in the past decade. Despite new advances in antibiotic development, MRSA infections remain a considerable concern due to the ability of this organism to rapidly acquire resistance to new agents. In 2002, MRSA strains fully resistant to vancomycin were isolated in the United States.[1] Resistance to linezolid, a member of the oxazolidinone class has also been reported in some patients followed by prolonged antibiotic treatment in the United States.[2] There is therefore a continuing need to discover and characterize new classes of antibiotics to reduce the pressures of bacterial resistance. In the search for antibacterial compounds with activity against MDR *S. aureus* from plants, a number of species of the genus *Hypericum* have been investigated due to their ability to produce extracts with antibacterial activity toward multidrug-resistant (MDR) strains.[3]

Hypericum sampsonii Hance (Guttiferae) is a Chinese herbal medicine used in the treatment of numerous disorders such as backache, burns, diarrhea, snakebites, and swellings.[4] Because of its various bioactivities, this species has been investigated and polyprenylated benzophloroglucinol derivatives and xanthones have been isolated from this plant.[5–8] Guided by antibacterial screening using SA-1199B, an MDR strain of *S. aureus* that overexpresses the NorA efflux protein, the major characterized drug pump in *S. aureus,* the petroleum fraction of the roots from *H. sampsonii* was found to possess activity at a minimum inhibitory concentration (MIC) of 64 μg/mL. Herein, we report the phytochemical investigation on the nonpolar fraction of the root extract of *H. sampsonii*.

Results and Discussion

The powdered roots of *Hypericum sampsonii* were extracted with 95% EtOH, and the extract was fractionated into petroleum ether-, methanol-, and water-soluble fractions. The active petroleum ether-soluble fraction was rechromatographed on silica gel and RP-18 to afford four new polyisoprenylated benzophenone derivatives, denoted sampsoniones N (**1**), O (**2**), P (**3**), and Q (**4**), and four known compounds, sampsonione L, 7-epiclusianone (**5**), and sampsoniones B and R. Of these, **5** showed promising activity with an MIC of 7.3 μM (4 μg/mL) against SA-1199B, while the positive control drug norfloxacin showed activity at MIC 100 μM (32 μg/mL).

Sampsonione N (**1**) was obtained as an optically active colorless oil, $[\alpha]_D^{20}$ +22.0 (*c* 0.090, $CHCl_3$); HR-MALDI-MS of **1** indicated a molecular formula of $C_{33}H_{42}O_5$ ([M + Na]$^+$ 541.2924, calcd for $C_{33}H_{42}O_5Na^+$, 541.2915). The ^{13}C NMR spectrum of **1** (Table 1) showed signals for three carbonyls (δ_C 207.7, 193.0, 190.7), a benzoyl group (δ_C 173.2, 137.0, 128.2 × 2, 128.5 × 2, 132.7), eight methyls, four methylenes, four methines, and eight aliphatic quaternary carbons. These data, in combination with biogenetic considerations, suggested that the compound possessed a benzoylphloroglucinol structural feature.[5] The remaining 20 carbon signals were assigned to four isoprenyl moieties, and the NMR data for **1** were very similar to those of sampsonione M (**1a**), which was previously isolated from the same plant.[5] The difference between **1**and **1a** emerged at C-8, in which the substituent group was 3-methyl-2-butenyl in **1**, instead of a geranyl group in **1a**. Compound **1** was a new benzoylphloroglucinol and was named sampsonione N.

Sampsonione O (**2**), $C_{33}H_{42}O_5$, had the same molecular formula as **1**, and the ^{13}C NMR data (Table 1) of **2** were similar to those of **1** except for C-1, C-2, C-7, and C-8 (**1**: δ_C 69.0, 173.2, 190.7, and 63.5; **2**: δ_C 78.3, 188.7, 177.0, and 54.1). However, the dihydrofuran ring was formed through an oxygen atom at C-7 in **2** instead of at C-2 in **1** and was established by the presence in the HMBC

* To whom correspondence should be addressed. E-mail: muqing2000@yahoo.com.
† Fudan University, Shanghai.
‡ University of London.

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Table 1. NMR Data for Sampsoniones N (**1**) and O (**2**)

1					**2**				
no.	δH (*J* in Hz)	δC	HMBC[a]	ROESY	no.	δH (*J* in Hz)	δC[b]	HMBC[a]	ROESY
1		69.0			1		78.3		
2		173.2			2		188.7		
3					3		119.3		
4α	4.66, dd (9.0, 10.6)	93.4		5α, 5*β*, 21, 22	4α	2.81, dd (10.6, 15.3)	28.2	3, 5, 7, 20	5α
					4*β*	2.96, dd (6.7,15.3)		3, 5, 7, 20	22
5α	2.81, dd (10.6, 14.9)	26.9	2, 6, 20	4α	5α	4.79, dd (6.7, 10.6)	93.5	4, 7, 21, 22	4α, 21, 22
5*β*	2.96, dd (8.6, 14.5)		2, 4, 6, 20	4α, 21, 22					
6		118.5			6				
7		190.7			7		177.0		
8		63.5			8		54.1		
9a	2.16, dd (7.4,14.1)	40.6	7, 8, 10, 12, 23	10, 34	9a	2.18, dd (6.6, 14.1)	39.4	7, 8, 10, 11, 23	10, 23, 34
9b	2.12, dd (0.8,14.1)			10	9b	2.22, dd (1.2, 14.1)		7, 8, 10, 23	10, 23
10	1.50, m	48.5	1, 8, 9, 11, 28	9a, 9b, 28	10	1.53, m	48.8	1, 8, 9, 11, 28, 33, 34	9, 34
11		48.9			11		49.9		
12		207.7			12		207.7		
13		193.0			13		193.8		
14		137.0			14		137.2		
15	7.57, m	128.2	13, 17, 19	16	15	7.56, dd (1.1, 8.2)	128.8	13, 17, 19	16
16	7.35, t (8.2)	128.5	14, 18	15, 17	16	7.26, dt (1.6,2.0,9.0)	128.6	14, 18	15, 17
17	7.49, t (7.4)	132.7	15, 19	16, 18	17	7.39, m	132.8	15, 19	16, 18
18	7.35, t (8.2)	128.5	14, 16	17, 19	18	7.26, dt (1.6,2.0,9.0)	128.6	14, 16	17, 19
19	7.57, m	128.2	13, 15, 17	18	19	7.56, dd (1.1, 8.2)	128.8	13, 15, 17	18
20		70.7			20		72.5		
21	0.89, s	26.3	4, 20, 22	5*β*, 22, 31	21	1.31, s	26.5	5, 20, 22	5α, 22, 31
22	0.88, s	23.7	4, 20, 21	5*β*, 21	22	1.22, s	23.8	5, 20, 21	4*β*, 5α, 21, 31
23a	2.60, dd (6.7, 14.1)	29.8	7, 8, 9, 12, 24, 25	27	23	2.53, d (6.6)	30.1	7, 8, 9, 12, 24, 25	23, 27
23b	2.49, dd (7.8, 14.4)								
24	5.08, t (7.4)	119.2	23, 26, 27		24	5.03, t (6.6)	120.8	23, 26, 27	
25		134.8			25		135.6		
26	1.68, s	26.1	24, 25, 27		26	1.70, s	26.6	24, 25, 27	
27	1.71, s	18.2	24, 25, 26	23	27	1.70, s	18.8	24, 25, 26	
28a	2.24, br, d	29.0	10, 30	10, 33	28a	2.25, br, d	30.5	9, 10, 29, 30	33
28b	2.0, m			10	28b	1.95, m		10, 29, 30	
29	4.89, t (7.04)	124.6	28, 31, 32		29	4.95, t (7.0)	125.4	28, 31, 32	31
30		132.8			30		133.0		
31	1.67, s	25.8	30, 32	21	31	1.70, s	26.4	29, 30, 32	21, 22, 32
32	1.55, s	17.9	30, 31		32	1.58, s	18.5	29, 30, 31	31
33	1.48, s	23.5	1, 10, 11, 34	10, 28	33	1.49, s	23.2	1, 10, 11, 34	28a, 34
34	1.43, s	26.7	1, 10, 11, 33	9a, 10	34	1.33, s	27.3	1, 10, 11, 33	9a, 33

[a] Carbons that correlate with the proton resonance.

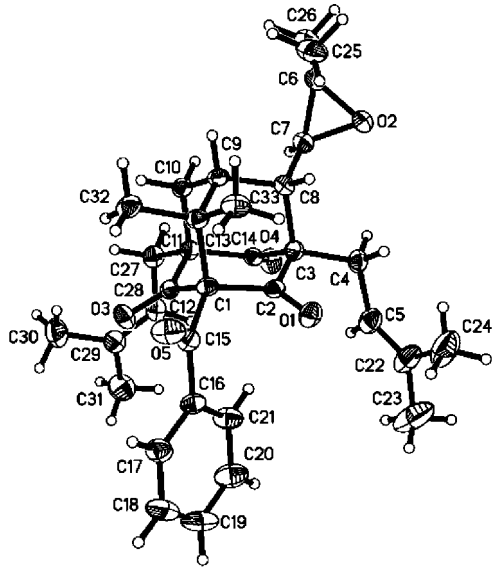

Figure 1. X-ray crystallography of sampsonione Q (**4**).

spectrum of a correlation between the methine proton of C-5 and the quaternary carbon at C-7 (177.0) of **2**. This assignment was supported by the presence of the corresponding correlation between H-23 and C-7. The structure for **2** is shown in Figure 1 and was confirmed by the ^{1}H–^{1}H COSY, HMQC, HMBC, and ROESY spectra, and compound **2** was named sampsonione O.

Sampsonione P (**3**) had a molecular formula of $C_{33}H_{42}O_5$ on the basis of HR-MALDI-MS ([M + Na]$^+$ 541.2924). The ^{13}C NMR and ^{1}H NMR data of **3** (Table 2) were compared with those of the known compound sampsonione L.[5] 2D NMR data also suggested that **3** and sampsonione L had the same skeleton and differed only with regard to the side chains attached to C-1 and C-3. Compound **3** possessed a 3-methyl-2-butenyl group and a benzoyl moiety. In the HMBC spectrum, the proton signals at δ_H 2.68 (H-13a) and 2.48 (H-13b) were correlated with the carbon signals at δ_C 68.5 (C-1), 194.0 (C-2), and 206.1 (C-12). In the ROESY spectrum, the proton signals at δ_H 2.68 (H-13a) and 2.48 (H-13b) were correlated with those at δ_H 1.20 (H-33) and 1.05 (H-34). These correlations suggested that the 3-methyl-2-butenyl group was connected to C-1 and the benzoyl group to C-3 in **3**, rather than the former connected to C-3 and the latter to C-1 as seen in sampsonione L.[5] Therefore, the structure of **3** was assigned as shown in Figure 1 and given the trivial name sampsonione P.

Sampsonione Q (**4**) was obtained as fine, colorless crystals, $[\alpha]_D^{20}$ −9.65 (*c* 0.401, $CHCl_3$); HR-MALDI-MS indicated a molecular formula of $C_{33}H_{40}O_5$ ([M + Na]$^+$ 539.2768, calcd for $C_{33}H_{40}O_5Na^+$, 539.2769). The analysis of 1D and 2D NMR spectra revealed that **4** was closely related to the adamantyl derivative sampsonione J, previously isolated from *H. sampsonii*.[5] The difference was in the side chain at C-11, with a geranyl group in sampsonione J being replaced by the 3-methyl-2-butenyl group in **4**. The α-configuration of H-8 in **4** was confirmed by the *W*-coupling between δ_H 2.51 (dt, *J* = 2.7, 5.5, 8.2, H-8) and 2.63 (dt, *J* = 2.7, 5.9, 14.1, H-10a), as well as an NOE interaction of H-8 with the C-32 methyl protons. Therefore, the structure of **4** was assigned as shown and named sampsonione Q, and the structure was confirmed by X-ray crystallography (Figure 1).

Compound **5** was isolated as colorless crystals, mp 98 °C, $[\alpha]_D^{20}$ −9.65 (*c* 0.401, $CHCl_3$) and existed as a mixture of 1,3-ene-one

Table 2. NMR Data for Sampsoniones P (**3**) and Q (**4**)

	3					**4**			
no.	δH (*J* in Hz)	δC	HMBC[a]	ROESY	no.	δH (*J* in Hz)	δC[b]	HMBC[a]	ROESY
1		68.5			1		81.8		
2		194.0			2		200.5		
3		116.6			3		72.5		
4		176.2			4a	2.82, dd (6.3, 15.3)	26.8	2, 3, 5, 22	24
					4b	2.44, dd (7.4, 15.3)		2, 3, 5, 8, 14, 22	24
5					5	4.96, br, t (7.1)	118.6	23, 24	23
6β	4.54, dd (5.4, 10.9)	91.5		9b, 26, 27	6		56.9		
7α	2.74, dd (10.9, 12.9)	30.6	6, 8, 9, 12, 25	7β, 26, 27					25
7β	1.78, dd (5.4, 12.9)		4, 8	7α, 9a	7β	2.69, d (8.6)	61.4	3, 8, 25	25
8		58.7			8α	2.51, dt (2.7, 5.5, 8.2)	55.8	7, 9	9, 26, 32
9a	2.12, dd (7.1, 14.5)	36.4	4, 8, 10, 28	7β, 10, 34	9	1.71, m	45.9	1, 3, 11	8, 10a, 10b
9b	2.34, d (14.5)		4, 8, 10, 11, 12, 28	6β, 10					26, 32, 33
10	1.51, m	46.2		9a, 9b, 28b, 29, 33, 34	10a	2.63, dt (2.7, 5.9, 14.1)	40.9	8, 13, 14	9, 33
					10b	2.28, dd (2.7, 14.1)		9, 11, 12, 13	9
11		47.5			11		68.7		
12		206.1			12		202.2		
13a	2.68, overlap	25.4	1, 12, 14, 15	13b, 14, 33	13		55.1		
13b	2.48, overlap		1, 2, 14, 15	13a, 14, 33, 34					
14	4.90, m	119.4		13a, 13b, 16	14		202.8		
15		135.0			15		192.8		
16	1.63, s	26.2	14, 15, 17	14	16		134.4		
17	1.60, s	18.1	14, 15, 16		17	7.16, m	129.3	15, 19, 21	18
18		191.7			18	7.27, m	127.9	16, 20	17, 19
19		137.5			19	7.42, m	132.5	17, 21	18, 20
20	7.68, dd (1.2, 8.2)	128.8	18, 22, 24	21	20	7.27, m	127.9	16, 18	19, 21
21	7.39, t (7.8)	128.5	19, 23	20, 22	21	7.16, m	129.3	15, 17, 19	20
22	7.52, t (6.3)	133.3	20, 24	21, 23	22		134.8		
23	7.39, t (7.8)	128.5	19, 21	22, 24	23	1.62, s	26.0	5, 22, 24	5, 24
24	7.68, dd (1.2, 8.2)	128.8	18, 20, 22	23	24	1.68, s	18.2	5, 22, 23	4, 23
25		70.4			25	1.33, s	24.7	7, 6, 26	7β
26	1.09, s	23.8	6, 25, 27	6β, 7α	26	1.29, s	19.1	7, 6, 25	8
27	1.10, s	26.7	6, 25, 26	6β, 7α	27	2.56, d (7.04)	27.4	10, 11, 12, 14, 28, 29	31
28a	2.21, m	29.0	29, 30	28b, 29, 33	28	5.18, br, t (7.1)	118.2	30, 31	30
28b	2.52, m			10, 28a, 29				30, 31	30
29	4.90, m	124.3		10, 28a, 28b, 31	29		135.4		
30		133.4			30	1.68, s	26.0	28, 29, 31	28, 31
31	1.69, s	25.9	29, 30, 32	29	31	1.66, s	18.1	28, 29, 30	27, 30
32	1.65, s	17.8	29, 30, 31		32	1.41, s	22.5	1, 9, 13, 33	8, 9
33	1.20, s	22.4	1, 10, 11, 34	10, 23a, 23b, 28a, 34	33	1.48, s	23.1	1, 9, 13, 32	9, 10a
34	1.05, s	26.9	1, 10, 11, 33	9a, 10, 23b, 33	34				

[a] Carbons that correlate with the proton resonance.

tautomers in the ratio of 3:2 in $CDCl_3$ solution. Its structure was confirmed as 7-epiclusianone by X-ray crystollagraphy (Figure 2).[9–12] In this experiment, **5** was isolated as the main constituent (2.8%) from 40 g of the petroleum ether residue. *S. aureus* strain SA-1199B, which is resistant to norfloxacin, overproduces the NorA MDR efflux protein, the major drug pump in *S. aureus*.[13] 7-Epiclusianone (**5**) showed promising activity against SA-1199B at an MIC of 7.3 μM (4 μg/mL), while norfloxacin showed activity at an MIC of 100 μM (32 μg/mL). Therefore, **5** was assumed to be the predominant active constituent of *H. sampsonii* root extract (256 μg/mL for the EtOH extract and 32 μg/mL for the petroleum fraction).

Other polyisoprenylated benzoylphloroglucinol derivatives were isolated and determined to be sampsonione B[5d] and sampsonione L,[5d] respectively. Also isolated was the known polyisoprenylated benzoylphloroglucinol derivative 1-benzoyl-7α-(1-hydroxy-1-methylethyl)-13,13-methyl-3,11-di(methyl-2-butenyl)tricycle[4.3.1.1^{3,11}]-undecane-2,12,14-trione,[14] which was given the trivial name sampsonione R. Absolute configurations of these compounds remain to be determined, and except for 7-epiclusianone (**5**), none of these metabolites exhibited activity against MDR *S. aureus* strain SA-1199B.

Experimental Section

General Experimental Procedures. Optical rotations were measured using a JASCO P-1020 polarimeter. IR spectra were recorded using an Avatar 360 ESP FTIR spectrophotometer and UV spectra on a Shimadzu UV-1600PC spectropolarimeter. ^{1}H and ^{13}C NMR spectra were obtained on a Varian Mercury Plus 400 MHz. Column chromatography was carried out with silica gel (10–40 μm, Merck) and ODS (C-18, 15–35 μm, Merck). Fractions obtained from column chromatography were monitored by TLC (silica gel HGF254, 10–40 μm, Yantai, Huanghai, China). ESI mass spectra were obtained on an Agilent 1100 Series LC/MSD spectrometer and HR-MALDI-MS spectra on an IonSpec 4.7 T FTMS. X-ray crystallographic analysis was carried out on a Bruker Smart Apex CCD diffractometer with graphite-monochromated Mo Kα radiation ($\lambda = 0.71073$ Å).

Plant Material. *Hypericum sampsonii* was collected from Cha Lin County in Hunan Province, China. A voucher specimen (No. HS-003) was deposited at the Natural Medicine Chemistry Laboratory of the School of Pharmacy, Fudan University. The plant was identified by Dr. Zhang Wen-Ju, Associate Professor in the Center of Biodiversity of the Biology School, Fudan University, China.

Extraction and Isolation. Powdered roots of the plant (1.1 kg) were extracted with 95% EtOH and afforded 90 g of extract after evaporation under vacuum at 45 °C. The extract was partitioned into petroleum ether- (40 g), methanol- (13 g), and water-soluble fractions.

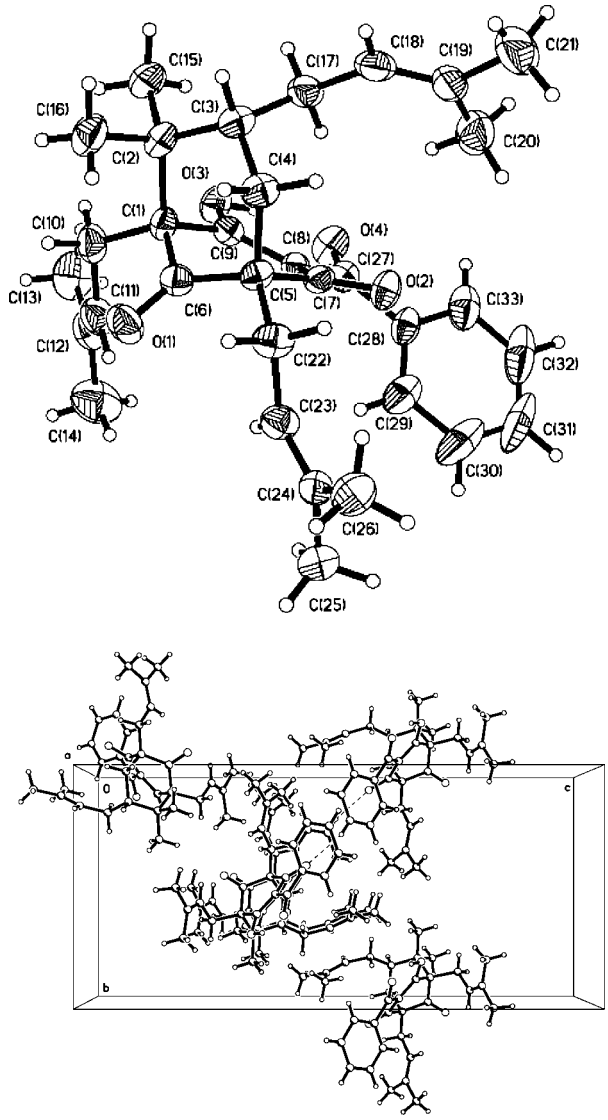

Figure 2. X-ray crystallography of 7-epiclusianone (**5**).

The petroleum ether-soluble fraction was subjected to column chromatography over silica gel, eluting with a gradient from petroleum ether to ethyl acetate and finally washed with methanol to afford 15 fractions (1–15). Fraction 1 (8.9g) was recrystallized from acetone to give **5** (1.1 g). Fraction 3 was chromatographed on silica gel columns eluted with petroleum ether–ethyl acetate to yield **4** (39.3 mg) and sampsonione B (19.4 mg). Fraction 8 was chromatographed on silica gel (petroleum ether–chloroform–acetone) and ODS (MeOH–H_2O) to yield **1** (9.7 mg), **2** (12.9 mg), **3** (2.4 mg), sampsonione L (3.9 mg), and sampsonione R (15.3 mg).

Sampsonione N (1): colorless oil; $[\alpha]_D^{20}$ +22.0 (*c* 0.090, $CHCl_3$); UV ($CHCl_3$) λ_{max} (log ε) 278 (3.88), 246 (3.99) nm; IR (film) ν 3434, 3064, 2965, 2925, 1724, 1699, 1655, 1632, 1600, 1580, 1446 cm^{-1}; ^1H NMR (400 MHz) and ^{13}C NMR ($CDCl_3$, 100 MHz) data, see Table 1; HR-MALDI-MS $[M + Na]^+$ 541.29245 (calcd for $C_{33}H_{42}O_5Na^+$, 541.2915).

Sampsonione O (2): colorless oil; $[\alpha]_D^{20}$ +87.9 (*c* 0.073, $CHCl_3$); UV ($CHCl_3$) λ_{max} (log ε) 284 (4.02), 248 (4.08), 216 (3.67) nm; IR (film) ν_{max} 3468, 3052, 2970, 2926, 2848, 1725, 1698, 1626, 1613, 1446 cm^{-1}; ^1H NMR (400 MHz) and ^{13}C NMR ($CDCl_3$, 100 MHz) data, see Table 1; HR-MALDI-MS $[M + Na]^+$ 541.29245 (calcd for $C_{33}H_{42}O_5Na^+$, 541.2930).

Sampsonione P (3): colorless oil; $[\alpha]_D^{20}$ +18.6 (*c* 0.022, $CHCl_3$); UV ($CHCl_3$) λ_{max} (log ε) 249 (4.08) nm; IR (film) ν_{max} 3479, 3056, 2969, 2924, 2851, 1732, 1682, 1631, 1448 cm^{-1}; ^1H NMR (400 MHz) and ^{13}C NMR ($CDCl_3$, 100 MHz) data, see Table 2; HR-MALDI-MS $[M + Na]^+$ 541.29245 (calcd for $C_{33}H_{42}O_5Na^+$, 541.2915).

Sampsonione Q (4): yellow oil; $[\alpha]_D^{20}$ –9.65 (*c* 0.401, $CHCl_3$); UV ($CHCl_3$) λ_{max} (log ε) 248 (3.98), 206 (3.27) nm; IR (film) ν_{max} 3055, 2962, 2923, 2851, 1745, 1704, 1597, 1582, 1447 cm^{-1}; ^1H NMR (400 MHz) and ^{13}C NMR ($CDCl_3$, 100 MHz) data, see Table 2; HR-MALDI-MS $[M + Na]^+$ 539.27680 (calcd for $C_{33}H_{40}O_5Na^+$, 539.2769).

X-ray Crystal Data for 4. Crystal data were as follows: colorless, fine crystal, C_{33} H_{40} O_5, fw 516.65, monoclinic, crystal size 0.15 × 0.12 × 0.05 mm, space group *P*2(1), a = 11.183(5) Å, b = 10.895(5) Å, c = 11.768(5) Å, V = 1427.1(11) $Å^3$, Z = 2, D_{calcd} = 1.202 g/cm^3, $F(000)$ = 556, reflections collected 7107, reflections unique 3266 (R_{int} = 0.0268), final R indices for $I > 2\sigma(I)$ R_1 = 0.0443, wR_2 = 0.1045, R indices for all data R_1 = 0.0561, wR_2 = 0.1104, completeness to 2θ (26.99) 99.8%, maximum transmission 0.9960, minimum transmission 0.9882. The structure was solved by direct methods using the program SHELXS. Refinement method was full-matrix least-squares on F^2, and goodness-of-fit on F^2 is 1.069. The X-ray diffraction material has also been deposited in the Cambridge Crystallographic Data Center (CCDC) as deposit no. CCDC 656236.

Bacteria. SA-1199B is a strain of *S. aureus* overproducing the NorA MDR efflux protein, the major drug pump in *S. aureus*, and was resistant to norfloxacin (MIC = 32 μg/mL). Additionally, some of this resistance is a result of a GrlA subunit substitution known to correlate with diminished fluoroquinolone susceptibility.[13]

Minimum Inhibitory Concentration (MIC) Assay. Bacteria were cultured on nutrient agar (Oxoid) and incubated for 24 h at 37 °C prior to MIC determination. The control antibiotic norflorxacin was obtained from Sigma Chemical Co. Mueller-Hinton broth (MHB; Oxoid) was adjusted to contain 20 and 10 mg/L of Ca^{2+} and Mg^{2+}, respectively. An inoculum density of 5 × 10^5 CFU of *S. aureus* was prepared in normal saline (9 g/L) by comparison with a 0.5 MacFarland turbidity standard. The inoculum (125 μL) was added to all wells, and the microtiter plate was incubated at 37 °C for 18 h. For MIC determination, 20 μL of a 5 mg/mL methanolic solution of 3-[4,5-dimethylthiazol-2-yl]-2,5-diphenyltetrazolium bromide (MTT; Sigma) was added to each of the wells and incubated for 20 min. Bacterial growth was indicated by a color change from yellow to dark blue. The MIC was recorded as the lowest concentration at which no growth was observed.[15]

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Supporting Information Available: NMR spectra of sampsoniones N–Q (**1**–**4**) and X-ray diffraction parameters of compound **5**. This material is available free of charge via the Internet at http://pubs.acs.org.

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