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Analytical Data and References of Compounds from Table 1

Entry #	¹ H-NMR ^a (300 MHz, δ [ppm])	¹³ C-NMR ^a (75.5 MHz, δ [ppm])	IR (film, ν [cm ⁻¹])	MS (m/z, rel. intensity)	Elemental Analysis
1 ¹	δ = 2.66-2.81 (m, 2 H), 3.25 (d, ³ J = 3.5 Hz, OH), 3.71 (s, 3 H), 5.12 (m, 1 H), 7.25-7.37 (m, 5 H)	δ = 43.6, 52.3, 70.6, 126.1, 128.3, 129.0, 142.9, 173.2			
2 ²	δ = 1.26 (t, ³ J = 7.1 Hz, 3 H), 2.70 (m, 2 H), 3.28 (d, ³ J = 2.1 Hz, 1H, OH), 4.19 (q, ³ J = 7.1 Hz, 2 H), 5.13 (m, 1 H), 7.27-7.39 (m, 5 H)				
3 ³					
4	δ = 1.00 (t, ³ J = 14.6 Hz, 3 H), 1.5 (m, 2 H), 2.48 (s, 1 H, OH), 3.52 (dd, ³ J = 9.3 Hz, ³ J = 2.1 Hz, 1 H), 3.70 (s, 3 H)	δ = 11.6, 20.8, 22.6, 25.0, 47.6, 52.3, 78.8, 178.6	3680-3100, 2960, 1870, 1730, 1465, 1435	(GC/EI) 161 (1.0, [M+H] ⁺), 131 (13.0, [M-C ₂ H ₅] ⁺), 102 (100.0, [M-C ₂ H ₃ O ₂] ⁺), 87 (30.8, [M-C ₄ H ₉ O] ⁺)	Calcd. for C ₈ H ₁₆ O ₃ (160.2): C: 60.0 H: 10.1 Found: C: 60.2 H: 9.9
5 ⁴	δ = 0.82 (d, ³ J = 6.7 Hz, 3 H), 0.97 (d, ³ J = 6.9 Hz, 3 H), 1.19 (s, 3 H), 1.38 (s, 3 H), 1.80 - 1.88 (m, 1 H), 2.88 (d, ³ J = 8.6 Hz, 1H, OH), 3.40 (dd, ³ J = 8.6 Hz, ³ J = 3.6 Hz), 3.69 (s, 3 H)	δ = 16.2, 21.7, 22.4, 23.8, 29.4, 46.0, 51.9, 81.7, 178.6			

6 ⁵					
7	$\delta = 1.27$ (s, 3 H), 1.28 (s, 3 H), 2.24 (d, $^3J = 5.3$ Hz, 1 H, 1H, OH), 2.54 (dd, $^2J = 13.6$ Hz, $^3J = 10.3$ Hz, 1 H), 2.81 (dd, $^2J = 13.6$ Hz, $^3J = 2.1$ Hz, 1 H), 3.69 (s, 3 H), 3.92 (ddd, $^3J = 10.3$ Hz, $^3J = 5.3$ Hz, $^3J = 2.1$ Hz, 1 H), 7.19-7.33 (m, 5 H)	$\delta = 20.7, 21.8, 38.4,$ $47.2, 52.0, 126.4,$ $128.5, 129.3, 129.7,$ $139.1, 177.7$ (see also copy of spectrum)	3620-3400, 3030, 2980, 2950, 1720, 1495, 1470, 1453, 1435	(GC/EI): 223 (3.6, [M+H] ⁺), 204 (26.6, [M-H ₂ O] ⁺), 145 (31.7, [204-C ₂ H ₃ O ₂] ⁺), 131 (100.0, [M-C ₇ H ₇] ⁺), HRMS calcd. for C ₁₃ H ₁₉ O ₃ ⁺ : [M+H ⁺] 223.1334, Found: 223.1287	Calcd for C ₁₃ H ₁₈ O ₃ (222.3): C: 70.3 H: 8.2 Found: C: 71.1 H: 7.9

^a All NMR spectra were recorded in CDCl₃ with tetramethylsilane as internal standard

- (1) Miller, R. E.; Nord, F. F. *J. Org. Chem.* **1951**, *16*, 728.
- (2) Maruoka, K.; Hashimoto, S.; Kitagawa, Y.; Yamamoto, H.; Nozaki, H. *J. Am. Chem. Soc.* **1977**, *99*, 7705.
- (3) Orsini, F.; Pelizzoni, F.; Ricca, G. *Tetrahedron Lett.* **1982**, *23*, 3945.
- (4) Aben, R. W.; Scheeren, J. W. *Synthesis* **1978**, 400.
- (5) Orsini, F.; Pelizzoni, F.; Pulici, M.; Vallarino, L. M. *J. Org. Chem.* **1994**, *59*, 1.

References and selected ^1H NMR Data of Compounds from Table 2

Entry #	$^1\text{H-NMR}$ (300 MHz, δ [ppm]) ^a
<i>anti-8</i> (9, 10)¹	0.86-1.02 (m, 6 H), 1.21 (d, $^3\text{J} = 7.1$ Hz, 1 H), 1.67-1.76 (m, 1 H), 2.67 (dq, $^3\text{J} = 7.1$ Hz, $^3\text{J} = 5.7$ Hz, 1 H), 2.90 (br, OH), 3.39 (dd, $^3\text{J} = 5.7$ Hz, $^3\text{J} = 5.7$ Hz, 1 H), 3.71 (s, 3 H)
<i>syn-8</i> (9, 10)¹	0.86-1.02 (m, 6 H), 1.19 (d, $^3\text{J} = 6.9$ Hz, 1 H), 1.67-1.76 (m, 1 H), 2.67 (dq, $^3\text{J} = 7.9$ Hz, $^3\text{J} = 6.9$ Hz, 1 H), 2.90 (br, OH), 3.58 (dd, $^3\text{J} = 7.9$ Hz, $^3\text{J} = 3.8$ Hz, 1 H), 3.71 (s, 3 H)
<i>anti-11</i>²	$\delta = 1.02$ (d, $^3\text{J} = 7.2$ Hz, 3 H), 1.44 (s, 9 H), 2.71 (dd, $^3\text{J} = 8.0$ Hz, $^3\text{J} = 7.2$ Hz, 1 H), 3.21 (d, $^3\text{J} = 4.6$ Hz, 1 H, OH), 4.70 (dd, $^3\text{J} = 8.0$ Hz, $^3\text{J} = 4.6$ Hz, 1 H), 7.25-7.35 (m, 5 H)
<i>syn-11</i>²	$\delta = 1.11$ (d, $^3\text{J} = 7.1$ Hz, 3 H), 1.37 (s, 9 H), 2.70 (dd, $^3\text{J} = 7.1$ Hz, $^3\text{J} = 4.3$ Hz, 1 H), 3.10 (d, $^3\text{J} = 3.1$ Hz, 1 H, OH), 5.02 (dd, $^3\text{J} = 4.3$ Hz, $^3\text{J} = 3.1$ Hz, 1 H), 7.25-7.35 (m, 5 H)
12, 13, 14¹	
<i>anti-15</i>¹	$\delta = 0.76$ (d, $^3\text{J} = 6.3$ Hz, 3 H), 0.86 (d, $^3\text{J} = 6.7$ Hz, 3 H), 1.79 (ddd, $^3\text{J} = 7.0$ Hz, $^3\text{J} = 6.7$ Hz, $^3\text{J} = 6.3$ Hz, 1 H), 2.35 (br, OH), 2.57 (dd, $^3\text{J} = 7.0$ Hz, $^3\text{J} = 6.2$ Hz), 3.57 (s, 3 H), 4.95 (m, 1 H), 7.24-7.36 (m, 5 H)
<i>syn-15</i>¹	$\delta = 0.79$ (d, $^3\text{J} = 8.7$ Hz, 3 H), 0.88 (d, $^3\text{J} = 6.9$ Hz, 3 H), 2.12 (ddd, $^3\text{J} = 8.7$ Hz, $^3\text{J} = 6.9$ Hz, $^3\text{J} = 4.6$ Hz, 1 H), 2.35 (br, OH), 2.74 (dd, $^3\text{J} = 8.1$ Hz, $^3\text{J} = 4.6$ Hz), 3.49 (s, 3 H), 4.97 (m, 1 H), 7.24-7.36 (m, 5 H)
<i>anti-16</i>¹	$\delta = 3.18$ (d, $^3\text{J} = 4.0$ Hz, 1 H, OH), 3.67 (s, 3 H), 3.88 (d, $^3\text{J} = 9.3$ Hz, 1 H), 5.17 (dd, $^3\text{J} = 9.3$ Hz, $^3\text{J} = 4.0$ Hz, 1 H), 7.07-7.33 (m, 10 H)
<i>syn-16</i>¹	$\delta = 2.69$ (d, $^3\text{J} = 2.1$ Hz, 1 H, OH), 3.51 (s, 3 H), 3.88 (d, $^3\text{J} = 7.5$ Hz, 1 H), 5.29 (dd, $^3\text{J} = 7.5$ Hz, $^3\text{J} = 2.1$ Hz, 1 H), 7.07-7.33 (m, 10 H)

17³	
18-I (anti ?)	$\delta = 0.69$ (d, $^3J = 6.9$ Hz, 3 H), 0.90 (d, $^3J = 6.9$ Hz, 3 H), 1.64 (s, 3 H), 3.14 (d, $^3J = 6.0$ Hz, 1 H, OH), 3.66 (s, 3 H), 4.17 (dd, $^3J = 6.0$ Hz, $^3J = 4.0$ Hz, 1 H), 7.24 - 7.38 (m, 5 H)
18-II (syn ?)	$\delta = 0.77$ (d, $^3J = 6.6$ Hz, 3 H), 0.92 (d, $^3J = 6.6$ Hz, 3 H), 1.62 (s, 3 H), 1.73 - 1.80 (m, 1 H), 2.02 (d, $^3J = 4.3$ Hz, 1 H, OH), 3.65 (s, 3 H), 4.06 (dd, $^3J = 4.9$ Hz, $^3J = 4.3$ Hz, 1 H), 7.25-7.49 (m, 5 H)
19-I (anti ?)	$\delta = 1.16$ (s, 3 H), 1.19 (s, 3 H), 1.25 (d, $^3J = 7.0$ Hz, 3 H), 2.78 (d, 7.7 Hz, OH), 2.92 (dq, $^3J = 7.0$ Hz, $^3J = 7.0$ Hz, 1 H), 3.57 (s, 3 H), 3.84 (dd, $^3J = 7.7$ Hz, $^3J = 7.0$ Hz, 1 H), 7.17 - 7.28 (m, 5 H)
19-II (syn ?)	$\delta = 1.23$ (s, 3 H), 1.27 (s, 3 H), 1.33 (d, $^3J = 7.1$ Hz, 3 H), 2.87 (br, OH), 2.98 (dq, $^3J = 7.1$ Hz, $^3J = 4.2$ Hz, 1 H), 3.26 (s, 3 H), 3.68 (br, 1 H), 7.19 - 7.28 (m, 5 H)
20⁴	

^a All NMR spectra were recorded in CDCl₃ with tetramethylsilane as internal standard

- (1) Canceill, J.; Basselier, J.-J.; Jacques, J. *Bull. Soc. Chim. Fr.* **1967**, 1024.
- (2) Heathcock, C. H.; Pirrung, M. C.; Sohn, J. E. *J. Org. Chem.* **1979**, 44, 4294.
- (3) Orsini, F.; Pelizzoni, F.; Pulici, M.; Vallarino, L. M. *J. Org. Chem.* **1994**, 59, 1.
- (4) Matsumoto, T.; Hosoda, Y.; Mori, K.; Fukui, K. *Bull. Chem. Soc. Jpn.* **1972**, 45, 3156.

