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Synthesis of Novel O-Acylated-D-ribono-1,5-lactones and Structural Assignment Supported by Conventional NOESY-NMR and X-ray Analysis

Marcus M. Sá,[*,a] *Gustavo P. Silveira,*[a] *Miguel S. B. Caro*[a] *and Javier Ellena*[b]

[a]*Departamento de Química, Universidade Federal de Santa Catarina, 88040-900 Florianópolis-SC, Brazil*

[b]*Instituto de Física de São Carlos, Universidade de São Paulo, CP 369, 13560-970 São Carlos-SP, Brazil*

Table S1. Crystal data and structure refinement

Empirical formula	$C_{19}H_{15}NO_8$
Formula weight	385.32
Temperature	120(2) K
Wavelength	0.71073 Å
Crystal system	Orthorhombic
Space group	$P2_12_12_1$
Unit cell dimensions	a = 6.7160(2) Å
	b = 11.2840(3) Å
	c = 22.2160(6) Å
Volume	1683.60(8) Å^3
Z	4
Density (calculated)	1.520 Mg m^{-3}
Absorption coefficient	0.121 mm^{-1}
F(000)	800
Crystal size	0.24 x 0.20 x 0.16 mm^3
Theta range for data collection	2.57 to 25.00°
Index ranges	$-7 \le h \le 7$, $-11 \le k \le 11$, $-26 \le l \le 26$
Reflections collected	2189
Independent reflections	2189 [R(int) = 0.0000]
Completeness to theta = 25.00°	79.6 %
Refinement method	Full-matrix least-squares on F^2
Computing[a]	COLLECT, HKL Denzo and Scalepack, SHELXS-97, SHELXL-97
Data / parameters	2189 / 253
Goodness-of-fit on F^2	1.040
Final R indices [I>2σ(I)]	R1 = 0.0311, wR2 = 0.0747
R indices (all data)	R1 = 0.0345, wR2 = 0.0774
Largest diff. peak and hole	0.169 and -0.215 e.Å^{-3}

[a] Data collection, data processing, structure solution and structure refinement, respectively.

Table S2. Atomic coordinates (× 10^4) and equivalent isotropic displacement parameters (Å^2 × 10^3). U(eq) is defined as one third of the trace of the orthogonalized U_{ij} tensor

	x	y	z	U(eq)
O(1)	5647(2)	7162(1)	2651(1)	26(1)
O(2)	4197(2)	5609(2)	2237(1)	32(1)
O(3)	9482(2)	5128(1)	2520(1)	22(1)
O(4)	9289(2)	6547(1)	3232(1)	24(1)
O(5)	7295(2)	5105(1)	1503(1)	23(1)
O(6)	6061(2)	6088(1)	704(1)	29(1)
O(7)	6025(3)	-179(2)	40(1)	37(1)
O(8)	6454(3)	710(2)	-814(1)	36(1)
N(1)	6279(3)	715(2)	-266(1)	26(1)
C(1)	5593(3)	6267(2)	2250(1)	21(1)
C(2)	7395(3)	6185(2)	1837(1)	20(1)
C(3)	9337(3)	6213(2)	2189(1)	20(1)
C(4)	9280(3)	7195(2)	2674(1)	23(1)
C(5)	7395(4)	7917(2)	2654(1)	26(1)
C(6)	10142(3)	5432(2)	3110(1)	20(1)
C(7)	9400(3)	4503(2)	3540(1)	20(1)
C(8)	7385(4)	4212(2)	3549(1)	27(1)
C(9)	6714(4)	3309(2)	3917(1)	31(1)
C(10)	8033(3)	2681(2)	4273(1)	28(1)
C(11)	10026(3)	2981(2)	4276(1)	28(1)
C(12)	10712(4)	3890(2)	3908(1)	23(1)
C(13)	6554(3)	5179(2)	938(1)	20(1)
C(14)	6490(3)	3994(2)	642(1)	18(1)
C(15)	6413(3)	3972(2)	13(1)	21(1)
C(16)	6370(3)	2893(2)	-288(1)	21(1)
C(17)	6343(3)	1868(2)	50(1)	21(1)
C(18)	6364(3)	1870(2)	673(1)	23(1)
C(19)	6468(3)	2943(2)	970(1)	22(1)

*e-mail: msa@qmc.ufsc.br

Table S3. Full bond lengths [Å] and angles [°]

O(1)-C(1)	1.348(2)	O(2)-C(1)-O(1)	120.18(18)
O(1)-C(5)	1.450(3)	O(2)-C(1)-C(2)	124.80(18)
O(2)-C(1)	1.196(3)	O(1)-C(1)-C(2)	115.01(18)
O(3)-C(6)	1.426(2)	O(5)-C(2)-C(3)	108.97(16)
O(3)-C(3)	1.431(2)	O(5)-C(2)-C(1)	109.11(17)
O(4)-C(6)	1.409(3)	C(3)-C(2)-C(1)	111.68(13)
O(4)-C(4)	1.438(2)	O(3)-C(3)-C(2)	107.75(16)
O(5)-C(13)	1.353(2)	O(3)-C(3)-C(4)	104.90(12)
O(5)-C(2)	1.428(2)	C(2)-C(3)-C(4)	110.63(17)
O(6)-C(13)	1.197(3)	O(4)-C(4)-C(5)	107.74(15)
O(7)-N(1)	1.228(3)	O(4)-C(4)-C(3)	103.62(16)
O(8)-N(1)	1.2234(19)	C(5)-C(4)-C(3)	112.81(16)
N(1)-C(17)	1.479(3)	O(1)-C(5)-C(4)	111.25(17)
C(1)-C(2)	1.522(3)	O(4)-C(6)-O(3)	105.36(14)
C(2)-C(3)	1.522(3)	O(4)-C(6)-C(7)	111.47(15)
C(3)-C(4)	1.546(3)	O(3)-C(6)-C(7)	108.28(16)
C(4)-C(5)	1.506(3)	C(12)-C(7)-C(8)	119.40(19)
C(6)-C(7)	1.503(3)	C(12)-C(7)-C(6)	120.8(2)
C(7)-C(12)	1.387(3)	C(8)-C(7)-C(6)	119.76(18)
C(7)-C(8)	1.393(3)	C(9)-C(8)-C(7)	120.0(2)
C(8)-C(9)	1.382(3)	C(8)-C(9)-C(10)	120.5(2)
C(9)-C(10)	1.383(3)	C(11)-C(10)-C(9)	119.9(2)
C(10)-C(11)	1.381(3)	C(10)-C(11)-C(12)	119.9(2)
C(11)-C(12)	1.390(3)	C(7)-C(12)-C(11)	120.3(2)
C(13)-C(14)	1.491(3)	O(6)-C(13)-O(5)	123.82(18)
C(14)-C(19)	1.392(3)	O(6)-C(13)-C(14)	124.70(15)
C(14)-C(15)	1.397(2)	O(5)-C(13)-C(14)	111.45(17)
C(15)-C(16)	1.390(3)	C(19)-C(14)-C(15)	120.52(18)
C(16)-C(17)	1.379(3)	C(19)-C(14)-C(13)	122.19(14)
C(17)-C(18)	1.385(2)	C(15)-C(14)-C(13)	117.28(18)
C(18)-C(19)	1.380(3)	C(16)-C(15)-C(14)	119.80(19)
C(1)-O(1)-C(5)	117.73(15)	C(17)-C(16)-C(15)	118.25(15)
C(6)-O(3)-C(3)	106.71(15)	C(16)-C(17)-C(18)	122.88(19)
C(6)-O(4)-C(4)	106.89(13)	C(16)-C(17)-N(1)	118.69(14)
C(13)-O(5)-C(2)	116.52(15)	C(18)-C(17)-N(1)	118.43(18)
O(8)-N(1)-O(7)	124.10(17)	C(19)-C(18)-C(17)	118.65(18)
O(8)-N(1)-C(17)	118.25(17)	C(18)-C(19)-C(14)	119.85(15)
O(7)-N(1)-C(17)	117.64(14)		

Table S4. Anisotropic displacement parameters ($Å^2 \times 10^3$). The anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2}U_{11} + ... + 2 h k a^* b^* U_{12}$

	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
O(1)	28(1)	23(1)	26(1)	-3(1)	6(1)	0(1)
O(2)	27(1)	30(1)	39(1)	1(1)	3(1)	-7(1)
O(3)	30(1)	18(1)	16(1)	1(1)	-2(1)	-2(1)
O(4)	39(1)	16(1)	18(1)	0(1)	0(1)	1(1)
O(5)	33(1)	20(1)	16(1)	-3(1)	-4(1)	2(1)
O(6)	40(1)	23(1)	23(1)	0(1)	-7(1)	4(1)
O(7)	51(1)	21(1)	40(1)	-2(1)	-1(1)	1(1)
O(8)	44(1)	37(1)	26(1)	-12(1)	-2(1)	3(1)
N(1)	22(1)	27(1)	29(1)	-7(1)	-4(1)	3(1)
C(1)	22(1)	18(1)	22(1)	3(1)	-1(1)	2(1)
C(2)	28(1)	14(1)	17(1)	-1(1)	1(1)	-1(1)
C(3)	24(1)	16(1)	19(1)	2(1)	3(1)	1(1)
C(4)	29(1)	21(1)	18(1)	2(1)	1(1)	-4(1)
C(5)	35(1)	19(1)	23(1)	-1(1)	1(1)	-5(1)
C(6)	20(1)	22(1)	18(1)	0(1)	-2(1)	-1(1)
C(7)	25(1)	18(1)	17(1)	-1(1)	2(1)	1(1)
C(8)	27(1)	26(1)	28(1)	8(1)	-2(1)	3(1)
C(9)	30(1)	31(1)	33(1)	6(1)	3(1)	-3(1)
C(10)	40(2)	23(1)	22(1)	2(1)	7(1)	2(1)
C(11)	39(2)	26(1)	18(1)	1(1)	-1(1)	12(1)
C(12)	26(1)	25(1)	19(1)	-3(1)	-1(1)	5(1)
C(13)	18(1)	25(1)	18(1)	0(1)	1(1)	0(1)
C(14)	15(1)	22(1)	19(1)	-2(1)	-1(1)	3(1)
C(15)	17(1)	26(1)	20(1)	1(1)	-1(1)	-1(1)
C(16)	18(1)	28(1)	19(1)	-2(1)	-2(1)	4(1)
C(17)	13(1)	24(1)	25(1)	-5(1)	-2(1)	1(1)
C(18)	22(1)	23(1)	25(1)	1(1)	-1(1)	2(1)
C(19)	23(1)	26(1)	17(1)	0(1)	0(1)	3(1)

Table S6. Complete list of torsion angles [°]

C(5)-O(1)-C(1)-O(2)	178.85(17)
C(5)-O(1)-C(1)-C(2)	-1.3(2)
C(13)-O(5)-C(2)-C(3)	-139.83(16)
C(13)-O(5)-C(2)-C(1)	97.99(18)
O(2)-C(1)-C(2)-O(5)	-9.1(3)
O(1)-C(1)-C(2)-O(5)	171.06(15)
O(2)-C(1)-C(2)-C(3)	-129.6(2)
O(1)-C(1)-C(2)-C(3)	50.5(2)
C(6)-O(3)-C(3)-C(2)	-136.10(16)
C(6)-O(3)-C(3)-C(4)	-18.2(2)
O(5)-C(2)-C(3)-O(3)	-51.57(18)
C(1)-C(2)-C(3)-O(3)	69.04(19)
O(5)-C(2)-C(3)-C(4)	-165.71(14)
C(1)-C(2)-C(3)-C(4)	-45.1(2)
C(6)-O(4)-C(4)-C(5)	143.29(17)
C(6)-O(4)-C(4)-C(3)	23.5(2)
O(3)-C(3)-C(4)-O(4)	-3.1(2)
C(2)-C(3)-C(4)-O(4)	112.88(17)
O(3)-C(3)-C(4)-C(5)	-119.29(17)
C(2)-C(3)-C(4)-C(5)	-3.4(2)
C(1)-O(1)-C(5)-C(4)	-49.87(19)
O(4)-C(4)-C(5)-O(1)	-63.09(18)
C(3)-C(4)-C(5)-O(1)	50.66(19)
C(4)-O(4)-C(6)-O(3)	-35.8(2)
C(4)-O(4)-C(6)-C(7)	-153.08(17)
C(3)-O(3)-C(6)-O(4)	33.6(2)
C(3)-O(3)-C(6)-C(7)	152.96(16)
O(4)-C(6)-C(7)-C(12)	-120.3(2)
O(3)-C(6)-C(7)-C(12)	124.27(19)
O(4)-C(6)-C(7)-C(8)	62.8(2)
O(3)-C(6)-C(7)-C(8)	-52.6(2)
C(12)-C(7)-C(8)-C(9)	-0.9(3)
C(6)-C(7)-C(8)-C(9)	176.02(19)
C(7)-C(8)-C(9)-C(10)	-0.5(3)
C(8)-C(9)-C(10)-C(11)	1.9(3)
C(9)-C(10)-C(11)-C(12)	-1.9(3)
C(8)-C(7)-C(12)-C(11)	1.0(3)
C(6)-C(7)-C(12)-C(11)	-175.95(18)
C(10)-C(11)-C(12)-C(7)	0.4(3)
C(2)-O(5)-C(13)-O(6)	2.8(3)
C(2)-O(5)-C(13)-C(14)	-178.92(17)
O(6)-C(13)-C(14)-C(19)	-159.5(2)
O(5)-C(13)-C(14)-C(19)	22.2(3)
O(6)-C(13)-C(14)-C(15)	19.3(3)
O(5)-C(13)-C(14)-C(15)	-158.93(18)
C(19)-C(14)-C(15)-C(16)	-1.8(3)
C(13)-C(14)-C(15)-C(16)	179.40(19)
C(14)-C(15)-C(16)-C(17)	2.0(3)
C(15)-C(16)-C(17)-C(18)	-0.2(3)
C(15)-C(16)-C(17)-N(1)	179.44(18)
O(8)-N(1)-C(17)-C(16)	7.2(3)
O(7)-N(1)-C(17)-C(16)	-171.8(2)
O(8)-N(1)-C(17)-C(18)	-173.08(18)
O(7)-N(1)-C(17)-C(18)	7.9(3)
C(16)-C(17)-C(18)-C(19)	-1.7(3)
N(1)-C(17)-C(18)-C(19)	178.59(18)
C(17)-C(18)-C(19)-C(14)	1.9(3)
C(15)-C(14)-C(19)-C(18)	-0.2(3)
C(13)-C(14)-C(19)-C(18)	178.6(2)