Crystal structure of O^1,O^2,O^3,O^6-tetraacetyl-O^4-[tetra-O-acetyl-β-D-glucopyranosyl]-α-D-glucopyranose (octa-O-acetyl-α-D-cellobiose), $C_{28}H_{38}O_{19}$

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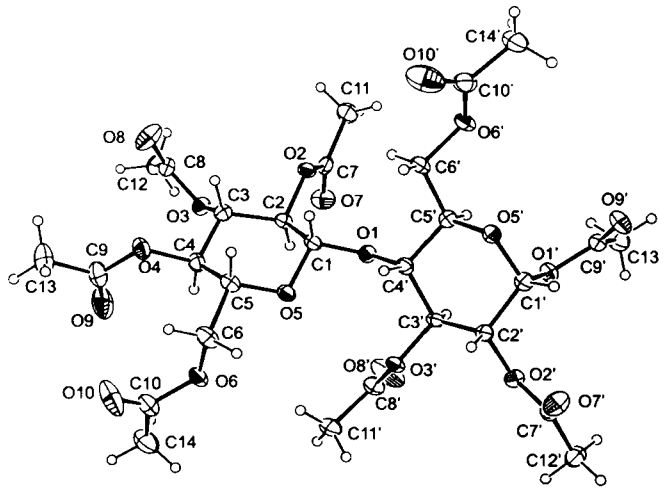

Abstract

$C_{28}H_{38}O_{19}$, orthorhombic, $P2_12_12_1$ (No. 19), a = 22.576(5) Å, b = 5.5979(8) Å, c = 26.120(6) Å, V = 3301.0 Å^3, Z = 4, $R_{gt}(F)$ = 0.038, $wR_{ref}(F^2)$ = 0.091, T = 220 K.

Source of material

Peracylated α-cellobiose (commercially available from Aldrich) reacts in acetone solution with $[PtMe_3(Me_2CO)_3]BF_4$ under complex formation. Experiments to recrystallize it from dichloromethane/*n*-hexane solution at 233 K led under cleavage of the ligand yielding well-shaped crystals of the title compound (**I**).

Discussion

Neutral carbohydrate ligands are unique, and their highly versatile coordination behavior is well documented in trimethylplatinum(IV) complexes [1]. In this respect, the solid state structure of **I** is of interest due to high conformational flexibility of the disaccharides and manifold donor sites of nearly equivalent oxygen atoms. **I** crystallizes as discrete molecules with weak intermolecular C–H···O interactions only (closest contact: –C(O)–CH_3···O=C(CH_3)–O–: d(C11···O10^{ii}) = 3.204(6) Å; symmetry code: (*i*) $x, y-1, z$). The C—C and C—O bond lengths are in the expected range [2]. Planes of acetoxy substituents include with their mean pyranose ring plane angles between 53.1(2)° and 86.9(1)°. The mean planes of the pyranosyl rings include an angle of 57.8(2)° (torsion angle H1–C1···C4′–H4′ 53(3)°). Both glycopyranosyl rings adopt a slightly distorted chair conformation as defined by the Cremer and Pople [3] puckering parameters. The O5 → C5 ring has a $^4C_1$ conformation with a total puckering amplitude Q = 0.595(4) Å and angles Θ = 11.9(4)° and Φ = 6(2)° (alternatively: q_2 = 0.123(4) Å, q_3 = 0.583(4) Å, Φ = 6(2)°). The ring O5′ → C5′ exhibits $^4C_1$ conformation as well, revealing a total puckering amplitude of Q = 0.554(4) Å and angles Θ = 5.9(4)° and Φ = 328(4)° (alternatively: q_2 = 0.056(4) Å, q_3 = 0.551(4) Å, Φ = 328(4)°). For both rings Q is slightly smaller than the Q value of an ideal cyclohexane chair (0.63 Å for d(C—C) = 1.54 Å [3]). From the Θ value, the O5′ → C5′ ring is less distorted than the O5 → C5 ring (5.9(4)° vs. 11.9(4)°). Generally, there are close similarities between the structure of the title compound and that of the corresponding β-isomer [4].

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Table 1. Data collection and handling.

Crystal:	colourless block, size 0.2 × 0.2 × 0.4 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	1.17 cm^{-1}
Diffractometer, scan mode:	Stoe IPDS, 100 frames, Δφ = 2°
$2\theta_{max}$:	50°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	17305, 3251
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 2328
$N(param)_{refined}$:	488
Programs:	SHELXS-97 [5], SHELXL-97 [6]

Table 2. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	x	y	z	U_{iso}
H(1)	4*a*	0.808(1)	1.226(7)	0.397(1)	0.021(8)
H(1′)	4*a*	0.670(1)	1.220(7)	0.200(1)	0.029(9)
H(2′)	4*a*	0.772(1)	1.206(7)	0.208(1)	0.031(9)
H(2)	4*a*	0.840(2)	0.745(9)	0.428(2)	0.05(1)
H(3′)	4*a*	0.765(1)	0.788(7)	0.270(1)	0.03(1)
H(3)	4*a*	0.858(1)	1.156(7)	0.492(1)	0.027(9)
H(4′)	4*a*	0.790(1)	1.227(8)	0.313(1)	0.029(9)
H(4)	4*a*	0.954(2)	0.948(8)	0.441(1)	0.04(1)
H(5′)	4*a*	0.682(1)	1.008(7)	0.322(1)	0.03(1)
H(5)	4*a*	0.897(2)	1.386(8)	0.406(1)	0.04(1)
H(6A)	4*a*	0.996(2)	1.419(8)	0.388(1)	0.05(1)
H(6B)	4*a*	0.961(2)	1.348(8)	0.334(2)	0.05(1)
H(6D)	4*a*	0.709(2)	1.464(9)	0.357(2)	0.06(1)
H(6E)	4*a*	0.702(2)	1.227(9)	0.394(2)	0.06(1)
H(11D)	4*a*	0.9574	0.6430	0.2501	0.056
H(11E)	4*a*	0.9537	0.8438	0.2918	0.056
H(11F)	4*a*	0.9437	0.9071	0.2340	0.056
H(11A)	4*a*	0.6725	0.5510	0.4988	0.060
H(11B)	4*a*	0.6690	0.8216	0.4836	0.060
H(11C)	4*a*	0.6989	0.7460	0.5353	0.060
H(12A)	4*a*	0.9195	0.5530	0.5746	0.049
H(12B)	4*a*	0.8567	0.5934	0.5987	0.049
H(12C)	4*a*	0.9129	0.7033	0.6250	0.049
H(12D)	4*a*	0.7459	0.8289	0.0584	0.051
H(12E)	4*a*	0.7443	0.6377	0.1022	0.051
H(12F)	4*a*	0.8047	0.7508	0.0854	0.051
H(13D)	4*a*	0.5279	0.6900	0.2378	0.065
H(13E)	4*a*	0.5898	0.5795	0.2514	0.065
H(13F)	4*a*	0.5696	0.5968	0.1941	0.065
H(13A)	4*a*	1.0902	1.3227	0.5309	0.094

Table 2. Continued.

Atom	Site	x	y	z	U_{iso}
H(13B)	4a	1.0295	1.4191	0.5513	0.094
H(13C)	4a	1.0543	1.5095	0.4987	0.094
H(14A)	4a	1.1403	0.9006	0.3645	0.077
H(14B)	4a	1.0914	0.8222	0.3253	0.077

Table 2. Continued.

Atom	Site	x	y	z	U_{iso}
H(14C)	4a	1.0846	0.7589	0.3834	0.077
H(14D)	4a	0.5269	1.7345	0.3678	0.117
H(14E)	4a	0.5231	1.5137	0.4046	0.117
H(14F)	4a	0.5248	1.4754	0.3452	0.117

Table 3. Atomic coordinates and displacement parameters (in $Å^2$).

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C(1)	4a	0.8231(1)	1.0660(8)	0.3902(1)	0.022(2)	0.036(3)	0.033(2)	0.004(2)	0.003(1)	-0.001(2)
C(1')	4a	0.6872(1)	1.1225(7)	0.2276(1)	0.031(2)	0.032(2)	0.033(2)	-0.002(2)	-0.001(2)	0.001(2)
C(2')	4a	0.7517(1)	1.0563(7)	0.2183(1)	0.029(2)	0.031(2)	0.030(2)	-0.001(2)	0.002(1)	0.001(2)
C(2)	4a	0.8298(1)	0.9124(8)	0.4381(1)	0.024(2)	0.033(3)	0.032(2)	0.004(2)	0.003(1)	0.001(2)
C(3')	4a	0.7792(1)	0.9413(8)	0.2648(1)	0.022(2)	0.036(3)	0.029(2)	0.001(2)	0.007(1)	0.000(2)
C(3)	4a	0.8746(1)	1.0210(7)	0.4748(1)	0.034(2)	0.022(2)	0.032(2)	0.005(2)	0.001(2)	0.000(2)
C(4')	4a	0.7672(1)	1.0867(8)	0.3132(1)	0.023(2)	0.033(2)	0.031(2)	-0.002(2)	0.001(1)	0.007(2)
C(4)	4a	0.9312(1)	1.0901(8)	0.4471(1)	0.027(2)	0.033(3)	0.042(2)	-0.001(2)	-0.003(2)	-0.003(2)
C(5')	4a	0.7013(2)	1.1409(8)	0.3179(1)	0.031(2)	0.036(3)	0.030(2)	-0.000(2)	0.002(2)	-0.000(2)
C(5)	4a	0.9181(1)	1.2275(8)	0.3983(1)	0.028(2)	0.035(3)	0.043(2)	0.000(2)	0.003(2)	0.005(2)
C(6)	4a	0.9723(2)	1.2942(9)	0.3674(2)	0.033(2)	0.045(3)	0.055(3)	0.000(2)	0.004(2)	0.013(2)
C(6')	4a	0.6884(2)	1.309(1)	0.3615(2)	0.024(2)	0.057(3)	0.051(3)	0.004(2)	0.001(2)	-0.018(2)
C(7')	4a	0.7554(1)	0.9739(8)	0.1289(1)	0.029(2)	0.046(3)	0.030(2)	-0.003(2)	-0.000(1)	0.005(2)
C(7)	4a	0.7506(2)	0.6822(8)	0.4739(1)	0.036(2)	0.040(3)	0.025(2)	-0.009(2)	-0.004(2)	-0.001(2)
C(8)	4a	0.8877(2)	0.8840(8)	0.5619(1)	0.035(2)	0.037(3)	0.032(2)	-0.001(2)	-0.006(2)	-0.002(2)
C(8')	4a	0.8731(2)	0.7416(8)	0.2660(2)	0.035(2)	0.038(3)	0.044(2)	0.006(2)	0.006(2)	0.001(2)
C(9')	4a	0.5958(1)	0.9135(8)	0.2230(1)	0.028(2)	0.046(3)	0.032(2)	-0.001(2)	0.003(1)	-0.004(2)
C(9)	4a	1.0200(2)	1.177(1)	0.4946(2)	0.039(2)	0.058(4)	0.057(3)	-0.001(2)	-0.014(2)	0.009(2)
C(10)	4a	1.0671(2)	1.1026(9)	0.3681(2)	0.028(2)	0.053(3)	0.058(3)	-0.004(2)	-0.002(2)	0.007(2)
C(10')	4a	0.6048(2)	1.560(1)	0.3747(2)	0.044(2)	0.079(4)	0.036(2)	0.026(3)	0.000(2)	0.005(2)
C(11')	4a	0.9377(2)	0.7880(9)	0.2599(2)	0.032(2)	0.057(3)	0.051(2)	0.011(2)	0.007(2)	-0.001(2)
C(11)	4a	0.6927(2)	0.7020(9)	0.5002(2)	0.039(2)	0.067(3)	0.045(2)	-0.012(2)	0.004(2)	0.004(2)
C(12)	4a	0.8949(2)	0.6641(7)	0.5928(1)	0.048(2)	0.040(3)	0.034(2)	-0.001(2)	-0.006(2)	0.004(2)
C(12')	4a	0.7633(2)	0.7807(8)	0.0903(1)	0.049(2)	0.043(3)	0.037(2)	0.001(2)	-0.000(2)	-0.002(2)
C(13')	4a	0.5684(2)	0.6739(8)	0.2269(2)	0.040(2)	0.047(3)	0.074(3)	-0.011(2)	0.001(2)	-0.005(2)
C(13)	4a	1.0513(2)	1.375(1)	0.5212(2)	0.060(3)	0.075(4)	0.100(4)	-0.019(3)	-0.030(3)	-0.005(3)
C(14)	4a	1.0986(2)	0.8765(9)	0.3596(2)	0.042(2)	0.059(4)	0.092(4)	0.006(2)	0.003(2)	-0.002(3)
C(14')	4a	0.5391(2)	1.572(1)	0.3729(2)	0.056(3)	0.176(7)	0.060(3)	0.061(4)	-0.002(2)	-0.015(4)
O(1)	4a	0.78616(9)	0.9446(5)	0.35611(8)	0.030(1)	0.034(2)	0.028(1)	-0.001(1)	-0.0009(9)	0.004(1)
O(1')	4a	0.65516(9)	0.9018(5)	0.23185(9)	0.026(1)	0.035(2)	0.039(1)	-0.001(1)	-0.0019(9)	0.000(1)
O(2')	4a	0.75748(9)	0.8861(5)	0.17709(8)	0.038(1)	0.032(2)	0.030(1)	0.003(1)	0.002(1)	0.002(1)
O(2)	4a	0.77353(9)	0.9013(5)	0.46373(8)	0.027(1)	0.034(2)	0.033(1)	-0.001(1)	0.0074(9)	-0.001(1)
O(3')	4a	0.84210(9)	0.9396(5)	0.25633(8)	0.022(1)	0.036(2)	0.035(1)	0.002(1)	0.0054(9)	0.004(1)
O(3)	4a	0.8873(1)	0.8332(5)	0.51159(9)	0.041(1)	0.031(2)	0.031(1)	0.005(1)	-0.002(1)	0.001(1)
O(4)	4a	0.9657(1)	1.2472(5)	0.4793(1)	0.032(1)	0.041(2)	0.056(2)	-0.004(1)	-0.007(1)	-0.006(1)
O(5)	4a	0.88022(9)	1.0837(5)	0.36719(9)	0.025(1)	0.044(2)	0.034(1)	0.001(1)	0.0036(9)	0.002(1)
O(5')	4a	0.68191(9)	1.2608(5)	0.27196(9)	0.029(1)	0.035(2)	0.036(1)	0.006(1)	-0.002(1)	-0.001(1)
O(6)	4a	1.00857(9)	1.0834(5)	0.35960(9)	0.025(1)	0.050(2)	0.051(2)	-0.003(1)	0.004(1)	-0.000(2)
O(6')	4a	0.6250(1)	1.3411(6)	0.3633(1)	0.026(1)	0.084(3)	0.067(2)	0.010(1)	0.001(1)	-0.033(2)
O(7)	4a	0.7757(1)	0.5004(6)	0.4632(1)	0.060(2)	0.035(2)	0.061(2)	-0.003(1)	0.012(1)	-0.004(2)
O(7')	4a	0.7483(1)	1.1834(6)	0.1201(1)	0.075(2)	0.038(2)	0.038(2)	0.001(2)	0.001(1)	0.008(1)
O(8)	4a	0.8816(2)	1.0832(6)	0.5786(1)	0.108(2)	0.039(2)	0.039(2)	0.004(2)	-0.009(2)	-0.004(2)
O(8')	4a	0.8521(1)	0.5550(6)	0.2786(2)	0.051(2)	0.039(2)	0.157(4)	0.009(2)	0.023(2)	0.023(2)
O(9')	4a	0.5707(1)	1.0966(6)	0.2133(1)	0.031(1)	0.045(2)	0.065(2)	0.004(1)	-0.003(1)	0.002(2)
O(9)	4a	1.0391(1)	0.9802(7)	0.4867(2)	0.059(2)	0.066(3)	0.104(3)	0.019(2)	-0.041(2)	-0.002(2)
O(10')	4a	0.6374(2)	1.7210(8)	0.3873(2)	0.080(3)	0.062(3)	0.119(3)	0.015(2)	0.034(2)	0.005(3)
O(10)	4a	1.0893(1)	1.2856(7)	0.3822(2)	0.038(2)	0.049(3)	0.158(4)	-0.001(2)	-0.021(2)	-0.001(2)

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