Crystal structure of 6,6-bis(methyl2,3-*O*-isopropylidene-6-deoxy-α-D-mannopyranoside), $C_{20}H_{34}O_{10}$

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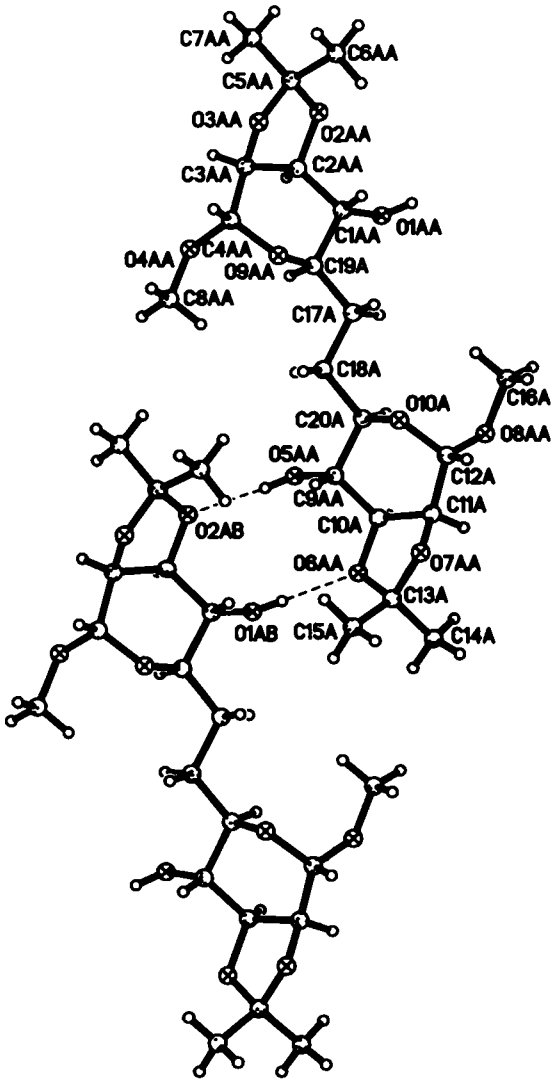

Abstract

$C_{20}H_{34}O_{10}$, monoclinic, $P12_11$ (No. 4), $a = 9.6895$ Å, $b = 23.0104$ Å, $c = 10.1973$ Å, $\beta = 92.239°$, $V = 2271.8$ Å^3, $Z = 4$, $R_{gt}(F) = 0.062$, $wR_{ref}(F^2) = 0.196$, $T = 293$ K.

Source of material

The title compound has been obtained by heating of methyl 6-deoxy-6-iodo-2,3-*O*-isopropylidene-α-D-mannopyranoside [1], Zn, NH_4Cl, vitamin B 12, and NH_2NHBoc in methanol [2,3]. The title compound was obtained after separation from the main product (2*R*/*S*,3*S*,4*S*,5*R*)-2-[-*N*-(*tert*-butoxycarbonyl)-hydrazino]-3-4-isopropylidendioxy-5-vinyltetrahydrofuran [ratio 3:1] by flash chromatographie using petroleum ether / ethyl acetate in the form of colourless crystals: mp 333 K – 335 K; $[\alpha]_{20}^{D} = +27$ (c = 0.50, in MeOH).

Discussion

The compound crystallizes with two independent molecules in the asymmetric unit. We observe a strong intermolecular hydrogen bond interaction between the hydroxy groups (O1, O5) and the oxygen atoms (O2, O6) of the dioxolan moieties. The H1A···O6A, H5A···O2A, H1B···O6B and H5B···O2B distances are 2.08 Å, 2.07 Å, 2.11Å and 2.06 Å, respectively. The bond angles of these hydrogen bonds are nearly linear (~180°).

Table 1. Data collection and handling.

Crystal:	colourless block, size 0.15 × 0.2 × 0.5 mm
Wavelength:	Cu K_α radiation (1.54178 Å)
μ:	8.56 cm^{-1}
Diffractometer, scan mode:	Siemens P4, ω
$2\theta_{max}$:	134.98°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	4876, 4195
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 3069
$N(param)_{refined}$:	546
Programs:	SHELXS-97 [4], SHELXL-97 [5]

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

Atom	Site	*x*	*y*	*z*	U_{iso}
H(1AA)	2*a*	0.6842	0.0150	1.0846	0.089
H(1A)	2*a*	0.5409	0.0767	1.0389	0.058
H(2A)	2*a*	0.7868	0.1442	1.0634	0.059
H(3A)	2*a*	0.6792	0.2294	1.0401	0.066
H(4A)	2*a*	0.4682	0.2196	0.8895	0.059
H(5A)	2*a*	0.7080	0.0677	0.3319	0.100
H(6A1)	2*a*	0.4754	0.1377	1.3956	0.157
H(6A2)	2*a*	0.4076	0.1203	1.2591	0.157
H(6A3)	2*a*	0.3692	0.1796	1.3241	0.157
H(7A1)	2*a*	0.6648	0.2156	1.4037	0.169
H(7A2)	2*a*	0.5648	0.2609	1.3352	0.169
H(7A3)	2*a*	0.7052	0.2453	1.2722	0.169
H(8A1)	2*a*	0.6579	0.2483	0.6237	0.120
H(8A2)	2*a*	0.5086	0.2528	0.6770	0.120
H(8A3)	2*a*	0.5694	0.1920	0.6408	0.120
H(9A)	2*a*	0.5224	0.0303	0.3662	0.061
H(10A)	2*a*	0.7002	–0.0663	0.3607	0.058
H(11A)	2*a*	0.5360	–0.1321	0.3768	0.061

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Table 2. Continued.

Atom	Site	x	y	z	U_{iso}
H(12A)	2a	0.3453	-0.0960	0.5148	0.071
H(14A)	2a	0.5524	-0.1285	0.0238	0.125
H(14B)	2a	0.4255	-0.1587	0.0860	0.125
H(14C)	2a	0.5673	-0.1553	0.1650	0.125
H(15A)	2a	0.4209	-0.0353	-0.0098	0.134
H(15B)	2a	0.3604	-0.0033	0.1109	0.134
H(15C)	2a	0.2879	-0.0593	0.0542	0.134
H(16A)	2a	0.5314	-0.1477	0.7849	0.156
H(16B)	2a	0.3804	-0.1322	0.7359	0.156
H(16C)	2a	0.4866	-0.0825	0.7694	0.156
H(17A)	2a	0.4909	0.0329	0.8013	0.071
H(17B)	2a	0.6459	0.0136	0.8025	0.071
H(18A)	2a	0.5070	0.0797	0.6051	0.076
H(18B)	2a	0.6687	0.0774	0.6129	0.076
H(19A)	2a	0.7099	0.1125	0.8318	0.057
H(20A)	2a	0.6536	-0.0259	0.5861	0.063
H(1BB)	2a	0.2024	0.2297	0.5032	0.096
H(1B)	2a	0.0277	0.2730	0.4419	0.057
H(2B)	2a	0.2364	0.3590	0.4673	0.057
H(3B)	2a	0.0843	0.4333	0.4531	0.066
H(4B)	2a	-0.0869	0.4144	0.2742	0.069
H(5B)	2a	0.1949	0.2782	-0.2674	0.093
H(6B1)	2a	-0.0788	0.3162	0.7810	0.113
H(6B2)	2a	-0.1185	0.2927	0.6403	0.113
H(6B3)	2a	-0.2046	0.3431	0.7008	0.113

Table 2. Continued.

Atom	Site	x	y	z	U_{iso}
H(7B1)	2a	0.0330	0.4131	0.8078	0.116
H(7B2)	2a	-0.0900	0.4431	0.7296	0.116
H(7B3)	2a	0.0612	0.4500	0.6824	0.116
H(8B1)	2a	0.1430	0.4545	0.0477	0.167
H(8B2)	2a	-0.0148	0.4553	0.0766	0.167
H(8B3)	2a	0.0581	0.3965	0.0441	0.167
H(9B)	2a	0.0427	0.2205	-0.2235	0.059
H(10B)	2a	0.2840	0.1499	-0.2251	0.058
H(11B)	2a	0.1641	0.0670	-0.2097	0.066
H(12B)	2a	-0.0347	0.0796	-0.0676	0.063
H(14D)	2a	0.1401	0.0760	-0.5721	0.155
H(14E)	2a	0.0328	0.0355	-0.5059	0.155
H(14F)	2a	0.1784	0.0460	-0.4375	0.155
H(15D)	2a	-0.0283	0.1576	-0.5815	0.131
H(15E)	2a	-0.0872	0.1809	-0.4505	0.131
H(15F)	2a	-0.1417	0.1221	-0.5107	0.131
H(16D)	2a	0.1714	0.0529	0.2026	0.121
H(16E)	2a	0.0197	0.0460	0.1457	0.121
H(16F)	2a	0.0770	0.1078	0.1829	0.121
H(17C)	2a	-0.0070	0.2286	0.2039	0.077
H(17D)	2a	0.1525	0.2174	0.2114	0.077
H(18C)	2a	-0.0020	0.2689	0.0047	0.077
H(18D)	2a	0.1548	0.2849	0.0210	0.077
H(19B)	2a	0.1858	0.3199	0.2367	0.063
H(20B)	2a	0.2109	0.1855	-0.0025	0.059

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}
O(1A)	2a	0.7240(4)	0.0388(2)	1.0396(4)	0.066(2)	0.048(2)	0.064(3)	0.013(2)	0.006(2)	0.006(2)
C(1A)	2a	0.6328(6)	0.0857(2)	1.0076(5)	0.056(3)	0.043(3)	0.045(3)	0.001(2)	0.006(2)	0.001(2)
O(2A)	2a	0.6630(4)	0.1390(2)	1.2137(4)	0.075(2)	0.055(2)	0.042(2)	0.020(2)	-0.002(2)	-0.005(2)
C(2A)	2a	0.6874(6)	0.1408(2)	1.0760(5)	0.055(3)	0.047(3)	0.046(3)	0.008(2)	0.002(2)	0.000(2)
O(3A)	2a	0.5070(5)	0.2018(2)	1.1181(4)	0.087(3)	0.078(3)	0.045(2)	0.037(3)	0.003(2)	0.000(2)
C(3A)	2a	0.6157(6)	0.1964(3)	1.0293(6)	0.059(3)	0.055(3)	0.050(3)	0.008(3)	-0.001(2)	-0.005(3)
O(4A)	2a	0.6467(4)	0.2234(2)	0.8085(4)	0.071(3)	0.060(2)	0.051(2)	-0.009(2)	0.002(2)	0.002(2)
C(4A)	2a	0.5522(6)	0.1958(2)	0.8900(5)	0.060(3)	0.040(3)	0.048(3)	0.001(2)	-0.001(2)	0.001(2)
O(5A)	2a	0.7255(4)	0.0406(2)	0.3821(4)	0.070(3)	0.065(3)	0.065(3)	-0.027(2)	-0.003(2)	0.002(2)
C(5A)	2a	0.5591(8)	0.1815(3)	1.2431(6)	0.092(4)	0.066(4)	0.043(3)	0.037(4)	0.000(3)	-0.004(3)
O(6A)	2a	0.5870(4)	-0.0455(2)	0.2006(4)	0.066(2)	0.063(2)	0.044(2)	-0.015(2)	0.002(2)	0.001(2)
C(6A)	2a	0.442(1)	0.1521(5)	1.3118(9)	0.106(6)	0.120(8)	0.091(6)	0.056(6)	0.038(5)	0.030(5)
O(7A)	2a	0.3945(4)	-0.0849(2)	0.2824(4)	0.064(2)	0.073(3)	0.047(2)	-0.014(2)	0.000(2)	-0.002(2)
C(7A)	2a	0.630(1)	0.2303(4)	1.3207(8)	0.21(1)	0.076(5)	0.054(4)	0.045(7)	-0.035(6)	-0.021(4)
O(8A)	2a	0.5183(6)	-0.1213(2)	0.6007(4)	0.109(4)	0.057(2)	0.043(2)	-0.007(3)	0.010(2)	0.005(2)
C(8A)	2a	0.5910(9)	0.2296(4)	0.6765(6)	0.107(6)	0.093(5)	0.040(3)	-0.029(5)	0.005(3)	0.008(3)
C(9A)	2a	0.6016(6)	0.0089(3)	0.4045(5)	0.052(3)	0.050(3)	0.051(3)	-0.007(2)	0.001(2)	0.004(2)
O(9A)	2a	0.5155(4)	0.1404(2)	0.8428(4)	0.053(2)	0.052(2)	0.053(2)	-0.006(2)	-0.005(2)	-0.002(2)
C(10A)	2a	0.6087(6)	-0.0495(2)	0.3412(5)	0.049(3)	0.049(3)	0.047(3)	-0.005(2)	-0.001(2)	-0.002(2)
O(10A)	2a	0.4487(4)	-0.0260(2)	0.5622(4)	0.060(2)	0.058(2)	0.059(2)	-0.009(2)	0.010(2)	-0.010(2)
C(11A)	2a	0.4990(6)	-0.0926(3)	0.3823(5)	0.059(3)	0.055(3)	0.038(3)	-0.006(3)	0.002(2)	0.000(2)
C(12A)	2a	0.4421(7)	-0.0835(3)	0.5174(6)	0.066(3)	0.058(3)	0.053(3)	-0.014(3)	0.004(3)	-0.008(3)
C(13A)	2a	0.4635(6)	-0.0774(3)	0.1625(6)	0.062(3)	0.064(4)	0.044(3)	-0.014(3)	0.003(2)	-0.001(3)
C(14A)	2a	0.506(1)	-0.1354(4)	0.1039(8)	0.119(6)	0.068(4)	0.064(4)	-0.008(4)	0.005(4)	-0.020(4)
C(15A)	2a	0.3752(8)	-0.0405(5)	0.0712(8)	0.070(4)	0.124(7)	0.073(5)	0.004(5)	-0.011(4)	0.025(5)
C(16A)	2a	0.476(1)	-0.1209(4)	0.7337(7)	0.19(1)	0.071(5)	0.048(4)	-0.023(6)	0.026(5)	0.003(4)
C(17A)	2a	0.5831(7)	0.0452(3)	0.7797(6)	0.082(4)	0.050(3)	0.045(3)	-0.007(3)	-0.001(3)	-0.004(3)
C(18A)	2a	0.5858(8)	0.0558(3)	0.6317(6)	0.088(4)	0.051(3)	0.050(3)	-0.018(3)	0.003(3)	-0.001(3)
C(19A)	2a	0.6221(6)	0.0968(2)	0.8607(5)	0.052(3)	0.041(3)	0.050(3)	-0.001(2)	0.002(2)	-0.001(2)
C(20A)	2a	0.5825(6)	0.0007(3)	0.5509(6)	0.060(3)	0.048(3)	0.048(3)	-0.005(3)	-0.004(2)	-0.005(2)
O(1B)	2a	0.2238(5)	0.2510(2)	0.4429(4)	0.069(2)	0.061(3)	0.064(3)	0.025(2)	0.011(2)	0.005(2)
C(1B)	2a	0.1143(6)	0.2896(2)	0.4123(5)	0.059(3)	0.037(3)	0.047(3)	0.004(2)	0.006(2)	0.002(2)
O(2B)	2a	0.1143(4)	0.3444(2)	0.6178(4)	0.053(2)	0.062(2)	0.042(2)	0.014(2)	0.002(2)	-0.003(2)
C(2B)	2a	0.1405(6)	0.3471(2)	0.4790(5)	0.049(3)	0.049(3)	0.044(3)	0.007(2)	0.004(2)	-0.004(2)
O(3B)	2a	-0.0737(4)	0.3854(2)	0.5090(4)	0.065(2)	0.068(3)	0.053(2)	0.020(2)	0.004(2)	-0.001(2)
C(3B)	2a	0.0436(6)	0.3954(3)	0.4310(6)	0.071(4)	0.043(3)	0.051(3)	0.004(3)	-0.003(3)	0.002(2)
O(4B)	2a	0.1023(6)	0.4247(2)	0.2233(4)	0.110(4)	0.058(2)	0.049(2)	-0.015(3)	0.002(2)	0.008(2)

Table 3. Continued.

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C(4B)	2*a*	0.0011(7)	0.3937(2)	0.2867(6)	0.084(4)	0.040(3)	0.047(3)	0.007(3)	–0.001(3)	–0.001(2)
O(5B)	2*a*	0.2294(4)	0.2563(2)	–0.2118(4)	0.065(2)	0.058(2)	0.063(3)	–0.015(2)	–0.009(2)	0.009(2)
C(5B)	2*a*	–0.0211(6)	0.3700(3)	0.6373(6)	0.054(3)	0.059(3)	0.052(3)	0.014(3)	0.006(2)	–0.002(3)
O(6B)	2*a*	0.1607(4)	0.1547(2)	–0.3862(4)	0.064(2)	0.064(2)	0.038(2)	–0.015(2)	0.001(2)	–0.004(2)
C(6B)	2*a*	–0.1139(7)	0.3267(3)	0.6949(8)	0.071(4)	0.063(4)	0.094(5)	0.006(3)	0.024(4)	0.011(4)
O(7B)	2*a*	–0.0045(4)	0.0986(2)	–0.2964(4)	0.070(2)	0.058(2)	0.055(2)	–0.015(2)	–0.005(2)	0.004(2)
C(7B)	2*a*	–0.0025(9)	0.4240(4)	0.7221(7)	0.095(5)	0.072(4)	0.064(4)	0.011(4)	0.011(4)	–0.006(4)
O(8B)	2*a*	0.1472(4)	0.0772(2)	0.0193(4)	0.073(3)	0.061(2)	0.051(2)	0.012(2)	0.002(2)	0.008(2)
C(8B)	2*a*	0.069(1)	0.4335(4)	0.0867(7)	0.19(1)	0.085(6)	0.054(4)	–0.036(7)	–0.003(6)	0.026(4)
C(9B)	2*a*	0.1340(6)	0.2107(3)	–0.1850(5)	0.055(3)	0.050(3)	0.043(3)	–0.002(2)	–0.002(2)	0.002(2)
O(9B)	2*a*	–0.0158(4)	0.3365(2)	0.2378(4)	0.073(3)	0.048(2)	0.052(2)	–0.001(2)	–0.007(2)	–0.003(2)
C(10B)	2*a*	0.1852(5)	0.1548(3)	–0.2462(5)	0.050(3)	0.050(3)	0.045(3)	–0.001(2)	–0.007(2)	–0.001(2)
O(10B)	2*a*	0.0146(4)	0.1594(2)	–0.0245(4)	0.059(2)	0.049(2)	0.051(2)	0.003(2)	0.010(2)	–0.002(2)
C(11B)	2*a*	0.1058(7)	0.1015(3)	–0.2019(5)	0.075(4)	0.050(3)	0.038(3)	–0.003(3)	0.000(2)	–0.002(2)
C(12B)	2*a*	0.0492(6)	0.1033(3)	–0.0666(5)	0.061(3)	0.049(3)	0.046(3)	0.003(3)	0.002(2)	0.007(2)
C(13B)	2*a*	0.0499(7)	0.1139(3)	–0.4199(6)	0.080(4)	0.064(4)	0.044(3)	–0.023(3)	–0.003(3)	–0.004(3)
C(14B)	2*a*	0.105(1)	0.0635(4)	–0.4900(8)	0.170(9)	0.077(5)	0.065(5)	–0.029(6)	0.023(5)	–0.021(4)
C(15B)	2*a*	–0.0621(8)	0.1466(4)	–0.4978(8)	0.095(5)	0.095(6)	0.069(4)	–0.033(5)	–0.036(4)	0.017(4)
C(16B)	2*a*	0.100(1)	0.0704(4)	0.1480(6)	0.125(6)	0.070(4)	0.048(4)	0.012(5)	0.013(4)	0.013(3)
C(17B)	2*a*	0.0819(8)	0.2453(3)	0.1847(6)	0.100(5)	0.044(3)	0.047(3)	0.004(3)	0.003(3)	–0.004(3)
C(18B)	2*a*	0.0873(8)	0.2549(3)	0.0372(6)	0.099(5)	0.047(3)	0.047(3)	0.006(3)	0.005(3)	–0.005(3)
C(19B)	2*a*	0.1026(7)	0.2999(2)	0.2646(6)	0.069(3)	0.042(3)	0.047(3)	0.007(3)	0.005(3)	–0.001(2)
C(20B)	2*a*	0.1238(6)	0.2015(2)	–0.0385(5)	0.062(3)	0.041(3)	0.045(3)	0.000(2)	0.000(2)	–0.003(2)

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