Crystal structure of (1*S*, 4*S*, 5*S*, 8*R*)-8-nitrooxy-2,6-dioxabicyclo[3.3.0] octan-4-yl-3-(3,4-diacetoxyphenyl)acrylate, $C_{19}H_{19}NO_{11}$

Peng-Cheng Fan, Lin-Lin Jing, Lei He, Xiao-Fei Fan and Zheng-Ping Jia*

Department of Pharmacy, Lanzhou General Hospital of PLA, Lanzhou 730050, Gansu Province, P. R. China

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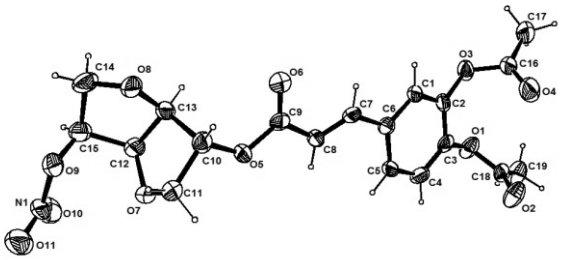

Abstract

$C_{19}H_{19}NO_{11}$, monoclinic, $P2_1$ (no. 4), a = 5.315(9) Å, b = 11.63(2) Å, c = 15.99(3) Å, β = 94.96(2)°, V = 984.7 Å^3, Z = 2, $R_{gt(F)}$ = 0.0877, $wR_{ref(F^2)}$ = 0.2586, T = 296 K.

Table 1. Data collection and handling.

Crystal:	colourless blocks, size 0.18×0.21×0.23 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	1.23 cm^{-1}
Diffractometer, scan mode:	Bruker APEX-II CCD, φ and ω
$2\theta_{max}$:	51°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	3737, 2974
$N(param)_{refined}$:	282
Programs:	SHELXS-97 [5], ORTEP-3 [6]

Source of material

The title compound was synthesized according to the literature [1]. 3-(3,4-diacetoxyphenyl)acrylic acid (2.64 g, 10 mmol), isosorbide mononitrate (1.91 g, 10 mmol, CAS No:16051-77-7, $[\alpha]_D$ = 168° (c = 1.0, EtOH) and DMAP (0.24 g, 1 mmol) were dissolved in 100 ml of dry THF, then DCC (4.12 g, 10 mmol) was added to the solution at 0°C. The mixture was stirred at room temperature for 5 h. The resulting mixture was filtered and concentrated *in vacuo*. The crude product was purified by column chromatography over silica gel using ethyl acetate/*n*-hexane (7:3) as eluent. Yield: 3.63 g (83%).Single crystals of the title compound suitable for *X*-ray diffraction were recrystallized from hexane/ethylacetate (1:1).

Experimental details

In the structure all of the H atoms were positioned geometrically and refined discernible using a riding model, with C–$H_{methine}$ = 0.98 Å; C–H_{methyl} = 0.96 Å; C–$H_{methylene}$ = 0.97 Å; C–H_{aryl} = 0.93 Å. The H atom isotropic displacement parameters were fixed; U_{iso} (H_{aryl}, $H_{methine}$) = 1.2 times U_{eq} of the parent atom; U_{iso} ($H_{methylene}$, H_{methyl}) = 1.5 times $U_{eq}$of the parent atom.

Discussion

The molecule is built up from the isosorbide mononitrate skeleton substituted on C6 by the 3-(3,4-diacetoxyphenyl)acrylic acid. Both of the two fused furanose rings display twisted conformation. The puckering parameters [2] for the C10–C11–O7–C12–C13 ring are $Q(2)$ = 0.342(9)Å and $\varphi(2)$ = 55.9(14)° and those for the C12–C13–O8–C14–C15 ring are $Q(2)$ = 0.374(9)Å and $\varphi(2)$ = 347.9(15)°. These values indicate that these two rings have different extent of puckering caused by the different substituent group in the ring. The pseudorotation parameters [3] for C10–C11–O7–C12–C13 ring are P = 142.7(8)° & $\tau(M)$ = 37.0(5)° for the C10–C13 reference bond with the closest pucker descriptor being twisted on C11–C10 and those for C12–C13–O8–C14–C15 ring are P = 78.9(9)° and $\tau(M)$ = 42.4(6)° for the C12–C15 reference bond with the closest puckering descriptor being twisted on C14–O8. Owing to the know absolute configuration of the starting isosorbide mononitrate, the absolute configuration of the title compound could be deduced to be 1*S*, 4*S*, 5*S*, 8*R*. The molecular packing is stabilized by weak non-classical C–H···O hydrogen bonds.

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

Atom	Site	x	y	z	U_{iso}
H(1)	2*a*	–0.0414	0.2992	0.2488	0.059
H(4)	2*a*	–0.1845	0.4991	0.4865	0.070
H(5)	2*a*	0.1359	0.5572	0.4076	0.060
H(7)	2*a*	0.3033	0.4213	0.2220	0.059
H(8)	2*a*	0.3478	0.6248	0.3012	0.055
H(10)	2*a*	0.9667	0.6718	0.1499	0.065
H(11A)	2*a*	1.0130	0.8254	0.2610	0.081
H(11B)	2*a*	1.1476	0.8470	0.1784	0.081
H(12)	2*a*	0.5129	0.9313	0.1051	0.073
H(13)	2*a*	0.5748	0.7483	0.0596	0.064
H(14A)	2*a*	0.7202	0.8704	–0.0684	0.101
H(14B)	2*a*	1.0091	0.9033	–0.0584	0.101
H(15)	2*a*	0.6912	1.0356	0.0052	0.078
H(17A)	2*a*	–0.5453	–0.0385	0.3322	0.133
H(17B)	2*a*	–0.6146	0.0467	0.2578	0.133
H(17C)	2*a*	–0.7491	0.0572	0.3410	0.133
H(19A)	2*a*	–0.8230	0.2243	0.5248	0.113
H(19B)	2*a*	–0.8155	0.3456	0.5676	0.113
H(19C)	2*a*	–0.7055	0.2383	0.6176	0.113

* Correspondence author (e-mail: zhengping_jia@yahoo.cn)

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)	2*a*	–0.073(2)	0.3417(6)	0.2960(4)	0.065(5)	0.034(4)	0.045(3)	0.002(4)	–0.010(3)	0.005(3)
C(2)	2*a*	–0.259(2)	0.3076(6)	0.3431(4)	0.062(5)	0.037(4)	0.056(4)	–0.012(4)	–0.021(4)	0.012(3)
C(3)	2*a*	–0.295(2)	0.3651(7)	0.4159(4)	0.050(5)	0.060(5)	0.042(4)	0.001(4)	–0.016(3)	0.015(3)
C(4)	2*a*	–0.154(2)	0.4588(7)	0.4382(4)	0.069(5)	0.068(6)	0.037(3)	0.003(5)	–0.005(4)	–0.001(3)
C(5)	2*a*	0.034(2)	0.4950(6)	0.3906(4)	0.060(5)	0.037(4)	0.048(4)	–0.004(4)	–0.014(3)	0.000(3)
C(6)	2*a*	0.069(2)	0.4369(6)	0.3162(4)	0.055(4)	0.043(4)	0.043(3)	0.007(4)	–0.012(3)	0.005(3)
C(7)	2*a*	0.262(2)	0.4745(7)	0.2620(4)	0.061(5)	0.051(5)	0.033(3)	0.002(4)	–0.008(3)	0.000(3)
C(8)	2*a*	0.380(1)	0.5693(7)	0.2616(4)	0.055(4)	0.037(4)	0.045(3)	–0.005(4)	–0.006(3)	0.002(3)
C(9)	2*a*	0.566(1)	0.5949(7)	0.2015(4)	0.053(5)	0.063(5)	0.037(3)	–0.012(4)	–0.014(3)	0.003(3)
C(10)	2*a*	0.851(2)	0.7345(7)	0.1606(4)	0.053(5)	0.056(5)	0.051(4)	–0.003(4)	–0.005(3)	0.009(3)
C(11)	2*a*	0.985(2)	0.8361(8)	0.2008(5)	0.087(6)	0.068(6)	0.046(4)	–0.023(5)	–0.002(4)	0.004(4)
C(12)	2*a*	0.692(2)	0.9130(7)	0.1031(4)	0.078(6)	0.055(5)	0.047(4)	–0.004(4)	–0.009(4)	0.004(3)
C(13)	2*a*	0.732(2)	0.7869(7)	0.0807(4)	0.076(5)	0.037(4)	0.045(3)	–0.001(4)	–0.010(3)	0.002(3)
C(14)	2*a*	0.865(3)	0.8839(8)	–0.0281(4)	0.160(1)	0.058(6)	0.038(4)	0.014(6)	–0.006(5)	0.005(4)
C(15)	2*a*	0.809(2)	0.9789(8)	0.0317(5)	0.096(7)	0.047(5)	0.050(4)	0.006(5)	–0.007(4)	0.002(3)
C(16)	2*a*	–0.398(2)	0.1172(7)	0.3516(5)	0.116(6)	0.046(4)	0.057(4)	–0.005(5)	–0.025(4)	0.005(3)
C(17)	2*a*	–0.593(2)	0.0391(9)	0.3178(6)	0.122(9)	0.065(6)	0.077(6)	–0.024(6)	–0.005(6)	0.015(5)
C(18)	2*a*	–0.488(2)	0.2951(7)	0.5340(4)	0.047(4)	0.049(4)	0.061(4)	0.009(4)	–0.012(3)	0.012(3)
C(19)	2*a*	–0.727(2)	0.2740(1)	0.5634(6)	0.054(5)	0.096(8)	0.077(5)	–0.002(5)	0.011(4)	0.024(5)
N(1)	2*a*	1.017(2)	1.1271(6)	0.1079(4)	0.122(8)	0.036(4)	0.060(4)	–0.025(5)	–0.007(5)	0.005(3)
O(1)	2*a*	–0.507(1)	0.3341(6)	0.4562(3)	0.051(3)	0.104(5)	0.058(3)	–0.010(3)	–0.014(2)	0.025(3)
O(2)	2*a*	–0.286(1)	0.2847(7)	0.5734(4)	0.055(4)	0.120(6)	0.072(4)	0.015(4)	0.004(3)	0.033(4)
O(3)	2*a*	–0.421(1)	0.2208(5)	0.3158(3)	0.077(4)	0.053(4)	0.069(3)	–0.025(3)	–0.024(3)	0.016(3)
O(4)	2*a*	–0.247(2)	0.0983(6)	0.4083(5)	0.145(6)	0.067(4)	0.108(4)	–0.006(4)	–0.062(4)	0.016(3)
O(5)	2*a*	0.664(1)	0.7002(4)	0.2130(3)	0.060(3)	0.045(3)	0.051(3)	–0.017(3)	–0.004(2)	0.002(2)
O(6)	2*a*	0.618(1)	0.5333(5)	0.1461(3)	0.094(5)	0.067(4)	0.054(3)	–0.017(3)	0.007(3)	–0.003(3)
O(7)	2*a*	0.830(1)	0.9299(5)	0.1821(3)	0.115(5)	0.055(4)	0.043(3)	–0.025(4)	0.001(3)	–0.004(2)
O(8)	2*a*	0.916(1)	0.7865(5)	0.0223(3)	0.117(5)	0.044(3)	0.046(3)	0.004(3)	0.007(3)	0.005(2)
O(9)	2*a*	1.041(1)	1.0286(5)	0.0591(3)	0.093(5)	0.044(3)	0.064(3)	–0.005(3)	0.009(3)	0.007(3)
O(10)	2*a*	0.816(2)	1.1673(7)	0.1127(5)	0.119(7)	0.060(4)	0.106(5)	0.005(5)	0.028(5)	–0.019(4)
O(11)	2*a*	1.221(2)	1.1635(7)	0.1366(4)	0.146(7)	0.067(5)	0.087(4)	–0.022(5)	–0.029(5)	0.012(4)

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