Crystal structure of 2,3-*O*-cyclohexylidene-*D*-glyceraldehyde *N*-benzylnitrone, $C_{16}H_{21}NO_3$

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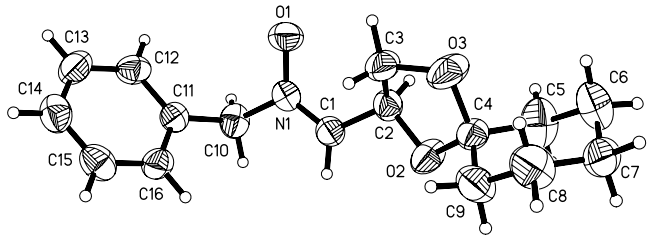

Abstract

$C_{16}H_{21}NO_3$, monoclinic, $P2_1$ (no. 4), a = 10.882(3) Å, b = 5.422(2) Å, c = 13.168(3) Å, β = 109.80(2)°, V = 731.0 Å^3, Z = 2, $R_{gt}(F)$ = 0.062, $wR_{ref}(F^2)$ = 0.162, T = 293 K.

Source of material

The title compound was obtained [1,2] by reaction of 2,3-*O*-cyclohexylidene-D-glyceraldehyde [3] and *N*-benzylhydroxylamine in dry dichloromethane in the presence of magnesium sulfate [4]. The product was purified by recrystallization from ethyl acetate/petroleum ether to give a spectroscopically and analytically pure solid, m.p. 97 - 100 °C (lit. [2]: 88 - 89 °C), $[\alpha]_D^{20}$ = 79 (c = 1.28, EtOH), lit. [2]: $[\alpha]_D^{20}$ = 82.5 (c = 1.00, EtOH).

Experimental details

H atoms were located on difference fourier map, but refined with fixed individual displacement parameters using a riding model with *d*(C—H) ranging from 0.93 to 0.98 Å. The Flack's parameter is: x = 1(2) which is meaningless for characterization of the absolute configuration.

Discussion

The title compound crystallizes with one independent molecule in the asymmetric unit. The slight lengthening of the N1–O1 distance of 1.293(3) Å is in accordance with polar character of the nitrone function. The N1=C1 double bond is characterized by a distance of 1.289(4) Å. The crystal packing is stabilized by a number of weak interactions. C1–H1 works as donor, and O1 of the nitrone function represents the acceptor. The C1···O1 distance is 3.405(4) Å and the H1···O1 distance is 2.48 Å with an angle C1–H1···O1 of 173°. Also a weak nearly linear interaction is formed between C9–H9A of a methylene group of the cyclohexyl moiety as donor and O3 of the dioxolane moiety. The distance of the C9···O3 contact is 3.387(5) Å and the H9A···O3 distance is 2.44 Å with an angle C9–H9A···O3 of 164°, which is nearly linear.

Table 1. Data collection and handling.

Crystal:	colourless block, size 0.25 × 0.3 × 0.7 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	0.86 cm^{-1}
Diffractometer, scan mode:	Nicolet P3, Wyckoff
$2\theta_{max}$:	53.98°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	6358, 3182
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 2460
$N(param)_{refined}$:	182
Programs:	SHELXS-97 [5], SHELXL-97 [6], SHELXTL-Plus [7]

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

Atom	Site	x	y	z	U_{iso}
H(1)	2*a*	0.3411	0.4725	0.9508	0.052
H(2)	2*a*	0.4415	0.1400	0.8487	0.055
H(3A)	2*a*	0.2703	–0.1257	0.7898	0.072
H(3B)	2*a*	0.1659	0.0877	0.7715	0.072
H(5A)	2*a*	0.4702	0.2139	0.6487	0.095
H(5B)	2*a*	0.4449	0.4941	0.6194	0.095
H(6A)	2*a*	0.4273	0.2840	0.4622	0.099
H(6B)	2*a*	0.3274	0.0968	0.4813	0.099
H(7A)	2*a*	0.2146	0.3741	0.3528	0.080
H(7B)	2*a*	0.2775	0.5962	0.4291	0.080
H(8A)	2*a*	0.0994	0.2568	0.4629	0.090
H(8B)	2*a*	0.0693	0.5355	0.4325	0.090
H(9A)	2*a*	0.2101	0.6604	0.5996	0.079
H(9B)	2*a*	0.1122	0.4693	0.6190	0.079
H(10A)	2*a*	0.3638	0.4154	1.1259	0.060
H(10B)	2*a*	0.4205	0.1606	1.1770	0.060
H(12)	2*a*	0.2779	–0.1331	1.2249	0.062
H(13)	2*a*	0.0850	–0.2026	1.2557	0.073
H(14)	2*a*	–0.0875	0.0637	1.1894	0.069
H(15)	2*a*	–0.0696	0.3987	1.0877	0.068
H(16)	2*a*	0.1250	0.4779	1.0602	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}
N(1)	2*a*	0.3467(2)	0.1502(4)	1.0140(2)	0.040(1)	0.047(1)	0.040(1)	0.002(1)	0.0136(9)	–0.003(1)
O(1)	2*a*	0.3472(2)	–0.0864(4)	1.0020(2)	0.076(2)	0.042(1)	0.056(1)	0.009(1)	0.024(1)	0.003(1)

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Table 3. Continued.

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C(1)	2*a*	0.3468(2)	0.3042(5)	0.9394(2)	0.043(1)	0.043(1)	0.046(2)	–0.004(1)	0.018(1)	–0.005(1)
O(2)	2*a*	0.3425(2)	0.4223(4)	0.7662(2)	0.082(2)	0.048(1)	0.037(1)	–0.022(1)	0.0144(9)	–0.0026(9)
C(2)	2*a*	0.3558(3)	0.2162(6)	0.8355(2)	0.048(2)	0.049(2)	0.042(1)	0.008(1)	0.018(1)	0.006(1)
O(3)	2*a*	0.2558(3)	0.0858(4)	0.6621(2)	0.137(2)	0.043(1)	0.049(1)	–0.030(1)	0.028(1)	–0.007(1)
C(3)	2*a*	0.2507(4)	0.0446(6)	0.7677(3)	0.098(2)	0.038(2)	0.049(2)	–0.012(2)	0.032(2)	–0.004(1)
C(4)	2*a*	0.2954(3)	0.3336(5)	0.6567(2)	0.055(2)	0.039(1)	0.040(1)	–0.012(1)	0.014(1)	–0.006(1)
C(5)	2*a*	0.4043(4)	0.333(1)	0.6098(3)	0.059(2)	0.117(4)	0.057(2)	0.001(2)	0.016(2)	0.004(2)
C(6)	2*a*	0.3559(4)	0.267(1)	0.4902(3)	0.090(3)	0.099(3)	0.069(2)	0.006(2)	0.042(2)	–0.013(2)
C(7)	2*a*	0.2465(4)	0.4281(8)	0.4274(3)	0.101(3)	0.058(2)	0.042(2)	–0.005(2)	0.023(2)	–0.005(2)
C(8)	2*a*	0.1370(4)	0.4210(9)	0.4722(3)	0.082(2)	0.082(3)	0.049(2)	0.021(2)	0.009(2)	0.000(2)
C(9)	2*a*	0.1839(4)	0.4885(7)	0.5915(3)	0.081(2)	0.064(2)	0.053(2)	0.020(2)	0.023(2)	–0.002(2)
C(10)	2*a*	0.3488(3)	0.2387(6)	1.1211(2)	0.052(2)	0.060(2)	0.037(1)	–0.013(1)	0.013(1)	–0.008(1)
C(11)	2*a*	0.2227(3)	0.1828(5)	1.1400(2)	0.049(1)	0.044(2)	0.029(1)	–0.004(1)	0.011(1)	–0.006(1)
C(12)	2*a*	0.2088(3)	–0.0233(5)	1.1981(2)	0.059(2)	0.043(2)	0.049(2)	0.008(1)	0.012(1)	0.006(1)
C(13)	2*a*	0.0931(3)	–0.0654(7)	1.2160(3)	0.078(2)	0.050(2)	0.059(2)	–0.012(2)	0.030(2)	0.005(2)
C(14)	2*a*	–0.0100(3)	0.0924(7)	1.1762(3)	0.055(2)	0.067(2)	0.054(2)	–0.008(2)	0.024(2)	–0.012(2)
C(15)	2*a*	0.0011(3)	0.2935(7)	1.1166(2)	0.052(2)	0.065(2)	0.050(2)	0.013(2)	0.013(1)	–0.003(2)
C(16)	2*a*	0.1175(3)	0.3396(6)	1.0995(2)	0.064(2)	0.045(2)	0.038(1)	0.005(1)	0.018(1)	0.007(1)

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