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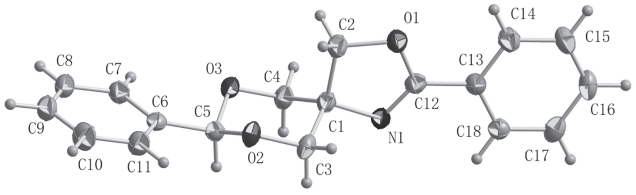
Crystal structure of 2,8-diphenyl-3,7,9-trioxa-1-azaspiro[4.5]dec-1-ene, $C_{18}H_{17}N_1O_3$



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Abstract

$C_{18}H_{17}N_1O_3$, monoclinic, $P2_1/c$ (no. 14), $a = 14.9752(8)$ Å, $b = 9.0793(5)$ Å, $c = 11.1030(6)$ Å, $\beta = 106.431(1)°$, $V = 1447.96(14)$ Å^3, $Z = 4$, $R_{gt}(F) = 0.0342$, $wR_{ref}(F^2) = 0.0903$, $T = 296(2)$ K.

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The crystal structure is shown in the figure. Tables 1 and 2 contain details on crystal structure and measurement conditions and a list of the atoms including atomic coordinates and displacement parameters.

Source of material

Tris(hydroxymethyl)aminomethane (TRIS) (2.00 g, 16.5 mmol) and benzaldehyde (5.31 g, 50 mmol) were stirred at 338 K in anhydrous toluene (50 mL) until the reaction mixture becomes clear. The solvent was evaporated to dryness and the residue dissolved in diethylether (50 mL). The etheral solution was washed with a saturated Na_2CO_3 solution (10 mL). The aqueous phase was extracted with diethylether and the combined organic phase was dried over anhydrous Na_2SO_4, then evaporated to a white powder. The latter was recrystallized from ethanol to afford colorless crystals (2.73 g, 56%).

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Table 1: Data collection and handling.

Crystal:	Block, colorless
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ:	0.09 mm^{-1}
Diffractometer, scan mode:	Bruker APEX-II, φ and ω-scans
$2\theta_{max}$, completeness:	25°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	26710, 2541, 0.018
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 2384
$N(param)_{refined}$:	199
Programs:	Bruker programs [1], SHELX [2, 3]

Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2).

Atom	x	y	z	U_{iso}*/U_{eq}
O3	0.06061(6)	0.17715(10)	0.24804(7)	0.0270(2)
O2	0.07766(6)	0.35823(9)	0.10810(7)	0.0275(2)
O1	−0.21461(6)	0.32501(10)	0.03002(9)	0.0349(2)
N1	−0.13695(7)	0.14691(11)	−0.04115(9)	0.0265(2)
C1	−0.06661(8)	0.21939(13)	0.06214(11)	0.0256(3)
C5	0.12246(8)	0.24476(13)	0.19006(11)	0.0233(3)
H5	0.1456	0.1706	0.1421	0.028*
C13	−0.30284(8)	0.18096(13)	−0.14404(11)	0.0261(3)
C12	−0.21330(9)	0.21354(14)	−0.05101(11)	0.0276(3)
C4	−0.01397(8)	0.10583(14)	0.15649(11)	0.0278(3)
H4A	−0.0559	0.0597	0.1976	0.033*
H4B	0.0109	0.0299	0.1136	0.033*
C7	0.23114(9)	0.24366(15)	0.40891(12)	0.0295(3)
H7	0.1974	0.1653	0.4277	0.035*
C6	0.20345(8)	0.30765(13)	0.29031(11)	0.0247(3)
C18	−0.30567(9)	0.07887(14)	−0.23862(11)	0.0282(3)
H18	−0.2510	0.0349	−0.2445	0.034*
C14	−0.38491(9)	0.24658(15)	−0.13635(13)	0.0337(3)
H14	−0.3835	0.3151	−0.0736	0.040*
C3	0.00384(9)	0.29752(14)	0.00848(11)	0.0273(3)
H3A	0.0293	0.2282	−0.0395	0.033*
H3B	−0.0269	0.3757	−0.0476	0.033*
C8	0.30866(9)	0.29594(16)	0.49914(12)	0.0338(3)
H8	0.3265	0.2526	0.5782	0.041*
C11	0.25427(9)	0.42562(15)	0.26460(12)	0.0340(3)
H11	0.2363	0.4702	0.1860	0.041*
C15	−0.46869(9)	0.20986(16)	−0.22206(14)	0.0404(3)
H15	−0.5236	0.2539	−0.2169	0.049*
C2	−0.12262(9)	0.32762(17)	0.11823(13)	0.0373(3)
H2A	−0.1245	0.2957	0.2008	0.045*
H2B	−0.0960	0.4257	0.1248	0.045*
C17	−0.38951(9)	0.04279(16)	−0.32366(13)	0.0353(3)
H17	−0.3912	−0.0255	−0.3867	0.042*

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Table 2 (continued)

Atom	x	y	z	U_{iso}*/U_{eq}
C10	0.33164(10)	0.47703(17)	0.35560(14)	0.0412(3)
H10	0.3651	0.5563	0.3378	0.049*
C9	0.35948(9)	0.41159(16)	0.47264(13)	0.0374(3)
H9	0.4121	0.4454	0.5330	0.045*
C16	−0.47099(9)	0.10798(17)	−0.31533(14)	0.0410(4)
H16	−0.5274	0.0832	−0.3726	0.049*

Experimental details

All hydrogen atoms were identified in difference Fourier syntheses.

Discussion

Synthetic routes employing oxazolidines as cyclic protection of aminoalcohols have been widely used for the asymmetric synthesis of chiral amines, aminoalcohols and/or aminoacids [4]. Moreover, such derivatives were assumed to have potential as prodrug forms, as they undergo conversion to the parent compound [5]. Indeed, oxazolidine derived from ephedrine has been proved to have sympathomimetic activity in several animal models [6, 7]. In both cases, the success of the approach relies on the cleavage of the 1,3-N,O ring.

Herein, we report on a new oxazolidine derivative, which was synthesized and characterized by single-crystal X-ray diffraction [2, 3]. In the structure, there are two new formed rings, C(1)—C(3)—O(2)—C(5)—O(3)—C(4) and C(1)—N(1)—C(12)—O(1)—C(2). The bond lengths of N(1)—C(1), N(1)—C(12) are 1.4746(15) Å and 1.2706(16) Å respectively. All geometric parameters are in the expected ranges [8, 9].

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