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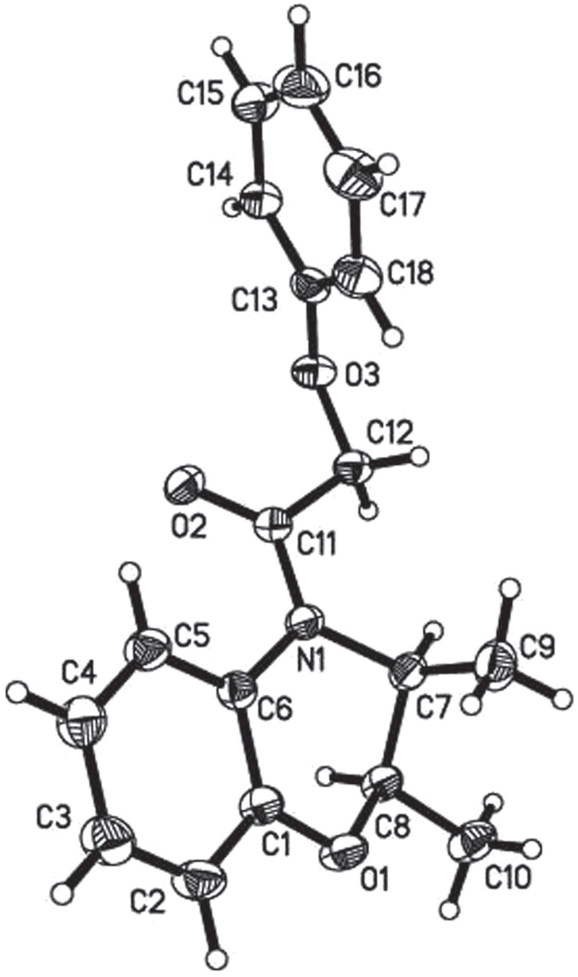
Crystal structure of 1-((2R,3S)-2,3-dimethyl-2,3-dihydro-4H-benzo[b][1,4]oxazin-4-yl)-2-phenoxyethan-1-one, $C_{18}H_{19}NO_3$



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Abstract

$C_{18}H_{19}NO_3$, triclinic, $P\bar{1}$ (no. 2), $a = 7.9928(16)$ Å, $b = 8.7582(18)$ Å, $c = 11.692(2)$ Å, $\alpha = 97.00(3)°$, $\beta = 105.63(3)°$, $y = 99.16(3)°$, $V = 766.4(3)$ Å^3, $Z = 2$, F(000) = 316, $R_{gt}(F) = 0.0447$, $wR_{ref}(F^2) = 0.1235$, $T = 293$ K.

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Table 1: Data collection and handling.

Crystal:	Colorless, block, size 0.16×0.25×0.31 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	0.88 cm^{-1}
Diffractometer, scan mode:	Rigaku RAXIS-RAPID, ω scans
$2\theta_{max}$:	54.96°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	7586, 3480
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 2561
$N(param)_{refined}$:	201
Programs:	SHELX [8], Diamond [9]

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

Atom	Site	*x*	*y*	*z*	U_{iso}
H(2)	2*i*	0.9478	0.8537	0.9414	0.067
H(3)	2*i*	0.9564	0.6098	0.8473	0.074
H(4)	2*i*	0.7494	0.4974	0.6647	0.069
H(5)	2*i*	0.5319	0.6268	0.5778	0.058
H(7)	2*i*	0.3781	1.0975	0.7035	0.048
H(8)	2*i*	0.6872	1.1589	0.7504	0.050
H(9A)	2*i*	0.2332	0.9055	0.7826	0.088
H(9B)	2*i*	0.3391	1.0447	0.8876	0.088
H(9C)	2*i*	0.4133	0.8907	0.8717	0.088
H(10A)	2*i*	0.5863	1.2425	0.9592	0.087
H(10B)	2*i*	0.5798	1.3431	0.8565	0.087
H(10C)	2*i*	0.7629	1.3221	0.9378	0.087
H(12A)	2*i*	0.1442	0.9519	0.5578	0.051
H(12B)	2*i*	0.2758	1.0805	0.5253	0.051
H(14)	2*i*	−0.0341	0.8114	0.1728	0.060
H(15)	2*i*	−0.2493	0.5874	0.0908	0.077
H(16)	2*i*	−0.3442	0.4293	0.2140	0.086
H(17)	2*i*	−0.2188	0.4935	0.4190	0.082
H(18)	2*i*	0.0008	0.7153	0.5043	0.065

Table 3: Atomic displacement parameters (Å^2).

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C(1)	2*i*	0.7340(2)	0.8877(1)	0.8163(1)	0.0449(7)	0.0383(6)	0.0383(6)	0.0088(5)	0.0059(5)	0.0047(5)
C(2)	2*i*	0.8639(2)	0.8082(2)	0.8681(1)	0.0528(8)	0.0541(8)	0.0499(8)	0.0161(7)	−0.0052(6)	0.0072(6)
C(3)	2*i*	0.8694(2)	0.6628(2)	0.8120(2)	0.0607(9)	0.0540(8)	0.0674(9)	0.0268(7)	0.0039(8)	0.0128(7)
C(4)	2*i*	0.7453(2)	0.5956(2)	0.7031(1)	0.0653(9)	0.0428(7)	0.0633(9)	0.0220(7)	0.0119(8)	0.0038(6)
C(5)	2*i*	0.6152(2)	0.6735(2)	0.6511(1)	0.0504(8)	0.0395(7)	0.0476(7)	0.0098(6)	0.0060(6)	0.0000(5)
C(6)	2*i*	0.6064(2)	0.8208(1)	0.7063(1)	0.0372(6)	0.0359(6)	0.0366(6)	0.0079(5)	0.0091(5)	0.0069(5)
C(7)	2*i*	0.4486(2)	1.0301(1)	0.7481(1)	0.0441(7)	0.0412(6)	0.0337(6)	0.0131(5)	0.0088(5)	0.0022(5)
C(8)	2*i*	0.6327(2)	1.1266(1)	0.8117(1)	0.0477(7)	0.0392(6)	0.0339(6)	0.0108(5)	0.0052(5)	0.0033(5)
C(9)	2*i*	0.3494(2)	0.9615(2)	0.8300(1)	0.0624(9)	0.0644(9)	0.0541(8)	0.0107(8)	0.0276(8)	0.0054(7)
C(10)	2*i*	0.6412(2)	1.2718(2)	0.8993(1)	0.071(1)	0.0442(7)	0.0476(7)	0.0127(7)	0.0054(7)	−0.0044(6)
C(11)	2*i*	0.3695(2)	0.8750(1)	0.5415(1)	0.0341(6)	0.0380(6)	0.0364(6)	0.0042(5)	0.0079(5)	0.0032(5)
C(12)	2*i*	0.2232(2)	0.9695(2)	0.5086(1)	0.0374(6)	0.0470(7)	0.0384(6)	0.0095(5)	0.0034(5)	0.0025(5)
C(13)	2*i*	0.0042(2)	0.7863(2)	0.3469(1)	0.0319(6)	0.0437(7)	0.0446(7)	0.0105(5)	0.0052(5)	0.0059(5)
C(14)	2*i*	−0.0709(2)	0.7473(2)	0.2229(1)	0.0420(7)	0.0595(8)	0.0442(7)	0.0113(6)	0.0058(6)	0.0030(6)
C(15)	2*i*	−0.1999(2)	0.6136(2)	0.1740(2)	0.0514(9)	0.066(1)	0.0612(9)	0.0100(8)	0.0027(7)	−0.0110(8)
C(16)	2*i*	−0.2561(2)	0.5189(2)	0.2471(2)	0.0525(9)	0.0501(9)	0.096(1)	−0.0004(7)	0.0084(9)	−0.0053(9)
C(17)	2*i*	−0.1809(2)	0.5579(2)	0.3694(2)	0.059(1)	0.0552(9)	0.090(1)	0.0047(8)	0.0199(9)	0.0220(9)
C(18)	2*i*	−0.0498(2)	0.6909(2)	0.4211(1)	0.0480(8)	0.0562(8)	0.0552(8)	0.0102(7)	0.0090(7)	0.0153(6)
N(1)	2*i*	0.4683(1)	0.9032(1)	0.65934(8)	0.0369(5)	0.0373(5)	0.0337(5)	0.0099(4)	0.0042(4)	0.0007(4)
O(1)	2*i*	0.7393(1)	1.0316(1)	0.87973(8)	0.0564(6)	0.0444(5)	0.0374(5)	0.0155(4)	−0.0047(4)	−0.0014(4)
O(2)	2*i*	0.3923(1)	0.7820(1)	0.46342(7)	0.0503(5)	0.0545(6)	0.0360(4)	0.0163(4)	0.0055(4)	−0.0043(4)
O(3)	2*i*	0.1251(1)	0.9253(1)	0.38537(7)	0.0400(5)	0.0503(5)	0.0383(4)	0.0047(4)	0.0007(4)	0.0087(4)

The crystal structure is shown in the figure. Tables 1–3 contain details of the measurement method and a list of the atoms including atomic coordinates and displacement parameters.

Source of material

2.28 g (14 mmol) 2,3-dimethyl-3,4-dihydro-2H-1,4-benzoxazine, 1.6 g (15.2 mmol), anhydrous Na_2CO_3, and 20 mL benzene were mixed. 1.7 mL of phenoxyacetonyl chloride (17.6 mmol) was added slowly at room temperature and the mixture was stirred for 30 min. Then the mixture was filtered and rinsed with water. The organic phase was dried over anhydrous $MgSO_4$, and the benzene was removed under vacuum. The crude product was recrystallized from EtOAc and light petroleum. The product was collected as white solid in yield 61.2%.

Experimental details

The C—H atoms were then constrained to an ideal geometry, with C—H distances of 0.93–0.98 Å. The U_{iso} values of the hydrogen atoms of methyl groups were set to $1.5U_{eq}(C_{methyl})$ and the U_{iso} values of all other hydrogen atons were set to $1.2U_{eq}(C)$.

Discussion

Benzoxazine and its derivatives have exhibited various biological activities, such as herbicides, fungicides, anti-diabetic, anxiolytic, cardiovascular and neuroprotective agents or antidepressants [1–3]. Moreover, they also act as useful intermeditates in the syntheses of compounds with therapeutic interest [4, 5]. In continuation of our interest in the development of N-containing heterocyclic compounds, herein we report a 1,4-benzoxazine [6, 7]. In the crystal structure, the oxazine ring and the annulated benzene ring are almost coplanar with the dihedral angle being 5.56(4)° (see the figure). The dihedral angle of benzoxazine ring and the phenyl moiety is 71.54(4)°. The torsion angle of N(1)—C(7)—C(8)—O(1) is −64.79(1)°, forming a half-chair conformation. It should be mentioned that there is a p-π-p-π conjunctive effect between O1, benzene ring [C1, C2, C3, C4, C5, and C6], N1, C11-O2 [C = O], which result in shortened bond lengths for O1-C1, C6-N1, and N1-C11. The shortest H···centroid distance is 3.2 Å and the shortest H···O distance is a geometrically forced intramolecular distance.

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