Crystal structure of (1*S*,2*R*,4*S*)-1-((phenylamino)methyl)-4-(prop-1-en-2-yl)cyclohexane-1,2-diol), $C_{16}H_{23}NO_2$

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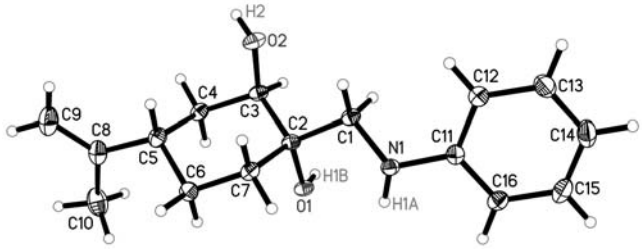
Received February 9, 2011, accepted and available on-line April 27, 2011; CCDC no. 1267/3399



Abstract

$C_{16}H_{23}NO_2$, orthorhombic, $P2_12_12_1$ (no. 19), a = 5.9637(3) Å, b = 8.8317(5) Å, c = 27.809(1) Å, V = 1464.7 Å^3, Z = 4, $R_{gt}(F)$ = 0.026, $wR_{ref}(F^2)$ = 0.040, T = 150 K.

Source of material

A mixture of aniline (5.1 mmol) and epoxy perillyl alcohol (5 mmol) was added to 0.25 mmol of $Ca(CF_3CO_2)_2$ under solvent-free conditions. The mixture was stirred at 40 °C for 72 h. After the reaction was finished, the mixture was extracted with AcOEt (3 × 10 mL), dried over Na_2SO_4 and the solvent was removed at reduced pressure. Colorless crystals of the title compound were isolated by column chromatography on silica gel using hexane/ethyl acetate as eluent (yield 55 %).

Experimental details

Hydrogen atoms attached to N1, O1 and O2 were located in the difference Fourier map and refined isotropically. All other hydrogen atoms were placed in idealized positions and refined using a riding model. The absolute configuration of the title structure could not be determined using data collected with Mo K_α radiation (Flack parameter x = 0.1(10)).

Discussion

Aminodiol motif is present in many pharmacologically important compounds such as antiviral glycosidase inhibitors [1,2], sphingolipids [3,4], antibacterial and antiprotozoal [5]. Also, aminodiols have been found to act as HIV protease inhibitors [6,7]. Furthermore, aminodiols may serve as useful starting materials for the synthesis of biologically active compounds, e.g., 3-amino-1,2-butanediol derivative, a key intermediate in the synthesis of anticancer agent ES-285 [8]. Chiral aminodiols and their derivatives also find application as catalysts for enantioselective transformations [9,10]. Different approaches to obtain aminodiol derivatives have been developed [11-13]. Aminolysis of 1,2-epoxides represents one of the valuable pathway to produce commercially important aminoalcohols and aminodiols from olefins [14-16].

The absolute configuration has been assigned by reference to unchanging the chiral centres in the synthetic procedure. The cyclohexane ring is in a chair conformation, with puckering parameters Q, θ and φ of 0.565(2) Å, 172.8(1)° and –31(1)°, respectively [19]. In the crystal structure, molecules interact *via* N1–H1A···O2 and O2–H2···O1 intermolecular hydrogen bonds to form chains parallel to [100]. The chains are further linked into layers parallel to the (001) plane by O1–H1B···N1 hydrogen bonds.

Table 1. Data collection and handling.

Crystal:	colorless needle, size 0.08 × 0.16 × 0.47 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	0.77 cm^{-1}
Diffractometer, scan mode:	STOE IPDS II, ω
$2\theta_{max}$:	51°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	9453, 2721
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 1941
$N(param)_{refined}$:	185
Programs:	SHELXS-97, SHELXL-97 [20]

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

Atom	Site	x	y	z	U_{iso}
H(1C)	4*a*	0.2794	0.5111	0.7883	0.031
H(1D)	4*a*	0.2056	0.3386	0.7803	0.031
H(3)	4*a*	0.1020	0.6135	0.7143	0.031
H(4A)	4*a*	0.2151	0.5950	0.6350	0.034
H(4B)	4*a*	–0.0472	0.5571	0.6367	0.034
H(5)	4*a*	0.0329	0.2945	0.6332	0.035
H(6A)	4*a*	0.4090	0.2120	0.6216	0.034
H(6B)	4*a*	0.4948	0.3831	0.6273	0.034
H(7A)	4*a*	0.2723	0.2133	0.7013	0.031
H(7B)	4*a*	0.5337	0.2543	0.7015	0.031
H(9A)	4*a*	–0.0613	0.3247	0.5112	0.060
H(9B)	4*a*	–0.1539	0.2582	0.5628	0.060
H(10A)	4*a*	0.2597	0.4718	0.5064	0.078

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Table 2. Continued.

Atom	Site	*x*	*y*	*z*	U_{iso}
H(10B)	4*a*	0.4439	0.4259	0.5456	0.078
H(10C)	4*a*	0.2964	0.5755	0.5529	0.078
H(12)	4*a*	0.2648	0.3128	0.8642	0.039
H(13)	4*a*	0.3373	0.3287	0.9463	0.049
H(14)	4*a*	0.6854	0.4077	0.9739	0.054
H(15)	4*a*	0.9675	0.4677	0.9189	0.046
H(16)	4*a*	0.8995	0.4484	0.8368	0.037
H(1A)	4*a*	0.653(2)	0.408(2)	0.7756(5)	0.033(4)
H(1B)	4*a*	0.480(3)	0.642(2)	0.7119(6)	0.066(5)
H(2)	4*a*	–0.187(3)	0.457(2)	0.7094(6)	0.068(7)

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)	4*a*	0.3223(2)	0.4147(2)	0.77270(4)	0.0214(8)	0.0232(7)	0.0318(8)	0.0017(7)	–0.0006(6)	–0.0007(6)
C(2)	4*a*	0.3326(2)	0.4377(1)	0.71836(5)	0.0195(7)	0.0199(7)	0.0299(7)	–0.0030(7)	0.0024(7)	0.0027(6)
C(3)	4*a*	0.1144(2)	0.5092(2)	0.70053(4)	0.0206(8)	0.0213(7)	0.0350(8)	–0.0010(7)	0.0014(7)	0.0012(6)
C(4)	4*a*	0.1032(2)	0.5201(1)	0.64617(4)	0.0248(9)	0.0263(8)	0.0327(8)	0.0006(7)	–0.0050(7)	0.0018(6)
C(5)	4*a*	0.1479(3)	0.3681(1)	0.62143(5)	0.031(1)	0.0258(8)	0.0320(8)	–0.0021(8)	–0.0043(7)	0.0010(6)
C(6)	4*a*	0.3775(3)	0.3104(2)	0.63731(4)	0.029(1)	0.0219(7)	0.0326(8)	0.0024(7)	–0.0016(7)	–0.0027(6)
C(7)	4*a*	0.3834(3)	0.2910(1)	0.69181(4)	0.0237(9)	0.0217(7)	0.0327(8)	0.0005(7)	–0.0033(6)	–0.0005(6)
C(8)	4*a*	0.1223(3)	0.3804(2)	0.56749(5)	0.050(1)	0.0254(8)	0.0343(9)	0.0054(8)	–0.0099(8)	–0.0021(6)
C(9)	4*a*	–0.0467(3)	0.3150(2)	0.54505(6)	0.064(1)	0.046(1)	0.0415(9)	0.006(1)	–0.0184(9)	–0.0046(8)
C(10)	4*a*	0.2957(3)	0.4714(2)	0.54075(6)	0.078(2)	0.045(1)	0.0331(8)	–0.002(1)	–0.0023(9)	0.0054(8)
C(11)	4*a*	0.5742(3)	0.3803(1)	0.84209(5)	0.0306(9)	0.0199(7)	0.0263(8)	0.0047(7)	–0.0023(7)	–0.0003(5)
C(12)	4*a*	0.4084(3)	0.3443(2)	0.87508(5)	0.030(1)	0.0356(9)	0.0310(8)	0.0009(8)	–0.0005(7)	0.0003(6)
C(13)	4*a*	0.4516(3)	0.3542(2)	0.92388(5)	0.043(1)	0.048(1)	0.0319(9)	0.0099(9)	0.0038(8)	0.0020(7)
C(14)	4*a*	0.6578(3)	0.4003(2)	0.94035(5)	0.056(1)	0.049(1)	0.0301(8)	0.018(1)	–0.0091(9)	–0.0082(7)
C(15)	4*a*	0.8246(3)	0.4358(2)	0.90779(5)	0.039(1)	0.0336(9)	0.044(1)	0.0064(9)	–0.0157(8)	–0.0079(7)
C(16)	4*a*	0.7836(3)	0.4250(2)	0.85906(5)	0.0286(9)	0.0264(8)	0.0382(9)	0.0027(8)	–0.0025(7)	–0.0033(7)
O(1)	4*a*	0.5139(2)	0.5399(1)	0.70720(3)	0.0204(6)	0.0203(5)	0.0360(5)	–0.0016(4)	0.0012(4)	0.0012(4)
O(2)	4*a*	–0.0653(2)	0.4193(1)	0.71907(4)	0.0208(6)	0.0378(6)	0.0437(6)	–0.0029(5)	–0.0002(5)	0.0081(5)
N(1)	4*a*	0.5391(2)	0.3634(1)	0.79207(4)	0.0212(7)	0.0278(6)	0.0265(7)	0.0000(6)	0.0012(6)	0.0027(5)

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