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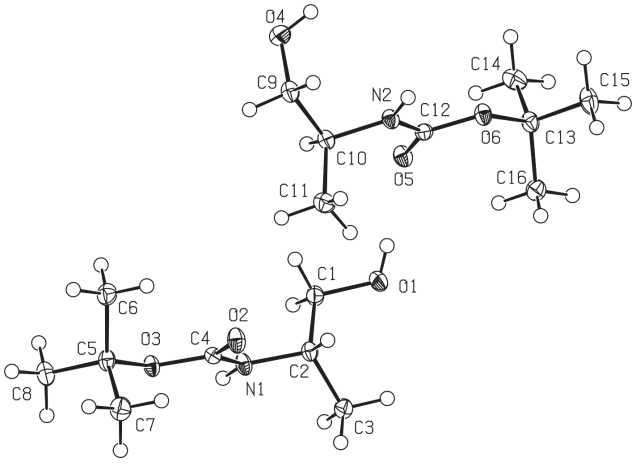
Crystal structure of (*S*)-*tert*-butyl-(1-hydroxy-propan-2-yl)carbamate, $C_8H_{17}NO_3$



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Abstract

$C_8H_{17}NO_3$, monoclinic, $P2_1$ (no. 4), $a = 10.4369(3)$ Å, $b = 9.4976(2)$ Å, $c = 10.8153(3)$ Å, $\beta = 114.911(1)°$, $V = 972.33(4)$ Å^3, $Z = 4$, $R_{gt}(F) = 0.0287$, $wR_{ref}(F^2) = 0.0733$, $T = 173(2)$ K.

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The asymmetric unit of the title 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

Pivalic anhydride (4.36 g, 20 mmol) was added to an ice-cooled solution of *S*-(+)-alaninol (1.50 g, 20 mmol) and triethylamine (2.78 ml, 20 mmol) in tetrahydrofuran (60 mL). The mixture was warmed to room temperature, stirred for

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Table 1: Data collection and handling.

Crystal:	Block, colorless
Size:	0.43 × 0.36 × 0.28 mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ:	0.09m^{-1}
Diffractometer, scan mode:	Bruker APEX-II, φ and ω-scans
$2\theta_{max}$, completeness:	25.5°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	17778, 3584, 0.030
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 3544
$N(param)_{refined}$:	227
Programs:	Bruker programs [1], SHELX [2], WinGX, ORTEP [3]

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

Atom	x	y	z	U_{iso}*/U_{eq}
C1	0.61468(18)	0.5823(2)	0.31520(17)	0.0178(4)
H1A	0.6150	0.4783	0.3098	0.021*
H1B	0.5226	0.6164	0.2464	0.021*
C2	0.63136(17)	0.62616(19)	0.45700(16)	0.0148(3)
H2	0.6189	0.7305	0.4585	0.018*
C3	0.77532(18)	0.5861(2)	0.56814(17)	0.0203(4)
H3A	0.7830	0.6207	0.6564	0.031*
H3B	0.8499	0.6286	0.5479	0.031*
H3C	0.7856	0.4835	0.5716	0.031*
C4	0.41021(17)	0.6268(2)	0.48752(16)	0.0145(3)
C5	0.18738(17)	0.58148(19)	0.50802(17)	0.0164(4)
C6	0.09532(18)	0.6659(2)	0.38241(18)	0.0211(4)
H6A	0.1428	0.7544	0.3810	0.032*
H6B	0.0044	0.6862	0.3850	0.032*
H6C	0.0794	0.6113	0.3003	0.032*
C7	0.2261(2)	0.6627(2)	0.63998(19)	0.0237(4)
H7A	0.2859	0.6039	0.7172	0.036*
H7B	0.1399	0.6883	0.6497	0.036*
H7C	0.2776	0.7484	0.6381	0.036*
C8	0.11521(19)	0.4430(2)	0.51041(19)	0.0211(4)
H8A	0.0951	0.3910	0.4260	0.032*
H8B	0.0266	0.4621	0.5182	0.032*
H8C	0.1775	0.3867	0.5886	0.032*
C9	0.22184(17)	0.9615(2)	−0.03227(17)	0.0180(4)
H9A	0.1577	0.9266	0.0071	0.022*
H9B	0.2232	1.0656	−0.0268	0.022*
C10	0.36996(17)	0.90638(19)	0.05321(16)	0.0158(4)
H10	0.3722	0.8031	0.0361	0.019*
C11	0.4097(2)	0.9294(2)	0.20473(17)	0.0239(4)
H11A	0.5074	0.8991	0.2580	0.036*
H11B	0.3464	0.8744	0.2320	0.036*

Table 2 (continued)

Atom	x	y	z	U_{iso}*/U_{eq}
H11C	0.4007	1.0295	0.2215	0.036*
C12	0.58674(17)	0.9137(2)	0.01628(15)	0.0146(4)
C13	0.80358(17)	0.96344(19)	−0.01468(17)	0.0172(4)
C14	0.7720(2)	0.8678(2)	−0.13682(19)	0.0247(4)
H14A	0.7301	0.7797	−0.1240	0.037*
H14B	0.8599	0.8470	−0.1454	0.037*
H14C	0.7057	0.9149	−0.2197	0.037*
C15	0.86212(19)	1.1045(2)	−0.0338(2)	0.0252(4)
H15A	0.7949	1.1488	−0.1180	0.038*
H15B	0.9523	1.0895	−0.0398	0.038*
H15C	0.8769	1.1658	0.0439	0.038*
C16	0.90338(18)	0.8970(2)	0.11981(18)	0.0216(4)
H16A	0.9103	0.9579	0.1955	0.032*
H16B	0.9972	0.8858	0.1208	0.032*
H16C	0.8669	0.8046	0.1296	0.032*
N1	0.51840(15)	0.55661(16)	0.47953(14)	0.0167(3)
H1H	0.5212	0.4644	0.4883	0.020*
N2	0.47144(14)	0.97762(16)	0.01340(14)	0.0161(3)
H2A	0.4558	1.0659	−0.0134	0.019*
O1	0.72568(12)	0.63768(15)	0.28467(12)	0.0202(3)
H1	0.6914	0.6933	0.2183	0.030*
O2	0.39770(13)	0.75448(14)	0.48617(13)	0.0208(3)
O3	0.31662(12)	0.53303(13)	0.49685(12)	0.0159(3)
O4	0.16730(13)	0.92146(15)	−0.17056(12)	0.0248(3)
H4	0.1934	0.9795	−0.2139	0.037*
O5	0.61246(13)	0.78670(13)	0.03847(12)	0.0183(3)
O6	0.66989(12)	1.00624(13)	−0.00871(12)	0.0175(3)

90 minutes and concentrated *in vacuo*. The residue was dissolved in ethylacetate (50 mL), washed twice with 50 mL of water, brine (50 mL), dried with Na_2SO_4 and concentrated *in vacuo* to afford the title compound (3.46 g, yield > 90%) as a colourless, crystalline solid [5, 6].

Experimental details

All hydrogen atoms were positioned geometrically, allowed to ride on their parent atoms and refined isotropically. The absolute structure was determined by refinement of the Flack parameter (0.10(15) [2]) using Parsons method [4].

Discussion

Protection of amines, amino acids and amino alcohol with a *tert*-butyloxycarbonyl (Boc) group can be conducted under aqueous or anhydrous condition, using a base and anhydrous Boc_2O. *N*-Protected amino alcohols are versatile building blocks in organic synthesis and have many applications in the synthesis of important chemicals and pharmeceuticals. We have reported one of the application of Boc protected alaninol derivatives previously [5–7].

The crystal structure of the title compound contains two symmetrically non-equivalent molecules in the asymmetric unit. Each molecule consists of a *tert*-butoxy unit and a *N*-[2-hydroxy-1-methylethyl]amidyl moiety, which confirms the successful synthesis of the title compound. The two molecules also have of a stereogenic centre with *S*-configuration at C2 in one molecule and C10 in the other (*cf*. the figure). Classical intermolecular N—H···O hydrogen bonding is observed between the amide hydrogen atoms and carbonyl oxygen atoms (N1···O2′ = 2.977(2) Å; ′ = $-x+1$, $y-1/2$, $-z+1$; N2···O5″ = 3.047(2) Å; ″ = $-x+1, y+1/2, -z$) of neighbouring molecules. Linking in this manner forms chains, which extent along the *b* axis. The OH groups are involved in classical O—H···O hydrogen bonding with carbonyl oxygen atoms (O1···O5 = 2.800(2) Å) and OH groups (O4···O1″ = 2.855(2) Å) of neighboring molecules. The O—H···O hydrogen bonding patterns sew the chains together resulting in a two dimensional network. All bond lengths and angles are in the expected ranges for this class of compounds [8].

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