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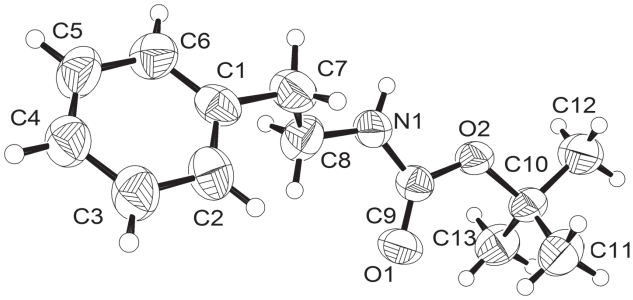
Crystal structure of *tert*-butyl 2-phenylethylcarbamate, $C_{13}H_{19}NO_2$



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Abstract

$C_{13}H_{19}NO_2$, monoclinic, $P2_1/n$ (no. 14), $a = 5.2692(3)$ Å, $b = 13.8663(9)$ Å, $c = 17.8020(13)$ Å, $\beta = 93.323(6)°$, $V = 1298.50(15)$, $Z = 4$, $R_{gt}(F) = 0.0590$, $wR_{ref}(F^2) = 0.1932$, $T = 293$ K.

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The asymmetric unit of the title structure is shown in the figure. Tables 1 and 2 contain details of the measurement method and a list of the atoms including atomic coordinates and displacement parameters.

Source of material

tert-Butyl 2-phenylethylcarbamate was synthesized from the reaction of 2-phenylethylamine with 1.2 equivalents of di-*tert*-butyl dicarbonate in the presence of 1.5 equivalents of triethylamine in dichloromethane at 0 °C for 15 minutes and

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Table 1: Data collection and handling.

Crystal:	Colourless needle
	Size 0.57 × 0.18 × 0.10 mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ:	0.8 cm^{-1}
Diffractometer, scan mode:	SuperNova, ω
$2\theta_{max}$, completeness:	59.8°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	6696, 3146, 0.022
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 1985
$N(param)_{refined}$:	186
Programs:	SHELX [14], CrysAlis^{PRO} [15], WinGX [16]

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

Atom	x	y	z	U_{iso}*/U_{eq}
O1	−0.1282(3)	0.15249(12)	−0.02887(9)	0.0674(5)
O2	0.1718(2)	0.24524(10)	0.03205(8)	0.0595(4)
C1[a]	0.3832(2)	−0.01016(10)	−0.21915(8)	0.056(2)
C2[a]	0.1667(2)	−0.00308(10)	−0.26730(8)	0.0677(17)
H2[a]	0.0548	0.0481	−0.2625	0.081*
C3[a]	0.1176(2)	−0.07251(10)	−0.32257(8)	0.0730(16)
H3[a]	−0.0272	−0.0678	−0.3548	0.088*
C4[a]	0.2850(2)	−0.14901(10)	−0.32968(8)	0.0682(17)
H4[a]	0.2521	−0.1955	−0.3667	0.082*
C5[a]	0.5015(2)	−0.15610(10)	−0.28153(8)	0.0739(17)
H5[a]	0.6134	−0.2073	−0.2863	0.089*
C6[a]	0.5506(2)	−0.08667(10)	−0.22626(8)	0.0676(17)
H6[a]	0.6954	−0.0914	−0.1940	0.081*
C7[a]	0.4229(17)	0.0638(8)	−0.1572(5)	0.070(3)
H7A[a]	0.6014	0.0643	−0.1405	0.084*
H7B[a]	0.3815	0.1270	−0.1777	0.084*
C8[a]	0.2677(7)	0.0468(3)	−0.0900(2)	0.070(3)
H8A[a]	0.0906	0.0388	−0.1068	0.084*
H8B[a]	0.3242	−0.0121	−0.0649	0.084*
C1A[b]	0.3508(7)	−0.0104(3)	−0.2191(2)	0.055(3)
C2A[b]	0.1686(7)	−0.0229(3)	−0.2779(2)	0.068(2)
H2A[b]	0.0330	0.0198	−0.2839	0.082*
C3A[b]	0.1890(7)	−0.0994(3)	−0.3277(2)	0.078(2)
H3A[b]	0.0671	−0.1078	−0.3670	0.093*
C4A[b]	0.3917(7)	−0.1633(3)	−0.3186(2)	0.075(2)
H4A[b]	0.4053	−0.2145	−0.3519	0.090*
C5A[b]	0.5739(7)	−0.1508(3)	−0.2598(2)	0.087(2)
H5A[b]	0.7095	−0.1936	−0.2538	0.105*

Table 2 (continued)

Atom	x	y	z	U_{iso}*/U_{eq}
C6A[b]	0.5535(7)	−0.0743(3)	−0.2100(2)	0.076(2)
H6A[b]	0.6754	−0.0660	−0.1707	0.091*
C7A[b]	0.349(2)	0.0737(9)	−0.1667(6)	0.064(3)
H7A1[b]	0.5195	0.1005	−0.1611	0.076*
H7A2[b]	0.2380	0.1231	−0.1887	0.076*
C8A[b]	0.2655(13)	0.0486(4)	−0.0927(4)	0.066(4)
H8A1[b]	0.0885	0.0292	−0.0980	0.080*
H8A2[b]	0.3630	−0.0064	−0.0738	0.080*
C9	0.0918(4)	0.17255(14)	−0.01289(11)	0.0506(5)
C10	−0.0073(4)	0.30852(16)	0.06855(12)	0.0579(5)
C11	−0.1721(5)	0.36138(18)	0.01000(16)	0.0782(7)
H11A	−0.0665	0.3920	−0.0251	0.117*
H11B	−0.2708	0.4093	0.0341	0.117*
H11C	−0.2840	0.3164	−0.0162	0.117*
C12	0.1684(5)	0.3775(2)	0.11144(18)	0.0922(10)
H12A	0.2756	0.3423	0.1472	0.138*
H12B	0.0698	0.4237	0.1374	0.138*
H12C	0.2718	0.4105	0.0770	0.138*
C13	−0.1626(5)	0.25076(18)	0.12148(14)	0.0718(7)
H13A	−0.2796	0.2101	0.0928	0.108*
H13B	−0.2556	0.2939	0.1519	0.108*
H13C	−0.0514	0.2116	0.1533	0.108*
N1	0.2909(3)	0.12580(13)	−0.03773(10)	0.0613(5)
H1	0.4407	0.1437	−0.0217	0.074*

[a]Occupancy: 0.558(8); [b]Occupancy: 0.442(8).

then under reflux for 1 h. The crude product was purified by crystallization from hexane to give the title compound (90%) as colourless crystals, mp 56–57 °C (lit. 56.1–56.4 °C [1]; 54–55 °C [2]; 55–56 °C [3]).

Experimental details

The methylbenzene segment of the molecule is disordered and was refined with the occupancies 56(1)% and 44(1)%. The aromatic ring was constrained into a regular hexagon with C—C distances of 1.39 Å. All H atoms were placed in calculated positions and refined using a riding model. For the methyl groups, C—H bonds were fixed at 0.96 Å and U_{iso}(H) set to 1.5U_{eq}(C) with free rotation around the C—C bond. For the rest of the hydrogens, U_{iso}(H) was set to 1.2U_{eq}(C) with aromatic C—H and N—H distances of 0.93 and 0.86 Å, respectively.

Discussion

Various carbamate and thiocarbamate derivatives show antimicrobial activities [4–6] and various synthetic procedures have been reported for the production of carbamates. Convenient and efficient syntheses involve reactions of amino acids with *Boc*-benzotriazoles in the presence of triethylamine in aqueous acetonitrile at room temperature [7], of amines with phenyl 4,5-dichloro-6-oxopyridazine-1(6*H*)-carboxylate in tetrahydrofuran (THF) at room temperature [8], of nitriles with an excess of di-*tert*-butyl dicarbonate in the presence of a catalytic amount of nickel boride in methanol at room temperature [9], of aromatic carboxylic acids with di-*tert*-butyl dicarbonate in the presence of sodium azide, tetrabutylammonium bromide and zinc(II) trifluoromethanesulfonate in THF at 40 °C [10] and of nitro aromatics with excess chloroformates in the presence of zinc and ammonium chloride in aqueous THF at 0 °C [11]. High yields of substituted derivatives can be produced from regioselective lithiation of aryl carbamates using lithium reagents, at room temperature, followed by treatment of the lithium intermediates obtained *in situ* with electrophiles [12, 13].

The asymmetric unit of the title structure consists of one molecule with a disordered benzyl fragment. All bond lengths and angles are in the expected ranges. In the crystal structure, the amide group is involved in a N—H···O hydrogen bond (N···O distance = 3.078(2)Å, N—H···O angle = 153.3°) leading to the formation of *C*(4) chains along [100].

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