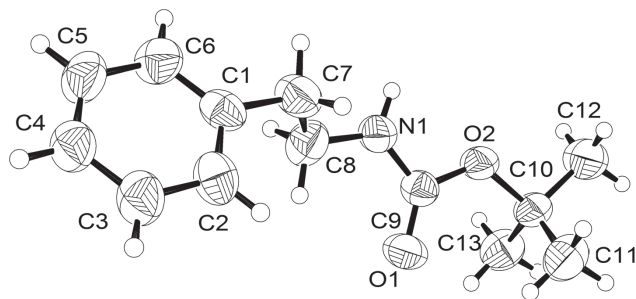


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# Crystal structure of *tert*-butyl 2-phenylethylcarbamate, C<sub>13</sub>H<sub>19</sub>NO<sub>2</sub>



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## Abstract

C<sub>13</sub>H<sub>19</sub>NO<sub>2</sub>, monoclinic, *P*2<sub>1</sub>/*n* (no. 14), *a* = 5.2692(3) Å, *b* = 13.8663(9) Å, *c* = 17.8020(13) Å, β = 93.323(6)°, *V* = 1298.50(15), *Z* = 4, *R*<sub>gt</sub>(*F*) = 0.0590, *wR*<sub>ref</sub>(*F*<sup>2</sup>) = 0.1932, *T* = 293 K.

CCDC no.: 1487250

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 Kα radiation (0.71073 Å)
μ:	0.8 cm <sup>-1</sup>
Diffractometer, scan mode:	SuperNova, ω
2θ <sub>max</sub> , completeness:	59.8°, >99%
<i>N</i> ( <i>hkl</i> ) <sub>measured</sub> , <i>N</i> ( <i>hkl</i> ) <sub>unique</sub> , <i>R</i> <sub>int</sub> :	6696, 3146, 0.022
Criterion for <i>I</i> <sub>obs</sub> , <i>N</i> ( <i>hkl</i> ) <sub>gt</sub> :	<i>I</i> <sub>obs</sub> > 2 σ( <i>I</i> <sub>obs</sub> ), 1985
<i>N</i> ( <i>param</i> ) <sub>refined</sub> :	186
Programs:	SHELX [14], CrysAlis <sup>PRO</sup> [15], WinGX [16]

Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å<sup>2</sup>).

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

Table 2 (continued)

Atom	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> <sub>iso</sub> <sup>*</sup> / <i>U</i> <sub>eq</sub>
C6A <sup>b</sup>	0.5535(7)	-0.0743(3)	-0.2100(2)	0.076(2)
H6A <sup>b</sup>	0.6754	-0.0660	-0.1707	0.091*
C7A <sup>b</sup>	0.349(2)	0.0737(9)	-0.1667(6)	0.064(3)
H7A1 <sup>b</sup>	0.5195	0.1005	-0.1611	0.076*
H7A2 <sup>b</sup>	0.2380	0.1231	-0.1887	0.076*
C8A <sup>b</sup>	0.2655(13)	0.0486(4)	-0.0927(4)	0.066(4)
H8A1 <sup>b</sup>	0.0885	0.0292	-0.0980	0.080*
H8A2 <sup>b</sup>	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*

<sup>a</sup>Occupancy: 0.558(8); <sup>b</sup>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*<sub>iso</sub>(H) set to 1.5*U*<sub>eq</sub>(C) with free rotation around the C–C bond. For the rest of the hydrogens, *U*<sub>iso</sub>(H) was set to 1.2*U*<sub>eq</sub>(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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