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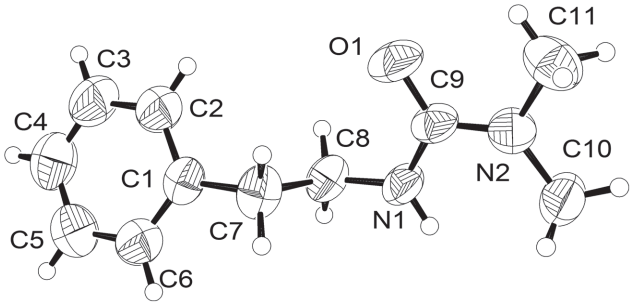
Crystal structure of 1,1-dimethyl-3-(2-phenylethyl)urea, $C_{11}H_{16}N_2O$



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Abstract

$C_{11}H_{16}N_2O$, orthorhombic, *Pbca* (no. 61), $a = 10.7388(6)$ Å, $b = 9.8449(5)$ Å, $c = 21.1259(14)$ Å, $V = 2233.5(2)$ Å^3, $Z = 8$, $R_{gt}(F) = 0.0582$, $wR_{ref}(F^2) = 0.1795$, $T = 293$ K.

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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

1,1-Dimethyl-3-(2-phenylethyl)urea was synthesized from reaction of (2-phenylethyl)amine with dimethylcarbamoyl chloride (1.1 mole equivalents) in the presence of triethylamine (1.4 mole equivalents) in dichloromethane under reflux for 1 h. Recrystallization using a mixture of ethyl acetate

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Table 1: Data collection and handling.

Crystal:	Colourless, block Size 0.37 × 0.17 × 0.10 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	0.8 cm^{-1}
Diffractometer, scan mode:	SuperNova, ω
$2\theta_{max}$, completeness:	59.4°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	7627, 2680, 0.025
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 1669
$N(param)_{refined}$:	129
Programs:	SHELX [13], CrysAlis^{PRO} [14], WinGX [15]

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

Atom	x	y	z	U_{iso}*/U_{eq}
C1	0.48541(18)	0.06211(19)	0.35228(8)	0.0580(5)
C2	0.5322(2)	0.1652(2)	0.31509(11)	0.0743(6)
H2	0.6015	0.2135	0.3288	0.089*
C3	0.4777(3)	0.1979(3)	0.25766(12)	0.0921(8)
H3	0.5107	0.2678	0.2333	0.111*
C4	0.3765(3)	0.1288(3)	0.23680(13)	0.0993(8)
H4	0.3409	0.1505	0.1980	0.119*
C5	0.3273(2)	0.0284(3)	0.27259(13)	0.0975(8)
H5	0.2573	−0.0183	0.2586	0.117*
C6	0.3810(2)	−0.0053(2)	0.33001(11)	0.0784(6)
H6	0.3463	−0.0746	0.3542	0.094*
C7	0.54501(19)	0.0256(2)	0.41422(9)	0.0687(6)
H7A	0.4881	−0.0306	0.4384	0.082*
H7B	0.5599	0.1080	0.4382	0.082*
C8	0.66752(18)	−0.04998(19)	0.40590(9)	0.0618(5)
H8A	0.6514	−0.1347	0.3839	0.074*
H8B	0.7222	0.0041	0.3794	0.074*
C9	0.79739(16)	0.01667(16)	0.49494(10)	0.0565(5)
C10	0.8638(2)	−0.1564(2)	0.57266(11)	0.0833(7)
H10A	0.7839	−0.1822	0.5891	0.125*
H10B	0.9248	−0.1612	0.6058	0.125*
H10C	0.8868	−0.2170	0.5390	0.125*
C11	0.9276(2)	0.0821(2)	0.58375(12)	0.0848(7)
H11A	0.9358	0.1629	0.5587	0.127*
H11B	1.0088	0.0470	0.5934	0.127*
H11C	0.8846	0.1031	0.6224	0.127*
N1	0.73091(15)	−0.07966(14)	0.46471(7)	0.0604(4)
H1	0.7261	−0.1599	0.4806	0.072*
N2	0.85761(15)	−0.01881(16)	0.54863(8)	0.0675(5)
O1	0.80282(14)	0.13384(12)	0.47359(8)	0.0751(5)

and diethyl ether (1:3 by volume) gave the title compound (~99%) as colourless crystals, mp 89–90 °C (lit. 88–90 °C [1]; 81–82 °C [2]).

Experimental details

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 1.5 U_{eq}(C) with free rotation around the C—C bond (HFIX 137 in SHELX [13]). For the rest of the hydrogens, U_{iso}(H) was set to 1.2 U_{eq}(C) with aromatic C—H and N—H distances of 0.93 and 0.86 Å, respectively.

Discussion

The synthesis of substituted ureas is of great interest for both academia and industry since ureas represent various biologically active compounds [2–4]. Urea derivatives can be synthesized using various efficient procedures [5–7]. Regioselective lithiation of aromatic ureas with lithium reagents followed by reaction with electrophiles at low temperatures is considered to be one of the most clean, efficient and convenient processes for the production of a large number of substituted derivatives in high yields [8–11].

The asymetric unit (Figure) of the crystal structure consists of a molecule of $C_{11}H_{16}N_2O$. In the crystal, the amide group is involved in N—H···O hydrogen bonding (N···O distance = 2.850(2) Å, N—H···O angle = 152.4°) leading to the formation of C(4) chains [12] along the [010] direction.

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