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N-Benzylacetamide

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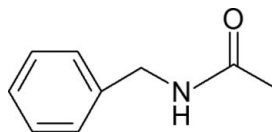
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Key indicators: single-crystal X-ray study; $T = 298$ K; mean $\sigma(\text{C}-\text{C}) = 0.002$ Å; R factor = 0.039; wR factor = 0.096; data-to-parameter ratio = 20.4.

Molecules of *N*-benzylacetamide, $\text{C}_9\text{H}_{11}\text{NO}$, are interconnected by a framework of weak intermolecular $\text{N}-\text{H}\cdots\text{O}$ hydrogen bonds. The molecules form infinite hydrogen-bonded chains, parallel to the *a* direction.

Related literature

For related literature, see: Desiraju & Steiner (1999); Flakus & Michta (2003, 2004, 2005); Flakus & Pyzik (2006); Flakus *et al.* (2003, 2007); Kotera *et al.* (1968).



Experimental

Crystal data

$\text{C}_9\text{H}_{11}\text{NO}$
 $M_r = 149.19$
Monoclinic, $P2_1/c$
 $a = 4.8383$ (10) Å
 $b = 14.906$ (3) Å
 $c = 11.663$ (2) Å
 $\beta = 100.04$ (3)°

$V = 828.3$ (3) Å³
 $Z = 4$
Mo $K\alpha$ radiation
 $\mu = 0.08$ mm⁻¹
 $T = 298$ (2) K
 $0.60 \times 0.12 \times 0.01$ mm

Data collection

Oxford Diffraction KM-4 CCD
Sapphire3 diffractometer
Absorption correction: none
7651 measured reflections

2718 independent reflections
1377 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.017$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.039$
 $wR(F^2) = 0.096$
 $S = 1.00$
2718 reflections
133 parameters

H atoms treated by a mixture of independent and constrained refinement
 $\Delta\rho_{\text{max}} = 0.18$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.18$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{N1}-\text{H1N}\cdots\text{O1}^i$	0.90 (1)	2.02 (1)	2.906 (1)	168.7 (9)

Symmetry code: (i) $x + 1, y, z$.

Data collection: *CrysAlis CCD* (Oxford Diffraction, 2007); cell refinement: *CrysAlis CCD*; data reduction: *CrysAlis RED* (Oxford Diffraction, 2007); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *publCIF* (Westrip, 2007).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: AV3099).

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supplementary materials

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N-Benzylacetamide

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Comment

We report here the synthesis, isolation of single crystals and structure determination of *N*-benzylacetamide, (I).

The title compound is subject of our study of a generation mechanism of IR spectra of hydrogen-bonded molecular crystals (Flakus & Michta, 2004, 2005; Flakus *et al.*, 2007; Flakus, Tyl & Jones, 2003; Flakus & Pyzik, 2006). The spectral studies were preceded by determination of the crystal X-ray structure. Measurement of the IR spectra and theoretical analysis of the results concerned *e.g.* the linear dichroic effects, the H/D isotopic and temperature effects, observed in the solid-state IR spectra of the hydrogen and of the deuterium bond at the frequency ranges of the $\nu_{\text{N-H}}$ and $\nu_{\text{N-D}}$ bands, respectively. Some spectacular effects are especially visible for those systems, where the proton-acceptor atom is oxygen (Flakus & Michta, 2003).

The structure of (I) with the atomic numbering scheme is presented in Fig. 1. Molecules of (I) are interconnected by a framework of intermolecular N—H \cdots O hydrogen bonds, *viz.* N1—H1N \cdots O1, respectively, as shown in Fig. 2 and detailed in Table 1. The Fig. 2 also shows that in the crystal structure of (I) the molecules interact *via* N—H \cdots O hydrogen bonds, forming infinite chains perpendicular to the *b* axis. The values of the H—*A* and *D* \cdots *A* distances and the *D*—H \cdots *A* angle (Table 1) characterize this bond as a weak hydrogen bond (Desiraju & Steiner, 1999), and agree with relevant data for *N*-benzylacetamide forming intermolecular N—H \cdots O hydrogen bonds [*D* \cdots *A* = 2.90 (12) Å and *D*—H \cdots *A* = 168.7 (9)°]. The weakening of the intermolecular hydrogen bond in (I) is supported by IR spectroscopic data. The band of the isolated N—H stretching vibration, $\nu_{\text{N-H}}$, was located in the 3400–3100 cm⁻¹ frequency range.

Experimental

Benzylamine (30 g, 0.28 mol) was added to a solution of acetic acid (170 g, 2.83 mol) at 130–150°C with stirring. The reaction mixture was then brought to reflux for 6 h, allowed to cool and excess of acetic acid solution was evaporated under reduced pressure to give a white precipitate. The precipitate was dissolved in petroleum ether and the solution was left for crystallization at room temperature. After a few days, the deposited white crystals were collected and recrystallized from petroleum ether, giving single, needle-shaped crystals of quality suitable for X-ray measurement. The yield 34 g, 80%; m.p. 57°C; literature m.p. 58 – 59°C (Kotera *et al.*, 1968).

The IR spectra of *N*-benzylacetamide crystals were measured by a transmission method in the frequency ranges of the proton and deuteron stretching vibration bands, $\nu_{\text{N-H}}$ and $\nu_{\text{N-D}}$. Spectral experiment were performed at room temperature and at the temperature of liquid nitrogen (77 K), using polarized radiation. The solid-state spectra were measured with the 2 cm⁻¹ resolution for the normal incidence of the radiation beam, using the FT—IR Nicolet Magna 560 spectrometer.

Refinement

Some of the hydrogen atoms were located in a difference Fourier map and refined freely; other H atoms were placed in calculated positions 0,98 Å (methyl C) and refined as riding with $U_{iso}(\text{H}) = 1,5U_{eq}(\text{C})$ for the methyl H atoms.

Figures

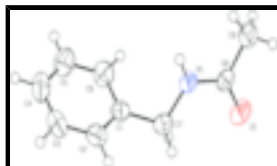


Fig. 1. The conformation of *N*-benzylacetamide molecule with the atom numbering scheme. Atomic displacement ellipsoids represent 50% probability level.

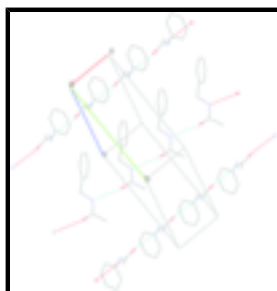


Fig. 2. The crystal packing of the title compound, viewed approximately down the *b* axis. The intermolecular N—H...O interactions are represented by dashed lines.

N-benzylacetamide

Crystal data

$\text{C}_9\text{H}_{11}\text{NO}$

$M_r = 149.19$

Monoclinic, $P2_1/c$

$a = 4.8383(10) \text{ \AA}$

$b = 14.906(3) \text{ \AA}$

$c = 11.663(2) \text{ \AA}$

$\beta = 100.04(3)^\circ$

$V = 828.3(3) \text{ \AA}^3$

$Z = 4$

$F_{000} = 320$

$D_x = 1.196 \text{ Mg m}^{-3}$

Mo $K\alpha$ radiation

$\lambda = 0.71073 \text{ \AA}$

$\mu = 0.08 \text{ mm}^{-1}$

$T = 298(2) \text{ K}$

Needle, white

$0.60 \times 0.12 \times 0.01 \text{ mm}$

Data collection

Oxford Diffraction KM-4 CCD Sapphire3 diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

$T = 298(2) \text{ K}$

θ scans

Absorption correction: none

7651 measured reflections

1377 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.017$

$\theta_{\text{max}} = 32.8^\circ$

$\theta_{\text{min}} = 3.3^\circ$

$h = -3 \rightarrow 7$

$k = -22 \rightarrow 22$

$l = -17 \rightarrow 16$

2718 independent reflections

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.039$	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2) = 0.096$	$w = 1/[\sigma^2(F_o^2) + (0.0459P)^2]$
$S = 1.00$	where $P = (F_o^2 + 2F_c^2)/3$
2718 reflections	$(\Delta/\sigma)_{\max} < 0.001$
133 parameters	$\Delta\rho_{\max} = 0.18 \text{ e } \text{\AA}^{-3}$
Primary atom site location: structure-invariant direct methods	$\Delta\rho_{\min} = -0.18 \text{ e } \text{\AA}^{-3}$
	Extinction correction: none

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R -factor wR and goodness of fit S are based on F^2 , conventional R -factors R are based on F , with F set to zero for negative F^2 . The threshold expression of $F^2 > 2\sigma(F^2)$ is used only for calculating R -factors(gt) *etc.* and is not relevant to the choice of reflections for refinement. R -factors based on F^2 are statistically about twice as large as those based on F , and R -factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
N1	0.17528 (16)	0.41069 (5)	0.71931 (7)	0.0389 (2)
O1	-0.22568 (12)	0.44356 (5)	0.78353 (6)	0.0548 (2)
C1	0.22770 (18)	0.31836 (6)	0.55164 (8)	0.0382 (2)
C2	0.3398 (2)	0.23405 (7)	0.54011 (9)	0.0454 (3)
C3	0.4952 (2)	0.21655 (8)	0.45370 (9)	0.0520 (3)
C4	0.5422 (2)	0.28301 (9)	0.37859 (9)	0.0553 (3)
C5	0.4327 (3)	0.36742 (9)	0.38894 (9)	0.0569 (3)
C6	0.2780 (2)	0.38484 (8)	0.47474 (9)	0.0490 (3)
C7	0.0568 (2)	0.33683 (8)	0.64518 (10)	0.0471 (3)
C8	0.02600 (17)	0.45714 (6)	0.78448 (8)	0.0372 (2)
C9	0.1794 (2)	0.52948 (8)	0.85890 (9)	0.0546 (3)
H1N	0.362 (2)	0.4186 (7)	0.7291 (9)	0.052 (3)*
H2	0.302 (2)	0.1875 (7)	0.5928 (10)	0.061 (3)*
H3	0.572 (2)	0.1552 (8)	0.4457 (10)	0.068 (3)*
H4	0.648 (3)	0.2737 (8)	0.3205 (11)	0.070 (4)*
H5	0.465 (3)	0.4141 (9)	0.3392 (11)	0.083 (4)*

supplementary materials

H6	0.213 (2)	0.4431 (8)	0.4854 (10)	0.060 (3)*
H9A	0.1117	0.5871	0.8296	0.082*
H9B	0.3766	0.5251	0.8573	0.082*
H9C	0.1483	0.5227	0.9375	0.082*
H17	-0.141 (3)	0.3521 (7)	0.6096 (9)	0.063 (3)*
H27	0.045 (2)	0.2809 (8)	0.6986 (10)	0.067 (3)*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0224 (4)	0.0499 (5)	0.0451 (5)	-0.0047 (3)	0.0080 (3)	-0.0065 (4)
O1	0.0251 (4)	0.0719 (5)	0.0702 (5)	-0.0032 (3)	0.0162 (3)	-0.0056 (4)
C1	0.0290 (4)	0.0436 (6)	0.0405 (5)	-0.0056 (4)	0.0021 (4)	-0.0050 (4)
C2	0.0484 (6)	0.0404 (6)	0.0456 (6)	-0.0050 (4)	0.0035 (5)	-0.0017 (5)
C3	0.0539 (6)	0.0490 (7)	0.0506 (6)	0.0076 (5)	0.0022 (5)	-0.0127 (6)
C4	0.0531 (7)	0.0732 (8)	0.0406 (6)	0.0062 (6)	0.0107 (5)	-0.0091 (6)
C5	0.0627 (7)	0.0624 (8)	0.0475 (6)	0.0040 (6)	0.0152 (5)	0.0105 (6)
C6	0.0486 (6)	0.0437 (6)	0.0554 (6)	0.0086 (5)	0.0114 (5)	0.0035 (5)
C7	0.0355 (5)	0.0545 (7)	0.0529 (6)	-0.0121 (5)	0.0123 (5)	-0.0099 (5)
C8	0.0265 (4)	0.0462 (6)	0.0404 (5)	0.0004 (4)	0.0101 (4)	0.0049 (4)
C9	0.0422 (6)	0.0658 (7)	0.0599 (7)	-0.0076 (5)	0.0203 (5)	-0.0174 (6)

Geometric parameters (\AA , $^\circ$)

N1—C8	1.3305 (12)	C4—C5	1.3786 (18)
N1—C7	1.4544 (13)	C4—H4	0.928 (12)
N1—H1N	0.897 (11)	C5—C6	1.3747 (16)
O1—C8	1.2325 (10)	C5—H5	0.937 (14)
C1—C2	1.3845 (14)	C6—H6	0.940 (11)
C1—C6	1.3863 (14)	C7—H17	1.000 (12)
C1—C7	1.5046 (14)	C7—H27	1.048 (12)
C2—C3	1.3831 (16)	C8—C9	1.4976 (14)
C2—H2	0.966 (11)	C9—H9A	0.9600
C3—C4	1.3674 (16)	C9—H9B	0.9600
C3—H3	0.998 (12)	C9—H9C	0.9600
C8—N1—C7	122.45 (8)	C5—C6—C1	121.00 (11)
C8—N1—H1N	119.5 (7)	C5—C6—H6	120.6 (7)
C7—N1—H1N	117.3 (7)	C1—C6—H6	118.3 (7)
C2—C1—C6	118.04 (10)	N1—C7—C1	111.11 (8)
C2—C1—C7	120.70 (9)	N1—C7—H17	108.9 (6)
C6—C1—C7	121.26 (9)	C1—C7—H17	110.3 (6)
C3—C2—C1	120.92 (10)	N1—C7—H27	107.8 (6)
C3—C2—H2	121.0 (7)	C1—C7—H27	112.2 (6)
C1—C2—H2	118.0 (7)	H17—C7—H27	106.3 (9)
C4—C3—C2	120.15 (11)	O1—C8—N1	122.90 (9)
C4—C3—H3	119.9 (7)	O1—C8—C9	120.84 (8)
C2—C3—H3	120.0 (7)	N1—C8—C9	116.25 (8)
C3—C4—C5	119.73 (11)	C8—C9—H9A	109.5

C3—C4—H4	122.2 (8)	C8—C9—H9B	109.5
C5—C4—H4	118.0 (8)	H9A—C9—H9B	109.5
C6—C5—C4	120.15 (12)	C8—C9—H9C	109.5
C6—C5—H5	118.8 (8)	H9A—C9—H9C	109.5
C4—C5—H5	121.1 (8)	H9B—C9—H9C	109.5

Hydrogen-bond geometry (\AA , $^\circ$)

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
N1—H1N \cdots O1 ⁱ	0.90 (1)	2.02 (1)	2.906 (1)	168.7 (9)

Symmetry codes: (i) $x+1, y, z$.

Fig. 1

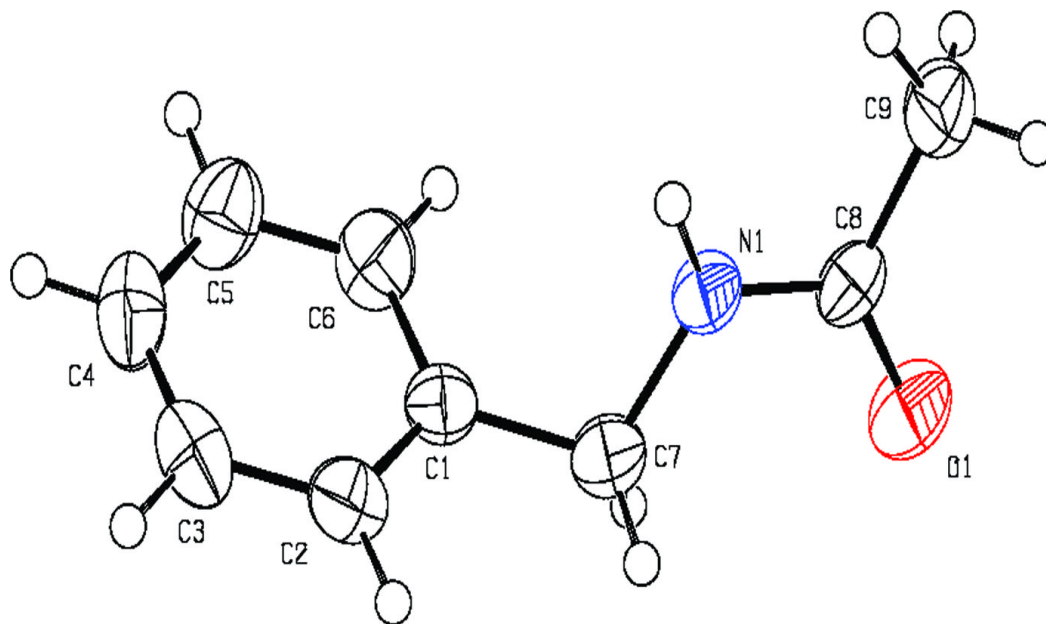


Fig. 2

