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N-Isopropylbenzamide

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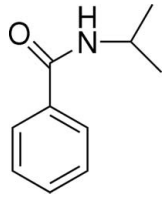
Key indicators: single-crystal X-ray study; T = 150 K; mean σ(C–C) = 0.007 Å; R factor = 0.058; wR factor = 0.148; data-to-parameter ratio = 7.8.

In the title compound, $C_{10}H_{13}NO$, the dihedral angle between the amide group and the phenyl ring is 30.0 (3)°. In the crystal structure, intermolecular N—H···O hydrogen bonds link molecules into one-dimensional chains along the *a* axis.

Related literature

For related literature, see: Clayden *et al.* (2006); Kopka *et al.* (2005); Smart (2001); Van Waarde *et al.* (2004); Stephenson, Wilson *et al.* (2008); Stephenson, van Oosten *et al.* (2008).



Experimental

Crystal data

$C_{10}H_{13}NO$
M_r = 163.21
Monoclinic, $P2_1$
a = 5.0093 (7) Å
b = 10.1250 (13) Å
c = 9.6714 (14) Å
β = 104.133 (7)°
V = 475.68 (11) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.07 mm^{-1}
T = 150 (1) K
0.14 × 0.13 × 0.08 mm

Data collection

Bruker Nonius KappaCCD diffractometer
Absorption correction: multi-scan (*SORTAV*; Blessing 1995)
T_{min} = 0.954, T_{max} = 0.996
2462 measured reflections
887 independent reflections
621 reflections with $I > 2\sigma(I)$
R_{int} = 0.061

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.057
$wR(F^2)$ = 0.148
S = 1.06
887 reflections
114 parameters
1 restraint
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.18 e Å^{-3}
$\Delta\rho_{min}$ = −0.21 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1N···O1^{i}	0.83 (5)	2.10 (5)	2.890 (5)	160 (5)

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

Data collection: *COLLECT* (Nonius, 2002); cell refinement: *DENZO-SMN* (Otwinowski & Minor, 1997); data reduction: *DENZO-SMN*; program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1994); program(s) used to refine structure: *SHELXTL* (Sheldrick, 2008); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB2729).

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supporting information

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N-Isopropylbenzamide

Erik M. van Oosten, Alan J. Lough and Neil Vasdev

S1. Comment

The isopropylamine moiety is a common structural feature in many pharmaceutical compounds, in particular among *β*-adrenergic receptor antagonists (*β*-blockers) (Van Waarde *et al.*, 2004; Kopka *et al.*, 2005). Recent work in our laboratory (Stephenson, Wilson *et al.*, 2008; Stephenson, van Oosten *et al.*, 2008) and others (Van Waarde *et al.*, 2004; Kopka *et al.*, 2005) has focused on developing *β*-blockers labeled with the positron emitting isotope fluorine-18 ($t_{1/2}$ = 109.7 min) at the isopropyl moiety for medical imaging with positron emission tomography. It is established that substitution of fluorine into a drug often enhances its biological properties (Smart, 2001). Our goal is to structurally characterize the isopropylamine group for comparison with fluorinated analogs developed in our laboratory. Herein we report the single-crystal X-ray structure of the title compound, (I), (Fig. 1).

The dihedral angle between the essentially planar set of atoms C7/O1/N1/C8 [r.m.s. deviation 0.006 Å] and the benzene ring (C1–C6) in (I) is 30.0 (3)°. In the crystal structure, intermolecular N—H···O hydrogen bonds link molecules into one-dimensional chains along the *a* axis (Table 1, Fig. 2).

S2. Experimental

N-Isopropylbenzamide was made according to a literature procedure (Clayden *et al.*, 2006), with minor modifications. Benzoyl chloride (0.825 ml, 7.11 mmol) was added to CH_2Cl_2 (17 ml, 0.4 *M*) under nitrogen. The mixture was cooled in an ice bath to 273 K and stirred for 10 min. Isopropylamine (1.8 ml, 21.33 mmol) was added dropwise. Upon completion of this addition the ice bath was removed and the reaction mixture was stirred at room temperature for 1.5 h. When the starting material was consumed (monitored by TLC) the reaction mixture was diluted with H_2O (150 ml), extracted with CH_2Cl_2 (3 × 50 ml), washed with H_2O (2 × 100 ml) followed by brine (2 × 100 ml), dried over Na_2SO_4, and concentrated. No further purification was necessary. Colourless blocks of (I) were obtained by slow evaporation of a solution of the title compound in $CDCl_3$. ^{1}H NMR ($CDCl_3$, 300 MHz) d = 7.78–7.67 (m, 2H), 7.51–7.36 (m, 3H), 5.99 (br, 1H), 4.37–4.18 (m, 1H), 1.25 (d, J = 6.5 Hz, 6H) ^{13}C NMR ($CDCl_3$, 75 MHz) d = 166.9, 135.2, 131.5, 128.7, 127.0, 42.1, 23.1.

S3. Refinement

In the absence of significant anamlous dispersion effects, Friedel pairs were merged before refinement. The H atoms bonded to C atoms were placed in calculated positions with C—H = 0.95 Å and 0.98 Å (methyl). They were included in the refinement in the riding-model approximation with U_{iso}(H) = 1.2U_{eq}(C) or U_{iso}(H) = 1.5U_{eq}(C) for methyl H atoms. The position of the H atom bonded to the N atom was refined independently with U_{iso}(H) = 1.2U_{eq}(N).

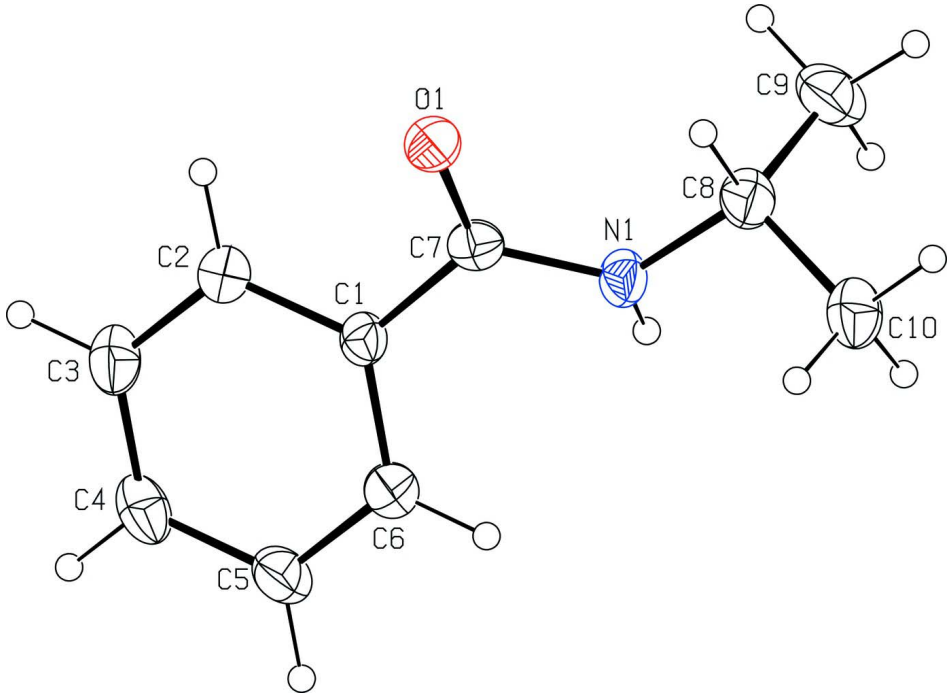

Figure 1

The molecular structure of (I) showing 30% probability displacement ellipsoids (arbitrary spheres for H atoms).

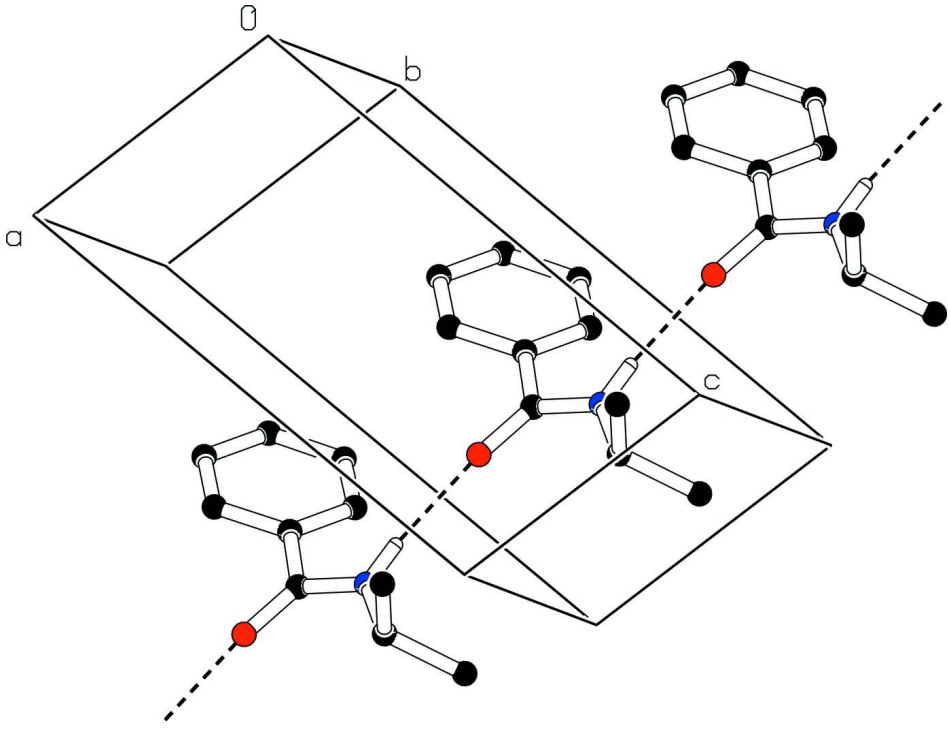

Figure 2

Part of the crystal structure of (I) showing hydrogen bonds as dashed lines.

N-Isopropylbenzamide

Crystal data

$C_{10}H_{13}NO$
$M_r = 163.21$
Monoclinic, $P2_1$
Hall symbol: P 2yb
$a = 5.0093$ (7) Å
$b = 10.1250$ (13) Å
$c = 9.6714$ (14) Å
$\beta = 104.133$ (7)°
$V = 475.68$ (11) Å^3
$Z = 2$
$F(000) = 176$
$D_x = 1.140$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 2462 reflections
$\theta = 3.0$–$25.0°$
$\mu = 0.07$ mm^{-1}
$T = 150$ K
Block, colourless
0.14 × 0.13 × 0.08 mm

Data collection

Bruker Nonius KappaCCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
Detector resolution: 9 pixels mm^{-1}
φ scans and ω scans with κ offsets
Absorption correction: multi-scan (*SORTAV*; Blessing 1995)
$T_{min} = 0.954$, $T_{max} = 0.996$
2462 measured reflections
887 independent reflections
621 reflections with $I > 2\sigma(I)$
$R_{int} = 0.061$
$\theta_{max} = 25.0°$, $\theta_{min} = 3.0°$
$h = -5 \rightarrow 5$
$k = -12 \rightarrow 10$
$l = -11 \rightarrow 11$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.057$
$wR(F^2) = 0.148$
$S = 1.06$
887 reflections
114 parameters
1 restraint
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0784P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.18$ e Å^{-3}
$\Delta\rho_{min} = -0.21$ e Å^{-3}

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 $F^2 > \sigma(F^2)$ 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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.6751 (6)	0.1703 (3)	0.8045 (3)	0.0424 (8)
N1	0.2696 (8)	0.1717 (4)	0.8673 (4)	0.0411 (10)
H1N	0.103 (11)	0.188 (5)	0.839 (5)	0.049*

C1	0.3226 (9)	0.3127 (4)	0.6749 (5)	0.0350 (11)
C2	0.4247 (10)	0.3165 (5)	0.5544 (5)	0.0452 (13)
H2A	0.5601	0.2545	0.5432	0.054*
C3	0.3286 (11)	0.4116 (6)	0.4493 (5)	0.0535 (14)
H3A	0.3957	0.4126	0.3655	0.064*
C4	0.1377 (10)	0.5034 (5)	0.4666 (6)	0.0516 (14)
H4A	0.0734	0.5682	0.3951	0.062*
C5	0.0391 (11)	0.5014 (5)	0.5883 (6)	0.0523 (14)
H5A	−0.0912	0.5656	0.6010	0.063*
C6	0.1296 (9)	0.4064 (5)	0.6915 (5)	0.0451 (13)
H6A	0.0597	0.4051	0.7744	0.054*
C7	0.4356 (8)	0.2117 (4)	0.7864 (4)	0.0362 (12)
C8	0.3461 (10)	0.0743 (5)	0.9808 (5)	0.0474 (13)
H8A	0.5489	0.0586	0.9996	0.057*
C9	0.1984 (13)	−0.0561 (5)	0.9351 (6)	0.0648 (16)
H9A	0.2525	−0.0903	0.8512	0.097*
H9B	−0.0011	−0.0417	0.9116	0.097*
H9C	0.2488	−0.1200	1.0132	0.097*
C10	0.2833 (11)	0.1271 (6)	1.1160 (5)	0.0556 (15)
H10A	0.3743	0.2126	1.1400	0.083*
H10B	0.3510	0.0647	1.1942	0.083*
H10C	0.0839	0.1380	1.1013	0.083*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0319 (17)	0.0442 (18)	0.0515 (17)	0.0024 (15)	0.0112 (13)	0.0032 (16)
N1	0.0326 (19)	0.046 (2)	0.047 (2)	0.003 (2)	0.0149 (18)	0.012 (2)
C1	0.033 (2)	0.035 (2)	0.039 (3)	−0.006 (2)	0.012 (2)	0.000 (2)
C2	0.049 (3)	0.044 (3)	0.046 (3)	0.001 (2)	0.019 (2)	0.003 (2)
C3	0.057 (3)	0.061 (3)	0.046 (3)	−0.003 (3)	0.019 (3)	0.012 (3)
C4	0.049 (3)	0.051 (3)	0.050 (3)	−0.002 (3)	0.004 (3)	0.020 (3)
C5	0.053 (3)	0.040 (3)	0.066 (3)	0.007 (3)	0.017 (3)	0.013 (3)
C6	0.046 (3)	0.042 (3)	0.051 (3)	0.003 (2)	0.017 (2)	0.005 (3)
C7	0.035 (3)	0.034 (3)	0.037 (2)	−0.004 (2)	0.005 (2)	−0.008 (2)
C8	0.041 (3)	0.054 (3)	0.048 (3)	0.012 (3)	0.012 (2)	0.017 (3)
C9	0.085 (4)	0.043 (3)	0.064 (3)	0.010 (3)	0.014 (3)	0.012 (3)
C10	0.062 (3)	0.062 (4)	0.044 (3)	−0.001 (3)	0.015 (2)	0.012 (3)

Geometric parameters (Å, º)

O1—C7	1.242 (5)	C5—C6	1.380 (7)
N1—C7	1.337 (6)	C5—H5A	0.9500
N1—C8	1.455 (6)	C6—H6A	0.9500
N1—H1N	0.83 (5)	C8—C10	1.516 (7)
C1—C2	1.383 (6)	C8—C9	1.525 (8)
C1—C6	1.392 (6)	C8—H8A	1.0000
C1—C7	1.493 (6)	C9—H9A	0.9800

C2—C3	1.398 (8)	C9—H9B	0.9800
C2—H2A	0.9500	C9—H9C	0.9800
C3—C4	1.373 (7)	C10—H10A	0.9800
C3—H3A	0.9500	C10—H10B	0.9800
C4—C5	1.384 (7)	C10—H10C	0.9800
C4—H4A	0.9500		

C7—N1—C8	124.0 (4)	O1—C7—N1	122.2 (4)
C7—N1—H1N	118 (4)	O1—C7—C1	121.0 (4)
C8—N1—H1N	117 (4)	N1—C7—C1	116.7 (4)
C2—C1—C6	119.2 (4)	N1—C8—C10	109.9 (4)
C2—C1—C7	118.3 (4)	N1—C8—C9	110.4 (4)
C6—C1—C7	122.4 (4)	C10—C8—C9	111.5 (4)
C1—C2—C3	120.0 (5)	N1—C8—H8A	108.3
C1—C2—H2A	120.0	C10—C8—H8A	108.3
C3—C2—H2A	120.0	C9—C8—H8A	108.3
C4—C3—C2	120.3 (5)	C8—C9—H9A	109.5
C4—C3—H3A	119.9	C8—C9—H9B	109.5
C2—C3—H3A	119.9	H9A—C9—H9B	109.5
C3—C4—C5	119.8 (5)	C8—C9—H9C	109.5
C3—C4—H4A	120.1	H9A—C9—H9C	109.5
C5—C4—H4A	120.1	H9B—C9—H9C	109.5
C6—C5—C4	120.2 (5)	C8—C10—H10A	109.5
C6—C5—H5A	119.9	C8—C10—H10B	109.5
C4—C5—H5A	119.9	H10A—C10—H10B	109.5
C5—C6—C1	120.5 (4)	C8—C10—H10C	109.5
C5—C6—H6A	119.8	H10A—C10—H10C	109.5
C1—C6—H6A	119.8	H10B—C10—H10C	109.5

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*N*···O1^{i}	0.83 (5)	2.10 (5)	2.890 (5)	160 (5)

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