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N,*N*-Diisopropyl-1-naphthamide

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The crystal structure of *N*,*N*-diisopropyl-1-naphthamide, $C_{17}H_{21}NO$, has been determined at 180 K. The molecular conformation is such that the plane of the amide group is approximately perpendicular to the plane of the naphthalene unit. C—H···O interactions link the molecules into chains running along the *b* direction.

Key indicators

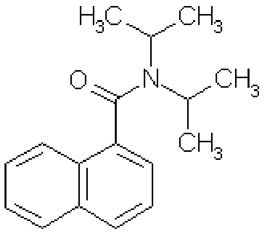
Single-crystal X-ray study
T = 180 K
Mean σ(C–C) = 0.004 Å
R factor = 0.065
wR factor = 0.148
Data-to-parameter ratio = 14.2

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Comment

The use of directed orthometallation involving amide-type functional groups in general, and tertiary amides in particular, has revolutionized the synthesis of complex benzenoid aromatic compounds (Snieckus, 1990).

It is generally assumed that O—Li coordination is maintained from the reactive complex through to the products, but in the case of the tertiary amides such coordination is problematic. A tertiary amide group, even in a simple aromatic amide, lies twisted out of the plane of the benzene ring for steric reasons (Bowles *et al.*, 1997), discouraging direct coordination of an oxygen lone pair to a 2-lithio substituent. It is clear that the twist angle affects the rate of lithiation (Beak *et al.*, 1993), but even amides which are nearly perpendicular, *e.g.* naphthamides, undergo efficient ortholithiation (Clayden *et al.*, 1999).



(I)

In light of the effect of amide twist angle on lithiation, we have investigated both aromatic tertiary amides and their lithiated derivatives. We report here the structure of *N*,*N*-diisopropyl-1-naphthamide, (I), determined at 180 K.

The molecular conformation in (I) is such that the plane of the amide group forms an angle of 83.4 (2)° with the least-squares plane through the naphthalene unit. C—H···O interactions exist between the H atom in the 5-position of the napthalene ring and the carboxyl group of a neighbouring molecule [H5···O1^{i} = 2.41 Å, C5—H5···O1^{i} = 161.7°; symmetry code: (i) $1 - x$, $-\frac{1}{2} + y$, $\frac{1}{2} - z$]. These interactions link the molecules into chains running along the *b* direction.

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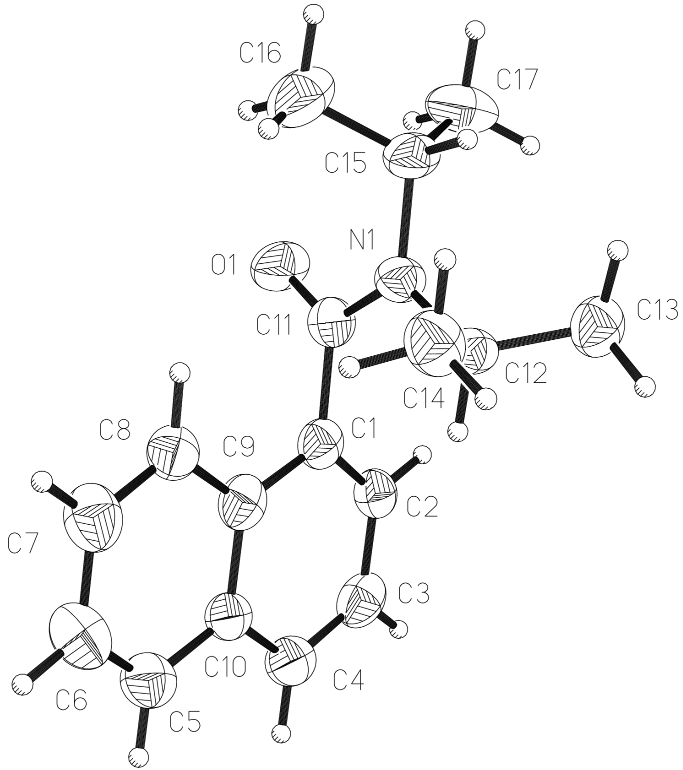

Figure 1
The molecular unit in (I) showing displacement ellipsoids at the 50% probability level (*XP*; Sheldrick, 1993).

Experimental

1-Naphthoyl chloride (5.4 ml, 35.8 mmol) was added dropwise to diisopropylamine (9.8 ml, 74.7 mmol) in Et_2O (50 ml) at 273 K and the mixture was stirred for 24 h at room temperature after which time H_2O (40 ml) was added. The aqueous phase was extracted with Et_2O (3 × 30 ml) and the organic extracts washed with H_2O, 1 *M* HCl and saturated $NaHCO_3$ (25 ml each) and dried ($MgSO_4$). Removal of the solvent afforded the title compound, which was recrystallized from tetrahydrofuran.

Crystal data

$C_{17}H_{21}NO$
$M_r = 255.35$
Monoclinic, $P2_1/c$
$a = 7.5403$ (10) Å
$b = 13.6498$ (19) Å
$c = 14.4042$ (11) Å
$\beta = 101.958$ (8)°
$V = 1450.4$ (3) Å^3
$Z = 4$

$D_x = 1.169$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 13372 reflections
$\theta = 1.0$–25.0°
$\mu = 0.07$ mm^{-1}
$T = 180$ (2) K
Plate, colourless
0.21 × 0.14 × 0.05 mm

Data collection

Nonius KappaCCD diffractometer
Thin-slice ω and φ scans
Absorption correction: multi-scan (*SORTAV*; Blessing, 1995)
$T_{\min} = 0.992$, $T_{\max} = 0.997$
8393 measured reflections
2494 independent reflections

1725 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.064$
$\theta_{\max} = 24.9°$
$h = -7 \rightarrow 8$
$k = -16 \rightarrow 16$
$l = -17 \rightarrow 16$

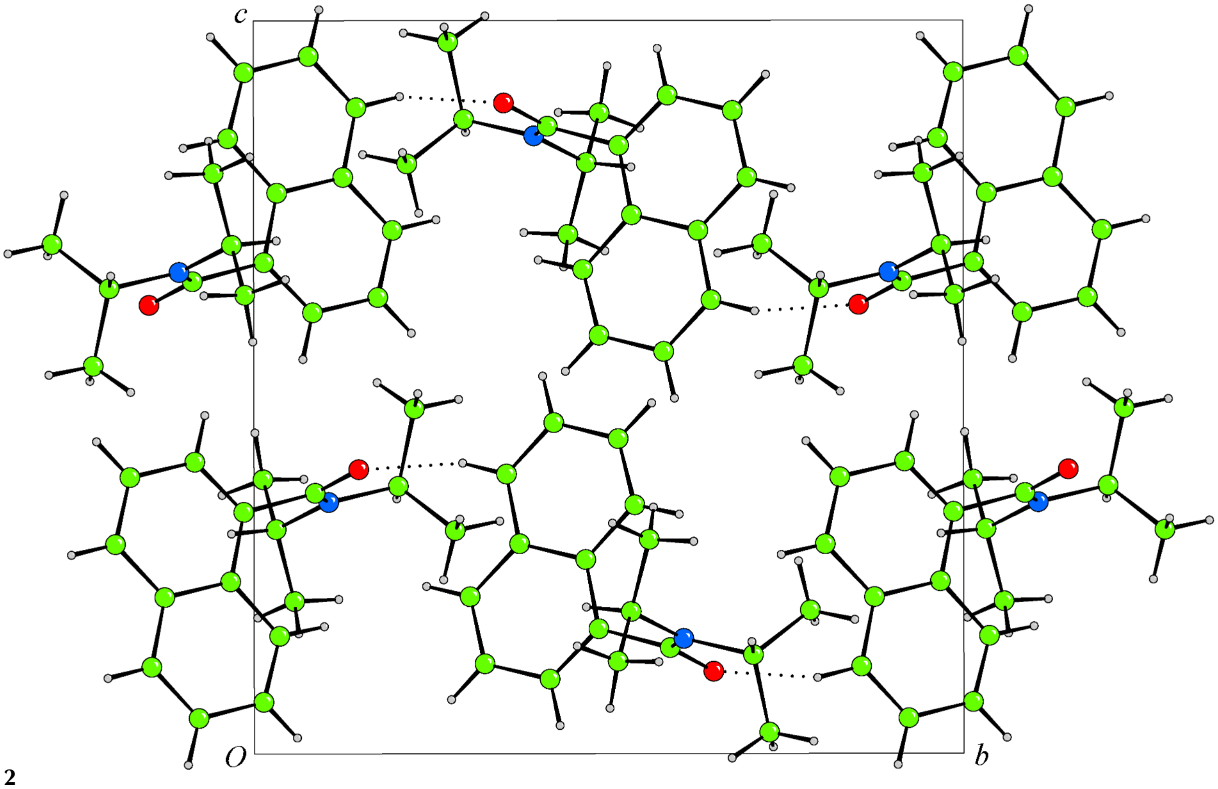

Figure 2
Projection onto (100) showing molecules of (I) linked by C—H···O interactions into chains running along the *b* direction. (*CAMERON*; Watkin *et al.*, 1996)

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.065$
$wR(F^2) = 0.148$
$S = 1.07$
2494 reflections
176 parameters
H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0467P)^2 + 0.8144P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.026$
$\Delta\rho_{\max} = 0.27$ e Å^{-3}
$\Delta\rho_{\min} = -0.21$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

O1—C11	1.236 (3)	C5—C6	1.360 (4)
N1—C11	1.351 (3)	C5—C10	1.409 (4)
N1—C12	1.481 (3)	C6—C7	1.394 (4)
N1—C15	1.485 (3)	C7—C8	1.358 (4)
C1—C2	1.364 (3)	C8—C9	1.422 (3)
C1—C9	1.412 (3)	C9—C10	1.432 (4)
C1—C11	1.519 (4)	C12—C13	1.514 (4)
C2—C3	1.411 (4)	C12—C14	1.518 (3)
C3—C4	1.376 (4)	C15—C16	1.521 (4)
C4—C10	1.405 (4)	C15—C17	1.521 (4)
C11—N1—C12	122.7 (2)	C1—C9—C10	118.3 (2)
C11—N1—C15	120.4 (2)	C8—C9—C10	118.4 (2)
C12—N1—C15	116.88 (19)	C4—C10—C5	122.0 (2)
C2—C1—C9	120.4 (2)	C4—C10—C9	120.0 (2)
C2—C1—C11	120.2 (2)	C5—C10—C9	117.9 (2)
C9—C1—C11	119.2 (2)	O1—C11—N1	122.7 (2)
C1—C2—C3	121.5 (2)	O1—C11—C1	118.4 (2)
C4—C3—C2	119.6 (2)	N1—C11—C1	118.9 (2)
C3—C4—C10	120.3 (2)	N1—C12—C13	112.2 (2)
C6—C5—C10	122.4 (3)	N1—C12—C14	112.2 (2)
C5—C6—C7	119.2 (3)	C13—C12—C14	110.9 (2)
C8—C7—C6	121.5 (3)	N1—C15—C16	112.5 (2)
C7—C8—C9	120.6 (3)	N1—C15—C17	112.1 (2)
C1—C9—C8	123.4 (2)	C16—C15—C17	112.9 (2)

Table 2
Hydrogen-bonding geometry (Å, °).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C5—H5···O1^{i}	0.95	2.41	3.327 (3)	162

Symmetry code: (i) $1 - x, y - \frac{1}{2}, \frac{1}{2} - z$.

All H atoms were placed geometrically and allowed to ride during subsequent refinement with an isotropic displacement parameter fixed at $1.2U_{\rm eq}$ for the carbon to which the hydrogen was attached. For the methyl groups, $U_{\rm iso}$ was fixed at $1.5U_{\rm eq}$ for the carbon.

Data collection: *COLLECT* (Nonius BV, 1998); cell refinement: *HKL SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *HKL DENZO* and *SCALEPACK* (Otwinowski & Minor, 1997); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); software used to prepare material for publication: *SHELXL*97.

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