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N-(1-Adamantyl)acetamide

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Abstract

N-(1-Adamantyl)acetamide, $C_{12}H_{19}NO$, crystallizes in space group *C*2/*c*. The acetamide groups related by a c-glide plane are linked by an N—H—O hydrogen bond. Conformation of the molecule is substantially the same as that found in its metanol-water solvte and that determined recently at 178 K.

Comment

N-(1-adamantyl)acetamide (1) forms a methanol-water solvate from an acetone-methanol solution (Kashino, Tateno, Hamada & Haisa, 1997). It has been found that (1) also forms unsolvated crystals from an acetone solution. The structure of (1) has also been determined very recently at 178 K by Pröhl, Blaschette & Jones (1997).

In the crystals, an N—H···O hydrogen bond is formed between the acetamide groups related by a c-glide plane (x, 1 − y, −1/2 + z)[N—H 0.87 (2), H···O 2.10 (2), N···O 2.962 (3) Å, N—H···O 169 (2) °]. The N···O distance is significantly larger than 2.928 (2) Å at 178 K (Pröhl, Blaschette & Jones, 1997). This glide type of hydrogen bond corresponds to one of the typical type of hydrogen bonds formed between the amide groups (Haisa *et al.*, 1980; Leiserowitz & Tuval, 1978). A twofold axis in the crystal is surrounded by hydrophobic adamantane moieties.

Conformations around the C1—N bonds are similar to those found in the methanol-water solvate; among three C—C1—N—C angles two correspond to *gauche* [−60.9 (2) and 61.0 (2) °], and one corresponds to *trans* [179.9 (2) °].

Intramolecular C—H···O interactions [C8···O 3.068 (3), H8A···O 2.45 (2) Å, C8—H8A···O 121 (1); C2···O 3.108 (3), H2B···O 2.53 (2) Å, C2—H2B···O 117 (1) °] are also found as in the solvate. These geometries are in good agreement with those at 178 K (Pröhl, Blaschette & Jones, 1997).

Experimental

Crystals were grown by slow evaporation from an acetone solution of *N*-(1-adamantyl)acetamide (Aldrich 13,710–3).

Refinement

Data collection and cell refinement were carried out with MSC/AFC Data Collection and Refinement Software (Rigaku Corporation, 1990). The structure was solved by direct methods using *MITHRIL* (Gilmore, 1984) and refined by full-matrix least squares using *TEXSAN* (Molecular Structure Corporation, 1985). H atoms were located from a difference Fourier map. and refined isotropically. The displacement ellipsoids were drawn with the aid of *ORTEP* II (Johnson, 1976). The calculations were performed on a VAX 3100 computer using *TEXSAN* at the X-ray Laboratory of Okayama University.

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Computing details

N-(1-Adamantyl)acetamide

Crystal data

$C_{12}H_{19}NO$	$V = 2231\ (2)\ Å^3$
$M_r = 193.29$	$Z = 8$
Monoclinic, $C2/c$	Mo $K\alpha$
$a = 24.778\ (9)$ Å	$\mu = 0.07\ mm^{-1}$
$b = 9.442\ (3)$ Å	$T = 296$ K
$c = 9.537\ (6)$ Å	0.40 × 0.40 × 0.13 mm
$\beta = 91.27\ (4)^o$	

Data collection

Rigaku AFC-5R diffractometer	$R_{int} = 0.023$
Absorption correction: none	3 standard reflections
2736 measured reflections	every 97 reflections
2574 independent reflections	intensity decay: 1.4%
1635 reflections with $I > 1.5\sigma(I)$	

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.057$	204 parameters
$wR(F^2) = 0.054$	All H-atom parameters refined
$S = 1.25$	$\Delta\rho_{max} = 0.21\ e\ Å^{-3}$
1635 reflections	$\Delta\rho_{min} = -0.16\ e\ Å^{-3}$

Table 1
Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N—H1N···O^{i}	0.87 (2)	2.10 (2)	2.962 (3)	169 (2)

Symmetry codes: (i) $x, -y+1, z-1/2$.

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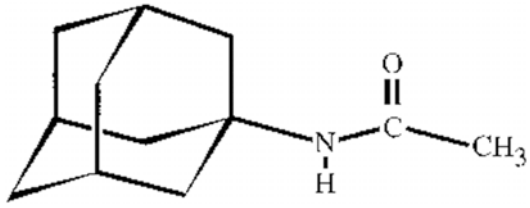
Scheme 1



supplementary materials

N-(1-Adamantyl)acetamide

Crystal data

$C_{12}H_{19}NO$	F_{000} = 848
M_r = 193.29	D_x = 1.151 Mg m^{-3} D_m = 1.14 Mg m^{-3} D_m measured by flotation in KI aqueous solution
Monoclinic, *C*2/*c*	Mo *K*α radiation λ = 0.71073 Å
a = 24.778 (9) Å	Cell parameters from 25 reflections
b = 9.442 (3) Å	θ = 9.5–11.0º
c = 9.537 (6) Å	μ = 0.07 mm^{-1}
β = 91.27 (4)º	*T* = 296 K
V = 2231 (2) Å^3	Plate, colorless
Z = 8	0.40 × 0.40 × 0.13 mm

Data collection

Rigaku AFC-5R diffractometer	θ_{max} = 27.5º
ω/2θ scans	*h* = −32→32
Absorption correction: none	*k* = 0→12
2736 measured reflections	*l* = 0→12
2574 independent reflections	3 standard reflections
1635 reflections with $I > 1.5\sigma(I)$	every 97 reflections
R_{int} = 0.023	intensity decay: 1.4%

Refinement

Refinement on *F*	Weighting scheme based on measured s.u.'s $w = 1/\sigma^2(F)$
$R[F^2 > 2\sigma(F^2)]$ = 0.057	$(\Delta/\sigma)_{max}$ = 0.06
$wR(F^2)$ = 0.054	$\Delta\rho_{max}$ = 0.21 e Å^{-3}
S = 1.25	$\Delta\rho_{min}$ = −0.16 e Å^{-3}
1635 reflections	Extinction correction: $I_{corr}=I_o(1+gI_c)$
204 parameters	Extinction coefficient: 0.266E-5
All H-atom parameters refined	

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

	x	*y*	*z*	U_{iso}*/U_{eq}
O	0.29287 (6)	0.5505 (2)	0.5695 (1)	0.0640 (9)
N	0.32020 (6)	0.4388 (2)	0.3732 (2)	0.0441 (9)
C1	0.36199 (7)	0.3467 (2)	0.4373 (2)	0.039 (1)
C2	0.33680 (8)	0.2437 (2)	0.5414 (2)	0.048 (1)
C3	0.3803 (1)	0.1464 (2)	0.6040 (2)	0.057 (1)

C4	0.4063 (1)	0.0621 (3)	0.4871 (3)	0.068 (1)
C5	0.4314 (1)	0.1634 (3)	0.3832 (2)	0.062 (1)
C6	0.4749 (1)	0.2520 (3)	0.4580 (3)	0.071 (1)
C7	0.4490 (1)	0.3355 (3)	0.5753 (2)	0.060 (1)
C8	0.40594 (9)	0.4345 (2)	0.5123 (2)	0.051 (1)
C9	0.4233 (1)	0.2355 (3)	0.6795 (2)	0.063 (1)
C10	0.3879 (1)	0.2613 (3)	0.3209 (2)	0.052 (1)
C11	0.28887 (8)	0.5289 (2)	0.4425 (2)	0.046 (1)
C12	0.2467 (1)	0.6055 (3)	0.3557 (3)	0.061 (1)
H1N	0.3150 (7)	0.432 (2)	0.283 (2)	0.046 (5)*
H2A	0.3088 (9)	0.189 (2)	0.492 (2)	0.065 (6)*
H2B	0.3188 (8)	0.296 (2)	0.617 (2)	0.056 (6)*
H3	0.3639 (8)	0.080 (2)	0.670 (2)	0.060 (6)*
H4A	0.434 (1)	0.001 (2)	0.529 (2)	0.078 (7)*
H4B	0.378 (1)	0.004 (2)	0.443 (2)	0.071 (7)*
H5	0.4472 (8)	0.108 (2)	0.307 (2)	0.058 (6)*
H6A	0.502 (1)	0.187 (3)	0.498 (2)	0.090 (8)*
H6B	0.492 (1)	0.319 (3)	0.395 (3)	0.10 (1)*
H7	0.4765 (9)	0.389 (2)	0.625 (2)	0.063 (6)*
H8A	0.3887 (8)	0.489 (2)	0.586 (2)	0.061 (6)*
H8B	0.4219 (8)	0.499 (2)	0.442 (2)	0.065 (6)*
H9A	0.451 (1)	0.171 (2)	0.720 (2)	0.075 (7)*
H9B	0.4067 (9)	0.290 (2)	0.757 (2)	0.068 (7)*
H10A	0.3611 (9)	0.210 (2)	0.265 (2)	0.065 (6)*
H10B	0.4030 (9)	0.323 (2)	0.252 (2)	0.062 (6)*
H12A	0.243 (1)	0.571 (3)	0.263 (3)	0.080 (7)*
H12B	0.256 (1)	0.701 (4)	0.354 (3)	0.13 (1)*
H12C	0.213 (1)	0.613 (3)	0.405 (3)	0.10 (1)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O	0.086 (1)	0.072 (1)	0.0345 (7)	0.0305 (9)	−0.0057 (7)	−0.0044 (7)
N	0.050 (1)	0.052 (1)	0.0296 (8)	0.0117 (8)	−0.0088 (7)	−0.0010 (8)
C1	0.041 (1)	0.041 (1)	0.0344 (9)	0.0056 (9)	−0.0080 (8)	−0.0017 (8)
C2	0.048 (1)	0.050 (1)	0.046 (1)	0.001 (1)	−0.005 (1)	0.003 (1)
C3	0.066 (1)	0.049 (1)	0.054 (1)	0.005 (1)	−0.009 (1)	0.014 (1)
C4	0.078 (2)	0.047 (1)	0.079 (2)	0.017 (1)	−0.017 (1)	−0.004 (1)
C5	0.067 (2)	0.063 (2)	0.057 (1)	0.024 (1)	0.000 (1)	−0.011 (1)
C6	0.050 (1)	0.080 (2)	0.085 (2)	0.018 (1)	−0.006 (1)	0.004 (2)
C7	0.050 (1)	0.062 (1)	0.067 (1)	0.001 (1)	−0.024 (1)	−0.006 (1)
C8	0.052 (1)	0.046 (1)	0.052 (1)	0.000 (1)	−0.012 (1)	−0.003 (1)
C9	0.069 (2)	0.070 (2)	0.049 (1)	0.019 (1)	−0.021 (1)	0.004 (1)
C10	0.057 (1)	0.057 (1)	0.041 (1)	0.009 (1)	−0.004 (1)	−0.004 (1)
C11	0.053 (1)	0.049 (1)	0.036 (1)	0.009 (1)	−0.0039 (8)	0.0035 (9)
C12	0.062 (2)	0.074 (2)	0.046 (1)	0.022 (1)	−0.006 (1)	0.006 (1)

Geometric parameters (Å, °)

O—C11	1.230 (2)	C2—H2A	0.98 (2)
N—C1	1.475 (2)	C2—H2B	0.99 (2)
N—C11	1.336 (3)	C3—H3	0.98 (2)
C1—C2	1.532 (3)	C4—H4A	0.97 (2)
C1—C8	1.533 (3)	C4—H4B	0.98 (2)
C1—C10	1.525 (3)	C5—H5	0.98 (2)
C2—C3	1.529 (3)	C6—H6A	0.97 (3)
C3—C4	1.524 (3)	C6—H6B	0.98 (3)
C3—C9	1.526 (3)	C7—H7	0.96 (2)
C4—C5	1.520 (3)	C9—H9A	0.99 (2)
C5—C6	1.530 (4)	C9—H9B	0.99 (2)
C5—C10	1.530 (3)	C10—H10A	0.97 (2)
C6—C7	1.521 (4)	C10—H10B	0.96 (2)
C7—C8	1.532 (3)	C12—H12A	0.95 (2)
C7—C9	1.521 (3)	C12—H12B	0.94 (3)
C11—C12	1.505 (3)	C12—H12C	0.96 (3)
C1—N—C11	125.5 (2)	C3—C4—H4B	108 (1)
N—C1—C2	110.5 (2)	C5—C4—H4A	111 (1)
N—C1—C8	111.1 (2)	C5—C4—H4B	112 (1)
N—C1—C10	108.2 (1)	H4A—C4—H4B	109 (2)
C2—C1—C8	109.7 (2)	C4—C5—H5	109 (1)
C2—C1—C10	108.6 (2)	C6—C5—H5	110 (1)
C8—C1—C10	108.7 (2)	C10—C5—H5	109 (1)
C1—C2—C3	109.9 (2)	C5—C6—H6A	108 (2)
C2—C3—C4	109.5 (2)	C5—C6—H6B	113 (2)
C2—C3—C9	109.4 (2)	C7—C6—H6A	110 (1)
C4—C3—C9	109.4 (2)	C7—C6—H6B	108 (2)
C3—C4—C5	109.5 (2)	H6A—C6—H6B	110 (2)
C4—C5—C6	109.5 (2)	C6—C7—H7	109 (1)
C4—C5—C10	109.7 (2)	C8—C7—H7	111 (1)
C6—C5—C10	109.5 (2)	C9—C7—H7	108 (1)
C5—C6—C7	108.8 (2)	C1—C8—H8A	108 (1)
C6—C7—C8	109.2 (2)	C1—C8—H8B	108 (1)
C6—C7—C9	110.4 (2)	C7—C8—H8A	110 (1)
C8—C7—C9	109.7 (2)	C7—C8—H8B	111 (1)
C1—C8—C7	109.5 (2)	H8A—C8—H8B	111 (2)
C3—C9—C7	109.4 (2)	C7—C9—H9A	110 (1)
C1—C10—C5	109.9 (2)	C7—C9—H9B	110 (1)
O—C11—N	123.9 (2)	C3—C9—H9A	108 (1)
O—C11—C12	120.1 (2)	C3—C9—H9B	110 (1)
N—C11—C12	116.0 (2)	H9A—C9—H9B	109 (2)
C1—N—H1N	117 (1)	C1—C10—H10A	112 (1)
C11—N—H1N	117 (1)	C1—C10—H10B	111 (1)
C1—C2—H2A	109 (1)	C5—C10—H10A	112 (1)
C1—C2—H2B	111 (1)	C5—C10—H10B	110 (1)
C3—C2—H2A	111 (1)	H10A—C10—H10B	102 (2)

C3—C2—H2B	110 (1)	C11—C12—H12A	113 (1)
H2A—C2—H2B	107 (2)	C11—C12—H12B	107 (2)
C2—C3—H3	110 (1)	C11—C12—H12C	111 (2)
C4—C3—H3	109 (1)	H12A—C12—H12B	109 (2)
C9—C3—H3	110 (1)	H12A—C12—H12C	116 (2)
C3—C4—H4A	108 (1)	H12B—C12—H12C	99 (3)
C1—N—C11—O	3.4 (3)	C8—C1—N—C11	−60.9 (2)
C2—C1—N—C11	61.0 (2)	C10—C1—N—C11	179.9 (2)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N—H1N···O^{i}	0.87 (2)	2.10 (2)	2.962 (3)	169 (2)

Symmetry codes: (i) *x*, −*y*+1, *z*−1/2.