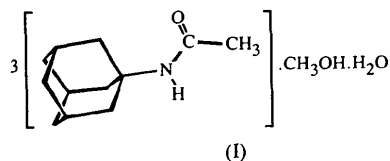


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gen-bonding ability on the symmetry of the crystals of adamantane derivatives. However, the crystal structures of such derivatives have been reported only for 1-adamantanol (Amoureux *et al.*, 1979) and 1-adamantanecarboxylic acid (Harvey *et al.*, 1986; Bélanger-Gariépy *et al.*, 1990). In the present work, we establish the structure of a new methanol–water solvate of *N*-(1-adamantyl)acetamide, (I).



Molecules in the asymmetric unit are shown in Fig. 1. The hydrogen-bonding geometry is summarized in Table 1. In addition to four hydrogen bonds in the

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Methanol–Water Solvate of *N*-(1-Adamantyl)acetamide

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Abstract

It has been found that *N*-(1-adamantyl)acetamide forms a methanol–water solvate, $3C_{12}H_{19}NO \cdot CH_4O \cdot H_2O$. The acetyl amino groups in the asymmetric unit are linked by hydrogen bonds *via* the methanol or water molecules. The asymmetric units are linked by hydrogen bonds between acetyl amino groups to form a ribbon. The ribbons are linked by hydrogen bonds between the water and methanol molecules. The conformations of the three acetyl amino groups with respect to the adamantane moieties are essentially the same.

Comment

The molecule of adamantane has high symmetry, *T_d*, and adamantane crystallizes in the highest space group, *Fm $\bar{3}$ m* (Nordman & Schmitkons, 1965; Amoureux *et al.*, 1980; Amoureux & Bee, 1980). In view of the development of crystal structure systems and the design of organic crystals, it is of interest to study the effects of some simple functional substituents having hydro-

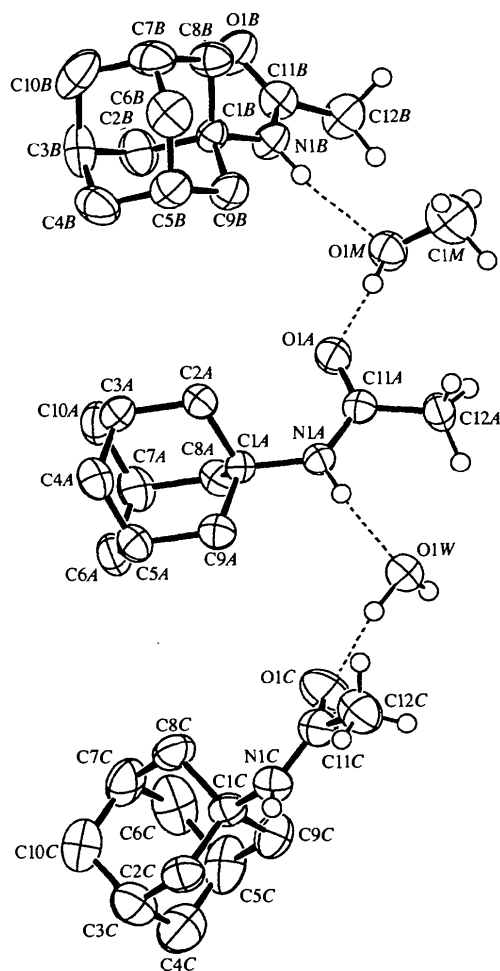


Fig. 1. The molecular structure of the asymmetric unit showing 50% probability displacement ellipsoids. H atoms attached to adamantyl C atoms have been omitted for clarity. Hydrogen bonds are shown by dashed lines.

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asymmetric unit, hydrogen bonds between acetyl amino groups link asymmetric units related by a **b** translation, resulting in the formation of a ribbon. Ribbons, related by a centre of symmetry, are linked by hydrogen bonds between the water and methanol molecules. Thus, one side of the ribbon forms a hydrophilic region and the other side forms a hydrophobic region.

In the three independent molecules (*A*, *B* and *C*) of *N*-(1-adamantyl)acetamide, the conformations around the C1—N1 bonds are similar; for each molecule, two of the three C—C—N—C angles correspond to *gauche* conformations, and the other corresponds to a *trans* conformation. Thus, the conformation around the C1—N1 bond is not significantly influenced by the crystallographic environment. It is of note that the O atoms of the amide groups participate in intramolecular C—H···O interactions [C···O 3.043 (6)–3.141 (6), H···O 2.44 (4)–2.56 Å and C—H···O 119 (3)–124 (3)°]. Such interactions have been noted for 1,3-(2-hydril-*F*-adamantyl) bis(trifluoroacetate), based on ¹H NMR and IR spectra (Adcock & Zhang, 1995).

Experimental

Crystals were grown by slow evaporation from a methanol-acetone solution (1:1 *v/v*) of *N*-(1-adamantyl)acetamide (Aldrich 13,710-3). A crystal was sealed in a glass capillary to avoid efflorescence during diffraction data measurements.

Crystal data

3C₁₂H₁₉NO·CH₄O·H₂O

M_r = 629.9

Triclinic

P $\bar{1}$

a = 14.622 (2) Å

b = 17.901 (3) Å

c = 6.908 (1) Å

α = 92.43 (1)°

β = 90.88 (1)°

γ = 92.35 (1)°

V = 1804.8 (5) Å³

Z = 2

D_x = 1.159 Mg m⁻³

D_m = 1.15 Mg m⁻³

D_m measured by flotation in aqueous KI solution

Data collection

Rigaku AFC-5R diffractometer

$\omega/2\theta$ scans

Absorption correction: none

6947 measured reflections

6370 independent reflections

3515 reflections with

I > 1.5 σ (*I*)

R_{int} = 0.030

Mo *K* α radiation

λ = 0.71073 Å

Cell parameters from 25 reflections

θ = 11.0–11.5°

μ = 0.076 mm⁻¹

T = 295 K

Plate developed in {100}

0.50 × 0.33 × 0.32 mm

Colourless

θ_{\max} = 26.0°

h = -18 → 18

k = -23 → 23

l = 0 → 9

3 standard reflections

every 97 reflections

intensity decay: 0.7%

Refinement

Refinement on *F*

R = 0.072

wR = 0.064

S = 1.16

3515 reflections

479 parameters

H atoms: see below

w = 1/ σ^2 (*F*)

(Δ/σ)_{max} = 0.004

$\Delta\rho_{\max}$ = 0.26 e Å⁻³

$\Delta\rho_{\min}$ = -0.25 e Å⁻³

Extinction correction:

*I*_{corr} = *I*₀(1 + *gI*_c)

Extinction coefficient:

g = 1.02 × 10⁻⁶

Scattering factors from *International Tables for X-ray Crystallography* (Vol. IV)

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

D—H···A	D—H	H···A	D···A	D—H···A
O1W—H2W···O1C	0.96 (5)	1.76 (5)	2.695 (4)	165 (5)
O1W—H1W···O1M ⁱ	0.94 (4)	1.90 (5)	2.820 (5)	169 (5)
O1M—H1OM···O1A	0.91 (4)	1.76 (4)	2.661 (4)	169 (4)
N1A—H1NA···O1W	0.89 (4)	2.03 (4)	2.906 (4)	170 (4)
N1B—H1NB···O1M	0.85 (4)	2.17 (4)	3.013 (5)	171 (4)
N1C—H1NC···O1B ⁱ	0.89 (4)	1.99 (4)	2.880 (4)	177 (4)

Symmetry codes: (i) 1 - *x*, 1 - *y*, 1 - *z*; (ii) *x*, *y* - 1, *z*.

Data collection and cell refinement were carried out with *MSCI/AF C 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. The H atoms involved in the hydrogen bonds and C—H···O interactions were refined isotropically. The coordinates of the other H atoms were fixed; their isotropic displacement parameters were assumed to be the same as the equivalent isotropic displacement parameters of the C atoms to which they are attached. The displacement ellipsoid plots were drawn with the aid of *ORTEPII* (Johnson, 1976). The calculations were performed on a VAX 3100 computer using *TEXSAN* at the X-ray Laboratory of Okayama University, Japan.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: FR1075). Services for accessing these data are described at the back of the journal.

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