O37—C31	1.392 (3)	C22—C23	1.392 (5)
O37—H37	0.91 (5)	C23—C24	1.374 (4)
C1—C2	1.386 (4)	C24—C25	1.390 (5)
C1—C6	1.383 (4)	C24—C29	1.506 (5)
C2—C3	1.394 (4)	C25—C26	1.387 (5)
C2—C40	1.512 (5)	C26—C30	1.520 (4)
C3—C4	1.386 (5)	C29—C80	1.511 (6)
C4—C5	1.388 (4)	C30—C32	1.517 (4)
C4—C9	1.501 (4)	C31—C32	1.389 (4)
C5—C6	1.395 (4)	C31—C36	1.397 (4)
C6—C10	1.511 (4)	C32—C33	1.404 (4)
C9—C60	1.483 (6)	C33—C34	1.386 (5)
C10—C12	1.521 (5)	C34—C35	1.382 (5)
C11—C12	1.387 (5)	C35—C36	1.381 (4)
C11—C16	1.399 (4)	C36—C40	1.518 (5)
C12—C13	1.389 (5)	C60—C70	1.277 (5)
C13—C14	1.378 (5)	C80—C90	1.249 (6)

O7—C1—C2	117.9 (3)	C21—C22—C23	117.5 (3)
C2—C1—C6	122.9 (3)	C22—C23—C24	122.7 (4)
O7—C1—C6	119.3 (2)	C23—C24—C29	120.8 (3)
C1—C2—C40	122.7 (3)	C23—C24—C25	117.9 (4)
C3—C2—C40	120.3 (2)	C25—C24—C29	121.2 (3)
C1—C2—C3	117.0 (3)	C24—C25—C26	122.4 (3)
C2—C3—C4	123.1 (3)	C25—C26—C30	121.4 (3)
C3—C4—C5	117.0 (3)	C21—C26—C30	120.9 (3)
C3—C4—C9	121.4 (3)	C21—C26—C25	117.8 (3)
C5—C4—C9	121.7 (3)	C24—C29—C80	115.8 (3)
C4—C5—C6	122.7 (3)	C26—C30—C32	113.0 (3)
C1—C6—C10	122.7 (3)	O37—C31—C32	117.7 (3)
C5—C6—C10	119.8 (3)	C32—C31—C36	122.7 (3)
C1—C6—C5	117.4 (2)	O37—C31—C36	119.7 (3)
C4—C9—C60	115.3 (3)	C30—C32—C31	123.3 (2)
C6—C10—C12	113.2 (2)	C30—C32—C33	119.5 (3)
O17—C11—C16	118.7 (3)	C31—C32—C33	117.3 (3)
C12—C11—C16	122.8 (3)	C32—C33—C34	121.0 (4)
O17—C11—C12	118.6 (3)	C33—C34—C35	119.6 (3)
C10—C12—C13	121.1 (3)	C34—C35—C36	121.6 (4)
C10—C12—C11	121.3 (3)	C31—C36—C40	122.0 (2)
C11—C12—C13	117.7 (3)	C31—C36—C35	117.9 (3)
C12—C13—C14	121.3 (3)	C35—C36—C40	120.2 (3)
C13—C14—C15	119.7 (4)	C2—C40—C36	112.3 (3)
C14—C15—C16	121.3 (4)	C9—C60—C70	129.6 (3)
C15—C16—C20	120.2 (3)	C29—C80—C90	129.0 (4)
C11—C16—C20	122.5 (3)	C1—O7—H7	118 (2)
C11—C16—C15	117.3 (3)	C11—O17—H17	106 (3)
C16—C20—C22	113.2 (2)	C21—O27—H27	115 (2)
O27—C21—C26	119.8 (3)	C31—O37—H37	112 (2)
O27—C21—C22	118.5 (3)	O7—H7···O17	166 (4)
C22—C21—C26	121.7 (3)	O17—H17···O27	165 (3)
C20—C22—C23	121.2 (3)	O27—H27···O37	168 (4)
C20—C22—C21	121.3 (3)	O7—H37···O37	164 (4)

Data were collected in the $\omega/2\theta$-scan mode [scan width (ω): 1.3 + 0.35tanθ], using graphite-monochromated Mo $K\alpha$ radiation. The intensity data were corrected for Lorentz and polarization effects and for long time-scale variation. No absorption correction was applied. The structure was solved with *MULTAN* (Germain *et al.*, 1971) and refined by full-matrix least squares. Refinements (on F^2) were made, using all reflections. Weights for each reflection in the refinement were $w = 1/[\sigma^2(F_o^2)]$, $\sigma(F_o^2) = \sigma^2(I) + (pF_o^2)^2$; the value of the instability factor p was determined as 0.04. All calculations were performed with *SDP* (B. A. Frenz & Associates Inc., 1983). All heavy atoms were refined with anisotropic displacement parameters. H atoms were placed at calculated positions (C—H 0.95 Å) and treated as riding atoms, except for the hydroxyl H atoms, which were found from a difference Fourier synthesis and refined with isotropic displacement parameters.

Data collection: *CAD-4 EXPRESS* (Enraf–Nonius, 1992). Cell refinement: *CAD-4 EXPRESS*. Data reduction: *CAD-4 EXPRESS*.

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3-Methyl-1,5-dinitro-3-azabicyclo[3.3.1]non-7-ene

O. V. SHISHKIN,[a] YU. M. ATROSCHENKO,[b] S. S. GITIS,[b] E. N. ALIFANOVA[b] AND I. V. SHAKHKELDYAN[b]

[a]*A. N. Nesmeyanov Institute of Organoelement Compounds of the Russian Academy of Sciences, 28 Vavilov Str., Moscow 117813, Russia, and* [b]*Tula State Pedagogical University, 125 Lenina Ave., Tula 300026, Russia. E-mail: oleg@xray.ineos.ac.ru*

Abstract

The 3-azabicyclo[3.3.1]non-7-ene skeleton of the title compound, $C_9H_{13}N_3O_4$, has a sofa–chair conformation. There are two molecules in the asymmetric unit with different orientations of their nitro groups.

$C_9H_{13}N_3O_4$

Comment

There are two molecules (A and B) in the asymmetric unit of the title compound, (I), with different orientations of their nitro groups. The nitro groups are orthogonal at C(1) and synperiplanar at C(5) with respect to the C(1)—C(8) and C(5)—C(6) bonds, respectively. The O(2)—N(1)—C(1)—C(8) torsion angles are $-83.5\,(4)$ and $94.9\,(4)°$ in A and B, respectively, while the corresponding O(4)—N(2)—C(5)—C(6) torsion angles are $-19.0\,(5)$ and $-21.7\,(5)°$. The cyclohexene ring has a sofa conformation: the displacement of C(9) from the plane defined by the other five ring atoms is $-0.760\,(4)$ Å in molecule A, while it is $0.768\,(4)$ Å in molecule B. The piperidine ring adopts a chair conformation with N(3) and C(9) displaced from the plane of the remaining ring atoms by $0.657\,(3)$ and $-0.778\,(4)$ Å, respectively, in A and by $-0.672\,(3)$ and $0.760\,(4)$ Å, respectively, in B.

(I)
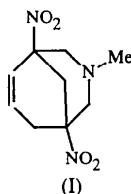
The conformation of the polycyclic skeleton of the molecule is very similar to that of the bicyclo[3.3.1]-non-7-ene derivative (Evans *et al.*, 1987) but unlike the chair–chair conformations of 3-azabicyclo[3.3.1]-nonan-7-one (Kaftory & Dunitz, 1976) and 3-oxabi-cyclo[3.3.1]nonan-7-one (Kaftory & Dunitz, 1975). The methyl group has an antiperiplanar orientation with respect to the C(4)—C(5) and C(1)—C(2) bonds. The C(5)—C(4)—N(3)—C(10) torsion angle is $-177.9\,(3)$ in molecule A and $175.9\,(3)°$ in molecule B.

A short O(1)$\cdots$H(8B)($\frac{1}{2}-x$, $-\frac{1}{2}+y$, z) intermolecular contact of 2.36 Å was observed; the sum of the corresponding van der Waals radii (Zefirov & Zorky, 1989) is 2.45 Å.
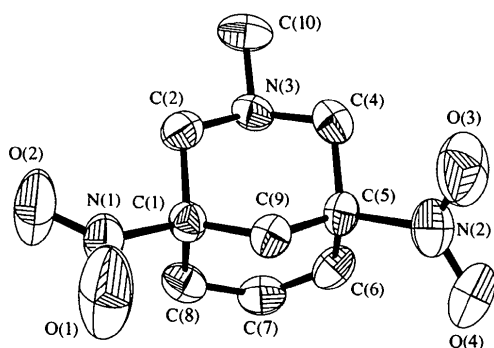

Fig. 1. A view of one of the independent molecules of (I) in the asymmetric unit, showing 50% probability displacement ellipsoids. H atoms have been omitted for clarity.

Experimental

The title compound was synthesized according to the method of Severin *et al.* (1963).

Crystal data

$C_9H_{13}N_3O_4$	Mo $K\alpha$ radiation
$M_r = 227.22$	$\lambda = 0.71073$ Å
Orthorhombic	Cell parameters from 24
$Pbca$	reflections
$a = 12.890\,(6)$ Å	$\theta = 10–12°$
$b = 11.187\,(4)$ Å	$\mu = 0.113$ mm^{-1}
$c = 29.565\,(13)$ Å	$T = 293\,(2)$ K
$V = 4263\,(3)$ Å^3	Block
$Z = 16$	$0.50 \times 0.20 \times 0.20$ mm
$D_x = 1.416$ Mg m^{-3}	Colourless
D_m not measured	

Data collection

Syntex $P2_1$ diffractometer	$\theta_{max} = 25.05°$
$2\theta/\omega$ scans	$h = 0 \rightarrow 15$
Absorption correction: none	$k = 0 \rightarrow 13$
3780 measured reflections	$l = 0 \rightarrow 35$
3780 independent reflections	2 standard reflections
1680 reflections with	every 98 reflections
$I > 2\sigma(I)$	intensity variation: $\pm 2.0\%$

Refinement

Refinement on F^2	$\Delta\rho_{max} = 0.22$ e Å^{-3}
$R(F) = 0.080$	$\Delta\rho_{min} = -0.20$ e Å^{-3}
$wR(F^2) = 0.146$	Extinction correction:
$S = 1.365$	*SHELXTL/PC* (Sheldrick,
3755 reflections	1993)
292 parameters	Extinction coefficient:
H atoms riding	0.0023 (6)
$w = 1/[\sigma^2(F_o^2) + (0.081P)^2$	Scattering factors from
$+ 0.692P]$	*International Tables for*
where $P = (F_o^2 + 2F_c^2)/3$	*Crystallography* (Vol. C)
$(\Delta/\sigma)_{max} = 0.001$	

The two molecules in the asymmetric unit are related by an approximate twofold screw axis (equation: $0.03x - 0.02y + z = 0$; translation: 3.57 Å). However, analysis of the measured data set and the small value of the shift along the y direction indicate that this axis is not crystallographic but is only local to pairs of molecules. The standard uncertainties on the C—C distances do not exceed 0.005 Å.

Data collection: *P3* (Siemens, 1989). Cell refinement: *P3*. Data reduction: *SHELXTL/PC* (Sheldrick, 1993). Program(s) used to solve structure: *SHELXTL/PC*. Program(s) used to refine structure: *SHELXTL/PC*. Molecular graphics: *SHELXTL/PC*. Software used to prepare material for publication: *SHELXTL/PC*.

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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).

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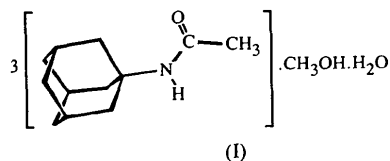
Molecules in the asymmetric unit are shown in Fig. 1. The hydrogen-bonding geometry is summarized in Table 1. In addition to four hydrogen bonds in the

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

SETSUO KASHINO, SHUICHI TATENO, NORITAKA HAMADA AND MASAO HAISA†

Department of Chemistry, Faculty of Science, Okayama University, Tsushima, Okayama 700, Japan. E-mail: kashinos@cc.okayama-u.ac.jp

Abstract

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

Comment

The molecule of adamantane has high symmetry, *Td*, 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-
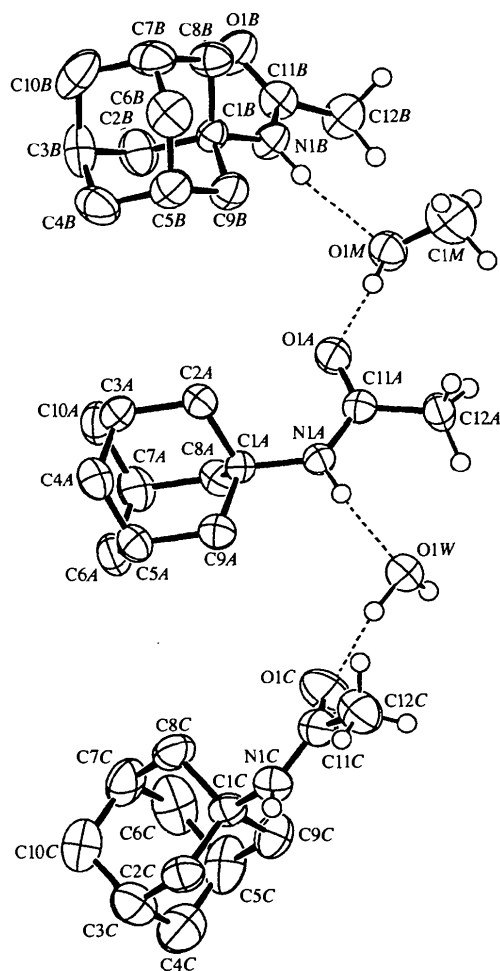
† Emeritus Professor of Okayama University.

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.