Table 1. *Selected geometric parameters (Å, °)*

I1—C10	2.116 (4)	N1′—C2′	1.351 (6)
I2—C5	2.097 (4)	N1′—C11′	1.457 (6)
I3—C5′	2.101 (5)	C2—C10	1.392 (6)
N1—C2	1.360 (6)	C2′—C10	1.398 (6)
N1—C11	1.452 (5)		
C2—N1—C11	126.6 (4)	C2—C10—C2′	124.5 (4)
C2′—N1′—C11′	126.3 (4)	C2—C10—I1	118.6 (3)
N1—C2—C10	121.9 (4)	C2′—C10—I1	116.8 (3)
N1′—C2′—C10	121.5 (4)		

Methyl groups were refined as rigid groups allowed to rotate but not tip from the starting position. Other H atoms were included with a riding model.

Data collection: *DIF*4 (Stoe & Cie, 1992*a*). Cell refinement: *DIF*4. Data reduction: *REDU*4 (Stoe & Cie, 1992*b*). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *XP* (Siemens, 1994*b*). Software used to prepare material for publication: *SHELXL*93.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: FG1330). Services for accessing these data are described at the back of the journal.

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1-Acetamidoadamantane†

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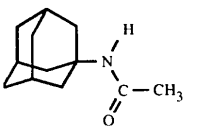
Abstract

In the title compound, $C_{12}H_{19}NO$, the bond lengths and angles are closely similar to those of two cage-substituted 1-acetamidoadamantanes. The molecules are linked into polymeric chains through a weak N—H···O hydrogen bond [H···O 2.04 (2), N···O 2.928 (2) Å and N—H···O 168 (1)°].

Comment

The title compound, (1), was originally synthesized *via* typical Ritter reactions from 1-hydroxy- or 1-bromoadamantane and acetonitrile in the presence of concentrated sulfuric acid (Stetter, Schwarz & Hirschhorn, 1959; Stetter, Mayer, Schwarz & Wulff, 1960). In a more direct approach, the 1-adamantyl carbocation was generated by oxidation of adamantane with nitric acid and then reacted with acetonitrile or acetamide (Bakke & Storm, 1989; Klimochkin, Bagrii, Dolgopolova & Moiseev, 1988). We have now obtained (1) by another method based on the well known process of silver(I)-promoted halide abstraction from 1-halogenoadamantanes (*e.g.* Kevill & Weitl, 1970) and determined its structure as part of the identification procedure.



(1)

The molecular structure of (1) is shown in Fig. 1. The bond lengths and angles are closely similar to the cor-

† Alternative name: 1-acetamidotricyclo[3.3.1.1^{3,7}]decane.

responding values in 1-acetamido-3-(4-tolyl)adamantane (Prozorovskii, Tafeenko, Rybakov, Shokova & Kovalev, 1987) and 1-acetamido-2,2,5,6,6-pentamethyladamantane (Amini, Bishop, Craig, Rae & Scudder, 1989). The amide group in (1) adopts an approximately *syn*-planar conformation [torsion angle O—C11—N—C1 2.8 (2)°], the carbonyl C11 atom lying 0.005 (2) Å out of the plane defined by N, O and C12. The conformational orientation of the acetamido substituent relative to the adamantyl cage is shown by the torsion angles C11—N—C1—C*n* (*n* = 2, 8, 10) in Table 1; the antiperiplanar conformation (for *n* = 10) confers approximate mirror symmetry on the molecule. Within the cage, the C—C bond lengths vary from 1.528 (2) to 1.537 (2) Å (mean 1.532 Å); the C—C—C angles at the methylene and methine C atoms lie in the ranges 109.18 (13)–109.95 (13) (mean 109.64) and 109.27 (13)–109.95 (14)° (mean 109.50°), respectively.

As shown in Fig. 2, a weak N—H···O hydrogen bond [*cf.* Table 2; H···O—C11 167.6 (4)°] joins glide-plane-related molecules into polymeric chains parallel to the *z* axis. The structure also displays one inter- and two intramolecular C—H···O sequences (Table 2) that can be viewed as hydrogen bonds (Desiraju, 1996).

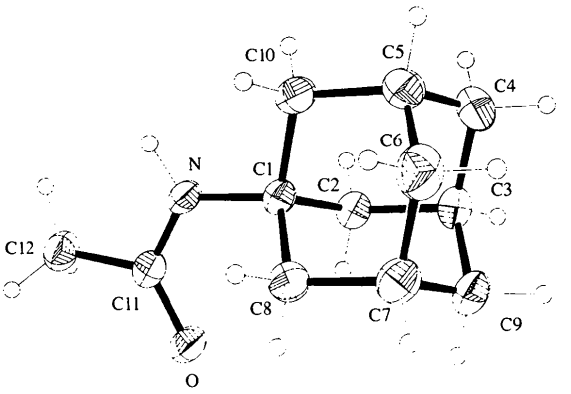


Fig. 1. Structure of the title compound in the crystal. Ellipsoids represent 50% probability levels. H-atom radii are arbitrary.

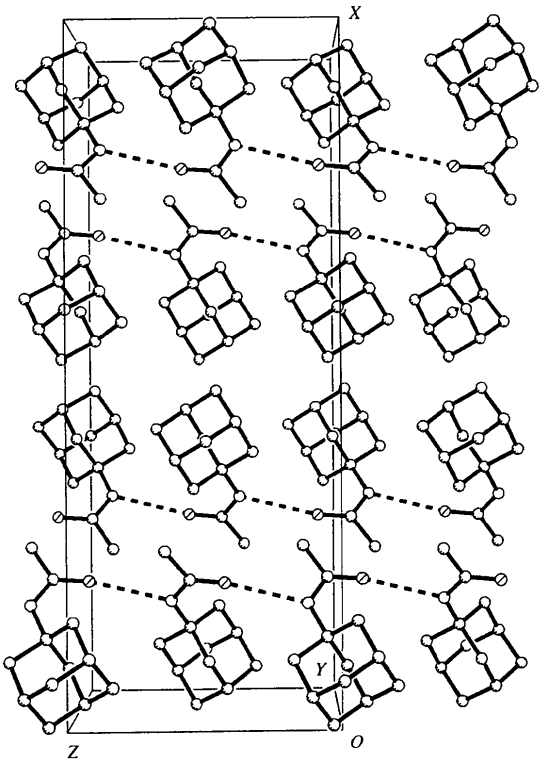


Fig. 2. Packing diagram viewed along the *y* axis. H atoms are omitted. Hydrogen bonds are indicated by broken lines.

Experimental

Anhydrous $Ag[N(SO_2Me)_2]$ was prepared as described elsewhere (Cammenga, Epple, Blaschette & Näveke, 1989). At ambient temperature, a solution of 4.3 g (20 mmol) 1-bromoadamantane in MeCN (30 ml) was added dropwise to a stirred solution of 5.6 g (20 mmol) of the Ag salt in the same solvent (100 ml). After refluxing for 3 h, the stoichiometric amount of AgBr was removed by filtration and the red–brown solution was poured into cold water (200 ml); the hydrolysis caused (1) to separate as a colourless solid, which was recrystallized from diethyl ether/petroleum ether; yield 3.2 g (83%), m.p. 422 K.

Crystal data

$C_{12}H_{19}NO$
M_r = 193.28
Monoclinic
$C2/c$
a = 24.312 (5) Å
b = 9.464 (2) Å
c = 9.454 (3) Å
β = 90.06 (2)°
V = 2175.5 (9) Å^3
Z = 8
D_x = 1.180 Mg m^{-3}
D_m not measured
Mo $K\alpha$ radiation
λ = 0.71073 Å
Cell parameters from 40 reflections
θ = 10.0–11.5°
μ = 0.074 mm^{-1}
T = 178 (2) K
Prism
0.50 × 0.45 × 0.30 mm
Colourless

Data collection

Siemens *R*3 diffractometer
ω scans
Absorption correction: none
2852 measured reflections
1915 independent reflections
1355 reflections with $I > 2\sigma(I)$
R_{int} = 0.022
θ_{max} = 25.0°
$h = -28 \rightarrow 28$
$k = -11 \rightarrow 0$
$l = -11 \rightarrow 4$
3 standard reflections every 147 reflections
intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.039
$wR(F^2)$ = 0.115
S = 1.029
1914 reflections
132 parameters
H atoms: see below
$\Delta\rho_{max}$ = 0.150 e Å^{-3}
$\Delta\rho_{min}$ = −0.166 e Å^{-3}
Extinction correction: *SHELXL*93 (Sheldrick, 1993)
Extinction coefficient: 0.0055 (10)

$w = 1/[\sigma^2(F_o^2) + (0.059P)^2 + 0.5656P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$

Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 1. *Selected geometric parameters (Å, °)*

N—C11	1.345 (2)	O—C11	1.235 (2)
N—C1	1.474 (2)	C11—C12	1.501 (2)
C11—N—C1	125.13 (12)	C2—C1—C10	108.61 (12)
N—C1—C2	110.62 (11)	C8—C1—C10	108.44 (12)
N—C1—C8	111.12 (12)	O—C11—N	123.53 (14)
C2—C1—C8	110.07 (12)	O—C11—C12	120.02 (14)
N—C1—C10	107.91 (11)	N—C11—C12	116.44 (13)
C11—N—C1—C2	61.6 (2)	C1—N—C11—O	2.8 (2)
C11—N—C1—C8	−60.9 (2)	C1—N—C11—C12	−176.49 (13)
C11—N—C1—C10	−179.69 (13)		

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N—H0···O^{i}	0.91 (2)	2.04 (2)	2.928 (2)	168 (1)
C12—H12*A*···O^{i}	0.98	2.42	3.287 (2)	148
C2—H2*B*···O	0.99	2.51	3.107 (2)	118
C8—H8*A*···O	0.99	2.46	3.064 (2)	119

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

H-atom treatment: NH coordinates free, *U* fixed; rigid methyl group (allowed to rotate but not tip); others riding. Despite the β angle being close to 90°, no higher Laue symmetry was appropriate.

Data collection: *P*3 (Nicolet XRD Corporation, 1987). Cell refinement: *P*3. Data reduction: *XDISK* (Nicolet XRD Corporation, 1987). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *XP* (Siemens, 1994). Software used to prepare material for publication: *SHELXL*93.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: FG1344). Services for accessing these data are described at the back of the journal.

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3-Cyano-6-(4-methoxyphenyl)-5-methyl-4-methylthio-2*H*-pyran-2-one

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Abstract

In the title compound, $C_{15}H_{13}NO_3S$, the two six-membered rings are inclined at an angle of 38.9 (1)°. The conformations of the ring substituents are discussed and compared with the situation when the 5-methyl substituent is replaced with an H atom.

Comment

Various 4-hydroxy-2*H*-pyran-2-ones and their ether derivatives have been found to be effective antibacterial and antifungal agents (Israili & Smissman, 1976; Kretzschmar, Meyer, Teschendorf & Zoellner, 1969). The addition of enolate anions derived from aryl–alkyl ketones to doubly active ketene dithioacetals has been shown to give 4-methylthio-2-pyrones (δ-lactones) in low to moderate yields (Hatada *et al.*, 1975). These lactones have been found to be vulnerable to nucleophilic attack because of the presence of several electrophilic centres, and provide a single-step synthesis for a variety of heterocycles such as pyrazoles, isoxazoles, 2-aminopyridines and 2-iminopyridines; these reactions illustrate the tremendous synthetic potential of such compounds (Ram, Verma, Hussaini & Shoeb, 1991; Ram, Hussaini, Singh & Shoeb, 1993; Hussaini *et al.*, 1994; Ram *et al.*, 1994). Lactones with a methyl group in the C-5 position have not been synthesized previously and so we have synthesized several new 3-cyano-5-methyl-4-methylthio-6-aryl-2*H*-pyran-2-ones with a view to screening them for antibacterial and antifungal activity.