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4,4a,4b,5,6,7,9,9a-Octahydro-1,4-methano-7-methyl-5-nitro-1*H*-fluoren-9-one

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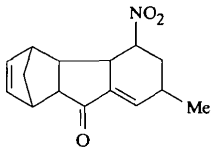
Abstract

In the title compound, $C_{15}H_{17}NO_3$, the six-membered ring adopts an envelope conformation and the nitro and methyl groups are attached in equatorial and bi-axial positions, respectively. In the norbornene ring system, the six-membered ring adopts a boat conformation and the two five-membered rings formed by the bridging C atom adopt envelope conformations. The fused five-membered ring is in a conformation intermediate between a flattened envelope and a half-chair. In the solid state, the C=O group is involved in weak C—H···O hydrogen bonds.

Comment

Hydrindane skeletons are the ubiquitous structural subunits of numerous naturally occurring organic compounds (Ho, 1988). Consequently, approaches to the synthesis of this framework remain relevant objectives in chemical synthesis, particularly stereoselective synthesis. In connection with our programme on the synthesis of kinamycin antibiotics which bear hydrindane subunits (Mal & Hazra, 1996), the title compound, (I), was prepared as a precursor of a hydrindane. In order to establish the stereochemistry of the substituents, the X-ray structure determination of (I) was carried out.



(I)

The bond lengths and angles observed in this structure are comparable with those observed in related structures (Chinnakali *et al.*, 1999; Fun *et al.*, 1998); C4—C5 and C9—C10 show double-bond character. The six-membered ring *A* adopts an envelope conformation, with C1 deviating from the mean plane by −0.378 (1) Å; the asymmetry parameter ΔC_s(C1) = 6.6 (1)° (Duax *et al.*, 1976). The nitro group is attached equatorially to ring *A*, whereas the methyl group is attached in the bi-axial position. The fused five-membered ring *B* is in a conformation which is intermediate between a flattened envelope and a half-chair [ΔC_s(C5) = 1.5 (2)° and ΔC_2(C7) = 5.9 (2)°], with C5 and C13 deviating from the mean plane by −0.095 (1) and 0.080 (1) Å, respectively. The six-membered ring *C* of the norbornene ring system adopts a boat conformation [ΔC_s(C8) = 3.1 (2)° and ΔC_s(C9—C10) = 0.9 (2)°]. The two five-membered rings in the norbornene moiety, formed by the bridging atom C14, adopt an envelope conformation. The dihedral angles formed by the mean plane through ring *B* with those through rings *A* and *C* are 19.74 (8) and 84.83 (10)°, respectively. The C1/N1/O1/O2 plane through the nitro group forms a dihedral angle of 52.4 (1)° with ring *A*. In the crystal, the screw-related molecules are linked by weak C12—H12···O3$(1-x, 2-y, z-\frac{1}{2})$ hydrogen bonds to form an infinite chain along the *c* direction (Table 2).

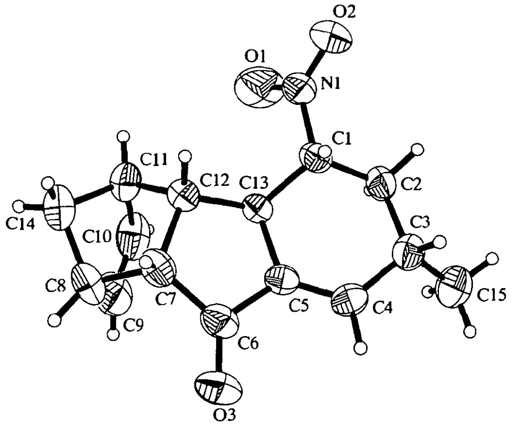


Fig. 1. The structure of (I) showing 50% probability displacement ellipsoids and the atom-numbering scheme. H atoms are shown as spheres of arbitrary radii.

Experimental

The preparation of the title compound, (I), was achieved in one step by the tributylphosphine-catalyzed condensation of methacrolein with 2,3,3a,4,7,7a-hexahydro-4,7-methano-3-nitromethyl-1*H*-indene-1-one. Single crystals of (I) were obtained by recrystallization from a dilute solution of the compound in ethanol (m.p. 433–435 K).

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Crystal data

$C_{15}H_{17}NO_3$
$M_r = 259.30$
Orthorhombic
$Pca2_1$
$a = 18.0675\,(9)$ Å
$b = 7.7986\,(4)$ Å
$c = 9.2847\,(4)$ Å
$V = 1308.2\,(1)$ Å^3
$Z = 4$
$D_x = 1.317$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 6180 reflections
$\theta = 2.25–28.31°$
$\mu = 0.092$ mm^{-1}
$T = 293\,(2)$ K
Block
0.48 × 0.40 × 0.40 mm
Colourless

Data collection

Siemens SMART CCD area-detector diffractometer
ω scans
Absorption correction: none
8746 measured reflections
3233 independent reflections
2823 reflections with $I > 2\sigma(I)$

$R_{\rm int} = 0.028$
$\theta_{\rm max} = 28.31°$
$h = 0 \rightarrow 23$
$k = 0 \rightarrow 10$
$l = -12 \rightarrow 12$
Intensity decay: negligible

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.037$
$wR(F^2) = 0.098$
$S = 1.05$
3233 reflections
173 parameters
H atoms riding
$w = 1/[\sigma^2(F_o^2) + (0.0513P)^2 + 0.0867P]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\rm max} < 0.001$
$\Delta\rho_{\rm max} = 0.15$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.11$ e Å^{-3}
Extinction correction: none
Scattering factors from *International Tables for Crystallography* (Vol. C)

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

O1—N1	1.202 (2)	N1—C1	1.516 (2)
O2—N1	1.217 (2)	C4—C5	1.327 (2)
O3—C6	1.214 (2)	C9—C10	1.321 (3)
C8—C14—C11	93.8 (1)		
O1—N1—C1—C13	−22.5 (2)	O2—N1—C1—C2	−81.2 (2)

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C12—H12···O3$^{\rm i}$	0.98	2.49	3.337 (2)	144

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

After checking their presence in the difference map, the H atoms were fixed geometrically and allowed to ride on the atoms to which they were attached; rotating-group refinement was used for the methyl groups.

Data collection: *SMART* (Siemens, 1996). Cell refinement: *SAINT* (Siemens, 1996). Data reduction: *SAINT*. Program(s) used to solve structure: *SHELXTL* (Sheldrick, 1997). Program(s) used to refine structure: *SHELXTL*. Molecular graphics: *SHELXTL*. Software used to prepare material for publication: *SHELXTL* and *PLATON* (Spek, 1990).

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: SK1281). Services for accessing these data are described at the back of the journal.

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(2*RS*,5*RS*)-2,5-Bis[3-(4-methylphenyl)-3-oxopropyl]cyclopentan-1-one

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Abstract

In the title compound, $C_{25}H_{28}O_3$, the cyclopentane ring adopts a half-chair conformation, with one of the side chains attached in the equatorial position and the other in a bi-axial orientation. The mean plane through the cyclopentane ring forms dihedral angles of 30.9 (2) and 24.3 (2)° with the two phenyl rings. The crystal structure is stabilized by C—H···O hydrogen bonds and C—H···π interactions.

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