

Orthorhombic
Pbca
 $a = 9.4810(9) \text{ \AA}$
 $b = 19.8360(19) \text{ \AA}$
 $c = 13.1780(14) \text{ \AA}$
 $V = 2478.3(4) \text{ \AA}^3$
 $Z = 8$
 $D_x = 1.288 \text{ Mg m}^{-3}$
 D_m not measured

Data collection

Enraf–Nonius CAD-4
 diffractometer
 ω - 2θ scans
 Absorption correction: none
 2306 measured reflections
 2174 independent reflections
 1528 reflections with
 $I > 2\sigma(I)$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.036$
 $wR(F^2) = 0.135$
 $S = 0.951$
 2174 reflections
 176 parameters
 H atoms treated by a
 mixture of independent
 and constrained refinement
 $w = 1/[\sigma^2(F_o^2) + (0.1P)^2]$
 where $P = (F_o^2 + 2F_c^2)/3$

Cell parameters from 25
 reflections
 $\theta = 13\text{--}15^\circ$
 $\mu = 0.085 \text{ mm}^{-1}$
 $T = 296 \text{ K}$
 Prism
 $0.50 \times 0.25 \times 0.22 \text{ mm}$
 Yellow

$R_{\text{int}} = 0.012$
 $\theta_{\text{max}} = 24.97^\circ$
 $h = 0 \rightarrow 11$
 $k = -23 \rightarrow 1$
 $l = 0 \rightarrow 15$
 3 standard reflections
 frequency: 60 min
 intensity decay: 0.5%

$(\Delta/\sigma)_{\text{max}} = 0.011$
 $\Delta\rho_{\text{max}} = 0.139 \text{ e \AA}^{-3}$
 $\Delta\rho_{\text{min}} = -0.166 \text{ e \AA}^{-3}$
 Extinction correction:
SHELXL97
 Extinction coefficient:
 0.0073 (16)
 Scattering factors from
International Tables for
Crystallography (Vol. C)

Supplementary data for this paper are available from the IUCr electronic archives (Reference: FG1463). Services for accessing these data are described at the back of the journal.

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Table 1. Selected geometric parameters (\AA , $^\circ$)

N1—N11	1.4045 (18)	N12—C13	1.345 (2)
N1—C3	1.2956 (19)	C13—C14	1.486 (2)
N2—C3	1.343 (2)	C14—C19	1.383 (2)
N11—C13	1.2982 (19)		
N11—N1—C3	111.55 (12)	N1—C3—N2	125.94 (15)
N1—N11—C13	112.02 (12)	N11—C13—N12	124.92 (15)
C3—N1—N11—C13	173.53 (13)	N12—C13—C14—N15	16.6 (2)
N2—C3—C4—N5	2.9 (2)		

Table 2. Hydrogen-bonding geometry (\AA , $^\circ$)

D—H...A	D—H	H...A	D...A	D—H...A
N2—H2B...N15 ⁱ	0.89 (2)	2.24 (2)	3.073 (2)	154.9 (17)
N12—H12B...N11 ⁱⁱ	0.89 (2)	2.262 (19)	3.0918 (19)	154.8 (17)

Symmetry codes: (i) $x - \frac{1}{2}, y, \frac{3}{2} - z$; (ii) $\frac{1}{2} + x, y, \frac{3}{2} - z$.

Data collection: *CAD-4/PC* (Enraf–Nonius, 1993). Cell refinement: *CAD-4/PC*. Data reduction: *XCAD4* (Harms & Wocadlo, 1996). Program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997b). Program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997a). Molecular graphics: *PLUTON92* and *PLATON92* (Spek, 1992). Software used to prepare material for publication: *SHELXL97*.

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1,9a-Epoxy-1,2,3,4,4a,4b,5,8,8a,9a-decahydro-2-methyl-4-nitro-5,8-methano-9H-fluoren-9-one

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Abstract

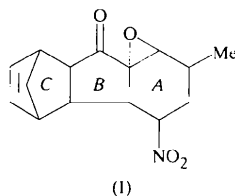
The six-membered ring of the title compound, $\text{C}_{15}\text{H}_{17}\text{NO}_4$, adopts a half-chair conformation, and the

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methyl and nitro groups are attached equatorially to it. The five-membered ring fused to it is in a flattened envelope conformation. In the norbornene ring system, the six-membered ring adopts a fairly symmetrical boat conformation and the two five-membered rings formed by the bridging C atom adopt envelope conformations. The mean plane through the fused five-membered ring forms dihedral angles of 31.14 (5) and 84.28 (7)° with the mean planes of the adjacent six-membered rings.

Comment

The *cis*-hydrindane carbon skeleton is found to occur in many bioactive sesquiterpenoids and steroids (Corey & Cheng, 1989). It is also used as a synthon for the synthesis of a wide range of natural products (Ho, 1988). Though a number of methodologies for the synthesis of *cis*-hydrindane exist in the literature, the preparation of stereo-defined substituted hydrindanes poses a considerable challenge to the synthetic chemists. We have recently developed a methodology for the stereo-controlled fabrication of *cis*-hydrindanes (Mal & Hazra, 1998). This route has yielded the precursor fluorenone, in which a *cis*-hydrindane structure is embedded. Although NMR study of the title compound, (I), revealed the relative stereochemistry of the substituents of ring A, their orientation with respect to ring C could not be ascertained. As a result, the X-ray structure analysis was undertaken to deduce the full stereostructure of the compound.



The bond lengths observed in this structure show normal values (Allen *et al.*, 1987); C9—C10 shows double-bond character. The six-membered ring A adopts a half-chair conformation, with asymmetry parameter $\Delta C_2(C2-C1) = 0.0058(8)$; the methyl and nitro groups are attached equatorially to it. The fused five-membered ring (B) adopts a flattened envelope conformation [$\Delta C_5(C5) = 0.0019(8)$], with C5 deviating from the C6—C7—C12—C13 plane by 0.141 (2) Å. The six-membered ring (C) of the norbornene ring system is a fairly symmetrical boat. The mean plane through the fused five-membered ring forms dihedral angles of 31.14 (5) and 84.28 (7)° with the mean planes of the adjacent six-membered rings. The epoxy plane is inclined at an angle of 73.10 (7)° with respect to the attached A ring and the nitro group is twisted by 47.6 (1)°. The mean planes through rings A and C form a dihedral angle of 59.08 (7)°. The geometry of the norbornene ring system is the same, within experimental

error, as other norbornene systems (Beddoes *et al.*, 1993; Dvorkin *et al.*, 1987). The crystal structure is stabilized by van der Waals interactions.

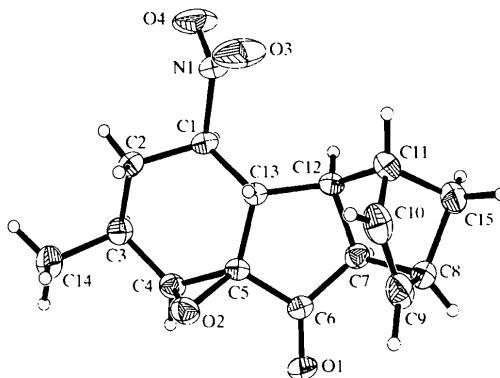


Fig. 1. The structure of the title compound showing 50% probability displacement ellipsoids and the atom-numbering scheme.

Experimental

Tricyclo[5.2.1.0^{2,6}]deca-3,8-dien-5-one was treated with nitromethane in the presence of 1,8-diazabicyclo[5.4.0]undecene (DBU) to give an oil, which was subsequently treated with methacrolein, again in the presence of DBU, to give 2,3,4,4a,4b,5,8,8a-octahydro-2-methyl-4-nitro-5,8-methano-9H-fluoren-9-one. This was then epoxidized to give the title compound, which was crystallized from ethanol by cooling to 258 K.

Crystal data

C₁₅H₁₇NO₄
M_r = 275.30
 Monoclinic
*P*2₁/*c*
a = 11.5320 (2) Å
b = 11.2967 (2) Å
c = 10.3406 (2) Å
 β = 96.312 (1)°
V = 1338.94 (4) Å³
Z = 4
D_x = 1.366 Mg m⁻³
D_m not measured

Mo *K*α radiation
 λ = 0.71073 Å
 Cell parameters from 4864 reflections
 θ = 1.5–33.0°
 μ = 0.099 mm⁻¹
T = 293 (2) K
 Block
 0.48 × 0.40 × 0.28 mm
 Colourless

Data collection

Siemens SMART CCD area-detector diffractometer
 ω scans
 Absorption correction: none
 8611 measured reflections
 3057 independent reflections

2334 reflections with $I > 2\sigma(I)$
 $R_{int} = 0.024$
 $\theta_{max} = 27.5^\circ$
 $h = -14 \rightarrow 14$
 $k = 0 \rightarrow 14$
 $l = 0 \rightarrow 13$

Refinement

Refinement on *F*²
 $R[F^2 > 2\sigma(F^2)] = 0.048$
 $wR(F^2) = 0.132$

$(\Delta/\sigma)_{max} < 0.001$
 $\Delta\rho_{max} = 0.249 \text{ e \AA}^{-3}$
 $\Delta\rho_{min} = -0.190 \text{ e \AA}^{-3}$

$S = 1.054$
 3056 reflections
 249 parameters
 All H atoms refined
 $w = 1/[\sigma^2(F_o^2) + (0.0610P)^2 + 0.3308P]$
 where $P = (F_o^2 + 2F_c^2)/3$

Extinction correction: none
 Scattering factors from
*International Tables for
 Crystallography* (Vol. C)

Siemens (1996*b*). *SHELXTL Reference Manual*. Version 5. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
 Siemens (1996*c*). *SMART Software Reference Manual*. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.

Table 1. Selected bond lengths (Å)

O1—C6	1.215 (2)	O4—N1	1.205 (2)
O2—C5	1.450 (2)	N1—C1	1.519 (2)
O2—C4	1.452 (2)	C4—C5	1.470 (2)
O3—N1	1.202 (2)	C9—C10	1.321 (3)

The data collection covered a hemisphere of reciprocal space by a combination of three sets of exposures; each set had a different φ angle (0, 88 and 180°) for the crystal and each exposure of 30 s covered 0.3° in ω . The crystal-to-detector distance was 4 cm and the detector swing angle was -35° . Coverage of the unique set is over 99% complete. Crystal decay was monitored by repeating 30 initial frames at the end of data collection and analysing the duplicate reflections; it was found to be negligible. The structure was solved by direct methods and refined by full-matrix least-squares techniques. All H atoms were located from a difference Fourier map and refined isotropically.

Data collection: *SMART* (Siemens, 1996*c*). Cell refinement: *SAINT* (Siemens, 1996*a*). Data reduction: *SAINT*. Program(s) used to solve structure: *SHELXTL* (Siemens, 1996*b*). Program(s) used to refine structure: *SHELXTL*. Molecular graphics: *SHELXTL*. Software used to prepare material for publication: *SHELXTL* and *PARST* (Nardelli, 1995).

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: HA1220). Services for accessing these data are described at the back of the journal.

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2-(2-Hydroxybenzylidene)-1-(2-picoloyl)-hydrazine Hemihydrate

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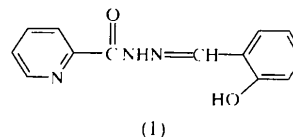
Abstract

In the title compound [2-hydroxybenzaldehyde 2-pyridylcarbonylhydrazone hemihydrate, $C_{13}H_{11}N_3O_2 \cdot 0.5H_2O$ or (1)·0.5H₂O], molecules of (1) are linked pairwise through intermolecular N—H···N hydrogen bonds. These pairs alternate with hydrogen-bonded water molecules to form one-dimensional chains.

Comment

Compared to simple hydrazone Schiff bases, acyl, aroyl and heteroaryl Schiff bases have an additional O-donor atom from the carbonyl group, whose presence introduces a wider range of properties (Dutta & Hossain, 1985). These compounds may therefore function as polydentate ligands and some reports of their complexes have appeared (Dutta & Sarkar, 1981; Aggarwal *et al.*, 1981; Dutta & Das, 1985).

Molecules of (1) are essentially planar [r.m.s. deviation from the least-squares mean plane is only 0.051 (7) Å]. The O1···N1 separation of 2.669 (8) Å, which is less than the sum of the corresponding van der Waals radius sum (2.85 Å), suggests a possible



intramolecular hydrogen bond, as does the O1—H1···N1 bond angle of 144.9 (5)°. Molecules of (1) are linked into pairs by intermolecular N2—H2···N3' hydrogen bonds (Fig. 1) [N2···N3' 3.116 (5) Å and