Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

6,9-Methano-4-methyl-3a,4,5a,6,9,10-hexahydro-5-oxa-1*H*,3*H*-furano[3,4-c]isoindol-3-one

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Received 12 February 2001
Accepted 16 February 2001
Online 28 February 2001

Key indicators

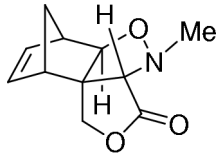
Single-crystal X-ray study
T = 200 K
Mean σ(C–C) = 0.002 Å
R factor = 0.043
wR factor = 0.112
Data-to-parameter ratio = 16.6

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

The regio- and stereochemistry of the title compound, $C_{11}H_{13}NO_3$, has been established. The geometric parameters show normal values.

Comment

Recently, we reported the first examples of intramolecular 1,3-dipolar cycloadditions of norbornadiene-tethered nitrones (Tranmer *et al.*, 2000). Although eight different regio- and stereoisomers could be formed in the cycloaddition of *N*-methyl-α-bicyclo[2.2.1]hepta-2,5-dien-2-ylmethoxycarbonyl nitrone, a single cycloadduct, (I), was obtained. The regio- and stereochemistry of the cycloadduct was established by our single-crystal X-ray diffraction analysis as shown in the scheme and Fig. 1.



(I)

Experimental

N-Methyl-α-bicyclo[2.2.1]hepta-2,5-dien-2-ylmethoxycarbonyl nitrone, which was generated *in situ* by the addition of *N*-methyl hydroxylamine, pyridine and 4 Å molecular sieves to oxoacetic acid bicyclo[2.2.1]hepta-2,5-dien-2-ylmethyl ester in toluene, undergoes spontaneous intramolecular cycloaddition at 358 K to provide cycloadduct (I) as the only regio- and stereoisomer. Suitable crystals were grown from an ethyl acetate/hexanes (2:8) mixture.

Crystal data

$C_{11}H_{13}NO_3$
M_r = 207.22
Orthorhombic, *Pbca*
a = 8.6209 (2) Å
b = 13.9266 (4) Å
c = 16.4518 (6) Å
V = 1975.2 (1) Å^3
Z = 8
D_x = 1.394 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 10 386 reflections
θ = 2.6–27.5°
μ = 0.10 mm^{-1}
T = 200 (1) K
Block, colourless
0.27 × 0.25 × 0.22 mm

Data collection

Nonius KappaCCD diffractometer
φ s, and ω scans with κ offsets
Absorption correction: multi-scan (*DENZO–SMN*; Otwinowski & Minor, 1997)
T_{min} = 0.973, T_{max} = 0.978
10 386 measured reflections
2254 independent reflections
1745 reflections with $I > 2\sigma(I)$
R_{int} = 0.022
θ_{max} = 27.5°
$h = 0 \rightarrow 11$
$k = 0 \rightarrow 18$
$l = 0 \rightarrow 21$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0478P)^2 + 0.5639P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)] = 0.043$	$(\Delta/\sigma)_{max} < 0.001$
$wR(F^2) = 0.112$	$\Delta\rho_{max} = 0.20$ e Å^{-3}
$S = 1.04$	$\Delta\rho_{min} = -0.17$ e Å^{-3}
2254 reflections	
136 parameters	
H-atom parameters constrained	

Table 1
Selected geometric parameters (Å, °).

O1—C8	1.3490 (18)	N6—C10	1.454 (2)
O1—C2	1.4541 (18)	N6—C7	1.4811 (17)
C2—C3	1.5363 (18)	C7—C8	1.5055 (18)
C3—C7	1.5190 (19)	C8—O9	1.2016 (17)
C3—C4	1.5555 (18)	C11—C12	1.519 (2)
C3—C11	1.5566 (18)	C11—C15	1.5341 (19)
C4—O5	1.4314 (17)	C12—C13	1.326 (2)
C4—C14	1.541 (2)	C13—C14	1.520 (2)
O5—N6	1.4726 (15)	C14—C15	1.540 (2)

H atoms were treated as riding atoms with C—H distances in the range 0.95–1.00 Å.

Data collection: *COLLECT* (Nonius, 1997); cell refinement: *DENZO–SMN* (Otwinowski & Minor, 1997); data reduction: *DENZO–SMN*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*II (Johnson, 1976) and *PLATON* (Spek, 2001); software used to prepare material for publication: *SHELXL*97 and *PRPKAPPA* (Ferguson, 1999).

We thank Dr. Sean Conway of the IUCr Chester staff and Professor George Ferguson for assistance with CIF preparation.

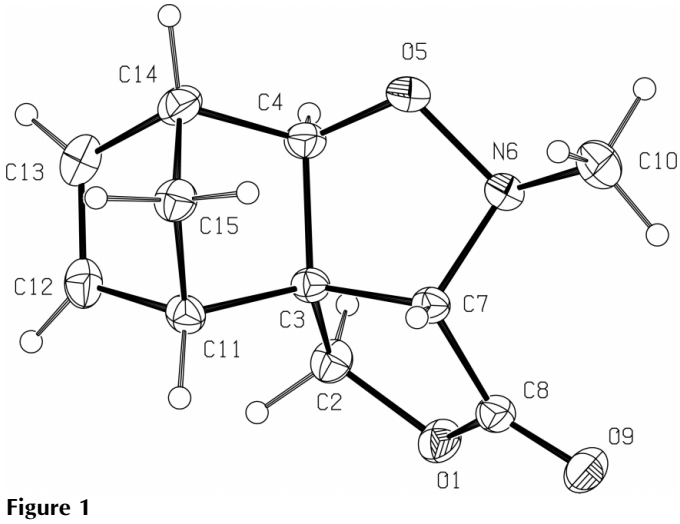


Figure 1
A view of (I) with the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level.

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