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2,3,3a,4,5a,6,7,8-Octahydro-5a-methyl-6,9-methano-1*H*,9*H*-5-oxa-4-azacyclopenta[*c*]indene

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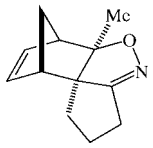
The regio- and stereochemistry of the title compound, $C_{12}H_{15}NO$, has been established by X-ray analysis. The molecular dimensions are normal.

Comment

Recently, we reported the first examples of intramolecular 1,3-dipolar cycloadditions of norbornadiene-tethered nitrile oxides (Yip *et al.*, 1999). Although four different regio- and stereoisomers could be formed in the cycloaddition of 4-(2′-norbornadienyl)butyl nitrile oxide, a single cycloadduct (I) was obtained. The regio- and stereochemistry was established by our single-crystal X-ray diffraction analysis.



(I)

Experimental

4-(2′–Norbornadienyl)butyl nitrile oxide which was generated *in situ* by the addition of di-*tert*-butylcarbonate and 4-(dimethylamino)-pyridine to 1-nitro-4-(2′-norbornadienyl)butane in toluene, undergoes spontaneous intramolecular cycloaddition at 363 K to provide cycloadduct (I) as the only regio- and stereoisomer. Suitable crystals were grown from ethyl acetate/hexanes (2:8) mixture.

Crystal data

$C_{12}H_{15}NO$	$D_x = 1.267$ Mg m^{-3}
$M_r = 189.25$	Mo $K\alpha$ radiation
Monoclinic, $P2_1/n$	Cell parameters from 25 reflections
$a = 7.640$ (2) Å	$\theta = 9.47$–$12.13°$
$b = 12.7121$ (14) Å	$\mu = 0.080$ mm^{-1}
$c = 10.2205$ (7) Å	$T = 294$ (1) K
$\beta = 91.668$ (11)°	Plate, colorless
$V = 992.2$ (3) Å^3	0.42 × 0.42 × 0.19 mm
$Z = 4$	

Data collection

Enraf–Nonius CAD-4 diffractometer	$\theta_{max} = 25.50°$
$\theta/2\theta$ scans	$h = -9 \rightarrow 9$
1997 measured reflections	$k = 0 \rightarrow 15$
1860 independent reflections	$l = 0 \rightarrow 12$
1314 reflections with $I > 2\sigma(I)$	3 standard reflections
$R_{int} = 0.011$	frequency: 120 min
	intensity decay: 6.2%

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0523P)^2 + 0.0760P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)] = 0.0442$	$(\Delta/\sigma)_{max} < 0.001$
$wR(F^2) = 0.1090$	$\Delta\rho_{max} = 0.171$ e Å^{-3}
$S = 1.044$	$\Delta\rho_{min} = -0.137$ e Å^{-3}
1860 reflections	Extinction correction: *SHELXL*97
129 parameters	Extinction coefficient: 0.103 (7)
H-atom parameters constrained	

Molecule (I) crystallized in the monoclinic system; space group $P2_1/n$ from the systematic absences. H atoms were treated as riding atoms (C—H 0.93–0.98 Å).

Data collection: *CAD*-4-*PC Software* (Enraf–Nonius, 1992); cell refinement: *SET*4 and *CELDIM* (Enraf–Nonius, 1992); data reduction: *DATRD*2 in *NRCVAX*96 (Gabe *et al.*, 1989); program(s) used to solve structure: *SOLVER* in *NRCVAX*96; program(s) used to refine structure: *NRCVAX*96 and *SHELXL*97 (Sheldrick, 1997); software used to prepare material for publication: *NRCVAX*96, *SHELXL*97 and *WordPerfect* macro *PREP*8 (Ferguson, 1998).

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References

Enraf–Nonius (1992). *CAD*-4-*PC Software*. Version 1.1. Enraf–Nonius, Delft, Holland.
Ferguson, G. (1998). *PREP*8. University of Guelph, Canada.
Gabe, E. J., Le Page, Y., Charland, J.-P., Lee, F. L. & White, P. S. (1989). *J. Appl. Cryst.* **22**, 384–387.
Sheldrick, G. M. (1997). *SHELXL*97. University of Göttingen, Germany.
Yip, C., Handerson, S., Jordan. R. & Tam, W. (1999). *Org. Lett.* **1**, 791–794.