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Supplementary data for this paper are available from the IUCr electronic archives (Reference: HA1239). Services for accessing these data are described at the back of the journal.

## References

Brown, C. J. (1966). *Acta Cryst.* **21**, 1–5.

Byriel, K. A., Kennard, C. H. L., Lynch, D. E., Smith, G. & Thompson, J. G. (1992). *Aust. J. Chem.* **45**, 969–981.

Disalvo, F. J. (1990). *Science*, **247**, 649–655.

Goswami, S., Mahapatra, A. K., Ghosh, K., Nigam, G. D., Chinnakali, K. & Fun, H.-K. (1999). *Acta Cryst.* C**55**, 87–89.

Li, Q. & Mak, T. C. W. (1997). *Acta Cryst.* B**53**, 252–261.

Lynch, D. E., Smith, G., Freney, D., Byriel, K. A. & Kennard, C. H. L. (1994). *Aust. J. Chem.* **47**, 1097–1115.

Nardelli, M. (1995). *J. Appl. Cryst.* **28**, 659.

Scheinbeim, J. B. & Schempp, E. (1976). *Acta Cryst.* B**32**, 566–571.

Sheldrick, G. M. (1997). *SHELXTL. Structure Determination Programs.* Version 5.10. Bruker Analytical X-ray Systems Inc., Madison, Wisconsin, USA.

Siemens (1996). *SMART* and *SAINT. Area Detector Control and Integration Software.* Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.

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*Acta Cryst.* (1999). C**55**, 585-586

# 5,10-Dimethoxy-1,4-methano-1,4,4a,11a-tetrahydrobenzo[*b*]fluoren-11-one oxime

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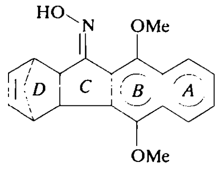
## Abstract

In the title compound, $C_{20}H_{19}NO_3$, the oxime moiety C=N—O—H adopts a *trans* conformation. Furthermore, the molecule as a whole exists in a dimeric form through hydrogen bonding between the oxime groups.

## Comment

The title compound was prepared in connection with our work on the synthesis of phenanthroviridin, an anticancer drug (Gore *et al.*, 1992). The X-ray structure determination of the title compound, (I), was undertaken in order to determine the stereochemistry of the oxime moiety, which is very important for the planned Beckmann rearrangement.



(I)

The bond lengths and angles observed in this structure have normal values (Allen *et al.*, 1987; Beddoes *et al.*, 1993); the C9—C10 and N1—C6 bonds show double-bond character. The oxime moiety C=N—O—H adopts a *trans* conformation. The six-membered ring, *D*, of the norbornene ring system is a fairly symmetrical boat with asymmetry parameters $\Delta C_s$(C7—C12) = 0.019 (1) and $\Delta C_s$(C8) = 0.024 (1) (Nardelli, 1983). The two five-membered rings in the norbornene moiety formed by the bridging atom C14 adopt envelope conformations. The angles between the three-atom bridge plane C8—C14—C11 and four-atom planes of the six-membered rings (C8, C9, C10, C11 and C7, C8, C11, C12) are 52.0 (2) and 60.2 (2)°, respectively. The C9—C10—C7—C12 plane is nearly perpendicular [dihedral angle 87.59 (8)°] to the five-membered ring *C*. In the solid state, inversion-related molecules exist in dimeric form through O—H···N hydrogen bonding (Table 2).

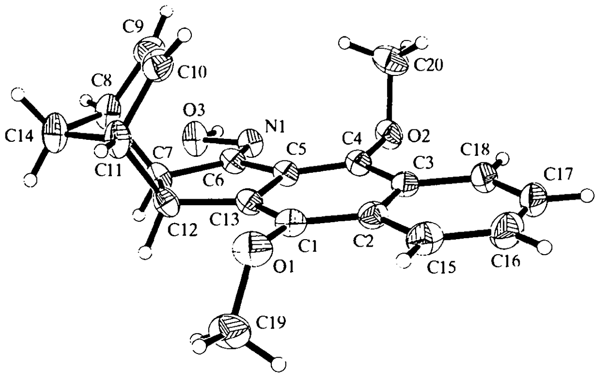


Fig. 1. The structure of the title compound showing 50% probability displacement ellipsoids and the atom-numbering scheme.

## Experimental

To synthesize the title compound, 3-phenylsulphonylphthalide (1.100 mg, 4.0 mmol) was allowed to react with tricyclo-

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[5.2.1$^{2,6}$]deca-3,8-diene-5-one (644 mg, 4.4 mmol) in the presence of lithium *tert*-butoxide (960 mg, 12.0 mmol) in tetrahydrofuran at 213 K ($CHCl_2$/liquid $N_2$ bath) under an argon atmosphere, which provided a solid mass after quenching with a 20% HCl solution. The solid was refluxed for 2 h with $Me_2SO_4$ (1.000 g, 8.0 mmol) in the presence of $K_2CO_3$ (2.800 g, 20.0 mmol) in acetone. After the usual work-up, the dimethylated product was refluxed for 1 h with $NH_3Cl{\cdot}OH$ (280 mg, 4.0 mmol) and $CH_3COONa$ (330 mg, 0.4 mmol) in ethanol. Cooling of the reaction mixture furnished the title compound in 65% overall yield (836 mg). Single crystals were grown by cooling a hot methanol solution of the compound.

*Crystal data*

$C_{20}H_{19}NO_3$
$M_r = 321.36$
Triclinic
$P\bar{1}$
$a = 8.8166\ (6)$ Å
$b = 9.9326\ (6)$ Å
$c = 9.9764\ (6)$ Å
$\alpha = 76.875\ (2)°$
$\beta = 76.582\ (1)°$
$\gamma = 74.331\ (1)°$
$V = 805.52\ (9)$ Å$^3$
$Z = 2$
$D_x = 1.325$ Mg m$^{-3}$
$D_m$ not measured

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 2150 reflections
$\theta = 2.93–27.50°$
$\mu = 0.089$ mm$^{-1}$
$T = 293\ (2)$ K
Block
0.30 × 0.24 × 0.20 mm
Colourless

*Data collection*

Siemens SMART CCD area-detector diffractometer
$\omega$ scans
Absorption correction: none
5269 measured reflections
3569 independent reflections

2448 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.024$
$\theta_{\rm max} = 27.50°$
$h = -10 \rightarrow 11$
$k = -12 \rightarrow 12$
$l = 0 \rightarrow 12$

*Refinement*

Refinement on $F^2$
$R[F^2 > 2\sigma(F^2)] = 0.058$
$wR(F^2) = 0.171$
$S = 1.02$
3569 reflections
223 parameters
H atoms: see text
$w = 1/[\sigma^2(F_o^2) + (0.0866P)^2 + 0.2047P]$
where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\rm max} < 0.001$
$\Delta\rho_{\rm max} = 0.25$ e Å$^{-3}$
$\Delta\rho_{\rm min} = -0.21$ e Å$^{-3}$
Extinction correction: none
Scattering factors from *International Tables for Crystallography* (Vol. C)

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

| | | | |
|---|---|---|---|
| O1—C1 | 1.384 (2) | O3—N1 | 1.411 (2) |
| O1—C19 | 1.408 (3) | N1—C6 | 1.279 (2) |
| O2—C4 | 1.384 (2) | C9—C10 | 1.339 (4) |
| O2—C20 | 1.430 (3) | | |
| C11—C14—C8 | 93.72 (17) | | |
| C19—O1—C1—C2 | −99.7 (2) | C5—C4—O2—C20 | 87.1 (2) |
| C20—O2—C4—C3 | −93.4 (2) | C13—C1—O1—C19 | 83.9 (3) |
| O3—N1—C6—C5 | 177.57 (16) | | |

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| O3—H1O3···N1$^{\rm i}$ | 0.92 (3) | 1.96 (3) | 2.811 (2) | 154 (3) |

Symmetry code: (i) $1 - x, -y, 2 - z$.

The data collection covered over a hemisphere of reciprocal space by a combination of three sets of exposures; each set had a different $\varphi$ angle (0, 88 and 180°) for the crystal and each exposure of 30 s covered 0.3° in $\omega$. 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 the data collection and analysing the intensity of duplicate reflections, and was found to be negligible.

H atoms were fixed geometrically (except for the OH group) and allowed to ride on the atoms to which they are attached. Rotating-group refinement (AFIX137) was used for the two methyl groups present in the structure.

Data collection: *SMART* (Siemens, 1996*a*). Cell refinement: *SAINT* (Siemens, 1996*b*). Data reduction: *SAINT*. Program(s) used to solve structure: *SHELXTL* (Sheldrick, 1996). 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: BK1435). Services for accessing these data are described at the back of the journal.

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## References

Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans.* 2, pp. S1–19.
Beddoes, R. L., Gorman, A. A. & McNeeney, S. P. (1993). *Acta Cryst.* C**49**, 1811–1813.
Gore, M. P., Gould, S. J. & Weller, D. D. (1992). *J. Org. Chem.* **57**, 2774–2783.
Nardelli, M. (1983). *Acta Cryst.* C**39**, 1141–1142.
Nardelli, M. (1995). *J. Appl. Cryst.* **28**, 659.
Sheldrick, G. M. (1996). *SHELXTL*. Version 5. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
Siemens (1996*a*). *SMART. Area Detector Control Software.* Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
Siemens (1996*b*). *SAINT. Area Detector and Integration Software.* Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.