1985), *SHELX*76 (Sheldrick, 1976) and *PARST* (Nardelli, 1983). Calculations were performed using a MicroVAX II computer.

The authors thank Professor Vasantha Pattabhi for disscussions and one of us (VK) thanks the University of Madras for financial support.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates, complete geometry and torsion angles have been deposited with the IUCr (Reference: SZ1027). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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A Tetralin Derivative

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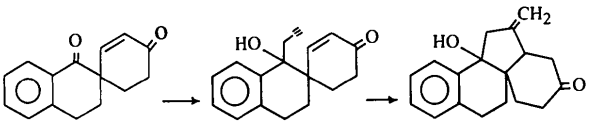
(*Received 3 March 1995; accepted 18 July 1995*)

Abstract

In the structure of 1-hydroxy-1-propynyl-1,2,3,4-tetrahydronaphthalene-2-spiro-(2′-cyclohexen-4′-one), $C_{18}H_{18}O_2$, the C_{sp^2} atom at the spiro junction (axial) is *trans* to the propargyl chain (pseudo-axial). The crystal structure is stabilized by O—H···O hydrogen bonds.

Comment

The structure determination of the title compound, (2), is a continuation of our studies in tin-mediated vinyl radical cyclization of similarly substituted substrates (Janarthanam, Balakumar & Rajagopalan, 1994). Our efforts were directed towards the synthesis of compound (3) as a model for Retigeranic acid-type molecules (Janarthanam, Shanmugam & Rajagopalan, 1993).



(1) (2) (3)

In the scheme above, the stereochemistry at C1 [carbonyl in starting material (1)] is fixed by the fact that the introduction of the three-carbon unit is a stereo-electronically controlled step. During the radical cyclization, *i.e.* (2) → (3), a new stereocenter is generated at atom C4′. As the stereochemistry at C1 is fixed, the extent of stereocontrol obtainable at C4′ becomes amenable for study. Thus, the treatment of compound (1) with propargyl sesquialuminium bromide in tetrahydrofuran at 195 K for 3 h gave compound (2) as a mixture of diastereomers in the ratio 90:10. It was of interest to establish the stereochemistry of the major diastereomer with regard to the orientation of the propargyl chain and the enone moiety. The present study reports the X-ray crystallographic investigation of this orientation, as well as the conformation of the partially unsaturated rings.

The central ring adopts a sofa conformation (ΔC_2 = 7.2°) and the enone ring adopts a half-chair conformation (ΔC_s = 8.2°) (Duax, Weeks & Rohrer, 1976). The bond lengths and angles agree with those of similar systems (Nethaji, 1987; Geetha & Rajan, 1991). The sp^2 C4′ atom at the spiro junction (axial) is *trans* to the propargyl chain (pseudo-axial).

The molecular packing in the unit cell involves O—H···O4 hydrogen bonding [O1···O4($-x$, $-y+1$, $-z+2$) 2.905 (2), H···O4 2.06 (3) Å and O1—H···O4 155 (3)°].

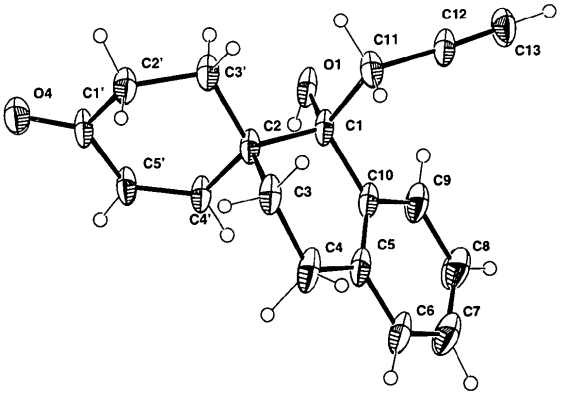


Fig. 1. A perspective diagram of the title compound with ellipsoids plotted at the 30% probability level.

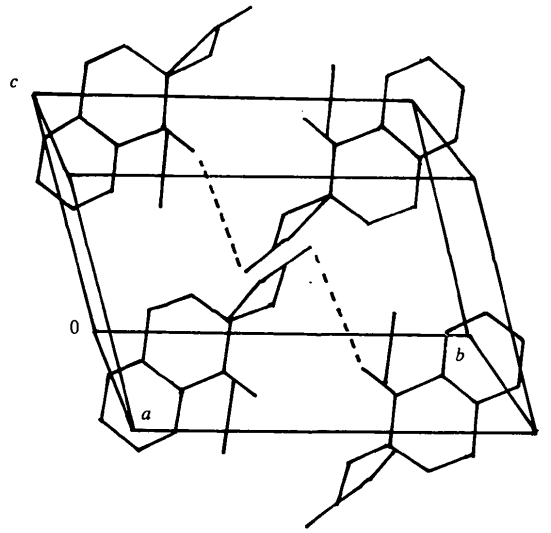


Fig. 2. Unit-cell packing diagram of the title molecule viewed down the *a* axis.

Experimental

Crystals of the title compound (2) were obtained from methanol.

Crystal data

$C_{18}H_{18}O_2$
M_r = 266.33
Triclinic
$P\bar{1}$
a = 7.947 (2) Å
b = 11.949 (3) Å
c = 7.749 (2) Å
α = 106.19 (2)°
β = 96.79 (2)°
γ = 84.70 (2)°
V = 700.3 (3) Å^3
Z = 2
D_x = 1.263 Mg m^{-3}
D_m = 1.25 (2) Mg m^{-3}
D_m measured by flotation in a bromoform/benzene solution

Cu $K\alpha$ radiation
λ = 1.5418 Å
Cell parameters from 25 reflections
θ = 20–30°
μ = 0.60 mm^{-1}
T = 293 K
Needle
0.2 × 0.2 × 0.15 mm
Colorless

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: empirical *via* ψ scans (North, Phillips & Mathews, 1968)
T_{min} = 0.90, T_{max} = 0.96
2385 measured reflections
2385 independent reflections

1757 observed reflections [$I > 3\sigma(I)$]
θ_{max} = 70°
$h = -9 \rightarrow 9$
$k = -14 \rightarrow 14$
$l = 0 \rightarrow 9$
3 standard reflections
frequency: 120 min
intensity decay: 3%

Refinement

Refinement on F
R = 0.039
wR = 0.046

$(\Delta/\sigma)_{max}$ = 0.002
$\Delta\rho_{max}$ = 0.28 e Å^{-3}
$\Delta\rho_{min}$ = −0.35 e Å^{-3}
S = 0.888
1757 reflections
253 parameters
All H-atom parameters refined
$w = 1/[\sigma^2(F) + 0.003639F^2]$

Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV, Table 2.3.1)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters (Å^2)*

$U_{eq} = (1/3)\Sigma_i\Sigma_j U_{ij} a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j.$

	x	y	z	U_{eq}
O1	0.3512 (2)	0.36876 (11)	0.8720 (2)	0.0452 (7)
O4	−0.0382 (2)	0.63760 (13)	1.3648 (2)	0.0550 (9)
C1	0.3892 (2)	0.2959 (2)	0.9913 (2)	0.0381 (9)
C2	0.2955 (2)	0.3488 (2)	1.1660 (2)	0.0380 (9)
C3	0.3290 (3)	0.2639 (2)	1.2872 (3)	0.0485 (11)
C4	0.2651 (4)	0.1439 (2)	1.1949 (3)	0.0622 (14)
C5	0.2802 (3)	0.1017 (2)	0.9941 (3)	0.0474 (11)
C6	0.2282 (3)	−0.0092 (2)	0.8995 (3)	0.0615 (14)
C7	0.2355 (4)	−0.0510 (2)	0.7166 (3)	0.0668 (15)
C8	0.2966 (4)	0.0167 (2)	0.6226 (3)	0.0641 (15)
C9	0.3457 (3)	0.1273 (2)	0.7128 (3)	0.0518 (12)
C10	0.3384 (2)	0.1719 (2)	0.8988 (2)	0.0405 (10)
C11	0.5825 (2)	0.3007 (2)	1.0397 (3)	0.0455 (11)
C12	0.6880 (2)	0.2503 (2)	0.8911 (3)	0.0454 (10)
C13	0.7845 (3)	0.2117 (2)	0.7820 (3)	0.0586 (13)
C1′	0.0566 (2)	0.5502 (2)	1.3170 (2)	0.0445 (10)
C2′	0.2350 (3)	0.5417 (2)	1.4031 (3)	0.0457 (11)
C3′	0.3549 (2)	0.4702 (2)	1.2675 (3)	0.0417 (10)
C4′	0.1083 (2)	0.3575 (2)	1.1089 (2)	0.0409 (10)
C5′	0.0019 (2)	0.4479 (2)	1.1764 (2)	0.0451 (11)

Table 2. *Selected geometric parameters (Å, °)*

O1—C1	1.428 (3)	C5—C10	1.400 (4)
O4—C1′	1.225 (2)	C6—C7	1.372 (3)
C1—C2	1.569 (2)	C7—C8	1.380 (4)
C1—C10	1.524 (3)	C8—C9	1.380 (3)
C1—C11	1.540 (2)	C9—C10	1.397 (3)
C2—C3	1.551 (4)	C11—C12	1.463 (3)
C2—C3′	1.532 (3)	C12—C13	1.177 (3)
C2—C4′	1.504 (2)	C1′—C2′	1.501 (3)
C3—C4	1.517 (3)	C1′—C5′	1.454 (3)
C4—C5	1.512 (3)	C2′—C3′	1.526 (3)
C5—C6	1.397 (3)	C4′—C5′	1.333 (3)
O1—C1—C11	104.0 (2)	O4—C1′—C5′	122.0 (2)
O1—C1—C10	110.9 (1)	O4—C1′—C2′	121.8 (2)
O1—C1—C2	109.6 (2)		
O1—C1—C11—C12	−65.0 (2)	O1—C1—C2—C3′	−61.5 (2)
O1—C1—C10—C5	−147.1 (2)	O1—C1—C2—C4′	58.7 (2)
O1—C1—C10—C9	31.3 (3)	O4—C1′—C5′—C4′	172.3 (2)
O1—C1—C2—C3	176.2 (2)	O4—C1′—C2′—C3′	−146.1 (2)

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989). Cell refinement: *SDP* (Frenz, 1978). Data reduction: *SDP*. Program(s) used to solve structure: *SHELXS86* (Sheldrick, 1985). Program(s) used to refine structure: *SHELX76* (Sheldrick, 1976). Molecular graphics: *ORTEP*II (Johnson, 1976). Geometrical calculations: *PARST* (Nardelli, 1983).

One of the authors (VK) thanks the University of Madras and CSIR for financial support.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates, complete geometry and torsion angles have been deposited with the IUCr (Reference: CF1014). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Acta Cryst. (1996). C**52**, 1985–1987

Nearly Centrosymmetric (*S*)-7-(2,6-Dichlorobenzyl)-8-(3-oxocyclopentyl)-1,3-dipropyl-7*H*-purine-2,6-dione

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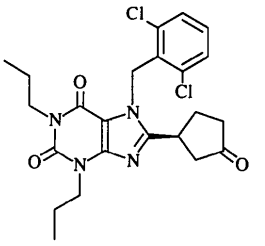
(*Received 19 December 1995; accepted 20 February 1996*)

Abstract

The structure of the title compound, $C_{23}H_{26}Cl_2N_4O_3$, with two molecules in the asymmetric unit, is essentially centrosymmetric apart from the cyclopentanone rings. It was possible, nevertheless, to determine the absolute configuration unambiguously.

Comment

The title compound, (I), is the slightly higher affine (*S*)-(−) enantiomer of KFM 19 (Schingnitz, Küfner-Mühl, Ensinger, Lehr & Kuhn, 1991). It belongs to a group of selective adenosine A_1-antagonists with therapeutic potential for the treatment of dementia and related cognitive deficiencies. The structure analysis was undertaken in order to determine the absolute configuration of the stereogenic centre.



(I)

The compound turned out to be the *S* stereoisomer (Fig. 1). Apart from the cyclopentanone rings, the two molecules in the asymmetric unit show a nearly perfect centrosymmetric arrangement (Fig. 2). The cyclopentan-

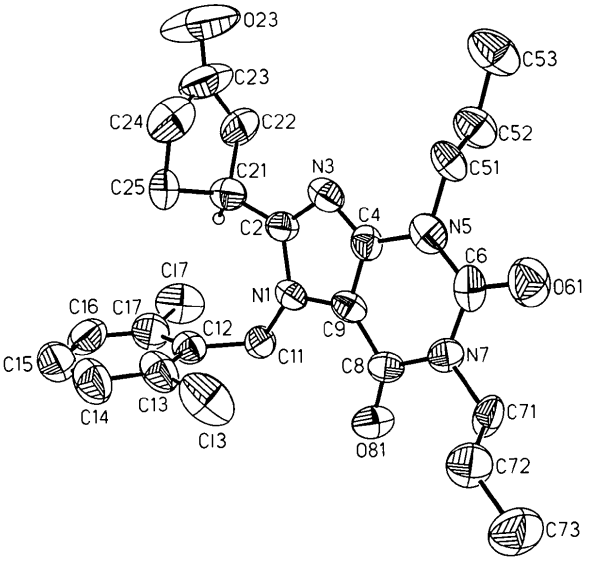


Fig. 1. View of one molecule of the title compound showing the atomic labelling. Only the tertiary H atom is shown and ellipsoids are plotted at the 50% probability level.

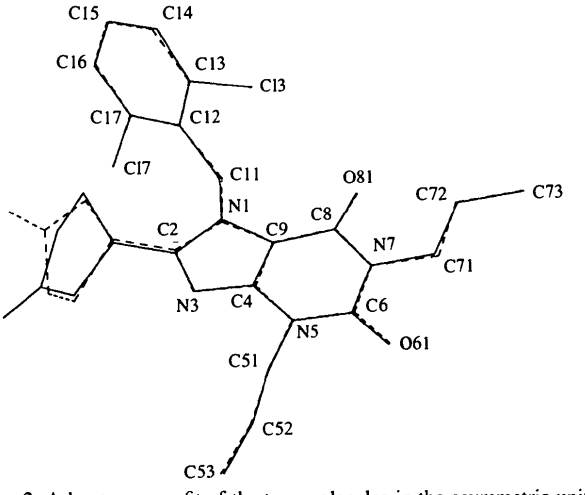


Fig. 2. A least-squares fit of the two molecules in the asymmetric unit with one molecule inverted. Fitted atoms are labelled.