$V = 943.5$ (3) Å^3
$Z = 4$
$D_x = 1.374$ Mg m^{-3}
0.40 × 0.40 × 0.06 mm
Yellow
Crystal source: Aldrich

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: none
1738 measured reflections
1664 independent reflections
1029 observed reflections [$I > 2\sigma(I)$]
$R_{\rm int} = 0.0296$
$\theta_{\rm max} = 24.98°$
$h = -5 \rightarrow 5$
$k = 0 \rightarrow 19$
$l = 0 \rightarrow 15$
2 standard reflections
frequency: 60 min
intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.0543$
$wR(F^2) = 0.1759$
$S = 0.786$
1664 reflections
172 parameters
$w = 1/[\sigma^2(F_o^2) + (0.2096P)^2 + 0.1467P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} = -0.062$
$\Delta\rho_{\rm max} = 0.154$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.253$ e Å^{-3}
Extinction correction: none
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)

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

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

	x	y	z	$U_{\rm eq}$
N10	−0.0070 (5)	0.23733 (15)	0.2255 (2)	0.0473 (7)
C14	−0.3202 (6)	0.2200 (2)	0.0597 (2)	0.0414 (7)
C11	0.0230 (6)	0.3362 (2)	0.0888 (2)	0.0419 (7)
C13	−0.2183 (6)	0.1939 (2)	0.1638 (2)	0.0426 (7)
C9	−0.2017 (6)	0.2928 (2)	0.0185 (2)	0.0441 (7)
C12	0.1149 (6)	0.30593 (15)	0.1917 (2)	0.0400 (7)
C5	−0.3350 (7)	0.1234 (2)	0.2031 (2)	0.0565 (8)
O1	−0.2819 (5)	0.31831 (12)	−0.07342 (13)	0.0641 (7)
C4	0.3352 (6)	0.3476 (2)	0.2584 (2)	0.0511 (8)
C8	−0.5374 (7)	0.1732 (2)	−0.0014 (2)	0.0515 (8)
C1	0.1504 (7)	0.4071 (2)	0.0573 (2)	0.0503 (7)
C2	0.3601 (7)	0.4480 (2)	0.1235 (2)	0.0603 (8)
C7	−0.6469 (7)	0.1050 (2)	0.0375 (3)	0.0644 (9)
C6	−0.5450 (8)	0.0796 (2)	0.1412 (3)	0.0660 (9)
C3	0.4538 (7)	0.4167 (2)	0.2247 (2)	0.0566 (8)

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

Angles at C other than those given below are in the range 118–122°.

N10—C12	1.355 (4)	C13—C5	1.398 (4)
N10—C13	1.363 (3)	C9—O1	1.250 (3)
N10—H10	0.96 (4)	C12—C4	1.406 (4)
C14—C8	1.406 (4)	C5—C6	1.363 (5)
C14—C13	1.411 (3)	C4—C3	1.354 (5)
C14—C9	1.443 (4)	C8—C7	1.350 (4)
C11—C1	1.389 (4)	C1—C2	1.366 (4)
C11—C12	1.410 (3)	C2—C3	1.400 (4)
C11—C9	1.456 (4)	C7—C6	1.402 (5)
C12—N10—C13	123.0 (2)	C14—C9—C11	116.9 (2)
O1—C9—C14	122.8 (2)		

All nine H atoms were located from the Fourier difference map and their positions and isotropic displacement parameters were refined.

Data collection: CAD-4 diffractometer software. Cell refinement: CAD-4 diffractometer software. Data reduction: CAD-4 diffractometer software. Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990*a*). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *PLUTO* (Motherwell & Clegg, 1978). Software used to prepare material for publication: *SHELXL*93.

We acknowledge support from the SERC (CASE studentship for GDP).

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: HA1122). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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(1β,5α,6β)-10,10-(1,2-Ethylenedioxy)-1,5-dimethylbicyclo[4.4.0]decan-4-one

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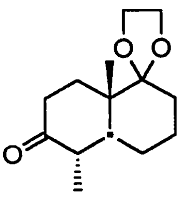
Abstract

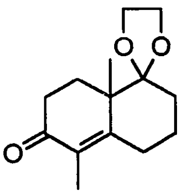
The title compound (1) [alternative name: 5,8a-dimethyl(decahydronaphthalene)-1-spiro-2′-(1′,3′-dioxolan)-6-one, $C_{14}H_{22}O_3$] was obtained as the major prod-

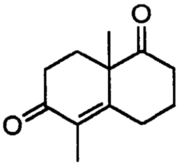
uct upon Pd/CaCO$_3$-catalyzed hydrogenation of the corresponding enone (2). The analysis of the crystal structure of (1) has established that the monoacetal is *cis*-fused and that both rings adopt nearly perfect chair forms. The C(5) methyl group is in an α position, resulting directly from *syn*-hydrogenation of the enone. While there are two possible chair–chair conformations for *cis*-decalins, the title compound crystallized in the conformation which places the C(5) methyl group in an equatorial position.

Comment

Studies directed at the synthesis of the natural product arenarol, a *cis*-fused bicyclo[4.4.0]decane (Schmitz, Lakshmi, Powell & van der Helm, 1984), have involved the preparation of various *cis*-decalones including the title compound, (1). Hydrogenation of the corresponding enone (2) over a Pd/CaCO$_3$ catalyst produced a mixture of diastereomers in a 9:1 ratio based on integration of ^{1}H NMR spectra. Both products have been characterized as *cis*-fused isomers. This was in contrast to the hydrogenation of the corresponding diketone (3), which gave an approximate 3:2 mixture of C(5)-methyl diastereomers of the corresponding *cis*-fused decalin along with approximately 10% of the *trans*-fused analog (Park, Scott & Wiemer, 1994).


(1)


(2)


(3)

The stereo-controlled synthesis of highly substituted *cis*-fused decalins is dependent on precise stereochemical knowledge. As there are two possible chair–chair conformations of *cis*-fused decalones (Heathcock, Ratcliffe & Van, 1972; Huffman & Balke, 1988), the stereochemistry of the C(5) methyl group could not be determined by spectroscopic methods. The determination of the crystal structure of (1) allowed the stereochemistry at C(5) to be assigned unambiguously, as well as establishing the overall conformation of the molecule.

The *cis*-fused bicyclic ring in the title compound adopts a nearly perfect chair–chair conformation. The C(5) methyl group proved to be in the α equatorial position, which must result from β *syn*-hydrogenation in order to attain the overall conformation.

Studies on the reactivity of the title compound have revealed preferred formation of the C(3) enolate over the C(5) isomer (Park, Scott & Wiemer, 1994). The C(3)—C(4) bond is shorter than the C(4)—C(5) bond, which may suggest a tendency towards C(3)—C(4) enol formation.

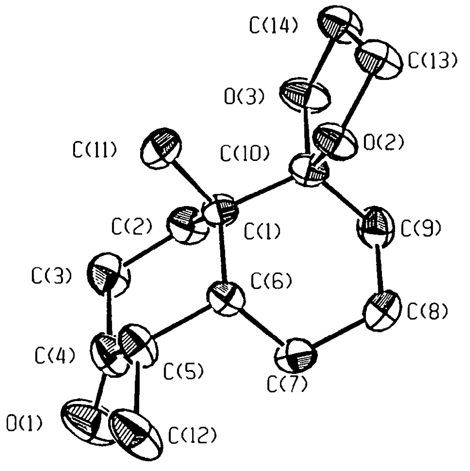


Fig. 1. An *ORTEP* (Johnson, 1965) drawing of the crystal structure of the title compound. Displacement ellipsoids are shown at the 35% probability level.

Experimental

Catalytic hydrogenation of the enone (2) over Pd/CaCO$_3$ in pyridine was performed under 60 psi of hydrogen [1 psi $\simeq$ 6.895 × 10^3 Pa] at room temperature over 2 d to give a mixture of title compound and the C(5) epimer in a 9:1 ratio. The major product was separated by column chromatography and dissolved in ether at room temperature. The solution was placed in a closed vial and allowed to stand at 258 K for 3 d. The remaining solvent was decanted, and the colorless crystals were washed quickly with cold ether (258 K) and dried under air.

Crystal data

$C_{14}H_{22}O_3$
$M_r = 238.3$
Monoclinic
$P2_1/c$
$a = 7.889\ (1)$ Å
$b = 19.490\ (3)$ Å
$c = 8.559\ (1)$ Å
$\beta = 101.70\ (1)^\circ$
$V = 1288.7\ (6)$ Å^3
$Z = 4$
$D_x = 1.23$ Mg m^{-3}

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 13–16^\circ$
$\mu = 0.079$ mm^{-1}
$T = 295$ K
Irregular cube
0.28 × 0.25 × 0.22 mm
Colorless

Data collection

Enraf–Nonius CAD-4 diffractometer
θ–2θ scans [range (0.8 + 0.35tanθ)°, speed 1.67–5.0° min^{-1}, background determined by the Lehman–Larson method]
Absorption correction: none
5325 measured reflections
2622 independent reflections

1840 observed reflections [$I > 2\sigma(I)$]
$R_{\rm int} = 0.058$ (on I)
$\theta_{\rm max} = 30^\circ$
$h = -10 \rightarrow 10$
$k = -18 \rightarrow 25$
$l = -11 \rightarrow 5$
4 standard reflections
frequency: 240 min
intensity decay: <2.0%

Refinement

Refinement on F
$R = 0.046$
$wR = 0.072$
$S = 1.43$
1840 reflections
154 parameters
H atoms were included in the structure-factor calculations at fixed positions with C—H = 0.95 Å
$w = 1/[\sigma^2(F) + (0.04F)^2]$
$(\Delta/\sigma)_{\max} = 0.03$
$\Delta\rho_{\max} = 0.22$ e Å^{-3}
$\Delta\rho_{\min} = -0.15$ e Å^{-3}
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV, Table 2.2B)

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

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

	x	y	z	$U_{\rm eq}$
O(1)	0.4615 (2)	0.8721 (1)	0.1527 (2)	0.0978 (5)
O(2)	0.9159 (1)	0.85509 (6)	0.8481 (1)	0.0504 (3)
O(3)	1.0664 (1)	0.93121 (6)	0.7265 (1)	0.0598 (4)
C(1)	0.8336 (2)	0.86594 (8)	0.5612 (2)	0.0404 (4)
C(2)	0.8426 (2)	0.91508 (9)	0.4218 (2)	0.0531 (5)
C(3)	0.7603 (3)	0.8857 (1)	0.2588 (2)	0.0655 (5)
C(4)	0.5842 (3)	0.8565 (1)	0.2549 (2)	0.0613 (5)
C(5)	0.5703 (2)	0.80745 (9)	0.3882 (2)	0.0550 (5)
C(6)	0.6438 (2)	0.84245 (8)	0.5514 (2)	0.0422 (4)
C(7)	0.5260 (2)	0.9009 (1)	0.5847 (2)	0.0552 (5)
C(8)	0.5950 (2)	0.9355 (1)	0.7431 (2)	0.0632 (5)
C(9)	0.7778 (2)	0.96122 (9)	0.7524 (2)	0.0588 (5)
C(10)	0.8976 (2)	0.90382 (8)	0.7212 (2)	0.0432 (4)
C(11)	0.9534 (2)	0.8043 (1)	0.5562 (2)	0.0617 (5)
C(12)	0.3883 (3)	0.7788 (1)	0.3735 (3)	0.0865 (6)
C(13)	1.0684 (2)	0.8728 (1)	0.9620 (2)	0.0599 (5)
C(14)	1.1795 (2)	0.9087 (1)	0.8675 (2)	0.0585 (5)

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

O(1)—C(4)	1.205 (2)	C(3)—C(4)	1.496 (3)
O(2)—C(10)	1.428 (2)	C(4)—C(5)	1.510 (3)
O(2)—C(13)	1.428 (2)	C(5)—C(6)	1.556 (2)
O(3)—C(10)	1.427 (2)	C(5)—C(12)	1.522 (3)
O(3)—C(14)	1.417 (2)	C(6)—C(7)	1.533 (2)
C(1)—C(2)	1.543 (2)	C(7)—C(8)	1.513 (2)
C(1)—C(6)	1.552 (2)	C(8)—C(9)	1.514 (3)
C(1)—C(10)	1.548 (2)	C(9)—C(10)	1.523 (2)
C(1)—C(11)	1.534 (2)	C(13)—C(14)	1.483 (3)
C(2)—C(3)	1.526 (2)		
C(10)—O(2)—C(13)	107.4 (1)	C(6)—C(5)—C(12)	114.1 (2)
C(10)—O(3)—C(14)	109.5 (1)	C(1)—C(6)—C(5)	111.0 (1)
C(2)—C(1)—C(6)	109.6 (1)	C(1)—C(6)—C(7)	112.6 (1)
C(2)—C(1)—C(10)	109.3 (1)	C(5)—C(6)—C(7)	111.3 (1)
C(2)—C(1)—C(11)	109.8 (1)	C(6)—C(7)—C(8)	112.1 (1)
C(6)—C(1)—C(10)	109.1 (1)	C(7)—C(8)—C(9)	111.2 (2)
C(6)—C(1)—C(11)	111.1 (1)	C(8)—C(9)—C(10)	111.4 (1)
C(10)—C(1)—C(11)	107.8 (1)	O(2)—C(10)—O(3)	106.0 (1)
C(1)—C(2)—C(3)	113.4 (1)	O(2)—C(10)—C(1)	108.7 (1)
C(2)—C(3)—C(4)	112.6 (2)	O(2)—C(10)—C(9)	109.0 (1)
O(1)—C(4)—C(3)	121.8 (2)	O(3)—C(10)—C(1)	109.9 (1)
O(1)—C(4)—C(5)	122.5 (2)	O(3)—C(10)—C(9)	109.2 (1)
C(3)—C(4)—C(5)	115.7 (1)	C(1)—C(10)—C(9)	113.8 (1)
C(4)—C(5)—C(6)	109.2 (1)	O(2)—C(13)—C(14)	104.6 (1)
C(4)—C(5)—C(12)	112.4 (1)	O(3)—C(14)—C(13)	105.7 (1)

The structure was determined by direct methods using *MolEN* (Fair, 1990). Data collection: *CAD*-4 *Software* (Enraf–Nonius, 1989). Cell refinement: local program. Data reduction: *MolEN*. Program(s) used to refine structure: *MolEN*.

We thank Mr M. A. Capron for his help in preparing the X-ray data tables. Financial support from the National Institutes of Health and the University of Iowa Graduate College, in the form of an assistantship for KP, is gratefully acknowledged.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates, bond distances and angles involving non-H atoms and torsion angles have been deposited with the IUCr (Reference: BK1068). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Oxime of 1-Methyl-3-trifluoroacetylpyrrolidin-2-one

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Abstract

The title compound, 3-(1-hydroxyimino-2,2,2-trifluoroethyl)-1-methylpyrrolidin-2-one, $C_7H_9F_3N_2O_2$, is obtained by condensation of 1-methyl-3-trifluoroacetylpyrrolidin-2-one and hydroxylamine without ring opening of the lactam. The least-squares planes through the pyrrolidine and oxime groups are approximately perpendicular to each other.