First publ. in: Acta Crystallographica, Section C, 49 (1993), 9, pp. 1654-1655

Structure of (±)-1β-*tert*-Butoxy-3aα,4β,5,6,7,7a-hexahydro-7aβ-methyl-5-oxo-4α-indancarboxylic Acid Methyl Ester at 153 K

EHMKE POHL, REGINE HERBST-IRMER AND GEORGE M. SHELDRICK

Institut für Anorganische Chemie, Universität Göttingen, Tammannstrasse 4, 3400 Göttingen, Germany

SIEGLINDE VAN HOMMELEN AND ULRICH GROTH

Institut für Organische Chemie, Universität Göttingen, Tammannstrasse 2, 3400 Göttingen, Germany

(*Received* 6 *October* 1992; *accepted* 15 *February* 1993)

Abstract

The X-ray structure of (±)-1β-*tert*-butoxy-3aα,4β,5,6,-7,7a-hexahydro-7aβ-methyl-5-oxo-4α-indancarboxylic acid methyl ester is reported, in which the six-membered structure determination was undertaken to investigate the stereospecificity of this step. Colourless crystals were obtained by slow evaporation from a mixture of diethyl ether–pentane at room temperature. The six-membered ring adopts a pseudo chair conformation and the five-membered ring an envelope conformation. The bond distances are comparable with the corresponding distances in other hexahydroindan derivatives (Schomer, Sheldrick & Wagner, 1978; D'Angelo *et al.*, 1983; Caine *et al.*, 1987).

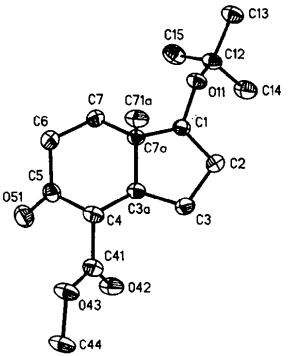

Fig. 1. Structure of the title compound showing 50% probability displacement ellipsoids. The H atoms are omitted for clarity.

Experimental

Crystal data

$C_{16}H_{26}O_4$
M_r = 282.37
Monoclinic
$P2_1/c$
a = 12.420 (2) Å
b = 11.180 (2) Å
c = 11.979 (2) Å
β = 110.91 (2)°
V = 1553.8 (3) Å^3
Z = 4
D_x = 1.207 Mg m^{-3}
Mo $K\alpha$ radiation
λ = 0.71073 Å
Cell parameters from 40 reflections
θ = 10-12.5°
μ = 0.085 mm^{-1}
T = 153 (2) K
Blocks
0.4 × 0.3 × 0.3 mm
Colourless

Data collection

Stoe-Siemens AED four-circle diffractometer
Profile data from $2\theta/\omega$ scans
Absorption correction: none
3755 measured reflections
2757 independent reflections
2043 observed reflections [$I > 2\sigma(I)$]
R_{int} = 0.0974
θ_{max} = 25.07°
$h = -14 \rightarrow 14$
$k = -13 \rightarrow 13$
$l = -14 \rightarrow 14$
3 standard reflections
frequency: 90 min
intensity variation: none

Refinement

Refinement on F^2
Final $R1$ = 0.0434 for $F > 4\sigma F$
$wR2$ = 0.1131 for all data
S = 1.047
2756 reflections
203 parameters
Calculated weights $w = 1/[\sigma^2(F_o^2) + (0.0447P)^2 + 0.8231P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.000
$\Delta\rho_{max}$ = 0.183 e Å^{-3}
$\Delta\rho_{min}$ = −0.181 e Å^{-3}
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)

Data collection: *DIF*4 (Stoe & Cie, 1988). Cell refinement: *DIF*4. Data reduction: *REDU*4 (Stoe & Cie, 1988). Program(s) used to solve structure: *SHELXS*-90 (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL*-92 (Sheldrick, 1992). Molecular graphics: *SHELXTL-Plus* (Sheldrick, 1991). Software used to prepare material for publication: *SHELXL*-92.

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

$U_{eq} = \frac{1}{3}\sum_i\sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j$.

	x	y	z	U_{eq}
C1	0.6876 (2)	0.0500 (2)	0.4445 (2)	0.0262 (9)
O11	0.57892 (10)	0.02326 (11)	0.35486 (11)	0.0271 (7)
C12	0.5487 (2)	0.0912 (2)	0.2450 (2)	0.0287 (10)
C13	0.4240 (2)	0.0572 (2)	0.1769 (2)	0.0345 (10)
C14	0.5578 (2)	0.2245 (2)	0.2709 (2)	0.0428 (12)
C15	0.6231 (2)	0.0544 (2)	0.1747 (2)	0.0430 (12)
C2	0.6781 (2)	0.1198 (2)	0.5510 (2)	0.0348 (11)
C3	0.7839 (2)	0.0825 (2)	0.6604 (2)	0.0319 (10)
C3a	0.8498 (2)	−0.0025 (2)	0.60771 (15)	0.0252 (9)
C4	0.9328 (2)	−0.0924 (2)	0.6887 (2)	0.0280 (9)
C41	1.0263 (2)	−0.0362 (2)	0.7929 (2)	0.0288 (10)
C44	1.1527 (2)	−0.0709 (2)	0.9908 (2)	0.0416 (11)
O42	1.06479 (12)	0.06239 (13)	0.79478 (12)	0.0384 (8)
O43	1.06165 (11)	−0.11227 (12)	0.88501 (11)	0.0352 (7)
C5	0.9869 (2)	−0.1630 (2)	0.6128 (2)	0.0306 (11)
O51	1.09036 (12)	−0.17390 (12)	0.64227 (13)	0.0389 (8)
C6	0.9037 (2)	−0.2157 (2)	0.4990 (2)	0.0386 (12)
C7	0.8120 (2)	−0.1263 (2)	0.4246 (2)	0.0305 (10)
C7a	0.75570 (15)	−0.0623 (2)	0.50172 (15)	0.0238 (9)
C71a	0.6791 (2)	−0.1476 (2)	0.5401 (2)	0.0323 (10)

Table 2. *Geometric parameters* (Å, °)

C1—O11	1.425 (2)	C4—C41	1.506 (3)
C1—C7a	1.533 (2)	C4—C5	1.529 (3)
C1—C2	1.536 (3)	C41—O42	1.198 (2)
O11—C12	1.448 (2)	C41—O43	1.337 (2)
C12—C15	1.513 (3)	C44—O43	1.441 (2)
C12—C14	1.518 (3)	C5—O51	1.211 (2)
C12—C13	1.518 (3)	C5—C6	1.506 (3)
C2—C3	1.546 (3)	C6—C7	1.539 (3)
C3—C3a	1.530 (3)	C7—C7a	1.521 (3)
C3a—C4	1.517 (2)	C7a—C71a	1.529 (3)
C3a—C7a	1.539 (2)		
O11—C1—C7a	112.84 (14)	C3a—C4—C5	107.72 (14)
O11—C1—C2	113.53 (15)	O42—C41—O43	124.0 (2)
C7a—C1—C2	103.79 (14)	O42—C41—C4	125.7 (2)
C1—O11—C12	116.31 (13)	O43—C41—C4	110.3 (2)
O11—C12—C15	110.6 (2)	C41—O43—C44	116.4 (2)
O11—C12—C14	110.7 (2)	O51—C5—C6	122.5 (2)
C15—C12—C14	111.4 (2)	O51—C5—C4	121.7 (2)
O11—C12—C13	103.97 (14)	C6—C5—C4	115.8 (2)
C15—C12—C13	110.0 (2)	C5—C6—C7	113.4 (2)
C14—C12—C13	110.0 (2)	C7a—C7—C6	111.0 (2)
C1—C2—C3	105.85 (15)	C7—C7a—C71a	111.1 (2)
C3a—C3—C2	103.65 (14)	C7—C7a—C1	114.37 (15)
C4—C3a—C3	119.29 (15)	C71a—C7a—C1	110.0 (2)
C4—C3a—C7a	112.45 (15)	C7—C7a—C3a	109.09 (15)
C3—C3a—C7a	104.44 (14)	C71a—C7a—C3a	113.23 (14)
C41—C4—C3a	113.6 (2)	C1—C7a—C3a	98.52 (14)
C41—C4—C5	109.6 (2)		

This work was supported by the Deutsche Forschungsgemeinschaft and the Fonds der Chemischen Industrie.

Lists of structure factors, anisotropic displacement parameters and H-atom coordinates have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71118 (13 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: SE1022]

References

Caine, D., McCloskey, C. J., Atwood, J. L., Bott, S. G., Zhang, H. M. & VanDerveer, D. (1987). *J. Org. Chem.* **52**, 1280-1284.

D'Angelo, J., Ortuno, R. M., Brault, J. F., Ficini, J. F., Riche, C. & Bouchaudy, J. F. (1983). *Tetrahedron Lett.* **24**, 1489-1492.

Micheli, R. A., Hajos, Z. G., Cohen, N., Parrish, D. R., Portland, L. A., Sciamanna, W., Scott, M. A. & Wehrli, P. A. (1975). *J. Org. Chem.* **40**, 675-681.

Neises, B. & Steglich, W. (1978). *Angew. Chem.* **90**, 556-557; *Angew. Chem. Int. Ed. Engl.* **17**, 522-523.

Schomer, U., Sheldrick, W. S. & Wagner, F. (1978). *J. Chem. Soc. Perkin Trans.* 1, pp. 336-340.

Sheldrick, G. M. (1990). *Acta Cryst.* A**46**, 467-473.

Sheldrick, G. M. (1991). *SHELXTL-Plus*. Release 4.1. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.

Sheldrick, G. M. (1992). *SHELXL*-92. *Program for Crystal Structure Refinement*. Univ. of Göttingen, Germany.

Stoe & Cie (1988). *DIF*4. *Diffractometer Control Program*. Stoe & Cie, Darmstadt, Germany.

Stoe & Cie (1988). *REDU*4. *Data Reduction Program*. Stoe & Cie, Darmstadt, Germany.