Acta Cryst. (1991). C47, 1349–1350

Structure of 11-*tert*-Butyldiphenylsilyl Ether of (±)-15-*epi*-Benzodioxaneprostacyclin Methyl Ester

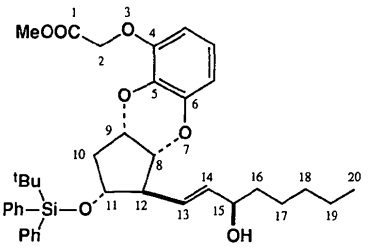
BY MOTOO SHIRO, SHOZO TAKECHI AND SACHIO MORI

Shionogi Research Laboratories, Shionogi & Co. Ltd, Fukushima-ku, Osaka 553, *Japan*

(*Received* 5 *November* 1990; *accepted* 12 *December* 1990)

Abstract. Methyl {12-(*tert*-Butyldiphenylsiloxy)-11-[(3*R**,1*E*)-3-hydroxy-1-octenyl]-2,9-dioxatricyclo-[8.3.0^{1,10}.0^{3,8}]trideca-3(8),4,6-trien-4-yl}oxyacetate, $C_{38}H_{48}O_7Si$, $M_r = 644{\cdot}88$, monoclinic, $P2_1/c$, $a = 9{\cdot}885$ (1), $b = 10{\cdot}278$ (1), $c = 36{\cdot}284$ (1) Å, $\beta = 93{\cdot}18$ (1)°, $V = 3680{\cdot}5$ (6) Å^3, $Z = 4$, $D_x = 1{\cdot}164$ g cm^{-3}, λ(Cu $K\alpha$) = 1·54178 Å, $\mu = 9{\cdot}22$ cm^{-1}, $F(000) = 1384$, $T = 295$ K, $R = 0{\cdot}065$ for 3573 reflections. A hydrogen bond is made between the hydroxyl group at position 15 (prostaglandin numbering) and the O atom at 3, O···O = 2·822 (5) Å.

Experimental. Colorless crystals from Et_2O/*n*-pentane. Crystal dimensions 0·3 × 0·3 × 0·2 mm. Rigaku AFC-5*R* diffractometer, graphite-monochromatized Cu $K\alpha$. Unit-cell parameters from 2θ angles for 15 reflections in the range $38 \le 2\theta \le 40°$.



(I)

Intensities to $2\theta = 110°$ in h 0/10, k 0/10, l −38/38, ω–2θ scans, ω-scan width $(1{\cdot}2 + 0{\cdot}2\tan\theta)°$, three standard reflections monitored every 100 measurements showed no significant change. 4627 unique reflections measured, 3636 intensities observed [$F_o > 3\sigma(F_o)$]. $R_{int} = 0{\cdot}040$. No absorption corrections applied. Structure solved by *MULTAN*84 (Main, Germain & Woolfson, 1984) and refined by block-diagonal least squares to minimize $\sum(w|\Delta F|^2)$. H atoms, except for those of C(16)–C(20), located on a difference density map. Positional for all atoms and anisotropic thermal parameters for non-H atoms refined. Temperature factor of each H atom set equal to B_{eq} of the bonded atom. $w = [\sigma^2(F_o) + 0{\cdot}00401|F_o|^2]^{-1}$, $w = 0$ for 50 reflections with $w^{1/2}|\Delta F| \ge 4$ and 13 very strong ones. Final $R = 0{\cdot}065$, $wR = 0{\cdot}094$ and $S = 1{\cdot}241$. Max. $\Delta/\sigma = 0{\cdot}5$ in the final cycle. Several peaks (0·66–0·40 e Å^{-3} in peak height), found around the atoms C(16)–C(20) in the final difference density map, are far from the positions geometrically estimated for H atoms. We assumed this part to be disordered, but could not confirm it on the map. Atomic scattering factors

Table 1. *Atomic coordinates* (× 10^4) *and equivalent isotropic temperature factors* (Å^2 × 10) *with e.s.d.'s in parentheses*

$B_{eq} = (4/3)\sum_i\sum_j \beta_{ij}\mathbf{a}_i.\mathbf{a}_j$.

	x	y	z	B_{eq}
C(1)	5566 (4)	6600 (4)	7056 (1)	61 (1)
C(2)	5432 (4)	5754 (4)	7382 (1)	62 (1)
O(3)	6642 (3)	5037 (3)	7472 (1)	65 (1)
C(4)	6857 (3)	3896 (4)	7289 (1)	52 (1)
C(5)	7976 (3)	3196 (3)	7433 (1)	47 (1)
C(6)	8261 (4)	2001 (3)	7277 (1)	51 (1)
O(7)	9344 (3)	1263 (3)	7405 (1)	61 (1)
C(8)	10027 (3)	1703 (3)	7742 (1)	50 (1)
C(9)	10075 (4)	3160 (4)	7784 (1)	54 (1)
C(10)	10495 (4)	3355 (4)	8191 (1)	55 (1)
C(11)	10248 (3)	2040 (3)	8379 (1)	47 (1)
C(12)	9418 (3)	1239 (3)	8095 (1)	46 (1)
C(13)	9453 (3)	−186 (4)	8158 (1)	51 (1)
C(14)	8362 (4)	−928 (4)	8174 (1)	59 (1)
C(15)	8356 (5)	−2357 (4)	8242 (1)	67 (1)
C(16)	7623 (8)	−2582 (6)	8619 (1)	123 (3)
C(17)	7501 (9)	−3916 (9)	8722 (1)	150 (3)
C(18)	6750 (12)	−3846 (13)	9140 (3)	223 (6)
C(19)	6120 (15)	−4667 (27)	9187 (5)	318 (11)
C(20)	5603 (11)	−4621 (14)	9577 (2)	210 (6)
O(21)	6506 (3)	6716 (4)	6873 (1)	91 (1)
O(22)	4403 (3)	7250 (3)	6995 (1)	85 (1)
C(23)	4357 (6)	8302 (6)	6728 (2)	106 (2)
C(24)	6057 (4)	3419 (4)	7000 (1)	61 (1)
C(25)	6383 (5)	2224 (4)	6847 (1)	68 (1)
C(26)	7467 (4)	1539 (4)	6984 (1)	62 (1)
O(27)	8716 (2)	3707 (2)	7728 (1)	55 (1)
O(28)	7567 (4)	−2946 (3)	7950 (1)	88 (1)
O(29)	9582 (2)	2210 (2)	8713 (1)	53 (1)
Si(30)	9951·4 (9)	1661·8 (9)	9130·5 (3)	45·5 (2)
C(31)	11019 (4)	2902 (4)	9399 (1)	65 (1)
C(32)	12197 (6)	3338 (6)	9171 (2)	99 (2)
C(33)	10140 (7)	4092 (5)	9482 (2)	103 (2)
C(34)	11618 (6)	2328 (6)	9762 (1)	101 (2)
C(35)	8248 (4)	1399 (4)	9320 (1)	60 (1)
C(36)	8012 (5)	1326 (6)	9691 (1)	92 (2)
C(37)	6734 (6)	1115 (8)	9812 (2)	126 (3)
C(38)	5672 (6)	954 (9)	9561 (2)	135 (3)
C(39)	5857 (5)	988 (9)	9189 (2)	125 (3)
C(40)	7175 (4)	1234 (6)	9074 (1)	88 (2)
C(41)	10850 (4)	58 (4)	9130 (1)	54 (1)
C(42)	10263 (5)	−1015 (4)	9294 (1)	68 (1)
C(43)	10886 (6)	−2220 (4)	9305 (2)	95 (2)
C(44)	12134 (7)	−2365 (5)	9159 (2)	106 (2)
C(45)	12742 (5)	−1340 (6)	9005 (2)	93 (2)
C(46)	12111 (4)	−131 (5)	8985 (1)	74 (1)

0108-2701/91/061349-02$03.00

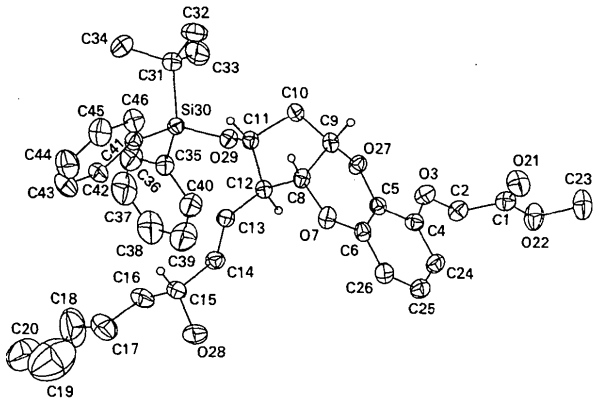


Fig. 1. Perspective view of the title compound, with the atom-numbering system.

were from *International Tables for X-ray Crystallography* (1974, Vol. IV). Calculations on a FACOM M340*R* computer at our laboratories. Final atomic coordinates and equivalent isotropic temperature factors are given in Table 1. Selected bond lengths and angles are listed in Table 2.* A perspective view of the molecule with the atom-numbering system is presented in Fig. 1.

The temperature factors of C(18), C(19) and C(20) are very large and exhibit a large anisotropy. The bond lengths and angles relevant to the atoms, for which the libration corrections have not been applied, are less reliable.

* Lists of anisotropic temperature factors of the non-H atoms, H-atom coordinates, all bond lengths and angles, and structure factors have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53827 (36 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 2. *Selected bond lengths* (Å) *and angles* (°) *with e.s.d.'s in parentheses*

C(1)—C(2)	1·480 (6)	C(1)—O(21)	1·178 (6)
C(1)—O(22)	1·337 (5)	C(2)—O(3)	1·427 (5)
O(3)—C(4)	1·370 (5)	C(4)—C(5)	1·396 (5)
C(4)—C(24)	1·369 (6)	C(5)—C(6)	1·388 (5)
C(5)—O(27)	1·367 (5)	C(6)—O(7)	1·372 (5)
C(6)—C(26)	1·371 (6)	O(7)—C(8)	1·437 (5)
C(8)—C(9)	1·506 (5)	C(8)—C(12)	1·522 (5)
C(9)—C(10)	1·525 (6)	C(9)—O(27)	1·460 (5)
C(10)—C(11)	1·540 (5)	C(11)—C(12)	1·523 (5)
C(11)—O(29)	1·422 (5)	C(12)—C(13)	1·482 (5)
O(22)—C(23)	1·451 (8)	C(24)—C(25)	1·393 (6)
C(25)—C(26)	1·353 (6)	O(29)—Si(30)	1·638 (4)
Si(30)—C(31)	1·890 (4)	Si(30)—C(35)	1·873 (4)
Si(30)—C(41)	1·873 (4)		

C(2)—C(1)—O(21)	128·2 (4)	C(2)—C(1)—O(22)	108·2 (4)
O(21)—C(1)—O(22)	123·6 (4)	C(1)—C(2)—O(3)	112·0 (3)
C(2)—O(3)—C(4)	118·8 (3)	O(3)—C(4)—C(5)	113·6 (4)
O(3)—C(4)—C(24)	125·4 (4)	C(5)—C(4)—C(24)	120·9 (4)
C(4)—C(5)—C(6)	118·5 (3)	C(4)—C(5)—O(27)	118·2 (3)
C(6)—C(5)—O(27)	123·2 (3)	C(5)—C(6)—O(7)	121·6 (4)
C(5)—C(6)—C(26)	120·2 (4)	O(7)—C(6)—C(26)	118·3 (4)
C(6)—O(7)—C(8)	115·8 (3)	O(7)—C(8)—C(9)	114·2 (3)
O(7)—C(8)—C(12)	115·4 (3)	C(9)—C(8)—C(12)	103·8 (3)
C(8)—C(9)—C(10)	103·5 (3)	C(8)—C(9)—O(27)	110·2 (3)
C(10)—C(9)—O(27)	106·4 (3)	C(9)—C(10)—C(11)	105·8 (3)
C(10)—C(11)—C(12)	105·5 (3)	C(10)—C(11)—O(29)	111·2 (3)
C(12)—C(11)—O(29)	112·7 (3)	C(8)—C(12)—C(11)	100·2 (3)
C(8)—C(12)—C(13)	115·6 (3)	C(11)—C(12)—C(13)	115·0 (3)
C(1)—O(22)—C(23)	118·5 (4)	C(4)—C(24)—C(25)	119·0 (4)
C(24)—C(25)—C(26)	120·5 (4)	C(6)—C(26)—C(25)	120·8 (4)
C(5)—O(27)—C(9)	114·2 (3)	C(11)—O(29)—Si(30)	131·0 (3)
O(29)—Si(30)—C(31)	109·5 (2)	O(29)—Si(30)—C(35)	103·3 (2)
O(29)—Si(30)—C(41)	112·5 (2)	C(31)—Si(30)—C(35)	113·3 (2)
C(31)—Si(30)—C(41)	110·0 (2)	C(35)—Si(30)—C(41)	108·0 (2)

Related literature. The title compound has been discussed by Mori & Takechi (1990).

References

MAIN, P., GERMAIN, G. & WOOLFSON, M. M. (1984). *MULTAN*84. *A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data.* Univs. of York, England, and Louvain, Belgium.

MORI, S. & TAKECHI, S. (1990). *Heterocycles*, **31**, 1189–1193.

Acta Cryst. (1991). C**47**, 1350–1352

Structure of 2-Methyl-2-(5,5-dimethyl-3-hydantoinyl)propanamide

BY KABUIKA N. BUTAMINA* AND LEWIS J. RADONOVICH†

Department of Chemistry, University of North Dakota, Grand Forks, ND 58202, *USA*

(*Received* 26 *June* 1990; *accepted* 7 *December* 1990)

Abstract. $C_9H_{15}N_3O_3$ (1), $M_r = 213{\cdot}24$, orthorhombic, *Pbca*, $a = 6{\cdot}323$ (3), $b = 16{\cdot}25$ (1), $c = 20{\cdot}16$ (1) Å, $V = 2071{\cdot}4$ Å^3, $Z = 8$, $D_m = 1{\cdot}362$ (2), $D_x = 1{\cdot}367$ g cm^{-3}, λ(Mo $K\alpha$) = 0·7107 Å, $\mu = 0{\cdot}973$ cm^{-1}, $F(000) = 912$, $T = 295$ K, $R(F) = 0{\cdot}043$ and $wR(F) = 0{\cdot}048$ for 2301 intensity data with $F > 3\sigma(F)$. The observed bond parameters are similar to those in 5,5-dimethylhydantoin except for an increase of 0·03 Å in the C—N bond lengths at the substituted ring N atom.

* Current address: Antioch College, Yellow Springs, Ohio 45387, USA.

† Author to whom correspondence should be addressed.

0108-2701/91/061350-03$03.00