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(22*R*)-3β-*tert*-Butyldimethylsilyloxy-22-methylchola-5,23-diene

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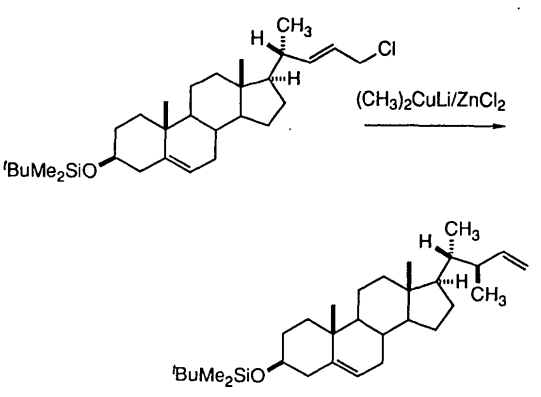
Abstract

The structure determination of the title compound, $C_{31}H_{54}OSi$, based on single-crystal X-ray diffraction, shows that the absolute configuration at C22 is *R*.

Comment

The diastereoselectivity of nucleophilic additions to olefins bearing a chiral center on an adjacent C atom provides useful information for studies on the factors that control stereoselectivities of organic reactions (Nakamura, Sekiya, Arai & Aoki, 1989). In relation to studies on the diastereoselectivity of the S_N2'-allylation reaction of organocopper reagents, the reaction shown in the scheme below was examined and found to give the methylated product (1) as a single diastereomer (Arai, Kawasuji & Nakamura, 1993). Since no spectroscopic techniques were effective in determining the stereochemistry of this steroidal product, the X-ray crystal structure analysis was performed. The perspective view obtained (Fig. 1) reveals the absolute configuration at C22 to be *R*.



(1)

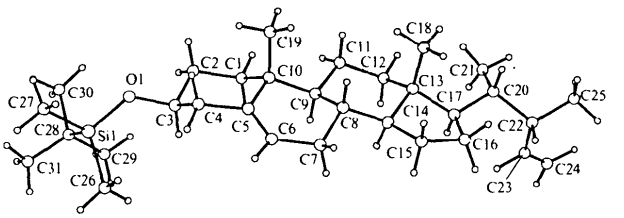


Fig. 1. Molecular structure of (22*R*)-3β-*tert*-butyldimethylsilyloxy-22-methylchola-5,23-diene with atomic numbering.

Experimental

Crystal data

$C_{31}H_{54}OSi$	Cu $K\alpha$ radiation
M_r = 470.85	λ = 1.5418 Å
Orthorhombic	Cell parameters from 20 reflections
$P2_12_12_1$	θ = 45–55°
a = 12.021 (1) Å	μ = 0.818 mm^{-1}
b = 40.218 (5) Å	T = 296 K
c = 6.171 (1) Å	Plate
V = 2984 (1) Å^3	0.5 × 0.45 × 0.25 mm
Z = 4	Colorless
D_x = 1.05 Mg m^{-3}	

Data collection

Rigaku AFC-4 diffractometer	R_{int} = 0.0348
$\omega/2\theta$ scans	θ_{max} = 62.5°
Absorption correction: none	h = 0 → 13
2826 measured reflections	k = 0 → 46
2815 independent reflections	l = 0 → 7
2491 observed reflections [$F > 3\sigma(F)$]	3 standard reflections monitored every 50 reflections
	intensity variation: 2%

Refinement

Refinement on F	$(\Delta/\sigma)_{max}$ = 0.3
R = 0.068	$\Delta\rho_{max}$ = 0.171 e Å^{-3}
wR = 0.108	$\Delta\rho_{min}$ = −0.316 e Å^{-3}
S = 0.723	Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)
2491 reflections	
376 parameters	
Only H-atom U's refined	
$w = 1/[\sigma^2(F) + 0.020545F^2]$	

Program(s) used to refine structure: *SHELX76* (Sheldrick, 1976). Molecular graphics: *PLUTO* (Motherwell & Clegg, 1978).

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}
Si1	0.08219 (11)	0.44582 (3)	0.4803 (3)	0.0676
O1	0.0684 (3)	0.41670 (7)	0.2955 (6)	0.0758
C1	−0.0336 (3)	0.32938 (9)	0.2162 (10)	0.0645
C2	−0.0299 (4)	0.36746 (10)	0.1921 (11)	0.0726
C3	0.0634 (4)	0.38161 (9)	0.3288 (9)	0.0661
C4	0.1742 (3)	0.36631 (10)	0.2569 (10)	0.0683
C5	0.1716 (3)	0.32842 (10)	0.2673 (8)	0.0580
C6	0.2516 (3)	0.31167 (10)	0.3669 (9)	0.0646
C7	0.2584 (3)	0.27486 (9)	0.3801 (9)	0.0634
C8	0.1790 (3)	0.25693 (9)	0.2247 (8)	0.0557
C9	0.0656 (3)	0.27450 (9)	0.2229 (7)	0.0506
C10	0.0736 (3)	0.31148 (10)	0.1489 (7)	0.0515
C11	−0.0210 (3)	0.25421 (10)	0.0955 (11)	0.0680
C12	−0.0308 (3)	0.21760 (10)	0.1655 (9)	0.0614
C13	0.0834 (3)	0.20003 (10)	0.1554 (7)	0.0529
C14	0.1629 (3)	0.22074 (9)	0.2952 (8)	0.0542
C15	0.2648 (3)	0.19915 (10)	0.3282 (10)	0.0680
C16	0.2181 (3)	0.16360 (10)	0.3314 (11)	0.0715
C17	0.0926 (3)	0.16619 (9)	0.2725 (9)	0.0589
C18	0.1225 (5)	0.19723 (12)	−0.0785 (10)	0.0743
C19	0.0929 (5)	0.31457 (12)	−0.0943 (9)	0.0736
C20	0.0493 (4)	0.13462 (10)	0.1605 (11)	0.0706
C21	−0.0767 (4)	0.13683 (11)	0.1116 (12)	0.0854
C22	0.0718 (5)	0.10262 (11)	0.3011 (13)	0.0874
C23	0.0060 (6)	0.10092 (13)	0.5023 (14)	0.1041
C24	−0.0533 (8)	0.07745 (17)	0.5850 (17)	0.1290
C25	0.0628 (7)	0.07117 (15)	0.1660 (19)	0.1186
C26	0.1675 (5)	0.43148 (17)	0.7151 (14)	0.1014
C27	0.1545 (7)	0.48062 (15)	0.3390 (14)	0.1073
C28	−0.0576 (5)	0.45955 (13)	0.5827 (11)	0.0860
C29	−0.1120 (7)	0.43049 (19)	0.708 (2)	0.1261
C30	−0.1311 (8)	0.4687 (3)	0.3865 (19)	0.1379
C31	−0.0458 (9)	0.4902 (2)	0.7324 (18)	0.1385

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

Si1—O1	1.643 (4)	C10—C19	1.523 (7)
Si1—C26	1.867 (8)	C12—C11	1.539 (6)
Si1—C27	1.864 (7)	C13—C12	1.546 (5)
Si1—C28	1.878 (6)	C13—C17	1.545 (6)
O1—C3	1.427 (5)	C13—C18	1.522 (7)
C1—C2	1.539 (5)	C14—C13	1.533 (6)
C1—C10	1.533 (6)	C14—C15	1.515 (5)
C2—C3	1.514 (7)	C16—C15	1.536 (6)
C3—C4	1.533 (6)	C17—C16	1.556 (6)
C4—C5	1.526 (6)	C17—C20	1.537 (6)
C5—C6	1.326 (6)	C20—C21	1.547 (7)
C5—C10	1.545 (6)	C20—C22	1.576 (8)
C6—C7	1.485 (5)	C22—C23	1.473 (11)
C7—C8	1.534 (6)	C22—C25	1.519 (10)
C8—C9	1.535 (5)	C23—C24	1.288 (11)
C8—C14	1.531 (5)	C28—C29	1.548 (11)
C9—C11	1.539 (6)	C28—C30	1.543 (13)
C10—C9	1.559 (5)	C28—C31	1.546 (11)
O1—Si1—C26	112.0 (3)	C9—C11—C12	114.5 (4)
O1—Si1—C27	104.9 (3)	C13—C12—C11	111.0 (3)
O1—Si1—C28	110.6 (2)	C14—C13—C12	106.4 (3)
C26—Si1—C27	109.8 (3)	C14—C13—C17	99.8 (3)
C26—Si1—C28	108.7 (3)	C14—C13—C18	112.4 (4)
C27—Si1—C28	110.7 (3)	C12—C13—C17	116.6 (3)
Si1—O1—C3	127.5 (3)	C12—C13—C18	110.3 (4)
C2—C1—C10	114.6 (4)	C17—C13—C18	110.9 (4)
C1—C2—C3	110.0 (4)	C8—C14—C13	115.8 (4)
O1—C3—C2	108.8 (4)	C8—C14—C15	118.8 (3)
O1—C3—C4	108.6 (3)	C13—C14—C15	105.5 (3)
C2—C3—C4	109.3 (4)	C14—C15—C16	103.9 (3)
C3—C4—C5	111.8 (3)	C17—C16—C15	106.8 (3)
C4—C5—C10	115.9 (4)	C13—C17—C16	103.7 (3)
C4—C5—C6	120.8 (4)	C13—C17—C20	119.6 (4)
C10—C5—C6	123.3 (4)	C16—C17—C20	112.2 (3)
C5—C6—C7	124.9 (4)	C17—C20—C21	111.8 (4)
C6—C7—C8	113.6 (4)	C17—C20—C22	111.7 (5)
C7—C8—C9	109.9 (3)	C21—C20—C22	108.8 (4)
C7—C8—C14	110.4 (4)	C20—C22—C25	111.4 (6)
C9—C8—C14	109.1 (3)	C20—C22—C23	114.2 (4)
C10—C9—C8	112.8 (3)	C25—C22—C23	112.7 (5)
C10—C9—C11	113.4 (3)	C22—C23—C24	131.7 (7)
C8—C9—C11	111.1 (3)	Si1—C28—C29	108.9 (4)
C1—C10—C5	107.8 (3)	Si1—C28—C30	108.6 (5)
C1—C10—C9	108.5 (3)	Si1—C28—C31	110.7 (5)
C1—C10—C19	110.9 (4)	C29—C28—C30	109.4 (6)
C5—C10—C9	109.2 (3)	C29—C28—C31	109.9 (7)
C5—C10—C19	108.3 (4)	C30—C28—C31	109.4 (6)
C9—C10—C19	112.1 (4)		

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates, torsion angles and complete geometry have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71544 (27 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: AS1074]

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2-(4-Pentanoyloxyphenyl)-6-pentanoyloxybenzoxazole

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Abstract

In 2-[4-(pentanoyloxy)phenyl]benzoxazol-6-yl pentanoate, $C_{23}H_{25}NO_5$, the phenyl and benzoxazole rings are not strictly coplanar, the dihedral angle between the corresponding least-squares planes being 10.8 (6)°. A torsion angle of the *gauche* type is present in the aliphatic tail bonded to the benzo ring. Molecules of the title compound are packed in layers piled up along **a**. The lateral packing of the molecules shows face-to-face and face-to-edge contacts among the aromatic rings.