Crystal structure of (1*S*,5*R*,6*S*,7*R*)-7-(*tert*-butyldiphenylsilyloxy)-6-vinyl-2-oxabicyclo[3.3.0]octan-3-one, C₂₅H₃₀O₃Si

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Abstract

C₂₅H₃₀O₃Si, monoclinic, *P*12₁1 (No. 4), *a* = 9.750(1) Å, *b* = 7.691(1) Å, *c* = 16.055(2) Å, β = 102.99(1)°, *V* = 1173.1 Å³, *Z* = 2, *R*_{gt}(*F*) = 0.045, *wR*_{ref}(*F*²) = 0.125, *T* = 293 K.

Source of material

Enantiomerically pure (1*S*,5*R*,6*S*,7*R*)-7-(*tert*-butyldiphenylsilyloxy)-6-vinyl-2-oxabicyclo[3.3.0]octan-3-one was obtained by an allylmethylation/intramolecular [2+2] cycloaddition/Baeyer-Villiger oxidation sequence and was isolated as the minor diastereoisomer [1]. Crystals suitable for X-ray diffraction analysis were grown by slow crystallization from a ethyl acetate hexane solution.

Experimental details

All the hydrogen atoms were refined with a common isotropic temperature factor (*U*_{iso} = 0.116(2) Å²).

Discussion

The results of X-ray diffraction analysis unambiguously confirm the absolute and relative configuration. As shown on the figure, the absolute configuration is 1*S*,5*R*,6*S*,7*R*. The conformation of the bicyclic skeleton is different from that observed for the major isomer I [2]. The lactone is now a flat envelop Δ*C*s (C4) = 0.5° [3] while the cyclopentane is an half chair with the twist on the C(6)–C(7) bond Δ*C*2 (C1) = 2.5°. The bulky silyloxy substituent is in pseudo-axial position.

Table 1. Data collection and handling.

Crystal:	colourless parallelepiped, size 0.26 × 0.30 × 0.36 mm
Wavelength:	Cu K _α radiation (1.54178 Å)
μ:	10.48 cm ^{−1}
Diffractometer, scan mode:	Huber, θ/2θ
2θ _{max} :	134.98°
<i>N</i> (<i>hkl</i>) _{measured} , <i>N</i> (<i>hkl</i>) _{unique} :	4238, 4018
Criterion for <i>I</i> _{obs} , <i>N</i> (<i>hkl</i>) _{gt} :	<i>I</i> _{obs} > 2 σ(<i>I</i> _{obs}), 3932
<i>N</i> (<i>param</i>) _{refined} :	297
Programs:	SHELXS-86 [4], SHELXL-93 [5], PLATON [6]

Table 2. Atomic coordinates and displacement parameters (in Å²).

Atom	Site	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso}
H(15A)	2a	0.1853(5)	0.3278(4)	0.2870(3)	0.116(2)
H(15B)	2a	0.3468(5)	0.3059(4)	0.3260(3)	0.116
H(15C)	2a	0.2694(5)	0.1782(4)	0.2546(3)	0.116
H(16A)	2a	0.5208(4)	0.3829(5)	0.2379(3)	0.116
H(16B)	2a	0.4661(4)	0.4355(5)	0.1419(3)	0.116
H(16C)	2a	0.4445(4)	0.2452(5)	0.1714(3)	0.116
H(17A)	2a	0.1008(5)	0.4234(6)	0.1377(3)	0.116
H(17B)	2a	0.1862(5)	0.2699(6)	0.1096(3)	0.116
H(17C)	2a	0.2077(5)	0.4603(6)	0.0800(3)	0.116
H(19)	2a	0.4720(3)	0.5028(6)	0.3982(2)	0.116
H(20)	2a	0.6447(4)	0.6038(9)	0.5125(2)	0.116
H(21)	2a	0.7287(3)	0.8788(8)	0.5136(2)	0.116
H(22)	2a	0.6430(4)	1.0592(5)	0.4019(3)	0.116
H(23)	2a	0.4711(3)	0.9660(4)	0.2880(2)	0.116
H(25)	2a	0.5259(3)	0.7887(5)	0.1565(2)	0.116
H(26)	2a	0.5358(4)	0.9316(6)	0.0325(2)	0.116
H(27)	2a	0.3335(4)	1.0290(5)	−0.0579(2)	0.116
H(28)	2a	0.1204(3)	0.9813(5)	−0.0239(2)	0.116
H(29)	2a	0.1084(3)	0.8317(4)	0.0979(2)	0.116
H(1)	2a	−0.063(4)	0.729(6)	0.456(3)	0.116
H(4A)	2a	−0.107(5)	0.527(7)	0.242(3)	0.116
H(4B)	2a	−0.243(5)	0.547(7)	0.281(3)	0.116
H(5)	2a	−0.189(5)	0.764(6)	0.324(3)	0.116
H(6)	2a	−0.062(4)	0.954(6)	0.298(3)	0.116
H(7)	2a	0.165(4)	0.934(6)	0.294(3)	0.116
H(8A)	2a	0.219(4)	0.708(6)	0.424(3)	0.116
H(8B)	2a	0.127(4)	0.894(6)	0.432(3)	0.116
H(10)	2a	−0.102(5)	0.733(6)	0.157(3)	0.116
H(11A)	2a	−0.194(4)	0.998(7)	0.076(3)	0.116
H(11B)	2a	−0.176(6)	1.063(7)	0.174(3)	0.116

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Table 3. Atomic coordinates and displacement parameters (in Å²).

Atom	Site	x	y	z	U ₁₁	U ₂₂	U ₃₃	U ₁₂	U ₁₃	U ₂₃
C(1)	2a	-0.0067(3)	0.6965(4)	0.4080(2)	0.077(1)	0.093(2)	0.064(1)	0.006(1)	0.031(1)	-0.009(1)
O(2)	2a	0.0217(3)	0.5130(4)	0.4214(2)	0.117(2)	0.098(2)	0.083(1)	0.010(1)	0.017(1)	0.012(1)
C(3)	2a	-0.0509(4)	0.4207(5)	0.3584(2)	0.114(3)	0.095(2)	0.098(2)	-0.014(2)	0.052(2)	0.000(2)
C(4)	2a	-0.1303(4)	0.5296(6)	0.2889(2)	0.075(2)	0.121(3)	0.090(2)	-0.037(2)	0.024(2)	-0.019(2)
C(5)	2a	-0.1163(3)	0.7153(4)	0.3226(2)	0.053(1)	0.104(2)	0.072(1)	0.010(1)	0.027(1)	0.002(1)
C(6)	2a	-0.0533(3)	0.8522(4)	0.2733(2)	0.062(1)	0.077(2)	0.079(2)	0.016(1)	0.023(1)	0.000(1)
C(7)	2a	0.1061(2)	0.8258(3)	0.3026(2)	0.060(1)	0.059(1)	0.070(1)	0.001(1)	0.019(1)	-0.008(1)
C(8)	2a	0.1237(3)	0.7919(5)	0.3982(2)	0.071(2)	0.100(2)	0.066(1)	-0.000(1)	0.016(1)	-0.024(2)
O(9)	2a	-0.0447(6)	0.2639(5)	0.3603(3)	0.256(5)	0.083(2)	0.182(4)	-0.015(2)	0.081(4)	0.003(2)
C(10)	2a	-0.1126(3)	0.8561(7)	0.1783(2)	0.063(2)	0.153(3)	0.084(2)	0.021(2)	0.018(1)	0.032(2)
C(11)	2a	-0.1653(7)	0.997(1)	0.1375(5)	0.124(4)	0.26(1)	0.131(4)	0.078(5)	0.033(3)	0.091(5)
O(12)	2a	0.1495(1)	0.6794(2)	0.26051(8)	0.0482(6)	0.0619(8)	0.0560(7)	-0.0003(6)	0.0158(5)	-0.0020(6)
Si(13)	2a	0.30394(5)	0.65311(7)	0.23655(3)	0.0463(3)	0.0616(3)	0.0459(3)	0.0035(2)	0.0134(2)	0.0033(2)
C(14)	2a	0.3068(3)	0.4178(3)	0.2060(2)	0.078(2)	0.062(1)	0.078(2)	0.009(1)	0.028(1)	0.001(1)
C(15)	2a	0.2740(5)	0.2960(4)	0.2748(3)	0.130(3)	0.067(2)	0.144(3)	0.011(2)	0.071(3)	0.019(2)
C(16)	2a	0.4475(4)	0.3655(5)	0.1876(3)	0.117(3)	0.088(2)	0.149(3)	0.018(2)	0.079(3)	-0.011(2)
C(17)	2a	0.1894(5)	0.3903(6)	0.1258(3)	0.159(4)	0.081(2)	0.133(3)	-0.001(2)	-0.013(3)	-0.042(2)
C(18)	2a	0.4502(2)	0.7232(4)	0.3287(1)	0.0459(9)	0.093(2)	0.051(1)	0.005(1)	0.0144(8)	-0.000(1)
C(19)	2a	0.5048(3)	0.6163(6)	0.3976(2)	0.074(2)	0.140(3)	0.066(1)	-0.013(2)	0.005(1)	0.029(2)
C(20)	2a	0.6096(4)	0.6775(9)	0.4668(2)	0.083(2)	0.194(5)	0.056(1)	0.008(3)	0.004(1)	0.016(2)
C(21)	2a	0.6595(3)	0.8400(8)	0.4677(2)	0.062(2)	0.170(4)	0.076(2)	0.006(2)	0.012(1)	-0.038(2)
C(22)	2a	0.6086(4)	0.9464(5)	0.4016(3)	0.088(2)	0.108(2)	0.105(2)	0.006(2)	0.007(2)	-0.049(2)
C(23)	2a	0.5048(3)	0.8895(4)	0.3328(2)	0.071(2)	0.082(2)	0.084(2)	0.010(1)	0.005(1)	-0.024(1)
C(24)	2a	0.3150(2)	0.7898(3)	0.1417(1)	0.061(1)	0.068(1)	0.049(1)	0.0013(9)	0.0180(9)	0.0032(9)
C(25)	2a	0.4427(3)	0.8249(5)	0.1200(2)	0.064(1)	0.102(2)	0.069(1)	0.005(1)	0.028(1)	0.012(1)
C(26)	2a	0.4491(4)	0.9122(6)	0.0458(2)	0.087(2)	0.128(3)	0.087(2)	-0.004(2)	0.047(2)	0.019(2)
C(27)	2a	0.3290(4)	0.9701(5)	-0.0080(2)	0.109(2)	0.120(3)	0.062(1)	0.002(2)	0.033(2)	0.026(2)
C(28)	2a	0.2026(3)	0.9409(5)	0.0121(2)	0.086(2)	0.113(2)	0.058(1)	0.002(2)	0.008(1)	0.019(1)
C(29)	2a	0.1960(3)	0.8513(4)	0.0856(2)	0.064(1)	0.094(2)	0.058(1)	-0.002(1)	0.015(1)	0.010(1)

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