Crystal structure of (1*R*,2*S*,3*R*,5*S*)-2-(dimethylphenylsilyl)-6-oxobicyclo[3.2.0]hept-3-yl acetate, $C_{17}H_{22}O_3Si$

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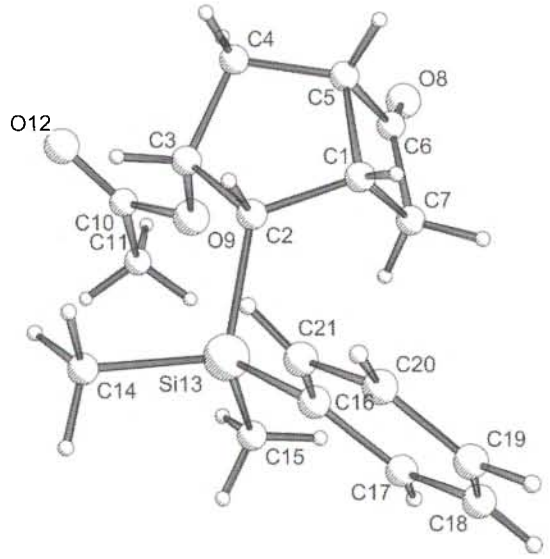

Abstract

$C_{17}H_{22}O_3Si$, orthorhombic, $P2_12_12_1$ (No. 19), a = 11.221(1) Å, b = 11.261(1) Å, c = 13.165(2) Å, V = 1663.5 Å^3, Z = 4, $R_{gt}(F)$ = 0.035, $wR(F^2)$ = 0.105, T = 293 K.

Source of material

Enantiopure (1*R*,2*S*,3*R*,5*S*)-2-(dimethylhenylsilyl)-6-oxobicyclo[3.2.0]hept-3-yl acetate has been synthesized by an allylmetallation/intramolecular [2+2] cycloaddition sequence [1].

Discussion

The configuration at the four stereogenic centers is 1*R*,2*S*,3R,5*S*. The 5-membered ring adopted an envelope conformation $\Delta C_{S(C3)}$ = 3.2° [2]. The cyclobutanone is planar with endocyclic torsion angles less than 2°.

Table 1. Data collection and handling.

Crystal:	colourless, parallelipiped, size 0.12 × 0.16 × 0.24 mm
Wavelength:	Cu K_α radiation (1.54178 Å)
μ:	13.04 cm^{-1}
Diffractometer, scan mode:	Huber, θ/2θ
$2\theta_{max}$:	135.08°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	3303, 3000
Criterion for F_{obs}, $N(hkl)_{gt}$:	$F_{obs} > 2\,\sigma(F_{obs})$, 2892
$N(param)_{refined}$:	249
Programs:	SHELXS-86 [3], SHELXL-93 [4], PLUTO [5]

Table 2. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	x	y	z	U_{iso}
H(11A)	4*a*	0.2144(3)	0.4490(3)	0.4678(2)	0.093(2)
H(11B)	4*a*	0.1722(3)	0.5720(3)	0.5104(2)	0.093(2)
H(11C)	4*a*	0.0840(3)	0.4929(3)	0.4476(2)	0.093(2)
H(1)	4*a*	0.349(3)	0.096(3)	0.758(2)	0.093(2)
H(2)	4*a*	0.155(3)	0.103(3)	0.743(2)	0.093(2)
H(3)	4*a*	0.060(3)	0.270(3)	0.704(3)	0.093(2)
H(4A)	4*a*	0.154(3)	0.305(3)	0.851(3)	0.093(2)
H(4B)	4*a*	0.204(3)	0.411(3)	0.794(3)	0.093(2)
H(5)	4*a*	0.354(3)	0.260(3)	0.865(3)	0.093(2)
H(7A)	4*a*	0.496(3)	0.166(3)	0.663(2)	0.093(2)
H(7B)	4*a*	0.401(3)	0.227(3)	0.593(3)	0.093(2)
H(14A)	4*a*	-0.041(3)	0.143(3)	0.559(2)	0.093(2)
H(14B)	4*a*	-0.025(3)	0.033(3)	0.631(3)	0.093(2)
H(14C)	4*a*	-0.019(3)	0.001(3)	0.499(3)	0.093(2)
H(15A)	4*a*	0.314(3)	0.117(3)	0.452(2)	0.093(2)
H(15B)	4*a*	0.196(3)	0.092(3)	0.393(3)	0.093(2)
H(15C)	4*a*	0.209(3)	0.192(3)	0.441(3)	0.093(2)
H(17)	4*a*	0.367(3)	-0.092(3)	0.488(3)	0.093(2)
H(18)	4*a*	0.443(3)	-0.265(3)	0.530(3)	0.093(2)
H(19)	4*a*	0.371(3)	-0.388(3)	0.658(3)	0.093(2)
H(20)	4*a*	0.195(3)	-0.321(3)	0.749(2)	0.093(2)
H(21)	4*a*	0.131(3)	-0.131(3)	0.711(3)	0.093(2)

Table 3. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C(1)	4*a*	0.3276(2)	0.1598(2)	0.7304(2)	0.064(1)	0.0401(9)	0.053(1)	0.0091(9)	-0.005(1)	0.0016(8)
C(2)	4*a*	0.1993(2)	0.1460(2)	0.6920(2)	0.059(1)	0.0412(9)	0.0460(9)	-0.0019(8)	0.0049(8)	0.0041(7)
C(3)	4*a*	0.1436(2)	0.2688(2)	0.7026(2)	0.057(1)	0.050(1)	0.050(1)	0.0056(8)	0.0081(8)	0.0053(8)
C(4)	4*a*	0.2006(2)	0.3231(2)	0.7966(2)	0.086(2)	0.054(1)	0.050(1)	0.013(1)	0.007(1)	-0.0031(9)
C(5)	4*a*	0.3259(2)	0.2722(2)	0.8002(2)	0.081(1)	0.049(1)	0.048(1)	0.006(1)	-0.011(1)	-0.0024(8)

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Table 3. Continued.

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C(6)	4*a*	0.4134(2)	0.3303(2)	0.7280(2)	0.065(1)	0.054(1)	0.076(2)	0.000(1)	–0.012(1)	0.002(1)
C(7)	4*a*	0.4203(2)	0.2206(2)	0.6608(2)	0.059(1)	0.067(1)	0.067(1)	0.000(1)	0.000(1)	–0.007(1)
O(8)	4*a*	0.4575(2)	0.4260(2)	0.7244(2)	0.103(2)	0.055(1)	0.120(2)	–0.014(1)	–0.012(1)	0.003(1)
O(9)	4*a*	0.1751(1)	0.3378(1)	0.6125(1)	0.0561(7)	0.0491(7)	0.0551(7)	0.0063(6)	0.0023(6)	0.0092(6)
C(10)	4*a*	0.1078(2)	0.4330(2)	0.5912(2)	0.055(1)	0.051(1)	0.073(1)	0.0014(9)	–0.0097(9)	0.010(1)
C(11)	4*a*	0.1482(3)	0.4920(3)	0.4957(2)	0.085(2)	0.067(1)	0.083(2)	–0.009(1)	–0.014(1)	0.023(1)
O(12)	4*a*	0.0271(2)	0.4651(2)	0.6439(2)	0.075(1)	0.078(1)	0.111(2)	0.027(1)	0.012(1)	0.024(1)
Si(13)	4*a*	0.16818(5)	0.06017(5)	0.57140(4)	0.0589(3)	0.0454(3)	0.0515(3)	–0.0073(2)	–0.0002(2)	0.0021(2)
C(14)	4*a*	0.0019(2)	0.0527(3)	0.5606(3)	0.063(1)	0.084(2)	0.095(2)	–0.010(1)	–0.013(1)	0.006(2)
C(15)	4*a*	0.2302(3)	0.1234(3)	0.4518(2)	0.101(2)	0.067(1)	0.052(1)	–0.011(1)	0.006(1)	0.003(1)
C(16)	4*a*	0.2324(2)	–0.0918(2)	0.5943(2)	0.069(1)	0.045(1)	0.057(1)	–0.0067(9)	–0.0068(9)	–0.0081(8)
C(17)	4*a*	0.3302(2)	–0.1337(2)	0.5410(2)	0.073(1)	0.063(1)	0.072(1)	–0.002(1)	–0.009(1)	–0.014(1)
C(18)	4*a*	0.3812(3)	–0.2426(3)	0.5644(3)	0.090(2)	0.074(2)	0.095(2)	0.020(1)	–0.028(2)	–0.031(2)
C(19)	4*a*	0.3347(4)	–0.3111(2)	0.6412(3)	0.139(3)	0.053(1)	0.096(2)	0.023(2)	–0.047(2)	–0.020(2)
C(20)	4*a*	0.2401(4)	–0.2711(2)	0.6951(3)	0.161(3)	0.053(2)	0.083(2)	–0.005(2)	–0.016(2)	0.007(1)
C(21)	4*a*	0.1875(3)	–0.1626(2)	0.6717(2)	0.110(2)	0.052(1)	0.075(2)	–0.006(1)	0.006(2)	0.001(1)

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References

1. Adam, J. M.; Ghosez, L.; Houk, K. N.: A Convergent Stategy for the Asymmetric Synthesis of Enantiomerically Pure Bicyclic Compounds Using a Silicon-directed Cycloaddition Reaction: the Synthesis of Enantiomerically Pure Bicyclo[3.2.0]hept-2-en-6-one. Angew. Chem. Int. Ed. 1999, in print.
2. Duax, W. L.; Weeks, C. M.; Rohrer, D. C.: Quantitative Analysis of Ring Conformation. In: *Topics in stereochemistry*, vol 9, p. 271. J. Wiley New York 1976.
3. Sheldrick, G. M.: Phase Annealing in SHELX-90: Direct Methods for Larger Structures. Acta Crystallogr. A**46** (1990) 476.
4. Sheldrick, G. M.: SHELXL-93 Program for the Refinement of Crystal Structures. University of Göttingen, Germany 1993.
5. Motherwell, W. D. S.; Clegg W.: PLUTO. Program for Plotting Molecular and Crystal Structures, University of Cambridge, England 1978.