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Crystal structure of 1,8-dimethyl-7α-methoxy-10α-(triisopropylsilyloxy)-(2β)-12-oxatricyclo[6.3.1.0^{2,7}]dodecane-3β,4α-diol, $[(C_3H_7)_3SiO](CH_3)_2(OH)_2(OCH_3)(C_{11}H_{12}O)$

J. Wittenberg[I], H. M. R. Hoffmann[I] and R. Wartchow*,[II]

[I] Universität Hannover, Institut für Organische Chemie, Schneiderberg 1B, D-30167 Hannover, Germany
[II] Universität Hannover, Institut für Anorganische Chemie, Callinstr. 9, D-30167 Hannover, Germany

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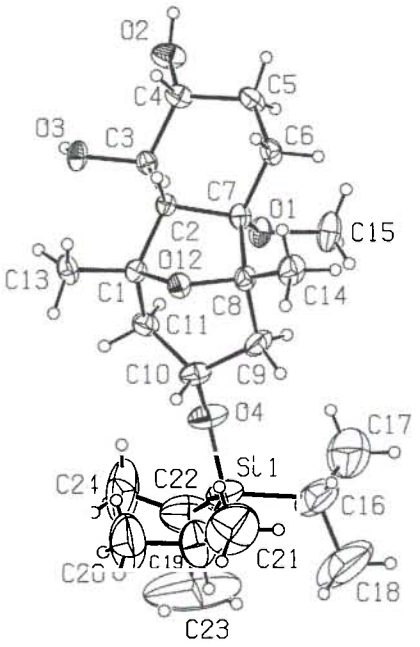

Abstract

$C_{23}H_{44}O_5Si$, monoclinic, $P12_1/a1$ (No. 14), a = 13.117(2) Å, b = 8.736(1) Å, c = 22.362(3) Å, β = 101.36(2)°, V = 2512.3 Å^3, Z = 4, $R_{gt}(F)$ = 0.060, $wR_{all}(F^2)$ = 0.115, T = 300 K.

Source of material

The preparation is described in [1].

Discussion

Because the crystal was turned about only one axis, not all reflections could be measured within the given $2\theta_{max}$: the completeness of the unique data set is 93%. H1 and H2 were refined with distance restraints assuming hydrogen bonds to O atoms of neighbouring molecules, H3 to H44 as riding H's.

The title compound is an intermediate *en route* to dictyoxetane, a marine natural product with an unusual dioxatricyclic skeleton. Cf. also [2].

* Correspondence author
(e-mail: Wartchow@mbox.aca.uni-hannover.de)

Table 1. Data collection and handling.

Crystal:	colourless plate ‖ (001), size 0.08 × 0.41 × 0.81 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	1.22 cm^{-1}
Diffractometer, scan mode:	Stoe IPDS, 180 exposures, Δφ = 1.1°
$2\theta_{max}$:	48°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	15101, 3670
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 1502
$N(param)_{refined}$:	270
Programs:	SHELXS-86 [3], SHELXL-93 [4], PLATON [5]

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

Atom	Site	x	y	z	U_{iso}
H(1)	4e	0.189(4)	0.661(4)	0.483(1)	0.10(2)
H(2)	4e	0.1897(9)	0.279(5)	0.433(2)	0.11(2)
H(3)	4e	0.4465(3)	0.3992(4)	0.4446(2)	0.036
H(4)	4e	0.2494(4)	0.4250(4)	0.3705(2)	0.042
H(5)	4e	0.3343(4)	0.5365(5)	0.4899(2)	0.051
H(6)	4e	0.3594(4)	0.7741(5)	0.4532(2)	0.059
H(7)	4e	0.3031(4)	0.7311(5)	0.3870(2)	0.059
H(8)	4e	0.4792(4)	0.7412(4)	0.3847(2)	0.052
H(9)	4e	0.5078(4)	0.6366(4)	0.4429(2)	0.052
H(10)	4e	0.4707(4)	0.4552(5)	0.2535(2)	0.069
H(11)	4e	0.5824(4)	0.3832(5)	0.2636(2)	0.069
H(12)	4e	0.5269(4)	0.1499(5)	0.2804(2)	0.065
H(13)	4e	0.3783(4)	0.0883(5)	0.3129(2)	0.057
H(14)	4e	0.3344(4)	0.2552(5)	0.3016(2)	0.057
H(15)	4e	0.4877(3)	0.0190(4)	0.4048(2)	0.076
H(16)	4e	0.3945(3)	0.0784(4)	0.4329(2)	0.076
H(17)	4e	0.5082(3)	0.1319(4)	0.4604(2)	0.076
H(18)	4e	0.6496(3)	0.5617(5)	0.4028(2)	0.088
H(19)	4e	0.6332(3)	0.6186(5)	0.3351(2)	0.088
H(20)	4e	0.6960(3)	0.4680(5)	0.3547(2)	0.088
H(21)	4e	0.3125(4)	0.6859(5)	0.2464(2)	0.114
H(22)	4e	0.4337(4)	0.6711(5)	0.2644(2)	0.114
H(23)	4e	0.3730(4)	0.7645(5)	0.3060(2)	0.114
H(24)	4e	0.5406(7)	0.3504(9)	0.1312(3)	0.224
H(25)	4e	0.3842(7)	0.4545(9)	0.1376(3)	0.267
H(26)	4e	0.3415(7)	0.4045(9)	0.0700(3)	0.267
H(27)	4e	0.4330(7)	0.5226(9)	0.0847(3)	0.267
H(28)	4e	0.5546(8)	0.193(1)	0.0497(4)	0.378
H(29)	4e	0.5355(8)	0.366(1)	0.0313(4)	0.378
H(30)	4e	0.4440(8)	0.248(1)	0.0165(4)	0.378
H(31)	4e	0.5671(6)	−0.0287(8)	0.1212(3)	0.175
H(32)	4e	0.4915(7)	−0.1835(8)	0.1835(4)	0.290
H(33)	4e	0.5547(7)	−0.0974(8)	0.2404(4)	0.290
H(34)	4e	0.6132(7)	−0.1925(8)	0.1982(4)	0.290

Table 2. Continued.

Atom	Site	x	y	z	U_{iso}
H(35)	4*e*	0.6615(7)	0.176(1)	0.1637(4)	0.285
H(36)	4*e*	0.7128(7)	0.019(1)	0.1863(4)	0.285
H(37)	4*e*	0.6542(7)	0.114(1)	0.2285(4)	0.285
H(38)	4*e*	0.2704(7)	0.131(1)	0.0938(3)	0.243
H(39)	4*e*	0.3718(8)	0.025(2)	0.0272(4)	0.528

Table 2. Continued.

Atom	Site	x	y	z	U_{iso}
H(40)	4*e*	0.2734(8)	–0.077(2)	0.0280(4)	0.528
H(41)	4*e*	0.3825(8)	–0.128(2)	0.0646(4)	0.528
H(42)	4*e*	0.2574(8)	0.0086(9)	0.1808(4)	0.301
H(43)	4*e*	0.3124(8)	–0.1353(9)	0.1597(4)	0.301
H(44)	4*e*	0.2033(8)	–0.0836(9)	0.1231(4)	0.301

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}
Si(1)	4*e*	0.4449(1)	0.1356(2)	0.14892(6)	0.071(1)	0.098(1)	0.0488(9)	0.004(1)	0.0153(8)	–0.0143(9)
O(1)	4*e*	0.3594(2)	0.5412(3)	0.3138(1)	0.041(2)	0.050(2)	0.046(2)	–0.003(2)	0.000(2)	0.014(2)
O(2)	4*e*	0.1924(3)	0.5979(4)	0.4509(2)	0.033(2)	0.067(2)	0.080(3)	0.003(2)	0.017(2)	–0.022(2)
O(3)	4*e*	0.2591(3)	0.2853(3)	0.4421(1)	0.028(2)	0.046(2)	0.061(2)	–0.007(2)	0.013(2)	0.013(2)
O(4)	4*e*	0.4149(3)	0.2006(4)	0.2106(1)	0.058(3)	0.113(3)	0.050(2)	0.000(2)	0.012(2)	–0.027(2)
C(1)	4*e*	0.4476(4)	0.2391(4)	0.3803(2)	0.026(3)	0.039(3)	0.049(3)	–0.006(2)	0.014(2)	–0.004(2)
C(2)	4*e*	0.4051(3)	0.3855(4)	0.4034(2)	0.028(3)	0.032(3)	0.031(2)	–0.004(2)	0.004(2)	0.001(2)
C(3)	4*e*	0.2947(4)	0.4114(4)	0.4106(2)	0.031(3)	0.032(3)	0.041(3)	–0.002(2)	0.006(2)	0.002(2)
C(4)	4*e*	0.2967(4)	0.5578(5)	0.4483(2)	0.032(4)	0.042(3)	0.051(3)	0.002(2)	0.005(2)	–0.008(2)
C(5)	4*e*	0.3495(4)	0.6928(5)	0.4231(2)	0.043(4)	0.033(3)	0.073(3)	–0.001(3)	0.017(3)	–0.012(2)
C(6)	4*e*	0.4561(4)	0.6556(4)	0.4062(2)	0.039(3)	0.033(3)	0.058(3)	–0.005(2)	0.012(2)	0.002(2)
C(7)	4*e*	0.4422(3)	0.5136(4)	0.3657(2)	0.031(3)	0.037(3)	0.042(3)	–0.007(2)	0.003(3)	0.002(2)
C(8)	4*e*	0.5385(4)	0.4374(5)	0.3454(2)	0.032(3)	0.039(3)	0.051(3)	–0.004(2)	0.016(2)	0.004(2)
C(9)	4*e*	0.5176(4)	0.3838(5)	0.2783(2)	0.058(4)	0.073(4)	0.049(3)	–0.009(3)	0.027(3)	–0.001(3)
C(10)	4*e*	0.4703(4)	0.2249(5)	0.2717(2)	0.051(4)	0.071(4)	0.041(3)	–0.003(3)	0.010(3)	–0.017(3)
C(11)	4*e*	0.3974(4)	0.1958(5)	0.3145(2)	0.048(4)	0.044(3)	0.052(3)	–0.003(2)	0.013(3)	–0.011(2)
O(12)	4*e*	0.5537(2)	0.2970(3)	0.3811(1)	0.030(2)	0.039(2)	0.057(2)	0.002(2)	0.013(2)	0.003(2)
C(13)	4*e*	0.4607(3)	0.1049(4)	0.4235(2)	0.047(4)	0.032(3)	0.073(3)	0.004(2)	0.012(3)	0.012(2)
C(14)	4*e*	0.6386(3)	0.5300(5)	0.3609(2)	0.037(4)	0.065(3)	0.077(3)	–0.006(3)	0.021(3)	0.005(3)
C(15)	4*e*	0.3705(4)	0.6768(5)	0.2799(2)	0.077(5)	0.077(4)	0.067(3)	–0.002(3)	0.001(3)	0.041(3)
C(16)	4*e*	0.4771(7)	0.3083(9)	0.1060(3)	0.21(1)	0.161(7)	0.096(5)	0.095(7)	0.083(5)	0.057(5)
C(17)	4*e*	0.4027(7)	0.4330(9)	0.0990(3)	0.24(1)	0.158(8)	0.146(7)	0.033(7)	0.051(7)	0.051(6)
C(18)	4*e*	0.5054(8)	0.276(1)	0.0455(4)	0.36(2)	0.30(1)	0.144(7)	0.14(1)	0.158(9)	0.098(7)
C(19)	4*e*	0.5596(6)	0.0082(8)	0.1615(3)	0.084(6)	0.109(6)	0.150(6)	–0.015(5)	0.008(5)	0.015(5)
C(20)	4*e*	0.5543(7)	–0.1281(8)	0.1992(4)	0.17(1)	0.120(7)	0.31(1)	0.031(6)	0.113(8)	0.079(7)
C(21)	4*e*	0.6550(7)	0.086(1)	0.1872(4)	0.15(1)	0.22(1)	0.210(9)	0.073(8)	0.064(8)	0.046(8)
C(22)	4*e*	0.3184(7)	0.047(1)	0.1085(3)	0.19(1)	0.183(8)	0.102(6)	–0.021(7)	–0.005(6)	–0.071(6)
C(23)	4*e*	0.3384(8)	–0.042(2)	0.0516(4)	0.21(1)	0.60(2)	0.24(1)	–0.11(1)	0.034(9)	–0.32(1)
C(24)	4*e*	0.2688(8)	–0.0487(9)	0.1461(4)	0.26(1)	0.182(8)	0.144(7)	–0.155(8)	–0.007(7)	0.035(6)

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References

1. Wittenberg, J.: Stereoselektive Darstellung von oxa- und dioxatricyclischen Substrukturen des Dictyoxetans. Dissertation, Universität Hannover, Germany 1998.
2. Wittenberg, J.; Beil, W.; Hoffmann, H. M. R.: Synthesis of Dioxatricyclic Segments of Dictyoxetane. Oxygenated 6,8-Dimethyl-2,7-dioxatricyclo[4.2.1.$0^{3,8}$]nonanes show Antitumor Activity. Tetrahedron Lett. **39** (1998) 8259-8262 .
3. Sheldrick, G. M.: SHELXS-86, program for crystal structure determination. University of Göttingen, Germany 1993.
4. Sheldrick, G. M.: SHELXL-93, a program for refining crystal structures. University of Göttingen, Germany 1993.
5. Spek, A. L.: PLATON, an integrated tool for the analysis of the results of a single crystal structure determination. Acta Crystallogr. Suppl. A**46** (1990) C-34.