Crystal structure of 2,2,7,7-tetramethyldinaphtho[2,1-c;1',2'-e]-1,2,7-oxadisilepin, (C₁₀H₆Si(CH₃)₂)₂O

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Table 1. Parameters used for the X-ray data collection

Crystal:	colorless, white, spherical form, size 0.3 x 0.3 x 0.3 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	1.79 cm ⁻¹
Diffractometer:	Enraf Nonius CAD4
Scan mode:	ω
$T_{\text{measurement}}$:	293 K
$2\theta_{\text{max}}$:	56°
$N(hkl)_{\text{unique}}$:	6648
Criterion for I_o :	$I_o > 2 \sigma(I_o)$
$N(\text{param})_{\text{refined}}$:	496
Program:	SHELXL-93

Source of material: Reaction of 0.75 mmol 2,2'-bis(trimethylsilyl)-1,1'-binaphthyl with 1.39 mmol of boron tribromide in dichloromethane at 195 K. The mixture was held at this temperature for one hour, was allowed to warm to room temperature overnight and was then hydrolysed with water. Usual work up and recrystallisation from toluene yielded 58.4% of the title compound (see ref. 1).

The crystal structure of 2,2,7,7-tetramethyldinaphtho[2,1-c;1',2'-e]-1,2,7-oxadisilepin is comparable with the structures of 2,2'-bis(trimethylsilyl)-1,1'-binaphthyl and the corresponding germyl compound (see refs. 2, 3). There are also two independent molecules forming the asymmetric unit. Here the two silicon atoms of a molecule are connected through an oxygen atom, forming a 7-membered ring. This has an effect on the angle between the two naphthyl planes (70.2°) of a molecule and the packing of the molecules in the unit cell. For the other compounds (see ref. 2, 3) the planes are orientated right-angled to each other (88–90°), but here the naphthyl planes from one molecule to another are parallel orientated. This could be a reason for closer packing of the molecules and a minor volume of the unit cell.

C₄₈H₄₈O₂Si₄, triclinic, $P\bar{1}$ (No. 2), $a = 8.654(2)$ Å, $b = 11.502(3)$ Å, $c = 22.821(3)$ Å, $\alpha = 77.76(1)^\circ$, $\beta = 86.46(1)^\circ$, $\gamma = 72.20(1)^\circ$, $V = 2113.6$ Å³, $Z = 2$, $R(F) = 0.060$, $R_w(F^2) = 0.113$.

Table 2. Final atomic coordinates and displacement parameters (in Å²)

Atom	Site	x	y	z	U_{iso}
H(3)	2i	0.077(2)	0.573(2)	0.3495(8)	0.060
H(4)	2i	0.162(2)	0.724(2)	0.2852(8)	0.082
H(5)	2i	0.289(3)	0.678(2)	0.1952(8)	0.116
H(6)	2i	0.375(3)	0.475(2)	0.1799(8)	0.089
H(8)	2i	0.350(2)	0.258(2)	0.2078(7)	0.074
H(9)	2i	0.284(2)	0.107(2)	0.2758(7)	0.075
H(13)	2i	-0.156(2)	0.472(2)	0.2971(7)	0.059
H(14)	2i	-0.421(2)	0.589(2)	0.2866(9)	0.103
H(15)	2i	-0.578(2)	0.650(2)	0.370(1)	0.111
H(16)	2i	-0.470(2)	0.589(2)	0.459(1)	0.101
H(18)	2i	-0.239(2)	0.446(2)	0.5302(8)	0.072
H(19)	2i	0.026(2)	0.316(2)	0.5428(7)	0.070
H(41A)	2i	0.157(9)	-0.057(3)	0.330(2)	0.162
H(41B)	2i	0.19(1)	-0.130(3)	0.397(2)	0.162
H(41C)	2i	0.326(4)	-0.083(5)	0.360(4)	0.162
H(42A)	2i	-0.084(2)	0.036(4)	0.451(2)	0.103
H(42B)	2i	-0.144(3)	0.101(7)	0.386(1)	0.103
H(42C)	2i	-0.123(3)	0.181(4)	0.431(3)	0.103
H(43A)	2i	0.33(1)	0.215(2)	0.570(1)	0.103
H(43B)	2i	0.432(7)	0.083(8)	0.5601(7)	0.103
H(43C)	2i	0.246(8)	0.11(1)	0.574(1)	0.103
H(44A)	2i	0.553(2)	0.189(5)	0.450(2)	0.097
H(44B)	2i	0.440(5)	0.327(3)	0.433(3)	0.097
H(44C)	2i	0.452(6)	0.234(7)	0.390(1)	0.097
H(23)	2i	1.160(2)	0.030(2)	0.2013(8)	0.079
H(24)	2i	1.427(3)	-0.093(2)	0.2141(9)	0.086
H(25)	2i	1.571(2)	-0.153(2)	0.1346(9)	0.088
H(26)	2i	1.460(2)	-0.078(2)	0.0366(7)	0.071
H(28)	2i	1.236(2)	0.042(2)	-0.0306(8)	0.081
H(29)	2i	0.990(2)	0.179(2)	-0.0439(6)	0.061

Table 2. (Continued)

Atom	Site	x	y	z	U_{iso}
H(33)	2i	0.927(2)	-0.077(2)	0.1503(7)	0.064
H(34)	2i	0.838(2)	-0.222(2)	0.2162(9)	0.092
H(35)	2i	0.704(3)	-0.179(2)	0.3024(9)	0.078
H(36)	2i	0.652(2)	0.012(2)	0.3230(7)	0.082
H(38)	2i	0.650(2)	0.225(2)	0.2923(6)	0.085
H(39)	2i	0.722(2)	0.381(2)	0.2283(7)	0.067
H(45A)	2i	0.577(7)	0.415(7)	-0.0594(8)	0.100
H(45B)	2i	0.67(1)	0.279(3)	-0.066(1)	0.100
H(45C)	2i	0.764(7)	0.38(1)	-0.0726(9)	0.100

Table 2. (Continued)

Atom	Site	x	y	z	U_{iso}
H(46A)	2i	0.581(3)	0.210(6)	0.106(2)	0.098
H(46B)	2i	0.503(7)	0.220(7)	0.045(2)	0.098
H(46C)	2i	0.464(4)	0.337(2)	0.074(3)	0.098
H(47A)	2i	1.141(2)	0.343(6)	0.115(2)	0.126
H(47B)	2i	1.091(3)	0.481(3)	0.079(4)	0.126
H(47C)	2i	1.090(3)	0.373(8)	0.047(2)	0.126
H(48A)	2i	0.842(6)	0.572(3)	0.165(2)	0.111
H(48B)	2i	0.667(5)	0.574(4)	0.150(3)	0.111
H(48C)	2i	0.767(8)	0.639(2)	0.101(1)	0.111

Table 3. Final atomic coordinates and displacement parameters (in Å²)

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Si(1)	2i	0.2801(6)	0.1947(5)	0.4717(2)	0.043(3)	0.053(3)	0.050(3)	-0.019(4)	-0.003(2)	-0.010(2)
Si(2)	2i	0.1375(7)	0.0819(5)	0.3929(2)	0.069(4)	0.059(4)	0.059(3)	-0.029(4)	-0.009(3)	-0.019(3)
O(1)	2i	0.257(2)	0.076(1)	0.4486(5)	0.07(1)	0.058(8)	0.064(7)	-0.03(1)	-0.007(7)	-0.008(6)
C(1)	2i	0.115(2)	0.341(2)	0.3463(7)	0.03(1)	0.05(1)	0.049(8)	0.00(1)	0.007(7)	-0.031(7)
C(2)	2i	0.163(2)	0.436(2)	0.3030(6)	0.05(1)	0.05(1)	0.042(8)	-0.03(1)	-0.015(8)	0.014(7)
C(3)	2i	0.132(2)	0.555(2)	0.3147(8)	0.04(1)	0.05(1)	0.044(9)	0.01(2)	0.001(9)	-0.009(9)
C(4)	2i	0.181(2)	0.645(2)	0.2767(8)	0.08(2)	0.06(1)	0.06(1)	-0.02(2)	-0.02(1)	0.01(1)
C(5)	2i	0.264(3)	0.615(2)	0.2236(8)	0.11(2)	0.13(2)	0.06(1)	-0.08(2)	-0.02(1)	0.04(1)
C(6)	2i	0.309(3)	0.494(2)	0.2125(8)	0.08(2)	0.08(2)	0.06(1)	-0.02(2)	-0.00(1)	0.01(1)
C(7)	2i	0.255(2)	0.403(2)	0.2512(6)	0.04(1)	0.06(1)	0.029(7)	-0.00(1)	-0.011(7)	0.012(7)
C(8)	2i	0.294(2)	0.278(2)	0.2423(7)	0.07(2)	0.07(1)	0.059(9)	-0.01(1)	0.014(9)	-0.050(8)
C(9)	2i	0.254(2)	0.188(2)	0.2820(7)	0.08(1)	0.07(1)	0.052(9)	-0.03(1)	0.010(9)	-0.019(8)
C(10)	2i	0.163(2)	0.220(1)	0.3348(6)	0.03(1)	0.035(9)	0.039(7)	0.01(1)	-0.016(7)	0.005(6)
C(11)	2i	0.016(2)	0.372(1)	0.3976(5)	0.03(1)	0.028(8)	0.034(7)	-0.00(1)	-0.011(6)	0.004(6)
C(12)	2i	-0.150(2)	0.449(2)	0.3886(8)	0.05(1)	0.04(1)	0.06(1)	0.00(1)	0.019(9)	-0.029(8)
C(13)	2i	-0.218(2)	0.492(2)	0.3304(7)	0.023(9)	0.05(1)	0.06(1)	0.02(1)	-0.010(8)	-0.029(9)
C(14)	2i	-0.374(2)	0.563(2)	0.3246(9)	0.07(1)	0.12(2)	0.09(1)	-0.05(2)	-0.03(1)	-0.00(1)
C(15)	2i	-0.471(2)	0.600(2)	0.375(1)	0.04(1)	0.10(2)	0.14(2)	-0.02(1)	0.03(1)	-0.04(2)
C(16)	2i	-0.407(2)	0.562(2)	0.427(1)	0.03(1)	0.09(2)	0.15(2)	-0.02(1)	0.00(1)	-0.06(1)
C(17)	2i	-0.250(2)	0.484(2)	0.4399(7)	0.04(1)	0.07(1)	0.059(9)	-0.03(1)	0.002(8)	-0.021(9)
C(18)	2i	-0.178(2)	0.429(2)	0.4963(8)	0.04(1)	0.07(1)	0.08(1)	-0.01(1)	0.03(1)	-0.04(1)
C(19)	2i	-0.016(2)	0.350(2)	0.5043(7)	0.06(1)	0.06(1)	0.067(9)	-0.02(1)	-0.021(8)	-0.015(9)
C(20)	2i	0.080(2)	0.321(2)	0.4546(7)	0.05(1)	0.06(1)	0.051(9)	-0.03(1)	0.000(8)	-0.019(8)
C(41)	2i	0.211(3)	-0.064(2)	0.3670(9)	0.16(3)	0.06(2)	0.11(2)	-0.04(2)	-0.02(2)	-0.03(1)
C(42)	2i	-0.080(2)	0.103(2)	0.4183(7)	0.05(1)	0.09(1)	0.08(1)	-0.04(1)	0.016(9)	-0.03(1)
C(43)	2i	0.327(2)	0.144(2)	0.5540(6)	0.09(2)	0.07(1)	0.045(8)	-0.04(2)	-0.025(9)	0.009(9)
C(44)	2i	0.452(2)	0.242(2)	0.4313(7)	0.025(9)	0.07(1)	0.10(1)	-0.01(1)	0.005(9)	-0.02(1)
Si(3)	2i	0.7205(6)	0.3051(5)	0.0283(2)	0.049(4)	0.048(3)	0.042(2)	-0.015(4)	-0.004(2)	-0.001(2)
Si(4)	2i	0.8615(7)	0.4193(5)	0.1063(2)	0.050(4)	0.037(3)	0.059(3)	-0.011(4)	-0.007(3)	-0.001(2)
O(2)	2i	0.749(1)	0.427(1)	0.0503(5)	0.054(9)	0.044(7)	0.052(6)	-0.013(9)	-0.019(6)	0.002(5)
C(21)	2i	0.980(2)	0.135(2)	0.0998(7)	0.04(1)	0.05(1)	0.07(1)	-0.02(1)	0.020(8)	-0.036(8)
C(22)	2i	1.141(2)	0.057(2)	0.1106(6)	0.035(8)	0.06(1)	0.049(8)	-0.027(9)	-0.008(6)	-0.016(7)
C(23)	2i	1.219(2)	0.010(2)	0.1676(8)	0.07(1)	0.07(2)	0.06(1)	-0.04(1)	0.00(1)	-0.02(1)
C(24)	2i	1.380(3)	-0.066(2)	0.1760(9)	0.05(2)	0.04(1)	0.10(1)	0.04(1)	-0.02(1)	-0.04(1)
C(25)	2i	1.465(2)	-0.100(2)	0.1290(9)	0.06(1)	0.04(1)	0.12(2)	0.01(1)	-0.04(1)	-0.03(1)
C(26)	2i	1.398(2)	-0.055(2)	0.0695(7)	0.06(1)	0.05(1)	0.062(8)	-0.01(1)	0.023(8)	-0.027(8)
C(27)	2i	1.237(2)	0.024(2)	0.0624(9)	0.029(9)	0.05(1)	0.10(1)	-0.01(1)	0.019(9)	-0.04(1)
C(28)	2i	1.175(2)	0.068(2)	0.0019(8)	0.07(1)	0.10(2)	0.05(1)	-0.05(2)	0.01(1)	-0.03(1)

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