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Structures of a Cyclic Diimidodisiloxane and its Parent Dianhydride

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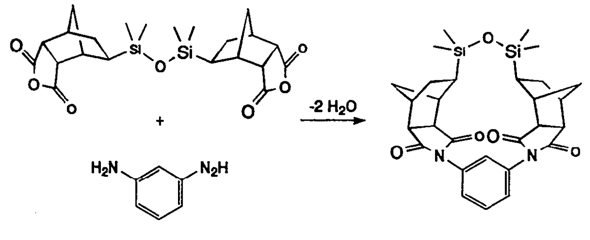
Abstract. (1): (±)-5,5′-(1,1,3,3-Tetramethyldisiloxane-1,3-diyl)bis{(1*R**,2*S**,3*S**,4*R**,5*S**)-bicyclo[2.2.1]heptene-2,3-dicarboxylic anhydride}, $C_{22}H_{30}O_7Si_2$, $M_r = 462.6$, monoclinic, Cc, $a = 8.519\,(6)$, $b = 22.21\,(1)$, $c = 13.36\,(2)$ Å, $\beta = 108.06\,(9)°$, $V = 2403.4$ Å^3, $Z = 4$, $D_x = 1.28$ g cm^{-3}, $\lambda(\text{Mo }K\alpha) = 0.71069$ Å, $\mu = 1.9$ cm^{-1}, $F(000) = 984$, room temperature, final $R = 0.0908$ for 1942 unique observed reflections. (2): (±)-(2*R**,3*S**,3a*S**,12a*S**,13*S**,14*R**,15a*R**,16*S**,20*S**,20a*R**)-2,3,3a,13,14,15,15a,16,17,19,20,20a-Dodecahydro-17,17,19,19-tetramethyl-2,20:3,5:11,13:14,16-tetramethano-6,10-metheno-5*H*-dicyclopent[*d*,*o*][1,7,13,2,18]oxadiazadisilacyclooctadecene-4,12,23,25(1*H*,12a*H*)-tetrone, $C_{28}H_{34}N_2O_5Si_2$, $M_r = 534.8$, orthorhombic, $Pnca$, $a = 11.567\,(3)$, $b = 12.585\,(4)$, $c = 18.430\,(4)$ Å, $V = 2683.0$ Å^3, $Z = 4$, $D_x = 1.32$ g cm^{-3}, $\lambda(\text{Mo }K\alpha) = 0.71069$ Å, $\mu = 1.7$ cm^{-1}, $F(000) = 1136$, room temperature, final $R = 0.0833$ for 998 unique observed reflections. The structures of the silicone dianhydride (1) and its unstrained cyclocondensation product with *m*-phenylenediamine (2) have been determined. The bond lengths and angles of the two compounds are very similar except for the Si—O—Si bond angle.

Introduction. This laboratory has been active in the synthesis of functionalized disiloxanes and the preparation of polyimidosiloxanes from them. In particular we have investigated the dianhydride 5,5′-(1,1,3,3-tetramethyldisiloxane-1,3-diyl)bis(bicyclo[2.2.1]heptene-2,3-dicarboxylic anhydride) (1) and the polymer and cyclooligomers which result from its condensation with *m*-phenylenediamine, as shown in the scheme below. In the preparation of the polyimide we have been able to isolate the cyclic monomer (2) in high yield. The monomer was also isolated from a polymerization mixture where the molecular weight was built by siloxane equilibrations (Buese, 1990). In that process, the enthalpy of polymerization is zero. Hence, the cyclic monomer (2) is free of strain. Crystals of (1) and (2) were isolated and determined to be the D,L isomers rather than the *meso* isomers. The first difference in the composition of atoms between these two disiloxanes occurs five bonds from the Si atoms. The configuration of substituents about each atom up to that point is identical. This paper describes the structure of the two disiloxanes, (1) and (2). It will be shown that the common segments of these two unstrained structures are significantly different only in conformation and in the Si—O—Si bond angle.



Experimental. The synthesis of (1) has been reported by Ryang (1983). The white solid had a melting

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Table 1. *Experimental details*

	(1)	(2)
Crystal habit	Plate	Plate
Size (mm)	0.2 × 0.5 × 0.1	0.2 × 0.2 × 0.1
Lattice parameters		
No. of reflections	24	20
2θ range (°)	5–22	4–16
Reflection range		
h	0 to 11	0 to 13
k	0 to 27	0 to 14
l	−16 to 16	0 to 20
Max. $(\sin\theta)/\lambda$ (Å^{-1})	0.595	0.538
Check reflections	$22\bar{1}$ $06\bar{1}$	202 113
Variation (%)	1 1	1 1
Reflections collected	4663	2035
Unique observed [$F > 3\sigma(F)$]	1942	998
No. of parameters	278	169
R	0.0908	0.0833
wR	0.1206	0.0579
Weighting $w^{-1} = \sigma^2(F) +$	$0.0127F^2$	$0.00008F^2$
S	0.804	1.290
Difference peaks (e Å^{-3})		
Min.	−0.701	−0.480
Max.	0.698	0.443
Max. Δ/σ	0.001	−0.351

Table 2. *Atomic coordinates and equivalent isotropic thermal parameters for* (1)

U_{eq} is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U_{eq} (Å^2 × 10^3)
Si(1)	0.0000	0.1757 (1)	0.0000	49 (1)
Si(1*a*)	−0.0138 (3)	0.0371 (1)	−0.0015 (2)	44 (1)
O(1)	0.0421 (7)	0.1053 (2)	−0.0187 (5)	53 (2)
O(2)	0.3354 (16)	0.3505 (4)	0.0091 (10)	122 (5)
O(2*a*)	0.1697 (15)	−0.1466 (4)	−0.1133 (7)	113 (5)
O(3)	0.705 (16)	0.3492 (4)	−0.3316 (8)	129 (5)
O(3*a*)	0.5524 (15)	−0.1359 (4)	0.1915 (8)	119 (5)
O(4)	0.1819 (11)	0.3598 (3)	−0.1581 (8)	101 (4)
O(4*a*)	0.3506 (13)	−0.1513 (3)	0.0440 (7)	97 (4)
C(1)	0.0968 (14)	0.1970 (5)	0.1348 (8)	81 (4)
C(1*a*)	−0.1830 (12)	0.0131 (4)	−0.1171 (8)	68 (4)
C(2)	- 0.2282 (10)	0.1865 (4)	−0.0388 (10)	74 (4)
C(2*a*)	−0.0833 (12)	0.0342 (4)	0.1179 (8)	70 (4)
C(3)	0.0866 (8)	0.2243 (3)	−0.0851 (7)	52 (3)
C(3*a*)	0.1680 (10)	−0.0145 (3)	0.0203 (6)	49 (3)
C(4)	0.0014 (13)	0.2206 (5)	−0.2044 (8)	79 (4)
C(4*a*)	0.3059 (12)	−0.0082 (4)	0.1289 (7)	60 (3)
C(5)	0.1445 (15)	0.2117 (4)	−0.2491 (9)	81 (4)
C(5*a*)	0.4634 (10)	−0.0032 (4)	0.0972 (8)	63 (3)
C(6)	0.2589 (15)	0.1722 (4)	−0.1647 (9)	78 (4)
C(6*a*)	0.4038 (10)	0.0352 (3)	−0.0027 (8)	60 (3)
C(7)	0.2699 (9)	0.2137 (3)	−0.0739 (7)	54 (3)
C(7*a*)	0.2643 (10)	−0.0085 (3)	−0.0588 (6)	52 (3)
C(8)	0.3377 (12)	0.2709 (4)	−0.1152 (9)	73 (4)
C(8*a*)	0.3672 (12)	−0.0662 (4)	−0.0564 (7)	65 (4)
C(9)	0.2548 (14)	0.2709 (4)	−0.2295 (9)	78 (4)
C(9*a*)	0.5021 (11)	−0.0634 (4)	0.0504 (7)	62 (3)
C(10)	0.1533 (16)	0.3282 (5)	−0.2522 (11)	94 (5)
C(10*a*)	0.4827 (15)	−0.1184 (4)	0.1084 (9)	78 (4)
C(11)	0.2949 (14)	0.3289 (4)	−0.0745 (10)	76 (4)
C(11*a*)	0.2863 (15)	−0.1250 (4)	−0.0490 (9)	79 (4)

Table 3. *Atomic coordinates and equivalent isotropic thermal parameters for* (2)

U_{eq} is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U_{eq} (Å^2 × 10^3)
Si(1)	0.1821 (2)	0.6109 (2)	0.4251 (1)	33 (1)
O(1)	0.2500	0.5000	0.4407 (4)	41 (3)
O(2)	0.1510 (5)	0.7441 (5)	0.1867 (3)	64 (2)
O(3)	−0.618 (5)	0.4463 (5)	0.1415 (3)	65 (3)
N(1)	0.0641 (5)	0.5862 (5)	0.1557 (3)	41 (3)
C(1)	0.0901 (7)	0.6396 (6)	0.5050 (4)	52 (3)
C(2)	0.2853 (6)	0.7222 (6)	0.4159 (4)	50 (4)
C(3)	0.1015 (6)	0.5982 (6)	0.3375 (4)	32 (3)
C(4)	0.0330 (6)	0.4917 (7)	0.3234 (4)	40 (3)
C(5)	−0.0906 (7)	0.5291 (7)	0.3060 (4)	51 (4)
C(6)	−0.1017 (6)	0.6248 (6)	0.3562 (4)	54 (3)
C(7)	0.0048 (7)	0.6837 (6)	0.3259 (4)	38 (3)
C(8)	−0.0294 (7)	0.6881 (6)	0.2439 (4)	46 (3)
C(9)	−0.0972 (7)	0.5857 (7)	0.2323 (4)	45 (3)
C(10)	−0.0369 (8)	0.5291 (7)	0.1719 (5)	47 (4)
C(11)	0.0721 (8)	0.6825 (7)	0.1936 (4)	46 (3)
C(12)	0.1579 (8)	0.5427 (7)	0.1142 (4)	46 (4)
C(13)	0.2500	0.5000	0.1510 (6)	46 (5)
C(14)	0.1564 (8)	0.5444 (6)	0.0388 (4)	55 (4)
C(15)	0.2500	0.5000	0.0030 (6)	67 (5)

range of 399–426 K. Analysis by liquid chromatography exhibited two peaks of equal area. Three recrystallizations from toluene yielded a few fine white needles which melted at 444–446 K; these crystals were of insufficient quality for X-ray analysis. The powdery residue from the mother liquor was dissolved in an acetone:water mixture. Upon standing overnight, a few large colorless crystals were formed which melted sharply at 404–405 K and gave a single peak by liquid chromatographic analysis. A suitable large crystal was selected for X-ray analysis.

The acid equilibration of a 1 wt% solution of poly{*N*,*N*′-*m*-phenylenebis[(1*R**,2*S**,3*S**,4*R**,5*S**)-5-(pentamethyldisiloxanyl)-2,3-norbornanedicarboximide]} (4.0 g) in 1,2-dichlorobenzene at 323 K and 50 mm Hg was carried out. After 24 h the catalyst was quenched and the solution filtered and concentrated. Thin-layer chromatography analysis of this residue indicated a large portion of cyclic oligomers with (2) being the largest component. Many large crystals formed from a concentrated acetone solution of the residue (41% isolated yield). The crystals melted sharply at 576 K with decomposition. A suitable large crystal was selected from those recovered for X-ray analysis.

All data were collected in air at room temperature using a Nicolet *P*3*F* automated single-crystal diffractometer and Mo $K\alpha$ radiation; intensities measured by θ–2θ scans. Lp corrections applied; anomalous-dispersion corrections not applied; D_m values not determined. Each of the structures was solved using direct methods and the *SHELXTL* program package (Sheldrick, 1981). All H-atom positions were located by a difference Fourier synthesis and, in the final stages of full-matrix least-squares refinement (on F), constrained to have bond lengths of 0.96 Å, appropriate bond angles and isotropic thermal parameters of 0.06 Å^2. All non-H atoms were treated anisotropically. No absorption correction was applied to either of the structures; no correction for secondary extinction was made. Details of the data collection are presented in Table 1. All values of R are for observed reflections. The sizes of the R values for both compounds presumably reflect the thinness of the crystals and the temperature at which the data were collected. The difference peaks for (1) are close to Si, while those for (2) are close to C(10). The residual

Table 4. *Bond angles* (°)

(1)

Si(1)—O(1)—Si(1*a*)	141.2 (5)	O(1)—Si(1*a*)—C(1*a*)	109.8 (4)
O(1)—Si(1)—C(1)	110.5 (4)	O(1)—Si(1*a*)—C(2*a*)	109.7 (4)
O(1)—Si(1)—C(2)	109.8 (4)	O(1)—Si(1*a*)—C(3*a*)	109.1 (4)
O(1)—Si(1)—C(3)	108.1 (4)	C(1*a*)—Si(1*a*)—C(2*a*)	109.6 (5)
C(1)—Si(1)—C(2)	111.1 (6)	C(1*a*)—Si(1*a*)—C(3*a*)	110.4 (4)
C(1)—Si(1)—C(3)	108.1 (5)	C(2*a*)—Si(1*a*)—C(3*a*)	108.2 (4)
C(2)—Si(1)—C(3)	109.2 (4)	Si(1*a*)—C(3*a*)—C(4*a*)	115.9 (6)
Si(1)—C(3)—C(4)	116.7 (6)	Si(1*a*)—C(3*a*)—C(7*a*)	115.7 (5)
Si(1)—C(3)—C(7)	115.5 (5)	C(4*a*)—C(3*a*)—C(7*a*)	102.9 (7)
C(4)—C(3)—C(7)	103.4 (8)	C(3*a*)—C(4*a*)—C(5*a*)	102.8 (8)
C(3)—C(4)—C(5)	103.6 (8)	C(4*a*)—C(5*a*)—C(6*a*)	101.5 (7)
C(4)—C(5)—C(6)	101.0 (10)	C(4*a*)—C(5*a*)—C(9*a*)	111.2 (7)
C(4)—C(5)—C(9)	109.3 (9)	C(6*a*)—C(5*a*)—C(9*a*)	100.5 (8)
C(6)—C(5)—C(9)	98.3 (8)	C(5*a*)—C(6*a*)—C(7*a*)	94.1 (6)
C(5)—C(6)—C(7)	96.3 (8)	C(3*a*)—C(7*a*)—C(6*a*)	102.5 (7)
C(3)—C(7)—C(6)	102.0 (7)	C(3*a*)—C(7*a*)—C(8*a*)	109.6 (7)
C(3)—C(7)—C(8)	108.3 (6)	C(6*a*)—C(7*a*)—C(8*a*)	99.5 (7)
C(6)—C(7)—C(8)	98.7 (8)	C(7*a*)—C(8*a*)—C(9*a*)	104.0 (7)
C(7)—C(8)—C(9)	104.8 (7)	C(7*a*)—C(8*a*)—C(11*a*)	117.1 (9)
C(7)—C(8)—C(11)	114.5 (10)	C(9*a*)—C(8*a*)—C(11*a*)	101.7 (7)
C(9)—C(8)—C(11)	106.6 (8)	C(5*a*)—C(9*a*)—C(8*a*)	102.6 (7)
C(5)—C(9)—C(8)	103.9 (9)	C(5*a*)—C(9*a*)—C(10*a*)	115.3 (9)
C(5)—C(9)—C(10)	113.0 (9)	C(8*a*)—C(9*a*)—C(10*a*)	106.3 (7)
C(8)—C(9)—C(10)	105.6 (10)	O(3*a*)—C(10*a*)—O(4*a*)	119.8 (11)
O(3)—C(10)—O(4)	120.3 (11)	O(3*a*)—C(10*a*)—C(9*a*)	132.3 (11)
O(3)—C(10)—C(9)	131.3 (13)	O(4*a*)—C(10*a*)—C(9*a*)	107.9 (8)
O(4)—C(10)—C(9)	108.2 (10)	O(2*a*)—C(11*a*)—O(4*a*)	120.5 (10)
O(2)—C(11)—O(4)	119.2 (10)	O(2*a*)—C(11*a*)—C(8*a*)	127.0 (10)
O(2)—C(11)—C(8)	132.3 (10)	O(4*a*)—C(11*a*)—C(8*a*)	112.4 (8)
O(4)—C(11)—C(8)	108.5 (9)	C(10*a*)—O(4*a*)—C(11*a*)	111.6 (9)
C(10)—O(4)—C(11)	110.8 (9)		

(2)

Si(1)—O(1)—Si(1*a*)	159.6 (6)	C(9)—C(8)—C(11)	106.1 (7)
O(1)—Si(1)—C(1)	107.7 (4)	C(5)—C(9)—C(8)	104.1 (7)
O(1)—Si(1)—C(2)	110.8 (4)	C(5)—C(9)—C(10)	114.5 (8)
O(1)—Si(1)—C(3)	108.7 (4)	C(8)—C(9)—C(10)	105.5 (7)
C(1)—Si(1)—C(2)	107.3 (5)	O(3)—C(10)—N(1)	122.5 (9)
C(1)—Si(1)—C(3)	114.6 (4)	O(3)—C(10)—C(9)	129.8 (9)
C(2)—Si(1)—C(3)	107.8 (4)	N(1)—C(10)—C(9)	107.6 (8)
Si(1)—C(3)—C(4)	117.6 (6)	O(2)—C(11)—N(1)	123.7 (8)
Si(1)—C(3)—C(7)	114.5 (6)	O(2)—C(11)—C(8)	129.0 (8)
C(4)—C(3)—C(7)	101.7 (6)	N(1)—C(11)—C(8)	107.3 (8)
C(3)—C(4)—C(5)	103.9 (7)	C(10)—N(1)—C(11)	113.1 (7)
C(4)—C(5)—C(6)	101.2 (7)	C(10)—N(1)—C(12)	123.1 (7)
C(4)—C(5)—C(9)	111.9 (7)	C(11)—N(1)—C(12)	123.0 (7)
C(6)—C(5)—C(9)	99.6 (7)	N(1)—C(12)—C(13)	118.2 (8)
C(5)—C(6)—C(7)	95.4 (7)	N(1)—C(12)—C(14)	121.2 (8)
C(3)—C(7)—C(6)	101.1 (7)	C(13)—C(12)—C(14)	120.6 (9)
C(3)—C(7)—C(8)	109.6 (7)	C(12)—C(13)—C(12*a*)	120.8 (11)
C(6)—C(7)—C(8)	99.5 (7)	C(12)—C(14)—C(15)	117.4 (9)
C(7)—C(8)—C(9)	103.6 (7)	C(14)—C(15)—C(14*a*)	123.1 (11)
C(7)—C(8)—C(11)	113.5 (7)		

Table 5. *Bond lengths* (Å)

(1)

Si(1)—O(1)	1.641 (6)	Si(1*a*)—O(1)	1.626 (6)
Si(1)—C(1)	1.796 (11)	Si(1*a*)—C(1*a*)	1.835 (10)
Si(1)—C(2)	1.866 (9)	Si(1*a*)—C(2*a*)	1.867 (13)
Si(1)—C(3)	1.878 (10)	Si(1*a*)—C(3*a*)	1.876 (9)
C(3)—C(4)	1.535 (13)	C(3*a*)—C(4*a*)	1.563 (11)
C(3)—C(7)	1.540 (11)	C(3*a*)—C(7*a*)	1.532 (14)
C(4)—C(5)	1.528 (19)	C(4*a*)—C(5*a*)	1.531 (15)
C(5)—C(6)	1.520 (14)	C(5*a*)—C(6*a*)	1.531 (14)
C(5)—C(9)	1.590 (15)	C(5*a*)—C(9*a*)	1.554 (13)
C(6)—C(7)	1.504 (14)	C(6*a*)—C(7*a*)	1.537 (11)
C(7)—C(8)	1.566 (14)	C(7*a*)—C(8*a*)	1.546 (12)
C(8)—C(9)	1.469 (16)	C(8*a*)—C(9*a*)	1.530 (12)
C(8)—C(11)	1.487 (15)	C(8*a*)—C(11*a*)	1.495 (14)
C(9)—C(10)	1.516 (15)	C(9*a*)—C(10*a*)	1.484 (15)
O(2)—C(11)	1.165 (18)	O(2*a*)—C(11*a*)	1.194 (14)
O(3)—C(10)	1.172 (16)	O(3*a*)—C(10*a*)	1.152 (14)
O(4)—C(10)	1.394 (17)	O(4*a*)—C(10*a*)	1.393 (14)
O(4)—C(11)	1.406 (13)	O(4*a*)—C(11*a*)	1.326 (14)

(2)

Si(1)—O(1)	1.627 (8)	C(8)—C(9)	1.524 (14)
Si(1)—C(1)	1.852 (12)	C(8)—C(11)	1.498 (14)
Si(1)—C(2)	1.849 (12)	C(9)—C(10)	1.495 (14)
Si(1)—C(3)	1.873 (12)	O(2)—C(11)	1.204 (12)
C(3)—C(4)	1.579 (13)	O(3)—C(10)	1.218 (13)
C(3)—C(7)	1.566 (13)	N(1)—C(10)	1.404 (13)
C(4)—C(5)	1.540 (13)	N(1)—C(11)	1.401 (12)
C(5)—C(6)	1.523 (14)	N(1)—C(12)	1.436 (13)
C(5)—C(9)	1.535 (14)	C(12)—C(13)	1.373 (12)
C(6)—C(7)	1.542 (14)	C(12)—C(14)	1.390 (13)
C(7)—C(8)	1.564 (14)	C(14)—C(15)	1.386 (12)

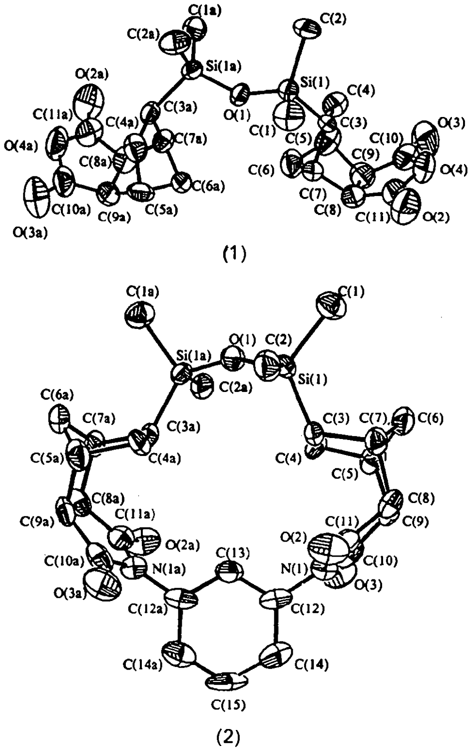

Fig. 1. Thermal ellipsoid (50% probability) plots of (1) (top) and (2) (bottom).

electron density failed to refine as either disorder or additional molecules, such as solvent. Scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV, pp. 55, 99, 149).

Discussion. Coordinates and thermal factors are given in Tables 2 and 3 for (1) and (2), respectively. The two halves of (2) are related by a crystallographic center of symmetry. Bond angles are given in Table 4 and bond lengths are given in Table 5.*

As seen in Fig. 1 the dianhydride (1) assumes a very different conformation to that of the cyclic imide (2). The size of the cavity of the cyclic imide is insufficient to permit the rotation of the phenylene group through the cavity. As seen in Table 5, the differences in the lengths of equivalent bonds in the two compounds, including the Si—O bonds, are very

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* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54356 (20 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

small. The bond angles (Table 4) of equivalent sites in the two molecules are also similar except for those of the Si—O—Si angle which differ by 18.4°.

The Si—O—Si bond angle of disiloxanes has been observed over an unusually large range. Typically, unstrained disiloxanes adopt a bent configuration with the Si—O—Si angle ranging from 140 to 160° (see for example Wiese, Wannagat, Thewalt & Debaerdemaeker, 1987; Chandra, Lo, Hitchcock & Lappert, 1987; Michalczyk, Fink, Haller, West & Michl, 1986; Karle, Karle & Nielsen, 1986; Fink, Haller, West & Michl, 1984; Curtis, D'Errico, Duffy, Epstein & Bell, 1983; Eaborn, Hitchcock & Lickiss, 1983; Verdegaal, De Kok, Westerink, Van Boom & Romers, 1981; Yanovskii, Struchkov, Kalinin, Ismailov & Myakushev, 1981; Voronkov, Nesterov, Gusev, Alexeev & Lotarev, 1981; Barrow, Ebsworth & Harding, 1979) although, in cases where the organosubstituents are bulky, a linear configuration with Si—O—Si equal to 180° has been observed (Gusev, Los, Vlasenko, Zhun & Sheludyakov, 1984; Gusev, Antipin, Yufit, Struchkov, Sheludyakov, Zhun & Vlasenko, 1983; Glidewell & Liles, 1977, 1981). All of the disiloxanes whose structures have been determined differ in structure by at least one of the substituents on the silicon atoms. There has not been a previous example of X-ray diffraction studies where the difference in structures is remote to the silicon atom. Furthermore, as stated in the *Introduction*, (2) is unstrained. These structures illustrate the large number of conformations the siloxane may assume and the relative ease with which the Si—O—Si bond angle may be distorted.

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8-Cyclohexyl-16*H*-dinaphtho[2,1-*d*:1′,2′-*g*][1,3,2]dioxaphosphocine 8-Oxide

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Abstract. $C_{27}H_{25}O_3P$, $M_r = 428{\cdot}47$, triclinic, $P\bar{1}$, $a = 9{\cdot}5223\,(12)$, $b = 11{\cdot}1702\,(9)$, $c = 11{\cdot}4109\,(13)$ Å, $\alpha = 75{\cdot}938\,(11)$, $\beta = 76{\cdot}451\,(10)$, $\gamma = 68{\cdot}651\,(12)°$, $V = 1082{\cdot}2\,(2)$ Å^3, $Z = 2$, $D_m = 1{\cdot}32$, $D_x = 1{\cdot}315$ g cm^{-3}, λ(Cu $K\alpha$) = 1·5418 Å, $\mu = 13{\cdot}2$ cm^{-1}, $F(000) = 452$, $T = 298$ K, final $R = 0{\cdot}055$ for 1937 observed reflections. The dioxaphosphocine ring exhibits a distorted boat conformation with the phosphoryl O

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