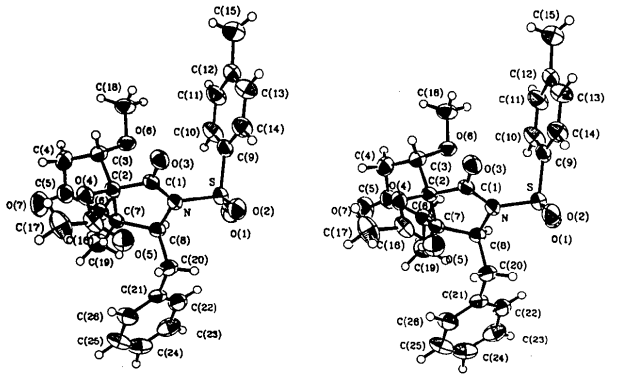
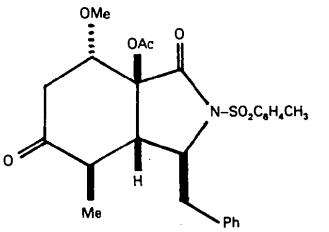


Fig. 1. Stereoview of the molecule (one enantiomorph; the crystals are racemic) with crystallographic numbering scheme.

$0{\cdot}35\tan\theta)°$ at 1·3–10° min^{-1}, extended 25% on each side for background measurement, three standard reflections showed negligible variations in intensity, Lp but no absorption corrections, 5772 independent reflections measured, 3188 with $I \geq 3\sigma(I)$, where $\sigma^2(I) = S + 4(B_1 + B_2) + (0{\cdot}04I)^2$, S = scan, B_1 and B_2 = background counts. Structure by direct methods, refined by full-matrix least squares on F, H atoms from a difference map, $w = 1/\sigma^2(F)$, scattering factors from *International Tables for X-ray Crystallography* (1974), locally written, or locally modified versions of standard computer programs, final $R = 0{\cdot}036$, $wR = 0{\cdot}047$ for 3188 reflections, $S = 1{\cdot}84$, 316 parameters (non-H atoms, plus 116 H-atom parameters), $R = 0{\cdot}091$ for all 5772 reflections, $\Delta/\sigma = 0{\cdot}06$ (mean), 0·93 [maximum, for x of H(17*b*)], maximum final difference density −0·3 to 0·2 e $Å^{-3}$. Atomic parameters are in Table 1, bond lengths and angles in Table 2, and a view of the molecule in Fig. 1.*

Related literature. Bates & Ramaswamy (1983) describe the use of the title compound (I) in a possible synthetic route to cytochalasin mould metabolites.



(I)

We thank Dr G. S. Bates for crystals, the Natural Sciences and Engineering Research Council of Canada for financial support, and the University of British Columbia Computing Centre for assistance.

* Lists of anisotropic thermal parameters, hydrogen positions, bond lengths and angles involving H, torsion angles, and structure factors have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51050 (34 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Acta Cryst. (1988). C**44**, 1850–1851

Structure of *tert*-Butyldiphenylsilyl *tert*-Butyldiphenylsilanecarboxylate

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Abstract. (^{t}Bu)$Ph_2SiCOOSiPh_2(^t$Bu), $C_{33}H_{38}O_2Si_2$, M_r = 522·84, monoclinic, $C2/c$, a = 19·277 (3), b = 10·220 (1), c = 31·312 (4) Å, β = 103·30 (1)°, V = 6003·4 (13) $Å^3$, Z = 8, D_x = 1·157 g cm^{-3}, Mo $K\alpha_1$, λ = 0·70930 Å, μ = 1·4 cm^{-1}, $F(000)$ = 2240, T = 295 K, R = 0·038 for 3484 observed reflections. The molecule contains a central Si—C(O)—O—Si grouping, torsion angle 159·6 (1)°, Si—C = 1·924 (3), Si—O = 1·702 (2) Å, with planar geometry at the carboxyl C atom, and tetrahedral geometries at the Si atoms; molecular dimensions are within normal ranges.

Experimental. {100}, {111}, {001}, ±($\bar{1}$13) developed, 0·43 × 0·34 × 0·38 mm, Enraf–Nonius CAD-4F diffractometer, lattice parameters from 25 reflections with θ = 14–17°. Intensities for $\theta \leq 27{\cdot}5°$, hkl: −25 to 25, 0 to 13, 0 to 40, ω–2θ scan, ω-scan width $(0{\cdot}57 + 0{\cdot}35\tan\theta)°$ at 1·0–10° min^{-1}, extended 25% on each side for background measurement, standard reflections showed negligible variations in intensity, Lp but no absorption corrections, 6868 independent reflections measured, 3484 with $I \geq 3\sigma(I)$, where $\sigma^2(I) = S + 4(B_1 + B_2) + (0{\cdot}04I)^2$, S = scan, B_1 and

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Table 1. *Positional (fractional* $\times 10^4$, Si $\times 10^5$) *and equivalent isotropic thermal parameters* ($U \times 10^3$ Å^2), *with e.s.d.'s in parentheses*

	x	y	z	U_{eq}*
Si(1)	16145 (4)	27857 (8)	8260 (3)	34
Si(2)	36178 (4)	12249 (7)	16433 (3)	31
O(1)	2883 (1)	1575 (2)	1242 (1)	39
O(2)	2928 (1)	3749 (2)	1283 (1)	59
C(1)	2595 (1)	2776 (3)	1146 (1)	37
C(2)	1086 (1)	2198 (3)	1233 (1)	42
C(3)	1173 (2)	717 (3)	1289 (1)	65
C(4)	1355 (2)	2848 (4)	1683 (1)	63
C(5)	293 (2)	2520 (3)	1068 (1)	62
C(6)	1400 (1)	4517 (3)	643 (1)	38
C(7)	1528 (2)	5558 (3)	935 (1)	56
C(8)	1379 (2)	6836 (3)	799 (1)	68
C(9)	1108 (2)	7104 (4)	370 (2)	71
C(10)	974 (2)	6098 (4)	71 (1)	81
C(11)	1117 (2)	4824 (3)	209 (1)	60
C(12)	1496 (1)	1675 (3)	339 (1)	37
C(13)	2057 (2)	1041 (3)	217 (1)	53
C(14)	1949 (2)	218 (4)	−142 (1)	64
C(15)	1276 (2)	23 (3)	−392 (1)	59
C(16)	711 (2)	631 (3)	−286 (1)	57
C(17)	822 (2)	1448 (3)	77 (1)	48
C(18)	4463 (1)	1755 (3)	1482 (1)	39
C(19)	4636 (2)	3209 (3)	1524 (1)	64
C(20)	4390 (2)	1332 (3)	1004 (1)	63
C(21)	5094 (2)	1013 (3)	1773 (1)	62
C(22)	3591 (1)	−602 (3)	1644 (1)	37
C(23)	3230 (2)	−1313 (3)	1281 (1)	47
C(24)	3248 (2)	−2676 (4)	1277 (1)	65
C(25)	3624 (2)	−3335 (3)	1635 (2)	75
C(26)	3982 (2)	−2672 (4)	2002 (1)	69
C(27)	3961 (2)	−1324 (3)	2003 (1)	51
C(28)	3443 (1)	1857 (3)	2168 (1)	34
C(29)	3579 (2)	3132 (3)	2316 (1)	50
C(30)	3365 (2)	3591 (4)	2682 (1)	67
C(31)	3013 (2)	2787 (4)	2906 (1)	68
C(32)	2868 (2)	1525 (4)	2772 (1)	60
C(33)	3084 (2)	1060 (3)	2407 (1)	47

* $U_{eq} = \frac{1}{3} \times$ trace of the diagonalized **U** tensor.

B_2 = background counts. Structure by Patterson and Fourier methods, refined by full-matrix least squares on F, H atoms in calculated positions, $w = 1/\sigma^2(F)$, scattering factors from *International Tables for X-ray Crystallography* (1974), locally written, or locally modified versions of standard computer programs, final $R = 0.038$, $wR = 0.040$ for 3484 reflections, $S = 1.46$, 334 parameters, $R = 0.148$ for all 6868 reflections, $\Delta/\sigma = 0.06$ (mean), 0.29 (maximum), maximum final difference density −0.5 to 0.4 e Å^{-3}. Atomic parameters are in Table 1, bond lengths and angles in Table 2, and a view of the molecule in Fig. 1.*

Related literature. Acetyltriphenylsilane (Chieh & Trotter, 1969).

We thank Dr G. S. Bates for crystals, the Natural Sciences and Engineering Research Council of Canada for financial support and the University of British Columbia Computing Centre for assistance.

* Lists of anisotropic thermal parameters, hydrogen positions, torsion angles and structure factors have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51054 (35 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 2. *Bond lengths* (Å) *and angles* (°), *with e.s.d.'s in parentheses*

Si(1)–C(1)	1.924 (3)	C(12)–C(17)	1.388 (4)
Si(1)–C(2)	1.902 (3)	C(13)–C(14)	1.382 (4)
Si(1)–C(6)	1.876 (3)	C(14)–C(15)	1.367 (4)
Si(1)–C(12)	1.872 (3)	C(15)–C(16)	1.360 (4)
Si(2)–O(1)	1.702 (2)	C(16)–C(17)	1.385 (4)
Si(2)–C(18)	1.892 (3)	C(18)–C(19)	1.522 (4)
Si(2)–C(22)	1.868 (3)	C(18)–C(20)	1.535 (4)
Si(2)–C(28)	1.865 (3)	C(18)–C(21)	1.541 (4)
O(1)–C(1)	1.353 (3)	C(22)–C(23)	1.394 (4)
O(2)–C(1)	1.208 (3)	C(22)–C(27)	1.395 (4)
C(2)–C(3)	1.528 (4)	C(23)–C(24)	1.394 (4)
C(2)–C(4)	1.536 (4)	C(24)–C(25)	1.363 (5)
C(2)–C(5)	1.532 (4)	C(25)–C(26)	1.376 (5)
C(6)–C(7)	1.388 (4)	C(26)–C(27)	1.378 (4)
C(6)–C(11)	1.380 (4)	C(28)–C(29)	1.388 (4)
C(7)–C(8)	1.383 (4)	C(28)–C(33)	1.395 (4)
C(8)–C(9)	1.352 (5)	C(29)–C(30)	1.385 (4)
C(9)–C(10)	1.375 (5)	C(30)–C(31)	1.359 (5)
C(10)–C(11)	1.379 (5)	C(31)–C(32)	1.365 (5)
C(12)–C(13)	1.387 (4)	C(32)–C(33)	1.387 (4)

C(1)–Si(1)–C(2)	105.18 (13)	Si(1)–C(12)–C(13)	123.4 (2)
C(1)–Si(1)–C(6)	106.55 (13)	Si(1)–C(12)–C(17)	120.4 (2)
C(1)–Si(1)–C(12)	110.05 (13)	C(13)–C(12)–C(17)	116.2 (3)
C(2)–Si(1)–C(6)	112.84 (13)	C(12)–C(13)–C(14)	121.8 (3)
C(2)–Si(1)–C(12)	111.70 (13)	C(13)–C(14)–C(15)	120.0 (3)
C(6)–Si(1)–C(12)	110.25 (13)	C(14)–C(15)–C(16)	120.1 (3)
O(1)–Si(2)–C(18)	111.36 (12)	C(15)–C(16)–C(17)	119.6 (3)
O(1)–Si(2)–C(22)	101.11 (12)	C(12)–C(17)–C(16)	122.2 (3)
O(1)–Si(2)–C(28)	106.81 (11)	Si(2)–C(18)–C(19)	116.3 (2)
C(18)–Si(2)–C(22)	108.20 (12)	Si(2)–C(18)–C(20)	106.8 (2)
C(18)–Si(2)–C(28)	118.45 (13)	Si(2)–C(18)–C(21)	108.2 (2)
C(22)–Si(2)–C(28)	109.53 (13)	C(19)–C(18)–C(20)	109.1 (3)
Si(2)–O(1)–C(1)	125.7 (2)	C(19)–C(18)–C(21)	107.6 (2)
Si(1)–C(1)–O(1)	115.1 (2)	C(20)–C(18)–C(21)	108.7 (2)
Si(1)–C(1)–O(2)	124.0 (2)	Si(2)–C(22)–C(23)	121.9 (2)
O(1)–C(1)–O(2)	120.8 (2)	Si(2)–C(22)–C(27)	121.4 (2)
Si(1)–C(2)–C(3)	109.2 (2)	C(23)–C(22)–C(27)	116.6 (3)
Si(1)–C(2)–C(4)	111.0 (2)	C(22)–C(23)–C(24)	121.3 (3)
Si(1)–C(2)–C(5)	110.7 (2)	C(23)–C(24)–C(25)	119.8 (3)
C(3)–C(2)–C(4)	108.5 (3)	C(24)–C(25)–C(26)	120.8 (3)
C(3)–C(2)–C(5)	109.0 (3)	C(25)–C(26)–C(27)	119.0 (4)
C(4)–C(2)–C(5)	108.4 (3)	C(22)–C(27)–C(26)	122.5 (3)
Si(1)–C(6)–C(7)	121.9 (2)	Si(2)–C(28)–C(29)	124.2 (2)
Si(1)–C(6)–C(11)	121.7 (2)	Si(2)–C(28)–C(33)	118.9 (2)
C(7)–C(6)–C(11)	116.4 (3)	C(29)–C(28)–C(33)	116.5 (3)
C(6)–C(7)–C(8)	121.8 (3)	C(28)–C(29)–C(30)	121.7 (3)
C(7)–C(8)–C(9)	120.2 (4)	C(29)–C(30)–C(31)	120.1 (3)
C(8)–C(9)–C(10)	119.6 (3)	C(30)–C(31)–C(32)	120.2 (3)
C(9)–C(10)–C(11)	120.0 (3)	C(31)–C(32)–C(33)	119.9 (3)
C(6)–C(11)–C(10)	121.9 (3)	C(28)–C(33)–C(32)	121.6 (3)

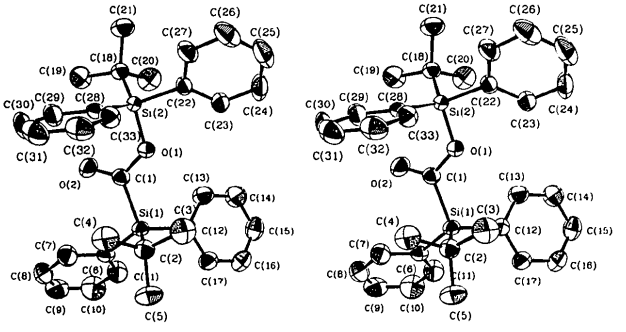

Fig. 1. Stereoview of the molecule.

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