

Table 3. Torsion angles (°)

The sign convention is as defined by Klyne & Prelog (1960).

C(102)-C(1)-C(2)-C(3)	1.5 (13)	C(6)-C(61)-C(101)-C(10)	-168.9 (7)	C(4)-C(41)-C(102)-C(1)	8.7 (11)
C(2)-C(1)-C(102)-C(41)	-7.6 (12)	C(7)-C(61)-C(101)-C(10)	60.2 (9)	O(5)-C(41)-C(102)-C(1)	-171.3 (7)
O(11)-C(1)-C(102)-C(41)	172.0 (7)	C(61)-C(7)-C(8)-C(9)	53.4 (10)	C(41)-O(5)-C(6)-C(61)	-48.8 (8)
C(2)-C(1)-O(11)-C(15)	-4.3 (12)	C(7)-C(8)-C(9)-C(14)	-175.7 (8)	C(41)-O(5)-C(6)-C(13)	-167.9 (7)
C(1)-C(2)-C(3)-C(4)	3.9 (13)	C(14)-C(9)-C(10)-C(101)	178.8 (7)	O(5)-C(6)-C(61)-C(101)	62.3 (8)
C(2)-C(3)-C(4)-C(41)	-2.8 (12)	C(9)-C(10)-C(101)-C(102)	176.3 (7)	C(12)-C(6)-C(61)-C(101)	-57.9 (11)
Br-C(4)-C(41)-C(102)	177.7 (6)	C(61)-C(101)-C(102)-C(41)	6.3 (11)	C(13)-C(6)-C(61)-C(101)	174.1 (8)
C(3)-C(4)-C(41)-C(102)	-3.8 (12)	C(10)-C(101)-C(102)-C(41)	129.4 (8)	C(101)-C(61)-C(7)-C(8)	-58.7 (9)
C(102)-C(41)-O(5)-C(6)	15.4 (10)	O(11)-C(1)-C(2)-C(3)	-178.0 (8)	C(6)-C(61)-C(101)-C(102)	-41.9 (10)
C(4)-C(41)-C(102)-C(101)	-172.1 (8)	C(2)-C(1)-C(102)-C(101)	173.2 (8)	C(7)-C(61)-C(101)-C(102)	-172.8 (7)
O(5)-C(41)-C(102)-C(101)	8.0 (12)	O(11)-C(1)-C(102)-C(101)	-7.3 (11)	C(7)-C(8)-C(9)-C(10)	-51.3 (11)
C(41)-O(5)-C(6)-C(12)	74.1 (9)	C(102)-C(1)-O(11)-C(15)	176.2 (8)	C(8)-C(9)-C(10)-C(101)	54.1 (9)
O(5)-C(6)-C(61)-C(7)	-169.7 (7)	C(2)-C(3)-C(4)-Br	175.7 (7)	C(9)-C(10)-C(101)-C(61)	-59.1 (9)
C(12)-C(6)-C(61)-C(7)	70.1 (10)	Br-C(4)-C(41)-O(5)	-2.3 (10)	C(61)-C(101)-C(102)-C(1)	-174.5 (8)
C(13)-C(6)-C(61)-C(7)	-57.9 (10)	C(3)-C(4)-C(41)-O(5)	176.1 (7)	C(10)-C(101)-C(102)-C(1)	-51.4 (10)
C(6)-C(61)-C(7)-C(8)	172.2 (7)	C(4)-C(41)-O(5)-C(6)	-164.6 (7)		

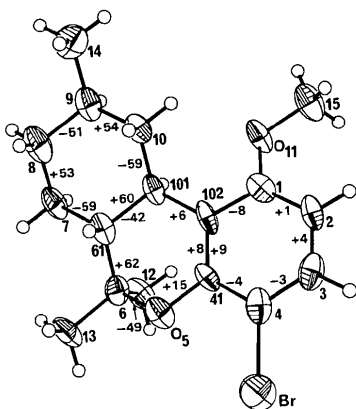


Fig. 1. The molecule of (II), showing selected ring torsion angles (°) (e.s.d.'s 1°) and the atom labelling.

We thank the Verband der Chemischen Industrie for financial support. Calculations and plotting were performed on an Eclipse S-250 minicomputer (which simultaneously controlled the diffractometer) with the *SHELXTL* system written by GMS; data were collected with the diffractometer control program written by Dr W. Clegg, University of Göttingen.

References

- BUCOURT, R. & HAINAUT, D. (1965). *Bull. Soc. Chim. Fr.* pp. 1366-1378.
 KLYNE, W. & PRELOG, V. (1960). *Experientia*, **16**, 521-523.
 TIETZE, L. F., VON KIEDROWSKI, G., HARMS, K., CLEGG, W. & SHELDRIK, G. M. (1980). *Angew. Chem.* **92**, 130-131.

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(2*S*,6*aR*,9*R*,10*aR*)-6,6,9-Trimethyl-1-oxo-2-phenylseleno-1,2,3,4,6*a*,7,8,9,10,10*a*-decahydro-6*H*-dibenzo[*b,d*]pyran

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Abstract. C₂₂H₂₈O₂Se, *M_r* = 405.38, monoclinic, *P*2₁, *a* = 10.298 (3), *b* = 9.915 (2), *c* = 10.835 (2) Å, β = 114.62 (4)°, *U* = 1004.2 Å³, *Z* = 4, *D_x* = 1.341 Mg m⁻³, μ(Mo *K*α) = 1.86 mm⁻¹. The structure was solved

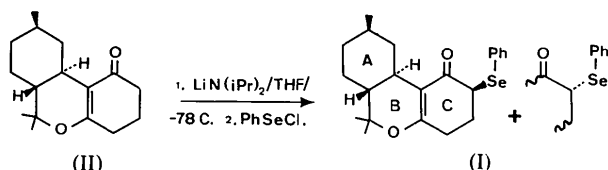
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with the aid of a novel Fourier-recycling procedure and refined to *R* = 0.0496 for 2388 independent data. The presence of the Se atom enabled the absolute configuration to be determined by refining both enantio-

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mers with complex scattering factors; the inverse structure converged to $R = 0.0708$.

Introduction. The sample of (I) was prepared as one of two isomers by the action of $\text{LiN}(i\text{-Pr})_2$ in THF at 195 K followed by PhSeCl on (6a*R*,9*R*,10a*R*)-6,6,9-trimethyl-1-oxo-1,2,3,4,6a,7,8,9,10,10a-decahydro-6*H*-dibenzo[*b,d*]pyran (II).



NMR spectra indicated that the two isomers differed in their configurations at C(2), the atom at which substitution had taken place. A crystal structure determination was, however, required to show which isomer adopted which configuration, and to establish the conformation of the cyclohexenone ring. The significant f'' for the Se atom (2.4 e) made possible the assignment of the absolute configuration of the product, and hence starting material, by X-ray diffraction. Both were optically active in solution.

The product crystallized out of diisopropyl ether/petroleum ether under argon. A crystal $0.3 \times 0.5 \times 0.3$ mm was sealed in a Lindemann-glass capillary. Since the compound was optically active the systematic absences determined the space group as $P2_1$. Data were collected on a Stoe-Siemens four-circle diffractometer with a control program written by Clegg (1981). Cell dimensions were obtained from diffractometer-angle measurements of 40 strong reflexions with $20 < 2\theta < 25^\circ$. With a view to the determination of absolute configuration, Friedel opposites were measured immediately after one another (to eliminate the effect of any long-term fluctuations), and in such a way that the directions of the incident and diffracted beams were interchanged (to cancel the effect of absorption to a first approximation, assuming a reasonably symmetrical crystal). Empirical absorption corrections based on measurements at different azimuthal angles only marginally improved the merging R index. 3070 reflexions were measured; equivalents (but not Friedel opposites) were merged to give 2388 unique data with $F > 4\sigma(F)$, which were used for all calculations, performed on a DG Eclipse minicomputer (which also controlled the diffractometer) with the *SHELXTL* system written by GMS.

The Se coordinates were deduced from the Patterson function; location of the remaining atoms by standard methods would have been difficult because a Fourier map phased by the Se atom alone contains a double image of the structure, with a mirror plane perpendicular to b . The problem was resolved by a novel E -Fourier-recycling procedure (Sheldrick, 1980) in which peaks were eliminated one at a time in order to

optimize an R index based on the agreement of point-atom structure factors with the observed E values. The remaining peaks were then used, together with the Se atom, to phase the next E map. Four cycles of this procedure led automatically to virtually the complete structure (excluding H). The structure was refined with

Table 1. Atom coordinates ($\times 10^4$) and isotropic temperature factors ($\text{\AA}^2 \times 10^3$)

	x	y	z	U or U_{eq}
Se	1763 (1)	2511 (2)	1822 (1)	71 (1)*
C(1)	843 (7)	874 (6)	-615 (6)	52 (3)*
C(2)	811 (7)	896 (7)	782 (6)	58 (3)*
H(2)	1311	104	1253	73
C(3)	-714 (7)	886 (7)	646 (7)	65 (3)*
H(3a)	-1144	35	274	75
H(3b)	-706	1001	1528	75
C(4)	-1594 (7)	2010 (6)	-278 (6)	58 (3)*
H(4a)	-1362	2840	223	63
H(4b)	-2587	1809	-551	63
C(41)	-1328 (7)	2184 (5)	-1531 (6)	51 (3)*
O(5)	-2429 (4)	2874 (4)	-2488 (4)	55 (2)*
C(6)	-2502 (7)	2903 (6)	-3878 (6)	58 (3)*
C(61)	-992 (7)	3104 (6)	-3771 (6)	55 (2)*
H(61)	-697	3942	-3291	63
C(7)	-888 (8)	3294 (7)	-5153 (7)	67 (3)*
H(7a)	-1498	4025	-5640	81
H(7b)	-1192	2480	-5675	81
C(8)	630 (8)	3607 (8)	-4925 (8)	73 (4)*
H(8a)	677	3641	-5791	89
H(8b)	886	4471	-4490	89
C(9)	1700 (6)	2568 (10)	-4045 (6)	66 (3)*
H(9)	1495	1720	-4515	81
C(10)	1550 (6)	2399 (9)	-2709 (6)	63 (3)*
H(10a)	2195	1708	-2181	74
H(10b)	1786	3235	-2219	74
C(101)	12 (7)	1996 (6)	-2960 (6)	51 (2)*
H(101)	-195	1160	-3453	57
C(102)	-187 (7)	1712 (6)	-1681 (6)	48 (2)*
O(11)	1676 (5)	137 (4)	-819 (5)	65 (2)*
C(12)	-3214 (9)	1598 (8)	-4560 (8)	82 (4)*
H(12a)	-3306	1658	-5477	93
H(12b)	-4133	1422	-4566	93
H(12c)	-2571	878	-4100	93
C(13)	-3441 (8)	4130 (9)	-4502 (7)	79 (3)*
H(13a)	-3658	4179	-5452	87
H(13b)	-2970	4944	-4062	87
H(13c)	-4310	4024	-4386	87
C(14)	3204 (8)	2930 (9)	-3812 (9)	90 (4)*
H(14a)	3865	2282	-3223	100
H(14b)	3391	3798	-3382	100
H(14c)	3313	2980	-4649	100
C(1')	3595 (6)	2563 (12)	1640 (5)	66 (3)*
C(2')	4311 (8)	1446 (9)	1568 (7)	82 (4)*
H(2')	3961	556	1600	86
C(3')	5585 (10)	1674 (11)	1439 (9)	106 (5)*
H(3')	6113	914	1341	122
C(4')	6094 (10)	2931 (13)	1444 (8)	106 (4)*
H(4')	6999	3056	1404	128
C(5')	5305 (10)	4032 (13)	1516 (9)	100 (4)*
H(5')	5662	4924	1508	123
C(6')	4018 (10)	3863 (10)	1590 (8)	98 (4)*
H(6')	3446	4619	1614	113

* U_{eq} is $\frac{1}{3}$ of the trace of the orthogonalized \mathbf{U} matrix.

isotropic riding H [$U(\text{H})$ was set to 1.2 times the equivalent U of the C atom to which the H was attached; C–H was fixed at 0.96 Å and H–C–H at 109.5°], and the remaining atoms anisotropic. Complex scattering factors and weights $w = 1/[\sigma^2(F) + 0.0005F^2]$ were employed. Refinement converged to R'

$= \sum w^{1/2} \Delta / \sum w^{1/2} |F_o| = 0.0532$ and corresponding $R = 0.0496$. The inverse structure refined to $R' = 0.0776$, $R = 0.0708$ and so could be rejected. Final atomic coordinates are given in Table 1,* bond lengths, angles and selected torsion angles in Tables 2 and 3.

Table 2. Bond lengths (Å) and angles (°)

C(1)–C(2)	1.528 (12)	C(1)–C(102)	1.459 (9)
C(1)–O(11)	1.216 (10)	C(2)–Se	1.969 (8)
C(2)–C(3)	1.516 (12)	C(3)–C(4)	1.519 (10)
C(4)–C(41)	1.503 (12)	C(41)–O(5)	1.360 (7)
C(6)–O(5)	1.476 (9)	C(6)–C(61)	1.523 (11)
C(61)–C(7)	1.556 (12)	C(7)–C(8)	1.510 (13)
C(8)–C(9)	1.520 (11)	C(10)–C(9)	1.527 (11)
C(10)–C(101)	1.543 (11)	C(101)–C(61)	1.514 (9)
C(101)–C(102)	1.510 (11)	C(102)–C(41)	1.336 (12)
C(12)–C(6)	1.518 (11)	C(13)–C(6)	1.526 (11)
C(14)–C(9)	1.505 (12)	C(1')–Se	1.977 (8)
C(1')–C(2')	1.351 (15)	C(1')–C(6')	1.369 (16)
C(2')–C(3')	1.395 (16)	C(3')–C(4')	1.351 (18)
C(4')–C(5')	1.382 (18)	C(5')–C(6')	1.371 (16)
C(2)–Se–C(1')	103.4 (5)	C(2)–C(1)–C(102)	117.9 (7)
C(2)–C(1)–O(11)	119.8 (6)	C(102)–C(1)–O(11)	122.3 (8)
Se–C(2)–C(1)	111.6 (6)	Se–C(2)–C(3)	108.0 (6)
C(1)–C(2)–C(3)	110.6 (6)	C(2)–C(3)–C(4)	111.7 (8)
C(3)–C(4)–C(41)	113.4 (7)	C(4)–C(41)–O(5)	109.8 (7)
C(4)–C(41)–C(102)	124.7 (6)	O(5)–C(41)–C(102)	125.5 (7)
C(41)–O(5)–C(6)	117.3 (6)	O(5)–C(6)–C(61)	108.0 (5)
O(5)–C(6)–C(12)	106.8 (7)	C(61)–C(6)–C(12)	114.7 (7)
O(5)–C(6)–C(13)	102.7 (7)	C(61)–C(6)–C(13)	111.6 (7)
C(12)–C(6)–C(13)	112.0 (6)	C(6)–C(61)–C(7)	114.8 (6)
C(6)–C(61)–C(101)	112.2 (7)	C(7)–C(61)–C(101)	110.0 (7)
C(61)–C(7)–C(8)	110.4 (6)	C(7)–C(8)–C(9)	113.2 (8)
C(8)–C(9)–C(10)	109.6 (8)	C(8)–C(9)–C(14)	111.7 (8)
C(10)–C(9)–C(14)	111.8 (6)	C(9)–C(10)–C(101)	111.3 (6)
C(61)–C(101)–C(102)	108.5 (7)	C(61)–C(101)–C(102)	110.3 (7)
C(10)–C(101)–C(102)	114.1 (6)	C(1)–C(102)–C(41)	119.3 (7)
C(1)–C(102)–C(101)	120.1 (7)	C(41)–C(102)–C(101)	120.6 (6)
Se–C(1')–C(2')	123.4 (9)	Se–C(1')–C(6')	111.2 (8)
C(2')–C(1')–C(6')	125.4 (9)	C(1')–C(2')–C(3')	115.6 (10)
C(2')–C(3')–C(4')	121.9 (12)	C(3')–C(4')–C(5')	119.5 (12)
C(4')–C(5')–C(6')	120.8 (12)	C(1')–C(6')–C(5')	116.6 (11)

Discussion. A view of the molecule is shown in Fig. 1. The absolute configuration agrees with that expected chemically for the starting material. The cyclohexane ring *A* adopts the chair conformation, and dihydropyran ring *B* a half-chair, with maximum deviations from ideal torsion angles 4.8 and 3.8° respectively.

* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 35579 (17 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

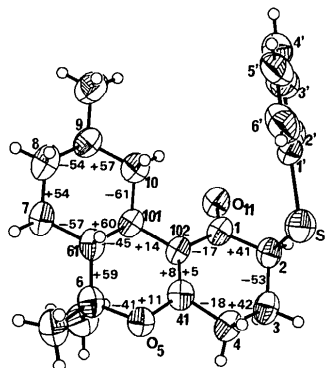


Fig. 1. Compound (I), with 50% probability thermal ellipsoids for non-hydrogen atoms, and the torsion angles (°) in the ring.

Table 3. Torsion angles (°)

The sign convention is as defined by Klyne & Prelog (1960).

C(1')–Se–C(2)–C(1)	–48.6 (6)	C(61)–C(7)–C(8)–C(9)	54.5 (9)	C(4)–C(41)–C(102)–C(1)	5.0 (10)
C(2)–Se–C(1')–C(2')	–33.8 (6)	C(7)–C(8)–C(9)–C(14)	–178.7 (8)	O(5)–C(41)–C(102)–C(1)	–173.9 (6)
C(102)–C(1)–C(2)–Se	–78.9 (7)	C(14)–C(9)–C(10)–C(101)	–178.7 (8)	C(41)–O(5)–C(6)–C(61)	–41.4 (7)
O(11)–C(1)–C(2)–Se	103.6 (7)	C(9)–C(10)–C(101)–C(102)	175.8 (7)	C(41)–O(5)–C(6)–C(13)	–159.4 (6)
C(2)–C(1)–C(102)–C(41)	–17.0 (9)	C(61)–C(101)–C(102)–C(41)	14.1 (8)	O(5)–C(6)–C(61)–C(101)	58.9 (7)
O(11)–C(1)–C(102)–C(41)	160.4 (7)	C(10)–C(101)–C(102)–C(41)	136.5 (7)	C(12)–C(6)–C(61)–C(101)	–60.1 (8)
Se–C(2)–C(3)–C(4)	69.1 (7)	C(6')–C(1')–C(2')–C(3')	0.3 (11)	C(13)–C(6)–C(61)–C(101)	171.1 (6)
C(2)–C(3)–C(4)–C(41)	42.3 (8)	C(2')–C(1')–C(6')–C(5')	–2.7 (12)	C(101)–C(61)–C(7)–C(8)	–57.3 (8)
C(3)–C(4)–C(41)–C(102)	–18.3 (9)	C(2')–C(3')–C(4')–C(5')	–3.1 (13)	C(6)–C(61)–C(101)–C(102)	–45.3 (7)
C(102)–C(41)–O(5)–C(6)	11.2 (9)	C(4')–C(5')–C(6')–C(1')	2.3 (13)	C(7)–(61)–C(101)–C(102)	–174.5 (6)
C(4)–C(41)–C(102)–C(101)	–177.3 (6)	C(1')–Se–C(2)–C(3)	–170.4 (6)	C(7)–C(8)–C(9)–C(10)	–54.2 (9)
O(5)–C(41)–C(102)–C(101)	3.7 (10)	C(2)–Se–C(1')–C(6')	145.8 (6)	C(8)–C(9)–C(10)–C(101)	56.9 (10)
C(41)–O(5)–C(6)–C(12)	82.6 (7)	C(102)–C(1)–C(2)–C(3)	41.4 (9)	C(9)–C(10)–C(101)–C(61)	–60.8 (9)
O(5)–C(6)–C(61)–C(7)	–174.5 (6)	O(11)–C(1)–C(2)–C(3)	–136.1 (7)	C(61)–C(101)–C(102)–C(1)	–168.3 (6)
C(12)–C(6)–C(61)–C(7)	66.5 (8)	C(2)–C(1)–C(102)–C(101)	165.3 (6)	C(10)–C(101)–C(102)–C(1)	–45.9 (9)
C(13)–C(6)–C(61)–C(7)	–62.3 (8)	O(11)–C(1)–C(102)–C(101)	–17.3 (10)	Se–C(1')–C(2')–C(3')	179.9 (6)
C(6)–C(61)–C(7)–C(8)	175.0 (7)	C(1)–C(2)–C(3)–C(4)	–53.3 (8)	Se–C(1')–C(6')–C(5')	177.6 (7)
C(6)–C(61)–C(101)–C(10)	–171.0 (6)	C(3)–C(4)–C(41)–O(5)	160.8 (6)	C(1')–C(2')–C(3')–C(4')	2.7 (12)
C(7)–C(61)–C(101)–C(10)	59.9 (7)	C(4)–C(41)–O(5)–C(6)	–167.9 (6)	C(3')–C(4')–C(5')–C(6')	0.4 (13)

The methyl substituent on C(9) is equatorial; C(9) has the *R* configuration according to the notation of Cahn, Ingold & Prelog (1966). The cyclohexenone ring *C* is approximately a half-chair (the maximum deviation from ideal cyclohexene half-chair torsion angles is 8.7°), with an axial phenylselenyl substituent. This conformation minimizes repulsion between the Se atom and the carbonyl group. The plane of the phenyl ring is at right angles to that of the molecular skeleton, which enables efficient molecular packing.

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References

- CAHN, R. S., INGOLD, C. K. & PRELOG, V. (1966). *Angew. Chem.* **78**, 413–447.
 CLEGG, W. (1981). *Acta Cryst.* **A37**. In the press.
 KLYNE, W. & PRELOG, V. (1960). *Experientia.* **16**, 521–523.
 SHELDRICK, G. M. (1980). To be published.

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Dodeca(phenylsilasesquioxane)*

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Abstract. C₇₂H₆₀O₁₈Si₁₂, *M_r* = 1550.3, tetragonal, *P4/n*, *a* = 17.449 (4), *c* = 14.156 (4) Å, *U* = 4310.0 Å³, *D_x* = 1.194 Mg m⁻³, *Z* = 2, *F*(000) = 1608; final *R* = 0.077 for 1666 unique reflexions. The (PhSi)₁₂O₁₈ molecule contains four ten-membered and four eight-membered silicon–oxygen rings and has crystallographic 4 (*S₄*) symmetry. The Si₁₂O₁₈ core approximates to 42*m* (*D_{2d}*) symmetry.

with *I* > 2σ(*I*); the discrepancy index for equivalent reflexions was 0.067.

The structure was solved by direct methods, and refined to a minimum value of ∑ *w*Δ² [Δ = |*F_o*| –

Table 1. *Atomic coordinates* (×10⁴) *and equivalent isotropic thermal parameters* (Å² × 10⁴)

U_{eq} = 1/3 of the trace of the orthogonalized *U* matrix.

	<i>x</i>	<i>y</i>	<i>z</i>	<i>U_{eq}</i>
Si(1)	7745 (1)	4178 (1)	5953 (2)	394 (7)
Si(2)	6981 (1)	4119 (1)	4003 (2)	408 (7)
Si(3)	8095 (1)	3184 (1)	2724 (2)	408 (7)
O(1)	7355 (3)	4427 (3)	4973 (3)	450 (18)
O(2)	7650 (3)	3855 (3)	3289 (4)	485 (20)
O(3)	8799 (3)	2881 (3)	3350 (4)	495 (20)
O(4)	6464 (3)	3381 (3)	4274 (3)	513 (19)
O(5)	7500	2500	2508 (5)	507 (29)
C(11)	8158 (4)	5037 (4)	6507 (6)	459 (30)
C(12)	8281 (5)	5074 (6)	7463 (7)	752 (43)
C(13)	8581 (7)	5723 (8)	7888 (9)	1026 (55)
C(14)	8770 (7)	6329 (7)	7372 (12)	1038 (61)
C(15)	8664 (7)	6314 (6)	6416 (11)	1121 (60)
C(16)	8350 (6)	5675 (5)	5985 (8)	801 (40)
C(21)	6410 (5)	4875 (5)	3423 (6)	564 (34)
C(22)	5794 (6)	5184 (6)	3890 (9)	1036 (53)
C(23)	5355 (7)	5779 (8)	3400 (12)	1293 (72)
C(24)	5516 (9)	5999 (8)	2547 (10)	1325 (71)
C(25)	6091 (8)	5636 (7)	2078 (8)	1203 (60)
C(26)	6509 (7)	5080 (7)	2513 (8)	1070 (53)
C(31)	8470 (4)	3558 (4)	1599 (5)	412 (27)
C(32)	7990 (5)	3757 (6)	858 (7)	701 (39)
C(33)	8241 (6)	4056 (6)	34 (7)	899 (46)
C(34)	9013 (6)	4164 (6)	–91 (7)	845 (46)
C(35)	9503 (6)	3987 (6)	608 (7)	835 (46)
C(36)	9235 (5)	3690 (5)	1452 (6)	639 (36)

Introduction. Very small crystals of this compound were obtained in small quantities from an attempt to recrystallize the silicon–nitrogen ring compound (PhSiF.NMe)₃ from acetone. The nature of the material obtained was established by a preliminary crystal structure determination based on data collected at high speed from a crystal of dimensions *ca* 0.1 mm. A second data set was subsequently obtained from a slightly larger crystal, including measurements of several sets of equivalent reflexions. All measurements were made with a Stoe–Siemens AED diffractometer (Clegg, 1981) and graphite-monochromated Mo *K*α radiation (λ = 0.71069 Å). 11444 intensities were obtained in the range 7 < 2θ < 50°. Cell dimensions were derived from the setting angles of 42 automatically centred reflexions.

Equivalent reflexions were averaged and the two data sets combined, without application of absorption corrections (μ = 0.23 mm⁻¹), to give 1666 unique data

* 1,3,5,7,9,11,13,15,17,19,21,23-Dodecaphenylheptacyclo-[11.11.1.1^{3,9}.1^{5,23}.1^{7,19}.1^{11,17}.1^{15,21}]dodecasiloxane.