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The Crystal and Molecular Structure of Decaphenylcyclopentasilane, $C_{60}H_{50}Si_5$

BY LÁSZLÓ PÁRKÁNYI

Central Research Institute for Chemistry of the Hungarian Academy of Sciences, H-1525-Budapest, POB 17, Hungary

KÁLMÁN SASVÁRI*

Physics Department, University of York, York YO1 5DD, England

AND JEAN PAUL DECLERCQ AND GABRIEL GERMAIN

Laboratory of Chemical Physics and Crystallography, University of Louvain, B-1348 Louvain-la-Neuve, Belgium

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$C_{60}H_{50}Si_5$ is monoclinic, space group $C2/c$, with $a = 39{\cdot}721\ (8)$, $b = 12{\cdot}862\ (3)$, $c = 22{\cdot}701\ (4)$ Å, $\beta = 99{\cdot}62\ (2)°$, $Z = 8$. The structure was refined to $R = 0{\cdot}113$ for 5504 non-zero reflections. The nucleus of the molecule is a five-membered Si ring puckered in a form intermediate between C_s and C_2 symmetry. The Si–Si lengths vary between 2·371 and 2·413 Å (average 2·396 Å) and the Si–Si–Si angles between 102·7 and 106·7° (average 104·5°). The Si–C lengths range from 1·869 to 1·917 Å (average 1·895 Å). The planes of the Si–C bond pairs of the $SiPh_2$ groups are almost perpendicular to the adjoining Si–Si–Si plane with a maximum deviation of 10·5°.

Introduction

The nucleus of decaphenylcyclopentasilane (DPHCPSI) is a five-membered Si ring similar to gaseous cyclopentasilane, Si_5H_{10}, which has been studied by electron diffraction (Smith, Seip, Hengge & Bauer, 1976). An analysis of the decaphenyl derivative was carried out to determine the conformation of the cyclopentasilane ring and the Si–Si lengths in a system where ring strain is likely to be greater than in cyclopentasilane but smaller than in octaphenylcyclotetrasilane (OPHCTSI: Párkányi, Sasvári & Barta, 1978).

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* Present address: Central Research Institute for Chemistry of the Hungarian Academy of Sciences, H-1525-Budapest, POB 17, Hungary.

Experimental

Crystals were obtained from benzene/ethanol as thin transparent plates. Crystal data are: a = 39·721 (8), b = 12·862 (3), c = 22·701 (4) Å, β = 99·62 (2)°, V = 11 434·7 Å^3, $C_{60}H_{50}Si_5$, M_r = 911·483, D_x = 1·059, D_m = 1·04 g cm^{-3}, μ(Mo $K\alpha$, λ = 0·71069 Å) = 1·60 cm^{-1}, space group $C2/c$, Z = 8. The intensities of 6125 independent reflections, of which 2089 were unobserved [$F_o \leq 5\sigma(F_o)$], were collected on a Syntex $P2_1$ automatic four-circle diffractometer with monochromated Mo $K\alpha$ radiation. 621 reflections with $I \leq 0$ were omitted from the refinement to reduce computing time. Cell parameters and their e.s.d.'s were calculated from diffractometer data by least squares. The space group was determined from systematic absences. After data reduction the absolute scale and approximate temperature factor (B = 3·7 Å^2) were obtained from a Wilson plot using all reflexions. No absorption correction was made.

Determination and refinement of the structure

The phase problem was solved with *MULTAN* 77 (Main, Woolfson, Lessinger, Germain & Declercq, 1977). E values were calculated from the molecular scattering factors of the SiPh groups and Si_5 ring (Main, 1976); the K curve (Karle, Hauptman & Christ, 1958) was applied in the scaling procedure. 400 reflections ($|E| \geq 1·83$) and 3200 sign relationships

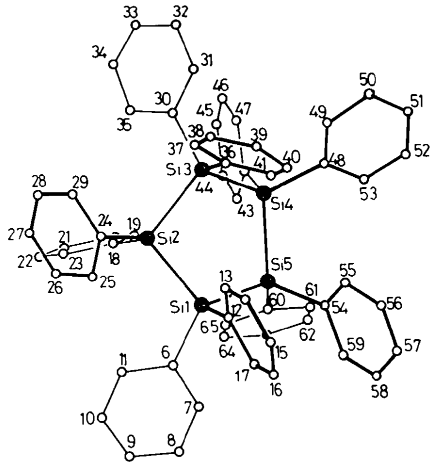

Fig. 1. A view of the molecule, with the atomic numbering, projected on to the best plane of the cyclopentasilane ring. H atoms are omitted for clarity.

Table 1. *Final fractional coordinates* (× 10^4) *for the non-hydrogen atoms*

	x	y	z		x	y	z
Si(1)	1913 (1)	5760 (2)	1379 (1)	C(34)	786 (4)	4211 (15)	3457 (7)
Si(2)	1744 (1)	5422 (2)	2314 (1)	C(35)	1002 (4)	4558 (12)	3055 (6)
Si(3)	1162 (1)	5967 (3)	2151 (1)	C(36)	1149 (3)	7427 (9)	2200 (6)
Si(4)	937 (1)	5328 (3)	1169 (1)	C(37)	1232 (4)	7901 (11)	2740 (7)
Si(5)	1420 (1)	5339 (3)	650 (1)	C(38)	1214 (5)	8977 (15)	2808 (10)
C(6)	2312 (3)	5029 (9)	1267 (5)	C(39)	1122 (5)	9576 (14)	2300 (11)
C(7)	2400 (3)	4893 (10)	710 (5)	C(40)	1052 (7)	9136 (16)	1755 (10)
C(8)	2706 (3)	4490 (10)	622 (6)	C(41)	1065 (5)	8045 (12)	1702 (8)
C(9)	2938 (3)	4198 (10)	1115 (6)	C(42)	750 (3)	3979 (10)	1273 (5)
C(10)	2858 (3)	4290 (10)	1680 (6)	C(43)	844 (3)	3055 (11)	1019 (6)
C(11)	2552 (3)	4693 (9)	1757 (5)	C(44)	697 (4)	2114 (12)	1127 (7)
C(12)	2006 (3)	7177 (9)	1267 (5)	C(45)	431 (4)	2112 (13)	1481 (7)
C(13)	1932 (3)	7962 (9)	1647 (5)	C(46)	339 (4)	2982 (14)	1732 (7)
C(14)	2005 (4)	9013 (11)	1532 (7)	C(47)	496 (4)	3903 (12)	1619 (6)
C(15)	2153 (4)	9281 (11)	1052 (7)	C(48)	552 (3)	6083 (10)	769 (5)
C(16)	2239 (4)	8496 (12)	671 (7)	C(49)	351 (5)	6636 (18)	1060 (7)
C(17)	2172 (4)	7471 (10)	784 (6)	C(50)	35 (5)	7042 (20)	787 (9)
C(18)	1787 (3)	3961 (9)	2435 (5)	C(51)	−56 (4)	6949 (16)	208 (8)
C(19)	1558 (3)	3272 (11)	2073 (6)	C(52)	143 (6)	6388 (22)	−91 (8)
C(20)	1585 (4)	2224 (11)	2142 (7)	C(53)	439 (4)	5973 (17)	181 (7)
C(21)	1817 (4)	1788 (10)	2577 (6)	C(54)	1399 (3)	6404 (11)	64 (5)
C(22)	2052 (3)	2436 (11)	2934 (5)	C(55)	1266 (4)	7373 (11)	127 (7)
C(23)	2029 (3)	3525 (9)	2860 (5)	C(56)	1309 (5)	8185 (14)	−265 (9)
C(24)	2006 (3)	6055 (9)	2983 (5)	C(57)	1487 (5)	8000 (15)	−739 (8)
C(25)	2323 (3)	6532 (9)	2967 (5)	C(58)	1618 (5)	7007 (14)	−812 (7)
C(26)	2518 (3)	6911 (9)	3479 (6)	C(59)	1579 (4)	6231 (12)	−424 (6)
C(27)	2410 (3)	6846 (10)	4023 (5)	C(60)	1440 (3)	4026 (10)	263 (5)
C(28)	2095 (3)	6414 (10)	4057 (5)	C(61)	1216 (3)	3849 (11)	−265 (6)
C(29)	1892 (3)	5996 (10)	3543 (5)	C(62)	1199 (4)	2834 (15)	−497 (7)
C(30)	914 (3)	5478 (10)	2725 (5)	C(63)	1398 (4)	2027 (12)	−239 (8)
C(31)	612 (4)	6044 (15)	2806 (7)	C(64)	1623 (4)	2225 (11)	276 (7)
C(32)	404 (4)	5631 (19)	3203 (8)	C(65)	1646 (3)	3201 (10)	524 (6)
C(33)	491 (4)	4713 (19)	3509 (7)				

were used. The E map calculated with the sign set of the highest combined figure of merit revealed the positions of all Si and 23 C atoms. A weighted Fourier synthesis calculated with the phases obtained from the previous 28 atoms revealed all the remaining non-hydrogen atoms. The parameters of the non-hydrogen atoms were refined by anisotropic least squares. *SHELX* 77 (Sheldrick, 1977) was used first, but as a convergent refinement could only be achieved with unit weights, refinement was repeated with XRAY 72 (Stewart, Kruger, Ammon, Dickinson & Hall, 1972) and Cruickshank's (1961) weighting scheme. The H atoms were generated geometrically and were included in fixed positions. The final structure factor calculation gave R values of 0·095 and 0·113 for 4036 observed and 5504 non-zero reflections respectively. The final coordinates of the non-hydrogen atoms are listed in Table 1. The scattering factors were from Cromer & Mann (1968).*

Discussion

The molecule with the numbering of the atoms is shown in Fig. 1. Relevant bond lengths and angles are given in Tables 2 and 3.

The nucleus of the molecule is a puckered five-membered Si ring. The puckering is indicated by the endocyclic torsion angles (Table 4). The ring conformation is intermediate between C_s and C_2 symmetry with the following asymmetry parameters (Duax, Weeks & Rohrer, 1976): $\Delta C_s[\mathrm{Si}(2)] = 9·0$ and $\Delta C_2[\mathrm{Si}(5)] = 9·3$; the pseudorotation phase angle (Altona, Geise & Romers, 1968) $\Delta = -15·9°$ [if φ_0 is assigned to the rotation about Si(2)–Si(3)].

The Si–Si lengths range from 2·371 to 2·413 Å (average 2·395 Å), which is somewhat longer than in 1,2,3,4-tetra-*tert*-butyltetramethylcyclotetrasilane (2·377 Å; Hurt, Calabrese & West, 1975) and in OPHCTSI (2·377 Å; Párkányi *et al.*, 1978), where these bonds have equal lengths. These findings seem to agree with the results of quantum-chemical calculations (Nagy, 1977); *i.e.* the Si–Si bond order in polysilanes decreases with the degree of oligomerization. This conclusion is not supported, however, by the considerably shorter Si–Si bond (2·342 Å) found in gaseous cyclopentasilane (Smith *et al.*, 1976). This latter value is similar to results obtained for Si–Si bonds in disilanes studied by electron diffraction (2·340 Å; Beagley, Monaghan & Hewitt, 1971) and by microwave spectroscopy (2·332 Å; Cox & Varma, 1966). The Si–Si–Si angles vary between 102·7 and 106·7° (average 104·5°), which agrees well with the mean value (104·2°) reported for cyclopentasilane.

The Si···Si non-bonded distances (Table 5) are considerably longer than those in OPHCTSI (3·35 Å) because of ring puckering. The planes of the Si–C bond pairs at each Si atom are nearly perpendicular to the corresponding Si–Si–Si plane or to the best plane of the Si$_5$ ring (Table 6). For all Si–C bond pairs the

Table 2. *Relevant bond lengths* (Å) *with their e.s.d.'s in parentheses*

Si(1)–Si(2)	2·371 (3)	Si(2)–C(24)	1·877 (11)
Si(2)–Si(3)	2·385 (5)	Si(3)–C(30)	1·869 (12)
Si(3)–Si(4)	2·404 (3)	Si(3)–C(36)	1·882 (12)
Si(4)–Si(5)	2·413 (5)	Si(4)–C(42)	1·917 (13)
Si(5)–Si(1)	2·405 (4)	Si(4)–C(48)	1·908 (12)
Si(1)–C(6)	1·896 (12)	Si(5)–C(54)	1·902 (13)
Si(1)–C(12)	1·885 (11)	Si(5)–C(60)	1·911 (13)
Si(2)–C(18)	1·903 (11)		

Table 3. *Relevant bond angles* (°) *with their e.s.d.'s in parentheses*

Si(5)–Si(1)–Si(2)	104·8 (2)	Si(1)–Si(2)–C(24)	116·3 (7)
Si(1)–Si(2)–Si(3)	102·7 (3)	Si(3)–Si(2)–C(18)	112·0 (7)
Si(2)–Si(3)–Si(4)	104·1 (3)	Si(3)–Si(2)–C(24)	113·0 (7)
Si(3)–Si(4)–Si(5)	104·4 (2)	Si(2)–Si(3)–C(30)	113·8 (8)
Si(4)–Si(5)–Si(1)	106·7 (3)	Si(2)–Si(3)–C(36)	108·6 (7)
C(6)–Si(1)–C(12)	105·9 (10)	Si(4)–Si(3)–C(30)	112·0 (7)
C(18)–Si(2)–C(24)	106·7 (10)	Si(4)–Si(3)–C(36)	112·7 (7)
C(30)–Si(3)–C(36)	105·8 (11)	Si(3)–Si(4)–C(42)	106·7 (7)
C(42)–Si(4)–C(48)	102·9 (11)	Si(3)–Si(4)–C(48)	114·3 (7)
C(54)–Si(5)–C(60)	108·4 (12)	Si(5)–Si(4)–C(42)	114·9 (8)
Si(5)–Si(1)–C(6)	113·5 (7)	Si(5)–Si(4)–C(48)	113·8 (8)
Si(5)–Si(1)–C(12)	106·3 (7)	Si(4)–Si(5)–C(54)	113·6 (8)
Si(2)–Si(1)–C(6)	113·4 (8)	Si(4)–Si(5)–C(60)	108·5 (7)
Si(2)–Si(1)–C(12)	112·9 (7)	Si(1)–Si(5)–C(54)	104·8 (7)
Si(1)–Si(2)–C(18)	106·2 (7)	Si(1)–Si(5)–C(60)	115·1 (7)

Table 4. *Torsion angles* (°) *in the* Si$_5$ *ring*

Si(1)–Si(2)–Si(3)–Si(4)	41·9
Si(2)–Si(3)–Si(4)–Si(5)	−30·2
Si(3)–Si(4)–Si(5)–Si(1)	7·3
Si(4)–Si(5)–Si(1)–Si(2)	18·4
Si(5)–Si(1)–Si(2)–Si(3)	−37·0

Table 5. Si–Si *non-bonded distances* (Å) *and their inclination angles* (°) *in the* Si$_5$ *ring*

Si(1)–Si(3)	3·713	Si(1)–Si(3)–Si(5)	37·3
Si(3)–Si(5)	3·805	Si(3)–Si(5)–Si(2)	36·6
Si(5)–Si(2)	3·783	Si(5)–Si(2)–Si(4)	37·2
Si(2)–Si(4)	3·776	Si(2)–Si(4)–Si(1)	36·1
Si(4)–Si(1)	3·866	Si(4)–Si(1)–Si(3)	36·9

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* Lists of structure factors, hydrogen atomic coordinates, full tables of bond lengths and angles, and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 33858 (38 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

inclination angles of the Si—C bonds with respect to the corresponding Si—Si—Si plane are different on the two sides of the Si_5 ring and in an opposite sense at sequential Si atoms, with the exception of those at Si(1) and Si(5) (Table 7). This feature is similar to that found in OPHCTSI where the differences of the angles are as high as 28°; in the present molecule they are much lower and vary between 4 and 10°. This is a direct consequence of the longer Si···Si distances; *i.e.* the phenyl rings are pushed further from the center of the Si_5 ring and will therefore have the same close packing on both sides of the ring with less movement in the phenyl positions. Puckering may also decrease ring strain in the cyclopentasilane ring; thus the lengths of the Si—Si bonds are probably not influenced by strain (*cf.* bond lengths in OPHCTSI).

The Si—C distances (mean 1·895 Å, with a maximum deviation of 0·26 Å) fall in the range of reported values (1·84–1·93 Å). The phenyl rings are planar and are tilted to the corresponding Si—C axis by angles ranging from 0·3 to 9·1° (Table 8). Bond lengths in the phenyl rings are rather distorted but the results do not justify discussion. The angles between the phenyl planes in the $SiPh_2$ moieties fall in the range 68·1–88·7° (Table 9) and show greater differences than those in OPHCTSI (78·2–84·1°).

Table 6. *Dihedral angles* (°) *of the* C—Si—C *planes* (*a*) *with the corresponding* Si—Si—Si *planes and* (*b*) *with the least-squares plane of the* Si_5 *ring*

		(*a*)	(*b*)
C(6)—Si(1)—C(12)	Si(5)—Si(1)—Si(2)	87·3	91·9
C(18)—Si(2)—C(24)	Si(1)—Si(2)—Si(3)	86·5	87·8
C(30)—Si(3)—C(36)	Si(2)—Si(3)—Si(4)	79·5	95·9
C(42)—Si(4)—C(48)	Si(3)—Si(4)—Si(5)	86·5	79·6
C(54)—Si(5)—C(60)	Si(4)—Si(5)—Si(1)	95·6	88·8

Table 7. *Inclination angles* (°) *of the* Si—C *bond pairs to the corresponding* Si—Si—Si *planes*

Si(5)—Si(1)—Si(2)	Si(1)—C(6)	−49·3
	Si(1)—C(12)	56·5
Si(1)—Si(2)—Si(3)	Si(2)—C(18)	−58·3
	Si(2)—C(24)	48·1
Si(2)—Si(3)—Si(4)	Si(3)—C(30)	−50·8
	Si(3)—C(36)	54·9
Si(3)—Si(4)—Si(5)	Si(4)—C(42)	−54·3
	Si(4)—C(48)	48·3
Si(4)—Si(5)—Si(1)	Si(5)—C(54)	56·2
	Si(5)—C(60)	−51·4

Table 8. *The inclination angles* (°) *of* Si—C *bonds to their phenyl planes*

Si(1)—C(6)	C(6)···C(11)	7·7
Si(1)—C(12)	C(12)···C(17)	1·2
Si(2)—C(18)	C(18)···C(23)	0·4
Si(2)—C(24)	C(24)···C(29)	4·3
Si(3)—C(30)	C(30)···C(35)	4·8
Si(3)—C(36)	C(36)···C(41)	1·7
Si(4)—C(42)	C(42)···C(47)	0·3
Si(4)—C(48)	C(48)···C(53)	8·0
Si(5)—C(54)	C(54)···C(59)	9·1
Si(5)—C(60)	C(60)···C(65)	7·7

Table 9. *Dihedral angles* (°) *of the phenyl planes on the same* Si *atom*

C(6)···C(11)	C(12)···C(17)	74·3
C(18)···C(23)	C(24)···C(29)	75·4
C(30)···C(35)	C(36)···C(41)	69·2
C(42)···C(47)	C(48)···C(53)	88·7
C(54)···C(59)	C(60)···C(65)	68·1

Table 10. *Non bonded* H···H *distances* <2·50 Å

The fractional atomic coordinates (×10^3) are given in square brackets.

H(7)[223, 510, 35]···H(17)[221, 691, 50]	2·36
H(31)[55, 671, 258]···H(49)[43, 677, 150]	2·45
H(32)[19, 601, 326]···H(50)[−12, 740, 103]	2·45
H(33)[34, 442, 377]···H(52)[7, 373, 447]$^{\rm I}$	2·24
H(41)[101, 772, 130]···H(55)[114, 750, 46]	2·06

Symmetry code: (I) $x, 1 - y, \frac{1}{2} + z$.

Close intra- and intermolecular H···H distances are listed in Table 10. There is only one intermolecular close contact <2·50 Å. H(19) with fractional coordinates (0·137, 0·357, 0·176) is in a peculiar position over the Si_5 ring maintaining about the same non-bonded distance (2·97 and 3·03 Å) with Si(2) and Si(4) respectively.

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Structure Cristalline de l'Oxymétavincadifformine $C_{21}H_{26}N_2O_4$

PAR NOËL RODIER

Laboratoire de Chimie Minérale, Faculté des Sciences Pharmaceutiques et Biologiques, rue J.-B. Clément,
92290 *Châtenay-Malabry, France*

RENÉ CÉOLIN

Laboratoire de Chimie Minérale, Faculté de Pharmacie, 2bis boulevard Tonnelé, 37032 *Tours CEDEX, France*

ET GUY CROQUELOIS, NICOLE KUNESCH ET JACQUES POISSON

Laboratoire de Chimie des Substances Naturelles, Faculté des Sciences Pharmaceutiques et Biologiques, rue J.-B. Clément, 92290 *Châtenay-Malabry, France*

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Oxymetavincadifformine is orthorhombic, space group $P2_12_12_1$, with $a = 9{\cdot}711$ (3), $b = 13{\cdot}812$ (5), $c = 13{\cdot}955$ (4) Å. $Z = 4$, $D_c = 1{\cdot}313$ g cm^{-3}. Data were collected on an automatic diffractometer using Mo $K\alpha$ radiation. The structure was solved by direct methods and refined by a least-squares procedure to a final R of 0·049 for 1887 independent reflections. The H atoms were located and their positional parameters refined. The molecule contains a benzene, two other six-membered rings and two five-membered rings; its shape, rather closed, is stabilized by an intramolecular hydrogen bond.

Introduction

Croquelois, Kunesch & Poisson (1974) et Croquelois (1975) ont préparé l'oxymétavincadifformine $C_{21}H_{26}N_2O_4$ par une transposition oxydative de la vincadifformine sous l'action de l'acide *m*-chloroperbenzoïque et ont déterminé sa formule stéréochimique. Le but de l'étude cristallographique est de vérifier la structure proposée (Fig. 1).

Le cristal utilisé mesure environ 0,40 × 0,40 × 0,24 mm. Il a été obtenu en taillant à la lame de rasoir un échantillon issu de la recristallisation du produit dans le mélange éther–acétone.

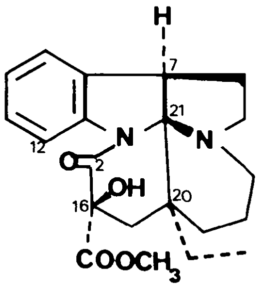

Fig. 1. Oxymétavincadifformine.