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Structure of a Highly Crowded Linear Trisilane

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Abstract. Hexamethyl-2-(1,2,3-tri-*tert*-butylcyclopropenyl)-2-(2,4,6-triisopropylphenyl)trisilane, $C_{36}H_{68}$ -Si₃, $M_r = 585 \cdot 2$, monoclinic, $P2_1/c$, a = 15.907 (2), b = 11.537 (3), c = 20.928 (3) Å, $\beta = 94.31$ (1),° V = 3830 (1) Å³, Z = 4, $D_x = 1.02$ g cm⁻³, λ (Mo $K\alpha$) = 0.71069 Å, $\mu = 1.40$ cm⁻¹, F(000) = 1304, T = 298 K, R = 0.043 for 2115 unique reflections $[I \ge 3\sigma(I)]$. The molecular geometry of this compound is distinguished by extreme crowding resulting in long Si—Si [2.441 (2) and 2.464 (2) Å] and Si—C [1.973 (4) and 1.988 (4) Å] bonds about the central Si atom. The Si—Si angle is 92.60 (7)°, significantly compressed from a tetrahedral value. A number of short non-bonding contacts between peripheral groups (≤ 3.5 Å) are observed.

Introduction. Hexamethyl-2-(1,2,3-tri-*tert*-butylcyclopropenyl)-2-(2,4,6-triisopropylphenyl)trisilane (R =isopropyl, shown below), is a photochemical source of highly hindered silylenes and silacyclobutadienes (Puranik & Fink, 1989). This highly crowded molecule is unusual because of the juxtaposition of extremely bulky groups at the central Si atom. An X-ray structure determination was undertaken in order to explore the structural consequences of the steric crowding in this molecule, and to present comparisons with the structures of two closely similar compounds where R = H and Me (Puranik, Johnson & Fink, 1989).



Experimental. A colorless plate-shaped crystal, 0.40 $\times 0.40 \times 0.17$ mm, was obtained from ethanolhexane: intensity data collected on an Enraf-Nonius CAD-4 diffractometer at 298 K, Mo Ka radiation (graphite monochromator), lattice parameters from setting of 25 reflections with $4 < \theta < 13^{\circ}$; $\omega - 2\theta$ scans, with $1 \le 2\theta \le 50^\circ$; scan width $(0.8 + 0.2\tan\theta)^\circ$; $0 \le h$ $\leq 18, 0 \leq k \leq 13, -24 \leq l \leq 24;$ 6975 total reflections, 6457 unique, 2115 observed with $I \ge 3\sigma(I)$: 2.2% variation in three standard reflections measured every 7200 s; Lorentz-polarization corrections made; absorption corrections based on ψ scans of five reflections near $\chi = 90$ (min. and max. transmission factors are 0.97 and 0.99, respectively); linear decay correction applied to data; $R_{int} = 0.026$. The structure was solved by direct methods using MULTAN11/82 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1982) in the Structure Determination Package (Frenz, 1985); refined on F by full-matrix least squares with minimization $[\sum (|F_{o}| - |F_{c}|)^{2} / \sum w(F_{o})^{2}]^{1/2}, \quad w = 1/\sigma F^{2} \text{ using the}$ same program. All non-H atoms were refined anisotropically; H atoms were located in a difference map and included as fixed contributions riding on attached C atoms with isotropic thermal parameters 1.3 times those of respective C atoms; the remainder of the H atoms were placed in calculated positions and treated as above. The refinement is not based on a model which includes disorder. The non-H atoms with high thermal parameters, C(15), C(16), C(17), C(29), C(29A), C(29B), and associated H atoms were removed after final refinement, followed by a reexamination of the difference Fourier map. The ΔF map revealed no additional electron density which could be attributed to the presence of disorder of the associated tert-butyl or para-isopropyl groups in question. Reassignment of the C atoms followed by

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Si(1)

Si(1)

Si(1) C(1)

Si(1) C(7)

Si(2)

Si(2)

Si(2)

Si(3)

Si(3)

Si(3)

C(1)

C(1)

C(2)

C(2)

C(3)

C(4)

C(4)

C(5)

C(6)

C(7)

C(7)

C(7)

Si(2)

Si(2)

Si(2)

Si(3)

Si(3)

C(1)

Si(1) Si(2)

Si(1)

Si(1)

C(22)

C(22) Si(2)

C(23) Si(2)

Si(1)

Si(1) Si(3)

Si(1) Si(3)

C(25)

C(25)

C(26) Si(3)

Si(1) C(1)

Si(1) C(1)

C(2)

C(1)

C(I)

C(11)

C(11)

C(12)

C(8)

C(8)

C(8)

C(15)

C(15)

C(16)

C(9)

C(9)

Si(2)

Si(3)

C(22)

C(23)

C(24)

C(25)

C(26)

C(27)

C(2)

C(6)

C(3) C(28)

C(4)

C(5)

C(29)

C(30)

C(8)

C(9)

C(10)

Si(1)

Si(1)

Si(1)

Si(1)

Si(1)

Si(1)

Si(2)

Si(2)

Si(2)

Si(3)

Si(3)

Si(3)

C(1)

C(2)

C(2)

C(10)

C(10)

C(10)

C(14)

C(14)

C(14)

C(14)

C(14)

C(14)

C(18) C(19)

C(18)

C(18)

C(6)

2.441 (2)

2.464 (2)

1.973 (4)

1.988 (4)

1.886(5)

1.886 (6)

1.883 (5)

1.884 (6)

1.895 (5)

1.872 (5)

1.413(5)

1.415 (5)

1.402 (6)

1.513 (6)

1.380 (6)

1.356 (7)

1.514(7)

1.390 (6)

1-509 (6)

1·548 (6) 1·533 (6)

1·556 (6) Si(3) 92

C(1)

C(7)

C(1)

C(7)

C(7)

C(22)

C(23)

C(24)

C(23)

C(24)

C(24)

C(25)

C(26)

C(27)

C(26)

C(27)

C(27)

C(2)

C(6)

C(6)

C(3)

C(28)

C(12)

C(13)

C(13)

C(15)

C(16)

C(17)

C(16)

C(17)

C(17)

C(20)

C(21)

92.60 (7)

116.6 (1)

111.6(1)

105.3 (1)

121.8 (1)

108.7(2)

110.4(2)

108.4(2)

125.4 (2)

107.8 (3)

101-3 (3)

102.4 (3)

117.1 (2)

105.2 (2)

121.7(2)

104.5 (3)

98.8 (3)

108.4 (2)

122.0 (3)

122.0 (3)

116.0 (4)

120.3 (4)

125.8 (4)

107.1 (4)

108.4 (4)

106.4 (4)

111.9 (4)

109.1 (4)

111-1 (4)

108-6 (5)

109.5 (5)

106.5 (4)

111-8 (4)

109.6 (4)

111-3 (4)

Table 1. Atomic coordinates and equivalent isotropic thermal parameters with e.s.d.'s in parentheses

Table 2. Non-hydrogen bond distances (Å) and angles (°)

C(8)

C(8)

C(9)

C(10)

C(10)

C(10)

C(14)

C(14)

C(14)

C(18)

C(18)

C(18)

C(28)

C(28)

C(29)

C(29)

C(30)

C(30)

C(3)

C(2)

C(3)

C(3)

C(5)

C(4)

C(1)

C(1)

C(5)

Si(1)

Si(1)

Si(1)

C(8)

C(8)

C(9)

C(7)

C(7)

C(9)

C(7)

C(7)

C(8)

C(7)

C(7)

C(7)

C(19)

C(19)

C(20)

C(2)

C(2)

C(4)

C(4)

C(6)

C(9)

C(14)

C(18)

C(11)

C(12)

C(13)

C(15)

C(16)

C(17)

C(19)

C(20)

C(21)

C(28A)

C(28B)

C(29A)

C(29B)

C(30A)

C(30B)

C(2)

C(3)

C(4)

C(4)

C(4)

C(5)

C(6)

C(6)

C(6)

C(7)

C(7)

C(7)

C(7)

C(7)

C(7)

C(8)

C(8)

C(8)

C(9)

C(9)

C(9)

C(10)

C(10)

C(10) C(13)

C(18)

C(18)

C(18)

C(28)

C(28)

C(29)

C(29)

C(30)

C(28A) C(28)

C(29*A*) C(29) C(6) C(30)

C(30A) C(30)

1.279 (6)

1.529 (7)

1.509 (6)

1.522 (6)

1.524 (6)

1.526 (6)

1.505 (7)

1.517 (7)

1.513 (6)

1.533 (7)

1.526 (7)

1.523 (6)

1.515 (7)

1 405 (8)

1·448 (9) 1·508 (7)

1.531 (7)

113.9 (4)

123-1 (5)

116.0 (5)

122.9 (5)

121-1 (6)

124.1 (5)

120·5 (4) 125·1 (4)

114.5 (4)

118.8 (3)

119.4 (3)

117.1 (3)

116.7 (3)

64.8 (3)

149.9 (4)

144.4 (5)

66-1 (3)

147.2 (4)

145.5 (5)

111.1 (4)

113.0 (4)

110.6 (4)

109.1 (5)

107.9 (4)

107.1 (4)

112.8 (4)

113 2 (4)

107.5 (4)

114.6 (6)

112.4 (6)

 $111 \cdot 1$ (7)

111.0 (4)

113.0 (5)

111.2 (4)

49·1 (3) 118·4 (4)

C(28)

C(4)

C(5)

C(29)

C(29)

C(6)

C(5)

C(30)

C(30)

C(8)

C(9)

C(10)

C(9)

C(10)

C(10)

C(14)

C(14)

C(8)

C(18)

C(18)

C(1)

C(12)

C(20)

C(21)

C(21)

C(28A)

C(28B)

C(28B)

C(29A)

C(29B)

C(29B)

C(30A)

C(30B)

C(30B)

C(9)

$B_{eq} = (4/3)[a^2\beta_{11} + b^2\beta_{22} + c^2\beta_{33} + (2ab\cos\gamma)\beta_{12} + (2a\cos\beta)\beta_{12} + (2a\beta)\beta_{12} + (2a\beta)$	13
$+ (2bc\cos\alpha)\beta_{23}].$	

	x	у	Ζ	$B_{\rm eq}({\rm \AA}^2)$
Si(1)	0.76681 (8)	0.2204(1)	0.89677 (6)	2.92 (3)
Si(2)	0.6582 (1)	0.0710 (2)	0.89713 (7)	4.91 (4)
Si(3)	0.8708 (1)	0.0846 (2)	0.85748 (7)	4.73 (4)
$\mathbf{C}(\mathbf{i})$	0.7511 (3)	0.3408 (4)	0.8300 (2)	2.8 (1)
C(2)	0.8201 (3)	0.3961 (4)	0.8039 (2)	3.2(1)
C(3)	0.8065 (3)	0.4875 (5)	0.7603 (2)	4.4 (1)
C(4)	0.7272 (3)	0.5276 (5)	0.7399 (2)	5.0(1)
C(5)	0.6616 (3)	0.4721 (5)	0.7642 (2)	4.8(1)
C(6)	0.6702 (3)	0.3802 (4)	0.8071 (2)	3.4 (1)
C(7)	0.7849 (3)	0.2975 (4)	0.9817 (2)	2.8(1)
C(8)	0.7475 (3)	0.4199 (4)	0.9903 (2)	3.3 (1)
C(9)	0.7085 (3)	0.3375 (4)	1.0163 (2)	3.5(1)
C(10)	0.8649 (3)	0.2622 (5)	1.0245 (2)	3.6(1)
C(11)	0.9444 (3)	0.3086 (5)	0.9974 (2)	5.0(1)
C(12)	0.8644 (3)	0.3070 (5)	1.0930 (2)	5.4 (2)
C(13)	0.8720 (3)	0.1305 (5)	1.0293 (2)	5.2 (1)
C(14)	0.7549 (3)	0.5515 (4)	0.9848 (2)	4-3(1)
C(15)	0.7630 (5)	0.5893 (6)	0.9166 (3)	11.5 (3)
C(16)	0.6769 (4)	0.6074 (5)	1.0087 (3)	7.9 (2)
C(17)	0.8292 (4)	0.5968 (5)	1.0269 (3)	8.4 (2)
C(18)	0.6426 (3)	0.3071 (5)	1.0619 (2)	4.9(1)
C(19)	0.6445 (3)	0.1779 (5)	1.0789 (3)	6.3 (2)
C(20)	0.5544 (3)	0.3386 (6)	1 0312 (3)	6.6 (2)
C(21)	0.6556 (4)	0.3757 (6)	1.1242 (2)	7.5 (2)
C(22)	0.6898 (4)	~ 0.0447 (5)	0.9580 (3)	7.6 (2)
C(23)	0.6480 (4)	0.0004 (6)	0.8156 (3)	8.0 (2)
C(24)	0.5449 (3)	0.0961 (6)	0.9137 (3)	7.1 (2)
C(25)	0.8631 (4)	- 0.0715 (5)	0.8827 (3)	7.3 (2)
C(26)	0.8483 (3)	0.0816 (6)	0.7672 (2)	6.8 (2)
C(27)	0.9868 (3)	0.1059 (5)	0.8757 (3)	6.4 (2)
C(28)	0.9122 (3)	0.3674 (5)	0.8196 (2)	4.7 (1)
C(28A)	0.9580 (3)	0.3375 (6)	0.7604 (3)	7.7 (2)
C(28B)	0.9607 (3)	0.4642 (6)	0.8545 (3)	7.8 (2)
C(29)	0.7131 (4)	0.6315 (5)	0.6962 (3)	7.3 (2)
C(29A)	0.7505 (7)	0.6226 (7)	0.6376 (3)	15.7 (4)
C(29B)	0.7356 (8)	0.7395 (7)	0.7282 (4)	18.1 (5)
C(30)	0.5879 (3)	0.3312 (5)	0.8265 (2)	4.4 (1)
C(30A)	0.5397 (3)	0.2734 (6)	0.7705 (3)	7 7 (2)
C(30B)	0.5335 (3)	0.4214 (6)	0.8578 (3)	7.5 (2)

routine refinement revealed the presence of H(15A), H(16A), H(17A), H(29) and H(29B2). The remainder of the H atoms on these methyl groups were placed in calculated positions; final R = 0.043, wR = 0.051; $(\Delta/\sigma)_{max} = 0.00$; S = 1.61 for 352 variables; final difference map $\Delta \rho_{max} = \pm 0.2$ e Å⁻³; atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV, Table 2.2A).

Discussion. Atomic coordinates are reported in Table 1;* non-H bond lengths and angles in Table 2. An *ORTEP* (Johnson, 1976) plot of the title compound is shown in Fig. 1. A packing diagram, viewed along the b crystallographic axis, is shown in Fig. 2.

In this molecule, the allylic *tert*-butyl group nearly eclipses one of the trimethylsilyl groups with a C(10)-C(7)-Si(1)-Si(3) torsion angle of $4.0 (4)^{\circ}$. The aromatic and cyclopropene ring planes intersect

at an angle of	45·7 (4)°. ′	The methy	yl groups	of the
ortho-isopropyl	substituen	ts are dire	ected away	from
the $Si(1)$ — $C(1)$	vector in	order to	minimize	steric
interactions.				

The crowding around the central Si atom of the trisilane results in abnormally long bond lengths to this atom. The respective Si(1)—C(1) and Si(1)—C(7) bond lengths of 1.973 (4) and 1.988 (4) Å are greatly extended from a normal value of 1.86 Å (Armitage, 1982), and are comparable with the largest reported Si—C bond length of 2.012 Å (Weidenbruch, Blintjer, Peters & von Schnering, 1986). The Si—Si bond lengths of 2.441 (2) and 2.264 (2) Å are also higher than the typical value of

^{*} 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 53172 (89 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

2.34 Å (Armitage, 1982). The extreme steric requirements of the aromatic and cyclopropene substituents are also responsible for the small Si—Si bond angle of 92.60 (7)°.

Comparisons of selected bond parameters with two closely analogous cyclopropenyltrisilanes, where R = H and Me, are shown in Table 3. The three trisilanes form a series in which the *ortho* and *para* aromatic substituents become progressively larger. The size of the *ortho* substituent profoundly affects the degree of steric crowding at the central Si atom. It is seen that all bond lengths at Si(1) uniformly increase with the size of the *ortho* substituent. A parallel compression of the Si—Si—Si angle is also found with increasing size of the *ortho* substituents.

The steric crowding at Si(1) also results in some distortion of the aromatic ring in the title compound. The ring C atoms deviate significantly from planarity

Fig. 1. ORTEP drawing of title compound with atom labeling; thermal ellipsoids are at the 50% probability level.



Fig. 2. Packing diagram view along the b axis.

Table 3. Comparison of selected bond lengths (Å) and angles (°) for (2,4,6-R₃C₃H₃)('Bu₃C₃)Si(SiMe₃)₂

R	d(Si—aryl)	d[Si-(cyclopropenyl)]	d(Si—Si)	θ(Si—Si—Si)
Н*	1.893 (5)	1.940 (4)	2.396 (2), 2.398 (2)	102.09 (7)
Me*	1.940 (4)	1.959 (4)	2.426 (2), 2.455 (2)	93.97 (6)
<i>i</i> -Pr	1.973 (4)	1.988 (4)	2.441 (2), 2.464 (2)	92.60 (7)

* Puranik, Johnson & Fink (1989).

 Table 4. Non-bonding contacts (Å) for the title compound

Intramolecular (≤	3·5 Å)		
C(22)…C(25)	3.291 (9)	C(26)…C(28A)	3.44 (1)
C(24)C(30)	3.368 (9)	C(27)…C(28)	3.418 (8)
Intermolecular (≤	4∙0 Å)		
Intermolecular (\leq C(12)···C(26)	4·0 Å) 3·891 (9)	C(22)···C(29 <i>A</i>)	3.91 (1)

 $(\chi^2 = 0.32$ relative to the mean plane) and assume a boat-like conformation. Within the ring, longer C—C bond distances are observed to the siliconbearing carbon. Thus, C(1)—C(2) and C(1)—C(6) are 1.413 (5) and 1.415 (5) Å, respectively, as compared to the analogous distances to the *para* carbon which are 1.380 (6) [C(3)—C(4)] and 1.356 (7) Å [C(4)—C(5)]. The extended bond distances at C(1) are certainly due to repulsive interactions between the large silyl substituent and the *ortho*-isopropyl groups of the aromatic ring.

A number of short intramolecular and intermolecular non-bonding contacts are also evident in the title compound. The shortest intramolecular contacts are between peripheral methyl groups on the *tert*-butyl, trimethylsilyl, and *ortho*-isopropyl groups (Table 4). Some short intermolecular contacts are also present and are listed in Table 4.

It is likely that the high thermal parameters observed for the *para*-isopropyl and one of the vinylic *tert*-butyl groups can be attributed to the packing of the molecules in the crystal. The *para*isopropyl group (Fig. 2) is oriented in a manner which could allow movement of the group, but not actual rotational disorder. The *tert*-butyl group in question also appears to exhibit movement allowed by packing in the crystal, but again, there is no evidence for true rotational disorder.

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Structure of the Naturally Occurring Sesquiterpene Lactone 8-Epiisolipidiol

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Abstract. Decahydro-4.8-dihydroxy-3.9-dimethyl-6methyleneazuleno[4,5-b]furan-2(3H)-one, $C_{15}H_{21}O_4$, $M_r = 265.3$, orthorhombic, $P2_12_12_1$, a = 5.755 (1), b = 13.891 (2), c = 17.511 (2) Å, V = 1399.9 (4) Å³, Z = 4, $D_x = 1.25 \text{ Mg m}^{-3}$, $\lambda(\text{Cu } K\alpha) = 1.54178 \text{ Å}$, μ $= 0.65 \text{ mm}^{-1}$, F(000) = 572, T = 295 K, R = 0.055, wR = 0.070 for 1685 observed reflections. The threering guaianolide system contains an exo-methylenecycloheptane moiety cis-fused with the cyclopentane [at C(1)-C(5)] and *trans*-annelated with the α -methyl- γ -lactone [at C(6)—C(7)]. The two fivemembered rings are in envelope conformations while the seven-membered ring is about midway between chair and twist-chair forms. The secondary hydroxyl groups are at C(3) and C(8), and are both β -orientated; they participate in intermolecular hydrogen bonds, O(8) both as a donor and an acceptor, and O(3) as a donor only.

Introduction. Chemotaxonomic studies are becoming increasingly important in the classification of plants. So far from the plants of the tribe *Lactuceae* (*Compositae* family) predominantly guaiane-type sesquiterpene lactones related to lactucin (I) have been isolated. Recently a series of structurally related 8β -hydroxyguaian-6,12-olides with additional oxygen functions at C(3) have been isolated from *Crepis* species (Barbetti, Casinovi, Santurbano & Longo, 1979; Kisiel, 1983a, b, 1984), *Andryala* (Bohlmann & Gupta, 1982; Massanet, Collado, Macias, Luis & Vergara, 1984), *Ixeris* (Nishimura, Miyse, Ueno, Noro, Kuroyanagi & Fukushima, 1985) and

Prenanthes (Miyase, Yamada & Fukushima, 1987). The presence of these guaianolides in plants of the tribe Lactuceae might be of chemotaxonomical importance, since their C(8) epimers appear mostly in plants of the tribes Cynareae. Vernonieae and Mutisieae. 8-Epiisolipidiol (II) was obtained for the first time from Crepis capillaris (L.) Wallr. (Kisiel, 1983a), and its 8α -hydroxy analogue was isolated from Amberboa lippii D. C. (Cynareae) (Gonzalez, Garcia Marrero & Breton, 1970). An initial molecular structure, (II), was proposed for 8-epiisolipidiol based on chemical and spectroscopic methods (Kisiel, 1983a). Confirmation of the structure was sought through crystallographic analysis.



Experimental. 8-Epiisolipidiol (II) was extracted from the aerial parts of *Crepis capillaris* (L.) Wallr. with ethanol and isolated by silica-gel column chromatography.

A crystal approximately $0.2 \times 0.3 \times 0.3$ mm, obtained from methanol solution, was used for X-ray analysis. Syntex P2₁ diffractometer, θ -2 θ scan, variable scan rate, graphite-monochromated Cu K α radiation, cell parameters by least-squares refinement

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