Acta Cryst. (1982). B**38**, 1648–1649

2,2,4,4,6,6-Hexa-*tert*-butylcyclotrisiloxane

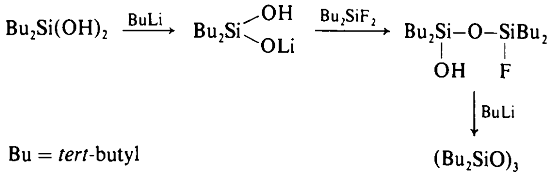
By William Clegg

Anorganisch-Chemisches Institut der Universität, Tammannstrasse 4, *D*-3400 *Göttingen, Federal Republic of Germany*

(*Received* 14 *August* 1981; *accepted* 22 *December* 1981)

Abstract. $C_{24}H_{54}O_3Si_3$, $M_r = 475{\cdot}0$, rhombohedral, $R\bar{3}c$, $a = 10{\cdot}103\,(1)$, $c = 48{\cdot}967\,(5)$ Å on hexagonal axes, $V = 4328$ Å^3, $Z = 6$, $D_x = 1{\cdot}09$ Mg m^{-3}; final $R = 0{\cdot}047$ for 1102 unique diffractometer data. The Si_3O_3 ring is planar, with internal angles at Si and O of 105·1 (1) and 134·9 (1)°. The Si–O bond length is 1·654 (1) Å.

Introduction. Compounds in which Si is bonded to two *tert*-butyl groups and to two functional groups (OH or NH_2) are remarkably resistant to condensation reactions, although analogous compounds with different alkyl groups react readily to give various chain and ring compounds containing Si—O—Si and Si—N—Si units. This difference has been attributed to the steric bulk of the *tert*-butyl group, which inhibits attack at the Si atom (Sommer & Tyler, 1954; Weidenbruch, Pesel & Hieu, 1980). The title compound has, however, been produced *via* the reaction scheme shown (Klingebiel, 1981; Clegg, Klingebiel & Sheldrick, 1982).



$$Bu_2Si(OH)_2 \xrightarrow{BuLi} Bu_2Si\begin{smallmatrix}OH\\OLi\end{smallmatrix} \xrightarrow{Bu_2SiF_2} \underset{OH}{Bu_2Si}-O-\underset{F}{SiBu_2} \xrightarrow{BuLi} (Bu_2SiO)_3$$

Bu = *tert*-butyl

A block crystal of size *ca* 0·4 mm was examined on a Stoe–Siemens AED diffractometer (Mo $K\alpha$ radiation, graphite monochromator, $\lambda = 0{\cdot}71069$ Å). All measurements were made at room temperature. Cell dimensions were derived by least-squares refinement from the 2θ values ($20 < 2\theta < 25°$) of 80 accurately centred reflexions; 2θ was measured as the difference in ω values of the centred positions on either side of the direct beam, in order to minimize the effects of systematic errors.

Intensity data were collected in a θ/ω scan mode for reflexions with $2\theta < 60°$ (Clegg, 1981). No corrections were made for absorption ($\mu = 0{\cdot}18$ mm^{-1}).

The structure was solved by automatic direct methods, and refined by a blocked-cascade least-squares method to a minimum value of $\sum w\Delta^2$ [$\Delta = |F_o| - |F_c|$; $w^{-1} = \sigma^2(F) + 0{\cdot}0005F^2$], from 1102 unique reflexions with $I > 2\sigma(I)$. H atoms were constrained so that C—H = 0·96 Å, H—C—H = 109·5°, and $U(H) = 1{\cdot}2U_{eq}(C)$. All other atoms were assigned anisotropic thermal parameters. Final values of R and of R_g [$=(\sum w\Delta^2/\sum wF_o^2)^{1/2}$] were 0·047 and 0·056 respectively. A final difference synthesis contained no features outside the range ±0·3 e Å^{-3}. Atomic scattering factors were taken from *International Tables for X-ray Crystallography* (1974).

Table 1. *Atomic coordinates* ($\times 10^5$) *and equivalent isotropic thermal parameters* (Å$^2 \times 10^4$)

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

	x	y	z	U_{eq}
Si	17460 (6)	0	75000	300 (2)
O	0	−15003 (14)	75000	321 (6)
C(1)	28111 (20)	−82 (20)	78217 (4)	396 (7)
C(2)	45518 (22)	10540 (27)	78011 (5)	562 (10)
C(3)	24894 (25)	−16477 (23)	78800 (4)	535 (9)
C(4)	23097 (26)	5371 (27)	80710 (4)	546 (10)

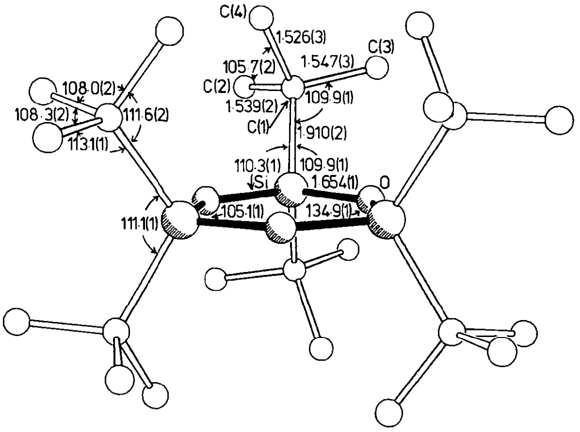


Fig. 1. Projection of $(Bu_2SiO)_3$ almost edge-on to the Si_3O_3 plane, showing atom labelling, bond lengths (Å) and angles (°).

Atomic coordinates are given in Table 1 and bond lengths and angles in Fig. 1.*

Discussion. The $(Bu_2SiO)_3$ molecule (Bu = *tert*-butyl) has crystallographic 32 (D_3) symmetry (Fig. 1), the molecular centre of mass being the special position $0,0,\frac{3}{4}$. The Si_3O_3 ring is, therefore, strictly planar. In other cyclotrisiloxane structures, deviations from planarity are slight (Bokii, Zakharova & Struchkov, 1972; Shklover, Bokii, Struchkov, Andrianov, Zavin & Svistunov, 1974*a*,*b*; Shklover, Adaasuren, Kotrelev, Zhdanova, Svistunov & Struchkov, 1980). Bond lengths and angles within this ring are also typical of such structures.

The compound is chemically very stable and yields well formed polyhedral crystals, giving narrow X-ray reflexions of significant intensity well beyond the 2θ limit to which data were collected. Thermal parameters are unusually low in comparison with other organosilicon–oxygen and –nitrogen compounds studied in our laboratory (Table 1). Presumably the sterically bulky *tert*-butyl groups, which prevent a facile synthesis of $(Bu_2SiO)_3$, are also responsible for these properties.

I thank Dr U. Klingebiel for supplying crystals, and the Verband der Chemischen Industrie for financial support. All computer programs were written by WC and Professor G. M. Sheldrick for a Data General S250 Eclipse mini-computer.

References

Bokii, N. G., Zakharova, G. N. & Struchkov, Yu. T. (1972). *J. Struct. Chem. (USSR)*, **13**, 267–272.

Clegg, W. (1981). *Acta Cryst.* A**37**, 22–28.

Clegg, W., Klingebiel, U. & Sheldrick, G. M. (1982). *Z. Naturforsch. Teil B*, **37**. In the press.

International Tables for X-ray Crystallography (1974). Vol. IV, pp. 99, 149. Birmingham: Kynoch Press.

Klingebiel, U. (1981). *Chem. Ber.* **114**, 3366–3371.

Shklover, V. E., Adaasuren, P., Kotrelev, G. V., Zhdanova, E. A., Svistunov, V. S. & Struchkov, Yu. T. (1980). *J. Struct. Chem. (USSR)*, **21**, 198–202.

Shklover, V. E., Bokii, N. G., Struchkov, Yu. T., Andrianov, K. A., Zavin, B. G. & Svistunov, V. S. (1974*a*). *J. Struct. Chem. (USSR)*, **15**, 79–85.

Shklover, V. E., Bokii, N. G., Struchkov, Yu. T., Andrianov, K. A., Zavin, B. G. & Svistunov, V. S. (1974*b*). *J. Struct. Chem. (USSR)*, **15**, 741–748.

Sommer, L. H. & Tyler, L. J. (1954). *J. Am. Chem. Soc.* **76**, 1030–1033.

Weidenbruch, M., Pesel, H. & Hieu, D. V. (1980). *Z. Naturforsch. Teil B*, **35**, 31–34.

---

* Lists of structure factors, anisotropic thermal parameters, H-atom parameters and a thermal ellipsoid plot have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 36613 (20 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Acta Cryst. (1982). B**38**, 1649–1652

Structure of (*E*)-3,4-Bis(*p*-methoxyphenyl)-2,2,5,5-tetramethyl-3-hexene

By Tullio Pilati and Massimo Simonetta

Istituto di Chimica Fisica e Centro CNR, Università, Via Golgi 19, 20133 *Milano, Italy*

(*Received* 3 *December* 1981; *accepted* 22 *December* 1981)

Abstract. $C_{24}H_{32}O_2$, $M_r = 352{\cdot}52$, monoclinic, $P2_1/c$, $a = 10{\cdot}792\,(3)$, $b = 11{\cdot}930\,(3)$, $c = 8{\cdot}326\,(6)$ Å, $\beta = 93{\cdot}97\,(4)°$, $Z = 2$, $D_c = 1{\cdot}095$ Mg m^{-3}. The molecule, itself centrosymmetric, lies at a symmetry centre with two statistically disordered orientations. The structure was refined with constraints on both geometrical and thermal parameters to an $R = 0{\cdot}076$ on 1306 independent reflections classified as observed.

Introduction. In line with our interest in the geometries and conformations of highly strained molecules, we have undertaken the study of the crystal structure of the title compound (hereinafter DTBDMPE), to confirm experimentally the molecular conformation of (*E*)-2,2,5,5-tetramethyl-3,4-diphenyl-3-hexene (DTBDPE) recently determined by force-field calculations (Favini, Simonetta, Sottocornola & Todeschini, 1981). Colourless crystals were obtained by slow evaporation from saturated methyl ethyl ketone solution. A single crystal, sealed into a glass capillary to prevent sublimation, was mounted on a Syntex $P\bar{1}$ diffractometer. The intensity data were collected up to $\theta = 23°$, using Mo $K\alpha$ radiation ($\lambda = 0{\cdot}71069$ Å, graphite monochromator). A total of 1483 independent

0567-7408/82/051649-04$01.00 © 1982 International Union of Crystallography