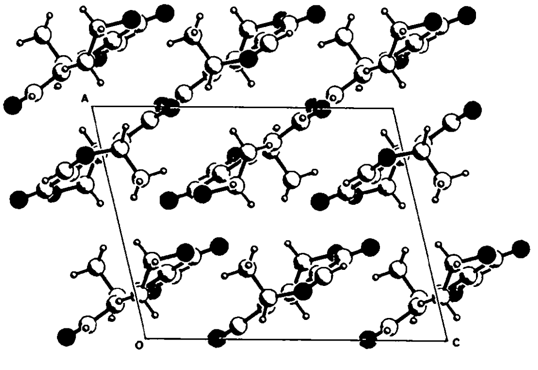

Fig. 2. Projection of the crystal structure down the *b* axis. Oxygen atoms are represented as black spheres.

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Structure of 1,1,3,3-Tetraisopropyl-1,3-disiloxanediol, $C_{12}H_{30}O_3Si_2$

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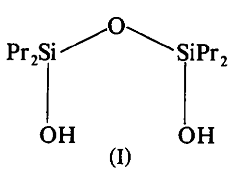
Abstract. $M_r = 278{\cdot}5$, triclinic, $P\bar{1}$, $a = 8{\cdot}968\,(1)$, $b = 9{\cdot}008\,(1)$, $c = 21{\cdot}898\,(3)$ Å, $\alpha = 82{\cdot}21\,(1)$, $\beta = 89{\cdot}69\,(1)$, $\gamma = 85{\cdot}08\,(1)°$, $U = 1746{\cdot}2$ Å^3, $Z = 4$, $D_x = 1{\cdot}059$ Mg m^{-3}, $\lambda(\text{Mo } K\alpha) = 0{\cdot}71069$ Å, $\mu = 0{\cdot}19$ mm^{-1}, $F(000) = 616$, $T = 291$ K, $R = 0{\cdot}045$ for 4662 observed reflections. The two crystallographically independent molecules have mean Si—O(Si) and Si—O(H) bond lengths of 1·639 (2) and 1·619 (2) Å and a mean Si—O—Si angle of 164·1 (2)°. Molecules are linked together by hydrogen bonds, to form chains parallel to the *b* axis.

Introduction. Hydrolysis of Pr_2SiF_2 (Pr = isopropyl) with KOH (Klingebiel, 1983) yields the title compound (I) and the six-membered ring siloxane $(Pr_2SiO)_3$, a homologue of the *tert*-butyl compound $(Bu_2SiO)_3$, the structure of which has been reported (Clegg, 1982).



(I)

Experimental. Crystals obtained from *n*-hexane and mounted in capillaries, 0·5 × 0·45 × 0·25 mm, Stoe–Siemens AED diffractometer, unit-cell parameters refined from 2θ angles for 50 reflections centred at $\pm\omega$ ($20 < 2\theta < 25°$), intensities measured for 6124 reflections with $2\theta < 50°$ and $h \geq 0$, real-time profile-fitting (Clegg, 1981), no significant intensity variation for three standard reflections; no absorption correction, 6124 unique reflections (no equivalents measured), 4662 with $F > 4\sigma(F)$; structure solved by automatic multisolution direct methods, blocked-cascade refinement on F, $w^{-1} = \sigma^2(F) + 0{\cdot}0005F^2$, alkyl H atoms constrained to give C—H = 0·96 Å, H—C—H = 109·5°, $U(\text{H}) = 1{\cdot}2U_{\text{eq}}(\text{C})$, hydroxyl H atoms refined freely with isotropic U, anisotropic thermal parameters for all non-H atoms, no extinction correction, 371 parameters, $R = 0{\cdot}045$, $wR = 0{\cdot}054$, slope of normal probability plot = 1·32, max. shift/e.s.d. = 0·2, mean = 0·04, largest peak in final difference map = 0·27 e Å^{-3}, largest hole = −0·24 e Å^{-3}, scattering factors from *International Tables for X-ray Crystallography* (1974), programs used: *SHELXTL* (Sheldrick, 1978), diffractometer control program by WC.

Discussion. Atomic coordinates and bond lengths are given in Tables 1 and 2 respectively.* The two

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* Tables of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 38469 (31 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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crystallographically independent molecules (one is shown in Fig. 1) differ significantly only in torsion angles of the isopropyl substituents. The Si—O bond lengths and Si—O—Si angles lie within the wide ranges already observed in siloxanes, silicates and related structures (Barrow, Ebsworth & Harding, 1979; Baur, 1980).

Torsion angles for the four H—O—Si—O groups are 152 (2), −39 (2), −175 (2) and −37 (2)°. Within each molecule, the first OH group [O(1) or O(3)] is turned away from the second [O(2) or O(4)], but the second is directed towards the first. Each OH group forms a hydrogen bond [O···O = 2·719 (5)–2·849 (5) Å] to an OH group of another molecule. Of the four crystallographically independent hydrogen bonds produced, three hold molecules together in pairs, and the fourth links these dimers into chains parallel to the *b* axis (Fig. 2).

I thank Dr U. Klingebiel for supplying crystals, and the Verband der Chemischen Industrie for financial support.

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

$$U_{\rm eq} = \tfrac{1}{3}\textstyle\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$U_{\rm eq}$
Si(1)	1836 (1)	−70 (1)	1660 (1)	403 (2)
Si(2)	3374 (1)	−3293 (1)	1423 (1)	407 (2)
O(1)	2333 (2)	−100 (1)	2383 (1)	521 (6)
H(1)	2452 (24)	603 (25)	2468 (10)	640 (65)
O(2)	3113 (2)	−4469 (1)	2045 (1)	513 (6)
H(2)	3097 (23)	−4049 (23)	2302 (10)	576 (62)
O(12)	2427 (2)	−1692 (1)	1472 (1)	463 (5)
C(11)	2723 (3)	1478 (2)	1171 (1)	550 (9)
C(12)	2442 (5)	1510 (4)	484 (1)	1097 (17)
C(13)	4379 (3)	1499 (3)	1304 (1)	826 (13)
C(14)	−249 (3)	120 (3)	1629 (1)	637 (10)
C(15)	−952 (4)	−118 (4)	1027 (2)	1020 (16)
C(16)	−918 (4)	1572 (4)	1844 (2)	1234 (19)
C(21)	5385 (3)	−2977 (3)	1353 (1)	701 (11)
C(22)	5962 (3)	−2475 (4)	1946 (2)	954 (14)
C(23)	6339 (4)	−4373 (4)	1203 (2)	1368 (22)
C(24)	2599 (3)	−4116 (3)	771 (1)	676 (10)
C(25)	2815 (5)	−3154 (4)	159 (1)	1330 (21)
C(26)	951 (3)	−4392 (4)	866 (2)	1012 (15)
Si(3)	7986 (1)	3836 (1)	6361 (1)	416 (2)
Si(4)	6798 (1)	7217 (1)	6519 (1)	400 (2)
O(3)	7190 (2)	2956 (2)	6962 (1)	560 (6)
H(3)	7376 (26)	2192 (26)	7066 (11)	766 (73)
O(4)	7237 (2)	7301 (2)	7239 (1)	530 (6)
H(4)	7188 (22)	6728 (22)	7443 (9)	509 (59)
O(34)	7321 (2)	5567 (1)	6339 (1)	467 (5)
C(31)	7389 (3)	3129 (3)	5653 (1)	624 (10)
C(32)	5686 (4)	3175 (4)	5613 (1)	947 (14)
C(33)	8022 (4)	3965 (4)	5062 (1)	918 (14)
C(34)	10040 (3)	3723 (3)	6471 (1)	692 (11)
C(35)	10445 (3)	4196 (4)	7096 (1)	876 (13)
C(36)	10827 (4)	2188 (4)	6398 (2)	1235 (19)
C(41)	4738 (3)	7576 (3)	6400 (1)	657 (10)
C(42)	3893 (3)	6387 (4)	6775 (2)	1011 (16)
C(43)	4156 (4)	9146 (4)	6526 (2)	1242 (19)
C(44)	7854 (3)	8654 (2)	6052 (1)	553 (9)
C(45)	7434 (4)	8866 (3)	5368 (1)	904 (14)
C(46)	9531 (3)	8324 (3)	6140 (1)	877 (14)

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

Si(1)—O(1)	1·644 (2)	Si(1)—O(12)	1·616 (2)
Si(1)—C(11)	1·871 (2)	Si(1)—C(14)	1·864 (3)
Si(2)—O(2)	1·636 (2)	Si(2)—O(12)	1·626 (2)
Si(2)—C(21)	1·852 (3)	Si(2)—C(24)	1·859 (3)
O(1)—H(1)	0·701 (23)	O(2)—H(2)	0·720 (22)
C(11)—C(12)	1·522 (4)	C(11)—C(13)	1·517 (4)
C(14)—C(15)	1·513 (5)	C(14)—C(16)	1·524 (5)
C(21)—C(22)	1·538 (5)	C(21)—C(23)	1·532 (5)
C(24)—C(25)	1·515 (4)	C(24)—C(26)	1·528 (4)
Si(3)—O(3)	1·635 (2)	Si(3)—O(34)	1·615 (1)
Si(3)—C(31)	1·852 (3)	Si(3)—C(34)	1·851 (3)
Si(4)—O(4)	1·640 (2)	Si(4)—O(34)	1·619 (2)
Si(4)—C(41)	1·861 (3)	Si(4)—C(44)	1·860 (2)
O(3)—H(3)	0·702 (22)	O(4)—H(4)	0·640 (18)
C(31)—C(32)	1·527 (4)	C(31)—C(33)	1·538 (4)
C(34)—C(35)	1·541 (4)	C(34)—C(36)	1·526 (5)
C(41)—C(42)	1·513 (4)	C(41)—C(43)	1·525 (4)
C(44)—C(45)	1·529 (4)	C(44)—C(46)	1·516 (4)

O(1)—Si(1)—O(12)	106·1 (1)	O(1)—Si(1)—C(11)	109·4 (1)
O(12)—Si(1)—C(11)	111·0 (1)	O(1)—Si(1)—C(14)	107·4 (1)
O(12)—Si(1)—C(14)	108·7 (1)	C(11)—Si(1)—C(14)	113·9 (1)
O(2)—Si(2)—O(12)	109·5 (1)	O(2)—Si(2)—C(21)	109·5 (1)
O(12)—Si(2)—C(21)	108·5 (1)	O(2)—Si(2)—C(24)	106·3 (1)
O(12)—Si(2)—C(24)	108·3 (1)	C(21)—Si(2)—C(24)	114·6 (1)
Si(1)—O(1)—H(1)	114·9 (17)	Si(2)—O(2)—H(2)	107·4 (16)
Si(1)—O(12)—Si(2)	164·3 (1)	Si(1)—C(11)—C(12)	113·3 (2)
Si(1)—C(11)—C(13)	113·2 (2)	C(12)—C(11)—C(13)	110·9 (3)
Si(1)—C(14)—C(15)	116·1 (2)	Si(1)—C(14)—C(16)	112·6 (2)
C(15)—C(14)—C(16)	111·1 (3)	Si(2)—C(21)—C(22)	110·5 (2)
Si(2)—C(21)—C(23)	112·2 (2)	C(22)—C(21)—C(23)	110·1 (3)
Si(2)—C(24)—C(25)	111·7 (2)	Si(2)—C(24)—C(26)	111·9 (2)
C(25)—C(24)—C(26)	110·6 (3)	O(3)—Si(3)—O(34)	104·2 (1)
O(3)—Si(3)—C(31)	109·7 (1)	O(34)—Si(3)—C(31)	108·6 (1)
O(3)—Si(3)—C(34)	110·7 (1)	O(34)—Si(3)—C(34)	108·9 (1)
C(31)—Si(3)—C(34)	114·2 (1)	O(4)—Si(4)—O(34)	110·1 (1)
O(4)—Si(4)—C(41)	110·4 (1)	O(34)—Si(4)—C(41)	108·6 (1)
O(4)—Si(4)—C(44)	105·6 (1)	O(34)—Si(4)—C(44)	109·7 (1)
C(41)—Si(4)—C(44)	112·5 (1)	Si(3)—O(3)—H(3)	122·0 (19)
Si(4)—O(4)—H(4)	120·1 (19)	Si(3)—O(34)—Si(4)	163·8 (1)
Si(3)—C(31)—C(32)	111·2 (2)	Si(3)—C(31)—C(33)	112·9 (2)
C(32)—C(31)—C(33)	110·6 (2)	Si(3)—C(34)—C(35)	111·1 (2)
Si(3)—C(34)—C(36)	113·7 (2)	C(35)—C(34)—C(36)	110·7 (3)
Si(4)—C(41)—C(42)	112·0 (2)	Si(4)—C(41)—C(43)	112·2 (2)
C(42)—C(41)—C(43)	110·9 (3)	Si(4)—C(44)—C(45)	112·8 (2)
Si(4)—C(44)—C(46)	111·9 (2)	C(45)—C(44)—C(46)	110·9 (2)

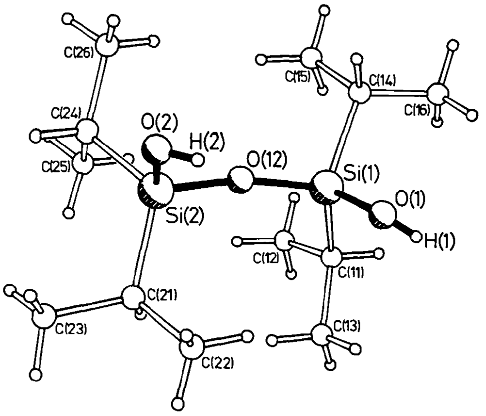

Fig. 1. View of one of the molecules, with atom labelling.

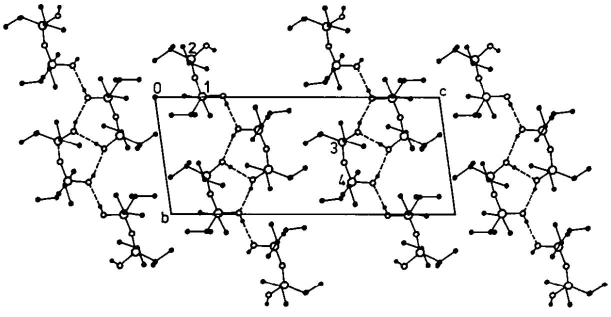

Fig. 2. The crystal packing seen in parallel projection along the *a* axis. The Si atoms are numbered.

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Tétrahydro-5,6,7,7a Benzo[3,4]cyclobuta[1,2-*b*]benzazocine[1],* $C_{17}H_{15}N$

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(*Reçu le* 27 *mai* 1982, *accepté le* 17 *février* 1983)

Abstract. $M_r = 233{\cdot}3$, orthorhombic, $P2_12_12_1$, $a = 27{\cdot}32\,(2)$, $b = 7{\cdot}149\,(4)$, $c = 6{\cdot}596\,(3)$ Å, $V = 1288{\cdot}3$ Å^3, $Z = 4$, $D_x = 1{\cdot}20$ g cm^{-3}, $\lambda(\text{Mo } K\alpha) = 0{\cdot}7107$ Å, $\mu(\text{Mo } K\alpha) = 0{\cdot}65$ cm^{-1}, $F(000) = 496$, $T = 295$ K. The structure was solved by direct methods and refined by a full-matrix least-squares technique. $R = 0{\cdot}053$ for 1476 independent reflections [$I > 2\sigma(I)$]. The structure consists of layers of molecules parallel to (100). Its cohesion is due to van der Waals interactions. The molecule is built of four rings: *A* and *D* are benzene rings and the two others, *B* and *C*, contain four and eight atoms respectively. The ring-junction configuration is *cis* for *B*/*C*.

Introduction. En milieu acide, la décomposition d'un azide tertiaire benzocyclobuténique conduit généralement à un indole comme l'indique la Fig. 1 (Adam, 1981; Adam, Andrieux & Plat, 1981). Dans les mêmes conditions, l'azide représenté par la Fig. 2 donne un produit non indolique de formule brute $C_{17}H_{15}N$. L'étude cristallographique de ce dernier a été entreprise dans le but de déterminer sa structure moléculaire que les méthodes classiques (RMN de ^{1}H et de ^{13}C, IR, UV)

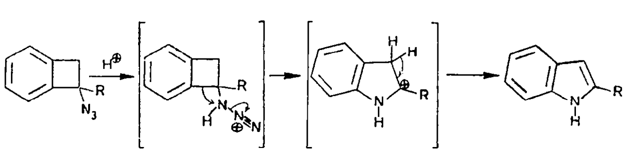

Fig. 1. Formation d'indole par décomposition en milieu acide d'un azide tertiaire benzocyclobuténique.

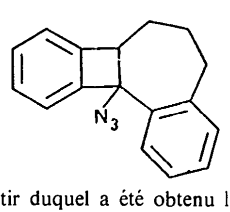

Fig. 2. Azide à partir duquel a été obtenu la tétrahydro-5,6,7,7a benzo[3,4]cyclobuta[1,2-*b*]benzazocine[1]. (Numérotation de l'auteur. Voir la note précédente.)

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* Note de l'éditeur: La numérotation atomique utilisée dans le titre est la numérotation conforme aux règles de nomenclature de l'IUPAC. La numérotation utilisée par les auteurs dans le reste de ce memoire conduit à nommer ce composé tétrahydro-9b,10,11, 12 benzo[3,4]cyclobuta[1,2-*b*]benzazocine[1].

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