Table 1. *Fractional coordinates (× 10⁴) and equivalent isotropic thermal parameters (Å² × 10³)*

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

	x	y	z	U_{eq}
C(1)	7982 (9)	2250 (9)	3060 (2)	51 (2)
C(2)	7325 (12)	1498 (10)	2677 (2)	60 (2)
C(3)	5248 (12)	1997 (9)	2602 (2)	54 (2)
C(4)	4084 (10)	1578 (9)	2945 (2)	50 (2)
C(5)	4721 (9)	2310 (8)	3344 (2)	45 (1)
C(6)	3530 (10)	1791 (9)	3697 (2)	52 (2)
C(7)	3709 (9)	58 (9)	3816 (2)	48 (1)
C(8)	5747 (8)	−329 (7)	3910 (2)	38 (1)
C(9)	6925 (8)	23 (8)	3540 (1)	37 (1)
C(10)	6782 (9)	1787 (8)	3421 (2)	41 (1)
C(11)	8950 (9)	−486 (8)	3609 (2)	43 (1)
C(12)	9174 (9)	−2186 (8)	3762 (2)	45 (1)
C(13)	8018 (9)	−2454 (7)	4138 (2)	38 (1)
C(14)	5974 (8)	−2060 (8)	4037 (2)	39 (1)
C(15)	4898 (10)	−2734 (10)	4395 (2)	59 (2)
C(16)	5987 (11)	−4251 (9)	4513 (2)	58 (2)
C(17)	7747 (10)	−4197 (8)	4259 (2)	44 (1)
C(18)	8738 (12)	−1498 (8)	4497 (2)	48 (2)
C(19)	4580 (12)	4134 (8)	3314 (3)	63 (2)
O(1)	9374 (7)	−4738 (5)	4486 (2)	50 (1)
C(20)	9603 (12)	−6306 (8)	4523 (2)	51 (2)
C(21)	11241 (13)	−6731 (9)	4768 (3)	71 (2)
O(2)	8539 (10)	−7254 (6)	4385 (2)	83 (2)
O(3)	4784 (8)	2669 (8)	2300 (2)	77 (2)
Br	8865 (2)	2132 (2)	2229 (1)	92 (1)
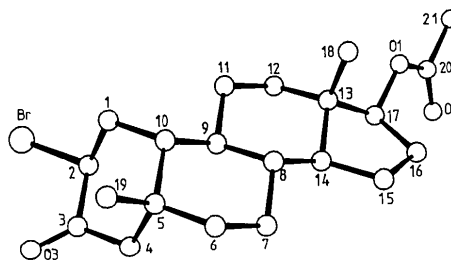
Fig. 1. Perspective view (*ORTEP*) of the title compound with the numbering scheme.

The bromo-ketone moiety is planar with the Br—C(2)—C(3)—O(3) dihedral angle 0·3 (9)° and a Br⋯O(3) distance of 3·0 Å. The packing is unexceptional.

Related literature. 5β-R steroids: Boeyens, Bull & van Rooyen (1980); Sedee, Beijersbergen van Henegouwen, Guijt & Haasnoot (1985); Kirk & Hartshorn (1968).

References

BOEYENS, J. C. A., BULL, J. R. & VAN ROOYEN, P. H. (1980). *S. Afr. J. Chem.* **33**, 45–50.

CREMER, D. & POPLE, J. A. (1975). *J. Am. Chem. Soc.* **97**, 1354–1358.

International Tables for X-ray Crystallography (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)

JOHNSON, C. K. (1965). *ORTEP*. Report ORNL-3794. Oak Ridge National Laboratory, Tennessee, USA.

KIRK, D. N. & HARTSHORN, M. P. (1968). *Steroid Reaction Mechanisms*, pp. 257–262. Amsterdam: Elsevier.

MAIN, P., FISKE, S. J., HULL, S. E., LESSINGER, L., GERMAIN, G., DECLERCQ, J.-P. & WOOLFSON, M. M. (1980). *MULTAN80. A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data.* Univs. of York, England, and Louvain, Belgium.

SEDEE, A. G. J., BEIJERSBERGEN VAN HENEGOUWEN, G. M. J., GUIJT, W. & HAASNOOT, C. A. G. (1985). *J. Org. Chem.* **50**, 4182–4187.

SHELDRICK, G. M. (1976). *SHELX76*. Program for crystal structure determination. Univ. of Cambridge, England.

A 1,1,3,3-Tetraphenyl-1,3-disiloxanediol Pyridinium Chloride Lattice Complex

BY M. A. HOSSAIN

Department of Applied Chemistry, Dhaka University, Dhaka-1000, Bangladesh

M. T. RAHMAN AND G. RASUL

Department of Chemistry, Dhaka University, Dhaka-1000, Bangladesh

AND M. B. HURSTHOUSE AND B. HUSSAIN

Department of Chemistry, Queen Mary College, London E1 4NS, England

Abstract. $C_{24}H_{22}O_3Si_2.C_5H_6N^+.Cl^-$, $M_r = 530\cdot16$, monoclinic, $P2_1/a$, $a = 20\cdot508$ (7), $b = 9\cdot785$ (1), $c = 14\cdot042$ (3) Å, $\beta = 90\cdot36$ (2)°, $V = 2818\cdot8$ (8) Å³, $Z = 4$, $D_x = 1\cdot247$ g cm⁻³, Mo $K\alpha$, $\lambda = 0\cdot71069$ Å, $\mu = 1\cdot57$ cm⁻¹, $F(000) = 1108$, $T = 298$ K, $R = 0\cdot043$, $wR = 0\cdot053$ for 3083 observed data. The Cl⁻ ion is involved in a fairly strong interaction with the proton on the pyridine N atom [Cl⋯H(10) 1·95 (2) Å] and

Table 1. *Fractional atomic coordinates* (×10⁴) *and equivalent isotropic temperature factors* ($\text{Å}^2 \times 10^3$) *for* $C_{24}H_{22}O_3Si_2.C_5H_6N^+.Cl^-$

	x	y	z	U_{eq}*
Cl	8275 (1)	9418 (1)	9408 (1)	62·8 (3)
O(1)	9622 (1)	600 (3)	8813 (2)	58 (1)
Si(1)	9562·5 (4)	1790 (1)	8009 (1)	40·5 (3)
O(2)	9180 (1)	1261 (2)	7070 (2)	53 (1)
Si(2)	8691·3 (4)	171 (1)	6566 (1)	40·2 (3)
O(3)	8054 (1)	−90 (3)	7216 (2)	56 (1)
C(11)	9153 (1)	−1446 (2)	6358 (2)	41 (1)
C(12)	9780 (1)	−1378 (2)	5979 (2)	56 (1)
C(13)	10127 (1)	−2577 (2)	5802 (2)	72 (2)
C(14)	9847 (1)	−3843 (2)	6003 (2)	77 (2)
C(15)	9221 (1)	−3911 (2)	6381 (2)	85 (2)
C(16)	8874 (1)	−2713 (2)	6559 (2)	64 (1)
C(21)	8400 (1)	965 (2)	5434 (2)	48 (1)
C(22)	8390 (1)	214 (2)	4590 (2)	72 (2)
C(23)	8173 (1)	821 (2)	3747 (2)	99 (2)
C(24)	7966 (1)	2179 (2)	3748 (2)	113 (3)
C(25)	7975 (1)	2930 (2)	4593 (2)	105 (2)
C(26)	8192 (1)	2323 (2)	5436 (2)	78 (2)
C(31)	10414 (1)	2235 (2)	7682 (1)	39 (1)
C(32)	10541 (1)	2671 (2)	6756 (1)	52 (1)
C(33)	11171 (1)	3057 (2)	6503 (1)	70 (2)
C(34)	11675 (1)	3006 (2)	7175 (1)	74 (2)
C(35)	11547 (1)	2570 (2)	8101 (1)	65 (2)
C(36)	10917 (1)	2184 (2)	8355 (1)	49 (1)
C(41)	9104 (1)	3316 (2)	8456 (2)	42 (1)
C(42)	8435 (1)	3425 (2)	8289 (2)	58 (1)
C(43)	8099 (1)	4587 (2)	8587 (2)	73 (2)
C(44)	8432 (1)	5639 (2)	9051 (2)	75 (2)
C(45)	9101 (1)	5530 (2)	9217 (2)	76 (2)
C(46)	9438 (1)	4368 (2)	8920 (2)	61 (1)
N(10)	3662 (1)	3254 (3)	731 (2)	58 (1)
C(20)	3272 (2)	2191 (4)	858 (3)	60 (1)
C(30)	4247 (2)	3349 (4)	1159 (3)	63 (2)
C(40)	4451 (2)	2324 (4)	1743 (3)	69 (2)
C(50)	4050 (2)	1218 (5)	1884 (3)	75 (2)
C(60)	3456 (2)	1163 (4)	1434 (3)	71 (2)

* U_{eq} according to Hamilton (1959).

Table 2. *Selected bond lengths* (Å) *and angles* (°) *for* $C_{24}H_{22}O_3Si_2.C_5H_6N^+.Cl^-$

Si(1)–O(1)	1·626 (5)	O(2)–Si(1)	1·615 (4)
C(31)–Si(1)	1·860 (4)	C(41)–Si(1)	1·875 (4)
Si(2)–O(2)	1·623 (4)	O(3)–Si(2)	1·618 (5)
C(11)–Si(2)	1·868 (4)	C(21)–Si(2)	1·864 (5)
C(20)–N(10)	1·325 (6)	C(30)–N(10)	1·342 (6)
C(60)–C(20)	1·344 (7)	C(40)–C(30)	1·360 (7)
C(50)–C(40)	1·374 (7)	C(60)–C(50)	1·371 (7)
H(1)–O(1)	0·847 (39)	H(3)–O(3)	0·725 (39)
H(10)–N(10)	1·096 (41)	H(20)–C(20)	1·006 (39)
H(30)–C(30)	0·950 (41)	H(40)–C(40)	0·923 (34)
H(50)–C(50)	0·988 (41)	H(60)–C(60)	0·970 (40)
O(2)–Si(1)–O(1)	111·8 (2)	C(31)–Si(1)–O(1)	105·8 (2)
C(31)–Si(1)–O(2)	109·1 (2)	C(41)–Si(1)–O(1)	111·9 (2)
C(41)–Si(1)–O(2)	106·6 (2)	C(41)–Si(1)–C(31)	111·7 (2)
Si(2)–O(2)–Si(1)	149·6 (2)	O(3)–Si(2)–O(2)	110·8 (2)
C(11)–Si(2)–O(2)	108·2 (2)	C(11)–Si(2)–O(3)	111·5 (2)
C(21)–Si(2)–O(2)	107·0 (2)	C(21)–Si(2)–O(3)	106·9 (2)
C(21)–Si(2)–C(11)	112·3 (2)	C(12)–C(11)–Si(2)	119·2 (3)
C(16)–C(11)–Si(2)	120·8 (3)	C(22)–C(21)–Si(2)	120·5 (3)
C(26)–C(21)–Si(2)	119·5 (3)	C(32)–C(31)–Si(1)	118·9 (3)
C(36)–C(31)–Si(1)	121·1 (2)	C(30)–N(10)–C(20)	122·3 (4)
C(60)–C(20)–N(10)	120·0 (5)	C(40)–C(30)–N(10)	119·3 (5)
C(50)–C(40)–C(30)	119·1 (5)	C(60)–C(50)–C(40)	119·7 (5)
C(50)–C(60)–C(20)	119·6 (5)		
Si(1)–O(1)–H(1)	119·5 (27)	H(3)–O(3)–Si(2)	116·0 (32)
H(10)–N(10)–C(20)	120·3 (21)	H(10)–N(10)–C(30)	117·4 (21)
H(20)–C(20)–N(10)	112·6 (22)	H(20)–C(20)–C(60)	127·4 (22)
H(30)–C(30)–N(10)	115·3 (25)	H(30)–C(30)–C(40)	125·3 (25)
H(40)–C(40)–C(30)	116·2 (22)	H(40)–C(40)–C(50)	124·6 (22)
H(50)–C(50)–C(40)	122·4 (23)	H(50)–C(50)–C(60)	117·7 (24)
H(60)–C(60)–C(20)	121·5 (24)	H(60)–C(60)–C(50)	118·8 (24)

weaker interactions with the silanol H atoms [Cl··· H(1), H(3) 2·28, 2·42 (3) Å]. The Si—O—Si angle in the disiloxane link is 149·6 (2)°.

Experimental. The title compound was obtained in an attempt to prepare a siloxy derivative of titanium by the reaction between tetraphenyldisiloxanediol and titanium tetrachloride in the presence of pyridine. The colourless crystals were obtained as ill formed polyhedral blocks directly from the reaction mixture. A specimen of dimensions 0·25 × 0·22 × 0·22 mm was used for the X-ray investigation. CAD-4 diffractometer, graphite-monochromatized Mo $K\alpha$ radiation; orientation matrix and cell dimensions from least-squares refinement on setting angles for 25 well spaced reflections (10 < θ < 13°). Data collection *via* $\omega/2\theta$ scan mode 1·5 < θ < 25°, $\pm h$, $+k$, $+ l$; 3 intensity, 2 orientation control reflections, no instability; 5501 reflections measured, 4971 unique (R_{int} = 0·02), 3083 observed [$I > 3\sigma(I)$]. h = 0–24, k = 0–11, l = 0–16. Structure solved *via* direct methods (*SHELXS*86; Sheldrick, 1986) and refined by full-matrix least squares on f (*SHELX*76; Sheldrick, 1976). Absorption correction by *DIFABS* (Walker & Stuart, 1983); min., max. transmission coefficient 0·93, 1·10. Weighting scheme used: $w = [\sigma^2(F) + 0·00015F_o^2]^{-1}$. Phenyl rings treated as idealized hexagons, H atoms included with group U_{iso}
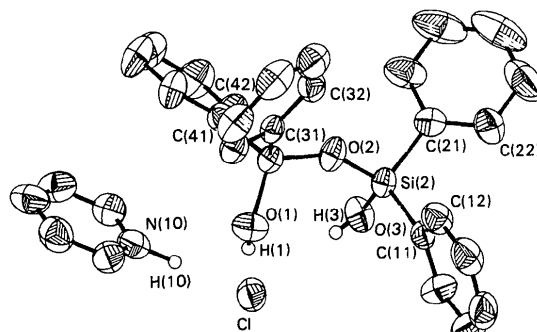

Fig. 1. Diagram of the asymmetric unit of the lattice complex.

values; pyridine H atoms located experimentally and freely refined isotropically. All non-H atoms refined anisotropically. Largest $\Delta/\sigma = 0·2$ [U_{11} H(10)], max., min. $\Delta\rho = +0·25$, $-0·23$ e Å⁻³. Atomic scattering factor data as included in *SHELX*76. Final atomic parameters of non-H atoms are given in Table 1;* selected bond lengths and angles are given in Table 2. A diagram of the complex, with atom numbering, is shown in Fig. 1.

Related literature. The structure of tetraphenyldisiloxanediol itself contains three independent molecules of

the diol with Si—O—Si link angles of 147·4 (3), 157·1 (4) and 162·5 (4)° (Hossain, 1980; Hossain & Hursthouse, 1987). In the tetraisopropyl derivative the two independent molecules present have link angles of 163·8 (1) and 164·1 (1)° (Clegg, 1983).

References

CLEGG, W. (1983). Acta Cryst. C39, 901–903.

HAMILTON, W. C. (1959). Acta Cryst. 12, 609–610.
HOSSAIN, M. A. (1980). PhD thesis, Univ. of London, England.
HOSSAIN, M. A. & HURSTHOUSE, M. B. (1987). J. Cryst. Spectrosc. Res. In the press.
SHELDRICK, G. M. (1976). SHELX76. Program for crystal structure determination. Univ. of Cambridge, England.
SHELDRICK, G. M. (1986). SHELXS86. Program for crystal structure solution. Univ. of Göttingen, Federal Republic of Germany.
WALKER, N. & STUART, D. (1983). Acta Cryst. A39, 158–166.

Structure of a Hydroxyiodolactam

By M. M. Olmstead,† M. J. Kurth and S. H. Bloom

Department of Chemistry, University of California, Davis, California 95616, USA

Abstract. 1-(1-Hydroxymethylpropyl)-5-iodomethyl-2-pyrrolidone, $C_9H_{16}INO_2$, $M_r = 297\cdot14$, monoclinic, $P2_1/c$, $a = 7\cdot419$ (1), $b = 19\cdot562$ (3), $c = 8\cdot156$ (1) Å, $\beta = 114\cdot58$ (1)°, $V = 1076\cdot5$ (3) Å^3, $Z = 4$, D_x (130 K) $= 1\cdot83$ g cm^{-3}, Mo $K\alpha$, $\lambda = 0\cdot71069$ Å, $\mu = 29\cdot1$ cm^{-1}, $F(000) = 584$, $T = 130$ (1) K, $R = 0\cdot045$ for 1953 unique reflections. The molecules crystallize as intermolecular hydrogen-bonded pairs. The $O(2)\cdots O(1^i)$ distance is $2\cdot760$ (9) Å, (i) $= -1 + x$, $\frac{1}{2} - y$, $z - \frac{1}{2}$. There is an I$\cdots$I intermolecular contact of $3\cdot942$ (4) Å. The bond lengths and angles are all normal.

Experimental. Compound prepared by iodolactamization of 2-(3-butenyl)-4,5-dihydro-4-ethyloxazole as a mixture of (5R*,1'R*)- and (5R*,1'S*)-isomers [(1) and (2), respectively]. Purification by radial chromatography (LiChroprep™ Si60 PF, EtOAc:cyclohexane 1:1) gave (1) as a crystalline solid and (2) as an oil. Recrystallization of (1) from cyclohexane gave colorless needles and plates; crystal dimensions 0·25 × 0·25 × 1·00 mm; Syntex $P2_1$ diffractometer, locally modified LT-1 apparatus, $T = 130$ K, Mo $K\alpha$ radiation, graphite monochromator; cell dimensions from least-squares fit of 10 reflections with $17 < 2\theta < 26°$; space group $P2_1/c$ based on conditions $h0l$, $l = 2n$, $0k0$, $k = 2n$, range of absorption correction factors 1·83–2·45; data collected to $2\theta_{max} = 50°$ with hkl ranges 0 to 8, 0 to 23, −9 to 9, respectively; ω scans, 2·0° range, 60° min^{-1} speed, 1·8° offset for background; two check reflections monitored every 200 reflections displayed only random fluctuations with a maximum deviation from the average

† Author to whom correspondence should be addressed.

Table 1. *Atomic coordinates* ($\times 10^4$) *and equivalent isotropic thermal parameters* (Å^2 $\times 10^3$)

Equivalent isotropic U defined as one-third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U_{eq}
I	444 (1)	4678 (1)	2908 (1)	31 (1)
O(1)	6607 (8)	2690 (3)	1824 (7)	33 (2)
O(2)	484 (8)	2601 (3)	−886 (7)	32 (2)
N	4022 (9)	3445 (3)	839 (7)	21 (2)
C(1)	5726 (12)	3182 (4)	2088 (10)	25 (3)
C(2)	6356 (12)	3581 (4)	3830 (10)	29 (3)
C(3)	5146 (11)	4240 (4)	3157 (10)	28 (3)
C(4)	3309 (11)	4026 (4)	1534 (10)	23 (3)
C(5)	1591 (12)	3821 (4)	1996 (11)	27 (3)
C(6)	3186 (12)	3226 (4)	−1076 (10)	28 (3)
C(7)	3724 (13)	3719 (5)	−2226 (11)	36 (3)
C(8)	5877 (12)	3887 (5)	−1535 (10)	31 (3)
C(9)	993 (13)	3098 (4)	−1869 (10)	32 (3)

intensity of less than 2%; 2108 reflections measured, 1953 unique data, $R_{int} = 0\cdot008$, 1758 observed [$I > 3\sigma(I)$] used in the solution and refinement (based on F); structure solved by heavy-atom methods; absorption correction [program $XABS$, a method which obtains an absorption tensor from $F_o - F_c$ differences; Hope & Moezzi (1987)] applied, blocked-cascade least-squares refinement, 124 parameters; all non-H atoms with anisotropic thermal parameters. H atoms bonded to C were included using a riding model with C—H of 0·96 Å and isotropic thermal parameters set equal to 1·2 times the equivalent isotropic thermal parameter of the bonded C atom (this requires the use of three additional parameters in order to define the methyl group); the hydroxyl H atom was refined using the constraint that it be staggered with respect to the C in a Newman projection down the O—C bond and U_{iso}