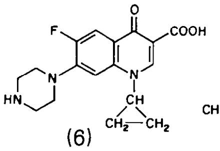
hypothesis. However, the more active compounds (and oxolinic acid, whose activity is approximately next in the series) have, in addition, a very electronegative substituent (F or O) at C(6), which might be a hydrogen-bond acceptor. Furthermore, the N(1) substituent may be important. Albrecht (1977) has noted that ethyl or propyl groups show maximum antibacterial activity in a series of related compounds with ten different substituents in the 1-position; a hydrophobic interaction with the gyrase protein seems possible. In the three compounds with *N*-ethyl groups studied by X-ray diffraction, the *N*-ethyl conformations are remarkably similar (see above), and the N(1) substituents in (6) and (7) can be thought of as spatially analogous. In all these cases there is some conformational similarity to the natural nucleosides, suggesting a possible template-blocking role for these drugs.

We wish to thank Dr R. D. Westland, Warner-Lambert Co., Pharmaceutical Research Division, for the gift of the material.



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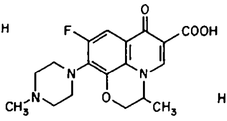

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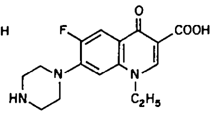

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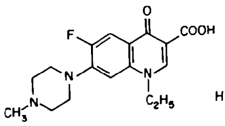

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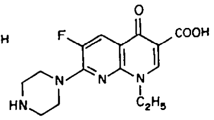

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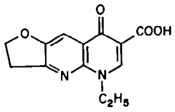

(11)

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Acta Cryst. (1985). C**41**, 1055–1057

2,5-Dichloro-2,5-dimethyl-2,5-disilahexane, $C_6H_{16}Cl_2Si_2$

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(*Received* 22 *February* 1985; *accepted* 11 *March* 1985)

Abstract. $M_r = 215{\cdot}3$, triclinic, $P\bar{1}$, $a = 6{\cdot}143$ (2), $b = 6{\cdot}179$ (3), $c = 9{\cdot}074$ (3) Å, $\alpha = 104{\cdot}52$ (3), $\beta = 93{\cdot}07$ (3), $\gamma = 117{\cdot}13$ (3)°, $V = 291{\cdot}0$ (2) Å^3, $Z = 1$, $D_x = 1{\cdot}228$ Mg m^{-3}, Mo $K\alpha$, $\lambda = 0{\cdot}71069$ Å, $\mu = 0{\cdot}697$ mm^{-1}, $F(000) = 114$, $T = 150$ K, $R = 0{\cdot}020$ for 769 observed reflections. The molecule is centrosymmetric; the CSiCCSiC moiety is nearly planar. The Si–Cl bond is rather long [2·091 (1) Å]; other bond

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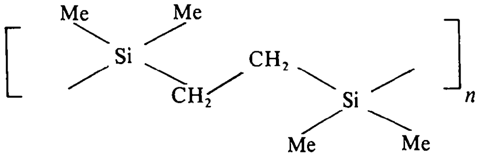
lengths and angles are normal. No intermolecular distances are shorter than the sum of the van der Waals radii.

Introduction. The crystal structure determination of the title compound (I) has been carried out in order to obtain information on the probable chain geometry of the carbosilane polymer poly[(dimethylsilylene)-ethylene(dimethylsilylene)]



as (I) represents a model of its single unit. We plan to study several other molecules of this series with a larger chain length.

Experimental. Transparent (unstable in the atmosphere), irregular-shaped single crystal $0{\cdot}7 \times 0{\cdot}5 \times 0{\cdot}2$ mm used for measurements of unit-cell parameters (20 reflections with $25 < 2\theta < 28°$) and intensities of 1334 reflections ($0 \leq h \leq 7$, $-8 \leq k \leq 8$, $-11 \leq l \leq 11$). Syntex $P2_1$ diffractometer (graphite monochromator, $\theta/2\theta$ scan, $2\theta_{\max} = 56°$). No significant variation in intensities of 2 standard reflections measured after every 100 reflections. No absorption or secondary-extinction corrections. Structure solved by Patterson method revealing Si and Cl atoms and refined by block-diagonal least squares with anisotropic thermal parameters for non-hydrogen atoms using 769 independent reflections with $I > 4\sigma(I)$ and minimizing $\sum w(|F_o| - |F_c|)^2$; $w = [\sigma^2(F_o) + 0{\cdot}03F_o^2]^{-1}$. Scattering factors from *International Tables for X-ray Crystallography* (1974). H atoms located by a difference synthesis and refined isotropically. Final $R = 0{\cdot}020$, $wR = 0{\cdot}027$, $S = 0{\cdot}93$, max. $\Delta/\sigma = 0{\cdot}5$, final electron density fluctuations $\pm 0{\cdot}4$ e Å^{-3}. All calculations carried out with an Eclipse S/200 computer using the *INEXTL* programs (Gerr, Yanovsky & Struchkov, 1983).

Discussion. Atomic coordinates are given in Table 1,* bond lengths, bond angles and torsion angles in Table 2. A perspective view of the molecule with the atom numbering is shown in Fig. 1.

The previously supposed centrosymmetric structure of the molecule is confirmed. The SiCCSi moiety has a planar *trans* conformation in all molecules investigated, with a symmetric configuration of the substituents (Shibayeva, Atovmyan, Rozenberg & Stryukov, 1973; Tacke, Niedner, Frohnecke, Ernst & Sheldrick, 1980). The SiCC bond angle of 114·3 (1)° is significantly greater than the tetrahedron value, this feature also being unexceptional. The orientation of substituents at Si atoms with respect to the central planar moiety seems to be less rigid. In the structure described by Tacke *et al.* (1980) the planar chain substituent $-CH_2CH_2CH_2N^+Me_3$ is in the *gauche* configuration with respect to the central moiety of the molecule. However, small non-bulky substituents seem to prefer the *trans* configuration. Thus in molecule (I) this configuration is nearly ideal, and the deviation of C(1) from the zigzag CSiCCSiC plane by 0·098 (2) Å is

Table 1. *Atomic coordinates* ($\times 10^5$ *for* Si *and* Cl; $\times 10^4$ *for* C; $\times 10^3$ *for* H) *and equivalent isotropic temperature factors* (Å^2)

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

	x	y	z	$B_{\rm eq}$ or $B_{\rm iso}$
Si	12439 (7)	84677 (7)	71543 (4)	1·34 (1)
Cl	−21603 (7)	84790 (7)	70636 (4)	2·27 (1)
C(1)	3704 (3)	11829 (3)	7563 (2)	2·30 (6)
C(2)	1060 (3)	6392 (3)	5215 (2)	1·57 (5)
C(3)	1531 (3)	7146 (3)	8725 (2)	2·30 (6)
H(11)	370 (4)	1292 (4)	858 (3)	3·2 (4)
H(12)	347 (4)	1258 (4)	673 (3)	3·4 (4)
H(13)	522 (4)	1196 (4)	765 (3)	4·0 (5)
H(21)	255 (3)	638 (3)	518 (2)	1·7 (3)
H(22)	81 (4)	720 (4)	444 (2)	2·1 (4)
H(31)	33 (4)	552 (4)	853 (3)	3·7 (5)
H(32)	315 (4)	718 (4)	880 (3)	3·7 (5)
H(33)	156 (5)	818 (4)	965 (3)	4·4 (5)

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

Si—Cl	2·091 (1)	C(1)SiCl	106·67 (6)
Si—C(1)	1·851 (2)	C(2)SiCl	105·98 (5)
Si—C(2)	1·862 (2)	C(3)SiCl	106·98 (6)
Si—C(3)	1·848 (2)	C(1)SiC(2)	112·45 (8)
C(2)—C(2′)	1·540 (2)	C(1)SiC(3)	112·15 (8)
C(1)—H(11)	1·00 (2)	C(2)SiC(3)	112·11 (8)
C(1)—H(12)	1·02 (2)	SiC(2)C(2′)	114·3 (1)
C(1)—H(13)	0·90 (3)		
C(2)—H(21)	0·92 (2)	C(1)SiC(2)C(2′)	−176·7 (1)
C(2)—H(22)	1·00 (2)	C(3)SiC(2)C(2′)	55·83 (9)
C(3)—H(31)	0·90 (2)	ClSiC(2)C(2′)	−60·54 (5)
C(3)—H(32)	0·98 (3)		
C(3)—H(33)	0·91 (3)		

A prime indicates an atom at $\bar{x}$, $1-y$, $1-z$.

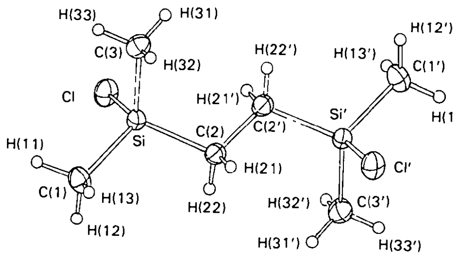


Fig. 1. A perspective view of the molecule with the atom-numbering scheme. H atoms are shown as spheres of arbitrary size.

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* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 42119 (9 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

possibly caused by the presence of Cl substituents at the Si atoms.

The Si—Cl bond length of 2·091 (1) Å is somewhat longer than the values obtained earlier [2·074 (1) and 2·080 (1) Å in $SiPh_3Cl$ (Lobkovsky, Fokin & Semenenko, 1981); it is still shorter in other (however, less accurate) studies]. The remaining bond lengths and angles are unexceptional.

In the crystal of (I) there are no intermolecular distances shorter than the sums of the van der Waals radii. The shortest intermolecular contact involving a Cl atom is Cl···H(33)($-x, 2-y, 2-z$) 3·06 (3) Å.

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Acta Cryst. (1985). C**41**, 1057–1059

Structure of 9-Hydroxy-3,7-dimethyl-2,3,4,5,6,7-hexahydro-2,7-methano-1*H*-3-benzazonine Hydrobromide, $C_{15}H_{22}NO^+.Br^-$

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(*Received* 24 *August* 1984; *accepted* 17 *January* 1985)

Abstract. $M_r = 312·25$, orthorhombic, $P2_12_12_1$, $a = 10·766$ (5), $b = 16·137$ (7), $c = 8·291$ (3) Å, $V = 1440$ (1) Å^3, $Z = 4$, $D_x = 1·440$ Mg m^{-3}, λ(Cu $K\alpha$) = 1·5402 Å, μ(Cu $K\alpha$) = 3·8 mm^{-1}, $F(000) = 648$, $T = 293$ K. $R = 0·052$ for 1589 observed reflections. The title compound is a synthetic analgesic which has a *C*-ring-enlarged benzomorphan structure. The N-containing ring protrudes from the molecular plane, and the H atom at the cationic N atom orientates toward the benzene ring. The distance of the N atom from the center of the benzene ring is 4·45 (1) Å.

Introduction. During extensive efforts to search for better analgesics, many kinds of chemical modifications have been applied to the morphine structure. Synthetic compounds containing only a portion of the morphine structure proved to be as effective as, or more effective than, the parent compound. Among them, the 6,7-benzomorphan compounds proved to be a particularly interesting and important class of analgesics and to give the most promising starting point in designing strong analgesics with low undesirable side effects, because the selective ligands for multiple opiate receptors were most likely to be found in this series (Gilbert & Martin, 1976). Some of them show useful narcotic antagonist activity.

By comparing the X-ray structures of several opiates (Gylbert, 1973; Hardy & Ahmed, 1975; Cochran & Abola, 1975), some structural requirements for possession of analgesic activity are proposed and discussed. Hardy & Ahmed (1975) ascribed the biological inactivity of 3-hydroxy-*N*-methyl-(+)-morphinan to the orientation of the lone electron pair of the N atom based on its X-ray structure. Opheim & Cox (1976) and Shiller, Yam & Lis (1977) suggested that the cationic form of the opiate drug is the active species, to interact with the receptor *via* ionic association. They also deduced that the distance of the cationic N atom from the benzene ring is important for analgesic activity. Moreover, Belleau, Conway, Ahmed & Hardy (1974) pointed out the importance of the relative geometry between the spacial orientation of the N lone electron pair and the benzene ring.

To elucidate the structure–activity relationships and to verify the proposals described above, several modified benzomorphans, with, for example, the *C* ring enlarged to a seven-membered ring or the N atom shifted to the next position in the carbon ring, were synthesized and analyzed crystallographically (Shiotani, Kometani & Mitsuhashi, 1975; Shiotani, Kometani, Mitsuhashi, Nozawa, Kurobe & Futsukaichi, 1976; Itai, Iitaka, Kometani & Shiotani, 1985). All these compounds were shown to possess fairly strong analgesic activities and the relationships between the X-ray structures and activities have been

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