

**Related literature.** The method of preparation of this possible anti-AIDS compound has been described by Van Aerschot, Herdewijn, Balzarini, Pauwels & De Clercq (1989).

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## Structure of 2,2',6,6'-Tetrakis[(dimethylammonium)methyl]biphenyl Tetrachloride *n*.Hydrate ( $n \approx 1.72$ )

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**Abstract.** 2,2',6,6'-Biphenyltetrayltetrakis[methyl(dimethylammonium)] tetrachloride 1.72-hydrate,  $C_{24}H_{42}N_4^{4+}.4Cl^{-}.1.72H_2O$ ,  $M_r = 559.42$ , monoclinic,  $I2/a$ ,  $a = 13.121$  (16),  $b = 11.030$  (7),  $c = 20.807$  (9) Å,  $\beta = 102.43$  (6)°,  $V = 2941$  (4) Å<sup>3</sup>,  $Z = 4$ ,  $D_x = 1.263$ ,  $D_m = 1.29$  (2) Mg m<sup>-3</sup>,  $\lambda(Cu K\alpha) = 1.54184$  Å,  $\mu = 39.3$  cm<sup>-1</sup>,  $F(000) = 1196.8$ ,  $T = 295$  K,  $R = 0.131$  for 1160 unique observed diffractometer data [ $I \geq 3\sigma(I)$ ]. Only crystals with a flaky habit, of poor quality were available, but the resulting moderate quality of the data is sufficient to secure the conformational and packing features of the title compound. The cation and an ordered water molecule are situated on a crystallographic twofold axis. The two phenyl rings are approximately perpendicular [ $83.3$  (8)°] due to the fourfold *ortho* substitution. N—H...Cl and O—H...Cl hydrogen bonds link the ions and ordered H<sub>2</sub>O into infinite chains running in the *a* direction. Voids in this structure contain additional disordered solvate molecules.

**Experimental.** The density was determined by flotation in an ethanol/CCl<sub>4</sub> mixture. The crystals crack and turn white after drying and were therefore mounted in glass capillaries. The crystals have a flaky habit causing broad reflection profiles. The

crystals diffract poorly at higher  $\theta$  values. An optimum specimen was selected by photographic techniques. Data were collected on an Enraf-Nonius CAD-4F diffractometer for a transparent, colorless crystal (0.24 × 0.06 × 1.4 mm). The cell parameters of the primitive triclinic unit cell [ $a = 11.030$  (7),  $b = 12.350$  (5),  $c = 13.121$  (16) Å,  $\alpha = 69.46$  (7),  $\beta = 102.43$  (6),  $\gamma = 63.37$  (4)°] were calculated by least squares from the SET4 setting angles of 25 reflections with  $14 \leq \theta \leq 17^\circ$ . The metrical symmetry for this cell is *I*-centered monoclinic (Spek, 1988). Data set collected in the triclinic setting. 5847 reflections were scanned [ $h - 13:13$ ,  $k - 14:15$ ,  $l 0:15$ ;  $\theta \leq 70^\circ$ ;  $\omega/2\theta$ -scan mode;  $\Delta\omega = (1.50 + 0.14\tan\theta)^\circ$ ; Ni-filtered Cu  $K\alpha$  radiation]. One reference reflection (124) showed a linear increase of 10% during 114 h of X-ray exposure time. The data were corrected for Lp and for absorption [*ABSORB* (Spek, 1983); correction factors 1.24–3.71] and transformed to monoclinic, resulting in the unique set of 1326 reflections [ $I > 3\sigma(I)$ ] used in the structure determination.  $\sigma^2(I) = \sigma_{cs}^2(I) + (pI)^2$  (McCandlish, Stout & Andrews, 1975) with  $p = 0.031$ . All non-H atoms were found by direct methods followed by peak optimization (*SHELXS86*; Sheldrick, 1986). The structure was refined on *F* by full-matrix least-squares procedures using anisotropic thermal parameters for all non-H atoms (*SHELX76*; Sheldrick, 1976). All H atoms

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

$$U_{eq} = (1/3) \sum_i \sum_j U_{ij} a_i^* a_j^* a_i \cdot a_j$$

	x	y	z	$U_{eq}(\text{\AA}^2)$
Cl(1)	0.0434 (3)	0.1347 (4)	0.1969 (2)	0.043 (1)
Cl(2)	0.1681 (3)	0.4216 (5)	0.1128 (2)	0.058 (2)
O	$\frac{1}{2}$	0.266 (2)	0	0.059 (7)
N(1)	0.4305 (9)	0.608 (1)	0.1060 (6)	0.031 (4)
N(2)	0.2531 (9)	1.046 (1)	-0.1639 (6)	0.034 (4)
C(1)	0.303 (1)	0.859 (2)	-0.0086 (7)	0.030 (5)
C(2)	0.325 (1)	0.923 (2)	-0.0600 (7)	0.039 (5)
C(3)	0.424 (1)	0.925 (2)	-0.0744 (7)	0.035 (5)
C(4)	0.498 (1)	0.844 (2)	-0.0388 (8)	0.037 (5)
C(5)	0.479 (1)	0.781 (2)	0.0107 (9)	0.040 (6)
C(6)	0.382 (1)	0.780 (2)	0.0270 (7)	0.031 (5)
C(7)	0.360 (1)	0.717 (2)	0.0866 (7)	0.035 (5)
C(8)	0.406 (2)	0.511 (2)	0.0592 (9)	0.058 (7)
C(9)	0.420 (1)	0.574 (2)	0.1733 (8)	0.053 (6)
C(10)	0.243 (1)	1.020 (2)	-0.0942 (7)	0.034 (5)
C(11)	0.230 (1)	0.940 (2)	-0.2076 (8)	0.052 (6)
C(12)	0.181 (1)	1.148 (2)	-0.1888 (8)	0.040 (5)

Table 2. Bond distances ( $\text{\AA}$ ), angles ( $^\circ$ ) and hydrogen-bond geometry ( $\text{\AA}$ ,  $^\circ$ )

N(1)—C(7)	1.52 (2)	C(1)—C(6)	1.44 (2)	
N(1)—C(8)	1.43 (2)	C(2)—C(3)	1.40 (2)	
N(1)—C(9)	1.48 (2)	C(2)—C(10)	1.58 (2)	
N(2)—C(10)	1.51 (2)	C(3)—C(4)	1.41 (2)	
N(2)—C(11)	1.47 (2)	C(4)—C(5)	1.31 (2)	
N(2)—C(12)	1.50 (2)	C(5)—C(6)	1.38 (2)	
C(1)—C(1)'	1.50 (2)	C(6)—C(7)	1.51 (2)	
C(1)—C(2)	1.36 (2)			
C(7)—N(1)—C(8)	112 (1)	C(1)—C(2)—C(10)	118 (1)	
C(7)—N(1)—C(9)	106 (1)	C(3)—C(2)—C(10)	118 (1)	
C(8)—N(1)—C(9)	114 (1)	C(2)—C(3)—C(4)	117 (1)	
C(10)—N(2)—C(11)	113 (1)	C(3)—C(4)—C(5)	121 (1)	
C(10)—N(2)—C(12)	107 (1)	C(4)—C(5)—C(6)	123 (2)	
C(11)—N(2)—C(12)	111 (1)	C(1)—C(6)—C(5)	118 (1)	
C(1)'—C(1)—C(2)	123 (1)	C(1)—C(6)—C(7)	117 (1)	
C(1)'—C(1)—C(6)	119 (1)	C(5)—C(6)—C(7)	124 (1)	
C(2)—C(1)—C(6)	118 (1)	N(1)—C(7)—C(6)	112 (1)	
C(1)—C(2)—C(3)	123 (2)	N(2)—C(10)—C(2)	111 (1)	
D—H...A	D—A	D—H	H...A	D—H...A
N(1)—H(1)...Cl(2) <sup>ii</sup>	3.107 (13)	0.980 (17)	2.199 (13)	153.5 (12)
N(2)—H(2)...Cl(1) <sup>iii</sup>	3.059 (13)	0.980 (17)	2.121 (13)	159.6 (13)
O—H(103)...Cl(2) <sup>i</sup>	3.269 (11)	0.981 (14)	2.288 (11)	179.5 (16)

Symmetry code: (i)  $\frac{1}{2} - x, -y, z$ ; (ii)  $\frac{1}{2} + x, 1 - y, z$ ; (iii)  $\frac{1}{2} - x, 1 + y, -z$ .

were introduced at calculated positions (C—H, N—H, O—H = 0.98  $\text{\AA}$ ) and refined in the riding mode on their carrier atoms with three isotropic thermal parameters ( $U = 0.020\text{--}0.044 \text{\AA}^2$ ). Convergence reached at  $R = 0.151$ ,  $wR = 17.3$ ,  $w = 1$ . An irregular diffuse electron density was found at 3.8  $\text{\AA}$  from Cl(1) and 3.4  $\text{\AA}$  from Cl(2). This electron density was interpreted as originating from an additional disordered water molecule. To take the electron density into account in the refinement the BYPASS method was applied (van der Sluis & Spek, 1990). An electron count yields 28.7 electrons, equivalent to 0.72 molecules per biphenyl moiety. Convergence was reached at  $R = 0.131$  [ $wR = 0.169$ ,  $w = 1.0/[\sigma^2(F) + 0.00268F^2]$ ,  $S = 6.84$ , 153 parameters;

$(\Delta/\sigma)_{\max} = 0.1$ ,  $\langle \Delta/\sigma \rangle = 0.02$ }. Residual density within the range  $-0.69$  to  $1.02 \text{ e \AA}^{-3}$ . The relatively high final  $R$  value is related to the low quality of the data obtainable with the available sample and consistent with the flaky habit of the crystals. Scattering factors from Cromer & Mann (1968), anomalous-dispersion corrections from Cromer & Liberman (1970). The final atomic coordinates and equivalent isotropic temperature factors are listed in Table 1.\* The program package *EUCLID* (Spek, 1982) was used for geometrical calculations and illustrations. All calculations were carried out on a microVAX cluster. Table 1 gives atomic coordinates and Table 2 gives bond lengths, bond angles and hydrogen-bond geometry. Fig. 1 shows a thermal ellipsoid plot and Fig. 2 shows a projection of the structure along  $b$ .

\* Lists of anisotropic thermal parameters, H-atom positions, bond angles and distances involving H atoms, torsion angles, and structure factors have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52904 (12 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

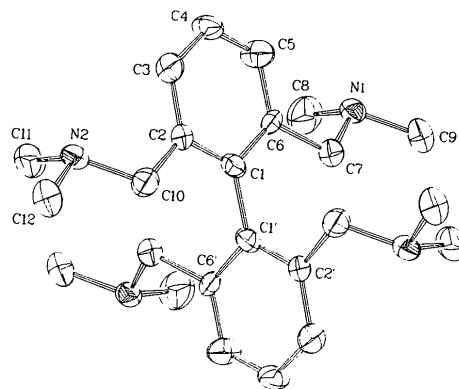


Fig. 1. Thermal-motion ellipsoid plot (50% probability level) of the cation projected down the twofold axis with adopted labeling.

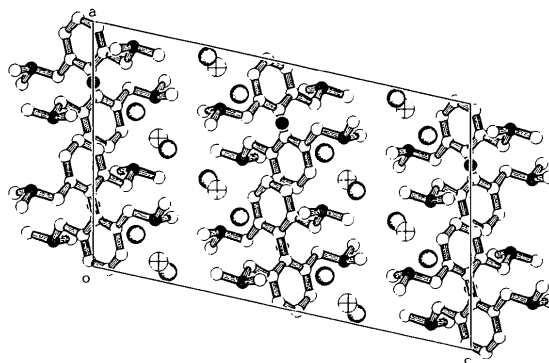


Fig. 2. Projection of the structure down the  $b$  axis. The centres of the disordered solvent areas have been indicated by crossed circles.

**Related literature.** Crystals of the title compound were obtained as an unintentional reaction product of the synthesis of related tantalum chlorine complexes (van der Zeijden, 1988; Abbenhuis, Grove, van Koten, van der Sluis & Spek, 1990).

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## Structure of *endo,endo*-2,3-Diphenyl-2,3-epoxybornane

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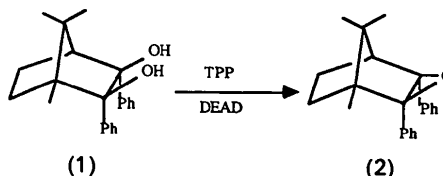
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**Abstract.** C<sub>22</sub>H<sub>24</sub>O, *M<sub>r</sub>* = 304.43, monoclinic, *C*2, *a* = 18.862 (3), *b* = 9.958 (2), *c* = 10.262 (1) Å, β = 117.47 (1)°, *V* = 1709.9 (4) Å<sup>3</sup>, *Z* = 4, *D<sub>x</sub>* = 1.182 g cm<sup>-3</sup>, λ(Mo *K*α) = 0.71073 Å, μ = 0.66 cm<sup>-1</sup>, *F*(000) = 656, *T* = 298 (2) K, *R* = 0.039 and *wR* = 0.039 for 2238 observed [*I*<sub>o</sub> ≥ 2.5σ(*I*)] reflections including Friedel pairs. The molecule is a diphenyl-substituted bornane system fused to an *exo*-oriented epoxide, typical of all known compounds of this type. The central six-membered ring is bent with an interplanar angle of 106.6 (1)°. The planar flap containing the apical C atom of the bornane adopts an unsymmetrical disposition relative to the two coalesced envelopes, canting toward the unsubstituted end of the bornane backbone with intersection angles of 122.8 (2) and 130.7 (2)°. The plane of the epoxide ring forms a dihedral angle of 111.3 (1)° at the epoxide–bornane interface. The torsion angles described by O—C—C—C(apical) are -27.9 (2) and 33.0 (3)°, and the distance between the centroids of the phenyl rings is 5.126 (3) Å. The three-membered

epoxide ring is characterized by (i) long C—O single-bond lengths [1.442 (2) and 1.454 (2) Å], (ii) small intra-ring bond angles [59.7 (1), 58.9 (1) and 61.4 (1)°], and (iii) a short C—C single-bond length [1.479 (3) Å]. These can be explained through an increase in *p*-orbital contribution in the C—O bonds with a concomitant decrease in *p*-character in the C—C bond. Other distances and angles are within published ranges for similar molecules.

**Experimental.** The title molecule (2) was prepared by dehydration of the precursor *endo,endo*-2,3-diphenylbornane-2,3-diol (1) via a *syn*-Mitsunobu reaction using the reagents triphenylphosphine (TPP) and diethyl azodicarboxylate (DEAD). Crystals were obtained from ethanol.



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Colorless triangular crystals, 0.36 × 0.36 × 0.36 mm; mounted in a thin-wall capillary, Nicolet