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Structures of the *cis* and *trans* Isomers of 2,4,6,6-Tetrachloro-2,4-bis(dimethylamino)cyclotri(phosphazene)*

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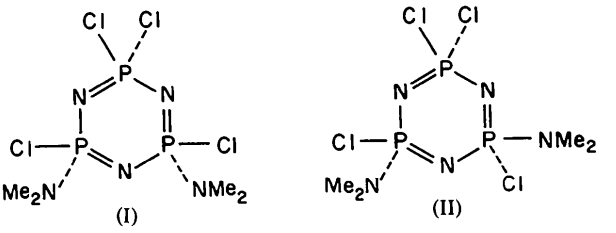
Abstract

The crystal structures of the *cis* and *trans* isomers of $N_3P_3Cl_4[N(CH_3)_2]_2$, $C_4H_{12}Cl_4N_5P_3$, have been determined. The *cis* isomer is orthorhombic, *Pnma*, a = 8·878 (2), b = 14·588 (3), c = 11·655 (3) Å, Z = 4, d_o = 1·602, d_c = 1·605 Mg m^{-3}; R = 0·060 and R_w = 0·069 for 1267 observed reflexions. The molecule has a crystallographic plane of symmetry, and its phosphazene ring is in a slight chair conformation. The *trans* isomer is triclinic, $P\bar{1}$, a = 8·578 (4), b = 11·920 (4), c = 8·512 (4) Å, α = 90·16 (3), β = 118·82 (3), γ = 93·38 (5)°, Z = 2, d_o = 1·595, d_c = 1·592 Mg m^{-3}; R = 0·066 and R_w = 0·075 for 2283 observed reflexions. The phosphazene ring in this molecule has a slight twist-boat conformation and is much flatter than in the *cis* isomer. The results indicate equal endocyclic P–N lengths, 1·577 (3) Å, in the $Cl(NMe_2)P$–N–$PCl(NMe_2)$ segment of both isomers. Slightly different P–N lengths, 1·568 (3) and 1·583 (3) Å, are observed in the Cl_2P–N–$PCl(NMe_2)$ segments of both, with the stronger bond next to the Cl_2 substituents. The observed P–Cl lengths compare favourably with the calculated values from the ^{35}Cl NQR frequencies. They are considerably shorter, 1·985 (3)–1·991 (3) Å, at the geminal than at the non-geminal P atoms, 2·027 (3)–2·052 (2) Å. The exocyclic P–N adjoining the elongated P–Cl are considerably shorter, 1·615 (4)–1·617 (4) Å, than where two NMe_2 are geminally substituted {1·640 (3) Å in geminal $N_3P_3Cl_3[N(CH_3)_2]_2$}.

Introduction

Connelly, Dalgleish, Harkins, Keat, Porte, Raitt & Shaw (1978) have discussed the use of ^{35}Cl NQR spectroscopy as an effective probe in studying Cl-containing derivatives of cyclotri(phosphazenes). Their technique was based partly on already available crystallographic results, and to demonstrate its applicability they predicted the unknown molecular structures and P–Cl lengths of the *cis* and *trans* isomers of $N_3P_3Cl_4(NMe_2)_2$, (I) and (II). The present analysis of these two compounds is intended to examine the accuracy of those predictions, and to provide additional results that might help to refine the proposed technique.



Experimental

Both compounds were crystallized from light petroleum as thin transparent plates. On extended exposure to X-rays, the crystals became opaque and their intensities dropped steadily. The cell parameters were measured on a Picker diffractometer with Cu radiation $\lambda(K\alpha_1)$ = 1·54050, $\lambda(K\alpha_2)$ = 1·54434 Å, from high-order axial reflexions [2θ = 58 to 97° for (I) and 62–76° for (II)] and their equivalents. The intensities were measured with Nb-filtered Mo radiation by the θ–2θ scan method at a 2θ scanning speed of 2° min^{-1}. The 2θ scan ranges were varied from 2·0 to 2·4°, and backgrounds were measured for 10 s with a stationary counter at the start and end of each scan. For each compound, two standard reflexions were scanned at regular intervals for scaling, and their intensities showed a maximum drop of 20%. The measurements covered all the unique reflexions in the range 2θ = 0–55°. The intensities were corrected for background, scale and Lorentz and polarization effects, but not for absorption. The intensities of the *trans* isomer were collected twice with two different crystals; the two sets were then averaged after proper scaling. The crystal data are presented in Table 1.

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Table 1. *Crystal data*

Formula: $C_4H_{12}Cl_4N_5P_3$; $M_r = 364{\cdot}91$

Isomer	*cis*	*trans*
Space group	*Pnma*	$P\bar{1}$
a (Å)	8·878 (2)	8·578 (4)
b (Å)	14·588 (3)	11·920 (4)
c (Å)	11·655 (3)	8·512 (4)
α (°)		90·16 (3)
β (°)		118·82 (3)
γ (°)		93·38 (5)
V (Å^3)	1509·5	760·7
Z	4	2
d_c (Mg m^{-3})	1·605	1·592
d_o (in KI solution) (Mg m^{-3})	1·602	1·595
Temperature (K)	297	295
μ(Cu $K\alpha$) (mm^{-1})	10·28	10·20
μ(Mo $K\alpha$) (mm^{-1})	1·08	1·08
Crystal dimensions (mm)	0·1, 0·3, 0·6	0·2, 0·3, 0·5
Reflexions measured	1799	3482
Reflexions observed (=m)	1267	2283
Parameters (=n)	95	193
Observations/parameters	13·3	11·8
R (obs. data)	0·060	0·066
R_w	0·069	0·075
Mean (shift/e.s.d.)	0·15	0·08
Maximum (shift/e.s.d.)	0·57	0·67
$[\sum w(\Delta F)^2/(m-n)]^{1/2}$	1·22	0·91

Structure determination

Both structures were determined by symbolic addition (Karle & Karle, 1963). For the orthorhombic *cis* isomer, three origin-defining reflexions and one symbol produced the signs of the 350 reflexions with $|E| > 1{\cdot}30$, and the corresponding E map gave the positions of the P, Cl and N atoms. The C atoms were then located from a Fourier map. For the triclinic *trans* isomer, it was necessary to start with a larger phase set comprising three for origin definition, two permuted with + and − signs, and two symbols. The permutation with the lowest number of $\sum_2$ contradictions, excluding the trivial solution with all positive signs, produced the correct solution. The corresponding E map which was calculated with 416 terms with $|E| > 1{\cdot}40$ gave the positions of all the non-hydrogen atoms. Difference maps, calculated after partial refinement, gave all 12 H positions in the *trans* isomer, but only four of the six non-equivalent H atoms in the *cis* isomer. These atoms were included in the refinement with isotropic temperature factors.

The refinement was by block-diagonal least squares (one block per atom), minimizing $\sum w(|F_o| - |F_c|)^2$, where $w = \{1 + [(|F_o| - p_2)/p_1]^2\}^{-1}$. The values of p_1 and p_2 were selected as 20 and 20 for the *cis* isomer, and 25 and 8 for the *trans* isomer, to make $\langle w(\Delta F)^2 \rangle$ independent of the $|F_o|$ amplitudes. Only reflexions with significant net counts were included in the refinement. A few weak reflexions (seven for the *cis* and three for the *trans*) showing the effect of multipole diffraction were excluded. The final R indices and other refinement indicators are included in Table 1. Scattering factors were those of Hanson, Herman, Lea & Skillman (1964) for the non-hydrogen atoms, and of Stewart, Davidson & Simpson (1965) for H. All calculations were performed with the NRC system of programs (Ahmed, Hall, Pippy & Huber, 1973).

Table 2. *Atomic parameters* ($\times 10^4$, *for* H $\times 10^3$) *for* *cis*-$N_3P_3Cl_4(NMe_2)_2$

	x	y	z	B_{eq} or B (Å^2)
N(1)	4005 (5)	1563 (3)	4972 (4)	4·2
P(2)	2586 (1)	1567 (1)	5799 (1)	3·1
N(3)	2150 (6)	2500 (0)	6395 (5)	3·7
P(6)	4615 (2)	2500 (0)	4486 (1)	3·4
Cl(1)	801 (2)	1164 (1)	4801 (2)	6·6
Cl(3)	4462 (4)	2500 (0)	2786 (2)	7·1
Cl(4)	6844 (2)	2500 (0)	4669 (2)	5·4
N(7)	2763 (5)	772 (3)	6756 (3)	4·0
C(1)	3121 (8)	−160 (4)	6362 (6)	5·6
C(2)	1861 (12)	822 (5)	7794 (7)	9·8
H(1,1)	358 (6)	−51 (4)	690 (5)	5·0 (1·3)
H(1,2)	409 (7)	−18 (5)	585 (6)	8·0 (1·7)
H(1,3)	214 (8)	−45 (5)	621 (6)	9·1 (1·9)
H(2,1)	205 (7)	52 (4)	825 (6)	7·3 (1·6)

Table 3. *Atomic parameters* ($\times 10^4$, *for* H $\times 10^3$) *for* *trans*-$N_3P_3Cl_4(NMe_2)_2$

	x	y	z	B_{eq} or B (Å^2)
N(1)	−1483 (6)	2352 (5)	27 (6)	5·0
P(2)	−3293 (2)	2427 (1)	76 (2)	3·9
N(3)	−3249 (6)	2444 (6)	1949 (6)	6·0
P(4)	−1425 (2)	2572 (1)	3756 (2)	3·8
N(5)	371 (6)	2600 (5)	3654 (6)	5·4
P(6)	332 (2)	2407 (1)	1820 (2)	3·9
Cl(1)	−4274 (3)	3890 (2)	−1085 (3)	6·4
Cl(2)	−1370 (3)	1200 (2)	5197 (3)	5·4
Cl(3)	1975 (3)	3572 (2)	1623 (2)	6·5
Cl(4)	1580 (3)	1032 (2)	1923 (3)	6·6
N(7)	−4718 (6)	1441 (4)	−1210 (6)	4·5
N(8)	−1380 (7)	3639 (5)	4955 (6)	4·5
C(1)	−4864 (10)	1227 (7)	−2979 (9)	6·5
C(2)	−6393 (9)	1244 (7)	−1152 (11)	5·8
C(3)	−3028 (12)	3968 (8)	4867 (12)	6·6
C(4)	247 (11)	3880 (6)	6687 (9)	6·7
H(1,1)	−517 (11)	38 (7)	−327 (11)	10·1 (2·2)
H(1,2)	−372 (10)	124 (6)	−301 (10)	8·2 (1·9)
H(1,3)	−576 (10)	164 (7)	−377 (10)	9·2 (2·1)
H(2,1)	−684 (11)	52 (7)	−147 (11)	10·2 (2·3)
H(2,2)	−630 (12)	126 (7)	29 (12)	10·8 (2·5)
H(2,3)	−715 (11)	182 (7)	−159 (12)	11·6 (2·5)
H(3,1)	−315 (12)	371 (8)	592 (12)	11·7 (2·6)
H(3,2)	−404 (11)	381 (7)	359 (11)	10·4 (2·3)
H(3,3)	−276 (12)	478 (8)	510 (12)	12·6 (2·6)
H(4,1)	151 (9)	360 (6)	673 (9)	7·7 (1·8)
H(4,2)	8 (12)	358 (8)	748 (12)	11·8 (2·5)
H(4,3)	32 (9)	465 (6)	708 (9)	7·8 (1·8)

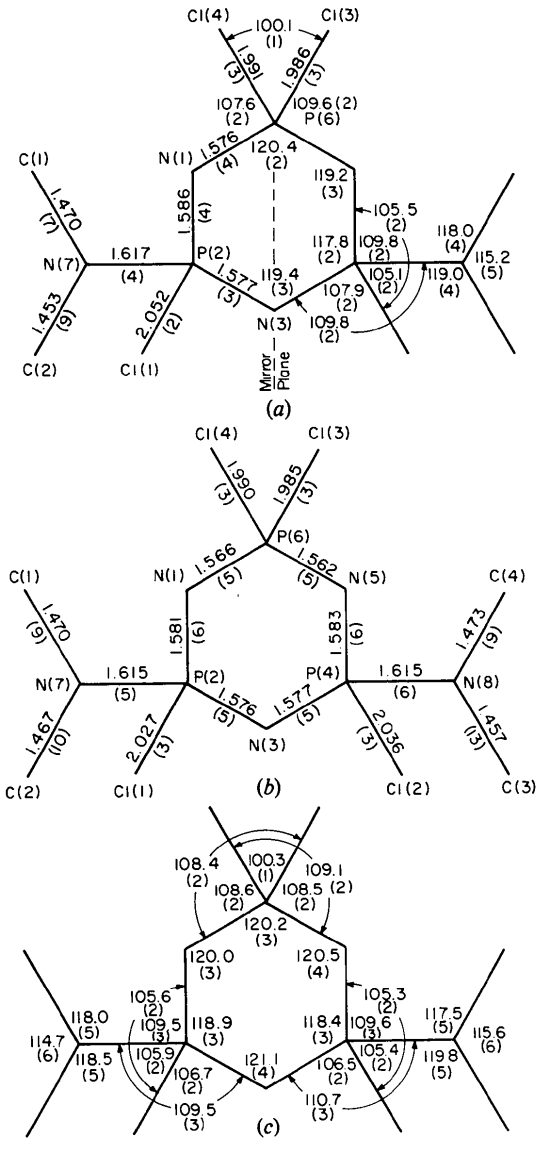

Fig. 1. Bond lengths (Å) and angles (°) (*a*) for the *cis* isomer; (*b*) and (*c*) for the *trans* isomer.

The refined atomic parameters are given in Tables 2 and 3,* bond lengths and angles in Fig. 1.

Discussion

Bond lengths

The cyclic P–N bonds in each $Cl(NMe_2)P–N–PCl(NMe_2)$ segment are of equal lengths, 1·577 (3) Å, regardless of whether the two Cl substituents are *cis* or *trans*, similar to the observations made in the corresponding segments of the *cis* and *trans* isomers of the non-geminal $N_3P_3Cl_3(NMe_2)_3$ (Ahmed & Pollard, 1972*a*; Ahmed & Gabe, 1975). On the other hand, the cyclic $Cl_2P^{x}–N^{y}PCl(NMe_2)$ segments have x and y as 1·576 (4) and 1·586 (4) Å in *cis*-$N_3P_3Cl_4(NMe_2)_2$, and 1·564 (5) and 1·582 (6) Å in the *trans* isomer. The differences $y - x$ of 0·010 (6) and 0·018 (8) Å are only possibly significant if considered by themselves. However, the difference $y - x$ becomes significant if the present results are combined with those of the geminal $N_3P_3Cl_3(NMe_2)_3$ (Ahmed & Pollard, 1972*b*), as shown in the top half of Table 4. The mean $y - x$ for the three compounds is 0·019 (4) Å for which $t = 4·8$ and $P < 0·001$, indicating a stronger P–N bond near the Cl_2 substituents. Considerably larger $y - x$ differences of 0·063 (5) and 0·059 (5) Å have been observed in the $Cl_2P^{x}–N^{y}P(NMe_2)_2$ and $Cl(NMe_2)P^{x}–N^{y}P(NMe_2)_2$ segments, respectively, of geminal $N_3P_3Cl_3(NMe_2)_3$ with the shorter bond always adjacent to the Cl substituent(s). A summary of the cyclic P–N lengths in these compounds is presented in the second half of Table 4. The mean of x and y is 1·577 Å for each P–N–P segment in Table 4(*b*), indicating that Cl and NMe_2 are of equal electronegativity, and that the difference $y - x$ should be attributed to more efficient orbital overlaps with Cl than with NMe_2.

Table 4. *Effect of the* Cl *and* NMe_2 *substituents on the endocyclic* P–N *lengths* (Å)

(*a*) For $Cl_2P^{x}–N^{y}PCl(NMe_2)$ segments

	x	y	$y - x$
Geminal $N_3P_3Cl_3(NMe_2)_3$	1·563 (4)	1·592 (4)	0·029 (6)
cis-$N_3P_3Cl_4(NMe_2)_2$	1·576 (4)	1·586 (4)	0·010 (6)
trans-$N_3P_3Cl_4(NMe_2)_2$	1·564 (5)	1·582 (6)	0·018 (8)
Mean	1·568 (3)	1·587 (3)	0·019 (4)

(*b*) Summary of the cyclic P–N

	x	y	$y - x$
$Cl_2P^{x}–N^{y}P(NMe_2)_2$[(i)]	1·545 (4)	1·608 (4)	0·063 (5)
$Cl(NMe_2)P–N–P(NMe_2)_2$[(i)]	1·547 (4)	1·606 (4)	0·059 (5)
$Cl_2P–N–PCl(NMe_2)$[(ii)]	1·568 (3)	1·587 (3)	0·019 (4)
$Cl(NMe_2)P–N–PCl(NMe_2)$[(iii)]	1·577 (3)	1·577 (3)	0·000 (4)

(i) In geminal $N_3P_3Cl_3(NMe_2)_3$.
(ii) Mean from Table 4(*a*).
(iii) Average of *cis*- and *trans*-$N_3P_3Cl_4(NMe_2)_2$.

The P–Cl lengths are in the ranges 1·986 (3)–2·052 (2) and 1·985 (3)–2·036 (3) Å in the *cis* and *trans* isomers, respectively. They are shortest at the geminally substituted P(6), and longest at P(2) and P(4) where P are non-geminally substituted. The observed lengths correlate fairly well with the values predicted from the revised relationship

$$d(\text{P–Cl}) = -0·0152\,\nu + 2·4166 \text{ Å}$$

connecting bond lengths, d (Å), and ^{35}Cl NQR frequencies, ν (MHz) (Connelly, Harkins, Porte, Shaw & van de Grampel, 1980), Table 5.

* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 35124 (26 pp.). Copies may be obtained though The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 5. *Observed and calculated* P—Cl *lengths from X-ray and* ^{35}Cl *NQR frequencies*

(*a*) X-ray results (Å).
(*b*) From d(P—Cl) = −0·0152 ν + 2·4166 Å.

[d at room temperature; ν (MHz) at 77 K from Figs. 3 and 4 of Connelly *et al.* (1978).]

		(*a*)	(*b*)
cis-$N_3P_3Cl_4(NMe_2)_2$			
ν_1	24·06	2·052 (2)	2·051 (5)
ν_2	27·38	1·991 (3)	2·000 (5)
ν_3	28·11	1·986 (3)	1·989 (5)
trans-$N_3P_3Cl_4(NMe_2)_2$			
ν_1	24·17	2·036 (3)	2·049 (5)
ν_2	24·67	2·027 (3)	2·042 (5)
ν_3	27·51	1·990 (3)	1·998 (5)
ν_4	27·61	1·985 (3)	1·997 (5)

The exocyclic P—NMe_2 are of lengths 1·617 (4) and 1·615 (5) Å, which are comparable to the mean of 1·620 (2) Å for the non-geminal P atoms of the three structures of $N_3P_3Cl_3(NMe_2)_3$. The corresponding value for P—$(NMe_2)_2$ in geminal $N_3P_3Cl_3(NMe_2)_3$ is considerably longer, 1·640 (3) Å. Thus, the results are consistently indicative of a longer P—Cl accompanied by a shorter P—NMe_2 where P is non-geminally substituted, than where it is geminally substituted.

Valency angles

The valency angles in the present structures are closely comparable to those reported for geminal and non-geminal $N_3P_3Cl_3(NMe_2)_3$ (Ahmed & Pollard, 1972*a*,*b*; Ahmed & Gabe, 1975). The cyclic P—N—P angles are 119·2 (3)–121·1 (4)°, and N—P—N 117·8 (2)–118·9 (3)° at P(2) and P(4) and 120·2 (3)–120·4 (2)° at P(6). The exocyclic Cl—P—Cl angles are 100·1 (1)–100·3 (1)° while the Cl—P—NMe_2 angles are 105·1 (2)–105·9 (2)°.

Molecular conformation

Projections of the two molecules on the mean planes through N(1), P(2), P(4) and N(5) are shown in Fig. 2, with the displacements of the ring atoms from those planes, and the endocyclic torsion angles.

The *cis* molecule has a crystallographic mirror plane through N(3), P(6), Cl(3) and Cl(4). Its phosphazene ring adopts a slight chair conformation with N(1), P(2), P(2′) and N(1′) exactly coplanar, while N(3) and P(6) are displaced by −0·317 (5) and 0·121 (2) Å from that plane. The two planes N(1)—P(6)—N(1′) and P(2)—N(3)—P(2′) make angles of 8·9 and 23·5°, respectively, with the seat of the chair. This molecular

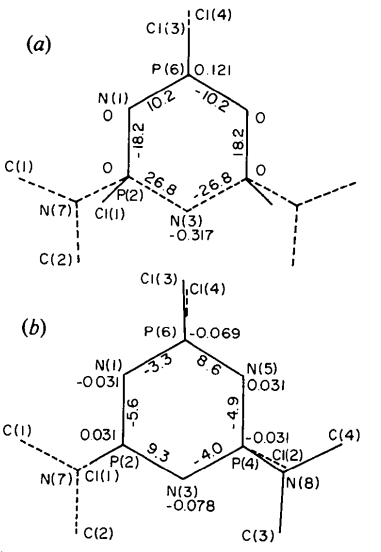

Fig. 2. Molecular projections on the mean planes of N(1), P(2), P(4) and N(5); displacements (Å) of the ring atoms from those mean planes; and the endocyclic torsion angles for (*a*) the *cis* and (*b*) the *trans* isomer. E.s.d.'s are ≤0·6° for torsion angles, and ≤0·004 Å for the displacements.

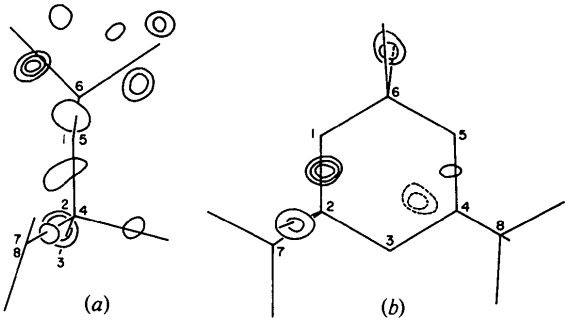

Fig. 3. Composite drawings of the residual electron distributions (e Å^{-3}) from the final difference maps for (*a*) the *cis* and (*b*) the *trans* isomer. Contour lines start at ±0·3, then at intervals of ±0·1 e Å^{-3}.

conformation was correctly predicted by Connelly *et al.* (1978).

The *trans* molecule has no crystallographic symmetry but its bond lengths and angles are almost identical across the plane through N(3), P(6), Cl(3) and Cl(4). Its phosphazene ring is in a slight twist-boat conformation, as shown by the torsion angles and the displacements given in Fig. 2(*b*). This ring is on the whole much flatter than that of the *cis* isomer. Its conformation was not quite correctly predicted by Connelly *et al.* (1978), but the observed and predicted conformations would correspond if the P(6)Cl_2 residue of the predicted conformation is raised by about 0·15 Å. The quadrupole resonance frequencies ν_3 and ν_4 must then be re-assigned.

Each of the exocyclic N atoms has a non-planar coordination. In the *cis* isomer, N(7) is 0·246 (4) Å

from the C(1), C(2), P(2) plane, and the mean angle at N(7) is only 117·4°. The corresponding values in the *trans* isomer are −0·261 (5) Å and 117·1° at N(7), and 0·236 (5) Å and 117·6° at N(8).

Residual electron densities

Two composite drawings of the residual electron distributions in the final difference maps of the two isomers are shown in Fig. 3(*a*),(*b*), where only the significant peaks and troughs are drawn. They occur in the ranges −0·28 to 0·54 and −0·44 to 0·56 e $Å^{-3}$ for the *cis* and *trans* isomers respectively, and are considered significant beyond ±0·3 e $Å^{-3}$. While most of the peaks are typical of bonding electrons, there is some residual density in the PCl_2 plane on the Cl(3)···Cl(4) vector, especially in the *trans* isomer.

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Structure and Synthesis of 14α-Ethyl-5α-cholest-7-ene-3β,15α-diol Di-*p*-bromobenzoate

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Abstract

14α-Ethyl-5α-cholest-7-ene-3β,15α-diol has been shown to be a very potent inhibitor of sterol biosynthesis in cultured animal cells. The chemical synthesis and crystal structure of 14α-ethyl-5α-cholest-7-ene-3β,15α-diol di-*p*-bromobenzoate are described. The compound crystallized in the space group $P2_1$, two molecules per unit cell, with cell dimensions: a = 10·948 (9), b = 6·332 (2), c = 28·983 (8) Å, β = 97·33 (5)°. The structure was solved by the heavy-atom method and refined by block-matrix anisotropic least squares to R = 7·9%. Both the 14-ethyl group and the O substituent at C(15) are on the α side of the steroid nucleus. Ring *B* has a half-chair conformation and the *C–D* ring juncture is *trans*.

Introduction

A number of 15-oxygenated sterols have been found to be potent inhibitors of sterol biosynthesis in animal cells in culture (Schroepfer, Parish, Chen & Kandutsch, 1976, 1977; Schroepfer, Parish & Kandutsch, 1977, 1979; Schroepfer, Raulston & Kandutsch, 1977; Schroepfer, Pascal & Kandutsch, 1980; Schroepfer, Parish, Tsuda, Raulston & Kandutsch, 1979). Moreover, several of these 15-oxygenated sterols have been shown to have significant hypocholesterolemic activity in animals (Raulston, Mishaw, Parish & Schroepfer, 1976; Kisic, Monger, Parish, Satterfield, Raulston & Schroepfer, 1977; Schroepfer, Monger, Taylor, Chamberlain, Parish, Kisic & Kandutsch, 1977; Kisic, Taylor, Chamberlain, Parish & Schroepfer, 1978). The most potent of these inhibitors of sterol biosynthesis described to date are 14α-ethyl-5α-cholest-7-ene-3β,15α-diol (Schroepfer, Parish & Kandutsch, 1977; Schroepfer, Parish, Tsuda, Raulston & Kandutsch, 1979) and its corresponding 3-keto derivative, 14α-ethyl-15α-hydroxy-5α-cholest-7-en-3-one (Schroepfer, Raulston & Kandutsch, 1977; Schroepfer, Parish, Tsuda, Raulston & Kandutsch, 1979). These compounds caused a 50% inhibition of sterol synthesis in *L* cells at 5 × 10^{-8} *M* and 6 × 10^{-9} *M*, respectively. In

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