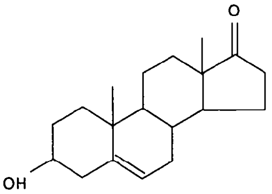
C—H and O—H bond distances were constrained to 1·00 (2) Å and the H atoms were given one of four common temperature factors (methyl or non-methyl in molecule *A* or *B*). Atomic scattering factors from *SHELX76*. Final $\Delta/\sigma \le 0·5$, $\Delta\rho$ max. = 0·1, $\Delta\rho$ min. = $-0·1$ e Å^{-3}. Molecular geometries were generated by the *GX* package (Mallinson & Muir, 1985).



(I)

Atomic coordinates are listed in Table 1, bond lengths and valency angles in Table 2 and short intermolecular O···O contacts in Table 3.* The atomic arrangement is shown in Fig. 1.

Related literature. DHEA may be obtained commercially or can be synthesized in high yield (Hosoda, Fukushima & Fishman, 1973). The hydrate of DHEA was isolated by us during a synthesis of 3,5-cycloandrost-6β-ol-17-one. Its conformation may be compared with crystal structures of other 5-ene steroids such as androst-5-ene-3,17-dione (Carrell, Glusker, Covey, Batzold & Robinson, 1978) and 17β-hydroxy-7α-methyl-5-androsten-3-one (Cox, Mkandawire & Mallinson, 1981).

Table 3. *Short* O···O *contacts* (Å) *with e.s.d.'s*

O(1)···O(3^{i})	2·87 (1)
O(1)···O(2^{ii})	2·87 (1)
O(2)···O(23^{iii})	3·03 (2)
O(3)···O(21)	2·77 (1)
O(3)···O(23^{iv})	2·76 (2)
O(21)···O(23)	2·76 (2)

Coordinates transposed by: (i) $2 - x$, $-\frac{1}{2} - y$, $-\frac{1}{2} - z$; (ii) x, $-1 + y$, $-1 + z$; (iii) $1\frac{1}{2} - x$, $-y$, $-\frac{1}{2} + z$; (iv) x, y, $-1 + z$.

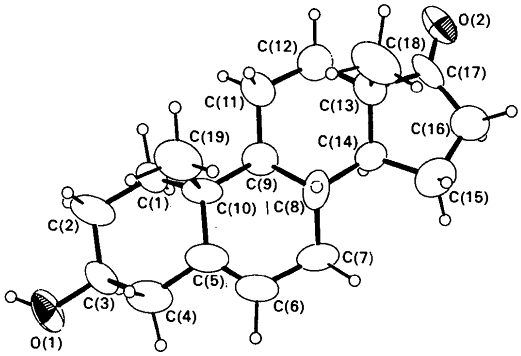


Fig. 1. The atomic arrangement in molecule *A* (50% probability ellipsoids).

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* Lists of structure factors, anisotropic thermal parameters, H-atom positions and torsion angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52309 (15 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Diisopropylammonium Chloride

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Abstract. $C_6H_{16}N^+.Cl^-$, M_r = 137·65, orthorhombic, $P2_12_12_1$, a = 7·825 (4), b = 8·257 (1), c = 13·268 (2) Å, V = 857·3 (5) Å^3, Z = 4, D_x = 1·066 g cm^{-3}, λ(Mo $K\alpha$) = 0·71073 Å, μ = 3·6 cm^{-1}, $F(000)$ = 304, T = 295 K, R = 0·030 for 556 observations (of 901 unique data). Each H on the N atom is hydrogen bonded to a Cl atom. N—H···Cl bond lengths (N—Cl distance) are 3·180 (3) and 3·163 (3) Å with N—H···Cl angles 176 (2) and 175 (2)° respectively. Each Cl atom is involved in two

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Table 1. *Coordinates and equivalent isotropic thermal parameters*

The equivalent isotropic thermal parameter, for atoms refined anisotropically, is defined by the equation: $B_{eq} = 8\pi^2(U_{11} + U_{22} + U_{33})/3$.

	x	y	z	B_{eq}(Å^2)
H′	0·937 (5)	0·304 (3)	0·602 (2)	3·6 (7)*
H″	0·800 (4)	0·298 (3)	0·534 (2)	2·7 (6)*
Cl	0·7237 (1)	0·2496 (1)	0·37219 (6)	4·05 (2)
N	0·8286 (4)	0·3262 (3)	0·5974 (2)	2·84 (5)
C1	0·8009 (5)	0·5046 (4)	0·6134 (2)	3·72 (8)
C2	0·6136 (6)	0·5464 (5)	0·6037 (4)	6·8 (1)
C3	0·9084 (7)	0·5930 (5)	0·5376 (3)	5·2 (1)
C4	0·7373 (5)	0·2114 (4)	0·6666 (3)	3·87 (7)
C5	0·7809 (6)	0·2491 (5)	0·7757 (3)	5·35 (9)
C6	0·7878 (6)	0·0401 (4)	0·6379 (3)	4·75 (9)

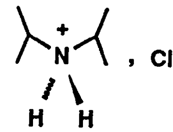
* B_{iso}.

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

N—C1	1·504 (4)	C1—C2	1·511 (6)
C4—C5	1·519 (5)	N—C4	1·501 (4)
C1—C3	1·501 (5)	C4—C6	1·517 (5)
N—H′	0·87 (3)	N—H″	0·90 (2)
C1—N—C4	117·6 (3)	C2—C1—C3	112·0 (3)
C5—C4—C6	111·9 (3)	N—C1—C2	110·6 (3)
N—C4—C5	110·3 (3)	N—C1—C3	107·5 (3)
N—C4—C6	108·1 (3)		
C1—N—H′	109 (2)	C1—N—H″	110 (2)
C4—N—H′	107 (2)	C4—N—H″	107 (2)
H′—N—H″	105 (3)		

hydrogen bonds. The cation has an approximate twofold axis of symmetry.

Experimental. Colorless needles of (1), m.p. 389–390 K, were isolated as an ether-insoluble by-product of a reaction and recrystallized in methanol/ether.



(1)

Crystal size 0·55 × 0·18 × 0·15 mm, capillary mounted, space group from systematic absences $h00$ with h odd, $0k0$ with k odd and $00l$ with l odd, cell dimensions from setting angles of 25 reflections having $10 < \theta < 13°$. Data collection on an Enraf–Nonius CAD-4 diffractometer, Mo $K\alpha$ radiation, graphite monochromator, ω–2θ scans designed for $I = 25\sigma(I)$, subject to max. scan time = 120 s, scan rates varied 0·69–4·00° min^{-1}. Two octants, $2 < 2\theta < 50°$, $-9 \leq h \leq 9$, $0 \leq k \leq 9$, $0 \leq l \leq 15$ were collected. Data corrected for background, Lorentz and polarization effects. No absorption correction was made. Standard reflections 200, 040, 004 indicated a 0·8% intensity decrease, for which a linear correction was applied. The two octants were averaged, $R_{int} = 0{\cdot}029$, to yield 901 unique data, 556 observed with $I > 3\sigma(I)$. Structure solved by direct methods, using *MULTAN* (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1982), refined by full-matrix least squares where the function minimized was $\sum w(|F_o| - |F_c|)^2$ and weights were assigned as $w = 4F_o^2\mathrm{Lp}[S^2(C + R^2B) + (0{\cdot}02F_o^2)^2]^{-1}$, where S = scan rate, C = total integrated peak count, R = scan time/background counting time, B = total background count, Lp = Lorentz–polarization factor, using Enraf–Nonius *SDP* (Frenz & Okaya, 1980), scattering factors of Cromer & Waber (1974) and anomalous coefficients of Cromer (1974). Non-H atoms refined anisotropically; H atoms located by ΔF and included as fixed contributions with C—H 0·95 Å and $B = 1{\cdot}3B_{eq}$ of the bonded C atom, except for the two H atoms on the N which were refined isotropically. The final cycle included 82 variables and converged (largest $\Delta/\sigma < 0{\cdot}01$) with R

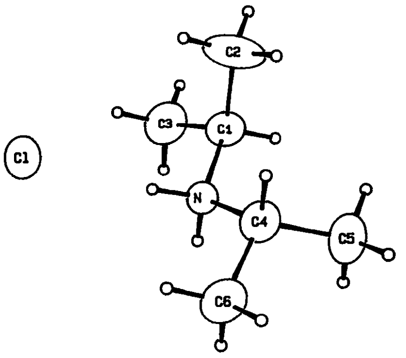


Fig. 1. Numbering scheme and thermal ellipsoids drawn at the 40% probability level. H atoms are drawn as circles with the same arbitrary radius.

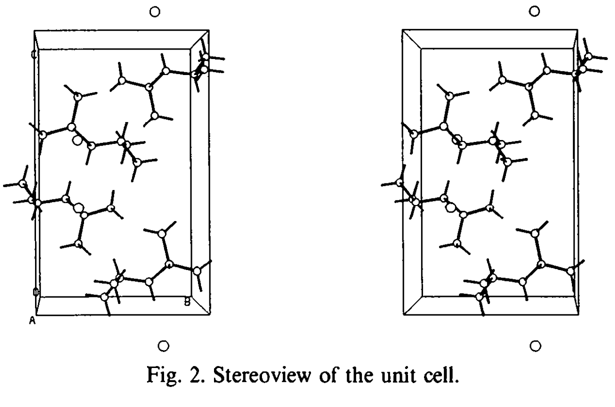


Fig. 2. Stereoview of the unit cell.

$= 0{\cdot}030$, $wR = 0{\cdot}033$, $R(\text{all}) = 0{\cdot}102$ and $S = 1{\cdot}295$. Max. residual density 0·22, min. $-0{\cdot}18$ e Å^{-3}, extinction coefficient $g = 9\,(10) \times 10^{-8}$. Coordinates* are given in Table 1; bond distances and angles are given in Table 2. The molecule is illustrated in Fig. 1, and a diagram of the unit cell is shown in Fig. 2.

Related literature. Crystal structures of diisopropylammonium diisopropyldithiocarbamate (Wahlberg, 1978) and of the diisopropylammonium salt of an organoplatinate (Dell'Amico, Calderazzo & Pelizzi, 1979).

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Structure of Peperomin B

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Abstract. $C_{23}H_{26}O_8$, $M_r = 430{\cdot}46$, monoclinic, $P2_1$, $a = 11{\cdot}044\,(1)$, $b = 9{\cdot}500\,(2)$, $c = 11{\cdot}696\,(2)$ Å, $\beta = 112{\cdot}39\,(1)°$, $U = 1135$ Å^3, $Z = 2$, $D_x = 1{\cdot}2600$ Mg m^{-3}, $\lambda(\text{Mo } K\alpha) = 0{\cdot}71069$ Å, $\mu = 0{\cdot}104$ mm^{-1}, $F(000) = 456$, $T = 297\,(5)$ K, $R(F) = 0{\cdot}034$ for 2191 observed reflections [$I > 3\sigma(I)$]. The molecule has 1*S*, 2*S* and 4*S* chiral centers. This work confirmed the unusual *seco* structure and stereochemistry of peperomin B. The three rings (*A*, *B*, *C*) make rather large dihedral angles with each other.

Experimental. Recrystallized from a 25% aqueous solution of 2-propanol–methanol. CAD-4 diffractometer, graphite monochromator, translucent prismatic crystal (0·35 × 0·40 × 0·50 mm) used for data collection, unit-cell parameters from 25 reflections with $11 < 2\theta < 25°$, data collected by ω–2θ scans, with scan parameters $2(0{\cdot}8 + 0{\cdot}35\tan\theta)°$ and with scan speed 20/10 to 20/3° min^{-1}, three standard reflections ($\bar{5}35$, $\overline{53}5$, $35\bar{1}$) checked every 2 h varied within $2\sigma(I)$. Max. $(\sin\theta)/\lambda = 0{\cdot}7035$ Å^{-1} ($-15 \le h \le 15$, $0 \le k \le 13$, $0 \le l \le 16$), 3490 reflections collected, 2191 significant with $I > 3\sigma(I)$. Empirical absorption correction based on azimuthal rotation from reflections 021, 020 and $0\bar{2}0$ (North, Phillips & Mathews, 1968). Minimum, maximum and average correction factors were 0·94, 0·99, 0·97, respectively. The structure was solved by direct methods, using *MULTAN*82 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1982). Block-diagonal least-squares refinement was carried out on positional and anisotropic thermal parameters of non-H atoms over 2191 reflections. The function minimized is $\sum w(|F_o| - |F_c|)$, where $w = 1/\sigma^2(F)$ from counting statistics. Positions of the H atoms were geometrically calculated and not refined. In the last stage of least-squares calculation, using the *CRYLSQ* program (Olthof-Hazekamp, 1988), the $R(F)$ factor reduced to 0·034, $S = 0{\cdot}375$, $(\Delta\rho)_{\max} = 0{\cdot}25$ e Å^{-3}, $(\Delta/\sigma)_{\max} = 0{\cdot}0037$. Atomic scattering factors were taken from *International Tables for X-ray Crystallography* (1974). Final positional and iso-

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