= 0·030, wR = 0·033, R(all) = 0·102 and S = 1·295. Max. residual density 0·22, min. −0·18 e Å^{-3}, extinction coefficient g = 9 (10) × 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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* Lists of H-atom coordinates, anisotropic thermal parameters and structure-factor amplitudes have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52238 (10 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Structure of Peperomin B

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Abstract. $C_{23}H_{26}O_8$, M_r = 430·46, monoclinic, $P2_1$, a = 11·044 (1), b = 9·500 (2), c = 11·696 (2) Å, β = 112·39 (1)°, U = 1135 Å^3, Z = 2, D_x = 1·2600 Mg m^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, μ = 0·104 mm^{-1}, F(000) = 456, T = 297 (5) K, $R(F)$ = 0·034 for 2191 observed reflections [$I > 3\sigma(I)$]. The molecule has 1S, 2S and 4S 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 < 2θ < 25°, data collected by ω–2θ scans, with scan parameters 2(0·8 + 0·35tanθ)° and with scan speed 20/10 to 20/3° min^{-1}, three standard reflections ($\bar{5}35$, $\bar{5}\bar{3}5$, $35\bar{1}$) checked every 2 h varied within 2$\sigma(I)$. Max. (sinθ)/λ = 0·7035 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 = 0·375, $(\Delta\rho)_{\max}$ = 0·25 e Å^{-3}, $(\Delta/\sigma)_{\max}$ = 0·0037. Atomic scattering factors were taken from *International Tables for X-ray Crystallography* (1974). Final positional and iso-

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Table 1. *Atomic positional coordinates and thermal parameters of non-*H *atoms*

	x	y	z	U_{iso}(Å^2)
C(1)	0·9051 (2)	0·5606 (3)	0·2608 (2)	0·044 (2)
C(2)	0·9425 (3)	0·4935 (3)	0·1601 (3)	0·052 (2)
C(3)	0·8267 (3)	0·4121 (4)	0·0682 (3)	0·066 (3)
C(4)	1·0478 (3)	0·3791 (4)	0·2070 (3)	0·066 (3)
C(5)	1·0063 (4)	0·2733 (4)	0·1023 (3)	0·078 (3)
C(6)	1·1888 (4)	0·4239 (6)	0·2421 (5)	0·112 (5)
C(7)	0·7905 (2)	0·6626 (3)	0·2117 (2)	0·043 (2)
C(8)	0·7657 (3)	0·7414 (3)	0·1047 (3)	0·049 (2)
C(9)	0·6664 (3)	0·8388 (4)	0·0766 (3)	0·052 (2)
C(10)	0·5365 (4)	1·0222 (5)	0·0033 (5)	0·099 (4)
C(11)	0·5940 (3)	0·8575 (4)	0·1473 (3)	0·062 (2)
C(12)	0·6153 (3)	0·7798 (5)	0·2507 (3)	0·076 (3)
C(13)	0·7160 (3)	0·6803 (4)	0·2824 (3)	0·060 (2)
C(14)	0·5596 (7)	0·7197 (11)	0·4190 (5)	0·260 (9)
C(15)	1·0186 (2)	0·6350 (3)	0·3602 (2)	0·044 (2)
C(16)	1·0439 (3)	0·6024 (3)	0·4829 (3)	0·049 (2)
C(17)	1·1422 (3)	0·6743 (4)	0·5765 (4)	0·052 (2)
C(18)	1·2173 (3)	0·7735 (4)	0·5483 (4)	0·054 (2)
C(19)	1·1915 (3)	0·8057 (4)	0·4240 (4)	0·054 (4)
C(20)	1·0919 (3)	0·7367 (3)	0·3302 (3)	0·049 (2)
C(21)	1·2563 (3)	0·9342 (5)	0·2817 (5)	0·081 (3)
C(22)	1·4330 (4)	0·7736 (7)	0·6921 (7)	0·105 (4)
C(23)	1·0975 (3)	0·5468 (4)	0·7305 (4)	0·068 (3)
O(1)	0·8835 (3)	0·2969 (3)	0·0240 (3)	0·084 (2)
O(2)	1·0711 (3)	0·1815 (3)	0·0860 (3)	0·105 (3)
O(3)	0·6252 (2)	0·9302 (3)	−0·0225 (3)	0·076 (2)
O(4)	0·5011 (2)	0·9615 (3)	0·0962 (3)	0·090 (2)
O(5)	0·5363 (3)	0·8020 (5)	0·3140 (5)	0·139 (3)
O(6)	1·2702 (2)	0·9058 (3)	0·4063 (3)	0·075 (2)
O(7)	1·3124 (2)	0·8467 (3)	0·6408 (3)	0·074 (2)
O(8)	1·1707 (2)	0·6530 (3)	0·6999 (3)	0·066 (2)

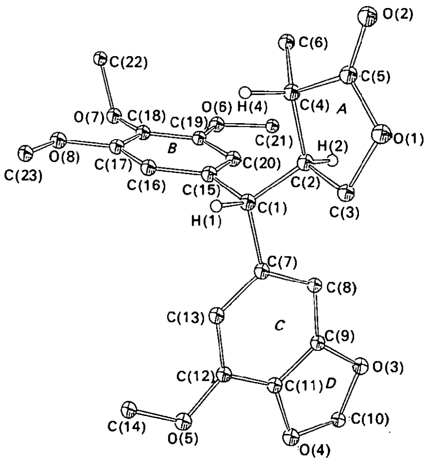

Fig. 1. *ORTEP* (Johnson, 1976) perspective view showing the atom-numbering scheme. H atoms at the chiral centers are shown as open circles; other atoms are shown as shaded spheres. The breaking of the C(6)—C(20) bond results in a large twist angle between the trimethoxybenzene and the γ-butyrolactone rings.

tropic thermal parameters are listed in Table 1* and selected bond distances and angles in Table 2. The molecule and numbering scheme are shown in Fig. 1 and a stereoview of the unit cell is presented in Fig. 2.

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

C(1)—C(2)	1·528 (5)	C(11)—O(4)	1·385 (4)
C(1)—C(7)	1·522 (4)	C(12)—C(13)	1·397 (5)
C(1)—C(15)	1·520 (3)	C(12)—O(5)	1·359 (5)
C(2)—C(3)	1·531 (4)	C(13)—C(7)	1·382 (5)
C(2)—C(4)	1·533 (4)	C(14)—O(5)	1·395 (8)
C(3)—O(1)	1·451 (5)	C(15)—C(16)	1·389 (4)
C(4)—C(5)	1·514 (5)	C(15)—C(20)	1·389 (4)
C(4)—C(6)	1·513 (6)	C(16)—C(17)	1·391 (4)
C(5)—O(1)	1·335 (4)	C(17)—C(18)	1·377 (5)
C(5)—O(2)	1·188 (5)	C(17)—O(8)	1·369 (4)
C(7)—C(8)	1·393 (4)	C(18)—C(19)	1·404 (4)
C(8)—C(9)	1·376 (4)	C(18)—O(7)	1·375 (3)
C(9)—O(3)	1·380 (4)	C(19)—C(20)	1·387 (4)
C(10)—O(3)	1·429 (6)	C(19)—O(6)	1·356 (4)
C(10)—O(4)	1·410 (7)	C(21)—O(6)	1·433 (5)
C(11)—C(9)	1·363 (5)	C(22)—O(7)	1·417 (5)
C(11)—C(12)	1·358 (5)	C(23)—O(8)	1·422 (5)

C(2)—C(1)—C(7)	113·9 (2)	C(11)—C(12)—O(5)	118·1 (3)
C(2)—C(1)—C(15)	113·4 (2)	C(13)—C(12)—O(5)	124·8 (4)
C(7)—C(1)—C(15)	108·5 (2)	C(7)—C(13)—C(12)	121·2 (3)
C(1)—C(2)—C(3)	110·7 (3)	C(1)—C(15)—C(16)	118·0 (3)
C(1)—C(2)—C(4)	114·3 (2)	C(1)—C(15)—C(20)	121·4 (2)
C(3)—C(2)—C(4)	102·1 (3)	C(16)—C(15)—C(20)	120·6 (2)
C(2)—C(3)—O(1)	105·8 (3)	C(15)—C(16)—C(17)	119·6 (3)
C(2)—C(4)—C(5)	103·2 (2)	C(16)—C(17)—C(18)	120·6 (3)
C(2)—C(4)—C(6)	117·4 (3)	C(16)—C(17)—O(8)	123·6 (3)
C(5)—C(4)—C(6)	111·6 (4)	C(18)—C(17)—O(8)	115·8 (2)
C(4)—C(5)—O(1)	111·0 (3)	C(17)—C(18)—C(19)	119·6 (2)
C(4)—C(5)—O(2)	127·0 (3)	C(17)—C(18)—O(7)	120·5 (3)
O(1)—C(5)—O(2)	122·0 (3)	C(19)—C(18)—O(7)	119·9 (3)
C(1)—C(7)—C(8)	122·1 (3)	C(18)—C(19)—C(20)	120·2 (3)
C(1)—C(7)—C(13)	116·8 (3)	C(18)—C(19)—O(6)	114·9 (2)
C(8)—C(7)—C(13)	120·9 (3)	C(20)—C(19)—O(6)	124·9 (3)
C(7)—C(8)—C(9)	116·2 (3)	C(15)—C(20)—C(19)	119·5 (3)
C(8)—C(9)—C(11)	122·8 (3)	C(3)—O(1)—C(5)	110·3 (3)
C(8)—C(9)—O(3)	126·9 (3)	C(9)—O(3)—C(10)	103·9 (3)
C(11)—C(9)—O(3)	110·2 (3)	C(10)—O(4)—C(11)	104·3 (3)
O(3)—C(10)—O(4)	109·0 (3)	C(12)—O(5)—C(14)	116·9 (5)
C(9)—C(11)—C(12)	121·7 (3)	C(19)—O(6)—C(21)	117·4 (2)
C(9)—C(11)—O(4)	109·9 (3)	C(18)—O(7)—C(22)	113·5 (3)
C(12)—C(11)—O(4)	128·4 (4)	C(17)—O(8)—C(23)	116·5 (2)
C(11)—C(12)—C(13)	117·1 (4)		

C(1)—C(2)—C(3)—O(1)	−149·2 (3)	C(16)—C(15)—C(1)—C(7)	103·4 (3)
C(1)—C(2)—C(4)—C(5)	144·2 (3)	C(15)—C(1)—C(7)—C(13)	−79·7 (3)
C(2)—C(1)—C(15)—C(20)	53·5 (4)	C(15)—C(1)—C(7)—C(8)	96·5 (3)
C(4)—C(2)—C(1)—C(15)	61·5 (3)	C(1)—C(7)—C(8)—C(9)	−174·3 (2)
C(2)—C(1)—C)15)—C(16)	−129·0 (3)	C(7)—C(8)—C(9)—O(3)	178·8 (3)

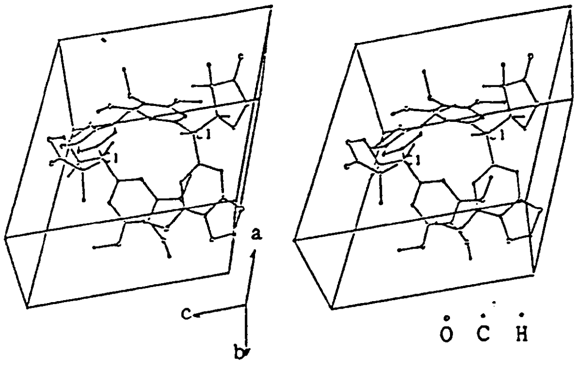

Fig. 2. Stereoscopic view, showing how the peperomin B molecules are packed in a unit cell.

Related literature. Peperomin B is one of the lignans separated from *Peperomia japonica* Makino (Piperaceae) as described in our recent work (Chen, Jan, Chen & Lee, 1989). An unusual aspect of this structure is the fission of the C(6)—C(20) bond which

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* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52237 (31 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

forms a six-membered ring between the γ-butyrolactone and the trimethoxybenzene ring in normal phenyltetrahydronaphthalene-type lignans.

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Reinvestigation of the Structure of Hypoxanthinium Nitrate Monohydrate

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Abstract. $C_5H_5N_4O^+.NO_3^-.H_2O$, $M_r = 217{\cdot}14$, orthorhombic, *Pnma*, $a = 13{\cdot}699$ (3), $b = 6{\cdot}264$ (4), $c = 10{\cdot}069$ (2) Å, $V = 864{\cdot}0$ Å^3, $Z = 4$, $D_x = 1{\cdot}669$ g cm^{-3}, λ(Mo $K\alpha$) = 0·71073 Å, $\mu = 1{\cdot}02$ cm^{-1}, $F(000) = 448$, room temperature, $R = 0{\cdot}054$ for 1154 observed reflections. As part of our investigations on metal–hypoxanthine complexes, the structure of hypoxanthine nitrate monohydrate was redetermined in order to provide more reliable data on protonated hypoxanthine for structural comparison purposes. The reinvestigation, confirming the general structural features of the compound reported by Rosenstein, Oberding, Hyde, Zubieta, Karlin & Seeman [*Cryst. Struct. Commun.* (1982), **11**, 1507–1513] resulted in bond distances and angles with e.s.d.'s of 0·003 Å and 0·2° for the hypoxanthinium cation and of 0·002 Å and 0·2° for the nitrate group. These e.s.d.'s are smaller by a factor of about seven compared with the values reported by Rosenstein *et al.* (1982), which had been obtained by a refinement based on 253 observed reflections only. In addition, the reinvestigation of the structure yielded new positions for the water H atoms and some significant differences in the positions of the hypoxanthine H atoms. Since all H atoms were localized in difference Fourier maps and successfully refined, the existence of N(1)-, N(7)- and N(9)-protonated hypoxanthinium cations, nitrate anions and non-protonated water molecules could be confirmed. The hypoxanthinium and the nitrate ions are planar due to unit-cell symmetry. Each hypoxanthinium cation is surrounded by three different nitrate groups and *vice versa*, forming layers perpendicular to the *b* axis of the cell with an interplanar spacing of $b/2 = 3{\cdot}132$ (4) Å. This layered structure is stabilized by an extended hydrogen-bonding system. The H atoms H(1), H(7) and H(9) of the hypoxanthinium ion are involved in hydrogen bonds of the type N—H···O with the adjacent nitrate groups, whereas the water molecules are hydrogen bonded to N(3) and O(6) of the hypoxanthinium cation and to O(1) of the nitrate group. The hydrogen bonds involving H(1) and H(42) are bifurcated. Despite the pronounced layer-type structure of the compound, no direct purine–purine stacking is observed. In contrast, the nitrate groups are stacked approximately on top of each other rotated by about 180°, forming infinite columns along the *b* axis of the cell.

Experimental. Single crystals of hypoxanthinium nitrate monohydrate were prepared by dissolving 200 mg (1·47 mmol) hypoxanthine in 120 ml 2·5*M* HNO_3 at 370 K. After crystallization for 3 weeks at room temperature, needle-shaped crystals suitable for X-ray investigations could be isolated from this solution. Composition: calculated C 27·66, H 3·25, N 32·25%; observed C 27·87, H 3·52, N 32·52%.

A transparent crystal with dimensions 0·72 × 0·25 × 0·21 mm was mounted along its needle axis (*b*) on an Enraf–Nonius CAD-4 diffractometer with graphite-monochromatized Mo $K\alpha$ radiation. The lattice constants were derived from least-squares

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