Fig. 3. Stereoscopic view with thermal ellipsoids at 30% probability.

Table 3. *Hydrogen-bond lengths* (Å) *and angles* (°)

Donor atom	Acceptor atom	Position of acceptor atom	Distance D···A	H···A	Angle	
O(1)	O(2')	$x, y, 1 + z$	2·768 (6)	1·81 (7)	O(1)—H(13)—O(2')	175 (7)
O(2)	O(M)	$\frac{1}{2} + x, \frac{1}{2} - y, 2 - z$	2·720 (8)	1·89 (7)	O(2)—H(3b)—O(M)	164 (7)
O(W)	O(1)	x, y, z	2·805 (8)	1·83 (5)	O(W)—H(O Wb)—O(1)	162 (4)
O(W)	O(2)	x, y, z	2·775 (8)	1·90 (5)	O(W)—H(O Wa)—O(2)	157 (5)
O(M)	O(W)	x, y, z	2·654 (9)	1·79 (7)	O(M)—H(O M)—O(W)	144 (6)

Discussion. The crystal structure is shown in Fig. 1. Five intermolecular hydrogen bonds (Table 3) connect the molecules to form a two-dimensional sheet.

The bond lengths and angles (Fig. 2) are normal within experimental error. A stereoscopic drawing of the molecule is shown in Fig. 3. The two condensed rings, *A* and *B*, are in the chair form. The six atoms of the pyrone ring (*C*) are planar within experimental error and form an angle of 60·4° with the plane of the methoxycarbonyl group. This explains the UV absorption, $\lambda_{max} = 260$ nm (Suzuki, Gohbara, Kosuge, Tamura, Ohashi & Sasada, 1976). While this paper was being prepared, the molecular structure of the monoacetate of this compound was reported (Goddard, Hatton, Howard, MacMillan & Gilmore, 1976). Its

structure is consistent with the present one, except for minor details.

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Iodophyllanthin

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Abstract. $C_{24}H_{32}I_2O_6$, $M_r = 670·3$, orthorhombic, $C222_1$, $a = 7·388$ (4), $b = 21·967$ (9), $c = 16·266$ (8) Å, $V = 2639·8$ Å³, $D_m = 1·63$, $Z = 4$, $D_x = 1·686$ g cm⁻³. Mo radiation, $R = 0·049$ for 1174 reflexions. The molecule lies on a crystallographic twofold axis, and has normal dimensions.

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Introduction. Unit-cell and intensity data (systematic absences: hkl, $h + k \neq 2n$, $00l$, $l \neq 2n$) were measured

on a Datex-automated GE XRD 6 diffractometer, with Mo $K\alpha$ radiation (Zr filter and pulse-height analyser), a θ–2θ scan, and check-reflexion scaling. Of 1586 reflexions with $2\theta \leq 60°$, 1176 with $I \geq 3\sigma(I)$ were classified as observed [$\sigma^2(I) = S + B + (0{\cdot}05\,S)^2$, where S = scan and B = background counts]. The crystal [prepared by treatment of phyllanthin with ICl (Row, Srinivasulu, Smith & Subba Rao, 1966)] had dimensions of about 0·2–0·3 mm, and no absorption correction was made (μ = 24 cm^{-1}, maximum error in $|F_o|$ about 6%). The structure was solved from Patterson and electron-density maps, and refined by full-matrix least-squares methods, with minimization of $\Sigma\,w(|F_o| - |F_c|)^2$; $w = (31 - 1{\cdot}2|F_o| + 0{\cdot}02|F_o|^2)^{-1}$ gave approximately uniform values of $w(|F_o| - |F_c|)^2$ over ranges of $|F_o|$. Atomic scattering factors and anomalous scattering factors for I were taken from *International Tables for X-ray Crystallography* (1962). Calculations were performed with local versions of standard crystallographic programs. The final R and R_w for 1174 reflexions* were 0·049 and 0·063 (001 and 020 were given zero weight because of suspected extinction errors); a final difference synthesis contained random fluctuations of ±0·5 e Å^{-3}, and H atoms could not be located. Final positional parameters are in Table 1. The analysis was undertaken to verify the conclusions from chemical studies, and the absolute configuration was assumed to be that determined by chemical methods (Row, Srinivasulu, Smith & Subba Rao, 1966; Row, Satyanarayana & Subba Rao, 1967).

* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 32372 (4 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

Table 1. *Positional parameters and standard deviations*
($\times 10^4$)

	x	y	z
I	1153 (1·5)	4129 (0·3)	5094 (0·5)
C(1)	1593 (14)	3377 (5)	4319 (8)
C(2)	2046 (15)	3446 (5)	3499 (7)
C(3)	2330 (18)	2910 (5)	3044 (7)
C(4)	2146 (18)	2341 (5)	3374 (7)
C(5)	1685 (16)	2276 (5)	4222 (7)
C(6)	1422 (15)	2810 (5)	4674 (6)
C(7)	2623 (26)	1820 (9)	2094 (10)
C(8)	1446 (24)	1644 (6)	5386 (10)
C(9)	2324 (15)	4060 (5)	3078 (7)
C(10)	989 (15)	4196 (4)	2361 (6)
C(11)	1550 (18)	4782 (5)	1927 (7)
C(12)	3922 (31)	5248 (9)	1202 (13)
O(1)	2320 (17)	1794 (4)	2982 (6)
O(2)	1588 (18)	1705 (4)	4514 (6)
O(3)	3227 (14)	4703 (4)	1538 (6)

Discussion. The general molecular structure is as determined by chemical methods (Row *et al.*, 1966, 1967; Krishna Rao & Murthy, 1968). The molecule has C_2 symmetry (Fig. 1) and lies on a twofold crystallographic axis parallel to **b**. The plane of an aromatic ring and its four immediate substituent atoms has the equation: $0{\cdot}9704X + 0{\cdot}0056Y + 0{\cdot}2412Z = 2{\cdot}8920$ (X,Y,Z are coordinates in Å); neighbouring substituents are displaced slightly from the plane in opposite directions [I −0·02 and C(9) +0·03 Å; O(1) −0·04 and O(2) +0·04 Å], suggesting minor steric interference between neighbouring groups. The two aromatic planes in the molecule are nearly parallel to each other (angle 1°) and to **b**, the shortest intramolecular distances between atoms of the two rings being about 3·8 Å. The conformation of the aliphatic chain is given by the
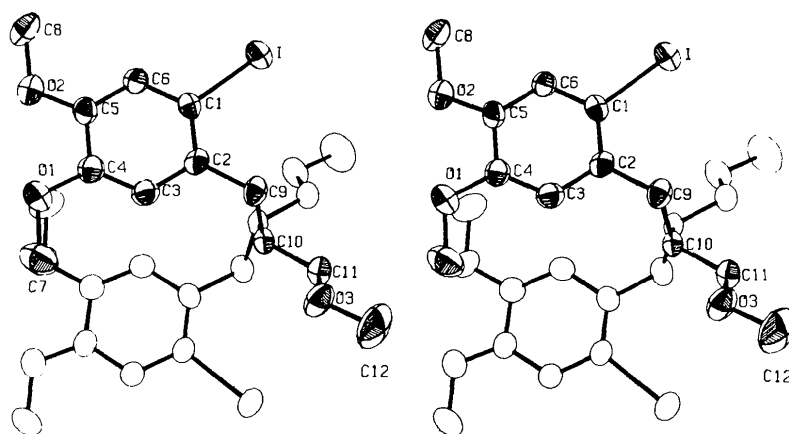

Fig. 1. A stereoview of the iodophyllanthin molecule along **a**, with 50% vibration ellipsoids. Atom numbering is for crystallographic convenience.

Table 2. *Bond lengths* (Å) *and angles* (°) ($\sigma = 0.01$ Å *for* C—I *and* ~ 0.02 Å *for other bonds*)

I—C(1)	2·10	C(2)—C(9)	1·53
C(1)—C(2)	1·38	C(9)—C(10)	1·56
C(2)—C(3)	1·41	C(10)—C(10')	1·53
C(3)—C(4)	1·37	C(10)—C(11)	1·53
C(4)—C(5)	1·43	C(7)—O(1)	1·46
C(5)—C(6)	1·40	C(8)—O(2)	1·43
C(6)—C(1)	1·38	C(11)—O(3)	1·40
C(4)—O(1)	1·37	C(12)—O(3)	1·41
C(5)—O(2)	1·34		
C(1)—C(2)—C(3)	117	C(2)—C(1)—I	122
C(2)—C(3)—C(4)	123	C(6)—C(1)—I	116
C(3)—C(4)—C(5)	120	C(1)—C(2)—C(9)	124
C(4)—C(5)—C(6)	117	C(3)—C(2)—C(9)	119
C(5)—C(6)—C(1)	122	C(3)—C(4)—O(1)	128
C(6)—C(1)—C(2)	122	C(5)—C(4)—O(1)	113
C(2)—C(9)—C(10)	115	C(4)—C(5)—O(2)	117
C(9)—C(10)—C(11)	110	C(6)—C(5)—O(2)	127
C(9)—C(10)—C(10')	113	C(4)—O(1)—C(7)	116
C(10')—C(10)—C(11)	113	C(5)—O(2)—C(8)	116
C(10)—C(11)—O(3)	110	C(11)—O(3)—C(12)	113

dihedral angles C(9)—C(10)—C(10')—C(9') and C(11)—C(10)—C(10')—C(11') of −156 and −46° respectively (−180 and −60° for ideal staggered, and −120 and 0° for ideal eclipsed conformations).

Bond lengths (Table 2) are close to normal values; mean lengths are C—I 2·10, C(sp^2)—C(sp^2) 1·39,

C(sp^2)—C(sp^3) 1·53, C(sp^3)—C(sp^3) 1·54, C(sp^2)—O 1·35, C(sp^3)—O 1·43 Å. The angles (Table 2) show some deviations from normal values, particularly those involving the substituents of the aromatic rings. C(2)—C(1)—I, 122, and C(1)—C(2)—C(9), 124°, suggest repulsion between the I and C(9) substituents (in accord with the displacements from the aromatic plane), but C(5)—C(4)—O(1), 113, and C(4)—C(5)—O(2), 117°, deviate from 120° in the opposite sense.

Intermolecular distances correspond to normal van der Waals interactions; the shortest of each type are: I···I 3·84, I···O 3·89, C···C 3·62, C···O 3·43 Å, and between other atom types, >3·9 Å.

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DL-Leucylglycylglycine (Lgg)

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Abstract. $C_{10}N_3O_4H_{19}$, monoclinic, $P2_1/c$, $a = 11·52(2)$, $b = 12·44(2)$, $c = 9·70(1)$ Å, $\beta = 102·6(2)°$, $Z = 4$, $D_c = 1·26$, $D_m = 1·28$ g cm^{-3}, $|\lambda(Cu K\alpha) = 1·5418$ Å]. The molecules are packed head-to-tail in rows parallel to **b**. The peptide is in a *trans* configuration and an extended conformation with the terminal carboxyl group nearly parallel to the peptide plane. The side chain of the leucyl residue is in a different conformation from that normally found.

Introduction. Single crystals were obtained by slow evaporation from an ethanol–dioxane mixture. Crystals were frequently twinned; only one was untwinned and large enough for structure analysis. Cell dimensions were determined from rotation and Weissenberg photographs and the intensities collected by the equi-inclination Weissenberg technique for *hkl*, $l = 0$–4, with the multiple-film method. The intensities were estimated visually and corrected for Lorentz, polarization and absorption factors. 750 reflexions were possible of which only 570 had observable intensities. Photographs about **b** or **a** could not be taken

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