(1·769 Å) and in a 1,3,4-thiadiazolidine-5-thione (Karle & Karle, 1965) (1·777 and 1·842 Å) but similar to that found in the morpholinoethyl-1,2,4-triazoline-3-thione (Gors *et al.*, 1977) (1·67 Å) and other 1,2,4-triazoline-3-thiones (Seccombe & Kennard, 1973*a,b,c*) (1·668, 1·673, 1·675 Å).

There are two kinds of C—N bond lengths within the triazolidine ring. Two [C(1)—N(1) = 1·484 (2) and C(1)—N(3) = 1·472 (2) Å] are single-bond lengths. The other two [C(2)—N(1) = 1·352 (2) and C(2)—N(2) = 1·364 (2) Å] are intermediate between single-bond and double-bond length, indicating considerable conjugation with the C—S bond.

The exocyclic angles at N(2) show considerable asymmetry, C(2)—N(2)—C(3) [131·4 (1)°] being significantly larger than N(3)—N(2)—C(3) [117·5 (1)°]. As previously noted (Branch & Nowell, 1985, 1986), this asymmetry appears to be characteristic of the triazolyl ring itself rather than due to the influence of any intra- or intermolecular interactions.

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Acta Cryst. (1988). C**44**, 1996–1999

Structure of Mangostin Acetate

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(*Received* 8 *March* 1988; *accepted* 20 *June* 1988)

Abstract. 1,3,6-Triacetoxy-7-methoxy-2,8-bis(3-methyl-2-butenyl)-9*H*-xanthen-9-one, $C_{30}H_{32}O_9$, M_r = 536·6, triclinic, $P\bar{1}$, a = 11·400 (4), b = 11·464 (3), c = 13·403 (1) Å, α = 70·03 (1), β = 106·57 (2), γ = 118·53 (3)°, V = 1430·5 (7) Å^3, Z = 2, D_m = 1·241 (3) (flotation), D_x = 1·246 Mg m^{-3}, Cu $K\alpha$ radiation, λ = 1·5418 Å, μ = 0·725 mm^{-1}, $F(000)$ = 568, T = 294 K, final R = 0·063 for 3022 reflections [$I > 3\sigma(I)$]. The tricyclic xanthone system is not planar. The mean planes of the two isoprenyl side chains are inclined at an angle of 159·8 (2)°.

Introduction. Xanthones are a class of compounds which have been shown to possess anti-inflammatory and CNS depressant activities (Gopalakrishnan,

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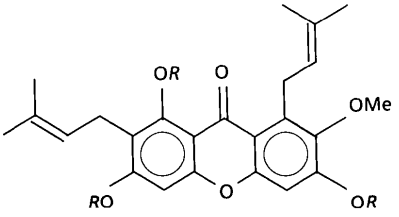
0108-2701/88/111996-04$03.00

Shankaranarayanan, Nazimudeen, Viswanathan & Kameswaran, 1980). Mangostin (I) is one of the xanthones isolated from the mangosteen fruit hulls (*Garcinia mangostana*, Guttieferae) and is reported to inhibit prostaglandin synthetase, similar to non-steroidal anti-inflammatory compounds (Shankaranarayanan, Gopalakrishnan & Kameswaran, 1979). The most significant feature is that the anti-inflammatory agents in clinical use aggravate gastric ulcers whereas mangostin and other xanthones possess anti-ulcer activity. In view of the interesting pharmacological activities displayed by mangostin, we decided to determine the single-crystal X-ray structure of its acetate derivative (II).



(I) R = H; (II) R = Ac

Experimental. The title compound was prepared by the acetylation of (I). A solution of 150 mg of (I) in 0·5 ml of acetic anhydride and 3 drops of pyridine was stirred at room temperature for 3 h. The precipitated solid was collected by vacuum filtration and recrystallized from benzene as pale yellow crystals. Crystal dimensions 0·13 × 0·08 × 0·12 mm. CAD-4 diffractometer, monochromated Cu $K\alpha$ radiation. Cell parameters from least-squares refinement of setting angles of 25 reflections (θ range from 25 to 35°). Intensity data for $0 < \theta < 55°$, $\omega/2\theta$ scans, two check reflections for every 98 data points did not vary significantly over the course of the data collection. Lp but no absorption correction, 4209 reflections (h 0→11, k −11→11, l −14→14) of which 3022 [$I > 3\sigma(I)$] used in calculations. Direct methods with *MULTAN*80 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980); an E map calculated from the set of phases with the highest figure of merit revealed the structure. Full-matrix least-squares refinement on F. Anisotropic temperature factors for C and O atoms, isotropic for H. H positions from a difference map. $w = 1·248/[\sigma^2(F_o) + 0·008F_o^2]$, final $R = 0·063$, $wR = 0·067$ for 3022 reflections, $R = 0·078$ for all reflections. $S = 1·47$; final ΔF map featureless, $(\Delta/\sigma)_{max} = 0·52$, final $\Delta\rho$ excursions −0·21–0·29 e Å^{-3}. No corrections for secondary extinction, scattering factors as in *SHELX* (Sheldrick, 1976). Calculations of geometrical data and crystal packing were computed using the program *PARST* (Nardelli, 1983). Calculations performed on an IBM 370 computer.

Table 1. *Fractional positional parameters and equivalent isotropic thermal parameters*

$B_{eq} = \frac{8}{3}\pi^2 \sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i . \mathbf{a}_j$.

	x	y	z	B_{eq}(Å^2)
C1	0·4001 (3)	0·0228 (3)	0·2264 (2)	4·1 (1)
C2	0·4195 (3)	0·1228 (3)	0·2741 (3)	4·1 (1)
C3	0·3444 (3)	0·0814 (3)	0·3591 (2)	3·7 (1)
C4	0·3485 (3)	0·1802 (3)	0·4100 (3)	4·3 (1)
C5	0·2571 (3)	0·1212 (3)	0·4895 (2)	3·6 (1)
C6	0·2381 (3)	0·1974 (3)	0·5413 (3)	4·1 (1)
C7	0·1574 (3)	0·1410 (4)	0·6207 (3)	4·3 (1)
C8	0·0895 (3)	−0·0025 (3)	0·6475 (2)	4·2 (1)
C9	0·1000 (3)	−0·0840 (3)	0·5997 (2)	4·5 (1)
C10	0·1833 (3)	−0·0194 (3)	0·5203 (2)	3·7 (1)
C11	0·2585 (3)	−0·0560 (3)	0·3905 (2)	4·1 (1)
C12	0·2381 (3)	−0·1537 (3)	0·3415 (3)	4·2 (1)
C13	0·3083 (3)	−0·1111 (3)	0·2587 (3)	4·2 (1)
C14	0·1786 (4)	−0·2456 (4)	0·1327 (3)	5·1 (2)
C15	0·1749 (5)	−0·3518 (5)	0·0899 (3)	6·8 (2)
C16	0·5832 (5)	0·0326 (6)	0·1698 (4)	7·7 (2)
C17	0·5159 (3)	0·2695 (4)	0·2317 (3)	4·9 (1)
C18	0·6501 (3)	0·3139 (4)	0·3025 (3)	4·8 (1)
C19	0·7694 (4)	0·3962 (3)	0·2676 (3)	5·4 (2)
C20	0·8962 (4)	0·4406 (5)	0·3473 (5)	8·1 (2)
C21	0·7859 (5)	0·4527 (5)	0·1509 (5)	8·0 (2)
C22	0·2717 (4)	0·4117 (3)	0·4181 (3)	4·8 (1)
C23	0·3654 (5)	0·5599 (4)	0·4044 (4)	7·6 (2)
C24	0·1454 (4)	0·2306 (4)	0·6751 (3)	5·4 (2)
C25	0·2763 (5)	0·3012 (4)	0·7435 (3)	6·4 (2)
C26	0·3066 (5)	0·2596 (4)	0·8486 (3)	6·2 (2)
C27	0·2166 (7)	0·1373 (7)	0·9185 (4)	9·0 (2)
C28	0·4455 (7)	0·3426 (6)	0·9058 (4)	9·5 (3)
C29	−0·0031 (4)	−0·1691 (4)	0·8045 (4)	5·4 (2)
C30	−0·1184 (5)	−0·2214 (6)	0·8618 (3)	7·3 (2)
O1	0·1846 (2)	−0·1077 (2)	0·4734 (2)	4·5 (1)
O2	0·2913 (2)	−0·2069 (2)	0·2075 (2)	5·1 (1)
O3	0·0977 (4)	−0·2022 (4)	0·1126 (3)	9·6 (3)
O4	0·4652 (3)	0·0580 (3)	0·1416 (2)	5·6 (1)
O5	0·4217 (3)	0·3022 (3)	0·3860 (3)	6·5 (1)
O6	0·3076 (2)	0·3402 (2)	0·5165 (2)	4·7 (1)
O7	0·1790 (3)	0·3603 (3)	0·3554 (2)	6·0 (1)
O8	−0·0035 (2)	−0·0596 (3)	0·7194 (2)	5·1 (1)
O9	0·0805 (3)	−0·2113 (3)	0·8259 (2)	6·9 (1)

Discussion. Final positional parameters of the non-H atoms are given in Table 1.* Bond distances and angles are given in Table 2. A perspective view of the molecule is given in Fig. 1. The geometric parameters of the xanthone nucleus are quite normal and agree with the values of bond distances and angles for other xanthones (Soderholm, Sonnerstam, Norrestam & Palm, 1976; Dobler & Schierlein, 1977; Fukuyama, Hamada, Tsukihara & Katsube, 1978).

The lengths of the C—O bonds in the three acetoxy groups [1·186 (4), 1·188 (7) and 1·173 (8) Å] are shorter than the usual C=O bond distance of 1·215 (5) Å proposed by Sutton (1965). However, as Low & Wilson (1984) noted, this bond is frequently shorter than expected for a conjugated C=O bond. This is further confirmed by Skrzat & Roszak (1986) based on the data retrieved for 102 acetoxy groups substituted on aromatic six-membered carbon rings. The C=O bond distance [O3—C14, 1·173 (8) Å] in

* 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 51159 (18 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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

O1–C10	1·369 (5)	C5–C6	1·405 (6)
O1–C11	1·379 (4)	C5–C10	1·387 (4)
O2–C13	1·396 (5)	C6–C7	1·385 (6)
O2–C14	1·370 (4)	C7–C8	1·405 (5)
O3–C14	1·173 (8)	C7–C24	1·519 (8)
O4–C1	1·374 (5)	C8–C9	1·363 (6)
O4–C16	1·430 (8)	C9–C10	1·396 (4)
O5–C4	1·219 (4)	C11–C12	1·381 (6)
O6–C6	1·399 (3)	C12–C13	1·369 (6)
O6–C22	1·361 (4)	C14–C15	1·492 (8)
O7–C22	1·186 (4)	C17–C18	1·503 (4)
O8–C8	1·404 (4)	C18–C19	1·339 (5)
O8–C29	1·380 (5)	C19–C20	1·509 (6)
O9–C29	1·188 (7)	C19–C21	1·504 (7)
C1–C2	1·396 (6)	C22–C23	1·492 (5)
C1–C13	1·382 (4)	C24–C25	1·515 (5)
C2–C3	1·433 (5)	C25–C26	1·324 (5)
C2–C17	1·516 (4)	C26–C27	1·474 (7)
C3–C4	1·486 (6)	C26–C28	1·538 (7)
C3–C11	1·388 (4)	C29–C30	1·478 (8)
C4–C5	1·465 (5)		

C10–O1–C11	119·4 (2)	C5–C10–C9	123·9 (3)
C13–O2–C14	117·5 (3)	O1–C10–C9	113·8 (3)
C1–O4–C16	114·5 (3)	O1–C10–C5	122·3 (3)
C6–O6–C22	118·0 (3)	O1–C11–C3	123·0 (3)
C8–O8–C29	119·6 (3)	C3–C11–C12	123·4 (3)
O4–C1–C13	118·9 (3)	O1–C11–C12	113·6 (3)
O4–C1–C2	120·4 (3)	C11–C12–C13	117·4 (3)
C2–C1–C13	120·6 (3)	C1–C13–C12	122·4 (3)
C1–C2–C17	118·5 (3)	O2–C13–C12	119·2 (3)
C1–C2–C3	118·0 (3)	O2–C13–C1	118·4 (3)
C3–C2–C17	123·5 (3)	O2–C14–O3	122·0 (4)
C2–C3–C11	118·2 (3)	O3–C14–C15	128·0 (4)
C2–C3–C4	122·8 (3)	O2–C14–C15	109·9 (4)
C4–C3–C11	118·9 (3)	C2–C17–C18	113·2 (3)
O5–C4–C3	122·1 (4)	C17–C18–C19	125·1 (3)
C3–C4–C5	115·4 (3)	C18–C19–C21	123·7 (4)
O5–C4–C5	122·4 (3)	C18–C19–C20	119·8 (4)
C4–C5–C10	120·4 (3)	C20–C19–C21	116·4 (4)
C4–C5–C6	124·6 (3)	O6–C22–O7	123·5 (3)
C6–C5–C10	114·9 (3)	O7–C22–C23	127·1 (4)
O6–C6–C5	119·8 (3)	O6–C22–C23	109·4 (3)
C5–C6–C7	124·5 (3)	C7–C24–C25	112·1 (4)
O6–C6–C7	115·6 (3)	C24–C25–C26	126·0 (4)
C6–C7–C24	121·3 (3)	C25–C26–C28	119·3 (4)
C6–C7–C8	115·8 (3)	C25–C26–C27	125·2 (4)
C8–C7–C24	122·9 (3)	C27–C26–C28	115·5 (4)
O8–C8–C7	116·1 (3)	O8–C29–O9	122·8 (4)
C7–C8–C9	123·4 (3)	O9–C29–C30	127·1 (4)
O8–C8–C9	120·1 (3)	O8–C29–C30	110·0 (4)
C8–C9–C10	117·3 (3)		

one of the acetoxy groups is shorter than the mean value of 1·188 (2) Å deduced by Skrzat & Roszak (1986). This may be due to the high thermal vibration of O3.

The tricyclic xanthone system in the present structure is not planar; also the dihedral angle between the planes of the outer rings, which are not significantly planar [$\sum(\Delta/\sigma)^2 = 65·9$ and 30·3], is 8·8 (1)°. The dihedral angle between the mean planes of the two isoprenyl side chains is 159·8 (2)°. Further, the mean planes of the two isoprenyl side chains form dihedral angles of 67·7 (2) and 116·3 (2)° with the mean plane of the xanthone moiety. The corresponding angle in other xanthones is 111·5 (1)° in garcinone B (Ravikumar & Rajan, 1987); 66·8 (3)° in 5-hydroxy-8,-9-dimethoxy-2,2-dimethyl-7-(3-methyl-2-butenyl)-2*H*,-6*H*-pyrano[3,2-*b*]xanthen-6-one (Ravikumar, Rajan & Padmanabhan, 1987); 95·4° in epishamixanthone (Fukuyama, Hamada, Tsukihara & Katsube, 1978) and 102° in morellin (Kartha & Ambady, 1982).

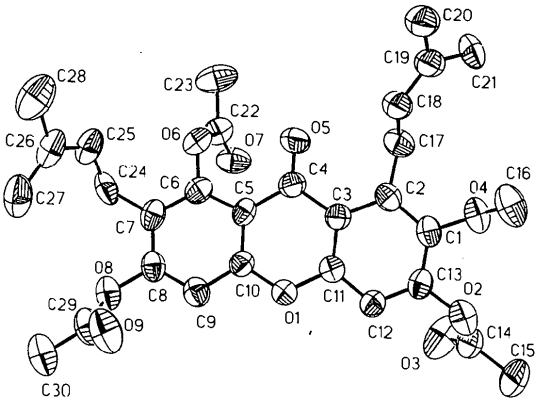


Fig. 1. *ORTEP* drawing (Johnson, 1976) of the title compound. Thermal ellipsoids are at 50% probability level.

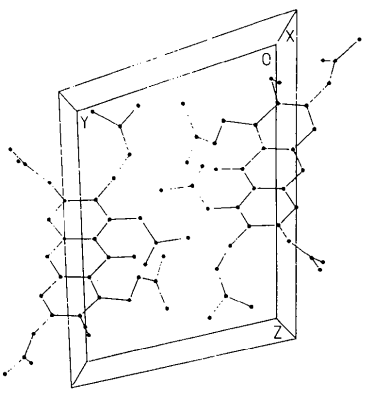


Fig. 2. Packing diagram of the contents of the unit cell looking down the *a* axis.

The three acetoxy groups are almost planar [$\sum(\Delta/\sigma)^2 = 0·20,21,6$ and 0·38] and they form dihedral angles of 111·5 (1), 79·4 (1) and 57·5 (1)°, respectively, with the mean plane of the xanthone ring system.

The packing of the molecules, illustrated in Fig. 2, is governed essentially by van der Waals forces.

This work was supported by the Council of Scientific Industrial Research, India.

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Acta Cryst. (1988). C**44**, 1999–2002

(2*S*)-2-Hydroxy-4-methylvaleryl-L-valyl-L-phenylalanyl Methyl Ester

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(*Received* 14 *March* 1988; *accepted* 6 *June* 1988)

Abstract. $C_{21}H_{32}N_2O_5$, $M_r = 392{\cdot}50$, orthorhombic, $P2_12_12_1$, $a = 10{\cdot}273\ (2)$, $b = 18{\cdot}862\ (3)$, $c = 24{\cdot}226\ (3)$ Å, $V = 4694{\cdot}2$ Å^3, $Z = 8$, $D_x = 1{\cdot}111$ Mg m^{-3}, $\lambda(\text{Cu } K\alpha) = 1{\cdot}5418$ Å, $\mu = 0{\cdot}652$ mm^{-1}, $F(000) = 1696$, room temperature, $R = 0{\cdot}051$ for 2053 observed reflections. In the region of the central valyl residue, the two peptide molecules in the asymmetric unit have very similar β-pleated sheet-type conformations. The oxygen O1 atoms which replace the classical N terminal participate in four different hydrogen-type interactions between the two independent molecules. The peptide units are *trans* and at least one of them shows significant deviation from planarity (9°).

Introduction. Renin is a proteolitic enzyme which cleaves the Leu-Leu peptide bond in equine (Skeggs, Kanh, Lentz & Shumway, 1957) or the Leu-Val bond in human substrates (Tewksbury, Dart & Travis, 1981). Inhibition of that reaction may have important therapeutic implications in the regulation of blood pressure. Among the number of ways explored in the search for potent inhibitors, one concerns the modification of the normal peptide bond with the object of producing a non-hydrolysable analogue. The natural peptide bond can be replaced by the reduced carbonyl analogue (Szelke, Leckie, Hallett, Jones, Suieras, Atrash & Lever, 1982), the hydroxy methylene analogue (Tree, Brown, Leckie, Lever, Manhem, Morton, Robertson, Szelke & Webb, 1984), the amino alcohol analogue (Dann, Stammers, Harris, Arrowsmith, Davies, Hardy & Morton, 1986), or the oxyacetyl analogue. The title compound is the tripeptide analogue obtained after transesterification of Leu-ψ(CO—O)-Leu-Val-Phe-OMe.

We describe in this report the crystal structure of the title compound (*O*-Leu-Val-Phe-OMe) and compare the conformations and the modes of interaction with those observed for Leu-Leu-Val-Tyr-OMe (Precigoux, Courseille, Geoffre & Leroy, 1987) and phenyloxyacetyl-Leu-Val-Phe-OMe (Geoffre, Leroy & Precigoux, 1986).

Experimental. The title compound *O*-Leu-Val-Phe-OMe was crystallized by slow diffusion of diisopropyl ether into a methanolic solution. Space group and preliminary unit-cell parameters determined from X-ray diffraction photographs. Crystal of dimensions $0{\cdot}1 \times 0{\cdot}2 \times 0{\cdot}4$ mm. Computer-controlled CAD-4 diffractometer, ω–2θ scan to a maximum Bragg angle of 50°, graphite-monochromated Cu $K\alpha$ radiation. 25 reflections in the range $10 \le \theta \le 32°$ used for cell-parameters refinement. Intensities not corrected for absorption. h: 0 to 10; k: 0 to 18; l: 0 to 24. Maximum variation in intensity of standard reflections 3%. 2738 reflections measured, 2053 with $I \ge 2\sigma(I)$ used in refinement. Structure solved by the direct-methods program *MITHRIL* (Gilmore, 1984). The non-H atoms were refined anisotropically and the H atoms, for the tertiary CH and the secondary CH_2 groups, were located geometrically and refined isotropically on F using the block-diagonal least-squares method. Refinement converged at $R = 0{\cdot}051$, $wR = 0{\cdot}053$, $S = 1{\cdot}0786$ (max. $\Delta/\sigma = 0{\cdot}07$). The weighting scheme was $w^{1/2} = 1$ if $|F_o| < p$ and $w^{1/2} = p/F_o$ if $|F_o| \ge p$ with $p = |F_o^2\ (\text{max.})/10|^{1/2}$. Maximum and minimum values in the difference Fourier map were 0·031 and

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