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Tlatlancuayin

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Key indicators

Single-crystal X-ray study
T = 120 K
Mean σ(C–C) = 0.002 Å
R factor = 0.036
wR factor = 0.095
Data-to-parameter ratio = 11.5

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

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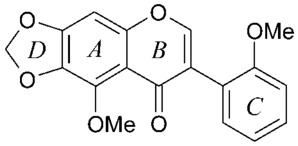
The biologically active title compound, 2′,5-dimethoxy-6,7-(methylenedioxy)isoflavone, $C_{18}H_{14}O_6$, was isolated from the aerial parts of *Iresine herbstii* Hook (Amaranthaceae). The 1,3-dioxolane ring, fused to the benzopyran ring system, adopts an envelope conformation. The dihedral angle between the benzopyran ring system and the 3-phenyl ring is 62.75 (6)°.

Comment

The title compound, (I), was originally isolated from *Iresine celosioides* (Amaranthaceae) (Djerassi *et al.*, 1954; Crabbé *et al.*, 1958) and later from the aerial parts of *Iresine herbstii* Hook (Amaranthaceae), a perennial herb native to tropical South America. *Iresine herbstii* is known as a 'tlatlancuaya' in central Mexico (Crabbé *et al.*, 1958). Extracts of this plant have been used by the natives of the northern Peruvian Andes and Mexico for the treatment of various diseases, including malaria and typhoid (Martinez, 1944; De Feo, 2003). From plants of the genus *Iresine* the following have also been isolated: the sesquiterpene lactone iresin, the title isoflavone (I) (Djerassi *et al.*, 1954; Djerassi & Burstein, 1958; Crabbé *et al.*, 1958), campesterol, β-sitosterol, 3′,4′,7-trihydroxy-6-methoxyflavone (Kubínová *et al.*, 1998) and betalains (Cai *et al.*, 2001). A purified aqueous extract from *Iresine herbstii* significantly reduced the locomotory activity, motor coordination and stereotyped behavior of mice (De Feo *et al.*, 1996), while an ethanolic extract inhibited lipoperoxidation (Kubínová *et al.*, 1998). We report here the crystal structure of (I).



(I)

The structure of (I) (Fig. 1 and Table 1) is similar to that of 5-methoxy-6,7-methylenedioxyflavone (Koh & Ng, 1993). The five-membered ring (D, see scheme) adopts an envelope conformation, with Cremer & Pople (1975) puckering parameters q_2 = 0.195 (4) Å and φ_2 = −179.7 (4)°, and a local pseudo-mirror running through atom C10 and the mid-point of the C5—C6 bond. The flap of the envelope is C10, which is displaced from the mean plane of the other four atoms by 0.255 (1) Å. The benzopyran fragment A/B and ring C are each essentially planar (the average deviations of contributing atoms from the least-squares planes are 0.03 and 0.01 Å, respectively). The dihedral angle between A/B and C is 62.75 (6)°. The methoxy group on ring C lies close to the ring plane [C11—C12—O6—C17 = −172.77 (12)°], while the

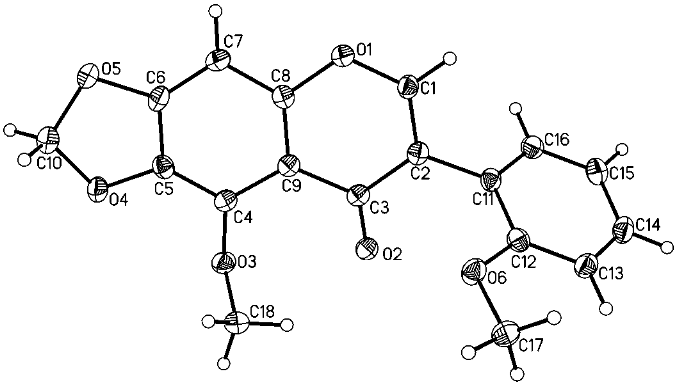


Figure 1
A view of the title compound. Displacement parameters for non-H atoms are drawn at the 50% probability level. H atoms are shown as small spheres of arbitrary radii.

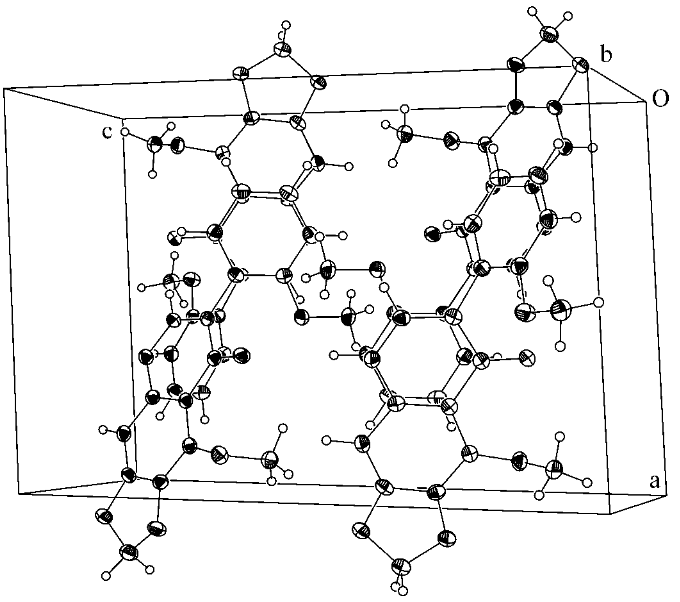


Figure 2
Part of the crystal structure of (I), showing stacking interactions between the *B* and *C* rings of adjacent tlatlancuayin molecules.

methoxy group on ring *A* is almost perpendicular to the ring plane [C9—C4—O3—C18 = 76.51 (17)°].

In the crystal structure of (I), the molecular packing is such that stacking interactions between rings *B* and *C* of adjacent tlatlancuayin molecules are optimized (Fig. 2). The dihedral angle between rings *B* and C^{i} [symmetry code: (i) $1 - x, y - \frac{1}{2}, \frac{1}{2} - z$] is 9.67 (4)°. If *Cg*1 is the centroid of ring *B* and *Cg*2 that of ring C^{i}, the *Cg*1···*Cg*2 distance is 3.650 (2) Å, the distance of *Cg*1 from the plane through the C^{i} ring atoms is 3.504 (1) Å, and the distance of *Cg*2 from the plane through the atoms of ring *B* is 3.409 (2) Å.

Experimental

Tlatlancuayin, together with other substances, was obtained from the dried and powdered aerial part of *Iresine herbstii* cultivated in the faculty greenhouse. After extraction with 95% ethanol and evaporation of the solvent *in vacuo*, the extract was repeatedly subjected to silica-gel chromatography, eluting with benzene and ethyl acetate (8:2, 97:3 *v*/*v*), yielding pure tlatlancuayin. The purity of tlatlancuayin obtained from the gel chromatography was proven using high-performance liquid chromatography (HP1100, DAD detector). The title compound was identified by comparing the spectroscopic data with the previously published data (Crabbé *et al.*, 1958). The light-brown well developed crystals were isolated from the eluted solution by evaporation of the solvent at room temperature. Analysis (Carlo–Erba 1180 instrument) calculated for $C_{18}H_{14}O_6$: C 66.26, H 4.32%; found: C 65.15, H 4.37%.

Crystal data

$C_{18}H_{14}O_6$
$M_r = 326.29$
Monoclinic, $P2_1/c$
$a = 11.262$ (3) Å
$b = 7.6872$ (19) Å
$c = 16.540$ (4) Å
$\beta = 93.26$ (2)°
$V = 1429.6$ (6) Å^3
$Z = 4$

$D_x = 1.516$ Mg m^{-3}
Mo *Kα* radiation
Cell parameters from 2626 reflections
$\theta = 2.6$–27.5°
$\mu = 0.12$ mm^{-1}
$T = 120$ (2) K
Prism, light brown
0.50 × 0.30 × 0.30 mm

Data collection

Kuma KM-4 CCD diffractometer
ω scans
Absorption correction: none
7787 measured reflections
2513 independent reflections
2191 reflections with $I > 2\sigma(I)$

$R_{\rm int} = 0.044$
$\theta_{\rm max} = 25.0°$
$h = -13 \rightarrow 13$
$k = -9 \rightarrow 6$
$l = -19 \rightarrow 19$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.036$
$wR(F^2) = 0.095$
$S = 1.00$
2513 reflections
219 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.05P)^2 + 0.6P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} < 0.001$
$\Delta\rho_{\rm max} = 0.21$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.23$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

O1—C1	1.3547 (18)	O5—C6	1.3759 (17)
O1—C8	1.3714 (18)	O6—C12	1.3715 (18)
O2—C3	1.2364 (17)	C1—C2	1.342 (2)
O3—C4	1.3712 (18)	C2—C11	1.494 (2)
O4—C5	1.3772 (18)	C5—C6	1.389 (2)
C4—O3—C18	114.63 (11)	C4—C9—C3	123.46 (13)
C12—O6—C17	116.46 (11)	O4—C10—O5	106.94 (12)
C1—C2—C3	120.23 (13)		
C9—C4—O3—C18	76.51 (17)	C11—C12—O6—C17	−172.77 (12)

Table 2
Hydrogen-bonding geometry (Å, °).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C18—H18*A*···O2	0.98	2.32	2.988 (2)	124
C1—H1···O2^{i}	0.95	2.41	3.3393 (19)	165
C7—H7···O5^{ii}	0.95	2.55	3.4276 (19)	153
C13—H13···O2^{iii}	0.95	2.59	3.496 (2)	158
C15—H15···O4^{iv}	0.95	2.60	3.377 (2)	139

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

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H atoms were positioned geometrically, with C—H = 0.95–0.99 Å, and with $U_{\rm iso}$(H) values equal to $1.2U_{\rm eq}$(C) [$1.5U_{\rm eq}$(C) for methyl H atoms].

Data collection: *CrysAlisCCD* (Oxford Diffraction, 2004); cell refinement: *CrysAlisRED* (Oxford Diffraction, 2004); data reduction: *CrysAlisRED*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*III (Burnett & Johnson, 1996); software used to prepare material for publication: *SHELXL*97 and *PARST* (Nardelli, 1995).

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