Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters (Å²)*

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

	x	y	z	$B_{eq/iso}$
N1	−0.2128 (7)	0.7957 (2)	0.6965 (2)	4.11 (8)
C1*A*	−0.3028 (8)	0.8593 (2)	0.7484 (2)	3.70 (8)
C1*B*	−0.2556 (9)	0.8249 (2)	0.8234 (2)	4.7 (1)
C1*G*	−0.3154 (13)	0.8826 (4)	0.8856 (3)	6.7 (2)
C1*D*1	−0.5833 (19)	0.9098 (8)	0.8868 (4)	11.9 (3)
C1*D*2	−0.237 (2)	0.8377 (5)	0.9536 (3)	9.9 (3)
C1′	−0.1541 (7)	0.9386 (2)	0.7356 (2)	3.61 (8)
O1′	0.0765 (5)	0.9372 (2)	0.7248 (2)	4.51 (7)
N2	−0.2915 (6)	1.0080 (2)	0.7374 (2)	4.05 (8)
C2*A*	−0.1791 (8)	1.0911 (2)	0.7326 (2)	4.2 (1)
C2*B*	−0.1516 (18)	1.1148 (3)	0.6537 (3)	7.9 (2)
C2′	−0.3464 (9)	1.1533 (2)	0.7713 (2)	4.6 (1)
O21′	−0.2578 (7)	1.2251 (1)	0.7785 (2)	6.1 (1)
O22′	−0.5644 (7)	1.1329 (2)	0.7906 (3)	7.1 (1)
Solvent molecule				
S1†	0.0659 (6)	0.1503 (2)	0.0329 (1)	7.88 (7)
S2†	−0.0570 (9)	0.0861 (2)	0.0137 (2)	6.9 (8)
O3	−0.2197 (12)	0.1414 (4)	0.0590 (2)	9.9 (2)
C1	0.020 (2)	0.1416 (5)	−0.0631 (4)	11.7 (4)
C2	0.207 (2)	0.0541 (7)	0.0562 (5)	11.6 (3)

† S1 and S2 were refined with site-occupancy factors of 0.62 and 0.38, respectively.

Table 2. *Selected torsion angles (°) involving non-H atoms*

N1—C1*A*—C1′—N2	(ψ_1)	137.3 (3)
C1*A*—C1′—N2—C2*A*	(ω_1)	174.4 (3)
N1—C1*A*—C1*B*—C1*G*	($^1\chi_1$)	175.6 (4)
C1*A*—C1*B*—C1*G*—C1D1	($^1\chi_{21}$)	59.1 (7)
C1*A*—C1*B*—C1*G*—C1D2	($^1\chi_{22}$)	−176.1 (5)
C1′—N2—C2*A*—C2′	(φ_2)	−151.8 (4)
N2—C2*A*—C2′—O21′	(ψ_{21})	171.9 (3)
N2—C2*A*—C2′—O22′	(ψ_{22})	−11.8 (5)

Table 3. *Hydrogen-bonding geometry (Å, °)*

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O21′^{i}	1.02 (7)	1.74 (7)	2.737 (5)	163 (5)
N1—H2···O3^{ii}	0.79 (7)	2.02 (7)	2.788 (6)	164 (7)
N1—H3···O22′^{iii}	0.69 (7)	2.21 (7)	2.875 (5)	161 (7)
N2—H6···O22′	0.79 (7)	2.33 (6)	2.654 (5)	106 (6)

Symmetry codes: (i) $-x, y-\frac{1}{2}, \frac{3}{2}-z$; (ii) $-\frac{1}{2}-x, 1-y, \frac{1}{2}+z$; (iii) $-1-x, y-\frac{1}{2}, \frac{3}{2}-z$.

H atoms of terminal groups in side chains showed abnormal B_j's and hence were only included in the structure-factor calculations. The other H atoms in the peptide molecule were refined isotropically.

Data collection: *SDP* (Enraf–Nonius, 1979). Cell refinement: *SDP*. Data reduction: *SDP*. Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1985). Program(s) used to refine structure: *SHELX*76 (Sheldrick, 1978). Molecular graphics: *PLUTO* (Motherwell & Clegg, 1976); *ORTEP* (Vickovic, 1994). Software used to prepare material for publication: *PARST* (Nardelli, 1983).

SNM thanks UGC for the award of a junior fellowship.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: VJ1038). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Dalspinosin

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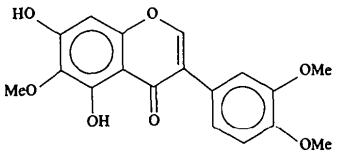
Abstract

The title compound, 3-(3,4-dimethoxyphenyl)-5,7-dihydroxy-6-methoxy-4*H*-1-benzopyran-4-one, $C_{18}H_{16}O_7$, consists of two phenyl rings (*A* and *B*) and a heterocyclic ring *C*. Rings *A* and *B* are planar and ring *C* is slightly puckered. The packing of the molecules in the unit cell is governed by van der Waals interactions and hydrogen bonds.

Comment

Dalspinosin (I) is an isoflavone derivative having a unique 3′,4′ arrangement of the methoxy groups in ring *B*. Isoflavanoids have oestrogenic, insecticidal, pesticidal and antifungal properties (Harborne, Mabry & Mabry, 1975). Fig. 1 is a perspective view of the molecular geometry showing numbering scheme adopted.



(I)

The two phenyl rings (*A* and *B*) are planar. The dihedral angle between rings *A* and *B* is 31.55 (6)°. Ring *C* is slightly puckered with q_2 = 0.070 (2) Å, θ_2 = 94 (1)° and φ_2 = −109 (2)° (Cremer & Pople, 1975) and ring *A* is twisted from the mean plane of ring *C* by 4.62 (5)°. The methoxy groups at C6, C3′ and C4′ are oriented slightly out of the plane of the rings to which they are attached (Shoja, 1992).

The mean bond lengths averaged over each type of bond agree well with the values observed in similar compounds (Kaneda, Iitaka & Shibata 1973; Breton, Precigoux, Courseille & Hospital, 1975). The structure is stabilized by van der Waals interactions and O—H···O and C—H···O hydrogen bonds (Desiraju, 1991).

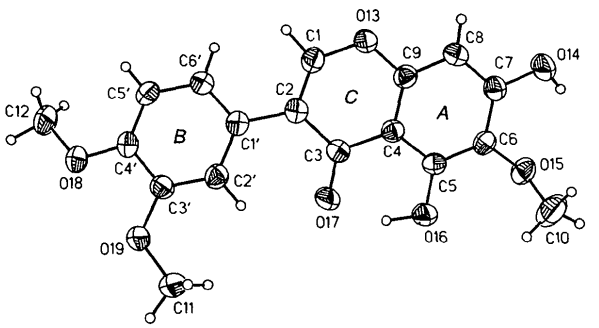


Fig. 1. Molecular structure showing 50% probability displacement ellipsoids.

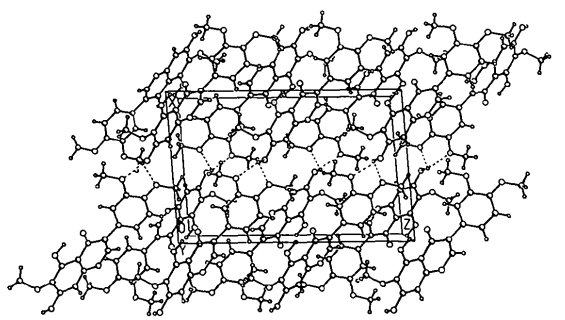


Fig. 2. Packing in the crystal viewed down the *b* axis.

Experimental

The compound was isolated from extracts of the roots of the plant *Dalbergia Spinosa*. Single crystals were obtained by slow evaporation of a methanol solution.

Crystal data

$C_{18}H_{16}O_7$
M_r = 344.31
Monoclinic
$P2_1/c$
a = 10.752 (1) Å
b = 8.711 (2) Å
c = 16.646 (1) Å
β = 94.4 (1)°
V = 1554.5 (4) Å^3
Z = 4
D_x = 1.471 Mg m^{-3}
D_m = 1.472 Mg m^{-3}

Mo $K\alpha$ radiation
λ = 0.71073 Å
Cell parameters from 23 reflections
θ = 20–34°
μ = 0.114 mm^{-1}
T = 293 K
Rectangular
0.1 × 0.1 × 0.05 mm
Pale yellow

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: none
2888 measured reflections
2732 independent reflections
2116 observed reflections [$I > 2\sigma(I)$]

R_{int} = 0.0307
θ_{max} = 25°
h = 0 → 12
k = 0 → 10
l = −19 → 19
2 standard reflections
frequency: 60 min
intensity decay: <2%

Refinement

Refinement on F^2
$R(F)$ = 0.0399
$wR(F^2)$ = 0.0998
S = 1.048
2732 reflections
290 parameters
H atoms refined isotropically
$w = 1/[\sigma^2(F_o^2) + (0.0612P)^2 + 0.5485P]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max}$ = 0.075
$\Delta\rho_{max}$ = 0.198 e Å^{-3}
$\Delta\rho_{min}$ = −0.225 e Å^{-3}
Extinction correction: none
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C) *Tables* 4.2.6.8 and 6.1.1.4

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters (Å^2)*

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

	x	y	z	B_{eq}
C1	0.9235 (2)	0.0711 (2)	0.1484 (1)	3.17 (5)
C2	1.0056 (1)	−0.0454 (2)	0.1484 (1)	2.71 (5)
C3	0.9891 (1)	−0.1579 (2)	0.0831 (1)	2.63 (4)
C4	0.8777 (1)	−0.1415 (2)	0.0294 (1)	2.54 (4)
C5	0.8458 (1)	−0.2495 (2)	−0.0323 (1)	2.64 (5)
C6	0.7348 (1)	−0.2362 (2)	−0.0792 (1)	2.71 (5)
C7	0.6527 (1)	−0.1154 (2)	−0.0652 (1)	2.87 (5)
C8	0.6823 (2)	−0.0068 (2)	−0.0062 (1)	3.16 (5)
C9	0.7936 (1)	−0.0215 (2)	0.0396 (1)	2.80 (5)
C1′	1.1054 (1)	−0.0600 (2)	0.2149 (1)	2.65 (5)
C2′	1.2204 (1)	−0.1303 (2)	0.2036 (1)	2.77 (5)
C3′	1.3097 (1)	−0.1477 (2)	0.2671 (1)	2.82 (5)
C4′	1.2878 (1)	−0.0917 (2)	0.3439 (1)	2.78 (5)
C5′	1.1763 (1)	−0.0208 (2)	0.3549 (1)	2.99 (5)
C6′	1.0857 (1)	−0.0059 (2)	0.2912 (1)	2.96 (5)
C10	0.7568 (3)	−0.3376 (4)	−0.2088 (1)	5.49 (9)
C11	1.4473 (2)	−0.2868 (3)	0.1868 (1)	4.34 (7)
C12	1.3536 (2)	−0.0844 (3)	0.4832 (1)	4.04 (7)
O13	0.8206 (1)	0.0883 (1)	0.0974 (1)	3.45 (4)
O14	0.5432 (1)	−0.1013 (2)	−0.1096 (1)	3.83 (4)
O15	0.6951 (1)	−0.3435 (1)	−0.1363 (1)	3.49 (3)
O16	0.9225 (1)	−0.3685 (1)	−0.0444 (1)	3.64 (4)
O17	1.0638 (1)	−0.2649 (1)	0.0742 (1)	3.81 (4)
O18	1.3805 (1)	−0.1183 (1)	0.4031 (1)	3.49 (4)
O19	1.4220 (1)	−0.2199 (2)	0.2623 (1)	3.82 (4)

Table 2. *Selected geometric parameters (Å)*

C1—C2	1.345 (3)	C8—C9	1.374 (3)
C1—O13	1.350 (3)	C9—O13	1.372 (2)
C2—C3	1.464 (3)	C1′—C2′	1.405 (3)
C2—C1′	1.486 (3)	C1′—C6′	1.387 (3)
C3—C4	1.446 (3)	C2′—C3′	1.380 (3)
C3—O17	1.247 (3)	C3′—C4′	1.405 (3)
C4—C5	1.415 (3)	C3′—O19	1.370 (3)
C4—C9	1.401 (3)	C4′—C5′	1.373 (3)
C5—C6	1.381 (3)	C4′—O18	1.366 (2)
C5—O16	1.349 (3)	C5′—C6′	1.390 (3)
C6—C7	1.404 (3)	C10—O15	1.422 (4)
C6—O15	1.377 (2)	C11—O19	1.429 (3)
C7—C8	1.384 (3)	C12—O18	1.417 (3)
C7—O14	1.347 (2)		

Table 3. *Hydrogen-bonding geometry (Å, °)*

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C2′—H2′···O17	0.94 (2)	2.32 (2)	2.880 (2)	118 (2)
C10—H10A···O16	0.95 (4)	2.64 (4)	3.159 (3)	115 (3)
O14—H14···O15	0.86 (4)	2.28 (4)	2.725 (2)	113 (3)
O16—H16···O17	1.00 (3)	1.64 (3)	2.562 (2)	150 (3)
C8—H8···O14^{i}	0.88 (3)	2.68 (3)	3.346 (3)	133 (2)
C5′—H5′···O16^{ii}	0.94 (2)	2.73 (2)	3.655 (3)	170 (2)
C12—H12B···O15^{ii}	0.97 (2)	2.77 (3)	3.373 (3)	121 (2)
C11—H11B···O15^{iii}	1.03 (3)	2.75 (3)	3.636 (3)	145 (2)
O16—H16···O16^{iii}	1.00 (3)	2.64 (3)	3.135 (2)	110 (2)
C11—H11C···O14^{iv}	1.00 (3)	2.72 (3)	3.621 (4)	150 (2)
O14—H14···O18^{v}	0.86 (4)	2.46 (4)	3.022 (2)	123 (3)
O14—H14···O19^{v}	0.86 (4)	2.13 (3)	2.871 (2)	145 (2)

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

The structure was solved by direct methods using *SHELXS*86 (Sheldrick, 1985). The initial *R* factor for the model proposed was 0.19. After a few cycles of full-matrix least-squares refinement, the *R* factor reduced to 0.11. All H atoms were located from the difference Fourier map and were refined isotropically.

Data collection: *Enraf–Nonius CAD-4 Software* (Enraf–Nonius, 1989). Cell refinement: *SDP* (Frenz, 1978). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1985). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *ORTEP* (Johnson, 1965). Software used to prepare material for publication: *PARST* (Nardelli, 1983).

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: DE1027). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Acetoxymethyl 4-Chloro-*N*-furfuryl-5-sulfamoylanthranilate, an Absorption Furosemide Prodrug

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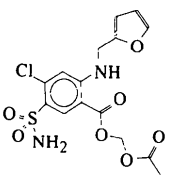
Abstract

The title compound, $C_{15}H_{15}ClN_2O_7S$, which was synthesized and characterized as the acetoxymethyl ester of 4-chloro-*N*-furfuryl-5-sulfamoylanthranilic acid (furosemide) is an absorption furosemide prodrug. The molecule crystallized in a triclinic unit cell, space group $P\bar{1}$. The crystal structure is stabilized by one intramolecular and two intermolecular hydrogen bonds.

Comment

Furosemide is a strong diuretic agent widely used in hypertensive crisis. The use of some acyloxymethyl esters of furosemide as prodrugs to improve the therapeutic success of this drug has been studied by Prandi, Fagiolino, Manta & Llera (1992).



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