Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

Tectorigenin monohydrate: an isoflavone from *Belamcanda chinensis*

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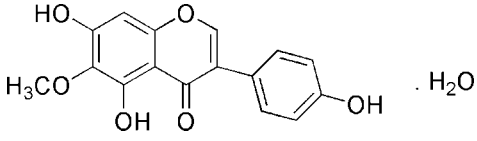
Received 20 September 2008; accepted 24 September 2008

Key indicators: single-crystal X-ray study; T = 113 K; mean σ(C–C) = 0.002 Å; R factor = 0.045; wR factor = 0.117; data-to-parameter ratio = 13.2.

The title compound [systematic name: 5,7-dihydroxy-3-(4-hydroxyphenyl)-6-methoxy-4*H*-chromen-4-one monohydrate], $C_{16}H_{12}O_6\cdot H_2O$, is isolated from *Belamcanda chinensis* and is said to have antimicrobiotic and anti-inflammatory effects. The chromen-4-one system and the benzene ring are inclined at a dihedral angle of 36.79 (6)°. Molecules are linked by inter- and intramolecular O—H···O hydrogen bonds.

Related literature

For general background, see: Oh *et al.* (2001). For a related structure, see: Gao *et al.* (2008).



Experimental

Crystal data

$C_{16}H_{12}O_6\cdot H_2O$ M_r = 318.27
Monoclinic, $P2_1/c$
a = 12.971 (3) Å
b = 14.652 (3) Å
c = 7.2930 (15) Å
β = 103.81 (3)°
V = 1346.0 (5) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.13 mm^{-1}
T = 113 (2) K
0.14 × 0.04 × 0.02 mm

Data collection

Rigaku Saturn CCD area-detector diffractometer
Absorption correction: multi-scan (*CrystalClear*; Rigaku/MSC, 2005)
T_{min} = 0.973, T_{max} = 0.998
9180 measured reflections
2967 independent reflections
2069 reflections with $I > 2\sigma(I)$
R_{int} = 0.085

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.044
$wR(F^2)$ = 0.117
S = 1.00
2967 reflections
224 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.33 e Å^{-3}
$\Delta\rho_{min}$ = −0.27 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O7—H7B···O2^{i}	0.77 (3)	2.57 (2)	2.971 (2)	114 (2)
O7—H7A···O6^{ii}	0.95 (3)	1.95 (3)	2.884 (2)	167 (2)
O6—H6···O1^{iii}	0.88 (2)	1.88 (2)	2.7368 (17)	167 (2)
O3—H3···O5	0.90 (2)	1.71 (2)	2.5658 (16)	159.6 (18)
O1—H1···O7^{iv}	0.87 (2)	1.83 (2)	2.6630 (17)	160.4 (19)

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

Data collection: *CrystalClear* (Rigaku/MSC, 2005); cell refinement: *CrystalClear*; data reduction: *CrystalClear*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT2795).

References

Gao, H., Li, G., Zhang, J. & Zeng, J. (2008). *Acta Cryst.* E**64**, o1538.
Oh, K. B., Kang, H. & Matsuoka, H. (2001). *Biosci. Biotechnol. Biochem.* **65**, 939–942.
Rigaku/MSC (2005). *CrystalClear*. Rigaku/MSC, The Woodlands, Texas, USA.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.

supplementary materials

Acta Cryst. (2008). E**64**, o2056 [**doi:10.1107/S1600536808030833**]

Tectorigenin monohydrate: an isoflavone from *Belamcanda chinensis*

B. Liu, Y. Ma, H. Gao and Q. Wu

Comment

The title compound [systematic name: 5,7-dihydroxy-3-(4-hydroxyphenyl)-6-methoxy-4*H*-chromen-4-one] was isolated from Belamcanda chinensis and is said to have antimicrobiotic and anti-inflammatory effects. We report here the crystal structure of its monohydrate. The two aromatic ring systems rings are inclined at a dihedral angle of 36.79 (6)°. The molecules are linked by intermolecular O—H···O hydrogen bonds (Table 1).

Experimental

The title compound was isolated from Belamcanda chinensis.

Refinement

H atoms bonded to C were positioned geometrically (C—H=0.95–0.98 Å), and refined as riding with U_{iso}(H)=1.2U_{eq}(C) or 1.5$_{eq}$(C_{methyl}). The coordinates of the H atoms bonded to O were refined with U_{iso}(H)=1.5U_{eq}(O).

Figures

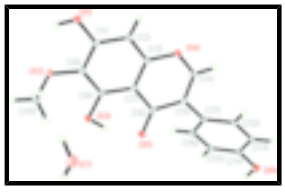


Fig. 1. A view of the title compound. Displacement ellopsoids are drawn at the 30% probability level and H atoms are shown as small spheres of arbitrary radii.

5,7-dihydroxy-3-(4-hydroxyphenyl)-6-methoxy-4H-chromen-4-one monohydrate

Crystal data

$C_{16}H_{12}O_6·H_2O$	F_{000} = 664
M_r = 318.27	D_x = 1.571 Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -P 2ybc	Cell parameters from 2562 reflections
a = 12.971 (3) Å	θ = 1.6–27.1°
b = 14.652 (3) Å	μ = 0.13 mm^{-1}
c = 7.2930 (15) Å	T = 113 (2) K
β = 103.81 (3)°	Block, colorless
V = 1346.0 (5) Å^3	0.14 × 0.04 × 0.02 mm
Z = 4	

Data collection

Rigaku Saturn CCD area-detector diffractometer	2967 independent reflections
Radiation source: rotating anode	2069 reflections with $I > 2\sigma(I)$
Monochromator: confocal	$R_{int} = 0.085$
Detector resolution: 7.31 pixels mm^{-1}	$\theta_{max} = 27.1°$
$T = 113(2)$ K	$\theta_{min} = 1.6°$
ω and φ scans	$h = -16\rightarrow16$
Absorption correction: multi-scan (CrystalClear; Rigaku/MSC, 2005)	$k = -15\rightarrow18$
$T_{min} = 0.973$, $T_{max} = 0.998$	$l = -9\rightarrow7$
9180 measured reflections	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.045$	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2) = 0.117$	$w = 1/[\sigma^2(F_o^2) + (0.0629P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.00$	$(\Delta/\sigma)_{max} = 0.001$
2967 reflections	$\Delta\rho_{max} = 0.33$ e Å^{-3}
224 parameters	$\Delta\rho_{min} = -0.27$ e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit *S* are based on F^2, conventional *R*-factors *R* are based on *F*, with *F* set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on *F*, and *R*- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.15492 (10)	0.72865 (7)	−0.00354 (17)	0.0204 (3)
H1	0.1297 (17)	0.6740 (14)	−0.033 (3)	0.031*
O2	0.26095 (9)	0.56579 (7)	0.09198 (16)	0.0191 (3)

O3	0.45733 (10)	0.56828 (7)	0.34259 (17)	0.0193 (3)
H3	0.5174 (18)	0.5889 (13)	0.420 (3)	0.029*
O4	0.45077 (9)	0.89559 (7)	0.32487 (17)	0.0173 (3)
O5	0.61065 (9)	0.66388 (7)	0.53713 (16)	0.0188 (3)
O6	1.02531 (10)	0.87836 (8)	0.95714 (18)	0.0221 (3)
H6	1.0680 (18)	0.8325 (15)	0.952 (3)	0.033*
C1	0.25505 (13)	0.72879 (10)	0.1088 (2)	0.0159 (4)
C2	0.30232 (13)	0.81198 (9)	0.1645 (2)	0.0161 (4)
H2	0.2660	0.8676	0.1254	0.019*
C3	0.40402 (13)	0.81206 (9)	0.2788 (2)	0.0142 (4)
C4	0.55224 (13)	0.89830 (10)	0.4301 (2)	0.0157 (4)
H4	0.5850	0.9566	0.4520	0.019*
C5	0.61050 (13)	0.82575 (10)	0.5063 (2)	0.0141 (4)
C6	0.56447 (13)	0.73485 (10)	0.4688 (2)	0.0142 (4)
C7	0.45876 (13)	0.73227 (10)	0.3443 (2)	0.0141 (4)
C8	0.40834 (13)	0.64784 (9)	0.2855 (2)	0.0148 (4)
C9	0.30772 (14)	0.64647 (9)	0.1659 (2)	0.0155 (4)
C10	0.22428 (15)	0.50987 (11)	0.2257 (3)	0.0243 (5)
H10A	0.1689	0.5425	0.2702	0.036*
H10B	0.2838	0.4963	0.3332	0.036*
H10C	0.1952	0.4527	0.1648	0.036*
C11	0.71993 (13)	0.83905 (9)	0.6228 (2)	0.0153 (4)
C12	0.74392 (14)	0.91262 (9)	0.7482 (2)	0.0157 (4)
H12	0.6895	0.9541	0.7595	0.019*
C13	0.84645 (14)	0.92549 (9)	0.8561 (2)	0.0172 (4)
H13	0.8622	0.9759	0.9401	0.021*
C14	0.92533 (13)	0.86514 (10)	0.8412 (2)	0.0165 (4)
C15	0.90422 (14)	0.79282 (10)	0.7145 (2)	0.0197 (4)
H15	0.9594	0.7526	0.7010	0.024*
C16	0.80091 (14)	0.78018 (10)	0.6076 (2)	0.0185 (4)
H16	0.7857	0.7302	0.5225	0.022*
O7	0.04211 (12)	0.58284 (9)	0.8508 (2)	0.0333 (4)
H7A	0.033 (2)	0.5862 (15)	0.718 (4)	0.050*
H7B	0.076 (2)	0.5390 (18)	0.876 (4)	0.050*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0144 (7)	0.0182 (6)	0.0251 (7)	0.0000 (4)	−0.0024 (5)	−0.0017 (4)
O2	0.0203 (7)	0.0157 (5)	0.0204 (6)	−0.0030 (4)	0.0031 (5)	−0.0024 (4)
O3	0.0178 (7)	0.0122 (5)	0.0247 (7)	0.0012 (4)	−0.0012 (5)	0.0003 (4)
O4	0.0142 (6)	0.0122 (5)	0.0233 (6)	0.0001 (4)	0.0001 (5)	0.0003 (4)
O5	0.0170 (6)	0.0151 (5)	0.0228 (6)	0.0026 (4)	0.0018 (5)	0.0022 (4)
O6	0.0142 (7)	0.0193 (6)	0.0287 (7)	0.0001 (5)	−0.0033 (5)	−0.0036 (4)
C1	0.0118 (8)	0.0211 (8)	0.0142 (8)	0.0007 (6)	0.0023 (7)	−0.0008 (5)
C2	0.0150 (9)	0.0167 (7)	0.0167 (8)	0.0031 (6)	0.0039 (7)	0.0011 (5)
C3	0.0144 (8)	0.0137 (7)	0.0156 (8)	−0.0020 (6)	0.0060 (7)	−0.0013 (5)
C4	0.0135 (8)	0.0167 (7)	0.0169 (8)	−0.0019 (6)	0.0038 (7)	−0.0018 (5)

C5	0.0129 (8)	0.0162 (7)	0.0144 (8)	0.0003 (6)	0.0054 (7)	−0.0015 (5)
C6	0.0142 (9)	0.0158 (7)	0.0135 (8)	0.0009 (6)	0.0052 (7)	0.0000 (5)
C7	0.0118 (8)	0.0161 (7)	0.0149 (8)	0.0013 (6)	0.0043 (7)	0.0006 (5)
C8	0.0165 (9)	0.0141 (7)	0.0146 (8)	0.0008 (6)	0.0053 (7)	0.0006 (5)
C9	0.0182 (9)	0.0141 (7)	0.0149 (8)	−0.0021 (6)	0.0054 (7)	−0.0012 (5)
C10	0.0250 (10)	0.0205 (8)	0.0285 (10)	−0.0043 (7)	0.0090 (8)	0.0021 (6)
C11	0.0152 (9)	0.0140 (7)	0.0159 (8)	−0.0019 (6)	0.0023 (7)	0.0016 (5)
C12	0.0162 (9)	0.0136 (7)	0.0181 (8)	0.0019 (6)	0.0056 (7)	0.0015 (5)
C13	0.0199 (10)	0.0125 (7)	0.0186 (8)	−0.0029 (6)	0.0033 (7)	−0.0018 (5)
C14	0.0132 (8)	0.0169 (7)	0.0181 (8)	−0.0018 (6)	0.0013 (7)	0.0022 (5)
C15	0.0153 (9)	0.0183 (7)	0.0253 (9)	0.0030 (6)	0.0045 (7)	−0.0031 (6)
C16	0.0177 (9)	0.0179 (7)	0.0192 (9)	−0.0006 (6)	0.0030 (7)	−0.0047 (5)
O7	0.0329 (9)	0.0253 (6)	0.0358 (9)	0.0005 (6)	−0.0037 (7)	−0.0060 (5)

Geometric parameters (Å, °)

O1—C1	1.3602 (18)	C5—C11	1.483 (2)
O1—H1	0.87 (2)	C6—C7	1.453 (2)
O2—C9	1.3784 (16)	C7—C8	1.4171 (19)
O2—C10	1.439 (2)	C8—C9	1.386 (2)
O3—C8	1.3453 (17)	C10—H10A	0.9800
O3—H3	0.90 (2)	C10—H10B	0.9800
O4—C4	1.3568 (18)	C10—H10C	0.9800
O4—C3	1.3717 (16)	C11—C16	1.384 (2)
O5—C6	1.2434 (17)	C11—C12	1.400 (2)
O6—C14	1.3823 (18)	C12—C13	1.387 (2)
O6—H6	0.88 (2)	C12—H12	0.9500
C1—C2	1.381 (2)	C13—C14	1.376 (2)
C1—C9	1.400 (2)	C13—H13	0.9500
C2—C3	1.382 (2)	C14—C15	1.390 (2)
C2—H2	0.9500	C15—C16	1.393 (2)
C3—C7	1.3917 (19)	C15—H15	0.9500
C4—C5	1.345 (2)	C16—H16	0.9500
C4—H4	0.9500	O7—H7A	0.95 (3)
C5—C6	1.458 (2)	O7—H7B	0.77 (3)
C1—O1—H1	113.5 (13)	O2—C9—C8	121.26 (13)
C9—O2—C10	114.16 (13)	O2—C9—C1	118.99 (13)
C8—O3—H3	100.2 (12)	C8—C9—C1	119.62 (13)
C4—O4—C3	118.50 (11)	O2—C10—H10A	109.5
C14—O6—H6	112.1 (13)	O2—C10—H10B	109.5
O1—C1—C2	118.13 (13)	H10A—C10—H10B	109.5
O1—C1—C9	120.40 (13)	O2—C10—H10C	109.5
C2—C1—C9	121.47 (13)	H10A—C10—H10C	109.5
C1—C2—C3	118.09 (13)	H10B—C10—H10C	109.5
C1—C2—H2	121.0	C16—C11—C12	118.55 (14)
C3—C2—H2	121.0	C16—C11—C5	120.86 (13)
O4—C3—C2	116.81 (12)	C12—C11—C5	120.58 (15)
O4—C3—C7	120.39 (13)	C13—C12—C11	120.59 (15)
C2—C3—C7	122.80 (13)	C13—C12—H12	119.7

C5—C4—O4	125.74 (13)	C11—C12—H12	119.7
C5—C4—H4	117.1	C14—C13—C12	119.88 (14)
O4—C4—H4	117.1	C14—C13—H13	120.1
C4—C5—C6	118.67 (14)	C12—C13—H13	120.1
C4—C5—C11	119.95 (13)	C13—C14—O6	117.93 (14)
C6—C5—C11	121.36 (13)	C13—C14—C15	120.68 (14)
O5—C6—C7	121.41 (13)	O6—C14—C15	121.39 (15)
O5—C6—C5	123.52 (14)	C14—C15—C16	118.98 (16)
C7—C6—C5	115.08 (12)	C14—C15—H15	120.5
C3—C7—C8	117.94 (13)	C16—C15—H15	120.5
C3—C7—C6	121.37 (13)	C11—C16—C15	121.29 (14)
C8—C7—C6	120.69 (13)	C11—C16—H16	119.4
O3—C8—C9	119.11 (12)	C15—C16—H16	119.4
O3—C8—C7	120.86 (13)	H7A—O7—H7B	102 (2)
C9—C8—C7	120.03 (13)		
O1—C1—C2—C3	179.92 (17)	C10—O2—C9—C8	73.9 (2)
C9—C1—C2—C3	0.2 (3)	C10—O2—C9—C1	−110.33 (18)
C4—O4—C3—C2	−177.22 (16)	O3—C8—C9—O2	−5.6 (3)
C4—O4—C3—C7	2.1 (3)	C7—C8—C9—O2	173.72 (17)
C1—C2—C3—O4	177.11 (17)	O3—C8—C9—C1	178.63 (17)
C1—C2—C3—C7	−2.2 (3)	C7—C8—C9—C1	−2.1 (3)
C3—O4—C4—C5	−4.9 (3)	O1—C1—C9—O2	6.3 (3)
O4—C4—C5—C6	2.7 (3)	C2—C1—C9—O2	−173.97 (17)
O4—C4—C5—C11	−178.42 (17)	O1—C1—C9—C8	−177.84 (17)
C4—C5—C6—O5	−177.81 (18)	C2—C1—C9—C8	1.9 (3)
C11—C5—C6—O5	3.3 (3)	C4—C5—C11—C16	−139.51 (19)
C4—C5—C6—C7	2.0 (3)	C6—C5—C11—C16	39.4 (3)
C11—C5—C6—C7	−176.86 (16)	C4—C5—C11—C12	39.3 (3)
O4—C3—C7—C8	−177.26 (17)	C6—C5—C11—C12	−141.85 (18)
C2—C3—C7—C8	2.0 (3)	C16—C11—C12—C13	−0.7 (3)
O4—C3—C7—C6	2.6 (3)	C5—C11—C12—C13	−179.49 (16)
C2—C3—C7—C6	−178.17 (18)	C11—C12—C13—C14	−0.5 (3)
O5—C6—C7—C3	175.31 (19)	C12—C13—C14—O6	−177.31 (16)
C5—C6—C7—C3	−4.5 (3)	C12—C13—C14—C15	2.1 (3)
O5—C6—C7—C8	−4.8 (3)	C13—C14—C15—C16	−2.4 (3)
C5—C6—C7—C8	175.30 (17)	O6—C14—C15—C16	176.94 (16)
C3—C7—C8—O3	179.49 (17)	C12—C11—C16—C15	0.3 (3)
C6—C7—C8—O3	−0.4 (3)	C5—C11—C16—C15	179.12 (17)
C3—C7—C8—C9	0.2 (3)	C14—C15—C16—C11	1.2 (3)
C6—C7—C8—C9	−179.67 (17)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O7—H7B···O2^{i}	0.77 (3)	2.57 (2)	2.971 (2)	114 (2)
O7—H7A···O6^{ii}	0.95 (3)	1.95 (3)	2.884 (2)	167 (2)
O6—H6···O1^{iii}	0.88 (2)	1.88 (2)	2.7368 (17)	167 (2)
O3—H3···O5	0.90 (2)	1.71 (2)	2.5658 (16)	159.6 (18)

O1—H1···O7^{iv}	0.87 (2)	1.83 (2)	2.6630 (17)	160.4 (19)

Symmetry codes: (i) *x*, *y*, *z*+1; (ii) *x*−1, −*y*+3/2, *z*−1/2; (iii) *x*+1, *y*, *z*+1; (iv) *x*, *y*, *z*−1.

Fig. 1