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Bergenin monohydrate, a constituent of *Hurmiria balsamifera*, at 120 K

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The title compound, 3,4,4a,10b-tetrahydro-3,4,8,10-tetrahydroxy-2-(hydroxymethyl)-9-methoxypyrano[3,2-*c*][2]benzo-*H*)-one monohydrate, $C_{14}H_{16}O_9 \cdot H_2O$, crystallized in space group $P2_12_12_1$ from methanol. The data collection was made at low temperature and the results obtained agree with those reported in another publication for the same compound determined at room temperature [Shang-Zhen, Hua, Xu-Wei, Zhong-Yuan & Guo-Zhi (1989). *J. Struct. Chem.* **8**, 305–310]. However, there are small differences in the cell constants and some geometrical parameters.

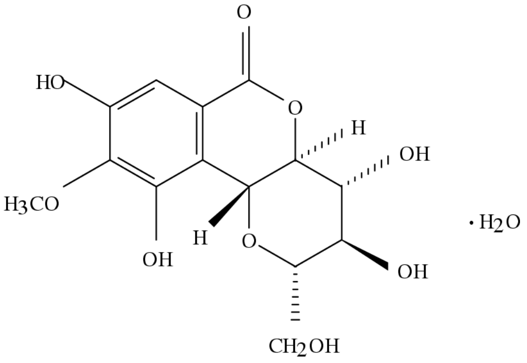
Key indicators

Single-crystal X-ray study
T = 120 K
Mean σ(C–C) = 0.004 Å
R factor = 0.037
wR factor = 0.129
Data-to-parameter ratio = 8.6

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

Comment

Bergenin, a natural isocoumarin, has been isolated from a variety of plants (Piacente *et al.*, 1996; Prithiviray *et al.*, 1997; Hua *et al.*, 1998; Lim *et al.*, 2000) and was reported to exhibit several biological activities, such as hepatoprotective (Lim *et al.*, 2000), antihepatotoxicity on carbon tetrachloride-intoxicated hepatocytes (Kim *et al.*, 2000), antifungal (Prithiviray *et al.*, 1997) and anti-HIV (Piacente *et al.*, 1996). This substance has been synthesized through an intramolecular *c*-glycosylation reaction (Rousseau & Martin, 2000) and its structure was determined by X-ray analysis of its 3,4,8,10,11-pentaacetate derivative (Frick *et al.*, 1991). Its structure was determined by conventional spectroscopic methods including IR, UV and NMR spectra of both ^{1}H and ^{13}C nuclei. However, as the spectroscopic data do not permit unambiguous definition of the stereochemistry, a single-crystal structure determination was undertaken.



(I)

In the title compound, (I) (Fig. 1), atoms C5, C12, O5, O6 and O7 are in the same least-squares plane through ring *A*, within experimental error, as shown by the value of σ_{av} [defined as $(\Sigma d_i^2/N-3)^{1/2}$] of 0.102 Å for the 11 atoms. Atoms O3 and C4 are −0.258 (2) and −0.795 (2) Å out of that plane, respectively. Therefore, ring *B* is in a distorted half-chair

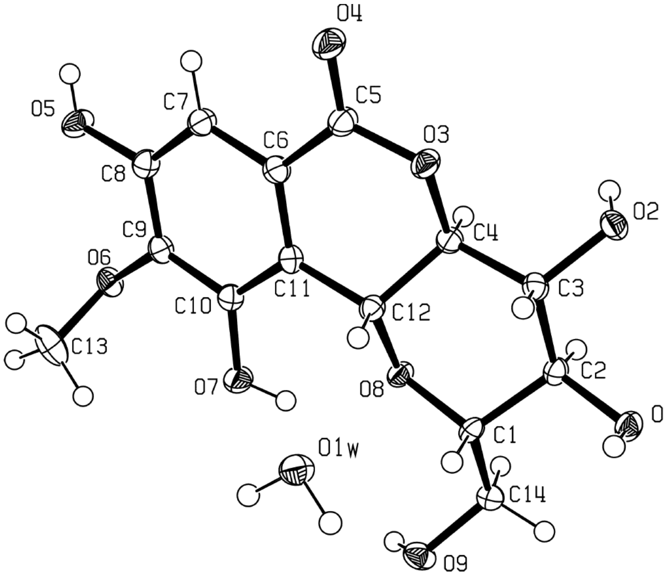


Figure 1
View of the molecule of (I), showing the atom labelling, with 50% probability displacement ellipsoids.

conformation, the Cremer & Pople (1975) ring-puckering parameters being q_2 = 0.424 (1), q_3 = 0.249 (1) Å, Q = 0.492 (1) Å, θ = 59.5 (2)° and φ = 340.3 (2)°. The H atoms on C12 and C4 at the junction of rings *B* and *C* are in *trans* positions. Ring *C* is essentially in a chair conformation [puckering parameters for six-membered ring: q_2 = 0.107 (1), q_3 = −0.585 (1) Å, Q = 0.594 (1) Å, θ = 169.7 (1)° and φ = 76.8 (7)°]. Atom C2 is located −1.108 (2) Å out of the least-squares plane through ring *A*, whereas C12 is in that plane. The O1 and O2 hydroxyl groups, as well as the CH_2OH group, are in equatorial positions, maintaining a *trans* relation with the neighbouring C atom.

The overall geometry of rings *B* and *C* is in good agreement with the very similar geometry for these moieties observed in a similar compound (Frick *et al.*, 1991). Determination of the absolute configuration of (I) was not possible in the present X-ray analysis, owing to the absence of suitable anomalous scatterers within the molecule.

The molecules are joined through intra- and intermolecular O—H···O hydrogen bonds and the water molecule forms hydrogen bonds with different groups of the bergenin molecule (details are in Table 2).

The unit cell and some geometrical parameters (Table 1), obtained at low temperature for the title compound, show small differences when compared with those reported for the same compound determined at room temperature by Shang-Zhen *et al.* (1989).

Experimental

Bergenin was isolated from an ethyl acetate fraction, obtained from an ethanol extract after partition of the solvents, from the leaves of *Hurmiria balsamifera (Aubl.) St. Hil (Humiriaseae)* by chromatography on silica gel, eluting with different C_6H_{14}–ethyl acetate ratios, and was recrystallized from methanol.

Crystal data

$C_{14}H_{16}O_9{\cdot}H_2O$
$M_r = 346.28$
Orthorhombic, $P2_12_12_1$
$a = 7.484$ (2) Å
$b = 13.901$ (3) Å
$c = 14.132$ (4) Å
$V = 1470.2$ (7) Å^3
$Z = 4$
$D_x = 1.564$ Mg m^{-3}

Mo $K\alpha$ radiation
Cell parameters from 1951 reflections
$\theta = 1.0$–27.5°
$\mu = 0.14$ mm^{-1}
$T = 120$ (2) K
Prism, colourless
0.28 × 0.16 × 0.14 mm

Data collection

Nonius KappaCCD diffractometer
φ scans, and ω scans with κ offsets
Absorption correction: none
1943 measured reflections
1943 independent reflections

1713 reflections with $I > 2\sigma(I)$
$\theta_{\max} = 27.5°$
$h = 0 \rightarrow 9$
$k = 0 \rightarrow 17$
$l = 0 \rightarrow 18$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.037$
$wR(F^2) = 0.129$
$S = 1.26$
1943 reflections
225 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.076P)^2 + 0.0815P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.001$
$\Delta\rho_{\max} = 0.36$ e Å^{-3}
$\Delta\rho_{\min} = -0.44$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

O1—C2	1.429 (3)	O9—C14	1.429 (3)
O2—C3	1.428 (3)	C1—C14	1.516 (4)
O3—C5	1.353 (3)	C1—C2	1.539 (4)
O3—C4	1.447 (3)	C2—C3	1.529 (4)
O6—C9	1.372 (3)	C3—C4	1.515 (3)
O6—C13	1.434 (4)	C4—C12	1.517 (4)
O8—C12	1.438 (3)	C5—C6	1.480 (4)
O8—C1	1.447 (3)		
C5—O3—C4	117.7 (2)	O3—C4—C12	110.7 (2)
C9—O6—C13	112.8 (2)	O4—C5—O3	116.6 (2)
C12—O8—C1	110.52 (19)	O6—C9—C8	118.5 (2)
O8—C1—C14	107.50 (19)	O8—C12—C11	110.9 (2)
O8—C1—C2	109.5 (2)	O8—C12—C4	107.7 (2)
O3—C4—C3	107.2 (2)	O9—C14—C1	111.8 (2)

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1′···O2^{i}	0.84	1.88	2.714 (2)	170
O2—H2′···O9^{ii}	0.84	1.89	2.725 (2)	171
O5—H5′···O1*W*^{iii}	0.84	1.78	2.613 (2)	169
O7—H7′···O8	0.84	1.94	2.668 (2)	144
O9—H9′···O5^{iv}	0.84	2.00	2.799 (2)	158
O1*W*—H11*W*···O1^{i}	0.88	1.88	2.753 (2)	174
O1*W*—H12*W*···O3^{v}	0.91	2.57	3.265 (2)	133
O1*W*—H12*W*···O4^{v}	0.91	1.93	2.834 (2)	170
C2—H2···O4^{vi}	1.00	2.34	3.327 (3)	170
C4—H4···O6^{vii}	1.00	2.51	3.427 (2)	152
C12—H12···O1*W*	1.00	2.39	3.306 (3)	152
C13—H13*A*···O7	0.98	2.44	3.019 (3)	117

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

o610 C. S. Caldas et al. • $C_{14}H_{16}O_9{\cdot}H_2O$ Acta Cryst. (2002). E58, o609–o611

H atoms were positioned on stereochemical grounds, and were refined with fixed geometry, each riding on a carrier atom, with an isotropic displacement parameter of 1.5 (for hydroxyl-H atoms) or 1.2 (for the other H atoms) times the equivalent isotropic displacement parameter of the attached atom. In addition, for both water-H atoms, the isotropic displacement parameter was refined. The methyl and hydroxyl groups were allowed to rotate, but not tip.

Data collection: *COLLECT* (Nonius, 1999); cell refinement: *HKL SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *HKL SCALEPACK*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*-3 (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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Acta Cryst. (2002). E**58**, o609–o611 C. S. Caldas *et al.* • $C_{14}H_{16}O_9 \cdot H_2O$ **o611**