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3,5-Dihydroxy-2-hydroxymethyl-4*H*-pyran-4-one

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.004 Å
R factor = 0.061
wR factor = 0.182
Data-to-parameter ratio = 11.6

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

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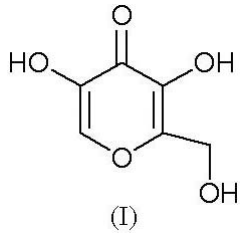
In the title compound, $C_6H_6O_5$, an extract from *Gaultheria leucocarpa* B1 var. crenulata, the asymmetric unit consists of two nearly parallel molecules with different conformations linked by a strong intermolecular O—H···O hydrogen bond. The pyranoid ring in both molecules is planar, and the dihedral angle formed by these planes is 7.14 (15)°. The crystal packing is stabilized by O—H···O intermolecular hydrogen bonds, resulting in the formation of a two-dimensional network.

Comment

The title compound, (I), commonly known as 3-hydroxykojic acid, was first isolated from the culture broth of *Gluconobacter cerinus* var. ammoniac (Terada *et al.*, 1961). In the course of our systematic search for bioactive substances from Chinese traditional and herbal medicine, we have studied the leaves of *Gaultheria leucocarpa* B1 var. crenulata, which was widely used for the treatment of rheumatoid arthritis, swelling pain, trauma, chronic tracheitis, cold and vertigo (Jiangsu New Medical College, 1977), and obtained the title compound.



(I)

As shown in Fig. 1, the asymmetric unit of (I) consists of two nearly parallel molecules linked by a strong intermolecular O5—H6···O10 hydrogen bond. The two molecules differ in the orientation of the hydroxy groups. The pyranoid ring in both molecules is planar, and the dihedral angle between the two planes is 7.14 (15)°.

Although hydroxy groups O2—H2, O4—H3, O7—H8 and O9—H9 are adjacent to the carbonyl C3═O3 and C9═O8 bonds, no significant intramolecular O—H···O hydrogen-bond interactions are observed in the crystal structure. The crystal packing is stabilized by strong intermolecular O—H···O hydrogen bonds involving the hydroxy groups and carbonyl atoms O3 and O8, resulting in the formation of a two-dimensional sheet (Fig. 2 and Table 1).

Experimental

The leaves and stems of *G. leucocarpa* B1 var. crenulata (Kutz, T. Z. Hsu) were collected in the Daming Mountain of Guangxi Province,

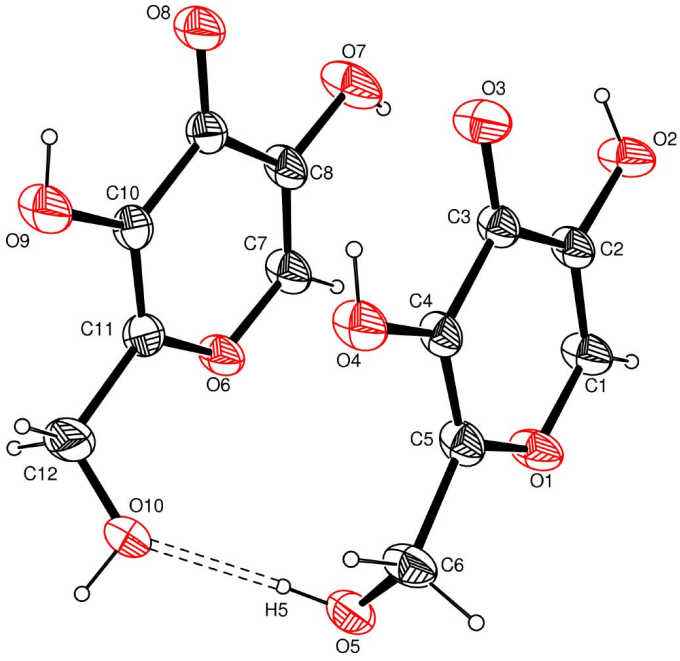


Figure 1
An *ORTEP* (Farrugia, 1997; Burnett & Johnson, 1996) view of the asymmetric unit of (I), showing the atom-labeling scheme and 50% probability displacement ellipsoids. The hydrogen bond is shown with dashed lines.

People's Republic of China, and identified by Professor Bing-Yang Ding of Wenzhou Normal University. The air-dried and powdered plant materials (4 kg) were extracted with 95% EtOH. The EtOH extracts were evaporated under reduced pressure to give a residue which was suspended in distilled water and partitioned successively with petroleum ether, $CHCl_3$, EtOAc and *n*-BuOH. The EtOAC extract (100 g) was introduced into a silica gel column and eluted with $CHCl_3$–MeOH (10:1) to obtain the title compound (30 mg).

Crystal data

$C_6H_6O_5$
M_r = 158.11
Triclinic, $P\overline{1}$
a = 8.2045 (9) Å
b = 8.9704 (10) Å
c = 9.6826 (10) Å
α = 93.026 (2)°
β = 109.337 (2)°
γ = 109.787 (2)°
V = 621.71 (12) Å^3
Z = 4
D_x = 1.689 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 1957 reflections
θ = 4.9–56.1°
μ = 0.15 mm^{-1}
T = 293 (2) K
Block, colorless
0.51 × 0.50 × 0.39 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.914, T_{max} = 0.939
3405 measured reflections
2387 independent reflections
2007 reflections with $I > 2\sigma(I)$
R_{int} = 0.063
θ_{max} = 26.0°
$h = -8 \rightarrow 10$
$k = -11 \rightarrow 11$
$l = -10 \rightarrow 11$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.061
$wR(F^2)$ = 0.182
S = 1.08
2387 reflections
205 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.1098P)^2 + 0.1959P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.55 e Å^{-3}
$\Delta\rho_{min}$ = −0.39 e Å^{-3}

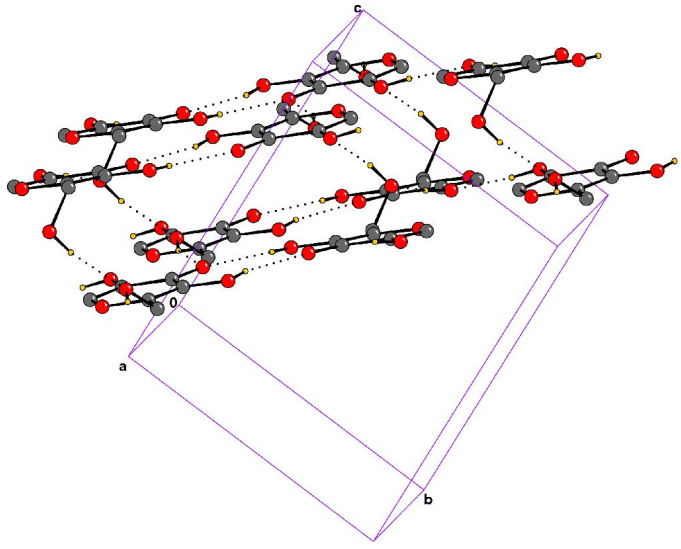


Figure 2
A *CAMERON* (Watkin *et al.*, 1993) packing plot of (I), viewed approximately along the crystallographic *a* axis. H atoms not involved in hydrogen bonding have been omitted for clarity and hydrogen bonds are shown as dashed lines.

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

D—H···A	D—H	H···A	D···A	D—H···A
O2—H2···O5^{i}	0.82	1.85	2.597 (2)	151.4
O2—H2···O3	0.82	2.35	2.756 (2)	111.5
O4—H4···O8^{ii}	0.82	2.04	2.765 (2)	147.9
O4—H4···O3	0.82	2.33	2.755 (2)	112.8
O5—H5···O10	0.82	1.85	2.657 (3)	168.3
O7—H7···O2^{iii}	0.82	1.85	2.655 (2)	166.6
O9—H9···O3^{ii}	0.82	1.90	2.614 (2)	145.7
O9—H9···O8	0.82	2.37	2.784 (2)	112.0
O10—H10···O8^{iv}	0.82	2.06	2.849 (2)	160.7
O10—H10···O7^{iv}	0.82	2.32	2.839 (3)	121.9

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

All H atoms were located in a difference Fourier map, but they were introduced in calculated positions and treated as riding on their parent atoms [C—H = 0.93–0.97 Å, O—H = 0.82 Å, and U_{iso}(H) = 1.2U_{eq}(C) and 1.5U_{eq}(C,O)].

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT* (Bruker, 2000); data reduction: *SAINT* and *SHELXTL* (Bruker, 2000); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEPIII* (Burnett & Johnson, 1996), *ORTEP-3 for Windows* (Farrugia, 1997) and *CAMERON* (Watkin *et al.*, 1993); software used to prepare material for publication: *SHELXTL*.

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