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(1*S*,3a*R*,4*S*,6a*R*)-1-(4-Hydroxy-3-methoxyphenyl)-4-(4-hydroxy-3-methylphenyl)perhydrofuro[3,4-*c*]-furan-3a,6a-diol hexahydrate

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.004 Å
H-atom completeness 77%
Disorder in solvent or counterion
R factor = 0.043
wR factor = 0.129
Data-to-parameter ratio = 9.4

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

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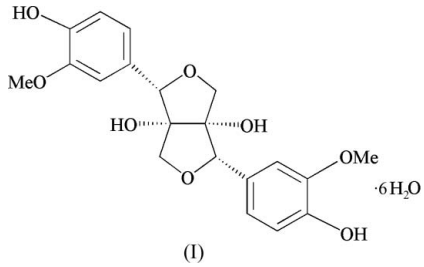
In the title compound, $C_{20}H_{22}O_8\cdot 6H_2O$, the asymmetric unit contains one half-molecule with the other half generated by a crystallographic twofold axis of symmetry. It was extracted from the leaves of *Prinsepia utilis Royle* and displays powerful anti-oxidant activity.

Comment

Prinsepia utilis Royle is a shrub plant growing at an altitude of 1000–2500 m in the south of China and India. Its leaves have been used for various skin diseases, and also for rheumatism, in traditional Chinese medicine. In order to investigate its bioactive natural products, we have undertaken chemical studies on *Prinsepia utilis Royle*, and have obtained an anti-oxidant lignan, (I), from an ethyl acetate extract of its leaves. This lignan, prinsepiol, was previously described by Kilidhar *et al.* (1982). It displays powerful anti-oxidant activity (Piccinelli *et al.*, 2004). An X-ray crystal structure determination of (I) was carried out in order to elucidate its structure and the results are presented here.



(I)

The molecular structure of (I) is illustrated in Fig. 1. The asymmetric unit contains one half-molecule. The other half is generated by a crystallographic twofold axis of symmetry; this axis passes through the mid-point of the C1—C1A bond [symmetry code: (A) $-x+1, -y, z$] and is parallel to the *c* axis of the unit cell. The bond distance C1—C1A of 1.546 (4) Å confirms its single-bond character. The atoms of the methoxy group attached to the benzene ring do not deviate substantially from the plane of the ring; the maximum deviation of 0.0833 (3) Å is observed for atom C10. The dihedral angle between the two benzene rings is 70.9 (3)°. As atom C2 is part of a five-membered ring, it has a distorted tetrahedral geometry, with the O2—C2—C1 [104.65 (19)°] and C4—C2—C1 [115.7 (2)°] angles deviating significantly from ideal tetrahedral values. The packing of the molecules in the solid state is stabilized by O—H···O intermolecular interactions; Table 1 lists the hydrogen-bond contacts. The relative configuration of the two asymmetric C atoms in the asymmetric unit and the *cis* orientation of the fused five-membered rings are established by this determination.

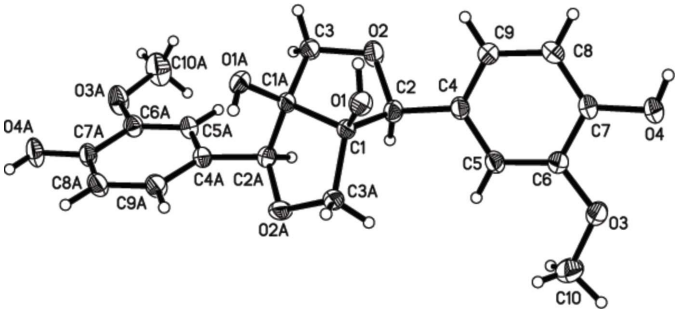


Figure 1
A view of the molecule (I) showing the atom-labeling scheme. Displacement ellipsoids are drawn at the 30% probability level. Water molecules have been omitted.

Experimental

The leaves of *Prinsepia utilis Royle* were dried at 313 K in the dark. The material (1.3 kg) was refluxed three times with 95% EtOH (10 l each time). The extract was concentrated under reduced pressure to give a residue (190 g) which was partitioned between ethyl acetate and water. The EtOAc layer was concentrated to a residue of 28 g. Chromatographic separation was performed on a silica-gel column, using solvents of increasing polarity as mobile phases to give 16 fractions. Fraction 11 (2 g) was chromatographed on Sephadex LH-20 (MeOH) to give five fractions (11.1–11.5). Fraction 11.4 (280 mg) was separated by high-performance liquid chromatography (HPLC) to give 12 fractions (11.4.1–11.4.12). Fraction 11.4.3 (31 mg) was separated by HPLC to give the pure title compound, (I) (m.p. 464–465 K). ^{13}C NMR (CD_3OD): δ 148.8, 147.6, 129.7, 121.7, 115.7, 113.0, 89.2, 88.5, 76.9, 56.5. Crystals suitable for X-ray structure analysis were obtained by slow evaporation of a solution in methanol and water at room temperature.

Crystal data

$C_{20}H_{22}O_8\cdot 6H_2O$
M_r = 498.47
Orthorhombic, $P2_12_12$
a = 19.060 (3) Å
b = 6.6887 (11) Å
c = 9.3752 (16) Å
V = 1195.2 (3) Å^3
Z = 2
D_x = 1.385 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 1916 reflections
θ = 2.4–22.1°
μ = 0.12 mm^{-1}
T = 293 (2) K
Block, colorless
0.26 × 0.20 × 0.16 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.972, T_{max} = 0.981
7991 measured reflections
1692 independent reflections
1270 reflections with $I > 2\sigma(I)$
R_{int} = 0.038
θ_{max} = 28.0°
$h = -24 \rightarrow 25$
$k = -6 \rightarrow 8$
$l = -12 \rightarrow 12$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.043
$wR(F^2)$ = 0.129
S = 1.04
1692 reflections
180 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0873P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.28 e Å^{-3}
$\Delta\rho_{min}$ = −0.17 e Å^{-3}
Extinction correction: *SHELXL97*
Extinction coefficient: 0.005 (4)

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

D—H···A	D—H	H···A	D···A	D—H···A
O5—H5*C*···O10^{i}	0.85	2.23	3.041 (19)	160
O5—H5*C*···O8^{ii}	0.85	1.86	2.663 (7)	158
O5—H5*B*···O5^{ii}	0.85	2.11	2.925 (5)	164
O5—H5*A*···O3^{iii}	0.85	2.25	2.989 (3)	145
O5—H5*A*···O4^{iii}	0.85	2.21	2.929 (3)	142
O4—H4···O1^{iii}	0.82	1.85	2.656 (2)	166
O1—H1···O5	0.82	1.93	2.732 (3)	167

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

All H atoms were positioned geometrically (C—H = 0.93–0.97 Å and O—H = 0.82–0.85 Å) and refined as riding. For the CH and CH_2 groups, U_{iso}(H) values were set equal to $1.2U_{eq}$(carrier atom) and for the methyl groups they were set equal to $1.5U_{eq}$(carrier atom). The asymmetric unit contains three H_2O molecules. One of these, O5, was ordered (*i.e.* it displayed full occupancy), although one of its H atoms was disordered equally over two sites. The other two water molecules were distributed over five sites, O6–O10, with partial occupancies of 0.64 (1), 0.36 (1), 0.692 (7), 0.183 (7) and 0.125 (9), respectively. H atoms on the disordered waters were not found or positioned, but they were assumed to be present when calculating the molecular weight of the asymmetric unit and the crystal density. Anomalous dispersion effects were too small to establish the absolute configuration, so the Friedel equivalents were merged in the refinement.

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT* (Bruker, 1997); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1997); software used to prepare material for publication: *SHELXTL*.

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