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Key indicators

# Syringin monohydrate

In the title compound, $C_{17}H_{24}O_9 \cdot H_2O$, the syringin [4-(3-hydroxy-1-propenyl)-2,6-dimethoxyphenyl-β-D-glucopyranoside] molecule contains a benzene ring, two methoxy groups, an allyl group and a D-glucose fragment which adopts a chair conformation. A molecular column running along the a axis is formed *via* O—H···O and C—H···O hydrogen bonds and C—H···π interactions. The columns are assembled into a three-dimensional framework by an O—H···O hydrogen bond.

Comment

The title compound, (I), was first obtained from the radix of *Acanthopanax senticosus* by Bernays (1841) and found to possess immunomodulatory activities (Cho *et al.*, 2001) and anti-inflammatory and antinociceptive activities (Choi *et al.*, 2004). It was isolated from the heartwood of *Osmanthus armatus* during the course of our ongoing studies on the chemical constituents of *Osmanthus* (Yin *et al.*, 2006), which is distributed in southeast China and contains mainly lignan, phenylpropanoid and secoiridiod glycosides. The structure of syringin was elucidated on the basis of spectroscopic methods such as ^{1}H and ^{13}C NMR (Wang, 1980; Karasawa, 1986; Wu *et al.*, 1999). We report here the crystal structure of (I) (Fig. 1).

(I)
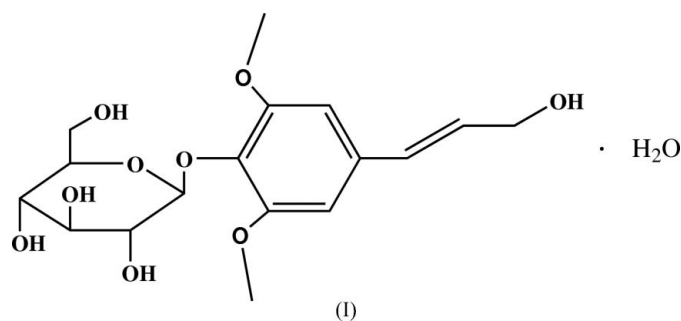
The syringin molecule, except for the D-glucose fragment, is planar with an r.m.s. deviation of 0.0535 Å for atoms C1–C11 and O1–O4. The D-glucose ring adopts a chair conformation with puckering parameters $Q = 0.585$ (6) Å, $\theta = 5.6$ (6)° and $\Phi = 45$ (7)° (Cremer & Pople, 1975) as does that in phillyrin (Yin *et al.*, 2006). The hydroxymethyl group and the benzene ring have a *trans* configuration. In the crystal structure of (I), the syringin and water molecules are linked *via* O—H···O hydrogen bonds, and the syringin molecules related by translation along the a axis are connected by C—H···O hydrogen bonds and C—H···π interactions (Table 1; Cg is the centroid of the benzene ring), forming a molecular column along the a axis (Fig. 2). The columns are further linked by an
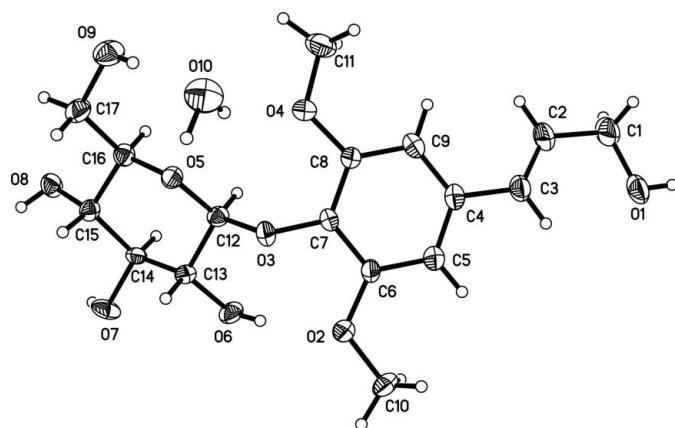
Figure 1
A view of the asymmetric unit of the title compound, showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level. H atoms are shown as spheres of arbitrary radii.
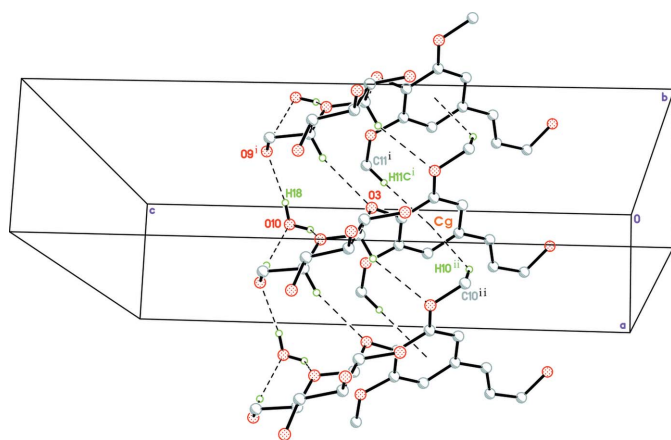

Figure 2
A partial packing diagram of (I), showing the hydrogen bonds and C—H···π interactions (dashed lines). For the sake of clarity, H atoms not involved in these interactions have been omitted (symmetry codes as in Table 1).

$O8$—$H8$···$O1^{iii}$ hydrogen bond (Table 1), forming a three-dimensional framework.

Experimental

The dried heartwood (2 kg) was extracted three times with boiling ethanol (95%, 3 × 2 l). After removal of the solvent under reduced pressure, the extract was suspended in water and then partitioned successively with light petroleum, $CHCl_3$, EtOAc and n-BuOH. The n-BuOH fraction was chromatographed on a silica gel column, using a gradient mixture of $CHCl_3$–CH_3OH as eluant. The fraction eluted with $CHCl_3$–CH_3OH (4:1 v/v) was further purified by sephadex LH-20 column chromatography using $CHCl_3$–MeOH (1:1 v/v) as eluant to afford the title compound (2.0 g, m.p. 464–465 K). $[\alpha]_D = -5.8$ (c, 0.01, MeOH). ESI–MS (m/z): 407 $[M+Cl]^-$. ^1H NMR (MeOD): δ 6.75 (2H, s, H-3, H-5), 6.55 (1H, d, $J = 15.9$ Hz, H-7), 6.33 (1H, dt, $J_1 = 15.9$ Hz, $J_2 = 5.5$ Hz, H-8), 4.87(1H, d, $J = 7.5$, H-1'), 4.22 (2H, d, $J = 5.5$ Hz, H-9), 3.86 (6H, s, H-10,11), 3.80–3.30 (6H, m, sugar H); ^{13}C NMR (MeOD): δ 154.4 (C-2,6), 136.0 (C-7), 135.3 (C-1), 131.3 (C-7), 130.1 (C-8), 105.5 (C-3,5), 105.4 (C-1'), 78.4 (C-5'), 77.8 (C-3'), 75.7 (C-2'), 71.4 (C-4'), 63.5 (C-9), 62.6 (C-6'), 57.0 (C-10,11). Crystals

suitable for diffraction analysis were obtained by slow evaporation of a methanol solution at room temperature.

Crystal data

$C_{17}H_{24}O_9 \cdot H_2O$	$Z = 4$
$M_r = 390.38$	$D_x = 1.362$ Mg m^{-3}
Orthorhombic, $P2_12_12_1$	Mo $K\alpha$ radiation
$a = 4.855$ (2) Å	$\mu = 0.11$ mm^{-1}
$b = 19.519$ (3) Å	$T = 298$ (2) K
$c = 20.084$ (3) Å	Prism, colourless
$V = 1903.3$ (9) Å^3	$0.49 \times 0.15 \times 0.10$ mm

Data collection

Bruker SMART 1000 CCD area-detector diffractometer	9643 measured reflections
φ and ω scans	1986 independent reflections
Absorption correction: multi-scan (*SADABS*; Bruker, 2000)	1241 reflections with $I > 2\sigma(I)$
$T_{min} = 0.980$, $T_{max} = 0.989$	$R_{int} = 0.085$
	$\theta_{max} = 25.0°$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0605P)^2$
$R[F^2 > 2\sigma(F^2)] = 0.057$	$+ 3.1656P]$
$wR(F^2) = 0.168$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.02$	$(\Delta/\sigma)_{max} < 0.001$
1986 reflections	$\Delta\rho_{max} = 0.25$ e Å^{-3}
251 parameters	$\Delta\rho_{min} = -0.33$ e Å^{-3}
H-atom parameters constrained	

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

D—H···A	D—H	H···A	D···A	D—H···A
$O6$—$H6$···$O2$	0.82	2.10	2.916 (7)	172
$O6$—$H6$···$O3$	0.82	2.47	2.827 (5)	107
$O9$—$H9$···$O10$	0.82	2.07	2.881 (8)	172
$O10$—$H18$···$O9^i$	0.85	2.06	2.902 (9)	172
$O10$—$H19$···$O5$	0.85	2.36	2.921 (6)	124
$C3$—$H3$···$O1$	0.93	2.38	2.768 (7)	105
$C13$—$H13$···$O8^i$	0.98	2.40	3.334 (8)	160
$C12$—$H12$···$O2^{ii}$	0.98	2.63	3.538 (8)	154
$C16$—$H16$···$O3^{ii}$	0.98	2.54	3.493 (8)	163
$C17$—$H17A$···$O8$	0.97	2.59	2.938 (8)	101
$O8$—$H8$···$O1^{iii}$	0.82	1.92	2.730 (6)	168
$C10$—$H10C$···Cg^i	0.96	3.00	3.805 (6)	142
$C11$—$H11C$···Cg^{ii}	0.96	2.92	3.812 (9)	155

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

H atoms of the water molecule and the hydroxyl groups were located initially in a difference Fourier map and then constrained to ride on the parent O atom with O—H = 0.82–0.85 Å and with $U_{iso}(H) = 1.5U_{eq}(O)$. The methyl H atoms were constrained to an ideal geometry with C—H = 0.96 Å and $U_{iso}(H) = 1.5U_{eq}(C)$, but were allowed to rotate freely about the C—C bonds. All remaining H atoms were placed in geometrically idealized positions (C—H = 0.93–0.98 Å) and constrained to ride on their parent atoms with $U_{iso}(H) = 1.2U_{eq}(C)$. In the absence of significant anomalous scattering effects, Friedel pairs were merged; the absolute configuration was assigned on the basis of the known configuration of D-glucose.

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT* (Bruker, 2000); 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, 2000); software used to prepare material for publication: *SHELXTL* and *PLATON* (Spek, 2003).

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