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Key indicators

Single-crystal X-ray study
 $T = 293$ K
 Mean $\sigma(\text{C}-\text{C}) = 0.006$ Å
 R factor = 0.067
 wR factor = 0.125
 Data-to-parameter ratio = 11.9

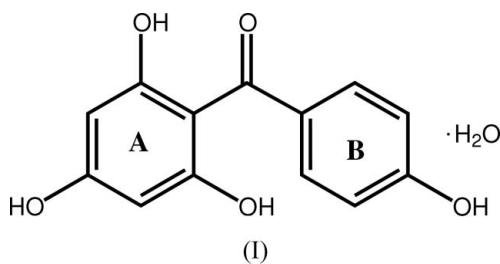
For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

(4-Hydroxyphenyl)(2,4,6-trihydroxyphenyl)methanone monohydrate

The title compound, $\text{C}_{13}\text{H}_{10}\text{O}_5$, is a benzophenone derivative containing phloroglucinol and *p*-hydroxybenzoyl groups. The *p*-hydroxybenzoyl group is twisted out of the plane of the phloroglucinol group as a result of steric hindrance [dihedral angle = $50.2(1)^\circ$]. In the crystal packing, screw-related molecules are linked by $\text{O}-\text{H}\cdots\text{O}$ hydrogen bonds to form a zigzag layer parallel to $(10\bar{1})$. Adjacent layers are interlinked by $\text{O}-\text{H}\cdots\text{O}$ hydrogen bonds involving the solvent water molecules.

Comment

Iris germanica Linn. is a decorative plant which blooms in May and June. The flowers are large and deep blue or purple–blue in colour. It belongs to the family Iridaceae, which is popular for its flowers. In ancient Greece and Rome, *Iris* root was used in perfumery. The fresh root possesses diuretic, emetic and cathartic properties. Phytochemical studies revealed that the plant possesses flavonoids, isoflavons and flavonols (Orhan *et al.*, 2003; Atta-ur-Rahman *et al.*, 2003). The title compound, (I), known as iriflophenone, is an important benzophenone derivative, which is present in various species of the genus *Iris*, such as *I. germanica* and *I. florentina* (Nishikawa & Robinson, 1922; Dhar & Kalla, 1974; Arisawa *et al.*, 1973). Compound (I) has been isolated for the first time from *I. germanica*. We report here the X-ray crystal structure of (I) (Fig. 1).



The $\text{C}7-\text{C}8$ bond [$1.451(5)$ Å] is shorter than the $\text{C}1-\text{C}7$ bond [$1.475(5)$ Å]; this difference is presumably due to the electron-donating effect of the two *ortho*-substituted hydroxy groups to the $\text{C}9$ and $\text{C}13$ positions in the phloroglucinol ring. There is electron delocalization over the $\text{O}3-\text{C}9=\text{C}8-\text{C}7=\text{O}2$ and $\text{O}5-\text{C}13-\text{C}8-\text{C}7=\text{O}2$ fragments. As a result, the $\text{C}8=\text{C}9$ [$1.418(4)$ Å] and $\text{C}8-\text{C}13$ [$1.408(5)$ Å] bonds are slightly longer than the $\text{C}9-\text{C}10$ [$1.371(5)$ Å], $\text{C}10=\text{C}11$ [$1.382(5)$ Å], $\text{C}11-\text{C}12$ [$1.381(5)$ Å] and $\text{C}12=\text{C}13$ [$1.371(5)$ Å] bonds. As a result of the steric interactions, the *p*-hydroxybenzoyl group at the $\text{C}8$ position is twisted away from the phloroglucinol ring; the dihedral angle between the $\text{C}1-\text{C}7/\text{O}1$ and $\text{C}8-\text{C}13/\text{O}3-\text{O}5$ planes is $50.2(1)^\circ$.

Received 23 September 2005
 Accepted 4 October 2005
 Online 8 October 2005

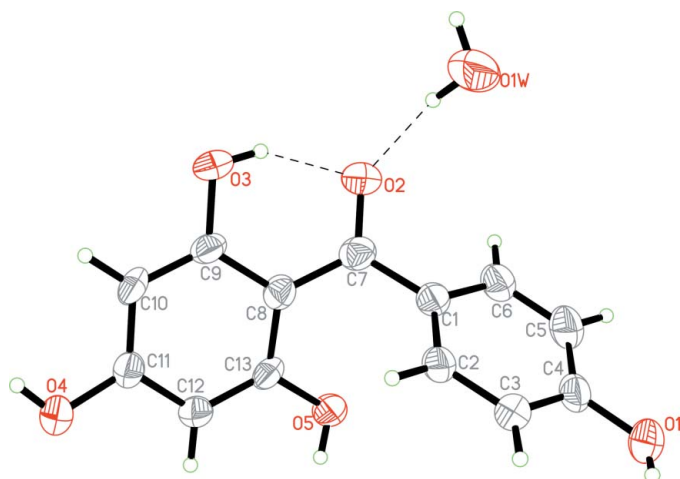


Figure 1
The molecular structure of (I), showing 50% probability displacement ellipsoids and the atom-numbering scheme. Hydrogen bonds are shown as dashed lines.

An intramolecular O3—H3A···O2 hydrogen bond generates a ring of graph-set motif $S(6)$ (Bernstein *et al.*, 1995). In the crystal packing, screw-related molecules are linked by O4—H4A···O3ⁱ and O5—H5A···O1ⁱⁱ (symmetry codes are given in Table 1) hydrogen bonds to form a zigzag layer parallel to (10 $\bar{1}$). The adjacent layers are linked together by O—H···O hydrogen bonds involving the water molecules (Fig. 2).

Experimental

The air-dried rhizomes of *I. germanica* were crushed into a moderately coarse powder (3 kg) and were defatted with hexane (12 l) at room temperature. Plant material was extracted with ethanol–water (80:20, 25 × 3 l) and the extract was vacuum-dried (765 g). The extract was further fractionated with dichloromethane (145 g, 6 l), ethyl acetate (50 g, 6 l) and *n*-butanol (202 g, 6 l). The ethyl acetate extract (15.5 g) was subjected to silica gel column chromatography with increasing polarities of petroleum ether–dichloromethane and then with methanol. Compound (I) was isolated at 1% methanol–dichloromethane in 22 mg ($14.67 \times 10^{-2}\%$, $R_F = 0.22$). It was recrystallized from dichloromethane–methanol (9.7:0.3) to give yellow single crystals (m.p. 482–483 K).

Crystal data

$C_{13}H_{10}O_5 \cdot H_2O$	$D_x = 1.445 \text{ Mg m}^{-3}$
$M_r = 264.23$	Mo $K\alpha$ radiation
Monoclinic, $P2_1/c$	Cell parameters from 1444 reflections
$a = 13.305 (2) \text{ \AA}$	$\theta = 1.5\text{--}25.0^\circ$
$b = 10.365 (2) \text{ \AA}$	$\mu = 0.12 \text{ mm}^{-1}$
$c = 8.8087 (17) \text{ \AA}$	$T = 293 (2) \text{ K}$
$\beta = 90.949 (4)^\circ$	Needle, yellow
$V = 1214.6 (4) \text{ \AA}^3$	$0.39 \times 0.07 \times 0.05 \text{ mm}$
$Z = 4$	

Data collection

Siemens SMART CCD area-detector diffractometer	2134 independent reflections
ω scans	1100 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	$R_{\text{int}} = 0.080$
$T_{\text{min}} = 0.957$, $T_{\text{max}} = 0.994$	$\theta_{\text{max}} = 25.0^\circ$
5991 measured reflections	$h = -13 \rightarrow 15$
	$k = -12 \rightarrow 12$
	$l = -10 \rightarrow 10$

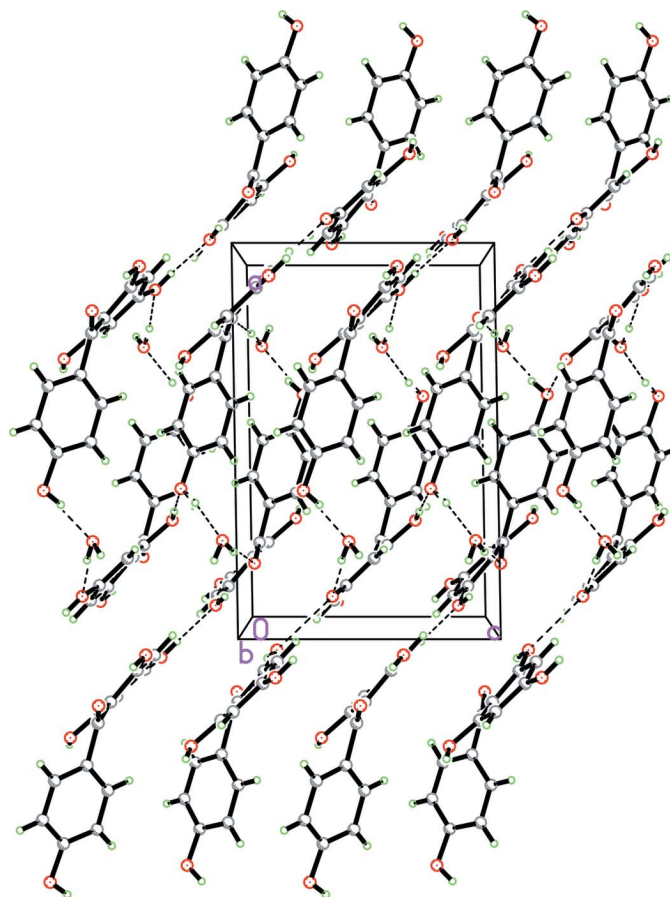


Figure 2
Molecular packing of (I), viewed along the b axis. Hydrogen bonds are shown as dashed lines.

Refinement

Refinement on F^2	H atoms treated by a mixture of independent and constrained refinement
$R[F^2 > 2\sigma(F^2)] = 0.067$	$wR(F^2) = 0.125$
$S = 1.03$	$w = 1/[\sigma^2(F_o^2) + (0.0342P)^2]$
2134 reflections	where $P = (F_o^2 + 2F_c^2)/3$
180 parameters	$(\Delta/\sigma)_{\text{max}} = 0.001$
	$\Delta\rho_{\text{max}} = 0.19 \text{ e \AA}^{-3}$
	$\Delta\rho_{\text{min}} = -0.19 \text{ e \AA}^{-3}$

Table 1
Hydrogen-bond geometry (\AA , $^\circ$).

$D\text{--}H\cdots A$	$D\text{--}H$	$H\cdots A$	$D\cdots A$	$D\text{--}H\cdots A$
O3—H3A···O2	0.82	1.79	2.529 (3)	149
O4—H4A···O3 ⁱ	0.82	1.94	2.724 (4)	160
O5—H5A···O1 ⁱⁱ	0.82	1.89	2.703 (3)	176
O1—H1A···O1W ⁱⁱⁱ	0.82	1.81	2.621 (5)	171
O1W—H1W1···O4 ^{iv}	0.87 (5)	2.00 (5)	2.848 (5)	167 (5)
O1W—H2W1···O2	0.90 (6)	1.87 (6)	2.751 (5)	166 (5)

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

H atoms of the water molecule were located in a difference map and refined isotropically. All other H atoms were positioned geometrically (C—H = 0.93 \AA and O—H = 0.82 \AA) and allowed to

ride on the parent atoms, with $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{C})$ for hydroxy H atoms and $1.2U_{\text{eq}}(\text{C})$ for others.

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1996); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 1997); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL* and *PLATON* (Spek, 2003).

The authors thank the Higher Education Commission, Government of Pakistan, and also the Malaysian Government and Universiti Sains Malaysia for the Scientific Advancement Grant Allocation (SAGA) grant No. 304/PFIZIK/635003/A118.

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