

**(5,6-Dihydroxy-2-methyl-1-benzofuran-3-yl)
phenyl ketone**Li-Xia Pei,^a Xian-Zhang Bu,^b
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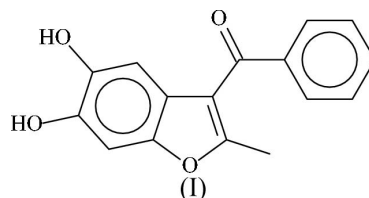
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The molecules of the title compound, C₁₆H₁₂O₄, are linked by
hydrogen bonds involving the two hydroxy and carbonyl O
atoms, forming a three-dimensional network.

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CommentThe synthesis of the anti-tumour compound tanshinone and its
analogues requires the use of polyphenolic benzo[*b*]furan
reagents (Aisa *et al.*, 2003), as does the synthesis of manso-
none F (Suh *et al.*, 2000). The title compound, (I) (Fig. 1) was
synthesized by condensing catechol and benzoylacetone in a
Michael synthesis for a biological screening trial.The benzofuran ring system of (I) is twisted by 26.9 (1)°
with respect to the ketonic (C7–C10–C11–O4) fragment,
and the phenyl ring is twisted by 35.7 (1)° with respect to it.
The molecules are linked by hydrogen bonds (Table 1) into a
three-dimensional network.**Key indicators**

Single-crystal X-ray study

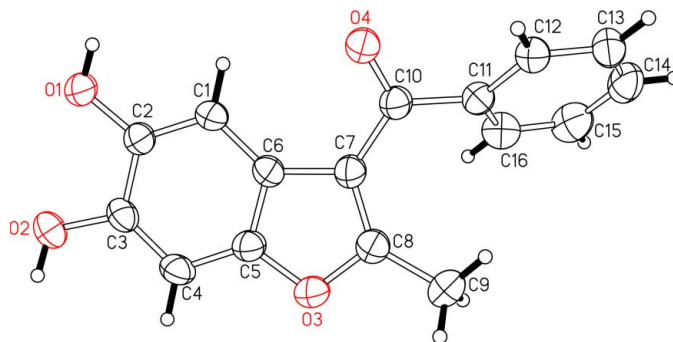
T = 295 K

Mean $\sigma(\text{C}-\text{C}) = 0.002 \text{ \AA}$

R factor = 0.037

wR factor = 0.114

Data-to-parameter ratio = 14.3

For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.**Experimental**To a solution of catechol (0.55 g, 5 mmol) in a mixture of ethanol and
water (9:1, 50 ml), benzoylacetone (0.81 g, 5 mmol) and pyridine
(0.40 g, 5 mmol) were added. Sodium iodate (1.19 g, 6 mmol) was
then added to the stirred solution. The progress of the reaction was
monitored by thin-layer chromatography. The solid product was
separated and the solution evaporated. The resulting material was**Figure 1**The molecular structure of (I). Displacement ellipsoids are drawn at the
50% probability level and H atoms are shown as spheres of arbitrary
radii.

extracted with ethyl acetate and the extract was washed with dilute hydrochloric acid, followed by distilled water, before being dried over sodium sulfate. Removal of the solvent gave the crude product, which was purified by column chromatography to give pure compound (**1**) in about 30% yield. Elemental analysis, calculated for $C_{16}H_{12}O_4$: C 71.64, H 4.51%; found: C 71.75, H 4.59%.

Crystal data

$C_{16}H_{12}O_4$
 $M_r = 268.26$
 Orthorhombic, *Pbca*
 $a = 6.7936$ (9) Å
 $b = 16.988$ (2) Å
 $c = 21.515$ (3) Å
 $V = 2483.0$ (6) Å³
 $Z = 8$
 $D_x = 1.435$ Mg m⁻³

Mo $K\alpha$ radiation
 Cell parameters from 909 reflections
 $\theta = 1.9$ – 27.1°
 $\mu = 0.10$ mm⁻¹
 $T = 295$ (2) K
 Block, colourless
 $0.52 \times 0.31 \times 0.25$ mm

Data collection

Bruker SMART area-detector diffractometer
 φ and ω scans
 Absorption correction: none
 13 857 measured reflections
 2725 independent reflections

2165 reflections with $I > 2\sigma(I)$
 $R_{int} = 0.020$
 $\theta_{max} = 27.1^\circ$
 $h = -8 \rightarrow 8$
 $k = -21 \rightarrow 20$
 $l = -24 \rightarrow 27$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.037$
 $wR(F^2) = 0.114$
 $S = 1.00$
 2725 reflections
 190 parameters
 H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0626P)^2 + 0.7622P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{max} = 0.001$
 $\Delta\rho_{max} = 0.22$ e Å⁻³
 $\Delta\rho_{min} = -0.18$ e Å⁻³
 Extinction correction: none

Table 1

Hydrogen-bond geometry (Å, °).

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
O1–H1 $o \cdots$ O4 ⁱ	0.86 (1)	1.86 (1)	2.711 (1)	177 (2)
O2–H2 $o \cdots$ O1 ⁱⁱ	0.85 (1)	2.11 (2)	2.817 (2)	141 (2)

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

The carbon-bound H atoms were placed in calculated positions (C–H = 0.93 Å for aromatic H atoms and C–H = 0.96 Å for methyl H atoms), and were included in the refinement in the riding-model approximation, with $U_{iso}(H) = 1.2U_{eq}(C)$ for aromatic H atoms and $1.5U_{eq}(C)$ for methyl H atoms. The oxygen-bound H atoms were refined with a distance restraint of 0.85 (1) Å.

Data collection: SMART (Bruker, 2001); cell refinement: SAINT (Bruker, 2001); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 1997); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: ORTEPII (Johnson, 1976); software used to prepare material for publication: SHELXL97.

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