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Bis(3-formyl-4-hydroxy-5-methoxyphenyl)-methane

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Key indicators

Single-crystal X-ray study
T = 296 K
Mean σ(C–C) = 0.003 Å
R factor = 0.046
wR factor = 0.109
Data-to-parameter ratio = 13.4

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

In the title compound, $C_{17}H_{16}O_3$, the asymmetric unit contains one half-molecule; a twofold rotation axis bisects the molecule. The structure is stabilized by O—H···N intramolecular hydrogen bonds and C—H···π and π–π intermolecular interactions.

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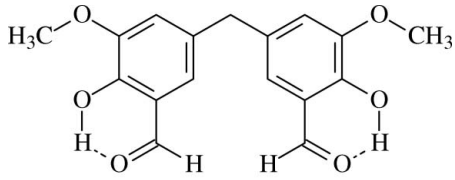
Comment

Hydroxy-substituted benzaldehyde reagents used for condensation with primary amines, hydrazines, hydroxylamine and other primary amine derivatives afford imine derivatives which can function as ligands towards a number of metal cations (Loudon, 2002; Khandar & Nejati, 2000; Khandar & Rezvani, 1999).



(I)

The asymmetric unit of the title compound, (I), contains one half-molecule; a twofold rotation axis passes through C9 (Fig. 1). The bond lengths and angles (Table 1) are in normal ranges (Allen *et al.*, 1987).

In (I), molecules have strong intramolecular O—H···O hydrogen bonds (Table 2) and they are linked through C8—H8*a*···*Cg*1 (*Cg*1 is the centroid of the C1–C6 ring) and *Cg*1···*Cg*1 intermolecular interactions (Fig. 2). For the C8—

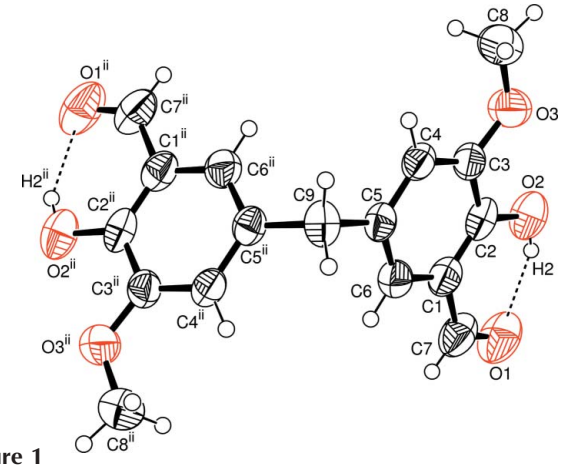


Figure 1
The molecular structure of (I), with the atomic numbering scheme. Displacement ellipsoids are drawn at the 50% probability level. The intramolecular hydrogen bonds are shown as dashed lines. [Symmetry code: (i) $1-x, y, \frac{1}{2}-z$.]

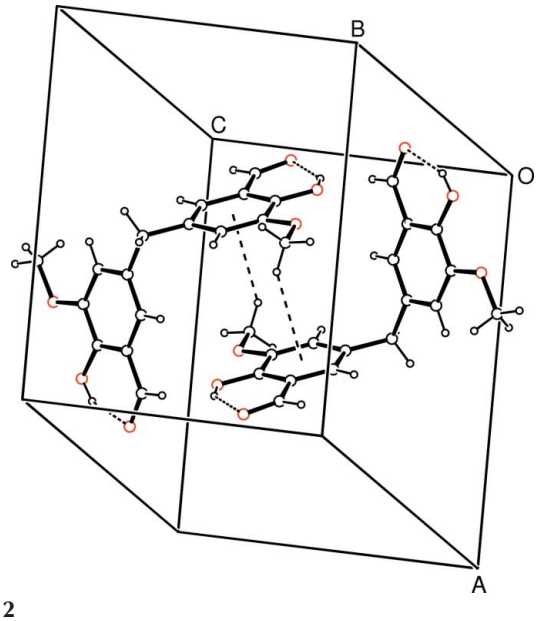


Figure 2
A packing diagram of (I) with the C—H···π intermolecular interactions shown as dashed lines.

H8*a*···*Cg*1 contact, the distance between atom H8*a* and the aromatic ring centroid is 3.01 (3) Å (symmetry code: 1 − *x*, 1 − *y*, −*z*). There is also π–π stacking between adjacent molecules at (*x*, *y*, *z*) and (−*x*, *y*, $\frac{1}{2}$ − *z*), with distances of 3.608 (16) Å between the rings centroids and perpendicular distances of 2.481 (16) Å between the rings.

Experimental

A mixture of *o*-vanillin (0.1 mol) and formaldehyde (0.1 mol) was stirred at 393 K for 2 h. The solution was added to boiling ethyl alcohol and stirred at 393 K for 20 min and cooled to room temperature. The precipitate was filtered off and recrystallized from ethyl alcohol by slow evaporation (yield 1.58 g, 10%, m.p. 424–426 K).

Crystal data

$C_{17}H_{16}O_6$
M_r = 316.30
Monoclinic, $C2/c$
a = 14.960 (3) Å
b = 8.2889 (11) Å
c = 13.249 (3) Å
β = 115.015 (15)°
V = 1488.8 (5) Å^3
Z = 4
D_x = 1.411 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 3016 reflections
θ = 2.9–27.5°
μ = 0.11 mm^{-1}
T = 296 (2) K
Plate, yellow
0.40 × 0.24 × 0.07 mm

Data collection

Stoe IPDS-II diffractometer
ω scans
Absorption correction: integration (*X-RED32*; Stoe & Cie, 2002)
T_{min} = 0.963, T_{max} = 0.993
6042 measured reflections
1470 independent reflections
828 reflections with $I > 2\sigma(I)$
R_{int} = 0.065
θ_{max} = 26.0°
h = −18 → 18
k = −10 → 10
l = −16 → 16

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.046
$wR(F^2)$ = 0.109
S = 1.00
1470 reflections
110 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.049P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.12 e Å^{-3}
$\Delta\rho_{min}$ = −0.11 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

C1—C2	1.388 (3)	C3—C4	1.381 (3)
C1—C6	1.399 (3)	C4—C5	1.400 (3)
C2—O2	1.360 (2)	C5—C6	1.368 (3)
C2—C3	1.395 (3)	C7—O1	1.219 (3)
C2—C1—C7	120.9 (2)	O3—C3—C4	124.8 (2)
O2—C2—C3	118.0 (2)	C5—C9—C5^{i}	113.6 (3)
C6—C1—C7—O1	−176.1 (2)	C4—C5—C9—C5^{i}	108.23 (19)
C6—C5—C9—C5^{i}	−72.25 (18)	C4—C3—O3—C8	−1.6 (3)

Symmetry code: (i) $-x+1, y, -z+\frac{1}{2}$.

Table 2
Hydrogen-bond geometry (Å, °).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O2—H2···O1	0.82	1.94	2.648 (3)	145

Atom H9, attached to C9, was located in a difference map and refined isotropically [C—H = 0.98 (2) Å]. The remaining H atoms were positioned geometrically [0.82 (OH), 0.93 (CH) and 0.96 Å (CH_3)] and constrained to ride on their parent atoms with U_{iso}(H) values of 1.5 (1.2 for methine) times U_{eq}(C,O).

Data collection: *X-AREA* (Stoe & Cie, 2002); cell refinement: *X-AREA*; data reduction: *X-RED32*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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