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6,6′-Dihydroxy-5,5′-dimethoxy-3,3′-methylenedibenzaldehyde

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Key indicators

Single-crystal X-ray study
T = 294 K
Mean σ(C–C) = 0.003 Å
R factor = 0.040
wR factor = 0.125
Data-to-parameter ratio = 14.4

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

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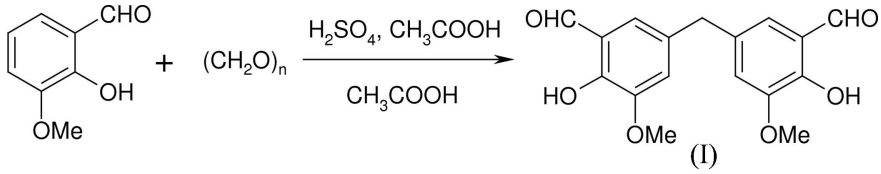
In the title compound, $C_{17}H_{16}O_6$, the asymmetric unit contains one half-molecule. A twofold rotation axis passes through the C atom linking the two rings. Intramolecular O—H···O hydrogen bonds seem to be effective in stabilizing the molecular structure.

Comment

Dinuclear ligands are useful in the preparation of dinuclear complexes which are excellent asymmetric catalysts owing to their large molecular weights (Wei & Atwood, 1997; Janssen *et al.*, 1997) and several active sites.



(I)

The title compound, (I), was synthesized according to reported methods with little modification (Marvel & Tarköy, 1957; Sun & Tang, 2004). In our case, it was synthesized according to the reported methods with little modification.

The asymmetric unit contains only one half-molecule. A twofold rotation axis passes through atom C1. Intramolecular O—H···O hydrogen bonds (Fig. 1 and Table 1) seem to be effective in stabilizing the molecular structure.

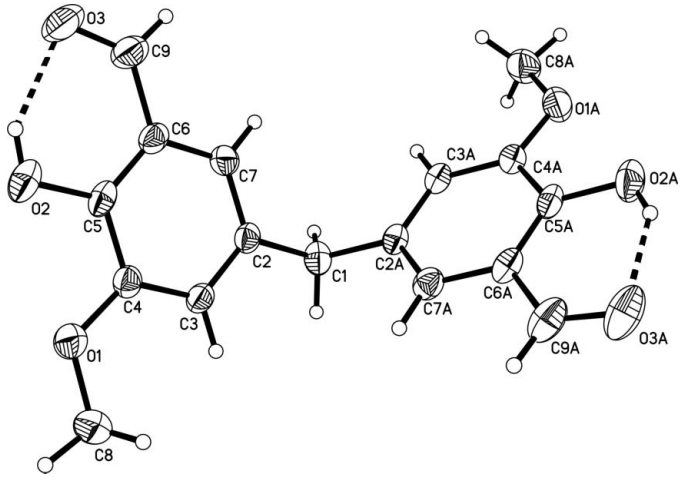


Figure 1
The molecular structure of (I), with the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level. Dashed lines indicate intramolecular O-H···O hydrogen bonds. [Symmetry code: (A) $-x, y, -z+\frac{3}{2}$.]

Experimental

The title compound was synthesized from the reaction of 3-methoxysalicylaldehyde (15.2 g, 100 mmol) and paraformaldehyde (4.5 g, 50 mmol) in glacial acetic acid (18 ml) for 24h at 363-368 K with concentrated sulfuric acid (0.5 ml) as catalyst. The compound was separated by column chromatography on silica-gel eluted with ethyl acetate–hexane (4:1). Pale yellow crystals suitable for X-ray analysis were obtained by crystallization at 298K from ethyl acetate (yield 3.7 g, 23%, m.p. 414-415K).

Crystal data

$C_{17}H_{16}O_6$
M_r = 316.30
Monoclinic, $C2/c$
a = 14.940 (4) Å
b = 8.262 (2) Å
c = 13.236 (4) Å
β = 114.816 (4)°
V = 1483.0 (7) Å^3
Z = 4
D_x = 1.417 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 1065 reflections
θ = 2.9–26.2°
μ = 0.11 mm^{-1}
T = 294 (2) K
Block, yellow
0.30 × 0.18 × 0.12 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.942, T_{max} = 0.987
4053 measured reflections
1541 independent reflections
902 reflections with $I > 2\sigma(I)$
R_{int} = 0.031
θ_{max} = 26.6°
$h = -18 \rightarrow 13$
$k = -10 \rightarrow 8$
$l = -12 \rightarrow 16$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.040
$wR(F^2)$ = 0.125
S = 1.00
1541 reflections
107 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0608P)^2 + 0.326P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.15 e Å^{-3}
$\Delta\rho_{min}$ = −0.17 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O2—H2···O3	0.82	1.93	2.645 (3)	145

The H atoms were positioned geometrically [0.82 (OH), 0.93 and 0.97 (CH) and 0.96 Å (CH_3)] and constrained to ride on their parent atoms, with U_{iso}(H) = 1.2U_{eq}(C) [1.5U_{eq}(C,O) for methyl and hydroxyl H atoms].

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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