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4-Hydroxy-2,6-dimethoxybenzaldehyde

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.004 Å
R factor = 0.054
wR factor = 0.120
Data-to-parameter ratio = 9.8

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

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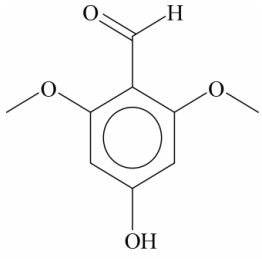
The structure of the title compound, $C_9H_{10}O_4$, is presented and compared with 4-hydroxy-3,5-dimethoxybenzaldehyde. Both form infinite hydrogen-bonded chains.

Comment

In the course of our research on functionalized PPV oligomers with specific optical and electronic characteristics, we have been examining benzaldehydes that can be used as precursor materials. In this context, we have synthesized the title compound, (I) (Fig. 1). As the molecular structure of 4-hydroxy-3,5-dimethoxybenzaldehyde, (II), was published recently by Kolev *et al.* (2004), this contribution presents a comparison of structural parameters and intermolecular contacts.



(I)

The bond lengths and angles in (I) contain no surprises. The molecules in (I) are all oriented in parallel layers, perpendicular to the glide planes and parallel to the screw axes. In contrast, the molecules of (II) have a different orientation with respect to the symmetry elements, and this generates a substantially different packing in the unit cell. Yet, the volume of the unit cell of (I) is nearly identical to that of (II). The particular placement of the methoxy groups in (I) results in the following intramolecular interactions involving the aldehyde function: O6···O21 = 2.704 (4) Å and O2···H21 = 2.19 (3) Å.

As in (II), the hydroxy group displays an intermolecular O4—H4···O21^{i} bridge [1.76 (4) Å and 168 (4)°; symmetry code: (i) $-x, y-\frac{1}{2}, \frac{3}{2}-z$] linking the molecules into infinite chains, here in the [010] direction. This interaction is indicated in Fig. 2. In (I), the hydroxy group also displays a contact between H3 and the the lone pairs of O4, characterized by C3—H3···O4^{ii} [2.54 (3) Å, 171 (2)°; symmetry code: (ii) $1-x, -y, 1-z$].

Another interesting interaction, often evident in 2,4,6-trimethoxy-substituted benzenes (Vande Velde *et al.*, 2004), is the OCH_3–π contact C26—H26*c*···*Cg*1^{iii} [3.06 (3) Å and 150 (2)°; symmetry code: (iii) $-x, -y, 2-z$; where *Cg*1

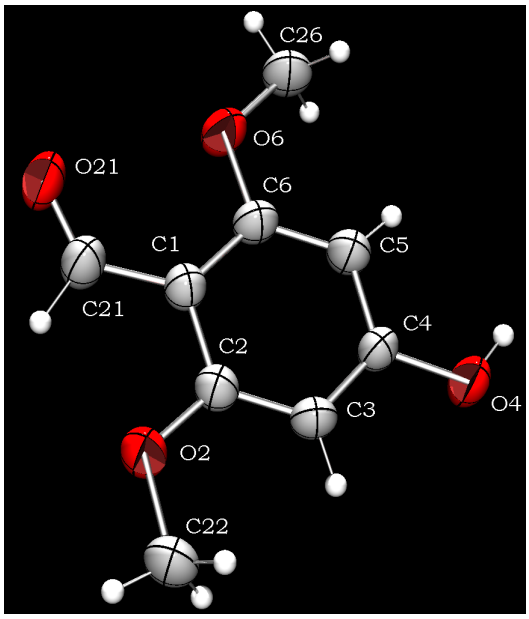


Figure 1
The structure and numbering scheme of (I). Displacement ellipsoids are drawn at the 50% probability level.

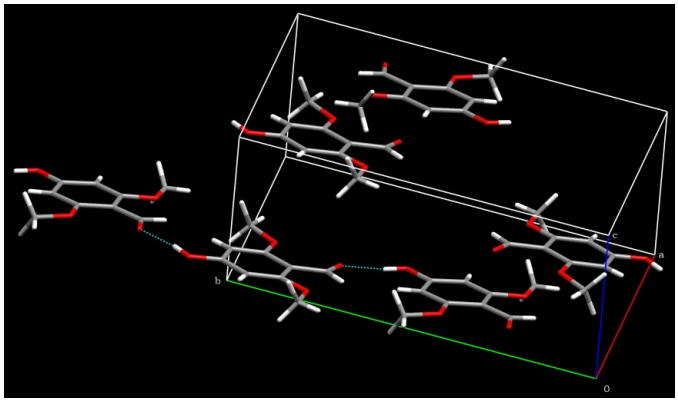


Figure 2
Unit-cell diagram of (I); hydrogen bonds are represented by dotted lines.

indicates the centroid of the ring]. This interaction is also evidenced by the distance of 2.88 (3) Å between H26*c* and C5^{iii}.

The list of interactions is completed by two more C—H···*n*(O) contacts which are barely below the sum of the van der Waals radii, namely O2···H22*a*^{iv} [2.70 (3) Å; symmetry code: (iv) $x, \frac{1}{2}-y, \frac{1}{2}+z$] and H22*c*···O21^{v} [2.71 (3) Å; symmetry code: (v) $1+x, y, z$].

Experimental

All starting materials were obtained from Acros or Aldrich and used as received. The reaction pathway as described by Albericio *et al.* (1990) was followed and the resulting product was recrystallized from ethanol, which yielded crystals suitable for the diffraction experiment.

Crystal data

$C_9H_{10}O_4$
M_r = 182.17
Monoclinic, $P2_1/c$
a = 7.518 (3) Å
b = 14.801 (10) Å
c = 7.678 (6) Å
β = 91.91 (5)°
V = 853.9 (9) Å^3
Z = 4
D_x = 1.417 Mg m^{-3}
Cell parameters from 25 reflections
θ = 6.7–18.5°
μ = 0.11 mm^{-1}
T = 293 (2) K
Prism, yellow–orange
0.36 × 0.32 × 0.26 mm

Data collection

Enraf–Nonius MACH3 diffractometer
$\omega/2\theta$ scans
3263 measured reflections
1548 independent reflections
748 reflections with $I > 2\sigma(I)$
R_{int} = 0.144
θ_{max} = 25.3°
$h = -9 \rightarrow 0$
$k = -17 \rightarrow 17$
$l = -9 \rightarrow 9$
3 standard reflections
frequency: 60 min
intensity decay: −2%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.054
$wR(F^2)$ = 0.120
S = 0.97
1548 reflections
158 parameters
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.0361P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.23 e Å^{-3}
$\Delta\rho_{min}$ = −0.27 e Å^{-3}

All H atoms were refined independently, with isotropic displacement parameters, giving C—H distances in the range 0.86 (3)–1.03 (3) Å and an O—H distance of 0.89 (4) Å. The R_{int} value of 0.144 is mainly due to weak diffraction from the crystal; a large part of the reflections are weak and these contribute to a great extent to the value of R_{int}.

Data collection: *CAD*-4 *EXPRESS* (Enraf–Nonius, 1994); cell refinement: *CAD*-4 *EXPRESS*; data reduction: *XCAD*4 (Harms & Wocadlo, 1995); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*-3 *for Windows* (Farrugia, 1997) and *MERCURY* (Version 1.2.1; Bruno *et al.*, 2002); software used to prepare material for publication: *WinGX* (Version 1.64; Farrugia, 1999) and *PLATON* (Spek, 2003).

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