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3,4-Dimethoxybenzaldehyde

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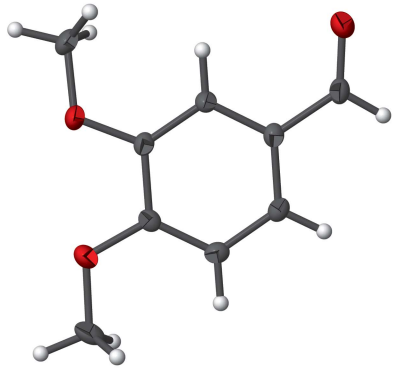
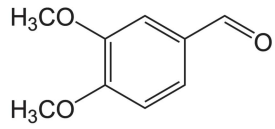
Structural data: full structural data are available from iucrdata.iucr.org

In the title compound, $C_9H_{10}O_3$, one of the methoxy C atoms deviates from the plane of the aromatic ring by 0.337 (2) Å. Crystallization was hindered by oiling out in various solvents. The crystal contains neither hydrogen bonds nor aromatic π–π stacking.

3D view



Chemical scheme



Structure description

The title compound, shown in Fig. 1, has a strong vanilla fragrance. It crystallizes in the orthorhombic space group $Pna2_1$. Differential scanning calorimetry measurements were performed to screen for polymorphic transitions, but none were observed between 150 K and the melting point of the title compound, 319 K (not shown).

The compound oils out in water and several organic solvents. Similar behaviour has been observed for the closely related molecule vanillin (Svärd *et al.*, 2007). For the crystal structure of vanillin-I, see: Velavan *et al.* (1995).

In the crystal, no hydrogen bonds are present.

Synthesis and crystallization

Commercial 3,4-dimethoxybenzaldehyde (99% pure, Aldrich) was used for the crystallization. A few crystals of the commercial powder were added to an aqueous saturated solution of 3,4-dimethoxybenzaldehyde at room temperature. Subsequently, the temperature was cycled between 298 and 303 K. After 2 weeks colourless needles were grown, suitable for single-crystal X-ray diffraction.

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 1.

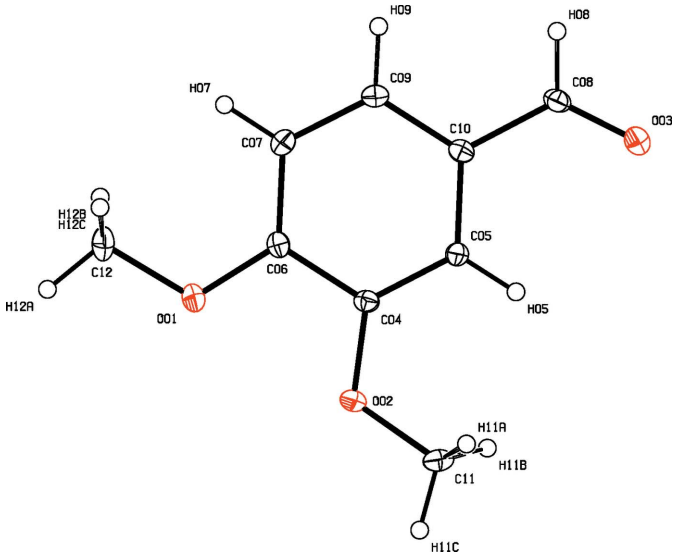


Figure 1
The molecular structure of the title compound, showing displacement ellipsoids drawn at the 30% probability level.

Table 1
Experimental details.

Crystal data	
Chemical formula	$C_9H_{10}O_3$
M_r	166.17
Crystal system, space group	Orthorhombic, $Pna2_1$
Temperature (K)	150
a, b, c (Å)	11.374 (2), 14.363 (3), 5.050 (2)
V (Å^3)	825.0 (4)
Z	4
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.10
Colour	Colourless
Crystal size (mm)	0.35 × 0.15 × 0.13
Data collection	
Diffractometer	Bruker D8 Quest APEX3
Absorption correction	Multi-scan (*SADABS*; Bruker, 2001)
T_{min}, T_{max}	0.90, 0.99
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	34976, 2608, 2237
R_{int}	0.050
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.035, 0.097, 1.03
No. of reflections	2608
No. of parameters	111
No. of restraints	1
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.34, −0.19
Absolute structure	Flack x determined using 930 quotients $[(I^+)-(I^-)]/[(I^+)+(I^-)]$ (Parsons *et al.*, 2013)
Absolute structure parameter	0.2 (3)

Computer programs: *APEX3* and *SAINT* (Bruker, 2012), *PEAKREF* (Schreurs, 2013), *SHELXT2014* (Sheldrick, 2015*a*), *SHELXL2014* (Sheldrick, 2015*b*), *PLATON* (Spek, 2009) and *ShelXLe* (Sheldrick, 2015*a*).

References

Bruker (2001). *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.

Bruker (2012). *APEX3* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.

Parsons, S., Flack, H. D. & Wagner, T. (2013). *Acta Cryst.* B**69**, 249–259.

Schreurs, A. M. M. (2013). *PEAKREF*. Utrecht University, The Netherlands.

Sheldrick, G. M. (2015*a*). *Acta Cryst.* A**71**, 3–8.

Sheldrick, G. M. (2015*b*). *Acta Cryst.* C**71**, 3–8.

Spek, A. L. (2009). *Acta Cryst.* D**65**, 148–155.

Svärd, M., Gracin, S. & Rasmuson, A. C. (2007). *J. Pharm. Sci.* **96**, 2390–2398.

Velavan, R., Sureshkumar, P., Sivakumar, K. & Natarajan, S. (1995). *Acta Cryst.* C**51**, 1131–1133.

full crystallographic data

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3,4-Dimethoxybenzaldehyde

Crystal data

$C_9H_{10}O_3$
M_r = 166.17
Orthorhombic, $Pna2_1$
a = 11.374 (2) Å
b = 14.363 (3) Å
c = 5.050 (2) Å
V = 825.0 (4) Å^3
Z = 4
F(000) = 352
D_x = 1.338 Mg m^{-3}
Melting point: 319 K
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 2237 reflections
θ = 2.2–30°
μ = 0.10 mm^{-1}
T = 150 K
Needle, colourless
0.35 × 0.15 × 0.13 mm

Data collection

Bruker D8 Quest APEX3 diffractometer
Radiation source: sealed tube
Graphite monochromator
Detector resolution: 10.4 pixels mm^{-1}
φ and ω scans
Absorption correction: multi-scan (SADABS; Bruker, 2001)
T_{min} = 0.90, T_{max} = 0.99
34976 measured reflections
2608 independent reflections
2237 reflections with $I > 2\sigma(I)$
R_{int} = 0.050
θ_{max} = 31.0°, θ_{min} = 2.3°
h = −16→15
k = −20→20
l = −7→7

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.035
$wR(F^2)$ = 0.097
S = 1.03
2608 reflections
111 parameters
1 restraint
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0572P)^2 + 0.1158P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.34 e Å^{-3}
$\Delta\rho_{min}$ = −0.19 e Å^{-3}
Absolute structure: Flack x determined using 930 quotients $[(I^+)-(I^-)]/[(I^+)+(I^-)]$ (Parsons *et al.*, 2013)
Absolute structure parameter: 0.2 (3)

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
O01	0.62471 (10)	0.74604 (8)	0.1630 (3)	0.0242 (3)
O02	0.51301 (9)	0.63612 (8)	0.4799 (3)	0.0244 (3)
O03	0.84277 (11)	0.46485 (8)	1.0130 (3)	0.0286 (3)
C04	0.63269 (12)	0.63367 (10)	0.5011 (4)	0.0177 (3)
C05	0.69492 (12)	0.57807 (10)	0.6762 (4)	0.0182 (3)
H05	0.6543	0.5369	0.7914	0.022*
C06	0.69401 (13)	0.69467 (10)	0.3273 (4)	0.0179 (3)
C07	0.81632 (13)	0.69977 (10)	0.3360 (4)	0.0200 (3)
H07	0.8574	0.7408	0.2212	0.024*
C08	0.88522 (14)	0.52368 (11)	0.8669 (4)	0.0219 (3)
H08	0.9681	0.5319	0.8726	0.026*
C09	0.87817 (13)	0.64383 (10)	0.5155 (4)	0.0198 (3)
H09	0.9615	0.6475	0.5233	0.024*
C10	0.81847 (12)	0.58287 (11)	0.6827 (4)	0.0181 (3)
C11	0.44789 (14)	0.59171 (13)	0.6874 (4)	0.0263 (3)
H11A	0.4698	0.6191	0.8582	0.040*
H11B	0.4658	0.5250	0.6888	0.040*
H11C	0.3635	0.6007	0.6572	0.040*
C12	0.68316 (17)	0.80898 (11)	−0.0137 (4)	0.0281 (4)
H12A	0.6248	0.8402	−0.1256	0.042*
H12B	0.7382	0.7742	−0.1254	0.042*
H12C	0.7264	0.8556	0.0893	0.042*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O01	0.0249 (6)	0.0236 (5)	0.0242 (6)	0.0029 (4)	−0.0010 (5)	0.0068 (5)
O02	0.0146 (5)	0.0269 (5)	0.0318 (6)	0.0019 (4)	−0.0009 (5)	0.0074 (5)
O03	0.0247 (6)	0.0284 (6)	0.0328 (7)	0.0016 (5)	−0.0036 (6)	0.0090 (6)
C04	0.0151 (6)	0.0163 (6)	0.0217 (7)	0.0005 (5)	−0.0003 (7)	−0.0014 (6)
C05	0.0170 (6)	0.0161 (6)	0.0215 (7)	−0.0003 (5)	0.0000 (7)	0.0020 (6)
C06	0.0214 (7)	0.0157 (6)	0.0166 (6)	0.0012 (5)	−0.0013 (6)	−0.0006 (6)
C07	0.0215 (7)	0.0192 (7)	0.0193 (7)	−0.0016 (6)	0.0031 (7)	0.0001 (6)
C08	0.0165 (7)	0.0239 (7)	0.0254 (8)	0.0014 (6)	−0.0036 (6)	0.0006 (7)
C09	0.0155 (6)	0.0219 (7)	0.0222 (8)	−0.0014 (5)	0.0001 (6)	−0.0016 (6)
C10	0.0166 (6)	0.0182 (6)	0.0194 (7)	0.0016 (5)	−0.0013 (7)	−0.0022 (6)
C11	0.0153 (6)	0.0316 (8)	0.0321 (8)	−0.0009 (6)	0.0032 (7)	0.0016 (7)
C12	0.0385 (9)	0.0213 (7)	0.0244 (8)	0.0026 (6)	0.0012 (8)	0.0075 (7)

Geometric parameters (Å, º)

O01—C06	1.361 (2)	C07—H07	0.9500
O01—C12	1.434 (2)	C08—C10	1.471 (2)
O02—C04	1.3659 (17)	C08—H08	0.9500
O02—C11	1.433 (2)	C09—C10	1.393 (2)
O03—C08	1.221 (2)	C09—H09	0.9500
C04—C05	1.386 (2)	C11—H11A	0.9800
C04—C06	1.423 (2)	C11—H11B	0.9800
C05—C10	1.4073 (19)	C11—H11C	0.9800
C05—H05	0.9500	C12—H12A	0.9800
C06—C07	1.394 (2)	C12—H12B	0.9800
C07—C09	1.401 (3)	C12—H12C	0.9800
C06—O01—C12	116.90 (14)	C10—C09—H09	119.7
C04—O02—C11	116.51 (14)	C07—C09—H09	119.7
O02—C04—C05	125.02 (15)	C09—C10—C05	120.23 (15)
O02—C04—C06	115.10 (14)	C09—C10—C08	119.66 (13)
C05—C04—C06	119.88 (12)	C05—C10—C08	120.11 (15)
C04—C05—C10	119.78 (14)	O02—C11—H11A	109.5
C04—C05—H05	120.1	O02—C11—H11B	109.5
C10—C05—H05	120.1	H11A—C11—H11B	109.5
O01—C06—C07	124.66 (14)	O02—C11—H11C	109.5
O01—C06—C04	115.20 (13)	H11A—C11—H11C	109.5
C07—C06—C04	120.12 (14)	H11B—C11—H11C	109.5
C06—C07—C09	119.45 (15)	O01—C12—H12A	109.5
C06—C07—H07	120.3	O01—C12—H12B	109.5
C09—C07—H07	120.3	H12A—C12—H12B	109.5
O03—C08—C10	125.30 (15)	O01—C12—H12C	109.5
O03—C08—H08	117.4	H12A—C12—H12C	109.5
C10—C08—H08	117.4	H12B—C12—H12C	109.5
C10—C09—C07	120.53 (14)		
C11—O02—C04—C05	12.6 (2)	O01—C06—C07—C09	179.45 (17)
C11—O02—C04—C06	−166.89 (15)	C04—C06—C07—C09	0.6 (2)
O02—C04—C05—C10	−178.91 (16)	C06—C07—C09—C10	0.6 (2)
C06—C04—C05—C10	0.6 (2)	C07—C09—C10—C05	−1.1 (2)
C12—O01—C06—C07	0.5 (2)	C07—C09—C10—C08	179.07 (15)
C12—O01—C06—C04	179.47 (14)	C04—C05—C10—C09	0.6 (2)
O02—C04—C06—O01	−0.6 (2)	C04—C05—C10—C08	−179.65 (16)
C05—C04—C06—O01	179.87 (14)	O03—C08—C10—C09	−176.38 (16)
O02—C04—C06—C07	178.39 (14)	O03—C08—C10—C05	3.8 (3)
C05—C04—C06—C07	−1.1 (2)		