research communications

The crystal structures of four dimethoxy-benzaldehyde isomers

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The crystal structures of four dimethoxybenzaldehyde ($C_9H_{10}O_3$) isomers, namely the 2,3-, 2,4-, 2,5- and 3,5- isomers, are reported and compared to the previously reported crystal structures of 3,4-dimethoxybenzaldehyde and 2,6-dimethoxybenzaldehyde. All dimethoxybenzaldehyde molecules in the crystal structures are nearly planar. The largest deviation (1.2 Å) from the aromatic plane is found for one of the methoxy groups of 2,3-dimethoxy-benzaldehyde. Upon rapid cooling of 3,4-dimethoxybenzaldehyde and 3,5-dimethoxybenzaldehyde, a metastable polymorph is formed. The crystal studied for the 3,5- isomer was refined as a two-component twin.

1. Chemical context

Dimethoxybenzaldehydes (DMBz) are often used as starting materials in condensation reactions forming Schiff base compounds. Schiff base compounds are versatile ligands in numerous metal–organic complexes that are used as a catalyst. Examples include C—O coupling reactions (Maity *et al.*, 2015), the Suzuiki–Miyaura reaction (Das & Linert, 2016), nitroaldol reactions (Handa *et al.*, 2008) and a wide variety of other reactions (Gupta & Sutar, 2008).
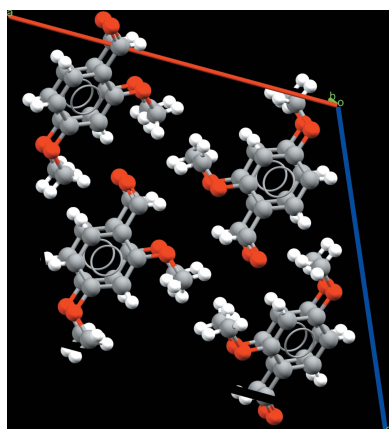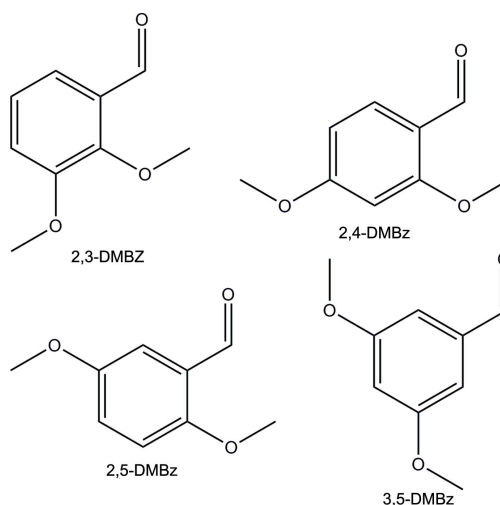
Whereas the crystal structures of nearly 100 DMBz derivatives have been published, not all of the crystal structures of the DMBz starting compounds are known. Only the crystal structures of 3,4-DMBz (de Ronde *et al.*, 2016) and 2,6-DMBz (Lemercier *et al.*, 2014) have been reported. In this work, we report the structures of the four other dimethoxy-benzaldehyde isomers, namely 2,3-DMBz (Fig. 1), 2,4-DMBz (Fig. 2), 2,5-DMBz (Fig. 3) and 3,5-DMBz (Fig. 4).

Table 1
Deviation from the aromatic plane (in Å).

	2,3-DMBz	2,4-DMBz	2,5-DMBz	2,6-DMBz (CSD refcode: LIZLAJ)	3,4-DMBz (CSD refcode: IQUGUY)	3,5-DMBz (molecule 1)	3,5-DMBz (molecule 2)
Aldehyde C	0.020	0.060	0.004	0.027	0.020	0.027	0.022
Aldehyde O	0.104	0.089	0.113	0.015	0.095	0.019	0.047
Methoxy 1 O	0.048	0.013	0.033	0.011	0.002	0.009	0.015
Methoxy 1 C	1.200	0.122	0.099	0.017	0.001	0.087	0.258
Methoxy 2 O	0.035	0.019	0.025	0.024	0.033	0.013	0.019
Methoxy 2 C	0.013	0.074	0.109	0.040	0.337	0.020	0.109

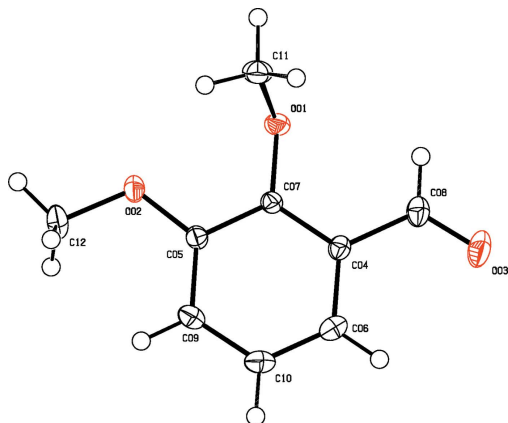Methoxy 1 and 2 are defined in the same order as the atomic labels, as shown in Fig. 4.

Figure 1
The molecular structure of 2,3-DMBz, showing displacement ellipsoids drawn at the 50% probability level.
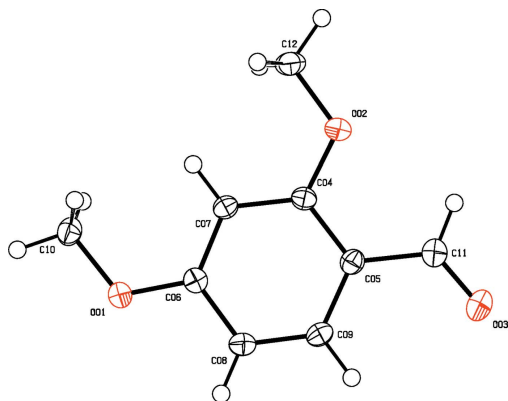

Figure 2
The molecular structure of 2,4-DMBz, showing displacement ellipsoids drawn at the 50% probability level.
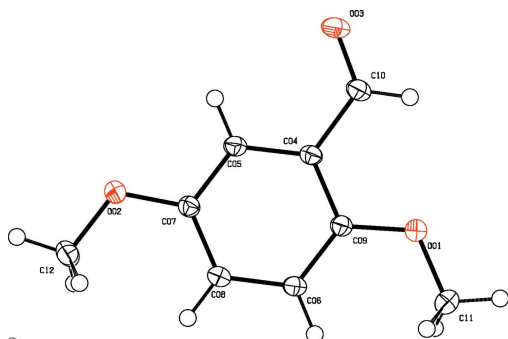

Figure 3
The molecular structure of 2,5-DMBz, showing displacement ellipsoids drawn at the 50% probability level.

2. Structural commentary

All four reported isomers crystallize in the monoclinic space group $P2_1/c$, which is also the case for the previously reported 2,6-DMBz (Lemercier *et al.*, 2014). On the other hand, 3,4-DMBz was reported to crystallize in space group $Pna2_1$ (de Ronde *et al.*, 2016). 3,5-DMBz has two molecules in the asymmetric unit, while the other crystal structures have one molecule in the asymmetric unit. The DMBz molecules in the crystal structures are almost planar (Table 1). The biggest deviation is found in the 2,3-DMBz in which one of the methoxy groups deviates by 1.2 Å from the aromatic plane.

3. Supramolecular features

In the crystal structure of 2,3-DMBz, one of the methoxy groups lies in the plane of the aromatic ring (see Fig. 5). The second methoxy group points towards the aldehyde group of a neighboring 2,3-DMBz molecule. In the crystal structure of 2,4-DMBz, shown in Fig. 6, π–π stacking interactions between the aromatic rings are present along the *b*-axis direction [centroid–centroid separation = 3.9638 (2) Å]. Similarly, in the crystal structure of 2,5-DMBz, aromatic π–π stacking interactions are present along the *a*-axis direction [centroid–centroid separation = 3.8780 (3) Å], as shown in Fig. 7. The crystal structures of 2,6-DMBz (Lemercier *et al.*, 2014), 3,4-DMBz (de Ronde *et al.*, 2016) and 3,5-DMBz do not exhibit aromatic π–π stacking interactions. As mentioned
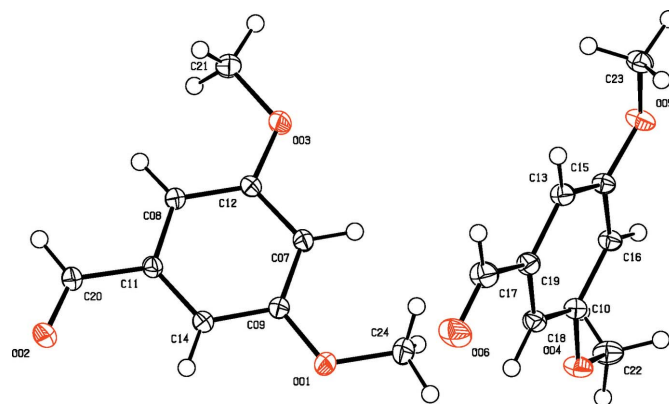

Figure 4
The molecular structure of 3,5-DMBz, showing displacement ellipsoids drawn at the 50% probability level.

Table 2
Melting point (in K) of DMBz as determined using the onset temperature of differential scanning calorimetry.

	2,3-DMBz	2,4-DMBz	2,5-DMBz	2,6-DMBz	3,4-DMBz	3,5-DMBz
Polymorph I (stable form)	322	341	321	368	317	319
Polymorph II					*	310

* Melting point could not be determined using differential scanning calorimetry.

above, only 3,5-DMBz has two molecules in the asymmetric unit, whereas the other crystal structures have one molecule in the asymmetric unit.
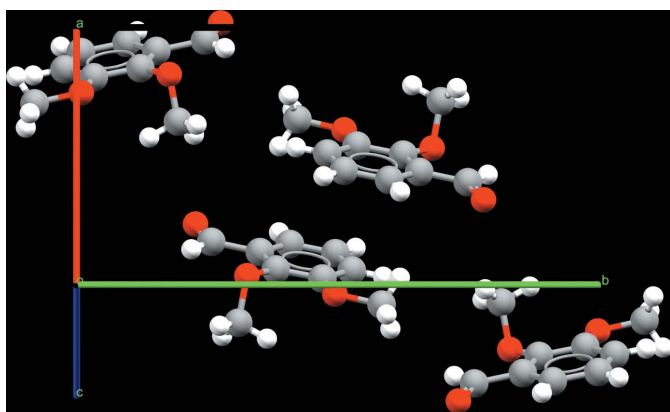

Figure 5
Crystal structure of 2,3-DMBz showing the orientation of the methoxy groups. One of the methoxy groups lies in the plane of the aromatic ring. The second methoxy group points towards the aldehyde group of a neighbouring 2,3-DMBz molecule.

Figure 6
A view along the *b* axis of the crystal structure of 2,4-DMBz, in which π–π stacking interactions between the aromatic rings are present.

4. Polymorphism

Polymorph screening using differential scanning calorimetry did not reveal any phase transitions for any DMBz between 133 K and the melting point of the compound (Table 2). On the other hand, a metastable polymorphic form was discovered after rapidly cooling from the melt for both 3,4-DMBz for which the crystal structure was reported previously (de Ronde *et al.* 2016) and 3,5-DMBz. In the course of hours, these polymorphic forms transformed into the stable forms. Powder X-ray diffraction measurements confirmed the existence of these metastable forms (3,4-DMBz: Figs. 8, 3, 5-DMBz: Fig. 9).

5. Database survey

A search in the Cambridge Structural Database (Version 5.39, update February 2018, Groom *et al.*, 2016) for dimethoxy-benzaldehydes derivatives yielded the crystal structure of 93 compounds, which can be subdivided into fourteen 2,3-DMBz derivatives (including two solvates), fifteen 2,4-DMBz derivatives (including four solvates), ten 2,5-DMBz derivatives (including two solvates), nine 2,6-DMBz derivatives (including one solvate), forty two 3,4-DMBz derivatives (including nine solvates) and three 3,5-DMBz derivatives.
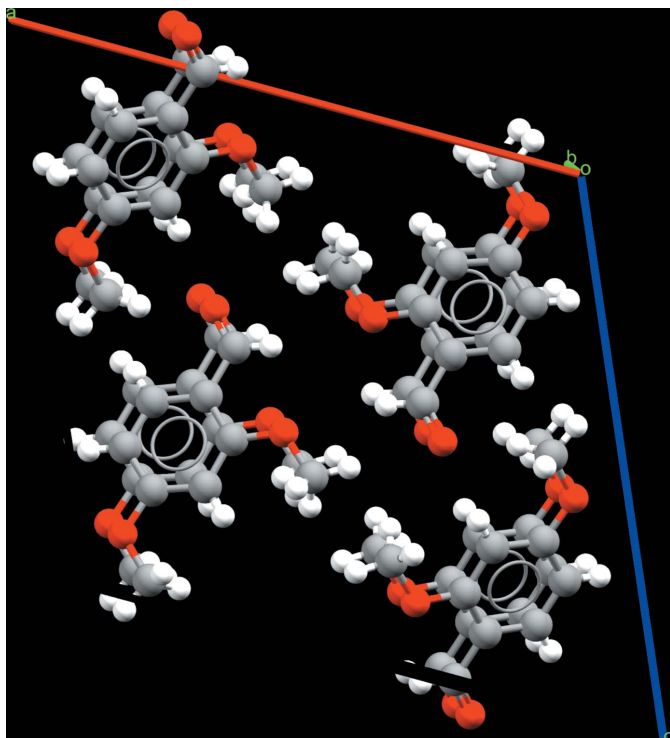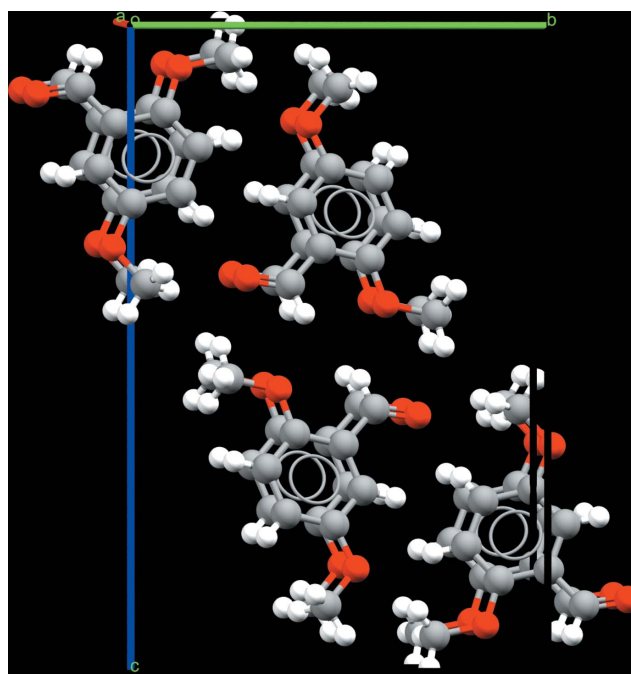

Figure 7
A view along the *a* axis of the crystal structure of 2,5-DMBz, in which π–π stacking interactions between the aromatic rings are present.

Table 3
Experimental details.

	2,3DMBz	2,4DMBz	2,5DMBz	3,5DMBz
Crystal data				
Chemical formula	$C_9H_{10}O_3$	$C_9H_{10}O_3$	$C_9H_{10}O_3$	$C_9H_{10}O_3$
M_r	166.17	166.17	166.17	166.17
Crystal system, space group	Monoclinic, $P2_1/c$	Monoclinic, $P2_1/c$	Monoclinic, $P2_1/n$	Monoclinic, $P2_1/c$
Temperature (K)	150	150	150	150
a, b, c (Å)	7.6152 (3), 15.5513 (6), 7.5891 (3)	15.1575 (8), 3.9638 (2), 14.6181 (8)	3.8780 (3), 11.5513 (7), 17.8153 (12)	11.7602 (5), 13.8957 (6), 11.4352 (5)
β (°)	115.8831 (18)	113.8388 (19)	91.808 (2)	118.642 (2)
V (Å^3)	808.59 (6)	803.35 (7)	797.66 (10)	1640.03 (13)
Z	4	4	4	8
Radiation type	Mo $K\alpha$	Mo $K\alpha$	Mo $K\alpha$	Mo $K\alpha$
μ (mm^{-1})	0.10	0.10	0.10	0.10
Crystal size (mm)	0.49 × 0.45 × 0.16	0.50 × 0.43 × 0.40	0.74 × 0.38 × 0.13	0.50 × 0.43 × 0.40
Data collection				
Diffractometer	Bruker D8 Quest *APEX3*	Bruker D8 Quest *APEX3*	Bruker D8 Quest *APEX3*	Bruker D8 Quest *APEX3*
Absorption correction	Multi-scan (*SADABS*; Krause et al., 2015)	Multi-scan (*SADABS*; Krause et al., 2015)	Multi-scan (*SADABS*; Krause et al., 2015)	Multi-scan (*SADABS*; Krause et al., 2015)
T_{min}, T_{max}	0.672, 0.747	0.685, 0.746	0.705, 0.747	0.703, 0.747
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	17821, 4126, 3160	15236, 2461, 2171	30235, 3873, 3276	53075, 7976, 6730
R_{int}	0.032	0.020	0.024	0.030
$(\sin\theta/\lambda)_{max}$ (Å^{-1})	0.849	0.714	0.834	0.836
Refinement				
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.043, 0.130, 1.02	0.039, 0.117, 1.03	0.039, 0.124, 1.02	0.042, 0.126, 1.05
No. of reflections	4126	2461	3873	7976
No. of parameters	111	111	111	222
H-atom treatment	H-atom parameters constrained	H-atom parameters constrained	H-atom parameters constrained	H-atom parameters constrained
$\Delta\rho_{max}, \Delta\rho_{min}$ (e Å^{-3})	0.60, −0.24	0.40, −0.24	0.54, −0.22	0.48, −0.26

Computer programs: *APEX3* and *SAINT* (Bruker, 2012), *PEAKREF* (Schreurs, 2013), *SHELXT2014/4* (Sheldrick, 2015a), *SHELXL2016/6* (Sheldrick, 2015b), *PLATON* (Spek, 2009) and *ShelXLe* (Hübschle et al., 2011).

Figure 8
Powder X-ray diffraction measurements of form I (black) and II (blue) of 3,4-DMBz. The powder pattern (red) was calculated from the crystal structure by de Ronde *et al.* (2016).
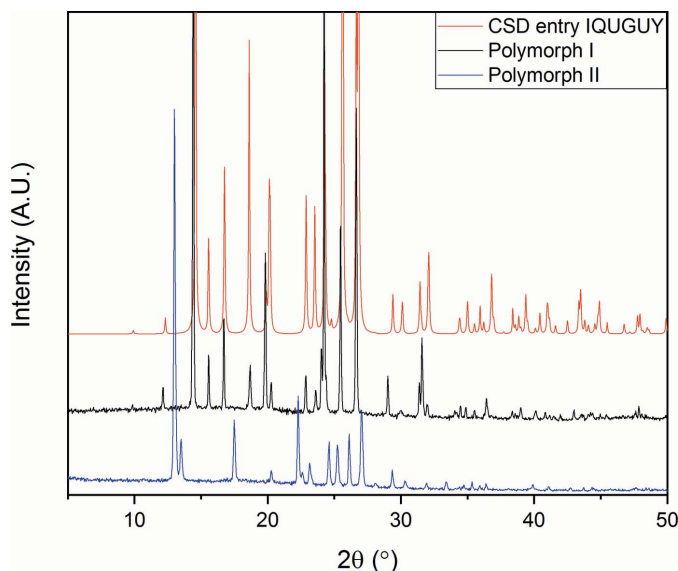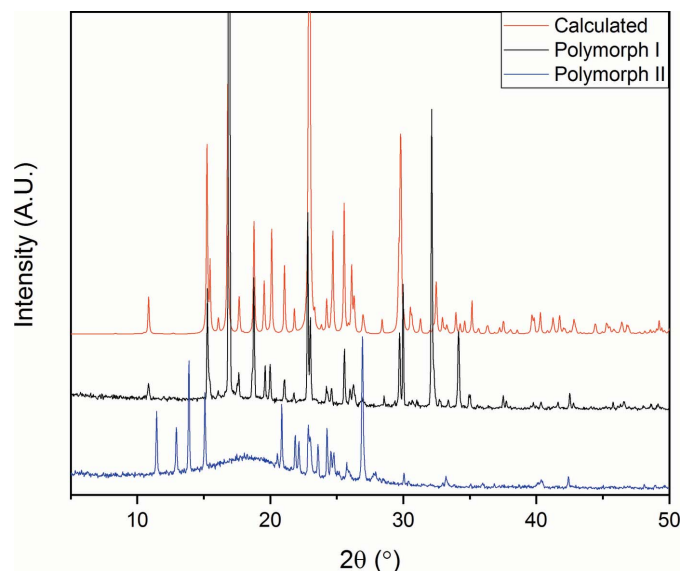
Figure 9
Powder X-ray diffraction measurements of form I (black) and II (blue) of 3,5-DMBz. The powder pattern (red) was calculated from the crystal structure.

6. Synthesis and crystallization

6.1. 2,3-dimethoxybenzaldehyde

30 mg of 2,3-dimethoxybenzaldehyde (97%, Fluorochem) was dissolved in 4 mL of isopropyl ether. Slow evaporation of a 1:1 mixture of this solution and heptane yielded colorless block-shaped crystals suitable for single crystal X-ray diffraction.

6.2. 2,4-dimethoxybenzaldehyde

25 mg of 2,4-dimethoxybenzaldeyhyde (98%, Aldrich) was dissolved in a 1:1 ratio of heptane/acetone (1.5 mL). Slow evaporation yielded colorless block-shaped crystals suitable for single crystal X-ray diffraction.

6.3. 2,5-dimethoxybenzaldehyde

1 g of 2,5-dimethoxybenzaldeyhyde (97%, Acros Organics) was dissolved in a mixture of heptane (1 mL) and acetone (1 mL). Slow evaporation yielded colorless needles suitable for single crystal X-ray diffraction.

6.4. 3,5-dimethoxybenzaldehyde

It was noted that 3,5-dimethoxybenzaldehyde (98%, Aldrich) oils out from solution, therefore the same method was used as had previously been employed for 3,4-dimethoxybenzaldehyde (de Ronde *et al.*, 2016). In short, a few crystals of the commercial powder were added to a saturated solution in water. Subsequently, the temperature was cycled between 298 and 303 K. This resulted in the growth of single crystals suitable for single-crystal X-ray diffraction in several weeks.

7. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 3. H atoms were positioned geometrically and refined as riding with C—H = 0.95–0.96 and $U_{iso}(H) = 1.2–1.5U_{eq}(C)$. The crystal of 3,5-DMBz studied was refined as a two-component twin.

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supporting information

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Computing details

For all structures, data collection: *APEX3* (Bruker, 2012); cell refinement: *PEAKREF* (Schreurs, 2013); data reduction: *SAINT* (Bruker, 2012); program(s) used to solve structure: *SHELXT2014/4* (Sheldrick, 2015a); program(s) used to refine structure: *SHELXL2016/6* (Sheldrick, 2015b); molecular graphics: *PLATON* (Spek, 2009), *ShelXLe* (Hübschle *et al.*, 2011).

2,3-Dimethoxybenzaldehyde (23DMBz)

Crystal data

$C_9H_{10}O_3$	$D_x = 1.365$ Mg m^{-3}
$M_r = 166.17$	Melting point: 322 K
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
$a = 7.6152$ (3) Å	Cell parameters from 6893 reflections
$b = 15.5513$ (6) Å	$\theta = 2.6$–$36.9°$
$c = 7.5891$ (3) Å	$\mu = 0.10$ mm^{-1}
$\beta = 115.8831$ (18)°	$T = 150$ K
$V = 808.59$ (6) Å^3	Block, colourless
$Z = 4$	$0.49 \times 0.45 \times 0.16$ mm
$F(000) = 352$	

Data collection

Bruker D8 Quest APEX3 diffractometer	17821 measured reflections
Radiation source: sealed tube	4126 independent reflections
Graphite monochromator	3160 reflections with $I > 2\sigma(I)$
Detector resolution: 10.4 pixels mm^{-1}	$R_{int} = 0.032$
φ and ω scans	$\theta_{max} = 37.1°$, $\theta_{min} = 2.6°$
Absorption correction: multi-scan (SADABS; Krause et al., 2015)	$h = -12 \rightarrow 12$
	$k = -26 \rightarrow 26$
$T_{min} = 0.672$, $T_{max} = 0.747$	$l = -12 \rightarrow 12$

Refinement

Refinement on F^2	Primary atom site location: structure-invariant direct methods
Least-squares matrix: full	Secondary atom site location: difference Fourier map
$R[F^2 > 2\sigma(F^2)] = 0.043$	
$wR(F^2) = 0.130$	Hydrogen site location: inferred from neighbouring sites
$S = 1.02$	
4126 reflections	H-atom parameters constrained
111 parameters	
0 restraints	

$w = 1/[\sigma^2(F_o^2) + (0.0743P)^2 + 0.0948P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$

$\Delta\rho_{max} = 0.60$ e Å^{-3}
$\Delta\rho_{min} = -0.24$ e Å^{-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 (Å^2)

	x	*y*	*z*	$U_{iso}*/U_{eq}$
O01	0.84423 (8)	0.67610 (3)	0.72112 (7)	0.01819 (11)
O02	0.93597 (9)	0.50880 (4)	0.76827 (8)	0.02194 (12)
O03	0.42554 (9)	0.77393 (5)	0.22027 (10)	0.03084 (15)
C04	0.60779 (10)	0.65162 (5)	0.38944 (10)	0.01633 (12)
C05	0.79724 (10)	0.53265 (4)	0.58899 (9)	0.01586 (12)
C06	0.50903 (11)	0.59449 (5)	0.23432 (10)	0.02083 (14)
H06	0.410967	0.615175	0.113675	0.025*
C07	0.75191 (9)	0.62098 (4)	0.56613 (9)	0.01466 (12)
C08	0.55760 (11)	0.74397 (5)	0.36618 (11)	0.02121 (14)
H08	0.632072	0.781899	0.470279	0.025*
C09	0.69865 (11)	0.47667 (5)	0.43414 (11)	0.01968 (13)
H09	0.729027	0.417067	0.448365	0.024*
C10	0.55498 (11)	0.50806 (5)	0.25774 (11)	0.02239 (15)
H10	0.487993	0.469473	0.152498	0.027*
C11	1.04331 (11)	0.69266 (5)	0.75867 (12)	0.02355 (15)
H11A	1.113534	0.638043	0.778082	0.035*
H11B	1.106109	0.728092	0.876679	0.035*
H11C	1.045946	0.723144	0.646820	0.035*
C12	0.98881 (13)	0.41998 (5)	0.79585 (12)	0.02495 (16)
H12A	0.872738	0.385147	0.769517	0.037*
H12B	1.086092	0.410593	0.931161	0.037*
H12C	1.043851	0.403253	0.705808	0.037*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O01	0.0179 (2)	0.0172 (2)	0.0179 (2)	−0.00017 (17)	0.00634 (17)	−0.00429 (17)
O02	0.0282 (3)	0.0151 (2)	0.0174 (2)	0.00471 (19)	0.0052 (2)	0.00247 (17)
O03	0.0228 (3)	0.0306 (3)	0.0343 (3)	0.0108 (2)	0.0081 (2)	0.0124 (3)
C04	0.0142 (3)	0.0176 (3)	0.0168 (3)	0.0012 (2)	0.0064 (2)	0.0019 (2)
C05	0.0180 (3)	0.0140 (3)	0.0157 (2)	0.0007 (2)	0.0075 (2)	0.0007 (2)
C06	0.0171 (3)	0.0265 (4)	0.0164 (3)	−0.0016 (2)	0.0049 (2)	−0.0001 (2)
C07	0.0147 (3)	0.0138 (3)	0.0153 (2)	0.0003 (2)	0.0064 (2)	−0.00084 (19)
C08	0.0185 (3)	0.0207 (3)	0.0250 (3)	0.0049 (2)	0.0100 (3)	0.0050 (3)
C09	0.0240 (3)	0.0156 (3)	0.0207 (3)	−0.0029 (2)	0.0110 (3)	−0.0034 (2)
C10	0.0229 (3)	0.0240 (3)	0.0189 (3)	−0.0061 (3)	0.0078 (3)	−0.0057 (2)

| C11 | 0.0193 (3) | 0.0225 (3) | 0.0254 (3) | −0.0045 (3) | 0.0067 (3) | −0.0055 (3) |
| C12 | 0.0324 (4) | 0.0170 (3) | 0.0272 (3) | 0.0085 (3) | 0.0147 (3) | 0.0064 (3) |

Geometric parameters (Å, º)

O01—C07	1.3750 (8)	C06—H06	0.9500
O01—C11	1.4383 (10)	C08—H08	0.9500
O02—C05	1.3601 (8)	C09—C10	1.3960 (11)
O02—C12	1.4284 (9)	C09—H09	0.9500
O03—C08	1.2175 (9)	C10—H10	0.9500
C04—C07	1.3945 (9)	C11—H11A	0.9800
C04—C06	1.4029 (10)	C11—H11B	0.9800
C04—C08	1.4767 (10)	C11—H11C	0.9800
C05—C09	1.3901 (10)	C12—H12A	0.9800
C05—C07	1.4085 (9)	C12—H12B	0.9800
C06—C10	1.3807 (12)	C12—H12C	0.9800
C07—O01—C11	112.47 (6)	C05—C09—H09	120.0
C05—O02—C12	117.16 (6)	C10—C09—H09	120.0
C07—C04—C06	119.94 (7)	C06—C10—C09	120.82 (7)
C07—C04—C08	120.08 (6)	C06—C10—H10	119.6
C06—C04—C08	119.98 (6)	C09—C10—H10	119.6
O02—C05—C09	124.88 (6)	O01—C11—H11A	109.5
O02—C05—C07	115.51 (6)	O01—C11—H11B	109.5
C09—C05—C07	119.59 (6)	H11A—C11—H11B	109.5
C10—C06—C04	119.71 (7)	O01—C11—H11C	109.5
C10—C06—H06	120.1	H11A—C11—H11C	109.5
C04—C06—H06	120.1	H11B—C11—H11C	109.5
O01—C07—C04	120.23 (6)	O02—C12—H12A	109.5
O01—C07—C05	119.76 (6)	O02—C12—H12B	109.5
C04—C07—C05	119.96 (6)	H12A—C12—H12B	109.5
O03—C08—C04	123.28 (8)	O02—C12—H12C	109.5
O03—C08—H08	118.4	H12A—C12—H12C	109.5
C04—C08—H08	118.4	H12B—C12—H12C	109.5
C05—C09—C10	119.98 (7)		
C12—O02—C05—C09	2.55 (11)	O02—C05—C07—O01	−1.05 (9)
C12—O02—C05—C07	−178.75 (6)	C09—C05—C07—O01	177.73 (6)
C07—C04—C06—C10	0.13 (11)	O02—C05—C07—C04	−178.28 (6)
C08—C04—C06—C10	−179.23 (7)	C09—C05—C07—C04	0.51 (10)
C11—O01—C07—C04	−108.70 (7)	C07—C04—C08—O03	−175.45 (7)
C11—O01—C07—C05	74.09 (8)	C06—C04—C08—O03	3.90 (11)
C06—C04—C07—O01	−177.61 (6)	O02—C05—C09—C10	178.31 (7)
C08—C04—C07—O01	1.75 (10)	C07—C05—C09—C10	−0.34 (11)
C06—C04—C07—C05	−0.40 (10)	C04—C06—C10—C09	0.03 (11)
C08—C04—C07—C05	178.96 (6)	C05—C09—C10—C06	0.08 (11)

2,4-Dimethoxybenzaldehyde (24DMBz)

Crystal data

$C_9H_{10}O_3$
$M_r = 166.17$
Monoclinic, $P2_1/c$
$a = 15.1575\ (8)$ Å
$b = 3.9638\ (2)$ Å
$c = 14.6181\ (8)$ Å
$\beta = 113.8388\ (19)°$
$V = 803.35\ (7)$ Å^3
$Z = 4$
$F(000) = 352$

$D_x = 1.374$ Mg m^{-3}
Melting point: 341 K
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 9286 reflections
$\theta = 2.8–30.5°$
$\mu = 0.10$ mm^{-1}
$T = 150$ K
Block, colourless
$0.50 \times 0.43 \times 0.40$ 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; Krause et al., 2015)
$T_{min} = 0.685$, $T_{max} = 0.746$

15236 measured reflections
2461 independent reflections
2171 reflections with $I > 2\sigma(I)$
$R_{int} = 0.020$
$\theta_{max} = 30.5°$, $\theta_{min} = 2.8°$
$h = -21 \rightarrow 21$
$k = -5 \rightarrow 5$
$l = -20 \rightarrow 19$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.039$
$wR(F^2) = 0.117$
$S = 1.03$
2461 reflections
111 parameters
0 restraints
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
$w = 1/[\sigma^2(F_o^2) + (0.0714P)^2 + 0.196P]$
   where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.40$ e Å^{-3}
$\Delta\rho_{min} = -0.24$ e Å^{-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 (Å^2)

	x	y	z	$U_{iso}*/U_{eq}$
O01	0.08102 (5)	0.65429 (18)	0.10801 (5)	0.02325 (16)
O02	0.39771 (5)	0.76391 (19)	0.37610 (5)	0.02571 (17)
O03	0.31680 (6)	0.2357 (2)	0.56154 (5)	0.0358 (2)
C04	0.30419 (6)	0.6579 (2)	0.33618 (6)	0.01822 (17)
C05	0.27290 (6)	0.4771 (2)	0.40045 (6)	0.01952 (18)
C06	0.14709 (6)	0.6063 (2)	0.20292 (6)	0.01766 (17)
C07	0.24207 (6)	0.7210 (2)	0.23720 (6)	0.01809 (17)

H07	0.263957	0.839754	0.193881	0.022*
C08	0.11380 (6)	0.4292 (2)	0.26563 (6)	0.01980 (18)
H08	0.048785	0.354354	0.241536	0.024*
C09	0.17672 (6)	0.3653 (2)	0.36266 (6)	0.02020 (18)
H09	0.154643	0.242605	0.405167	0.024*
C10	0.11345 (7)	0.8119 (2)	0.03878 (7)	0.02355 (19)
H10A	0.133339	1.044263	0.060154	0.035*
H10B	0.060848	0.813520	−0.028047	0.035*
H10C	0.168300	0.685728	0.036939	0.035*
C11	0.33908 (7)	0.3954 (3)	0.50273 (7)	0.0272 (2)
H11	0.403821	0.471787	0.524941	0.033*
C12	0.43374 (7)	0.9262 (3)	0.31058 (8)	0.0276 (2)
H12A	0.425511	0.776125	0.254432	0.041*
H12B	0.502361	0.977611	0.347363	0.041*
H12C	0.397996	1.135995	0.285113	0.041*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O01	0.0204 (3)	0.0285 (3)	0.0187 (3)	−0.0020 (2)	0.0057 (2)	0.0016 (2)
O02	0.0180 (3)	0.0327 (4)	0.0249 (3)	−0.0049 (3)	0.0071 (2)	0.0042 (3)
O03	0.0340 (4)	0.0489 (5)	0.0239 (3)	−0.0020 (3)	0.0111 (3)	0.0108 (3)
C04	0.0169 (3)	0.0182 (4)	0.0204 (4)	0.0001 (3)	0.0084 (3)	−0.0010 (3)
C05	0.0211 (4)	0.0201 (4)	0.0186 (4)	0.0008 (3)	0.0093 (3)	0.0001 (3)
C06	0.0189 (4)	0.0163 (3)	0.0181 (3)	0.0013 (3)	0.0077 (3)	−0.0022 (3)
C07	0.0194 (4)	0.0176 (3)	0.0194 (4)	0.0003 (3)	0.0100 (3)	−0.0001 (3)
C08	0.0191 (4)	0.0194 (4)	0.0229 (4)	−0.0016 (3)	0.0106 (3)	−0.0018 (3)
C09	0.0229 (4)	0.0198 (4)	0.0218 (4)	−0.0003 (3)	0.0131 (3)	0.0000 (3)
C10	0.0269 (4)	0.0249 (4)	0.0188 (4)	−0.0002 (3)	0.0092 (3)	0.0011 (3)
C11	0.0251 (4)	0.0335 (5)	0.0215 (4)	−0.0011 (4)	0.0078 (3)	0.0034 (3)
C12	0.0215 (4)	0.0296 (5)	0.0337 (5)	−0.0017 (3)	0.0134 (4)	0.0068 (4)

Geometric parameters (Å, º)

O01—C06	1.3567 (10)	C07—H07	0.9500
O01—C10	1.4347 (11)	C08—C09	1.3756 (12)
O02—C04	1.3629 (10)	C08—H08	0.9500
O02—C12	1.4326 (11)	C09—H09	0.9500
O03—C11	1.2201 (12)	C10—H10A	0.9800
C04—C07	1.3934 (11)	C10—H10B	0.9800
C04—C05	1.4077 (11)	C10—H10C	0.9800
C05—C09	1.4057 (12)	C11—H11	0.9500
C05—C11	1.4608 (12)	C12—H12A	0.9800
C06—C07	1.3954 (11)	C12—H12B	0.9800
C06—C08	1.4007 (11)	C12—H12C	0.9800
C06—O01—C10	117.43 (7)	C08—C09—H09	119.2
C04—O02—C12	117.63 (7)	C05—C09—H09	119.2

O02—C04—C07	122.59 (7)	O01—C10—H10A	109.5
O02—C04—C05	116.35 (7)	O01—C10—H10B	109.5
C07—C04—C05	121.05 (7)	H10A—C10—H10B	109.5
C09—C05—C04	118.29 (7)	O01—C10—H10C	109.5
C09—C05—C11	120.43 (8)	H10A—C10—H10C	109.5
C04—C05—C11	121.24 (8)	H10B—C10—H10C	109.5
O01—C06—C07	123.34 (8)	O03—C11—C05	124.40 (9)
O01—C06—C08	115.29 (7)	O03—C11—H11	117.8
C07—C06—C08	121.37 (8)	C05—C11—H11	117.8
C04—C07—C06	118.72 (8)	O02—C12—H12A	109.5
C04—C07—H07	120.6	O02—C12—H12B	109.5
C06—C07—H07	120.6	H12A—C12—H12B	109.5
C09—C08—C06	118.95 (8)	O02—C12—H12C	109.5
C09—C08—H08	120.5	H12A—C12—H12C	109.5
C06—C08—H08	120.5	H12B—C12—H12C	109.5
C08—C09—C05	121.60 (8)		

C12—O02—C04—C07	−4.23 (12)	O01—C06—C07—C04	−179.94 (7)
C12—O02—C04—C05	175.40 (8)	C08—C06—C07—C04	−0.36 (12)
O02—C04—C05—C09	179.48 (8)	O01—C06—C08—C09	179.01 (7)
C07—C04—C05—C09	−0.88 (13)	C07—C06—C08—C09	−0.60 (12)
O02—C04—C05—C11	−2.82 (13)	C06—C08—C09—C05	0.83 (13)
C07—C04—C05—C11	176.82 (8)	C04—C05—C09—C08	−0.11 (13)
C10—O01—C06—C07	4.17 (12)	C11—C05—C09—C08	−177.83 (8)
C10—O01—C06—C08	−175.43 (7)	C09—C05—C11—O03	−1.27 (16)
O02—C04—C07—C06	−179.28 (8)	C04—C05—C11—O03	−178.93 (10)
C05—C04—C07—C06	1.10 (12)		

2,5-Dimethoxybenzaldehyde (25DMBz)

Crystal data

$C_9H_{10}O_3$	$D_x = 1.384$ Mg m^{-3}
$M_r = 166.17$	Melting point: 321 K
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
$a = 3.8780$ (3) Å	Cell parameters from 9955 reflections
$b = 11.5513$ (7) Å	$\theta = 2.3–36.2°$
$c = 17.8153$ (12) Å	$\mu = 0.10$ mm^{-1}
$\beta = 91.808$ (2)°	$T = 150$ K
$V = 797.66$ (10) Å^3	Needle, colourless
$Z = 4$	$0.74 \times 0.38 \times 0.13$ mm
$F(000) = 352$	

Data collection

Bruker D8 Quest APEX3 diffractometer	30235 measured reflections
Radiation source: sealed tube	3873 independent reflections
Graphite monochromator	3276 reflections with $I > 2\sigma(I)$
Detector resolution: 10.4 pixels mm^{-1}	$R_{int} = 0.024$
φ and ω scans	$\theta_{max} = 36.4°$, $\theta_{min} = 2.1°$
Absorption correction: multi-scan (SADABS; Krause et al., 2015)	$h = -6 \rightarrow 6$
	$k = -14 \rightarrow 19$
$T_{min} = 0.705$, $T_{max} = 0.747$	$l = -29 \rightarrow 29$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	
$R[F^2 > 2\sigma(F^2)] = 0.039$	Hydrogen site location: inferred from neighbouring sites
$wR(F^2) = 0.124$	
$S = 1.02$	H-atom parameters constrained
3873 reflections	$w = 1/[\sigma^2(F_o^2) + (0.0691P)^2 + 0.1755P]$
111 parameters	where $P = (F_o^2 + 2F_c^2)/3$
0 restraints	$(\Delta/\sigma)_{max} = 0.001$
Primary atom site location: structure-invariant direct methods	$\Delta\rho_{max} = 0.54$ e Å^{-3}
	$\Delta\rho_{min} = -0.22$ e Å^{-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 (Å^2)

	x	y	z	U_{iso}*/U_{eq}
O01	0.70027 (15)	0.37815 (5)	0.56706 (3)	0.02308 (11)
O02	0.32551 (16)	0.55079 (5)	0.84287 (3)	0.02348 (12)
O03	0.81653 (19)	0.71583 (5)	0.60790 (3)	0.02970 (14)
C04	0.64196 (16)	0.53211 (5)	0.65291 (3)	0.01623 (11)
C05	0.54777 (16)	0.57451 (5)	0.72267 (3)	0.01719 (11)
H05	0.580616	0.654118	0.734217	0.021*
C06	0.44894 (18)	0.34122 (6)	0.68849 (4)	0.01849 (12)
H06	0.413247	0.261704	0.677056	0.022*
C07	0.40630 (16)	0.50128 (5)	0.77542 (3)	0.01656 (11)
C08	0.35490 (17)	0.38467 (6)	0.75789 (4)	0.01814 (12)
H08	0.255150	0.334576	0.793501	0.022*
C09	0.59531 (16)	0.41403 (5)	0.63571 (3)	0.01655 (11)
C10	0.79366 (19)	0.61160 (6)	0.59830 (4)	0.02189 (13)
H10	0.878085	0.579933	0.553220	0.026*
C11	0.6662 (2)	0.25766 (6)	0.55053 (4)	0.02393 (14)
H11A	0.787349	0.212440	0.589721	0.036*
H11B	0.766517	0.241337	0.501841	0.036*
H11C	0.421362	0.236493	0.548665	0.036*
C12	0.1987 (2)	0.47519 (7)	0.89872 (4)	0.02583 (15)
H12A	−0.020111	0.441090	0.880789	0.039*
H12B	0.161901	0.518945	0.944911	0.039*
H12C	0.367146	0.413466	0.908934	0.039*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O01	0.0330 (3)	0.0181 (2)	0.0185 (2)	−0.00401 (19)	0.00611 (18)	−0.00154 (16)
O02	0.0348 (3)	0.0174 (2)	0.0187 (2)	0.00073 (19)	0.00818 (19)	0.00082 (16)
O03	0.0450 (3)	0.0186 (2)	0.0256 (3)	−0.0097 (2)	0.0032 (2)	0.00350 (18)

C04	0.0178 (2)	0.0145 (2)	0.0164 (2)	−0.00141 (18)	0.00017 (18)	0.00262 (18)
C05	0.0197 (2)	0.0141 (2)	0.0178 (2)	−0.00057 (19)	0.00054 (19)	0.00188 (18)
C06	0.0222 (3)	0.0146 (2)	0.0186 (2)	−0.00275 (19)	0.0011 (2)	0.00149 (18)
C07	0.0179 (2)	0.0156 (2)	0.0163 (2)	0.00086 (19)	0.00157 (18)	0.00161 (18)
C08	0.0205 (3)	0.0158 (2)	0.0183 (2)	−0.00184 (19)	0.00202 (19)	0.00261 (18)
C09	0.0182 (2)	0.0155 (2)	0.0160 (2)	−0.00112 (19)	0.00031 (18)	0.00083 (18)
C10	0.0277 (3)	0.0191 (3)	0.0189 (3)	−0.0051 (2)	0.0018 (2)	0.0032 (2)
C11	0.0306 (3)	0.0194 (3)	0.0219 (3)	−0.0011 (2)	0.0020 (2)	−0.0034 (2)
C12	0.0322 (4)	0.0232 (3)	0.0227 (3)	0.0033 (3)	0.0109 (3)	0.0042 (2)

Geometric parameters (Å, º)

O01—C09	1.3655 (8)	C06—C09	1.3954 (9)
O01—C11	1.4280 (9)	C06—H06	0.9500
O02—C07	1.3757 (8)	C07—C08	1.3957 (9)
O02—C12	1.4232 (9)	C08—H08	0.9500
O03—C10	1.2189 (9)	C10—H10	0.9500
C04—C05	1.3952 (9)	C11—H11A	0.9800
C04—C09	1.4083 (9)	C11—H11B	0.9800
C04—C10	1.4738 (9)	C11—H11C	0.9800
C05—C07	1.3901 (9)	C12—H12A	0.9800
C05—H05	0.9500	C12—H12B	0.9800
C06—C08	1.3936 (9)	C12—H12C	0.9800
C09—O01—C11	116.90 (5)	O01—C09—C04	116.67 (5)
C07—O02—C12	116.67 (6)	C06—C09—C04	119.30 (6)
C05—C04—C09	119.89 (6)	O03—C10—C04	123.47 (7)
C05—C04—C10	119.37 (6)	O03—C10—H10	118.3
C09—C04—C10	120.73 (6)	C04—C10—H10	118.3
C07—C05—C04	120.57 (6)	O01—C11—H11A	109.5
C07—C05—H05	119.7	O01—C11—H11B	109.5
C04—C05—H05	119.7	H11A—C11—H11B	109.5
C08—C06—C09	120.27 (6)	O01—C11—H11C	109.5
C08—C06—H06	119.9	H11A—C11—H11C	109.5
C09—C06—H06	119.9	H11B—C11—H11C	109.5
O02—C07—C05	116.31 (6)	O02—C12—H12A	109.5
O02—C07—C08	124.18 (6)	O02—C12—H12B	109.5
C05—C07—C08	119.51 (6)	H12A—C12—H12B	109.5
C06—C08—C07	120.44 (6)	O02—C12—H12C	109.5
C06—C08—H08	119.8	H12A—C12—H12C	109.5
C07—C08—H08	119.8	H12B—C12—H12C	109.5
O01—C09—C06	124.03 (6)		
C09—C04—C05—C07	−0.32 (9)	C11—O01—C09—C04	177.58 (6)
C10—C04—C05—C07	−179.56 (6)	C08—C06—C09—O01	178.78 (6)
C12—O02—C07—C05	−176.57 (6)	C08—C06—C09—C04	−0.97 (10)
C12—O02—C07—C08	3.41 (10)	C05—C04—C09—O01	−178.60 (6)
C04—C05—C07—O02	179.25 (6)	C10—C04—C09—O01	0.63 (9)

C04—C05—C07—C08	−0.72 (10)	C05—C04—C09—C06	1.16 (9)
C09—C06—C08—C07	−0.07 (10)	C10—C04—C09—C06	−179.60 (6)
O02—C07—C08—C06	−179.05 (6)	C05—C04—C10—O03	−6.37 (11)
C05—C07—C08—C06	0.92 (10)	C09—C04—C10—O03	174.39 (7)
C11—O01—C09—C06	−2.17 (10)		

3,5-Dimethoxybenzaldehyde (35DMBz)

Crystal data

$C_9H_{10}O_3$
$M_r = 166.17$
Monoclinic, $P2_1/c$
$a = 11.7602$ (5) Å
$b = 13.8957$ (6) Å
$c = 11.4352$ (5) Å
$\beta = 118.642$ (2)°
$V = 1640.03$ (13) Å³
$Z = 8$
$F(000) = 704$

$D_x = 1.346$ Mg m⁻³
Melting point: 319 K
Mo *Kα* radiation, $\lambda = 0.71073$ Å
Cell parameters from 9794 reflections
$\theta = 2.5–36.4°$
$\mu = 0.10$ mm⁻¹
$T = 150$ K
Block, colourless
0.50 × 0.43 × 0.40 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; Krause et al., 2015)
$T_{min} = 0.703$, $T_{max} = 0.747$

53075 measured reflections
7976 independent reflections
6730 reflections with $I > 2\sigma(I)$
$R_{int} = 0.030$
$\theta_{max} = 36.5°$, $\theta_{min} = 2.5°$
$h = −19 \rightarrow 19$
$k = −23 \rightarrow 22$
$l = −19 \rightarrow 19$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.042$
$wR(F^2) = 0.126$
$S = 1.05$
7976 reflections
222 parameters
0 restraints
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
$w = 1/[\sigma^2(F_o^2) + (0.0699P)^2 + 0.3147P]$
  where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.48$ e Å⁻³
$\Delta\rho_{min} = −0.25$ e Å⁻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.

Refinement. Refined as a two-component twin.

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

	x	*y*	*z*	$U_{iso}*/U_{eq}$
O01	0.10858 (7)	0.50152 (5)	0.16802 (7)	0.02241 (12)

O02	−0.14028 (7)	0.31769 (5)	0.35652 (7)	0.02335 (13)
O03	−0.07159 (7)	0.74381 (5)	0.32465 (7)	0.02290 (13)
O04	0.38859 (7)	0.67098 (5)	0.07120 (6)	0.02131 (12)
O05	0.56249 (7)	0.91448 (4)	0.40230 (6)	0.02143 (12)
O06	0.64980 (9)	0.48884 (5)	0.50429 (8)	0.03135 (16)
C07	0.01767 (7)	0.62606 (5)	0.24757 (8)	0.01641 (12)
H07	0.055272	0.676140	0.220862	0.020*
C08	−0.11303 (7)	0.57541 (5)	0.34891 (7)	0.01585 (12)
H08	−0.163797	0.590389	0.390752	0.019*
C09	0.03688 (7)	0.53026 (6)	0.22608 (8)	0.01607 (12)
C10	0.46028 (7)	0.69980 (6)	0.20008 (8)	0.01623 (12)
C11	−0.09212 (7)	0.47970 (5)	0.32589 (7)	0.01525 (12)
C12	−0.05762 (7)	0.64810 (5)	0.30898 (8)	0.01594 (12)
C13	0.60966 (8)	0.74637 (6)	0.47010 (8)	0.01768 (13)
H13	0.659966	0.761890	0.561868	0.021*
C14	−0.01837 (8)	0.45589 (6)	0.26492 (8)	0.01680 (12)
H14	−0.005616	0.390505	0.249828	0.020*
C15	0.55203 (7)	0.81845 (5)	0.37503 (7)	0.01604 (12)
C16	0.47689 (7)	0.79570 (6)	0.23988 (7)	0.01637 (12)
H16	0.437535	0.845382	0.175813	0.020*
C17	0.65223 (10)	0.57391 (7)	0.52876 (9)	0.02399 (16)
H17	0.696151	0.592882	0.619685	0.029*
C18	0.51868 (8)	0.62611 (6)	0.29356 (8)	0.01800 (13)
H18	0.508552	0.560727	0.266097	0.022*
C19	0.59175 (8)	0.65038 (6)	0.42732 (8)	0.01768 (13)
C20	−0.14979 (8)	0.40334 (6)	0.37109 (8)	0.01879 (13)
H20	−0.197577	0.422663	0.414534	0.023*
C21	−0.14813 (10)	0.76918 (6)	0.38642 (10)	0.02406 (16)
H21A	−0.235885	0.743391	0.333753	0.036*
H21B	−0.152196	0.839410	0.391306	0.036*
H21C	−0.108775	0.742127	0.476544	0.036*
C22	0.30925 (9)	0.74150 (7)	−0.02458 (8)	0.02306 (15)
H22A	0.251864	0.771607	0.004783	0.035*
H22B	0.257053	0.710378	−0.111097	0.035*
H22C	0.364524	0.790773	−0.032972	0.035*
C23	0.64453 (9)	0.94220 (6)	0.53742 (8)	0.02092 (14)
H23A	0.611474	0.914527	0.594092	0.031*
H23B	0.645839	1.012531	0.544393	0.031*
H23C	0.732586	0.918592	0.566549	0.031*
C24	0.17317 (10)	0.57404 (7)	0.13303 (11)	0.02795 (19)
H24A	0.108750	0.616172	0.064624	0.042*
H24B	0.226038	0.543502	0.098340	0.042*
H24C	0.228988	0.612158	0.212125	0.042*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O01	0.0291 (3)	0.0174 (3)	0.0313 (3)	−0.0012 (2)	0.0229 (3)	−0.0018 (2)

O02	0.0322 (3)	0.0155 (3)	0.0262 (3)	−0.0037 (2)	0.0170 (3)	−0.0008 (2)
O03	0.0310 (3)	0.0124 (2)	0.0347 (3)	0.0003 (2)	0.0233 (3)	−0.0003 (2)
O04	0.0257 (3)	0.0174 (3)	0.0159 (2)	0.0009 (2)	0.0060 (2)	−0.00314 (19)
O05	0.0276 (3)	0.0133 (2)	0.0164 (2)	0.0012 (2)	0.0050 (2)	−0.00162 (18)
O06	0.0421 (4)	0.0170 (3)	0.0329 (4)	0.0070 (3)	0.0164 (3)	0.0048 (3)
C07	0.0178 (3)	0.0140 (3)	0.0191 (3)	−0.0010 (2)	0.0103 (2)	0.0003 (2)
C08	0.0182 (3)	0.0144 (3)	0.0166 (3)	−0.0007 (2)	0.0097 (2)	0.0002 (2)
C09	0.0173 (3)	0.0151 (3)	0.0179 (3)	−0.0006 (2)	0.0101 (2)	−0.0004 (2)
C10	0.0170 (3)	0.0156 (3)	0.0160 (3)	0.0003 (2)	0.0078 (2)	−0.0015 (2)
C11	0.0176 (3)	0.0138 (3)	0.0148 (3)	−0.0011 (2)	0.0081 (2)	0.0007 (2)
C12	0.0181 (3)	0.0128 (3)	0.0179 (3)	−0.0005 (2)	0.0094 (2)	−0.0001 (2)
C13	0.0202 (3)	0.0162 (3)	0.0158 (3)	0.0023 (2)	0.0079 (2)	0.0009 (2)
C14	0.0201 (3)	0.0138 (3)	0.0185 (3)	−0.0007 (2)	0.0109 (2)	−0.0001 (2)
C15	0.0174 (3)	0.0137 (3)	0.0163 (3)	0.0012 (2)	0.0075 (2)	−0.0006 (2)
C16	0.0180 (3)	0.0144 (3)	0.0154 (3)	0.0011 (2)	0.0070 (2)	−0.0005 (2)
C17	0.0305 (4)	0.0185 (3)	0.0221 (3)	0.0059 (3)	0.0119 (3)	0.0045 (3)
C18	0.0210 (3)	0.0144 (3)	0.0193 (3)	0.0011 (2)	0.0102 (3)	−0.0002 (2)
C19	0.0206 (3)	0.0148 (3)	0.0183 (3)	0.0027 (2)	0.0098 (3)	0.0019 (2)
C20	0.0232 (3)	0.0161 (3)	0.0197 (3)	−0.0030 (2)	0.0125 (3)	0.0002 (2)
C21	0.0301 (4)	0.0173 (3)	0.0324 (4)	0.0023 (3)	0.0212 (4)	−0.0013 (3)
C22	0.0249 (4)	0.0231 (4)	0.0165 (3)	0.0040 (3)	0.0062 (3)	−0.0012 (3)
C23	0.0248 (3)	0.0175 (3)	0.0170 (3)	−0.0018 (3)	0.0072 (3)	−0.0029 (2)
C24	0.0334 (4)	0.0236 (4)	0.0400 (5)	−0.0051 (3)	0.0281 (4)	−0.0032 (4)

Geometric parameters (Å, º)

O01—C09	1.3594 (10)	C13—C19	1.4014 (11)
O01—C24	1.4297 (11)	C13—H13	0.9500
O02—C20	1.2144 (10)	C14—H14	0.9500
O03—C12	1.3624 (10)	C15—C16	1.4000 (10)
O03—C21	1.4292 (11)	C16—H16	0.9500
O04—C10	1.3609 (10)	C17—C19	1.4797 (12)
O04—C22	1.4321 (11)	C17—H17	0.9500
O05—C15	1.3623 (9)	C18—C19	1.3898 (11)
O05—C23	1.4275 (10)	C18—H18	0.9500
O06—C17	1.2120 (12)	C20—H20	0.9500
C07—C09	1.3918 (11)	C21—H21A	0.9800
C07—C12	1.4025 (11)	C21—H21B	0.9800
C07—H07	0.9500	C21—H21C	0.9800
C08—C12	1.3923 (11)	C22—H22A	0.9800
C08—C11	1.4003 (11)	C22—H22B	0.9800
C08—H08	0.9500	C22—H22C	0.9800
C09—C14	1.4014 (11)	C23—H23A	0.9800
C10—C16	1.3914 (11)	C23—H23B	0.9800
C10—C18	1.3996 (11)	C23—H23C	0.9800
C11—C14	1.3884 (11)	C24—H24A	0.9800
C11—C20	1.4795 (11)	C24—H24B	0.9800
C13—C15	1.3922 (11)	C24—H24C	0.9800

C09—O01—C24	117.83 (7)	O06—C17—H17	117.6
C12—O03—C21	116.74 (7)	C19—C17—H17	117.6
C10—O04—C22	117.72 (7)	C19—C18—C10	118.77 (7)
C15—O05—C23	116.88 (6)	C19—C18—H18	120.6
C09—C07—C12	119.48 (7)	C10—C18—H18	120.6
C09—C07—H07	120.3	C18—C19—C13	121.69 (7)
C12—C07—H07	120.3	C18—C19—C17	119.99 (7)
C12—C08—C11	118.39 (7)	C13—C19—C17	118.32 (7)
C12—C08—H08	120.8	O02—C20—C11	124.56 (8)
C11—C08—H08	120.8	O02—C20—H20	117.7
O01—C09—C07	123.95 (7)	C11—C20—H20	117.7
O01—C09—C14	115.37 (7)	O03—C21—H21A	109.5
C07—C09—C14	120.68 (7)	O03—C21—H21B	109.5
O04—C10—C16	123.58 (7)	H21A—C21—H21B	109.5
O04—C10—C18	115.74 (7)	O03—C21—H21C	109.5
C16—C10—C18	120.68 (7)	H21A—C21—H21C	109.5
C14—C11—C08	121.94 (7)	H21B—C21—H21C	109.5
C14—C11—C20	120.39 (7)	O04—C22—H22A	109.5
C08—C11—C20	117.66 (7)	O04—C22—H22B	109.5
O03—C12—C08	124.06 (7)	H22A—C22—H22B	109.5
O03—C12—C07	115.08 (7)	O04—C22—H22C	109.5
C08—C12—C07	120.86 (7)	H22A—C22—H22C	109.5
C15—C13—C19	118.47 (7)	H22B—C22—H22C	109.5
C15—C13—H13	120.8	O05—C23—H23A	109.5
C19—C13—H13	120.8	O05—C23—H23B	109.5
C11—C14—C09	118.66 (7)	H23A—C23—H23B	109.5
C11—C14—H14	120.7	O05—C23—H23C	109.5
C09—C14—H14	120.7	H23A—C23—H23C	109.5
O05—C15—C13	124.69 (7)	H23B—C23—H23C	109.5
O05—C15—C16	114.44 (7)	O01—C24—H24A	109.5
C13—C15—C16	120.86 (7)	O01—C24—H24B	109.5
C10—C16—C15	119.52 (7)	H24A—C24—H24B	109.5
C10—C16—H16	120.2	O01—C24—H24C	109.5
C15—C16—H16	120.2	H24A—C24—H24C	109.5
O06—C17—C19	124.76 (9)	H24B—C24—H24C	109.5
C24—O01—C09—C07	3.19 (13)	C23—O05—C15—C13	3.63 (12)
C24—O01—C09—C14	−176.62 (8)	C23—O05—C15—C16	−176.23 (7)
C12—C07—C09—O01	−179.69 (7)	C19—C13—C15—O05	−179.27 (8)
C12—C07—C09—C14	0.11 (12)	C19—C13—C15—C16	0.58 (12)
C22—O04—C10—C16	−10.72 (12)	O04—C10—C16—C15	179.72 (7)
C22—O04—C10—C18	169.48 (8)	C18—C10—C16—C15	−0.50 (12)
C12—C08—C11—C14	−0.02 (11)	O05—C15—C16—C10	179.48 (7)
C12—C08—C11—C20	178.95 (7)	C13—C15—C16—C10	−0.38 (12)
C21—O03—C12—C08	0.13 (12)	O04—C10—C18—C19	−179.06 (7)
C21—O03—C12—C07	179.77 (8)	C16—C10—C18—C19	1.13 (12)
C11—C08—C12—O03	179.42 (7)	C10—C18—C19—C13	−0.93 (12)

C11—C08—C12—C07	−0.20 (11)	C10—C18—C19—C17	178.64 (8)
C09—C07—C12—O03	−179.50 (7)	C15—C13—C19—C18	0.09 (12)
C09—C07—C12—C08	0.15 (12)	C15—C13—C19—C17	−179.49 (8)
C08—C11—C14—C09	0.28 (11)	O06—C17—C19—C18	4.47 (15)
C20—C11—C14—C09	−178.66 (7)	O06—C17—C19—C13	−175.94 (10)
O01—C09—C14—C11	179.50 (7)	C14—C11—C20—O02	−1.69 (13)
C07—C09—C14—C11	−0.32 (12)	C08—C11—C20—O02	179.33 (8)