The crystal structures of four dimethoxybenzaldehyde isomers

Sander J. T. Brugman, Anthonius H. J. Engwerda, Emma Kalkman, Erik de Ronde, Paul Tinnemans* and Elias Vlieg

Radboud University, Institute for Molecules and Materials, Heyendaalseweg 135, 6525 AJ Nijmegen, The Netherlands.
*Correspondence e-mail: p.tinnemans@science.ru.nl

The crystal structures of four dimethoxybenzaldehyde (C₉H₁₀O₃) 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-dimethoxybenzaldehyde. 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 Suzuki–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 dimethoxybenzaldehyde isomers, namely 2,3-DMBz (Fig. 1), 2,4-DMBz (Fig. 2), 2,5-DMBz (Fig. 3) and 3,5-DMBz (Fig. 4).
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 \( Pna_2_1 \) (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, \( \pi-\pi \) 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 \( \pi-\pi \) 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 \( \pi-\pi \) stacking interactions. As mentioned

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### Table 1

<table>
<thead>
<tr>
<th></th>
<th>2,3-DMBz</th>
<th>2,4-DMBz</th>
<th>2,5-DMBz</th>
<th>2,6-DMBz</th>
<th>3,4-DMBz</th>
<th>3,5-DMBz (molecule 1)</th>
<th>3,5-DMBz (molecule 2)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Aldehyde C</td>
<td>0.020</td>
<td>0.060</td>
<td>0.004</td>
<td>0.027</td>
<td>0.020</td>
<td>0.027</td>
<td>0.022</td>
</tr>
<tr>
<td>Aldehyde O</td>
<td>0.104</td>
<td>0.089</td>
<td>0.113</td>
<td>0.015</td>
<td>0.095</td>
<td>0.019</td>
<td>0.047</td>
</tr>
<tr>
<td>Methoxy 1 O</td>
<td>0.048</td>
<td>0.013</td>
<td>0.033</td>
<td>0.011</td>
<td>0.002</td>
<td>0.009</td>
<td>0.015</td>
</tr>
<tr>
<td>Methoxy 1 C</td>
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<td>0.122</td>
<td>0.099</td>
<td>0.017</td>
<td>0.001</td>
<td>0.087</td>
<td>0.258</td>
</tr>
<tr>
<td>Methoxy 2 O</td>
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<td>0.019</td>
<td>0.025</td>
<td>0.024</td>
<td>0.033</td>
<td>0.013</td>
<td>0.019</td>
</tr>
<tr>
<td>Methoxy 2 C</td>
<td>0.013</td>
<td>0.074</td>
<td>0.109</td>
<td>0.040</td>
<td>0.337</td>
<td>0.020</td>
<td>0.109</td>
</tr>
</tbody>
</table>

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

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**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.

**Figure 4**
The molecular structure of 3,5-DMBz, showing displacement ellipsoids drawn at the 50% probability level.
above, only 3,5-DMBz has two molecules in the asymmetric unit, whereas the other crystal structures have one molecule in the asymmetric unit.

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 dimethoxybenzaldehydes 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.

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

<table>
<thead>
<tr>
<th></th>
<th>2,3-DMBz</th>
<th>2,4-DMBz</th>
<th>2,5-DMBz</th>
<th>2,6-DMBz</th>
<th>3,4-DMBz</th>
<th>3,5-DMBz</th>
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</thead>
<tbody>
<tr>
<td>Polymorph I (stable form)</td>
<td>322</td>
<td>341</td>
<td>321</td>
<td>368</td>
<td>317</td>
<td>319</td>
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<tr>
<td>Polymorph II</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>*</td>
<td>310</td>
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</table>

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

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.

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.

<table>
<thead>
<tr>
<th>Chemical data</th>
<th>2,3DMBz</th>
<th>2,4DMBz</th>
<th>2,5DMBz</th>
<th>3,5DMBz</th>
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</thead>
<tbody>
<tr>
<td>Chemical formula</td>
<td>C₉H₁₀O₃</td>
<td>C₉H₁₀O₃</td>
<td>C₉H₁₀O₃</td>
<td>C₉H₁₀O₃</td>
</tr>
<tr>
<td>Mr</td>
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<td>166.17</td>
<td>166.17</td>
<td>166.17</td>
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<td>Monoclinic, P2₁/c</td>
<td>Monoclinic, P2₁/c</td>
<td>Monoclinic, P2₁/c</td>
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<td>Temperature (K)</td>
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<td>150</td>
<td>150</td>
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<tr>
<td>a, b, c (Å)</td>
<td>7.6152 (3), 15.5513 (6), 7.5891 (3)</td>
<td>15.1575 (8), 3.9638 (2), 14.6181 (8)</td>
<td>113.8838 (19)</td>
<td>797.66 (10)</td>
</tr>
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<td>β (°)</td>
<td>115.8831 (18)</td>
<td>113.8388 (19)</td>
<td>91.808 (2)</td>
<td>118.642 (2)</td>
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<tr>
<td>V (Å³)</td>
<td>808.59 (6)</td>
<td>803.35 (7)</td>
<td>3.8780 (3), 11.5513 (7), 17.8153 (12)</td>
<td>1640.03 (13)</td>
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<tr>
<td>Z</td>
<td>4</td>
<td>4</td>
<td>4</td>
<td>8</td>
</tr>
<tr>
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<td>Mo Kα</td>
<td>Mo Kα</td>
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<td>μ (mm⁻¹)</td>
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<td>0.10</td>
<td>0.10</td>
<td>0.10</td>
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<td>Crystal size (mm)</td>
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<td>0.50 x 0.43 x 0.40</td>
<td>0.74 x 0.38 x 0.13</td>
<td>0.50 x 0.43 x 0.40</td>
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Data collection

<table>
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<th>Diffractometer</th>
<th>Bruker D8 Quest APEX3</th>
<th>Bruker D8 Quest APEX3</th>
<th>Bruker D8 Quest APEX3</th>
<th>Bruker D8 Quest APEX3</th>
</tr>
</thead>
<tbody>
<tr>
<td>Absorption correction</td>
<td>Multi-scan (SADABS; Krause et al., 2015)</td>
<td>Multi-scan (SADABS; Krause et al., 2015)</td>
<td>Multi-scan (SADABS; Krause et al., 2015)</td>
<td>Multi-scan (SADABS; Krause et al., 2015)</td>
</tr>
<tr>
<td>Tmin, Tmax</td>
<td>0.672, 0.747</td>
<td>0.685, 0.746</td>
<td>0.705, 0.747</td>
<td>0.703, 0.747</td>
</tr>
<tr>
<td>No. of measured, independent and observed [I &gt; 2σ(I)] reflections</td>
<td>17821, 4126, 3160</td>
<td>15236, 2461, 2171</td>
<td>30235, 3873, 3276</td>
<td>53075, 7976, 6730</td>
</tr>
<tr>
<td>R(int)</td>
<td>0.032</td>
<td>0.020</td>
<td>0.024</td>
<td>0.030</td>
</tr>
<tr>
<td>(sin θ/λ)max (Å⁻¹)</td>
<td>0.849</td>
<td>0.714</td>
<td>0.834</td>
<td>0.836</td>
</tr>
</tbody>
</table>

Refinement

| R[F² > 2σ(F²)], wR(F²), 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 | 222 | 222 |
| H-atom treatment | H-atom parameters constrained | H-atom parameters constrained | H-atom parameters constrained | H-atom parameters constrained |
| Δρmax, Δρmin (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 ShelXL (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-dimethoxybenzaldehyde (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-dimethoxybenzaldehyde (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 \( U_{iso}(H) = 1.2–1.5 U_{eq}(C) \). The crystal of 3,5-DMBz studied was refined as a two-component twin.

References

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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), ShelXL (Hübschle et al., 2011).

2,3-Dimethoxybenzaldehyde (23DMBz)

Crystal data

C₉H₁₀O₃

Mᵣ = 166.17

Monoclinic, P2₁/c

a = 7.6152 (3) Å

b = 15.5513 (6) Å

c = 7.5891 (3) Å

β = 115.8831 (18)°

V = 808.59 (6) Å³

Z = 4

F(000) = 352

Density (calculated)

Dₐ = 1.365 Mg m⁻³

Melting point

322 K

Mo Kα radiation, λ = 0.71073 Å

Cell parameters from 6893 reflections

θ = 2.6–36.9°

µ = 0.10 mm⁻¹

T = 150 K

Block, colourless

0.49 × 0.45 × 0.16 mm

Data collection

Bruker D8 Quest APEX3
diffractometer

17821 measured reflections

4126 independent reflections

3160 reflections with I > 2σ(I)

R[F²] = 0.032

θ = 2.6–36.9°, θ_min = 2.6°

h = −12→12

k = −26→26

l = −12→12

Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites

H-atoms: there is no unique way of placing them.

Refinement

Refinement on F²

Least-squares matrix: full

R[F² > 2σ(F²)] = 0.043

wR(F²) = 0.130

S = 1.02

4126 reflections

111 parameters

0 restraints

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Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites

H-atoms: there is no unique way of placing them.
supporting information

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

\[\Delta \rho_{\text{max}} = 0.60 \text{ e Å}^{-3}\]
\[\Delta \rho_{\text{min}} = -0.24 \text{ e Å}^{-3}\]

**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 (Å²)**

<table>
<thead>
<tr>
<th></th>
<th>x</th>
<th>y</th>
<th>z</th>
<th>(U_{\text{iso}}/U_{\text{eq}})</th>
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2,4-Dimethoxybenzaldehyde (24DMBz)

Crystal data

C₉H₁₀O₃  
Mr = 166.17  
Monoclinic, P2₁/c  
a = 15.1575 (8) Å  
b = 3.9638 (2) Å  
c = 14.6181 (8) Å  
α = 113.8388 (19)°  
V = 803.35 (7) Å³  
Z = 4  
F(000) = 352  
Dx = 1.374 Mg m⁻³  
Melting point: 341 K  
Mo Kα radiation, λ = 0.71073 Å  
Cell parameters from 9286 reflections  
θ = 2.8–30.5°  
μ = 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)  
Tmin = 0.685, Tmax = 0.746  
15236 measured reflections  
2461 independent reflections  
2171 reflections with I > 2σ(I)  
R(int) = 0.020  
θmax = 30.5°, θmin = 2.8°  
h = −21→21  
k = −5→5  
l = −20→19

Refinement

Refinement on F²  
Least-squares matrix: full  
R[F² > 2σ(F²)] = 0.039  
wR(F²) = 0.117  
S = 1.03  
2461 reflections  
111 parameters  
0 restraints  
Secondary atom site location: difference Fourier map  
Hydrogen site location: inferred from neighbouring sites  
H-atom parameters constrained  
w = 1/σ²(F²) + (0.0714P)² + 0.196P]  
where P = (F² + 2Fc²)/3  
(Δ/σ)max < 0.001  
Δρmax = 0.40 e Å⁻³  
Δρmin = −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 (Å²)

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Atomic displacement parameters (Å²)

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Geometric parameters (Å, °)

| 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 |
sup-6

2,5-Dimethoxybenzaldehyde (25DMBz)

Crystal data

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<th>Symmetry</th>
<th>Space group</th>
<th>( a )</th>
<th>( b )</th>
<th>( c )</th>
<th>( \beta )</th>
<th>( V )</th>
<th>( Z )</th>
<th>( D_r )</th>
<th>Melting point</th>
<th>( \lambda )</th>
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<td>( P_2_1/n )</td>
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<td>17.8153 (12) Å</td>
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<td>1.384 Mg m⁻³</td>
<td>321 K</td>
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Cell parameters from 9955 reflections

\( \theta = 2.3-36.2° \)
\( \mu = 0.10 \) mm⁻¹
\( T = 150 \) K

Needle, colourless

0.74 × 0.38 × 0.13 mm

Data collection

Bruker D8 Quest APEX3

diffactometer

Radiation source: sealed tube

Graphite monochromator

Detector resolution: 10.4 pixels mm⁻¹

\( \varphi \) and \( \omega \) scans

Absorption correction: multi-scan

(SADABS; Krause et al., 2015)

\( T_{\text{min}} = 0.705, T_{\text{max}} = 0.747 \)

30235 measured reflections

3873 independent reflections

3276 reflections with \( I > 2\sigma(I) \)

\( R_{\text{int}} = 0.024 \)

\( \theta_{\text{max}} = 36.4°, \theta_{\text{min}} = 2.1° \)

\( h = -6 \rightarrow 6 \)

\( k = -14 \rightarrow 19 \)

\( l = -29 \rightarrow 29 \)

Refinement

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.039$
$wR(F^2) = 0.124$
$S = 1.02$
3873 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
$w = 1/[\sigma^2(F_o^2) + (0.0691P)^2 + 0.1755P]$
where $P = (F_o^2 + 2F_c^2)/3$
$\Delta\sigma/\sigma_{\text{max}} = 0.001$
$\Delta\rho_{\text{max}} = 0.54 \text{ e Å}^{-3}$
$\Delta\rho_{\text{min}} = -0.22 \text{ e Å}^{-3}$

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 (Å²)

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<th>y</th>
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Atomic displacement parameters (Å²)

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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) 0.00054 (19) 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, °)

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<td>O01—C09—C06</td>
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<td>C11—O01—C09—C04</td>
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</table>
3,5-Dimethoxybenzaldehyde (35DMBz)

Crystal data

C₉H₁₀O₃  
Mr = 166.17  
Monoclinic, P2₁/c  
a = 11.7602 (5) Å  
b = 13.8957 (6) Å  
c = 11.4352 (5) Å  
β = 118.642 (2)°  
V = 1640.03 (13) Å³  
Z = 8  
F(000) = 704  
Dₜ = 1.346 Mg m⁻³  
Melting point: 319 K  
Mo Kα radiation, λ = 0.71073 Å  
Cell parameters from 9794 reflections  
θ = 2.5–36.4°  
µ = 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)

Tmin = 0.703, Tmax = 0.747

53075 measured reflections

7976 independent reflections

6730 reflections with I > 2σ(I)

Rint = 0.030

θmax = 36.5°, θmin = 2.5°

h = −19→19

k = −23→22

l = −19→19

Refinement

Refinement on F²  
Secondary atom site location: difference Fourier map

Least-squares matrix: full

R[F² > 2σ(F²)] = 0.042

wR(F²) = 0.126

S = 1.05

7976 reflections

222 parameters

0 restraints

Primary atom site location: structure-invariant

direct methods

Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained

w = 1/[σ²(Fo²) + (0.0699P)² + 0.3147P]  
where P = (Fo² + 2Fc²)/3

(Δ/σ)max = 0.001

Δρmax = 0.48 e Å⁻³

Δρmin = −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)

<table>
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<tr>
<th>x</th>
<th>y</th>
<th>z</th>
<th>Ueq</th>
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<tbody>
<tr>
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<td>0.10858 (7)</td>
<td>0.50152 (5)</td>
<td>0.16802 (7)</td>
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### Atomic displacement parameters (Å²)

<table>
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<tr>
<th></th>
<th>( U^{11} )</th>
<th>( U^{22} )</th>
<th>( U^{33} )</th>
<th>( U^{12} )</th>
<th>( U^{13} )</th>
<th>( U^{23} )</th>
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<tr>
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<td>0.0291 (3)</td>
<td>0.0174 (3)</td>
<td>0.0313 (3)</td>
<td>-0.0012 (2)</td>
<td>0.0229 (3)</td>
<td>-0.0018 (2)</td>
</tr>
</tbody>
</table>

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sup-10
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)  −0.0031 (2)
O05  0.0276 (3)  0.0133 (2)  0.0164 (2)  0.0012 (2)  0.0050 (2)  −0.0016 (2)
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)  0.0234 (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, °)

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—C08—C11  118.39 (7)  C18—C19—C17  119.99 (7)
C12—C08—H08  120.8  C13—C19—C17  118.32 (7)
C11—C08—H08  120.8  O02—C20—C11  124.56 (8)
O01—C09—C07  123.95 (7)  O02—C20—H20  117.7
O01—C09—C14  115.37 (7)  C11—C20—H20  117.7
C07—C09—C14  120.68 (7)  O03—C21—H21A  109.5
O04—C10—C16  123.58 (7)  O03—C21—H21B  109.5
O04—C10—C18  115.74 (7)  O03—C21—H21C  109.5
C16—C10—C18  120.68 (7)  H21A—C21—H21B  109.5
C14—C11—C08  121.94 (7)  H21A—C21—H21C  109.5
C14—C11—C20  120.39 (7)  H21B—C21—H21C  109.5
C08—C11—C20  117.66 (7)  O04—C22—H22A  109.5
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C03—C12—C07  115.08 (7)  O04—C22—H22C  109.5
C08—C12—C07  120.86 (7)  H22A—C22—H22B  109.5
C15—C13—C19  118.47 (7)  H22A—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)  O01—C24—H24C  109.5
C10—C16—H16  120.2  H24A—C24—H24C  109.5
C15—C16—H16  120.2  H24B—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)
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