Crystal structures of methyl 3,5-dimethylbenzoate, 3,5-bis(bromomethyl)phenyl acetate and 5-hydroxybenzene-1,3-dicarbaldehyde

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The crystal structures of the title compounds, methyl 3,5-dimethylbenzoate \((\text{C}_{10}\text{H}_{12}\text{O}_{2}; \text{1})\), 3,5-bis(bromomethyl)phenyl acetate \((\text{C}_{10}\text{H}_{10}\text{Br}_{2}\text{O}_{2}; \text{2})\) and 5-hydroxybenzene-1,3-dicarbaldehyde \((\text{C}_{8}\text{H}_{6}\text{O}_{3}; \text{3})\) were determined by single-crystal X-ray analysis. The crystals of \text{1} are composed of strands of C—H···O bonded molecules, which are further arranged into layers. As a result of the presence of two bromomethyl substituents in compound \text{2}, molecular dimers formed by crystallographically non-equivalent molecules are connected to structurally different two-dimensional aggregates in which the bromine atoms participate in Br···Br bonds of type I and type II. In the case of compound \text{3}, which possesses three donor/acceptor substituents, the molecular association in the crystal creates a close three-dimensional network comprising Caryl—H···Ohydroxy, Cformyl—H···Oformyl and O—H···Oformyl bonds.

1. Chemical context

Studies on molecular recognition of carbohydrates by artificial receptors revealed that macrocyclic compounds bearing two flexible side-arms represent effective and selective receptors for complexation of glucopyranosides. The binding properties of these compounds depend on the nature of their building blocks, among others, the type of bridging units that connect two aromatic platforms (Lippe & Mazik, 2013, 2015; Amrhein et al., 2016, 2021; Amrhein & Mazik, 2021). The design of such receptor architectures was inspired by the results of our crystallographic studies on receptor–carbohydrate complexes (Mazik et al., 2005; for recent examples, see Köhler et al., 2020, 2021). For the syntheses of macrocycles consisting of benzene-based bridges, various 2- or 5-substituted benzene-1,3-dicarbaldehydes have proven to be useful starting materials. Benzene derivatives with methyl or bromomethyl groups in positions 1 and 3 are used to prepare the latter compounds. The crystal structures of three 1,3,5-substituted benzenes, serving as precursors for the syntheses of the macrocyclic compounds mentioned above, are described in this work.
2. Structural commentary

The title compounds 1 and 3 crystallize in the monoclinic system (space group $P2_1/c$, $Z = 4$), whereas compound 2 crystallizes in the triclinic space group $P\overline{1}$ with two independent but conformationally similar molecules (A and B) in the asymmetric unit of the cell. In compound 1 (Fig. 1), the plane through the methyloxycarbonyl unit is tilted at an angle of 8.70 (8)° with respect to the benzene ring. In the independent molecules of 2 (Fig. 2), the planes passing through the ester units are inclined at angles of 62.9 (1) and 81.3 (1)°, respectively, to the plane of their arene ring. The two bromine atoms of each molecule are located on opposite sides of the benzene ring. In the crystal of the 5-hydroxybenzene-1,3-dicarboxaldehyde (3) (Fig. 3), the molecule deviates slightly from planarity, with the formyl groups rotated out of the benzene ring at angles of 4.43 (16) and 4.04 (16)°.

3. Supramolecular features

In the crystal structure of 1, the molecules are arranged into layers extending parallel to the crystallographic [101] plane (see Fig. 4). Within a given layer, the molecules are linked in strands via C—H···O=C bonds [$d(H \cdots O) 2.57 \text{ Å}$; Table 1], with a methyl H atom acting as the donor. No directional interactions are present between the molecular strands of a layer. With the participation of a H atom of the methyl ester unit, the linkage between the molecules of adjacent layers occurs by C—H···π contacts (Nishio et al., 2009) with a H···Cg distance of 2.77 Å. Fig. 5 shows a packing excerpt of the crystal structure viewed in the direction of the layer normal.

The excerpt of the crystal structure of 2 shown in Fig. 6 reveals two different inversion-symmetric dimers as the smallest supramolecular entities, in which the molecules are
linked in an identical manner by C—H···O=C and C—

H···Br bonds (Table 2) (Desiraju & Steiner, 1999). These
dimers, however, form differently structured domains within
the crystal. The dimers formed by molecule A are connected
via Br···Br bonds (Pedireddy et al., 1999) of type I [d(Br···Br) =
3.562 (1) Å; \( \theta_1 = 150.2^\circ; \theta_2 = 158.5^\circ \)] and of type II
[d(Br···Br) = 3.859 (1) Å; \( \theta_1 = 135.0^\circ; \theta_2 = 84.6^\circ \)] as well as

C—H···Br hydrogen bonds to form two-dimensional aggreg-
gates extending parallel to crystallographic [011] plane, in
which the bromine atoms contribute to the formation of a
cyclic four-membered synthon (Br₄) and an eight-membered
bonding motif (Fig. 7). The structure of the domains created
by molecule B is fundamentally different from those formed
by molecule A. In them, the dimers are linked in a strand-like
fashion via type 1 Br···Br interactions [d(Br···Br) =
3.638 (1) Å; \( \theta_1 = 152.3^\circ; \theta_2 = 145.9^\circ \)] (Fig. 7b), which are part of

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**Table 1**

Hydrogen-bond geometry (Å, °) for 1.

| D—H···A | D—H | H···A | D···A | D—H···A |
|--------|------|------|-------|---------|
| C10—H10B···O1' | 0.98 | 2.57 | 3.5215 (19) | 163 |
| C8—HB···Cg1'' | 0.98 | 2.76 | 3.445 (2) | 127 |

Symmetry codes: (i) \( x, -y, z \); (ii) \( x, y, z \); (iii) \( x, y, z \).

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**Table 2**

Hydrogen-bond geometry (Å, °) for 2.

| D—H···A | D—H | H···A | D···A | D—H···A |
|--------|------|------|-------|---------|
| C10A—H10D···O2A' | 0.97 | 2.28 | 3.236 (3) | 168 |
| C10A—H10C···Br1A' | 0.97 | 2.89 | 3.836 (3) | 164 |
| C8A—HB43···O2 | 0.96 | 2.58 | 3.521 (4) | 168 |
| C10—H10B···Br2A' | 0.97 | 3.01 | 3.757 (3) | 135 |
| C10—H10A···O2'' | 0.97 | 2.58 | 3.449 (3) | 150 |
| C9—HB9···Br2'' | 0.97 | 2.95 | 3.854 (3) | 156 |
| C9—HB4···O2'' | 0.97 | 2.45 | 3.334 (3) | 151 |

Symmetry codes: (i) \( x, -y, -z \); (ii) \( x, -y, -z, 1 \); (iii) \( -x, -y, -z \).

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**Table 3**

Hydrogen-bond geometry (Å, °) for 3.

| D—H···A | D—H | H···A | D···A | D—H···A |
|--------|------|------|-------|---------|
| C2—H2···O1' | 0.95 | 2.43 | 3.354 (16) | 160 |
| C8—HB···O2'' | 0.95 | 2.58 | 3.1973 (18) | 123 |
| O1—H1···O3'' | 0.85 (2) | 1.91 (2) | 2.6795 (13) | 150 (2) |

Symmetry codes: (i) \( -x, -y, -z \); (ii) \( -x, -y, -z, 1 \); (iii) \( -x, -y, -z, 1 \).

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an eight-membered ring motif. In the direction of the crystallographic a-axis, the connection of the dimers occurs through π−π (face-to-face) interactions (Tiekink & Zukerman-Schpector, 2012) with a centroid–centroid distance of 3.653 (1) Å and an offset of 1.592 Å between the interacting arene rings.

Viewing the crystal structure of compound 3 in the direction of the a-axis reveals a stacking arrangement of molecules (Fig. 8). Along the stacking axis the centroid-centroid distance of 3.735 (1) Å between consecutive molecules indicates the presence of offset π−π interactions. As is obvious from Fig. 9, showing the mode of non-covalent bonding in the crystal, the

![Figure 8](image)

**Figure 8**
Packing diagram of 3 viewed down the a-axis. Dashed lines represent hydrogen bonds.

![Figure 9](image)

**Figure 9**
Mode of intermolecular non-covalent interactions in the crystal structure of 3. The cyclic supramolecular synthons are marked by colour highlighting.

**Table 4**
Experimental details.

|   | 1                  | 2                  | 3                  |
|---|--------------------|--------------------|--------------------|
| Chemical data | C₈H₆O₃             | C₆H₆Br₂O₂           | C₆H₆O₃             |
| Mᵣ               | 164.20             | 322.00             | 150.13             |
| Crystal system, space group | Monoclinic, P₂₁/n | Triclinic, P̅T | Monoclinic, P₂₁/n |
| Temperature (K) | 153                | 130                | 153                |
| a, b, c (Å)      | 8.4631 (6), 7.9793 (4), 13.4042 (9) | 7.7936 (2), 9.1655 (2), 17.2292 (4) | 88.1637 (12), 80.9050 (12), 65.8659 (11) |
| α, β, γ (°)      | 90, 98.835 (6), 90 | 90, 98.835 (6), 90 | 90, 98.835 (6), 90 |
| V (Å³)           | 894.44 (10)        | 1108.30 (5)        | 671.64 (4)         |
| Z                | 4                  | 4                  | 4                  |
| μ (mm⁻¹)         | 0.08               | 7.29               | 12                 |
| Crystal size (mm) | 0.40 × 0.25 × 0.16 | 0.46 × 0.39 × 0.27 | 0.42 × 0.28 × 0.19 |

Data collection

| Diffractometer | Stoe IPDS 2T | Bruker Kappa APEXII CCD area detector | Bruker Kappa APEXII CCD area detector |
|----------------|-------------|-------------------------------------|-------------------------------------|
| Absorption correction | – | Multi-scan (SADABS; Bruker, 2014) | – |
| Tₘᵛₜ, Tₘᵚₓ | – | 0.134, 0.244 | – |
| No. of measured, independent and observed [I > 2σ(I)] reflections | 7437, 1762, 1449 | 29065, 5842, 5305 | 11533, 1819, 1519 |
| Rₓₓ, (sin θ/λ)max (Å⁻¹) | 0.046 | 0.033 | 0.058 |
| 0.617 | 0.680 | 0.691 |

Refinement

| R[F² > 2σ(F²)], wR(F²), S | 0.041, 0.116, 1.05 | 0.028, 0.070, 1.04 | 0.047, 0.131, 1.06 |
| No. of reflections | 1762 | 5842 | 1819 |
| No. of parameters | 112 | 255 | 104 |
| H-atom treatment | H-atom parameters constrained | H-atom parameters constrained | H atoms treated by a mixture of independent and constrained refinement |

Computer programs: X-AREA and X-RED (Stoe & Cie, 2002), APEX2 and SAINT (Bruker, 2014), SIR2014 (Burla et al., 2015), SHELXS97 (Sheldrick, 2008), SHELXL (Sheldrick, 2015), ShelXle (Harmschle et al., 2011), XP (Sheldrick, 2008), ORTEP-3 for Windows and WinGX (Farrugia, 2012) and publCIF (Westrip, 2010).
while crystals of were obtained by slow evaporation from a hexane solution, while its O atom forms a C—H...O bond [C2—H2...O1 = 2.43 Å, 159.6°; Table 2], thus creating a supramolecular synthon with the graph set R2(17) (Etter, 1990; Etter et al., 1990; Bernstein et al., 1995) in which four molecules take part. The OH group is also involved in formation of an inversion-symmetric ring motif of the structure R2(8). Another supramolecular motif corresponding to the R2(14) graph set is formed by the formyl groups of inversion-related molecules.

4. Database survey
A search in the Cambridge Structural Database (CSD, Version 5.43, update November 2021; Groom et al., 2016) for benzene derivatives containing the corresponding substituents resulted in several hits, but with relatively strong structural differences from the searched structures. The compound with the closest relation to 1 is ethyl 2,3,5,6-tetramethylbenzoate (FICVET; Pinkus et al., 2005), the crystal structure of which features C—H...O and C—H...π interactions. In the case of bromomethyl-substituted benzenes, the crystal structures of 1,2,4,5-tetrakis(bromomethyl)-3,6-dimethoxybenzene, 1,2,4,5-tetrakis(bromomethyl)-3,6-bis(hexyloxy)benzene and 1,2,4,5-tetrakis(bromomethyl)-3,6-bis(2-ethylbutyloxy)benzene (BASZIG, BASZOM, BASZUS; Velde et al. 2012) as well as 1,3,5-tris(bromomethyl)-2,4,6-trimethoxybenzene (IDOBag; Koch et al. 2013) are worth mentioning. The crystal structure of IDOBAG, for example, is characterized by the presence of C—H...O and C—H...Br hydrogen bonds as well as C—Br...Br halogen bonds of type II, as observed also in the crystal structure of 2. In the crystal structure of 2-hydroxy-isophthalaldehyde (NEJJOB; Zondervan et al. 1997), an analogue of 3, the molecules interact via O—H...O hydrogen bonds, forming chains. In addition, the hydroxy group is involved in an intramolecular O—H...O hydrogen bond with the neighbouring carbonyl oxygen atom.

5. Synthesis and crystallization
Compounds 1–3 were prepared according to literature procedures (Kurz & Göbel, 1996; Battaini et al., 2003; Star et al., 2003). Suitable crystals of compounds 2 and 3 for X-ray analysis were obtained by slow evaporation from a hexane solution, while crystals of 1 were grown from a subcooled melt.

6. Refinement
Crystal data, data collection and structure refinement details are summarized in Table 4. Hydrogen atom H1 in 3 was located in a difference-Fourier map and freely refined. Other H atoms were positioned geometrically and refined isotropically using a riding model with C—H = 0.93–0.98 Å and Uiso(H) = 1.2–1.5Ueq(C).

Acknowledgements
Open access funding by the Publication Fund of the Technische Universität Bergakademie Freiberg is gratefully acknowledged.

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Crystal structures of methyl 3,5-dimethylbenzoate, 3,5-bis(bromomethyl)phenyl acetate and 5-hydroxybenzene-1,3-dicarbaldehyde

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Computing details

Data collection: \textit{X-AREA} (Stoe \& Cie, 2002) for (1); \textit{APEX2} (Bruker, 2014) for (2), (3). Cell refinement: \textit{X-AREA} (Stoe \& Cie, 2002) for (1); \textit{SAINT} (Bruker, 2014) for (2), (3). Data reduction: \textit{X-RED} (Stoe \& Cie, 2002) for (1); \textit{SAINT} (Bruker, 2014) for (2), (3). Program(s) used to solve structure: \textit{SIR2014} (Burla et al., 2015) for (1); \textit{SHELXS97} (Sheldrick, 2008) for (2), (3). Program(s) used to refine structure: \textit{SHELXL} (Sheldrick, 2015) for (1), (2); \textit{SHELXL2014/7} (Sheldrick, 2015) for (3). Molecular graphics: \textit{XP} (Sheldrick, 2008) for (1); \textit{ORTEP-3 for Windows} (Farrugia, 2012) for (2), (3). Software used to prepare material for publication: \textit{WinGX} (Farrugia, 2012), \textit{publCIF} (Westrip, 2010), \textit{ShelXle} (Hübschle et al., 2011) for (1); \textit{SHELXTL} (Sheldrick, 2008) for (2), (3).

Methyl 3,5-dimethylbenzoate (1)

\textbf{Crystal data}

\begin{tabular}{ll}
C\textsubscript{10}H\textsubscript{12}O\textsubscript{2} & \\
M\textsubscript{r} = 164.20 & \\
Monoclinic, \textit{P}2\textsubscript{1}/\textit{n} & \\
a = 8.4631 (6) Å & \\
b = 7.9793 (4) Å & \\
c = 13.4042 (9) Å & \\
\(\beta = 98.835 (6)^\circ\) & \\
\(V = 894.44 (10) \text{ Å}^3\) & \\
Z = 4 & \\
\end{tabular}

\textbf{Data collection}

\begin{tabular}{ll}
Stoe IPDS 2T & 1762 independent reflections \\
diffractometer & 1449 reflections with \(I > 2\sigma(I)\) \\
Radiation source: sealed X-ray tube, 12 x 0.4 mm long-fine focus & \\
Plane graphite monochromator & \\
Detector resolution: 6.67 pixels mm\textsuperscript{-1} & \\
rotation method scans & \\
7437 measured reflections & \\
\end{tabular}

\textbf{Refinement}

\begin{tabular}{ll}
Refinement on \(F^2\) & 112 parameters \\
Least-squares matrix: full & 0 restraints \\
\(R[F^2 > 2\sigma(F^2)] = 0.041\) & Hydrogen site location: inferred from \\
\(wR(F^2) = 0.116\) & neighbouring sites \\
\(S = 1.05\) & H-atom parameters constrained \\
1762 reflections & \\
\end{tabular}
where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\text{max}} < 0.001$

**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 ($\AA^2$)**

|    | x          | y          | z          | $U_{\text{iso}}$/$U_{\text{eq}}$ |
|----|------------|------------|------------|-------------------------------|
| O1 | 0.27708 (14) | 0.33954 (14) | 0.48553 (8) | 0.0433 (3) |
| O2 | 0.20755 (12) | 0.58613 (12) | 0.54594 (7) | 0.0309 (3) |
| C1 | 0.12649 (14) | 0.34326 (16) | 0.62305 (9) | 0.0244 (3) |
| C2 | 0.10405 (16) | 0.16993 (17) | 0.62357 (10) | 0.0276 (3) |
| H2 | 0.1406 | 0.1027 | 0.5733 | 0.033* |
| C3 | 0.02860 (16) | 0.09507 (16) | 0.69720 (10) | 0.0283 (3) |
| C4 | −0.02303 (16) | 0.19629 (17) | 0.77063 (10) | 0.0279 (3) |
| H4 | −0.0747 | 0.1458 | 0.8211 | 0.033* |
| C5 | −0.00996 (15) | 0.36934 (17) | 0.77210 (10) | 0.0257 (3) |
| C6 | 0.07405 (15) | 0.44202 (17) | 0.69705 (10) | 0.0249 (3) |
| H6 | 0.0894 | 0.5599 | 0.6965 | 0.030* |
| C7 | 0.21123 (15) | 0.41859 (17) | 0.54403 (10) | 0.0271 (3) |
| C8 | 0.29088 (18) | 0.6711 (2) | 0.47431 (11) | 0.0361 (4) |
| H8A | 0.2808 | 0.7926 | 0.4822 | 0.054* |
| H8B | 0.2442 | 0.6387 | 0.4056 | 0.054* |
| H8C | 0.4042 | 0.6398 | 0.4866 | 0.054* |
| C9 | 0.00480 (19) | −0.09303 (17) | 0.69749 (12) | 0.0383 (4) |
| H9A | −0.0136 | −0.1291 | 0.7647 | 0.057* |
| H9B | 0.1005 | −0.1487 | 0.6805 | 0.057* |
| H9C | −0.0879 | −0.1231 | 0.6475 | 0.057* |
| C10 | −0.05669 (18) | 0.47866 (18) | 0.85204 (11) | 0.0333 (3) |
| H10A | 0.0340 | 0.5428 | 0.8870 | 0.050* |
| H10B | −0.1010 | 0.4080 | 0.9008 | 0.050* |
| H10C | −0.1392 | 0.5560 | 0.8201 | 0.050* |

**Atomic displacement parameters ($\AA^2$)**

|       | $U_{11}$ | $U_{22}$ | $U_{33}$ | $U_{12}$ | $U_{13}$ | $U_{23}$ |
|-------|----------|----------|----------|----------|----------|----------|
| O1    | 0.0488 (7) | 0.0417 (6) | 0.0461 (7) | 0.0006 (5) | 0.0280 (5) | −0.0072 (5) |
| O2    | 0.0345 (6) | 0.0299 (6) | 0.0308 (5) | −0.0021 (4) | 0.0126 (4) | 0.0040 (4) |
| C1    | 0.0207 (6) | 0.0273 (7) | 0.0253 (7) | 0.0015 (5) | 0.0035 (5) | −0.0001 (5) |
| C2    | 0.0262 (7) | 0.0260 (7) | 0.0300 (7) | 0.0039 (5) | 0.0025 (5) | −0.0042 (5) |
| C3    | 0.0269 (7) | 0.0232 (7) | 0.0329 (7) | 0.0006 (5) | −0.0012 (5) | 0.0017 (5) |
| C4    | 0.0283 (7) | 0.0284 (7) | 0.0265 (7) | −0.0026 (5) | 0.0023 (5) | 0.0046 (5) |
| C5    | 0.0249 (7) | 0.0274 (7) | 0.0247 (6) | 0.0005 (5) | 0.0034 (5) | −0.0001 (5) |
| C6    | 0.0245 (6) | 0.0221 (6) | 0.0280 (7) | 0.0004 (5) | 0.0040 (5) | 0.0004 (5) |
Geometric parameters (Å, °)

| Bond/Angle | Distance 1 | Distance 2 | Angle 1  | Angle 2  |
|------------|------------|------------|----------|----------|
| O1—C7      | 1.2073 (16) | C5—C6      | 1.3956 (18) |
| O2—C7      | 1.3375 (17) | C5—C10     | 1.5121 (18) |
| O2—C8      | 1.4448 (16) | C6—H6      | 0.9500   |
| C1—C6      | 1.3917 (18) | C8—H8A     | 0.9800   |
| C1—C2      | 1.3961 (19) | C8—H8B     | 0.9800   |
| C1—C7      | 1.4936 (17) | C8—H8C     | 0.9800   |
| C2—C3      | 1.3900 (19) | C9—H9A     | 0.9800   |
| C2—C2      | 0.9500     | C9—H9B     | 0.9800   |
| C3—C4      | 1.3944 (19) | C9—H9C     | 0.9800   |
| C3—C9      | 1.5144 (19) | C10—H10A   | 0.9800   |
| C4—C5      | 1.3931 (19) | C10—H10B   | 0.9800   |
| C4—H4      | 0.9500     | C10—H10C   | 0.9800   |
| C7—O2      | 116.20 (11) | O1—C7—C1   | 124.76 (13) |
| C6—C1      | 119.92 (12) | O2—C7—C1   | 111.94 (11) |
| C6—C1      | 121.19 (12) | O2—C8—H8A  | 109.5    |
| C2—C1      | 118.86 (12) | O2—C8—H8B  | 109.5    |
| C3—C2      | 120.46 (12) | H8A—C8—H8B | 109.5    |
| C3—C2      | 119.8       | O2—C8—H8C  | 109.5    |
| C1—C2      | 119.8       | H8A—C8—H8C | 109.5    |
| C2—C3      | 118.70 (12) | H8B—C8—H8C | 109.5    |
| C2—C3      | 120.23 (13) | C3—C9—H9A  | 109.5    |
| C4—C3      | 121.07 (13) | C3—C9—H9B  | 109.5    |
| C5—C4      | 121.89 (12) | H9A—C9—H9B | 109.5    |
| C5—C4      | 119.1       | C3—C9—H9C  | 109.5    |
| C3—C4      | 119.1       | H9A—C9—H9C | 109.5    |
| C4—C5      | 118.46 (12) | H9B—C9—H9C | 109.5    |
| C4—C5      | 121.74 (12) | C5—C10—H10A| 109.5    |
| C6—C5      | 119.80 (12) | C5—C10—H10B| 109.5    |
| C1—C6      | 120.57 (12) | H10A—C10—H10B| 109.5    |
| C1—C6      | 119.7       | C5—C10—H10C| 109.5    |
| C5—C6      | 119.7       | H10A—C10—H10C| 109.5    |
| O1—C7      | 123.30 (12) | H10B—C10—H10C| 109.5    |
| C6—C1      | 0.43 (19)   | C7—C1—C6—C5+| −178.18 (12) |
| C7—C1      | 178.69 (11) | C4—C5—C6—C1| −0.48 (19)  |
| C1—C2      | −0.42 (19)  | C10—C5—C6—C1| 179.82 (12) |
| C1—C2      | −179.91 (13) | C8—O2—C7—O1| −1.2 (2)     |
| C2—C3      | 0.0 (2)     | C8—O2—C7—C1| 178.09 (11)  |
| C9—C3      | 179.44 (13) | C6—C1—C7—O1| 170.41 (14)  |
| C3—C4      | 0.5 (2)     | C2—C1—C7—O1| −7.8 (2)      |
Hydrogen-bond geometry (Å, °)

Cg1 represents the centroid of the C1–C6 ring.

| D—H···A         | D—H   | H···A   | D···A    | D—H···A |
|-----------------|--------|---------|----------|---------|
| C10—H10B···O1i  | 0.98   | 2.57    | 3.5215 (19) | 163     |
| C8—H8B···Cg1ii  | 0.98   | 2.76    | 3.445 (2)  | 127     |

Symmetry codes: (i) x−1/2, −y+1/2, z+1/2; (ii) −x+1/2, y+3/2, −z+3/2.

3,5-Bis(bromomethyl)phenyl acetate (2)

Crystal data

C_{10}H_{10}Br_{2}O_{2}

Mr = 322.00

Triclinic, P\overline{1}

a = 7.7936 (2) Å

b = 9.1655 (2) Å

c = 17.2292 (4) Å

α = 88.1637 (12)°

β = 90.8050 (12)°

γ = 65.8659 (11)°

V = 1108.30 (5) Å³

Z = 4

F(000) = 624

D_{\text{c}} = 1.930 Mg m⁻³

Mo Kα radiation, λ = 0.71073 Å

Cell parameters from 9654 reflections

θ = 2.7–36.8°

µ = 7.29 mm⁻¹

T = 130 K

Irregular, colourless

0.46 × 0.39 × 0.27 mm

Data collection

Bruker Kappa APEXII CCD area detector diffractometer

φ and ω scans

Absorption correction: multi-scan

(SADABS; Bruker, 2014)

T_{\text{min}} = 0.134, T_{\text{max}} = 0.244

29065 measured reflections

5842 independent reflections

5305 reflections with I > 2σ(I)

R_{int} = 0.033

θ_{max} = 28.9°, θ_{min} = 1.2°

h = −10→10

k = −12→12

l = −23→22

Refinement

Refinement on F²

Least-squares matrix: full

R[F² > 2σ(F²)] = 0.028

wR(F²) = 0.070

S = 1.04

5842 reflections

255 parameters

0 restraints

Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained

w = 1/[σ²(Fc³) + (0.0273P)^2 + 2.052P]

where P = (Fc³ + 2Fc²)/3

(Δ/σ)_{max} = 0.001

Δρ_{max} = 1.21 e Å⁻³

Δρ_{min} = −0.98 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)

|      | x          | y          | z          | $U_{iso}^{*}/U_{eq}$ |
|------|------------|------------|------------|----------------------|
| Br1  | -0.08475(4)| 0.81672(3) | 0.00562(2) | 0.02904(7)           |
| Br2  | 0.48472(4) | 0.08778(3) | 0.11939(2) | 0.03302(8)           |
| O1   | 0.5485(3)  | 0.6991(2)  | 0.19580(10)| 0.0223(3)            |
| O2   | 0.8281(3)  | 0.4838(2)  | 0.16971(12)| 0.0300(4)            |
| C1   | 0.4550(3)  | 0.6259(3)  | 0.15806(14)| 0.0180(4)            |
| C2   | 0.3764(3)  | 0.7009(3)  | 0.09361(13)| 0.0177(4)            |
| H2   | 0.3906     | 0.7927     | 0.0756     | 0.021              |
| C3   | 0.2757(3)  | 0.6375(3)  | 0.05580(13)| 0.0168(4)           |
| C4   | 0.2561(3)  | 0.5002(3)  | 0.08395(14)| 0.0183(4)           |
| H4   | 0.1896     | 0.4570     | 0.0587     | 0.022              |
| C5   | 0.3346(3)  | 0.4268(3)  | 0.14936(14)| 0.0189(4)           |
| C6   | 0.4351(3)  | 0.4903(3)  | 0.18721(14)| 0.0190(4)           |
| H6   | 0.4879     | 0.4425     | 0.2312     | 0.023              |
| C7   | 0.7388(4)  | 0.6174(3)  | 0.19617(14)| 0.0204(4)           |
| C8   | 0.8159(4)  | 0.7190(3)  | 0.23218(15)| 0.0262(5)           |
| H8A  | 0.9257     | 0.6518     | 0.2548     | 0.039              |
| H8B  | 0.7203     | 0.7887     | 0.2725     | 0.039              |
| H8C  | 0.8515     | 0.7817     | 0.1925     | 0.039              |
| C9   | 0.1944(3)  | 0.7141(3)  | -0.01535(14)| 0.0221(5)         |
| H9A  | 0.2349     | 0.6337     | -0.0575    | 0.026              |
| H9B  | 0.2432     | 0.7937     | -0.0326    | 0.026              |
| C10  | 0.3063(4)  | 0.2828(3)  | 0.18047(17)| 0.0258(5)           |
| H10A | 0.1767     | 0.2972     | 0.1783     | 0.031              |
| H10B | 0.3249     | 0.2712     | 0.2351     | 0.031              |
| Br1A | 0.43346(3) | 0.44424(3) | 0.61006(2) | 0.02337(6)          |
| Br2A | 0.92345(4) | -0.40729(3)| 0.60976(2) | 0.02744(7)          |
| O1A  | 0.9262(3)  | 0.0059(2)  | 0.34359(11)| 0.0285(4)           |
| O2A  | 0.6523(3)  | 0.1522(3)  | 0.30204(12)| 0.0443(6)           |
| C1A  | 0.8337(3)  | 0.0113(3)  | 0.42092(14)| 0.0200(4)           |
| C2A  | 0.7921(3)  | 0.1409(3)  | 0.47025(16)| 0.0219(5)           |
| H2A  | 0.8118     | 0.2296     | 0.4509     | 0.026              |
| C3A  | 0.7203(3)  | 0.1375(3)  | 0.54912(15)| 0.0210(5)           |
| C4A  | 0.6907(3)  | 0.0042(3)  | 0.57655(14)| 0.0192(4)           |
| H4A  | 0.6413     | 0.0022     | 0.6292     | 0.023              |
| C5A  | 0.7340(3)  | -0.1266(3) | 0.52649(13)| 0.0171(4)           |
| C6A  | 0.8055(3)  | -0.1220(3) | 0.44763(13)| 0.0178(4)           |
| H6A  | 0.8340     | -0.2079    | 0.4133     | 0.021              |
| C7A  | 0.8205(4)  | 0.0866(3)  | 0.28849(14)| 0.0219(5)           |
| C8A  | 0.9420(4)  | 0.0791(4)  | 0.21112(16)| 0.0317(6)           |
| H8A1 | 0.9001     | 0.0369     | 0.1712     | 0.048              |
| H8A2 | 1.0722     | 0.0109     | 0.2145     | 0.048              |
| H8A3 | 0.9317     | 0.1846     | 0.1980     | 0.048              |
| C9A  | 0.6879(4)  | 0.2709(3)  | 0.60537(19)| 0.0323(6)           |
| H9A1 | 0.7829     | 0.3134     | 0.5893     | 0.039              |
| H9A2 | 0.7036     | 0.2291     | 0.6574     | 0.039              |
### Atomic displacement parameters (Å²)

|      | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{32}$ | $U^{33}$ | $U^{23}$ |
|------|----------|----------|----------|----------|----------|----------|
| Br1  | 0.02417 (13) | 0.02849 (13) | 0.03098 (14) | -0.00553 (10) | -0.01021 (10) | 0.00568 (10) |
| Br2  | 0.03039 (14) | 0.01534 (12) | 0.05357 (18) | -0.00887 (10) | -0.00889 (12) | 0.00249 (11) |
| O1   | 0.0273 (9) | 0.0173 (8) | 0.0254 (9) | -0.0095 (7) | -0.0116 (7) | 0.0016 (7) |
| O2   | 0.0238 (9) | 0.0277 (10) | 0.0385 (11) | -0.0103 (8) | -0.0035 (8) | -0.0084 (8) |
| C1   | 0.0183 (10) | 0.0160 (10) | 0.0198 (11) | -0.0063 (8) | -0.0051 (8) | -0.0011 (8) |
| C2   | 0.0205 (10) | 0.0141 (10) | 0.0182 (10) | -0.0069 (8) | -0.0028 (8) | 0.0009 (8) |
| C3   | 0.0170 (10) | 0.0163 (10) | 0.0139 (10) | -0.0042 (8) | -0.0005 (8) | -0.0014 (8) |
| C4   | 0.0159 (10) | 0.0169 (10) | 0.0218 (11) | -0.0068 (8) | -0.0020 (8) | -0.0019 (8) |
| C5   | 0.0152 (10) | 0.0162 (10) | 0.0228 (11) | -0.0055 (8) | 0.0010 (8) | 0.0012 (8) |
| C6   | 0.0194 (10) | 0.0174 (10) | 0.0186 (10) | -0.0056 (8) | -0.0049 (8) | 0.0037 (8) |
| C7   | 0.0248 (11) | 0.0231 (11) | 0.0166 (10) | -0.0126 (9) | -0.0045 (9) | 0.0028 (9) |
| C8   | 0.0337 (13) | 0.0302 (13) | 0.0228 (12) | -0.0199 (11) | -0.0081 (10) | 0.0016 (10) |
| C9   | 0.0238 (11) | 0.0252 (12) | 0.0163 (11) | -0.0088 (9) | -0.0040 (9) | 0.0007 (9) |
| C10  | 0.0232 (12) | 0.0225 (12) | 0.0330 (13) | -0.0118 (10) | -0.0019 (10) | 0.0063 (10) |
| Br1A | 0.02379 (12) | 0.01843 (11) | 0.01960 (11) | -0.00036 (9) | -0.00319 (9) | 0.00021 (9) |
| Br2A | 0.02581 (13) | 0.02373 (13) | 0.03068 (14) | -0.00786 (10) | -0.00669 (10) | 0.00987 (10) |
| O1A  | 0.0182 (8) | 0.0409 (11) | 0.0200 (9) | -0.0068 (8) | -0.0022 (7) | 0.0120 (8) |
| O2A  | 0.0272 (10) | 0.0641 (15) | 0.0212 (10) | 0.0028 (10) | -0.0076 (8) | 0.0062 (10) |
| C1A  | 0.0124 (9) | 0.0250 (11) | 0.0187 (11) | -0.0037 (8) | -0.0039 (8) | 0.0067 (9) |
| C2A  | 0.0141 (10) | 0.0168 (10) | 0.0339 (13) | -0.0041 (8) | -0.0091 (9) | 0.0083 (9) |
| C3A  | 0.0131 (10) | 0.0183 (11) | 0.0285 (12) | -0.0013 (8) | -0.0081 (9) | -0.0012 (9) |
| C4A  | 0.0136 (10) | 0.0228 (11) | 0.0175 (10) | -0.0032 (8) | -0.0035 (8) | -0.0003 (8) |
| C5A  | 0.0116 (9) | 0.0193 (10) | 0.0195 (11) | -0.0048 (8) | -0.0047 (8) | 0.0027 (8) |
| C6A  | 0.0144 (9) | 0.0192 (10) | 0.0180 (10) | -0.0041 (8) | -0.0051 (8) | 0.0000 (8) |
| C7A  | 0.0280 (12) | 0.0197 (11) | 0.0194 (11) | -0.0101 (10) | -0.0076 (9) | 0.0039 (9) |
| C8A  | 0.0399 (15) | 0.0364 (15) | 0.0227 (13) | -0.0211 (13) | -0.0022 (11) | 0.0086 (11) |
| C9A  | 0.0200 (12) | 0.0241 (13) | 0.0471 (17) | -0.0002 (10) | -0.0115 (11) | -0.0131 (12) |
| C10A | 0.0176 (10) | 0.0240 (12) | 0.0267 (12) | -0.0091 (9) | -0.0050 (9) | 0.0045 (9) |

### Geometric parameters (Å, °)

|      |      |      |      |      |      |      |
|------|------|------|------|------|------|------|
| Br1—C9 | 1.962 (2) | Br1A—C9A | 1.960 (3) |
| Br2—C10 | 1.965 (3) | Br2A—C10A | 1.979 (2) |
| O1—C7 | 1.362 (3) | O1A—C7A | 1.353 (3) |
| O1—C1 | 1.407 (3) | O1A—C1A | 1.403 (3) |
| O2—C7 | 1.196 (3) | O2A—C7A | 1.184 (3) |
| C1—C2 | 1.379 (3) | C1A—C2A | 1.379 (4) |
| C1—C6 | 1.383 (3) | C1A—C6A | 1.380 (3) |
| C2—C3 | 1.392 (3) | C2A—C3A | 1.390 (4) |
| C2—H2 | 0.9300 | C2A—H2A | 0.9300 |
| C3—C4 | 1.392 (3) | C3A—C4A | 1.389 (3) |
| Bond                  | Distance (Å) |
|-----------------------|--------------|
| C3—C9                 | 1.492 (3)    |
| C4—C5                 | 1.389 (3)    |
| C4—H4                 | 0.9300       |
| C5—C6                 | 1.391 (3)    |
| C5—C10                | 1.495 (3)    |
| C6—H6                 | 0.9300       |
| C7—C8                 | 1.492 (3)    |
| C8—H8A                | 0.9600       |
| C8—H8B                | 0.9600       |
| C9—H9A                | 0.9700       |
| C9—H9B                | 0.9700       |
| C10—H10A              | 0.9700       |
| C10—H10B              | 0.9700       |
| C7—O1—C1              | 118.16 (18)  |
| C2—C1—C6              | 122.2 (2)    |
| C2—C1—O1              | 116.6 (2)    |
| C6—C1—O1              | 121.1 (2)    |
| C1—C2—C3              | 119.2 (2)    |
| C1—C2—H2              | 120.4        |
| C3—C2—H2              | 120.4        |
| C4—C3—C2              | 119.3 (2)    |
| C4—C3—C9              | 120.7 (2)    |
| C2—C3—C9              | 120.0 (2)    |
| C5—C4—C3              | 120.8 (2)    |
| C5—C4—H4              | 119.6        |
| C3—C4—H4              | 119.6        |
| C4—C5—C6              | 119.9 (2)    |
| C4—C5—C10             | 120.1 (2)    |
| C6—C5—C10             | 120.0 (2)    |
| C1—C6—H6              | 120.7        |
| C5—C6—H6              | 120.7        |
| O2—C7—O1              | 123.3 (2)    |
| O2—C7—C8              | 126.2 (2)    |
| O1—C7—C8              | 110.5 (2)    |
| C7—C8—H8A             | 109.5        |
| C7—C8—H8B             | 109.5        |
| H8A—C8—H8B            | 109.5        |
| C7—C8—H8C             | 109.5        |
| H8A—C8—H8C            | 109.5        |
| C3—C9—Br1             | 111.76 (16)  |
| C3—C9—H9A             | 109.3        |
| Br1—C9—H9A            | 109.3        |
| C3—C9—H9B             | 109.3        |
| Br1—C9—H9B            | 109.3        |
Hydrogen-bond geometry (Å, °)

| D—H···A | D—H  | H···A  | D···A  | D—H···A |
|---------|-------|--------|--------|---------|
| C10A—H10D···O2 4i | 0.97  | 2.28   | 3.236 (3) | 168     |
| C10A—H10C···Br1 4i | 0.97  | 2.89   | 3.836 (3) | 164     |
| C8A—H8A···O2 4i | 0.96  | 2.58   | 3.521 (4) | 168     |
| C10—H10B···Br2 4i | 0.97  | 3.01   | 3.757 (3) | 135     |
| C10—H10A···O2 4ii | 0.97  | 2.58   | 3.449 (3) | 150     |
| C9—H9B···Br2 3iii | 0.97  | 2.95   | 3.854 (3) | 156     |
| C9—H9A···O2 3iii | 0.97  | 2.45   | 3.334 (3) | 151     |

Symmetry codes: (i) x+1, y, z; (ii) x, y, z; (iii) x-1, y+1, z.

5-Hydroxybenzene-1,3-dicarbaldehyde (3)

Crystal data

| C₈H₆O₃ | b = 11.9549 (4) Å |
|        | M₀ = 150.13 |
| Monoclinic, P2₁/n | c = 15.0846 (5) Å |
| a = 3.7345 (1) Å | β = 94.212 (2)° |
| V = 671.64 (4) Å³ |

Acta Cryst. (2022), E78, 682-686
$Z = 4$

$F(000) = 312$

$D_x = 1.485 \text{ Mg m}^{-3}$

Mo $K\alpha$ radiation, $\lambda = 0.71073 \text{ Å}$

Cell parameters from 6158 reflections

$\theta = 2.7–30.5^\circ$

$\mu = 0.12 \text{ mm}^{-1}$

$T = 153 \text{ K}$

Rod, colourless

$0.42 \times 0.28 \times 0.19 \text{ mm}$

**Data collection**

Bruker Kappa APEXII CCD area detector

$\phi$ and $\omega$ scans

11533 measured reflections

1819 independent reflections

1519 reflections with $I > 2\sigma(I)$

$R_{int} = 0.058$

$\theta_{\text{max}} = 29.4^\circ$, $\theta_{\text{min}} = 2.7^\circ$

$h = -5 \rightarrow 4$

$k = -16 \rightarrow 16$

$l = -20 \rightarrow 20$

Refinement

Refinement on $F^2$

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.047$

$wR(F^2) = 0.131$

$S = 1.06$

1819 reflections

104 parameters

0 restraints

Hydrogen site location: mixed

H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0692PF)^2 + 0.2868P]$

where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\text{max}} < 0.001$

$\Delta\rho_{\text{max}} = 0.33 \text{ e Å}^{-3}$

$\Delta\rho_{\text{min}} = -0.28 \text{ e Å}^{-3}$

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ($\text{Å}^2$)

|     | x         | y         | z         | $U_{\text{iso}}/U_{eq}$ |
|-----|-----------|-----------|-----------|-------------------------|
| O1  | 0.6355 (3) | 0.48950 (8) | 0.38804 (7) | 0.0332 (3) |
| O2  | 1.0631 (3) | 0.10838 (8) | 0.62211 (7) | 0.0336 (3) |
| O3  | 0.2117 (3) | 0.11521 (8) | 0.23777 (6) | 0.0291 (3) |
| C1  | 0.6468 (4) | 0.37999 (10) | 0.40378 (8) | 0.0214 (3) |
| C2  | 0.8207 (3) | 0.34507 (10) | 0.48515 (8) | 0.0207 (3) |
| H2  | 0.9189     | 0.4000     | 0.5254     | 0.025*     |
| C3  | 0.8496 (3) | 0.23282 (10) | 0.50697 (7) | 0.0197 (3) |
| C4  | 0.7080 (4) | 0.15111 (10) | 0.44830 (8) | 0.0214 (3) |
| H4  | 0.7294     | 0.0740     | 0.4631     | 0.026*     |
| C5  | 0.5354 (3) | 0.18440 (10) | 0.36798 (8) | 0.0206 (3) |
| C6  | 0.5036 (3) | 0.29757 (10) | 0.34512 (8) | 0.0204 (3) |
| H6  | 0.3850     | 0.3191     | 0.2899     | 0.024*     |
| C7  | 1.0363 (4) | 0.20285 (11) | 0.59351 (8) | 0.0235 (3) |
| H7  | 1.1419     | 0.2615     | 0.6290     | 0.028*     |
| C8  | 0.3862 (4) | 0.09684 (11) | 0.30752 (9) | 0.0253 (3) |
| H8  | 0.4289     | 0.0210     | 0.3240     | 0.030*     |
| H1  | 0.519 (7)  | 0.5065 (19) | 0.3394 (16) | 0.056 (7)* |
Atomic displacement parameters ($\AA^2$)

|    | $U^{11}$  | $U^{22}$  | $U^{33}$  | $U^{12}$  | $U^{13}$  | $U^{23}$  |
|----|-----------|-----------|-----------|-----------|-----------|-----------|
| O1 | 0.0477 (7)| 0.0190 (5)| 0.0297 (5)| −0.0017 (4)| −0.0184 (5)| 0.0031 (4) |
| O2 | 0.0445 (7)| 0.0281 (5)| 0.0269 (5)| 0.0034 (4) | −0.0066 (4)| 0.0052 (4) |
| O3 | 0.0329 (6)| 0.0298 (5)| 0.0233 (5)| −0.0024 (4)| −0.0071 (4)| −0.0047 (4)|
| C1 | 0.0228 (7)| 0.0204 (6)| 0.0203 (5)| −0.0004 (4)| −0.0036 (4)| 0.0005 (4) |
| C2 | 0.0214 (7)| 0.0217 (6)| 0.0183 (5)| −0.0005 (4)| −0.0034 (4)| −0.0010 (4)|
| C3 | 0.0184 (6)| 0.0228 (6)| 0.0175 (5)| 0.0005 (4) | −0.0012 (4)| 0.0005 (4) |
| C4 | 0.0227 (7)| 0.0206 (5)| 0.0204 (5)| 0.0000 (4) | −0.0011 (4)| 0.0002 (4) |
| C5 | 0.0193 (6)| 0.0234 (6)| 0.0187 (5)| −0.0008 (4)| −0.0011 (4)| −0.0025 (4)|
| C6 | 0.0194 (6)| 0.0236 (6)| 0.0175 (5)| −0.0006 (4)| −0.0023 (4)| −0.0003 (4)|
| C7 | 0.0248 (7)| 0.0257 (6)| 0.0195 (5)| 0.0022 (5) | −0.0027 (4)| 0.0006 (4) |
| C8 | 0.0267 (7)| 0.0248 (6)| 0.0236 (6)| −0.0025 (5)| −0.0026 (5)| −0.0026 (5)|

Geometric parameters ($\AA$, °)

|    |    |    |    |    |    |    |
|----|----|----|----|----|----|----|
| O1—C1 | 1.3541 (15) | C3—C7 | 1.4781 (16) |
| O1—H1 | 0.85 (2) | C4—C5 | 1.3882 (16) |
| O2—C7 | 1.2105 (16) | C4—H4 | 0.9500 |
| O3—C8 | 1.2163 (16) | C5—C6 | 1.3991 (17) |
| C1—C6 | 1.3870 (16) | C5—C8 | 1.4709 (17) |
| C1—C2 | 1.4022 (16) | C6—C7 | 0.9500 |
| C2—C3 | 1.3840 (17) | C7—H7 | 0.9500 |
| C2—H2 | 0.9500 | C8—H8 | 0.9500 |
| C3—C4 | 1.3958 (16) | C4—C5—C6 | 121.19 (11) |
| C1—O1—H1 | 113.2 (16) | C4—C5—C8 | 117.88 (11) |
| C1—C1—C2 | 124.40 (11) | C6—C5—C8 | 120.92 (11) |
| C6—C1—C2 | 115.87 (11) | C1—C6—C5 | 119.43 (11) |
| C3—C2—C1 | 119.73 (11) | C1—C6—H6 | 120.3 |
| C3—C2—H2 | 119.9 | C5—C6—H6 | 120.3 |
| C1—C2—H2 | 119.9 | O2—C7—C3 | 124.21 (12) |
| C2—C3—C4 | 120.56 (11) | O2—C7—H7 | 117.9 |
| C2—C3—C7 | 117.95 (11) | C3—C7—H7 | 117.9 |
| C4—C3—C7 | 121.49 (11) | O3—C8—C5 | 124.23 (12) |
| C5—C4—C3 | 118.86 (11) | O3—C8—H8 | 117.9 |
| C5—C4—H4 | 120.6 | C5—C8—H8 | 117.9 |
| C3—C4—H4 | 120.6 | O1—C1—C6—C5 | 179.13 (13) |
| C6—C1—C2—C3 | −179.31 (12) | C2—C1—C6—C5 | −0.1 (2) |
| C1—C2—C3—C4 | 0.0 (2) | C4—C5—C6—C1 | −0.1 (2) |
| C1—C2—C3—C7 | 179.88 (12) | C8—C5—C6—C1 | 179.87 (12) |
| C2—C3—C4—C5 | −0.5 (2) | C2—C3—C7—O2 | 176.10 (14) |
| C7—C3—C4—C5 | 179.96 (12) | C4—C3—C7—O2 | −4.3 (2) |
| C3—C4—C5—C6 | 0.3 (2) | C4—C5—C8—O3 | 175.61 (14) |
C3—C4—C5—C8  \(-179.58 (12)\)  C6—C5—C8—O3  \(-4.3 (2)\)

| D—H···A | D—H | H···A | D···A    | D—H···A |
|---------|------|------|----------|---------|
| C2—H2···O1\(^i\) | 0.95 | 2.43 | 3.3354 (16) | 160 |
| C8—H8···O2\(^a\) | 0.95 | 2.58 | 3.1973 (18) | 123 |
| O1—H1···O3\(^iii\) | 0.85 (2) | 1.91 (2) | 2.6795 (13) | 150 (2) |

Symmetry codes: (i) \(-x+2, -y+1, -z+1\); (ii) \(-x+1, -y, -z+1\); (iii) \(-x+1/2, y+1/2, -z+1/2\).