research communications

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

Ben Ebersbach, Wilhelm Seichter and Monika Mazik*

Technische Universität Bergakademie Freiberg, Leipziger Str. 29, D-09596 Freiberg/Sachsen, Germany. *Correspondence e-mail: monika.mazik@chemie.tu-freiberg.de

The crystal structures of the title compounds, methyl 3,5-dimethylbenzoate ($C_{10}H_{12}O_2$; **1**), 3,5-bis(bromomethyl)phenyl acetate ($C_{10}H_{10}Br_2O_2$; **2**) and 5-hydroxybenzene-1,3-dicarbaldehyde ($C_8H_6O_3$; **3**) were determined by single-crystal X-ray analysis. The crystals of **1** are composed of strands of C—H$\cdots$O=C bonded molecules, which are further arranged into layers. As a result of the presence of two bromomethyl substituents in compound **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$\cdots$Br bonds of type I and type II. In the case of compound **3**, which possesses three donor/acceptor substituents, the molecular association in the crystal creates a close three-dimensional network comprising C_{aryl}—H$\cdots$O$_{hydroxy}$, C_{formyl}—H$\cdots$O$_{formyl}$ and O—H$\cdots$O$_{formyl}$ 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-di-carbaldehydes 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.


**1**   **2**   **3**
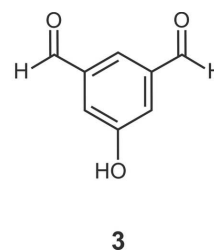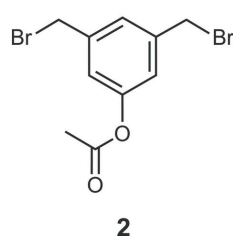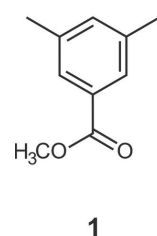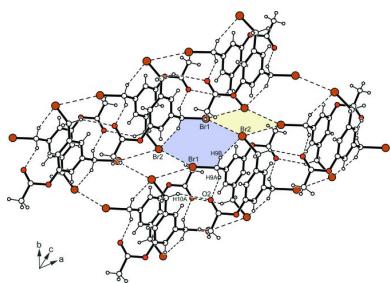
2. Structural commentary

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 $P\bar{1}$ 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-dicarbalde-hyde (**3**) (Fig. 3), the molecule deviates slightly from planarity,
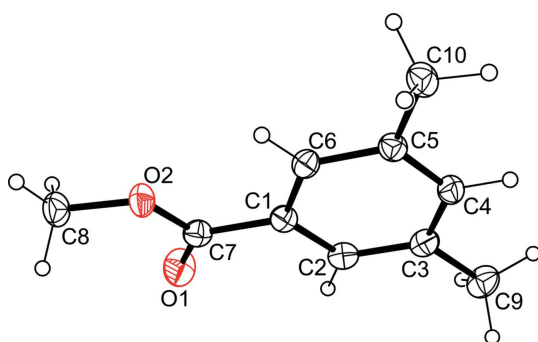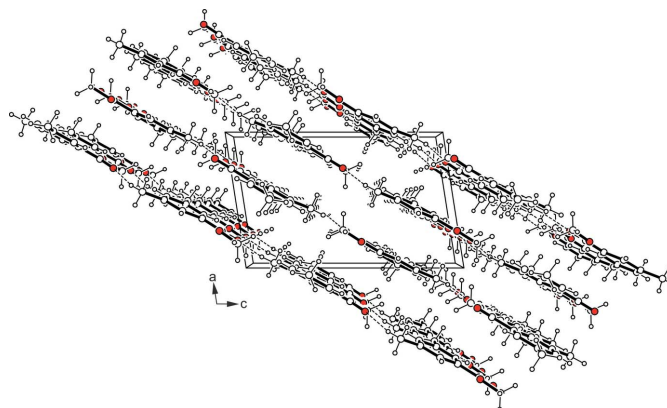

Figure 1
Perspective view of the molecular structure of **1**. Anisotropic displacement ellipsoids are drawn at the 50% probability level.
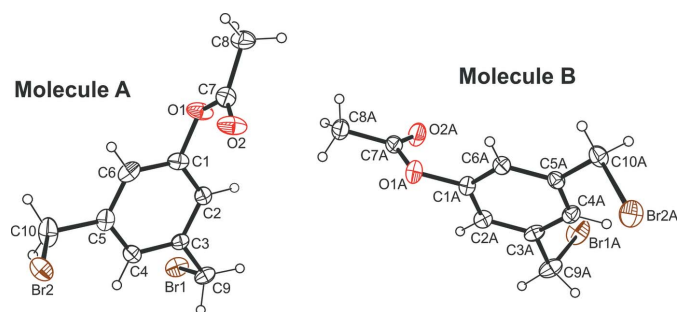
Figure 2
Perspective view of the molecular structure of **2**. Anisotropic displacement ellipsoids are drawn at the 50% probability level.
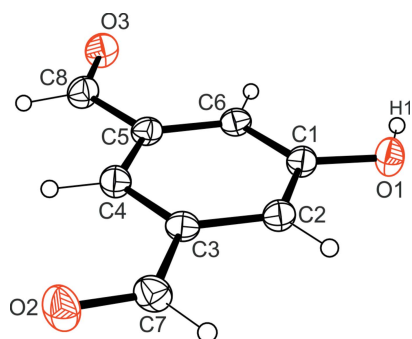
Figure 3
Perspective view of the molecular structure of **3**. Anisotropic displacement ellipsoids are drawn at the 50% probability level.

Figure 4
Packing diagram of **1** viewed down the crystallographic *b*-axis.

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⋯O) 2.57 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
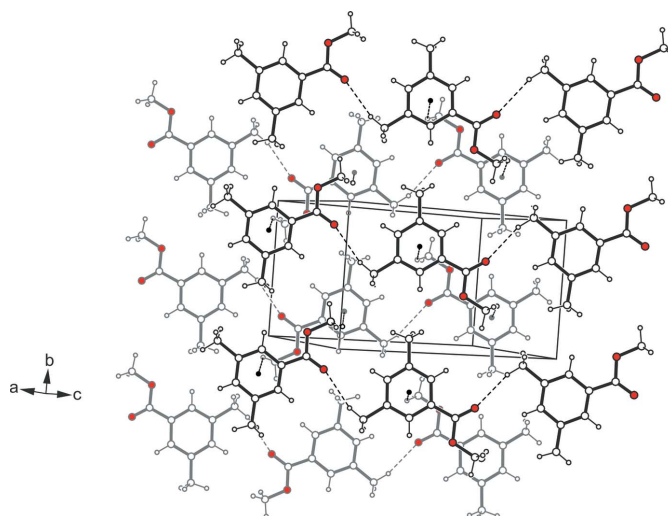
Figure 5
Excerpt of the packing structure of **1** viewed in the direction of the layer normal. Dashed lines represent hydrogen-bonding interactions.
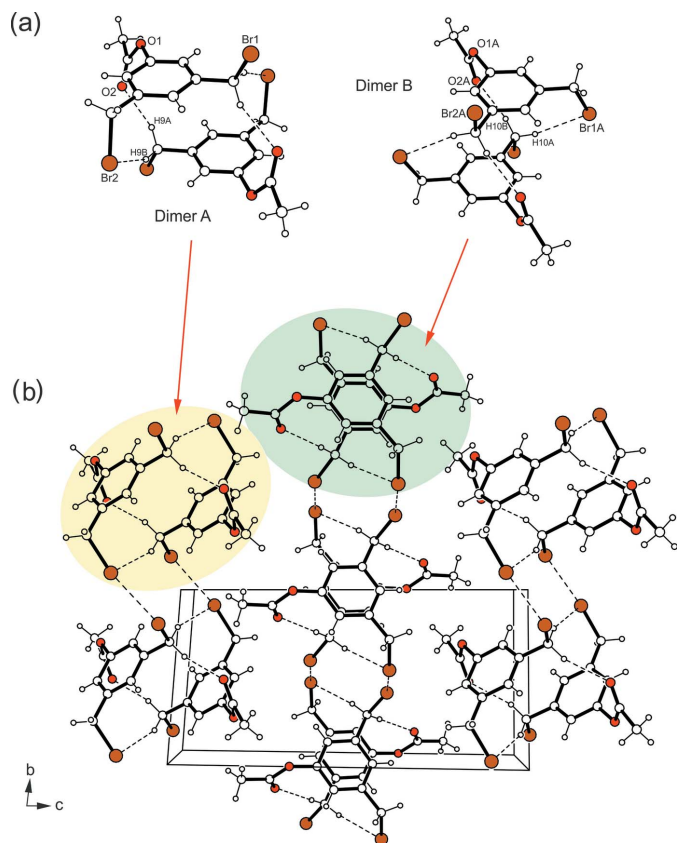
Figure 6
(*a*) Structures of the dimers formed by molecule *A* (left) and molecule *B* (right) in the crystal structure of **2**. (*b*) Packing structure of **2** viewed down the *a*-axis. Hydrogen bonds and Br···Br interactions are shown as dashed lines.

Table 1
Hydrogen-bond geometry (Å, °) for **1**.

*Cg*1 represents the centroid of the C1–C6 ring.

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
C10—H10B···O1^i	0.98	2.57	3.5215 (19)	163
C8—H8B···Cg1^{ii}	0.98	2.76	3.445 (2)	127

Symmetry codes: (i) $x - \frac{1}{2}, -y + \frac{1}{2}, z + \frac{1}{2}$; (ii) $-x + \frac{1}{2}, y + \frac{3}{2}, -z + \frac{3}{2}$.

Table 2
Hydrogen-bond geometry (Å, °) for **2**.

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
C10A—H10D···O2A^i	0.97	2.28	3.236 (3)	168
C10A—H10C···Br1A^i	0.97	2.89	3.836 (3)	164
C8A—H8A3···O2	0.96	2.58	3.521 (4)	168
C10—H10B···Br2A^i	0.97	3.01	3.757 (3)	135
C10—H10A···O2^{ii}	0.97	2.58	3.449 (3)	150
C9—H9B···Br2^{iii}	0.97	2.95	3.854 (3)	156
C9—H9A···O2^{iii}	0.97	2.45	3.334 (3)	151

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

Table 3
Hydrogen-bond geometry (Å, °) for **3**.

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
C2—H2···O1^i	0.95	2.43	3.3354 (16)	160
C8—H8···O2^{ii}	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) $-x + \frac{1}{2}, y + \frac{1}{2}, -z + \frac{1}{2}$.

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; $\theta_1 = 150.2°$, $\theta_2 = 158.5°$] and of type II [*d*(Br···Br) = 3.859 (1) Å; $\theta_1 = 135.0°$, $\theta_2 = 84.6°$] as well as

C—H···Br hydrogen bonds to form two-dimensional aggregates extending parallel to crystallographic [011] plane, in which the bromine atoms contribute to the formation of a cyclic four-membered synthon (Br$_4$) and an eight-membered bonding motif (Fig. 7*a*). 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 I Br···Br interactions [*d*(Br···Br) = 3.638 (1) Å; $\theta_1 = 152.3°$, $\theta_2 = 145.9°$] (Fig. 7*b*), which are part of
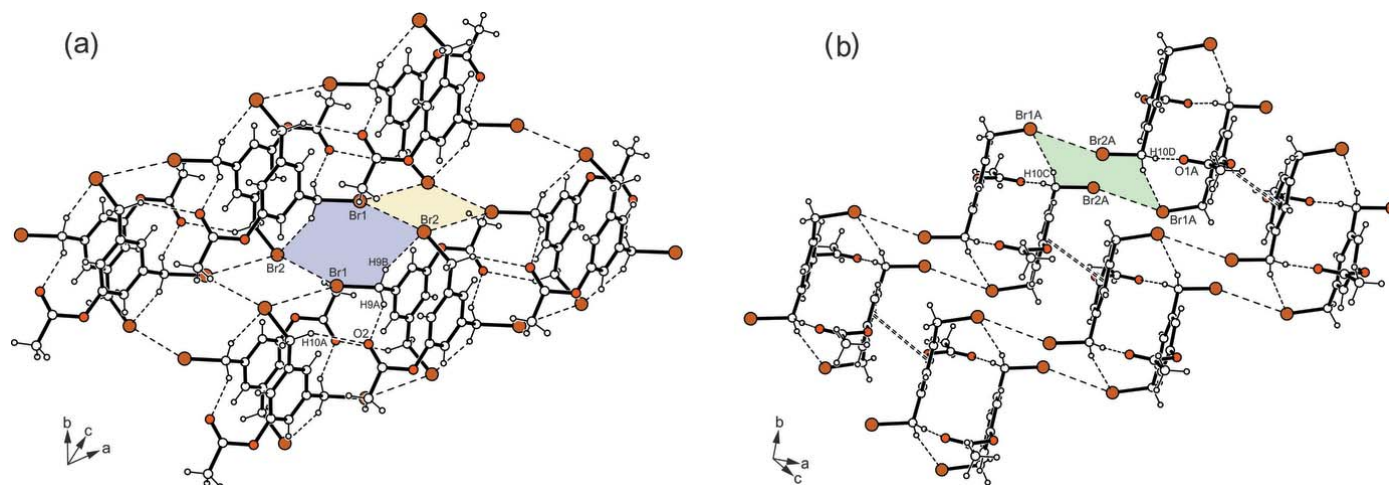
Figure 7
Patterns of intermolecular interactions created by (*a*) molecule *A* and (*b*) molecule *B* in the crystal structure of **2**.

Table 4
Experimental details.

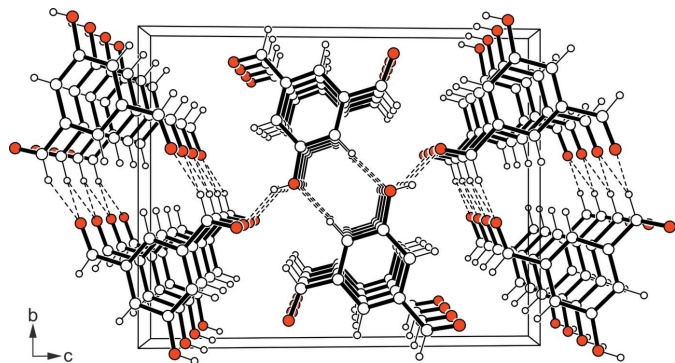|	1	2	3
Crystal data			
Chemical formula	$C_{10}H_{12}O_2$	$C_{10}H_{10}Br_2O_2$	$C_8H_6O_3$
M_r	164.20	322.00	150.13
Crystal system, space group	Monoclinic, $P2_1/n$	Triclinic, $P\bar{1}$	Monoclinic, $P2_1/n$
Temperature (K)	153	130	153
a, b, c (Å)	8.4631 (6), 7.9793 (4), 13.4042 (9)	7.7936 (2), 9.1655 (2), 17.2292 (4)	3.7345 (1), 11.9549 (4), 15.0846 (5)
α, β, γ (°)	90, 98.835 (6), 90	88.1637 (12), 80.9050 (12), 65.8659 (11)	90, 94.212 (2), 90
V (Å^3)	894.44 (10)	1108.30 (5)	671.64 (4)
Z	4	4	4
Radiation type	Mo $K\alpha$	Mo $K\alpha$	Mo $K\alpha$
μ (mm^{-1})	0.08	7.29	0.12
Crystal size (mm)	$0.40 \times 0.25 \times 0.16$	$0.46 \times 0.39 \times 0.27$	$0.42 \times 0.28 \times 0.19$
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)	–
T_{min}, T_{max}	–	0.134, 0.244	–
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	7437, 1762, 1449	29065, 5842, 5305	11533, 1819, 1519
R_{int}	0.046	0.033	0.058
$(\sin\theta/\lambda)_{max}$ (Å^{-1})	0.617	0.680	0.691
Refinement			
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, 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
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.24, −0.19	1.21, −0.98	0.33, −0.28

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* (Hübschle *et al.*, 2011), *XP* (Sheldrick, 2008), *ORTEP-3 for Windows* and *WinGX* (Farrugia, 2012) and *publCIF* (Westrip, 2010).

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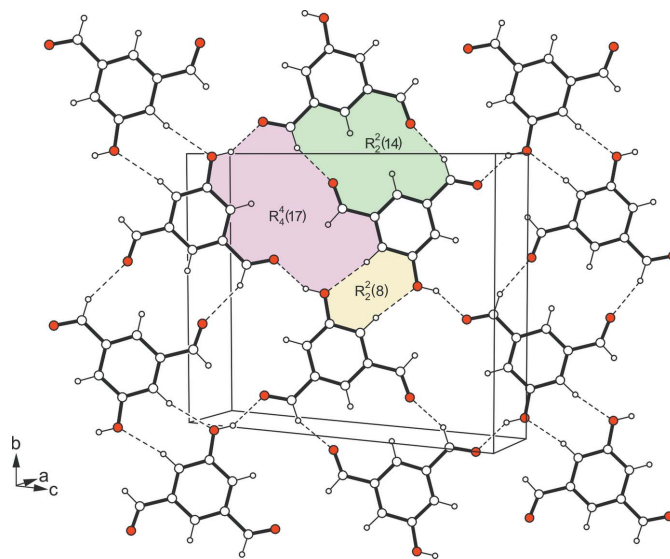
Figure 9
Mode of intermolecular non-covalent interactions in the crystal structure of **3**. The cyclic supramolecular synthons are marked by colour highlighting.

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

H atom of the hydroxy group forms an intermolecular O—H⋯O bond [O1—H1⋯O3 = 1.91 (2) Å, 150 (2)°; Table 3], while its O atom forms a C—H⋯O bond [C2—H2⋯O1 = 2.43 Å, 159.6°; Table 3], thus creating a supramolecular synthon with the graph set $R_4^4(17)$ (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 $R_2^2(8)$. Another supramolecular motif corresponding to the $R_2^2(14)$ 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 derivates 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-ethylbutoxy)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 $U_{iso}(H) = 1.2$–$1.5U_{eq}(C)$.

References

Amrhein, F., Lippe, J. & Mazik, M. (2016). *Org. Biomol. Chem.* **14**, 10648–10659.

Amrhein, F. & Mazik, M. (2021). *Eur. J. Org. Chem.* pp. 6282–6303.

Amrhein, F., Schwarzer, A. & Mazik, M. (2021). *Acta Cryst.* E**77**, 233–236.

Battaini, G., Monzani, E., Perotti, A., Para, C., Casella, L., Santagostini, L., Gullotti, M., Dillinger, R., Näther, C. & Tuczek, F. (2003). *J. Am. Chem. Soc.* **125**, 4185–4198.

Bernstein, J., Davis, R. E., Shimoni, L. & Chang, N.-L. (1995). *Angew. Chem. Int. Ed. Engl.* **34**, 1555–1573.

Bruker, (2014). *APEX2* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.

Burla, M. C., Caliandro, R., Carrozzini, B., Cascarano, G. L., Cuocci, C., Giacovazzo, C., Mallamo, M., Mazzone, A. & Polidori, G. (2015). *J. Appl. Cryst.* **48**, 306–309.

Desiraju, G. R. & Steiner, T. (1999). In *The Weak Hydrogen Bond*. Oxford University Press.

Etter, M. C. (1990). *Acc. Chem. Res.* **23**, 120–126.

Etter, M. C., MacDonald, J. C. & Bernstein, J. (1990). *Acta Cryst.* B**46**, 256–262.

Farrugia, L. J. (2012). *J. Appl. Cryst.* **45**, 849–854.

Groom, C. R., Bruno, I. J., Lightfoot, M. P. & Ward, S. C. (2016). *Acta Cryst.* B**72**, 171–179.

Hübschle, C. B., Sheldrick, G. M. & Dittrich, B. (2011). *J. Appl. Cryst.* **44**, 1281–1284.

Koch, N., Seichter, W. & Mazik, M. (2013). *Acta Cryst.* E**69**, o679.

Köhler, L., Seichter, W. & Mazik, M. (2020). *Eur. J. Org. Chem.* pp. 7023–7034.

Köhler, L., Hübler, C., Seichter, W. & Mazik, M. (2021). *RSC Adv.* **11**, 22221–22229.

Kurz, K. & Göbel, M. W. (1996). *Helv. Chim. Acta*, **79**, 1967–1979.

Lippe, J. & Mazik, M. (2013). *J. Org. Chem.* **78**, 9013–9020.

Lippe, J. & Mazik, M. (2015). *J. Org. Chem.* **80**, 1427–1439.

Mazik, M., Cavga, H. & Jones, P. G. (2005). *J. Am. Chem. Soc.* **127**, 9045–9052.

Nishio, M., Umezawa, Y., Honda, K., Tsuboyama, S. & Suezawa, H. (2009). *CrystEngComm*, **11**, 1757–1788.

Pedireddy, V. R., Reddy, D. S., Goud, B. S., Craig, D. C., Rae, A. D. & Desiraju, G. R. (1999). *J. Chem. Soc. Perkin Trans. 2*, pp. 2353–2360.

Pinkus, A. G., Klausmeyer, K. K., Feazell, R. P. & Lin, E. C. H. Y. (2005). *Acta Cryst.* E**61**, o662–o663.

Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.

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

Star, A., Liu, Y., Grant, K., Ridvan, L., Stoddart, J. F., Steuerman, D. W., Diehl, M. R., Boukai, A. & Heath, J. R. (2003). *Macromolecules*, **36**, 553–560.

Stoe & Cie (2002). *X-AREA* and *X-RED*. Stoe & Cie GmbH, Darmstadt, Germany.

Tiekink, E. R. T. & Zukerman-Schpector, J. (2012). In *The Importance of Pi-Interactions in Crystal Engineering. Frontiers in Crystal Engineering*. Chichester: Wiley.

Velde, C. M. L. V., Zeller, M. & Azov, V. A. (2012). *J. Mol. Struct.* **1016**, 109–117.

Westrip, S. P. (2010). *J. Appl. Cryst.* **43**, 920–925.

Zondervan, C., van den Beuken, E. K., Kooijman, H., Spek, A. L. & Feringa, B. L. (1997). *Tetrahedron Lett.* **38**, 3111–3114.

supporting information

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

Ben Ebersbach, Wilhelm Seichter and Monika Mazik

Computing details

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

Methyl 3,5-dimethylbenzoate (1)

Crystal data

$C_{10}H_{12}O_2$	$F(000) = 352$
$M_r = 164.20$	$D_x = 1.219$ Mg m^{-3}
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
$a = 8.4631$ (6) Å	Cell parameters from 7437 reflections
$b = 7.9793$ (4) Å	$\theta = 2.7–27.2°$
$c = 13.4042$ (9) Å	$\mu = 0.08$ mm^{-1}
$\beta = 98.835$ (6)°	$T = 153$ K
$V = 894.44$ (10) Å^3	Piece, colorless
$Z = 4$	$0.40 \times 0.25 \times 0.16$ mm

Data collection

Stoe IPDS 2T diffractometer	1762 independent reflections
	1449 reflections with $I > 2\sigma(I)$
Radiation source: sealed X-ray tube, 12 x 0.4 mm long-fine focus	$R_{int} = 0.046$
Plane graphite monochromator	$\theta_{max} = 26.0°$, $\theta_{min} = 2.7°$
Detector resolution: 6.67 pixels mm^{-1}	$h = -10\rightarrow9$
rotation method scans	$k = -9\rightarrow9$
7437 measured reflections	$l = -16\rightarrow16$

Refinement

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 neighbouring sites
$wR(F^2) = 0.116$	
$S = 1.05$	H-atom parameters constrained
1762 reflections	

$w = 1/[\sigma^2(F_o^2) + (0.0548P)^2 + 0.2723P]$
  where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$

$\Delta\rho_{max} = 0.24$ e Å^{-3}
$\Delta\rho_{min} = -0.19$ 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}
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	−0.00096 (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 (Å²)

	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)

C7	0.0223 (6)	0.0316 (7)	0.0276 (7)	0.0001 (5)	0.0045 (5)	−0.0024 (5)
C8	0.0320 (8)	0.0441 (9)	0.0337 (8)	−0.0061 (6)	0.0098 (6)	0.0092 (6)
C9	0.0418 (9)	0.0237 (8)	0.0480 (9)	−0.0012 (6)	0.0024 (7)	0.0011 (6)
C10	0.0388 (8)	0.0339 (8)	0.0300 (7)	−0.0009 (6)	0.0140 (6)	−0.0028 (6)

Geometric parameters (Å, º)

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—H2	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—C8	116.20 (11)	O1—C7—C1	124.76 (13)
C6—C1—C2	119.92 (12)	O2—C7—C1	111.94 (11)
C6—C1—C7	121.19 (12)	O2—C8—H8A	109.5
C2—C1—C7	118.86 (12)	O2—C8—H8B	109.5
C3—C2—C1	120.46 (12)	H8A—C8—H8B	109.5
C3—C2—H2	119.8	O2—C8—H8C	109.5
C1—C2—H2	119.8	H8A—C8—H8C	109.5
C2—C3—C4	118.70 (12)	H8B—C8—H8C	109.5
C2—C3—C9	120.23 (13)	C3—C9—H9A	109.5
C4—C3—C9	121.07 (13)	C3—C9—H9B	109.5
C5—C4—C3	121.89 (12)	H9A—C9—H9B	109.5
C5—C4—H4	119.1	C3—C9—H9C	109.5
C3—C4—H4	119.1	H9A—C9—H9C	109.5
C4—C5—C6	118.46 (12)	H9B—C9—H9C	109.5
C4—C5—C10	121.74 (12)	C5—C10—H10A	109.5
C6—C5—C10	119.80 (12)	C5—C10—H10B	109.5
C1—C6—C5	120.57 (12)	H10A—C10—H10B	109.5
C1—C6—H6	119.7	C5—C10—H10C	109.5
C5—C6—H6	119.7	H10A—C10—H10C	109.5
O1—C7—O2	123.30 (12)	H10B—C10—H10C	109.5
C6—C1—C2—C3	0.43 (19)	C7—C1—C6—C5	−178.18 (12)
C7—C1—C2—C3	178.69 (11)	C4—C5—C6—C1	−0.48 (19)
C1—C2—C3—C4	−0.42 (19)	C10—C5—C6—C1	179.82 (12)
C1—C2—C3—C9	−179.91 (13)	C8—O2—C7—O1	−1.2 (2)
C2—C3—C4—C5	0.0 (2)	C8—O2—C7—C1	178.09 (11)
C9—C3—C4—C5	179.44 (13)	C6—C1—C7—O1	170.41 (14)
C3—C4—C5—C6	0.5 (2)	C2—C1—C7—O1	−7.8 (2)

| C3—C4—C5—C10 | −179.82 (12) | C6—C1—C7—O2 | −8.91 (17) |
| C2—C1—C6—C5 | 0.03 (19) | C2—C1—C7—O2 | 172.85 (12) |

Hydrogen-bond geometry (Å, º)

*Cg*1 represents the centroid of the C1–C6 ring.

D—H⋯*A*	*D*—H	H⋯*A*	*D*⋯*A*	*D*—H⋯*A*
C10—H10*B*⋯O1^i	0.98	2.57	3.5215 (19)	163
C8—H8*B*⋯*Cg*1^{ii}	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

$C_{10}H_{10}Br_2O_2$	$Z = 4$
$M_r = 322.00$	$F(000) = 624$
Triclinic, $P\overline{1}$	$D_x = 1.930$ Mg m^{-3}
$a = 7.7936$ (2) Å	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
$b = 9.1655$ (2) Å	Cell parameters from 9654 reflections
$c = 17.2292$ (4) Å	$\theta = 2.7$–36.8°
$\alpha = 88.1637$ (12)°	$\mu = 7.29$ mm^{-1}
$\beta = 80.9050$ (12)°	$T = 130$ K
$\gamma = 65.8659$ (11)°	Irregular, colourless
$V = 1108.30$ (5) Å^3	$0.46 \times 0.39 \times 0.27$ mm

Data collection

Bruker Kappa APEXII CCD area detector diffractometer	5842 independent reflections
	5305 reflections with $I > 2\sigma(I)$
φ and ω scans	$R_{int} = 0.033$
Absorption correction: multi-scan (SADABS; Bruker, 2014)	$\theta_{max} = 28.9°$, $\theta_{min} = 1.2°$
	$h = -10 \rightarrow 10$
$T_{min} = 0.134$, $T_{max} = 0.244$	$k = -12 \rightarrow 12$
29065 measured reflections	$l = -23 \rightarrow 22$

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	
$R[F^2 > 2\sigma(F^2)] = 0.028$	H-atom parameters constrained
$wR(F^2) = 0.070$	$w = 1/[\sigma^2(F_o^2) + (0.0273P)^2 + 2.052P]$
$S = 1.04$	where $P = (F_o^2 + 2F_c^2)/3$
5842 reflections	$(\Delta/\sigma)_{max} = 0.001$
255 parameters	$\Delta\rho_{max} = 1.21$ e Å^{-3}
0 restraints	$\Delta\rho_{min} = -0.98$ 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*

C10A	0.7074 (3)	−0.2710 (3)	0.55685 (15)	0.0225 (5)
H10C	0.6986	−0.3315	0.5136	0.027*
H10D	0.5896	−0.2386	0.5938	0.027*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	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)	0.0010 (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 (8)
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, º)

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)

C3—C9	1.492 (3)	C3A—C9A	1.498 (4)
C4—C5	1.389 (3)	C4A—C5A	1.392 (3)
C4—H4	0.9300	C4A—H4A	0.9300
C5—C6	1.391 (3)	C5A—C6A	1.391 (3)
C5—C10	1.495 (3)	C5A—C10A	1.488 (3)
C6—H6	0.9300	C6A—H6A	0.9300
C7—C8	1.492 (3)	C7A—C8A	1.494 (4)
C8—H8A	0.9600	C8A—H8A1	0.9600
C8—H8B	0.9600	C8A—H8A2	0.9600
C8—H8C	0.9600	C8A—H8A3	0.9600
C9—H9A	0.9700	C9A—H9A1	0.9700
C9—H9B	0.9700	C9A—H9A2	0.9700
C10—H10A	0.9700	C10A—H10C	0.9700
C10—H10B	0.9700	C10A—H10D	0.9700
C7—O1—C1	118.16 (18)	C7A—O1A—C1A	118.43 (19)
C2—C1—C6	122.2 (2)	C2A—C1A—C6A	121.8 (2)
C2—C1—O1	116.6 (2)	C2A—C1A—O1A	119.6 (2)
C6—C1—O1	121.1 (2)	C6A—C1A—O1A	118.3 (2)
C1—C2—C3	119.2 (2)	C1A—C2A—C3A	119.3 (2)
C1—C2—H2	120.4	C1A—C2A—H2A	120.3
C3—C2—H2	120.4	C3A—C2A—H2A	120.3
C4—C3—C2	119.3 (2)	C4A—C3A—C2A	119.4 (2)
C4—C3—C9	120.7 (2)	C4A—C3A—C9A	120.0 (2)
C2—C3—C9	120.0 (2)	C2A—C3A—C9A	120.5 (2)
C5—C4—C3	120.8 (2)	C3A—C4A—C5A	121.0 (2)
C5—C4—H4	119.6	C3A—C4A—H4A	119.5
C3—C4—H4	119.6	C5A—C4A—H4A	119.5
C4—C5—C6	119.9 (2)	C6A—C5A—C4A	119.2 (2)
C4—C5—C10	120.1 (2)	C6A—C5A—C10A	120.1 (2)
C6—C5—C10	120.0 (2)	C4A—C5A—C10A	120.7 (2)
C1—C6—C5	118.6 (2)	C1A—C6A—C5A	119.3 (2)
C1—C6—H6	120.7	C1A—C6A—H6A	120.3
C5—C6—H6	120.7	C5A—C6A—H6A	120.3
O2—C7—O1	123.3 (2)	O2A—C7A—O1A	122.4 (2)
O2—C7—C8	126.2 (2)	O2A—C7A—C8A	126.0 (2)
O1—C7—C8	110.5 (2)	O1A—C7A—C8A	111.6 (2)
C7—C8—H8A	109.5	C7A—C8A—H8A1	109.5
C7—C8—H8B	109.5	C7A—C8A—H8A2	109.5
H8A—C8—H8B	109.5	H8A1—C8A—H8A2	109.5
C7—C8—H8C	109.5	C7A—C8A—H8A3	109.5
H8A—C8—H8C	109.5	H8A1—C8A—H8A3	109.5
H8B—C8—H8C	109.5	H8A2—C8A—H8A3	109.5
C3—C9—Br1	111.76 (16)	C3A—C9A—Br1A	112.24 (17)
C3—C9—H9A	109.3	C3A—C9A—H9A1	109.2
Br1—C9—H9A	109.3	Br1A—C9A—H9A1	109.2
C3—C9—H9B	109.3	C3A—C9A—H9A2	109.2
Br1—C9—H9B	109.3	Br1A—C9A—H9A2	109.2

H9A—C9—H9B	107.9	H9A1—C9A—H9A2	107.9
C5—C10—Br2	111.29 (17)	C5A—C10A—Br2A	110.38 (16)
C5—C10—H10A	109.4	C5A—C10A—H10C	109.6
Br2—C10—H10A	109.4	Br2A—C10A—H10C	109.6
C5—C10—H10B	109.4	C5A—C10A—H10D	109.6
Br2—C10—H10B	109.4	Br2A—C10A—H10D	109.6
H10A—C10—H10B	108.0	H10C—C10A—H10D	108.1
C7—O1—C1—C2	−116.6 (2)	C7A—O1A—C1A—C2A	−81.9 (3)
C7—O1—C1—C6	66.7 (3)	C7A—O1A—C1A—C6A	104.9 (3)
C6—C1—C2—C3	−0.8 (4)	C6A—C1A—C2A—C3A	0.2 (3)
O1—C1—C2—C3	−177.5 (2)	O1A—C1A—C2A—C3A	−172.7 (2)
C1—C2—C3—C4	0.2 (3)	C1A—C2A—C3A—C4A	−0.4 (3)
C1—C2—C3—C9	−178.3 (2)	C1A—C2A—C3A—C9A	175.3 (2)
C2—C3—C4—C5	0.4 (3)	C2A—C3A—C4A—C5A	0.8 (3)
C9—C3—C4—C5	178.8 (2)	C9A—C3A—C4A—C5A	−175.0 (2)
C3—C4—C5—C6	−0.3 (3)	C3A—C4A—C5A—C6A	−0.9 (3)
C3—C4—C5—C10	177.8 (2)	C3A—C4A—C5A—C10A	178.1 (2)
C2—C1—C6—C5	0.9 (4)	C2A—C1A—C6A—C5A	−0.3 (3)
O1—C1—C6—C5	177.4 (2)	O1A—C1A—C6A—C5A	172.73 (19)
C4—C5—C6—C1	−0.3 (3)	C4A—C5A—C6A—C1A	0.6 (3)
C10—C5—C6—C1	−178.5 (2)	C10A—C5A—C6A—C1A	−178.4 (2)
C1—O1—C7—O2	−4.0 (3)	C1A—O1A—C7A—O2A	−5.1 (4)
C1—O1—C7—C8	174.9 (2)	C1A—O1A—C7A—C8A	175.6 (2)
C4—C3—C9—Br1	70.6 (2)	C4A—C3A—C9A—Br1A	−95.8 (3)
C2—C3—C9—Br1	−111.0 (2)	C2A—C3A—C9A—Br1A	88.5 (3)
C4—C5—C10—Br2	80.7 (2)	C6A—C5A—C10A—Br2A	99.1 (2)
C6—C5—C10—Br2	−101.1 (2)	C4A—C5A—C10A—Br2A	−79.9 (2)

Hydrogen-bond geometry (Å, º)

D—H···A	D—H	H···A	D···A	D—H···A
C10A—H10D···O2A[i]	0.97	2.28	3.236 (3)	168
C10A—H10C···Br1A[i]	0.97	2.89	3.836 (3)	164
C8A—H8A3···O2	0.96	2.58	3.521 (4)	168
C10—H10B···Br2A[i]	0.97	3.01	3.757 (3)	135
C10—H10A···O2[ii]	0.97	2.58	3.449 (3)	150
C9—H9B···Br2[iii]	0.97	2.95	3.854 (3)	156
C9—H9A···O2[iii]	0.97	2.45	3.334 (3)	151

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

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

Crystal data

$C_8H_6O_3$	$b = 11.9549$ (4) Å
$M_r = 150.13$	$c = 15.0846$ (5) Å
Monoclinic, $P2_1/n$	$\beta = 94.212$ (2)°
$a = 3.7345$ (1) Å	$V = 671.64$ (4) Å³

$Z = 4$
$F(000) = 312$
$D_x = 1.485$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 6158 reflections

$\theta = 2.7–30.5°$
$\mu = 0.12$ mm^{-1}
$T = 153$ K
Rod, colourless
$0.42 \times 0.28 \times 0.19$ mm

Data collection

Bruker Kappa APEXII CCD area detector
   diffractometer
φ and ω scans
11533 measured reflections
1819 independent reflections
1519 reflections with $I > 2\sigma(I)$

$R_{int} = 0.058$
$\theta_{max} = 29.4°$, $\theta_{min} = 2.7°$
$h = -5 \rightarrow 4$
$k = -16 \rightarrow 16$
$l = -20 \rightarrow 20$

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
$w = 1/[\sigma^2(F_o^2) + (0.0692P)^2 + 0.2868P]$
   where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.33$ e Å^{-3}
$\Delta\rho_{min} = -0.28$ 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}
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)	0.37799 (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 (Å²)

	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 (Å, º)

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—H6	0.9500
C2—C3	1.3840 (17)	C7—H7	0.9500
C2—H2	0.9500	C8—H8	0.9500
C3—C4	1.3958 (16)		
C1—O1—H1	113.2 (16)	C4—C5—C6	121.19 (11)
O1—C1—C6	124.40 (11)	C4—C5—C8	117.88 (11)
O1—C1—C2	115.87 (11)	C6—C5—C8	120.92 (11)
C6—C1—C2	119.73 (11)	C1—C6—C5	119.43 (11)
C3—C2—C1	120.23 (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—C2—C3	−179.31 (12)	O1—C1—C6—C5	179.13 (13)
C6—C1—C2—C3	0.0 (2)	C2—C1—C6—C5	−0.1 (2)
C1—C2—C3—C4	0.3 (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) |

Hydrogen-bond geometry (Å, º)

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[ii]	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.