

Comparison of the crystal structures of methyl 4-bromo-2-(methoxymethoxy)benzoate and 4-bromo-3-(methoxymethoxy)benzoic acid

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CCDC references: 1457944; 1457943

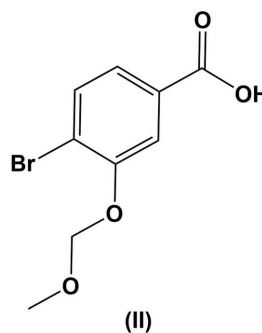
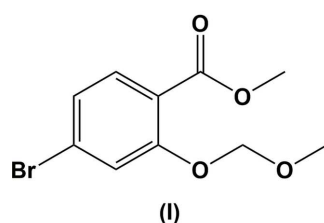
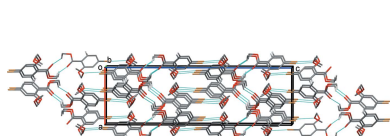
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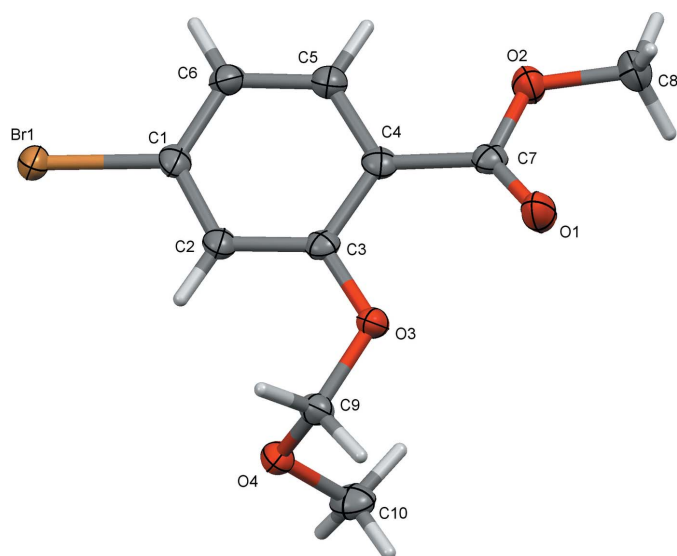
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The title compounds, C₁₀H₁₁BrO₄, (I), and C₉H₉BrO₄, (II), are derivatives of bromo–hydroxy–benzoic acids. Compound (II) crystallizes with two independent molecules (*A* and *B*) in the asymmetric unit. In both (I) and (II), the O—CH₂—O—CH₃ side chain is not in its fully extended conformation; the O—C—O—C torsion angle is 67.3 (3)° in (I), and –65.8 (3) and –74.1 (3)° in molecules *A* and *B*, respectively, in compound (II). In the crystal of (I), molecules are linked by C—H···O hydrogen bonds, forming *C*(5) chains along [010]. The chains are linked by short Br···O contacts [3.047 (2) Å], forming sheets parallel to the *bc* plane. The sheets are linked *via* C—H··· π interactions, forming a three-dimensional architecture. In the crystal of (II), molecules *A* and *B* are linked to form *R*₂²(8) dimers *via* two strong O—H···O hydrogen bonds. These dimers are linked into ···*A*–*B*···*A*–*B*···*A*–*B*··· [*C*₂(15)] chains along [011] by C—H···O hydrogen bonds. The chains are linked by slipped parallel π – π interactions [inter-centroid distances = 3.6787 (18) and 3.8431 (17) Å], leading to the formation of slabs parallel to the *bc* plane.

1. Chemical context

Ester derivatives of many compounds exhibit a variety of pharmacological properties, such as anticancer, antitumor and antimicrobial activities (Anadu *et al.*, 2006; Bartzatt *et al.*, 2004; Bi *et al.*, 2012). Salicylic acid and derivatives of salicylic acid are of great biological importance. For example, they are known for their analgesic and anti-inflammatory activities in the treatment of rheumatoid arthritis (Anderson *et al.*, 2014; Hardie, 2013). They are also known for their use as anti-bacterial and antimycobacterial agents (Silva *et al.*, 2008). In view of the above, compounds (I) and (II) were synthesized and we report herein on their crystal structures.



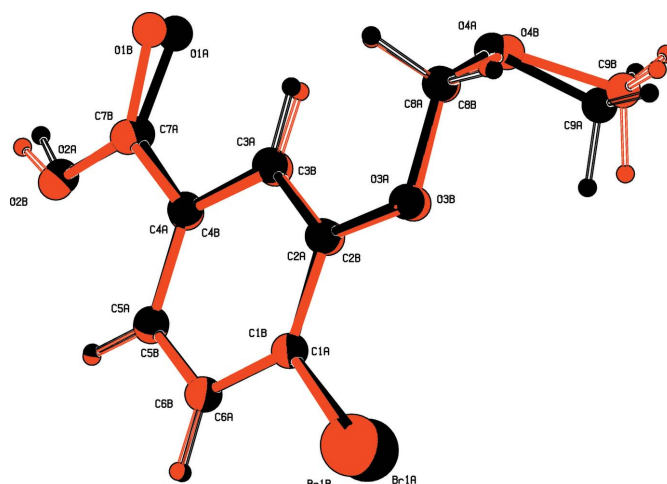

Figure 1

A view of the molecular structure of compound (I), showing the atom labelling. Displacement ellipsoids are drawn at the 50% probability level.

2. Structural commentary

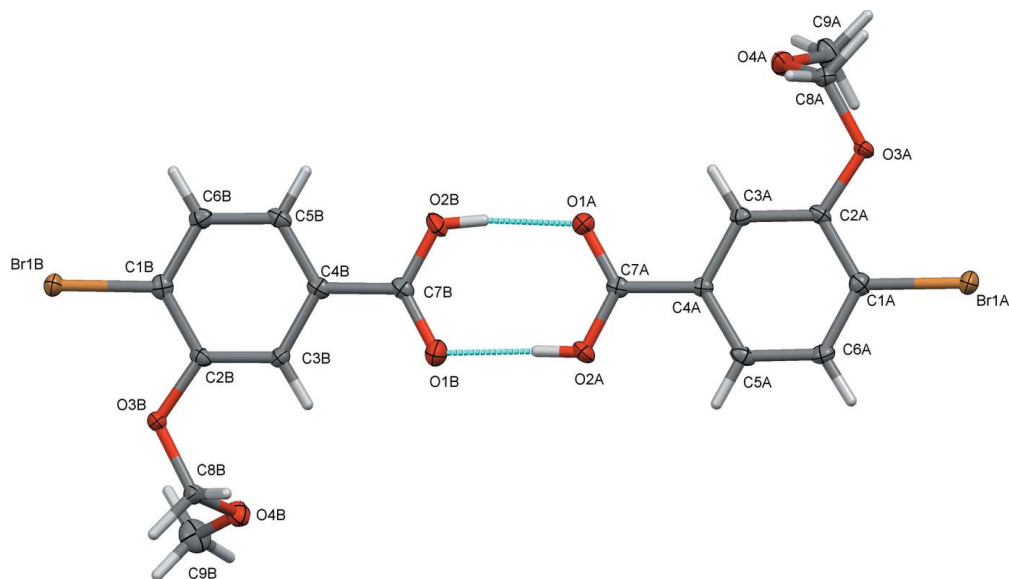
The molecular structure of compound (I), is illustrated in Fig. 1. The $-\text{O}-\text{CH}_2-\text{O}-\text{CH}_3$ side chain is not in its fully extended conformation, with torsion angle $\text{O}3-\text{C}9-\text{O}4-\text{C}10$ being $67.3(3)^\circ$. The dihedral angle between the benzene ring and the ester segment ($\text{O}1/\text{C}7/\text{O}2/\text{C}8$) is $14.5(2)^\circ$, while the plane through atoms $\text{C}10/\text{O}4/\text{C}9$ of the methoxymethoxy side chain is inclined to the benzene ring by $82.5(3)^\circ$.

The molecular structure of compound (II), is illustrated in Fig. 2. It crystallizes with two independent molecules (*A* and *B*) in the asymmetric unit. The conformations of the two molecules differ in the torsion angles of the $-\text{O}-\text{CH}_2-\text{O}-\text{CH}_3$


Figure 3

A view of the molecular fit of molecules *A* (black) and *B* (red) of compound (II).

side chains and the orientation of the $-\text{COO}-$ group with respect to the benzene ring, as shown in the *AutoMolFit* diagram (Fig. 3; Spek, 2009). The $-\text{O}-\text{CH}_2-\text{O}-\text{CH}_3$ side chains in molecules *A* and *B* are not in their fully extended conformation; torsion angle $\text{O}3\text{A}-\text{C}8\text{A}-\text{O}4\text{A}-\text{C}9\text{A}$ in molecule *A* is $-65.8(3)^\circ$, and torsion angle $\text{O}3\text{B}-\text{C}8\text{B}-\text{O}4\text{B}-\text{C}9\text{B}$ in molecule *B* is $-74.1(3)^\circ$. The dihedral angle between the benzene ring and the plane through atoms $\text{C}8\text{A}/\text{O}4\text{A}/\text{C}9\text{A}$ of the methoxymethoxy side chain in molecule *A* is $79.2(3)^\circ$, while the corresponding dihedral angle in molecule *B*, between the benzene ring and plane $\text{C}9\text{B}/\text{O}4\text{B}/\text{O}8\text{B}$ is $67.1(3)^\circ$. This is less than in compound (I) and further, the dihedral angle between the benzene ring and the $-\text{COO}-$ group is $6.6(4)^\circ$ in *A* and $9.1(4)^\circ$ in *B*; also less than observed in compound (I), *viz.* $14.5(2)^\circ$.


Figure 2

A view of the molecular structure of compound (II), showing the atom labelling. Displacement ellipsoids are drawn at the 50% probability level. $\text{O}-\text{H}\cdots\text{O}$ hydrogen bonds are shown as dashed lines (see Table 2).

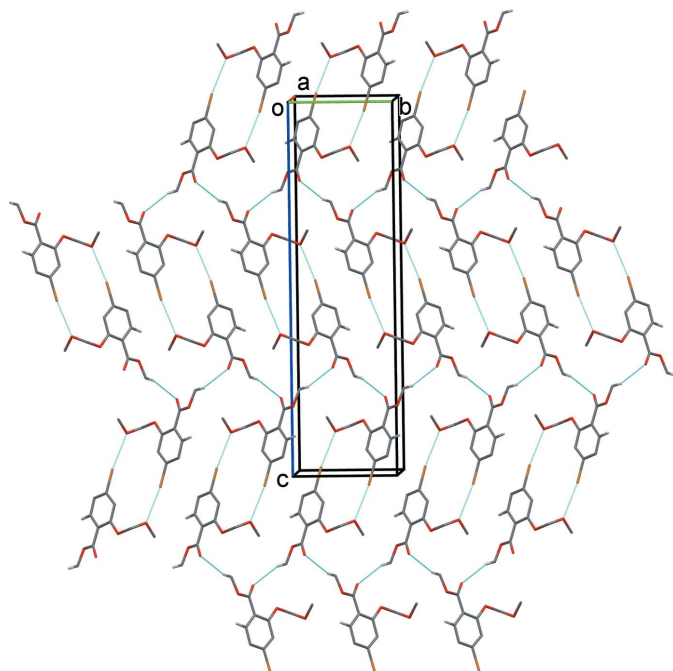


Figure 4
A view along the *a* axis of the crystal packing of compound (I). C–H...O and Br...O interactions are shown as dashed lines (see Table 1). H atoms not involved in these interactions have been omitted for clarity.

3. Supramolecular features

In the crystal of (I), molecules are linked by structure-directing C8–H8A...O1 hydrogen bonds (Table 1 and Fig. 4), forming *C*(5) chains along the *b* axis. Adjacent chains are linked by short Br1...O4ⁱ contacts [$d_{\text{Br}\cdots\text{O}} = 3.047(2) \text{ \AA}$; symmetry code (i): $-x, -y, -z + 1$] leading to the formation of sheets parallel to plane (100). The sheets are linked by C5–H5... π interactions (centroid of the benzene ring C1–C6) along the *a*-axis direction, forming a three-dimensional structure (Table 1 and Fig. 5).

In the crystal of (II), molecules *A* and *B* are linked *via* two strong O–H...O hydrogen bonds, namely, O2A–H2A...O1B and O2B–H2B...O1A, forming dimers with an $R_2^2(8)$ ring motif (Table 2 and Fig. 6). Adjacent dimers are linked by C8B–H8B2...O3A hydrogen bonds (Table 2), forming chains along [011]. The chains are linked *via* slipped parallel π – π interactions between *B* molecules [$Cg2\cdots Cg2^{\text{ii}}$ distance = $3.6792(18) \text{ \AA}$; *Cg*2 is the centroid of ring C1B–C6B;

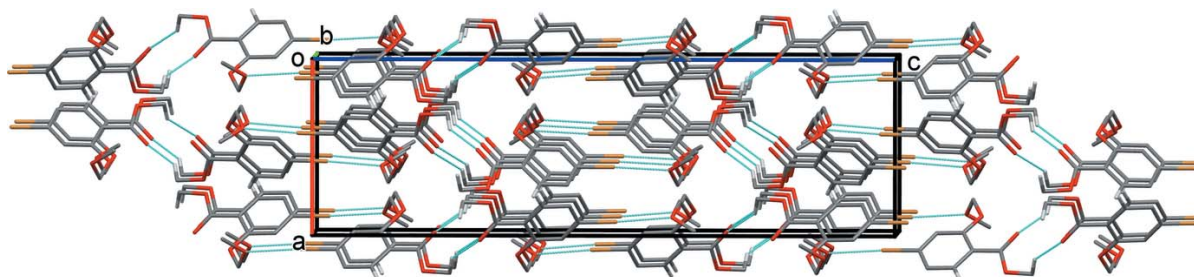


Figure 5
A view along the *b* axis of the crystal packing of compound (I). C–H...O and Br...O interactions are shown as dashed lines (see Table 1). H atoms not involved in these interactions have been omitted for clarity.

Table 1
Hydrogen-bond geometry (\AA , $^\circ$) for (I).

*Cg*1 is the centroid of the C1–C6 benzene ring.

<i>D</i> –H... <i>A</i>	<i>D</i> –H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> –H... <i>A</i>
C8–H8A...O1 ⁱ	0.98	2.58	3.439 (5)	147
C5–H5... <i>Cg</i> 1 ⁱⁱ	0.95	2.95	3.765 (4)	129

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

Table 2
Hydrogen-bond geometry (\AA , $^\circ$) for (II).

<i>D</i> –H... <i>A</i>	<i>D</i> –H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> –H... <i>A</i>
O2A–H2A...O1B	0.84 (5)	1.80 (5)	2.635 (4)	178 (5)
O2B–H2B...O1A	0.82 (5)	1.81 (5)	2.621 (4)	167 (5)
C8B–H8B2...O3A ⁱ	0.99	2.52	3.420 (4)	150

inter-planar distance = $3.3691(12) \text{ \AA}$; slippage = 1.477 \AA ; symmetry code (ii): $-x, -y + 2, -z + 1$], and between *A* and *B* molecules [$Cg1\cdots Cg2^{\text{iii}}$ = $3.8431(17) \text{ \AA}$; *Cg*1 is the centroid of the ring C1A–C6A; inter-planar distance = $3.5538(12) \text{ \AA}$; slippage 1.98 \AA ; symmetry code (iii): $-x + 1, -y + 1, -z + 1$], thus forming slabs lying parallel to the *bc* plane (Fig. 7).

4. Synthesis and crystallization

Synthesis of methyl 4-bromo-2-(methoxymethoxy) benzoate (I)

To a stirred solution of methyl 4-bromo-2-hydroxybenzoate (1.0 g, 4.32 mmol) in dichloromethane (15 ml) (DCM) was added *N,N*-diisopropylethylamine (1.5 ml, 8.65 mmol) (DIPEA), followed by chloromethyl methyl ether (0.49 ml, 6.49 mmol) (MOM-Cl), at 273 K and the reaction mixture was stirred at room temperature overnight. The reaction mixture was then diluted with water (50 ml) and the organic layer was extracted with ethyl acetate ($2 \times 50 \text{ ml}$). The combined organic layers were washed successively with water, brine, dried over anhydrous magnesium sulfate (MgSO_4), filtered and the filtrate was concentrated under reduced pressure. The crude product was purified by column chromatography using ethyl acetate:hexane (1:9) as eluent to afford (I) as an off-white coloured solid (yield: 0.851 g, 71.4%; m.p.: 353 K). $^1\text{H NMR}$ ($\text{DMSO}-d_6$, 400 MHz, p.p.m.): $\delta = 3.39$ (3H,

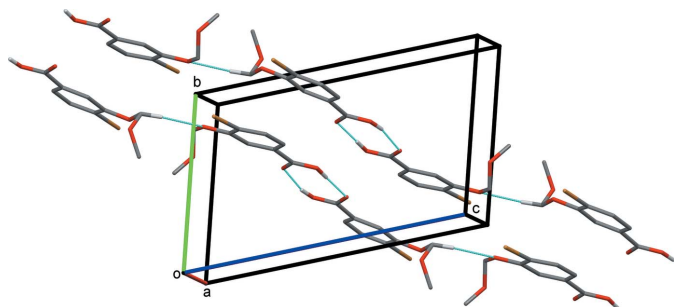


Figure 6
A partial view along the *a* axis of the crystal packing of compound (II). O—H···O and C—H···O hydrogen bonds are shown as dashed lines (see Table 2). H atoms not involved in these interactions have been omitted for clarity.

s), 3.79 (3H, *s*), 5.29 (2H, *s*) 7.29 (1H, *dd*, *J* = 1.20 Hz, 1.20 Hz), 7.44 (1H, *s*), 7.60 (1H, *d*, *J* = 8.00 Hz).

Synthesis of 4-bromo-3-(methoxymethoxy)benzoic acid (II)

A mixture of methyl 4-bromo-3-(methoxymethoxy)benzoate (1 g, 3.63 mmol), 10% aqueous potassium hydroxide (0.61 g, 3.0 mmol), tetrahydrofuran (5 ml) and methanol (20 ml) was stirred at room temperature for 2 h. The mixture was then concentrated to remove organic solvents and the aqueous layer was acidified with 6 *N* hydrochloric acid. The precipitated solid was filtered, dried under vacuum to afford (II) as a white solid (yield: 0.86g, 91%; m.p.: 433 K). ¹H NMR (DMSO-*d*₆, 400 MHz, p.p.m.): δ = 3.39 (3H, *s*), 5.28 (2H, *s*),

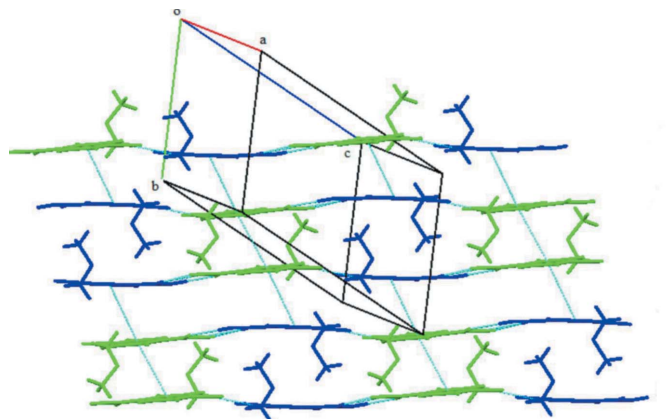


Figure 7
A view of the π - π stacking observed in the crystal of (II); molecule *A* green, molecule *B* blue.

7.26 (1H, *dd*, *J* = 1.20 Hz, 1.20 Hz), 7.40 (1H, *s*), 7.59 (1H, *d*, *J* = 8.00 Hz), 12.90 (1H, *s*).

Single crystals of compounds (I) and (II), suitable for X-ray diffraction studies, were obtained by solvent evaporation using methanol:chloroform (2:1) as the solvent mixture.

5. Refinement details

Crystal data, data collection and structure refinement details are summarized in Table 3. The H atoms of the OH groups in

Table 3
Experimental details.

	(I)	(II)
Crystal data		
Chemical formula	C ₁₀ H ₁₁ BrO ₄	C ₉ H ₆ BrO ₄
<i>M_r</i>	275.10	261.07
Crystal system, space group	Orthorhombic, <i>Pbca</i>	Triclinic, <i>P</i> $\bar{1}$
Temperature (K)	173	173
<i>a</i> , <i>b</i> , <i>c</i> (Å)	8.8487 (13), 8.1514 (11), 29.284 (4)	7.7211 (3), 9.6881 (4), 14.2627 (6)
α , β , γ (°)	90, 90, 90	73.635 (1), 77.664 (1), 69.577 (1)
<i>V</i> (Å ³)	2112.2 (5)	951.40 (7)
<i>Z</i>	8	4
Radiation type	Cu <i>K</i> α	Cu <i>K</i> α
μ (mm ⁻¹)	5.27	5.82
Crystal size (mm)	0.29 × 0.22 × 0.19	0.28 × 0.25 × 0.22
Data collection		
Diffractometer	Bruker APEXII	Bruker APEXII
Absorption correction	Multi-scan (<i>SADABS</i> ; Bruker, 2009)	Multi-scan (<i>SADABS</i> ; Bruker, 2009)
<i>T_{min}</i> – <i>T_{max}</i>	0.286, 0.367	0.245, 0.278
No. of measured, independent and observed [<i>I</i> > 2 σ (<i>I</i>)] reflections	8999, 1751, 1720	11112, 3031, 2930
<i>R_{int}</i>	0.052	0.040
(<i>sin</i> θ / λ) _{max} (Å ⁻¹)	0.590	0.585
Refinement		
<i>R</i> [<i>F</i> ² > 2 σ (<i>F</i> ²)], <i>wR</i> (<i>F</i> ²), <i>S</i>	0.052, 0.139, 1.14	0.039, 0.120, 1.09
No. of reflections	1751	3031
No. of parameters	138	261
No. of restraints	0	2
H-atom treatment	H-atom parameters constrained	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å ⁻³)	1.87, -0.97	0.67, -1.08

Computer programs: *APEX2*, *SAINT-Plus* and *XPREP* (Bruker, 2009), *SHELXS97* and *SHELXL97* (Sheldrick, 2008) and *Mercury* (Macrae *et al.*, 2008).

(II) were located in a difference Fourier map and refined with a distance restraint: O–H = 0.84 (5) Å. The C-bound H atoms in (I) and (II) were positioned with idealized geometry and refined using a riding model: C–H = 0.95–0.99 Å, with $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{C-methyl})$ and $1.2U_{\text{eq}}(\text{C})$ for other H atoms. In the final cycles of refinement reflection (0 0 2) in (I) and reflections (4 1 0), (6 – 4 6), (5 – 5 7), (4 2 0) and (0 – 1 6) in (II) were omitted due to large differences in F^2_{obs} and F^2_{calc} , considerably improving the values of $R1$, $wR2$, and GOF.

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Comparison of the crystal structures of methyl 4-bromo-2-(methoxymethoxy)-benzoate and 4-bromo-3-(methoxymethoxy)benzoic acid

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Computing details

For both compounds, data collection: *APEX2* (Bruker, 2009); cell refinement: *APEX2* and *SAINT-Plus* (Bruker, 2009); data reduction: *SAINT-Plus* and *XPREP* (Bruker, 2009). Program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008) for (I); *SHELXS97* (Sheldrick, 2008) for (II). For both compounds, program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008). Molecular graphics: *Mercury* (Macrae *et al.*, 2008) and *PLATON* (Spek, 2009) for (I); *Mercury* (Macrae *et al.*, 2008) for (II). Software used to prepare material for publication: *SHELXL97* (Sheldrick, 2008) and *PLATON* (Spek, 2009) for (I); *SHELXL97* (Sheldrick, 2008) for (II).

(I) Methyl 4-bromo-2-(methoxymethoxy)benzoate

Crystal data

$C_{10}H_{11}BrO_4$

$M_r = 275.10$

Orthorhombic, *Pbca*

Hall symbol: -P 2ac 2ab

$a = 8.8487$ (13) Å

$b = 8.1514$ (11) Å

$c = 29.284$ (4) Å

$V = 2112.2$ (5) Å³

$Z = 8$

$F(000) = 1104$

$D_x = 1.730$ Mg m⁻³

Melting point: 353 K

Cu $K\alpha$ radiation, $\lambda = 1.54178$ Å

Cell parameters from 108 reflections

$\theta = 5.8$ – 65.5°

$\mu = 5.27$ mm⁻¹

$T = 173$ K

Prism, colourless

$0.29 \times 0.22 \times 0.19$ mm

Data collection

Bruker APEXII

diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

phi and φ scans

Absorption correction: multi-scan

(*SADABS*; Bruker, 2009)

$T_{\min} = 0.286$, $T_{\max} = 0.367$

8999 measured reflections

1751 independent reflections

1720 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.052$

$\theta_{\max} = 65.5^\circ$, $\theta_{\min} = 5.8^\circ$

$h = -10 \rightarrow 10$

$k = -9 \rightarrow 9$

$l = -33 \rightarrow 26$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.052$

$wR(F^2) = 0.139$

$S = 1.14$

1751 reflections

138 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

$$w = 1/[\sigma^2(F_o^2) + (0.0922P)^2 + 2.3421P]$$

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

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

$$\Delta\rho_{\max} = 1.87 \text{ e } \text{\AA}^{-3}$$

$$\Delta\rho_{\min} = -0.97 \text{ e } \text{\AA}^{-3}$$

Special details

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

Refinement. Refinement of F^2 against ALL reflections. The weighted R -factor wR and goodness of fit S are based on F^2 , conventional R -factors R are based on F , with F set to zero for negative F^2 . The threshold expression of $F^2 > 2\sigma(F^2)$ is used only for calculating R -factors(gt) *etc.* and is not relevant to the choice of reflections for refinement. R -factors based on F^2 are statistically about twice as large as those based on F , and R -factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.0849 (4)	0.2964 (5)	0.54070 (13)	0.0228 (8)
C2	-0.0101 (4)	0.2114 (4)	0.56954 (11)	0.0224 (7)
H2	-0.0720	0.1256	0.5581	0.027*
C3	-0.0142 (4)	0.2529 (4)	0.61565 (12)	0.0206 (8)
C4	0.0827 (3)	0.3778 (4)	0.63243 (12)	0.0215 (7)
C5	0.1783 (4)	0.4566 (4)	0.60112 (11)	0.0250 (7)
H5	0.2437	0.5404	0.6120	0.030*
C6	0.1816 (4)	0.4177 (4)	0.55544 (11)	0.0256 (7)
H6	0.2478	0.4723	0.5349	0.031*
C7	0.0819 (3)	0.4299 (4)	0.68120 (12)	0.0218 (8)
C8	0.2085 (4)	0.5894 (5)	0.73653 (12)	0.0315 (8)
H8A	0.1242	0.6655	0.7414	0.047*
H8B	0.3043	0.6476	0.7408	0.047*
H8C	0.2016	0.4992	0.7585	0.047*
C9	-0.1952 (4)	0.0417 (4)	0.63083 (11)	0.0232 (7)
H9A	-0.2664	0.0089	0.6553	0.028*
H9B	-0.2556	0.0779	0.6042	0.028*
C10	-0.0340 (4)	-0.1675 (4)	0.65656 (13)	0.0326 (8)
H10A	0.0292	-0.0858	0.6720	0.049*
H10B	0.0293	-0.2584	0.6459	0.049*
H10C	-0.1099	-0.2093	0.6780	0.049*
O1	-0.0134 (3)	0.3974 (3)	0.70900 (8)	0.0347 (6)
O2	0.2014 (3)	0.5246 (3)	0.69070 (8)	0.0269 (6)
O3	-0.1048 (2)	0.1763 (3)	0.64664 (8)	0.0224 (5)
O4	-0.1082 (3)	-0.0932 (3)	0.61840 (8)	0.0260 (6)
Br1	0.08685 (4)	0.23443 (5)	0.477705 (13)	0.0262 (3)

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.031 (2)	0.0162 (18)	0.021 (2)	0.0058 (12)	0.0015 (12)	-0.0016 (15)

C2	0.0291 (18)	0.0165 (16)	0.0216 (18)	0.0018 (13)	0.0001 (14)	-0.0006 (13)
C3	0.0233 (18)	0.0155 (16)	0.0229 (19)	0.0029 (11)	-0.0006 (15)	0.0031 (11)
C4	0.0242 (16)	0.0158 (17)	0.0244 (18)	0.0042 (12)	-0.0004 (12)	0.0002 (14)
C5	0.0301 (17)	0.0200 (17)	0.0249 (17)	-0.0020 (13)	0.0003 (13)	0.0004 (13)
C6	0.0327 (17)	0.0194 (17)	0.0248 (17)	-0.0029 (13)	0.0060 (14)	0.0022 (13)
C7	0.0255 (17)	0.0155 (17)	0.0245 (18)	0.0027 (12)	0.0020 (12)	0.0013 (14)
C8	0.0391 (19)	0.031 (2)	0.0242 (17)	-0.0048 (16)	-0.0024 (16)	-0.0059 (14)
C9	0.0244 (16)	0.0196 (17)	0.0255 (17)	-0.0025 (13)	-0.0020 (13)	-0.0008 (13)
C10	0.0369 (19)	0.0235 (19)	0.038 (2)	0.0020 (17)	-0.0027 (16)	0.0071 (15)
O1	0.0416 (15)	0.0373 (15)	0.0253 (13)	-0.0121 (12)	0.0078 (11)	-0.0078 (11)
O2	0.0282 (12)	0.0298 (13)	0.0228 (12)	-0.0046 (10)	-0.0002 (10)	-0.0039 (10)
O3	0.0267 (11)	0.0189 (12)	0.0216 (12)	-0.0042 (9)	0.0033 (9)	-0.0019 (10)
O4	0.0364 (13)	0.0200 (13)	0.0217 (13)	0.0003 (10)	0.0016 (9)	-0.0009 (9)
Br1	0.0372 (4)	0.0236 (3)	0.0177 (4)	-0.00163 (13)	0.00120 (12)	0.00077 (12)

Geometric parameters (Å, °)

C1—C6	1.377 (5)	C7—O2	1.339 (4)
C1—C2	1.378 (5)	C8—O2	1.444 (4)
C1—Br1	1.913 (4)	C8—H8A	0.9800
C2—C3	1.392 (5)	C8—H8B	0.9800
C2—H2	0.9500	C8—H8C	0.9800
C3—O3	1.363 (4)	C9—O4	1.391 (4)
C3—C4	1.418 (5)	C9—O3	1.435 (4)
C4—C5	1.403 (5)	C9—H9A	0.9900
C4—C7	1.490 (5)	C9—H9B	0.9900
C5—C6	1.375 (5)	C10—O4	1.431 (4)
C5—H5	0.9500	C10—H10A	0.9800
C6—H6	0.9500	C10—H10B	0.9800
C7—O1	1.202 (4)	C10—H10C	0.9800
C6—C1—C2	123.2 (3)	O2—C8—H8A	109.5
C6—C1—Br1	119.1 (3)	O2—C8—H8B	109.5
C2—C1—Br1	117.6 (3)	H8A—C8—H8B	109.5
C1—C2—C3	119.2 (3)	O2—C8—H8C	109.5
C1—C2—H2	120.4	H8A—C8—H8C	109.5
C3—C2—H2	120.4	H8B—C8—H8C	109.5
O3—C3—C2	123.3 (3)	O4—C9—O3	112.3 (3)
O3—C3—C4	117.0 (3)	O4—C9—H9A	109.1
C2—C3—C4	119.6 (3)	O3—C9—H9A	109.1
C5—C4—C3	117.8 (3)	O4—C9—H9B	109.1
C5—C4—C7	119.9 (3)	O3—C9—H9B	109.1
C3—C4—C7	122.2 (3)	H9A—C9—H9B	107.9
C6—C5—C4	122.9 (3)	O4—C10—H10A	109.5
C6—C5—H5	118.6	O4—C10—H10B	109.5
C4—C5—H5	118.6	H10A—C10—H10B	109.5
C5—C6—C1	117.2 (3)	O4—C10—H10C	109.5
C5—C6—H6	121.4	H10A—C10—H10C	109.5

C1—C6—H6	121.4	H10B—C10—H10C	109.5
O1—C7—O2	122.7 (3)	C7—O2—C8	116.0 (3)
O1—C7—C4	126.2 (3)	C3—O3—C9	117.6 (3)
O2—C7—C4	111.1 (3)	C9—O4—C10	112.6 (3)
C6—C1—C2—C3	2.5 (5)	Br1—C1—C6—C5	-178.9 (2)
Br1—C1—C2—C3	179.8 (2)	C5—C4—C7—O1	164.0 (3)
C1—C2—C3—O3	179.8 (3)	C3—C4—C7—O1	-14.1 (5)
C1—C2—C3—C4	-2.0 (5)	C5—C4—C7—O2	-14.2 (4)
O3—C3—C4—C5	179.1 (3)	C3—C4—C7—O2	167.8 (3)
C2—C3—C4—C5	0.8 (5)	O1—C7—O2—C8	-1.3 (5)
O3—C3—C4—C7	-2.8 (5)	C4—C7—O2—C8	176.9 (3)
C2—C3—C4—C7	178.9 (3)	C2—C3—O3—C9	2.7 (4)
C3—C4—C5—C6	0.0 (5)	C4—C3—O3—C9	-175.6 (3)
C7—C4—C5—C6	-178.1 (3)	O4—C9—O3—C3	67.5 (3)
C4—C5—C6—C1	0.4 (5)	O3—C9—O4—C10	67.3 (3)
C2—C1—C6—C5	-1.7 (5)		

Hydrogen-bond geometry (Å, °)

Cg1 is the centroid of the C1—C6 benzene ring.

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
C8—H8 <i>A</i> ...O1 ⁱ	0.98	2.58	3.439 (5)	147
C5—H5...Cg1 ⁱⁱ	0.95	2.95	3.765 (4)	129

Symmetry codes: (i) $-x, y+1/2, -z+3/2$; (ii) $-x+1/2, y-1/2, z$.**(II) 4-Bromo-3-(methoxymethoxy)benzoic acid***Crystal data*C₉H₉BrO₄ $M_r = 261.07$ Triclinic, $P\bar{1}$

Hall symbol: -P 1

 $a = 7.7211$ (3) Å $b = 9.6881$ (4) Å $c = 14.2627$ (6) Å $\alpha = 73.635$ (1)° $\beta = 77.664$ (1)° $\gamma = 69.577$ (1)° $V = 951.40$ (7) Å³ $Z = 4$ $F(000) = 520$ $D_x = 1.823$ Mg m⁻³

Melting point: 433 K

Cu $K\alpha$ radiation, $\lambda = 1.54178$ Å

Cell parameters from 123 reflections

 $\theta = 3.3$ – 64.4 ° $\mu = 5.82$ mm⁻¹ $T = 173$ K

Prism, colourless

 $0.28 \times 0.25 \times 0.22$ mm*Data collection*

Bruker APEXII

diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

phi and φ scans

Absorption correction: multi-scan

(SADABS; Bruker, 2009)

 $T_{\min} = 0.245$, $T_{\max} = 0.278$

11112 measured reflections

3031 independent reflections

2930 reflections with $I > 2\sigma(I)$ $R_{\text{int}} = 0.040$ $\theta_{\text{max}} = 64.4$ °, $\theta_{\text{min}} = 3.3$ ° $h = -8 \rightarrow 8$ $k = -11 \rightarrow 11$ $l = -15 \rightarrow 16$

*Refinement*Refinement on F^2

Least-squares matrix: full

 $R[F^2 > 2\sigma(F^2)] = 0.039$ $wR(F^2) = 0.120$ $S = 1.09$

3031 reflections

261 parameters

2 restraints

Primary atom site location: structure-invariant
direct methodsSecondary atom site location: difference Fourier
mapHydrogen site location: inferred from
neighbouring sitesH atoms treated by a mixture of independent
and constrained refinement $w = 1/[\sigma^2(F_o^2) + (0.0844P)^2 + 0.9411P]$ where $P = (F_o^2 + 2F_c^2)/3$ $(\Delta/\sigma)_{\max} = 0.001$ $\Delta\rho_{\max} = 0.67 \text{ e } \text{\AA}^{-3}$ $\Delta\rho_{\min} = -1.08 \text{ e } \text{\AA}^{-3}$ *Special details*

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

Refinement. Refinement of F^2 against ALL reflections. The weighted R -factor wR and goodness of fit S are based on F^2 , conventional R -factors R are based on F , with F set to zero for negative F^2 . The threshold expression of $F^2 > 2\sigma(F^2)$ is used only for calculating R -factors(gt) *etc.* and is not relevant to the choice of reflections for refinement. R -factors based on F^2 are statistically about twice as large as those based on F , and R -factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
Br1B	-0.40349 (5)	1.06722 (4)	0.30367 (2)	0.01682 (17)
Br1A	1.35506 (5)	0.17904 (4)	0.90000 (2)	0.01972 (17)
O3B	-0.0105 (3)	1.0670 (3)	0.23290 (16)	0.0159 (5)
O3A	0.9612 (3)	0.1908 (2)	0.96819 (15)	0.0164 (5)
O4B	0.2558 (3)	1.1441 (2)	0.20751 (16)	0.0204 (5)
O4A	0.6543 (3)	0.3227 (3)	1.02268 (16)	0.0223 (5)
O1A	0.5360 (3)	0.4837 (3)	0.70491 (16)	0.0210 (5)
O2A	0.7281 (3)	0.5390 (3)	0.56876 (16)	0.0184 (5)
O1B	0.4376 (3)	0.7142 (3)	0.47778 (18)	0.0219 (5)
O2B	0.2466 (4)	0.6594 (3)	0.61509 (17)	0.0220 (5)
C4A	0.8598 (4)	0.3942 (3)	0.7147 (2)	0.0113 (6)
C7B	0.2753 (5)	0.7246 (3)	0.5251 (2)	0.0141 (6)
C4B	0.1108 (5)	0.8140 (3)	0.4734 (2)	0.0138 (7)
C2A	0.9761 (5)	0.2599 (3)	0.8698 (2)	0.0135 (6)
C7A	0.6971 (5)	0.4759 (3)	0.6597 (2)	0.0125 (6)
C5A	1.0392 (5)	0.3920 (3)	0.6689 (2)	0.0150 (7)
H5A	1.0601	0.4358	0.6006	0.018*
C3A	0.8282 (5)	0.3270 (3)	0.8147 (2)	0.0134 (6)
H3A	0.7058	0.3274	0.8447	0.016*
C3B	0.1349 (5)	0.9014 (3)	0.3777 (2)	0.0127 (6)
H3B	0.2555	0.9065	0.3476	0.015*
C2B	-0.0179 (5)	0.9803 (3)	0.3272 (2)	0.0133 (7)
C5B	-0.0660 (5)	0.8083 (3)	0.5191 (2)	0.0153 (7)
H5B	-0.0822	0.7511	0.5846	0.018*

C6B	-0.2167 (5)	0.8863 (3)	0.4684 (2)	0.0153 (7)
H6B	-0.3375	0.8823	0.4987	0.018*
C1B	-0.1937 (5)	0.9706 (3)	0.3735 (2)	0.0148 (7)
C6A	1.1867 (5)	0.3251 (3)	0.7243 (2)	0.0155 (7)
H6A	1.3099	0.3221	0.6942	0.019*
C9A	0.7006 (6)	0.3967 (4)	1.0828 (3)	0.0285 (8)
H9A1	0.7188	0.3296	1.1481	0.043*
H9A2	0.5993	0.4899	1.0894	0.043*
H9A3	0.8157	0.4213	1.0522	0.043*
C8A	0.7818 (5)	0.1825 (4)	1.0171 (2)	0.0174 (7)
H8A1	0.7332	0.1324	0.9816	0.021*
H8A2	0.7952	0.1192	1.0847	0.021*
C8B	0.1691 (5)	1.0616 (4)	0.1787 (2)	0.0154 (7)
H8B1	0.2491	0.9549	0.1876	0.018*
H8B2	0.1561	1.1016	0.1077	0.018*
C1A	1.1533 (5)	0.2628 (3)	0.8237 (2)	0.0155 (7)
C9B	0.1791 (6)	1.3037 (4)	0.1746 (3)	0.0292 (8)
H9B1	0.1718	1.3290	0.1037	0.044*
H9B2	0.2588	1.3541	0.1875	0.044*
H9B3	0.0539	1.3378	0.2100	0.044*
H2A	0.634 (6)	0.593 (5)	0.541 (3)	0.035*
H2B	0.330 (6)	0.612 (5)	0.650 (3)	0.035*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1B	0.0115 (3)	0.0212 (2)	0.0153 (2)	-0.00265 (17)	-0.00350 (16)	-0.00218 (16)
Br1A	0.0121 (3)	0.0260 (2)	0.0181 (2)	-0.00483 (17)	-0.00408 (16)	-0.00011 (16)
O3B	0.0121 (12)	0.0222 (11)	0.0109 (11)	-0.0052 (10)	-0.0023 (9)	0.0005 (9)
O3A	0.0138 (12)	0.0219 (12)	0.0085 (10)	-0.0029 (10)	-0.0007 (9)	0.0005 (9)
O4B	0.0222 (13)	0.0189 (11)	0.0203 (12)	-0.0083 (10)	-0.0063 (9)	0.0003 (9)
O4A	0.0164 (13)	0.0267 (12)	0.0164 (11)	0.0021 (10)	-0.0016 (9)	-0.0052 (9)
O1A	0.0144 (14)	0.0289 (12)	0.0159 (11)	-0.0062 (11)	-0.0022 (9)	0.0004 (9)
O2A	0.0200 (13)	0.0212 (11)	0.0105 (11)	-0.0058 (10)	-0.0034 (9)	0.0023 (9)
O1B	0.0158 (13)	0.0243 (12)	0.0233 (12)	-0.0044 (10)	-0.0052 (10)	-0.0018 (10)
O2B	0.0237 (14)	0.0244 (12)	0.0149 (12)	-0.0091 (11)	-0.0088 (10)	0.0067 (9)
C4A	0.0135 (17)	0.0104 (13)	0.0110 (14)	-0.0045 (12)	-0.0002 (12)	-0.0042 (11)
C7B	0.0193 (18)	0.0122 (14)	0.0120 (15)	-0.0055 (13)	-0.0022 (12)	-0.0036 (11)
C4B	0.0182 (18)	0.0120 (14)	0.0125 (15)	-0.0053 (13)	-0.0016 (12)	-0.0042 (11)
C2A	0.0177 (18)	0.0124 (14)	0.0092 (15)	-0.0037 (13)	-0.0008 (12)	-0.0022 (12)
C7A	0.0156 (17)	0.0110 (14)	0.0125 (15)	-0.0046 (12)	-0.0007 (12)	-0.0053 (11)
C5A	0.0194 (18)	0.0115 (14)	0.0113 (14)	-0.0028 (13)	0.0009 (12)	-0.0027 (11)
C3A	0.0137 (17)	0.0145 (14)	0.0130 (15)	-0.0063 (13)	-0.0002 (12)	-0.0032 (12)
C3B	0.0108 (16)	0.0137 (14)	0.0137 (15)	-0.0039 (12)	-0.0006 (12)	-0.0038 (12)
C2B	0.0173 (18)	0.0143 (14)	0.0096 (15)	-0.0066 (13)	-0.0016 (12)	-0.0025 (11)
C5B	0.0214 (18)	0.0152 (14)	0.0102 (14)	-0.0073 (13)	-0.0012 (12)	-0.0025 (11)
C6B	0.0153 (18)	0.0180 (15)	0.0151 (15)	-0.0067 (13)	0.0005 (12)	-0.0073 (12)
C1B	0.0169 (18)	0.0116 (14)	0.0166 (16)	-0.0022 (13)	-0.0034 (13)	-0.0061 (12)

C6A	0.0147 (18)	0.0166 (15)	0.0161 (15)	-0.0076 (13)	0.0015 (13)	-0.0044 (12)
C9A	0.033 (2)	0.0260 (18)	0.0227 (18)	-0.0002 (16)	-0.0086 (15)	-0.0083 (14)
C8A	0.0133 (18)	0.0233 (16)	0.0134 (15)	-0.0059 (14)	-0.0005 (12)	-0.0013 (12)
C8B	0.0127 (17)	0.0217 (16)	0.0125 (15)	-0.0066 (13)	-0.0011 (12)	-0.0038 (12)
C1A	0.0160 (18)	0.0133 (14)	0.0158 (16)	-0.0020 (13)	-0.0020 (13)	-0.0041 (12)
C9B	0.037 (2)	0.0185 (16)	0.033 (2)	-0.0084 (16)	-0.0077 (16)	-0.0052 (14)

Geometric parameters (Å, °)

Br1B—C1B	1.897 (3)	C2A—C1A	1.391 (5)
Br1A—C1A	1.901 (3)	C5A—C6A	1.387 (5)
O3B—C2B	1.371 (4)	C5A—H5A	0.9500
O3B—C8B	1.428 (4)	C3A—H3A	0.9500
O3A—C2A	1.374 (4)	C3B—C2B	1.385 (5)
O3A—C8A	1.430 (4)	C3B—H3B	0.9500
O4B—C8B	1.390 (4)	C2B—C1B	1.400 (5)
O4B—C9B	1.426 (4)	C5B—C6B	1.373 (5)
O4A—C8A	1.383 (4)	C5B—H5B	0.9500
O4A—C9A	1.426 (4)	C6B—C1B	1.380 (5)
O1A—C7A	1.260 (4)	C6B—H6B	0.9500
O2A—C7A	1.279 (4)	C6A—C1A	1.383 (5)
O2A—H2A	0.83 (3)	C6A—H6A	0.9500
O1B—C7B	1.275 (4)	C9A—H9A1	0.9800
O2B—C7B	1.271 (4)	C9A—H9A2	0.9800
O2B—H2B	0.82 (3)	C9A—H9A3	0.9800
C4A—C5A	1.395 (5)	C8A—H8A1	0.9900
C4A—C3A	1.398 (4)	C8A—H8A2	0.9900
C4A—C7A	1.482 (5)	C8B—H8B1	0.9900
C7B—C4B	1.476 (5)	C8B—H8B2	0.9900
C4B—C5B	1.394 (5)	C9B—H9B1	0.9800
C4B—C3B	1.402 (4)	C9B—H9B2	0.9800
C2A—C3A	1.386 (5)	C9B—H9B3	0.9800
C2B—O3B—C8B	117.7 (2)	C5B—C6B—C1B	120.4 (3)
C2A—O3A—C8A	118.1 (2)	C5B—C6B—H6B	119.8
C8B—O4B—C9B	113.7 (3)	C1B—C6B—H6B	119.8
C8A—O4A—C9A	113.3 (3)	C6B—C1B—C2B	121.2 (3)
C7A—O2A—H2A	116 (3)	C6B—C1B—Br1B	118.9 (3)
C7B—O2B—H2B	124 (4)	C2B—C1B—Br1B	119.9 (2)
C5A—C4A—C3A	120.9 (3)	C1A—C6A—C5A	119.5 (3)
C5A—C4A—C7A	120.5 (3)	C1A—C6A—H6A	120.2
C3A—C4A—C7A	118.5 (3)	C5A—C6A—H6A	120.2
O2B—C7B—O1B	123.3 (3)	O4A—C9A—H9A1	109.5
O2B—C7B—C4B	117.5 (3)	O4A—C9A—H9A2	109.5
O1B—C7B—C4B	119.1 (3)	H9A1—C9A—H9A2	109.5
C5B—C4B—C3B	120.6 (3)	O4A—C9A—H9A3	109.5
C5B—C4B—C7B	119.9 (3)	H9A1—C9A—H9A3	109.5
C3B—C4B—C7B	119.4 (3)	H9A2—C9A—H9A3	109.5

O3A—C2A—C3A	124.7 (3)	O4A—C8A—O3A	113.1 (3)
O3A—C2A—C1A	116.6 (3)	O4A—C8A—H8A1	109.0
C3A—C2A—C1A	118.6 (3)	O3A—C8A—H8A1	109.0
O1A—C7A—O2A	123.3 (3)	O4A—C8A—H8A2	109.0
O1A—C7A—C4A	118.9 (3)	O3A—C8A—H8A2	109.0
O2A—C7A—C4A	117.8 (3)	H8A1—C8A—H8A2	107.8
C6A—C5A—C4A	119.2 (3)	O4B—C8B—O3B	113.0 (3)
C6A—C5A—H5A	120.4	O4B—C8B—H8B1	109.0
C4A—C5A—H5A	120.4	O3B—C8B—H8B1	109.0
C2A—C3A—C4A	119.8 (3)	O4B—C8B—H8B2	109.0
C2A—C3A—H3A	120.1	O3B—C8B—H8B2	109.0
C4A—C3A—H3A	120.1	H8B1—C8B—H8B2	107.8
C2B—C3B—C4B	119.7 (3)	C6A—C1A—C2A	122.0 (3)
C2B—C3B—H3B	120.1	C6A—C1A—Br1A	119.1 (3)
C4B—C3B—H3B	120.1	C2A—C1A—Br1A	119.0 (2)
O3B—C2B—C3B	124.8 (3)	O4B—C9B—H9B1	109.5
O3B—C2B—C1B	116.4 (3)	O4B—C9B—H9B2	109.5
C3B—C2B—C1B	118.8 (3)	H9B1—C9B—H9B2	109.5
C6B—C5B—C4B	119.3 (3)	O4B—C9B—H9B3	109.5
C6B—C5B—H5B	120.4	H9B1—C9B—H9B3	109.5
C4B—C5B—H5B	120.4	H9B2—C9B—H9B3	109.5
O2B—C7B—C4B—C5B	-8.5 (4)	C4B—C3B—C2B—C1B	0.0 (4)
O1B—C7B—C4B—C5B	170.4 (3)	C3B—C4B—C5B—C6B	1.7 (4)
O2B—C7B—C4B—C3B	172.9 (3)	C7B—C4B—C5B—C6B	-176.9 (3)
O1B—C7B—C4B—C3B	-8.3 (4)	C4B—C5B—C6B—C1B	-0.6 (4)
C8A—O3A—C2A—C3A	2.2 (4)	C5B—C6B—C1B—C2B	-0.8 (4)
C8A—O3A—C2A—C1A	-178.5 (3)	C5B—C6B—C1B—Br1B	176.5 (2)
C5A—C4A—C7A—O1A	-175.6 (3)	O3B—C2B—C1B—C6B	180.0 (3)
C3A—C4A—C7A—O1A	0.8 (4)	C3B—C2B—C1B—C6B	1.1 (4)
C5A—C4A—C7A—O2A	2.4 (4)	O3B—C2B—C1B—Br1B	2.7 (4)
C3A—C4A—C7A—O2A	178.9 (3)	C3B—C2B—C1B—Br1B	-176.2 (2)
C3A—C4A—C5A—C6A	-1.5 (4)	C4A—C5A—C6A—C1A	-0.4 (4)
C7A—C4A—C5A—C6A	174.8 (3)	C9A—O4A—C8A—O3A	-65.8 (3)
O3A—C2A—C3A—C4A	-180.0 (3)	C2A—O3A—C8A—O4A	-65.6 (3)
C1A—C2A—C3A—C4A	0.7 (4)	C9B—O4B—C8B—O3B	-74.1 (3)
C5A—C4A—C3A—C2A	1.3 (4)	C2B—O3B—C8B—O4B	-76.2 (3)
C7A—C4A—C3A—C2A	-175.1 (3)	C5A—C6A—C1A—C2A	2.5 (4)
C5B—C4B—C3B—C2B	-1.4 (4)	C5A—C6A—C1A—Br1A	-177.4 (2)
C7B—C4B—C3B—C2B	177.2 (3)	O3A—C2A—C1A—C6A	178.0 (3)
C8B—O3B—C2B—C3B	7.7 (4)	C3A—C2A—C1A—C6A	-2.6 (4)
C8B—O3B—C2B—C1B	-171.1 (3)	O3A—C2A—C1A—Br1A	-2.2 (4)
C4B—C3B—C2B—O3B	-178.8 (3)	C3A—C2A—C1A—Br1A	177.2 (2)

Hydrogen-bond geometry (\AA , $^\circ$)

<i>D</i> —H \cdots <i>A</i>	<i>D</i> —H	H \cdots <i>A</i>	<i>D</i> \cdots <i>A</i>	<i>D</i> —H \cdots <i>A</i>
O2A—H2A \cdots O1B	0.84 (5)	1.80 (5)	2.635 (4)	178 (5)

<i>O2B—H2B…O1A</i>	0.82 (5)	1.81 (5)	2.621 (4)	167 (5)
<i>C8B—H8B2…O3Aⁱ</i>	0.99	2.52	3.420 (4)	150

Symmetry code: (i) $x-1, y+1, z-1$.