

Acta Crystallographica Section E

Structure Reports

Online

ISSN 1600-5368

3,5-Dibromo-2-hydroxybenzoic acid

Chong-Bo Liu,^a Dan-Dan Chen^b and Hui-Liang Wen^{c*}

^aCollege of Environmental and Chemical Engineering, Nanchang University of Aeronautics, Nanchang 330063, People's Republic of China, ^bDepartment of Chemistry, Nanchang University, Nanchang 330047, People's Republic of China, and ^cState Key Laboratory of Food Science and Technology, Nanchang University, Nanchang 330047, People's Republic of China
Correspondence e-mail: hlwen70@163.com

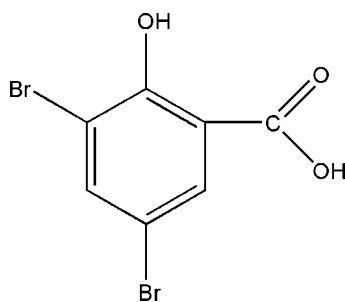
Received 16 November 2007; accepted 22 November 2007

Key indicators: single-crystal X-ray study; $T = 293$ K; mean $\sigma(\text{C}-\text{C}) = 0.005$ Å; R factor = 0.031; wR factor = 0.061; data-to-parameter ratio = 14.4.

The title compound, $\text{C}_7\text{H}_4\text{Br}_2\text{O}_3$, has an intramolecular $\text{O}-\text{H}\cdots\text{O}=\text{C}$ hydrogen bond and aggregates to form hydrogen-bonded dimers *via* $\text{O}-\text{H}\cdots\text{O}$ interactions. The formation of zigzag one-dimensional molecular tapes *via* $\text{C}-\text{H}\cdots\text{Br}$ interactions and $\pi-\pi$ stacking interactions (interplanar separation = 3.42 Å) completes the crystal structure.

Related literature

For related literature, see: Chiari *et al.* (1981); Jin & Xiao (2005).



Experimental

Crystal data

$\text{C}_7\text{H}_4\text{Br}_2\text{O}_3$
 $M_r = 295.92$

Monoclinic, $C2/c$
 $a = 10.770$ (3) Å

$b = 11.082$ (3) Å
 $c = 14.879$ (4) Å
 $\beta = 105.606$ (3)°
 $V = 1710.4$ (8) Å³
 $Z = 8$

Mo $K\alpha$ radiation
 $\mu = 9.44$ mm⁻¹
 $T = 293$ (2) K
 $0.50 \times 0.31 \times 0.21$ mm

Data collection

Bruker SMART CCD diffractometer
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)
 $T_{\min} = 0.088$, $T_{\max} = 0.243$
(expected range = 0.050–0.138)

6362 measured reflections
1599 independent reflections
1286 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.032$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.031$
 $wR(F^2) = 0.061$
 $S = 1.79$
1599 reflections

111 parameters
H-atom parameters constrained
 $\Delta\rho_{\text{max}} = 0.82$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.81$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{O1}-\text{H1}\cdots\text{O2}^i$	0.82	1.87	2.684 (3)	175
$\text{O3}-\text{H3}\cdots\text{O2}$	0.82	1.93	2.648 (3)	145
$\text{C3}-\text{H3A}\cdots\text{Br1}^{ii}$	0.93	2.89	3.810 (3)	172

Symmetry codes: (i) $-x + \frac{5}{2}, -y - \frac{1}{2}, -z + 1$; (ii) $-x + \frac{5}{2}, -y + \frac{1}{2}, -z + 1$.

Data collection: SMART (Bruker, 1998); cell refinement: SAINT (Bruker, 1998); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 1997); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: SHELXTL (Bruker, 1998); software used to prepare material for publication: SHELXTL.

This work was supported by the National Natural Science Foundation of China (grant No. 20662007).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: GG2052).

References

- Bruker (1998). SHELXTL, SAINT and SMART (Version 5.0). Bruker AXS Inc., Madison, Wisconsin, USA.
Chiari, G., Fronczek, F. R., Davis, S. T. & Gandour, R. D. (1981). *Acta Cryst.* **B37**, 1623–1625.
Jin, L.-F. & Xiao, F.-P. (2005). *Acta Cryst.* **E61**, o1198–o1199.
Sheldrick, G. M. (1996). SADABS. University of Göttingen, Germany.
Sheldrick, G. M. (1997). SHELXS97 and SHELXL97. University of Göttingen, Germany.

supplementary materials

Acta Cryst. (2008). E64, o3 [doi:10.1107/S1600536807062320]

3,5-Dibromo-2-hydroxybenzoic acid

C.-B. Liu, D.-D. Chen and H.-L. Wen

Comment

The compound 2-hydroxybenzoic acid (salicylic acid) and its derivatives have been widely studied as medicines or important active pharmaceutical intermediates (Chiari *et al.*, 1981; Jin & Xiao, 2005). Herein, we report the crystal structure of the title compound, (I).

In (I) (Fig. 1), the atoms O1, O2, O3, Br1, Br2, C1 and aromatic ring {C2, ..., C7} are essentially coplanar with a mean deviation of 0.014 Å. The crystal packing is stabilized by intramolecular and intermolecular O—H...O and C—H...Br hydrogen bonds (Table 1).

Experimental

Crystals appropriate for data collection were obtained by recrystallization from ethanol (m.p. 500–501 K).

Refinement

The hydroxyl H atom and the carboxylate H atom were located from difference Fourier map but were refined using the riding model with O—H distance restrained to 0.82 Å and $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{O})$; while all other H atoms were placed at geometrical idealized positions with C—H = 0.93 Å and $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$.

Figures

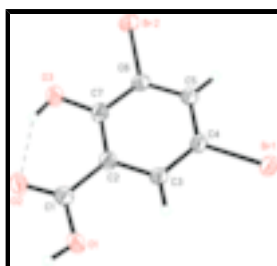


Fig. 1. The molecular structure of the title compound depicted with 30% probability displacement ellipsoids.

3,5-Dibromo-2-hydroxybenzoic acid

Crystal data

$\text{C}_7\text{H}_4\text{Br}_2\text{O}_3$

$M_r = 295.92$

Monoclinic, $C2/c$

Hall symbol: $-C 2yc$

$F_{000} = 1120$

$D_x = 2.298 \text{ Mg m}^{-3}$

Melting point: 500 K

Mo $K\alpha$ radiation

$\lambda = 0.71073 \text{ \AA}$

supplementary materials

$a = 10.770 (3) \text{ \AA}$	Cell parameters from 2346 reflections
$b = 11.082 (3) \text{ \AA}$	$\theta = 2.7\text{--}26.3^\circ$
$c = 14.879 (4) \text{ \AA}$	$\mu = 9.44 \text{ mm}^{-1}$
$\beta = 105.606 (3)^\circ$	$T = 293 (2) \text{ K}$
$V = 1710.4 (8) \text{ \AA}^3$	Block, light-yellow
$Z = 8$	$0.50 \times 0.31 \times 0.21 \text{ mm}$

Data collection

Bruker SMART CCD diffractometer	1599 independent reflections
Radiation source: fine-focus sealed tube	1286 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{\text{int}} = 0.032$
$T = 293(2) \text{ K}$	$\theta_{\text{max}} = 25.5^\circ$
φ and ω scans	$\theta_{\text{min}} = 2.7^\circ$
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	$h = -13 \rightarrow 13$
$T_{\text{min}} = 0.088$, $T_{\text{max}} = 0.243$	$k = -13 \rightarrow 13$
6362 measured reflections	$l = -17 \rightarrow 18$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.031$	H-atom parameters constrained
$wR(F^2) = 0.061$	$w = 1/[\sigma^2(F_o^2)]$
$S = 1.79$	where $P = (F_o^2 + 2F_c^2)/3$
1599 reflections	$(\Delta/\sigma)_{\text{max}} = 0.001$
111 parameters	$\Delta\rho_{\text{max}} = 0.82 \text{ e \AA}^{-3}$
Primary atom site location: structure-invariant direct methods	$\Delta\rho_{\text{min}} = -0.81 \text{ e \AA}^{-3}$
	Extinction correction: none

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'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	$U_{\text{iso}}^*/U_{\text{eq}}$
Br1	1.06441 (4)	0.35585 (3)	0.42603 (3)	0.07017 (19)
Br2	0.68570 (4)	0.02983 (4)	0.22295 (3)	0.05891 (17)
O1	1.2480 (2)	-0.0861 (2)	0.49804 (18)	0.0543 (7)
H1	1.2875	-0.1464	0.5219	0.081*
O2	1.1105 (2)	-0.2248 (2)	0.41973 (16)	0.0545 (7)
O3	0.8866 (2)	-0.1506 (2)	0.31093 (18)	0.0560 (7)
H3	0.9422	-0.2005	0.3341	0.084*
C1	1.1390 (3)	-0.1185 (3)	0.4393 (2)	0.0422 (9)
C2	1.0523 (3)	-0.0171 (3)	0.3998 (2)	0.0376 (8)
C3	1.0907 (3)	0.1018 (3)	0.4246 (2)	0.0403 (9)
H3A	1.1712	0.1172	0.4654	0.048*
C4	1.0092 (3)	0.1959 (3)	0.3885 (2)	0.0412 (9)
C5	0.8890 (3)	0.1766 (3)	0.3280 (2)	0.0414 (9)
H5	0.8349	0.2410	0.3040	0.050*
C6	0.8509 (3)	0.0593 (3)	0.3040 (2)	0.0395 (9)
C7	0.9306 (3)	-0.0390 (3)	0.3386 (2)	0.0387 (8)

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.0482 (3)	0.0329 (2)	0.1040 (4)	-0.00311 (18)	-0.0233 (2)	0.0025 (2)
Br2	0.0371 (2)	0.0526 (3)	0.0726 (3)	-0.00593 (18)	-0.0101 (2)	-0.0044 (2)
O1	0.0511 (16)	0.0353 (14)	0.0606 (17)	0.0064 (12)	-0.0126 (13)	0.0036 (12)
O2	0.0583 (17)	0.0327 (15)	0.0596 (17)	0.0041 (12)	-0.0064 (13)	-0.0017 (12)
O3	0.0514 (16)	0.0348 (15)	0.0690 (18)	-0.0019 (12)	-0.0056 (13)	-0.0074 (13)
C1	0.039 (2)	0.043 (2)	0.041 (2)	0.0019 (17)	0.0051 (17)	0.0040 (17)
C2	0.040 (2)	0.033 (2)	0.037 (2)	0.0013 (16)	0.0032 (17)	0.0044 (15)
C3	0.0332 (19)	0.037 (2)	0.043 (2)	-0.0024 (16)	-0.0032 (16)	0.0013 (16)
C4	0.041 (2)	0.0296 (19)	0.047 (2)	-0.0045 (15)	0.0024 (18)	0.0026 (16)
C5	0.0325 (19)	0.036 (2)	0.050 (2)	0.0017 (16)	0.0011 (17)	0.0028 (17)
C6	0.0326 (19)	0.041 (2)	0.041 (2)	-0.0032 (16)	0.0026 (16)	-0.0023 (16)
C7	0.042 (2)	0.035 (2)	0.037 (2)	-0.0032 (17)	0.0082 (17)	-0.0017 (16)

Geometric parameters (\AA , $^\circ$)

Br1—C4	1.905 (3)	C2—C3	1.401 (4)
Br2—C6	1.890 (3)	C2—C7	1.402 (5)
O1—C1	1.311 (4)	C3—C4	1.375 (5)
O1—H1	0.8200	C3—H3A	0.9300
O2—C1	1.233 (4)	C4—C5	1.381 (5)
O3—C7	1.348 (4)	C5—C6	1.380 (4)
O3—H3	0.8200	C5—H5	0.9300
C1—C2	1.478 (5)	C6—C7	1.397 (5)
C1—O1—H1	109.5	C3—C4—Br1	118.3 (3)

supplementary materials

C7—O3—H3	109.5	C5—C4—Br1	119.9 (3)
O2—C1—O1	122.7 (3)	C6—C5—C4	118.4 (3)
O2—C1—C2	122.9 (3)	C6—C5—H5	120.8
O1—C1—C2	114.4 (3)	C4—C5—H5	120.8
C3—C2—C7	119.5 (3)	C5—C6—C7	122.0 (3)
C3—C2—C1	120.0 (3)	C5—C6—Br2	119.4 (3)
C7—C2—C1	120.4 (3)	C7—C6—Br2	118.6 (3)
C4—C3—C2	119.9 (3)	O3—C7—C6	118.2 (3)
C4—C3—H3A	120.1	O3—C7—C2	123.2 (3)
C2—C3—H3A	120.1	C6—C7—C2	118.5 (3)
C3—C4—C5	121.7 (3)		
O2—C1—C2—C3	-179.7 (3)	C4—C5—C6—C7	-0.5 (5)
O1—C1—C2—C3	1.3 (5)	C4—C5—C6—Br2	179.0 (3)
O2—C1—C2—C7	1.2 (6)	C5—C6—C7—O3	-179.3 (3)
O1—C1—C2—C7	-177.8 (3)	Br2—C6—C7—O3	1.2 (4)
C7—C2—C3—C4	-0.3 (5)	C5—C6—C7—C2	0.5 (5)
C1—C2—C3—C4	-179.3 (3)	Br2—C6—C7—C2	-179.0 (2)
C2—C3—C4—C5	0.2 (5)	C3—C2—C7—O3	179.7 (3)
C2—C3—C4—Br1	178.9 (2)	C1—C2—C7—O3	-1.2 (5)
C3—C4—C5—C6	0.2 (5)	C3—C2—C7—C6	-0.1 (5)
Br1—C4—C5—C6	-178.5 (2)	C1—C2—C7—C6	179.0 (3)

Hydrogen-bond geometry (Å, °)

<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>
O1—H1 \cdots O2 ⁱ	0.82	1.87	2.684 (3)	175
O3—H3 \cdots O2	0.82	1.93	2.648 (3)	145
C3—H3A \cdots Br1 ⁱⁱ	0.93	2.89	3.810 (3)	172

Symmetry codes: (i) $-x+5/2, -y-1/2, -z+1$; (ii) $-x+5/2, -y+1/2, -z+1$.

Fig. 1

