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5-Bromo-2-hydroxybenzene-1,3-dicarbaldehyde

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Key indicators

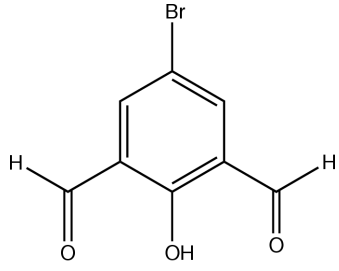
Single-crystal X-ray study
T = 203 K
Mean σ(C–C) = 0.006 Å
R factor = 0.045
wR factor = 0.122
Data-to-parameter ratio = 11.8

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

In the crystal structure of the title compound, $C_8H_5BrO_3$, two molecules are linked about inversion centres by C—H···Br hydrogen bonds. The structure is also characterized by intra- and intermolecular hydrogen bonds between hydroxyl and aldehyde O atoms.

Comment

5-Bromo-2-hydroxybenzene-1,3-dicarbaldehyde, (I), is a precursor in the synthesis of Robson-type ligands (Pilkington & Robson, 1970; Fenton, 1999). Bond lengths and angles are in the usual ranges. The molecule is planar with only minor distortions from the mean plane formed by non-H atoms; the deviations range from 0.004 (4) (for C6) to 0.126 (3) Å (O7). The crystal structure is also characterized by intra- and intermolecular hydrogen bonds (Table 1).



(I)

Experimental

The title compound was synthesized according to previously reported methods (Taniguchi, 1984; Xie *et al.*, 1994). To a solution of 4-bromo-2,6-dihydroxymethylphenol (40 g, 0.17 mol) in $CHCl_3$ (1 l), MnO_2 (232 g, 2.66 mol) was added. The resulting mixture was stirred under reflux for 48 h and cooled to room temperature. Then the solution was filtered and the solid was washed twice with 100 ml of $CHCl_3$. The washings were added to the original filtrate and the combined solution was evaporated to yield a yellow solid (22 g, 55% yield). Crystals were obtained by the slow evaporation of a $CHCl_3$ solution. ^{1}H NMR (200 MHz, $CDCl_3$, p.p.m.): 11.55 (*s*, 1H), 10.20 (*s*, 2H), 8.07 (*s*, 2H).

Crystal data

$C_8H_5BrO_3$
M_r = 229.03
Monoclinic, $P2_1/n$
a = 7.5206 (15) Å
b = 3.9367 (8) Å
c = 25.881 (5) Å
β = 96.84 (3)°
V = 760.8 (3) Å^3
Z = 4
D_x = 2.000 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 3595 reflections
θ = 1.6–26.8°
μ = 5.36 mm^{-1}
T = 203 (2) K
Needle, yellow
0.15 × 0.06 × 0.04 mm

Data collection

Siemens SMART CCD diffractometer
ω scans (1271 frames, 0.30°, 10 s, detector distance 5.5 cm, detector angle 23.0°)
Absorption correction: multi-scan (Blessing, 1995)
T_{min} = 0.528, T_{max} = 0.807
3595 measured reflections
1476 independent reflections
1280 reflections with $I > 2\sigma(I)$
R_{int} = 0.108
θ_{max} = 26.8°
$h = -7 \rightarrow 9$
$k = -4 \rightarrow 4$
$l = -31 \rightarrow 31$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.045
$wR(F^2)$ = 0.122
S = 1.15
1476 reflections
125 parameters
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.0504P)^2 + 1.1160P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.021
$\Delta\rho_{max}$ = 0.62 e Å^{-3}
$\Delta\rho_{min}$ = −0.77 e Å^{-3}

Table 1
Hydrogen-bonding geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O4—H4···O8	0.89	1.79	2.608 (7)	151
O4—H4···O4^{i}	0.89	2.47	2.935 (7)	113
C2—H2···Br^{ii}	1.00	2.93	3.921 (5)	173
C6—H6···O7^{iii}	0.95	2.57	3.495 (9)	164
C7—H7···O4	0.94	2.40	2.779 (8)	104

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

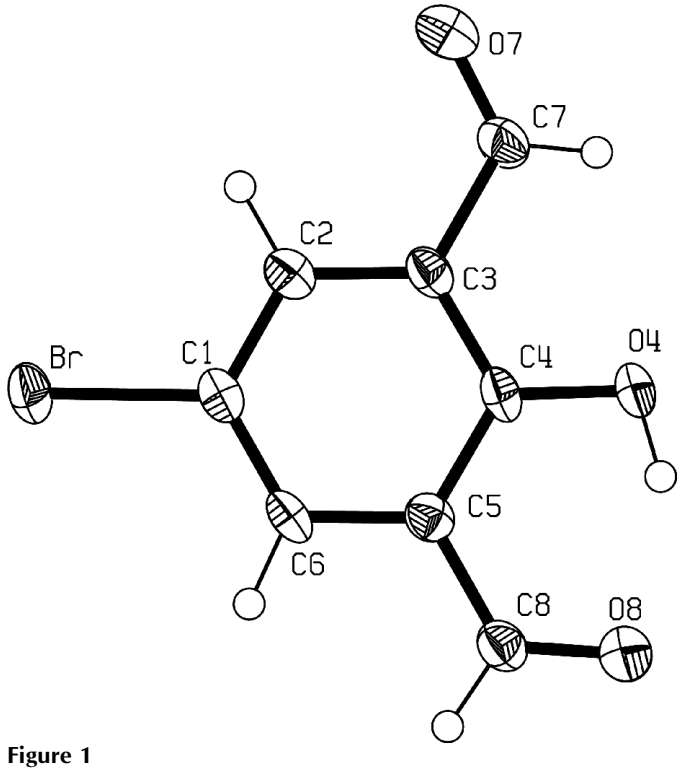


Figure 1
ORTEP view (*SHELXTL*; Siemens, 1996) of 5-bromo-2-hydroxybenzene-1,3-dicarbaldehyde showing displacement ellipsoids at the 50% probability level.

Data collection: *SMART* (Siemens, 1994–1996); cell refinement: *SAINT* (Siemens, 1994–1996); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *SHELXTL* (Siemens, 1996); software used to prepare material for publication: *SHELXL*97.

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