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3,5-Dibromosalicyl alcohol hemihydrate

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.009 Å
R factor = 0.041
wR factor = 0.117
Data-to-parameter ratio = 13.7

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

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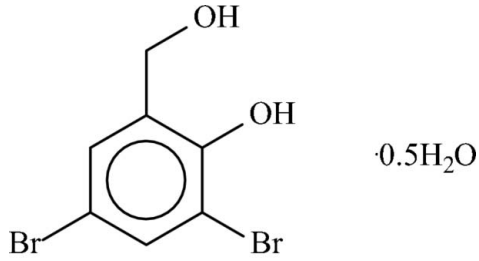
The asymmetric unit of the title compound, $C_7H_6Br_2O_2\cdot 0.5H_2O$, contains two independent 3,5-dibromosalicyl alcohol molecules and one water molecule. The salicyl alcohol molecules interact with each other and with the water molecule through O—H···O hydrogen bonding, giving rise to a layer structure.

Comment

The biostatic compound (Arct *et al.*, 1964) 3,5-dibromosalicyl alcohol can be synthesized by the direct bromination of salicyl alcohol (Brink, 1965) or from the reaction of 2,4-dibromophenol with trioxymethylene (Ziegler *et al.*, 1943). It is readily obtained by the reduction of 3,5-dibromosalicylaldehyde (see *Experimental*). The compound crystallizes from water as a hemihydrate, (I); there are two independent molecules of 3,5-dibromosalicyl alcohol, and they interact with the water molecule and with each other, forming a hydrogen-bonded layer structure (Fig. 1 and Table 1).



(I)

Experimental

3,5-Dibromosalicylaldehyde (4.1 g, 0.02 mol) was reduced by sodium borohydride (0.76 g, 0.02 mol) in ethanol (50 ml). The mixture was stirred for 2 h until the organic reactant could no longer be detected by thin-layer chromatography (petroleum ether/ethyl acetate, 5/3 *v*/*v*). The mixture was filtered and the solvent removed. Single crystals were obtained by recrystallization from water (melting point 411 K, yield 90%).

Crystal data

$C_7H_6Br_2O_2\cdot 0.5H_2O$
M_r = 290.95
Triclinic, $P\overline{1}$
a = 7.034 (1) Å
b = 8.484 (1) Å
c = 15.569 (2) Å
α = 99.682 (2)°
β = 91.695 (2)°
γ = 101.661 (2)°
V = 895.0 (2) Å^3
Z = 4
D_x = 2.159 Mg m^{-3}
Mo *Kα* radiation
μ = 9.01 mm^{-1}
T = 293 (2) K
Block, colourless
0.48 × 0.20 × 0.18 mm

Data collection

Bruker APEX2 area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.098, T_{max} = 0.294
(expected range = 0.066–0.197)
4711 measured reflections
3107 independent reflections
2351 reflections with $I > 2\sigma(I)$
R_{int} = 0.026
θ_{max} = 25.0°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.041
$wR(F^2)$ = 0.117
S = 1.04
3107 reflections
227 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0641P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.65 e Å^{-3}
$\Delta\rho_{min}$ = −0.49 e Å^{-3}
Extinction correction: *SHELXL97*
Extinction coefficient: 0.027 (2)

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1*o*···O1*w*	0.85 (1)	2.05 (4)	2.791 (6)	146 (6)
O2—H2*o*···O4^{i}	0.85 (1)	1.99 (2)	2.828 (6)	169 (7)
O4—H4*o*···O1*w*	0.85 (1)	1.97 (3)	2.771 (6)	157 (7)
O1*w*—H1*w*1···O2^{ii}	0.85 (1)	2.05 (2)	2.862 (6)	160 (6)
O1*w*—H1*w*2···O2^{iii}	0.85 (1)	1.98 (2)	2.803 (6)	165 (6)

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

Carbon-bound H atoms were placed at calculated positions (C—H = 0.93–0.97 Å) and were included in the refinement in the riding-model approximation, with U_{iso}(H) values set at 1.2 times U_{eq}(C). The hydroxy and water H atoms were located in a difference Fourier map, and were refined with distance restraints of O—H = 0.85 (1) Å and H···H = 1.39 (1) Å; their displacement parameters were similarly tied.

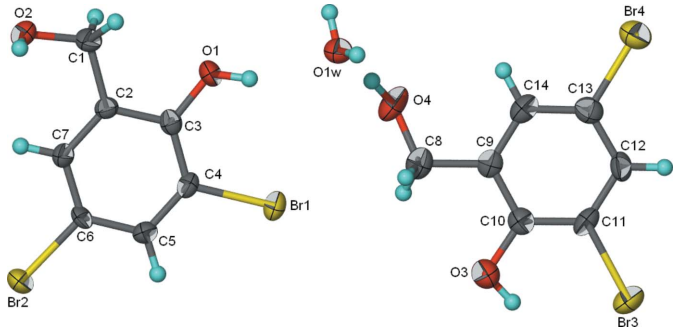


Figure 1
The asymmetric unit of (I). Displacement ellipsoids are drawn at the 50% probability level, and H atoms as spheres of arbitrary radii.

Data collection: *APEX2* (Bruker, 2004); cell refinement: *SAINT* (Bruker, 2004); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *X-SEED* (Barbour, 2001); software used to prepare material for publication: *SHELXL97*.

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References

Arct, J., Eckstein, Z. & Krzywicka, H. P. (1964). *Przem. Chem.* **43**, 87–91. (In Polish.)
Barbour, L. J. (2001). *J. Supramol. Chem.* **1**, 189–191.
Brink, M. (1965). *Acta Chem. Scand.* **19**, 255–256.
Bruker (2004). *APEX2* (Version 6.36A) and *SAINT* (Version 6.36A). Bruker AXS Inc., Madison, Wisconsin, USA.
Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
Sheldrick, G. M. (1997). *SHELXL97* and *SHELXS97*. University of Göttingen, Germany.
Ziegler, E., Meralla, H. & Simmler, I. (1943). *Ber. Deutsch Chem. Ges. Reihe B*, **76**, 664–649. (In German.)