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2-Bromofumaric acid

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Single crystals of 2-bromofumaric acid, $C_4H_3BrO_4$, were obtained from an aqueous solution of racemic 2,3-dibromosuccinic acid and (−)-quinine. The title compound crystallizes with two molecules in the asymmetric unit. The structure is stabilized by O—H···O hydrogen bonds forming alternating chains.

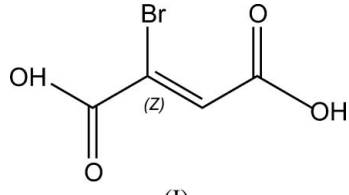
Key indicators

Single-crystal X-ray study
T = 299 K
Mean σ(C–C) = 0.005 Å
R factor = 0.037
wR factor = 0.072
Data-to-parameter ratio = 16.2

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

Comment

We are currently studying the structures and the chemical behaviour of some simple dicarboxylic acids, such as 2,3-dibromosuccinic acid. The structure of the racemate of this acid was determined some time ago (Bolte & Degen, 2000). Recently, we reported the structure of the *meso* form (Eriksson *et al.*, 2006). In an attempt to obtain the pure enantiomer, we attempted to grow crystals of salts of the acid with different chiral bases. However, the reaction with (−)-quinine yielded 2-bromofumaric acid, (I), apparently as the product of an elimination reaction.



(I)

The asymmetric unit of (I) contains two molecules of 2-bromofumaric acid, *A* and *B*, and their geometry is unexcep-

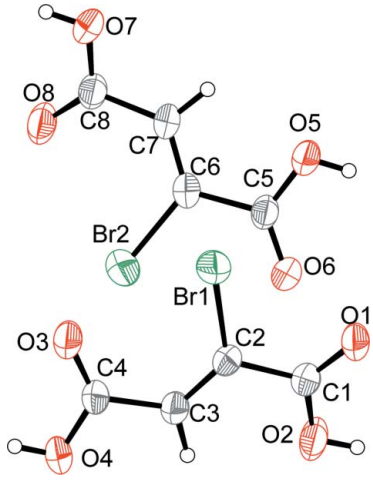


Figure 1
The contents of the asymmetric unit of (I). Displacement ellipsoids are drawn at the 50% probability level.

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tional (Fig. 1). Each *A* molecule binds through O—H···O hydrogen bonds *via* the carboxy groups to two adjacent *B* molecules and *vice versa*, to form closed hydrogen-bonded loops (Table 1 and Fig. 2). Hence, chains of the sequence. . . . *ABAB* . . . are obtained, which run along the (101) direction.

Experimental

The synthesis of racemic 2,3-dibromosuccinic acid has been described earlier (Fischer, 2006). The acid (276 mg) was dissolved together with (−)-quinine (Fluka, >98.0%; 163 mg) in demineralized water (5 ml). The solution was heated to 323 K for 1 h and then set aside for evaporation at room temperature. Single crystals of the title compound were obtained within a few days.

Crystal data

$C_4H_3BrO_4$
M_r = 194.97
Triclinic, $P\overline{1}$
a = 7.8290 (4) Å
b = 8.3791 (4) Å
c = 10.2443 (3) Å
α = 69.572 (4)°
β = 70.383 (3)°
γ = 79.963 (4)°
V = 591.97 (5) Å^3
Z = 4
D_x = 2.188 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 6.88 mm^{-1}
T = 299 K
Fragment, colourless
0.30 × 0.30 × 0.30 mm

Data collection

Bruker Nonius KappaCCD area-detector diffractometer
φ and ω scans
Absorption correction: numerical (Herrendorf & Bärnighausen, 1997)
T_{min} = 0.581, T_{max} = 0.661
11936 measured reflections
2701 independent reflections
1821 reflections with $I > 2\sigma(I)$
R_{int} = 0.052
θ_{max} = 27.5°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.037
$wR(F^2)$ = 0.072
S = 1.05
2701 reflections
167 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0231P)^2 + 0.495P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.33 e Å^{-3}
$\Delta\rho_{min}$ = −0.44 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O7—H7O···O1^{i}	0.82	1.89	2.702 (3)	168
O4—H4···O6^{ii}	0.82	1.93	2.752 (3)	175
O2—H2···O8^{iii}	0.82	1.81	2.621 (3)	169
O5—H5···O3^{iv}	0.82	1.76	2.574 (3)	170

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

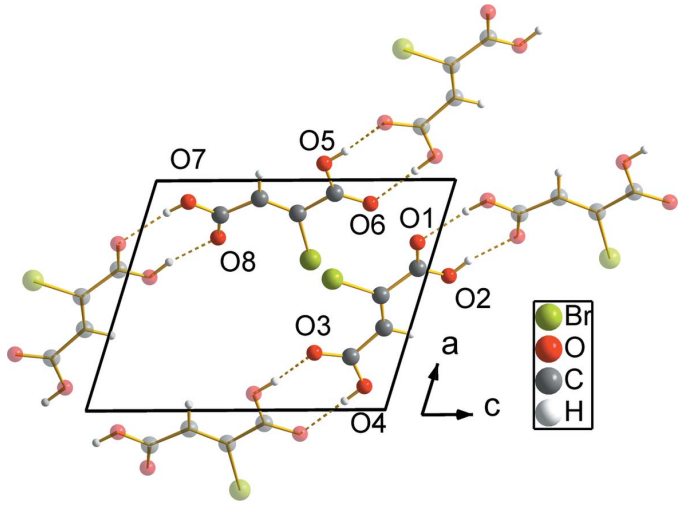

Figure 2
The hydrogen-bonding pattern in (I). Hydrogen bonds are drawn as dashed lines. Solid atoms represent the two molecules in the asymmetric unit.

All H atoms were placed in calculated positions, with C—H = 0.96 Å and O—H = 0.82 Å, and refined as riding, with U_{iso}(H) = 1.2U_{eq}(C) or 1.5U_{eq}(O).

Data collection: *COLLECT* (Nonius, 1999); cell refinement: *DIRAX/LSQ* (Duisenberg, 1992); data reduction: *EVALCCD* (Duisenberg *et al.*, 2003); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *DIAMOND* (Brandenburg, 2006); software used to prepare material for publication: *MAXUS* (Mackay *et al.*, 1999).

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