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rac-2,3-Dibromosuccinic acid

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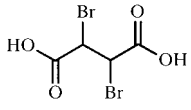
rac-2,3-Dibromosuccinic acid, $C_4H_4Br_2O_4$, is the product of the electrophilic addition of bromine to maleic acid. Whereas the carboxyl groups and the bromo ligands are in a *gauche* arrangement with respect to each other, the tertiary H atoms attached to the central C atoms are in a *trans* arrangement. The hydroxyl groups form hydrogen bonds with the carbonyl O atoms of neighbouring molecules.

Comment

We have prepared the title compound, (I), by a standard electrophilic addition reaction of bromine to maleic acid. The X-ray structure analysis was carried out in order to confirm the identity of the compound and to determine the conformation of the molecule. Bond lengths and angles do not show any unusual values. Whereas the carboxyl groups and bromo ligands are in a *gauche* arrangement with respect to each other, the tertiary H atoms attached to the central C atoms are in a *trans* arrangement. The crystal packing is stabilized by hydrogen bonds from the hydroxyl H atoms to the carbonyl O atoms of neighbouring molecules in such a way that a three-dimensional framework is formed. If the succinic acid is substituted with only one bromine ligand, as in (*R*)-2-bromo-1,4-butanedioic acid (Britten *et al.*, 1993), the conformation of the molecular skeleton and the hydrogen-bonding pattern are different; the carbon chain adopts a *trans* conformation and, as a result, the molecules crystallize in a linear fashion in a head-to-tail manner.



(I)

Experimental

The title compound was synthesized according to Hünig *et al.* (1979). After cooling a solution of 130 ml diethyl ether to 263 K, 0.11 mol bromine and 0.1 mol (11.6 g) maleic acid were added to the solution which was stirred continuously and warmed to room temperature. After 30 min, the solution was washed with sodium sulfite and water and then dried. The product was dissolved in petroleum ether and recrystallized.

Crystal data

$C_4H_4Br_2O_4$	D_x = 2.462 Mg m^{-3}
M_r = 275.89	Mo $K\alpha$ radiation
Monoclinic, $P2_1/c$	Cell parameters from 512 reflections
a = 12.725 (4) Å	θ = 1–20°
b = 6.1670 (10) Å	μ = 10.844 mm^{-1}
c = 10.228 (2) Å	T = 173 (2) K
β = 111.980 (10)°	Block, colourless
V = 744.3 (3) Å^3	0.30 × 0.20 × 0.15 mm
Z = 4	

Data collection

Siemens CCD three-circle diffractometer	R_{int} = 0.073
ω scans	θ_{max} = 26.37°
Absorption correction: empirical (*SADABS*; Sheldrick, 1996)	$h = -15 \rightarrow 15$
T_{min} = 0.090, T_{max} = 0.193	$k = -7 \rightarrow 7$
12 264 measured reflections	$l = -12 \rightarrow 12$
1517 independent reflections	123 standard reflections
1356 reflections with $I > 2\sigma(I)$	frequency: 1000 min
	intensity decay: none

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0464P)^2 + 0.0831P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)]$ = 0.031	$(\Delta/\sigma)_{max}$ = 0.001
$wR(F^2)$ = 0.081	$\Delta\rho_{max}$ = 1.16 e Å^{-3}
S = 1.119	$\Delta\rho_{min}$ = −0.72 e Å^{-3}
1517 reflections	Extinction correction: *SHELXL*97
94 parameters	Extinction coefficient: 0.0049 (8)
H-atom parameters constrained	

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

D—H···A	D—H	H···A	D···A	D—H···A
O12—H12···O41^{i}	0.84	1.87	2.702 (3)	172
O42—H42···O11^{ii}	0.84	1.91	2.706 (3)	159

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

All H atoms were located by difference Fourier synthesis and refined with fixed individual displacement parameters [U(H) = 1.2U_{eq}(C) or 1.2U_{eq}(O)] using a riding model with C—H = 1.00 Å or O—H = 0.84 Å. The OH groups were allowed to rotate about the C—O axis.

Data collection: *SMART* (Siemens, 1995); cell refinement: *SMART*; data reduction: *SAINT* (Siemens, 1995); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1990); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997).

References

Britten, J. F., Howard-Lock, H. E., Leblanc, D. & Lock, C. J. L. (1993). *Acta Cryst.* C**49**, 1222–1224.
Hünig, S., Märkl, G. & Sauer, J. (1979). In *Integriertes Organisches Praktikum*. Weinheim: VCH.
Sheldrick, G. M. (1990). *Acta Cryst.* A**46**, 467–473.
Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
Sheldrick, G. M. (1997). *SHELXL*97. University of Göttingen, Germany.
Siemens (1995). *SMART* and *SAINT*. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.