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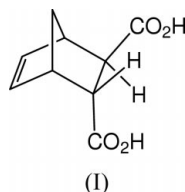
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## Key indicators

Single-crystal X-ray study  
 $T = 150$  K  
Mean  $\sigma(\text{C}-\text{C}) = 0.002$  Å  
 $R$  factor = 0.045  
 $wR$  factor = 0.106  
Data-to-parameter ratio = 13.8For details of how these key indicators were  
automatically derived from the article, see  
<http://journals.iucr.org/e>.2-Norbornene-*exo,endo*-5,6-dicarboxylic acidThe crystal structure of the title compound,  $\text{C}_9\text{H}_{10}\text{O}_4$ , contains infinite hydrogen-bonded chains of molecules. The norbornene skeleton is slightly twisted.

## Comment

The title compound, (I), alternatively called bicyclo[2.2.1]-hept-2-ene-*trans*-5,6-dicarboxylic acid, has been studied as part of a series of small-molecule models of organic polymers influencing the crystallization of inorganic salts, particularly  $\text{CaCO}_3$  (Megson, 1997; Feast *et al.*, 2002).

Compound (I) was prepared by the Diels–Alder addition of cyclopentadiene to fumaric acid (Diels & Alder, 1928; Alder & Stein, 1933). The asymmetric unit of (I) comprises one molecule. Both carboxyl groups form the usual pairs of hydrogen bonds with their inversion equivalents (Fig. 1), linking the molecules into an infinite zigzag chain, the general direction of which is parallel to the crystallographic  $a$  axis. *trans*-Substitution results in a slight twisting of the norbornene skeleton, the  $\text{C}2=\text{C}3$  and  $\text{C}5-\text{C}6$  bonds forming an angle of  $4.6(2)^\circ$ . On the other hand, the  $\text{C}5-\text{C}6$  bond in (I) is slightly shorter than the corresponding bonds in *cis*-dicarboxylic acid derivatives, *e.g.*  $1.566(2)$ – $1.574(2)$  Å (Bolte *et al.*, 2000, Batsanov & Hesselink, 2002*a,b,c*), due to lower steric repulsion between the carboxylic acid groups.

## Experimental

Fumaric acid (3.08 g, 27 mmol) and cyclopentadiene (2.3 ml, 2.3 g, 35 mmol) in 50 ml of dry THF were stirred at room temperature for 48 h. The solvent was removed using a rotary evaporator and the residue recrystallized from doubly distilled water, yielding 4.41 g (93%) of (I) [m.p. 458–459 K, *cf.* 460–461 K (Alder & Stein, 1933)].

## Crystal data

$\text{C}_9\text{H}_{10}\text{O}_4$   
 $M_r = 182.17$   
Monoclinic,  $P2_1/c$   
 $a = 12.698(1)$  Å  
 $b = 5.3906(3)$  Å  
 $c = 12.153(1)$  Å  
 $\beta = 92.56(1)^\circ$   
 $V = 831.04(11)$  Å<sup>3</sup>  
 $Z = 4$

$D_x = 1.456$  Mg m<sup>-3</sup>  
Mo  $K\alpha$  radiation  
Cell parameters from 471 reflections  
 $\theta = 10.2$ – $20.0^\circ$   
 $\mu = 0.12$  mm<sup>-1</sup>  
 $T = 150(2)$  K  
Plate, colourless  
 $0.40 \times 0.22 \times 0.16$  mm

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Dicarboxylic Diels–Alder products, Part 4. For Part 3, see Batsanov &amp; Hesselink (2002c).

## Data collection

SMART 1K CCD area-detector  
diffractometer  
 $\omega$  scans  
Absorption correction: none  
6167 measured reflections  
2187 independent reflections

1773 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.039$   
 $\theta_{\text{max}} = 29.0^\circ$   
 $h = -12 \rightarrow 17$   
 $k = -7 \rightarrow 6$   
 $l = -16 \rightarrow 14$

## Refinement

Refinement on  $F^2$   
 $R[F^2 > 2\sigma(F^2)] = 0.045$   
 $wR(F^2) = 0.106$   
 $S = 1.08$   
2187 reflections  
159 parameters  
All H-atom parameters refined

$w = 1/[\sigma^2(F_o^2) + (0.0291P)^2 + 0.6115P]$   
where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\text{max}} = 0.001$   
 $\Delta\rho_{\text{max}} = 0.32 \text{ e } \text{\AA}^{-3}$   
 $\Delta\rho_{\text{min}} = -0.22 \text{ e } \text{\AA}^{-3}$   
Extinction correction: *SHELXL97*  
Extinction coefficient: 0.024 (3)

Table 1

Selected geometric parameters ( $\text{\AA}$ ,  $^\circ$ ).

O1—C8	1.3111 (19)	C2—C3	1.330 (2)
O2—C8	1.2227 (19)	C3—C4	1.517 (2)
O3—C9	1.3173 (18)	C4—C7	1.544 (2)
O4—C9	1.2227 (19)	C4—C5	1.580 (2)
C1—C2	1.520 (2)	C5—C8	1.503 (2)
C1—C7	1.540 (2)	C5—C6	1.553 (2)
C1—C6	1.565 (2)	C6—C9	1.503 (2)
C2—C1—C7	100.88 (12)	C8—C5—C6	112.08 (12)
C2—C1—C6	106.29 (12)	C8—C5—C4	114.16 (12)
C7—C1—C6	98.70 (12)	C6—C5—C4	102.02 (11)
C3—C2—C1	107.72 (14)	C9—C6—C5	113.20 (12)
C2—C3—C4	107.76 (14)	C9—C6—C1	115.68 (12)
C3—C4—C7	100.57 (13)	C5—C6—C1	103.58 (11)
C3—C4—C5	103.27 (12)	C1—C7—C4	93.96 (12)
C7—C4—C5	101.33 (12)		
O2—C8—C5—C6	−49.76 (19)	C5—C6—C9—O4	20.6 (2)
C8—C5—C6—C9	−105.37 (14)		

Table 2

Hydrogen-bonding geometry ( $\text{\AA}$ ,  $^\circ$ ).

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
O1—H01 $\cdots$ O2 <sup>i</sup>	0.94 (3)	1.70 (3)	2.6350 (17)	173 (3)
O3—H03 $\cdots$ O4 <sup>ii</sup>	0.93 (2)	1.72 (2)	2.6446 (16)	176 (2)

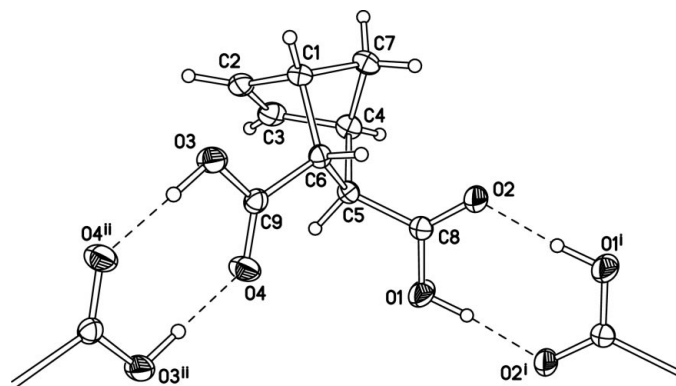
Symmetry codes: (i)  $-x, 1-y, 1-z$ ; (ii)  $1-x, 1-y, 1-z$ .

Figure 1

The molecular structure of (I), showing the intermolecular hydrogen bonding [symmetry codes: (i)  $-x, 1-y, 1-z$ ; (ii)  $1-x, 1-y, 1-z$ ]. Displacement ellipsoids are drawn at the 50% probability level.

All H atoms were refined isotropically; C—H distances were in the range 0.97 (2)–0.99 (2)  $\text{\AA}$ .

Data collection: *SMART* (Siemens, 1995); cell refinement: *SMART*; data reduction: *SAINT* (Siemens, 1995); 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*.

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