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#### Key indicators

Single-crystal X-ray study  
 T = 293 K  
 Mean  $\sigma(\text{C}-\text{C}) = 0.003 \text{ \AA}$   
 R factor = 0.043  
 wR factor = 0.115  
 Data-to-parameter ratio = 11.7

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

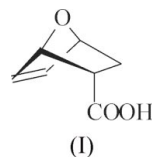
## (1*SR*,2*SR*,4*SR*)-7-Oxabicyclo[2.2.1]hept-5-ene-2-carboxylic acid

The title compound,  $\text{C}_7\text{H}_8\text{O}_3$ , has been synthesized and structurally characterized. The molecules are connected by  $\text{O}-\text{H}\cdots\text{O}$  hydrogen bonds to form centrosymmetric dimers.

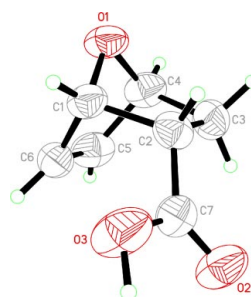
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#### Comment

Diels–Alder addition of furan and acrylic acid leads to the racemic title compound (Suami *et al.*, 1977) (1*SR*,2*SR*,4*SR*)-7-oxabicyclo[2.2.1]hept-5-ene-2-carboxylic acid, (I), of which we report the crystal structure. 7-Oxabicyclo[2.2.1]hept-5-enyl derivatives are extremely useful as key intermediates for the synthesis of important types of natural products and analogues, such as C-nucleosides and C-glycosides (Vogel *et al.*, 1990; Corey & Loh, 1993). After separation from the *exo*-diastereomers and optical resolution (Ogawa *et al.*, 1985) with chiral auxiliaries, the two enantiomerically pure *endo*-enantiomers give access to both L- and D-hexose derivatives through stereoselective transformations.

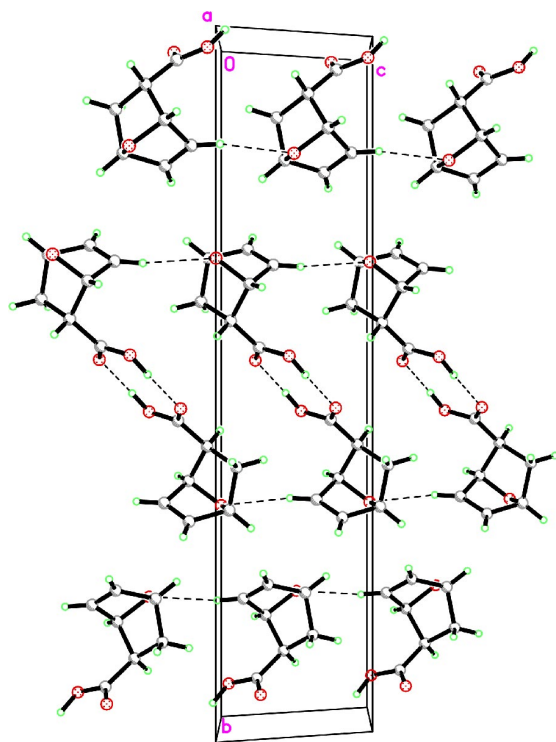


The crystal structure of (I) was determined in order to confirm its molecular structure. The ring system is nearly mirror symmetric: differences in equivalent bond lengths are smaller than 3.5 s.u.'s. The biggest difference in equivalent bond angles of 4.6 s.u.'s is observed at the carboxylate-substituted atom C2 (Fig. 1). The carboxylate bond distances [ $\text{C}7-\text{O}2 = 1.212(2) \text{ \AA}$  and  $\text{C}7-\text{O}3 = 1.301(2) \text{ \AA}$ ] correspond to typical distances [1.214 (19) and 1.308 (19)  $\text{ \AA}$ , respectively; Allen *et al.*, 1992]. The carboxylate group adopts a staggered conformation with respect to the  $\text{C}2-\text{C}1$  bond [ $\text{O}3-\text{C}7-\text{C}2-\text{C}1 = -47.7(2)^\circ$ ] and an almost eclipsed conformation with respect to the  $\text{C}2-\text{C}3$  bond [ $\text{O}2-\text{C}7-\text{C}2-\text{C}3 =$



**Figure 1**

The molecular structure of (I), showing the atomic numbering and 50% probability displacement ellipsoids.



**Figure 2**  
Packing diagram of (I), viewed down the *a* axis, showing hydrogen bonds as dashed lines.

15.7 (2)°], with a short intramolecular contact O2...H7 of 2.51 (2) Å. Two molecules are held together in a dimer located on a centre of inversion by the familiar carboxylic acid linkage (Leiserowitz, 1976). The two symmetry-equivalent hydrogen bonds with a length of 1.73 (3) Å are of intermediate strength and show an almost linear *D*–H...*A* angle of 178 (2)°. The eight-membered ring formed by the two carboxylate groups is almost planar, with an r.m.s. deviation of 0.018 Å from the mean plane through the C and O atoms. The dimers are stacked along *a*–*c*, with a weak O1...H6 hydrogen bond of 2.55 (2) Å between neighbouring molecules (Fig. 2).

## Experimental

A mixture of furan (204.00 g, 3 mol), acrylic acid (216.02 g, 3 mol) and hydroquinone (0.30 g, 2.7 mmol) was kept for 81 d under nitrogen at room temperature in the dark. The resulting racemic *endo* product, in the form of white crystals, was filtered off and twice recrystallized at 313 K from ethyl acetate/hexane [yield 40%, 168.0 g, 1.2 mol; m.p. 369–371 K (Suami *et al.*, 1977)]. The *exo* side-product could be concentrated in the mother liquor. MS: 140, 122, 111, 95, 81, 68, 54, 44, 39, 27. IR: 3436 (*m*), 3012 (*s*), 2962 (*s*), 1716 (*vs*), 1456 (*m*), 1320 (*s*), 1222 (*s*), 1198 (*s*). <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>, p.p.m.): 9.8 (very broad, COOH), 6.47 (*dd*, *J*<sub>6,5</sub> = 6, *J*<sub>6,1</sub> = 2 Hz, H<sub>6</sub>), 6.31 (*dd*, *J*<sub>5,6</sub> = 6, *J*<sub>5,4</sub> = 2 Hz, H<sub>5</sub>), 5.20 (*ddd*, *J*<sub>1,2-*exo*</sub> = 5, *J*<sub>1,6</sub> = 2, *J*<sub>1,4</sub> = 1 Hz, H<sub>1</sub>), 5.05 (*ddd*, *J*<sub>4,3-*exo*</sub> = 5, *J*<sub>4,5</sub> = 2, *J*<sub>4,1</sub> = 1 Hz, H<sub>4</sub>), 3.18 (*ddd*, *J*<sub>2,3-*exo*</sub> = 9, *J*<sub>2,1</sub> = 4, *J*<sub>2,3-*endo*</sub> = 4 Hz, H<sub>2-*exo*</sub>), 2.13 (*ddd*, *J*<sub>gem</sub> = 11, *J*<sub>3,2-*exo*</sub> = 9, *J*<sub>3,4</sub> = 4 Hz, H<sub>3-*exo*</sub>), 1.58 (*dd*, *J*<sub>gem</sub> = 11, *J*<sub>3,4</sub> = 4, *J*<sub>3,2-*exo*</sub> = 4 Hz, H<sub>3-*endo*</sub>) (Nelson & Allen, 1972). The *exo* product shows an additional <sup>1</sup>H NMR signal at 2.45 p.p.m.

## Crystal data

C<sub>7</sub>H<sub>8</sub>O<sub>3</sub>  
*M<sub>r</sub>* = 140.13  
Monoclinic, *P*2<sub>1</sub>/*n*  
*a* = 5.239 (4) Å  
*b* = 23.709 (19) Å  
*c* = 5.669 (5) Å  
*β* = 111.708 (13)°  
*V* = 654.2 (9) Å<sup>3</sup>  
*Z* = 4

*D<sub>x</sub>* = 1.423 Mg m<sup>−3</sup>  
Mo *Kα* radiation  
Cell parameters from 781 reflections  
*θ* = 3.4–27.0°  
*μ* = 0.11 mm<sup>−1</sup>  
*T* = 293 (2) K  
Prism, colourless  
0.55 × 0.42 × 0.35 mm

## Data collection

Siemens SMART 1K CCD area-detector diffractometer  
*ω* scans  
Absorption correction: none  
3688 measured reflections  
1439 independent reflections

1256 reflections with *I* > 2σ(*I*)  
*R*<sub>int</sub> = 0.014  
*θ*<sub>max</sub> = 27.8°  
*h* = −6 → 6  
*k* = −30 → 26  
*l* = −7 → 7

## Refinement

Refinement on *F*<sup>2</sup>  
*R* [*F*<sup>2</sup> > 2σ(*F*<sup>2</sup>)] = 0.043  
*wR*(*F*<sup>2</sup>) = 0.115  
*S* = 1.04  
1439 reflections  
123 parameters  
All H-atom parameters refined

$w = 1/[\sigma^2(F_o^2) + (0.0538P)^2 + 0.2006P]$   
where  $P = (F_o^2 + 2F_c^2)/3$   
(Δ/σ)<sub>max</sub> = 0.007  
Δρ<sub>max</sub> = 0.33 e Å<sup>−3</sup>  
Δρ<sub>min</sub> = −0.20 e Å<sup>−3</sup>

**Table 1**

Selected geometric parameters (Å, °).

O1–C1	1.434 (2)	C4–C5	1.504 (3)
O1–C4	1.441 (2)	C5–C6	1.313 (3)
C1–C2	1.554 (2)	C1–C6	1.498 (2)
C2–C3	1.536 (2)	C2–C7	1.495 (2)
C3–C4	1.542 (3)		
C1–O1–C4	95.53 (11)	O1–C4–C5	101.27 (14)
O1–C1–C6	101.97 (14)	O1–C4–C3	101.31 (13)
O1–C1–C2	100.53 (13)	C5–C4–C3	107.19 (13)
C6–C1–C2	106.75 (13)	C6–C5–C4	106.05 (16)
C7–C2–C3	116.27 (15)	C5–C6–C1	105.50 (16)
C7–C2–C1	113.50 (13)	O2–C7–O3	122.81 (15)
C3–C2–C1	101.46 (13)	O2–C7–C2	123.97 (15)
C2–C3–C4	100.62 (13)	O3–C7–C2	113.21 (15)

Data collection: *SMART* (Bruker, 2003); cell refinement: *SAINT* (Bruker, 2003); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2003); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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