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6,6′-Dioxo-2,2-bi(7-oxatricyclo[3.2.1.1^{3,8}]-nonane)-4,4′-dicarboxylic acid

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Key indicators

Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.005 Å
R factor = 0.040
wR factor = 0.089
Data-to-parameter ratio = 6.3

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

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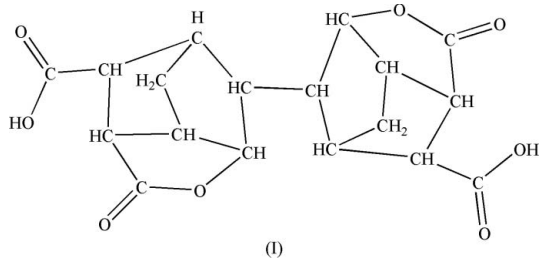
The title molecule, $C_{18}H_{18}O_8$, has C_2 molecular symmetry. In the crystal structure, each molecule is connected to four neighbours *via* intermolecular O—H···O hydrogen bonds to form a two-dimensional network parallel to (100).

Comment

Directional intermolecular interactions, particularly hydrogen bonding, are reliable and reproducible in generating molecular assemblies (Zaworotko, 1997; Braga & Grepioni, 2000). The design and use of reliable synthons lead to formation of supramolecular assemblies. The synthesis and crystal structure of the title compound, (I), confirms this as an efficient supramolecular synthon.



(I)

The molecule of (I) has two *endo*-norbornene skeletons related by a crystallographic twofold axis perpendicular to the bond C2—C2^{i} [symmetry code: (i) $-x+2, -y+2, z$] (Fig. 1). Its two carboxyl groups and two carbonyl groups are involved in intermolecular hydrogen bonding. Each molecule is hydrogen bonded to four adjacent molecules (Fig. 2 and Table 2), generating the motif described in graph-set-notation as $R_4^4(36)$ (Etter, 1990; Grell *et al.*, 2000).

Experimental

The title compound was prepared by dimerization of *endo*-norbornene-*cis*-5,6-dicarboxylic acid under acidic conditions. A solution (10 ml) of water containing trichloroacetic acid (2 mmol, 0.33 g) was added slowly to a solution (10 ml) of ethanol containing *endo*-norbornene-*cis*-5,6-dicarboxylic acid (2 mmol, 0.37 g). The mixture was stirred for several minutes and left to stand at room temperature for about three weeks; colourless plate-shaped crystals were obtained.

Crystal data

$C_{18}H_{18}O_8$
M_r = 362.32
Orthorhombic, *Aba*2
a = 9.8562 (12) Å
b = 13.4978 (17) Å
c = 11.8371 (14) Å
V = 1574.8 (3) Å^3
Z = 4
D_x = 1.528 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 1461 reflections
θ = 3.0–24.7°
μ = 0.12 mm^{-1}
T = 298 (2) K
Plate, colourless
0.29 × 0.25 × 0.10 mm

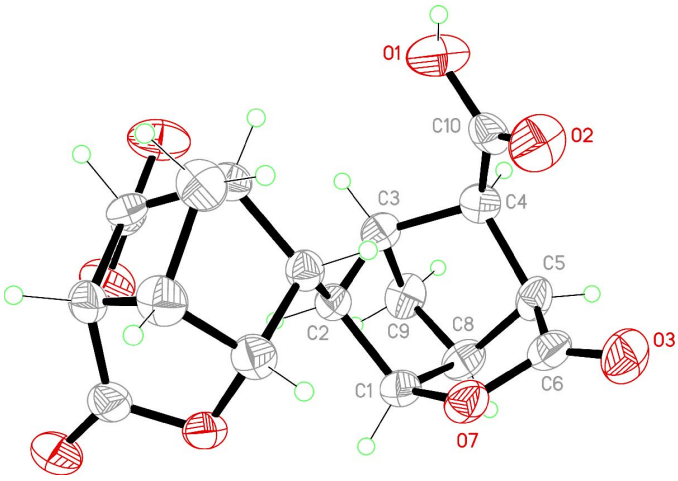


Figure 1
The structure of (I), with the atom-numbering scheme, showing displacement ellipsoids drawn at the 50% probability level.

Data collection

Bruker SMART APEX area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2002)
T_{min} = 0.966, T_{max} = 0.988
3985 measured reflections
750 independent reflections
736 reflections with $I > 2\sigma(I)$
R_{int} = 0.026
θ_{max} = 25.2°
$h = -11 \rightarrow 11$
$k = -13 \rightarrow 16$
$l = -14 \rightarrow 12$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.040
$wR(F^2)$ = 0.089
S = 1.19
750 reflections
119 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.029P)^2 + 1.6081P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.19 e Å^{-3}
$\Delta\rho_{min}$ = −0.22 e Å^{-3}

Table 1
Selected bond lengths (Å).

O1—C10	1.325 (5)	O3—C6	1.199 (4)
O2—C10	1.188 (4)	C2—C2^{i}	1.529 (6)

Symmetry code: (i) $2-x, 2-y, z$.

Table 2
Hydrogen-bonding geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1···O3^{ii}	0.82	1.92	2.689 (4)	157

Symmetry code: (ii) $2-x, \frac{3}{2}-y, z-\frac{1}{2}$.

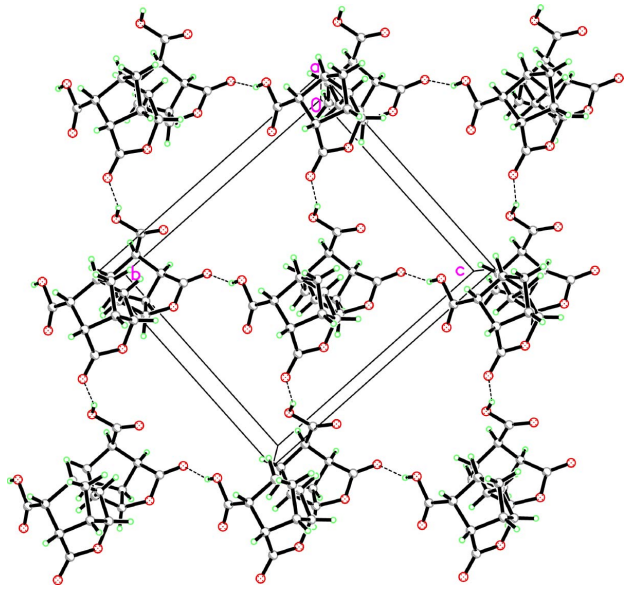


Figure 2
The hydrogen-bonding (dashed lines) generates the motif $R_4^4(36)$.

The H atoms were positioned geometrically and allowed to ride on their parent atoms at distances of Csp^3—H = 0.98 or 0.97 Å and U_{iso} = 1.5U_{eq}(C), or O—H = 0.82 Å and U_{iso} = 1.2U_{eq}(O). In the absence of significant anomalous scattering effects, Friedel pairs were merged.

Data collection: *SMART* (Bruker, 2002); cell refinement: *SAINT* (Bruker, 2002); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 2002); software used to prepare material for publication: *SHELXTL*.

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References

Braga, D. & Grepioni, F. (2000). *Acc. Chem. Res.* **33**, 601–608.
Bruker (2002). *SADABS* (Version 2.03), *SAINT* (Version 6.02), *SMART* (Version 5.62) and *SHELXTL* (Version 6.10). Bruker AXS Inc., Madison, Wisconsin, USA.
Etter, M. C. (1990). *Acc. Chem. Res.* **23**, 120–126.
Flack, H. D. (1983). *Acta Cryst.* A**39**, 876–881.
Grell, J., Bernstein, J. & Timhofer, G. (2000). *Acta Cryst.* B**56**, 166–179.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. Release 97-2. University of Göttingen, Germany.
Zaworotko, M. J. (1997). *Nature (London)*, **386**, 220–226.