

Acta Crystallographica Section E

Structure Reports

Online

ISSN 1600-5368

Bicyclo[2.2.2]oct-7-ene-2,3,5,6-tetra-carboxylic dianhydride

Tuoping Hu

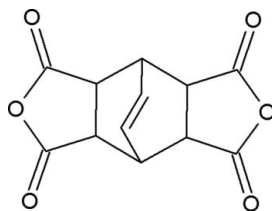
Department of Chemistry, North University of China, Taiyuan, Shanxi 030051, People's Republic of China

Correspondence e-mail: hutuopingsx@yahoo.com.cn

Received 5 April 2008; accepted 29 April 2008

 Key indicators: single-crystal X-ray study; $T = 296$ K; mean $\sigma(\text{C}-\text{C}) = 0.003$ Å; R factor = 0.043; wR factor = 0.100; data-to-parameter ratio = 12.9.

In the title compound, $\text{C}_{12}\text{H}_8\text{O}_6$, molecules are linked by weak $\text{C}-\text{H}\cdots\text{O}$ interactions involving all the potential donors, generating a three-dimensional network.



Experimental

Crystal data

$\text{C}_{12}\text{H}_8\text{O}_6$
 $M_r = 248.18$
 Monoclinic, $P2_1/n$
 $a = 7.627$ (2) Å
 $b = 13.877$ (3) Å
 $c = 9.823$ (2) Å
 $\beta = 100.68$ (2)°

$V = 1021.7$ (4) Å³
 $Z = 4$
 Mo $K\alpha$ radiation
 $\mu = 0.13$ mm⁻¹
 $T = 296$ (2) K
 $0.28 \times 0.16 \times 0.10$ mm

Data collection

Bruker SMART CCD area-detector diffractometer
 Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
 $T_{\min} = 0.921$, $T_{\max} = 0.987$

11412 measured reflections
 2119 independent reflections
 1460 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.051$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.042$
 $wR(F^2) = 0.099$
 $S = 1.01$
 2118 reflections

164 parameters
 H-atom parameters constrained
 $\Delta\rho_{\max} = 0.21$ e Å⁻³
 $\Delta\rho_{\min} = -0.16$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{C3}-\text{H3}\cdots\text{O2}^{\text{i}}$	0.98	2.50	3.313 (3)	140
$\text{C8}-\text{H8}\cdots\text{O3}^{\text{ii}}$	0.98	2.58	3.175 (2)	119

Symmetry codes: (i) $-x + \frac{3}{2}, y + \frac{1}{2}, -z + \frac{3}{2}$; (ii) $-x + \frac{1}{2}, y - \frac{1}{2}, -z + \frac{3}{2}$

Data collection: *SMART* (Bruker, 2007); cell refinement: *SAINT-Plus* (Bruker, 2007); data reduction: *SAINT-Plus*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL*.

The author acknowledges funding support from the Natural Science Foundation of Shanxi Province (2007011033), the Program of Technological Industrialization in Universities of Shanxi Province (20070308) and the Start-up Fund of the North University of China.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: SG2233).

References

- Bruker (2007). *SMART* and *SAINT-Plus*. Bruker AXS Inc., Madison, Wisconsin, USA.
 Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
 Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.

supplementary materials

Acta Cryst. (2008). E64, o1021 [doi:10.1107/S1600536808012452]

Bicyclo[2.2.2]oct-7-ene-2,3,5,6-tetracarboxylic dianhydride

T. Hu

Comment

The molecule of the title complex, (I) (Fig. 1), is neutral. Molecules are linked by C—H···O weak interactions involving all the potential donors, generating a three-dimensional network, as shown in Fig. 2. No conventional hydrogen bonding was found in the structure.

Experimental

The title compound was obtained unintentionally as the product of an attempted synthesis of a polymeric network complex of zinc with the bicyclo[2.2.2]oct-7-ene-2,3,5,6-tetracarboxylic acid. The title compound (0.4 mmol) and Zn(NO₃)₂·6H₂O (0.2 mmol) were dissolved in 15 ml distilled water, to which 2 drops of H₃PO₄ (w.t. 18%) was added. The solution was put into the oven at 50 centigrade degree for 1 day. Colourless prism crystals were collected by filtration.

Refinement

H atoms were positioned geometrically and refined using a riding model with C—H = 0.980 Å and 0.930 Å, respectively, with $U_{\text{iso}}(\text{H}) = 1.2$ times $U_{\text{eq}}(\text{C})$. Reflection -111 was omitted because it was eclipsed by the beam stop.

Figures

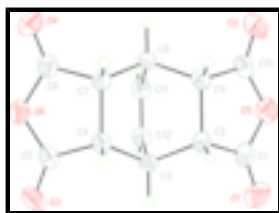


Fig. 1. Molecular structure showing 50% probability displacement ellipsoids.

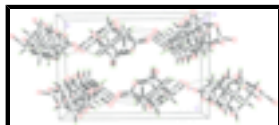


Fig. 2. Packing diagram viewed down the *a* axis.

Bicyclo[2.2.2]oct-7-ene-2,3,5,6-tetracarboxylic dianhydride

Crystal data

C₁₂H₈O₆

$M_r = 248.18$

Monoclinic, $P2_1/n$

$F_{000} = 512$

$D_x = 1.613 \text{ Mg m}^{-3}$

Mo $K\alpha$ radiation

$\lambda = 0.71073 \text{ \AA}$

supplementary materials

Hall symbol: -P 2yn

$a = 7.627(2) \text{ \AA}$

$b = 13.877(3) \text{ \AA}$

$c = 9.823(2) \text{ \AA}$

$\beta = 100.68(2)^\circ$

$V = 1021.7(4) \text{ \AA}^3$

$Z = 4$

Cell parameters from 2677 reflections

$\theta = 2.9\text{--}24.1^\circ$

$\mu = 0.13 \text{ mm}^{-1}$

$T = 296(2) \text{ K}$

Prism, colourless

$0.28 \times 0.16 \times 0.10 \text{ mm}$

Data collection

Bruker SMART CCD area-detector
diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

$T = 296(2) \text{ K}$

φ and ω scans

Absorption correction: multi-scan
(SADABS; Sheldrick, 1996)

$T_{\min} = 0.921$, $T_{\max} = 0.987$

11412 measured reflections

2119 independent reflections

1460 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.051$

$\theta_{\text{max}} = 26.5^\circ$

$\theta_{\text{min}} = 2.6^\circ$

$h = -9 \rightarrow 9$

$k = -17 \rightarrow 15$

$l = -11 \rightarrow 12$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.042$

$wR(F^2) = 0.099$

$S = 1.01$

2118 reflections

164 parameters

Primary atom site location: structure-invariant direct
methods

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring
sites

H-atom parameters constrained

$$w = 1/[\sigma^2(F_o^2) + (0.0334P)^2 + 0.3914P]$$

where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\text{max}} < 0.001$

$\Delta\rho_{\text{max}} = 0.21 \text{ e \AA}^{-3}$

$\Delta\rho_{\text{min}} = -0.16 \text{ e \AA}^{-3}$

Extinction correction: SHELXL97 (Sheldrick, 2008),

$$F_c^* = kFc[1 + 0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$$

Extinction coefficient: 0.005 (1)

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R -factor wR and goodness of fit S are based on F^2 , conventional R -factors R are based on F , with F set to zero for negative F^2 . The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating R -factors(gt) *etc.* and is not relevant to the choice of reflections for refinement. R -factors based on F^2 are statistically about twice as large as those based on F , and R -factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.7820 (3)	0.83700 (16)	0.6304 (2)	0.0449 (5)
C2	0.5829 (2)	0.84372 (13)	0.61717 (19)	0.0355 (4)
H2	0.5239	0.8381	0.5200	0.043*
C3	0.5305 (2)	0.93909 (13)	0.68048 (19)	0.0350 (4)
H3	0.5646	0.9952	0.6307	0.042*
C4	0.3256 (2)	0.93415 (13)	0.67248 (18)	0.0344 (4)
H4	0.2633	0.9301	0.5760	0.041*
C5	0.2627 (3)	1.01999 (15)	0.7435 (2)	0.0416 (5)
C6	0.1858 (3)	0.89126 (15)	0.8635 (2)	0.0434 (5)
C7	0.2788 (2)	0.84782 (13)	0.75613 (18)	0.0352 (4)
H7	0.1984	0.8039	0.6962	0.042*
C8	0.4506 (2)	0.79445 (13)	0.82409 (18)	0.0354 (4)
H8	0.4251	0.7413	0.8831	0.042*
C9	0.5347 (2)	0.75817 (13)	0.70190 (19)	0.0358 (4)
H9	0.4526	0.7145	0.6429	0.043*
C10	0.7097 (3)	0.70890 (16)	0.7535 (2)	0.0462 (5)
C11	0.5767 (2)	0.86683 (14)	0.90425 (19)	0.0392 (5)
H11	0.6221	0.8602	0.9984	0.047*
C12	0.6180 (2)	0.94061 (14)	0.83043 (19)	0.0394 (5)
H12	0.6951	0.9895	0.8686	0.047*
O1	0.88296 (19)	0.89053 (13)	0.58906 (17)	0.0638 (5)
O2	0.7409 (2)	0.64134 (12)	0.82843 (18)	0.0681 (5)
O3	0.2829 (2)	1.10382 (11)	0.72476 (16)	0.0596 (4)
O4	0.1289 (2)	0.85316 (12)	0.95463 (16)	0.0629 (5)
O5	0.84589 (17)	0.75576 (11)	0.70513 (15)	0.0537 (4)
O6	0.17498 (18)	0.99043 (10)	0.84767 (14)	0.0485 (4)

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0359 (11)	0.0528 (13)	0.0462 (12)	0.0033 (10)	0.0079 (9)	-0.0067 (10)
C2	0.0311 (9)	0.0378 (11)	0.0373 (10)	0.0016 (8)	0.0054 (7)	-0.0023 (8)
C3	0.0334 (9)	0.0298 (10)	0.0422 (10)	-0.0030 (8)	0.0076 (8)	0.0016 (8)
C4	0.0337 (9)	0.0331 (10)	0.0351 (9)	0.0039 (8)	0.0031 (7)	-0.0014 (8)
C5	0.0405 (11)	0.0389 (12)	0.0429 (11)	0.0098 (9)	0.0018 (9)	-0.0003 (9)
C6	0.0355 (10)	0.0470 (13)	0.0482 (12)	0.0008 (9)	0.0089 (9)	-0.0024 (10)
C7	0.0313 (9)	0.0327 (11)	0.0411 (10)	-0.0025 (8)	0.0052 (8)	-0.0035 (8)
C8	0.0375 (10)	0.0297 (10)	0.0390 (10)	0.0002 (8)	0.0070 (8)	0.0033 (8)
C9	0.0328 (9)	0.0309 (10)	0.0423 (10)	0.0017 (8)	0.0033 (8)	-0.0024 (8)
C10	0.0455 (12)	0.0419 (13)	0.0490 (12)	0.0112 (10)	0.0031 (9)	-0.0021 (10)
C11	0.0386 (10)	0.0415 (11)	0.0353 (10)	0.0047 (9)	0.0013 (8)	-0.0021 (9)
C12	0.0349 (10)	0.0369 (11)	0.0448 (11)	-0.0035 (8)	0.0034 (8)	-0.0078 (9)
O1	0.0392 (8)	0.0791 (12)	0.0768 (11)	-0.0069 (8)	0.0205 (8)	0.0052 (9)
O2	0.0689 (11)	0.0550 (10)	0.0784 (11)	0.0261 (8)	0.0083 (9)	0.0170 (9)

supplementary materials

O3	0.0726 (11)	0.0345 (9)	0.0716 (11)	0.0137 (7)	0.0131 (8)	0.0022 (8)
O4	0.0655 (10)	0.0696 (11)	0.0616 (10)	-0.0019 (8)	0.0328 (8)	0.0038 (8)
O5	0.0326 (7)	0.0582 (10)	0.0685 (10)	0.0107 (7)	0.0044 (7)	0.0018 (8)
O6	0.0500 (8)	0.0464 (9)	0.0516 (8)	0.0096 (6)	0.0161 (7)	-0.0056 (7)

Geometric parameters (Å, °)

C1—O1	1.194 (2)	C6—O6	1.386 (2)
C1—O5	1.384 (2)	C6—C7	1.502 (3)
C1—C2	1.503 (3)	C7—C8	1.545 (2)
C2—C9	1.533 (3)	C7—H7	0.9800
C2—C3	1.546 (3)	C8—C11	1.508 (3)
C2—H2	0.9800	C8—C9	1.546 (3)
C3—C12	1.500 (3)	C8—H8	0.9800
C3—C4	1.551 (2)	C9—C10	1.502 (3)
C3—H3	0.9800	C9—H9	0.9800
C4—C5	1.504 (3)	C10—O2	1.188 (2)
C4—C7	1.532 (3)	C10—O5	1.382 (3)
C4—H4	0.9800	C11—C12	1.326 (3)
C5—O3	1.192 (2)	C11—H11	0.9300
C5—O6	1.384 (2)	C12—H12	0.9300
C6—O4	1.189 (2)		
O1—C1—O5	120.12 (18)	C6—C7—C8	111.21 (15)
O1—C1—C2	129.6 (2)	C4—C7—C8	110.07 (15)
O5—C1—C2	110.29 (18)	C6—C7—H7	110.3
C1—C2—C9	104.20 (15)	C4—C7—H7	110.3
C1—C2—C3	110.54 (15)	C8—C7—H7	110.3
C9—C2—C3	109.79 (15)	C11—C8—C7	108.31 (15)
C1—C2—H2	110.7	C11—C8—C9	107.83 (15)
C9—C2—H2	110.7	C7—C8—C9	105.15 (14)
C3—C2—H2	110.7	C11—C8—H8	111.7
C12—C3—C2	107.81 (15)	C7—C8—H8	111.7
C12—C3—C4	108.10 (15)	C9—C8—H8	111.7
C2—C3—C4	105.96 (14)	C10—C9—C2	104.37 (15)
C12—C3—H3	111.6	C10—C9—C8	110.90 (15)
C2—C3—H3	111.6	C2—C9—C8	110.18 (14)
C4—C3—H3	111.6	C10—C9—H9	110.4
C5—C4—C7	104.15 (16)	C2—C9—H9	110.4
C5—C4—C3	110.29 (15)	C8—C9—H9	110.4
C7—C4—C3	109.89 (14)	O2—C10—O5	120.48 (18)
C5—C4—H4	110.8	O2—C10—C9	129.2 (2)
C7—C4—H4	110.8	O5—C10—C9	110.31 (17)
C3—C4—H4	110.8	C12—C11—C8	114.96 (16)
O3—C5—O6	119.82 (19)	C12—C11—H11	122.5
O3—C5—C4	129.8 (2)	C8—C11—H11	122.5
O6—C5—C4	110.36 (17)	C11—C12—C3	114.75 (17)
O4—C6—O6	120.26 (19)	C11—C12—H12	122.6
O4—C6—C7	129.5 (2)	C3—C12—H12	122.6
O6—C6—C7	110.22 (17)	C10—O5—C1	110.67 (15)

C6—C7—C4	104.49 (15)	C5—O6—C6	110.52 (16)
O1—C1—C2—C9	-175.3 (2)	C4—C7—C8—C9	-62.20 (17)
O5—C1—C2—C9	3.2 (2)	C1—C2—C9—C10	-1.16 (19)
O1—C1—C2—C3	-57.4 (3)	C3—C2—C9—C10	-119.57 (16)
O5—C1—C2—C3	121.13 (17)	C1—C2—C9—C8	117.95 (15)
C1—C2—C3—C12	-59.7 (2)	C3—C2—C9—C8	-0.46 (19)
C9—C2—C3—C12	54.68 (18)	C11—C8—C9—C10	61.6 (2)
C1—C2—C3—C4	-175.28 (15)	C7—C8—C9—C10	176.98 (15)
C9—C2—C3—C4	-60.87 (18)	C11—C8—C9—C2	-53.47 (18)
C12—C3—C4—C5	59.5 (2)	C7—C8—C9—C2	61.93 (17)
C2—C3—C4—C5	174.86 (15)	C2—C9—C10—O2	176.5 (2)
C12—C3—C4—C7	-54.75 (19)	C8—C9—C10—O2	57.9 (3)
C2—C3—C4—C7	60.60 (18)	C2—C9—C10—O5	-1.2 (2)
C7—C4—C5—O3	172.72 (19)	C8—C9—C10—O5	-119.86 (17)
C3—C4—C5—O3	54.9 (3)	C7—C8—C11—C12	-56.8 (2)
C7—C4—C5—O6	-4.85 (19)	C9—C8—C11—C12	56.5 (2)
C3—C4—C5—O6	-122.71 (16)	C8—C11—C12—C3	0.4 (2)
O4—C6—C7—C4	-177.8 (2)	C2—C3—C12—C11	-57.5 (2)
O6—C6—C7—C4	0.55 (19)	C4—C3—C12—C11	56.6 (2)
O4—C6—C7—C8	-59.1 (3)	O2—C10—O5—C1	-174.58 (19)
O6—C6—C7—C8	119.27 (16)	C9—C10—O5—C1	3.4 (2)
C5—C4—C7—C6	2.49 (18)	O1—C1—O5—C10	174.51 (19)
C3—C4—C7—C6	120.62 (15)	C2—C1—O5—C10	-4.2 (2)
C5—C4—C7—C8	-117.01 (16)	O3—C5—O6—C6	-172.38 (18)
C3—C4—C7—C8	1.1 (2)	C4—C5—O6—C6	5.5 (2)
C6—C7—C8—C11	-62.4 (2)	O4—C6—O6—C5	174.79 (18)
C4—C7—C8—C11	52.87 (19)	C7—C6—O6—C5	-3.7 (2)
C6—C7—C8—C9	-177.52 (15)		

Hydrogen-bond geometry (Å, °)

<i>D</i> —H \cdots <i>A</i>	<i>D</i> —H	H \cdots <i>A</i>	<i>D</i> \cdots <i>A</i>	<i>D</i> —H \cdots <i>A</i>
C3—H3 \cdots O2 ⁱ	0.98	2.50	3.313 (3)	140
C8—H8 \cdots O3 ⁱⁱ	0.98	2.58	3.175 (2)	119

Symmetry codes: (i) $-x+3/2, y+1/2, -z+3/2$; (ii) $-x+1/2, y-1/2, -z+3/2$.

Fig. 1

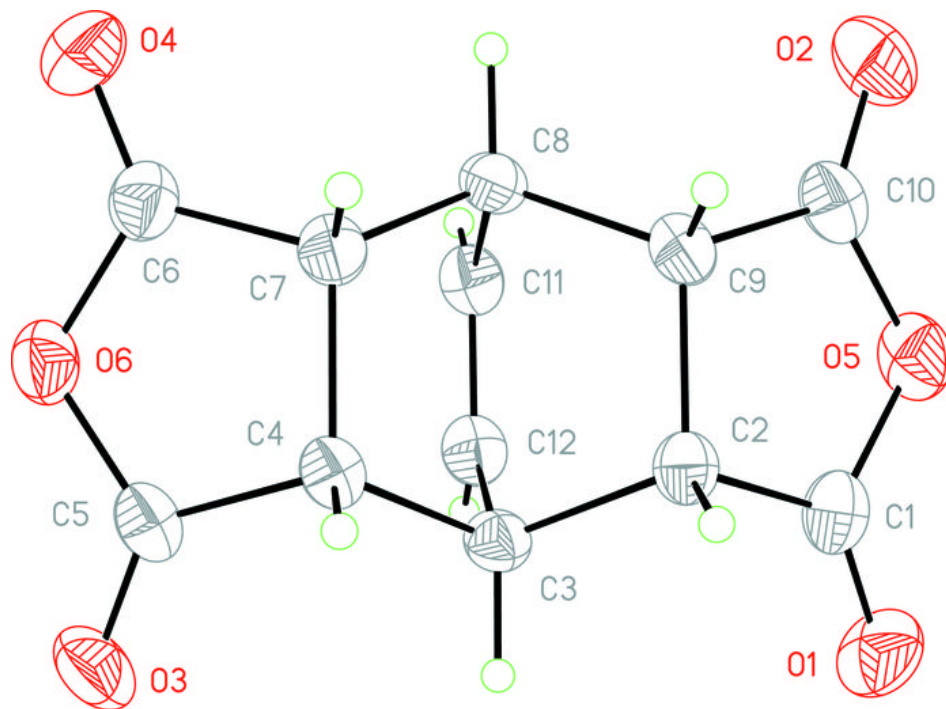


Fig. 2

