Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

Biphenyl-2,2′,4,4′-tetracarboxylic acid monohydrate

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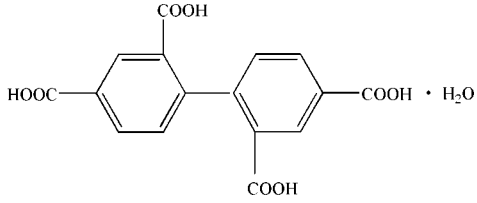
Received 11 August 2010; accepted 18 September 2010

Key indicators: single-crystal X-ray study; T = 296 K; mean σ(C–C) = 0.002 Å; R factor = 0.041; wR factor = 0.123; data-to-parameter ratio = 11.3.

In the title compound, $C_{16}H_{10}O_8\cdot H_2O$, the dihedral angle between the two benzene rings is 71.63 (5)°. In the crystal structure, pairs of inversion-related molecules are stacked [mean interplanar spacing = 3.5195 (18) Å], and O—H···O and C—H···O hydrogen bonds create a three-dimensional network.

Related literature

For general background to the use of aromatic carboxylates as building blocks for the construction of various architectures, see: Li *et al.* (2008); Du *et al.* (2007). For previous studies on the synthesis of aromatic carboxylate hydrates, see: Jiang *et al.* (2008); Li *et al.* (2009).



Experimental

Crystal data

$C_{16}H_{10}O_8\cdot H_2O$
M_r = 348.26
Triclinic, $P\overline{1}$
a = 7.1765 (1) Å
b = 9.4677 (2) Å
c = 11.9301 (2) Å
α = 106.013 (1)°
β = 100.098 (1)°
γ = 96.753 (1)°
V = 755.18 (2) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.13 mm^{-1}
T = 296 K
0.22 × 0.20 × 0.19 mm

Data collection

Bruker APEXII CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2007)
T_{min} = 0.972, T_{max} = 0.976
13177 measured reflections
2663 independent reflections
2441 reflections with $I > 2\sigma(I)$
R_{int} = 0.019

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.041
$wR(F^2)$ = 0.123
S = 1.03
2663 reflections
236 parameters
3 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.22 e Å^{-3}
$\Delta\rho_{min}$ = −0.41 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O6—H6A···O9	0.82	1.79	2.6076 (18)	174
O1—H1A···O2^{i}	0.82	1.87	2.680 (2)	169
O3—H3A···O4^{ii}	0.82	1.84	2.6400 (17)	166
O7—H7A···O5^{iii}	0.82	1.82	2.6280 (17)	171
O9—H9B···O8^{iv}	0.85 (1)	1.92 (1)	2.7616 (19)	170 (2)
O9—H9A···O8^{v}	0.84 (1)	2.20 (1)	2.983 (2)	154 (2)
C12—H12···O3^{vi}	0.93	2.57	3.451 (2)	159

Symmetry codes: (i) $-x+2, -y+3, -z+1$; (ii) $-x, -y+2, -z+1$; (iii) $x, y-1, z$; (iv) $-x, -y+2, -z+2$; (v) $x, y+1, z$; (vi) $-x+1, -y+2, -z+1$.

Data collection: *APEX2* (Bruker, 2007); cell refinement: *SAINT* (Bruker, 2007); data reduction: *SAINT*; 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*.

We thank the Universities and Colleges Natural Science Foundation of Henan (2009 A150011) and the Natural Science Foundation of China (200903036) for support.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: PK2260).

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Li, C.-P., Tian, Y.-L. & Guo, Y.-M. (2008). *Inorg. Chem. Commun.* **11**, 1405–1408.
Li, F., Wang, W.-W., Ji, X., Cao, C.-C. & Zhu, D.-Y. (2009). *Acta Cryst.* E**65**, o244.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.

supporting information

Acta Cryst. (2010). E**66**, o2626 [doi:10.1107/S1600536810037438]

Biphenyl-2,2′,4,4′-tetracarboxylic acid monohydrate

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S1. Comment

Aromatic carboxylates have been proven to be effective building blocks in constructing various architectures (Li *et al.*, 2008; Du *et al.*, 2007). Many crystal structures of aromatic carboxylic hydrates have been reported (Jiang *et al.*, 2008; Li *et al.*, 2009). In this paper, we report the synthesis and structure of a new aromatic carboxylic hydrate, biphenyl-2, 2′, 4, 4′ -tetracarboxylic acid monohydrate, (Fig. 1).

The dihedral angle between the two benzene rings of biphenyl-2, 2′, 4, 4′ -tetracarboxylic acid monohydrate is 71.63 (5)°, which is markedly different from 42.30 (11)° found in the biphenyl-2, 3, 3′, 4′-tetracarboxylic acid monohydrate (Jiang *et al.*, 2008). This might be a result of the hydrogen bonding ineractions of the title compound. The lattice water molecule links with biphenyl-2, 2′, 4, 4′-tetracarboxylic acid *via* O—H···O hydrogen bonding. The extensive O—H···O hydrogen bonding and a weak intermolecular C—H···O hydrogen bond helps to stabilize the crystal structure (Fig. 2 and Table 1). In addition, pairs of inversion related molecules are stacked with mean interplanar spacing = 3.5195 (18)Å)

S2. Experimental

A mixture of $C_{16}H_{10}O_8$ (0.3360 g), $BaCl_2$ (0.3451 g) and water (12 ml) was stirred at room temperature for 6 h. The solution was filtered and the filtrate was left to stand undisturbed. Upon slow evaporation at room temperature, a colorless crystalline solid appeared about a month later. The resulting colorless blocks were filtered off washed with water and dried at ambient temperature.

S3. Refinement

The H atoms of water molecules were located in difference Fourier maps and refined. All other hydrogen atoms were included in calculated positions and refined using a riding model with isotropic thermal parameters derived from the parent atoms (C—H = 0.93Å, O—H = 0.82 Å, U_{iso} (H) = 1.2Ueq (C) or 1.5Ueq (O)).

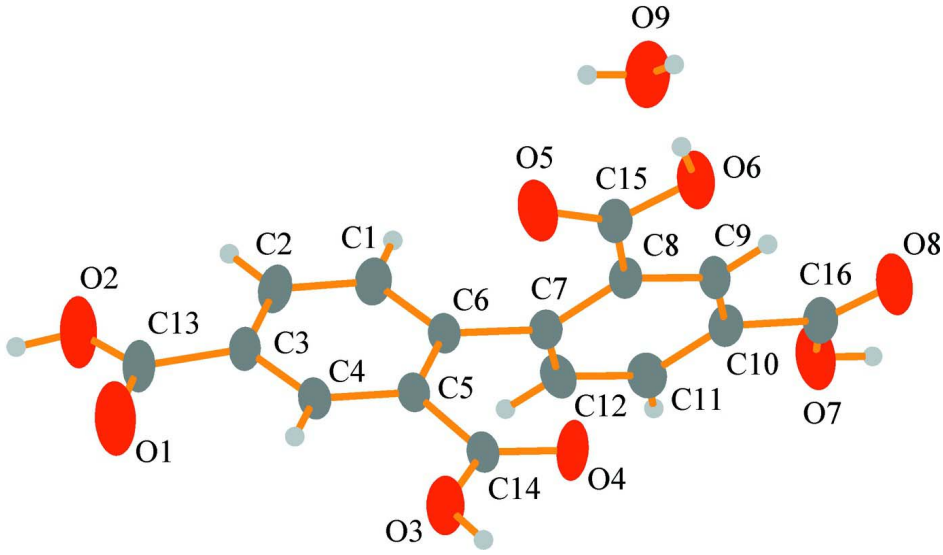

Figure 1

The structure of the title compound showing the atom-labeling scheme. Displacement ellipsoids are drawn at the 50% probability level

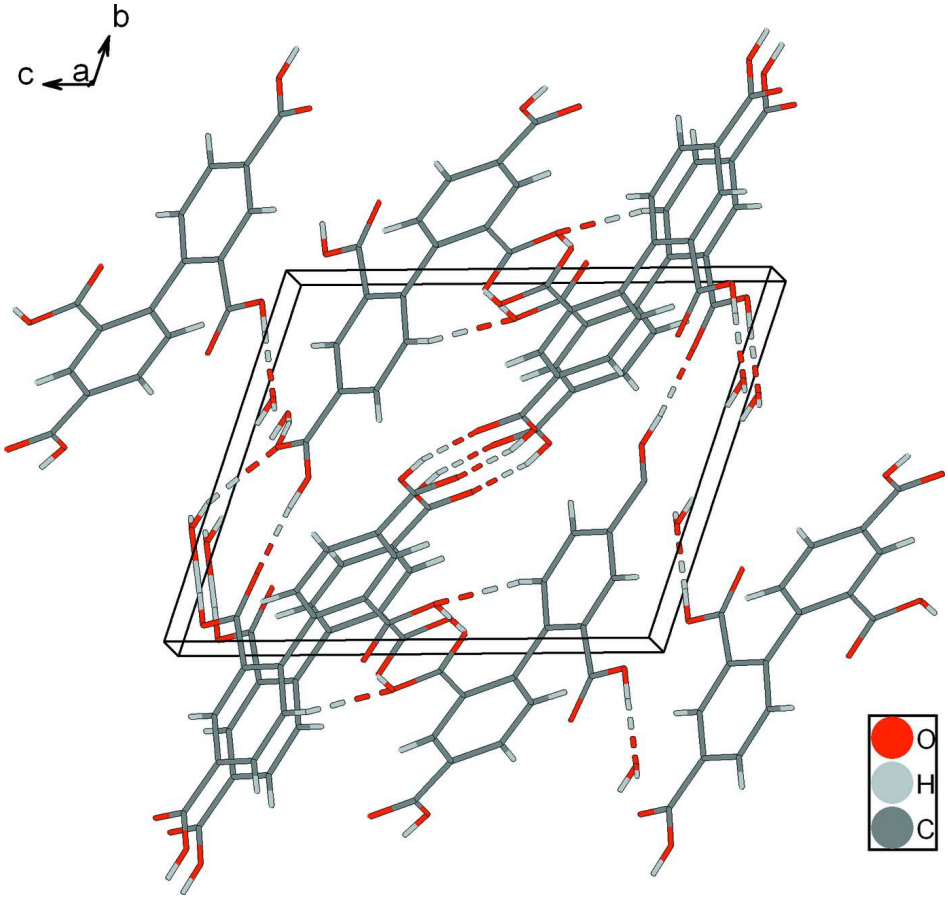

Figure 2

The O···H—O and C—H···O hydrogen bonds of biphenyl-2, 2′, 4, 4′ -tetracarboxylic acid monhydrate.

Biphenyl-2,2′,4,4′-tetracarboxylic acid monohydrate

Crystal data

$C_{16}H_{10}O_8 \cdot H_2O$
$M_r = 348.26$
Triclinic, $P\overline{1}$
Hall symbol: -P 1
$a = 7.1765$ (1) Å
$b = 9.4677$ (2) Å
$c = 11.9301$ (2) Å
$\alpha = 106.013$ (1)°
$\beta = 100.098$ (1)°
$\gamma = 96.753$ (1)°
$V = 755.18$ (2) Å^3
$Z = 2$
$F(000) = 360$
$D_x = 1.532$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 9316 reflections
$\theta = 2.8$–27.5°
$\mu = 0.13$ mm^{-1}
$T = 296$ K
Block, yellow
0.22 × 0.20 × 0.19 mm

Data collection

Bruker APEXII CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2007)
$T_{min} = 0.972$, $T_{max} = 0.976$
13177 measured reflections
2663 independent reflections
2441 reflections with $I > 2\sigma(I)$

R_{int} = 0.019
θ_{max} = 25.0°, θ_{min} = 1.8°
h = −8→8
k = −11→11
l = −14→14

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.041
$wR(F^2)$ = 0.123
S = 1.03
2663 reflections
236 parameters
3 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0701P)^2 + 0.3599P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.22 e Å^{-3}
$\Delta\rho_{min}$ = −0.41 e Å^{-3}

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 (Å²)

	x	y	z	U_{iso}*/U_{eq}
O1	0.7651 (3)	1.4462 (2)	0.51405 (19)	0.0815 (6)
H1A	0.8362	1.4851	0.4799	0.122*
O2	1.0447 (2)	1.4309 (2)	0.61981 (17)	0.0734 (5)
O3	0.19262 (18)	1.12724 (18)	0.49778 (13)	0.0522 (4)
H3A	0.0770	1.0941	0.4759	0.078*
O4	0.17196 (18)	0.97698 (17)	0.60878 (13)	0.0518 (4)
O5	0.3535 (2)	1.20930 (14)	0.88045 (14)	0.0531 (4)
O6	0.1817 (2)	1.07466 (14)	0.96298 (13)	0.0448 (3)
H6A	0.1502	1.1551	0.9916	0.067*
O7	0.4508 (2)	0.46084 (14)	0.83504 (15)	0.0531 (4)
H7A	0.4132	0.3873	0.8539	0.080*
O8	0.2313 (2)	0.55361 (14)	0.92899 (13)	0.0474 (4)
O9	0.1086 (2)	1.34013 (16)	1.05603 (16)	0.0556 (4)
C1	0.7674 (2)	1.1428 (2)	0.77090 (16)	0.0376 (4)
H1	0.8355	1.1113	0.8306	0.045*
C2	0.8611 (3)	1.2492 (2)	0.73154 (17)	0.0398 (4)
H2	0.9908	1.2877	0.7640	0.048*
C3	0.7614 (2)	1.29848 (19)	0.64325 (15)	0.0341 (4)
C4	0.5674 (2)	1.24238 (18)	0.59864 (14)	0.0308 (4)
H4	0.4990	1.2783	0.5419	0.037*
C5	0.4723 (2)	1.13359 (17)	0.63672 (13)	0.0273 (3)

C6	0.5744 (2)	1.08125 (17)	0.72423 (14)	0.0283 (4)
C7	0.4997 (2)	0.95149 (17)	0.76245 (14)	0.0278 (3)
C8	0.3751 (2)	0.95295 (17)	0.84121 (14)	0.0275 (3)
C9	0.3250 (2)	0.82523 (17)	0.87296 (14)	0.0288 (4)
H9	0.2409	0.8259	0.9241	0.035*
C10	0.3984 (2)	0.69691 (17)	0.82967 (14)	0.0298 (4)
C11	0.5231 (3)	0.69593 (18)	0.75326 (15)	0.0346 (4)
H11	0.5729	0.6103	0.7236	0.041*
C12	0.5734 (2)	0.82211 (19)	0.72119 (15)	0.0337 (4)
H12	0.6588	0.8206	0.6707	0.040*
C13	0.8643 (3)	1.4007 (2)	0.59059 (18)	0.0441 (5)
C14	0.2659 (2)	1.07440 (18)	0.57925 (14)	0.0289 (4)
C15	0.3025 (2)	1.09080 (18)	0.89507 (15)	0.0305 (4)
C16	0.3493 (2)	0.56452 (18)	0.86928 (15)	0.0326 (4)
H9B	0.0046 (19)	1.370 (2)	1.0685 (19)	0.049*
H9A	0.177 (2)	1.4042 (19)	1.037 (2)	0.049*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0562 (10)	0.1074 (15)	0.0975 (14)	−0.0142 (10)	0.0034 (9)	0.0776 (12)
O2	0.0451 (9)	0.0946 (13)	0.0903 (13)	−0.0128 (8)	0.0080 (8)	0.0581 (11)
O3	0.0305 (7)	0.0725 (10)	0.0609 (9)	−0.0063 (6)	−0.0048 (6)	0.0472 (8)
O4	0.0309 (7)	0.0645 (9)	0.0665 (9)	−0.0116 (6)	−0.0020 (6)	0.0463 (8)
O5	0.0791 (10)	0.0269 (7)	0.0717 (10)	0.0150 (6)	0.0410 (8)	0.0268 (6)
O6	0.0483 (8)	0.0325 (7)	0.0667 (9)	0.0130 (6)	0.0330 (7)	0.0210 (6)
O7	0.0648 (9)	0.0334 (7)	0.0836 (10)	0.0199 (7)	0.0408 (8)	0.0341 (7)
O8	0.0514 (8)	0.0381 (7)	0.0709 (9)	0.0136 (6)	0.0310 (7)	0.0326 (7)
O9	0.0501 (9)	0.0410 (8)	0.0905 (11)	0.0181 (6)	0.0342 (8)	0.0280 (8)
C1	0.0303 (9)	0.0426 (10)	0.0421 (9)	−0.0012 (7)	0.0001 (7)	0.0243 (8)
C2	0.0291 (9)	0.0432 (10)	0.0462 (10)	−0.0070 (7)	0.0026 (7)	0.0210 (8)
C3	0.0327 (9)	0.0336 (9)	0.0372 (9)	−0.0034 (7)	0.0087 (7)	0.0158 (7)
C4	0.0323 (9)	0.0319 (8)	0.0319 (8)	0.0013 (7)	0.0071 (7)	0.0172 (7)
C5	0.0268 (8)	0.0283 (8)	0.0293 (8)	0.0022 (6)	0.0083 (6)	0.0125 (6)
C6	0.0290 (8)	0.0278 (8)	0.0306 (8)	0.0020 (6)	0.0081 (6)	0.0130 (6)
C7	0.0270 (8)	0.0286 (8)	0.0292 (8)	0.0011 (6)	0.0027 (6)	0.0147 (6)
C8	0.0264 (8)	0.0256 (8)	0.0328 (8)	0.0024 (6)	0.0051 (6)	0.0144 (6)
C9	0.0288 (8)	0.0274 (8)	0.0350 (8)	0.0029 (6)	0.0103 (6)	0.0156 (6)
C10	0.0315 (8)	0.0252 (8)	0.0349 (8)	0.0028 (6)	0.0063 (7)	0.0142 (6)
C11	0.0409 (9)	0.0277 (8)	0.0401 (9)	0.0095 (7)	0.0136 (7)	0.0138 (7)
C12	0.0378 (9)	0.0340 (9)	0.0356 (9)	0.0068 (7)	0.0148 (7)	0.0164 (7)
C13	0.0344 (10)	0.0508 (11)	0.0503 (11)	−0.0077 (8)	0.0054 (8)	0.0289 (9)
C14	0.0274 (8)	0.0321 (8)	0.0310 (8)	0.0028 (6)	0.0075 (6)	0.0159 (6)
C15	0.0309 (8)	0.0277 (8)	0.0371 (8)	0.0049 (6)	0.0078 (7)	0.0165 (7)
C16	0.0337 (9)	0.0264 (8)	0.0422 (9)	0.0048 (6)	0.0097 (7)	0.0170 (7)

Geometric parameters (Å, º)

O1—C13	1.260 (2)	C3—C4	1.384 (2)
O1—H1A	0.8200	C3—C13	1.486 (2)
O2—C13	1.256 (2)	C4—C5	1.391 (2)
O3—C14	1.274 (2)	C4—H4	0.9300
O3—H3A	0.8200	C5—C6	1.405 (2)
O4—C14	1.243 (2)	C5—C14	1.489 (2)
O5—C15	1.207 (2)	C6—C7	1.498 (2)
O6—C15	1.309 (2)	C7—C12	1.391 (2)
O6—H6A	0.8200	C7—C8	1.405 (2)
O7—C16	1.309 (2)	C8—C9	1.390 (2)
O7—H7A	0.8200	C8—C15	1.484 (2)
O8—C16	1.211 (2)	C9—C10	1.385 (2)
O9—H9B	0.852 (9)	C9—H9	0.9300
O9—H9A	0.843 (9)	C10—C11	1.384 (2)
C1—C2	1.378 (2)	C10—C16	1.483 (2)
C1—C6	1.389 (2)	C11—C12	1.380 (2)
C1—H1	0.9300	C11—H11	0.9300
C2—C3	1.387 (3)	C12—H12	0.9300
C2—H2	0.9300		

C13—O1—H1A	109.5	C9—C8—C15	119.43 (14)
C14—O3—H3A	109.5	C7—C8—C15	120.93 (14)
C15—O6—H6A	109.5	C10—C9—C8	121.11 (15)
C16—O7—H7A	109.5	C10—C9—H9	119.4
H9B—O9—H9A	109.6 (14)	C8—C9—H9	119.4
C2—C1—C6	122.08 (16)	C11—C10—C9	119.36 (14)
C2—C1—H1	119.0	C11—C10—C16	120.69 (15)
C6—C1—H1	119.0	C9—C10—C16	119.91 (15)
C1—C2—C3	119.76 (16)	C12—C11—C10	119.95 (15)
C1—C2—H2	120.1	C12—C11—H11	120.0
C3—C2—H2	120.1	C10—C11—H11	120.0
C4—C3—C2	119.01 (15)	C11—C12—C7	121.64 (15)
C4—C3—C13	120.32 (16)	C11—C12—H12	119.2
C2—C3—C13	120.49 (16)	C7—C12—H12	119.2
C3—C4—C5	121.54 (15)	O2—C13—O1	123.66 (18)
C3—C4—H4	119.2	O2—C13—C3	118.64 (17)
C5—C4—H4	119.2	O1—C13—C3	117.59 (16)
C4—C5—C6	119.40 (14)	O4—C14—O3	122.26 (15)
C4—C5—C14	117.74 (14)	O4—C14—C5	121.23 (14)
C6—C5—C14	122.84 (14)	O3—C14—C5	116.51 (14)
C1—C6—C5	118.14 (14)	O5—C15—O6	122.33 (16)
C1—C6—C7	115.96 (14)	O5—C15—C8	123.22 (15)
C5—C6—C7	125.51 (14)	O6—C15—C8	114.42 (14)
C12—C7—C8	118.34 (14)	O8—C16—O7	123.12 (15)
C12—C7—C6	115.43 (14)	O8—C16—C10	124.19 (15)
C8—C7—C6	126.07 (14)	O7—C16—C10	112.68 (14)

C9—C8—C7	119.59 (15)		
C6—C1—C2—C3	0.5 (3)	C8—C9—C10—C11	−0.2 (2)
C1—C2—C3—C4	1.7 (3)	C8—C9—C10—C16	177.30 (15)
C1—C2—C3—C13	−173.46 (18)	C9—C10—C11—C12	0.2 (3)
C2—C3—C4—C5	−2.7 (3)	C16—C10—C11—C12	−177.33 (16)
C13—C3—C4—C5	172.56 (16)	C10—C11—C12—C7	−0.9 (3)
C3—C4—C5—C6	1.3 (2)	C8—C7—C12—C11	1.7 (2)
C3—C4—C5—C14	−176.96 (15)	C6—C7—C12—C11	177.43 (15)
C2—C1—C6—C5	−1.9 (3)	C4—C3—C13—O2	−168.9 (2)
C2—C1—C6—C7	171.29 (17)	C2—C3—C13—O2	6.2 (3)
C4—C5—C6—C1	1.0 (2)	C4—C3—C13—O1	7.5 (3)
C14—C5—C6—C1	179.14 (15)	C2—C3—C13—O1	−177.4 (2)
C4—C5—C6—C7	−171.49 (15)	C4—C5—C14—O4	179.96 (16)
C14—C5—C6—C7	6.6 (2)	C6—C5—C14—O4	1.8 (3)
C1—C6—C7—C12	−67.3 (2)	C4—C5—C14—O3	0.9 (2)
C5—C6—C7—C12	105.40 (19)	C6—C5—C14—O3	−177.24 (16)
C1—C6—C7—C8	108.06 (19)	C9—C8—C15—O5	173.05 (17)
C5—C6—C7—C8	−79.3 (2)	C7—C8—C15—O5	−4.3 (3)
C12—C7—C8—C9	−1.7 (2)	C9—C8—C15—O6	−5.2 (2)
C6—C7—C8—C9	−176.93 (15)	C7—C8—C15—O6	177.43 (15)
C12—C7—C8—C15	175.65 (15)	C11—C10—C16—O8	−174.54 (17)
C6—C7—C8—C15	0.5 (2)	C9—C10—C16—O8	8.0 (3)
C7—C8—C9—C10	1.0 (2)	C11—C10—C16—O7	6.9 (2)
C15—C8—C9—C10	−176.42 (14)	C9—C10—C16—O7	−170.53 (16)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O6—H6*A*···O9	0.82	1.79	2.6076 (18)	174
C4—H4···O3	0.93	2.37	2.706 (2)	101
C9—H9···O6	0.93	2.38	2.711 (2)	101
O1—H1*A*···O2^{i}	0.82	1.87	2.680 (2)	169
O3—H3*A*···O4^{ii}	0.82	1.84	2.6400 (17)	166
O7—H7*A*···O5^{iii}	0.82	1.82	2.6280 (17)	171
O9—H9*B*···O8^{iv}	0.85 (1)	1.92 (1)	2.7616 (19)	170 (2)
O9—H9*A*···O8^{v}	0.84 (1)	2.20 (1)	2.983 (2)	154 (2)
C12—H12···O3^{vi}	0.93	2.57	3.451 (2)	159

Symmetry codes: (i) −*x*+2, −*y*+3, −*z*+1; (ii) −*x*, −*y*+2, −*z*+1; (iii) *x*, *y*−1, *z*; (iv) −*x*, −*y*+2, −*z*+2; (v) *x*, *y*+1, *z*; (vi) −*x*+1, −*y*+2, −*z*+1.