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1,3-Di-4-pyridylpropane–2-hydroxybenzene-1,4-dicarboxylic acid (1/2)

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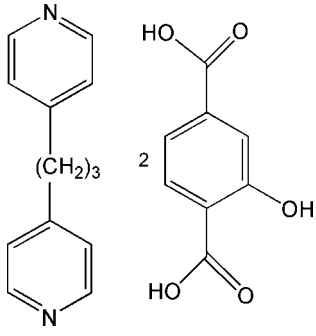
Key indicators: single-crystal X-ray study; T = 291 K; mean σ(C–C) = 0.004 Å; R factor = 0.056; wR factor = 0.173; data-to-parameter ratio = 13.1.

In the title compound, $C_{13}H_{14}N_2{\cdot}2C_8H_6O_5$, which crystallized in the monoclinic $C2/c$ space group, the 1,3-bis(4-pyridyl)propane molecules and 2-hydroxy-1,4-benzenedicarboxylic acid molecules are alternately linked by O—H···N and O—H···O hydrogen bonds into herringbone/zigzag chains.

Related literature

For general background, see: Bowers *et al.* (2005); Mukherjee *et al.* (2004). For the substitution of bromine for hydroxyl, see: Chen & Tong (2007); Zhang (2005).



Experimental

Crystal data

$C_{13}H_{14}N_2{\cdot}2C_8H_6O_5$
M_r = 562.52
Monoclinic, $C2/c$
a = 22.939 (11) Å
b = 4.781 (2) Å
c = 24.163 (11) Å
β = 96.542 (6)°
V = 2633 (2) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.11 mm^{-1}
T = 291 (2) K
0.35 × 0.19 × 0.05 mm

Data collection

Bruker CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 1997)
T_{min} = 0.963, T_{max} = 0.994
9178 measured reflections
2444 independent reflections
1335 reflections with $I > 2\sigma(I)$
R_{int} = 0.049

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.056
$wR(F^2)$ = 0.173
S = 1.03
2444 reflections
187 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.39 e Å^{-3}
$\Delta\rho_{min}$ = −0.18 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O4—H4···O5^{i}	0.82	1.82	2.631 (3)	172
O2—H2···N1	0.82	1.75	2.568 (3)	174
O1—H1···O3	0.82	1.79	2.516 (3)	147

Symmetry code: (i) $-x+1, -y+4, -z+1$.

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT* (Bruker, 1997); 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*.

The authors thank Luoyang Normal University for supporting this work.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: WW2129).

References

Bowers, J. R., Hopkins, G. W., Yap, G. P. A. & Wheeler, K. A. (2005). *Cryst. Growth Des.* **5**, 727–736.
Bruker (1997). *SMART*, *SAINT* and *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.
Chen, X. M. & Tong, M. L. (2007). *Acc. Chem. Res.* **40**, 162–170.
Mukherjee, P. S., Ghoshal, D., Zangrando, E., Mallah, T. & Chaudhuri, N. R. (2004). *Eur. J. Inorg. Chem.* **23**, 4675–4680.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Zhang, X. M. (2005). *Coord. Chem. Rev.* **249**, 1201–1219.

supporting information

Acta Cryst. (2008). E**64**, o2398 [doi:10.1107/S1600536808037835]

1,3-Di-4-pyridylpropane–2-hydroxybenzene-1,4-dicarboxylic acid (1/2)

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S1. Comment

The aromatic dicarboxylate are used extensively in the synthesis of coordination polymers (Mukherjee *et al.*, 2004) and the generation of hydrogen-bonding arrays of organic co-crystals (Bowers *et al.*, 2005). The 2-bromo-1,4-benzenedicarboxylic acid, possesses several interesting characteristics: (*a*) it has two carboxyl groups which may be completely or partially deprotonated, inducing rich coordination modes and allowing interesting structures with higher dimensions; (*b*) it can act not only as hydrogen-bond acceptor but also as hydrogen-bond donor, depending upon the number of deprotonated carboxyl groups. To propagate non-covalent interactions only along one direction, each unit must have two-point interactions with two adjacent neighbors. Dicarboxylic acids epitomize this model and exhibits a two-point contact per unit that can result in 1-D hydrogen bonding networks.

The crystal structure of the title compound comprises half of a 1,3-bis(4-pyridyl)propane (bpp) molecule and a 2-hydroxy-1,4-benzenedicarboxylic acid molecule per asymmetric unit (Fig. 1). One bpp molecule connects two adjacent 2-hydroxy-1,4-benzenedicarboxylic acid molecules *via* O—H···N hydrogen bonds, and complementary *via* O—H···O hydrogen bonds between adjacent dicarboxylic acid molecules, which extend into an one-dimensional herringbone/zigzag chain structure (Fig. 2). The dihedral angle between two pyridyl rings is 85.551 (11)°, and thus incorporation into supramolecular architecture imparts significant conformation change in the bpp molecule. The 1-D chain structure seems to suggest that the CH_2 chain in pyridyl bases is not an inert spacer but could have a conformational and structural role in controlling the supramolecular architecture.

S2. Experimental

1,3-Bis(4-pyridyl)propane (bpp) (0.5 mmol), 2-bromo-1,4-benzenedicarboxylic acid (1.0 mmol), and KOH (0.5 mmol) were added to water (12 ml) in a Teflon-lined stainless steel reactor. The mixture was heated at 435 K for 3 d, and then slowly cooled down to room temperature. Colorless crystals of the title compound were obtained. Elemental analysis – found: C, 61.81%; H, 4.56%; N, 4.92%; calc. for $C_{29}H_{26}N_2O_{10}$: C, 61.86%; H, 4.62%; N, 4.98%. Note the substitution of bromine for hydroxyl in the formation of title compound, which commonly happened under hydro(solvo)thermal conditions (Chen & Tong, 2007; Zhang, 2005).

S3. Refinement

H atoms were positioned geometrically and treated as riding, with C—H bonding lengths constrained to 0.93 (aromatic CH), or 0.97Å (methylene CH_2), and O—H=0.82Å, and with $U_{iso}(H) = 1.2U_{eq}(C)$ or $U_{iso}(H) = 1.5U_{eq}(O)$.

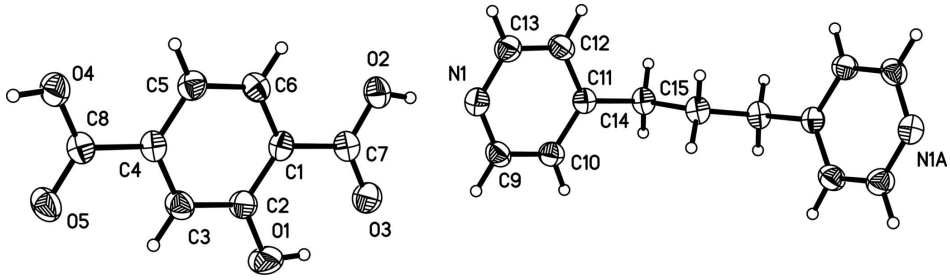

Figure 1

A view of the title compound, showing 30% probability displacement ellipsoids. Symmetry code: (A) -*x*, *y*, 1/2 - *z*.

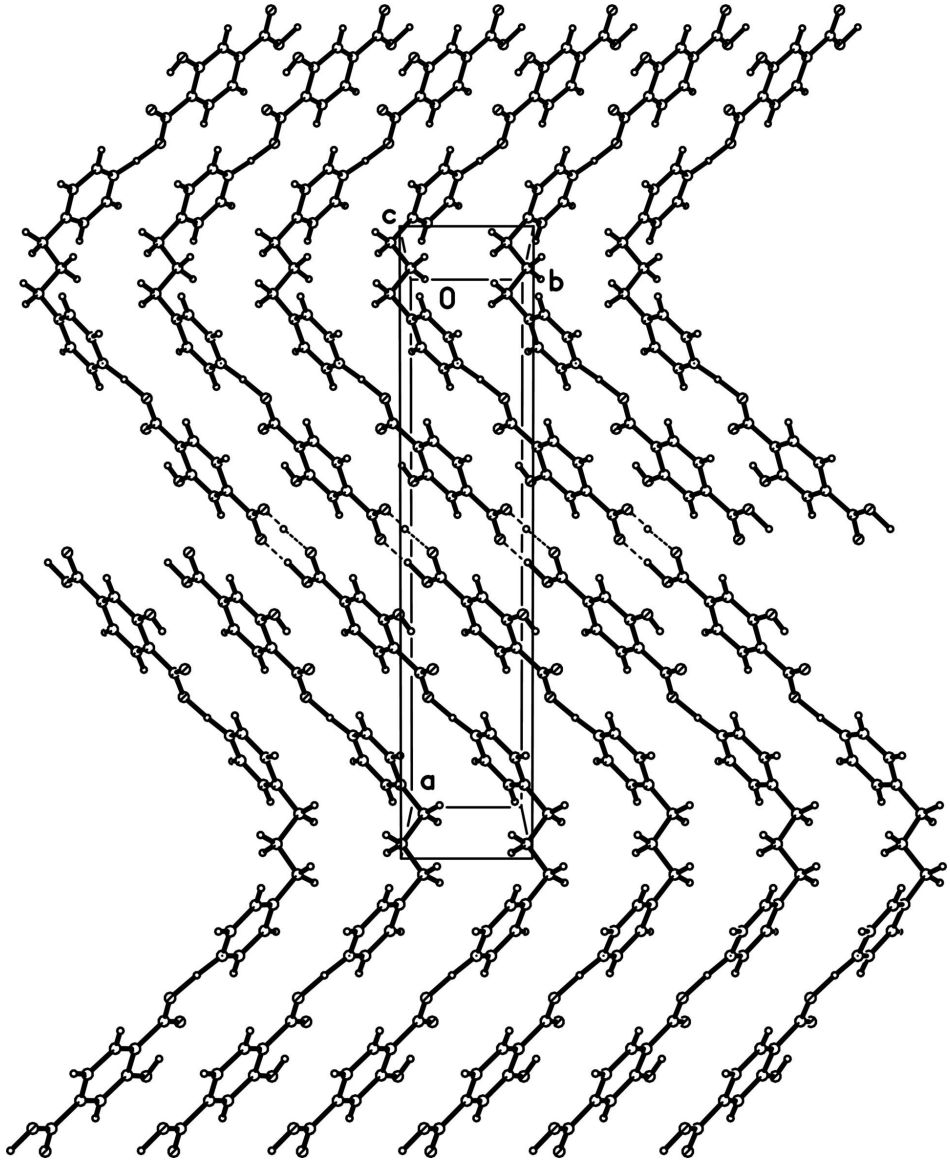

Figure 2

The unit-cell packing of the title compound, showing the hydrogen bonding interactions.

1,3-Di-4-pyridylpropane–2-hydroxybenzene-1,4-dicarboxylic acid (1/2)

Crystal data

$C_{13}H_{14}N_2 \cdot 2C_8H_6O_5$
$M_r = 562.52$
Monoclinic, $C2/c$
Hall symbol: -C 2yc
$a = 22.939$ (11) Å
$b = 4.781$ (2) Å
$c = 24.163$ (11) Å
$\beta = 96.542$ (6)°
$V = 2633$ (2) Å^3
$Z = 4$
$F(000) = 1176$
$D_x = 1.419$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 1102 reflections
$\theta = 3.4$–$23.3°$
$\mu = 0.11$ mm^{-1}
$T = 291$ K
Block, colorless
0.35 × 0.19 × 0.05 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω & φ scans
Absorption correction: multi-scan (*SADABS*; Bruker, 1997)
$T_{min} = 0.963$, $T_{max} = 0.994$
9178 measured reflections
2444 independent reflections
1335 reflections with $I > 2\sigma(I)$
$R_{int} = 0.049$
$\theta_{max} = 25.5°$, $\theta_{min} = 2.6°$
$h = -27 \rightarrow 27$
$k = -5 \rightarrow 5$
$l = -29 \rightarrow 28$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.056$
$wR(F^2) = 0.173$
$S = 1.03$
2444 reflections
187 parameters
0 restraints
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
$w = 1/[\sigma^2(F_o^2) + (0.0772P)^2 + 1.353P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.39$ e Å^{-3}
$\Delta\rho_{min} = -0.18$ 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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
O1	0.38415 (11)	1.0471 (6)	0.32590 (9)	0.0857 (9)	
H1	0.3557	0.9516	0.3141	0.129*	
O2	0.24082 (9)	0.8169 (5)	0.40221 (9)	0.0642 (7)	
H2	0.2203	0.6976	0.3852	0.096*	

O3	0.29118 (9)	0.7716 (5)	0.32941 (9)	0.0695 (7)	
O4	0.43663 (9)	1.8252 (5)	0.51792 (9)	0.0666 (7)	
H4	0.4624	1.9400	0.5274	0.100*	
O5	0.48854 (9)	1.7794 (5)	0.44642 (10)	0.0679 (7)	
N1	0.17662 (11)	0.4242 (5)	0.35544 (11)	0.0516 (7)	
C1	0.32537 (12)	1.0947 (6)	0.40090 (12)	0.0467 (7)	
C2	0.37382 (13)	1.1721 (6)	0.37430 (13)	0.0539 (8)	
C3	0.41262 (13)	1.3722 (6)	0.39812 (13)	0.0568 (8)	
H3	0.4451	1.4215	0.3805	0.068*	
C4	0.40338 (12)	1.4989 (6)	0.44770 (12)	0.0470 (7)	
C5	0.35515 (13)	1.4263 (6)	0.47459 (13)	0.0535 (8)	
H5	0.3486	1.5125	0.5078	0.064*	
C6	0.31687 (13)	1.2222 (6)	0.45088 (13)	0.0551 (8)	
H6	0.2849	1.1702	0.4690	0.066*	
C7	0.28337 (13)	0.8782 (6)	0.37506 (14)	0.0527 (8)	
C8	0.44604 (13)	1.7142 (6)	0.47143 (13)	0.0493 (8)	
C9	0.18299 (12)	0.3070 (7)	0.30666 (13)	0.0534 (8)	
H9	0.2147	0.3584	0.2881	0.064*	
C10	0.14366 (12)	0.1103 (6)	0.28276 (12)	0.0516 (8)	
H10	0.1493	0.0298	0.2488	0.062*	
C11	0.09577 (12)	0.0334 (6)	0.30954 (12)	0.0449 (7)	
C12	0.09152 (13)	0.1551 (7)	0.36110 (13)	0.0570 (8)	
H12	0.0609	0.1056	0.3813	0.068*	
C13	0.13223 (14)	0.3479 (7)	0.38230 (13)	0.0575 (8)	
H13	0.1285	0.4277	0.4168	0.069*	
C14	0.04828 (11)	−0.1557 (6)	0.28235 (12)	0.0499 (8)	
H14A	0.0318	−0.2651	0.3106	0.060*	
H14B	0.0648	−0.2837	0.2572	0.060*	
C15	0.0000	0.0194 (8)	0.2500	0.0498 (11)	
H15A	0.0176	0.1391	0.2241	0.060*	0.50
H15B	−0.0176	0.1391	0.2759	0.060*	0.50

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.109 (2)	0.092 (2)	0.0608 (15)	−0.0379 (16)	0.0301 (15)	−0.0327 (13)
O2	0.0605 (14)	0.0580 (14)	0.0726 (15)	−0.0154 (11)	0.0015 (12)	−0.0059 (12)
O3	0.0756 (15)	0.0679 (16)	0.0653 (15)	−0.0160 (12)	0.0091 (12)	−0.0159 (12)
O4	0.0658 (14)	0.0597 (14)	0.0748 (16)	−0.0169 (12)	0.0102 (12)	−0.0179 (13)
O5	0.0593 (14)	0.0617 (15)	0.0858 (16)	−0.0161 (12)	0.0222 (12)	−0.0191 (12)
N1	0.0513 (15)	0.0481 (15)	0.0543 (16)	−0.0003 (12)	0.0012 (13)	0.0053 (13)
C1	0.0496 (18)	0.0359 (16)	0.0527 (18)	−0.0006 (13)	−0.0027 (14)	−0.0012 (14)
C2	0.0597 (19)	0.0483 (18)	0.0543 (19)	−0.0076 (16)	0.0086 (15)	−0.0058 (16)
C3	0.059 (2)	0.0497 (19)	0.063 (2)	−0.0116 (16)	0.0148 (16)	−0.0048 (16)
C4	0.0456 (17)	0.0362 (17)	0.0578 (19)	0.0010 (13)	−0.0004 (15)	0.0018 (15)
C5	0.0593 (19)	0.0459 (18)	0.0552 (19)	−0.0024 (16)	0.0061 (15)	−0.0058 (15)
C6	0.0499 (18)	0.0527 (19)	0.063 (2)	−0.0059 (16)	0.0087 (15)	0.0009 (17)
C7	0.0475 (18)	0.0449 (18)	0.065 (2)	−0.0004 (14)	0.0028 (16)	0.0041 (17)

C8	0.0513 (18)	0.0350 (16)	0.061 (2)	0.0017 (14)	0.0033 (16)	−0.0043 (15)
C9	0.0450 (17)	0.055 (2)	0.061 (2)	−0.0051 (15)	0.0094 (15)	0.0089 (17)
C10	0.0518 (18)	0.0529 (19)	0.0509 (18)	0.0026 (15)	0.0093 (15)	0.0022 (15)
C11	0.0465 (17)	0.0364 (16)	0.0508 (17)	0.0044 (13)	0.0008 (14)	0.0045 (14)
C12	0.0504 (18)	0.064 (2)	0.059 (2)	−0.0045 (16)	0.0137 (15)	−0.0006 (17)
C13	0.062 (2)	0.058 (2)	0.0532 (19)	−0.0054 (17)	0.0070 (16)	−0.0081 (16)
C14	0.0467 (17)	0.0411 (17)	0.0607 (19)	−0.0015 (14)	0.0010 (14)	0.0004 (15)
C15	0.049 (2)	0.038 (2)	0.061 (3)	0.000	0.002 (2)	0.000

Geometric parameters (Å, º)

O1—C2	1.358 (3)	C5—C6	1.391 (4)
O1—H1	0.8200	C5—H5	0.9300
O2—C7	1.271 (3)	C6—H6	0.9300
O2—H2	0.8200	C9—C10	1.383 (4)
O3—C7	1.246 (4)	C9—H9	0.9300
O4—C8	1.283 (3)	C10—C11	1.387 (4)
O4—H4	0.8200	C10—H10	0.9300
O5—C8	1.244 (3)	C11—C12	1.389 (4)
N1—C13	1.320 (4)	C11—C14	1.507 (4)
N1—C9	1.328 (4)	C12—C13	1.369 (4)
C1—C6	1.386 (4)	C12—H12	0.9300
C1—C2	1.396 (4)	C13—H13	0.9300
C1—C7	1.501 (4)	C14—C15	1.530 (3)
C2—C3	1.386 (4)	C14—H14A	0.9700
C3—C4	1.380 (4)	C14—H14B	0.9700
C3—H3	0.9300	C15—C14^{i}	1.530 (3)
C4—C5	1.389 (4)	C15—H15A	0.9700
C4—C8	1.489 (4)	C15—H15B	0.9700
C2—O1—H1	109.5	N1—C9—C10	121.7 (3)
C7—O2—H2	109.5	N1—C9—H9	119.2
C8—O4—H4	109.5	C10—C9—H9	119.2
C13—N1—C9	119.3 (3)	C9—C10—C11	120.0 (3)
C6—C1—C2	118.9 (3)	C9—C10—H10	120.0
C6—C1—C7	121.2 (3)	C11—C10—H10	120.0
C2—C1—C7	119.9 (3)	C10—C11—C12	116.6 (3)
O1—C2—C3	119.6 (3)	C10—C11—C14	121.8 (3)
O1—C2—C1	120.4 (3)	C12—C11—C14	121.4 (3)
C3—C2—C1	119.9 (3)	C13—C12—C11	120.2 (3)
C4—C3—C2	120.6 (3)	C13—C12—H12	119.9
C4—C3—H3	119.7	C11—C12—H12	119.9
C2—C3—H3	119.7	N1—C13—C12	122.3 (3)
C3—C4—C5	120.4 (3)	N1—C13—H13	118.9
C3—C4—C8	118.6 (3)	C12—C13—H13	118.9
C5—C4—C8	121.0 (3)	C11—C14—C15	109.9 (2)
C4—C5—C6	118.8 (3)	C11—C14—H14A	109.7
C4—C5—H5	120.6	C15—C14—H14A	109.7

C6—C5—H5	120.6	C11—C14—H14B	109.7
C1—C6—C5	121.5 (3)	C15—C14—H14B	109.7
C1—C6—H6	119.3	H14A—C14—H14B	108.2
C5—C6—H6	119.3	C14—C15—C14^{i}	113.7 (3)
O3—C7—O2	124.0 (3)	C14—C15—H15A	108.8
O3—C7—C1	120.0 (3)	C14^{i}—C15—H15A	108.8
O2—C7—C1	116.0 (3)	C14—C15—H15B	108.8
O5—C8—O4	122.8 (3)	C14^{i}—C15—H15B	108.8
O5—C8—C4	120.2 (3)	H15A—C15—H15B	107.7
O4—C8—C4	117.0 (3)		

C6—C1—C2—O1	178.6 (3)	C2—C1—C7—O2	179.2 (3)
C7—C1—C2—O1	−1.8 (4)	C3—C4—C8—O5	0.9 (4)
C6—C1—C2—C3	0.4 (4)	C5—C4—C8—O5	−179.0 (3)
C7—C1—C2—C3	180.0 (3)	C3—C4—C8—O4	−179.1 (3)
O1—C2—C3—C4	−179.1 (3)	C5—C4—C8—O4	1.0 (4)
C1—C2—C3—C4	−0.8 (5)	C13—N1—C9—C10	1.2 (4)
C2—C3—C4—C5	0.3 (5)	N1—C9—C10—C11	0.7 (4)
C2—C3—C4—C8	−179.5 (3)	C9—C10—C11—C12	−2.3 (4)
C3—C4—C5—C6	0.6 (4)	C9—C10—C11—C14	173.2 (3)
C8—C4—C5—C6	−179.5 (3)	C10—C11—C12—C13	2.2 (4)
C2—C1—C6—C5	0.6 (5)	C14—C11—C12—C13	−173.4 (3)
C7—C1—C6—C5	−179.0 (3)	C9—N1—C13—C12	−1.4 (5)
C4—C5—C6—C1	−1.1 (4)	C11—C12—C13—N1	−0.3 (5)
C6—C1—C7—O3	178.2 (3)	C10—C11—C14—C15	−90.2 (3)
C2—C1—C7—O3	−1.4 (4)	C12—C11—C14—C15	85.1 (3)
C6—C1—C7—O2	−1.3 (4)	C11—C14—C15—C14^{i}	176.1 (3)

Symmetry code: (i) $-x, y, -z+1/2$.

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O4—H4···O5^{ii}	0.82	1.82	2.631 (3)	172
O2—H2···N1	0.82	1.75	2.568 (3)	174
O1—H1···O3	0.82	1.79	2.516 (3)	147

Symmetry code: (ii) $-x+1, -y+4, -z+1$.