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Butane-1,2,3,4-tetracarboxylic acid–4,4′-bipyridine (1/2)

Ning Zhang,[a] Xue-Min Shi,[a] Min Shao[b] and Ming-Xing Li[a]*

[a]Department of Chemistry, College of Science, Shanghai University, Shanghai 200444, People's Republic of China, and [b]Instrumental Analysis and Research Center, Shanghai University, Shanghai 200444, People's Republic of China
Correspondence e-mail: mx_li@mail.shu.edu.cn

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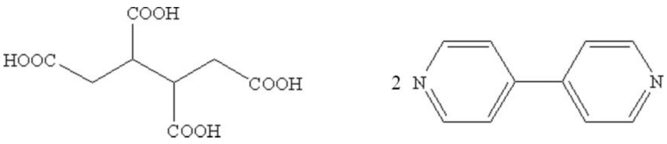
Key indicators: single-crystal X-ray study; T = 296 K; mean σ(C–C) = 0.005 Å; R factor = 0.061; wR factor = 0.176; data-to-parameter ratio = 12.0.

The hydrothermal reaction of butane-1,2,3,4-tetracarboxylic acid (H_4butca), 4,4′-bipyridine (bipy) and $Mn(SO_4)_2\cdot H_2O$ afforded a new co-crystal, $C_8H_{10}O_8\cdot 2C_{10}H_8N_2$ or H_4butca·2(bipy), in which strong O—H⋯N hydrogen-bonding and weak π–π stacking [centroid–centroid distance = 3.8459 (19) Å] interactions assemble the organic molecules into a three-dimensional supramolecular framework. C—H⋯O interactions are also present. The whole molecule has inversion symmetry.

Related literature

For the importance of hydrogen-bonding and π-π stacking interactions in supramolecular chemistry, crystal engineering and biological recognition, see: Wang *et al.* (2007). Many organic co-crystals have been assembled from *N*-heterocycles and polycarboxylic acids, see: Li *et al.* (2007). For the 1:1 co-crystal H_4butca·bipy, see: Najafpour *et al.* (2008).



Experimental

Crystal data

$C_8H_{10}O_8\cdot 2C_{10}H_8N_2$
M_r = 546.53
Triclinic, $P\overline{1}$
a = 7.4435 (11) Å
b = 8.6990 (13) Å
c = 11.438 (2) Å
α = 99.819 (3)°
β = 105.662 (3)°
γ = 111.361 (2)°
V = 633.57 (17) Å^3
Z = 1
Mo $K\alpha$ radiation
μ = 0.11 mm^{-1}
T = 296 K
0.30 × 0.30 × 0.30 mm

Data collection

Bruker APEXII CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996) T_{min} = 0.969, T_{max} = 0.969
3287 measured reflections
2196 independent reflections
1721 reflections with $I > 2\sigma(I)$
R_{int} = 0.013

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.061
$wR(F^2)$ = 0.176
S = 1.07
2196 reflections
183 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.78 e Å^{-3}
$\Delta\rho_{min}$ = −0.30 e Å^{-3}

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

D—H⋯A	D—H	H⋯A	D⋯A	D—H⋯A
C14—H14⋯O4	0.93	2.56	3.476 (3)	168
C9—H9⋯O2	0.93	2.51	3.425 (4)	167
O3—H3A⋯N1^{i}	0.82	1.78	2.595 (3)	170
O2—H2⋯N2^{ii}	0.82	1.84	2.642 (3)	167

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

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT* (Bruker, 2000); 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*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: AT2847).

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supporting information

Acta Cryst. (2009). E**65**, o2016 [doi:10.1107/S1600536809029237]

Butane-1,2,3,4-tetracarboxylic acid–4,4′-bipyridine (1/2)

Ning Zhang, Xue-Min Shi, Min Shao and Ming-Xing Li

S1. Comment

Organic cocrystals involving hydrogen-bonding (Table 1, Fig. 2) and *π*-*π* stacking [between the (N1/C5–C9) pyridine rings of 4,4′-bipyridine group, centroid-to-centroid distance = 3.8459 (19) Å] interactions are important in the areas of supramolecular chemistry, crystal engineering, and biological recognition (Wang *et al.*, 2007). Many organic cocrystals have been assembled from N-heterocycle and polycarboxylic acids (Li *et al.*, 2007). 4,4′-Bipyridine (bipy) and butane-1,2,3,4-tetracarboxylic acid (H_4butca) are important organic ligands in preparing metal complexes.

In our course of preparing ternary complexes containing butane-1,2,3,4-tetracarboxylic acid and 4,4′-bipyridine, a new 1:2 cocrystal compound of H_4butca.2bipy was prepared unexpectedly. Previously, a 1:1cocrystal of H_4butca.bipy has been synthesized by solution reaction (Najafpour, *et al.*, 2008). Herein we report the supramolecular framework of the title compound (I) (Fig. 1). The whole molecule has an inversion symmetry which lies on the midpoint of the C3—C3Aa bond of the butane-1,2,3,4-tetracarboxylic acid moiety.

S2. Experimental

A mixture of H_4butca (0.0468 g, 0.2 mmol), 4,4′-bipyridine (0.0468 g, 0.30 mmol), $Mn(SO_4)_2.H_2O$ (0.0510 g, 0.3 mmol) and H_2O (10 ml) was sealed in a 15 ml Teflon-lined stainless-steel reactor, which was heated at 413 K for 72 h. The reaction mixture was cooled to room temperature at a rate of 10 K h^{-1}. Light yellow block crystals suitable for X-ray diffraction were obtained in 56% yield (0.0273 g, based on H_4butca). Analysis calculated for $C_{28}H_{26}N_4O_8$ (%): C 61.53, H 4.795, N 10.25. Found: C 61.16, H 4.622, N 10.23. IR (KBr pellet, cm^{-1}): 3093w, 3040w, 2921w, 1711 s, 1601 s, 1539*m*, 1498*m*, 1414 s,1396 s, 1365*m*, 1285 s, 1217 s, 1128 s, 1071 s, 1005 s, 882*m*, 816 s, 626 s, 534*m*.

S3. Refinement

All H atoms were located geometrically, with C—H distances of 0.93 - 0.98 Å, O—H distances of 0.82 Å, and allowed to ride on their respective parent atoms with the constraint $U_{iso}(H) = 1.2U_{eq}(C)$ and $U_{iso}(H) = 1.5U_{eq}(O)$.

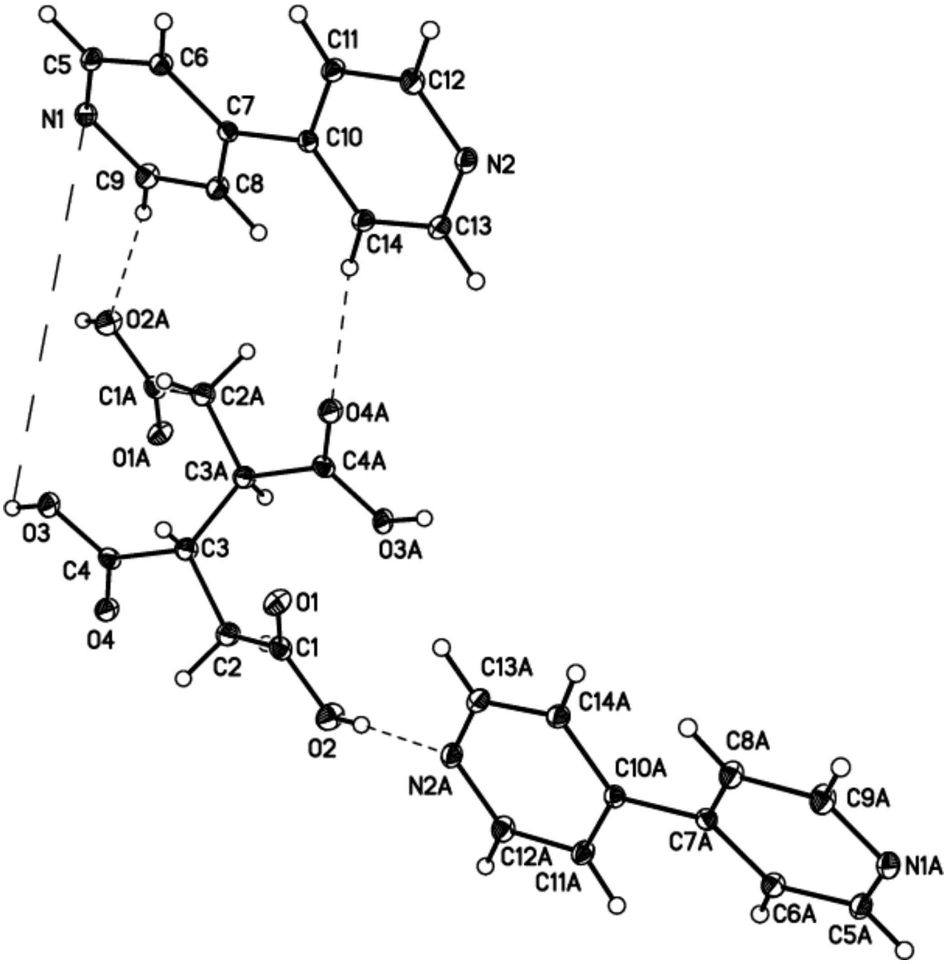

Figure 1

View of (I), showing displacement ellipsoids drawn at the 30% probability level.

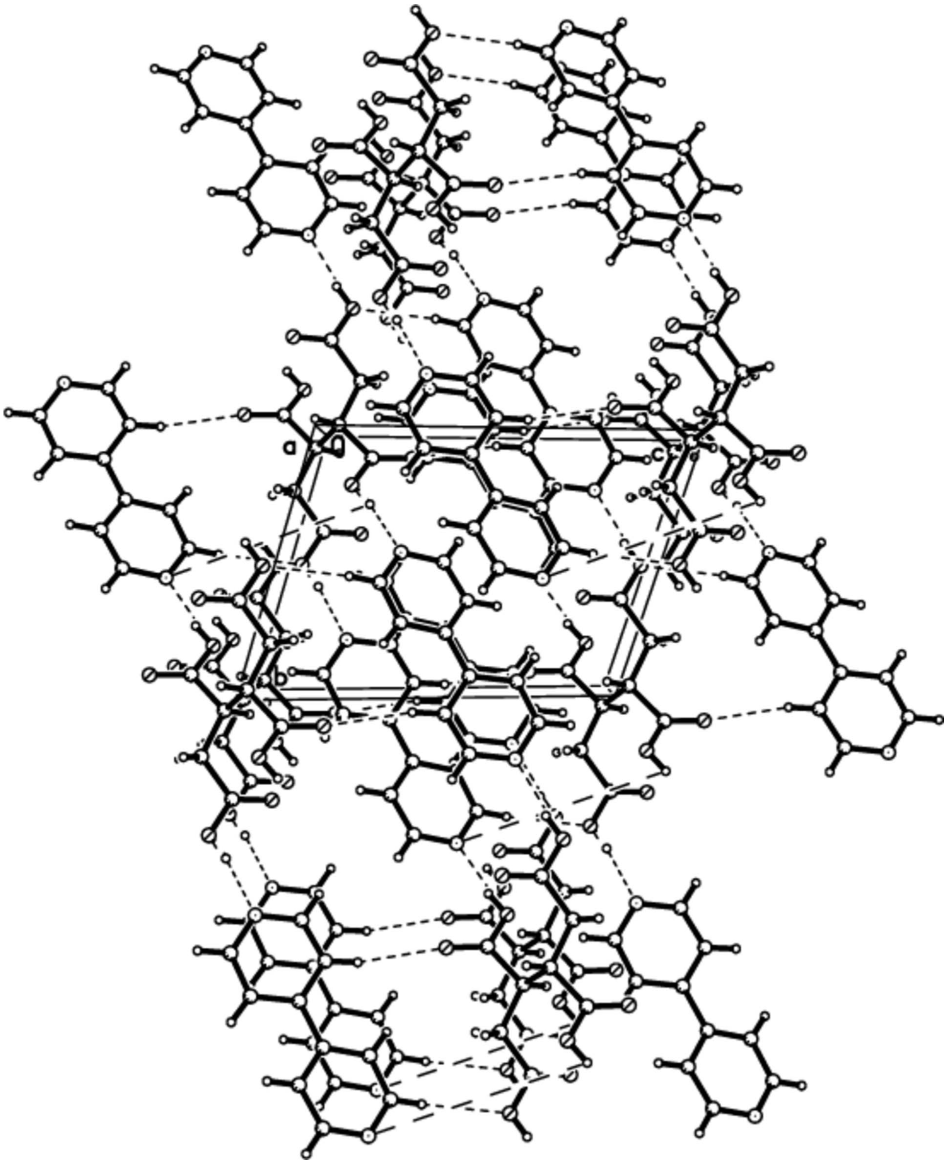

Figure 2

Crystal packing diagram of compound (I)

Butane-1,2,3,4-tetracarboxylic acid–4,4′-bipyridine (1/2)

Crystal data

$C_8H_{10}O_8 \cdot 2C_{10}H_8N_2$
$M_r = 546.53$
Triclinic, $P\overline{1}$
Hall symbol: -P 1
$a = 7.4435$ (11) Å
$b = 8.6990$ (13) Å
$c = 11.438$ (2) Å
$\alpha = 99.819$ (3)°
$\beta = 105.662$ (3)°
$\gamma = 111.361$ (2)°
$V = 633.57$ (17) Å^3
$Z = 1$
$F(000) = 286$
$D_x = 1.432$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 1387 reflections
$\theta = 2.6$–$26.6°$
$\mu = 0.11$ mm^{-1}
$T = 296$ K
Block, colourless
$0.30 \times 0.30 \times 0.30$ mm

Data collection

Bruker SMART APEXII CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{\min} = 0.969$, $T_{\max} = 0.969$
3287 measured reflections
2196 independent reflections
1721 reflections with $I > 2\sigma(I)$
$R_{\text{int}} = 0.013$
$\theta_{\max} = 25.0°$, $\theta_{\min} = 2.6°$
$h = -8 \rightarrow 8$
$k = -9 \rightarrow 10$
$l = -13 \rightarrow 9$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.061$
$wR(F^2) = 0.176$
$S = 1.07$
2196 reflections
183 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
$w = 1/[\sigma^2(F_o^2) + (0.0797P)^2 + 0.4629P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.78$ e Å^{-3}
$\Delta\rho_{\min} = -0.30$ 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}
C1	−0.1342 (5)	0.6302 (4)	−0.0696 (3)	0.0508 (7)
C2	−0.0232 (5)	0.7892 (4)	0.0456 (3)	0.0597 (9)
H2A	−0.1212	0.7939	0.0858	0.072*
H2B	0.0844	0.7742	0.1058	0.072*
C3	0.0728 (5)	0.9587 (3)	0.0242 (3)	0.0548 (8)
H3	0.1316	0.9400	−0.0410	0.066*
C4	0.2542 (5)	1.0895 (4)	0.1447 (3)	0.0498 (7)
C5	−0.2914 (4)	0.4885 (4)	0.4266 (3)	0.0479 (7)
H5	−0.3917	0.4111	0.4485	0.057*
C6	−0.1373 (4)	0.6347 (4)	0.5207 (3)	0.0449 (7)
H6	−0.1346	0.6548	0.6038	0.054*
C7	0.0146 (4)	0.7522 (3)	0.4900 (2)	0.0368 (6)
C8	−0.0007 (4)	0.7138 (4)	0.3640 (3)	0.0490 (7)
H8	0.0963	0.7888	0.3387	0.059*
C9	−0.1600 (5)	0.5644 (4)	0.2767 (3)	0.0543 (8)
H9	−0.1671	0.5408	0.1928	0.065*

C10	0.1856 (4)	0.9112 (3)	0.5876 (2)	0.0373 (6)
C11	0.2302 (4)	0.9316 (4)	0.7163 (3)	0.0467 (7)
H11	0.1531	0.8442	0.7436	0.056*
C12	0.3888 (4)	1.0814 (4)	0.8037 (3)	0.0515 (7)
H12	0.4160	1.0920	0.8897	0.062*
C13	0.4643 (4)	1.1920 (4)	0.6489 (3)	0.0500 (7)
H13	0.5455	1.2815	0.6249	0.060*
C14	0.3093 (4)	1.0477 (4)	0.5543 (3)	0.0452 (7)
H14	0.2869	1.0409	0.4692	0.054*
N1	−0.3041 (4)	0.4522 (3)	0.3059 (2)	0.0486 (6)
N2	0.5053 (3)	1.2116 (3)	0.7720 (2)	0.0480 (6)
O1	−0.1175 (4)	0.6219 (3)	−0.1724 (2)	0.0646 (6)
O2	−0.2488 (4)	0.5023 (3)	−0.0413 (2)	0.0675 (7)
H2	−0.3115	0.4168	−0.1043	0.101*
O3	0.4001 (3)	1.1949 (3)	0.11635 (17)	0.0535 (6)
H3A	0.4895	1.2695	0.1816	0.080*
O4	0.2601 (3)	1.0897 (3)	0.25184 (19)	0.0617 (6)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0491 (16)	0.0413 (16)	0.0448 (17)	0.0165 (13)	0.0013 (13)	0.0039 (13)
C2	0.0589 (19)	0.0442 (17)	0.0560 (19)	0.0128 (15)	0.0062 (15)	0.0121 (14)
C3	0.0543 (17)	0.0355 (15)	0.0479 (17)	0.0079 (13)	−0.0013 (14)	0.0065 (12)
C4	0.0508 (17)	0.0385 (15)	0.0391 (16)	0.0143 (13)	−0.0039 (13)	0.0051 (12)
C5	0.0422 (15)	0.0415 (15)	0.0449 (16)	0.0070 (13)	0.0106 (12)	0.0105 (12)
C6	0.0477 (15)	0.0451 (15)	0.0353 (14)	0.0143 (13)	0.0142 (12)	0.0103 (12)
C7	0.0358 (13)	0.0365 (13)	0.0354 (13)	0.0156 (11)	0.0095 (11)	0.0098 (11)
C8	0.0465 (16)	0.0501 (16)	0.0375 (15)	0.0092 (13)	0.0148 (12)	0.0096 (12)
C9	0.0551 (17)	0.0537 (17)	0.0353 (15)	0.0106 (14)	0.0126 (13)	0.0038 (13)
C10	0.0348 (13)	0.0363 (13)	0.0370 (14)	0.0146 (11)	0.0102 (11)	0.0079 (11)
C11	0.0471 (15)	0.0432 (15)	0.0377 (14)	0.0086 (13)	0.0138 (12)	0.0100 (12)
C12	0.0498 (16)	0.0526 (17)	0.0360 (15)	0.0120 (14)	0.0100 (13)	0.0068 (13)
C13	0.0447 (16)	0.0410 (15)	0.0536 (17)	0.0087 (13)	0.0148 (13)	0.0159 (13)
C14	0.0453 (15)	0.0460 (15)	0.0395 (15)	0.0142 (13)	0.0145 (12)	0.0150 (12)
N1	0.0450 (13)	0.0419 (13)	0.0417 (14)	0.0105 (11)	0.0060 (10)	0.0056 (10)
N2	0.0398 (12)	0.0424 (13)	0.0469 (14)	0.0088 (10)	0.0098 (10)	0.0079 (10)
O1	0.0678 (15)	0.0481 (13)	0.0711 (16)	0.0150 (11)	0.0286 (12)	0.0185 (11)
O2	0.0788 (16)	0.0500 (13)	0.0436 (12)	0.0084 (12)	0.0114 (11)	0.0034 (10)
O3	0.0456 (11)	0.0456 (12)	0.0373 (11)	0.0028 (9)	−0.0005 (9)	−0.0005 (9)
O4	0.0599 (13)	0.0513 (13)	0.0424 (12)	0.0047 (10)	0.0019 (10)	0.0097 (9)

Geometric parameters (Å, º)

C1—O1	1.209 (4)	C7—C10	1.484 (4)
C1—O2	1.285 (4)	C8—C9	1.374 (4)
C1—C2	1.513 (4)	C8—H8	0.9300
C2—C3	1.482 (4)	C9—N1	1.325 (4)

C2—H2A	0.9700	C9—H9	0.9300
C2—H2B	0.9700	C10—C11	1.383 (4)
C3—C4	1.536 (4)	C10—C14	1.395 (4)
C3—C3^{i}	1.542 (6)	C11—C12	1.374 (4)
C3—H3	0.9800	C11—H11	0.9300
C4—O4	1.214 (3)	C12—N2	1.327 (4)
C4—O3	1.301 (4)	C12—H12	0.9300
C5—N1	1.332 (4)	C13—N2	1.324 (4)
C5—C6	1.377 (4)	C13—C14	1.373 (4)
C5—H5	0.9300	C13—H13	0.9300
C6—C7	1.393 (4)	C14—H14	0.9300
C6—H6	0.9300	O2—H2	0.8200
C7—C8	1.386 (4)	O3—H3A	0.8200
O1—C1—O2	124.5 (3)	C6—C7—C10	121.7 (2)
O1—C1—C2	126.0 (3)	C9—C8—C7	119.6 (3)
O2—C1—C2	109.5 (3)	C9—C8—H8	120.2
C3—C2—C1	117.3 (3)	C7—C8—H8	120.2
C3—C2—H2A	108.0	N1—C9—C8	123.5 (3)
C1—C2—H2A	108.0	N1—C9—H9	118.3
C3—C2—H2B	108.0	C8—C9—H9	118.3
C1—C2—H2B	108.0	C11—C10—C14	116.6 (2)
H2A—C2—H2B	107.2	C11—C10—C7	121.5 (2)
C2—C3—C4	110.8 (2)	C14—C10—C7	121.8 (2)
C2—C3—C3^{i}	116.5 (3)	C12—C11—C10	119.8 (3)
C4—C3—C3^{i}	108.9 (3)	C12—C11—H11	120.1
C2—C3—H3	106.7	C10—C11—H11	120.1
C4—C3—H3	106.7	N2—C12—C11	123.5 (3)
C3^{i}—C3—H3	106.7	N2—C12—H12	118.2
O4—C4—O3	125.2 (2)	C11—C12—H12	118.2
O4—C4—C3	123.6 (3)	N2—C13—C14	124.0 (3)
O3—C4—C3	111.2 (3)	N2—C13—H13	118.0
N1—C5—C6	123.4 (3)	C14—C13—H13	118.0
N1—C5—H5	118.3	C13—C14—C10	119.2 (3)
C6—C5—H5	118.3	C13—C14—H14	120.4
C5—C6—C7	119.2 (2)	C10—C14—H14	120.4
C5—C6—H6	120.4	C9—N1—C5	117.3 (2)
C7—C6—H6	120.4	C13—N2—C12	116.9 (2)
C8—C7—C6	117.0 (2)	C1—O2—H2	109.5
C8—C7—C10	121.3 (2)	C4—O3—H3A	109.5

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

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C14—H14···O4	0.93	2.56	3.476 (3)	168
C9—H9···O2	0.93	2.51	3.425 (4)	167

O3—H3*A*···N1^{ii}	0.82	1.78	2.595 (3)	170
O2—H2···N2^{iii}	0.82	1.84	2.642 (3)	167

Symmetry codes: (ii) *x*+1, *y*+1, *z*; (iii) *x*−1, *y*−1, *z*−1.