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2,2′-Bipyridine–cyclopentane-1,2,3,4-tetracarboxylic acid (1/1)

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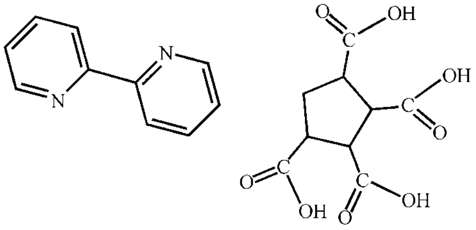
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.002 Å; R factor = 0.038; wR factor = 0.089; data-to-parameter ratio = 14.6.

The asymmetric unit of the title compound, $C_{10}H_8N_2\cdot C_9H_{10}O_8$, contains a half-molecule of 2,2′-bipyridine and a half-molecule of 1,2,3,4-cyclopentanetetracarboxylic acid, both components being completed by crystallographic inversion symmetry. In the crystal, the molecules are assembled into chains extending along [010] by O—H···N hydrogen bonds; adjacent chains are linked by O—H···O hydrogen bonds into a three-dimensional network.

Related literature

For general background to coordination polymers, see: Bowers *et al.* (2005); Bowes *et al.* (2003). For related structures, see: Chen *et al.* (2005).



Experimental

Crystal data

$C_{10}H_8N_2\cdot C_9H_{10}O_8$
M_r = 402.35
Orthorhombic, *Pnma*
a = 12.942 (3) Å
b = 25.118 (5) Å
c = 5.4353 (11) Å
V = 1766.8 (6) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.12 mm^{-1}
T = 293 K
0.44 × 0.36 × 0.27 mm

Data collection

Rigaku R-AXIS RAPID diffractometer
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
$T_{\min}$ = 0.949, $T_{\max}$ = 0.968
15967 measured reflections
2054 independent reflections
1499 reflections with $I > 2\sigma(I)$
$R_{\rm int}$ = 0.055

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.038
$wR(F^2)$ = 0.089
S = 1.02
2054 reflections
141 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{\max}$ = 0.19 e Å^{-3}
$\Delta\rho_{\min}$ = −0.16 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O2—H2B···O3$^{\rm i}$	0.87 (2)	1.80 (2)	2.6520 (16)	165 (2)
O4—H4A···N1$^{\rm ii}$	0.94 (2)	1.80 (2)	2.7335 (18)	169 (2)

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

Data collection: *RAPID-AUTO* (Rigaku, 1998); cell refinement: *RAPID-AUTO*; data reduction: *CrystalStructure* (Rigaku/MSC, 2004); 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: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: JH2304).

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supplementary materials

Acta Cryst. (2011). E**67**, o1945 [**doi:10.1107/S1600536811025827**]

2,2'-Bipyridine-cyclopentane-1,2,3,4-tetracarboxylic acid (1/1)

J.-L. Lin, X.-X. Guo and **W.-X. Huang**

Comment

In hydrogen-bonded adducts of simple di–, tri– and tetracarboxylic acids with tertiary diamines, the modes of hydrogen-bonded chains are often determined by hard and soft types. Our investigation builds on the associative behavior of carboxyl and pyridine functions. In this contribution, we report the title compound with bipyridine–cyclopentanetetracarboxylic acid cocrystal.

The asymmetric unit contains one 2,2'-bipyridine molecule and one 1,2,3,4–cyclopentanetetracarboxylic acid molecule. Both bipyridine and cyclopentanetetracarboxylic acid molecules are generated *via* crystallographic 2–fold rotation axes (Fig. 1), and C3 atoms are located at the Wyckoff 4c sites. The carboxylic groups of cyclopentanetetracarboxylic acid connect with the corresponding bipyridine molecules through O4–H4A···N1^{ii} hydrogen bonds generating a one-dimensional chain along [010] (Fig. 2). In this way, the adjacent one-dimensional chains are interconnected by O2–H2B···O3^{i} hydrogen bonds to give three-dimensional network parallel to (001) (Fig. 3).

Experimental

Under continuous stirring, a solution of 2,2'-bipyridine (0.1560 g, 1.00 mmol) in 10 ml CH_3OH was added dropwise to an aqueous solution of 1,2,3,4–cyclopentanetetracarboxylic acid (0.1230 g, 0.50 mmol) in 10 ml H_2O. The resulting mixture was further stirred for *ca* 30 min. After slow evaporation of the solution for one week at 35°C, colorless pillar sized crystals were obtained.

Refinement

H atoms bonded to C atoms were palced in geometrically calculated position and were refined using a riding model, with $U_{iso}(H) = 1.2 U_{eq}(C)$. H atoms attached to O atoms were found in a difference Fourier synthesis and were refined using a riding model, with the O—H distances fixed as initially found and with $U_{iso}(H)$ values set at 1.2 Ueq(O).

Figures

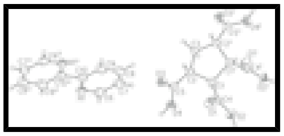

Fig. 1. *ORTEP* view of the title compound, The dispalcement ellipsoids are drawn at 45% probability dispalcement ellipsoids. [Symmetry codes: (i)*x*, –y+1/2, *z*;(ii)–x, *y* + 1, –z.]

Fig. 2. one-dimensional chain of the title cocrystal viewed along the *b* axis. O–H···N hydrogen bonds are shown as dashed lines.

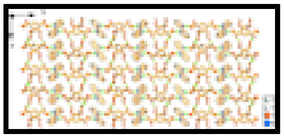

Fig. 3. Packing diagram of the title co-crystal viewed down the *c* axis. O–H···N and O–H···O hydrogen bonds are shown as dashed lines.

2,2'-Bipyridine–cyclopentane-1,2,3,4-tetracarboxylic acid (1/1)

Crystal data

$C_{10}H_8N_2 \cdot C_9H_{10}O_8$

$M_r = 402.35$

Orthorhombic, *Pnma*

Hall symbol: -P 2ac 2n

$a = 12.942\ (3)$ Å

$b = 25.118\ (5)$ Å

$c = 5.4353\ (11)$ Å

$V = 1766.8\ (6)$ Å^3

$Z = 4$

$F(000) = 840$

$D_x = 1.513$ Mg m^{-3}

Mo *K*α radiation, λ = 0.71073 Å

Cell parameters from 8201 reflections

θ = 3.2–27.5°

μ = 0.12 mm^{-1}

$T = 293$ K

Block, white

0.44 × 0.36 × 0.27 mm

Data collection

Rigaku R-AXIS RAPID diffractometer

Radiation source: fine-focus sealed tube

graphite

Detector resolution: 0 pixels mm^{-1}

ω scans

Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)

$T_{min} = 0.949$, $T_{max} = 0.968$

15967 measured reflections

2054 independent reflections

1499 reflections with $I > 2\sigma(I)$

$R_{int} = 0.055$

$\theta_{max} = 27.4°$, $\theta_{min} = 3.2°$

$h = -16 \rightarrow 16$

$k = -32 \rightarrow 32$

$l = -7 \rightarrow 7$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.038$

$wR(F^2) = 0.089$

$S = 1.02$

2054 reflections

141 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 atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.034P)^2 + 0.4998P]$ where $P = (F_o^2 + 2F_c^2)/3$

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

$\Delta\rho_{max} = 0.19$ e Å^{-3}

$\Delta\rho_{min} = -0.16$ 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	$U_{\rm iso}$*/$U_{\rm eq}$
C1	0.51833 (9)	0.28149 (5)	0.1032 (3)	0.0282 (3)
H1A	0.5337	0.2944	0.2694	0.034*
C2	0.40596 (10)	0.29744 (5)	0.0333 (3)	0.0311 (3)
H2A	0.3672	0.3004	0.1875	0.037*
C3	0.36186 (15)	0.2500	−0.1051 (4)	0.0354 (5)
H3A	0.3842	0.2500	−0.2754	0.043*
H3B	0.2869	0.2500	−0.0997	0.043*
O1	0.58022 (9)	0.31419 (5)	−0.2864 (2)	0.0560 (4)
O2	0.68873 (8)	0.30931 (4)	0.0286 (2)	0.0429 (3)
C4	0.59708 (10)	0.30368 (5)	−0.0745 (3)	0.0313 (3)
O3	0.33802 (8)	0.35651 (4)	−0.2754 (3)	0.0535 (4)
O4	0.44673 (9)	0.38926 (4)	0.0007 (2)	0.0438 (3)
C5	0.39410 (10)	0.34981 (6)	−0.0985 (3)	0.0341 (3)
N1	0.07695 (9)	0.51473 (5)	0.2707 (2)	0.0392 (3)
C6	0.14948 (12)	0.49161 (7)	0.4103 (3)	0.0456 (4)
H6A	0.1737	0.5103	0.5463	0.055*
C7	0.19052 (12)	0.44202 (7)	0.3654 (3)	0.0453 (4)
H7A	0.2406	0.4276	0.4683	0.054*
C8	0.15516 (13)	0.41452 (7)	0.1634 (3)	0.0448 (4)
H8A	0.1816	0.3811	0.1258	0.054*
C9	0.08006 (13)	0.43719 (6)	0.0176 (3)	0.0430 (4)
H9A	0.0553	0.4190	−0.1193	0.052*
C10	0.04132 (11)	0.48714 (6)	0.0747 (3)	0.0357 (3)
H2B	0.7334 (16)	0.3216 (8)	−0.077 (4)	0.074 (7)*
H4A	0.4380 (18)	0.4198 (10)	−0.098 (5)	0.095 (8)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0233 (6)	0.0356 (7)	0.0258 (7)	−0.0014 (5)	0.0007 (6)	−0.0040 (6)
C2	0.0225 (6)	0.0321 (7)	0.0389 (8)	−0.0010 (5)	0.0033 (6)	−0.0016 (6)
C3	0.0260 (9)	0.0286 (10)	0.0517 (14)	0.000	−0.0046 (10)	0.000

O1	0.0469 (7)	0.0863 (10)	0.0350 (7)	−0.0119 (6)	0.0054 (5)	0.0090 (6)
O2	0.0249 (5)	0.0494 (7)	0.0544 (8)	−0.0087 (5)	0.0019 (5)	0.0014 (6)
C4	0.0279 (7)	0.0290 (7)	0.0371 (9)	−0.0010 (6)	0.0046 (6)	−0.0054 (6)
O3	0.0430 (6)	0.0380 (6)	0.0796 (9)	−0.0087 (5)	−0.0285 (6)	0.0102 (6)
O4	0.0467 (6)	0.0340 (6)	0.0508 (7)	−0.0104 (5)	−0.0073 (5)	−0.0026 (5)
C5	0.0236 (6)	0.0311 (7)	0.0475 (9)	−0.0015 (6)	0.0004 (7)	−0.0026 (7)
N1	0.0387 (7)	0.0378 (7)	0.0411 (8)	−0.0084 (6)	−0.0029 (6)	0.0029 (6)
C6	0.0430 (8)	0.0494 (9)	0.0443 (10)	−0.0126 (8)	−0.0101 (8)	0.0036 (8)
C7	0.0390 (8)	0.0458 (9)	0.0510 (11)	−0.0051 (7)	−0.0069 (8)	0.0125 (8)
C8	0.0467 (9)	0.0394 (9)	0.0483 (10)	−0.0018 (7)	0.0023 (8)	0.0083 (8)
C9	0.0507 (9)	0.0386 (8)	0.0396 (9)	−0.0046 (7)	−0.0054 (8)	0.0021 (7)
C10	0.0375 (7)	0.0353 (8)	0.0343 (8)	−0.0093 (6)	0.0013 (7)	0.0057 (7)

Geometric parameters (Å, °)

C1—C4	1.5105 (19)	O4—C5	1.3177 (17)
C1—C2	1.5555 (18)	O4—H4A	0.94 (2)
C1—C1^{i}	1.582 (3)	N1—C6	1.339 (2)
C1—H1A	0.9800	N1—C10	1.352 (2)
C2—C5	1.506 (2)	C6—C7	1.376 (2)
C2—C3	1.5202 (19)	C6—H6A	0.9300
C2—H2A	0.9800	C7—C8	1.375 (2)
C3—C2^{i}	1.5202 (19)	C7—H7A	0.9300
C3—H3A	0.9700	C8—C9	1.377 (2)
C3—H3B	0.9700	C8—H8A	0.9300
O1—C4	1.2015 (19)	C9—C10	1.386 (2)
O2—C4	1.3194 (17)	C9—H9A	0.9300
O2—H2B	0.87 (2)	C10—C10^{ii}	1.490 (3)
O3—C5	1.2166 (19)		
C4—C1—C2	112.31 (12)	O2—C4—C1	111.99 (13)
C4—C1—C1^{i}	111.65 (7)	C5—O4—H4A	108.6 (14)
C2—C1—C1^{i}	104.92 (7)	O3—C5—O4	121.84 (14)
C4—C1—H1A	109.3	O3—C5—C2	123.90 (13)
C2—C1—H1A	109.3	O4—C5—C2	114.19 (13)
C1^{i}—C1—H1A	109.3	C6—N1—C10	117.60 (14)
C5—C2—C3	114.28 (13)	N1—C6—C7	124.23 (16)
C5—C2—C1	115.88 (11)	N1—C6—H6A	117.9
C3—C2—C1	105.67 (12)	C7—C6—H6A	117.9
C5—C2—H2A	106.8	C8—C7—C6	117.89 (16)
C3—C2—H2A	106.8	C8—C7—H7A	121.1
C1—C2—H2A	106.8	C6—C7—H7A	121.1
C2—C3—C2^{i}	103.21 (17)	C7—C8—C9	119.11 (16)
C2—C3—H3A	111.1	C7—C8—H8A	120.4
C2^{i}—C3—H3A	111.1	C9—C8—H8A	120.4
C2—C3—H3B	111.1	C8—C9—C10	120.02 (16)
C2^{i}—C3—H3B	111.1	C8—C9—H9A	120.0
H3A—C3—H3B	109.1	C10—C9—H9A	120.0

C4—O2—H2B	110.7 (14)	N1—C10—C9	121.14 (14)
O1—C4—O2	123.15 (14)	N1—C10—C10^{ii}	116.88 (17)
O1—C4—C1	124.84 (13)	C9—C10—C10^{ii}	121.98 (18)

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

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O2—H2B···O3^{iii}	0.87 (2)	1.80 (2)	2.6520 (16)	165 (2)
O4—H4A···N1^{iv}	0.94 (2)	1.80 (2)	2.7335 (18)	169 (2)

Symmetry codes: (iii) $x+1/2$, y, $-z-1/2$; (iv) $-x+1/2$, $-y+1$, $z-1/2$.

Fig. 1

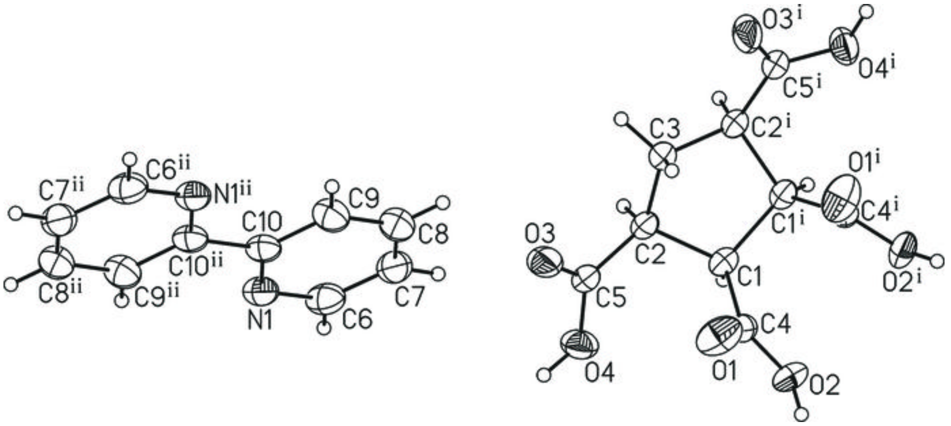
$C7^{ii}$
$C6^{ii}$
$N1^{ii}$
C10
C9
C8
$C10^{ii}$
$C8^{ii}$
$C9^{ii}$
N1
C6
C7
$O3^{i}$
$O4^{i}$
$C5^{i}$
C3
$C2^{i}$
$O1^{i}$
$C1^{i}$
$C4^{i}$
O3
C2
C1
C5
$O2^{i}$
C4
O4
O1
O2

Fig. 2

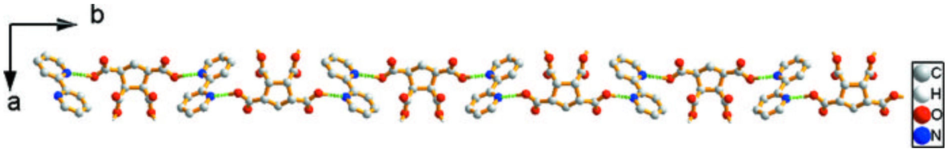

Fig. 3