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4,4′-Bipyridine acetic acid disolvate

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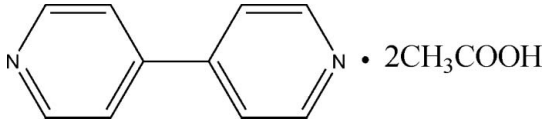
Key indicators: single-crystal X-ray study; T = 291 K; mean σ(C–C) = 0.006 Å; R factor = 0.050; wR factor = 0.119; data-to-parameter ratio = 8.6.

The crystal structure of the title compound, $C_{10}H_8N_2{\cdot}2C_2H_4O_2$, is built up from 4,4′-bipyridine and acetic acid molecules linked by strong O—H···N hydrogen bonds. The 4,4′-bipyridine and the two acetic acid molecules are further connected through weak C—H···O hydrogen bonds to form a supramolecular two-dimensional network parallel to the (001) plane. The two pyridine rings make a dihedral angle of 31.8 (1)°.

Related literature

For related literature, see: Dai *et al.* (2005); Li *et al.* (2005); Pedireddi *et al.* (1998); Wang *et al.* (2006). For structural analysis, see: Spek (2003).



Experimental

Crystal data

$C_{10}H_8N_2{\cdot}2C_2H_4O_2$
M_r = 276.29
Monoclinic, Pc
a = 3.893 (2) Å
b = 8.181 (5) Å
c = 22.563 (15) Å
β = 98.46 (3)°
V = 710.7 (7) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 291 (2) K
0.15 × 0.13 × 0.12 mm

Data collection

Rigaku R-AXIS RAPID diffractometer
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
T_{min} = 0.986, T_{max} = 0.988
6467 measured reflections
1595 independent reflections
995 reflections with $I > 2\sigma(I)$
R_{int} = 0.042

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.050
$wR(F^2)$ = 0.119
S = 1.04
1595 reflections
185 parameters
2 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.15 e Å^{-3}
$\Delta\rho_{min}$ = −0.13 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O2—H2A···N2	0.82	1.86	2.675 (5)	175
O4—H4A···N1	0.82	1.84	2.659 (5)	173
C7—H7···O1^{i}	0.93	2.62	3.526 (5)	165
C10—H10···O3^{ii}	0.93	2.37	3.273 (5)	164
C4—H4···O3^{iii}	0.93	2.56	3.397 (6)	150

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

Data collection: *RAPID-AUTO* (Rigaku, 1998); cell refinement: *RAPID-AUTO*; data reduction: *CrystalStructure* (Rigaku/MSC, 2002); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEPIII* (Burnett & Johnson, 1996); *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: DN2279).

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supporting information

Acta Cryst. (2008). E**64**, o46 [https://doi.org/10.1107/S1600536807062319]

4,4′-Bipyridine acetic acid disolvate

Ling Ye

S1. Comment

2,2-bipyridine is widely used to build up supramolecular network with carboxylic acid (Dai *et al.*, 2005; Li *et al.*, 2005; Pedireddi *et al.*, 1998; Wang *et al.*, 2006). Herein, we report the co-crystal structure of 2,2-bipyridine and acetic acid.

The asymmetric unit of (I) contains one 4,4-bipyridine molecule and two acetic acid molecules linked trough strong O—H···O hydogen bonds (Fig. 1). The two pyridine rings are both planar, with a RMS deviation of fitted atoms being 0.0033 Å and 0.0074 Å, respectively. The dihedral angle between them is 31.8 (1) °.

The 4,4-bipyridine and the two acetic acid molecules are further connected through C—H···O weak hydrogen bonds (*PLATON*, Spek, 2003) involving the carboxyl oxygen atoms (Table 1) to build up a supramolecular two dimensionnal network.parallel to the (0 0 1) plane (Fig. 2).

S2. Experimental

A mixture of 2,2-bipyridine (5 mmol, 0.78 g) and acetic acid (10 mmol, 0.60 g) in water (10 ml) was stirred for 2 h, and filtrate was allowed to evaporate at room temperature. Colorless single crystals of the title compound were formed after two weeks.

S3. Refinement

All H atoms attached to C atoms and O atom were fixed geometrically and treated as riding with C—H = 0.93 Å (aromatic) or 0.96 Å (methyl) and O—H = 0.82 Å with U_{iso}(H) = 1.2U_{eq}(C$_{aromatic}$ or O) or U_{iso}(H) = 1.5U_{eq}(Cmethyl).

In the absence of significant anomalous scattering, the absolute structure could not be reliably determined and then the Friedel pairs were merged and any references to the Flack parameter were removed.

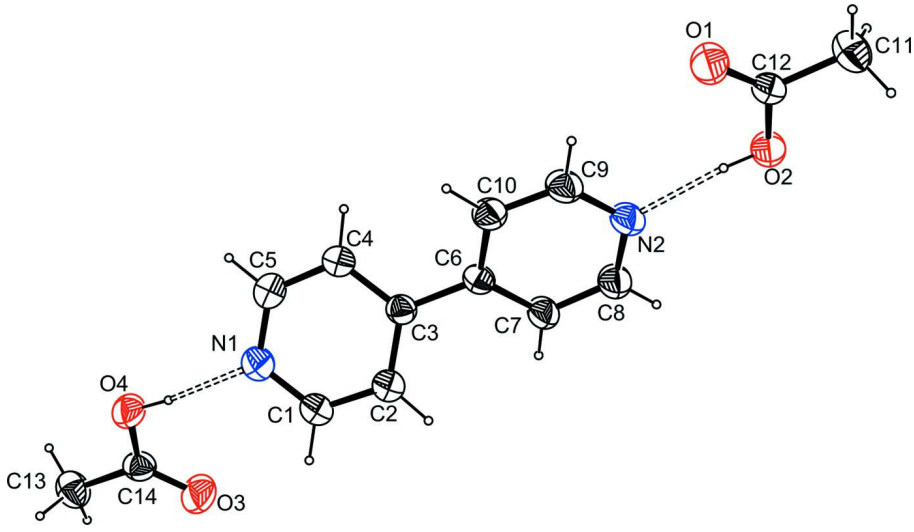

Figure 1

The asymmetric unit, with the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level. Hydrogen bonds are shown as dashed lines. H atoms are represented as small spheres of arbitrary radii.

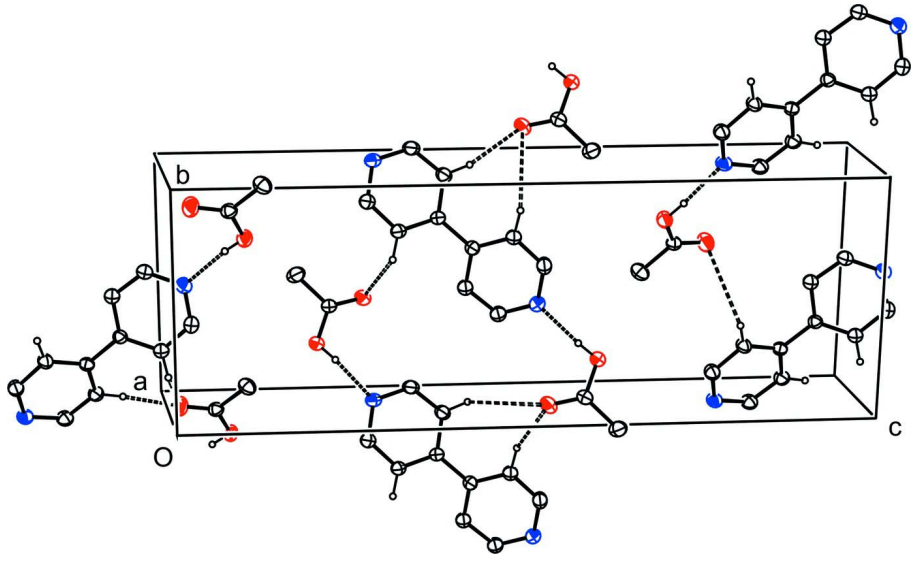

Figure 2

Packing view showing the Hydrogen bonding network. H atoms not involved in hydrogen bonds have been omitted for clarity.

4,4-Bipyridine acetic acid disolvate

Crystal data

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$c = 22.563\ (15)$ Å
$\beta = 98.46\ (3)°$
$V = 710.7\ (7)$ Å^3
$Z = 2$
$F(000) = 292$
$D_x = 1.291$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 4188 reflections

θ = 3.1–27.5°
μ = 0.10 mm^{-1}
T = 291 K

Block, colorless
0.15 × 0.13 × 0.12 mm

Data collection

Rigaku RAXIS-RAPID diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω scans
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
T_{min} = 0.986, T_{max} = 0.988

6467 measured reflections
1595 independent reflections
995 reflections with $I > 2\sigma(I)$
R_{int} = 0.042
θ_{max} = 27.5°, θ_{min} = 3.1°
h = −5→5
k = −10→10
l = −29→24

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.050
$wR(F^2)$ = 0.119
S = 1.04
1595 reflections
185 parameters
2 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
$w = 1/[\sigma^2(F_o^2) + (0.0545P)^2 + 0.0575P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.003
$\Delta\rho_{max}$ = 0.15 e Å^{-3}
$\Delta\rho_{min}$ = −0.13 e Å^{-3}

Special details

Experimental. (See detailed section in the paper)

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}
C13	−0.0181 (13)	−0.0149 (6)	0.6249 (2)	0.0728 (12)
H13A	0.1830	−0.0660	0.6470	0.109*
H13B	−0.1649	−0.0968	0.6039	0.109*
H13C	−0.1437	0.0423	0.6521	0.109*
C14	0.0919 (10)	0.1019 (5)	0.5814 (2)	0.0568 (10)
C1	0.4437 (12)	0.3978 (5)	0.47047 (19)	0.0697 (11)
H1	0.4572	0.2861	0.4636	0.084*
C2	0.4896 (13)	0.5031 (5)	0.4247 (2)	0.0640 (10)
H2	0.5306	0.4622	0.3879	0.077*
C3	0.4742 (9)	0.6695 (4)	0.43371 (16)	0.0488 (9)
C4	0.4131 (11)	0.7207 (5)	0.48979 (19)	0.0613 (11)
H4	0.4020	0.8316	0.4984	0.074*

C5	0.3695 (12)	0.6071 (5)	0.5321 (2)	0.0690 (12)
H5	0.3285	0.6441	0.5694	0.083*
C6	0.5176 (9)	0.7891 (4)	0.38616 (17)	0.0483 (9)
C7	0.4150 (11)	0.7538 (5)	0.32659 (19)	0.0599 (11)
H7	0.3218	0.6520	0.3152	0.072*
C8	0.4513 (11)	0.8706 (5)	0.2839 (2)	0.0654 (11)
H8	0.3770	0.8449	0.2440	0.078*
C9	0.6937 (12)	1.0494 (5)	0.35465 (19)	0.0649 (11)
H9	0.7942	1.1507	0.3646	0.078*
C10	0.6649 (11)	0.9422 (5)	0.39995 (18)	0.0599 (10)
H10	0.7426	0.9709	0.4395	0.072*
N1	0.3818 (10)	0.4468 (4)	0.52352 (16)	0.0655 (9)
O1	0.9176 (9)	1.3967 (4)	0.29266 (15)	0.0859 (11)
O3	0.0627 (10)	0.0786 (4)	0.52832 (15)	0.0938 (11)
C11	0.8647 (13)	1.5081 (6)	0.1948 (2)	0.0764 (14)
H11A	1.0155	1.5926	0.2131	0.115*
H11B	0.6438	1.5545	0.1789	0.115*
H11C	0.9664	1.4585	0.1630	0.115*
C12	0.8156 (11)	1.3817 (4)	0.2405 (2)	0.0565 (10)
N2	0.5861 (9)	1.0176 (4)	0.29663 (16)	0.0631 (9)
O2	0.6441 (8)	1.2545 (3)	0.21698 (13)	0.0698 (8)
H2A	0.6294	1.1864	0.2432	0.105*
O4	0.2338 (8)	0.2354 (3)	0.60567 (13)	0.0711 (9)
H4A	0.2902	0.2947	0.5794	0.107*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C13	0.074 (3)	0.066 (3)	0.075 (3)	−0.009 (2)	0.001 (2)	0.010 (2)
C14	0.056 (2)	0.051 (2)	0.061 (3)	0.0012 (18)	0.0008 (19)	−0.005 (2)
C1	0.097 (3)	0.050 (2)	0.063 (3)	0.005 (2)	0.013 (2)	0.004 (2)
C2	0.082 (3)	0.053 (2)	0.057 (2)	0.002 (2)	0.0112 (19)	0.000 (2)
C3	0.049 (2)	0.0470 (19)	0.049 (2)	0.0035 (17)	0.0013 (16)	0.0002 (17)
C4	0.078 (3)	0.048 (2)	0.058 (3)	0.0023 (19)	0.012 (2)	0.0001 (19)
C5	0.083 (3)	0.070 (3)	0.054 (2)	0.003 (2)	0.013 (2)	−0.001 (2)
C6	0.0467 (19)	0.0427 (18)	0.055 (2)	−0.0021 (16)	0.0050 (16)	0.0016 (17)
C7	0.075 (3)	0.048 (2)	0.054 (2)	−0.0082 (19)	0.003 (2)	0.002 (2)
C8	0.080 (3)	0.056 (2)	0.058 (2)	−0.009 (2)	0.004 (2)	0.000 (2)
C9	0.075 (3)	0.048 (2)	0.070 (3)	−0.0104 (19)	0.003 (2)	−0.002 (2)
C10	0.069 (3)	0.051 (2)	0.056 (2)	−0.0030 (19)	−0.001 (2)	−0.0051 (19)
N1	0.077 (2)	0.058 (2)	0.062 (2)	−0.0027 (17)	0.0123 (17)	0.0040 (18)
O1	0.119 (3)	0.072 (2)	0.062 (2)	−0.0298 (19)	−0.0012 (19)	−0.0039 (17)
O3	0.143 (3)	0.076 (2)	0.059 (2)	−0.021 (2)	0.002 (2)	−0.0126 (18)
C11	0.079 (3)	0.067 (3)	0.083 (4)	−0.012 (2)	0.008 (2)	0.015 (2)
C12	0.067 (3)	0.044 (2)	0.059 (3)	−0.0042 (19)	0.010 (2)	0.000 (2)
N2	0.076 (2)	0.0469 (19)	0.065 (2)	−0.0092 (16)	0.0076 (17)	0.0014 (17)
O2	0.096 (2)	0.0558 (17)	0.0559 (18)	−0.0206 (16)	0.0061 (16)	−0.0045 (13)
O4	0.098 (2)	0.0591 (18)	0.0567 (19)	−0.0146 (16)	0.0117 (16)	−0.0058 (14)

Geometric parameters (Å, º)

C13—C14	1.478 (6)	C6—C10	1.393 (5)
C13—H13A	0.9600	C7—C8	1.378 (6)
C13—H13B	0.9600	C7—H7	0.9300
C13—H13C	0.9600	C8—N2	1.326 (5)
C14—O3	1.201 (5)	C8—H8	0.9300
C14—O4	1.307 (5)	C9—N2	1.340 (5)
C1—N1	1.317 (6)	C9—C10	1.364 (6)
C1—C2	1.377 (6)	C9—H9	0.9300
C1—H1	0.9300	C10—H10	0.9300
C2—C3	1.380 (5)	O1—C12	1.191 (5)
C2—H2	0.9300	C11—C12	1.493 (6)
C3—C4	1.386 (6)	C11—H11A	0.9600
C3—C6	1.480 (5)	C11—H11B	0.9600
C4—C5	1.361 (6)	C11—H11C	0.9600
C4—H4	0.9300	C12—O2	1.306 (4)
C5—N1	1.328 (5)	O2—H2A	0.8200
C5—H5	0.9300	O4—H4A	0.8200
C6—C7	1.375 (5)		
C14—C13—H13A	109.5	C10—C6—C3	121.3 (3)
C14—C13—H13B	109.5	C6—C7—C8	119.5 (4)
H13A—C13—H13B	109.5	C6—C7—H7	120.3
C14—C13—H13C	109.5	C8—C7—H7	120.3
H13A—C13—H13C	109.5	N2—C8—C7	123.8 (4)
H13B—C13—H13C	109.5	N2—C8—H8	118.1
O3—C14—O4	121.5 (4)	C7—C8—H8	118.1
O3—C14—C13	124.4 (4)	N2—C9—C10	124.0 (4)
O4—C14—C13	114.0 (4)	N2—C9—H9	118.0
N1—C1—C2	123.6 (4)	C10—C9—H9	118.0
N1—C1—H1	118.2	C9—C10—C6	119.2 (4)
C2—C1—H1	118.2	C9—C10—H10	120.4
C1—C2—C3	119.5 (4)	C6—C10—H10	120.4
C1—C2—H2	120.2	C1—N1—C5	116.6 (4)
C3—C2—H2	120.2	C12—C11—H11A	109.5
C2—C3—C4	116.8 (4)	C12—C11—H11B	109.5
C2—C3—C6	122.2 (3)	H11A—C11—H11B	109.5
C4—C3—C6	121.0 (3)	C12—C11—H11C	109.5
C5—C4—C3	119.4 (4)	H11A—C11—H11C	109.5
C5—C4—H4	120.3	H11B—C11—H11C	109.5
C3—C4—H4	120.3	O1—C12—O2	124.1 (4)
N1—C5—C4	124.1 (4)	O1—C12—C11	123.6 (4)
N1—C5—H5	117.9	O2—C12—C11	112.3 (4)
C4—C5—H5	117.9	C8—N2—C9	116.3 (4)
C7—C6—C10	117.3 (3)	C12—O2—H2A	109.5
C7—C6—C3	121.4 (3)	C14—O4—H4A	109.5

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O2—H2*A*···N2	0.82	1.86	2.675 (5)	175
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