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4,4′-Bipyridinium 1,4-phenylenediacetate

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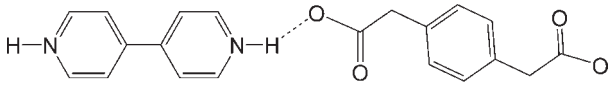
Key indicators: single-crystal X-ray study; T = 296 K; mean σ(C–C) = 0.004 Å; R factor = 0.058; wR factor = 0.193; data-to-parameter ratio = 12.7.

The title compound, $C_{10}H_{10}N_2^{2+}\cdot C_{10}H_8O_4^{2-}$, has inversion centres located at the geometric centres of the 1,4-phenylenediacetate anion and 4,4′-bipyridinium cation. The anions and cations are connected by N—H···O hydrogen bonds, forming one-dimensional supramolecular chains, which interact with each other *via* π–π interactions [centroid–centroid distance = 3.938 (2) Å], building a two-dimensional supramolecular sheet.

Related literature

For related complexes of 1,4-phenylenediacetic acid, see: Braverman & LaDuca (2007); Soares-Santos *et al.* (2008); Liu *et al.* (2009); Chen *et al.* (2006*a*,*b*).



Experimental

Crystal data

$C_{10}H_{10}N_2^{2+}\cdot C_{10}H_8O_4^{2-}$
M_r = 350.36
Triclinic, $P\overline{1}$
a = 4.579 (3) Å
b = 6.950 (5) Å
c = 13.859 (10) Å
α = 99.618 (9)°
β = 93.672 (9)°
γ = 97.373 (8)°
V = 429.6 (5) Å^3
Z = 1
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 296 K
0.25 × 0.20 × 0.18 mm

Data collection

Bruker SMART CCD area detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 1998)
T_{min} = 0.977, T_{max} = 0.983
2241 measured reflections
1499 independent reflections
1052 reflections with $I > 2\sigma(I)$
R_{int} = 0.014

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.058
$wR(F^2)$ = 0.193
S = 1.07
1499 reflections
118 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.41 e Å^{-3}
$\Delta\rho_{min}$ = −0.25 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O2	0.86	1.75	2.613 (3)	176

Data collection: *SMART* (Bruker, 2004); cell refinement: *SAINT* (Bruker, 2004); 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: JH2103).

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supporting information

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4,4′-Bipyridinium 1,4-phenylenediacetate

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S1. Comment

The flexible ligand of 1,4-phenylenediacetate is drawing much interest in constructing metal–organic framework or supramolecular molecules due to its flexibility and its multifunctional groups of carboxylato group and phenyl ring (Braverman & LaDuca 2007; Soares-Santos *et al.*, 2008; Liu *et al.*, 2009; Chen *et al.*, 2006*a*,*b*). We thus report here a supramolecular structrure formed by 4,4′-bipyridine and 1,4-phenylenediacetic acid. The title compound 4,4′-bipyridinium 1,4-phenylenediacetate (Fig. 1), $[C_{10}H_{10}N_2][C_{10}H_8O_4]$, has inversion centres located on the geometric centres of the 1,4-phenylenediacetate anion and 4,4′-bipyridinium cation. Each 4,4′-bipyridinium cation connects two 1,4-phenylenediacetate anions *via* N—H···O hydrogen bonds (Table 1), and *vice versa*. This leads to the formation of one dimensional supramolecular chains as shown in Fig. 2. The neighboring pyridyl rings from the adjacent one chains parallel to each other with perpendicular distance of 3.5654 (4) Å, a centre-to-centre distance of 3.938 (2) Å and an offset angle of 25.135 (7)°. These information suggest the existence of significant $\pi\cdots\pi$ stacking interaction between the two pyridyl rings, which results in the construction of two dimensional supramolecular sheets (Fig. 2) from the one dimensional chains.

S2. Experimental

A mixture of 1,4-phenylenediacetic acid (0.0584 g, 0.3 mmol), 4,4′-bipyridine (0.0312 g, 0.2 mmol), $Mn(CH_3COO)_2.4H_2O$ (0.0735 g, 0.3 mmol) and ethanol (2 ml) was sealed in a 23 ml Teflon-lined autoclave, heated at 140 °C for 4 d and cooled over a period of 48 h. Colorless crystals of the title compound were collected in a yield of 60% (0.0802 g). Found: C, 68.26; H, 5.40; N, 7.28. $C_{20}H_{18}N_2O_4$ requires C, 68.56; H, 5.18; N, 7.52%.

S3. Refinement

H atoms on the carbon and nitrogen atoms were placed at calculated positions (C–H = 0.93 Å and N–H = 0.86 Å) and were included in the refinement in the riding model approximation, with U_{iso}(H) = 1.2U_{eq}(C, N).

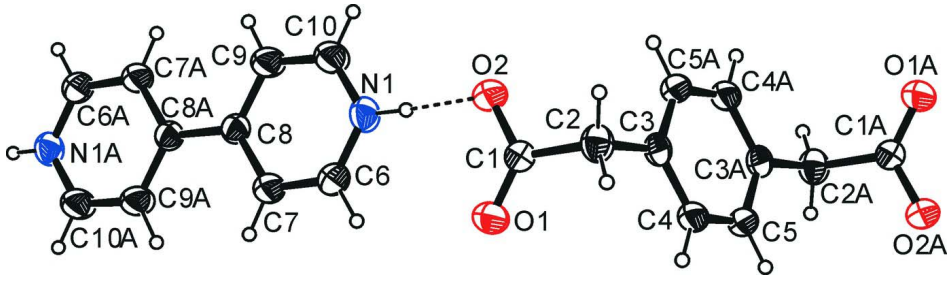

Figure 1

The molecular structure of the title compound with the atom-numbering scheme and 30% displacement ellipsoids.

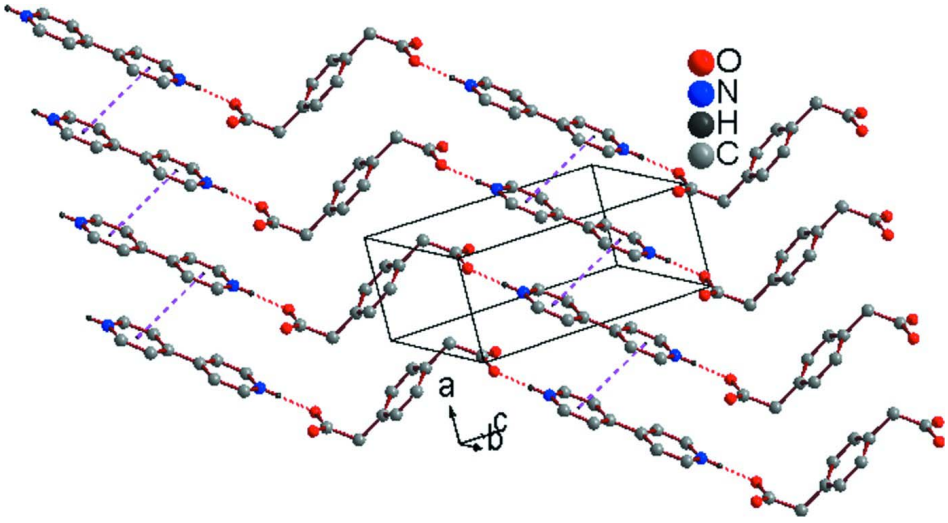

Figure 2

A view of the two-dimensional supramolecular sheet assembled by hydrogen bonds and $\pi\cdots\pi$ stacking interactions.

4,4′-Bipyridinium 1,4-phenylenediacetate

Crystal data

$C_{10}H_{10}N_2^{2+}\cdot C_{10}H_8O_4^{2-}$
$M_r = 350.36$
Triclinic, $P\overline{1}$
Hall symbol: -P 1
$a = 4.579\ (3)$ Å
$b = 6.950\ (5)$ Å
$c = 13.859\ (10)$ Å
$\alpha = 99.618\ (9)°$
$\beta = 93.672\ (9)°$
$\gamma = 97.373\ (8)°$
$V = 429.6\ (5)$ Å^3
$Z = 1$
$F(000) = 184$
$D_x = 1.354$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 736 reflections
$\theta = 3.0–23.8°$
$\mu = 0.10$ mm^{-1}
$T = 296$ K
Block, colourless
0.25 × 0.20 × 0.18 mm

Data collection

Bruker SMART CCD area detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 1998)
$T_{min} = 0.977$, $T_{max} = 0.983$
2241 measured reflections
1499 independent reflections
1052 reflections with $I > 2\sigma(I)$
$R_{int} = 0.014$
$\theta_{max} = 25.0°$, $\theta_{min} = 3.0°$
$h = -5\rightarrow5$
$k = -8\rightarrow7$
$l = -10\rightarrow16$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.058$
$wR(F^2) = 0.193$
$S = 1.07$
1499 reflections
118 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

$w = 1/[\sigma^2(F_o^2) + (0.0979P)^2 + 0.1429P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$

$\Delta\rho_{max} = 0.41$ e Å^{-3}
$\Delta\rho_{min} = -0.25$ 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}
O2	0.6746 (5)	0.5442 (3)	0.19005 (15)	0.0691 (7)
O1	0.7402 (6)	0.3340 (3)	0.28869 (16)	0.0812 (8)
N1	0.3813 (5)	0.7303 (4)	0.32281 (18)	0.0628 (7)
H1	0.4734	0.6645	0.2794	0.075*
C8	0.0807 (6)	0.9428 (4)	0.46281 (19)	0.0511 (7)
C4	0.6539 (6)	−0.0571 (4)	0.0761 (2)	0.0568 (8)
H4	0.7572	−0.0977	0.1273	0.068*
C5	0.4446 (7)	−0.1901 (4)	0.0152 (2)	0.0583 (8)
H5	0.4078	−0.3191	0.0262	0.070*
C3	0.7125 (6)	0.1349 (4)	0.06220 (19)	0.0522 (7)
C7	0.1869 (7)	0.7720 (4)	0.4784 (2)	0.0644 (8)
H7	0.1595	0.7255	0.5367	0.077*
C9	0.1311 (8)	1.0030 (5)	0.3742 (2)	0.0709 (9)
H9	0.0632	1.1163	0.3597	0.085*
C6	0.3343 (7)	0.6709 (4)	0.4066 (2)	0.0689 (9)
H6	0.4032	0.5558	0.4179	0.083*
C2	0.9333 (7)	0.2827 (5)	0.1320 (2)	0.0642 (8)
H2A	1.0804	0.2150	0.1606	0.077*
H2B	1.0338	0.3765	0.0963	0.077*
C1	0.7762 (6)	0.3900 (4)	0.2123 (2)	0.0524 (7)
C10	0.2832 (7)	0.8930 (5)	0.3077 (2)	0.0739 (10)
H10	0.3182	0.9365	0.2490	0.089*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O2	0.0924 (16)	0.0590 (13)	0.0641 (14)	0.0268 (11)	0.0119 (11)	0.0198 (10)
O1	0.119 (2)	0.0764 (15)	0.0619 (14)	0.0402 (14)	0.0238 (13)	0.0264 (12)
N1	0.0687 (16)	0.0644 (16)	0.0565 (15)	0.0193 (12)	0.0133 (12)	0.0033 (12)
C8	0.0531 (16)	0.0494 (15)	0.0509 (16)	0.0097 (12)	0.0035 (12)	0.0074 (12)
C4	0.0694 (18)	0.0581 (17)	0.0498 (16)	0.0235 (14)	0.0140 (14)	0.0151 (13)
C5	0.0774 (19)	0.0476 (16)	0.0568 (17)	0.0191 (14)	0.0214 (15)	0.0157 (13)

C3	0.0563 (16)	0.0547 (16)	0.0487 (15)	0.0168 (13)	0.0214 (12)	0.0049 (12)
C7	0.085 (2)	0.0583 (18)	0.0566 (18)	0.0252 (15)	0.0140 (15)	0.0156 (14)
C9	0.094 (2)	0.066 (2)	0.066 (2)	0.0364 (17)	0.0240 (17)	0.0240 (15)
C6	0.092 (2)	0.0569 (18)	0.065 (2)	0.0303 (17)	0.0141 (17)	0.0125 (15)
C2	0.0603 (17)	0.0675 (19)	0.0650 (19)	0.0138 (15)	0.0152 (14)	0.0048 (15)
C1	0.0589 (16)	0.0480 (15)	0.0503 (16)	0.0081 (13)	0.0052 (13)	0.0081 (12)
C10	0.095 (2)	0.076 (2)	0.063 (2)	0.0354 (19)	0.0246 (18)	0.0229 (16)

Geometric parameters (Å, º)

O2—C1	1.296 (3)	C5—H5	0.9300
O1—C1	1.202 (3)	C3—C5^{ii}	1.388 (4)
N1—C10	1.312 (4)	C3—C2	1.513 (4)
N1—C6	1.317 (4)	C7—C6	1.383 (4)
N1—H1	0.8600	C7—H7	0.9300
C8—C7	1.383 (4)	C9—C10	1.379 (4)
C8—C9	1.386 (4)	C9—H9	0.9300
C8—C8^{i}	1.488 (5)	C6—H6	0.9300
C4—C3	1.375 (4)	C2—C1	1.508 (4)
C4—C5	1.375 (4)	C2—H2A	0.9700
C4—H4	0.9300	C2—H2B	0.9700
C5—C3^{ii}	1.388 (4)	C10—H10	0.9300
C10—N1—C6	117.9 (3)	C10—C9—C8	119.3 (3)
C10—N1—H1	121.0	C10—C9—H9	120.4
C6—N1—H1	121.0	C8—C9—H9	120.4
C7—C8—C9	116.8 (3)	N1—C6—C7	122.9 (3)
C7—C8—C8^{i}	122.0 (3)	N1—C6—H6	118.5
C9—C8—C8^{i}	121.3 (3)	C7—C6—H6	118.5
C3—C4—C5	120.9 (3)	C1—C2—C3	109.8 (2)
C3—C4—H4	119.5	C1—C2—H2A	109.7
C5—C4—H4	119.5	C3—C2—H2A	109.7
C4—C5—C3^{ii}	121.1 (3)	C1—C2—H2B	109.7
C4—C5—H5	119.5	C3—C2—H2B	109.7
C3^{ii}—C5—H5	119.5	H2A—C2—H2B	108.2
C4—C3—C5^{ii}	118.0 (3)	O1—C1—O2	123.0 (3)
C4—C3—C2	120.7 (3)	O1—C1—C2	123.0 (3)
C5^{ii}—C3—C2	121.3 (3)	O2—C1—C2	113.9 (3)
C8—C7—C6	119.6 (3)	N1—C10—C9	123.5 (3)
C8—C7—H7	120.2	N1—C10—H10	118.3
C6—C7—H7	120.2	C9—C10—H10	118.3
C3—C4—C5—C3^{ii}	0.3 (4)	C8—C7—C6—N1	0.4 (5)
C5—C4—C3—C5^{ii}	−0.3 (4)	C4—C3—C2—C1	−92.6 (3)
C5—C4—C3—C2	177.3 (2)	C5^{ii}—C3—C2—C1	85.0 (3)
C9—C8—C7—C6	−0.2 (5)	C3—C2—C1—O1	90.7 (4)
C8^{i}—C8—C7—C6	179.7 (3)	C3—C2—C1—O2	−87.0 (3)
C7—C8—C9—C10	−0.5 (5)	C6—N1—C10—C9	−0.8 (5)

C8^{i}—C8—C9—C10	179.6 (3)	C8—C9—C10—N1	1.0 (6)
C10—N1—C6—C7	0.1 (5)		

Symmetry codes: (i) −*x*, −*y*+2, −*z*+1; (ii) −*x*+1, −*y*, −*z*.

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O2	0.86	1.75	2.613 (3)	176