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2,2′-Bipyridinium 5-nitroisophthalate 5-nitroisophthalic acid dihydrate

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Key indicators

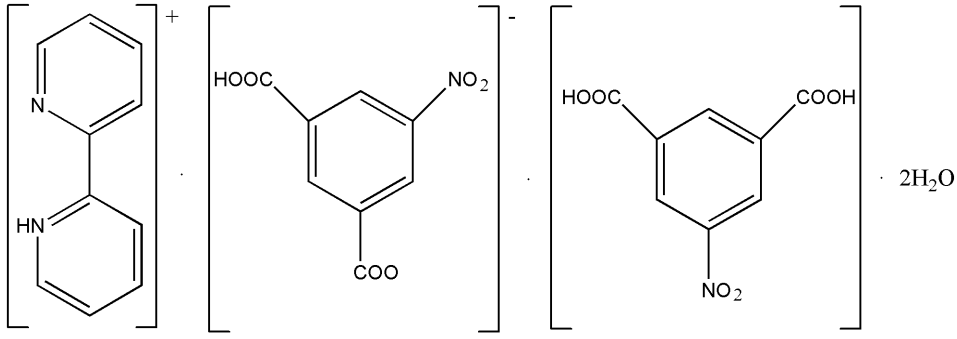
Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.004 Å
R factor = 0.073
wR factor = 0.157
Data-to-parameter ratio = 11.7

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

The title compound, $C_{10}H_9N_2^+\cdot C_8H_4NO_6^-\cdot C_8H_5NO_6\cdot 2H_2O$, consists of singly protonated 2,2′-bipyridinium cations, 5-nitroisophthalate anions, 5-nitroisophthalic acid and water molecules of crystallization, linked by N—H···O and O—H···O hydrogen bonds. The moieties are linked by multiple hydrogen bonds into an undulating sheet structure.

Comment

In the synthesis of crystal structures by design, the assembly of molecular units in predefined arrangements is a key goal (Desiraju, 1995, 1997; Braga *et al.*, 1998). Directional intermolecular interactions are the primary tools in achieving this goal and hydrogen bonding is currently the best among them (Zaworotko, 1997; Braga & Grepioni, 2000). In this paper, we report the structure of the title compound, (I).



(I)

Compound (I) consists of singly protonated 2,2′-bipyridinium cations, 5-nitroisophthalate anions, and 5-nitroisophthalic acid and solvent water molecules (Fig. 1). 2,2′-Bipyridinium cations are linked to the carboxy groups of the 5-nitroisophthalic acid molecules through N—H···O

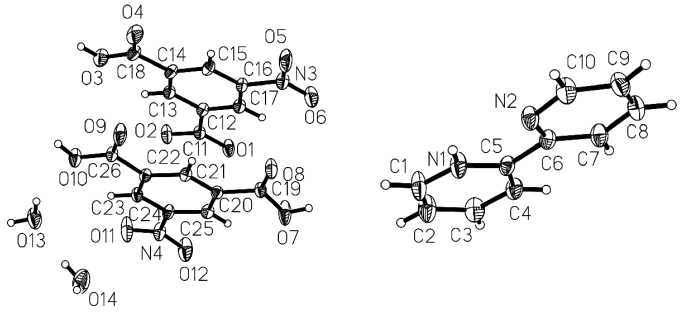


Figure 1
The asymmetric unit of (I), showing the atom-numbering scheme and displacement ellipsoids at the 50% probability level.

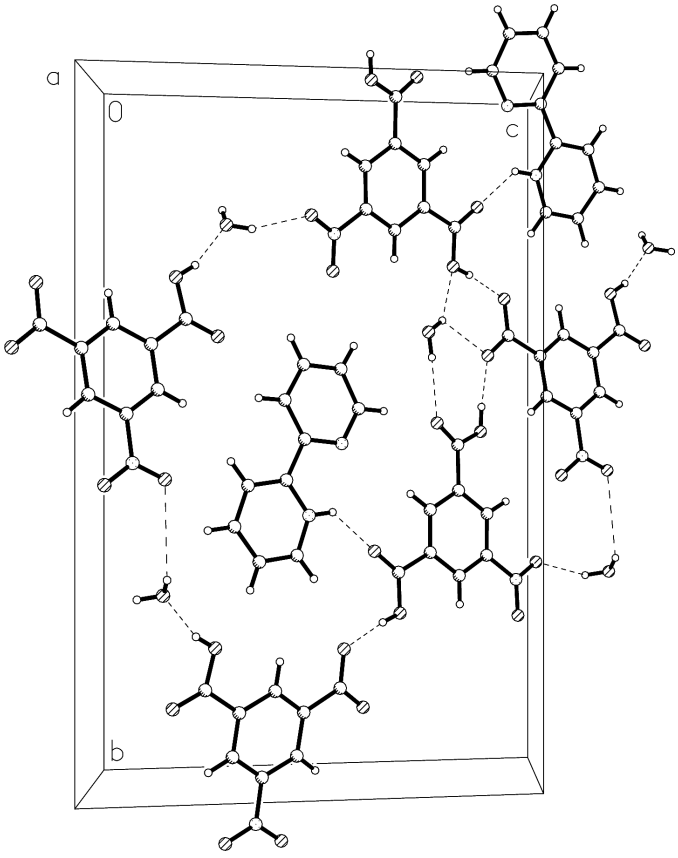


Figure 2
Perspective view of the layer structure of (I), assembled *via* hydrogen bonds, which are shown as dashed lines.

hydrogen-bonding interactions. The 5-nitroisophthalate anions and solvent water molecules form hydrogen bonds with both the carboxylic acid and nitro groups (Table 2). The cations, anions, solvent water molecules and 5-nitroisophthalic acid molecules interact through multimolecular interactions generating an undulating layer structure (Fig. 2).

Experimental

The title compound was synthesized by the hydrothermal method from a mixture of 5-nitroisophthalic acid (0.3 mmol), $La_2(SO_4)_3{\cdot}9H_2O$ (0.3 mmol), 2,2-bipyridine (0.3 mmol) and water (8.0 ml) in a 15 ml Telfon-lined stainless steel reactor. The solution was heated at 423 K for 4 d. After reaction, the vessel was cooled slowly to room temperature to give colorless crystals. The prismatic crystals were collected and washed with distilled water and dried in air.

Crystal data

$C_{10}H_9N_2^+{\cdot}C_8H_5NO_6^-{\cdot}C_8H_4NO_6{\cdot}2H_2O$
M_r = 614.48
Monoclinic, $P2_1/c$
a = 8.3294 (6) Å
b = 22.4059 (16) Å
c = 14.5461 (11) Å
β = 102.2410 (10)°
V = 2653.0 (3) Å^3
Z = 4
D_x = 1.538 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 4810 reflections
θ = 1.7–25.3°
μ = 0.13 mm^{-1}
T = 298 (2) K
Prism, colorless
0.35 × 0.20 × 0.17 mm

Data collection

Bruker SMART APEX area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2002)
T_{min} = 0.957, T_{max} = 0.979
14108 measured reflections
4810 independent reflections
4201 reflections with $I > 2\sigma(I)$
R_{int} = 0.028
θ_{max} = 25.3°
$h = -9 \rightarrow 9$
$k = -26 \rightarrow 25$
$l = -11 \rightarrow 17$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.073
$wR(F^2)$ = 0.157
S = 1.23
4810 reflections
412 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.051P)^2 + 1.822P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.24 e Å^{-3}
$\Delta\rho_{min}$ = −0.24 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

O1—C11	1.238 (3)	O10—C26	1.297 (3)
O2—C11	1.251 (3)	O11—N4	1.202 (3)
O3—C18	1.309 (4)	O12—N4	1.205 (3)
O4—C18	1.202 (3)	N1—C1	1.332 (4)
O5—N3	1.214 (3)	N1—C5	1.340 (4)
O6—N3	1.216 (3)	N2—C10	1.327 (4)
O7—C19	1.295 (3)	N2—C6	1.346 (4)
O8—C19	1.195 (3)	N3—C16	1.473 (4)
O9—C26	1.196 (3)	N4—C24	1.475 (3)
C1—N1—C5	123.8 (3)	N2—C10—C9	123.7 (3)
C10—N2—C6	117.0 (3)	O1—C11—O2	124.0 (3)
O5—N3—O6	123.5 (3)	O1—C11—C12	119.4 (2)
O5—N3—C16	117.9 (3)	O2—C11—C12	116.6 (2)
O6—N3—C16	118.7 (2)	O4—C18—O3	124.8 (3)
O11—N4—O12	123.9 (3)	O4—C18—C14	122.5 (3)
O11—N4—C24	118.3 (3)	O3—C18—C14	112.7 (2)
O12—N4—C24	117.8 (2)	O8—C19—O7	123.7 (3)
N1—C1—C2	119.3 (3)	O8—C19—C20	122.7 (3)
N1—C5—C4	117.4 (3)	O7—C19—C20	113.7 (2)
N1—C5—C6	116.0 (2)	O9—C26—O10	124.8 (3)
N2—C6—C7	122.6 (3)	O9—C26—C22	120.8 (2)
N2—C6—C5	114.2 (2)	O10—C26—C22	114.4 (2)

Table 2
Hydrogen-bonding geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O3—H3A···O14^{i}	0.82	1.79	2.582 (4)	162
O7—H7A···O1^{ii}	0.82	1.77	2.567 (3)	165
O10—H10A···O2^{iii}	0.82	1.74	2.553 (3)	170
O13—H13A···O8^{iv}	0.824 (12)	2.174 (13)	2.986 (3)	168 (3)
O13—H13B···O10	0.83 (3)	2.21 (2)	2.944 (4)	149 (2)
O14—H14A···O12^{iii}	0.82 (4)	2.55 (4)	3.042 (4)	120 (4)
O14—H14B···O13	0.82 (4)	2.02 (4)	2.830 (5)	171 (4)
N1—H1A···O9^{v}	0.86	2.06	2.746 (4)	136

Symmetry codes: (i) $x-1, y, z$; (ii) $1-x, 1-y, -z$; (iii) $x, \frac{1}{2}-y, \frac{1}{2}+z$; (iv) $1-x, y-\frac{1}{2}, \frac{1}{2}-z$; (v) $1-x, \frac{1}{2}+y, \frac{3}{2}-z$.

The water H atoms were refined subject to the restraint O—H = 0.82 (1) Å. The other H atoms were positioned geometrically (with orientation to fit the observed electron density for carboxyl O—H) and allowed to ride on their parent atoms at distances of 0.82 (O—H), 0.86 (N—H) and 0.93 Å (C—H), with U_{iso}(H) = 1.5U_{eq}(O) and 1.2U_{eq}(N or C). Three carboxyl H atoms are positioned geometrically,

the other carboxylic H atom cannot be positioned geometrically, and this H atom was added to atom N1 atom using the HFIX 43 instruction as no hydrogen bond was indicated by *SHELXL97*.

Data collection: *SMART* (Bruker, 2002); cell refinement: *SAINT* (Bruker, 2002); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEPII* (Johnson, 1976); software used to prepare material for publication: *SHELXL97*.

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