

4-(4-Pyridyl)pyridinium triiodide

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Key indicators

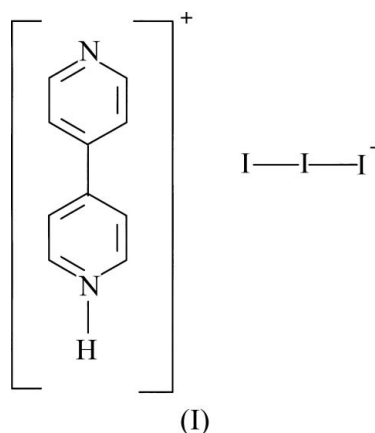
Single-crystal X-ray study
 $T = 100$ K
Mean $\sigma(\text{C}-\text{C}) = 0.007$ Å
 R factor = 0.036
 wR factor = 0.079
Data-to-parameter ratio = 38.2For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.

The title compound, $\text{C}_{10}\text{H}_9\text{N}_2^+\cdot\text{I}_3^-$, contains monoprotonated 4,4'-bipyridinium cations and triiodide anions. The cations assemble into infinite chains by way of $\text{N}-\text{H}\cdots\text{N}$ hydrogen bonds.

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Comment

We are presently interested in the syntheses and structures of dioxorhenium(V) halide salts (Kochel, 2006). The title compound, (I) (Fig. 1), arose as an unexpected product in this system. The asymmetric unit contains a monoprotonated 4,4'-bipyridinium cation and an I_3^- triiodide anion. The geometry of the I_3^- species (Table 1) is normal (Wieczorrek, 2000). The closest contact between I atoms in different anions is 3.8880 (6) Å for $\text{I}2\cdots\text{I}3^i$ [symmetry code: (i) $x, y - 1, z$]; this is 0.07 Å shorter than the van der Waals contact distance of 3.96 Å for two I atoms (Bondi, 1964). The dihedral angle between the aromatic rings in the cation is 33.5 (3)°.



The molecular packing in (I) can be characterized by a layer system (Fig. 2), in which layers of I_3^- anions alternate with sheets of 4,4'-bipyridinium cations, the sheets being parallel to (hkl) and stacked in the $[100]$ direction. Within the organic sheets, an $\text{N}-\text{H}\cdots\text{N}^i$ hydrogen bond links adjacent 4,4'-bipyridinium cations into infinite chains [$\text{N}1-\text{H}1\text{A} = 0.99$ Å, $\text{H}1\text{A}\cdots\text{N}2^i = 1.64$ Å, $\text{N}1\cdots\text{N}2^i = 2.622$ (6) Å and $\text{N}1-\text{H}1\text{A}\cdots\text{N}2^i = 174^\circ$; symmetry code: (i) $x, -1 + y, z$].

Experimental

A mixture of 0.36 g $(\text{NH}_4)_2\text{ReI}_6$ (Watt & Thompson, 1963), 4,4'-bipyridine (0.40 g) and a solution of 1 ml of HI in 50 ml of water was stirred for 5 h at a temperature of 320 K. The color of the reaction mixture changed from dark violet-red to yellow-brown. After reaction, the mixture was cooled and finally two types of crystals were

obtained, yellow plates of $C_{10}H_9N_2^+ \cdot ReO_4^-$ and yellow-brown prisms of the title compound. X-ray quality crystals of (I) were obtained by recrystallization of the selected crude product from an aqueous solution at room temperature.

Crystal data

$C_{10}H_9N_2^+ \cdot I_3^-$
 $M_r = 537.89$
 Triclinic, $P\bar{1}$
 $a = 8.8399$ (9) Å
 $b = 9.6529$ (8) Å
 $c = 9.7154$ (7) Å
 $\alpha = 107.743$ (7)°
 $\beta = 104.709$ (7)°
 $\gamma = 110.791$ (8)°
 $V = 674.38$ (13) Å³
 $Z = 2$
 $D_x = 2.649$ Mg m⁻³
 Mo $K\alpha$ radiation
 $\mu = 6.93$ mm⁻¹
 $T = 100$ (2) K
 Prism, yellow-brown
 $0.12 \times 0.06 \times 0.06$ mm

Data collection

Kuma KM-4-CCD diffractometer
 ω scans
 Absorption correction: numerical
 (*CrysAlis RED*; Oxford
 Diffraction, 2003)
 $T_{min} = 0.490$, $T_{max} = 0.681$
 10483 measured reflections
 5192 independent reflections
 3429 reflections with $I > 2\sigma(I)$
 $R_{int} = 0.050$
 $\theta_{max} = 36.6^\circ$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.036$
 $wR(F^2) = 0.079$
 $S = 0.92$
 5192 reflections
 136 parameters
 H-atom parameters constrained
 $w = 1/[\sigma^2(F_o^2) + (0.0339P)^2]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{max} < 0.001$
 $\Delta\rho_{max} = 3.20$ e Å⁻³
 $\Delta\rho_{min} = -1.41$ e Å⁻³

Table 1

Selected geometric parameters (Å, °).

I1–I3	2.9380 (5)	I1–I2	2.9505 (5)
I3–I1–I2	176.443 (13)		

The N-bound H atom was located in a difference map and refined as riding in its as-found relative position, with $U_{iso}(H) = 1.2U_{eq}(N)$ and N–H = 0.99 Å. The C-bound H atoms were positioned geometrically (C–H = 0.95 Å) and refined as riding, with $U_{iso}(H) = 1.2U_{eq}(\text{carrier})$. The highest peak in the final difference map is 1.38 Å from I3 and the deepest hole is 0.74 Å from I1.

Data collection: *CrysAlis CCD* (Oxford Diffraction, 2003); cell refinement: *CrysAlis RED* (Oxford Diffraction, 2003); data reduction: *CrysAlis RED*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1999); software used to prepare material for publication: *SHELXL97*.

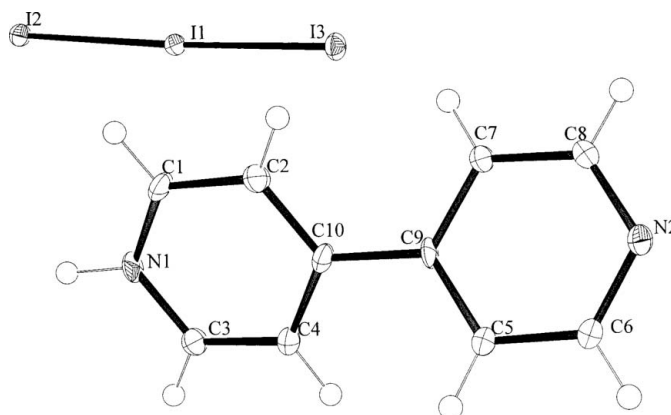


Figure 1

The asymmetric unit of (I), showing 50% displacement ellipsoids (arbitrary spheres for the H atoms).

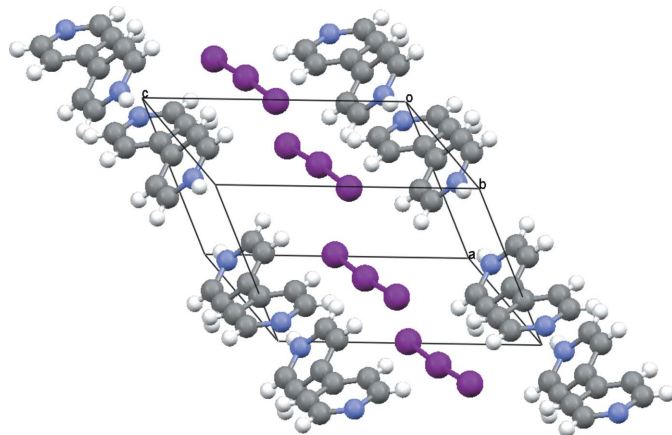


Figure 2

The packing of (I).

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