N-Methyl-N’-(3-phthalimidopropyl)-4,4′-bipyridinium diiodide
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Key indicators

Single-crystal X-ray study
T = 296 K
Mean σ(C–C) = 0.006 Å
R factor = 0.041
wR factor = 0.102
Data-to-parameter ratio = 18.6

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

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The asymmetric unit of the title compound, C_{22}H_{21}N_{3}O_{2}^{2+}.2I^–, consists of a substituted bipyridinium cation and two iodide anions. The dihedral angle between the two rings within the phthalimide moiety is 1.7 (3)°, where the r.m.s. deviations for the five- and six-membered rings are 0.006 and 0.004 Å, respectively. On the other hand, the two pyridinium rings are tilted at an angle of 3.9 (3)° because of the steric contacts between the H atoms at the 3-, 5-, 3′- and 5′-positions of the 4,4′-bipyridinium moiety. The phthalimide plane is tilted by 68.5 (1)° with respect to the pyridinium plane, directly attached to the propylene moiety.

Comment

A photochemical system made up of Ru(bpy)$_3^{2+}$ (bpy = 2,2′-bipyridine) and methylviologen (usually, N,N′-dimethyl-4,4′-bipyridinium dichloride) has been thought of as one of the promising candidates to achieve artificial photosynthetic devices. We previously reported that some amidate-bridged platinum dimers with the general formula [Pt$_2$(NH$_3$)$_4$-(µ-amidato)$_2$]$^{2+}$ (amidate = acetamidate, α-pyroldinonate, α-pyridonate, etc.) serve as effective H$_2$-producing catalysts in a well-known photosystem consisting of edta, Ru(bpy)$_3^{2+}$ and methylviologen (Sakai et al., 1993). Since then, various efforts have been made to develop a more effective system in which the chemical species mentioned above are linked together to give a single molecular device. The title compound, (I), was obtained as a precursor in such studies.

The molecular structure and the crystal packing diagram for (I) are shown in Figs. 1 and 2, respectively. All bond distances and angles in (I) are in the expected ranges.

Experimental

A solution of N-methyl-4,4′-bipyridinium iodide (1.0 mmol; Van Emon et al., 1986) and N-(3-bromopropyl)phthalimide (1.1 mmol) in methanol (20 ml) was refluxed for 2 d. The red prisms or plates deposited were collected by filtration and air-dried (yield: 52%). The purity has been checked by $^1$H NMR spectroscopy.
Crystal data

C_{22}H_{21}N_{3}O_{2}^{2+}.2I^{-}

M_r = 613.22
Triclinic, P\overline{1}

a = 6.0535 (5) Å
b = 7.4674 (6) Å
c = 25.776 (2) Å
\alpha = 83.715 (1)^\circ
\beta = 88.570 (2)^\circ
\gamma = 82.223 (1)^\circ
V = 1147.45 (16) Å\textsuperscript{3}

Z = 2
D_r = 1.775 Mg m\textsuperscript{-3}

Mo K\alpha radiation

Cell parameters from 3414 reflections

\theta = 2.8°–27.2°
\mu = 2.76 mm\textsuperscript{-1}
T = 296 (2) K
Plate, red

0.20 \times 0.20 \times 0.05 mm

Data collection

Bruker SMART APEX CCD-detector diffractometer

Absorption correction: multi-scan (SADABS; Sheldrick, 1996)

T_min = 0.586, T_max = 0.871
6983 measured reflections

4883 independent reflections
4060 reflections with I > 2\sigma(I)

Rint = 0.019

\( R_w = 0.041 \)
\( \theta_{\text{max}} = 27.5° \)
\( h = -7 \rightarrow 7 \)
\( k = -8 \rightarrow 9 \)
\( l = -24 \rightarrow 33 \)

Refinement

Refinement on \( F^2 \)

\( wR(F^2) = 0.102 \)
\( S = 1.05 \)

4883 reflections

H-atom parameters constrained

All H atoms were located at their idealized positions as riding atoms [C—H(aromatic) = 0.93 Å, C—H(methylene) = 0.97 Å and C—H(methyl) = 0.96 Å]. In the final difference Fourier synthesis, five residual peaks in the range 1.01–1.27 e Å\textsuperscript{-3} were observed within 0.94 Å of I atoms.

Data collection: SMART (Bruker, 2001); cell refinement: SAINT (Bruker, 2001); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 1997); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: KENX (Sakai, 2002); software used to prepare material for publication: SHELXL97 (Sheldrick, 1997), TEXSAN (Molecular Structure Corporation, 2001), KENX (Sakai, 2002) and ORTEPII (Johnson, 1976).

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References


