

N*-Methyl-*N'*-(3-phthalimidopropyl)-4,4'-bipyridinium diiodide*Ken Sakai,* Ken Okamoto,
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JapanCorrespondence e-mail:
ksakai@rs.kagu.tus.ac.jp**Key indicators**

Single-crystal X-ray study

 $T = 296$ KMean $\sigma(\text{C}-\text{C}) = 0.006$ 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>.

The asymmetric unit of the title compound, $\text{C}_{22}\text{H}_{21}\text{N}_3\text{O}_2^{2+} \cdot 2\text{I}^-$, consists of a substituted bipyridinium cation and two iodide anions. The dihedral angle between the two rings within the phthalimide moiety is $1.7(3)^\circ$, 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 $3.9(3)^\circ$ 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 $68.5(1)^\circ$ with respect to the pyridinium plane, directly attached to the propylene moiety.

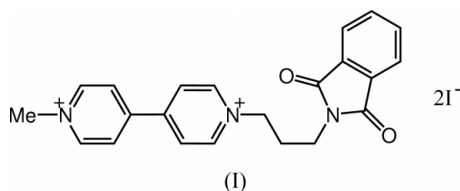
Received 20 May 2003

Accepted 4 June 2003

Online 17 June 2003

Comment

A photochemical system made up of $\text{Ru}(\text{bpy})_3^{2+}$ (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 $[\text{Pt}_2(\text{NH}_3)_4(\mu\text{-amidato})_2]^{2+}$ (amidate = acetamidate, α -pyrrolidinonate, α -pyridonate, *etc.*) serve as effective H_2 -producing catalysts in a well known photosystem consisting of edta, $\text{Ru}(\text{bpy})_3^{2+}$ 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 ^1H NMR spectroscopy.

Crystal data

$C_{22}H_{21}N_3O_2^{2+} \cdot 2I^-$
 $M_r = 613.22$
 Triclinic, $P\bar{1}$
 $a = 6.0535$ (5) Å
 $b = 7.4674$ (6) Å
 $c = 25.776$ (2) Å
 $\alpha = 83.715$ (1)°
 $\beta = 88.570$ (2)°
 $\gamma = 82.223$ (1)°
 $V = 1147.45$ (16) Å³

$Z = 2$
 $D_x = 1.775$ Mg m⁻³
 Mo $K\alpha$ radiation
 Cell parameters from 3414 reflections
 $\theta = 2.8$ – 27.2 °
 $\mu = 2.76$ mm⁻¹
 $T = 296$ (2) K
 Plate, red
 $0.20 \times 0.20 \times 0.05$ mm

Data collection

Bruker SMART APEX CCD-detector diffractometer
 ω scans
 Absorption correction: multi-scan (SADABS; Sheldrick, 1996)
 $T_{\min} = 0.586$, $T_{\max} = 0.871$
 6983 measured reflections

4883 independent reflections
 4060 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.019$
 $\theta_{\text{max}} = 27.5$ °
 $h = -7 \rightarrow 7$
 $k = -8 \rightarrow 9$
 $l = -24 \rightarrow 33$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.041$
 $wR(F^2) = 0.102$
 $S = 1.05$
 4883 reflections
 263 parameters
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.048P)^2 + 0.5905P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} = 0.001$
 $\Delta\rho_{\text{max}} = 1.27$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.44$ e Å⁻³

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⁻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).

This work was partly supported by a Grant-in-Aid for Scientific Research (No. 14340223) from the Ministry of Education, Science, Sports, and Culture of Japan.

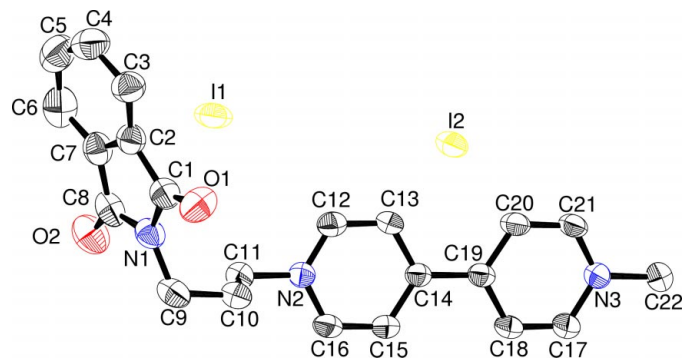


Figure 1

The structure of the independent cation and anions in (I), showing the atom-labeling scheme. Displacement ellipsoids are drawn at the 50% probability level.

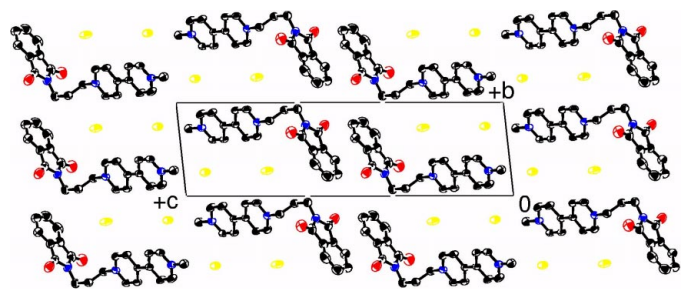


Figure 2

Crystal packing, viewed down the a axis of (I).

References

- Bruker (2001). *SAINT* (Version 6.22) and *SMART* (Version 5.625). Bruker AXS Inc., Madison, Wisconsin, USA.
 Johnson, C. K. (1976). *ORTEPII*. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.
 Molecular Structure Corporation (2001). *TEXSAN*. Version 1.11r1. MSC, 9009 New Trails Drive, The Woodlands, TX 77381-5209, USA.
 Sakai, K., Kizaki, Y., Tsubomura, T. & Matsumoto, K. (1993). *J. Mol. Catal.* **79**, 142–152.
 Sakai, K. (2002). *KENX*. GUI for *SHELXL97*. Tokyo University of Science, Japan.
 Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
 Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
 Van Emon, J., Hammock, B. & Seiber, J. N. (1986). *Anal. Chem.* **58**, 1866–1873.