Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

2,2′-Bipyridinium 1-oxide triiodide

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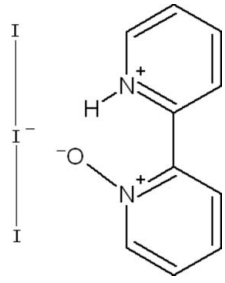
Received 5 October 2007; accepted 8 October 2007

Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.010 Å; R factor = 0.039; wR factor = 0.090; data-to-parameter ratio = 23.2.

The title compound, $C_{10}H_9N_2O^+\cdot I_3^-$, was obtained unintentionally as the product of an attempted synthesis of an iodoplumbate complex using NaI as a donor to provide I^-. The cation is planar (r.m.s. deviation for all non-H atoms is 0.024 Å) and has an intramolecular N—H···O hydrogen bond.

Related literature

For related literature, see: Corey *et al.* (1965).



Experimental

Crystal data

$C_{10}H_9N_2O^+\cdot I_3^-$
M_r = 553.89
Monoclinic, $P2_1/c$
a = 7.9714 (16) Å
b = 12.045 (2) Å
c = 15.434 (3) Å
β = 91.44 (3)°
V = 1481.4 (5) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 6.31 mm^{-1}
T = 293 (2) K
0.20 × 0.15 × 0.12 mm

Data collection

Rigaku Weissenberg IP diffractometer
Absorption correction: multi-scan (*TEXRAY*; Molecular Structure Corporation, 1999)
T_{min} = 0.14, T_{max} = 0.250
(expected range = 0.263–0.469)
13781 measured reflections
3393 independent reflections
2116 reflections with $I > 2\sigma(I)$
R_{int} = 0.041

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.039
$wR(F^2)$ = 0.090
S = 1.06
3393 reflections
146 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.80 e Å^{-3}
$\Delta\rho_{min}$ = −0.75 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2···O1	0.86	1.73	2.439 (6)	138

Data collection: *TEXRAY* (Molecular Structure Corporation, 1999); cell refinement: *TEXRAY*; data reduction: *TEXSAN* (Molecular Structure Corporation, 1999); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SORTX* (McArdle, 1995); software used to prepare material for publication: *SHELXL97*.

This work was supported by the Natural Science Foundation of Fujian Province of China (No. E0710008), the Special Foundation for Young Scientists of Fuzhou Unviersity (XRC0644), and the Science & Technology Promotion Foundation of Fuzhou University (XJJ-0605).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT2530).

References

Corey, E. J., Borror, A. L. & Foglia, T. (1965). *Inorg. Chem.* **30**, 288–290.
McArdle, P. (1995). *J. Appl. Cryst.* **28**, 65.
Molecular Structure Corporation (1999). *TEXRAY* (Version 1.10) and *TEXSAN* (Version 1.10). MSC, The Woodlands, Texas, USA.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.

  doi:10.1107/S1600536807049215

supplementary materials

Acta Cryst. (2007). E**63**, o4322 [**doi:10.1107/S1600536807049215**]

2,2'-Bipyridinium 1-oxide triiodide

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Comment

The three I atoms of the I_3^- anion are almost linear with an I2—I1—I3 angle of 178.89 (2)°. The protonated 2,2'-bipyridine-N-oxide molecule is planar (r.m.s. deviation for all non-H atoms 0.024 Å). The molecular conformation of the cation is stabilized by an N—H···O hydrogen bond.

Experimental

2,2'-bipyridine-N-oxide was prepared as reported in literature (Corey *et al.*, 1965). The title compound was obtained unintentionally as the product of an attempted synthesis of a iodoplumbate complex. 2,2'-bipyridine-N-oxide (0.17 g, 1.0 mmol) and $Pb(NO_3)_2$ (0.33 g, 1 mmol) were dissolved in 15 ml DMSO and stirred for 2 h, then addition $NaI{\cdot}2H_2O$ (0.55 g, 3 mmol) with continuous stirring for 2 h at room temperature. Finally, a clear yellow solution was obtained and adjusted to pH=4.0 using 10% HNO_3/DMSO, which was allowed to evaporate at room temperature. Blue block-like crystals formed over two weeks.

Refinement

All H atoms were positioned geometrically and refined using a riding model, with C—H = 0.93Å and N—H = 0.86Å and with U_{iso}(H) = 1.2Ueq(C,*N*).

Figures

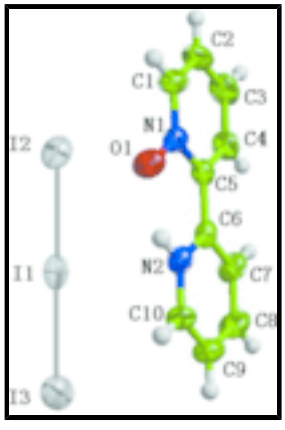

Fig. 1. The structure of the title compound showing the atom numbering scheme with ellipsoids drawn at the 50% probability level.

2,2'-Bipyridinium 1-oxide triiodide

Crystal data

$C_{10}H_9N_2O^+·I_3^-$	F_{000} = 1000
M_r = 553.89	D_x = 2.484 Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -P2ybc	Cell parameters from 25 reflections
a = 7.9714 (16) Å	θ = 12–18º
b = 12.045 (2) Å	μ = 6.31 mm^{-1}
c = 15.434 (3) Å	T = 293 (2) K
β = 91.44 (3)º	Cube, blue
V = 1481.4 (5) Å^3	0.20 × 0.15 × 0.12 mm
Z = 4	

Data collection

Rigaku Weissenberg IP diffractometer	2116 reflections with $I > 2\sigma(I)$
Radiation source: rotor target	R_{int} = 0.041
Monochromator: graphite	θ_{max} = 27.5º
T = 293(2) K	θ_{min} = 3.1º
ω scans	h = −10→10
Absorption correction: multi-scan (TEXRAY; Molecular Structure Corporation, 1999)	k = −15→15
T_{min} = 0.14, T_{max} = 0.250	l = −20→20
13781 measured reflections	Standard reflections: None
3393 independent reflections	

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)]$ = 0.039	$w = 1/[\sigma^2(F_o^2) + (0.0369P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2)$ = 0.090	$(\Delta/\sigma)_{max} < 0.001$
S = 1.06	$\Delta\rho_{max}$ = 0.80 e Å^{-3}
3393 reflections	$\Delta\rho_{min}$ = −0.75 e Å^{-3}
146 parameters	Extinction correction: SHELXL97 (Sheldrick, 1997), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Primary atom site location: structure-invariant direct methods	Extinction coefficient: 0.0010 (2)
Secondary atom site location: difference Fourier map	

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 $F^2 > 2\text{sigma}(F^2)$ 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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
I1	0.27866 (5)	0.31326 (4)	0.13335 (3)	0.07513 (17)
I2	0.41434 (6)	0.48743 (5)	0.24785 (3)	0.0888 (2)
I3	0.14733 (7)	0.14252 (5)	0.01697 (4)	0.1009 (2)
N1	0.8457 (5)	0.3845 (4)	0.2042 (3)	0.0575 (11)
O1	0.8225 (5)	0.2820 (3)	0.2317 (2)	0.0704 (11)
N2	0.7180 (6)	0.2285 (4)	0.0877 (3)	0.0588 (11)
H2	0.7394	0.2144	0.1415	0.071*
C1	0.9078 (7)	0.4603 (6)	0.2609 (4)	0.0728 (17)
H1	0.9334	0.4388	0.3175	0.087*
C2	0.9336 (9)	0.5660 (6)	0.2373 (5)	0.088 (2)
H2A	0.9770	0.6164	0.2777	0.106*
C3	0.8969 (10)	0.6006 (6)	0.1549 (6)	0.095 (2)
H3	0.9159	0.6736	0.1381	0.114*
C4	0.8312 (9)	0.5244 (5)	0.0974 (4)	0.0767 (17)
H4	0.8023	0.5472	0.0414	0.092*
C5	0.8065 (7)	0.4149 (5)	0.1202 (3)	0.0575 (13)
C6	0.7418 (7)	0.3317 (5)	0.0591 (3)	0.0573 (13)
C7	0.7041 (9)	0.3532 (6)	−0.0261 (4)	0.0774 (18)
H7	0.7169	0.4246	−0.0479	0.093*
C8	0.6471 (9)	0.2690 (8)	−0.0799 (4)	0.091 (2)
H8	0.6225	0.2834	−0.1380	0.109*
C9	0.6267 (9)	0.1642 (7)	−0.0472 (5)	0.090 (2)
H9	0.5887	0.1066	−0.0828	0.108*
C10	0.6627 (8)	0.1462 (5)	0.0369 (4)	0.0740 (17)
H10	0.6489	0.0756	0.0600	0.089*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
I1	0.0627 (3)	0.0830 (3)	0.0804 (3)	0.0191 (2)	0.01698 (19)	0.0211 (2)
I2	0.0840 (3)	0.1103 (4)	0.0719 (3)	0.0224 (3)	−0.0012 (2)	0.0038 (2)
I3	0.0921 (4)	0.0977 (4)	0.1132 (4)	0.0065 (3)	0.0090 (3)	−0.0074 (3)

N1	0.057 (3)	0.055 (3)	0.061 (3)	0.005 (2)	0.007 (2)	−0.004 (2)
O1	0.096 (3)	0.056 (2)	0.060 (2)	0.003 (2)	−0.004 (2)	0.0055 (19)
N2	0.064 (3)	0.058 (3)	0.054 (3)	0.005 (2)	0.006 (2)	0.004 (2)
C1	0.072 (4)	0.077 (5)	0.070 (4)	−0.004 (3)	−0.005 (3)	−0.018 (3)
C2	0.084 (5)	0.067 (5)	0.115 (6)	−0.018 (4)	0.012 (4)	−0.033 (4)
C3	0.107 (6)	0.059 (4)	0.120 (6)	−0.015 (4)	0.022 (5)	0.006 (4)
C4	0.089 (5)	0.062 (4)	0.079 (4)	−0.004 (3)	0.010 (3)	0.009 (3)
C5	0.056 (3)	0.055 (3)	0.061 (3)	0.002 (3)	0.009 (2)	0.002 (3)
C6	0.054 (3)	0.064 (4)	0.055 (3)	0.007 (3)	0.011 (2)	0.006 (3)
C7	0.099 (5)	0.078 (4)	0.055 (3)	0.006 (4)	0.006 (3)	0.009 (3)
C8	0.105 (6)	0.116 (6)	0.050 (4)	0.001 (5)	−0.006 (3)	−0.006 (4)
C9	0.096 (5)	0.098 (6)	0.075 (5)	−0.016 (4)	0.000 (4)	−0.030 (4)
C10	0.087 (5)	0.063 (4)	0.072 (4)	−0.013 (3)	0.006 (3)	−0.018 (3)

Geometric parameters (Å, °)

I1—I3	2.9080 (9)	C3—C4	1.371 (10)
I1—I2	2.9322 (9)	C3—H3	0.9300
N1—O1	1.320 (6)	C4—C5	1.380 (8)
N1—O1	1.320 (6)	C4—H4	0.9300
N1—C1	1.349 (7)	C5—C6	1.461 (8)
N1—C5	1.377 (7)	C6—C7	1.366 (8)
N2—C10	1.332 (7)	C7—C8	1.379 (9)
N2—C6	1.334 (7)	C7—H7	0.9300
N2—H2	0.8600	C8—C9	1.371 (10)
C1—C2	1.341 (9)	C8—H8	0.9300
C1—H1	0.9300	C9—C10	1.340 (9)
C2—C3	1.364 (10)	C9—H9	0.9300
C2—H2A	0.9300	C10—H10	0.9300
I3—I1—I2	178.89 (2)	C5—C4—H4	119.0
O1—N1—C1	118.5 (5)	N1—C5—C4	117.6 (5)
O1—N1—C1	118.5 (5)	N1—C5—C6	119.6 (5)
O1—N1—C5	121.4 (4)	C4—C5—C6	122.8 (5)
O1—N1—C5	121.4 (4)	N2—C6—C7	117.8 (6)
C1—N1—C5	120.1 (5)	N2—C6—C5	118.4 (5)
C10—N2—C6	123.0 (5)	C7—C6—C5	123.8 (6)
C10—N2—H2	118.5	C6—C7—C8	120.1 (6)
C6—N2—H2	118.5	C6—C7—H7	120.0
C2—C1—N1	121.6 (6)	C8—C7—H7	120.0
C2—C1—H1	119.2	C9—C8—C7	119.7 (6)
N1—C1—H1	119.2	C9—C8—H8	120.1
C1—C2—C3	120.8 (7)	C7—C8—H8	120.1
C1—C2—H2A	119.6	C10—C9—C8	118.7 (6)
C3—C2—H2A	119.6	C10—C9—H9	120.7
C2—C3—C4	118.0 (7)	C8—C9—H9	120.7
C2—C3—H3	121.0	N2—C10—C9	120.7 (6)
C4—C3—H3	121.0	N2—C10—H10	119.7
C3—C4—C5	122.0 (6)	C9—C10—H10	119.7
C3—C4—H4	119.0		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2···O1	0.86	1.73	2.439 (6)	138

Fig. 1