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Bis(2,6-dimethylpyridinium) dibromoiodate bromide

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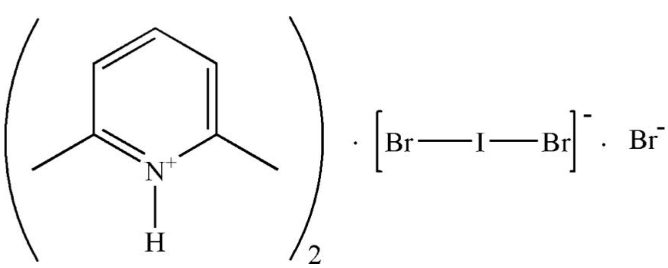
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.011 Å; R factor = 0.040; wR factor = 0.104; data-to-parameter ratio = 19.9.

In the title salt, $2C_7H_{10}N^+\cdot IBr_2^-\cdot Br^-$, each of the anions, *viz.* $[IBr_2]^-$ and Br^-, lie on a twofold axis. The IBr_2^- anion is almost linear, with a Br—I—Br angle of 178.25 (3)°. The cation is essentially planar (r.m.s. deviation = 0.0067 Å). In the crystal, each Br^- anion links two cations *via* N—H···Br···H—N hydrogen-bonding interactions.

Related literature

For background to this study, see: Kochel (2006). For comparison bond lengths and angles, see: Gardberg *et al.* (2002); Ahmadi *et al.* (2008).



Experimental

Crystal data

$2C_7H_{10}N^+\cdot Br_2I^-\cdot Br^-$
M_r = 582.92
Monoclinic, $C2/c$
a = 13.8627 (16) Å
b = 11.3622 (9) Å
c = 13.8957 (15) Å
β = 108.885 (13)°
V = 2070.9 (4) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 7.33 mm^{-1}
T = 293 K
0.34 × 0.28 × 0.15 mm

Data collection

Agilent Xcalibur Eos diffractometer
Absorption correction: analytical (*CrysAlis PRO*; Agilent, 2011)
T_{min} = 0.578, T_{max} = 0.733
4417 measured reflections
1834 independent reflections
1280 reflections with $I > 2\sigma(I)$
R_{int} = 0.034

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.040
$wR(F^2)$ = 0.104
S = 1.05
1834 reflections
92 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.52 e Å^{-3}
$\Delta\rho_{min}$ = −0.58 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1A···Br2	0.86	2.45	3.315 (5)	179

Data collection: *CrysAlis PRO* (Agilent, 2011); cell refinement: *CrysAlis PRO*; data reduction: *CrysAlis PRO*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL*.

The structure was determined at the Hamdi Mango Center for Scientific Research of the University of Jordan.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: PV2580).

References

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supporting information

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S1. Comment

Polyhalides display a variety of structures. Various compounds with interesting structures were found when protonated aromatic nitrogen bases were combined with polyhalides (Kochel, 2006). Herein, we report the crystal structure of $[(C_7H_{10}N)^+]_2$. $[IBr_2]^-$. Br^-, (**I**). Few crystals of (I) were found as an unexpected product from reaction mixture of CdI_2, HBr, 2,6-dimethylpyridine and Br_2 upon attempting to formulate $[(C_7H_{10}N)]_2$ $[CdBr_4]$ salt of 2,6-dimethylpyrinium.

The title salt is depicted in Fig. 1. The IBr_2^- anion is symmetrical and almost linear, Br1—I—Br1^i angle of 178.25 (3) °; (i) –*x* + 1, *y*, -*z* + 1/2], with I—Br1 distance of 2.7117 (9) Å. These values are in agreement with the values reported in the literature (Gardberg *et al.*, 2002). The molecular dimensions of the cation are as expected (Ahmadi *et al.*, 2008).

The cations are arranged as zigzag stacks parallel to the *c*-axis (Fig. 2). Moreover, alternating Br^- and IBr_2^- anions form stacks that separate the cations. Each bromide anion is hydrogen bonded *via* N1—H1A···Br2 with two cations along the *b*-axis (Table 1). There are no significant Br···Br or aryl···aryl interactions in the crystal structure; the shortest Br···Br separation is just greater than 5.0 Å and the shortest distance between the ring centroids is over 4.8 Å.

S2. Experimental

A solution of CdI_2 (0.37 g, 1 mmol) dissolved in 95% EtOH (10 ml) and 2 ml 60% HBr solution was added to a mixture of 2,6-dimethylpyridine (0.11 g, 1 mmol) dissolved in 95% EtOH (10 ml), 60% HBr (2 ml) and molecular bromine (2 ml). The resulting mixture was refluxed for 2 hr. On cooling few reddish crystals of the title complex were found mixed in the bulk of the precipitate formed which proved to be mainly 2,6-dimethylpyridinium bromide.

S3. Refinement

All H atoms were positioned geometrically and refined using a riding model, with N—H = 0.86 Å and C—H = 0.93 and 0.96 Å, for aryl and methyl H-atoms, respectively. The U_{iso}(H) were allowed at $1.5U_{eq}$(C methyl) or $1.2U_{eq}$(N/C non-methyl).

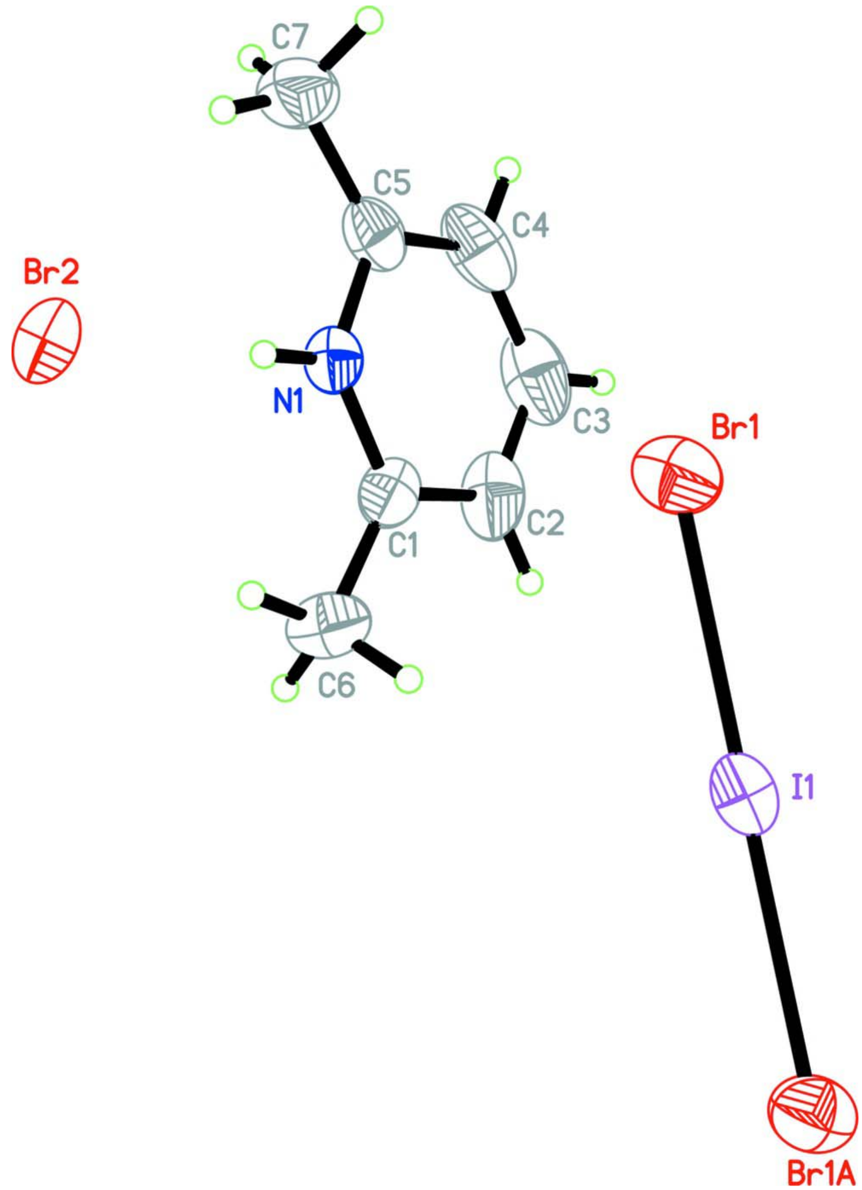

Figure 1

Molecular configuration and atom naming scheme for **I**. Displacement ellipsoids are drawn at the 30% probability level. A stands for the symmetry operation: -*x* + 1, *y*, -*z* + 1/2

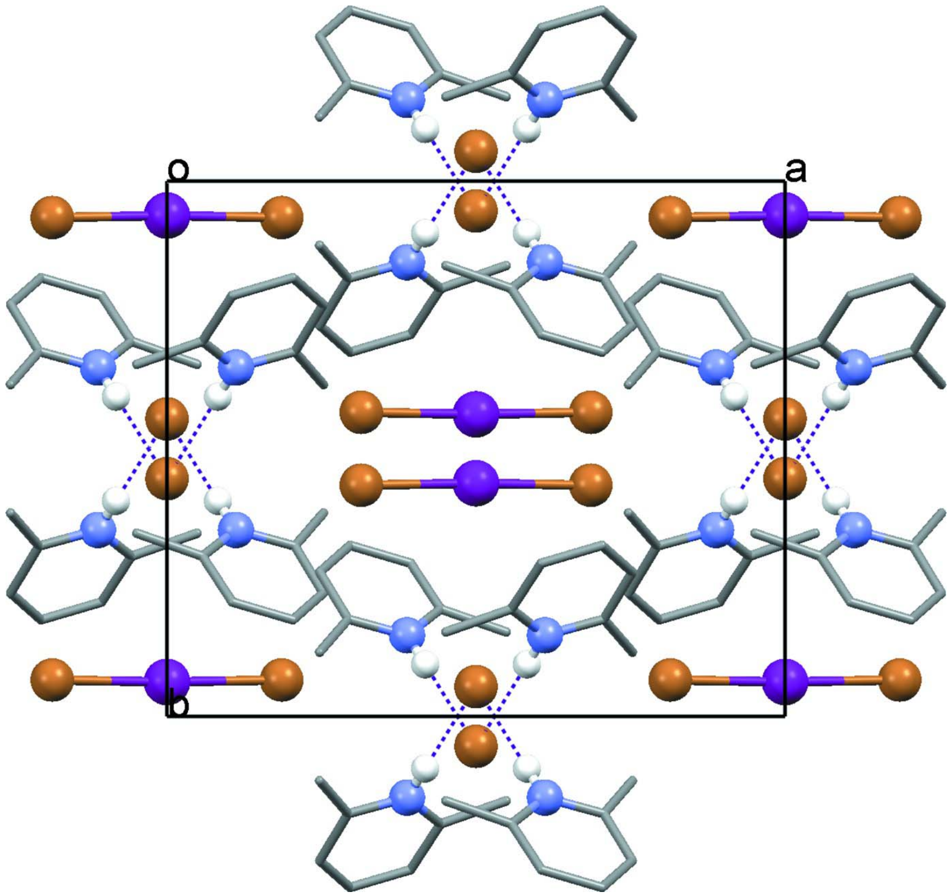

Figure 2

Packing diagram of **I**, down crystallographic *c* axis. Interspecies hydrogen bonds are shown as dashed lines.

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$2C_7H_{10}N^+\cdot Br_2I^-\cdot Br^-$
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Monoclinic, *C*2/*c*
Hall symbol: -C 2yc
a = 13.8627 (16) Å
b = 11.3622 (9) Å
c = 13.8957 (15) Å
β = 108.885 (13)°
V = 2070.9 (4) Å^3
Z = 4
F(000) = 1104
D_x = 1.870 Mg m^{-3}
Mo *Kα* radiation, *λ* = 0.71073 Å
Cell parameters from 1414 reflections
θ = 3.0–29.4°
μ = 7.33 mm^{-1}
T = 293 K
Block, orange
0.34 × 0.28 × 0.15 mm

Data collection

Agilent Xcalibur Eos diffractometer
Radiation source: Enhance (Mo) X-ray Source
Graphite monochromator
Detector resolution: 16.0534 pixels mm^{-1}
ω scans
Absorption correction: analytical (*CrysAlis PRO*; Agilent, 2011)
T_{min} = 0.578, T_{max} = 0.733
4417 measured reflections
1834 independent reflections
1280 reflections with $I > 2\sigma(I)$
R_{int} = 0.034

θ_{max} = 25.0°, θ_{min} = 3.1°
h = −16→12
k = −12→13
l = −16→16

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.040$
$wR(F^2) = 0.104$
S = 1.05
1834 reflections
92 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0423P)^2 + 1.4129P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.52 e Å^{-3}
$\Delta\rho_{min}$ = −0.58 e Å^{-3}

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds 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 $F^2 > \sigma(F^2)$ 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 (Å²)

	x	y	z	U_{iso}*/U_{eq}
I1	0.5000	0.56444 (5)	0.2500	0.0633 (2)
Br1	0.31439 (6)	0.56808 (6)	0.10417 (6)	0.0846 (3)
Br2	0.0000	0.55587 (7)	0.2500	0.0665 (3)
N1	0.1116 (3)	0.3483 (4)	0.1556 (3)	0.0529 (11)
H1A	0.0824	0.4027	0.1793	0.064*
C6	0.2531 (5)	0.3799 (6)	0.3092 (5)	0.081 (2)
H6A	0.2058	0.4369	0.3186	0.122*
H6B	0.3145	0.4188	0.3090	0.122*
H6C	0.2688	0.3237	0.3637	0.122*
C1	0.2069 (5)	0.3183 (5)	0.2109 (5)	0.0614 (16)
C5	0.0574 (5)	0.2992 (5)	0.0649 (5)	0.0654 (17)
C2	0.2529 (6)	0.2305 (6)	0.1722 (6)	0.085 (2)
H2A	0.3183	0.2057	0.2091	0.101*
C7	−0.0487 (5)	0.3421 (7)	0.0149 (5)	0.093 (2)
H7A	−0.0643	0.4021	0.0563	0.140*
H7B	−0.0955	0.2778	0.0069	0.140*
H7C	−0.0544	0.3742	−0.0506	0.140*
C4	0.1058 (8)	0.2137 (6)	0.0278 (6)	0.090 (2)
H4A	0.0720	0.1780	−0.0341	0.108*
C3	0.2038 (8)	0.1805 (6)	0.0814 (7)	0.098 (3)
H3A	0.2363	0.1235	0.0550	0.117*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
I1	0.0866 (5)	0.0525 (4)	0.0601 (4)	0.000	0.0366 (3)	0.000
Br1	0.0928 (6)	0.0837 (5)	0.0703 (5)	0.0069 (4)	0.0166 (4)	−0.0037 (4)
Br2	0.0573 (5)	0.0564 (5)	0.0949 (7)	0.000	0.0371 (5)	0.000
N1	0.059 (3)	0.046 (3)	0.059 (3)	0.006 (2)	0.026 (2)	0.005 (2)
C6	0.065 (4)	0.083 (5)	0.082 (5)	0.005 (4)	0.005 (4)	−0.001 (4)
C1	0.058 (4)	0.056 (4)	0.074 (4)	0.010 (3)	0.028 (3)	0.023 (3)
C5	0.088 (5)	0.058 (4)	0.058 (4)	−0.014 (4)	0.034 (4)	−0.002 (3)
C2	0.098 (6)	0.068 (5)	0.107 (6)	0.035 (4)	0.061 (5)	0.032 (4)
C7	0.076 (5)	0.116 (6)	0.078 (5)	−0.017 (5)	0.012 (4)	0.001 (4)
C4	0.152 (8)	0.063 (5)	0.068 (5)	−0.018 (5)	0.054 (5)	−0.014 (4)
C3	0.151 (8)	0.066 (5)	0.101 (6)	0.031 (5)	0.076 (6)	0.011 (5)

Geometric parameters (Å, º)

I1—Br1	2.7117 (9)	C1—C2	1.383 (9)
I1—Br1^{i}	2.7117 (9)	C5—C4	1.372 (9)
Br2—Br2^{ii}	0.0000	C5—C7	1.490 (9)
Br2—Br2	0.0000	C2—C3	1.350 (10)
N1—C1	1.340 (7)	C2—H2A	0.9300
N1—C5	1.361 (7)	C7—H7A	0.9600
N1—H1A	0.8600	C7—H7B	0.9600
C6—C1	1.483 (8)	C7—H7C	0.9600
C6—H6A	0.9600	C4—C3	1.373 (10)
C6—H6B	0.9600	C4—H4A	0.9300
C6—H6C	0.9600	C3—H3A	0.9300
Br1—I1—Br1^{i}	178.25 (3)	C4—C5—C7	125.8 (7)
Br2^{ii}—Br2—Br2	0 (10)	C3—C2—C1	120.7 (7)
C1—N1—C5	125.0 (5)	C3—C2—H2A	119.6
C1—N1—H1A	117.5	C1—C2—H2A	119.6
C5—N1—H1A	117.5	C5—C7—H7A	109.5
C1—C6—H6A	109.5	C5—C7—H7B	109.5
C1—C6—H6B	109.5	H7A—C7—H7B	109.5
H6A—C6—H6B	109.5	C5—C7—H7C	109.5
C1—C6—H6C	109.5	H7A—C7—H7C	109.5
H6A—C6—H6C	109.5	H7B—C7—H7C	109.5
H6B—C6—H6C	109.5	C5—C4—C3	120.6 (7)
N1—C1—C2	117.0 (6)	C5—C4—H4A	119.7
N1—C1—C6	117.4 (5)	C3—C4—H4A	119.7
C2—C1—C6	125.6 (6)	C2—C3—C4	120.1 (7)
N1—C5—C4	116.6 (6)	C2—C3—H3A	119.9
N1—C5—C7	117.6 (6)	C4—C3—H3A	119.9
C5—N1—C1—C2	−0.2 (9)	C6—C1—C2—C3	−179.9 (7)
C5—N1—C1—C6	−178.7 (5)	N1—C5—C4—C3	0.4 (9)

C1—N1—C5—C4	−0.9 (9)	C7—C5—C4—C3	−179.4 (7)
C1—N1—C5—C7	178.9 (5)	C1—C2—C3—C4	−2.2 (11)
N1—C1—C2—C3	1.8 (9)	C5—C4—C3—C2	1.1 (11)

Symmetry codes: (i) −*x*+1, *y*, −*z*+1/2; (ii) −*x*, *y*, −*z*+1/2.

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*A*···Br2	0.86	2.45	3.315 (5)	179