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1,1′-(Propane-1,3-diyl)dipyridinium dibromidodiiodidomercurate(II)

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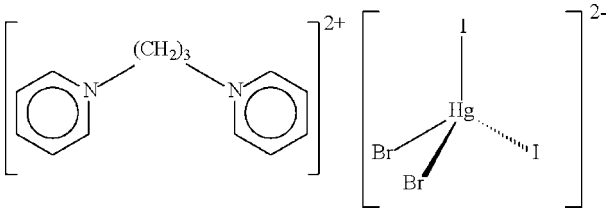
Key indicators: single-crystal X-ray study; T = 295 K; mean σ(C–C) = 0.012 Å; disorder in main residue; R factor = 0.039; wR factor = 0.100; data-to-parameter ratio = 20.1.

The Hg atom in the title compound, $(C_{13}H_{16}N_2)[HgBr_2I_2]$, is coordinated by four halogen atoms in a tetrahedral geometry. The four halogen atoms are each disordered between I and Br, with the I:Br ratios being 0.228 (3):0.772 (3), 0.333 (6):0.667 (6), 0.346 (6):0.654 (6) and 0.843 (7):0.157 (7).

Related literature

For related tetrahalidomercurates, see the preceding paper by Wang *et al.* (2007).



Experimental

Crystal data

$(C_{13}H_{16}N_2)[HgBr_2I_2]$
M_r = 802.74
Monoclinic, $P2_1/c$
a = 8.297 (2) Å
b = 15.431 (4) Å
c = 15.563 (4) Å
β = 95.984 (5)°
V = 1981.8 (9) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 15.02 mm^{-1}
T = 295 (2) K
0.30 × 0.30 × 0.26 mm

Data collection

Bruker APEX area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.011, T_{max} = 0.111
14070 measured reflections
3482 independent reflections
2630 reflections with $I > 2\sigma(I)$
R_{int} = 0.046

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.039
$wR(F^2)$ = 0.100
S = 1.01
3482 reflections
173 parameters
96 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 1.19 e Å^{-3}
$\Delta\rho_{min}$ = −0.75 e Å^{-3}

Table 1
Selected bond lengths (Å).

Hg1—I2	2.534 (14)	Hg1—Br4	2.78 (1)
Hg1—Br1	2.582 (6)	Hg1—I4	2.6963 (14)
Hg1—Br2	2.79 (1)	Hg1—I3	2.72 (3)
Hg1—Br3	2.67 (2)	Hg1—I1	2.868 (16)

Data collection: *SMART* (Bruker, 2004); cell refinement: *SAINT* (Bruker, 2004); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *X-SEED* (Barbour, 2001); software used to prepare material for publication: *publCIF* (Westrip, 2007).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CI2387).

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Westrip, S. P. (2007). *publCIF*. In preparation.

supplementary materials

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1,1'-(Propane-1,3-diyl)dipyridinium dibromidodiiodidomercurate(II)

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Comment

The preceding study reports the structure of a tetrahedral dibromidodichloridomercurate(II), which has been isolated as the 1,2-ethanedipyridinium salt (Wang *et al.*, 2007). Replacing the cation by 1,3-propanedipyridinium furnishes a similar tetrahalomercurate. The anion of the salt is composed 2.25 bromines and 1.75 iodines; the metal atom shows tetrahedral coordination (Fig. 1). Selected bond distances are given in Table 1.

Experimental

The salt was synthesized from the reaction of propane-1,3-dipyridinium dibromide (0.036 g, 0.1 mmol) in methanol (5 ml) and mercuric iodide (0.091 g, 0.2 mmol) in DMF (10 ml). The mixture was set aside for the formation of colorless crystals in 30% yield after several days.

Refinement

The four halogens lie in general positions. Initial attempts to refine the structure with either four bromines or four iodines gave unacceptably high *R*-indices and large peaks/deep holes. The four halogen atoms were then refined as four (Br+I) mixtures; one attempt allowed the mixtures to have the same displacement parameters as well as sharing the same site. A second attempt had the components having the same displacement parameters only. The second led to a formulation consisting of approximately of 2.25 Br and 1.75 I atoms. The use of a restraint that fixed the number of Br and I atoms as exactly 2.25 Br and 1.75 I led to occupancies of 0.772 (3), 0.667 (6), 0.654 (6) and 0.157 (7), respectively, for Br1, Br2, Br3 and Br4, and 0.228 (3), 0.333 (6), 0.346 (6) and 0.843 (7), respectively, for I1, I2, I3 and I4.

The anion is $[HgBr_{2.25}I_{1.75}]^{2-}$, but because it has nearly two bromine and two iodine atoms, it is regarded as $[HgBr_2I_2]$ for the purpose of naming the compound. The formulation is in fair agreement with CH&N elemental analysis. The refinement for a $[C_{13}H_{16}N_2]$ $[HgBr_{2.5}I_{1.5}]$ formulation is not significantly distinguishable in the *R* index, however.

Disorder also affected the cation; the pyridyl ring was refined as a rigid hexagon (C—C = C—N = 1.39 Å). The C(sp^3)—C(sp^3) distance was restrained to 1.50 (1) Å, and the N···C(sp^3) distance to 2.45 (1) Å. The displacement parameters of atoms of the cation were restrained to be nearly isotropic. C-bound H atoms were positioned geometrically (C–H = 0.93 and 0.97 Å), and were included in the refinement in the riding model approximation, with U_{iso}(H) = 1.2U_{eq}(C).

The final difference Fourier map had a large peak at 0.93 Å from Hg1, but was otherwise featureless.

Figures

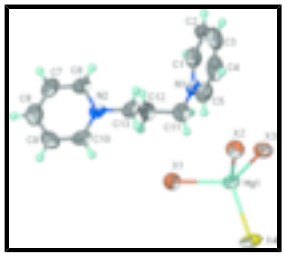

Fig. 1. The molecular structure of $[C_{13}H_{16}N_2][HgBr_2I_2]$. Displacement ellipsoids drawn at the 50% probability level. The bromine and iodine atoms are disordered; the figure depicts the anion as an $[HgX_4]^{2-}$ species. Hydrogen atoms are drawn as spheres of arbitrary radius.

1,1'-(Propane-1,3-diyl)dipyridinium dibromidodiiodidomercurate(II)

Crystal data

$(C_{13}H_{16}N_2)[HgBr_2I_2]$	F_{000} = 1438
M_r = 802.74	D_x = 2.690 Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -P 2ybc	Cell parameters from 3755 reflections
a = 8.297 (2) Å	θ = 2.6–23.5º
b = 15.431 (4) Å	μ = 15.02 mm^{-1}
c = 15.563 (4) Å	T = 295 (2) K
β = 95.984 (5)º	Block, colourless
V = 1981.8 (9) Å^3	0.30 × 0.30 × 0.26 mm
Z = 4	

Data collection

Bruker APEX area-detector diffractometer	3482 independent reflections
Radiation source: fine-focus sealed tube	2630 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.046
T = 295(2) K	θ_{max} = 25.0º
φ and ω scans	θ_{min} = 1.9º
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	h = −9→9
T_{min} = 0.011, T_{max} = 0.111	k = −18→18
14070 measured reflections	l = −18→18

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.039	H-atom parameters constrained
$wR(F^2)$ = 0.100	$w = 1/[\sigma^2(F_o^2) + (0.0445P)^2 + 4.75P]$ where $P = (F_o^2 + 2F_c^2)/3$

S = 1.01	$(\Delta/\sigma)_{max}$ = 0.001
3482 reflections	$\Delta\rho_{max}$ = 1.19 e Å^{-3}
173 parameters	$\Delta\rho_{min}$ = −0.75 e Å^{-3}
96 restraints	Extinction correction: none
Primary atom site location: structure-invariant direct methods	

Special details

Experimental. The crystals do not have well formed faces for a numerical correction. The value is somewhat larger than the cutoff of 3.0, but as the structure seems to have refined smoothly, the correction is alright.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
Hg1	0.25202 (5)	0.20536 (2)	0.28986 (2)	0.06365 (15)	
I1	0.478 (3)	0.3426 (13)	0.2693 (18)	0.0854 (11)	0.228 (3)
I2	0.419 (2)	0.0865 (8)	0.3686 (12)	0.0679 (12)	0.333 (6)
I3	0.134 (3)	0.1381 (19)	0.1346 (19)	0.0669 (17)	0.346 (6)
I4	0.0046 (2)	0.27339 (11)	0.36694 (10)	0.0720 (5)	0.843 (7)
Br1	0.4644 (13)	0.3249 (5)	0.2738 (7)	0.0854 (11)	0.772 (3)
Br2	0.4084 (16)	0.0665 (5)	0.3778 (9)	0.0679 (12)	0.667 (6)
Br3	0.164 (2)	0.1474 (15)	0.1304 (15)	0.0669 (17)	0.654 (6)
Br4	−0.0355 (19)	0.2486 (10)	0.3560 (10)	0.0720 (5)	0.157 (7)
N1	0.6071 (6)	0.1678 (3)	0.0538 (4)	0.0562 (18)	
C1	0.6774 (8)	0.1293 (5)	−0.0139 (4)	0.089 (3)	
H1	0.6873	0.1603	−0.0643	0.106*	
C2	0.7330 (9)	0.0444 (5)	−0.0062 (6)	0.118 (5)	
H2	0.7801	0.0186	−0.0515	0.142*	
C3	0.7182 (10)	−0.0020 (3)	0.0691 (8)	0.116 (5)	
H3	0.7554	−0.0588	0.0742	0.139*	
C4	0.6479 (10)	0.0366 (5)	0.1368 (6)	0.106 (4)	
H4	0.6380	0.0055	0.1872	0.127*	
C5	0.5923 (8)	0.1215 (5)	0.1292 (3)	0.075 (3)	
H5	0.5453	0.1472	0.1744	0.090*	
N2	0.9334 (6)	0.3771 (3)	0.1315 (4)	0.0609 (19)	
C6	1.0535 (7)	0.3751 (4)	0.0756 (4)	0.073 (3)	
H6	1.0577	0.3295	0.0368	0.087*	
C7	1.1673 (7)	0.4414 (5)	0.0775 (5)	0.082 (3)	
H7	1.2476	0.4401	0.0401	0.098*	
C8	1.1611 (8)	0.5096 (4)	0.1354 (5)	0.095 (4)	
H8	1.2372	0.5539	0.1367	0.114*	
C9	1.0410 (10)	0.5116 (4)	0.1913 (5)	0.087 (3)	
H9	1.0368	0.5572	0.2301	0.104*	
C10	0.9272 (7)	0.4453 (4)	0.1894 (4)	0.073 (3)	
H10	0.8468	0.4466	0.2268	0.087*	
C11	0.5536 (10)	0.2563 (6)	0.0483 (7)	0.075 (3)	
H11A	0.4913	0.2684	0.0963	0.090*	

H11B	0.4823	0.2639	−0.0046	0.090*
C12	0.6891 (10)	0.3202 (5)	0.0496 (6)	0.068 (3)
H12A	0.6450	0.3785	0.0488	0.081*
H12B	0.7442	0.3128	−0.0019	0.081*
C13	0.8085 (10)	0.3095 (6)	0.1278 (5)	0.067 (3)
H13A	0.7523	0.3125	0.1793	0.081*
H13B	0.8593	0.2530	0.1263	0.081*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Hg1	0.0598 (2)	0.0770 (3)	0.0551 (2)	0.00921 (18)	0.01045 (17)	−0.01326 (18)
I1	0.084 (2)	0.079 (3)	0.0980 (15)	−0.025 (2)	0.0357 (12)	−0.027 (3)
I2	0.0654 (16)	0.055 (4)	0.080 (3)	0.006 (3)	−0.0092 (15)	0.007 (3)
I3	0.064 (5)	0.081 (4)	0.0569 (19)	−0.001 (3)	0.011 (3)	−0.016 (2)
I4	0.0786 (10)	0.0844 (10)	0.0582 (6)	0.0220 (6)	0.0310 (6)	0.0064 (6)
Br1	0.084 (2)	0.079 (3)	0.0980 (15)	−0.025 (2)	0.0357 (12)	−0.027 (3)
Br2	0.0654 (16)	0.055 (4)	0.080 (3)	0.006 (3)	−0.0092 (15)	0.007 (3)
Br3	0.064 (5)	0.081 (4)	0.0569 (19)	−0.001 (3)	0.011 (3)	−0.016 (2)
Br4	0.0786 (10)	0.0844 (10)	0.0582 (6)	0.0220 (6)	0.0310 (6)	0.0064 (6)
N1	0.048 (4)	0.070 (5)	0.051 (4)	−0.007 (4)	0.005 (3)	0.002 (4)
C1	0.101 (9)	0.105 (9)	0.065 (7)	−0.037 (7)	0.031 (6)	−0.015 (6)
C2	0.119 (11)	0.089 (9)	0.152 (14)	−0.017 (8)	0.041 (10)	−0.057 (9)
C3	0.090 (9)	0.069 (8)	0.184 (16)	−0.016 (7)	−0.013 (10)	−0.020 (10)
C4	0.096 (9)	0.088 (9)	0.127 (12)	−0.025 (7)	−0.015 (9)	0.029 (8)
C5	0.073 (7)	0.094 (8)	0.060 (7)	−0.007 (6)	0.011 (5)	0.013 (6)
N2	0.055 (5)	0.071 (5)	0.058 (5)	0.005 (4)	0.009 (4)	0.002 (4)
C6	0.069 (7)	0.080 (7)	0.072 (7)	0.007 (5)	0.020 (5)	0.002 (5)
C7	0.069 (7)	0.098 (8)	0.082 (8)	−0.004 (6)	0.023 (6)	0.019 (6)
C8	0.078 (8)	0.098 (9)	0.106 (10)	−0.010 (7)	−0.009 (7)	0.004 (8)
C9	0.091 (8)	0.078 (7)	0.087 (8)	0.006 (6)	−0.009 (7)	−0.027 (6)
C10	0.068 (7)	0.095 (8)	0.057 (6)	0.022 (6)	0.008 (5)	−0.004 (5)
C11	0.069 (7)	0.076 (7)	0.077 (7)	0.005 (6)	−0.007 (6)	0.008 (5)
C12	0.065 (6)	0.063 (6)	0.073 (7)	−0.001 (5)	−0.007 (5)	0.010 (5)
C13	0.061 (6)	0.083 (7)	0.060 (6)	0.001 (5)	0.016 (5)	0.007 (5)

Geometric parameters (Å, °)

Hg1—I2	2.534 (14)	N2—C6	1.39
Hg1—Br1	2.582 (6)	N2—C10	1.39
Hg1—Br2	2.79 (1)	N2—C13	1.467 (9)
Hg1—Br3	2.67 (2)	C6—C7	1.39
Hg1—Br4	2.78 (1)	C6—H6	0.93
Hg1—I4	2.6963 (14)	C7—C8	1.39
Hg1—I3	2.72 (3)	C7—H7	0.93
Hg1—I1	2.868 (16)	C8—C9	1.39
N1—C1	1.39	C8—H8	0.93
N1—C5	1.39	C9—C10	1.39
N1—C11	1.435 (11)	C9—H9	0.93

C1—C2	1.39	C10—H10	0.93
C1—H1	0.93	C11—C12	1.494 (7)
C2—C3	1.39	C11—H11A	0.97
C2—H2	0.93	C11—H11B	0.97
C3—C4	1.39	C12—C13	1.495 (8)
C3—H3	0.93	C12—H12A	0.97
C4—C5	1.39	C12—H12B	0.97
C4—H4	0.93	C13—H13A	0.97
C5—H5	0.93	C13—H13B	0.97
I2—Hg1—Br1	102.8 (4)	C5—C4—C3	120.0
I2—Hg1—Br3	106.8 (7)	C5—C4—H4	120.0
Br1—Hg1—Br3	105.8 (4)	C3—C4—H4	120.0
I2—Hg1—I4	118.0 (4)	C4—C5—N1	120.0
Br1—Hg1—I4	108.9 (2)	C4—C5—H5	120.0
Br3—Hg1—I4	113.4 (4)	N1—C5—H5	120.0
I2—Hg1—I3	106.3 (8)	C6—N2—C10	120.0
Br1—Hg1—I3	111.9 (5)	C6—N2—C13	120.6 (6)
Br3—Hg1—I3	6.3 (6)	C10—N2—C13	119.3 (6)
I4—Hg1—I3	108.9 (5)	C7—C6—N2	120.0
I2—Hg1—Br4	116.2 (5)	C7—C6—H6	120.0
Br1—Hg1—Br4	119.1 (4)	N2—C6—H6	120.0
Br3—Hg1—Br4	105.3 (5)	C6—C7—C8	120.0
I4—Hg1—Br4	10.9 (3)	C6—C7—H7	120.0
I3—Hg1—Br4	100.3 (6)	C8—C7—H7	120.0
I2—Hg1—Br2	5.3 (6)	C9—C8—C7	120.0
Br1—Hg1—Br2	108.0 (4)	C9—C8—H8	120.0
Br3—Hg1—Br2	105.5 (6)	C7—C8—H8	120.0
I4—Hg1—Br2	114.7 (3)	C8—C9—C10	120.0
I3—Hg1—Br2	104.4 (7)	C8—C9—H9	120.0
Br4—Hg1—Br2	112.1 (4)	C10—C9—H9	120.0
I2—Hg1—I1	105.0 (7)	C9—C10—N2	120.0
Br1—Hg1—I1	2.3 (9)	C9—C10—H10	120.0
Br3—Hg1—I1	105.2 (6)	N2—C10—H10	120.0
I4—Hg1—I1	107.4 (5)	N1—C11—C12	113.5 (7)
I3—Hg1—I1	111.2 (7)	N1—C11—H11A	108.9
Br4—Hg1—I1	117.4 (6)	C12—C11—H11A	108.9
Br2—Hg1—I1	110.3 (6)	N1—C11—H11B	108.9
Br4—I4—Hg1	93.3 (16)	C12—C11—H11B	108.9
I4—Br4—Hg1	75.8 (16)	H11A—C11—H11B	107.7
C1—N1—C5	120.0	C11—C12—C13	111.9 (6)
C1—N1—C11	120.9 (6)	C11—C12—H12A	109.2
C5—N1—C11	119.1 (6)	C13—C12—H12A	109.2
C2—C1—N1	120.0	C11—C12—H12B	109.2
C2—C1—H1	120.0	C13—C12—H12B	109.2
N1—C1—H1	120.0	H12A—C12—H12B	107.9
C1—C2—C3	120.0	N2—C13—C12	111.1 (6)
C1—C2—H2	120.0	N2—C13—H13A	109.4
C3—C2—H2	120.0	C12—C13—H13A	109.4
C2—C3—C4	120.0	N2—C13—H13B	109.4

C2—C3—H3	120.0	C12—C13—H13B	109.4
C4—C3—H3	120.0	H13A—C13—H13B	108.0
I2—Hg1—I4—Br4	−83.3 (19)	I1—Hg1—Br4—I4	−23 (2)
Br1—Hg1—I4—Br4	160.2 (18)	C11—N1—C1—C2	178.0 (6)
Br3—Hg1—I4—Br4	42.6 (19)	C11—N1—C5—C4	−178.0 (6)
I3—Hg1—I4—Br4	37.9 (19)	C13—N2—C6—C7	177.5 (6)
Br2—Hg1—I4—Br4	−78.7 (19)	C13—N2—C10—C9	−177.5 (6)
I1—Hg1—I4—Br4	158.4 (19)	C1—N1—C11—C12	−68.6 (9)
I2—Hg1—Br4—I4	102.2 (18)	C5—N1—C11—C12	109.4 (8)
Br1—Hg1—Br4—I4	−22 (2)	N1—C11—C12—C13	−55.9 (12)
Br3—Hg1—Br4—I4	−139.9 (18)	C6—N2—C13—C12	−73.2 (9)
I3—Hg1—Br4—I4	−143.8 (19)	C10—N2—C13—C12	104.3 (8)
Br2—Hg1—Br4—I4	105.9 (18)	C11—C12—C13—N2	−175.5 (8)

Fig. 1