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Bis[4-(dimethylamino)pyridinium] tetrabromidocobaltate(II)

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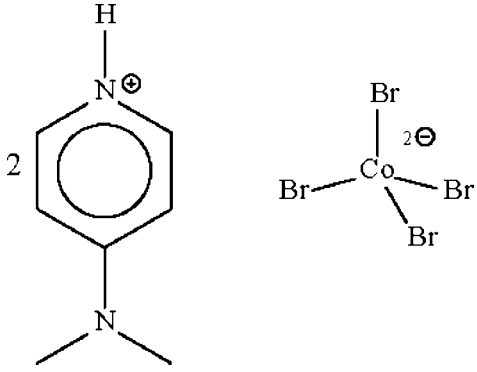
Key indicators: single-crystal X-ray study; T = 140 K; mean σ(C–C) = 0.003 Å; R factor = 0.021; wR factor = 0.054; data-to-parameter ratio = 21.5.

The metal atom in the title salt, $(C_7H_{11}N_2)_2[CoBr_4]$, shows a slightly distorted tetrahedral coordination. The cation forms an N—H···Br hydrogen bond to one of the two Br atoms. The Co^{II} atom lies on a special position of 2 site symmetry.

Related literature

For bis[4-(dimethylamino)pyridinium] tetrabromidocadmate(II) monohydrate, see: Lo & Ng (2009).



Experimental

Crystal data

$(C_7H_{11}N_2)_2[CoBr_4]$ M_r = 624.93
Monoclinic, $C2/c$
a = 10.4020 (2) Å
b = 12.1601 (2) Å
c = 16.9167 (2) Å
β = 104.270 (1)°
V = 2073.76 (6) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 8.54 mm^{-1}
T = 140 K
0.40 × 0.35 × 0.30 mm

Data collection

Bruker SMART APEX diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.131, T_{max} = 0.184
(expected range = 0.055–0.077)
8405 measured reflections
2386 independent reflections
2228 reflections with $I > 2\sigma(I)$
R_{int} = 0.024

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.021
$wR(F^2)$ = 0.054
S = 1.06
2386 reflections
111 parameters
1 restraint
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.49 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
N1—H1···Br1	0.87 (1)	2.71 (2)	3.454 (2)	144 (3)

Data collection: *APEX2* (Bruker, 2008); cell refinement: *SAINT* (Bruker, 2008); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *X-SEED* (Barbour, 2001); software used to prepare material for publication: *publCIF* (Westrip, 2009).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT2980).

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supplementary materials

Acta Cryst. (2009). E**65**, m859 [**doi:10.1107/S1600536809024398**]

Bis[4-(dimethylamino)pyridinium] tetrabromidocobaltate(II)

K. M. Lo and S. W. Ng

Experimental

Cobalt nitrate hexahydrate (0.89 g, 3 mmol) dissolved in a minimum volume of water was mixed with 4-dimethylaminopyridinium hydrobromide perbromide (1.1 g, 3 mmol) dissolved in 50 ml ethanol. The mixture was heated for 1 hour. The red solution slowly turned to blue solution. This was set aside for the growth of crystals.

Refinement

Hydrogen atoms were placed at calculated positions (C–H 0.95–0.98 Å) and were treated as riding on their parent atoms, with *U*(H) set to 1.2*U*~eq~(C). The amino H-atom was refined with a distance restraint of 0.84±0.01 Å.

Figures

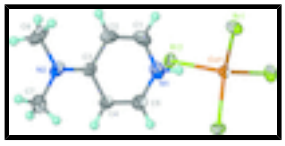

Fig. 1. Anisotropic displacement ellipsoid plot (Barbour, 2001) of the title compound at the 70% probability level. H atoms are drawn as spheres of arbitrary radius.

Bis[4-(dimethylamino)pyridinium] tetrabromidocobaltate(II)

Crystal data

$(C_7H_{11}N_2)_2[CoBr_4]$	F_{000} = 1204
M_r = 624.93	D_x = 2.002 Mg m^{-3}
Monoclinic, *C*2/*c*	Mo *K*α radiation, λ = 0.71073 Å
Hall symbol: -C 2yc	Cell parameters from 6526 reflections
a = 10.4020 (2) Å	θ = 2.5–28.4º
b = 12.1601 (2) Å	μ = 8.54 mm^{-1}
c = 16.9167 (2) Å	*T* = 140 K
β = 104.270 (1)º	Block, blue
V = 2073.76 (6) Å^3	0.40 × 0.35 × 0.30 mm
Z = 4	

Data collection

Bruker SMART APEX diffractometer	2386 independent reflections
Radiation source: fine-focus sealed tube	2228 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.024
T = 140 K	θ_{max} = 27.5º

ω scans	θ_{min} = 2.5º
Absorption correction: Multi-scan (SADABS; Sheldrick, 1996)	h = −11→13
T_{min} = 0.131, T_{max} = 0.184	k = −15→15
8405 measured reflections	l = −21→21

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.021	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2)$ = 0.054	$w = 1/[\sigma^2(F_o^2) + (0.0282P)^2 + 3.2278P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.06	$(\Delta/\sigma)_{max}$ = 0.001
2386 reflections	$\Delta\rho_{max}$ = 0.49 e Å^{-3}
111 parameters	$\Delta\rho_{min}$ = −0.75 e Å^{-3}
1 restraint	Extinction correction: none
Primary atom site location: structure-invariant direct methods	

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.

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

	x	y	z	U_{iso}*/U_{eq}
Br1	0.66045 (2)	0.777934 (17)	0.345907 (13)	0.02380 (7)
Br2	0.36829 (2)	0.555780 (17)	0.314578 (12)	0.02431 (7)
Co1	0.5000	0.67093 (3)	0.2500	0.01636 (9)
N1	0.7776 (2)	0.51482 (16)	0.39578 (13)	0.0268 (4)
H1	0.746 (3)	0.5670 (19)	0.3610 (16)	0.044 (9)*
N2	0.94584 (19)	0.27718 (16)	0.56095 (11)	0.0238 (4)
C1	0.8017 (2)	0.53686 (18)	0.47578 (15)	0.0267 (5)
H1A	0.7809	0.6076	0.4929	0.032*
C2	0.8555 (2)	0.46012 (18)	0.53283 (14)	0.0235 (4)
H2	0.8709	0.4770	0.5892	0.028*
C3	0.88849 (19)	0.35447 (17)	0.50763 (12)	0.0183 (4)
C4	0.8593 (2)	0.33468 (17)	0.42225 (12)	0.0194 (4)
H4	0.8782	0.2650	0.4024	0.023*
C5	0.8047 (2)	0.41504 (19)	0.36914 (13)	0.0243 (4)
H5	0.7851	0.4009	0.3122	0.029*
C6	0.9690 (3)	0.2937 (3)	0.64888 (14)	0.0364 (6)

H6A	0.8871	0.3194	0.6616	0.055*
H6B	1.0390	0.3488	0.6668	0.055*
H6C	0.9966	0.2241	0.6772	0.055*
C7	0.9865 (2)	0.17184 (19)	0.53287 (15)	0.0289 (5)
H7A	1.0364	0.1850	0.4916	0.043*
H7B	0.9076	0.1277	0.5090	0.043*
H7C	1.0426	0.1323	0.5792	0.043*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.01960 (12)	0.02113 (11)	0.02904 (12)	−0.00386 (8)	0.00292 (9)	−0.00728 (8)
Br2	0.03047 (13)	0.02293 (12)	0.02059 (11)	−0.00884 (8)	0.00829 (9)	0.00109 (7)
Co1	0.01504 (19)	0.01626 (17)	0.01751 (18)	0.000	0.00345 (14)	0.000
N1	0.0223 (10)	0.0229 (9)	0.0346 (10)	0.0040 (7)	0.0060 (8)	0.0059 (8)
N2	0.0206 (9)	0.0335 (10)	0.0166 (8)	0.0013 (7)	0.0029 (7)	0.0029 (7)
C1	0.0198 (11)	0.0233 (10)	0.0391 (13)	−0.0010 (8)	0.0110 (9)	−0.0074 (9)
C2	0.0178 (10)	0.0300 (11)	0.0245 (10)	−0.0046 (8)	0.0085 (8)	−0.0094 (8)
C3	0.0112 (9)	0.0249 (10)	0.0190 (9)	−0.0028 (7)	0.0042 (7)	−0.0007 (8)
C4	0.0170 (10)	0.0218 (9)	0.0191 (9)	0.0013 (8)	0.0040 (7)	−0.0012 (7)
C5	0.0214 (10)	0.0290 (11)	0.0212 (10)	0.0004 (9)	0.0030 (8)	0.0005 (8)
C6	0.0301 (13)	0.0608 (17)	0.0166 (10)	−0.0005 (12)	0.0027 (9)	0.0048 (10)
C7	0.0251 (12)	0.0268 (11)	0.0320 (12)	0.0034 (9)	0.0019 (9)	0.0071 (9)

Geometric parameters (Å, °)

Br1—Co1	2.4033 (3)	C2—C3	1.422 (3)
Br2—Co1	2.4019 (3)	C2—H2	0.9500
Co1—Br2^{i}	2.4019 (3)	C3—C4	1.421 (3)
Co1—Br1^{i}	2.4033 (3)	C4—C5	1.354 (3)
N1—C1	1.341 (3)	C4—H4	0.9500
N1—C5	1.348 (3)	C5—H5	0.9500
N1—H1	0.872 (10)	C6—H6A	0.9800
N2—C3	1.337 (3)	C6—H6B	0.9800
N2—C6	1.461 (3)	C6—H6C	0.9800
N2—C7	1.464 (3)	C7—H7A	0.9800
C1—C2	1.360 (3)	C7—H7B	0.9800
C1—H1A	0.9500	C7—H7C	0.9800
Br2—Co1—Br2^{i}	108.678 (18)	C4—C3—C2	116.79 (19)
Br2—Co1—Br1	112.808 (7)	C5—C4—C3	120.1 (2)
Br2^{i}—Co1—Br1	104.098 (8)	C5—C4—H4	119.9
Br2—Co1—Br1^{i}	104.099 (8)	C3—C4—H4	119.9
Br2^{i}—Co1—Br1^{i}	112.808 (7)	N1—C5—C4	121.0 (2)
Br1—Co1—Br1^{i}	114.444 (18)	N1—C5—H5	119.5
C1—N1—C5	121.0 (2)	C4—C5—H5	119.5
C1—N1—H1	119 (2)	N2—C6—H6A	109.5
C5—N1—H1	120 (2)	N2—C6—H6B	109.5

C3—N2—C6	121.6 (2)	H6A—C6—H6B	109.5
C3—N2—C7	120.84 (18)	N2—C6—H6C	109.5
C6—N2—C7	117.52 (19)	H6A—C6—H6C	109.5
N1—C1—C2	121.4 (2)	H6B—C6—H6C	109.5
N1—C1—H1A	119.3	N2—C7—H7A	109.5
C2—C1—H1A	119.3	N2—C7—H7B	109.5
C1—C2—C3	119.6 (2)	H7A—C7—H7B	109.5
C1—C2—H2	120.2	N2—C7—H7C	109.5
C3—C2—H2	120.2	H7A—C7—H7C	109.5
N2—C3—C4	120.97 (19)	H7B—C7—H7C	109.5
N2—C3—C2	122.24 (19)		
C5—N1—C1—C2	0.7 (3)	C1—C2—C3—N2	177.6 (2)
N1—C1—C2—C3	0.7 (3)	C1—C2—C3—C4	−1.6 (3)
C6—N2—C3—C4	−176.2 (2)	N2—C3—C4—C5	−178.1 (2)
C7—N2—C3—C4	3.1 (3)	C2—C3—C4—C5	1.0 (3)
C6—N2—C3—C2	4.7 (3)	C1—N1—C5—C4	−1.2 (3)
C7—N2—C3—C2	−176.0 (2)	C3—C4—C5—N1	0.3 (3)

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

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···Br1	0.87 (1)	2.71 (2)	3.454 (2)	144 (3)

Fig. 1