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Bis(1-adamantylammonium) tetrachloridocobaltate(II)

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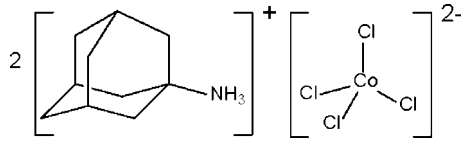
Key indicators: single-crystal X-ray study; T = 295 K; mean σ(C–C) = 0.004 Å; some non-H atoms missing; R factor = 0.041; wR factor = 0.100; data-to-parameter ratio = 20.7.

The Co^{II} atom in the title salt, $(C_{10}H_{18}N)_2[CoCl_4]$, exists in a tetrahedral coordination geometry. The asymmetric unit has two cations that lie on different special positions of site symmetry *m*; the anion lies on another special position of site symmetry *m*.

Related literature

Some amines do not form adducts with cobalt(II) chloride; in the reactions, the amines themselves are protonated. For 1,3-propanediammonium tetrachloridocobaltate, see: Guo *et al.* (1992); for tricyclohexylammonium chloride tetrachlorocobaltate, see: Geiser *et al.* (1984); for 4,4′-bipyridinium tetrachloridocobaltate, see: Barbour *et al.* (1996) and Gillon *et al.* (2000); for bis(4-dimethylamino)pyridinium tetrachloridocobaltate, see: Haddad *et al.* (2003).



Experimental

Crystal data

$(C_{10}H_{18}N)_2[CoCl_4]$
M_r = 505.24
Monoclinic, $C2/m$
a = 30.6005 (6) Å
b = 7.3046 (1) Å
c = 11.0009 (2) Å
β = 104.087 (1)°
V = 2385.02 (7) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 1.18 mm^{-1}
T = 295 (2) K
0.40 × 0.22 × 0.13 mm

Data collection

Bruker APEXII area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.709, T_{max} = 0.862
11019 measured reflections
2946 independent reflections
2115 reflections with $I > 2\sigma(I)$
R_{int} = 0.027

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.041
$wR(F^2)$ = 0.100
S = 1.04
2946 reflections
142 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.52 e Å^{-3}
$\Delta\rho_{min}$ = −0.49 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2E···Cl1^{i}	0.89	2.72	3.437 (3)	139
N2—H2E···Cl1^{i}	0.89	2.72	3.437 (3)	139
N2—H2D···Cl1	0.89	2.43	3.218 (3)	147
N2—H2C···Cl1^{ii}	0.89	2.43	3.218 (3)	148
N1—H1C···Cl1^{ii}	0.89	2.58	3.366 (2)	147
N1—H1B···Cl3^{iii}	0.89	2.46	3.322 (3)	164
N1—H1A···Cl1	0.89	2.58	3.366 (2)	147

Symmetry codes: (i) $-x+\frac{1}{2}, y+\frac{1}{2}, -z$; (ii) $x, -y+1, z$; (iii) $-x+\frac{1}{2}, -y+\frac{1}{2}, -z+1$.

Data collection: *APEX2* (Bruker, 2006); cell refinement: *SAINT* (Bruker, 2006); 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, 2008).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HJ2005).

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Bis(1-adamantylammonium) tetrachloridocobaltate(II)

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S1. Experimental

1-Aminoadamantane (6.05 g, 40 mmol) and salicylaldehyde (5.01 g, 41 mmol) were heated in ethanol (50 ml) for 1 h. The *N*-salicylidene-1-aminoadamantane that separated was collected in 70% yield, m.p. 366 K. Cobalt dichloride hexahydrate (1 mmol) dissolved in ethanol (10 ml) was reacted with the Schiff base (2 mmol) dissolved in alcohol (5 ml) to give a blue solution. Blue crystals separated from the solution after three weeks.

S2. Refinement

H atoms were generated geometrically (C–H 0.97 to 0.98 Å, N–H 0.89 |%A) and were included in the refinement in the riding model approximation, with U(H) set to 1.2 or 1.5U_{eq} of the parent atom.

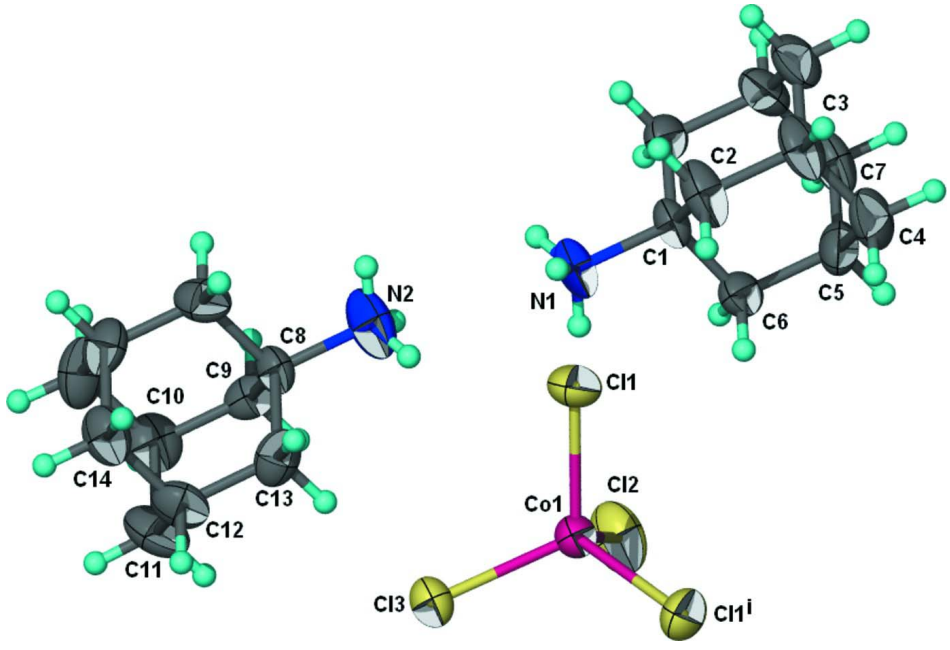

Figure 1

Thermal ellipsoid plot of 2[$C_{10}H_{18}N$] [$CoCl_4$]; displacement ellipsoids are drawn at the 50% probability level, and H atoms as spheres of arbitrary radius. [Symmery code (i) *x*, –*y*, *z*.]

Bis(1-adamantylammonium) tetrachloridocobaltate(II)

Crystal data

$(C_{10}H_{18}N)_2[CoCl_4]$
$M_r = 505.24$
Monoclinic, $C2/m$
Hall symbol: -C 2y
$a = 30.6005\ (6)$ Å
$b = 7.3046\ (1)$ Å
$c = 11.0009\ (2)$ Å
$\beta = 104.087\ (1)°$
$V = 2385.02\ (7)$ Å^3
$Z = 4$
$F(000) = 1060$
$D_x = 1.407$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 3080 reflections
$\theta = 2.6–22.3°$
$\mu = 1.18$ mm^{-1}
$T = 295$ K
Block, blue
0.40 × 0.22 × 0.13 mm

Data collection

Bruker APEXII area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min} = 0.709$, $T_{max} = 0.862$
11019 measured reflections
2946 independent reflections
2115 reflections with $I > 2\sigma(I)$
$R_{int} = 0.027$
$\theta_{max} = 27.5°$, $\theta_{min} = 1.9°$
$h = -28\rightarrow39$
$k = -9\rightarrow7$
$l = -14\rightarrow14$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.041$
$wR(F^2) = 0.100$
$S = 1.05$
2946 reflections
142 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
$w = 1/[\sigma^2(F_o^2) + (0.0368P)^2 + 2.5214P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.52$ e Å^{-3}
$\Delta\rho_{min} = -0.49$ e Å^{-3}

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 $F^2 > \sigma(F^2)$ 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}	Occ. (<1)
N1	0.18030 (9)	0.5000	0.3577 (3)	0.0615 (9)	
H1A	0.1921	0.4006	0.3315	0.092*	0.50
H1B	0.1863	0.4999	0.4410	0.092*	
H1C	0.1921	0.5995	0.3316	0.092*	0.50

C1	0.13007 (10)	0.5000	0.3050 (3)	0.0452 (8)	
C2	0.11951 (12)	0.5000	0.1618 (3)	0.0713 (13)	
H2A	0.1324	0.3923	0.1324	0.086*	0.50
H2B	0.1324	0.6077	0.1324	0.086*	0.50
C3	0.06830 (12)	0.5000	0.1108 (3)	0.0668 (12)	
H3	0.0611	0.5000	0.0190	0.080*	
C4	0.04858 (9)	0.3300 (5)	0.1559 (2)	0.0737 (9)	
H4A	0.0613	0.2214	0.1271	0.088*	
H4B	0.0162	0.3277	0.1221	0.088*	
C5	0.05938 (9)	0.3314 (4)	0.2990 (2)	0.0650 (8)	
H5	0.0465	0.2222	0.3286	0.078*	
C6	0.11079 (8)	0.3304 (4)	0.3507 (2)	0.0568 (7)	
H6A	0.1181	0.3289	0.4415	0.068*	
H6B	0.1236	0.2219	0.3220	0.068*	
C7	0.03975 (12)	0.5000	0.3436 (3)	0.0691 (12)	
H7A	0.0463	0.5000	0.4344	0.083*	
H7B	0.0073	0.5000	0.3116	0.083*	
N2	0.30310 (10)	0.5000	0.1192 (3)	0.0888 (13)	
H2C	0.2904	0.5989	0.1431	0.133*	0.50
H2D	0.2905	0.4000	0.1419	0.133*	0.50
H2E	0.2991	0.5011	0.0363	0.133*	
C8	0.35290 (10)	0.5000	0.1809 (3)	0.0483 (8)	
C9	0.35959 (12)	0.5000	0.3224 (3)	0.0530 (9)	
H9A	0.3457	0.6077	0.3484	0.064*	0.50
H9B	0.3457	0.3923	0.3484	0.064*	0.50
C10	0.40942 (14)	0.5000	0.3820 (3)	0.0678 (11)	
H10	0.4142	0.5000	0.4734	0.081*	
C11	0.43062 (10)	0.3295 (5)	0.3422 (3)	0.0803 (10)	
H11A	0.4167	0.2212	0.3675	0.096*	
H11B	0.4625	0.3266	0.3826	0.096*	
C12	0.42392 (9)	0.3305 (4)	0.1996 (2)	0.0650 (7)	
H12A	0.4377	0.2208	0.1738	0.078*	
C13	0.37368 (9)	0.3305 (4)	0.1389 (2)	0.0608 (7)	
H13A	0.3687	0.3304	0.0483	0.073*	
H13B	0.3598	0.2216	0.1634	0.073*	
C14	0.44527 (13)	0.5000	0.1588 (4)	0.0678 (11)	
H14A	0.4774	0.5000	0.1963	0.081*	
H14B	0.4408	0.5000	0.0684	0.081*	
Co1	0.244385 (15)	0.0000	0.28152 (4)	0.04942 (16)	
Cl1	0.22131 (2)	0.24581 (10)	0.15542 (7)	0.0678 (2)	
Cl2	0.21345 (4)	0.0000	0.44545 (11)	0.0907 (4)	
Cl3	0.32036 (3)	0.0000	0.34080 (8)	0.0561 (2)	

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0332 (15)	0.098 (3)	0.0501 (16)	0.000	0.0051 (12)	0.000
C1	0.0284 (15)	0.067 (2)	0.0389 (15)	0.000	0.0054 (12)	0.000

C2	0.0412 (19)	0.132 (4)	0.0404 (17)	0.000	0.0093 (15)	0.000
C3	0.044 (2)	0.120 (4)	0.0323 (16)	0.000	0.0007 (14)	0.000
C4	0.0519 (15)	0.091 (2)	0.0671 (16)	−0.0051 (16)	−0.0075 (12)	−0.0215 (16)
C5	0.0502 (15)	0.070 (2)	0.0668 (15)	−0.0193 (14)	−0.0016 (12)	0.0114 (14)
C6	0.0507 (14)	0.0539 (17)	0.0582 (14)	0.0007 (12)	−0.0012 (11)	0.0010 (12)
C7	0.0373 (19)	0.120 (4)	0.0495 (19)	0.000	0.0107 (15)	0.000
N2	0.0418 (18)	0.133 (4)	0.092 (3)	0.000	0.0173 (17)	0.000
C8	0.0323 (16)	0.061 (2)	0.0527 (18)	0.000	0.0119 (14)	0.000
C9	0.070 (2)	0.046 (2)	0.0535 (18)	0.000	0.0352 (17)	0.000
C10	0.074 (3)	0.091 (3)	0.0362 (17)	0.000	0.0097 (17)	0.000
C11	0.0753 (19)	0.097 (3)	0.0668 (17)	0.0301 (19)	0.0144 (14)	0.0262 (17)
C12	0.0609 (16)	0.067 (2)	0.0719 (16)	0.0209 (15)	0.0250 (13)	−0.0030 (14)
C13	0.0659 (16)	0.0614 (19)	0.0603 (14)	−0.0134 (14)	0.0253 (12)	−0.0185 (13)
C14	0.047 (2)	0.093 (3)	0.068 (2)	0.000	0.0234 (18)	0.000
Co1	0.0436 (3)	0.0467 (3)	0.0547 (3)	0.000	0.0057 (2)	0.000
Cl1	0.0664 (4)	0.0531 (4)	0.0775 (4)	0.0119 (3)	0.0051 (3)	0.0092 (3)
Cl2	0.0614 (6)	0.1401 (12)	0.0780 (7)	0.000	0.0311 (5)	0.000
Cl3	0.0433 (5)	0.0660 (6)	0.0573 (5)	0.000	0.0090 (4)	0.000

Geometric parameters (Å, º)

N1—C1	1.505 (4)	N2—H2D	0.8900
N1—H1A	0.8900	N2—H2E	0.8900
N1—H1B	0.8900	C8—C13	1.515 (3)
N1—H1C	0.8900	C8—C13^{i}	1.515 (3)
C1—C6^{i}	1.509 (3)	C8—C9	1.520 (4)
C1—C6	1.509 (3)	C9—C10	1.506 (5)
C1—C2	1.530 (4)	C9—H9A	0.9700
C2—C3	1.530 (5)	C9—H9B	0.9700
C2—H2A	0.9700	C10—C11	1.517 (4)
C2—H2B	0.9700	C10—C11^{i}	1.517 (4)
C3—C4^{i}	1.516 (4)	C10—H10	0.9800
C3—C4	1.516 (4)	C11—C12	1.531 (4)
C3—H3	0.9800	C11—H11A	0.9700
C4—C5	1.528 (4)	C11—H11B	0.9700
C4—H4A	0.9700	C12—C14	1.518 (4)
C4—H4B	0.9700	C12—C13	1.520 (4)
C5—C7	1.504 (4)	C12—H12A	0.9800
C5—C6	1.537 (3)	C13—H13A	0.9700
C5—H5	0.9800	C13—H13B	0.9700
C6—H6A	0.9700	C14—C12^{i}	1.518 (4)
C6—H6B	0.9700	C14—H14A	0.9700
C7—C5^{i}	1.504 (4)	C14—H14B	0.9700
C7—H7A	0.9700	Co1—Cl2	2.2313 (11)
C7—H7B	0.9700	Co1—Cl3	2.2567 (9)
N2—C8	1.510 (4)	Co1—Cl1	2.2738 (7)
N2—H2C	0.8900	Co1—Cl1^{ii}	2.2738 (7)

C1—N1—H1A	109.5	C8—N2—H2E	109.5
C1—N1—H1B	109.5	H2C—N2—H2E	109.5
H1A—N1—H1B	109.5	H2D—N2—H2E	109.5
C1—N1—H1C	109.5	N2—C8—C13	108.41 (19)
H1A—N1—H1C	109.5	N2—C8—C13^{i}	108.41 (19)
H1B—N1—H1C	109.5	C13—C8—C13^{i}	109.6 (3)
N1—C1—C6^{i}	108.50 (17)	N2—C8—C9	109.3 (3)
N1—C1—C6	108.50 (17)	C13—C8—C9	110.53 (19)
C6^{i}—C1—C6	110.3 (3)	C13^{i}—C8—C9	110.53 (19)
N1—C1—C2	109.7 (2)	C10—C9—C8	108.4 (3)
C6^{i}—C1—C2	109.94 (17)	C10—C9—H9A	110.0
C6—C1—C2	109.94 (17)	C8—C9—H9A	110.0
C1—C2—C3	108.5 (3)	C10—C9—H9B	110.0
C1—C2—H2A	110.0	C8—C9—H9B	110.0
C3—C2—H2A	110.0	H9A—C9—H9B	108.4
C1—C2—H2B	110.0	C9—C10—C11	109.4 (2)
C3—C2—H2B	110.0	C9—C10—C11^{i}	109.4 (2)
H2A—C2—H2B	108.4	C11—C10—C11^{i}	110.3 (4)
C4^{i}—C3—C4	110.0 (3)	C9—C10—H10	109.2
C4^{i}—C3—C2	109.49 (19)	C11—C10—H10	109.2
C4—C3—C2	109.49 (19)	C11^{i}—C10—H10	109.2
C4^{i}—C3—H3	109.3	C10—C11—C12	109.4 (2)
C4—C3—H3	109.3	C10—C11—H11A	109.8
C2—C3—H3	109.3	C12—C11—H11A	109.8
C3—C4—C5	109.2 (2)	C10—C11—H11B	109.8
C3—C4—H4A	109.8	C12—C11—H11B	109.8
C5—C4—H4A	109.8	H11A—C11—H11B	108.2
C3—C4—H4B	109.8	C14—C12—C13	109.4 (3)
C5—C4—H4B	109.8	C14—C12—C11	110.3 (3)
H4A—C4—H4B	108.3	C13—C12—C11	108.6 (2)
C7—C5—C4	109.8 (3)	C14—C12—H12A	109.5
C7—C5—C6	109.7 (2)	C13—C12—H12A	109.5
C4—C5—C6	109.0 (2)	C11—C12—H12A	109.5
C7—C5—H5	109.4	C8—C13—C12	108.7 (2)
C4—C5—H5	109.4	C8—C13—H13A	109.9
C6—C5—H5	109.4	C12—C13—H13A	109.9
C1—C6—C5	108.7 (2)	C8—C13—H13B	109.9
C1—C6—H6A	109.9	C12—C13—H13B	109.9
C5—C6—H6A	109.9	H13A—C13—H13B	108.3
C1—C6—H6B	109.9	C12^{i}—C14—C12	109.3 (3)
C5—C6—H6B	109.9	C12^{i}—C14—H14A	109.8
H6A—C6—H6B	108.3	C12—C14—H14A	109.8
C5—C7—C5^{i}	109.9 (3)	C12^{i}—C14—H14B	109.8
C5—C7—H7A	109.7	C12—C14—H14B	109.8
C5^{i}—C7—H7A	109.7	H14A—C14—H14B	108.3
C5—C7—H7B	109.7	Cl2—Co1—Cl3	112.11 (4)
C5^{i}—C7—H7B	109.7	Cl2—Co1—Cl1	111.30 (3)
H7A—C7—H7B	108.2	Cl3—Co1—Cl1	108.75 (3)

C8—N2—H2C	109.5	Cl2—Co1—Cl1^{ii}	111.30 (3)
C8—N2—H2D	109.5	Cl3—Co1—Cl1^{ii}	108.74 (3)
H2C—N2—H2D	109.5	Cl1—Co1—Cl1^{ii}	104.31 (4)

Symmetry codes: (i) x, $-y+1$, z; (ii) x, $-y$, z.

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2*E*···Cl1^{iii}	0.89	2.72	3.437 (3)	139
N2—H2*E*···Cl1^{iii}	0.89	2.72	3.437 (3)	139
N2—H2*D*···Cl1	0.89	2.43	3.218 (3)	147
N2—H2*C*···Cl1^{i}	0.89	2.43	3.218 (3)	148
N1—H1*C*···Cl1^{i}	0.89	2.58	3.366 (2)	147
N1—H1*B*···Cl3^{iv}	0.89	2.46	3.322 (3)	164
N1—H1*A*···Cl1	0.89	2.58	3.366 (2)	147

Symmetry codes: (i) x, $-y+1$, z; (iii) $-x+1/2$, $y+1/2$, $-z$; (iv) $-x+1/2$, $-y+1/2$, $-z+1$.