Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

N,*N*,*N*′,*N*′-Tetramethyl-*N*,*N*′-dipropylethane-1,2-diaminium tetrachloridocobaltate(II)

Sari M. Närhi, Jatta Kostamo, Janne Asikkala, Raija Oilunkaniemi* and Risto S. Laitinen

Department of Chemistry, PO Box 3000, FI-90014 University of Oulu, Finland
Correspondence e-mail: raija.oilunkaniemi@oulu.fi

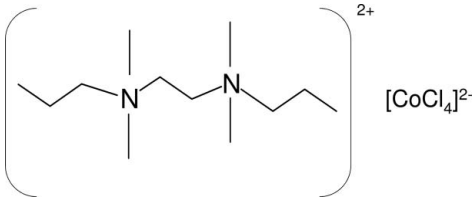
Received 9 November 2011; accepted 25 November 2011

Key indicators: single-crystal X-ray study; T = 120 K; mean σ(C–C) = 0.004 Å; R factor = 0.041; wR factor = 0.107; data-to-parameter ratio = 19.8.

The crystal structure of the title compound, $(C_{12}H_{30}N_2)[CoCl_4]$, is composed of discrete $(C_{12}H_{30}N_2)^{2+}$ cations and $[CoCl_4]^{2-}$ anions. The asymmetric unit contains a half-cation and a half-anion. The atoms of the cation occupy general positions about an inversion centre, which is located at the midpoint of the central C—C bond. The Co atoms lie on a twofold rotation axis. The slightly distorted tetrahedral coordination environment around the metal atom consists of two Cl atoms and their symmetry-related pairs.

Related literature

For the synthesis and structural characterization of $C_{12}H_{30}N_2^{2+}\cdot Cl_2^{2-}$, see: Närhi *et al.* (2011).



Experimental

Crystal data

$(C_{12}H_{30}N_2)[CoCl_4]$
M_r = 403.11
Monoclinic, $C2/c$
a = 13.583 (3) Å
b = 9.2334 (18) Å
c = 14.981 (3) Å
β = 101.83 (3)°
V = 1839.1 (6) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 1.51 mm^{-1}
T = 120 K
0.25 × 0.20 × 0.10 mm

Data collection

Bruker–Nonius KappaCCD diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2008*a*)
T_{min} = 0.705, T_{max} = 0.864
11798 measured reflections
1799 independent reflections
1596 reflections with $I > 2\sigma(I)$
R_{int} = 0.098

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.041
$wR(F^2)$ = 0.107
S = 1.08
1799 reflections
91 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.41 e Å^{-3}
$\Delta\rho_{min}$ = −0.45 e Å^{-3}

Table 1
Selected bond lengths (Å).

Co1—Cl1	2.2731 (9)	Co1—Cl2	2.2759 (8)

Data collection: *COLLECT* (Bruker, 2008); cell refinement: *DENZO-SMN* (Otwinowski & Minor, 1997); data reduction: *DENZO-SMN*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008*b*); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008*b*); molecular graphics: *DIAMOND* (Brandenburg, 1999); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

Financial support from the Academy of Finland is gratefully acknowledged.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HP2021).

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supporting information

Acta Cryst. (2011). E**67**, m1887 [https://doi.org/10.1107/S1600536811050744]

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S1. Comment

The asymmetric unit of $(C_{12}H_{30}N_2)[CoCl_4]$ consists of half of the cation and half of the anion (see Fig. 1). The N—C bond lengths in the cation range from 1.501 (3) to 1.530 (3) Å and the C—C bond lengths from 1.509 (4) to 1.525 (4) Å. These can be compared to the bond lengths in the related chloride and bromide (Närhi *et al.* 2011). In the title compound, the two *n*-propyl chains are almost coplanar with the N1—C1—C1^{ii}—N1^{ii} skeleton with all torsional angles *ca* 180 °, whereas in $(C_{12}H_{30}N_2)Cl_2$ and $(C_{12}H_{30}N_2)Br_2$ the *n*-propyl chains are in the *anti*-configuration with respect to the corresponding NCCN skeleton (Närhi *et al.* 2011). The cobalt atom shows a slightly distorted tetrahedral coordination geometry and the Co—Cl bond lengths of 2.2731 (9) Å and 2.2759 (8) Å are quite normal.

The packing of the title compound consists of layers of cations. The isolated anions lay between these layers with several hydrogen bonds connecting the anions and cations, as shown in Fig. 2. The packing of the molecules is shown in Fig. 3.

S2. Experimental

Addition of solution of $(C_{12}H_{30}N_2)Cl_2$ (0.118 g, 0.432 mmol) in 5 ml MeOH to solution of $CoCl_2 \cdot 6\ H_2O$ (0.103 g, 0.433 mmol) in 5 ml MeOH gave a purple solution from which the title compound was obtained as crystalline blue precipitate.

S3. Refinement

H atoms were positioned geometrically and refined using a riding model with C—H = 0.99 Å and with U_{iso}(H) = 1.2 U_{eq}(C) and 0.98 Å and U_{iso}(H) = 1.2 U_{eq}(C)for the methylene and methyl H atoms, respectively.

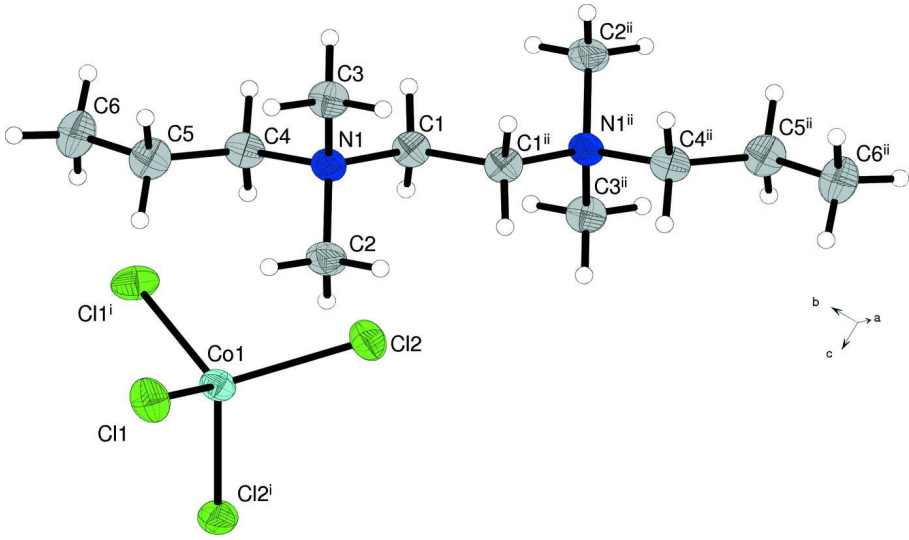

Figure 1

The molecular structure of the title compound indicating the numbering of the atoms. The thermal ellipsoids have been drawn at 50% probability. Symmetry code: (i) = -*x*, *y*, 0.5 - *z* (ii) = 0.5 - *x*, 0.5 - *y*, -*z*.

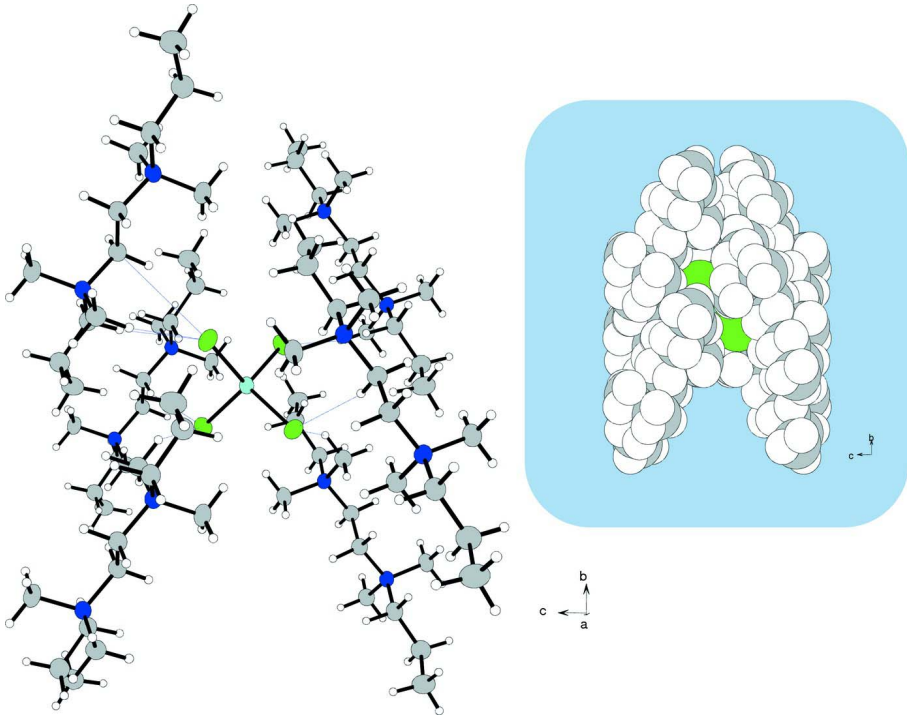

Figure 2

The closest contacts between an anion and the closest cations. The spacefilling presentation is shown in the insert.

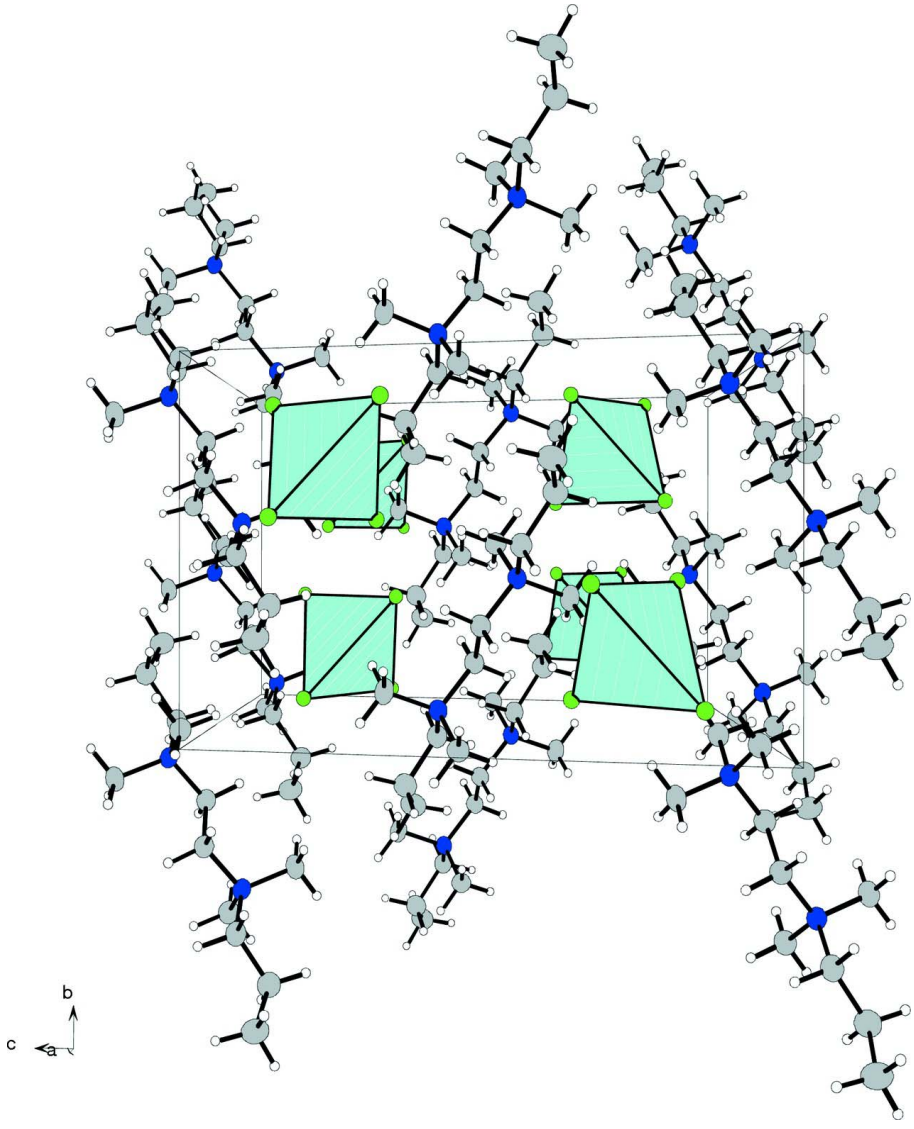

Figure 3

The packing of the molecules viewed along the *a* axis.

N,*N*,*N*′,*N*′-Tetramethyl-*N*,*N*′- dipropylethane-1,2-diaminium tetrachloridocobaltate(II)

Crystal data

$(C_{12}H_{30}N_2)[CoCl_4]$
$M_r = 403.11$
Monoclinic, *C*2/*c*
Hall symbol: -C 2yc
$a = 13.583\ (3)$ Å
$b = 9.2334\ (18)$ Å
$c = 14.981\ (3)$ Å
$\beta = 101.83\ (3)°$
$V = 1839.1\ (6)$ Å^3
$Z = 4$
$F(000) = 844$
$D_x = 1.456$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 1596 reflections
$\theta = 3.1–26.0°$
$\mu = 1.51$ mm^{-1}
$T = 120$ K
Plate, blue
0.25 × 0.20 × 0.10 mm

Data collection

Bruker–Nonius KappaCCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ scans, and ω scans with κ offsets
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2008*a*)
T_{min} = 0.705, T_{max} = 0.864
11798 measured reflections
1799 independent reflections
1596 reflections with $I > 2\sigma(I)$
R_{int} = 0.098
θ_{max} = 26.0°, θ_{min} = 3.1°
h = −15→16
k = −11→11
l = −18→18

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.041
$wR(F^2)$ = 0.107
S = 1.08
1799 reflections
91 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
$w = 1/[\sigma^2(F_o^2) + (0.0551P)^2 + 2.1498P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.41 e Å^{-3}
$\Delta\rho_{min}$ = −0.45 e Å^{-3}
Extinction correction: *SHELXL97* (Sheldrick, 2008), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.0131 (12)

Special details

Geometry. All s.u.'s (except the s.u. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell s.u.'s are taken into account individually in the estimation of s.u.'s in distances, angles and torsion angles; correlations between s.u.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell s.u.'s is used for estimating s.u.'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 $F^2 > 2\sigma(F^2)$ 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}
Co1	0.0000	0.27789 (5)	0.2500	0.0254 (2)
Cl1	0.12341 (5)	0.42242 (8)	0.32854 (5)	0.0369 (2)
Cl2	0.06661 (5)	0.13400 (8)	0.15413 (5)	0.0348 (2)
N1	0.24693 (16)	0.4259 (2)	0.07011 (14)	0.0271 (5)
C1	0.2084 (2)	0.3026 (3)	0.00547 (18)	0.0289 (6)
H1A	0.1775	0.3430	−0.0550	0.035*
H1B	0.1556	0.2494	0.0288	0.035*
C2	0.2940 (2)	0.3728 (3)	0.16405 (18)	0.0346 (7)
H2A	0.3542	0.3158	0.1612	0.052*
H2B	0.2457	0.3122	0.1874	0.052*
H2C	0.3129	0.4559	0.2047	0.052*
C3	0.3208 (2)	0.5173 (3)	0.03321 (19)	0.0322 (6)
H3A	0.3404	0.6007	0.0734	0.048*
H3B	0.2897	0.5514	−0.0280	0.048*
H3C	0.3805	0.4595	0.0302	0.048*

C4	0.15188 (19)	0.5122 (3)	0.07434 (18)	0.0309 (6)
H4A	0.1194	0.5404	0.0114	0.037*
H4B	0.1047	0.4474	0.0975	0.037*
C5	0.1662 (2)	0.6471 (3)	0.1325 (2)	0.0379 (7)
H5A	0.2160	0.7117	0.1130	0.046*
H5B	0.1915	0.6209	0.1972	0.046*
C6	0.0652 (2)	0.7244 (3)	0.1219 (2)	0.0420 (7)
H6A	0.0388	0.7451	0.0573	0.063*
H6B	0.0743	0.8154	0.1563	0.063*
H6C	0.0177	0.6625	0.1454	0.063*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Co1	0.0235 (3)	0.0307 (3)	0.0215 (3)	0.000	0.00327 (19)	0.000
Cl1	0.0295 (4)	0.0445 (5)	0.0335 (4)	−0.0022 (3)	−0.0012 (3)	−0.0116 (3)
Cl2	0.0375 (4)	0.0359 (4)	0.0341 (4)	−0.0039 (3)	0.0148 (3)	−0.0067 (3)
N1	0.0294 (11)	0.0279 (12)	0.0234 (11)	−0.0014 (9)	0.0042 (8)	−0.0016 (9)
C1	0.0251 (13)	0.0304 (14)	0.0292 (14)	−0.0022 (11)	0.0009 (10)	−0.0011 (11)
C2	0.0360 (15)	0.0416 (16)	0.0249 (13)	0.0076 (12)	0.0027 (11)	0.0036 (12)
C3	0.0308 (14)	0.0323 (15)	0.0330 (14)	−0.0063 (11)	0.0056 (11)	−0.0002 (11)
C4	0.0276 (13)	0.0348 (15)	0.0293 (14)	−0.0027 (11)	0.0037 (10)	0.0000 (11)
C5	0.0345 (15)	0.0369 (17)	0.0397 (16)	−0.0003 (12)	0.0014 (12)	−0.0016 (13)
C6	0.0363 (17)	0.0354 (17)	0.0523 (19)	0.0036 (12)	0.0044 (14)	−0.0043 (14)

Geometric parameters (Å, º)

Co1—Cl1	2.2731 (9)	C2—H2C	0.9800
Co1—Cl1^{i}	2.2731 (9)	C3—H3A	0.9800
Co1—Cl2	2.2759 (8)	C3—H3B	0.9800
Co1—Cl2^{i}	2.2759 (8)	C3—H3C	0.9800
N1—C3	1.501 (3)	C4—C5	1.509 (4)
N1—C2	1.504 (3)	C4—H4A	0.9900
N1—C1	1.516 (3)	C4—H4B	0.9900
N1—C4	1.530 (3)	C5—C6	1.525 (4)
C1—C1^{ii}	1.524 (5)	C5—H5A	0.9900
C1—H1A	0.9900	C5—H5B	0.9900
C1—H1B	0.9900	C6—H6A	0.9800
C2—H2A	0.9800	C6—H6B	0.9800
C2—H2B	0.9800	C6—H6C	0.9800
Cl1—Co1—Cl1^{i}	108.10 (5)	N1—C3—H3A	109.5
Cl1—Co1—Cl2	108.84 (3)	N1—C3—H3B	109.5
Cl1^{i}—Co1—Cl2	111.25 (3)	H3A—C3—H3B	109.5
Cl1—Co1—Cl2^{i}	111.25 (3)	N1—C3—H3C	109.5
Cl1^{i}—Co1—Cl2^{i}	108.84 (3)	H3A—C3—H3C	109.5
Cl2—Co1—Cl2^{i}	108.57 (4)	H3B—C3—H3C	109.5
C3—N1—C2	109.9 (2)	C5—C4—N1	116.4 (2)

C3—N1—C1	110.8 (2)	C5—C4—H4A	108.2
C2—N1—C1	112.2 (2)	N1—C4—H4A	108.2
C3—N1—C4	110.9 (2)	C5—C4—H4B	108.2
C2—N1—C4	109.4 (2)	N1—C4—H4B	108.2
C1—N1—C4	103.54 (19)	H4A—C4—H4B	107.3
N1—C1—C1^{ii}	112.4 (3)	C4—C5—C6	108.7 (2)
N1—C1—H1A	109.1	C4—C5—H5A	110.0
C1^{ii}—C1—H1A	109.1	C6—C5—H5A	110.0
N1—C1—H1B	109.1	C4—C5—H5B	110.0
C1^{ii}—C1—H1B	109.1	C6—C5—H5B	110.0
H1A—C1—H1B	107.9	H5A—C5—H5B	108.3
N1—C2—H2A	109.5	C5—C6—H6A	109.5
N1—C2—H2B	109.5	C5—C6—H6B	109.5
H2A—C2—H2B	109.5	H6A—C6—H6B	109.5
N1—C2—H2C	109.5	C5—C6—H6C	109.5
H2A—C2—H2C	109.5	H6A—C6—H6C	109.5
H2B—C2—H2C	109.5	H6B—C6—H6C	109.5
C3—N1—C1—C1^{ii}	63.7 (3)	C2—N1—C4—C5	61.9 (3)
C2—N1—C1—C1^{ii}	−59.5 (3)	C1—N1—C4—C5	−178.3 (2)
C4—N1—C1—C1^{ii}	−177.4 (3)	N1—C4—C5—C6	175.3 (2)
C3—N1—C4—C5	−59.4 (3)		

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