

Bis(diethylenetriamine)cobalt(III) hexachloridoindate(III)

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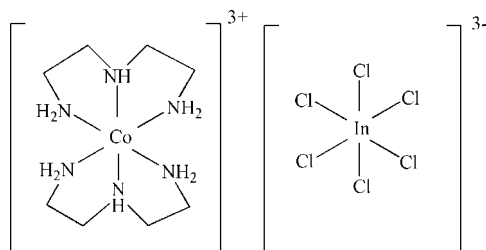
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Key indicators: single-crystal X-ray study; $T = 296$ K; mean $\sigma(\text{C}-\text{C}) = 0.003$ Å; R factor = 0.018; wR factor = 0.046; data-to-parameter ratio = 22.5.

The title compound, $[\text{Co}(\text{C}_4\text{H}_{13}\text{N}_3)_2][\text{InCl}_6]$, was synthesized under hydrothermal conditions. In the cation, the Co—N bond lengths lie in the range 1.967 (2)–1.9684 (15) Å. In the anion, the In^{III} atom is coordinated by six Cl atoms resulting in a slightly distorted octahedral geometry. Both metal atoms are located on special positions of site symmetry $2/m$. Furthermore, one Cl atom and one N atom are located on a mirror plane. N—H...Cl hydrogen bonds between cations and anions consolidate the crystal packing.

Related literature

For the use of chiral metal complexes as templates in the synthesis of open-framework metal phosphates and germanates, see: Stalder & Wilkinson (1997); Wang *et al.* (2003*a,b*); Pan *et al.* (2005, 2008). For the introduction of chiral metal complexes into coordination polymers, see: Pan *et al.* (2010*a,b*, 2011); Tong & Pan (2011). For In—Cl bond lengths in other hexachloridoindium compounds, see: Rothammel *et al.* (1998).



Experimental

Crystal data

$[\text{Co}(\text{C}_4\text{H}_{13}\text{N}_3)_2][\text{InCl}_6]$
 $M_r = 592.80$
 Orthorhombic, *Cccm*
 $a = 10.8925$ (5) Å
 $b = 14.7291$ (7) Å
 $c = 12.2205$ (6) Å

$V = 1960.62$ (16) Å³
 $Z = 4$
 Mo $K\alpha$ radiation
 $\mu = 2.84$ mm⁻¹
 $T = 296$ K
 $0.20 \times 0.18 \times 0.15$ mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
 Absorption correction: multi-scan (*SADABS*; Bruker, 2002)
 $T_{\text{min}} = 0.572$, $T_{\text{max}} = 0.653$

6910 measured reflections
 1282 independent reflections
 1139 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.021$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.018$
 $wR(F^2) = 0.046$
 $S = 1.06$
 1282 reflections

57 parameters
 H-atom parameters constrained
 $\Delta\rho_{\text{max}} = 0.34$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.37$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
N1—H1A...Cl ⁱ	0.90	2.62	3.4915 (17)	164
N1—H1B...Cl ⁱⁱ	0.90	2.61	3.3823 (18)	144
N1—H1B...Cl ⁱⁱⁱ	0.90	2.72	3.3957 (17)	133
N2—H2...Cl ^{iv}	0.91	2.79	3.5407 (19)	141
N2—H2...Cl ^v	0.91	2.79	3.5407 (19)	141

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

Data collection: *APEX2* (Bruker, 2002); cell refinement: *SAINT* (Bruker, 2002); data reduction: *SAINT*; 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*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: VM2092).

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supporting information

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S1. Comment

Chiral metal complexes are interesting templates for the synthesis of novel materials, because they are versatile and can be made with a wide of shapes, charges and particularly chirality. As templates, they have been used in the synthesis of open-framework metal phosphates and germanates, (for example: Stalder & Wilkinson, 1997; Wang *et al.*, 2003a; Pan *et al.*, 2005, 2008) and a new concept of chirality transfer of the chiral metal complex into the inorganic framework has been demonstrated (Wang *et al.*, 2003b). Recently, Pan *et al.* introduced the chiral metal complexes into coordination polymers (Pan *et al.*, 2010a, 2010b, 2011; Tong & Pan, 2011).

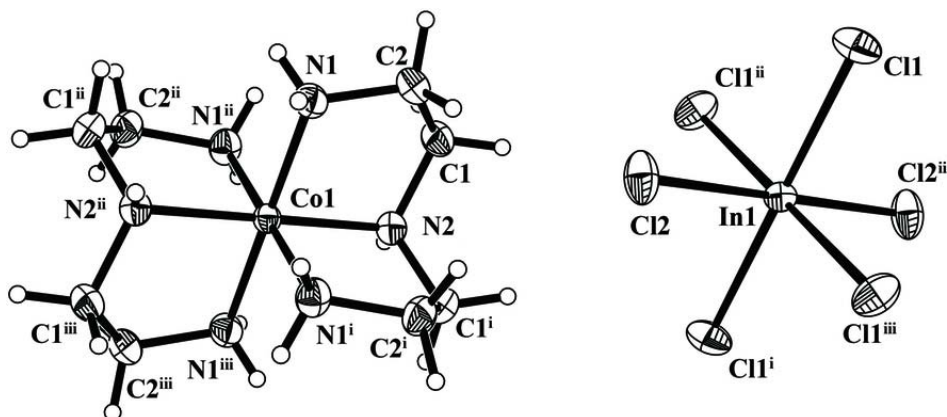
In this paper, we present a new hexachloro-indium templated by the metal complex $[\text{Co}(\text{dien})_2]^{3+}$. As shown in Fig. 1, the crystal structure consists of discrete $[\text{InCl}_6]^{3-}$ anions and $[\text{Co}(\text{dien})_2]^{3+}$ cations. In $[\text{InCl}_6]^{3-}$, the indium center is coordinated by six Cl atoms, resulting in a slightly distorted octahedral geometry. The In—Cl bond distances are in the range of 2.5024 (5)–2.5114 (7) Å, which is consistent with other hexachloro-indium compounds (Rothammel *et al.*, 1998). In $[\text{Co}(\text{dien})_2]^{3+}$, the cobalt center also displays a slightly distorted octahedral geometry and is bonded to six N atoms of two diethylenetriamines with the Co—N distances of 1.967 (2)–1.9684 (15) Å.

S2. Experimental

In a typical synthesis, a mixture of $\text{InCl}_3 \cdot 4\text{H}_2\text{O}$ (1 mmol), H_3PO_4 (4 mmol) $\text{Co}(\text{dien})_2\text{Cl}_3$ (0.25 mmol) and H_2O (10 ml) was added to a 20 ml Teflon-lined reactor under autogenous pressure at 100 °C for 3 days. Yellow block crystals were obtained.

S3. Refinement

All H atoms were positioned geometrically (C—H = 0.97 Å and N—H = 0.90–0.91 Å) and allowed to ride on their parent atoms, with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{parent atom})$.

**Figure 1**

A view of the structure of complex. Ellipsoids are drawn at the 50% probability level. [Symmetry codes: (i) $x, y, -z$; (ii) $-1/2 - x, -1/2 - y, z$; (iii) $-1/2 - x, -1/2 - y, -z$.]

Bis(diethylenetriamine)cobalt(III) hexachloridoindate(III)

Crystal data

$[\text{Co}(\text{C}_4\text{H}_{13}\text{N}_3)_2][\text{InCl}_6]$

$M_r = 592.80$

Orthorhombic, $Cccm$

Hall symbol: $-C 2 2 c$

$a = 10.8925 (5) \text{ \AA}$

$b = 14.7291 (7) \text{ \AA}$

$c = 12.2205 (6) \text{ \AA}$

$V = 1960.62 (16) \text{ \AA}^3$

$Z = 4$

$F(000) = 1176$

$D_x = 2.008 \text{ Mg m}^{-3}$

Mo $K\alpha$ radiation, $\lambda = 0.71073 \text{ \AA}$

$\theta = 2.3\text{--}28.3^\circ$

$\mu = 2.84 \text{ mm}^{-1}$

$T = 296 \text{ K}$

Block, yellow

$0.2 \times 0.18 \times 0.15 \text{ mm}$

Data collection

Bruker SMART APEX CCD area-detector diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

Detector resolution: $5.00 \text{ cm pixels mm}^{-1}$

φ and ω scans

Absorption correction: multi-scan

(*SADABS*; Bruker, 2002)

$T_{\min} = 0.572$, $T_{\max} = 0.653$

6910 measured reflections

1282 independent reflections

1139 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.021$

$\theta_{\max} = 28.3^\circ$, $\theta_{\min} = 2.3^\circ$

$h = -14 \rightarrow 11$

$k = -19 \rightarrow 18$

$l = -12 \rightarrow 16$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.018$

$wR(F^2) = 0.046$

$S = 1.06$

1282 reflections

57 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

$w = 1/[\sigma^2(F_o^2) + (0.0179P)^2 + 3.2797P]$

where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} < 0.001$

$\Delta\rho_{\max} = 0.34 \text{ e \AA}^{-3}$

$\Delta\rho_{\min} = -0.37 \text{ e \AA}^{-3}$

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 (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
In1	−0.2500	−0.2500	0.0000	0.02018 (8)
Co1	−0.2500	0.2500	0.0000	0.01769 (10)
Cl1	−0.38483 (5)	−0.31981 (4)	0.14285 (4)	0.03547 (12)
Cl2	−0.38713 (6)	−0.11293 (4)	0.0000	0.03569 (17)
N1	−0.35382 (15)	0.19629 (10)	0.11486 (13)	0.0265 (3)
H1A	−0.3447	0.2282	0.1772	0.080*
H1B	−0.4331	0.1999	0.0945	0.080*
N2	−0.1606 (2)	0.13392 (14)	0.0000	0.0228 (4)
H2	−0.0789	0.1466	0.0000	0.080*
C1	−0.18921 (19)	0.08352 (13)	0.10310 (16)	0.0289 (4)
H1C	−0.1353	0.1041	0.1612	0.080*
H1D	−0.1753	0.0191	0.0921	0.080*
C2	−0.32142 (19)	0.09943 (13)	0.13544 (17)	0.0295 (4)
H2A	−0.3748	0.0601	0.0931	0.080*
H2B	−0.3326	0.0853	0.2123	0.080*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
In1	0.01848 (12)	0.02348 (13)	0.01860 (12)	0.00027 (9)	0.000	0.000
Co1	0.0176 (2)	0.0165 (2)	0.0190 (2)	−0.00096 (17)	0.000	0.000
Cl1	0.0303 (2)	0.0454 (3)	0.0307 (2)	−0.0003 (2)	0.0080 (2)	0.0119 (2)
Cl2	0.0245 (3)	0.0231 (3)	0.0595 (5)	0.0016 (2)	0.000	0.000
N1	0.0266 (8)	0.0242 (7)	0.0286 (8)	−0.0011 (6)	0.0058 (6)	0.0023 (6)
N2	0.0220 (10)	0.0210 (10)	0.0255 (11)	0.0000 (8)	0.000	0.000
C1	0.0342 (10)	0.0247 (9)	0.0277 (9)	0.0031 (8)	−0.0027 (8)	0.0049 (7)
C2	0.0352 (11)	0.0234 (9)	0.0298 (10)	−0.0029 (8)	0.0031 (8)	0.0056 (7)

Geometric parameters (\AA , $^\circ$)

In1—Cl1 ⁱ	2.5024 (5)	N1—C2	1.491 (2)
In1—Cl1 ⁱⁱ	2.5024 (5)	N1—H1A	0.9000
In1—Cl1	2.5024 (5)	N1—H1B	0.9000
In1—Cl1 ⁱⁱⁱ	2.5024 (5)	N2—C1 ⁱ	1.495 (2)
In1—Cl2 ⁱⁱⁱ	2.5114 (7)	N2—C1	1.495 (2)
In1—Cl2	2.5114 (7)	N2—H2	0.9100

Co1—N2	1.967 (2)	C1—C2	1.512 (3)
Co1—N2 ^{iv}	1.967 (2)	C1—H1C	0.9700
Co1—N1 ⁱ	1.9684 (15)	C1—H1D	0.9700
Co1—N1 ^v	1.9684 (15)	C2—H2A	0.9700
Co1—N1 ^{iv}	1.9684 (15)	C2—H2B	0.9700
Co1—N1	1.9684 (15)		
Cl1 ⁱ —In1—Cl1 ⁱⁱ	180.0	N1 ⁱ —Co1—N1	90.97 (10)
Cl1 ⁱ —In1—Cl1	88.48 (3)	N1 ^v —Co1—N1	89.03 (10)
Cl1 ⁱⁱ —In1—Cl1	91.52 (3)	N1 ^{iv} —Co1—N1	180.00 (8)
Cl1 ⁱ —In1—Cl1 ⁱⁱⁱ	91.52 (3)	C2—N1—Co1	111.65 (12)
Cl1 ⁱⁱ —In1—Cl1 ⁱⁱⁱ	88.48 (3)	C2—N1—H1A	109.3
Cl1—In1—Cl1 ⁱⁱⁱ	180.0	Co1—N1—H1A	109.3
Cl1 ⁱ —In1—Cl2 ⁱⁱⁱ	91.076 (16)	C2—N1—H1B	109.3
Cl1 ⁱⁱ —In1—Cl2 ⁱⁱⁱ	88.924 (17)	Co1—N1—H1B	109.3
Cl1—In1—Cl2 ⁱⁱⁱ	91.076 (16)	H1A—N1—H1B	108.0
Cl1 ⁱⁱⁱ —In1—Cl2 ⁱⁱⁱ	88.924 (17)	C1 ⁱ —N2—C1	114.8 (2)
Cl1 ⁱ —In1—Cl2	88.924 (17)	C1 ⁱ —N2—Co1	109.18 (12)
Cl1 ⁱⁱ —In1—Cl2	91.076 (16)	C1—N2—Co1	109.18 (12)
Cl1—In1—Cl2	88.924 (17)	C1 ⁱ —N2—H2	107.8
Cl1 ⁱⁱⁱ —In1—Cl2	91.076 (16)	C1—N2—H2	107.8
Cl2 ⁱⁱⁱ —In1—Cl2	180.0	Co1—N2—H2	107.8
N2—Co1—N2 ^{iv}	180.0	N2—C1—C2	109.98 (16)
N2—Co1—N1 ⁱ	86.27 (6)	N2—C1—H1C	109.7
N2 ^{iv} —Co1—N1 ⁱ	93.73 (6)	C2—C1—H1C	109.7
N2—Co1—N1 ^v	93.73 (6)	N2—C1—H1D	109.7
N2 ^{iv} —Co1—N1 ^v	86.27 (6)	C2—C1—H1D	109.7
N1 ⁱ —Co1—N1 ^v	180.00 (10)	H1C—C1—H1D	108.2
N2—Co1—N1 ^{iv}	93.73 (6)	N1—C2—C1	109.24 (15)
N2 ^{iv} —Co1—N1 ^{iv}	86.27 (6)	N1—C2—H2A	109.8
N1 ⁱ —Co1—N1 ^{iv}	89.03 (10)	C1—C2—H2A	109.8
N1 ^v —Co1—N1 ^{iv}	90.97 (10)	N1—C2—H2B	109.8
N2—Co1—N1	86.27 (6)	C1—C2—H2B	109.8
N2 ^{iv} —Co1—N1	93.73 (6)	H2A—C2—H2B	108.3

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

Hydrogen-bond geometry ($\text{\AA}, ^\circ$)

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
N1—H1A \cdots Cl1 ^{vi}	0.90	2.62	3.4915 (17)	164
N1—H1B \cdots Cl2 ^{vii}	0.90	2.61	3.3823 (18)	144
N1—H1B \cdots Cl1 ^{viii}	0.90	2.72	3.3957 (17)	133
N2—H2 \cdots Cl1 ^{ix}	0.91	2.79	3.5407 (19)	141
N2—H2 \cdots Cl1 ^x	0.91	2.79	3.5407 (19)	141

Symmetry codes: (vi) $x, -y, -z+1/2$; (vii) $-x-1, -y, -z$; (viii) $-x-1, -y, z$; (ix) $x+1/2, y+1/2, z$; (x) $x+1/2, y+1/2, -z$.