

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

Qihui Wu, Shangwen Chen, Cailing Zhang, Xia Zhi and Zelin Chen*

Department of Materials and Chemical Engineering, Ministry of Education Key Laboratory of Application Technology of Hainan, Superior Resources Chemical Materials, Hainan University, Haikou 570228, Hainan Province, People's Republic of China

Correspondence e-mail: czl69995@163.com

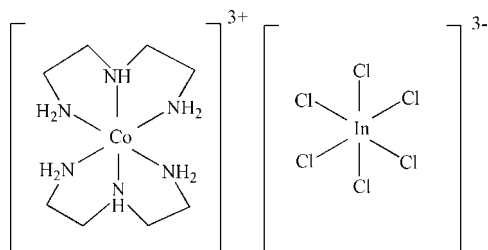
Received 26 April 2011; accepted 3 May 2011

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

$[\text{Co}(\text{C}_4\text{H}_{13}\text{N}_3)_2][\text{InCl}_6]$	$V = 1960.62$ (16) Å ³
$M_r = 592.80$	$Z = 4$
Orthorhombic, C_{ccm}	Mo $K\alpha$ radiation
$a = 10.8925$ (5) Å	$\mu = 2.84$ mm ⁻¹
$b = 14.7291$ (7) Å	$T = 296$ K
$c = 12.2205$ (6) Å	$0.20 \times 0.18 \times 0.15$ mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer	6910 measured reflections
Absorption correction: multi-scan (SADABS; Bruker, 2002)	1282 independent reflections
$T_{\min} = 0.572$, $T_{\max} = 0.653$	1139 reflections with $I > 2\sigma(I)$
	$R_{\text{int}} = 0.021$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.018$	57 parameters
$wR(F^2) = 0.046$	H-atom parameters constrained
$S = 1.06$	$\Delta\rho_{\text{max}} = 0.34$ e Å ⁻³
1282 reflections	$\Delta\rho_{\text{min}} = -0.37$ 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).

References

- Bruker (2002). *SADABS*, *APEX2* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.
- Pan, Q. H., Cheng, Q. & Bu, X.-H. (2010*b*). *CrystEngComm*, **12**, 4198–4204.
- Pan, Q. H., Cheng, Q. & Bu, X.-H. (2011). *Chem. J. Chin. Univ.* **32**, 527–531.
- Pan, Q. H., Li, J. Y. & Bu, X.-H. (2010*a*). *Micropor. Mesopor. Mater.* **132**, 453–457.
- Pan, Q. H., Yu, J. H. & Xu, R. R. (2005). *Chem. J. Chin. Univ.* **26**, 2199–2202.
- Pan, Q. H., Yu, J. H. & Xu, R. R. (2008). *Chem. Mater.* **20**, 370–372.
- Rothammel, W., Spengler, R. & Salah, A. B. (1998). *Acta Cryst.* **C54**, IUC9800059.
- Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.
- Stalder, S. M. & Wilkinson, A. P. (1997). *Chem. Mater.* **9**, 2168–2173.
- Tong, J. & Pan, Q. (2011). *Acta Cryst.* **E67**, m579–m580.
- Wang, Y., Yu, J. H. & Xu, R. R. (2003*a*). *Angew. Chem. Int. Ed.* **42**, 4089–4092.
- Wang, Y., Yu, J. H. & Xu, R. R. (2003*b*). *Chem. Eur. J.* **9**, 5048–5055.

supplementary materials

Acta Cryst. (2011). E67, m731 [doi:10.1107/S1600536811016758]

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

Q. Wu, S. Chen, C. Zhang, X. Zhi and Z. Chen

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.

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.

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 $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{parent atom})$.

Figures

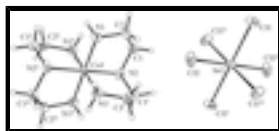


Fig. 1. A view of the structure of complex. Ellipsoids are drawn at the 50% probability level. [Symmetry codes: (i) $x, y, -z$; (ii) $-1/2 - x, -1/2 - y, z$; (iii) $-1/2 - x, -1/2 - y, -z$.]

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

Crystal data

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

$F(000) = 1176$

supplementary materials

$M_r = 592.80$

Orthorhombic, *Cccm*

Hall symbol: -C 2 2c

$a = 10.8925$ (5) Å

$b = 14.7291$ (7) Å

$c = 12.2205$ (6) Å

$V = 1960.62$ (16) Å³

$Z = 4$

$D_x = 2.008$ Mg m⁻³

Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

$\theta = 2.3$ – 28.3°

$\mu = 2.84$ mm⁻¹

$T = 296$ K

Block, yellow

$0.2 \times 0.18 \times 0.15$ mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer

Radiation source: fine-focus sealed tube graphite

Detector resolution: 5.00cm 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 $P = (F_o^2 + 2F_c^2)/3$

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

$\Delta\rho_{\max} = 0.34$ e Å⁻³

$\Delta\rho_{\min} = -0.37$ 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 (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
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)

supplementary materials

C11 ⁱ —In1—C11	88.48 (3)	N1 ^v —Co1—N1	89.03 (10)
C11 ⁱⁱ —In1—C11	91.52 (3)	N1 ^{iv} —Co1—N1	180.00 (8)
C11 ⁱ —In1—C11 ⁱⁱⁱ	91.52 (3)	C2—N1—Co1	111.65 (12)
C11 ⁱⁱ —In1—C11 ⁱⁱⁱ	88.48 (3)	C2—N1—H1A	109.3
C11—In1—C11 ⁱⁱⁱ	180.0	Co1—N1—H1A	109.3
C11 ⁱ —In1—C12 ⁱⁱⁱ	91.076 (16)	C2—N1—H1B	109.3
C11 ⁱⁱ —In1—C12 ⁱⁱⁱ	88.924 (17)	Co1—N1—H1B	109.3
C11—In1—C12 ⁱⁱⁱ	91.076 (16)	H1A—N1—H1B	108.0
C11 ⁱⁱⁱ —In1—C12 ⁱⁱⁱ	88.924 (17)	C1 ⁱ —N2—C1	114.8 (2)
C11 ⁱ —In1—C12	88.924 (17)	C1 ⁱ —N2—Co1	109.18 (12)
C11 ⁱⁱ —In1—C12	91.076 (16)	C1—N2—Co1	109.18 (12)
C11—In1—C12	88.924 (17)	C1 ⁱ —N2—H2	107.8
C11 ⁱⁱⁱ —In1—C12	91.076 (16)	C1—N2—H2	107.8
C12 ⁱⁱⁱ —In1—C12	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 C11 ^{vi}	0.90	2.62	3.4915 (17)	164
N1—H1B \cdots C12 ^{vii}	0.90	2.61	3.3823 (18)	144
N1—H1B \cdots C11 ^{viii}	0.90	2.72	3.3957 (17)	133
N2—H2 \cdots C11 ^{ix}	0.91	2.79	3.5407 (19)	141
N2—H2 \cdots C11 ^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) $x+1/2, y+1/2, -z$.

Fig. 1

