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Tris(1,10-phenanthroline)cobalt(II) triiodide

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Key indicators

Single-crystal X-ray study
T = 150 K
Mean σ(C–C) = 0.008 Å
R factor = 0.038
wR factor = 0.087
Data-to-parameter ratio = 18.3

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

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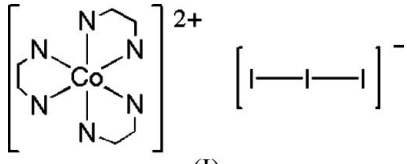
The asymmetric unit of the title compound, $[Co(C_{12}H_8N_2)_3](I_3)_2$, contains one $[Co(1,10\text{-phenanthroline})_3]^{2+}$ cation, half each of two centrosymmetric triiodide anions, and one complete triiodide anion. The title compound was synthesized solvothermally from $Co(NO_3)_2$, 1,10-phenanthroline, and SnI_2, where the SnI_2 reagent serves only as a source of I atoms.

Comment

Single crystals of the coordination compound tris(1,10-phenanthroline)cobalt(II) triiodide, (I), were isolated from the solvothermal reaction of $Co(NO_3)_2$, 1,10-phenanthroline (phen), and SnI_2. Though the synthesis includes tin(II) iodide as a reagent, the resultant product contains no tin. However, it is a well known phenomenon that tin(II) compounds are air-sensitive (Ryan & Xu, 2004), and since an inert environment was not used in the present synthesis, the absence of tin in the resulting compound is not surprising. Thus, the SnI_2 starting material functions only as a source of I atoms.



(I)

The asymmetric unit of (I) contains a $[Co(phen)_3]^{2+}$ cation in addition to three crystallographically distinct I_3^- anions, two of which are located about inversion centers (Fig. 1). For the non-centrosymmetric anion (I5—I6—I7), one I—I bond is slightly longer than the other and the anion deviates slightly from linearity. Both the cation and the anion of this compound have been observed in numerous other compounds, and the bond angles and distances for both species are typical (Table 1). The present compound is isostructural with $[Ni(phen)_3](I_3)_2$ (Freckmann & Tebbe, 1981). Additionally, the title compound is related to several other compounds having the same basic formula, $[M(phen)_3](I_3)_2$ (*M* = Mn or Fe; Horn *et al.*, 2002; Ramalakshmi *et al.*, 1999). However, these compounds crystallized in a different space group, and most of them contain solvents of crystallization.

Experimental

SnI_2 (0.3 mmol, 110 mg), $Co(NO_3)_2{\cdot}6H_2O$ (0.1 mmol, 29 mg), and 1,10-phenanthroline (0.3 mmol, 70 mg) were weighed and placed in a 23 ml Teflon-lined autoclave with absolute ethanol (10 ml) as the reaction solvent. The autoclave was subsequently sealed and heated

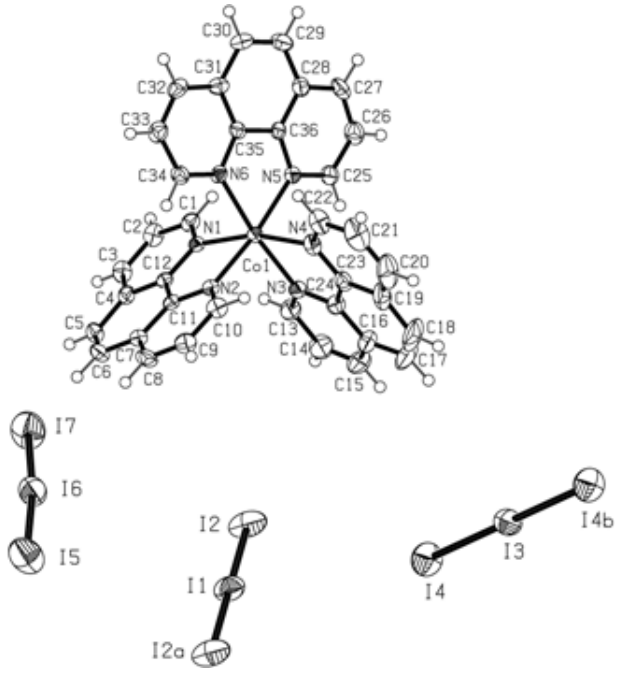


Figure 1
Displacement ellipsoid plot of (I), showing the atom-labeling scheme. Displacement ellipsoids are drawn at the 50% probability level. H atoms are drawn as circles of arbitrary radii [symmetry codes: (a) $1-x, -y, 1-z$; (b) $1-x, -y, -z$].

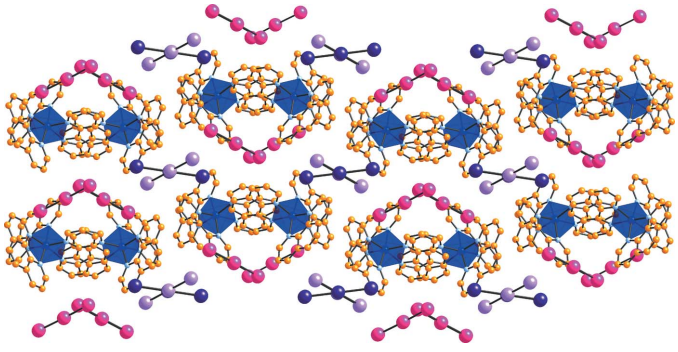


Figure 2
[100] view of the crystal packing in (I). Crystallographically independent I_3^- anions are shown in different colors. Other colors: Co dark blue, C yellow and N light blue.

at a rate of 1 K min^{-1} to 433 K. The temperature was held at 433 K for 3 d before it was decreased at a rate of 0.1 K min^{-1} to 353 K, where it was then held for 6 h. Finally, the temperature was decreased at a rate of 0.1 K min^{-1} to room temperature. Orange–brown crystals were isolated from the reaction and a suitable single crystal was selected for the X-ray diffraction experiment.

Crystal data

$[Co(C_{12}H_8N_2)_3](I_3)_2$
$M_r = 1360.94$
Monoclinic, $P2_1/c$
$a = 10.4187$ (5) Å
$b = 29.565$ (1) Å
$c = 12.9299$ (6) Å
$\beta = 93.395$ (10)°
$V = 3975.8$ (3) Å^3
$Z = 4$
$D_x = 2.274$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 8858 reflections
$\theta = 2.4$–26.4°
$\mu = 5.13$ mm^{-1}
$T = 150$ (1) K
Block, orange–brown
0.18 × 0.16 × 0.12 mm

Data collection

Bruker SMART APEX CCD diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2002)
$T_{min} = 0.434$, $T_{max} = 0.541$
42790 measured reflections
8145 independent reflections
7152 reflections with $I > 2\sigma(I)$
$R_{int} = 0.043$
$\theta_{max} = 26.4°$
$h = -13 \rightarrow 12$
$k = -36 \rightarrow 37$
$l = -16 \rightarrow 15$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.038$
$wR(F^2) = 0.087$
$S = 1.06$
8145 reflections
445 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0368P)^2 + 12.0369P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 2.11$ e Å^{-3}
$\Delta\rho_{min} = -1.07$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

I1—I2	2.9414 (4)	Co1—N4	2.118 (4)
I3—I4	2.9206 (4)	Co1—N6	2.123 (4)
I5—I6	2.9280 (5)	Co1—N5	2.131 (4)
I6—I7	2.8855 (5)	Co1—N3	2.150 (4)
Co1—N1	2.111 (4)	Co1—N2	2.151 (4)
$I2^i$—I1—I2	180	N1—Co1—N3	91.96 (16)
I4—I3—$I4^{ii}$	180	N4—Co1—N3	78.38 (17)
I7—I6—I5	175.733 (18)	N6—Co1—N3	172.73 (16)
N1—Co1—N4	165.56 (16)	N5—Co1—N3	94.72 (16)
N1—Co1—N6	90.52 (15)	N1—Co1—N2	78.16 (15)
N4—Co1—N6	100.39 (16)	N4—Co1—N2	91.56 (16)
N1—Co1—N5	101.39 (15)	N6—Co1—N2	93.97 (15)
N4—Co1—N5	90.19 (16)	N5—Co1—N2	172.05 (15)
N6—Co1—N5	78.09 (15)	N3—Co1—N2	93.23 (15)

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

H atoms were positioned geometrically and allowed to ride on their parent atoms, with C—H = 0.95 Å and $U_{iso}(H) = 1.2U_{eq}(C)$. The highest peak and depest hole are located 0.88 and 0.74 Å, respectively, from atom I7.

Data collection: *SMART-NT* (Bruker, 2001); cell refinement: *SAINT-Plus-NT* (Bruker, 2001); data reduction: *SAINT-Plus-NT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Sheldrick, 2000); software used to prepare material for publication: *SHELXTL*.

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