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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  $\sigma(\text{C}-\text{C}) = 0.008 \text{ \AA}$   
 R factor = 0.038  
 wR factor = 0.087  
 Data-to-parameter ratio = 18.3

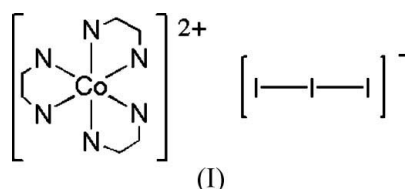
For details of how these key indicators were  
 automatically derived from the article, see  
<http://journals.iucr.org/e>.

The asymmetric unit of the title compound,  $[\text{Co}(\text{C}_{12}\text{H}_8\text{N}_2)_3] \cdot (\text{I}_3)_2$ , contains one  $[\text{Co}(1,10\text{-phenanthroline})_3]^{2+}$  cation, half each of two centrosymmetric triiodide anions, and one complete triiodide anion. The title compound was synthesized solvothermally from  $\text{Co}(\text{NO}_3)_2$ , 1,10-phenanthroline, and  $\text{SnI}_2$ , where the  $\text{SnI}_2$  reagent serves only as a source of I atoms.

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## Comment

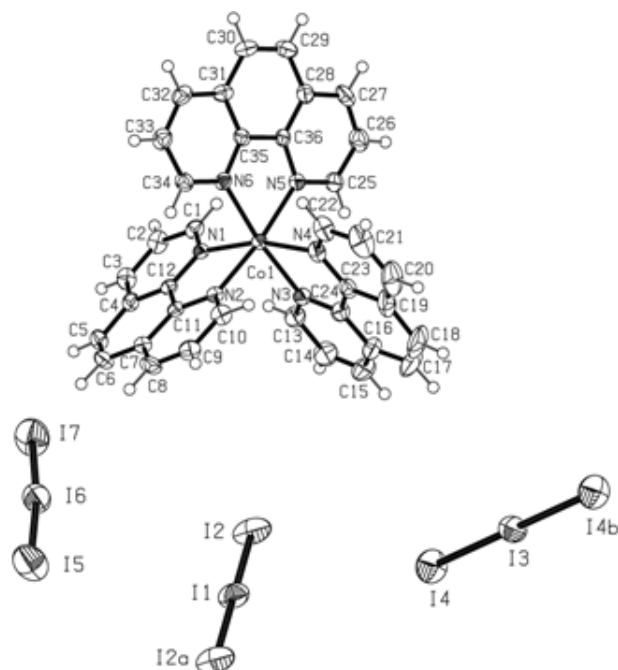
Single crystals of the coordination compound tris(1,10-phenanthroline)cobalt(II) triiodide, (I), were isolated from the solvothermal reaction of  $\text{Co}(\text{NO}_3)_2$ , 1,10-phenanthroline (phen), and  $\text{SnI}_2$ . Though the synthesis includes tin(II) iodide as a reagent, the resultant product 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  $\text{SnI}_2$  starting material functions only as a source of I atoms.



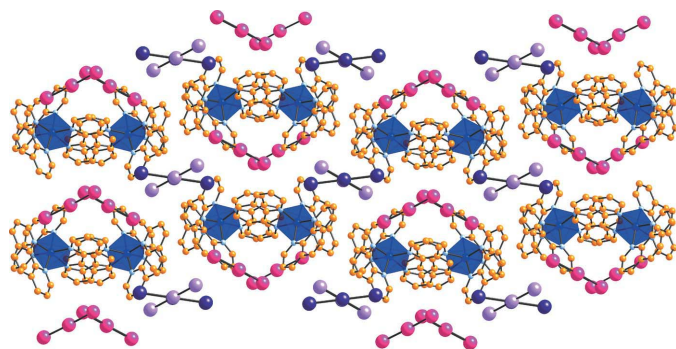
The asymmetric unit of (I) contains a  $[\text{Co}(\text{phen})_3]^{2+}$  cation in addition to three crystallographically distinct  $\text{I}_3^-$  anions, two of which are located about inversion centers (Fig. 1). For the non-centrosymmetric anion (I5–I6–I7), one I–I bond is slightly longer than the 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  $[\text{Ni}(\text{phen})_3](\text{I}_3)_2$  (Freckmann & Tebbe, 1981). Additionally, the title compound is related to several other compounds having the same basic formula,  $[\text{M}(\text{phen})_3](\text{I}_3)_2$  ( $M = \text{Mn}$  or  $\text{Fe}$ ; 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

$\text{SnI}_2$  (0.3 mmol, 110 mg),  $\text{Co}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$  (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  $1 \text{ K min}^{-1}$  to 433 K. The temperature was held at 433 K for 3 d before it was decreased at a rate of  $0.1 \text{ K min}^{-1}$  to 353 K, where it was then held for 6 h. Finally, the temperature was decreased at a rate of  $0.1 \text{ K min}^{-1}$  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

$[\text{Co}(\text{C}_{12}\text{H}_8\text{N}_2)_3](\text{I}_3)_2$   
 $M_r = 1360.94$   
 Monoclinic,  $P2_1/c$   
 $a = 10.4187(5) \text{ \AA}$   
 $b = 29.565(1) \text{ \AA}$   
 $c = 12.9299(6) \text{ \AA}$   
 $\beta = 93.395(10)^\circ$   
 $V = 3975.8(3) \text{ \AA}^3$   
 $Z = 4$

$D_x = 2.274 \text{ Mg m}^{-3}$   
 Mo  $K\alpha$  radiation  
 Cell parameters from 8858 reflections  
 $\theta = 2.4\text{--}26.4^\circ$   
 $\mu = 5.13 \text{ mm}^{-1}$   
 $T = 150(1) \text{ K}$   
 Block, orange-brown  
 $0.18 \times 0.16 \times 0.12 \text{ mm}$

#### Data collection

Bruker SMART APEX CCD diffractometer  
 $\omega$  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  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.043$   
 $\theta_{\max} = 26.4^\circ$   
 $h = -13 \rightarrow 12$   
 $k = -36 \rightarrow 37$   
 $l = -16 \rightarrow 15$

#### 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  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\max} = 0.001$   
 $\Delta\rho_{\max} = 2.11 \text{ e \AA}^{-3}$   
 $\Delta\rho_{\min} = -1.07 \text{ e \AA}^{-3}$

**Table 1**

Selected geometric parameters ( $\text{\AA}, ^\circ$ ).

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 <sup>i</sup> —I1—I2	180	N1—Co1—N3	91.96 (16)
I4—I3—I4 <sup>ii</sup>	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  $\text{C—H} = 0.95 \text{ \AA}$  and  $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ . The highest peak and deepest hole are located  $0.88$  and  $0.74 \text{ \AA}$ , 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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