Tris(1,10-Phenanthroline)Cobalt(II) Triiodide

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

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The asymmetric unit of the title compound, \([\text{Co(C}_{12}\text{H}_{8}\text{N}_2]_3\text{-}3\text{I}]_2\), contains one \([\text{Co(1,10-phenanthroline)}_3]^{2+}\) cation, half each of two centrosymmetric triiodide anions, and one complete triiodide anion. The title compound was synthesized solvothermally from \(\text{Co(NO}_3\}_{2}\), 1,10-phenanthroline, and \(\text{SnI}_2\), where the \(\text{SnI}_2\) 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 \(\text{Co(NO}_3\}_{2}\), 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 \([\text{Co(phen)}_3]^{2+}\) cation in addition to three crystallographically distinct \(\text{I}^-\) 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 \([\text{Ni(phen)}_3]_2\text{I}_2\) (Freckmann & Tebbe, 1981). Additionally, the title compound is related to several other compounds having the same basic formula, \([\text{M(phen)}_3]_2\text{I}_2\) (\(\text{M} = \text{Mn or Fe; Horn et al., 2002; Ramalakshmi et al., 1999}\)). However, these compounds crystallized in a different space group, and most of them contain solvents of crystallization.

Experimental

\(\text{SnI}_2\) (0.3 mmol, 110 mg), \(\text{Co(NO}_3\}_{2}\cdot6\text{H}_2\text{O}\) (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...
where it was then held for 6 h. Finally, the temperature was decreased for 3 d before it was decreased at a rate of 0.1 K min\(^{-1}\) (2005). E. Acta Cryst.

Crystal data

selected for the X-ray diffraction experiment.

were isolated from the reaction and a suitable single crystal was for 3 d before it was decreased at a rate of 0.1 K min\(^{-1}\) (2005). E. Acta Cryst.

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) \(1 - x, x, -y, -z\)].

Figure 2

[100] view of the crystal packing in (I). Crystallographically independent \(\text{I}^+\) 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

\[\text{[Co(C\(_2\text{H}_6\text{N}_3\text{)3}]\text{(I)2}}\]

\(M_i = 1360.94\)

Monoclinic, \(P_2_1/c\)

\(a = 10.4187\ (5)\ \text{Å}\)

\(b = 29.565\ (1)\ \text{Å}\)

\(c = 12.9299\ (6)\ \text{Å}\)

\(\beta = 93.395\ (10)^\circ\)

\(V = 3975.8\ (3)\ \text{Å}^3\)

\(Z = 4\)

\(D_x = 2.274\ \text{Mg m}^{-3}\)

Mo Kα radiation

Cell parameters from 8888 reflections

\(\theta = 2.4–26.4^\circ\)

\(\mu = 5.13\ \text{mm}^{-1}\)

\(T = 150\ (1)\ \text{K}\)

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-\(\omega\) scan
\(SADABS\); Sheldrick, 2002

\(T_{	ext{min}} = 0.434, T_{	ext{max}} = 0.541\)

42790 measured reflections

8145 independent reflections

17522 reflections with \(I > 2\sigma(I)\)

Refinement

Refinement on \(F^2\)

\(R[F^2 > 2\sigma(F^2)] = 0.038\)

\(wR(F^2) = 0.087\)

\(S = 1.06\)

8145 reflections

\(\Delta\rho_{	ext{max}} = 2.11\ \text{e Å}^{-3}\)

445 parameters

H-atom parameters constrained

Table 1

Selected geometric parameters (Å, \(^\circ\)).

\[\begin{array}{lccc}
\text{I1} & \text{I2} & \text{Col} & \text{N4} \\
2.9414 (4) & & 2.118 (4) & \\
\text{I3} & \text{I4} & \text{Col} & \text{N6} \\
2.9206 (4) & & 2.123 (4) & \\
\text{I5} & \text{I6} & \text{Col} & \text{N5} \\
2.9380 (5) & & 2.131 (4) & \\
\text{I5} & \text{I6} & \text{Col} & \text{N3} \\
2.69855 (5) & & 2.150 (4) & \\
\text{I5} & \text{I6} & \text{Col} & \text{N2} \\
2.111 (4) & & 2.151 (4) & \\
\text{I2} & \text{I1} & \text{N1} & \text{Col} & \text{N3} \\
180 & & 91.96 (16) & \\
\text{I4} & \text{I3} & \text{N1} & \text{Col} & \text{N4} \\
154 & & 78.38 (17) & \\
\text{I7} & \text{I6} & \text{N1} & \text{Col} & \text{N5} \\
175.733 (18) & & 172.73 (16) & \\
\text{N1} & \text{Col} & \text{N4} & & 94.72 (16) \\
165.56 (16) & & 91.56 (16) & \\
\text{N1} & \text{Col} & \text{N5} & & 91.97 (16) \\
100.52 (15) & & 91.56 (16) & \\
\text{N4} & \text{Col} & \text{N6} & & 93.97 (16) \\
10.91 (16) & & 93.23 (15) & \\
\text{N4} & \text{Col} & \text{N5} & & 93.23 (15) \\
78.09 (15) & & 93.23 (15) & \\
\text{N4} & \text{Col} & \text{N6} & & 93.23 (15) \\
100.52 (15) & & 93.23 (15) & \\
\end{array}\]

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

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

Data collection: SMART-NT (Bruker, 2001); cell refinement: SMART-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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References


