

Aquachloro[tris(methylpyridyl)amine]-cobalt(III) diperchlorate monohydrate

Timothy W. Failes

Centre for Heavy Metals Research, School of Chemistry, University of Sydney, NSW 2006, Australia

Correspondence e-mail: t.failes@chem.usyd.edu.au

Key indicators

Single-crystal X-ray study
 $T = 150$ K
 Mean $\sigma(\text{C}-\text{C}) = 0.004$ Å
 Disorder in solvent or counterion
 R factor = 0.037
 wR factor = 0.108
 Data-to-parameter ratio = 15.5

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

The tris(methylpyridyl)amine (tpa, $\text{C}_{18}\text{H}_{18}\text{N}_4$) ligand in the title complex, $[\text{CoCl}(\text{tpa})(\text{H}_2\text{O})](\text{ClO}_4)_2 \cdot \text{H}_2\text{O}$, is bound to the Co^{III} centre in a tripodal fashion through the four N-donor atoms, allowing the chloro and aqua ligands to occupy the remaining *cis* sites in the octahedral coordination geometry. The chloro ligand is coordinated *trans* to the tertiary amine N atom of the tpa ligand. A solvent water molecule is hydrogen bonded to the aqua ligand. The metal–ligand bond lengths are typical for Co^{III} complexes.

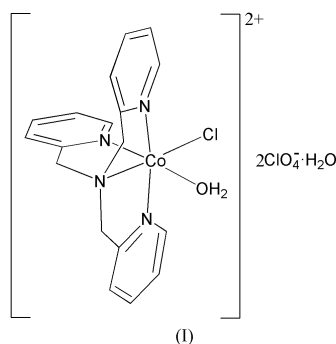
Received 26 April 2004

Accepted 10 May 2004

Online 15 May 2004

Comment

The title complex, (I) (Fig. 1), is a commonly used precursor to Co^{III} complexes, as is the complementary dichloro complex reported previously (Failes, 2003). The complex was first reported by Mandel *et al.* (1988) and the sample used in this study was prepared in a similar manner. However, the earlier report indicated that the complex contained 3.5 solvent water molecules per complex cation, whereas the structure in this study shows only a single solvent water molecule. The aqua ligand interacts with the solvent water molecule and a perchlorate anion by way of $\text{O}-\text{H}\cdots\text{O}$ hydrogen bonds (Table 2). Of the two perchlorate anions in (I), one is disordered.



Experimental

The complex was prepared according to a reported method (Mandel *et al.*, 1988). Crystals of (I) were grown by slow cooling of a methanol solution.

Crystal data

$[\text{CoCl}(\text{C}_{18}\text{H}_{18}\text{N}_4)(\text{H}_2\text{O})](\text{ClO}_4)_2 \cdot \text{H}_2\text{O}$
 $M_r = 619.68$
 Monoclinic, $P2_1/c$
 $a = 11.882$ (2) Å
 $b = 16.367$ (3) Å
 $c = 12.744$ (3) Å
 $\beta = 90.353$ (3)°
 $V = 2478.5$ (9) Å³
 $Z = 4$

$D_x = 1.661$ Mg m⁻³
 Mo $K\alpha$ radiation
 Cell parameters from 1006 reflections
 $\theta = 2.7$ – 27.4 °
 $\mu = 1.08$ mm⁻¹
 $T = 150$ (2) K
 Block, purple
 $0.49 \times 0.38 \times 0.19$ mm

Data collection

Bruker SMART1000 CCD
 diffractometer
 ω scans
 Absorption correction: multi-scan
 (SADABS; Sheldrick, 1996)
 $T_{\min} = 0.628$, $T_{\max} = 0.815$
 23 833 measured reflections

5830 independent reflections
 4486 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.027$
 $\theta_{\text{max}} = 28.0^\circ$
 $h = -15 \rightarrow 15$
 $k = -21 \rightarrow 21$
 $l = -16 \rightarrow 16$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.037$
 $wR(F^2) = 0.108$
 $S = 1.07$
 5830 reflections
 377 parameters
 H atoms treated by a mixture of
 independent and constrained
 refinement

$w = 1/[\sigma^2(F_o^2) + (0.0669P)^2 + 0.0165P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} = 0.001$
 $\Delta\rho_{\text{max}} = 0.51 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\text{min}} = -0.37 \text{ e } \text{\AA}^{-3}$

Table 1

Selected bond distances (Å).

Co1–N2	1.9232 (17)	Co1–N3	1.9357 (19)
Co1–N1	1.9260 (19)	Co1–N4	1.9510 (18)
Co1–O1	1.9298 (16)	Co1–Cl1	2.2207 (7)

Table 2

Hydrogen-bonding geometry (Å, °).

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
O1–H10A \cdots O5	0.82 (3)	1.88 (3)	2.699 (3)	177 (3)
O1–H10B \cdots O1W	1.02 (3)	1.61 (3)	2.587 (3)	159 (3)

One of the perchlorate anions is disordered and was modelled with the O atoms each occupying two sites, with occupancy factors refined and then fixed at 0.657 and 0.343. The H atoms attached to C were placed in calculated positions [$C(\text{Ar})-H = 0.95$ and $C(\text{CH}_2)-H = 0.99$ Å] using a riding model, with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{parent atom})$. The H atoms of the aqua ligand (H10A and H10B) and solvent water molecule (H1W and H2W) were located in Fourier difference maps and were freely refined with isotropic displacement parameters. Owing to the lack of observed hydrogen bonding and a close H1W \cdots H10B contact, the positions of atoms H1W and H2W should be regarded as less certain.

Data collection: SMART (Bruker, 1995); cell refinement: SAINT (Bruker, 1995); data reduction: SAINT and XPREP (Bruker, 1995);

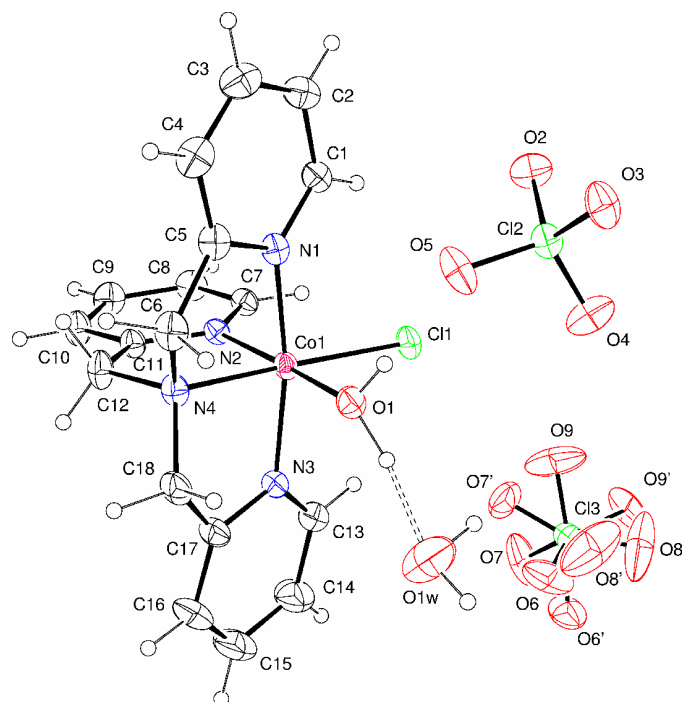


Figure 1

View of (I), with the crystallographic numbering scheme. Displacement ellipsoids are drawn at the 30% probability level. Both disordered perchlorate components are shown. The dashed line indicates a hydrogen bond.

program(s) used to solve structure: SHELXS97 (Sheldrick, 1997); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: ORTEP-3 for Windows (Farrugia, 1997); software used to prepare material for publication: WinGX (Farrugia, 1999).

References

- Bruker (1995). SMART, SAINT and XPREP. Bruker AXS Inc., Madison, Wisconsin, USA.
 Failes, T. W. (2003). Acta Cryst. E59, m616–m617.
 Farrugia, L. J. (1997). J. Appl. Cryst. 30, 565.
 Farrugia, L. J. (1999). J. Appl. Cryst. 32, 837–838.
 Mandel, J. B., Maricondi, C. & Douglas, B. E. (1988). Inorg. Chem. 27, 2990–2996.
 Sheldrick, G. M. (1996). SADABS. University of Göttingen, Germany.
 Sheldrick, G. M. (1997). SHELXS97 and SHELXL97. Release 97–2. University of Göttingen, Germany.