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Key indicators

Single-crystal X-ray study
 $T = 122$ K
Mean $\sigma(\text{C}-\text{C}) = 0.001$ Å
 R factor = 0.027
 wR factor = 0.060
Data-to-parameter ratio = 78.1For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.Trichloro(1,4,7-trimethyl-1,4,7-triazacyclo-
nonane)chromium(III)

The structure of the title complex, $[\text{CrCl}_3(\text{C}_9\text{H}_{21}\text{N}_3)]$, contains a distorted octahedral chromium(III) centre. The facial tridentate N -coordinating 1,4,7-trimethyl-1,4,7-triazacyclononane ligand and three chloride ions make up the first coordination sphere.

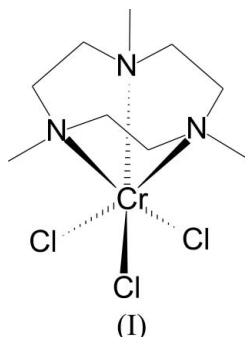
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Comment

The 1,4,7-trimethyl-1,4,7-triazacyclononane (tmtacn) ligand has become one of the classic ligands in coordination chemistry (Wieghardt *et al.*, 1982). In recent years, tmtacn–metal complexes have attracted attention in the area of ‘single-molecule magnets’, where tmtacn acts as a capping ligand in metal–cyanide cluster compounds, as described by Shores *et al.* (2004, and references therein). We report here the crystal structure of the title complex, $[\text{Cr}(\text{tmtacn})\text{Cl}_3]$, (I).



Complex (I) was prepared as a precursor for the above-mentioned metal–cyanide cluster compounds and is isostructural with the scandium(III) (Hajela *et al.*, 1997), titanium(III) (Bodner *et al.*, 1992), iron(III) (Silver & Trogler, 1995) and ruthenium(III) (Schneider *et al.*, 1994) analogues.

The bond lengths (Table 1) are similar to those of the tri-*n*-butyl-substituted analogous complex $\text{Cr}(\text{Bu}_3\text{tacn})\text{Cl}_3$, (Wu & Stahly, 1995) [$\text{Cr}-\text{N} = 2.142(2)$ – $2.154(2)$ Å; $\text{Cr}-\text{Cl} = 2.3196(9)$ – $2.3308(9)$ Å]. The $\text{Cr}-\text{Cl}$ and $\text{Cr}-\text{N}$ bond lengths are shorter than those of the scandium [$\text{Sc}-\text{Cl} = 2.406(2)$ Å; $\text{Sc}-\text{N} = 2.337(2)$ Å] and titanium [$\text{Ti}-\text{Cl} = 2.352(1)$ Å; $\text{Ti}-\text{N} = 2.256(3)$ Å] analogues, which is in agreement with the decrease of the relative ionic radii.

Experimental

Complex (I) was prepared by adding one equivalent of the tmtacn ligand to an acetonitrile solution of $[\text{Cr}(\text{THF})_3\text{Cl}_3]$. Crystals suitable for X-ray diffraction were obtained by slow evaporation of the filtered acetonitrile solution.

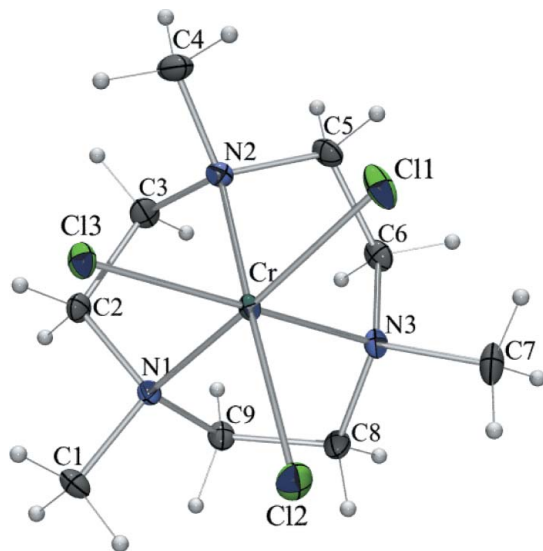


Figure 1
View of the molecular structure of $[\text{Cr}(\text{tmtacn})\text{Cl}_3]$, with the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level and H atoms are shown as small spheres of arbitrary radii.

Crystal data

$[\text{CrCl}_3(\text{C}_9\text{H}_{21}\text{N}_3)]$

$M_r = 329.64$

Monoclinic, $P2_1/c$

$a = 12.1518$ (14) Å

$b = 7.2055$ (5) Å

$c = 15.6894$ (14) Å

$\beta = 90.212$ (8)°

$V = 1373.8$ (2) Å³

$Z = 4$

$D_x = 1.594$ Mg m⁻³

Mo $K\alpha$ radiation

Cell parameters from 43956 reflections

$\theta = 1.7\text{--}45.0^\circ$

$\mu = 1.39$ mm⁻¹

$T = 122$ (1) K

Prism, green

$0.29 \times 0.28 \times 0.1$ mm

Data collection

Nonius KappaCCD area-detector diffractometer

ω and φ scans

Absorption correction: integration
Gaussian integration (Coppens, 1970)

$T_{\min} = 0.760$, $T_{\max} = 0.917$

88493 measured reflections

11318 independent reflections

9167 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.043$

$\theta_{\max} = 45.0^\circ$

$h = -24 \rightarrow 23$

$k = -14 \rightarrow 14$

$l = -31 \rightarrow 30$

Refinement

Refinement on F^2

$R[F^2 > 2\sigma(F^2)] = 0.027$

$wR(F^2) = 0.060$

$S = 1.12$

11318 reflections

145 parameters

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0185P)^2 + 0.4402P]$

where $P = (F_o^2 + 2F_c^2)/3$

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

$\Delta\rho_{\max} = 0.44$ e Å⁻³

$\Delta\rho_{\min} = -0.77$ e Å⁻³

Table 1

Selected geometric parameters (Å, °).

Cr—N1	2.1301 (5)	Cr—Cl1	2.3264 (2)
Cr—N2	2.1453 (6)	Cr—Cl2	2.3309 (3)
Cr—N3	2.1358 (6)	Cr—Cl3	2.3201 (3)
N1—Cr—N2	81.93 (2)	N3—Cr—Cl2	91.497 (17)
N1—Cr—N3	82.97 (2)	N1—Cr—Cl3	91.649 (17)
N3—Cr—N2	82.09 (2)	N2—Cr—Cl3	93.110 (17)
N1—Cr—Cl1	171.351 (16)	N3—Cr—Cl3	173.224 (16)
N2—Cr—Cl1	90.564 (17)	Cl1—Cr—Cl2	93.335 (9)
N3—Cr—Cl1	91.785 (17)	Cl3—Cr—Cl1	93.051 (10)
N1—Cr—Cl2	93.670 (16)	Cl3—Cr—Cl2	92.956 (9)
N2—Cr—Cl2	172.604 (17)		

H atoms were found in a difference Fourier map and were included in the refinement as atoms riding on the parent atom, with C—H bond lengths of 0.98 Å and 0.99 Å and $U_{\text{iso}}(\text{H})$ values of $1.2U_{\text{eq}}(\text{C})$.

Data collection: *COLLECT* (Nonius, 1999); cell refinement: *DIRAX* (Duisenberg, 1992); data reduction: *EVALCCD* (Duisenberg, 2003); program(s) used to solve structure: *SIR97* (Altomare *et al.*, 1999); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEPII* (Johnson, 1976); software used to prepare material for publication: *SHELXL97*.

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