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mer-Trichloro(2,2′,2″-terpyridine)chromium(III) dimethyl sulfoxide solvate

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The title compound, *mer*-[$CrCl_3(C_{15}H_{11}N_3)$]·$(CH_3)_2SO$, adopts a distorted octahedral configuration about the Cr^{III} atom.

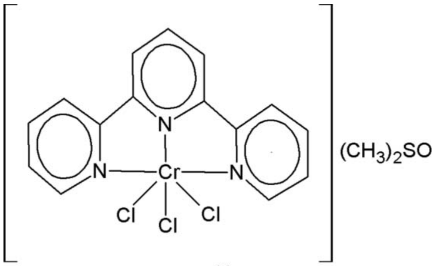
Key indicators

Single-crystal X-ray study
T = 101 K
Mean σ(C–C) = 0.003 Å
R factor = 0.029
wR factor = 0.072
Data-to-parameter ratio = 21.1

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

Comment

The synthesis and characterization of *mer*-[$CrCl_3$(terpy)] (terpy is 2,2′:6′,2″-terpyridine) were first reported about 30 years ago (Broomhead *et al.*, 1977), but no crystallographic data have been published to date. A search of the Cambridge Sructural Database (CSD, Version 5.27; Allen, 2002) revealed seven structures (Wickramasinghe *et al.*, 1982; Casellato *et al.*, 1991; Shiren *et al.*, 2002; Leung *et al.*, 1995) of chromium(III)–terpy complexes containing different non-halide mono- and multidentate ligands, none of which are similar to the title compound, (I). Various metallo-mono(terpy) complexes have also been reported, *viz.* M(terpy)Cl_n (M = Ga, Zn, Sn, Rh; n = 2, 3; Beran *et al.*, 1970; Einstein *et al.*, 1966; Einstein *et al.*, 1968; Pruchnik *et al.*, 2002), indicating the continued interest in such compounds.



(I)

The title compound, (I), contains a Cr^{III} atom which is octahedrally coordinated by three N atoms (N1, N2 and N3) of the terpy ligand and three Cl atoms (Cl1, Cl2 and Cl3) (Fig. 1). The mean Cr—N(outer) distance of 2.074 (2) Å is slightly longer than the Cr—N(central) bond distance [1.992 (1) Å]. A similar difference has also been observed in *mer*-[Cr(terpy)$_2$]$^{3+}$ (0.08 Å; Wickramasinghe *et al.*, 1982) and in the metallo-mono(terpy) complexes *mer*-[$GaCl_2$(terpy)] (0.078 Å; Beran *et al.*, 1970), *mer*-[$ZnCl_2$(terpy)] (0.12 Å; Einstein *et al.*, 1966) and *mer*-[$(CH_3)_2SnCl$(terpy)]$^+$ (0.08 Å; Einstein *et al.*, 1968).

The Cr—Cl1 [2.330 (1) Å] bond is *trans* to Cr—Cl3 [2.327 (1) Å] and the two bond lengths are very similar; they are significantly longer than the central Cr—Cl2 [2.291 (1) Å] bond. This can be ascribed to the larger *trans* influence of Cl *versus* N. A point of interest is the fact that the complexes *mer*-[$VCl_3(py)_3$] (Sorensen *et al.*, 1994) and *mer*-[$FeCl_3(py)_3$].py (py = pyridine) (Hoser *et al.*, 1983) both indicate a lengthened

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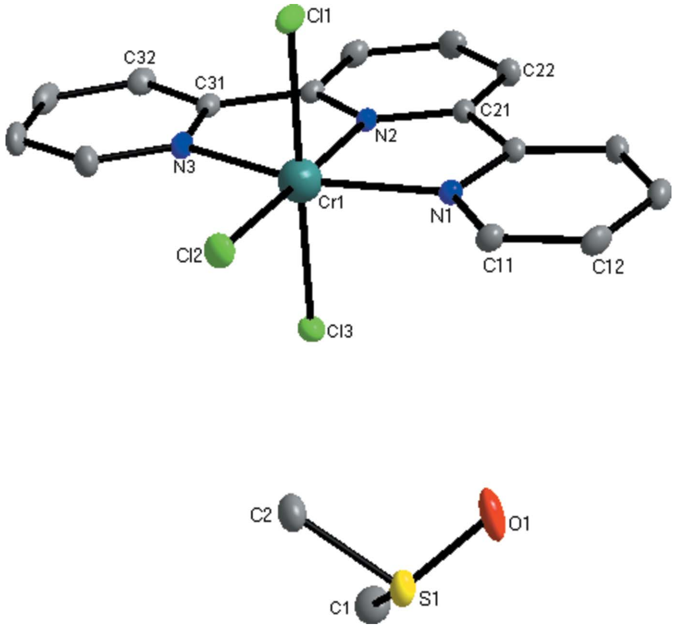

Figure 1
The molecular structure of (I) (50% probability displacement ellipsoids). For pyridine rings, the first digit indicates the ring number and the second indicates the number of the atom in the ring. Some labels have been omitted for clarity, but all rings are numbered in a similar fashion. H atoms have been omitted for clarity.

M—N(central) bond, as well as a shortened *M*—Cl(central) bond. This can thus eliminate the theory that the rigid nature of the terpyridyl ligand is the cause of the distortion of these bonds.

The other bond lengthss in complex (I) fall within the same ranges as in the corresponding metallo-terpy complexes (Beran *et al.*, 1970; Einstein *et al.*, 1966, 1968). The molecules within the structure are linked by an extensive network of hydrogen bonds (Table 2). The chromium(III) complex molecules and DMSO solvent molecules (at the centre of the unit cell) form infinite chains along the *a* axis (see Fig. 2).

The mean *cis*-N—Cr—N bond angle is 78.51 (6)° which is comparable to the bond angles in *mer*-$[\text{Cr(terpy)}_2]^{3+}$. The rigidity of the terpyridine group requires that these two N—Cr—N angles be much less than 90°. The three pyridine rings constituting the terpyridine ligand are also not coplanar. The dihedral angles between the two terminal rings and the central ring are 4.80 (7) (ring 1 *versus* 2) and 0.66 (7)° (ring 3 *versus* 2). The dihedral angles found in other metallo-mono-(terpyridine) complexes range from 1.3 to 6° (Beran *et al.*, 1970; Einstein *et al.*, 1966, 1968).

Experimental

Anhydrous chromium(III) chloride (0.15 g, 0.95 mmol) and 2,2′:6′,2″-terpyridine (0.10 g, 0.43 mmol) were dissolved in hexamethylphosphoramide (5 ml). A piece of zinc was added to catalyse dissolution of the chromium(III) chloride. The mixture was heated to 433 K and then immediately allowed to cool to room temperature. The resulting dark-green crystalline solid was filtered, the zinc removed, and washed with hexamethylphosphoramide followed by diethyl ether. The green solid was recrystallized from dimethylsulfoxide. Dark-green single crystals were obtained (yield: 0.16 g, 79%) suitable for X-ray crystallography. IR ν(C—N): 1316 cm^{-1}, ν(C$\cdots$C): 1600 cm^{-1}.

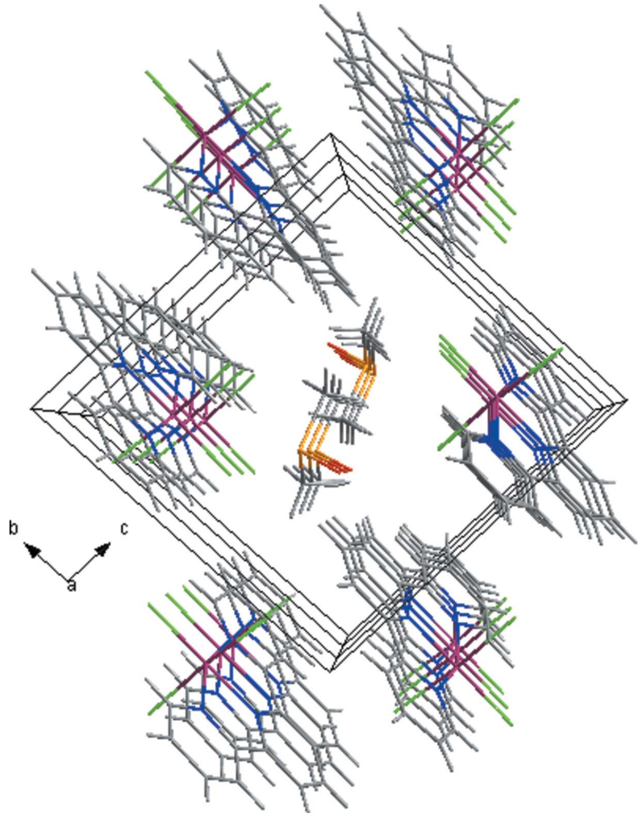

Figure 2
Perspective view of the packing of (I) (three unit cells deep) along the *a* axis.

Crystal data

$[\text{CrCl}_3(\text{C}_{15}\text{H}_{11}\text{N}_3)]\cdot\text{C}_2\text{H}_6\text{OS}$
$M_r = 469.75$
Triclinic, $P\overline{1}$
$a = 8.818$ (5) Å
$b = 10.672$ (5) Å
$c = 10.749$ (5) Å
$\alpha = 95.501$ (5)°
$\beta = 91.029$ (5)°
$\gamma = 98.580$ (5)°
$V = 995.1$ (9) Å^3
$Z = 2$
$D_x = 1.568$ Mg m^{-3}
Mo $K\alpha$ radiation
$\mu = 1.09$ mm^{-1}
$T = 101$ (2) K
Plate, green
0.22 × 0.21 × 0.04 mm

Data collection

Bruker X8 APEXII
  diffractometer
ω and φ scans
Absorption correction: multi-scan
  (*SADABS*; Bruker, 2004)
  $T_{\min} = 0.799$, $T_{\max} = 0.962$
13304 measured reflections
4966 independent reflections
4262 reflections with $I > 2\sigma(I)$
$R_{\text{int}} = 0.027$
$\theta_{\max} = 28.4°$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.029$
$wR(F^2) = 0.072$
$S = 1.04$
4966 reflections
235 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0289P)^2$
  $+ 0.5329P]$
  where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.001$
$\Delta\rho_{\max} = 0.40$ e Å^{-3}
$\Delta\rho_{\min} = -0.42$ e Å^{-3}

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Table 1
Selected geometric parameters (Å, °).

Cr1—N2	1.9921 (17)	Cr1—Cl2	2.2919 (12)
Cr1—N3	2.0712 (17)	Cr1—Cl3	2.3272 (10)
Cr1—N1	2.0782 (16)	Cr1—Cl1	2.3306 (11)
N2—Cr1—N3	78.61 (6)	N1—Cr1—Cl2	101.28 (5)
N2—Cr1—N1	78.42 (6)	N3—Cr1—Cl3	88.26 (5)
N3—Cr1—N1	157.02 (6)	N1—Cr1—Cl3	90.03 (5)
N3—Cr1—Cl2	101.70 (4)	Cl3—Cr1—Cl1	174.57 (2)

Table 2
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
C1—H1···Cl1^{i}	0.98	2.81	3.782 (2)	171
C2—H4···O1^{ii}	0.98	2.44	3.363 (3)	158
C2—H5···Cl3^{iii}	0.98	2.77	3.726 (2)	165
C14—H14···O1^{iv}	0.95	2.37	3.303 (3)	168
C22—H22···O1^{iv}	0.95	2.15	3.094 (2)	177
C24—H24···Cl1^{v}	0.95	2.75	3.635 (2)	155
C32—H31···Cl1^{v}	0.95	2.75	3.644 (2)	157
C33—H32···Cl1^{vi}	0.95	2.59	3.501 (3)	160

Symmetry codes: (i) $x, y, z-1$; (ii) $-x, -y+1, -z+1$; (iii) $-x+1, -y+1, -z+1$; (iv) $-x, -y, -z+1$; (v) $-x+1, -y, -z+2$; (vi) $x+1, y, z$.

The methyl and aromatic H atoms were placed in geometrically idealized positions (C—H = 0.93–0.96 Å) and constrained to ride on their parent atoms, with $U_{\rm iso}$(H) = 1.5$U_{\rm eq}$(C) and 1.2$U_{\rm eq}$(C), respectively.

Data collection: *APEX2* (Bruker, 2005); cell refinement: *SAINT-Plus* (Bruker, 2004); data reduction: *SAINT-Plus* and *XPREP* (Bruker, 2004); program(s) used to solve structure: *SHELXL97* (Sheldrick, 1997); program(s) used to refine structure: *SIR97* (Altomare *et al.*, 1999); molecular graphics: *DIAMOND* (Brandenburg & Putz, 2005); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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