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cis-Dichloridobis(1,10-phenanthroline)chromium(III) chloride

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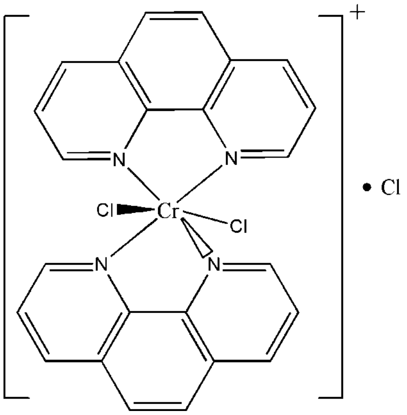
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.008 Å; disorder in solvent or counterion; R factor = 0.070; wR factor = 0.211; data-to-parameter ratio = 15.1.

In the title complex, $[CrCl_2(C_{12}H_8N_2)_2]Cl$, the Cr^{III} ion is situated on a twofold rotation axis and displays a slightly distorted octahedral $CrCl_2N_4$ coordination geometry. The Cr environment is composed of a *cis* arrangement of two 1,10-phenanthroline and two chloride ligands. The chloride counter-anion exhibits half-occupation and is equally disordered over two positions.

Related literature

For background to chromium(III) complexes, see: Vincent (2000). For the structure of a related Cr(III) complex with phenanthroline ligands, see: Birk *et al.* (2008).



Experimental

Crystal data

$[CrCl_2(C_{12}H_8N_2)_2]Cl$
M_r = 518.76
Monoclinic, $C2/c$
a = 15.446 (7) Å
b = 13.762 (6) Å
c = 12.536 (5) Å
β = 100.398 (6)°
V = 2621.1 (19) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.76 mm^{-1}
T = 298 K
0.40 × 0.10 × 0.10 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2000) T_{min} = 0.751, T_{max} = 0.928
5182 measured reflections
2265 independent reflections
1778 reflections with $I > 2\sigma(I)$
R_{int} = 0.039

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.070
$wR(F^2)$ = 0.211
S = 1.07
2265 reflections
150 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.92 e Å^{-3}
$\Delta\rho_{min}$ = −0.35 e Å^{-3}

Table 1
Selected bond lengths (Å).

Cr1—N1	2.062 (4)	Cr1—Cl1	2.2941 (15)
Cr1—N2	2.073 (4)		

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT* (Bruker, 2000); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: WM2436).

References

Birk, T., Bendix, J. & Weihe, H. (2008). *Acta Cryst.* E**64**, m369–m370.
Bruker (2000). *SMART* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.
Sheldrick, G. M. (2000). *SADABS*. University of Göttingen, Germany.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Vincent, J. B. (2000). *Acc. Chem. Res.* **33**, 503–510.

supplementary materials

Acta Cryst. (2011). E**67**, m139 [**doi:10.1107/S1600536810053845**]

cis-Dichloridobis(1,10-phenanthroline)chromium(III) chloride

X. Gao

Comment

Chromium is known to activate enzymes, maintain protein stability and enhance carbohydrate metabolism. Organic chromium (III) sources have been shown to enhance the availability of chromium. Nutritionists believe organic chromium should be supplemented in most animal diets. A search has therefore been underway to identify the biologically active form of chromium, *viz.* the biomolecule that binds chromium(III) and possesses an intrinsic function associated with insulin action in mammals (Vincent, 2000). We designed and synthesized a new chromium complex, $[CrCl_2(C_{12}H_8N_2)_2]Cl$.

The Cr atom has site symmetry 2 and exhibits a distorted octahedral coordination, defined of four N atoms [Cr—N1: 2.062 (4) Å, Cr—N2: 2.073 (4) Å] from two symmetry-related 1,10-phenanthroline ligands and of two symmetry-related Cl ligands [2.2941 (15) Å]. The ligands are in a *cis* arrangement (Fig. 1.). The two planar 1,10-phenanthroline ligands show an approximate perpendicular orientation to each other [N1—Cr1—N2A: 91.81 (14) °, N2—Cr1—N2A: 88.5 (2) °]. These values are similar to that of the structure of the related compound *cis*-difluoridobis(1,10-phenanthroline)chromium(III) perchlorate monohydrate (Birk *et al.*, 2008).

The crystal packing of the title compound is displayed in Fig. 2.

Experimental

The title compound was prepared by the following method. $CrCl_3\cdot 6H_2O$ (0.665 g, 0.0025 mol) and zinc powder (0.1 g, 0.0015 mol) were added into methanol (30 ml) and then refluxed for 1 h. To the mixture 1,10-phenanthroline (0.99 g, 0.0050 mol) in methanol (20 ml) was added dropwise and allowed to reflux for another 30 min. The solution was put aside and X-ray quality crystals were obtained after two days at room temperature.

Refinement

The H atoms were treated as riding atoms, with C—H (CH) = 0.93 Å, and with $U_{iso}(H) = 1.5\ U_{eq}$ (parent atom).

Figures

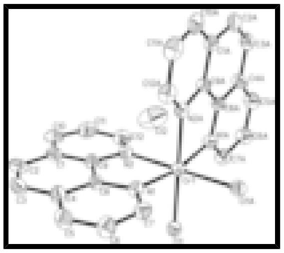


Fig. 1. The molecular structure of the title compound, showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level; H atoms were omitted for clarity. [Symmetry code A: $-x + 1, y, -z + 3/2$.]

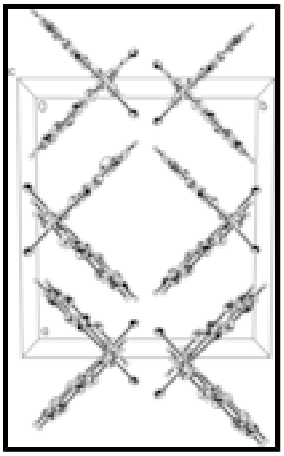


Fig. 2. The crystal structure of title compound, projected along the *c*-axis.

cis-Dichloridobis(1,10-phenanthroline)chromium(III) chloride

Crystal data

$[CrCl_2(C_{12}H_8N_2)_2]Cl$

M_r = 518.76

Monoclinic, $C2/c$

Hall symbol: -C 2yc

a = 15.446 (7) Å

b = 13.762 (6) Å

c = 12.536 (5) Å

β = 100.398 (6)°

V = 2621.1 (19) Å^3

Z = 4

$F(000)$ = 1052

D_x = 1.315 Mg m^{-3}

Mo $K\alpha$ radiation, λ = 0.71073 Å

Cell parameters from 1354 reflections

θ = 2.7–19.9°

μ = 0.76 mm^{-1}

T = 298 K

Block, red

0.40 × 0.10 × 0.10 mm

Data collection

Bruker SMART CCD area-detector diffractometer

Radiation source: fine-focus sealed tube

graphite

φ and ω scans

Absorption correction: multi-scan (*SADABS*; Sheldrick, 2000)

T_{min} = 0.751, T_{max} = 0.928

5182 measured reflections

2265 independent reflections

1778 reflections with $I > 2\sigma(I)$

R_{int} = 0.039

θ_{max} = 25.0°, θ_{min} = 2.4°

$h = -18 \rightarrow 18$

$k = -16 \rightarrow 16$

$l = -7 \rightarrow 14$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)]$ = 0.070

Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites

$wR(F^2) = 0.211$

$S = 1.07$

2265 reflections

150 parameters

0 restraints

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.1164P)^2 + 5.5982P]$
where $P = (F_o^2 + 2F_c^2)/3$

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

$\Delta\rho_{max} = 0.92$ e Å^{-3}

$\Delta\rho_{min} = -0.35$ e Å^{-3}

Special details

Experimental. the Cl-counter anion is only half occupied and equally disordered over two positions.

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2, conventional R-factors R are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating R-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	y	z	U_{iso}*/U_{eq}	Occ. (<1)
Cr1	0.5000	0.83468 (7)	0.7500	0.0338 (4)	
Cl1	0.60948 (7)	0.94883 (9)	0.76093 (10)	0.0481 (4)	
N1	0.5196 (2)	0.8199 (3)	0.9163 (3)	0.0386 (9)	
N2	0.5952 (2)	0.7267 (3)	0.7714 (3)	0.0383 (9)	
C1	0.6878 (3)	0.6322 (4)	0.9120 (4)	0.0467 (12)	
C2	0.7141 (4)	0.6151 (4)	1.0254 (5)	0.0600 (14)	
H2	0.7577	0.5695	1.0490	0.072*	
C3	0.6771 (4)	0.6636 (4)	1.0997 (5)	0.0576 (14)	
H3	0.6957	0.6507	1.1731	0.069*	
C4	0.6099 (3)	0.7344 (4)	1.0672 (4)	0.0495 (12)	
C5	0.5685 (4)	0.7889 (4)	1.1387 (4)	0.0582 (14)	
H5	0.5837	0.7789	1.2131	0.070*	
C6	0.5068 (4)	0.8555 (4)	1.0999 (4)	0.0555 (14)	
H6	0.4806	0.8923	1.1475	0.067*	
C7	0.4820 (3)	0.8693 (4)	0.9869 (4)	0.0478 (12)	
H7	0.4381	0.9142	0.9612	0.057*	
C8	0.5826 (3)	0.7539 (3)	0.9552 (4)	0.0380 (10)	
C9	0.6226 (3)	0.7021 (3)	0.8779 (4)	0.0370 (10)	
C10	0.7257 (4)	0.5865 (4)	0.8319 (5)	0.0588 (14)	
H10	0.7682	0.5387	0.8506	0.071*	
C11	0.6995 (4)	0.6130 (5)	0.7255 (5)	0.0650 (16)	
H11	0.7250	0.5839	0.6718	0.078*	
C12	0.6345 (4)	0.6836 (4)	0.6980 (5)	0.0531 (13)	
H12	0.6180	0.7012	0.6255	0.064*	

Cl2	0.6999 (6)	0.6655 (4)	0.4261 (5)	0.157 (3)	0.50

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cr1	0.0324 (6)	0.0364 (6)	0.0334 (6)	0.000	0.0081 (4)	0.000
Cl1	0.0390 (7)	0.0488 (7)	0.0549 (8)	−0.0079 (5)	0.0043 (5)	0.0066 (5)
N1	0.040 (2)	0.041 (2)	0.037 (2)	−0.0041 (17)	0.0121 (17)	−0.0010 (16)
N2	0.0376 (19)	0.039 (2)	0.040 (2)	0.0022 (16)	0.0107 (16)	0.0015 (17)
C1	0.040 (3)	0.047 (3)	0.053 (3)	0.002 (2)	0.007 (2)	0.006 (2)
C2	0.053 (3)	0.062 (3)	0.061 (3)	0.012 (3)	−0.001 (3)	0.015 (3)
C3	0.057 (3)	0.064 (4)	0.046 (3)	0.000 (3)	−0.005 (3)	0.010 (3)
C4	0.051 (3)	0.055 (3)	0.041 (3)	−0.009 (2)	0.004 (2)	−0.001 (2)
C5	0.070 (4)	0.068 (4)	0.037 (3)	−0.005 (3)	0.009 (2)	0.001 (3)
C6	0.069 (3)	0.062 (3)	0.039 (3)	−0.002 (3)	0.020 (3)	−0.012 (2)
C7	0.054 (3)	0.047 (3)	0.045 (3)	0.004 (2)	0.016 (2)	−0.005 (2)
C8	0.035 (2)	0.041 (2)	0.038 (2)	−0.0057 (19)	0.0053 (18)	0.0008 (19)
C9	0.034 (2)	0.036 (2)	0.041 (2)	−0.0036 (18)	0.0070 (19)	0.001 (2)
C10	0.052 (3)	0.055 (3)	0.071 (4)	0.021 (3)	0.016 (3)	0.008 (3)
C11	0.070 (4)	0.064 (4)	0.068 (4)	0.025 (3)	0.031 (3)	−0.004 (3)
C12	0.059 (3)	0.057 (3)	0.047 (3)	0.011 (3)	0.020 (3)	−0.002 (2)
Cl2	0.265 (8)	0.125 (4)	0.101 (4)	−0.037 (5)	0.086 (5)	−0.027 (3)

Geometric parameters (Å, °)

Cr1—N1	2.062 (4)	C3—C4	1.428 (8)
Cr1—N1^{i}	2.062 (4)	C3—H3	0.9300
Cr1—N2^{i}	2.073 (4)	C4—C5	1.408 (8)
Cr1—N2	2.073 (4)	C4—C8	1.417 (7)
Cr1—Cl1	2.2941 (15)	C5—C6	1.348 (8)
Cr1—Cl1^{i}	2.2941 (15)	C5—H5	0.9300
N1—C7	1.330 (6)	C6—C7	1.411 (7)
N1—C8	1.356 (6)	C6—H6	0.9300
N2—C12	1.330 (6)	C7—H7	0.9300
N2—C9	1.368 (6)	C8—C9	1.431 (6)
C1—C10	1.399 (8)	C10—C11	1.372 (8)
C1—C9	1.403 (7)	C10—H10	0.9300
C1—C2	1.426 (8)	C11—C12	1.395 (8)
C2—C3	1.353 (8)	C11—H11	0.9300
C2—H2	0.9300	C12—H12	0.9300
N1—Cr1—N1^{i}	168.7 (2)	C4—C3—H3	119.5
N1—Cr1—N2^{i}	91.81 (14)	C5—C4—C8	116.2 (5)
N1^{i}—Cr1—N2^{i}	80.07 (15)	C5—C4—C3	124.9 (5)
N1—Cr1—N2	80.07 (15)	C8—C4—C3	118.9 (5)
N1^{i}—Cr1—N2	91.81 (14)	C6—C5—C4	120.4 (5)
N2^{i}—Cr1—N2	88.5 (2)	C6—C5—H5	119.8
N1—Cr1—Cl1	92.00 (11)	C4—C5—H5	119.8

N1^{i}—Cr1—Cl1	95.73 (11)	C5—C6—C7	120.0 (5)
N2^{i}—Cr1—Cl1	175.09 (11)	C5—C6—H6	120.0
N2—Cr1—Cl1	89.14 (11)	C7—C6—H6	120.0
N1—Cr1—Cl1^{i}	95.73 (11)	N1—C7—C6	121.8 (5)
N1^{i}—Cr1—Cl1^{i}	92.00 (11)	N1—C7—H7	119.1
N2^{i}—Cr1—Cl1^{i}	89.14 (11)	C6—C7—H7	119.1
N2—Cr1—Cl1^{i}	175.09 (10)	N1—C8—C4	123.3 (4)
Cl1—Cr1—Cl1^{i}	93.56 (8)	N1—C8—C9	117.4 (4)
C7—N1—C8	118.3 (4)	C4—C8—C9	119.3 (4)
C7—N1—Cr1	128.4 (3)	N2—C9—C1	123.1 (4)
C8—N1—Cr1	113.2 (3)	N2—C9—C8	116.2 (4)
C12—N2—C9	117.7 (4)	C1—C9—C8	120.6 (4)
C12—N2—Cr1	129.2 (3)	C11—C10—C1	119.4 (5)
C9—N2—Cr1	113.0 (3)	C11—C10—H10	120.3
C10—C1—C9	117.3 (5)	C1—C10—H10	120.3
C10—C1—C2	124.2 (5)	C10—C11—C12	119.9 (5)
C9—C1—C2	118.5 (5)	C10—C11—H11	120.1
C3—C2—C1	121.7 (5)	C12—C11—H11	120.1
C3—C2—H2	119.1	N2—C12—C11	122.5 (5)
C1—C2—H2	119.1	N2—C12—H12	118.7
C2—C3—C4	121.1 (5)	C11—C12—H12	118.7
C2—C3—H3	119.5		

N1^{i}—Cr1—N1—C7	−137.2 (4)	Cr1—N1—C7—C6	−175.5 (4)
N2^{i}—Cr1—N1—C7	−93.5 (4)	C5—C6—C7—N1	−1.8 (8)
N2—Cr1—N1—C7	178.4 (4)	C7—N1—C8—C4	0.4 (7)
Cl1—Cr1—N1—C7	89.6 (4)	Cr1—N1—C8—C4	177.3 (4)
Cl1^{i}—Cr1—N1—C7	−4.2 (4)	C7—N1—C8—C9	−179.5 (4)
N1^{i}—Cr1—N1—C8	46.3 (3)	Cr1—N1—C8—C9	−2.6 (5)
N2^{i}—Cr1—N1—C8	90.0 (3)	C5—C4—C8—N1	−0.8 (7)
N2—Cr1—N1—C8	1.9 (3)	C3—C4—C8—N1	−179.7 (4)
Cl1—Cr1—N1—C8	−86.9 (3)	C5—C4—C8—C9	179.2 (4)
Cl1^{i}—Cr1—N1—C8	179.3 (3)	C3—C4—C8—C9	0.2 (7)
N1—Cr1—N2—C12	−177.2 (5)	C12—N2—C9—C1	−1.9 (7)
N1^{i}—Cr1—N2—C12	10.7 (4)	Cr1—N2—C9—C1	−178.7 (4)
N2^{i}—Cr1—N2—C12	90.7 (4)	C12—N2—C9—C8	176.5 (4)
Cl1—Cr1—N2—C12	−85.0 (4)	Cr1—N2—C9—C8	−0.3 (5)
Cl1^{i}—Cr1—N2—C12	151.5 (11)	C10—C1—C9—N2	0.1 (7)
N1—Cr1—N2—C9	−0.8 (3)	C2—C1—C9—N2	177.4 (5)
N1^{i}—Cr1—N2—C9	−172.9 (3)	C10—C1—C9—C8	−178.3 (5)
N2^{i}—Cr1—N2—C9	−92.9 (3)	C2—C1—C9—C8	−1.0 (7)
Cl1—Cr1—N2—C9	91.4 (3)	N1—C8—C9—N2	2.0 (6)
Cl1^{i}—Cr1—N2—C9	−32.1 (15)	C4—C8—C9—N2	−178.0 (4)
C10—C1—C2—C3	177.9 (5)	N1—C8—C9—C1	−179.6 (4)
C9—C1—C2—C3	0.8 (8)	C4—C8—C9—C1	0.5 (7)
C1—C2—C3—C4	−0.1 (9)	C9—C1—C10—C11	1.4 (8)

C2—C3—C4—C5	−179.3 (6)	C2—C1—C10—C11	−175.7 (6)
C2—C3—C4—C8	−0.4 (8)	C1—C10—C11—C12	−1.2 (9)
C8—C4—C5—C6	−0.2 (8)	C9—N2—C12—C11	2.2 (8)
C3—C4—C5—C6	178.7 (5)	Cr1—N2—C12—C11	178.4 (4)
C4—C5—C6—C7	1.4 (9)	C10—C11—C12—N2	−0.7 (9)
C8—N1—C7—C6	0.9 (7)		

Symmetry codes: (i) $-x+1$, y, $-z+3/2$.

Fig. 1

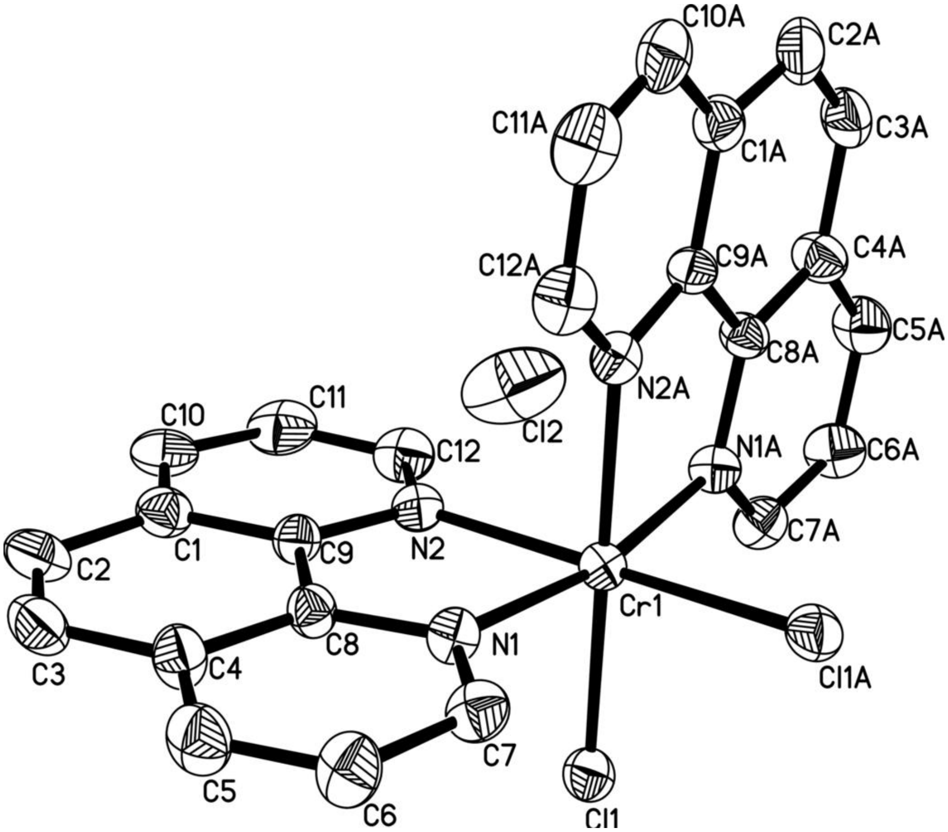

C10A
C2A
C11A
C1A
C3A
C12A
C9A
C4A
C8A
C5A
N2A
Cl2
C10
C11
C12
N1A
C6A
C7A
C1
C9
N2
C2
Cr1
C8
N1
C3
C4
Cl1A
C7
C5
C6
Cl1

Fig. 2