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trans-Dichloridobis(2,2-dimethylpropane-1,3-diamine- κ^2N,N')chromium(III) perchlorate

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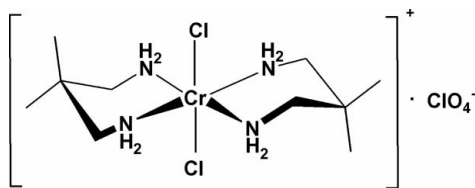
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Key indicators: single-crystal X-ray study; $T = 298$ K; mean $\sigma(\text{C}-\text{C}) = 0.005$ Å; disorder in solvent or counterion; R factor = 0.051; wR factor = 0.146; data-to-parameter ratio = 19.8.

In the title salt, $[\text{CrCl}_2(\text{C}_5\text{H}_{14}\text{N}_2)_2]\text{ClO}_4$, the Cr atom is in a *trans*- CrCl_2N_4 octahedral environment comprising the four N atoms of two chelating 2,2-dimethylpropane-1,3-diamine ligands and two Cl atoms. The two six-membered CrC_3N_2 rings in the cation adopt *anti* chair–chair conformations with respect to each other. The perchlorate anion is disordered over two positions in respect of the Cl and an O atom in a 6:4 ratio. $\text{N}-\text{H}\cdots\text{O}$ hydrogen bonds link the cations and anions into a layer structure.

Related literature

For the synthesis, see: House (1986). For related structures, see: Choi *et al.* (2002, 2007). For the spectroscopic properties, see: Choi (2000); Poon & Pun (1980).



Experimental

Crystal data

 $[\text{CrCl}_2(\text{C}_5\text{H}_{14}\text{N}_2)_2]\text{ClO}_4$
 $M_r = 426.71$

 Monoclinic, $P2_1/c$
 $a = 6.6373$ (6) Å

 $b = 20.767$ (2) Å
 $c = 13.878$ (2) Å
 $\beta = 100.249$ (9)°
 $V = 1882.4$ (4) Å³
 $Z = 4$

 Mo $K\alpha$ radiation
 $\mu = 1.05$ mm⁻¹
 $T = 298$ (2) K
 $0.32 \times 0.30 \times 0.25$ mm

Data collection

 Stoe Stadi-4 diffractometer
 Absorption correction: numerical
 (*X-SHAPE*; Stoe & Cie, 1996)
 $T_{\text{min}} = 0.805$, $T_{\text{max}} = 0.942$
 4305 measured reflections

 4305 independent reflections
 3453 reflections with $I > 2\sigma(I)$
 3 standard reflections
 frequency: 60 min
 intensity decay: 2.7%

Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.051$
 $wR(F^2) = 0.146$
 $S = 1.11$
 4305 reflections

 217 parameters
 H-atom parameters constrained
 $\Delta\rho_{\text{max}} = 0.54$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.81$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

| <i>D</i> — <i>H</i> ⋯ <i>A</i> | <i>D</i> — <i>H</i> | <i>H</i> ⋯ <i>A</i> | <i>D</i> ⋯ <i>A</i> | <i>D</i> — <i>H</i> ⋯ <i>A</i> |
|---|---------------------|---------------------|---------------------|--------------------------------|
| N1—H1B ⁱ ⋯O2 ⁱ | 0.90 | 2.29 | 3.030 (5) | 139 |
| N2—H2A ⁿ ⋯O3 ⁱⁱ | 0.90 | 2.23 | 3.099 (6) | 162 |
| N2—H2A ⁿ ⋯O4A ⁱⁱ | 0.90 | 2.42 | 3.183 (6) | 143 |
| N2—H2B ⁿ ⋯O4B | 0.90 | 2.36 | 3.217 (9) | 159 |
| N3—H3A ⁿ ⋯O4A ⁱⁱ | 0.90 | 2.60 | 3.482 (7) | 168 |
| N4—H4B ⁿ ⋯O2 ⁱ | 0.90 | 2.14 | 3.030 (5) | 172 |
| N4—H4A ⁿ ⋯O4A ⁱⁱⁱ | 0.90 | 2.54 | 3.403 (8) | 161 |

Symmetry codes: (i) $x, -y + \frac{1}{2}, z + \frac{1}{2}$; (ii) $x - 1, y, z$; (iii) $x - 1, -y + \frac{1}{2}, z + \frac{1}{2}$; (iv) $x + 1, y, z$.

Data collection: *STADI-4* (Stoe & Cie, 1996); cell refinement: *STADI-4*; data reduction: *X-RED* (Stoe & Cie, 1996); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: NG2499).

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supplementary materials

Acta Cryst. (2008). E64, m1429 [doi:10.1107/S1600536808032911]

***trans*-Dichloridobis(2,2-dimethylpropane-1,3-diamine- κ^2N,N')chromium(III) perchlorate**

J.-H. Choi, S. H. Lee and U. Lee

Comment

The $[\text{Cr}(\text{Me}_2\text{tn})_2\text{L}_2]^+$ (Me_2tn =2,2-dimethylpropane-1,3-diamine, L = monodentate) cation can exist as *trans* and *cis* geometric isomers. Especially, there are two possible conformations with respect to the six-membered rings in the *trans* isomer. The carbon atoms of the two chelate rings of the two conformers may be on the same side (*syn* conformer) or on opposite side (*anti* conformer) of the coordination plane (Choi *et al.*, 2002; Choi *et al.*, 2007). The *syn* or *anti* conformational stereochemistry of the six-membered chelate rings can not be readily determined by spectroscopic and physicochemical methods (Poon & Pun, 1980; Choi, 2000). In order to examine the influences of counter anions and packing forces of the crystal on the conformations, we have undertaken the X-ray structural analysis of *trans*- $[\text{Cr}(\text{Me}_2\text{tn})_2\text{Cl}_2]\text{ClO}_4$, (I).

The title complex has approximate C_i symmetry. The two chelate rings in the complex cation are only in anti chair-chair conformation with respect to each other (Fig.1). The Cr—N and Cr—Cl bond length are very similar to those of the *trans*- $[\text{Cr}(\text{Me}_2\text{tn})_2\text{Cl}_2]\text{Cl}$ (Choi *et al.*, 2007). However, the significant difference between these two crystal structures is the orientations with respect to the six-membered chelate rings of two Me_2tn ligands in the same *trans* geometry. The complex is stabilized by the formation of the extensive hydrogen bonds (Table 1).

Experimental

The complex *trans*- $[\text{Cr}(\text{Me}_2\text{tn})_2\text{Cl}_2]\text{ClO}_4$ was prepared according to the literature (House, 1986). The crystalline product deposited with ice-bath cooling was filtered off, and washed with cold 2-propanol and then diethyl ether. Recrystallization of the crude precipitate from 0.5M HCl and 70% HClO_4 solution afforded dark green crystals suitable for X-ray analysis. Anal. Found: C, 28.02; H, 6.50; N, 13.08%. Calc. for $\text{C}_{10}\text{H}_{28}\text{Cl}_3\text{CrN}_4\text{O}_4$: C, 28.15; H, 6.61; N, 13.13%.

Refinement

H atoms were positioned geometrically and refined as riding atoms, with C—H = 0.97 (methylene), 0.96 (methyl) Å, and N—H = 0.90 Å respectively, $\text{Uiso}(\text{H}) = 1.2\text{Ueq}(\text{C} \ \& \ \text{N})$. The Cl and one O atoms in the perchlorate anion are disordered over two positions with site-occupancy factors fixed at 0.60 (for atoms labelled A) and 0.40 (for atoms labelled B) in the final refinement.

Figures

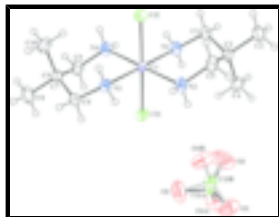


Fig. 1. Perspective view (30% probability level) of title compound.

trans-Dichloridobis(2,2-dimethylpropane-1,3-diamine- κ^2N,N')chromium(III) perchlorate

Crystal data

$[\text{CrCl}_2(\text{C}_5\text{H}_{14}\text{N}_2)_2]\text{ClO}_4$

$M_r = 426.71$

Monoclinic, $P2_1/c$

Hall symbol: $-P_2ybc$

$a = 6.6373$ (6) Å

$b = 20.767$ (2) Å

$c = 13.878$ (2) Å

$\beta = 100.249$ (9)°

$V = 1882.4$ (4) Å³

$Z = 4$

$F_{000} = 892$

$D_x = 1.506$ Mg m⁻³

Mo $K\alpha$ radiation

$\lambda = 0.71069$ Å

Cell parameters from 54 reflections

$\theta = 9.5\text{--}10.4^\circ$

$\mu = 1.05$ mm⁻¹

$T = 298$ (2) K

Block, green

$0.32 \times 0.30 \times 0.25$ mm

Data collection

Stoe Stadi-4
diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

$T = 298$ (2) K

$\omega/2-\theta$ scans

Absorption correction: Numerical
(X-SHAPE; Stoe & Cie, 1996)

$T_{\min} = 0.805$, $T_{\max} = 0.942$

4305 measured reflections

4305 independent reflections

3453 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.0000$

$\theta_{\max} = 27.5^\circ$

$\theta_{\min} = 1.8^\circ$

$h = -8 \rightarrow 8$

$k = 0 \rightarrow 26$

$l = 0 \rightarrow 18$

3 standard reflections

every 60 min

intensity decay: 2.7%

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.051$

$wR(F^2) = 0.146$

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0733P)^2 + 1.4729P]$

$S = 1.11$

4305 reflections

217 parameters

Primary atom site location: structure-invariant direct methods

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

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

$\Delta\rho_{\max} = 0.54 \text{ e } \text{\AA}^{-3}$

$\Delta\rho_{\min} = -0.81 \text{ e } \text{\AA}^{-3}$

Extinction correction: none

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds 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 $F^2 > 2\sigma(F^2)$ 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 (\AA^2)

| | x | y | z | $U_{\text{iso}}^*/U_{\text{eq}}$ | Occ. (<1) |
|------|--------------|--------------|--------------|----------------------------------|-----------|
| Cr | 0.43254 (6) | 0.23278 (2) | 0.57694 (3) | 0.03056 (15) | |
| Cl1 | 0.67222 (13) | 0.25634 (5) | 0.47898 (7) | 0.0540 (2) | |
| Cl2 | 0.18895 (12) | 0.21076 (4) | 0.67312 (7) | 0.0493 (2) | |
| Cl3A | 0.9541 (3) | 0.11988 (10) | 0.31249 (14) | 0.0471 (4) | 0.60 |
| Cl3B | 0.8697 (4) | 0.11095 (15) | 0.3227 (2) | 0.0434 (6) | 0.40 |
| O4B | 0.6845 (14) | 0.1083 (5) | 0.3541 (7) | 0.096 (3) | 0.40 |
| O4A | 1.1593 (9) | 0.1344 (3) | 0.3048 (5) | 0.0913 (18) | 0.60 |
| O1 | 0.8766 (6) | 0.06609 (19) | 0.2505 (3) | 0.0957 (12) | |
| O2 | 0.8510 (7) | 0.1748 (2) | 0.2793 (3) | 0.1124 (15) | |
| O3 | 0.9840 (9) | 0.1052 (3) | 0.4093 (3) | 0.140 (2) | |
| N1 | 0.6658 (4) | 0.20091 (13) | 0.6894 (2) | 0.0420 (6) | |
| H1AN | 0.7834 | 0.2010 | 0.6656 | 0.050* | |
| H1BN | 0.6795 | 0.2305 | 0.7375 | 0.050* | |
| N2 | 0.4181 (4) | 0.13895 (13) | 0.5210 (2) | 0.0466 (7) | |
| H2AN | 0.2998 | 0.1354 | 0.4782 | 0.056* | |
| H2BN | 0.5200 | 0.1348 | 0.4865 | 0.056* | |
| N3 | 0.1983 (4) | 0.26418 (12) | 0.46576 (19) | 0.0385 (6) | |
| H3AN | 0.1880 | 0.2354 | 0.4167 | 0.046* | |
| H3BN | 0.0806 | 0.2622 | 0.4893 | 0.046* | |
| N4 | 0.4506 (4) | 0.32609 (13) | 0.6350 (2) | 0.0448 (6) | |
| H4AN | 0.3526 | 0.3297 | 0.6719 | 0.054* | |
| H4BN | 0.5717 | 0.3295 | 0.6758 | 0.054* | |
| C1 | 0.6476 (5) | 0.13679 (16) | 0.7353 (2) | 0.0444 (7) | |
| H1A | 0.5282 | 0.1372 | 0.7667 | 0.053* | |
| H1B | 0.7668 | 0.1302 | 0.7860 | 0.053* | |
| C2 | 0.6295 (5) | 0.07995 (15) | 0.6641 (3) | 0.0411 (7) | |

supplementary materials

| | | | | |
|------|------------|--------------|------------|-------------|
| C3 | 0.6162 (7) | 0.01867 (19) | 0.7255 (3) | 0.0644 (11) |
| H3A | 0.6049 | -0.0184 | 0.6836 | 0.077* |
| H3B | 0.4982 | 0.0212 | 0.7565 | 0.077* |
| H3C | 0.7374 | 0.0151 | 0.7746 | 0.077* |
| C4 | 0.8169 (6) | 0.07509 (19) | 0.6154 (3) | 0.0569 (9) |
| H4A | 0.8011 | 0.0393 | 0.5708 | 0.068* |
| H4B | 0.9368 | 0.0688 | 0.6645 | 0.068* |
| H4C | 0.8311 | 0.1141 | 0.5801 | 0.068* |
| C5 | 0.4308 (5) | 0.08283 (16) | 0.5890 (3) | 0.0495 (8) |
| H5A | 0.4181 | 0.0435 | 0.5507 | 0.059* |
| H5B | 0.3161 | 0.0846 | 0.6235 | 0.059* |
| C6 | 0.2096 (5) | 0.32895 (16) | 0.4223 (2) | 0.0441 (7) |
| H6A | 0.0874 | 0.3356 | 0.3735 | 0.053* |
| H6B | 0.3261 | 0.3300 | 0.3889 | 0.053* |
| C7 | 0.2293 (5) | 0.38464 (15) | 0.4953 (2) | 0.0410 (7) |
| C8 | 0.2337 (8) | 0.4469 (2) | 0.4358 (4) | 0.0715 (12) |
| H8A | 0.2455 | 0.4833 | 0.4791 | 0.086* |
| H8B | 0.1095 | 0.4503 | 0.3886 | 0.086* |
| H8C | 0.3488 | 0.4459 | 0.4026 | 0.086* |
| C9 | 0.4311 (5) | 0.38267 (15) | 0.5681 (3) | 0.0469 (8) |
| H9A | 0.5435 | 0.3821 | 0.5320 | 0.056* |
| H9B | 0.4433 | 0.4217 | 0.6071 | 0.056* |
| C10 | 0.0472 (6) | 0.38715 (19) | 0.5480 (3) | 0.0571 (9) |
| H10A | 0.0640 | 0.4224 | 0.5935 | 0.069* |
| H10B | 0.0388 | 0.3475 | 0.5827 | 0.069* |
| H10C | -0.0762 | 0.3932 | 0.5010 | 0.069* |

Atomic displacement parameters (\AA^2)

| | U^{11} | U^{22} | U^{33} | U^{12} | U^{13} | U^{23} |
|------|-------------|-------------|-------------|--------------|--------------|---------------|
| Cr | 0.0243 (2) | 0.0293 (2) | 0.0375 (3) | 0.00052 (16) | 0.00378 (17) | -0.00218 (18) |
| Cl1 | 0.0375 (4) | 0.0574 (5) | 0.0726 (6) | 0.0075 (4) | 0.0244 (4) | 0.0073 (4) |
| Cl2 | 0.0377 (4) | 0.0561 (5) | 0.0578 (5) | 0.0062 (3) | 0.0185 (4) | 0.0123 (4) |
| Cl3A | 0.0561 (12) | 0.0449 (9) | 0.0375 (8) | 0.0080 (9) | 0.0011 (9) | -0.0001 (6) |
| Cl3B | 0.0381 (13) | 0.0507 (14) | 0.0405 (12) | 0.0016 (11) | 0.0046 (11) | 0.0027 (9) |
| O4B | 0.077 (5) | 0.122 (8) | 0.096 (6) | -0.025 (5) | 0.034 (5) | -0.052 (6) |
| O4A | 0.065 (3) | 0.100 (4) | 0.099 (4) | -0.011 (3) | -0.013 (3) | 0.000 (4) |
| O1 | 0.110 (3) | 0.091 (3) | 0.092 (2) | -0.029 (2) | 0.034 (2) | -0.038 (2) |
| O2 | 0.126 (3) | 0.077 (2) | 0.122 (3) | 0.025 (2) | -0.010 (3) | 0.024 (2) |
| O3 | 0.167 (5) | 0.178 (5) | 0.062 (2) | 0.004 (4) | -0.019 (3) | 0.017 (3) |
| N1 | 0.0319 (12) | 0.0373 (13) | 0.0526 (16) | 0.0014 (10) | -0.0036 (11) | -0.0012 (12) |
| N2 | 0.0489 (15) | 0.0369 (14) | 0.0502 (16) | 0.0038 (12) | -0.0017 (12) | -0.0068 (12) |
| N3 | 0.0332 (12) | 0.0391 (14) | 0.0420 (14) | 0.0029 (10) | 0.0031 (10) | -0.0021 (11) |
| N4 | 0.0485 (15) | 0.0358 (13) | 0.0460 (15) | 0.0014 (11) | -0.0029 (12) | -0.0048 (12) |
| C1 | 0.0439 (17) | 0.0408 (17) | 0.0467 (17) | 0.0064 (13) | 0.0035 (14) | 0.0064 (14) |
| C2 | 0.0373 (15) | 0.0323 (15) | 0.0554 (19) | 0.0035 (12) | 0.0126 (13) | 0.0059 (13) |
| C3 | 0.070 (3) | 0.044 (2) | 0.084 (3) | 0.0058 (18) | 0.024 (2) | 0.0195 (19) |
| C4 | 0.0474 (19) | 0.053 (2) | 0.076 (3) | 0.0068 (16) | 0.0259 (18) | 0.0017 (19) |

| | | | | | | |
|-----|-------------|-------------|-------------|--------------|-------------|--------------|
| C5 | 0.0434 (17) | 0.0322 (16) | 0.071 (2) | -0.0017 (13) | 0.0040 (16) | -0.0052 (15) |
| C6 | 0.0467 (17) | 0.0487 (18) | 0.0371 (16) | 0.0131 (14) | 0.0080 (13) | 0.0041 (14) |
| C7 | 0.0417 (16) | 0.0352 (15) | 0.0479 (17) | 0.0068 (12) | 0.0133 (13) | 0.0069 (13) |
| C8 | 0.083 (3) | 0.045 (2) | 0.086 (3) | 0.007 (2) | 0.015 (2) | 0.022 (2) |
| C9 | 0.0448 (18) | 0.0321 (15) | 0.064 (2) | -0.0009 (13) | 0.0094 (16) | -0.0003 (14) |
| C10 | 0.049 (2) | 0.052 (2) | 0.077 (3) | 0.0090 (16) | 0.0259 (19) | -0.0037 (18) |

Geometric parameters (Å, °)

| | | | |
|-----------|-------------|------------|-----------|
| Cr—N3 | 2.090 (3) | C1—H1A | 0.9700 |
| Cr—N2 | 2.093 (3) | C1—H1B | 0.9700 |
| Cr—N4 | 2.094 (3) | C2—C4 | 1.522 (4) |
| Cr—N1 | 2.100 (3) | C2—C5 | 1.530 (5) |
| Cr—Cl2 | 2.3179 (9) | C2—C3 | 1.543 (5) |
| Cr—Cl1 | 2.3212 (9) | C3—H3A | 0.9600 |
| Cl3A—Cl3B | 0.629 (2) | C3—H3B | 0.9600 |
| Cl3A—O3 | 1.357 (4) | C3—H3C | 0.9600 |
| Cl3A—O2 | 1.368 (4) | C4—H4A | 0.9600 |
| Cl3A—O4A | 1.418 (6) | C4—H4B | 0.9600 |
| Cl3A—O1 | 1.447 (4) | C4—H4C | 0.9600 |
| Cl3B—O3 | 1.308 (5) | C5—H5A | 0.9700 |
| Cl3B—O1 | 1.374 (5) | C5—H5B | 0.9700 |
| Cl3B—O4B | 1.377 (9) | C6—C7 | 1.528 (5) |
| Cl3B—O2 | 1.453 (5) | C6—H6A | 0.9700 |
| N1—C1 | 1.490 (4) | C6—H6B | 0.9700 |
| N1—H1AN | 0.9000 | C7—C10 | 1.520 (5) |
| N1—H1BN | 0.9000 | C7—C9 | 1.528 (5) |
| N2—C5 | 1.492 (4) | C7—C8 | 1.537 (5) |
| N2—H2AN | 0.9000 | C8—H8A | 0.9600 |
| N2—H2BN | 0.9000 | C8—H8B | 0.9600 |
| N3—C6 | 1.481 (4) | C8—H8C | 0.9600 |
| N3—H3AN | 0.9000 | C9—H9A | 0.9700 |
| N3—H3BN | 0.9000 | C9—H9B | 0.9700 |
| N4—C9 | 1.488 (4) | C10—H10A | 0.9600 |
| N4—H4AN | 0.9000 | C10—H10B | 0.9600 |
| N4—H4BN | 0.9000 | C10—H10C | 0.9600 |
| C1—C2 | 1.530 (5) | | |
| N3—Cr—N2 | 92.17 (10) | N1—C1—C2 | 114.6 (3) |
| N3—Cr—N4 | 88.82 (10) | N1—C1—H1A | 108.6 |
| N2—Cr—N4 | 179.01 (11) | C2—C1—H1A | 108.6 |
| N3—Cr—N1 | 179.46 (11) | N1—C1—H1B | 108.6 |
| N2—Cr—N1 | 87.78 (11) | C2—C1—H1B | 108.6 |
| N4—Cr—N1 | 91.24 (11) | H1A—C1—H1B | 107.6 |
| N3—Cr—Cl2 | 89.04 (8) | C4—C2—C5 | 111.9 (3) |
| N2—Cr—Cl2 | 92.25 (9) | C4—C2—C1 | 111.2 (3) |
| N4—Cr—Cl2 | 87.65 (9) | C5—C2—C1 | 111.7 (3) |
| N1—Cr—Cl2 | 90.42 (8) | C4—C2—C3 | 108.8 (3) |
| N3—Cr—Cl1 | 89.95 (8) | C5—C2—C3 | 106.4 (3) |
| N2—Cr—Cl1 | 88.28 (9) | C1—C2—C3 | 106.6 (3) |

supplementary materials

| | | | |
|-----------------|-------------|-----------------|-----------|
| N4—Cr—Cl1 | 91.84 (9) | C2—C3—H3A | 109.5 |
| N1—Cr—Cl1 | 90.59 (8) | C2—C3—H3B | 109.5 |
| Cl2—Cr—Cl1 | 178.87 (4) | H3A—C3—H3B | 109.5 |
| O3—Cl3A—O2 | 119.8 (4) | C2—C3—H3C | 109.5 |
| Cl3B—Cl3A—O4A | 170.0 (5) | H3A—C3—H3C | 109.5 |
| O3—Cl3A—O4A | 98.6 (4) | H3B—C3—H3C | 109.5 |
| O2—Cl3A—O4A | 103.2 (4) | C2—C4—H4A | 109.5 |
| O3—Cl3A—O1 | 112.9 (3) | C2—C4—H4B | 109.5 |
| O2—Cl3A—O1 | 109.9 (3) | H4A—C4—H4B | 109.5 |
| O4A—Cl3A—O1 | 111.3 (3) | C2—C4—H4C | 109.5 |
| O3—Cl3A—O4B | 70.5 (4) | H4A—C4—H4C | 109.5 |
| O2—Cl3A—O4B | 77.1 (4) | H4B—C4—H4C | 109.5 |
| O4A—Cl3A—O4B | 166.7 (4) | N2—C5—C2 | 114.0 (3) |
| O1—Cl3A—O4B | 80.6 (3) | N2—C5—H5A | 108.8 |
| O3—Cl3B—O1 | 121.3 (4) | C2—C5—H5A | 108.8 |
| O3—Cl3B—O4B | 96.3 (5) | N2—C5—H5B | 108.8 |
| O1—Cl3B—O4B | 110.6 (4) | C2—C5—H5B | 108.8 |
| O3—Cl3B—O2 | 117.1 (4) | H5A—C5—H5B | 107.7 |
| O1—Cl3B—O2 | 109.2 (3) | N3—C6—C7 | 115.0 (3) |
| O4B—Cl3B—O2 | 98.8 (6) | N3—C6—H6A | 108.5 |
| O3—Cl3B—O4A | 74.2 (3) | C7—C6—H6A | 108.5 |
| O1—Cl3B—O4A | 85.3 (3) | N3—C6—H6B | 108.5 |
| O4B—Cl3B—O4A | 164.1 (5) | C7—C6—H6B | 108.5 |
| O2—Cl3B—O4A | 75.2 (3) | H6A—C6—H6B | 107.5 |
| C1—N1—Cr | 119.62 (19) | C10—C7—C6 | 111.3 (3) |
| C1—N1—H1AN | 107.4 | C10—C7—C9 | 111.2 (3) |
| Cr—N1—H1AN | 107.4 | C6—C7—C9 | 112.3 (3) |
| C1—N1—H1BN | 107.4 | C10—C7—C8 | 108.8 (3) |
| Cr—N1—H1BN | 107.4 | C6—C7—C8 | 106.7 (3) |
| H1AN—N1—H1BN | 106.9 | C9—C7—C8 | 106.2 (3) |
| C5—N2—Cr | 119.9 (2) | C7—C8—H8A | 109.5 |
| C5—N2—H2AN | 107.3 | C7—C8—H8B | 109.5 |
| Cr—N2—H2AN | 107.3 | H8A—C8—H8B | 109.5 |
| C5—N2—H2BN | 107.3 | C7—C8—H8C | 109.5 |
| Cr—N2—H2BN | 107.3 | H8A—C8—H8C | 109.5 |
| H2AN—N2—H2BN | 106.9 | H8B—C8—H8C | 109.5 |
| C6—N3—Cr | 119.9 (2) | N4—C9—C7 | 113.7 (3) |
| C6—N3—H3AN | 107.3 | N4—C9—H9A | 108.8 |
| Cr—N3—H3AN | 107.3 | C7—C9—H9A | 108.8 |
| C6—N3—H3BN | 107.3 | N4—C9—H9B | 108.8 |
| Cr—N3—H3BN | 107.3 | C7—C9—H9B | 108.8 |
| H3AN—N3—H3BN | 106.9 | H9A—C9—H9B | 107.7 |
| C9—N4—Cr | 119.9 (2) | C7—C10—H10A | 109.5 |
| C9—N4—H4AN | 107.4 | C7—C10—H10B | 109.5 |
| Cr—N4—H4AN | 107.4 | H10A—C10—H10B | 109.5 |
| C9—N4—H4BN | 107.4 | C7—C10—H10C | 109.5 |
| Cr—N4—H4BN | 107.4 | H10A—C10—H10C | 109.5 |
| H4AN—N4—H4BN | 106.9 | H10B—C10—H10C | 109.5 |
| O2—Cl3A—Cl3B—O3 | -123.6 (4) | O1—Cl3A—O2—Cl3B | 67.3 (4) |

| | | | |
|-------------------|------------|------------------|------------|
| O4A—C13A—C13B—O3 | 20 (3) | O4B—C13A—O2—C13B | -7.5 (5) |
| O1—C13A—C13B—O3 | 123.2 (4) | O3—C13B—O2—C13A | 67.5 (5) |
| O4B—C13A—C13B—O3 | -80 (3) | O1—C13B—O2—C13A | -75.3 (4) |
| O3—C13A—C13B—O1 | -123.2 (4) | O4B—C13B—O2—C13A | 169.2 (6) |
| O2—C13A—C13B—O1 | 113.2 (3) | O4A—C13B—O2—C13A | 4.3 (4) |
| O4A—C13A—C13B—O1 | -103 (3) | O1—C13B—O3—C13A | 76.7 (5) |
| O4B—C13A—C13B—O1 | 157 (3) | O4B—C13B—O3—C13A | -164.5 (7) |
| O3—C13A—C13B—O4B | 80 (3) | O2—C13B—O3—C13A | -61.3 (5) |
| O2—C13A—C13B—O4B | -43 (3) | O4A—C13B—O3—C13A | 2.4 (4) |
| O4A—C13A—C13B—O4B | 100 (4) | O2—C13A—O3—C13B | 72.8 (5) |
| O1—C13A—C13B—O4B | -157 (3) | O4A—C13A—O3—C13B | -176.6 (6) |
| O3—C13A—C13B—O2 | 123.6 (4) | O1—C13A—O3—C13B | -59.0 (5) |
| O4A—C13A—C13B—O2 | 143 (3) | O4B—C13A—O3—C13B | 11.2 (5) |
| O1—C13A—C13B—O2 | -113.2 (3) | N2—Cr—N1—C1 | -41.0 (2) |
| O4B—C13A—C13B—O2 | 43 (3) | N4—Cr—N1—C1 | 138.9 (2) |
| O3—C13A—C13B—O4A | -20 (3) | Cl2—Cr—N1—C1 | 51.3 (2) |
| O2—C13A—C13B—O4A | -143 (3) | Cl1—Cr—N1—C1 | -129.2 (2) |
| O1—C13A—C13B—O4A | 103 (3) | N3—Cr—N2—C5 | -137.9 (2) |
| O4B—C13A—C13B—O4A | -100 (4) | N1—Cr—N2—C5 | 41.6 (2) |
| O3—C13B—O4B—C13A | 78 (3) | Cl2—Cr—N2—C5 | -48.8 (2) |
| O1—C13B—O4B—C13A | -155 (3) | Cl1—Cr—N2—C5 | 132.2 (2) |
| O2—C13B—O4B—C13A | -41 (2) | N2—Cr—N3—C6 | -141.5 (2) |
| O4A—C13B—O4B—C13A | 25.7 (16) | N4—Cr—N3—C6 | 38.6 (2) |
| O3—C13A—O4B—C13B | -96 (3) | Cl2—Cr—N3—C6 | 126.3 (2) |
| O2—C13A—O4B—C13B | 135 (3) | Cl1—Cr—N3—C6 | -53.2 (2) |
| O4A—C13A—O4B—C13B | -132 (3) | N3—Cr—N4—C9 | -39.7 (2) |
| O1—C13A—O4B—C13B | 22 (2) | N1—Cr—N4—C9 | 140.8 (2) |
| O3—C13A—O4A—C13B | 19 (3) | Cl2—Cr—N4—C9 | -128.8 (2) |
| O2—C13A—O4A—C13B | 142 (3) | Cl1—Cr—N4—C9 | 50.2 (2) |
| O1—C13A—O4A—C13B | -100 (3) | Cr—N1—C1—C2 | 59.1 (3) |
| O4B—C13A—O4A—C13B | 53 (3) | N1—C1—C2—C4 | 60.6 (4) |
| O3—C13B—O4A—C13A | -160 (3) | N1—C1—C2—C5 | -65.2 (4) |
| O1—C13B—O4A—C13A | 76 (3) | N1—C1—C2—C3 | 179.0 (3) |
| O4B—C13B—O4A—C13A | -105 (4) | Cr—N2—C5—C2 | -60.0 (4) |
| O2—C13B—O4A—C13A | -35 (3) | C4—C2—C5—N2 | -60.1 (4) |
| O3—C13B—O1—C13A | -75.2 (5) | C1—C2—C5—N2 | 65.2 (4) |
| O4B—C13B—O1—C13A | 173.4 (8) | C3—C2—C5—N2 | -178.8 (3) |
| O2—C13B—O1—C13A | 65.8 (4) | Cr—N3—C6—C7 | -56.9 (3) |
| O4A—C13B—O1—C13A | -6.8 (4) | N3—C6—C7—C10 | -60.2 (4) |
| O3—C13A—O1—C13B | 59.8 (5) | N3—C6—C7—C9 | 65.2 (4) |
| O2—C13A—O1—C13B | -76.8 (5) | N3—C6—C7—C8 | -178.8 (3) |
| O4A—C13A—O1—C13B | 169.6 (6) | Cr—N4—C9—C7 | 58.7 (3) |
| O4B—C13A—O1—C13B | -4.3 (5) | C10—C7—C9—N4 | 59.8 (4) |
| O3—C13A—O2—C13B | -65.8 (5) | C6—C7—C9—N4 | -65.7 (4) |
| O4A—C13A—O2—C13B | -173.9 (6) | C8—C7—C9—N4 | 178.0 (3) |

Hydrogen-bond geometry (Å, °)

| | | | | |
|---------------|-------|-------------|-------------|---------------|
| $D-H\cdots A$ | $D-H$ | $H\cdots A$ | $D\cdots A$ | $D-H\cdots A$ |
|---------------|-------|-------------|-------------|---------------|

supplementary materials

| | | | | |
|------------------------------|------|------|-----------|-----|
| N1—H1BN...O2 ⁱ | 0.90 | 2.29 | 3.030 (5) | 139 |
| N2—H2AN...O3 ⁱⁱ | 0.90 | 2.23 | 3.099 (6) | 162 |
| N2—H2AN...O4A ⁱⁱ | 0.90 | 2.42 | 3.183 (6) | 143 |
| N2—H2BN...O4B | 0.90 | 2.36 | 3.217 (9) | 159 |
| N3—H3AN...O4A ⁱⁱ | 0.90 | 2.60 | 3.482 (7) | 168 |
| N4—H4BN...O2 ⁱ | 0.90 | 2.14 | 3.030 (5) | 172 |
| N4—H4AN...O4A ⁱⁱⁱ | 0.90 | 2.54 | 3.403 (8) | 161 |
| N1—H1AN...Cl2 ^{iv} | 0.90 | 2.68 | 3.525 (3) | 156 |
| N3—H3BN...Cl1 ⁱⁱ | 0.90 | 2.69 | 3.533 (3) | 156 |

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

Fig. 1

