Supporting Information

Synthesis, Characterization and Crystal Structure of *trans*-Aquahydroxobis(2,2-dimethyl-1,3-propanediamine)chromium(III) Diperchlorate

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Abstract

Computing details

Data collection: PAL ADSC Quantum-210 ADX Program; cell refinement: HKL3000sm; data reduction: HKL3000sm; program(s) used to solve structure: SHELXL-2013-XS (Sheldrick, 2013); program(s) used to refine structure: SHELXL-2013-XL (Sheldrick, 2013); molecular graphics: Diamond 3; software used to prepare material for publication: WinGX,enCifer1.4.

References

NOT FOUND

(S57)

Crystal data

$C_{10}H_{32}CrN_4O_2{\cdot}2(ClO_4)$	$F(000) = 516$
$M_r = 491.29$	$D_x = 1.599$ Mg m^{-3}
Monoclinic, $P2_1/n$	Synchrotron radiation, $\lambda = 0.62998$ Å
$a = 5.9800$ (12) Å	Cell parameters from 32022 reflections
$b = 8.3120$ (17) Å	$\theta = 0.4$–$33.6°$
$c = 20.554$ (4) Å	$\mu = 0.63$ mm^{-1}
$\beta = 92.50$ (3)°	$T = 100$ K
$V = 1020.7$ (4) Å^3	Plate, pink
$Z = 2$	0.21 × 0.21 × 0.05 mm

Data collection

ADSC Q210 CCD area detector diffractometer	2590 reflections with $I > 2\sigma(I)$
Radiation source: PLSII 2D bending magnet	$R_{int} = 0.030$
ω scan	$\theta_{max} = 26.0°$, $\theta_{min} = 2.8°$
Absorption correction: empirical (using intensity measurements) HKL3000sm Scalepack	$h = -8 \rightarrow 8$
$T_{min} = 0.880$, $T_{max} = 0.969$	$k = -10 \rightarrow 10$
10076 measured reflections	$l = -28 \rightarrow 28$
2699 independent reflections	

Refinement

Refinement on F^2	2 restraints
Least-squares matrix: full	Hydrogen site location: mixed
$R[F^2 > 2\sigma(F^2)] = 0.032$	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2) = 0.084$	$w = 1/[\sigma^2(F_o^2) + (0.0451P)^2 + 0.7468P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.08$	$(\Delta/\sigma)_{max} < 0.001$
2699 reflections	$\Delta\rangle_{max} = 0.47$ e Å^{-3}
133 parameters	$\Delta\rangle_{min} = -0.90$ e Å^{-3}

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.

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

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
Cr1	0.0000	0.5000	0.0000	0.00553 (9)	
O1A	0.31838 (14)	0.43888 (12)	0.00828 (5)	0.01156 (18)	0.5
H1OA	0.346 (2)	0.3458 (13)	0.0158 (6)	0.017*	

O1W	0.31838 (14)	0.43888 (12)	0.00828 (5)	0.01156 (18)	0.5
H1OW	0.346 (2)	0.3458 (13)	0.0158 (6)	0.017*	0.5
H2OW	0.450 (7)	0.481 (6)	0.003 (2)	0.019 (11)*	0.5
N1	-0.06125 (17)	0.38506 (13)	0.08775 (5)	0.0098 (2)	
H1A	-0.1538	0.2889	0.0774	0.012*	
H1B	0.0842	0.3461	0.1065	0.012*	
N2	0.07680 (17)	0.71252 (13)	0.04989 (5)	0.0108 (2)	
H2A	0.2369	0.7077	0.0642	0.013*	
H2B	0.0604	0.8021	0.0183	0.013*	
C1	-0.1730 (2)	0.47657 (17)	0.13967 (6)	0.0120 (2)	
H1C	-0.1884	0.4055	0.1779	0.014*	
H1D	-0.3255	0.5069	0.1234	0.014*	
C2	-0.04742 (19)	0.62864 (17)	0.16150 (6)	0.0120 (2)	
C3	-0.0529 (2)	0.75578 (16)	0.10779 (6)	0.0130 (2)	
H3A	-0.2107	0.7749	0.0933	0.016*	
H3B	0.0070	0.8578	0.1263	0.016*	
C4	0.1925 (2)	0.58958 (19)	0.18554 (7)	0.0188 (3)	
H4A	0.1893	0.5129	0.2217	0.028*	
H4B	0.2677	0.6886	0.2004	0.028*	
H4C	0.2740	0.5422	0.1499	0.028*	
C5	-0.1737 (2)	0.7008 (2)	0.21806 (7)	0.0189 (3)	
H5A	-0.3258	0.7299	0.2027	0.028*	
H5B	-0.0950	0.7971	0.2344	0.028*	
H5C	-0.1808	0.6215	0.2532	0.028*	
Cl1P	0.39342 (5)	0.08511 (4)	0.09176 (2)	0.01381 (9)	
O1P	0.1962 (2)	0.07899 (14)	0.04848 (6)	0.0245 (2)	
O2P	0.3522 (2)	0.19316 (17)	0.14447 (6)	0.0291 (3)	
O3P	0.44502 (18)	-0.07253 (14)	0.11549 (7)	0.0248 (3)	
O4P	0.5746 (2)	0.14749 (17)	0.05515 (7)	0.0344 (3)	

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cr1	0.00268 (13)	0.00465 (15)	0.00940 (13)	-0.00100 (7)	0.00196 (8)	0.00061 (8)
O1A	0.0046 (4)	0.0094 (5)	0.0207 (4)	0.0004 (3)	0.0020 (3)	0.0039 (3)
O1W	0.0046 (4)	0.0094 (5)	0.0207 (4)	0.0004 (3)	0.0020 (3)	0.0039 (3)
N1	0.0100 (4)	0.0081 (5)	0.0118 (4)	-0.0005 (3)	0.0040 (3)	0.0022 (3)

N2	0.0109 (4)	0.0070 (5)	0.0149 (5)	-0.0027 (3)	0.0041 (3)	-0.0023 (4)
C1	0.0090 (5)	0.0148 (6)	0.0126 (5)	-0.0011 (4)	0.0049 (4)	0.0002 (4)
C2	0.0083 (5)	0.0154 (6)	0.0124 (5)	0.0004 (4)	0.0025 (4)	-0.0034 (4)
C3	0.0119 (5)	0.0108 (6)	0.0167 (5)	0.0000 (4)	0.0051 (4)	-0.0035 (4)
C4	0.0108 (5)	0.0249 (8)	0.0205 (6)	0.0017 (5)	-0.0016 (4)	-0.0035 (5)
C5	0.0159 (6)	0.0265 (8)	0.0146 (6)	0.0024 (5)	0.0045 (4)	-0.0066 (5)
Cl1P	0.01322 (15)	0.00886 (17)	0.01921 (16)	0.00014 (9)	-0.00086 (11)	0.00132 (10)
O1P	0.0274 (6)	0.0176 (6)	0.0273 (6)	0.0070 (4)	-0.0136 (4)	-0.0030 (4)
O2P	0.0359 (6)	0.0307 (7)	0.0202 (5)	0.0131 (5)	-0.0057 (4)	-0.0073 (4)
O3P	0.0169 (5)	0.0147 (6)	0.0420 (7)	0.0027 (4)	-0.0055 (4)	0.0119 (5)
O4P	0.0322 (6)	0.0259 (7)	0.0462 (8)	-0.0161 (5)	0.0168 (6)	-0.0035 (5)

Geometric parameters (Å, º)

Cr1—O1A	1.9707 (9)	C1—H1D	0.9900
Cr1—O1W^{i}	1.9707 (9)	C2—C3	1.5274 (19)
Cr1—O1A^{i}	1.9707 (9)	C2—C4	1.5318 (18)
Cr1—N2	2.0838 (11)	C2—C5	1.5354 (17)
Cr1—N2^{i}	2.0838 (11)	C3—H3A	0.9900
Cr1—N1	2.0875 (11)	C3—H3B	0.9900
Cr1—N1^{i}	2.0875 (11)	C4—H4A	0.9800
O1A—H1OA	0.805 (11)	C4—H4B	0.9800
N1—C1	1.4921 (16)	C4—H4C	0.9800
N1—H1A	0.9900	C5—H5A	0.9800
N1—H1B	0.9900	C5—H5B	0.9800
N2—C3	1.4926 (16)	C5—H5C	0.9800
N2—H2A	0.9900	Cl1P—O3P	1.4272 (11)
N2—H2B	0.9900	Cl1P—O2P	1.4369 (12)
C1—C2	1.5277 (19)	Cl1P—O4P	1.4422 (12)
C1—H1C	0.9900	Cl1P—O1P	1.4467 (12)
O1A—Cr1—O1A^{i}	180.0	C2—C1—H1C	108.8
O1A—Cr1—N2	89.12 (4)	N1—C1—H1D	108.8
O1W^{i}—Cr1—N2	90.88 (4)	C2—C1—H1D	108.8
O1A^{i}—Cr1—N2	90.88 (4)	H1C—C1—H1D	107.7
O1A—Cr1—N2^{i}	90.88 (4)	C3—C2—C1	111.43 (10)

O1W^{i}—Cr1—N2^{i}	89.12 (4)	C3—C2—C4	111.73 (11)
O1A^{i}—Cr1—N2^{i}	89.12 (4)	C1—C2—C4	111.16 (12)
N2—Cr1—N2^{i}	180.0	C3—C2—C5	106.32 (11)
O1A—Cr1—N1	90.73 (5)	C1—C2—C5	107.09 (10)
O1W^{i}—Cr1—N1	89.28 (5)	C4—C2—C5	108.85 (11)
O1A^{i}—Cr1—N1	89.28 (5)	N2—C3—C2	114.45 (10)
N2—Cr1—N1	90.34 (4)	N2—C3—H3A	108.6
N2^{i}—Cr1—N1	89.66 (4)	C2—C3—H3A	108.6
O1A—Cr1—N1^{i}	89.28 (5)	N2—C3—H3B	108.6
O1W^{i}—Cr1—N1^{i}	90.72 (5)	C2—C3—H3B	108.6
O1A^{i}—Cr1—N1^{i}	90.72 (5)	H3A—C3—H3B	107.6
N2—Cr1—N1^{i}	89.66 (4)	C2—C4—H4A	109.5
N2^{i}—Cr1—N1^{i}	90.34 (4)	C2—C4—H4B	109.5
N1—Cr1—N1^{i}	180.0	H4A—C4—H4B	109.5
Cr1—O1A—H1OA	117.1 (10)	C2—C4—H4C	109.5
C1—N1—Cr1	119.05 (8)	H4A—C4—H4C	109.5
C1—N1—H1A	107.6	H4B—C4—H4C	109.5
Cr1—N1—H1A	107.6	C2—C5—H5A	109.5
C1—N1—H1B	107.6	C2—C5—H5B	109.5
Cr1—N1—H1B	107.6	H5A—C5—H5B	109.5
H1A—N1—H1B	107.0	C2—C5—H5C	109.5
C3—N2—Cr1	119.05 (8)	H5A—C5—H5C	109.5
C3—N2—H2A	107.6	H5B—C5—H5C	109.5
Cr1—N2—H2A	107.6	O3P—Cl1P—O2P	110.96 (8)
C3—N2—H2B	107.6	O3P—Cl1P—O4P	110.63 (8)
Cr1—N2—H2B	107.6	O2P—Cl1P—O4P	109.04 (9)
H2A—N2—H2B	107.0	O3P—Cl1P—O1P	109.64 (7)
N1—C1—C2	113.70 (10)	O2P—Cl1P—O1P	108.88 (7)
N1—C1—H1C	108.8	O4P—Cl1P—O1P	107.61 (9)
Cr1—N1—C1—C2	-58.13 (13)	Cr1—N2—C3—C2	56.91 (13)
N1—C1—C2—C3	68.33 (13)	C1—C2—C3—N2	-67.91 (13)
N1—C1—C2—C4	-57.00 (14)	C4—C2—C3—N2	57.11 (14)
N1—C1—C2—C5	-175.78 (11)	C5—C2—C3—N2	175.74 (10)

Symmetry code: (i) -*x*, -*y*+1, -*z*.

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1*A*—H1*OA*···O1*P*	0.81 (1)	2.50 (1)	3.1965 (17)	146 (1)
O1*A*—H1*OA*···O4*P*	0.81 (1)	2.27 (1)	3.0022 (19)	152 (1)
O1*W*—H1*OW*···O1*P*	0.81 (1)	2.50 (1)	3.1965 (17)	146 (1)
O1*W*—H1*OW*···O4*P*	0.81 (1)	2.27 (1)	3.0022 (19)	152 (1)
N1—H1*A*···O4*P*^{ii}	0.99	2.04	2.9939 (17)	161
N1—H1*B*···O2*P*	0.99	2.16	3.1248 (17)	163
N2—H2*A*···O3*P*^{iii}	0.99	2.43	3.0983 (17)	125
N2—H2*B*···O1*P*^{iii}	0.99	2.51	3.1291 (17)	120
N2—H2*B*···O1*P*^{i}	0.99	2.24	3.0773 (17)	141

Symmetry codes: (i) -*x*, -*y*+1, -*z*; (ii) *x*-1, *y*, *z*; (iii) *x*, *y*+1, *z*.