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(Acetylacetonato)aqua(2,2′,2″-terpyridine)chromium(III) bis(perchlorate) dihydrate hexamethylphosphoramide solvate

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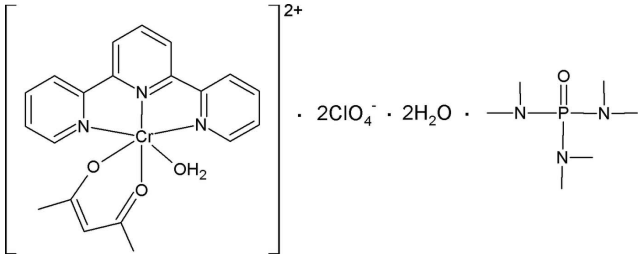
Key indicators: single-crystal X-ray study; T = 101 K; mean σ(C–C) = 0.002 Å; R factor = 0.030; wR factor = 0.080; data-to-parameter ratio = 18.3.

The title compound, $[Cr(C_5H_8O_2)(C_{15}H_{11}N_3)(H_2O)](ClO_4)_2\cdot 2H_2O\cdot C_6H_{18}N_3OP$, adopts a distorted octahedral configuration about the Cr^{III} atom, mainly due to the rigidity of the terpyridyl ligand. The N—Cr—N bite angles are significantly distorted from 90° at 78.3 (1) and 78.6 (1)°. The Cr—N bond distances vary between 1.998 (1) and 2.068 (1) Å. The crystal packing is controlled by hydrogen bonding and π-stacking, with an interplanar separation of 3.263 (2) Å.

Related literature

For similar complexes, see: Beran *et al.* (1970); Cloete *et al.* (2007); Einstein & Penfold (1966, 1968); Liu & Verkade (1998); Morosin (1965); Shiren & Tanaka (2002); Visser *et al.* (2005); Wickramasinghe *et al.* (1982). For the synthesis of the starting complex *mer*-[$CrCl_3$(terpy)], see: Cloete *et al.* (2007).



Experimental

Crystal data

$[Cr(C_5H_7O_2)(C_{15}H_{11}N_3)(H_2O)](ClO_4)_2\cdot 2(H_2O)\cdot C_6H_{18}N_3OP$
M_r = 816.53
Monoclinic, $P2_1/n$
a = 13.2524 (4) Å
b = 13.5607 (4) Å
c = 19.7271 (6) Å
β = 95.588 (1)°
V = 3528.35 (18) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.59 mm^{-1}
T = 101 (2) K
0.26 × 0.24 × 0.19 mm

Data collection

Bruker Kappa APEXII diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2004)
T_{min} = 0.850, T_{max} = 0.899
61605 measured reflections
8750 independent reflections
7534 reflections with $I > 2\sigma(I)$
R_{int} = 0.032

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.030
$wR(F^2)$ = 0.08
S = 1.02
8750 reflections
477 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.4 e Å^{-3}
$\Delta\rho_{min}$ = −0.42 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Cr1—O2	1.9268 (10)	Cr1—N2	1.9976 (12)
Cr1—O1	1.9356 (10)	Cr1—N1	2.0547 (12)
Cr1—O3	1.9684 (11)	Cr1—N3	2.0676 (12)
O2—Cr1—O1	91.05 (4)	N2—Cr1—N1	78.55 (5)
O2—Cr1—O3	87.51 (5)	N2—Cr1—N3	78.26 (5)

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

D—H···A	D—H	H···A	D···A	D—H···A
O14—H4A···O10	0.86 (3)	2.00 (3)	2.838 (2)	163 (3)
O14—H4B···O8^{i}	0.80 (3)	2.09 (3)	2.861 (2)	162 (2)
O3—H45···O13	0.72 (2)	1.93 (2)	2.6461 (17)	171 (2)
O13—H5A···O7^{ii}	0.75 (3)	2.14 (3)	2.8830 (18)	172 (3)
O13—H5B···O14^{iii}	0.89 (3)	1.84 (3)	2.7356 (19)	177 (2)
O3—H46···O12^{iv}	0.87 (3)	1.63 (3)	2.4989 (16)	175 (3)

Symmetry codes: (i) $-x+\frac{3}{2}, y-\frac{1}{2}, -z+\frac{1}{2}$; (ii) $-x+\frac{1}{2}, y+\frac{1}{2}, -z+\frac{1}{2}$; (iii) $-x+\frac{3}{2}, y+\frac{1}{2}, -z+\frac{1}{2}$; (iv) $x, y, z-1$.

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: *SIR97* (Altomare *et al.*, 1999); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *DIAMOND* (Brandenburg & Putz, 2005); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: PV2041).

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supplementary materials

Acta Cryst. (2007). E**63**, m3069-m3070 [**doi:10.1107/S1600536807056176**]

(Acetylacetonato)aqua(2,2',2''-terpyridine)chromium(III) bis(perchlorate) dihydrate hexamethylphosphoramide solvate

N. Cloete and H. G. Visser

Comment

The title compound, $[Cr(acac)(H_2O)(terpy)]\cdot 2(ClO_4)\cdot 2(H_2O)\cdot(PON_3C_6H_{18})$, (terpy = 2,2',2''-terpyridine and acac = acetyl acetonoate), (I), contains a chromium(III) atom which is octahedrally surrounded by three N atoms (N1, N2 and N3) of the terpy ligand, two O atoms from the acac ligand and another O atom from the coordinated H_2O molecule (Figure 1). The Cr-atom in (I) adopts a distorted octahedral geometry where the mean intraligand N—Cr—N bond angle is 78.4 (1)°, similar to bond angles in *mer*-$[CrCl_3(terpy)]$ (Cloete *et al.*, 2007). The rigidity of the terpyridyl group necessatates the above mentioned bite angles to be much smaller than 90°. The O1—Cr—O2 bond angle is 91.1 (1)° which is equal to the average intramolecular O—Cr—O bond angle reported in $[Cr(acac)_3$ (Morosin, 1965)].

The mean Cr—N(peripheral) distance of 2.062 (1) Å is slightly longer than the Cr—N(central) bond distance (1.998 (1) Å). This difference of approximately 0.08 Å has also been observed in *mer*-$[Cr(terpy)_2]^{3+}$ (Wickramasinghe *et al.*, 1982) and in the metallo-mono(terpy) complexes *mer*-$[CrCl_3(terpy)]$ (Cloete *et al.*, 2007), [*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 C—C bond distances for the acac ligand range between 1.384 (2) and 1.496 Å. These bond lengths are consistent with the partial double bond character of the acetylacetonate ligand in which the electrons are delocalized in the chelate ring.

The other bonds in complex (I) fall within the same range as the corresponding complexes (Beran *et al.*, 1970; Cloete *et al.*, 2007; Einstein *et al.*, 1966; Einstein *et al.*, 1968; Liu & Verkade, 1998; Shiren & Tanaka, 2002 and Visser *et al.*, 2005).

The $[Cr(acac)(H_2O)(terpy)]$ cations are packed diagonally across the *ac* plane with the perchlorate anions, hexamethylphosphoramide and water molecules filling the spaces in between (Fig. 2). The molecules within the structure are linked by an extensive network of intermolecular hydrogen bonds and non-conventinal H-bonds of the types C—H···O and C—H···N. The crystal packing also seems to be influenced by π-π stacking interactions between the terpy moieties (Fig. 2) with an interplanar separation of 3.263 (2) Å.

Experimental

$[Cr(terpy)Cl_3]$ (Cloete *et al.*, 2007) (1.5 g, 3.2 mmole) was dissolved in methanol (300 ml). $AgClO_4$ (0.662 g, 3.2 mmole) was added to the methanol solution and stirred at room temperature for 12 h. The solution was filtered. NaOH (0.13 g, 3.2 mmole) and acetylacetone (3.3 ml 3.2 mmole) was added to the filtrate. Reaction mixture was stirred for another 12 h after which the solution was acidified with $HClO_4$ (5 ml, 60% *v*/*v*) and stirred for two hours. H_2O (100 ml) was added and the solution was left to slowly evaporate at room temperature. Single red cystals were obtained after 1 day which were suitable for X-ray crystallography. Yield: 0.199 g (76.1%)

Refinement

The aromatic, methylene and methyl (hexamethylphosphoramide molecule) hydrogen atoms were placed in geometrically idealized positions and constrained to ride on their parent atoms, with U_{iso}(H) = 1.5U_{eq}(C) and 1.2U_{eq}(C), respectively. The hydrogen atoms on the methyl groups of the acac ligand were constrained to ride on their parent atoms and idealized as rotating CH_3 groups. Aqua H atoms were located in a difference Fourier map and then refined isotropically. The H atom on the C3 atom of the acac group was located in a difference Fourier map and then refined isotropically. The maximum and the minimum residual electron density are located 0.69 Å from N6 and 0.69 Å from Cr1, respectively, indicating no physical meaning.

Figures

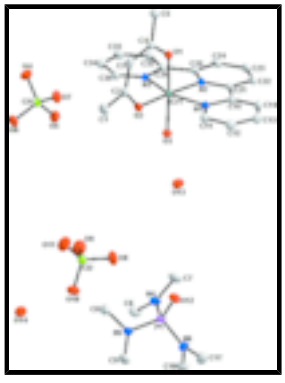


Fig. 1. : View of (I) with 50% probability displacement ellipsoids; H-atoms were omitted for clarity.

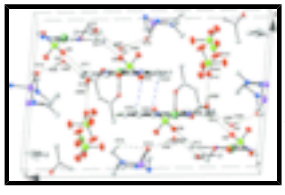


Fig. 2. : View of (I) along the *b* axis, illustrating selected H-bonds and π stacking. i) (−*x*, 2 − *y*, −*z*) ii) (*x*, *y*, −1 + *z*) iii) (3/2 − *x*, 1/2 + *y*, 1/2 − *z*)

(Acetylacetonato)aqua(2,2',2''-terpyridine)chromium(III) bis(perchlorate) dihydrate hexamethylphosphoramide solvate

Crystal data

$[Cr(C_5H_7O_2)(C_{15}H_{11}N_3)(H_2O_1)](ClO_4)_2·2(H_2O)·C_6H_{18}N_3OP$	F_{000} = 1700
	D_x = 1.537 Mg m^{-3}
M_r = 816.53	D_m = 1.54 Mg m^{-3}
	D_m measured by not measured
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -P 2yn	Cell parameters from 9416 reflections
a = 13.2524 (4) Å	θ = 2.5–28.3°
b = 13.5607 (4) Å	μ = 0.59 mm^{-1}
c = 19.7271 (6) Å	T = 101 (2) K
β = 95.588 (1)°	Cuboid, red

V = 3528.35 (18) Å^3
Z = 4

0.26 × 0.24 × 0.19 mm

Data collection

Bruker X8 APEXII 4K Kappa CCD diffractometer
T = 101(2) K
ω and φ scans
Absorption correction: multi-scan (SADABS; Bruker, 2004)
T_{min} = 0.850, T_{max} = 0.899
61605 measured reflections
8750 independent reflections

7534 reflections with $I > 2\sigma(I)$
R_{int} = 0.032
θ_{max} = 28.3º
θ_{min} = 1.8º
h = −17→17
k = −18→18
l = −24→26

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.030
$wR(F^2)$ = 0.08
S = 1.02
8750 reflections
477 parameters

H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0529P)^2 + 4.3676P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.4 e Å^{-3}
$\Delta\rho_{min}$ = −0.42 e Å^{-3}
Extinction correction: none

Special details

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.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
Cr1	0.126363 (16)	0.768594 (16)	0.120659 (11)	0.01100 (6)
Cl1	0.19255 (3)	0.33434 (3)	0.091107 (18)	0.01726 (8)
Cl2	0.69017 (3)	0.49729 (3)	0.254242 (19)	0.02045 (9)
O1	−0.02022 (8)	0.76518 (7)	0.11518 (5)	0.0145 (2)
O2	0.13586 (8)	0.67037 (8)	0.19157 (5)	0.0160 (2)
N2	0.12138 (9)	0.87546 (9)	0.05072 (6)	0.0127 (2)
N3	0.12156 (9)	0.68837 (9)	0.03127 (6)	0.0137 (2)
N1	0.12924 (9)	0.89261 (9)	0.18065 (6)	0.0141 (2)
O6	0.24232 (9)	0.25598 (9)	0.13100 (6)	0.0260 (3)
P1	0.43832 (3)	0.77800 (3)	0.995845 (19)	0.01538 (8)
O12	0.34383 (8)	0.82187 (9)	1.01873 (6)	0.0222 (2)

N4	0.42298 (10)	0.75554 (10)	0.91434 (7)	0.0199 (3)
N5	0.47021 (10)	0.66964 (10)	1.02906 (7)	0.0203 (3)
O13	0.39785 (10)	0.80887 (10)	0.23635 (7)	0.0252 (3)
O14	0.89534 (10)	0.29744 (12)	0.25721 (7)	0.0289 (3)
N6	0.53124 (10)	0.85556 (10)	1.01509 (7)	0.0206 (3)
C7	0.35895 (13)	0.81940 (15)	0.86919 (9)	0.0281 (4)
H7A	0.3191	0.8608	0.8957	0.042*
H7B	0.3148	0.7798	0.8388	0.042*
H7C	0.4007	0.8597	0.8433	0.042*
C8	0.39334 (13)	0.59136 (13)	1.02112 (10)	0.0288 (4)
H8A	0.3511	0.601	0.9793	0.043*
H8B	0.3524	0.5935	1.0587	0.043*
H8C	0.4262	0.5284	1.0202	0.043*
C9	0.53796 (14)	0.66036 (14)	1.09146 (9)	0.0299 (4)
H9A	0.5865	0.7131	1.0939	0.045*
H9B	0.5728	0.5983	1.0915	0.045*
H9C	0.4992	0.6635	1.1301	0.045*
C17	0.53491 (16)	0.91838 (14)	1.07527 (9)	0.0324 (4)
H17A	0.4708	0.9158	1.0941	0.049*
H17B	0.5489	0.9851	1.0628	0.049*
H17C	0.5874	0.8956	1.1086	0.049*
C6	0.48678 (14)	0.68963 (14)	0.87875 (9)	0.0298 (4)
H6A	0.5269	0.6497	0.9113	0.045*
H6B	0.5307	0.7278	0.853	0.045*
H6C	0.4448	0.6479	0.8485	0.045*
C10	0.62493 (13)	0.85386 (15)	0.98238 (10)	0.0298 (4)
H10A	0.6169	0.8114	0.9433	0.045*
H10B	0.679	0.8297	1.0139	0.045*
H10C	0.6407	0.9194	0.9683	0.045*
O3	0.27545 (8)	0.76660 (8)	0.12612 (6)	0.0162 (2)
H5A	0.3840 (19)	0.8279 (19)	0.2696 (14)	0.047 (8)*
H5B	0.465 (2)	0.8070 (18)	0.2371 (12)	0.045 (7)*
H4B	0.8704 (19)	0.244 (2)	0.2575 (13)	0.045 (7)*
H4A	0.852 (2)	0.342 (2)	0.2664 (16)	0.078 (10)*
H45	0.3038 (17)	0.7814 (16)	0.1576 (12)	0.030 (6)*
H46	0.3014 (19)	0.7886 (18)	0.0903 (14)	0.050 (7)*
C31	0.11945 (11)	0.74333 (11)	−0.02662 (7)	0.0144 (3)
O4	0.11986 (9)	0.29300 (9)	0.04007 (6)	0.0284 (3)
O5	0.26586 (9)	0.39128 (9)	0.05905 (6)	0.0263 (3)
C25	0.12033 (10)	0.85093 (11)	−0.01524 (7)	0.0145 (3)
C22	0.12551 (11)	1.04583 (11)	0.02463 (8)	0.0182 (3)
H22	0.128	1.1113	0.0387	0.022*
C34	0.11710 (12)	0.54218 (12)	−0.03595 (8)	0.0214 (3)
H34	0.1158	0.4737	−0.0382	0.026*
C13	0.12692 (13)	1.06842 (12)	0.25091 (9)	0.0252 (4)
H13	0.1256	1.1276	0.2747	0.03*
O7	0.14090 (9)	0.39758 (9)	0.13592 (6)	0.0250 (3)
C15	0.12453 (11)	0.97999 (11)	0.14630 (8)	0.0154 (3)
C24	0.12035 (11)	0.92365 (12)	−0.06455 (8)	0.0177 (3)

H24	0.1184	0.9073	−0.1104	0.021*
C14	0.12233 (12)	1.06858 (11)	0.18027 (9)	0.0212 (3)
H14	0.1178	1.1277	0.1563	0.025*
C12	0.13348 (13)	0.98032 (12)	0.28550 (9)	0.0233 (3)
H12	0.1375	0.9791	0.3328	0.028*
C2	0.06322 (11)	0.62566 (11)	0.21764 (7)	0.0153 (3)
C4	−0.07570 (11)	0.70747 (11)	0.14682 (7)	0.0148 (3)
C3	−0.03864 (11)	0.63886 (11)	0.19608 (8)	0.0175 (3)
H3	−0.0854	0.5995	0.2157	0.021*
C21	0.12382 (10)	0.96977 (11)	0.07163 (8)	0.0146 (3)
C5	−0.18745 (12)	0.71765 (12)	0.12873 (8)	0.0213 (3)
H5C	−0.2025	0.7109	0.0804	0.032*
H5D	−0.2093	0.7813	0.1428	0.032*
H5E	−0.2223	0.6672	0.1514	0.032*
C32	0.11698 (11)	0.69970 (12)	−0.08987 (8)	0.0193 (3)
H32	0.1161	0.7382	−0.1289	0.023*
C33	0.11581 (12)	0.59778 (13)	−0.09459 (8)	0.0228 (3)
H33	0.1142	0.5671	−0.1369	0.027*
O8	0.67088 (11)	0.59924 (9)	0.26969 (7)	0.0355 (3)
C35	0.12030 (11)	0.58980 (11)	0.02628 (8)	0.0173 (3)
H35	0.1216	0.5523	0.0658	0.021*
C11	0.13407 (11)	0.89343 (12)	0.24870 (8)	0.0183 (3)
H11	0.138	0.8337	0.272	0.022*
O9	0.69011 (12)	0.48494 (11)	0.18257 (7)	0.0400 (3)
O11	0.61525 (11)	0.43677 (10)	0.28030 (7)	0.0383 (3)
O10	0.78831 (11)	0.47091 (11)	0.28708 (9)	0.0482 (4)
C23	0.12333 (11)	1.02168 (12)	−0.04377 (8)	0.0196 (3)
H23	0.1239	1.0716	−0.0761	0.024*
C1	0.09592 (13)	0.55771 (12)	0.27532 (8)	0.0232 (3)
H1A	0.1417	0.5093	0.2604	0.035*
H1B	0.0376	0.5253	0.2903	0.035*
H1C	0.1294	0.5949	0.3123	0.035*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cr1	0.01239 (11)	0.00971 (11)	0.01106 (11)	0.00019 (8)	0.00199 (8)	0.00174 (8)
Cl1	0.01874 (17)	0.01400 (16)	0.01917 (18)	−0.00186 (13)	0.00244 (13)	0.00061 (13)
Cl2	0.02155 (18)	0.01963 (18)	0.01980 (18)	−0.00030 (14)	0.00005 (14)	0.00105 (14)
O1	0.0145 (5)	0.0144 (5)	0.0149 (5)	−0.0007 (4)	0.0027 (4)	0.0021 (4)
O2	0.0179 (5)	0.0144 (5)	0.0159 (5)	0.0002 (4)	0.0023 (4)	0.0037 (4)
N2	0.0106 (5)	0.0127 (6)	0.0150 (6)	0.0003 (4)	0.0024 (4)	0.0027 (5)
N3	0.0126 (6)	0.0140 (6)	0.0147 (6)	0.0000 (4)	0.0017 (4)	0.0011 (5)
N1	0.0134 (6)	0.0129 (6)	0.0161 (6)	0.0004 (4)	0.0027 (4)	0.0001 (5)
O6	0.0289 (6)	0.0195 (6)	0.0300 (6)	0.0038 (5)	0.0047 (5)	0.0084 (5)
P1	0.01319 (17)	0.01865 (19)	0.01444 (18)	0.00062 (14)	0.00207 (13)	0.00170 (14)
O12	0.0190 (5)	0.0292 (6)	0.0191 (6)	0.0052 (5)	0.0065 (4)	0.0047 (5)
N4	0.0168 (6)	0.0264 (7)	0.0165 (6)	0.0026 (5)	0.0008 (5)	−0.0011 (5)

N5	0.0177 (6)	0.0196 (7)	0.0229 (7)	−0.0013 (5)	−0.0013 (5)	0.0035 (5)
O13	0.0238 (7)	0.0298 (7)	0.0217 (6)	−0.0018 (5)	0.0006 (5)	−0.0059 (5)
O14	0.0271 (7)	0.0302 (7)	0.0295 (7)	−0.0027 (6)	0.0024 (5)	−0.0020 (6)
N6	0.0203 (6)	0.0215 (7)	0.0199 (6)	−0.0036 (5)	0.0015 (5)	−0.0006 (5)
C7	0.0242 (8)	0.0402 (10)	0.0193 (8)	0.0053 (7)	−0.0006 (6)	0.0048 (7)
C8	0.0249 (9)	0.0204 (8)	0.0404 (10)	−0.0033 (7)	0.0002 (7)	0.0049 (7)
C9	0.0309 (9)	0.0274 (9)	0.0294 (9)	0.0016 (7)	−0.0074 (7)	0.0079 (7)
C17	0.0436 (11)	0.0278 (9)	0.0245 (9)	−0.0035 (8)	−0.0041 (8)	−0.0047 (7)
C6	0.0297 (9)	0.0349 (10)	0.0249 (9)	0.0043 (8)	0.0036 (7)	−0.0109 (8)
C10	0.0177 (8)	0.0378 (10)	0.0339 (10)	−0.0062 (7)	0.0020 (7)	0.0026 (8)
O3	0.0148 (5)	0.0207 (6)	0.0132 (5)	0.0002 (4)	0.0017 (4)	0.0014 (4)
C31	0.0117 (6)	0.0169 (7)	0.0148 (7)	0.0009 (5)	0.0020 (5)	0.0021 (6)
O4	0.0258 (6)	0.0299 (7)	0.0285 (6)	−0.0049 (5)	−0.0022 (5)	−0.0083 (5)
O5	0.0258 (6)	0.0238 (6)	0.0302 (6)	−0.0049 (5)	0.0081 (5)	0.0067 (5)
C25	0.0098 (6)	0.0187 (7)	0.0153 (7)	0.0010 (5)	0.0023 (5)	0.0034 (6)
C22	0.0134 (7)	0.0134 (7)	0.0280 (8)	0.0000 (5)	0.0027 (6)	0.0052 (6)
C34	0.0225 (8)	0.0169 (7)	0.0246 (8)	0.0004 (6)	0.0015 (6)	−0.0055 (6)
C13	0.0270 (8)	0.0188 (8)	0.0299 (9)	−0.0021 (6)	0.0033 (7)	−0.0099 (7)
O7	0.0334 (7)	0.0190 (6)	0.0234 (6)	0.0038 (5)	0.0071 (5)	−0.0017 (5)
C15	0.0121 (6)	0.0135 (7)	0.0205 (7)	−0.0010 (5)	0.0020 (5)	0.0002 (6)
C24	0.0125 (7)	0.0246 (8)	0.0162 (7)	0.0019 (6)	0.0026 (5)	0.0062 (6)
C14	0.0215 (8)	0.0130 (7)	0.0293 (9)	−0.0015 (6)	0.0041 (6)	−0.0013 (6)
C12	0.0251 (8)	0.0254 (8)	0.0193 (8)	−0.0003 (7)	0.0023 (6)	−0.0055 (6)
C2	0.0234 (7)	0.0106 (7)	0.0126 (7)	−0.0005 (5)	0.0043 (5)	−0.0008 (5)
C4	0.0174 (7)	0.0143 (7)	0.0132 (7)	−0.0018 (5)	0.0037 (5)	−0.0031 (5)
C3	0.0205 (7)	0.0155 (7)	0.0173 (7)	−0.0042 (6)	0.0062 (6)	0.0021 (6)
C21	0.0106 (6)	0.0127 (7)	0.0207 (7)	−0.0004 (5)	0.0029 (5)	0.0026 (6)
C5	0.0160 (7)	0.0246 (8)	0.0235 (8)	−0.0020 (6)	0.0037 (6)	0.0015 (6)
C32	0.0176 (7)	0.0258 (8)	0.0145 (7)	0.0000 (6)	0.0014 (5)	0.0002 (6)
C33	0.0219 (8)	0.0285 (9)	0.0179 (7)	0.0001 (7)	0.0014 (6)	−0.0076 (6)
O8	0.0512 (8)	0.0200 (6)	0.0347 (7)	0.0055 (6)	0.0011 (6)	0.0000 (5)
C35	0.0177 (7)	0.0152 (7)	0.0189 (7)	0.0008 (6)	0.0018 (6)	0.0010 (6)
C11	0.0192 (7)	0.0190 (7)	0.0168 (7)	0.0001 (6)	0.0022 (6)	−0.0007 (6)
O9	0.0579 (9)	0.0408 (8)	0.0224 (7)	−0.0050 (7)	0.0103 (6)	−0.0030 (6)
O11	0.0415 (8)	0.0369 (8)	0.0384 (8)	−0.0133 (6)	0.0126 (6)	0.0047 (6)
O10	0.0316 (7)	0.0369 (8)	0.0707 (11)	0.0079 (6)	−0.0222 (7)	−0.0106 (8)
C23	0.0146 (7)	0.0200 (8)	0.0245 (8)	0.0001 (6)	0.0034 (6)	0.0121 (6)
C1	0.0283 (8)	0.0210 (8)	0.0208 (8)	0.0025 (6)	0.0049 (6)	0.0086 (6)

Geometric parameters (Å, °)

Cr1—O2	1.9268 (10)	C17—H17B	0.96
Cr1—O1	1.9356 (10)	C17—H17C	0.96
Cr1—O3	1.9684 (11)	C6—H6A	0.96
Cr1—N2	1.9976 (12)	C6—H6B	0.96
Cr1—N1	2.0547 (12)	C6—H6C	0.96
Cr1—N3	2.0676 (12)	C10—H10A	0.96
Cl1—O5	1.4352 (12)	C10—H10B	0.96
Cl1—O4	1.4376 (12)	C10—H10C	0.96

Cl1—O6	1.4430 (12)	O3—H45	0.72 (2)
Cl1—O7	1.4503 (12)	O3—H46	0.87 (3)
Cl2—O11	1.4222 (13)	C31—C32	1.379 (2)
Cl2—O9	1.4236 (14)	C31—C25	1.476 (2)
Cl2—O10	1.4405 (14)	C25—C24	1.385 (2)
Cl2—O8	1.4439 (13)	C22—C23	1.386 (2)
O1—C4	1.2776 (18)	C22—C21	1.389 (2)
O2—C2	1.2864 (18)	C22—H22	0.93
N2—C25	1.3418 (19)	C34—C33	1.380 (2)
N2—C21	1.3431 (19)	C34—C35	1.384 (2)
N3—C35	1.3403 (19)	C34—H34	0.93
N3—C31	1.3617 (19)	C13—C12	1.374 (2)
N1—C11	1.3376 (19)	C13—C14	1.389 (2)
N1—C15	1.3635 (19)	C13—H13	0.93
P1—O12	1.4953 (11)	C15—C14	1.377 (2)
P1—N4	1.6297 (13)	C15—C21	1.479 (2)
P1—N6	1.6356 (14)	C24—C23	1.391 (2)
P1—N5	1.6473 (14)	C24—H24	0.93
N4—C7	1.455 (2)	C14—H14	0.93
N4—C6	1.457 (2)	C12—C11	1.384 (2)
N5—C9	1.457 (2)	C12—H12	0.93
N5—C8	1.469 (2)	C2—C3	1.387 (2)
O13—H5A	0.75 (3)	C2—C1	1.495 (2)
O13—H5B	0.89 (3)	C4—C3	1.399 (2)
O14—H4B	0.80 (3)	C4—C5	1.496 (2)
O14—H4A	0.86 (3)	C3—H3	0.93
N6—C10	1.454 (2)	C5—H5C	0.96
N6—C17	1.458 (2)	C5—H5D	0.96
C7—H7A	0.96	C5—H5E	0.96
C7—H7B	0.96	C32—C33	1.385 (2)
C7—H7C	0.96	C32—H32	0.93
C8—H8A	0.96	C33—H33	0.93
C8—H8B	0.96	C35—H35	0.93
C8—H8C	0.96	C11—H11	0.93
C9—H9A	0.96	C23—H23	0.93
C9—H9B	0.96	C1—H1A	0.96
C9—H9C	0.96	C1—H1B	0.96
C17—H17A	0.96	C1—H1C	0.96
O2—Cr1—O1	91.05 (4)	N4—C6—H6B	109.5
O2—Cr1—O3	87.51 (5)	H6A—C6—H6B	109.5
O1—Cr1—O3	177.84 (5)	N4—C6—H6C	109.5
O2—Cr1—N2	176.74 (5)	H6A—C6—H6C	109.5
O1—Cr1—N2	90.76 (4)	H6B—C6—H6C	109.5
O3—Cr1—N2	90.76 (5)	N6—C10—H10A	109.5
O2—Cr1—N1	98.71 (5)	N6—C10—H10B	109.5
O1—Cr1—N1	90.80 (5)	H10A—C10—H10B	109.5
O3—Cr1—N1	91.00 (5)	N6—C10—H10C	109.5
N2—Cr1—N1	78.55 (5)	H10A—C10—H10C	109.5
O2—Cr1—N3	104.47 (5)	H10B—C10—H10C	109.5

O1—Cr1—N3	89.56 (5)	Cr1—O3—H45	118.6 (17)
O3—Cr1—N3	89.24 (5)	Cr1—O3—H46	115.2 (16)
N2—Cr1—N3	78.26 (5)	H45—O3—H46	113 (2)
N1—Cr1—N3	156.81 (5)	N3—C31—C32	121.40 (14)
O5—Cl1—O4	109.71 (8)	N3—C31—C25	114.47 (13)
O5—Cl1—O6	110.08 (7)	C32—C31—C25	124.13 (14)
O4—Cl1—O6	109.53 (8)	N2—C25—C24	120.25 (14)
O5—Cl1—O7	109.29 (7)	N2—C25—C31	113.06 (12)
O4—Cl1—O7	109.49 (7)	C24—C25—C31	126.68 (14)
O6—Cl1—O7	108.72 (7)	C23—C22—C21	118.31 (14)
O11—Cl2—O9	110.99 (9)	C23—C22—H22	120.8
O11—Cl2—O10	109.01 (10)	C21—C22—H22	120.8
O9—Cl2—O10	109.30 (10)	C33—C34—C35	119.05 (15)
O11—Cl2—O8	109.39 (9)	C33—C34—H34	120.5
O9—Cl2—O8	109.83 (9)	C35—C34—H34	120.5
O10—Cl2—O8	108.27 (8)	C12—C13—C14	119.61 (15)
C4—O1—Cr1	127.56 (9)	C12—C13—H13	120.2
C2—O2—Cr1	128.13 (9)	C14—C13—H13	120.2
C25—N2—C21	122.11 (13)	N1—C15—C14	121.21 (14)
C25—N2—Cr1	119.10 (10)	N1—C15—C21	114.15 (13)
C21—N2—Cr1	118.71 (10)	C14—C15—C21	124.63 (14)
C35—N3—C31	119.02 (13)	C25—C24—C23	118.39 (14)
C35—N3—Cr1	125.91 (10)	C25—C24—H24	120.8
C31—N3—Cr1	115.07 (10)	C23—C24—H24	120.8
C11—N1—C15	119.14 (13)	C15—C14—C13	119.07 (15)
C11—N1—Cr1	125.54 (10)	C15—C14—H14	120.5
C15—N1—Cr1	115.32 (10)	C13—C14—H14	120.5
O12—P1—N4	110.15 (7)	C13—C12—C11	118.86 (15)
O12—P1—N6	107.93 (7)	C13—C12—H12	120.6
N4—P1—N6	111.59 (7)	C11—C12—H12	120.6
O12—P1—N5	115.13 (7)	O2—C2—C3	124.09 (13)
N4—P1—N5	103.19 (7)	O2—C2—C1	114.96 (13)
N6—P1—N5	108.85 (7)	C3—C2—C1	120.94 (14)
C7—N4—C6	113.68 (13)	O1—C4—C3	124.53 (13)
C7—N4—P1	120.14 (11)	O1—C4—C5	115.50 (13)
C6—N4—P1	124.59 (11)	C3—C4—C5	119.97 (13)
C9—N5—C8	113.07 (14)	C2—C3—C4	124.34 (14)
C9—N5—P1	121.76 (12)	C2—C3—H3	117.8
C8—N5—P1	116.98 (11)	C4—C3—H3	117.8
H5A—O13—H5B	109 (2)	N2—C21—C22	120.24 (14)
H4B—O14—H4A	110 (3)	N2—C21—C15	113.14 (12)
C10—N6—C17	114.10 (14)	C22—C21—C15	126.62 (14)
C10—N6—P1	122.88 (12)	C4—C5—H5C	109.5
C17—N6—P1	121.96 (12)	C4—C5—H5D	109.5
N4—C7—H7A	109.5	H5C—C5—H5D	109.5
N4—C7—H7B	109.5	C4—C5—H5E	109.5
H7A—C7—H7B	109.5	H5C—C5—H5E	109.5
N4—C7—H7C	109.5	H5D—C5—H5E	109.5
H7A—C7—H7C	109.5	C31—C32—C33	119.22 (15)

H7B—C7—H7C	109.5	C31—C32—H32	120.4
N5—C8—H8A	109.5	C33—C32—H32	120.4
N5—C8—H8B	109.5	C34—C33—C32	119.33 (15)
H8A—C8—H8B	109.5	C34—C33—H33	120.3
N5—C8—H8C	109.5	C32—C33—H33	120.3
H8A—C8—H8C	109.5	N3—C35—C34	121.97 (14)
H8B—C8—H8C	109.5	N3—C35—H35	119
N5—C9—H9A	109.5	C34—C35—H35	119
N5—C9—H9B	109.5	N1—C11—C12	122.09 (15)
H9A—C9—H9B	109.5	N1—C11—H11	119
N5—C9—H9C	109.5	C12—C11—H11	119
H9A—C9—H9C	109.5	C22—C23—C24	120.68 (14)
H9B—C9—H9C	109.5	C22—C23—H23	119.7
N6—C17—H17A	109.5	C24—C23—H23	119.7
N6—C17—H17B	109.5	C2—C1—H1A	109.5
H17A—C17—H17B	109.5	C2—C1—H1B	109.5
N6—C17—H17C	109.5	H1A—C1—H1B	109.5
H17A—C17—H17C	109.5	C2—C1—H1C	109.5
H17B—C17—H17C	109.5	H1A—C1—H1C	109.5
N4—C6—H6A	109.5	H1B—C1—H1C	109.5

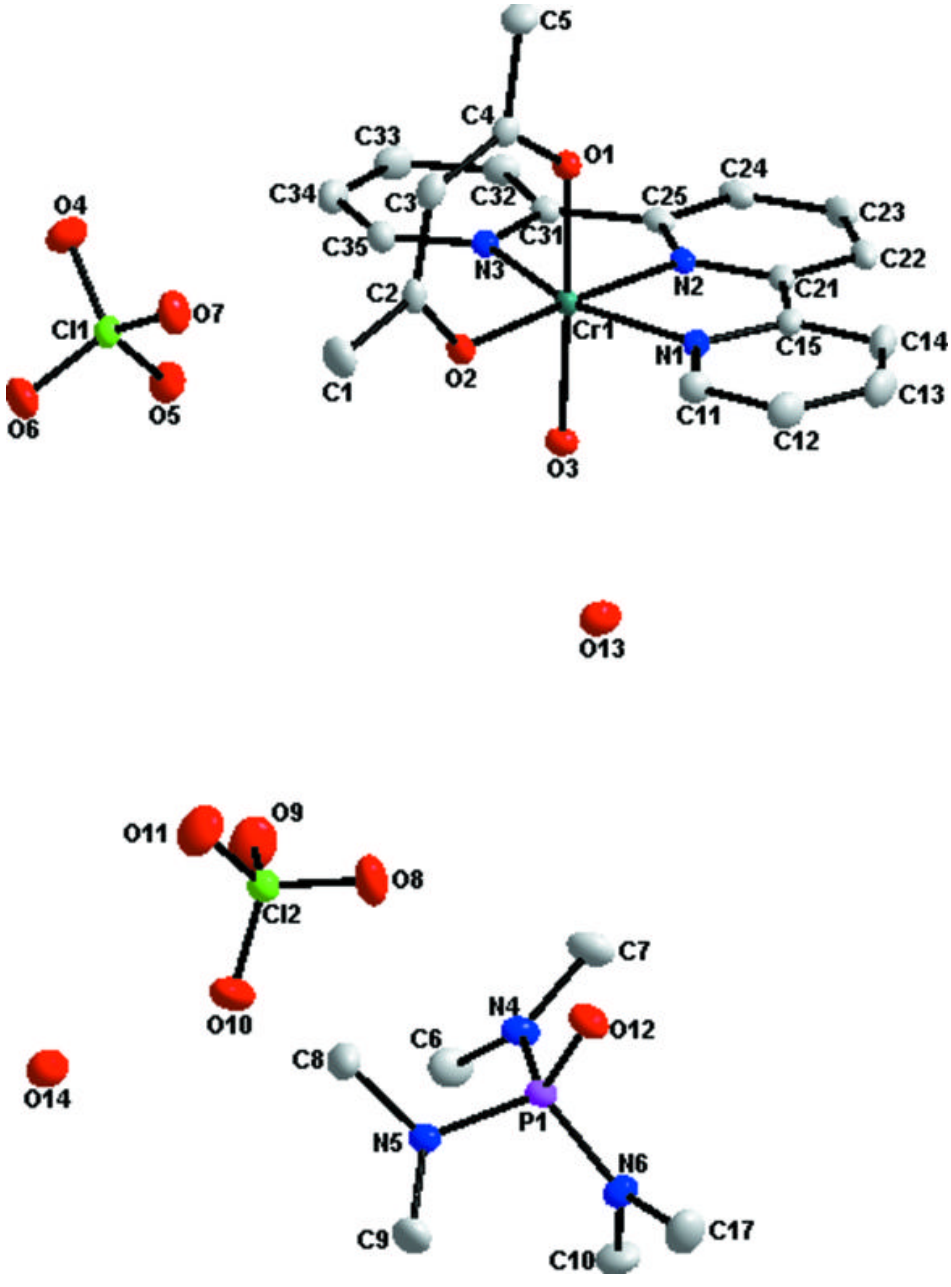
Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O14—H4A···O10	0.86 (3)	2.00 (3)	2.838 (2)	163 (3)
O14—H4B···O8^{i}	0.80 (3)	2.09 (3)	2.861 (2)	162 (2)
O3—H45···O13	0.72 (2)	1.93 (2)	2.6461 (17)	171 (2)
C1—H1A···O13^{ii}	0.96	2.77	3.384 (2)	123
C6—H6A···N5	0.96	2.52	3.007 (2)	111
C7—H7A···O12	0.96	2.47	2.976 (2)	113
C9—H9A···N6	0.96	2.54	3.043 (2)	113
C13—H13···O13^{iii}	0.93	2.48	3.289 (2)	145
O14—H4A···O10	0.86 (3)	2.00 (3)	2.838 (2)	163 (3)
O14—H4B···O8^{i}	0.80 (3)	2.09 (3)	2.861 (2)	162 (2)
O3—H45···O13	0.72 (2)	1.93 (2)	2.6461 (17)	171 (2)
C1—H1A···O13^{ii}	0.96	2.77	3.384 (2)	123
C6—H6A···N5	0.96	2.52	3.007 (2)	111
C7—H7A···O12	0.96	2.47	2.976 (2)	113
C9—H9A···N6	0.96	2.54	3.043 (2)	113
C13—H13···O13^{iii}	0.93	2.48	3.289 (2)	145
C17—H17A···O12	0.96	2.49	2.970 (2)	111
O13—H5A···O7^{iii}	0.75 (3)	2.14 (3)	2.8830 (18)	172 (3)
O13—H5B···O14^{iv}	0.89 (3)	1.84 (3)	2.7356 (19)	177 (2)
O3—H46···O12^{v}	0.87 (3)	1.63 (3)	2.4989 (16)	175 (3)
C6—H6C···O9^{vi}	0.96	2.57	3.466 (2)	155
C11—H11···O6^{iii}	0.93	2.59	3.322 (2)	136
C14—H14···O6^{vii}	0.93	2.48	3.199 (2)	134

C22—H22···O4^{vii}	0.93	2.47	3.367 (2)	163
C24—H24···O8^{viii}	0.93	2.53	3.415 (2)	159
C32—H32···O14^{ix}	0.93	2.57	3.289 (2)	135
C35—H35···O7	0.93	2.51	3.3811 (19)	156
C17—H17A···O12	0.96	2.49	2.970 (2)	111

Symmetry codes: (i) −*x*+3/2, *y*−1/2, −*z*+1/2; (ii) −*x*+1/2, *y*−1/2, −*z*+1/2; (iii) −*x*+1/2, *y*+1/2, −*z*+1/2; (iv) −*x*+3/2, *y*+1/2, −*z*+1/2; (v) *x*, *y*, *z*−1; (vi) −*x*+1, −*y*+1, −*z*+1; (vii) *x*, *y*+1, *z*; (viii) *x*−1/2, −*y*+3/2, *z*−1/2; (ix) −*x*+1, −*y*+1, −*z*.

Fig. 1


C5
C4
C33
O1
C24
C34
C3
C32
C25
C23
O4
C35
C31
C22
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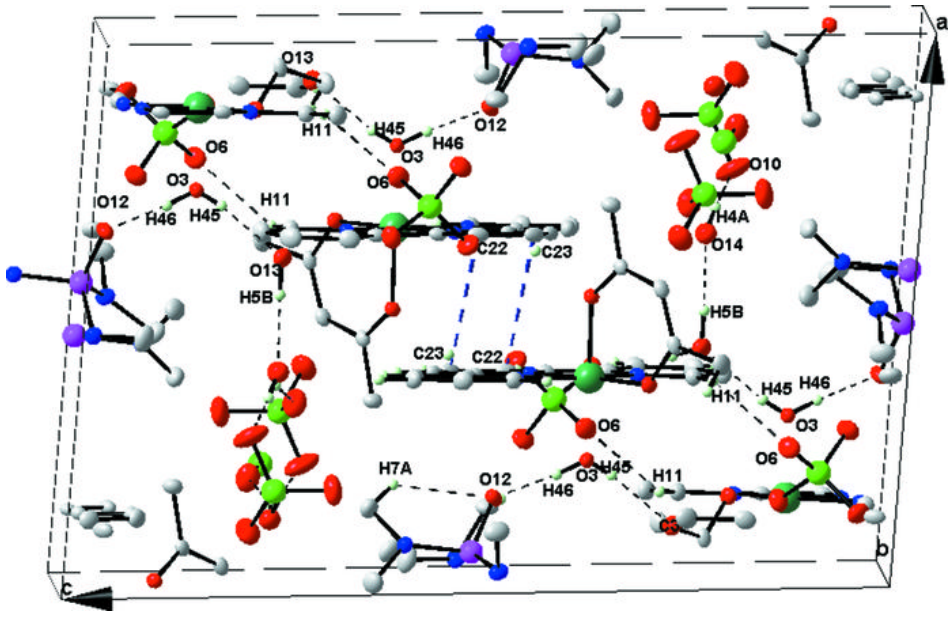
N2
C2
C21
Cl1
O7
Cr1
N1
C15
C14
C1
O2
C13
O6
O5
C11
C12
O3
O13
O11
O9
O8
Cl2
C7
N4
O10
C6
O12
C8
O14
P1
N5
N6
C9
C17
C10

Fig. 2


a
b
c
O13
H11
H45
O12
H46
O3
O6
O3
O12
H46
H45
H11
O6
O10
H4A
O14
C22
C23
O13
H5B
H5B
C23
C22
H11
H45
H46
O3
O6
O6
H7A
O12
O3
H45
H46
H11
C5