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Crystal structures of two cross-bridged chromium(III) tetraazamacrocycles

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Abstract

The crystal structure of dichlorido(4,10-dimethyl-1,4,7,10-tetraazabicyclo[5.5.2]tetradecane)chromium(III) hexafluoridophosphate, $[\text{CrCl}_2(\text{C}_{12}\text{H}_{26}\text{N}_4)]\text{PF}_6$, (I), has monoclinic symmetry

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(space group $P2_1/n$) at 150 K. The structure of the related dichlorido(4,11-dimethyl-1,4,8,11-tetraazabicyclo[6.6.2]hexadecane)chromium(III) hexafluoridophosphate, $[\text{CrCl}_2(\text{C}_{14}\text{H}_{30}\text{N}_4)]\text{PF}_6$, (II), also displays monoclinic symmetry (space group $P2_1/c$) at 150 K. In each case, the Cr^{III} ion is hexacoordinate with two *cis* chloride ions and two non-adjacent N atoms bound *cis* equatorially and the other two non-adjacent N atoms bound *trans* axially in a... [Read More](#)

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Crystal structures of two cross-bridged chromium(III) tetraazamacrocycles

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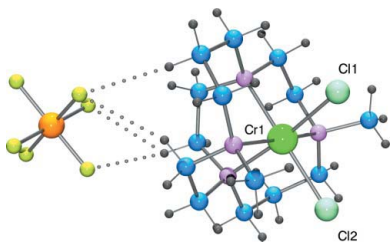
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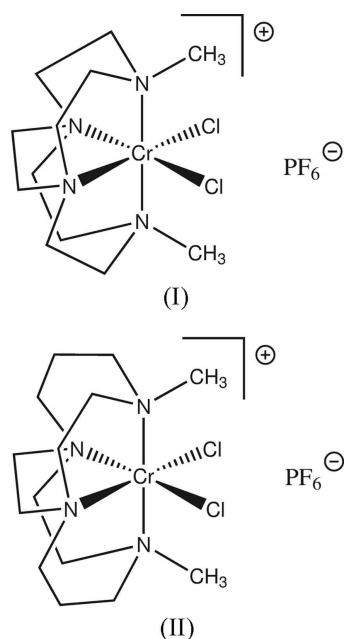
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The crystal structure of dichlorido(4,10-dimethyl-1,4,7,10-tetraazabicyclo[5.5.2]-tetradecane)chromium(III) hexafluoridophosphate, $[\text{CrCl}_2(\text{C}_{12}\text{H}_{26}\text{N}_4)]\text{PF}_6$, (I), has monoclinic symmetry (space group $P2_1/n$) at 150 K. The structure of the related dichlorido(4,11-dimethyl-1,4,8,11-tetraazabicyclo[6.6.2]hexadecane)chromium(III) hexafluoridophosphate, $[\text{CrCl}_2(\text{C}_{14}\text{H}_{30}\text{N}_4)]\text{PF}_6$, (II), also displays monoclinic symmetry (space group $P2_1/c$) at 150 K. In each case, the Cr^{III} ion is hexacoordinate with two *cis* chloride ions and two non-adjacent N atoms bound *cis* equatorially and the other two non-adjacent N atoms bound *trans* axially in a *cis*-V conformation of the macrocycle. The extent of the distortion from the preferred octahedral coordination geometry of the Cr^{III} ion is determined by the parent macrocycle ring size, with the larger cross-bridged cyclam ring in (II) better able to accommodate this preference and the smaller cross-bridged cyclen ring in (I) requiring more distortion away from octahedral geometry.

1. Chemical context

Ethylene cross-bridged tetraazamacrocycles were introduced to coordination chemists in 1990 by Weisman and Wong (Weisman *et al.*, 1990). Since then, their transition metal complexes have become important to the fields of oxidation catalysis (Yin *et al.*, 2007; Dong *et al.*, 2013), medical/biological imaging (Boswell *et al.*, 2004; Sprague *et al.*, 2007; Silversides *et al.*, 2011) and chemokine receptor antagonism (Lewis *et al.*, 2005; Valks *et al.*, 2006; Smith *et al.*, 2012) due to the combination of restricted macrocycle configuration and kinetic inertness inherent to these ligands. Chromium(III) complexes have played an important role in characterizing new ligands due to their relative kinetic inertness (Cotton & Wilkinson, 1988). Yet, to date, only one report of the chromium coordination chemistry of these macrobicyclic ligands has appeared in the literature (Maples *et al.*, 2009). In order to expand the range of metal ions that can be coordinated by these remarkable ligands (Hubin, 2003), we are exploring further the structural chemistry of chromium cross-bridged tetraazamacrocyclic complexes and report synthesis and crystal structures of dichlorido(4,10-dimethyl-1,4,7,10-tetraazabicyclo[5.5.2]tetradecane)chromium(III) hexafluoridophosphate, (I), and dichlorido(4,11-dimethyl-1,4,8,11-tetraazabicyclo[6.6.2]hexadecane)chromium(III)hexafluoridophosphate, (II).





2. Structural commentary

Each of the title compounds crystallizes with a single positively-charged metal complex and one PF_6^- anion in the asymmetric unit. The metal ion in each complex adopts a distorted octahedral geometry. The N atoms of each macrocycle occupy four coordination sites, while two chloride ions in a *cis* arrangement complete the coordination of Cr^{III} . This so-called *cis-V* conformation, expected to be dictated by the ligand cross-bridge, is apparent for both of the complexes structurally characterized here. Figs. 1 and 2 illustrate the local

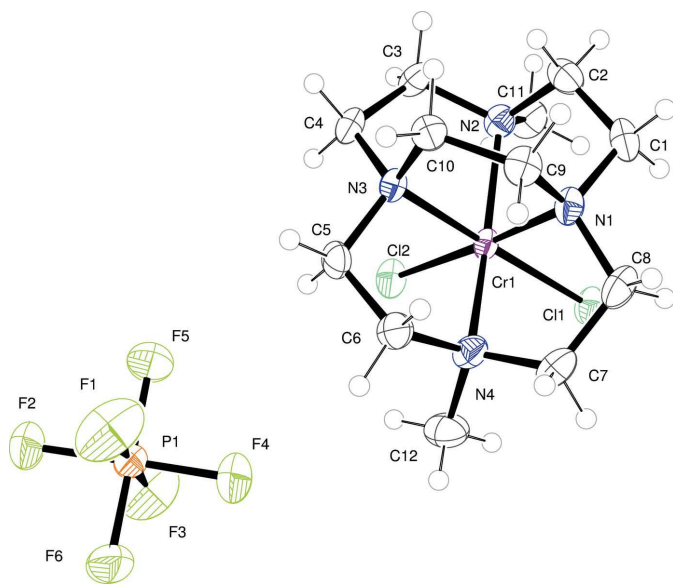


Figure 1
The molecular entities of (I), with atoms shown as displacement ellipsoids at the 50% probability level.

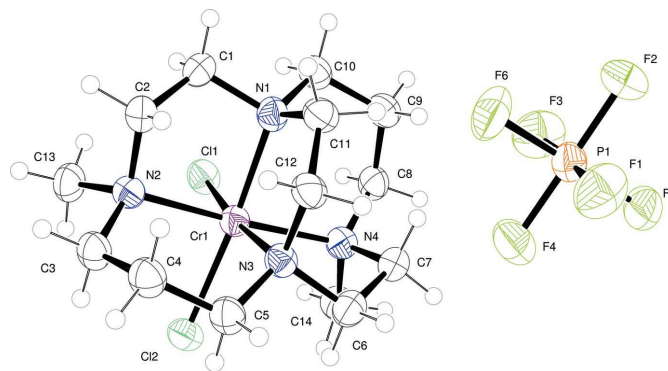


Figure 2
The molecular entities of (II), with atoms shown as displacement ellipsoids at the 50% probability level.

Table 1
Hydrogen-bond geometry (\AA , $^\circ$) for (I).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$C1-H1A\cdots F6^i$	0.99	2.53	3.347 (4)	140
$C1-H1B\cdots F3^{ii}$	0.99	2.46	3.410 (5)	162
$C2-H2A\cdots F4^i$	0.99	2.48	3.407 (4)	155
$C3-H3A\cdots F1^{iii}$	0.99	2.51	3.500 (4)	177
$C4-H4A\cdots F6^{iv}$	0.99	2.36	3.175 (4)	139
$C6-H6B\cdots F4$	0.99	2.30	3.238 (4)	158
$C1-H1A\cdots Cl1$	0.99	2.82	3.393 (4)	118
$C4-H4B\cdots Cl2$	0.99	2.63	3.137 (3)	112
$C5-H5A\cdots Cl2$	0.99	2.74	3.324 (3)	118
$C6-H6A\cdots Cl2^v$	0.99	2.77	3.447 (3)	126
$C8-H8B\cdots Cl1$	0.99	2.73	3.203 (4)	110
$C12-H12B\cdots Cl2$	0.98	2.78	3.329 (4)	116

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

geometry about Cr^{III} in (I) (dimethyl bridged-cyclen complex) and (II) (dimethyl bridged-cyclam complex), respectively. Apparently, neither the identity of the metal ion, nor that of the alkyl substituents affects this conformation. This same conformation has been seen in all known metal complexes with ethylene cross-bridged cyclam and cyclen ligands.

The ring size of the parent macrocycle alters the degree to which the metal ion is engulfed by the bridged macrocycle. This is most clearly evident in the $N2-Cr1-N4$ bond angle between two axially bound nitrogen atoms. This bond angle is $161.62(11)^\circ$ in the case of the smaller macrocycle, cyclen, while it is $171.44(14)^\circ$ for the cyclam complex. A larger bond angle, closer to linearity, indicates a better fit, or complementarity, between the ligand and the preferred octahedral geometry of the Cr^{III} ion. A more subtle difference in the $N1-Cr1-N3$ bond angles, *viz.* the equatorially bound N atoms, shows the same trend: this angle is $83.23(10)^\circ$ for the cyclen complex and $84.18(13)^\circ$ for the cyclam complex. Finally, the $Cr-N$ bond lengths are somewhat affected by the ligand size as well. The mean of the four $Cr-N$ bond lengths is 2.08 \AA in (I), while this average is 2.12 \AA in (II). The mean value for a number of $Cr-NR_3$ bonds in the literature is 2.093 \AA ($\sigma = 0.044 \text{ \AA}$) (Orpen *et al.*, 1989).

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

<i>D</i> –H··· <i>A</i>	<i>D</i> –H	H··· <i>A</i>	<i>D</i> ··· <i>A</i>	<i>D</i> –H··· <i>A</i>
C1–H1 <i>B</i> ···F5 ⁱ	0.99	2.41	3.318 (5)	153
C2–H2 <i>A</i> ···F1 ⁱⁱ	0.99	2.50	3.094 (5)	119
C2–H2 <i>A</i> ···F6 ⁱⁱ	0.99	2.53	3.223 (6)	127
C7–H7 <i>A</i> ···F4	0.99	2.55	3.098 (6)	115
C12–H12 <i>B</i> ···F2 ⁱⁱ	0.99	2.51	3.393 (6)	149
C3–H3 <i>A</i> ···Cl2	0.99	2.73	3.280 (5)	115
C5–H5 <i>B</i> ···Cl2	0.99	2.70	3.287 (5)	119
C8–H8 <i>A</i> ···Cl1	0.99	2.70	3.258 (5)	116
C8–H8 <i>B</i> ···Cl1 ⁱⁱⁱ	0.99	2.82	3.778 (4)	162
C10–H10 <i>B</i> ···Cl1	0.99	2.70	3.270 (5)	117
C13–H13 <i>A</i> ···Cl1	0.98	2.68	3.157 (5)	111
C13–H13 <i>A</i> ···Cl2 ^{iv}	0.98	2.73	3.572 (4)	144
C14–H14 <i>B</i> ···Cl2	0.98	2.69	3.141 (5)	108

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

3. Supramolecular features

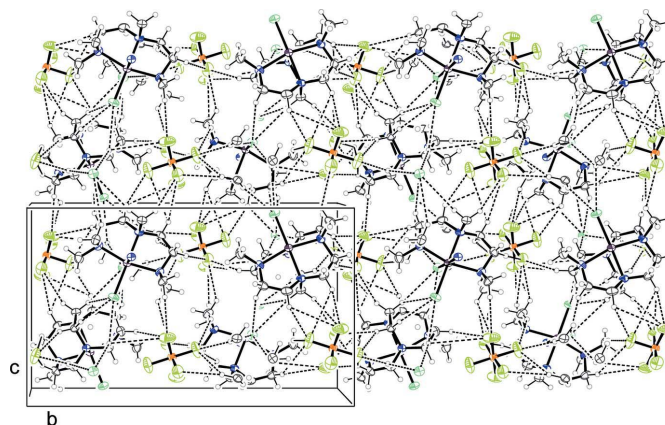
There are no classical hydrogen bonds present in either (I) and (II) but each structure contains a great many C–H···F and C–H···Cl interactions which generate three-dimensional arrays. These interactions were identified from the standard criterion that the distance from the hydrogen atom to the hydrogen-bond acceptor should not exceed the sum of the radius of the acceptor plus 2 Å. Tables 1 and 2 contain full details of these interactions for (I) and (II), respectively.

For (I), each PF₆[−] anion resides in a pocket between six metal complexes and there are C–H···F interactions to each of them. The mean C···F distance of those in Table 1 is 3.35 Å. Supplementary C–H···Cl intramolecular contacts are present and intermolecular interactions between neighbouring metal complexes are also observed. The overall effect of these intermolecular interactions is to generate an extended network. One way to describe this is in terms of puckered sheets of the cationic complex and PF₆[−] anions that extend in the *bc* plane. Between these sheets further C–H···F and C–H···Cl interactions assemble these layers in an *ABAB* fashion along *a* to generate a densely packed three-dimensional array as shown in Fig. 3.

For (II), the arrangement is rather similar and again a three-dimensional array is constructed from nonclassical hydrogen bonds between the cations and anions. The PF₆[−] anion is located in a pocket formed from four metal complexes in a distorted tetrahedral arrangement and forms C–H···F interactions to each of them, with a mean C···F distance of 3.23 Å. Further C–H···Cl interactions are also present. In a similar fashion to (I), these nonclassical interactions assemble the cations and anions into puckered sheets that extend in the *bc* plane. The sheets are then *ABAB* stacked along *a* as shown in Fig. 4.

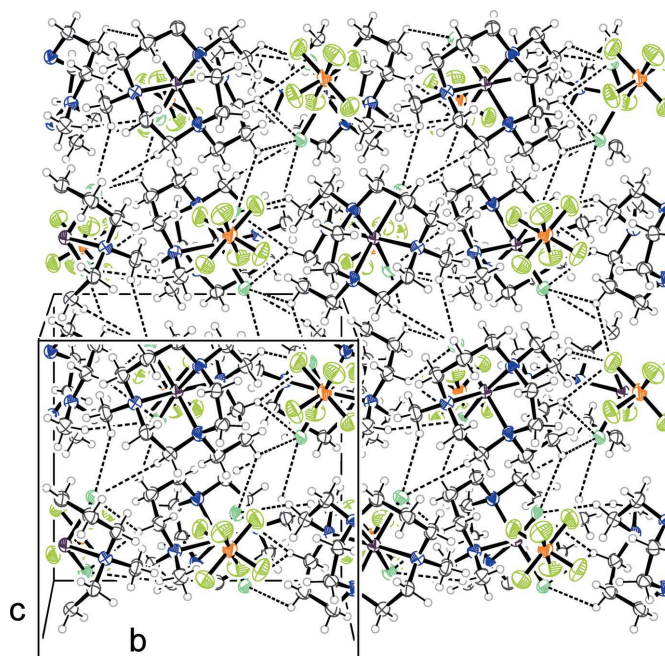
4. Database survey

The structures of three complexes that are directly analogous to (I) have been reported. These are the manganese (Hubin *et al.*, 2001), iron (McClain *et al.*, 2006) and cobalt (Hubin *et al.*,


Figure 3

Crystal packing of (I), viewed perpendicular to the *bc* plane. Dashed lines represent halide···H–C interactions.

2002) analogues. Each of these contains the metal in the trivalent state. For the Mn and Fe examples, the geometry about the metal is similar to that for Cr, but the bond angles formed by the two axially bound N atoms are smaller [155.01 (11) and 153.20 (9) °, respectively]. Similarly, the bite angles of the two equatorially bound N atoms are also noticeably smaller; these are 81.29 (11) and 78.62 (8)° for Mn and Fe analogues, respectively. However, the mean *M*–N bond length is longer for Mn and Fe examples: 2.153 and 2.167 Å, respectively. These differences in geometry reflect the smaller size of the Mn^{III} and Fe^{III} ions, and the possibility of a Jahn–Teller distortion for Mn^{III}, but the greater ligand field stabilization energy (LFSE) for Cr^{III} yields shorter bond


Figure 4

Crystal packing of (II), viewed perpendicular to the *bc* plane. Dashed lines represent halide···H–C interactions.

lengths. The Co^{III} analogue is rather different because it is in a low spin state. The axial N–Co–N bond angle is 168.8 (4)° and the equatorial bond angle is 87.2 (4)°. As expected, the mean bond length is shorter for the Co case at 1.978 Å. The smaller, low-spin Co^{III} ion fits into the pocket of the macrocycle better than Cr^{III}.

Chromium(III) complexes similar to (I) and (II) but crystallized with different anions have been reported before (Maples *et al.*, 2009). The chloride analogue of (I) has bond angles of 160.83 (19) and 83.50 (18)° about the chromium ion and a mean Cr–N bond length of 2.08 Å, which are in good agreement with (I), demonstrating the counter-anion has very little effect on the coordination about the metal. A cyclen-based macrocycle with benzyl groups replacing the methyl groups in (I), has key bond angles 160.35 (19) and 83.6 (2)° and a mean Cr–N bond length of 2.09 Å (Maples *et al.*, 2009). The pocket in the macrocycle is of a similar shape in this example but slightly enlarged because of the pendant benzyl groups.

The chloride analogue of (II) (Maples *et al.*, 2009) displays a similarly sized pocket; the N–Cr–N axial bond angle is 172.46 (11)° and the equatorial angle is 84.63 (11)°, while mean Cr–N bond length is 2.12 Å. In line with the observation in (I) and (II), the pocket of the cyclam-derived ligand is better able to accommodate the octahedrally surrounded Cr^{III} ion and displays larger bond lengths than the cyclen equivalent.

5. Synthesis and crystallization

The cross-bridged ligands were prepared according to literature procedures (Weisman *et al.*, 1990; Wong *et al.*, 2000). The title complexes were prepared by a procedure slightly modified from those found in Hubin *et al.* (2001) for other trivalent metal ions. In an inert atmosphere glove-box, 1 mmol of the respective ligand was dissolved in 20 ml of anhydrous dimethylformamide in a 50 ml Erlenmeyer flask. 1 mmol of anhydrous chromium(II) chloride was added to the stirring ligand solution. The reaction was allowed to stir at room temperature overnight. The solution was then filtered through filter paper and the solvent was removed under vacuum to give blue–violet solids. In the glove-box, this divalent complex was dissolved in 20 ml of methanol in a round-bottomed flask. Five equivalents of NH₄PF₆ (5 mmol, 0.815 g) were dissolved in the solution. The flask was removed from the glove-box with a stopper to protect it from air. In a fume hood, a stream of nitrogen gas was directed over the surface of the solution. Four to six drops of Br₂ were added and the reaction was stirred for 15 min. Bright purple precipitates formed immediately. The nitrogen gas was then allowed to bubble through the solution for 15 min to remove excess Br₂. The flask was then stoppered and placed in a freezer for 30 min to complete the precipitation. The purple solid product was collected by vacuum filtration on a glass frit and washed with methanol and then ether. Crystals suitable for X-ray diffraction (purple blocks) were grown from the slow evaporation of aqueous solutions of the product.

Table 3
Experimental details.

	(I)	(II)
Crystal data		
Chemical formula	[CrCl ₂ (C ₁₂ H ₂₆ N ₄)]PF ₆	[CrCl ₂ (C ₁₄ H ₃₀ N ₄)]PF ₆
<i>M_r</i>	494.24	522.29
Crystal system, space group	Monoclinic, <i>P</i> ₂ ₁ / <i>n</i>	Monoclinic, <i>P</i> ₂ ₁ / <i>c</i>
Temperature (K)	150	150
<i>a</i> , <i>b</i> , <i>c</i> (Å)	8.2271 (10), 19.957 (2), 12.0474 (17)	13.6801 (19), 12.437 (2), 12.3864 (17)
β (°)	96.374 (11)	102.028 (11)
<i>V</i> (Å ³)	1965.8 (4)	2061.1 (5)
<i>Z</i>	4	4
Radiation type	Mo <i>K</i> α	Mo <i>K</i> α
μ (mm ⁻¹)	1.00	0.95
Crystal size (mm)	0.10 × 0.10 × 0.08	0.15 × 0.15 × 0.06
Data collection		
Diffractometer	Stoe IPDS2	Stoe IPDS2
Absorption correction	Analytical [a face-indexed absorption correction was applied; <i>X</i> -AREA (Stoe & Cie, 2002)]	Analytical [a face-indexed absorption correction was applied; <i>X</i> -AREA (Stoe & Cie, 2002)]
<i>T</i> _{min} , <i>T</i> _{max}	0.827, 0.915	0.778, 0.901
No. of measured, independent and observed [<i>I</i> > 2 σ (<i>I</i>)] reflections	22999, 4501, 2798	13450, 4158, 2073
<i>R</i> _{int}	0.091	0.091
(sin θ / λ) _{max} (Å ⁻¹)	0.650	0.622
Refinement		
<i>R</i> [<i>F</i> ² > 2 σ (<i>F</i> ²)], <i>wR</i> (<i>F</i> ²), <i>S</i>	0.041, 0.096, 0.88	0.045, 0.109, 0.78
No. of reflections	4501	4158
No. of parameters	235	255
H-atom treatment	H-atom parameters constrained	H-atom parameters constrained
$\Delta\rho_{\max}$, $\Delta\rho_{\min}$ (e Å ⁻³)	0.77, -0.49	0.33, -0.79

Computer programs: *X*-AREA (Stoe & Cie, 2002), *SHELXS86*, *SHELXL2013* and *SHELXTL* (Sheldrick, 2008).

6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 3. H atoms were placed in idealised positions and refined using a riding model, with C–H = 0.98 and 0.99 Å for –CH₃ and –CH₂– groups, respectively, and with $U_{\text{iso}}(\text{H})$ values of, respectively, 1.5 and 1.2 times U_{eq} of the carrier atom.

In (I), there is evidence for a very small degree of disorder (10%) in the position of the PF₆[–] anions. Refinement with a second orientation for this anion did not lead to a substantial improve in the fit. A model with a single orientation was therefore retained.

The structure of (II) is presented in $P2_1/n$, consistent with manganese and cobalt analogues (Hubin *et al.*, 2001, 2002), rather than the $P2_1/c$ setting of the iron analogue (McClain *et al.*, 2006) which has $\beta \simeq 120^\circ$.

Acknowledgements

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Computing details

For both compounds, data collection: *X-AREA* (Stoe & Cie, 2002); cell refinement: *X-AREA* (Stoe & Cie, 2002); data reduction: *X-AREA* (Stoe & Cie, 2002); program(s) used to solve structure: *SHELXS86* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL2013* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL* (Sheldrick, 2008).

(I) Dichlorido(4,10-dimethyl-1,4,7,10-tetraazabicyclo[5.5.2]tetradecane)chromium(III) hexafluoridophosphate

Crystal data

[CrCl₂(C₁₂H₂₆N₄)]PF₆

M_r = 494.24

Monoclinic, *P*2₁/*n*

a = 8.2271 (10) Å

b = 19.957 (2) Å

c = 12.0474 (17) Å

β = 96.374 (11)°

V = 1965.8 (4) Å³

Z = 4

F(000) = 1012

D_x = 1.670 Mg m⁻³

Mo *K*α radiation, λ = 0.71073 Å

Cell parameters from 12040 reflections

θ = 2.7–31.2°

μ = 1.00 mm⁻¹

T = 150 K

Block, purple

0.10 × 0.10 × 0.08 mm

Data collection

Stoe IPDS2

diffractometer

Radiation source: fine-focus sealed tube

Detector resolution: 6.67 pixels mm⁻¹

ω-scans

Absorption correction: analytical

[a face-indexed absorption correction was applied; *X-AREA* (Stoe & Cie, 2002)]

T_{min} = 0.827, *T_{max}* = 0.915

22999 measured reflections

4501 independent reflections

2798 reflections with *I* > 2σ(*I*)

R_{int} = 0.091

θ_{max} = 27.5°, θ_{min} = 2.7°

h = -10→10

k = -25→25

l = -14→15

Refinement

Refinement on *F*²

Least-squares matrix: full

R[*F*² > 2σ(*F*²)] = 0.041

wR(*F*²) = 0.096

S = 0.88

4501 reflections

235 parameters

0 restraints

Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained

w = 1/[σ²(*F_o*²) + (0.0474*P*)²]

where *P* = (*F_o*² + 2*F_c*²)/3

(Δ/σ)_{max} < 0.001

Δρ_{max} = 0.77 e Å⁻³

Δρ_{min} = -0.49 e Å⁻³

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 (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
Cr1	0.21553 (5)	0.19169 (2)	0.20979 (4)	0.02173 (12)
Cl1	-0.06600 (7)	0.20275 (4)	0.17145 (7)	0.03159 (19)
Cl2	0.25268 (8)	0.23403 (4)	0.03552 (7)	0.03325 (19)
N1	0.2035 (3)	0.15406 (14)	0.3675 (2)	0.0274 (6)
N2	0.2399 (3)	0.09097 (13)	0.1641 (2)	0.0276 (6)
N3	0.4628 (3)	0.18082 (13)	0.2517 (2)	0.0234 (5)
N4	0.2408 (3)	0.28159 (14)	0.2997 (2)	0.0296 (6)
C1	0.1252 (4)	0.08715 (17)	0.3466 (3)	0.0348 (8)
H1A	0.0099	0.0927	0.3144	0.042*
H1B	0.1267	0.0621	0.4177	0.042*
C2	0.2193 (4)	0.04929 (17)	0.2664 (3)	0.0340 (7)
H2A	0.1606	0.0074	0.2435	0.041*
H2B	0.3283	0.0371	0.3042	0.041*
C3	0.4087 (3)	0.08276 (17)	0.1267 (3)	0.0328 (8)
H3A	0.3976	0.0776	0.0445	0.039*
H3B	0.4586	0.0412	0.1599	0.039*
C4	0.5235 (3)	0.14187 (17)	0.1595 (3)	0.0300 (7)
H4A	0.6348	0.1249	0.1837	0.036*
H4B	0.5296	0.1714	0.0940	0.036*
C5	0.5233 (3)	0.25153 (16)	0.2599 (3)	0.0298 (7)
H5A	0.5118	0.2726	0.1850	0.036*
H5B	0.6402	0.2524	0.2899	0.036*
C6	0.4221 (3)	0.28971 (17)	0.3373 (3)	0.0325 (8)
H6A	0.4467	0.2726	0.4145	0.039*
H6B	0.4515	0.3378	0.3372	0.039*
C7	0.1464 (4)	0.27476 (19)	0.3992 (3)	0.0370 (8)
H7A	0.0446	0.3014	0.3858	0.044*
H7B	0.2124	0.2937	0.4655	0.044*
C8	0.1016 (4)	0.20179 (19)	0.4244 (3)	0.0379 (8)
H8A	0.1186	0.1940	0.5061	0.045*
H8B	-0.0154	0.1939	0.3988	0.045*
C9	0.3681 (3)	0.14731 (18)	0.4344 (3)	0.0321 (7)
H9A	0.3697	0.1062	0.4805	0.038*
H9B	0.3867	0.1861	0.4854	0.038*
C10	0.5051 (3)	0.14384 (16)	0.3594 (3)	0.0275 (7)
H10A	0.6059	0.1633	0.3993	0.033*
H10B	0.5276	0.0963	0.3429	0.033*
C11	0.1158 (4)	0.06839 (19)	0.0721 (3)	0.0377 (8)
H11A	0.0059	0.0742	0.0947	0.057*

H11B	0.1338	0.0210	0.0559	0.057*
H11C	0.1262	0.0951	0.0050	0.057*
C12	0.1830 (4)	0.34219 (19)	0.2353 (4)	0.0428 (9)
H12A	0.0660	0.3378	0.2103	0.064*
H12B	0.2437	0.3469	0.1701	0.064*
H12C	0.2012	0.3819	0.2829	0.064*
P1	0.74031 (9)	0.44740 (4)	0.22662 (7)	0.0291 (2)
F1	0.8570 (3)	0.43093 (16)	0.3353 (2)	0.0756 (8)
F2	0.8987 (2)	0.46269 (12)	0.1662 (2)	0.0570 (6)
F3	0.6248 (3)	0.46395 (16)	0.1167 (2)	0.0744 (8)
F4	0.5828 (3)	0.43324 (12)	0.2878 (2)	0.0604 (7)
F5	0.7476 (3)	0.37224 (11)	0.1869 (3)	0.0675 (8)
F6	0.7324 (3)	0.52314 (12)	0.2644 (3)	0.0639 (7)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cr1	0.01590 (19)	0.0277 (3)	0.0219 (3)	−0.00047 (18)	0.00316 (17)	0.0011 (2)
Cl1	0.0156 (3)	0.0430 (5)	0.0361 (5)	0.0006 (3)	0.0023 (3)	0.0020 (4)
Cl2	0.0254 (3)	0.0468 (5)	0.0273 (4)	−0.0002 (3)	0.0018 (3)	0.0092 (4)
N1	0.0232 (11)	0.0368 (16)	0.0228 (14)	−0.0009 (10)	0.0049 (10)	0.0025 (12)
N2	0.0241 (11)	0.0302 (15)	0.0279 (15)	−0.0014 (10)	0.0011 (10)	−0.0035 (12)
N3	0.0173 (10)	0.0293 (14)	0.0237 (13)	−0.0008 (9)	0.0030 (9)	−0.0003 (11)
N4	0.0233 (11)	0.0322 (15)	0.0331 (16)	0.0037 (10)	0.0024 (11)	−0.0033 (12)
C1	0.0297 (15)	0.042 (2)	0.0334 (19)	−0.0101 (14)	0.0055 (14)	0.0085 (16)
C2	0.0342 (15)	0.0284 (18)	0.038 (2)	−0.0054 (13)	−0.0005 (14)	0.0049 (16)
C3	0.0265 (14)	0.0368 (19)	0.036 (2)	0.0060 (13)	0.0067 (13)	−0.0101 (16)
C4	0.0200 (13)	0.0403 (19)	0.0306 (18)	0.0043 (12)	0.0073 (12)	0.0006 (15)
C5	0.0197 (12)	0.0329 (18)	0.0364 (19)	−0.0056 (12)	0.0014 (12)	0.0017 (15)
C6	0.0244 (14)	0.0320 (19)	0.040 (2)	−0.0022 (12)	−0.0011 (13)	−0.0051 (15)
C7	0.0319 (15)	0.047 (2)	0.0324 (19)	0.0083 (15)	0.0065 (14)	−0.0095 (17)
C8	0.0336 (15)	0.052 (2)	0.0302 (18)	0.0077 (15)	0.0137 (14)	−0.0011 (17)
C9	0.0321 (15)	0.0389 (19)	0.0248 (17)	0.0005 (14)	0.0011 (13)	0.0016 (15)
C10	0.0249 (13)	0.0317 (17)	0.0244 (17)	0.0008 (12)	−0.0035 (12)	0.0032 (14)
C11	0.0313 (15)	0.045 (2)	0.035 (2)	−0.0027 (14)	−0.0049 (14)	−0.0101 (17)
C12	0.0393 (17)	0.034 (2)	0.053 (3)	0.0085 (15)	−0.0046 (16)	0.0024 (18)
P1	0.0267 (4)	0.0287 (4)	0.0326 (5)	0.0003 (3)	0.0059 (3)	−0.0026 (4)
F1	0.0721 (16)	0.109 (2)	0.0429 (15)	0.0339 (15)	−0.0032 (13)	0.0063 (16)
F2	0.0442 (11)	0.0542 (14)	0.0786 (18)	−0.0083 (10)	0.0329 (11)	−0.0070 (13)
F3	0.0674 (15)	0.107 (2)	0.0452 (15)	0.0192 (15)	−0.0114 (12)	0.0022 (15)
F4	0.0486 (12)	0.0574 (15)	0.0815 (19)	−0.0082 (10)	0.0357 (12)	−0.0007 (13)
F5	0.0616 (14)	0.0366 (13)	0.109 (2)	−0.0068 (10)	0.0292 (14)	−0.0234 (14)
F6	0.0456 (12)	0.0412 (14)	0.107 (2)	−0.0014 (10)	0.0159 (12)	−0.0269 (14)

Geometric parameters (Å, °)

Cr1—N3	2.053 (2)	C5—C6	1.522 (4)
Cr1—N1	2.056 (3)	C5—H5A	0.9900

Cr1—N4	2.094 (3)	C5—H5B	0.9900
Cr1—N2	2.100 (3)	C6—H6A	0.9900
Cr1—C12	2.3147 (9)	C6—H6B	0.9900
Cr1—C11	2.3215 (8)	C7—C8	1.541 (5)
N1—C8	1.486 (4)	C7—H7A	0.9900
N1—C1	1.492 (4)	C7—H7B	0.9900
N1—C9	1.503 (4)	C8—H8A	0.9900
N2—C11	1.491 (4)	C8—H8B	0.9900
N2—C2	1.512 (4)	C9—C10	1.523 (4)
N2—C3	1.516 (3)	C9—H9A	0.9900
N3—C4	1.486 (4)	C9—H9B	0.9900
N3—C5	1.496 (4)	C10—H10A	0.9900
N3—C10	1.499 (4)	C10—H10B	0.9900
N4—C12	1.486 (5)	C11—H11A	0.9800
N4—C7	1.505 (4)	C11—H11B	0.9800
N4—C6	1.519 (4)	C11—H11C	0.9800
C1—C2	1.507 (5)	C12—H12A	0.9800
C1—H1A	0.9900	C12—H12B	0.9800
C1—H1B	0.9900	C12—H12C	0.9800
C2—H2A	0.9900	P1—F1	1.570 (3)
C2—H2B	0.9900	P1—F3	1.577 (3)
C3—C4	1.535 (5)	P1—F5	1.578 (2)
C3—H3A	0.9900	P1—F6	1.582 (2)
C3—H3B	0.9900	P1—F4	1.585 (2)
C4—H4A	0.9900	P1—F2	1.591 (2)
C4—H4B	0.9900		
N3—Cr1—N1	83.23 (10)	N3—C5—C6	108.2 (2)
N3—Cr1—N4	85.68 (10)	N3—C5—H5A	110.1
N1—Cr1—N4	81.21 (11)	C6—C5—H5A	110.1
N3—Cr1—N2	80.93 (9)	N3—C5—H5B	110.1
N1—Cr1—N2	84.74 (11)	C6—C5—H5B	110.1
N4—Cr1—N2	161.62 (11)	H5A—C5—H5B	108.4
N3—Cr1—C12	91.97 (7)	N4—C6—C5	110.5 (3)
N1—Cr1—C12	175.19 (7)	N4—C6—H6A	109.6
N4—Cr1—C12	98.09 (8)	C5—C6—H6A	109.6
N2—Cr1—C12	94.91 (8)	N4—C6—H6B	109.6
N3—Cr1—C11	177.18 (8)	C5—C6—H6B	109.6
N1—Cr1—C11	93.99 (7)	H6A—C6—H6B	108.1
N4—Cr1—C11	93.50 (7)	N4—C7—C8	113.5 (3)
N2—Cr1—C11	99.27 (7)	N4—C7—H7A	108.9
C12—Cr1—C11	90.81 (3)	C8—C7—H7A	108.9
C8—N1—C1	113.3 (2)	N4—C7—H7B	108.9
C8—N1—C9	109.3 (3)	C8—C7—H7B	108.9
C1—N1—C9	110.9 (3)	H7A—C7—H7B	107.7
C8—N1—Cr1	106.2 (2)	N1—C8—C7	110.8 (2)
C1—N1—Cr1	103.6 (2)	N1—C8—H8A	109.5
C9—N1—Cr1	113.31 (17)	C7—C8—H8A	109.5

C11—N2—C2	108.1 (2)	N1—C8—H8B	109.5
C11—N2—C3	108.7 (2)	C7—C8—H8B	109.5
C2—N2—C3	111.8 (2)	H8A—C8—H8B	108.1
C11—N2—Cr1	114.0 (2)	N1—C9—C10	111.6 (3)
C2—N2—Cr1	106.88 (19)	N1—C9—H9A	109.3
C3—N2—Cr1	107.38 (18)	C10—C9—H9A	109.3
C4—N3—C5	114.0 (2)	N1—C9—H9B	109.3
C4—N3—C10	108.9 (2)	C10—C9—H9B	109.3
C5—N3—C10	111.3 (2)	H9A—C9—H9B	108.0
C4—N3—Cr1	106.11 (18)	N3—C10—C9	112.0 (2)
C5—N3—Cr1	103.27 (17)	N3—C10—H10A	109.2
C10—N3—Cr1	113.16 (16)	C9—C10—H10A	109.2
C12—N4—C7	109.0 (3)	N3—C10—H10B	109.2
C12—N4—C6	108.4 (2)	C9—C10—H10B	109.2
C7—N4—C6	110.4 (3)	H10A—C10—H10B	107.9
C12—N4—Cr1	114.8 (2)	N2—C11—H11A	109.5
C7—N4—Cr1	107.7 (2)	N2—C11—H11B	109.5
C6—N4—Cr1	106.47 (18)	H11A—C11—H11B	109.5
N1—C1—C2	108.3 (2)	N2—C11—H11C	109.5
N1—C1—H1A	110.0	H11A—C11—H11C	109.5
C2—C1—H1A	110.0	H11B—C11—H11C	109.5
N1—C1—H1B	110.0	N4—C12—H12A	109.5
C2—C1—H1B	110.0	N4—C12—H12B	109.5
H1A—C1—H1B	108.4	H12A—C12—H12B	109.5
C1—C2—N2	111.0 (3)	N4—C12—H12C	109.5
C1—C2—H2A	109.4	H12A—C12—H12C	109.5
N2—C2—H2A	109.4	H12B—C12—H12C	109.5
C1—C2—H2B	109.4	F1—P1—F3	179.36 (15)
N2—C2—H2B	109.4	F1—P1—F5	90.78 (16)
H2A—C2—H2B	108.0	F3—P1—F5	89.08 (17)
N2—C3—C4	113.5 (2)	F1—P1—F6	90.07 (16)
N2—C3—H3A	108.9	F3—P1—F6	90.06 (16)
C4—C3—H3A	108.9	F5—P1—F6	179.05 (17)
N2—C3—H3B	108.9	F1—P1—F4	91.76 (15)
C4—C3—H3B	108.9	F3—P1—F4	88.88 (15)
H3A—C3—H3B	107.7	F5—P1—F4	91.85 (13)
N3—C4—C3	110.3 (2)	F6—P1—F4	88.55 (13)
N3—C4—H4A	109.6	F1—P1—F2	88.05 (14)
C3—C4—H4A	109.6	F3—P1—F2	91.32 (15)
N3—C4—H4B	109.6	F5—P1—F2	89.00 (12)
C3—C4—H4B	109.6	F6—P1—F2	90.60 (13)
H4A—C4—H4B	108.1	F4—P1—F2	179.13 (14)
C8—N1—C1—C2	167.0 (3)	C7—N4—C6—C5	141.4 (3)
C9—N1—C1—C2	-69.6 (3)	Cr1—N4—C6—C5	24.8 (3)
Cr1—N1—C1—C2	52.3 (3)	N3—C5—C6—N4	-52.8 (3)
N1—C1—C2—N2	-51.4 (3)	C12—N4—C7—C8	140.9 (3)
C11—N2—C2—C1	-100.3 (3)	C6—N4—C7—C8	-100.1 (3)

C3—N2—C2—C1	140.1 (3)	Cr1—N4—C7—C8	15.7 (3)
Cr1—N2—C2—C1	22.8 (3)	C1—N1—C8—C7	-155.4 (3)
C11—N2—C3—C4	138.1 (3)	C9—N1—C8—C7	80.4 (3)
C2—N2—C3—C4	-102.6 (3)	Cr1—N1—C8—C7	-42.3 (3)
Cr1—N2—C3—C4	14.3 (3)	N4—C7—C8—N1	17.3 (4)
C5—N3—C4—C3	-157.4 (3)	C8—N1—C9—C10	-140.2 (3)
C10—N3—C4—C3	77.6 (3)	C1—N1—C9—C10	94.1 (3)
Cr1—N3—C4—C3	-44.5 (3)	Cr1—N1—C9—C10	-21.9 (3)
N2—C3—C4—N3	19.5 (4)	C4—N3—C10—C9	-140.6 (3)
C4—N3—C5—C6	166.7 (3)	C5—N3—C10—C9	92.9 (3)
C10—N3—C5—C6	-69.7 (3)	Cr1—N3—C10—C9	-22.9 (3)
Cr1—N3—C5—C6	52.0 (3)	N1—C9—C10—N3	28.9 (4)
C12—N4—C6—C5	-99.2 (3)		

Hydrogen-bond geometry (Å, °)

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
C1—H1 <i>A</i> ...F6 ⁱ	0.99	2.53	3.347 (4)	140
C1—H1 <i>B</i> ...F3 ⁱⁱ	0.99	2.46	3.410 (5)	162
C2—H2 <i>A</i> ...F4 ⁱ	0.99	2.48	3.407 (4)	155
C3—H3 <i>A</i> ...F1 ⁱⁱⁱ	0.99	2.51	3.500 (4)	177
C4—H4 <i>A</i> ...F6 ^{iv}	0.99	2.36	3.175 (4)	139
C6—H6 <i>B</i> ...F4	0.99	2.30	3.238 (4)	158
C1—H1 <i>A</i> ...C11	0.99	2.82	3.393 (4)	118
C4—H4 <i>B</i> ...C12	0.99	2.63	3.137 (3)	112
C5—H5 <i>A</i> ...C12	0.99	2.74	3.324 (3)	118
C6—H6 <i>A</i> ...C12 ^v	0.99	2.77	3.447 (3)	126
C8—H8 <i>B</i> ...C11	0.99	2.73	3.203 (4)	110
C12—H12 <i>B</i> ...C12	0.98	2.78	3.329 (4)	116

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

(II) Dichlorido(4,11-dimethyl-1,4,8,11-tetraazabicyclo[6.6.2]hexadecane)chromium(III) hexafluorophosphate

Crystal data

[CrCl₂(C₁₄H₃₀N₄)]PF₆

M_r = 522.29

Monoclinic, *P*2₁/*c*

a = 13.6801 (19) Å

b = 12.437 (2) Å

c = 12.3864 (17) Å

β = 102.028 (11)°

V = 2061.1 (5) Å³

Z = 4

F(000) = 1076

D_x = 1.683 Mg m⁻³

Mo *K*α radiation, λ = 0.71073 Å

Cell parameters from 9091 reflections

θ = 2.6–27.9°

μ = 0.95 mm⁻¹

T = 150 K

Block, purple

0.15 × 0.15 × 0.06 mm

Data collection

Stoe IPDS2

diffractometer

Radiation source: fine-focus sealed tube

Detector resolution: 6.67 pixels mm⁻¹

ω -scans

Absorption correction: analytical

[a face-indexed absorption correction was applied; *X-AREA* (Stoe & Cie, 2002)]

T_{min} = 0.778, *T_{max}* = 0.901

13450 measured reflections

4158 independent reflections
 2073 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.091$
 $\theta_{\text{max}} = 26.3^\circ$, $\theta_{\text{min}} = 2.6^\circ$

$h = -17 \rightarrow 16$
 $k = -15 \rightarrow 13$
 $l = -15 \rightarrow 15$

Refinement

Refinement on F^2
 Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.045$
 $wR(F^2) = 0.109$
 $S = 0.78$
 4158 reflections
 255 parameters
 0 restraints

Hydrogen site location: inferred from neighbouring sites
 H-atom parameters constrained
 $w = 1/[\sigma^2(F_o^2) + (0.052P)^2]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} < 0.001$
 $\Delta\rho_{\text{max}} = 0.33 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\text{min}} = -0.79 \text{ e } \text{\AA}^{-3}$

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 (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
Cr1	0.32108 (5)	0.56984 (5)	0.21705 (5)	0.03357 (19)
Cl1	0.46100 (9)	0.62992 (9)	0.33888 (8)	0.0431 (3)
Cl2	0.36519 (9)	0.66096 (9)	0.07251 (8)	0.0423 (3)
N1	0.2651 (3)	0.4975 (3)	0.3442 (3)	0.0367 (8)
N2	0.2251 (3)	0.7026 (3)	0.2372 (2)	0.0368 (9)
N3	0.1984 (3)	0.4956 (3)	0.1138 (2)	0.0331 (8)
N4	0.3972 (3)	0.4231 (3)	0.1961 (3)	0.0384 (9)
C1	0.2305 (4)	0.5917 (3)	0.4026 (3)	0.0406 (11)
H1A	0.2892	0.6311	0.4444	0.049*
H1B	0.1910	0.5656	0.4558	0.049*
C2	0.1674 (4)	0.6666 (4)	0.3209 (3)	0.0401 (11)
H2A	0.1057	0.6291	0.2834	0.048*
H2B	0.1478	0.7298	0.3600	0.048*
C3	0.1559 (3)	0.7420 (4)	0.1358 (3)	0.0396 (10)
H3A	0.1966	0.7733	0.0863	0.048*
H3B	0.1151	0.8008	0.1573	0.048*
C4	0.0858 (3)	0.6599 (4)	0.0706 (3)	0.0421 (11)
H4A	0.0419	0.6316	0.1183	0.051*
H4B	0.0425	0.6968	0.0074	0.051*
C5	0.1360 (3)	0.5661 (4)	0.0269 (3)	0.0388 (10)
H5A	0.0838	0.5211	-0.0193	0.047*
H5B	0.1793	0.5943	-0.0214	0.047*
C6	0.2446 (4)	0.4113 (4)	0.0536 (3)	0.0426 (11)
H6A	0.2768	0.4460	-0.0020	0.051*
H6B	0.1920	0.3623	0.0144	0.051*
C7	0.3218 (4)	0.3474 (4)	0.1338 (3)	0.0416 (11)

H7A	0.2891	0.3073	0.1856	0.050*
H7B	0.3549	0.2949	0.0931	0.050*
C8	0.4513 (3)	0.3706 (4)	0.3011 (3)	0.0408 (11)
H8A	0.5060	0.4190	0.3371	0.049*
H8B	0.4825	0.3035	0.2816	0.049*
C9	0.3879 (4)	0.3433 (4)	0.3847 (3)	0.0421 (11)
H9A	0.3353	0.2922	0.3496	0.051*
H9B	0.4309	0.3055	0.4474	0.051*
C10	0.3379 (3)	0.4366 (4)	0.4302 (3)	0.0405 (11)
H10A	0.3021	0.4089	0.4862	0.049*
H10B	0.3901	0.4869	0.4679	0.049*
C11	0.1794 (3)	0.4237 (4)	0.3005 (3)	0.0395 (10)
H11A	0.1286	0.4316	0.3463	0.047*
H11B	0.2038	0.3485	0.3080	0.047*
C12	0.1300 (3)	0.4441 (4)	0.1802 (3)	0.0402 (11)
H12A	0.1056	0.3749	0.1454	0.048*
H12B	0.0713	0.4913	0.1777	0.048*
C13	0.2856 (4)	0.7989 (4)	0.2819 (3)	0.0443 (11)
H13A	0.3262	0.7820	0.3548	0.053*
H13B	0.2407	0.8589	0.2883	0.053*
H13C	0.3294	0.8189	0.2318	0.053*
C14	0.4760 (4)	0.4407 (4)	0.1303 (4)	0.0469 (12)
H14A	0.5234	0.4952	0.1666	0.056*
H14B	0.4447	0.4653	0.0560	0.056*
H14C	0.5116	0.3731	0.1252	0.056*
P1	0.12310 (10)	0.10057 (10)	0.18374 (9)	0.0430 (3)
F1	0.0182 (2)	0.0745 (3)	0.1049 (2)	0.0696 (9)
F2	0.0958 (3)	0.0212 (2)	0.2740 (2)	0.0688 (9)
F3	0.2260 (2)	0.1297 (3)	0.2639 (2)	0.0718 (9)
F4	0.1495 (2)	0.1810 (2)	0.0940 (2)	0.0637 (8)
F5	0.1746 (2)	0.0057 (2)	0.1317 (2)	0.0615 (8)
F6	0.0718 (2)	0.1974 (2)	0.2371 (2)	0.0664 (9)

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cr1	0.0378 (4)	0.0298 (4)	0.0323 (3)	-0.0011 (3)	0.0054 (3)	-0.0001 (3)
Cl1	0.0451 (7)	0.0383 (6)	0.0423 (5)	-0.0058 (5)	0.0008 (5)	0.0013 (4)
Cl2	0.0466 (7)	0.0428 (7)	0.0379 (5)	-0.0023 (5)	0.0096 (5)	0.0057 (4)
N1	0.040 (2)	0.0317 (19)	0.0366 (17)	0.0027 (17)	0.0037 (16)	0.0002 (14)
N2	0.045 (2)	0.033 (2)	0.0333 (16)	0.0000 (17)	0.0082 (16)	0.0005 (14)
N3	0.032 (2)	0.0315 (19)	0.0345 (16)	-0.0029 (16)	0.0053 (15)	0.0008 (14)
N4	0.043 (2)	0.035 (2)	0.0374 (17)	0.0034 (18)	0.0077 (16)	0.0012 (15)
C1	0.052 (3)	0.035 (3)	0.035 (2)	0.002 (2)	0.011 (2)	-0.0052 (17)
C2	0.047 (3)	0.031 (2)	0.044 (2)	0.003 (2)	0.013 (2)	-0.0005 (18)
C3	0.040 (3)	0.036 (3)	0.040 (2)	0.004 (2)	0.0009 (19)	0.0013 (18)
C4	0.042 (3)	0.040 (3)	0.042 (2)	0.004 (2)	0.003 (2)	0.004 (2)
C5	0.039 (3)	0.037 (3)	0.037 (2)	0.001 (2)	0.0000 (19)	0.0011 (18)

C6	0.050 (3)	0.037 (3)	0.041 (2)	0.002 (2)	0.008 (2)	-0.0053 (19)
C7	0.052 (3)	0.034 (2)	0.040 (2)	0.003 (2)	0.011 (2)	-0.0031 (19)
C8	0.043 (3)	0.036 (3)	0.040 (2)	0.010 (2)	0.003 (2)	0.0025 (18)
C9	0.044 (3)	0.040 (3)	0.040 (2)	0.004 (2)	0.005 (2)	0.0066 (19)
C10	0.045 (3)	0.038 (3)	0.035 (2)	0.001 (2)	0.002 (2)	0.0054 (18)
C11	0.043 (3)	0.038 (2)	0.037 (2)	-0.005 (2)	0.007 (2)	0.0022 (19)
C12	0.047 (3)	0.035 (3)	0.039 (2)	-0.009 (2)	0.009 (2)	0.0011 (18)
C13	0.051 (3)	0.031 (3)	0.049 (2)	-0.004 (2)	0.007 (2)	-0.0061 (19)
C14	0.042 (3)	0.050 (3)	0.050 (2)	0.004 (2)	0.014 (2)	0.005 (2)
P1	0.0498 (8)	0.0337 (7)	0.0480 (6)	-0.0055 (6)	0.0157 (6)	-0.0035 (5)
F1	0.057 (2)	0.078 (2)	0.0717 (18)	-0.0152 (17)	0.0075 (15)	-0.0207 (16)
F2	0.097 (3)	0.057 (2)	0.0623 (16)	-0.0099 (18)	0.0399 (17)	0.0102 (14)
F3	0.061 (2)	0.088 (3)	0.0633 (17)	-0.0109 (18)	0.0054 (15)	-0.0088 (16)
F4	0.079 (2)	0.0471 (18)	0.0706 (17)	-0.0034 (16)	0.0279 (16)	0.0136 (14)
F5	0.084 (2)	0.0391 (16)	0.0712 (17)	0.0070 (16)	0.0400 (16)	-0.0038 (13)
F6	0.069 (2)	0.0465 (18)	0.092 (2)	-0.0049 (16)	0.0352 (17)	-0.0261 (15)

Geometric parameters (Å, °)

Cr1—N1	2.093 (3)	C6—C7	1.516 (6)
Cr1—N3	2.100 (3)	C6—H6A	0.9900
Cr1—N4	2.143 (4)	C6—H6B	0.9900
Cr1—N2	2.155 (4)	C7—H7A	0.9900
Cr1—C11	2.3012 (13)	C7—H7B	0.9900
Cr1—C12	2.3031 (12)	C8—C9	1.520 (6)
N1—C11	1.499 (5)	C8—H8A	0.9900
N1—C10	1.502 (5)	C8—H8B	0.9900
N1—C1	1.504 (5)	C9—C10	1.516 (6)
N2—C3	1.490 (5)	C9—H9A	0.9900
N2—C13	1.495 (5)	C9—H9B	0.9900
N2—C2	1.497 (5)	C10—H10A	0.9900
N3—C6	1.500 (5)	C10—H10B	0.9900
N3—C5	1.507 (5)	C11—C12	1.524 (5)
N3—C12	1.511 (5)	C11—H11A	0.9900
N4—C7	1.487 (6)	C11—H11B	0.9900
N4—C14	1.498 (5)	C12—H12A	0.9900
N4—C8	1.505 (5)	C12—H12B	0.9900
C1—C2	1.508 (6)	C13—H13A	0.9800
C1—H1A	0.9900	C13—H13B	0.9800
C1—H1B	0.9900	C13—H13C	0.9800
C2—H2A	0.9900	C14—H14A	0.9800
C2—H2B	0.9900	C14—H14B	0.9800
C3—C4	1.514 (6)	C14—H14C	0.9800
C3—H3A	0.9900	P1—F5	1.579 (3)
C3—H3B	0.9900	P1—F3	1.587 (3)
C4—C5	1.510 (6)	P1—F1	1.592 (3)
C4—H4A	0.9900	P1—F4	1.592 (3)
C4—H4B	0.9900	P1—F2	1.593 (3)

C5—H5A	0.9900	P1—F6	1.605 (3)
C5—H5B	0.9900		
N1—Cr1—N3	84.18 (13)	N3—C6—C7	110.4 (3)
N1—Cr1—N4	89.33 (13)	N3—C6—H6A	109.6
N3—Cr1—N4	84.16 (14)	C7—C6—H6A	109.6
N1—Cr1—N2	85.11 (13)	N3—C6—H6B	109.6
N3—Cr1—N2	88.80 (14)	C7—C6—H6B	109.6
N4—Cr1—N2	171.44 (14)	H6A—C6—H6B	108.1
N1—Cr1—C11	91.75 (10)	N4—C7—C6	108.7 (4)
N3—Cr1—C11	172.78 (10)	N4—C7—H7A	109.9
N4—Cr1—C11	89.84 (10)	C6—C7—H7A	109.9
N2—Cr1—C11	96.82 (10)	N4—C7—H7B	109.9
N1—Cr1—C12	173.17 (11)	C6—C7—H7B	109.9
N3—Cr1—C12	92.77 (9)	H7A—C7—H7B	108.3
N4—Cr1—C12	96.45 (9)	N4—C8—C9	115.9 (4)
N2—Cr1—C12	88.73 (9)	N4—C8—H8A	108.3
C11—Cr1—C12	91.90 (5)	C9—C8—H8A	108.3
C11—N1—C10	107.4 (3)	N4—C8—H8B	108.3
C11—N1—C1	110.5 (3)	C9—C8—H8B	108.3
C10—N1—C1	106.3 (3)	H8A—C8—H8B	107.4
C11—N1—Cr1	111.9 (2)	C10—C9—C8	116.6 (4)
C10—N1—Cr1	117.3 (3)	C10—C9—H9A	108.1
C1—N1—Cr1	103.2 (2)	C8—C9—H9A	108.1
C3—N2—C13	104.7 (3)	C10—C9—H9B	108.1
C3—N2—C2	110.4 (3)	C8—C9—H9B	108.1
C13—N2—C2	108.3 (3)	H9A—C9—H9B	107.3
C3—N2—Cr1	116.7 (2)	N1—C10—C9	114.0 (3)
C13—N2—Cr1	110.7 (3)	N1—C10—H10A	108.8
C2—N2—Cr1	105.9 (2)	C9—C10—H10A	108.8
C6—N3—C5	106.6 (3)	N1—C10—H10B	108.8
C6—N3—C12	110.3 (3)	C9—C10—H10B	108.8
C5—N3—C12	108.2 (3)	H10A—C10—H10B	107.7
C6—N3—Cr1	104.1 (3)	N1—C11—C12	113.9 (3)
C5—N3—Cr1	116.2 (3)	N1—C11—H11A	108.8
C12—N3—Cr1	111.2 (2)	C12—C11—H11A	108.8
C7—N4—C14	108.1 (3)	N1—C11—H11B	108.8
C7—N4—C8	109.7 (3)	C12—C11—H11B	108.8
C14—N4—C8	104.7 (3)	H11A—C11—H11B	107.7
C7—N4—Cr1	107.5 (3)	N3—C12—C11	113.9 (4)
C14—N4—Cr1	111.4 (3)	N3—C12—H12A	108.8
C8—N4—Cr1	115.4 (2)	C11—C12—H12A	108.8
N1—C1—C2	110.6 (3)	N3—C12—H12B	108.8
N1—C1—H1A	109.5	C11—C12—H12B	108.8
C2—C1—H1A	109.5	H12A—C12—H12B	107.7
N1—C1—H1B	109.5	N2—C13—H13A	109.5
C2—C1—H1B	109.5	N2—C13—H13B	109.5
H1A—C1—H1B	108.1	H13A—C13—H13B	109.5

N2—C2—C1	109.8 (4)	N2—C13—H13C	109.5
N2—C2—H2A	109.7	H13A—C13—H13C	109.5
C1—C2—H2A	109.7	H13B—C13—H13C	109.5
N2—C2—H2B	109.7	N4—C14—H14A	109.5
C1—C2—H2B	109.7	N4—C14—H14B	109.5
H2A—C2—H2B	108.2	H14A—C14—H14B	109.5
N2—C3—C4	116.7 (4)	N4—C14—H14C	109.5
N2—C3—H3A	108.1	H14A—C14—H14C	109.5
C4—C3—H3A	108.1	H14B—C14—H14C	109.5
N2—C3—H3B	108.1	F5—P1—F3	90.73 (18)
C4—C3—H3B	108.1	F5—P1—F1	91.12 (17)
H3A—C3—H3B	107.3	F3—P1—F1	178.15 (19)
C5—C4—C3	115.3 (4)	F5—P1—F4	89.96 (15)
C5—C4—H4A	108.5	F3—P1—F4	89.57 (17)
C3—C4—H4A	108.5	F1—P1—F4	90.36 (17)
C5—C4—H4B	108.5	F5—P1—F2	90.75 (16)
C3—C4—H4B	108.5	F3—P1—F2	90.55 (17)
H4A—C4—H4B	107.5	F1—P1—F2	89.50 (18)
N3—C5—C4	115.2 (3)	F4—P1—F2	179.28 (19)
N3—C5—H5A	108.5	F5—P1—F6	179.47 (19)
C4—C5—H5A	108.5	F3—P1—F6	88.75 (17)
N3—C5—H5B	108.5	F1—P1—F6	89.40 (17)
C4—C5—H5B	108.5	F4—P1—F6	89.91 (17)
H5A—C5—H5B	107.5	F2—P1—F6	89.38 (16)
C11—N1—C1—C2	-71.2 (4)	C8—N4—C7—C6	159.7 (3)
C10—N1—C1—C2	172.6 (4)	Cr1—N4—C7—C6	33.5 (4)
Cr1—N1—C1—C2	48.6 (4)	N3—C6—C7—N4	-56.2 (5)
C3—N2—C2—C1	159.5 (3)	C7—N4—C8—C9	-64.3 (5)
C13—N2—C2—C1	-86.4 (4)	C14—N4—C8—C9	-180.0 (4)
Cr1—N2—C2—C1	32.3 (4)	Cr1—N4—C8—C9	57.2 (4)
N1—C1—C2—N2	-56.7 (5)	N4—C8—C9—C10	-61.3 (5)
C13—N2—C3—C4	178.8 (4)	C11—N1—C10—C9	66.6 (5)
C2—N2—C3—C4	-64.9 (5)	C1—N1—C10—C9	-175.1 (4)
Cr1—N2—C3—C4	56.0 (5)	Cr1—N1—C10—C9	-60.3 (4)
N2—C3—C4—C5	-60.3 (5)	C8—C9—C10—N1	61.5 (5)
C6—N3—C5—C4	-177.6 (4)	C10—N1—C11—C12	-149.2 (4)
C12—N3—C5—C4	63.7 (5)	C1—N1—C11—C12	95.2 (4)
Cr1—N3—C5—C4	-62.1 (4)	Cr1—N1—C11—C12	-19.2 (5)
C3—C4—C5—N3	62.9 (5)	C6—N3—C12—C11	95.5 (4)
C5—N3—C6—C7	170.9 (4)	C5—N3—C12—C11	-148.2 (4)
C12—N3—C6—C7	-71.8 (4)	Cr1—N3—C12—C11	-19.6 (4)
Cr1—N3—C6—C7	47.6 (4)	N1—C11—C12—N3	25.8 (5)
C14—N4—C7—C6	-86.8 (4)		

Hydrogen-bond geometry (Å, °)

<i>D</i> —H··· <i>A</i>	<i>D</i> —H	H··· <i>A</i>	<i>D</i> ··· <i>A</i>	<i>D</i> —H··· <i>A</i>
C1—H1 <i>B</i> ···F5 ⁱ	0.99	2.41	3.318 (5)	153
C2—H2 <i>A</i> ···F1 ⁱⁱ	0.99	2.50	3.094 (5)	119
C2—H2 <i>A</i> ···F6 ⁱⁱ	0.99	2.53	3.223 (6)	127
C7—H7 <i>A</i> ···F4	0.99	2.55	3.098 (6)	115
C12—H12 <i>B</i> ···F2 ⁱⁱ	0.99	2.51	3.393 (6)	149
C3—H3 <i>A</i> ···Cl2	0.99	2.73	3.280 (5)	115
C5—H5 <i>B</i> ···Cl2	0.99	2.70	3.287 (5)	119
C8—H8 <i>A</i> ···Cl1	0.99	2.70	3.258 (5)	116
C8—H8 <i>B</i> ···Cl1 ⁱⁱⁱ	0.99	2.82	3.778 (4)	162
C10—H10 <i>B</i> ···Cl1	0.99	2.70	3.270 (5)	117
C13—H13 <i>A</i> ···Cl1	0.98	2.68	3.157 (5)	111
C13—H13 <i>A</i> ···Cl2 ^{iv}	0.98	2.73	3.572 (4)	144
C14—H14 <i>B</i> ···Cl2	0.98	2.69	3.141 (5)	108

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