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Chlorido(ethylenediamine-κ^2N)(η^5-pentamethylcyclopentadienyl)iridium(III) trifluoromethanesulfonate

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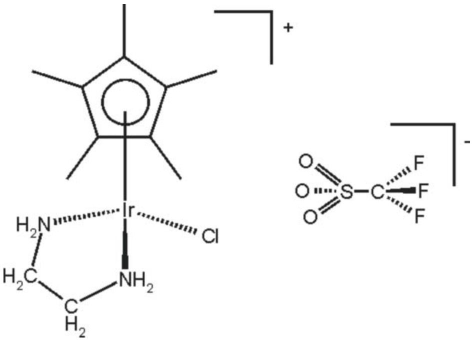
Key indicators: single-crystal X-ray study; T = 292 K; mean σ(C–C) = 0.012 Å; disorder in main residue; R factor = 0.037; wR factor = 0.079; data-to-parameter ratio = 13.1.

The title compound, $[Ir(\eta^5\text{-}C_5Me_5)Cl(en\text{-}\kappa^2N)](CF_3SO_3)$ (en is ethylenediamine) or $[Ir(C_{10}H_{15})Cl(C_2H_8N_2)](CF_3SO_3)$, contains a half-sandwich $(\eta^5\text{-}C_5Me_5)Ir^{III}$ fragment coordinated in a bidentate manner by the N atoms of an en ligand. N—H···O hydrogen bonds between the en amino groups and O atoms of the trifluoromethanesulfonate anions generate polymeric chains. The cation and anion both have C_s symmetry. The C atoms of the en ligand are disordered about a mirror plane. The crystal structure involves N—H···O, C—H···O and N—H···Cl hydrogen bonds.

Related literature

For related literature, see: Allen (2002); Bergs *et al.* (1997); Dadci *et al.* (1995); Gencaslan & Sheldrick (2005); Gleichmann *et al.* (1995); Grant *et al.* (2005); Korn & Sheldrick (1997); Poth *et al.* (2001); Schäfer & Sheldrick (2007); Scharwitz *et al.* (2007, 2007*a*,*b*,*c*); Youinou & Ziessel (1989).



Experimental

Crystal data

$[Ir(C_{10}H_{15})Cl(C_2H_8N_2)](CF_3O_3S)$
M_r = 572.04
Orthorhombic, $Pnma$
a = 15.400 (5) Å
b = 8.5728 (16) Å
c = 14.202 (5) Å
V = 1874.9 (10) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 7.42 mm^{-1}
T = 292 (2) K
0.22 × 0.20 × 0.16 mm

Data collection

Siemens P4 four-circle diffractometer
Absorption correction: ψ scan (*XPREP*; Sheldrick, 1995)
T_{min} = 0.211, T_{max} = 0.308
2309 measured reflections
1743 independent reflections
1308 reflections with $I > 2\sigma(I)$
R_{int} = 0.037
3 standard reflections every 97 reflections intensity decay: none

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.037
$wR(F^2)$ = 0.079
S = 0.99
1743 reflections
133 parameters
3 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.77 e Å^{-3}
$\Delta\rho_{min}$ = −0.93 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O2	0.90	2.09	2.954 (9)	162
C121—H121···O2	0.96	2.53	3.345 (12)	143
N1—H2···Cl1^i	0.90	2.60	3.464 (7)	160
N1′—H1′···O2	0.90	2.16	2.954 (9)	147

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

Data collection: *R3m/V* (Siemens, 1989); cell refinement: *R3m/V*; data reduction: *XDISK* (Siemens, 1989); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL-Plus* (Sheldrick, 1995); software used to prepare material for publication: *SHELXL97*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT2350).

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supplementary materials

Acta Cryst. (2007). E**63**, m1469-m1470 [**doi:10.1107/S1600536807019575**]

Chlorido(ethylenediamine-*κ*^{2}*N*)(*η*^{5}-pentamethylcyclopentadienyl)iridium(III) trifluoromethanesulfonate

M. Scharwitz, T. van Almsick and W. S. Sheldrick

Comment

Although many (pentamethylcyclopentadienyl)iridium(III) complexes with bidentate aromatic N-donor ligands such as 2,2-bipyridine (Dadci *et al.*, 1995; Youinou & Ziessel, 1989) or 1,10-phenanthroline (Gencaslan & Sheldrick, 2005) have been structurally characterized, few examples are known for ligands containing two or three amino or alkylamino N-donor functions. The Cambridge Structural Database (Version 5.27, December 2006; Allen, 2002) contain two entries [(η^5-C$_5$Me$_5$)Ir(1,2-diaminocyclohexane-κ^2*N*)](ClO$_4$)$_2$*2H$_2$O (Poth *et al.*, 2001) and [(η^5-C$_5$Me$_5$)IrCl(2-amino-3-dimethyl-aminopropionate-κ^2*N*)] *H$_2$O (Bergs *et al.*, 1997) for bidentate ligands of this type. Crystal structures have also been reported for [(η^5-C$_5$Me$_5$)Ir(1,4,7-triazacyclononane-κ^3*N*)](PF$_6$)$_2$ *CH$_3$NO$_2$ (Grant *et al.*, 2005) and [(η^5-C$_5$Me$_5$)Ir(dien-κ^3*N*)](CF$_3$SO$_3$)$_2$ (dien is diethylenetriamine) (Scharwitz *et al.*, 2007*c*), both of which contain κ^3*N* amino ligands. As part of our continuing studies on organoruthenium(II) and organoiridium(III) half-sandwhich complexes with N and S donor ligands (Gleichmann *et al.*, 1995, Korn & Sheldrick, 1997; Schäfer & Sheldrick, 2007; Scharwitz *et al.*, 2007*a*,b,c, Scharwitz *et al.*, 2007) we have now determined the structure of the title compound [(η^5-C$_5$Me$_5$)IrCl(en-κ^2*N*)](CF$_3$SO$_3$), (I).

The molecular structure of (I) is depicted in Fig. 1. Both the cation and anion exhibit crystallographic C$_s$ symmetry with the ethylenediamine carbon atoms C2 and C3 being disordered (s.o.f.s = 1/2) with symmetry-equivalent sites (i) [(i) = *x*, 0.5 − *y*, *z*] generated by the mirror plane. A twist δ conformation is observed for the five-membered chelate ring containing C2 and C3 with a λ conformation for the alternative disordered ring with C2^i and C3^i. Carbon atoms C2 and C3 are displaced respectively 0.258 (9) and −0.292 (19) Å from the best plane through the ring atoms. The Ir1—C13 distance of 2.127 (10) Å is somewhat shorter than the Ir1—C11 and Ir1—C12 distances of respectively 2.156 (7) and 2.162 (7) Å. Participation of the N1 atoms in N1—H1···O2 hydrogen bonds of length 2.954 (9) Å (H1···O2 = 2.09 Å, N1—H1···O2 = 161.8°) to symmetry-related trifluoromethanesulfonate counter-anions is observed (Fig. 1). Hydrogen bonding interactions of the type N1—H2···Cl1^i [(i) = 1 − *x*, −*y*, −*z*] (Table 1) link the cations and anions of (I) (Fig. 2).

Experimental

Ag(CF$_3$SO$_3$) (25.7 mg, 0.1 mmol) was added to a solution of [(η^5-C$_5$Me$_5$)IrCl$_2$]$_2$ (39.8 mg, 0.05 mmol) in acetone (10 ml) and stirred at room temperature for 30 min. After centrifugation of the precipitated AgCl and solvent removal from the resulting solution, the yellow residue was redissolved in CH$_3$OH/CH$_2$Cl$_2$ (10 ml, 1:1) and treated with 6.1 µl ethylendiamine (0.1 mmol). The reaction solution was heated for 2 h at 348 K and the solvent subsequently removed to afford (I), which was dissolved in methanol (3 ml) and reprecipitated with diethyl ether prior to drying in vacuum (yield 89%). Suitable crystals for X-ray analysis were grown by slow evaporation of a solution of (I) in CH$_3$OH/H$_2$O (1:). Elemental analysis found: C

27.4, H 4.5, N 5.2%; calculated for $C_{13}H_{23}ClF_3IrN_2O_3S$: C 27.3, H 4.1, N 4.9%. FAB-MS on a VG Autospec instrument (m/*z*): 537 (30) $[M—Cl]^+$, 423 (100) $[M—CF_3SO_3]^+$, 387 (60) $[M—CF_3SO_3—Cl]^+$.

Refinement

H atoms were constrained to idealized positions and refined using a riding model, with C—H distances of 0.97 Å for the methylene C atoms and 0.96 Å for the methyl groups; U_{iso}(H) = 1.2 U_{iso}(C) for methylene and 1.5 U_{iso}(C) for methyl groups. The methyl groups were allowed to rotate but not tip.

Figures

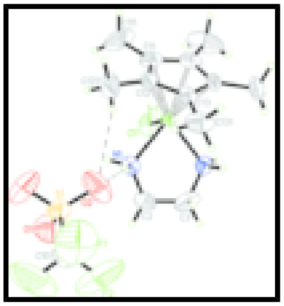

Fig. 1. Structure of the cation and counter anion of (I). Displacement ellipsoids are drawn at the 50% probability level.

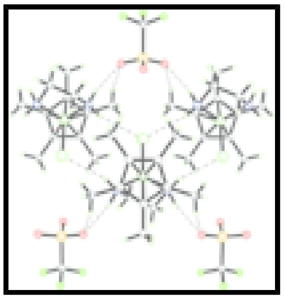

Fig. 2. Hydrogen bonding interactions in the crystal structure of (I) in a projection along [100].

Chlorido(ethylenediamine-κ^2*N*)(η^5-pentamethylcyclopentadienyl)iridium(III) trifluoromethanesulfonate

Crystal data

$[Ir(C_{10}H_{15})Cl(C_2H_8N_2)](CF_3O_3S_1)$	F_{000} = 1104
M_r = 572.04	D_x = 2.027 Mg m^{-3}
Orthorhombic, *Pnma*	Mo *K*α radiation λ = 0.71073 Å
Hall symbol: -P 2ac 2n	Cell parameters from 14 reflections
a = 15.400 (5) Å	θ = 3.5–12.7°
b = 8.5728 (16) Å	μ = 7.42 mm^{-1}
c = 14.202 (5) Å	*T* = 292 (2) K
V = 1874.9 (10) Å^3	Block, colourless
Z = 4	0.22 × 0.20 × 0.16 mm

Data collection

Siemens P4 four-circle diffractometer	$R_{\mathrm{int}} = 0.037$
Radiation source: fine-focus sealed tube	$\theta_{\max} = 25.0^\circ$
Monochromator: graphite	$\theta_{\min} = 2.7^\circ$
$T = 292(2)$ K	$h = -1\rightarrow18$
ω scans	$k = -1\rightarrow10$
Absorption correction: ψ scan (XPREP; Sheldrick, 1995)	$l = -1\rightarrow16$
$T_{\min} = 0.211$, $T_{\max} = 0.308$	3 standard reflections
2309 measured reflections	every 97 reflections
1743 independent reflections	intensity decay: 0.00%
1308 reflections with $I > 2\sigma(I)$	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.037$	H-atom parameters constrained
$wR(F^2) = 0.079$	$w = 1/[\sigma^2(F_o^2) + (0.0415P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 0.99$	$(\Delta/\sigma)_{\max} < 0.001$
1743 reflections	$\Delta\rho_{\max} = 0.77$ e Å^{-3}
133 parameters	$\Delta\rho_{\min} = -0.93$ e Å^{-3}
3 restraints	Extinction correction: none
Primary atom site location: structure-invariant direct methods	

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.

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

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

	x	y	z	U_{iso}*/U_{eq}	Occ. (<1)
Ir1	0.40783 (2)	0.2500	0.10854 (3)	0.03035 (15)	
Cl1	0.4968 (2)	0.2500	−0.0318 (2)	0.0512 (8)	

N1	0.5019 (3)	0.0926 (8)	0.1612 (4)	0.0391 (16)	0.50
H1	0.4808	0.0464	0.2133	0.047*	0.50
H2	0.5112	0.0177	0.1179	0.047*	0.50
C2	0.5844 (9)	0.166 (3)	0.1841 (14)	0.054 (5)	0.50
H21	0.6193	0.1805	0.1279	0.065*	0.50
H22	0.6168	0.1034	0.2287	0.065*	0.50
C3	0.5607 (14)	0.321 (3)	0.2265 (13)	0.068 (8)	0.50
H31	0.5318	0.3051	0.2864	0.081*	0.50
H32	0.6128	0.3818	0.2377	0.081*	0.50
N1'	0.5019 (3)	0.0926 (8)	0.1612 (4)	0.0391 (16)	0.50
H1'	0.4756	0.0139	0.1920	0.047*	0.50
H2'	0.5328	0.0520	0.1133	0.047*	0.50
C11	0.2866 (5)	0.1665 (10)	0.0516 (5)	0.053 (2)	
C111	0.2773 (7)	0.0629 (15)	−0.0350 (6)	0.106 (4)	
H111	0.2170	0.0526	−0.0509	0.159*	
H112	0.3012	−0.0382	−0.0219	0.159*	
H113	0.3080	0.1090	−0.0869	0.159*	
C12	0.2946 (5)	0.1144 (9)	0.1460 (6)	0.0400 (19)	
C121	0.2943 (6)	−0.0504 (9)	0.1767 (7)	0.070 (3)	
H121	0.3197	−0.0581	0.2383	0.105*	
H122	0.3274	−0.1119	0.1331	0.105*	
H123	0.2356	−0.0881	0.1787	0.105*	
C13	0.3020 (7)	0.2500	0.2048 (7)	0.036 (3)	
C131	0.3134 (8)	0.2500	0.3093 (8)	0.063 (4)	
H13A	0.3650	0.1932	0.3254	0.095*	0.50
H13B	0.2640	0.2013	0.3383	0.095*	0.50
H13C	0.3184	0.3555	0.3313	0.095*	0.50
S1	0.5106 (2)	−0.2500	0.3332 (2)	0.0544 (9)	
O1	0.5902 (7)	−0.2500	0.2830 (8)	0.105 (4)	
O2	0.4614 (5)	−0.1126 (9)	0.3224 (5)	0.099 (3)	
C10	0.5384 (16)	−0.2500	0.4557 (12)	0.096 (6)	
F1	0.4739 (11)	−0.2500	0.5110 (7)	0.149 (6)	
F2	0.5861 (6)	−0.1274 (14)	0.4760 (7)	0.192 (5)	

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Ir1	0.0303 (2)	0.0298 (2)	0.0309 (2)	0.000	0.0003 (2)	0.000
Cl1	0.0616 (19)	0.0525 (19)	0.0396 (15)	0.000	0.0153 (14)	0.000
N1	0.038 (3)	0.042 (4)	0.038 (4)	0.003 (3)	0.008 (3)	0.002 (3)
C2	0.039 (10)	0.065 (12)	0.059 (13)	0.006 (11)	0.002 (10)	0.003 (14)
C3	0.056 (13)	0.093 (19)	0.054 (14)	−0.017 (13)	−0.008 (10)	−0.018 (14)
N1'	0.038 (3)	0.042 (4)	0.038 (4)	0.003 (3)	0.008 (3)	0.002 (3)
C11	0.032 (4)	0.074 (6)	0.053 (4)	−0.005 (5)	−0.008 (4)	−0.006 (5)
C111	0.084 (8)	0.154 (12)	0.079 (7)	−0.010 (9)	−0.024 (7)	−0.070 (8)
C12	0.026 (4)	0.031 (4)	0.063 (5)	−0.003 (4)	−0.001 (4)	0.005 (4)
C121	0.056 (6)	0.032 (5)	0.122 (8)	−0.011 (5)	0.010 (6)	0.015 (6)
C13	0.032 (6)	0.036 (6)	0.039 (6)	0.000	0.007 (5)	0.000

C131	0.040 (7)	0.106 (12)	0.044 (7)	0.000	0.005 (6)	0.000
S1	0.059 (2)	0.059 (2)	0.0449 (18)	0.000	−0.0136 (17)	0.000
O1	0.080 (8)	0.139 (11)	0.098 (9)	0.000	0.011 (7)	0.000
O2	0.122 (6)	0.093 (6)	0.081 (5)	0.046 (5)	−0.004 (5)	0.030 (5)
C10	0.142 (18)	0.093 (15)	0.052 (10)	0.000	−0.033 (12)	0.000
F1	0.217 (15)	0.173 (13)	0.057 (6)	0.000	0.017 (8)	0.000
F2	0.206 (9)	0.233 (12)	0.137 (7)	−0.095 (8)	−0.065 (7)	−0.045 (8)

Geometric parameters (Å, °)

Ir1—N1'^{i}	2.116 (6)	C111—H112	0.9600
Ir1—N1^{i}	2.116 (6)	C111—H113	0.9600
Ir1—N1	2.116 (6)	C12—C13	1.436 (9)
Ir1—C13	2.127 (10)	C12—C121	1.479 (10)
Ir1—C11	2.156 (7)	C121—H121	0.9600
Ir1—C11^{i}	2.156 (7)	C121—H122	0.9600
Ir1—C12	2.162 (7)	C121—H123	0.9600
Ir1—C12^{i}	2.162 (7)	C13—C12^{i}	1.436 (9)
Ir1—Cl1	2.419 (3)	C13—C131	1.494 (14)
N1—C2	1.455 (10)	C131—H13A	0.9600
N1—H1	0.9000	C131—H13B	0.9600
N1—H2	0.9000	C131—H13C	0.9600
C2—C3	1.503 (13)	S1—O2^{ii}	1.409 (7)
C2—H21	0.9700	S1—O2	1.409 (7)
C2—H22	0.9700	S1—O2	1.409 (7)
C3—N1^{i}	1.49 (2)	S1—O1	1.417 (11)
C3—H31	0.9700	S1—C10	1.791 (16)
C3—H32	0.9700	O2—O2	0.00 (2)
C11—C12	1.417 (10)	C10—F1	1.27 (2)
C11—C11^{i}	1.431 (18)	C10—F2	1.315 (15)
C11—C111	1.525 (11)	C10—F2^{ii}	1.315 (15)
C111—H111	0.9600		
N1'^{i}—Ir1—N1^{i}	0.0 (5)	C12—C11—C11^{i}	108.4 (5)
N1'^{i}—Ir1—N1	79.2 (3)	C12—C11—C111	126.0 (8)
N1^{i}—Ir1—N1	79.2 (3)	C11^{i}—C11—C111	125.6 (6)
N1'^{i}—Ir1—C13	107.3 (3)	C12—C11—Ir1	71.1 (4)
N1^{i}—Ir1—C13	107.3 (3)	C11^{i}—C11—Ir1	70.6 (2)
N1—Ir1—C13	107.3 (3)	C111—C11—Ir1	125.2 (6)
N1'^{i}—Ir1—C11	159.5 (3)	C11—C111—H111	109.5
N1^{i}—Ir1—C11	159.5 (3)	C11—C111—H112	109.5
N1—Ir1—C11	120.9 (3)	H111—C111—H112	109.5
C13—Ir1—C11	65.0 (3)	C11—C111—H113	109.5
N1'^{i}—Ir1—C11^{i}	120.9 (3)	H111—C111—H113	109.5
N1^{i}—Ir1—C11^{i}	120.9 (3)	H112—C111—H113	109.5
N1—Ir1—C11^{i}	159.5 (3)	C11—C12—C13	107.6 (7)

C13—Ir1—C11^{i}	65.0 (3)	C11—C12—C121	125.4 (8)
C11—Ir1—C11^{i}	38.8 (5)	C13—C12—C121	127.0 (8)
N1'^{i}—Ir1—C12	143.9 (3)	C11—C12—Ir1	70.6 (4)
N1^{i}—Ir1—C12	143.9 (3)	C13—C12—Ir1	69.1 (5)
N1—Ir1—C12	97.0 (3)	C121—C12—Ir1	126.0 (6)
C13—Ir1—C12	39.1 (2)	C12—C121—H121	109.5
C11—Ir1—C12	38.3 (3)	C12—C121—H122	109.5
C11^{i}—Ir1—C12	64.7 (3)	H121—C121—H122	109.5
N1'^{i}—Ir1—C12^{i}	97.0 (3)	C12—C121—H123	109.5
N1^{i}—Ir1—C12^{i}	97.0 (3)	H121—C121—H123	109.5
N1—Ir1—C12^{i}	143.9 (3)	H122—C121—H123	109.5
C13—Ir1—C12^{i}	39.1 (2)	C12—C13—C12^{i}	108.1 (9)
C11—Ir1—C12^{i}	64.7 (3)	C12—C13—C131	126.0 (4)
C11^{i}—Ir1—C12^{i}	38.3 (3)	C12^{i}—C13—C131	126.0 (4)
C12—Ir1—C12^{i}	65.0 (4)	C12—C13—Ir1	71.8 (5)
N1'^{i}—Ir1—Cl1	84.45 (17)	C12^{i}—C13—Ir1	71.8 (5)
N1^{i}—Ir1—Cl1	84.45 (17)	C131—C13—Ir1	123.3 (8)
N1—Ir1—Cl1	84.45 (17)	C13—C131—H13A	109.5
C13—Ir1—Cl1	164.5 (3)	C13—C131—H13B	109.5
C11—Ir1—Cl1	100.5 (2)	H13A—C131—H13B	109.5
C11^{i}—Ir1—Cl1	100.5 (2)	C13—C131—H13C	109.5
C12—Ir1—Cl1	131.3 (2)	H13A—C131—H13C	109.5
C12^{i}—Ir1—Cl1	131.3 (2)	H13B—C131—H13C	109.5
C2—N1—Ir1	113.6 (12)	O2^{ii}—S1—O2	113.4 (7)
C2—N1—H1	108.9	O2^{ii}—S1—O2	113.4 (7)
Ir1—N1—H1	108.9	O2—S1—O2	0.0 (8)
C2—N1—H2	108.9	O2^{ii}—S1—O1	114.2 (4)
Ir1—N1—H2	108.9	O2—S1—O1	114.2 (4)
H1—N1—H2	107.7	O2—S1—O1	114.2 (4)
N1—C2—C3	105.1 (18)	O2^{ii}—S1—C10	103.5 (5)
N1—C2—H21	110.7	O2—S1—C10	103.5 (5)
C3—C2—H21	110.7	O2—S1—C10	103.5 (5)
N1—C2—H22	110.7	O1—S1—C10	106.4 (10)
C3—C2—H22	110.7	O2—O2—S1	0(10)
H21—C2—H22	108.8	F1—C10—F2	107.6 (12)
N1^{i}—C3—C2	109.6 (18)	F1—C10—F2^{ii}	107.6 (12)
N1^{i}—C3—H31	109.7	F2—C10—F2^{ii}	106.1 (19)
C2—C3—H31	109.7	F1—C10—S1	114.6 (16)
N1^{i}—C3—H32	109.7	F2—C10—S1	110.3 (11)
C2—C3—H32	109.7	F2^{ii}—C10—S1	110.3 (11)
H31—C3—H32	108.2		
N1'^{i}—Ir1—N1—C2	−12.7 (10)	N1'^{i}—Ir1—C12—C121	−93.8 (9)
N1^{i}—Ir1—N1—C2	−12.7 (10)	N1^{i}—Ir1—C12—C121	−93.8 (9)

C13—Ir1—N1—C2	−117.7 (9)	N1—Ir1—C12—C121	−12.4 (8)
C11—Ir1—N1—C2	171.6 (9)	C13—Ir1—C12—C121	−121.3 (10)
C11^{i}—Ir1—N1—C2	177.8 (9)	C11—Ir1—C12—C121	120.3 (10)
C12—Ir1—N1—C2	−156.4 (9)	C11^{i}—Ir1—C12—C121	157.9 (9)
C12^{i}—Ir1—N1—C2	−99.9 (9)	C12^{i}—Ir1—C12—C121	−159.6 (7)
Cl1—Ir1—N1—C2	72.7 (9)	Cl1—Ir1—C12—C121	76.4 (8)
Ir1—N1—C2—C3	38.5 (15)	C11—C12—C13—C12^{i}	2.6 (12)
N1—C2—C3—N1^{i}	−53.5 (14)	C121—C12—C13—C12^{i}	−176.9 (6)
N1'^{i}—Ir1—C11—C12	−109.7 (8)	Ir1—C12—C13—C12^{i}	63.0 (7)
N1^{i}—Ir1—C11—C12	−109.7 (8)	C11—C12—C13—C131	−178.7 (9)
N1—Ir1—C11—C12	58.2 (5)	C121—C12—C13—C131	1.8 (16)
C13—Ir1—C11—C12	−37.7 (4)	Ir1—C12—C13—C131	−118.3 (11)
C11^{i}—Ir1—C11—C12	−118.3 (4)	C11—C12—C13—Ir1	−60.4 (6)
C12^{i}—Ir1—C11—C12	−81.1 (6)	C121—C12—C13—Ir1	120.1 (8)
Cl1—Ir1—C11—C12	148.0 (4)	N1'^{i}—Ir1—C13—C12	−163.5 (4)
N1'^{i}—Ir1—C11—C11^{i}	8.6 (7)	N1^{i}—Ir1—C13—C12	−163.5 (4)
N1^{i}—Ir1—C11—C11^{i}	8.6 (7)	N1—Ir1—C13—C12	−79.7 (5)
N1—Ir1—C11—C11^{i}	176.5 (3)	C11—Ir1—C13—C12	37.0 (5)
C13—Ir1—C11—C11^{i}	80.56 (18)	C11^{i}—Ir1—C13—C12	79.9 (6)
C12—Ir1—C11—C11^{i}	118.3 (4)	C12^{i}—Ir1—C13—C12	116.9 (9)
C12^{i}—Ir1—C11—C11^{i}	37.2 (3)	Cl1—Ir1—C13—C12	58.4 (4)
Cl1—Ir1—C11—C11^{i}	−93.72 (9)	N1'^{i}—Ir1—C13—C12^{i}	79.7 (5)
N1'^{i}—Ir1—C11—C111	129.1 (9)	N1^{i}—Ir1—C13—C12^{i}	79.7 (5)
N1^{i}—Ir1—C11—C111	129.1 (9)	N1—Ir1—C13—C12^{i}	163.5 (4)
N1—Ir1—C11—C111	−63.0 (9)	C11—Ir1—C13—C12^{i}	−79.9 (6)
C13—Ir1—C11—C111	−158.9 (9)	C11^{i}—Ir1—C13—C12^{i}	−37.0 (5)
C11^{i}—Ir1—C11—C111	120.5 (8)	C12—Ir1—C13—C12^{i}	−116.9 (9)
C12—Ir1—C11—C111	−121.2 (10)	Cl1—Ir1—C13—C12^{i}	−58.4 (4)
C12^{i}—Ir1—C11—C111	157.7 (9)	N1'^{i}—Ir1—C13—C131	−41.90 (19)
Cl1—Ir1—C11—C111	26.8 (8)	N1^{i}—Ir1—C13—C131	−41.90 (19)
C11^{i}—C11—C12—C13	−1.6 (7)	N1—Ir1—C13—C131	41.90 (19)
C111—C11—C12—C13	179.8 (8)	C11—Ir1—C13—C131	158.5 (3)
Ir1—C11—C12—C13	59.5 (6)	C11^{i}—Ir1—C13—C131	−158.5 (3)
C11^{i}—C11—C12—C121	177.9 (7)	C12—Ir1—C13—C131	121.6 (4)
C111—C11—C12—C121	−0.7 (13)	C12^{i}—Ir1—C13—C131	−121.6 (4)
Ir1—C11—C12—C121	−121.0 (8)	Cl1—Ir1—C13—C131	180.000 (1)
C11^{i}—C11—C12—Ir1	−61.1 (2)	O2^{ii}—S1—O2—O2	0.00 (12)
C111—C11—C12—Ir1	120.3 (8)	O1—S1—O2—O2	0.0 (3)
N1'^{i}—Ir1—C12—C11	146.0 (5)	C10—S1—O2—O2	0.0 (2)
N1^{i}—Ir1—C12—C11	146.0 (5)	O2^{ii}—S1—C10—F1	−59.3 (4)
N1—Ir1—C12—C11	−132.7 (5)	O2—S1—C10—F1	59.3 (4)
C13—Ir1—C12—C11	118.5 (7)	O2—S1—C10—F1	59.3 (4)

C11^{i}—Ir1—C12—C11	37.6 (5)	O1—S1—C10—F1	180.000 (2)
C12^{i}—Ir1—C12—C11	80.1 (5)	O2^{ii}—S1—C10—F2	179.2 (14)
Cl1—Ir1—C12—C11	−43.9 (5)	O2—S1—C10—F2	−62.3 (17)
N1'^{i}—Ir1—C12—C13	27.5 (7)	O2—S1—C10—F2	−62.3 (17)
N1^{i}—Ir1—C12—C13	27.5 (7)	O1—S1—C10—F2	58.4 (15)
N1—Ir1—C12—C13	108.9 (5)	O2^{ii}—S1—C10—F2^{ii}	62.3 (17)
C11—Ir1—C12—C13	−118.5 (7)	O2—S1—C10—F2^{ii}	−179.2 (14)
C11^{i}—Ir1—C12—C13	−80.9 (5)	O2—S1—C10—F2^{ii}	−179.2 (14)
C12^{i}—Ir1—C12—C13	−38.4 (5)	O1—S1—C10—F2^{ii}	−58.4 (15)
Cl1—Ir1—C12—C13	−162.3 (4)		

Symmetry codes: (i) *x*, −*y*+1/2, *z*; (ii) *x*, −*y*−1/2, *z*.

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O2	0.90	2.09	2.954 (9)	162
C121—H121···O2	0.96	2.53	3.345 (12)	143
N1—H2···Cl1^{iii}	0.90	2.60	3.464 (7)	160
N1'—H1'···O2	0.90	2.16	2.954 (9)	147

Symmetry codes: (iii) −*x*+1, −*y*, −*z*.

Fig. 1

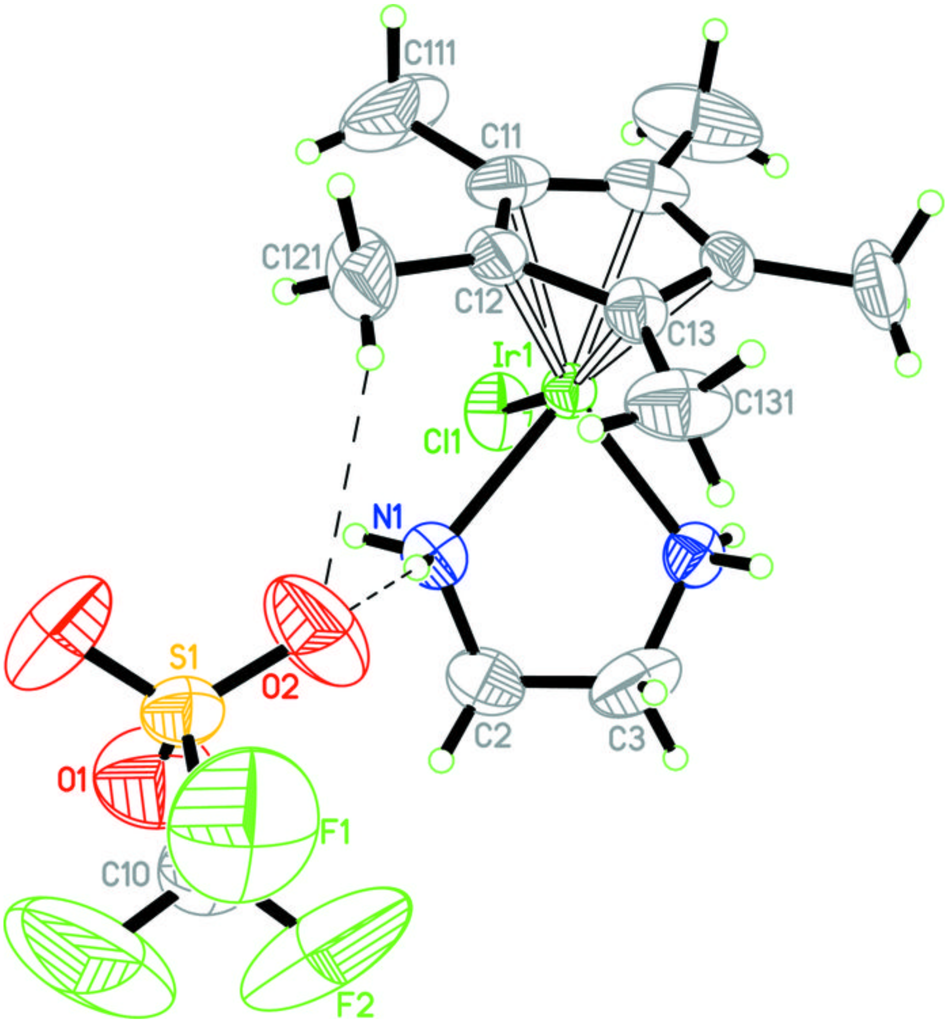

Fig. 2