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Key indicators

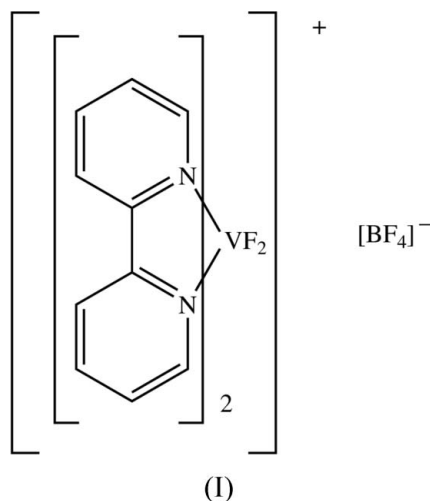
Single-crystal X-ray study
 $T = 120$ K
Mean $\sigma(\text{C}-\text{C}) = 0.004$ Å
 R factor = 0.048
 wR factor = 0.133
Data-to-parameter ratio = 15.7For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.Racemic *cis*-bis(2,2'-bipyridyl)difluorovanadium(III)
tetrafluoroborateThe title compound is a salt, $[\text{V}(\text{C}_{10}\text{H}_8\text{N}_2)_2\text{F}_2]^+ \cdot [\text{BF}_4]^-$, in which
pairs of cations, one each of Λ and Δ configuration, are linked
by a centrosymmetric π - π stacking interaction.

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Comment

The title compound, $[\text{V}(\text{bipy})_2\text{F}_2]^+ \cdot [\text{BF}_4]^-$ (bipy is 2,2'-
bipyridyl), (I), was obtained as an adventitious by-product
from the attempted preparation of [bis(2,2'-bipyridyl)(2,4-
pentanedionato-*O,O'*)vanadium(III)] tetrafluoroborate,
 $[\text{V}(\text{bipy})_2(\text{CH}_3\text{COCHCOCH}_3)_3]^+ \cdot [\text{BF}_4]^-$, from tris(2,4-
pentanedionato-*O,O'*)vanadium(III), $[\text{V}(\text{CH}_3\text{COCHCO}-$
 $\text{CH}_3)_3]$. The formation of (I) can be described as the complete
displacement of the ligands in tris(2,4-pentanedionato-
O,O')vanadium(III) and coordination instead of neutral 2,2'-
bipyridyl and of fluoride ions resulting from the adventitious
hydrolysis of part of the tetrafluoroborate component.The cations in (I) are chiral with approximate, but not exact,
local C_2 symmetry. However, the centrosymmetric space group
 $P\bar{1}$ accommodates equal numbers of Λ and Δ enantiomers;
the selected reference cation (Fig. 1) has the Λ configuration.
The individual bond angles (Table 1) around the octahedrally
coordinated metal centre show some considerable deviations
from the ideal values of 90 and 180°, ascribable both to the
rather small bite angles, *ca* 75°, of the bipyridyl ligands, and to
the mutual repulsion of the two F ligands. The V—N bonds
trans to the F ligands are significantly longer than the V—N
bonds *trans* to a bipyridyl N atom.In the two independent bipyridyl ligands, the dihedral
angles between the two ring planes are 3.0 (2) and 8.5 (2)° for
the ligands containing atoms N11 and N31, respectively. The

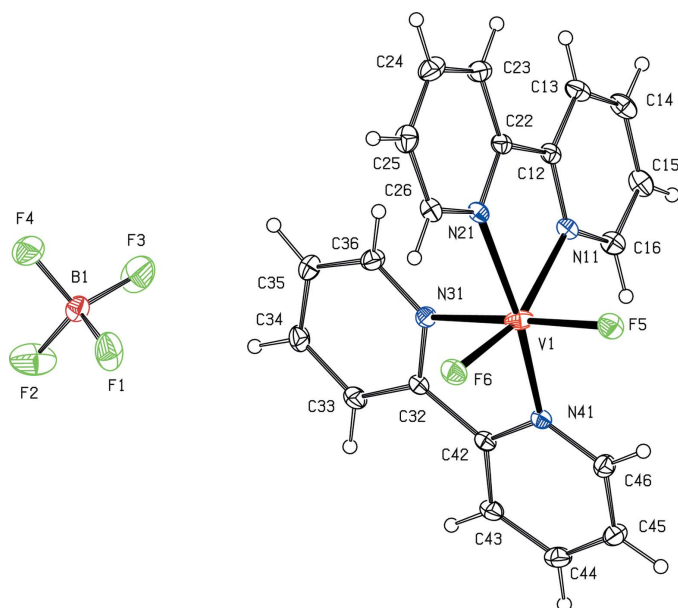


Figure 1
The ionic components of compound (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level.

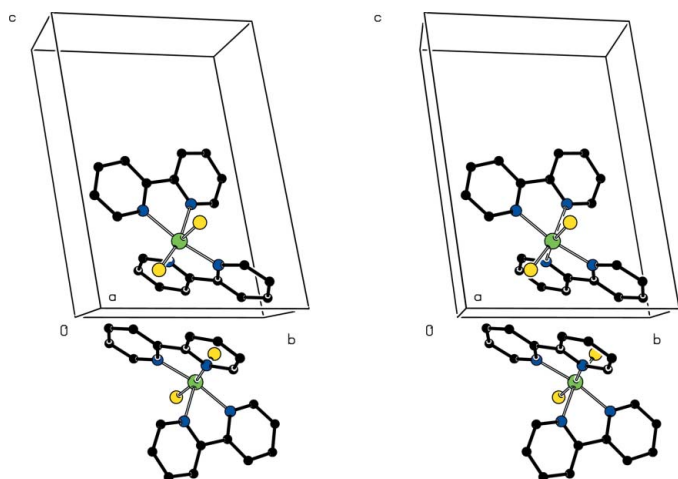


Figure 2
A stereoview of part of the crystal structure of compound (I), showing a pair of enantiomeric cations linked by a π - π stacking interaction. For the sake of clarity, H atoms have been omitted.

chelate ring containing atoms N11 and N21 is effectively planar, but that containing atoms N31 and N41 is distinctly puckered, adopting an envelope conformation with the ring folded across the N31...N41 vector.

The cations are linked into centrosymmetric pairs by means of a single aromatic π - π stacking interaction. The bipyridyl rings N11/C12–C16 at (x, y, z) and N21/C22–C26 at $(1 - x, 1 - y, -z)$ are almost parallel, with a dihedral angle between them of only $3.0(2)^\circ$. The ring-centroid separation is $3.56(2) \text{ \AA}$, and the interplanar spacing is *ca.* 3.40 \AA . This interaction thus links a racemic pair of cations across the centre of inversion at $(\frac{1}{2}, \frac{1}{2}, 0)$ (Fig. 2).

There are short C–H...F contacts, both between cations and between cations and anions (Table 2). Although inter-

actions of this type are exceptionally weak (Howard *et al.*, 1996), the concerted effect of cation...anion interactions involving three of the four F atoms in the anion may contribute to the restricted librational motion of the anion, which in simple salts often shows significant evidence for motion and/or disorder.

Experimental

A solution of tris(2,4-pentanedionato-*O,O'*)vanadium(III) (0.3 g) and 2-(2'-pyridyl)pyridinium tetrafluoroborate (0.42 g) in methanol (30 ml) was heated under reflux for 3 h in a dinitrogen atmosphere. After cooling of the reaction mixture, the solvent was removed under reduced pressure to yield the title compound, (I). Red crystals of (I) suitable for single-crystal X-ray diffraction were obtained by slow evaporation of a solution in acetone (m.p. 488 K, with charring).

Crystal data

$[\text{V}(\text{C}_{10}\text{H}_8\text{N}_2)_2\text{F}_2]\text{BF}_4$
 $M_r = 488.12$
 Triclinic, $P\bar{1}$
 $a = 8.0518(2) \text{ \AA}$
 $b = 9.3238(2) \text{ \AA}$
 $c = 13.5469(3) \text{ \AA}$
 $\alpha = 96.3421(12)^\circ$
 $\beta = 100.2723(14)^\circ$
 $\gamma = 94.9660(13)^\circ$
 $V = 988.70(4) \text{ \AA}^3$

$Z = 2$
 $D_x = 1.640 \text{ Mg m}^{-3}$
 Mo $K\alpha$ radiation
 Cell parameters from 4541 reflections
 $\theta = 3.6\text{--}27.5^\circ$
 $\mu = 0.57 \text{ mm}^{-1}$
 $T = 120(2) \text{ K}$
 Plate, red
 $0.24 \times 0.22 \times 0.04 \text{ mm}$

Data collection

Nonius KappaCCD area-detector diffractometer
 φ and ω scans
 Absorption correction: multi-scan (SADABS; Sheldrick, 2003)
 $T_{\text{min}} = 0.875, T_{\text{max}} = 0.977$
 20038 measured reflections

4541 independent reflections
 3970 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.033$
 $\theta_{\text{max}} = 27.5^\circ$
 $h = -10 \rightarrow 10$
 $k = -12 \rightarrow 12$
 $l = -17 \rightarrow 17$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.048$
 $wR(F^2) = 0.133$
 $S = 1.17$
 4541 reflections
 289 parameters
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0576P)^2 + 0.8934P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} < 0.001$
 $\Delta\rho_{\text{max}} = 0.53 \text{ e \AA}^{-3}$
 $\Delta\rho_{\text{min}} = -0.68 \text{ e \AA}^{-3}$

Table 1

Selected geometric parameters (\AA , $^\circ$).

V1–N11	2.1823 (19)	V1–N31	2.2247 (19)
V1–N21	2.114 (2)	V1–N41	2.1082 (19)
V1–F5	1.7232 (15)	V1–F6	1.7389 (15)
F5–V1–N11	88.08 (7)	N11–V1–N21	75.01 (7)
F5–V1–N21	99.97 (7)	N11–V1–N31	80.02 (7)
F5–V1–N31	160.97 (8)	N11–V1–N41	98.59 (7)
F5–V1–N41	92.76 (7)	N21–V1–N31	91.30 (7)
F6–V1–N11	160.98 (8)	N21–V1–N41	165.44 (8)
F6–V1–N21	90.18 (7)	N31–V1–N41	74.59 (7)
F6–V1–N31	88.60 (7)	F5–V1–F6	106.51 (7)
F6–V1–N41	92.94 (7)		

Table 2

Geometry of short inter-ion contacts (Å, °).

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
C13—H13 \cdots F5 ⁱ	0.95	2.50	3.265 (3)	137
C14—H14 \cdots F5 ⁱⁱ	0.95	2.42	3.145 (3)	133
C16—H16 \cdots F3 ⁱⁱⁱ	0.95	2.50	3.402 (3)	158
C24—H24 \cdots F2 ^{iv}	0.95	2.47	3.279 (4)	143
C33—H33 \cdots F6 ^v	0.95	2.42	3.209 (3)	140
C34—H34 \cdots F1	0.95	2.55	3.227 (3)	129
C43—H43 \cdots F6 ^v	0.95	2.46	3.180 (3)	132

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

All H atoms were located in difference maps and subsequently treated as riding atoms, with C—H distances of 0.95 Å and with $U_{\text{iso}}(\text{H}) = 1.2 U_{\text{eq}}(\text{C})$.

Data collection: *COLLECT* (Nonius, 1998); cell refinement: *DENZO* (Otwinowski & Minor, 1997) and *COLLECT*; data reduction: *DENZO* and *COLLECT*; program(s) used to solve structure: *OSCAIL* (McArdle, 2003) and *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *OSCAIL* and *SHELXL97*

(Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL97* and *PRPKAPPA* (Ferguson, 1999).

X-ray data were collected at the EPSRC X-ray Crystallographic Service, University of Southampton, UK; the authors thank the staff for all their help and advice.

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supporting information

Acta Cryst. (2005). E61, m1965–m1967 [doi:10.1107/S1600536805028060]

Racemic *cis*-bis(2,2'-bipyridyl)difluorovanadium(III) tetrafluoroborate

Savaridasson Jose Kavitha, Krishnaswamy Panchanatheswaran, John N. Low and Christopher Glidewell

S1. Comment

The title compound, $[\text{V}(\text{bipy})_2\text{F}_2]^+[\text{BF}_4]^-$ (bipy is 2,2'-bipyridyl), (I), was obtained as an adventitious by-product from the attempted preparation of $[\text{bis}(2,2'\text{-bipyridyl})(2,4\text{-pentanedionato-}O,O')\text{vanadium(III)}]$ tetrafluoroborate, $[\text{V}(\text{bipy})_2(\text{CH}_3\text{COCHCOCH}_3)_3]^+[\text{BF}_4]^-$, from tris(2,4-pentanedionato-*O,O'*)vanadium(III), $[\text{V}(\text{CH}_3\text{COCHCOCH}_3)_3]$. The formation of (I) can be described as the complete displacement of the ligands in tris(2,4-pentanedionato-*O,O'*)vanadium(III) and coordination instead of neutral 2,2'-bipyridyl and of fluoride ions resulting from the adventitious hydrolysis of part of the tetrafluoroborate component.

The cations in (I) are chiral with approximate, but not exact, local C_2 symmetry. However, the centrosymmetric space group $P\bar{1}$ accommodates equal numbers of Λ and Δ enantiomers; the selected reference cation (Fig. 1) has the Λ configuration. The individual bond angles (Table 1) around the octahedrally coordinated metal centre show some considerable deviations from the ideal values of 90 and 180°, ascribable both to the rather small bite angles, *ca* 75°, of the bipyridyl ligands, and to the mutual repulsion of the two F ligands. The V—N bonds *trans* to the F ligands are significantly longer than the V—N bonds *trans* to a bipyridyl N atom.

In the two independent bipyridyl ligands, the dihedral angles between the two ring planes are 3.0 (2) and 8.5 (2)° for the ligands containing atoms N11 and N31, respectively. The chelate ring containing atoms N11 and N21 is effectively planar, but that containing atoms N31 and N41 is distinctly puckered, adopting an envelope conformation with the ring folded across the N31⋯N41 vector.

The cations are linked into centrosymmetric pairs by means of a single aromatic π – π stacking interaction. The bipyridyl rings N11/C12–C16 at (*x*, *y*, *z*) and N21/C22–C26 at (1 – *x*, 1 – *y*, –*z*) are almost parallel, with a dihedral angle between them of only 3.0 (2)°. The ring–centroid separation is 3.558 (23) Å, and the interplanar spacing is *ca* 3.40 Å. This interaction thus links a racemic pair of cations across the centre of inversion at (1/2, 1/2, 0) (Fig. 2).

There are short C—H⋯F contacts, both between cations and between cations and anions (Table 2). Although interactions of this type are exceptionally weak (Howard *et al.*, 1996), the concerted effect of cation⋯anion interactions involving three of the four F atoms in the anion may contribute to the restricted librational motion of the anion, which in simple salts often shows significant evidence for motion and/or disorder.

S2. Experimental

A solution of tris(2,4-pentanedionato-*O,O'*)vanadium(III) (0.3 g) and 2-(2'-pyridyl)pyridinium tetrafluoroborate (0.42 g) in methanol (30 ml) was heated under reflux for 3 h in a dinitrogen atmosphere. After cooling of the reaction mixture, the solvent was removed under reduced pressure to yield the title compound, (I). Red crystals of (I) suitable for single-crystal X-ray diffraction were obtained by slow evaporation of a solution in acetone (m.p. 488 K, with charring).

S3. Refinement

All H atoms were located in difference maps and subsequently treated as riding atoms, with C—H distances of 0.95 Å and with $U_{\text{iso}}(\text{H}) = 1.2 U_{\text{eq}}(\text{C})$.

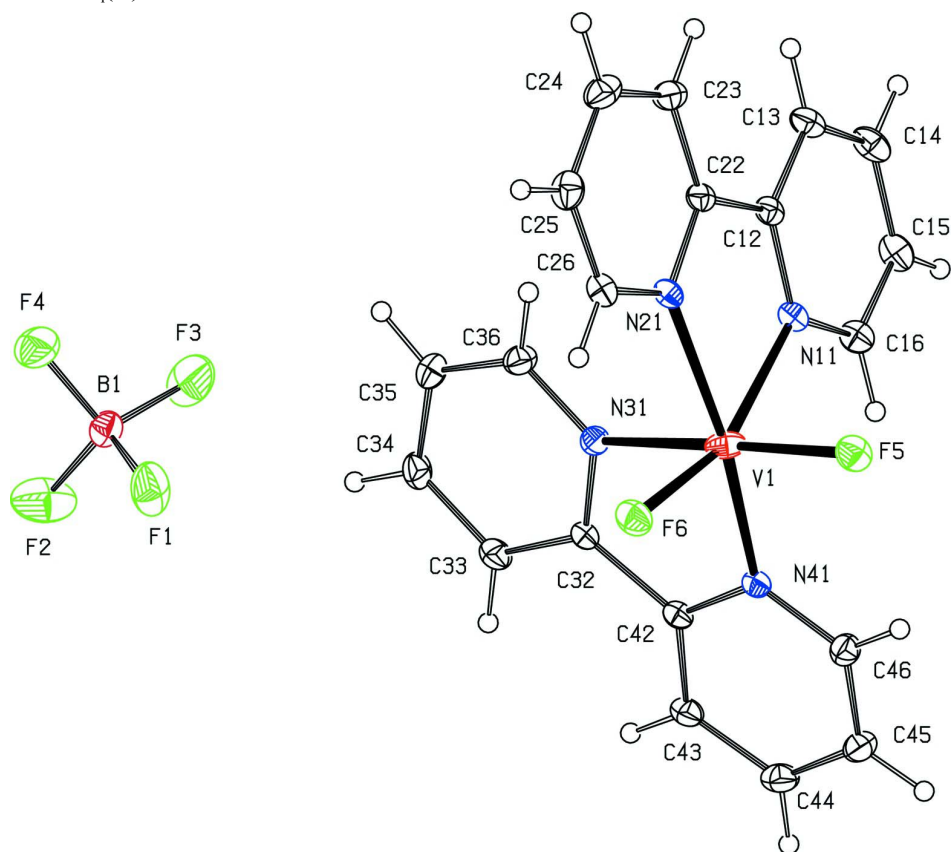


Figure 1

The ionic components of compound (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level.

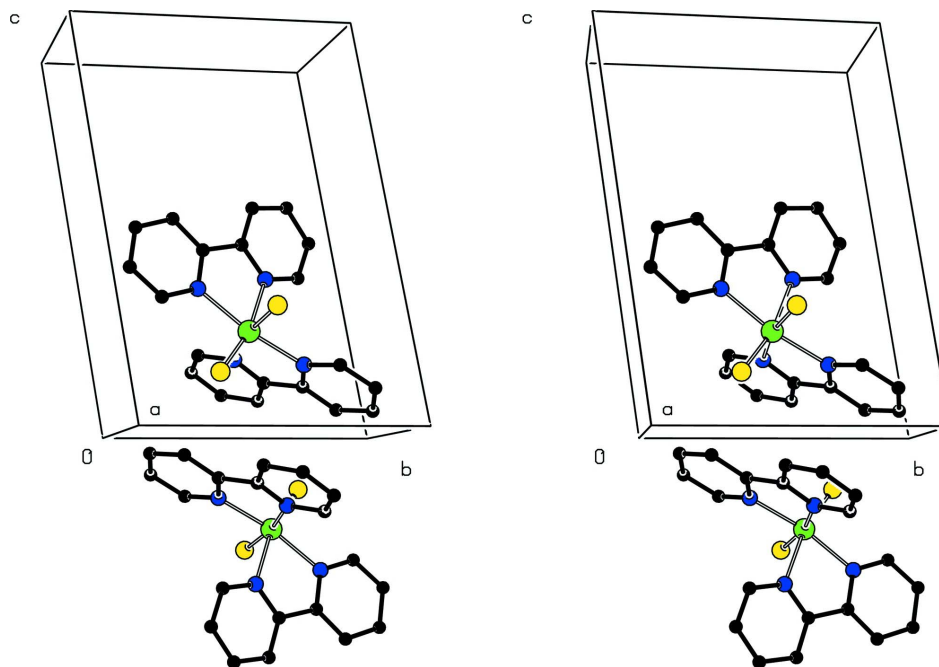


Figure 2

A stereoview of part of the crystal structure of compound (I), showing a pair of enantiomeric cations linked by a π - π stacking interaction. For the sake of clarity, H atoms have been omitted.

***cis*-bis(2,2'-bipyridyl)difluorovanadium(III) tetrafluoroborate**

Crystal data

[V(C₁₀H₈N₂)₂F₂]₂BF₄

$M_r = 488.12$

Triclinic, *P*1

Hall symbol: -P 1

$a = 8.0518$ (2) Å

$b = 9.3238$ (2) Å

$c = 13.5469$ (3) Å

$\alpha = 96.3421$ (12)°

$\beta = 100.2723$ (14)°

$\gamma = 94.9660$ (13)°

$V = 988.70$ (4) Å³

$Z = 2$

$F(000) = 492$

$D_x = 1.640$ Mg m⁻³

Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 4541 reflections

$\theta = 3.6$ – 27.5 °

$\mu = 0.57$ mm⁻¹

$T = 120$ K

Plate, red

$0.24 \times 0.22 \times 0.04$ mm

Data collection

Nonius KappaCCD area-detector
diffractometer

Radiation source: Bruker Nonius FR91 rotating
anode

Graphite monochromator

Detector resolution: 9.091 pixels mm⁻¹

φ and ω scans

Absorption correction: multi-scan
(*SADABS*; Sheldrick, 2003)

$T_{\min} = 0.875$, $T_{\max} = 0.977$

20038 measured reflections

4541 independent reflections

3970 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.033$

$\theta_{\max} = 27.5$ °, $\theta_{\min} = 3.6$ °

$h = -10 \rightarrow 10$

$k = -12 \rightarrow 12$

$l = -17 \rightarrow 17$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.048$

$wR(F^2) = 0.133$

$S = 1.17$

4541 reflections

289 parameters

0 restraints

Primary atom site location: structure-invariant
direct methods

Secondary atom site location: difference Fourier
map

Hydrogen site location: inferred from
neighbouring sites

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0576P)^2 + 0.8934P]$

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

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

$\Delta\rho_{\max} = 0.53 \text{ e } \text{\AA}^{-3}$

$\Delta\rho_{\min} = -0.68 \text{ e } \text{\AA}^{-3}$

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

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
V1	0.79578 (5)	0.46813 (5)	0.23463 (3)	0.02652 (14)
F1	0.8248 (2)	0.9922 (2)	0.71574 (16)	0.0565 (5)
F2	0.7214 (3)	1.0582 (3)	0.85451 (16)	0.0689 (7)
F3	0.5404 (2)	0.9771 (2)	0.7049 (2)	0.0695 (7)
F4	0.6933 (2)	1.19586 (19)	0.72924 (15)	0.0520 (5)
F5	0.81008 (19)	0.34016 (17)	0.13539 (11)	0.0319 (3)
F6	1.00199 (18)	0.54451 (17)	0.28618 (11)	0.0328 (3)
N11	0.5221 (2)	0.4474 (2)	0.17888 (14)	0.0213 (4)
N21	0.7537 (2)	0.6496 (2)	0.15632 (14)	0.0212 (4)
N31	0.7122 (2)	0.5766 (2)	0.36922 (14)	0.0217 (4)
N41	0.8016 (2)	0.3136 (2)	0.33735 (14)	0.0199 (4)
C12	0.4627 (3)	0.5507 (2)	0.12442 (16)	0.0204 (4)
C13	0.2907 (3)	0.5490 (3)	0.08569 (18)	0.0262 (5)
C14	0.1787 (3)	0.4382 (3)	0.10404 (19)	0.0300 (5)
C15	0.2397 (3)	0.3318 (3)	0.15904 (19)	0.0288 (5)
C16	0.4126 (3)	0.3389 (3)	0.19464 (18)	0.0260 (5)
C22	0.5938 (3)	0.6637 (2)	0.10969 (16)	0.0206 (4)
C23	0.5590 (3)	0.7772 (3)	0.05395 (19)	0.0276 (5)
C24	0.6906 (4)	0.8796 (3)	0.0470 (2)	0.0332 (6)
C25	0.8533 (3)	0.8668 (3)	0.0966 (2)	0.0311 (5)
C26	0.8816 (3)	0.7506 (3)	0.15031 (18)	0.0253 (5)
C32	0.7428 (3)	0.5080 (2)	0.45255 (17)	0.0196 (4)
C33	0.7266 (3)	0.5734 (3)	0.54698 (18)	0.0276 (5)
C34	0.6704 (3)	0.7102 (3)	0.55481 (19)	0.0298 (5)
C35	0.6290 (3)	0.7758 (3)	0.46860 (19)	0.0284 (5)
C36	0.6540 (3)	0.7069 (3)	0.37773 (19)	0.0265 (5)
C42	0.7883 (3)	0.3579 (2)	0.43366 (17)	0.0207 (4)
C43	0.8095 (3)	0.2667 (3)	0.50816 (18)	0.0254 (5)
C44	0.8433 (3)	0.1257 (3)	0.4819 (2)	0.0304 (5)
C45	0.8580 (3)	0.0807 (3)	0.3835 (2)	0.0298 (5)
C46	0.8377 (3)	0.1774 (3)	0.31348 (19)	0.0253 (5)
B1	0.6933 (4)	1.0550 (3)	0.7510 (2)	0.0295 (6)
H13	0.2506	0.6225	0.0473	0.031*
H14	0.0604	0.4356	0.0788	0.036*

H15	0.1645	0.2551	0.1723	0.035*
H16	0.4554	0.2646	0.2314	0.031*
H23	0.4463	0.7848	0.0209	0.033*
H24	0.6692	0.9576	0.0086	0.040*
H25	0.9446	0.9370	0.0939	0.037*
H26	0.9936	0.7414	0.1838	0.030*
H33	0.7536	0.5255	0.6052	0.033*
H34	0.6605	0.7580	0.6188	0.036*
H35	0.5842	0.8668	0.4717	0.034*
H36	0.6290	0.7537	0.3188	0.032*
H43	0.8010	0.3001	0.5757	0.031*
H44	0.8563	0.0605	0.5312	0.036*
H45	0.8817	-0.0155	0.3644	0.036*
H46	0.8497	0.1468	0.2461	0.030*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
V1	0.0245 (2)	0.0341 (3)	0.0276 (2)	0.01180 (17)	0.01147 (17)	0.01558 (17)
F1	0.0390 (10)	0.0626 (12)	0.0621 (12)	0.0103 (9)	0.0115 (9)	-0.0243 (10)
F2	0.0759 (15)	0.1036 (18)	0.0448 (11)	0.0383 (13)	0.0306 (11)	0.0327 (12)
F3	0.0325 (10)	0.0416 (11)	0.125 (2)	-0.0053 (8)	-0.0017 (11)	0.0019 (12)
F4	0.0554 (11)	0.0354 (10)	0.0634 (12)	0.0003 (8)	0.0020 (9)	0.0166 (9)
F5	0.0307 (8)	0.0374 (8)	0.0278 (8)	0.0049 (6)	0.0068 (6)	0.0027 (6)
F6	0.0255 (7)	0.0368 (8)	0.0338 (8)	-0.0008 (6)	0.0012 (6)	0.0042 (6)
N11	0.0186 (9)	0.0267 (10)	0.0182 (9)	0.0019 (7)	0.0029 (7)	0.0026 (7)
N21	0.0217 (9)	0.0255 (10)	0.0156 (9)	0.0016 (7)	0.0021 (7)	0.0026 (7)
N31	0.0248 (9)	0.0211 (9)	0.0194 (9)	0.0036 (7)	0.0043 (7)	0.0027 (7)
N41	0.0177 (9)	0.0219 (9)	0.0195 (9)	0.0018 (7)	0.0015 (7)	0.0034 (7)
C12	0.0206 (10)	0.0267 (11)	0.0144 (10)	0.0054 (8)	0.0045 (8)	0.0002 (8)
C13	0.0207 (11)	0.0379 (14)	0.0203 (11)	0.0075 (9)	0.0027 (9)	0.0034 (10)
C14	0.0178 (11)	0.0489 (16)	0.0227 (12)	0.0033 (10)	0.0039 (9)	0.0017 (11)
C15	0.0223 (11)	0.0371 (14)	0.0261 (12)	-0.0036 (10)	0.0061 (9)	0.0026 (10)
C16	0.0252 (12)	0.0309 (13)	0.0230 (12)	0.0008 (9)	0.0063 (9)	0.0070 (9)
C22	0.0230 (11)	0.0251 (11)	0.0147 (10)	0.0065 (9)	0.0050 (8)	0.0013 (8)
C23	0.0302 (12)	0.0282 (12)	0.0258 (12)	0.0097 (10)	0.0049 (10)	0.0058 (10)
C24	0.0437 (15)	0.0270 (13)	0.0332 (14)	0.0099 (11)	0.0120 (12)	0.0098 (10)
C25	0.0363 (14)	0.0270 (13)	0.0303 (13)	-0.0027 (10)	0.0103 (11)	0.0035 (10)
C26	0.0241 (11)	0.0287 (12)	0.0215 (11)	-0.0016 (9)	0.0030 (9)	0.0014 (9)
C32	0.0172 (10)	0.0223 (11)	0.0181 (10)	0.0009 (8)	0.0008 (8)	0.0021 (8)
C33	0.0318 (13)	0.0317 (13)	0.0183 (11)	0.0055 (10)	0.0014 (9)	0.0028 (9)
C34	0.0369 (14)	0.0294 (13)	0.0213 (12)	0.0033 (10)	0.0056 (10)	-0.0043 (9)
C35	0.0324 (13)	0.0230 (12)	0.0307 (13)	0.0057 (10)	0.0090 (10)	-0.0003 (10)
C36	0.0315 (12)	0.0248 (12)	0.0252 (12)	0.0078 (10)	0.0061 (10)	0.0065 (9)
C42	0.0170 (10)	0.0247 (11)	0.0196 (11)	0.0008 (8)	0.0011 (8)	0.0036 (8)
C43	0.0257 (11)	0.0294 (12)	0.0204 (11)	0.0027 (9)	0.0004 (9)	0.0065 (9)
C44	0.0293 (12)	0.0298 (13)	0.0327 (13)	0.0044 (10)	0.0013 (10)	0.0133 (10)
C45	0.0300 (13)	0.0220 (12)	0.0370 (14)	0.0058 (9)	0.0027 (10)	0.0057 (10)

C46	0.0253 (11)	0.0231 (11)	0.0275 (12)	0.0036 (9)	0.0055 (9)	0.0013 (9)
B1	0.0278 (14)	0.0258 (14)	0.0353 (15)	0.0022 (11)	0.0082 (12)	0.0021 (11)

Geometric parameters (Å, °)

V1—N11	2.1823 (19)	C26—H26	0.95
V1—N21	2.114 (2)	N31—C36	1.339 (3)
V1—F5	1.7232 (15)	N31—C32	1.354 (3)
V1—N31	2.2247 (19)	C32—C33	1.388 (3)
V1—N41	2.1082 (19)	C32—C42	1.484 (3)
V1—F6	1.7389 (15)	C33—C34	1.389 (4)
N11—C16	1.344 (3)	C33—H33	0.95
N11—C12	1.346 (3)	C34—C35	1.380 (4)
C12—C13	1.389 (3)	C34—H34	0.95
C12—C22	1.481 (3)	C35—C36	1.380 (3)
C13—C14	1.385 (4)	C35—H35	0.95
C13—H13	0.95	C36—H36	0.95
C14—C15	1.378 (4)	N41—C46	1.346 (3)
C14—H14	0.95	N41—C42	1.350 (3)
C15—C16	1.383 (3)	C42—C43	1.387 (3)
C15—H15	0.95	C43—C44	1.386 (4)
C16—H16	0.95	C43—H43	0.95
N21—C22	1.353 (3)	C44—C45	1.381 (4)
N21—C26	1.354 (3)	C44—H44	0.95
C22—C23	1.386 (3)	C45—C46	1.378 (4)
C23—C24	1.387 (4)	C45—H45	0.95
C23—H23	0.95	C46—H46	0.95
C24—C25	1.384 (4)	B1—F2	1.377 (4)
C24—H24	0.95	B1—F4	1.377 (3)
C25—C26	1.383 (4)	B1—F3	1.380 (3)
C25—H25	0.95	B1—F1	1.384 (3)
F5—V1—N11	88.08 (7)	C24—C25—H25	120.4
F5—V1—N21	99.97 (7)	N21—C26—C25	121.7 (2)
F5—V1—N31	160.97 (8)	N21—C26—H26	119.2
F5—V1—N41	92.76 (7)	C25—C26—H26	119.2
F6—V1—N11	160.98 (8)	C36—N31—C32	118.6 (2)
F6—V1—N21	90.18 (7)	C36—N31—V1	126.54 (16)
F6—V1—N31	88.60 (7)	C32—N31—V1	114.40 (14)
F6—V1—N41	92.94 (7)	N31—C32—C33	121.7 (2)
N11—V1—N21	75.01 (7)	N31—C32—C42	114.76 (19)
N11—V1—N31	80.02 (7)	C33—C32—C42	123.6 (2)
N11—V1—N41	98.59 (7)	C32—C33—C34	118.8 (2)
N21—V1—N31	91.30 (7)	C32—C33—H33	120.6
N21—V1—N41	165.44 (8)	C34—C33—H33	120.6
N31—V1—N41	74.59 (7)	C35—C34—C33	119.3 (2)
F5—V1—F6	106.51 (7)	C35—C34—H34	120.4
C16—N11—C12	119.1 (2)	C33—C34—H34	120.4

C16—N11—V1	124.34 (16)	C34—C35—C36	118.8 (2)
C12—N11—V1	116.51 (15)	C34—C35—H35	120.6
N11—C12—C13	121.5 (2)	C36—C35—H35	120.6
N11—C12—C22	114.97 (19)	N31—C36—C35	122.7 (2)
C13—C12—C22	123.5 (2)	N31—C36—H36	118.7
C14—C13—C12	118.8 (2)	C35—C36—H36	118.7
C14—C13—H13	120.6	C46—N41—C42	118.8 (2)
C12—C13—H13	120.6	C46—N41—V1	122.16 (16)
C15—C14—C13	119.6 (2)	C42—N41—V1	118.56 (15)
C15—C14—H14	120.2	N41—C42—C43	121.9 (2)
C13—C14—H14	120.2	N41—C42—C32	115.22 (19)
C14—C15—C16	118.7 (2)	C43—C42—C32	122.8 (2)
C14—C15—H15	120.6	C44—C43—C42	118.6 (2)
C16—C15—H15	120.6	C44—C43—H43	120.7
N11—C16—C15	122.1 (2)	C42—C43—H43	120.7
N11—C16—H16	118.9	C45—C44—C43	119.5 (2)
C15—C16—H16	118.9	C45—C44—H44	120.2
C22—N21—C26	119.2 (2)	C43—C44—H44	120.2
C22—N21—V1	118.69 (15)	C46—C45—C44	119.0 (2)
C26—N21—V1	122.16 (16)	C46—C45—H45	120.5
N21—C22—C23	121.4 (2)	C44—C45—H45	120.5
N21—C22—C12	114.7 (2)	N41—C46—C45	122.2 (2)
C23—C22—C12	123.8 (2)	N41—C46—H46	118.9
C22—C23—C24	119.3 (2)	C45—C46—H46	118.9
C22—C23—H23	120.4	F2—B1—F4	108.1 (2)
C24—C23—H23	120.4	F2—B1—F3	111.9 (3)
C25—C24—C23	119.2 (2)	F4—B1—F3	109.3 (2)
C25—C24—H24	120.4	F2—B1—F1	108.1 (2)
C23—C24—H24	120.4	F4—B1—F1	109.7 (2)
C26—C25—C24	119.3 (2)	F3—B1—F1	109.6 (2)
C26—C25—H25	120.4		
F5—V1—N11—C16	79.28 (19)	F5—V1—N31—C36	-124.0 (3)
F6—V1—N11—C16	-139.9 (2)	F6—V1—N31—C36	92.8 (2)
N41—V1—N11—C16	-13.23 (19)	N41—V1—N31—C36	-173.8 (2)
N21—V1—N11—C16	-179.9 (2)	N21—V1—N31—C36	2.6 (2)
N31—V1—N11—C16	-85.80 (19)	N11—V1—N31—C36	-71.9 (2)
F5—V1—N11—C12	-100.00 (16)	F5—V1—N31—C32	63.7 (3)
F6—V1—N11—C12	40.8 (3)	F6—V1—N31—C32	-79.49 (16)
N41—V1—N11—C12	167.50 (16)	N41—V1—N31—C32	13.94 (15)
N21—V1—N11—C12	0.86 (15)	N21—V1—N31—C32	-169.64 (16)
N31—V1—N11—C12	94.92 (16)	N11—V1—N31—C32	115.82 (16)
C16—N11—C12—C13	1.1 (3)	C36—N31—C32—C33	-4.5 (3)
V1—N11—C12—C13	-179.59 (17)	V1—N31—C32—C33	168.41 (18)
C16—N11—C12—C22	-178.86 (19)	C36—N31—C32—C42	173.5 (2)
V1—N11—C12—C22	0.5 (2)	V1—N31—C32—C42	-13.6 (2)
N11—C12—C13—C14	0.1 (3)	N31—C32—C33—C34	2.9 (4)
C22—C12—C13—C14	-179.9 (2)	C42—C32—C33—C34	-175.0 (2)

C12—C13—C14—C15	-0.7 (4)	C32—C33—C34—C35	1.3 (4)
C13—C14—C15—C16	0.0 (4)	C33—C34—C35—C36	-3.6 (4)
C12—N11—C16—C15	-1.8 (3)	C32—N31—C36—C35	2.0 (4)
V1—N11—C16—C15	178.94 (18)	V1—N31—C36—C35	-169.93 (18)
C14—C15—C16—N11	1.3 (4)	C34—C35—C36—N31	2.0 (4)
F5—V1—N21—C22	83.05 (17)	F5—V1—N41—C46	9.85 (18)
F6—V1—N21—C22	-170.15 (16)	F6—V1—N41—C46	-96.85 (18)
N41—V1—N21—C22	-67.7 (3)	N21—V1—N41—C46	161.0 (3)
N11—V1—N21—C22	-2.23 (16)	N11—V1—N41—C46	98.32 (18)
N31—V1—N21—C22	-81.54 (16)	N31—V1—N41—C46	175.41 (19)
F5—V1—N21—C26	-96.08 (18)	F5—V1—N41—C42	-178.07 (16)
F6—V1—N21—C26	10.72 (18)	F6—V1—N41—C42	75.23 (16)
N41—V1—N21—C26	113.2 (3)	N21—V1—N41—C42	-26.9 (4)
N11—V1—N21—C26	178.64 (19)	N11—V1—N41—C42	-89.60 (16)
N31—V1—N21—C26	99.33 (18)	N31—V1—N41—C42	-12.50 (15)
C26—N21—C22—C23	1.6 (3)	C46—N41—C42—C43	-0.5 (3)
V1—N21—C22—C23	-177.53 (17)	V1—N41—C42—C43	-172.87 (17)
C26—N21—C22—C12	-177.67 (19)	C46—N41—C42—C32	-178.12 (19)
V1—N21—C22—C12	3.2 (2)	V1—N41—C42—C32	9.5 (2)
N11—C12—C22—N21	-2.3 (3)	N31—C32—C42—N41	3.3 (3)
C13—C12—C22—N21	177.7 (2)	C33—C32—C42—N41	-178.7 (2)
N11—C12—C22—C23	178.4 (2)	N31—C32—C42—C43	-174.2 (2)
C13—C12—C22—C23	-1.5 (3)	C33—C32—C42—C43	3.7 (3)
N21—C22—C23—C24	-0.9 (4)	N41—C42—C43—C44	-0.8 (3)
C12—C22—C23—C24	178.3 (2)	C32—C42—C43—C44	176.7 (2)
C22—C23—C24—C25	-0.6 (4)	C42—C43—C44—C45	1.2 (4)
C23—C24—C25—C26	1.3 (4)	C43—C44—C45—C46	-0.3 (4)
C22—N21—C26—C25	-0.9 (3)	C42—N41—C46—C45	1.4 (3)
V1—N21—C26—C25	178.27 (18)	V1—N41—C46—C45	173.48 (18)
C24—C25—C26—N21	-0.6 (4)	C44—C45—C46—N41	-1.0 (4)

Hydrogen-bond geometry (\AA , $^\circ$)

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
C13—H13 \cdots F5 ⁱ	0.95	2.50	3.265 (3)	137
C14—H14 \cdots F5 ⁱⁱ	0.95	2.42	3.145 (3)	133
C16—H16 \cdots F3 ⁱⁱⁱ	0.95	2.50	3.402 (3)	158
C24—H24 \cdots F2 ^{iv}	0.95	2.47	3.279 (4)	143
C33—H33 \cdots F6 ^v	0.95	2.42	3.209 (3)	140
C34—H34 \cdots F1	0.95	2.55	3.227 (3)	129
C43—H43 \cdots F6 ^v	0.95	2.46	3.180 (3)	132

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