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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 = 0.043
 wR factor = 0.105
 Data-to-parameter ratio = 18.3

For details of how these key indicators were
 automatically derived from the article, see
<http://journals.iucr.org/e>.

(2,2'-Bipyridyl)bis(pentane-2,4-dionato)vanadium(III) perchlorate dichloromethane solvate

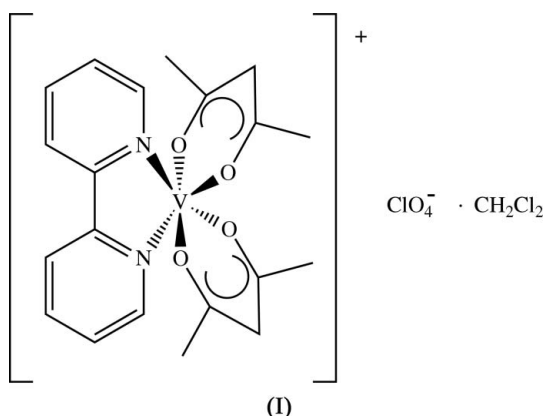
The title compound is a stoichiometrically solvated salt, $[\text{V}(\text{C}_5\text{H}_7\text{O}_2)_2(\text{C}_{10}\text{H}_8\text{N}_2)]\text{ClO}_4 \cdot \text{CH}_2\text{Cl}_2$. The ionic components are linked by three $\text{C}-\text{H} \cdots \text{O}$ hydrogen bonds into chains from which the solvent dichloromethane molecules are pendant, and pairs of antiparallel (inversion-related) chains are linked by a single $\pi-\pi$ stacking interaction.

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Comment

The nitrogen heterocycles 1,10-phenanthroline (phen) and 2,2'-bipyridine (bipy) are among the most widely utilized chelating ligands in coordination chemistry (Lever, 2003). We have recently prepared mixed-ligand vanadium(III) complexes containing both pentane-2,4-dionate (also called acetylacetonate; acac) and 1,10-phenanthroline ligands (Kavitha *et al.*, 2006) in order to assess their antidiabetic activity. Although phen and bipy have similar structures, there is a difference in their chelating ability, which has been attributed to the difference in the geometry of the free molecules (Reyzer & Brodbelt, 1999; Oresmaa *et al.*, 2002). The title complex, (I), which contains a 2,2'-bipyridine ligand, has been prepared in order to compare its structure with that of the 1,10-phenanthroline analogue and with the longer term aim of testing its antidiabetic activity.



In the cation, which has approximate twofold rotational symmetry, the V atom is octahedrally coordinated (Table 1) by three bidentate ligands (two acac and one bipy): each cation is thus chiral. The cation in the arbitrarily chosen asymmetric unit (Fig. 1) has a Δ configuration, but space-group symmetry generates a racemic mixture of Λ and Δ enantiomers. The component species are linked by three independent two-centre $\text{C}-\text{H} \cdots \text{O}$ hydrogen bonds and one planar three-centre $\text{C}-\text{H} \cdots (\text{O})_2$ hydrogen bond (Table 2). One further $\text{C}-\text{H} \cdots \text{O}$ hydrogen bond links the ionic aggregates into a

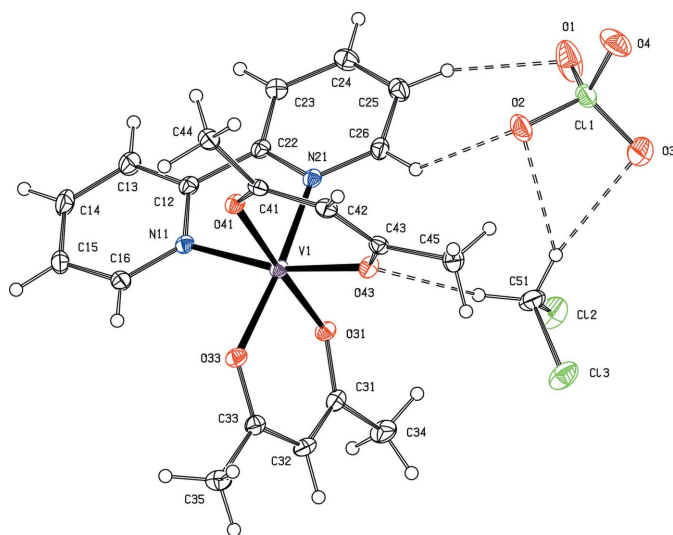


Figure 1
The asymmetric unit of (I), showing 30% displacement ellipsoids (arbitrary spheres for the H atoms). The C—H···O interactions are indicated by dashed lines.

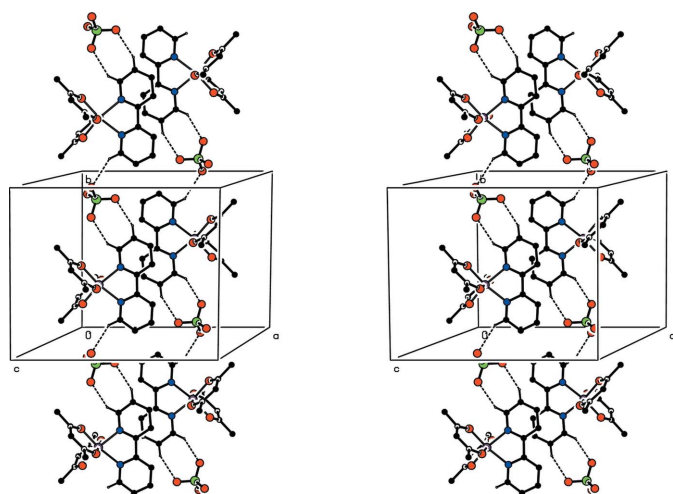


Figure 2
A stereoview of part of the crystal structure of (I), showing the formation of a π -stacked pair of hydrogen-bonded (dashed lines) chains. For clarity, the dichloromethane molecules have been omitted, as have the H atoms not involved in the hydrogen-bonding motifs shown.

$C(10)C(11)[R_2^2(7)]$ (Bernstein *et al.*, 1995) chain of rings running parallel to the [010] direction (Fig. 2); the dichloromethane molecules are pendant from this chain.

A single π - π stacking interaction links antiparallel pairs of these chains (Fig. 2). The rings (N21/C22–C26) in the cations at (x, y, z) and $(1-x, 1-y, 1-z)$ are strictly parallel, with an interplanar spacing of 3.426 (2) Å: the corresponding ring-centroid separation is 3.751 (2) Å, and the ring offset is 1.527 (2) Å. Similar C—H···O and π - π interactions were identified in the structure of the analogous phen complex $[V(acac)_2(phen)]ClO_4$ (Kavitha *et al.*, 2006). Otherwise, the bond lengths and angles of (I) present no unusual features, and they are very similar to those in the analogous phen complex.

Experimental

A solution of tris(pentane-2,4-dionato)vanadium(III) (0.30 g) and 2,2'-bipyridinium perchlorate (0.22 g) in methanol (30 ml) was heated under reflux for 3 h under an atmosphere of dinitrogen. The mixture was cooled to yield an orange solid which was crystallized by vapour diffusion of light petroleum into a solution in dichloromethane (m.p. 480 K).

Crystal data

$[V(C_5H_7O_2)_2(C_{10}H_8N_2)]ClO_4 \cdot CH_2Cl_2$
 $M_r = 589.71$
 Monoclinic, $P2_1/c$
 $a = 15.0676$ (5) Å
 $b = 12.5534$ (3) Å
 $c = 14.5533$ (5) Å
 $\beta = 111.4860$ (13)°
 $V = 2561.45$ (14) Å³
 $Z = 4$

$D_x = 1.529$ Mg m⁻³
 Mo $K\alpha$ radiation
 Cell parameters from 5869 reflections
 $\theta = 3.2$ – 27.5 °
 $\mu = 0.75$ mm⁻¹
 $T = 120$ (2) K
 Plate, orange
 $0.50 \times 0.20 \times 0.06$ mm

Data collection

Bruker–Nonius KappaCCD diffractometer
 φ and ω scans
 Absorption correction: multi-scan (SADABS; Sheldrick, 2003)
 $T_{min} = 0.706$, $T_{max} = 0.956$
 37694 measured reflections

5869 independent reflections
 4242 reflections with $I > 2\sigma(I)$
 $R_{int} = 0.058$
 $\theta_{max} = 27.5$ °
 $h = -19 \rightarrow 19$
 $k = -16 \rightarrow 16$
 $l = -18 \rightarrow 18$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.043$
 $wR(F^2) = 0.105$
 $S = 1.07$
 5869 reflections
 320 parameters
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0354P)^2 + 2.8417P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{max} = 0.001$
 $\Delta\rho_{max} = 0.36$ e Å⁻³
 $\Delta\rho_{min} = -0.48$ e Å⁻³

Table 1

Selected bond lengths (Å).

V1—O33	1.9487 (17)	V1—O31	1.9779 (17)
V1—O43	1.9526 (17)	V1—N11	2.116 (2)
V1—O41	1.9694 (16)	V1—N21	2.125 (2)

Table 2

Hydrogen-bond geometry (Å, °).

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
C16—H16···O4 ⁱ	0.95	2.51	3.349 (4)	147
C25—H25···O1	0.95	2.41	3.285 (4)	153
C26—H26···O2	0.95	2.47	3.249 (3)	139
C51—H51A···O43	0.99	2.53	3.429 (4)	150
C51—H51B···O2	0.99	2.59	3.404 (4)	140
C51—H51B···O3	0.99	2.44	3.397 (4)	161

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

All H atoms were located in a difference map and then treated as riding atoms, with C—H = 0.95 (ring H), 0.98 (methyl H) or 0.99 Å (CH₂), and with $U_{iso}(H) = 1.2U_{eq}(C)$ or $1.5U_{eq}(methyl C)$.

Data collection: COLLECT (Hooft, 1999); cell refinement: DENZO (Otwinowski & Minor, 1997) and COLLECT; data reduc-

tion: *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, England. The authors thank the staff for all their help and advice.

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