

Chloro[*N,N'*-dimethyl-*N,N'*-bis(2-pyridylmethyl)-ethane-1,2-diamine]oxovanadium(IV) perchlorateRune Kirk Egdal, Andrew D.  
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## Key indicators

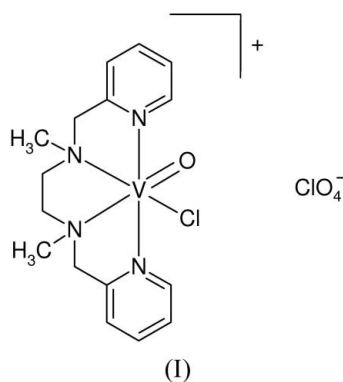
Single-crystal X-ray study  
 $T = 180$  K  
Mean  $\sigma(\text{C}-\text{C}) = 0.004$  Å  
 $R$  factor = 0.040  
 $wR$  factor = 0.102  
Data-to-parameter ratio = 16.7For details of how these key indicators were  
automatically derived from the article, see  
<http://journals.iucr.org/e>.

The title vanadium complex,  $[\text{VClO}(\text{C}_{16}\text{H}_{22}\text{N}_4)]\text{ClO}_4$ , of the tetradentate ligand *N,N'*-dimethyl-*N,N'*-bis(2-pyridylmethyl)-ethane-1,2-diamine was obtained from air oxidation of a mixture of the ligand and  $\text{VCl}_3$  in wet methanol.

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## Comment

The V atom of the title compound, (I) (Fig. 1), is coordinated by the tetradentate ligand *N,N'*-dimethyl-*N,N'*-bis(2-pyridylmethyl)ethane-1,2-diamine, with the two pyridine groups located *trans* to each other and forming a dihedral angle of  $66.5(1)^\circ$  between their least-squares planes. This conformation is comparable to that observed for other mononuclear transition-element complexes of the same ligand [see, for example, Raffard *et al.* (2002) and Li *et al.* (1991)]. The distorted octahedral coordination of the V atom is completed by an oxo and a chloro ligand, forming a geometry typical of that for similar  $\text{V}^{\text{IV}}$  complexes [see, for example, Brand *et al.* (1990), Otieno *et al.* (1996) and Tolis *et al.* (2001)]. The two V–N(pyridine) distances are 2.120(2) and 2.141(2) Å, whereas the V–N(amine) distances are longer and show considerable difference attributable to the *trans* influence of the vanadyl O atom; the V–N(amine) distances *cis* and *trans* to O1 are 2.1755(19) and 2.329(2) Å, respectively.



## Experimental

$\text{VCl}_3$  (48.7 mg, 0.310 mmol) and  $\text{C}_{16}\text{H}_{22}\text{N}_4 \cdot 4\text{HClO}_4$  (219.7 mg, 0.327 mmol) were added to methanol (5 ml). The suspension was heated to 434 K and 6 M NaOH (aqueous) (0.25 ml, 1.5 mmol) was added slowly to dissolve the ligand salt. The resulting blue solution was stirred at 343 K for 1 h, and blue crystals of the title complex were formed upon cooling to room temperature (yield: 104.7 mg, 0.222 mmol, 71.6%). Analysis calculated for  $\text{C}_{16}\text{H}_{22}\text{Cl}_2\text{N}_4\text{O}_5\text{V}$ : C 40.70, H 4.70, N 11.86%; found: C 40.72, H 4.63, N 11.72%.

## Crystal data

[VClO(C<sub>16</sub>H<sub>22</sub>N<sub>4</sub>)]ClO<sub>4</sub>  
*M<sub>r</sub>* = 472.22  
 Monoclinic, *P*2<sub>1</sub>/*c*  
*a* = 13.3031 (6) Å  
*b* = 8.9874 (4) Å  
*c* = 17.7551 (9) Å  
 $\beta$  = 106.408 (1)°  
*V* = 2036.35 (17) Å<sup>3</sup>  
*Z* = 4

*D<sub>x</sub>* = 1.540 Mg m<sup>-3</sup>  
 Mo *K*α radiation  
 Cell parameters from 3078 reflections  
 $\theta$  = 2.5–27.1°  
 $\mu$  = 0.79 mm<sup>-1</sup>  
*T* = 180 (2) K  
 Block, blue  
 0.34 × 0.20 × 0.20 mm

## Data collection

Bruker–Nonius X8 APEX-II CCD diffractometer  
 Thin-slice  $\omega$  and  $\varphi$  scans  
 Absorption correction: multi-scan (SADABS; Sheldrick, 2003)  
*T<sub>min</sub>* = 0.719, *T<sub>max</sub>* = 0.859  
 8836 measured reflections

4263 independent reflections  
 2835 reflections with *I* > 2σ(*I*)  
*R<sub>int</sub>* = 0.041  
 $\theta_{\max}$  = 28.3°  
*h* = −17 → 17  
*k* = −9 → 9  
*l* = −23 → 9

## Refinement

Refinement on *F*<sup>2</sup>  
*R* [*F*<sup>2</sup> > 2σ(*F*<sup>2</sup>)] = 0.040  
*wR* (*F*<sup>2</sup>) = 0.102  
*S* = 0.94  
 4263 reflections  
 255 parameters

H-atom parameters constrained  
 $w = 1/[\sigma^2(F_o^2) + (0.0546P)^2]$   
 where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\max} = 0.001$   
 $\Delta\rho_{\max} = 0.82 \text{ e \AA}^{-3}$   
 $\Delta\rho_{\min} = -0.42 \text{ e \AA}^{-3}$

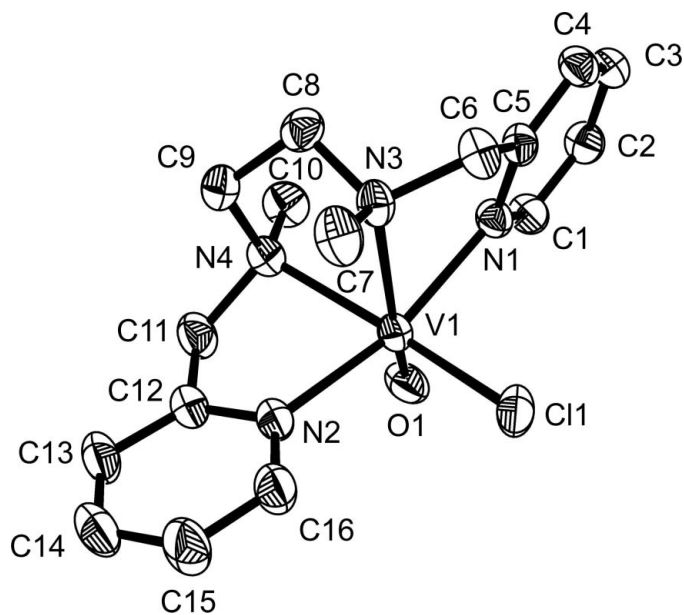


Figure 1

View of the cation of the title complex, showing displacement ellipsoids drawn at the 50% probability level. H atoms have been omitted for clarity.

Table 1

Selected geometric parameters (Å, °).

V1–O1	1.6024 (18)	V1–N4	2.1755 (19)
V1–N2	2.120 (2)	V1–Cl1	2.3184 (8)
V1–N1	2.141 (2)	V1–N3	2.329 (2)
O1–V1–N2	99.88 (8)	N1–V1–Cl1	92.22 (6)
O1–V1–N1	92.14 (8)	N4–V1–Cl1	162.45 (6)
N2–V1–N1	165.39 (8)	O1–V1–N3	160.69 (8)
O1–V1–N4	90.54 (8)	N2–V1–N3	92.70 (7)
N2–V1–N4	76.80 (8)	N1–V1–N3	73.63 (7)
N1–V1–N4	94.94 (7)	N4–V1–N3	78.06 (7)
O1–V1–Cl1	105.20 (7)	Cl1–V1–N3	88.67 (5)
N2–V1–Cl1	92.60 (6)		

H atoms bound to C atoms were positioned geometrically and allowed to ride during subsequent refinement, with C–H = 0.95 Å and  $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$  for the H atoms of the pyridyl rings, C–H = 0.99 Å and  $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$  for the methylene groups, and C–H = 0.98 Å and  $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{C})$  for the methyl groups. The methyl groups were also allowed to rotate about their local threefold axes.

Data collection: APEX2 (Bruker–Nonius, 2003); cell refinement: SAINT (Bruker, 2003); data reduction: SAINT; program(s) used to solve structure: SHELXTL (Sheldrick, 2000); program(s) used to refine structure: SHELXTL; molecular graphics: SHELXTL; software used to prepare material for publication: SHELXTL.

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