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# Dichlorooxo(*N*,*N*′,*N*″-trimethyl-1,4,7-triazacyclononane-$\kappa^3N$)vanadium(IV)

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**Key indicators**

Single-crystal X-ray study
*T* = 141 K
Mean σ(C–C) = 0.007 Å
*R* factor = 0.038
*wR* factor = 0.108
Data-to-parameter ratio = 14.8

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

In the title compound, [$VOCl_2(C_9H_{21}N_3)$], the V atom is at the center of a distorted octahedral coordination sphere and is coordinated by the three N atoms of the *N*,*N*′,*N*″-trimethyl-1,4,7-triazacyclononane ($Me_3$tacn) ligand, and by two chlorides and an oxo ligand.

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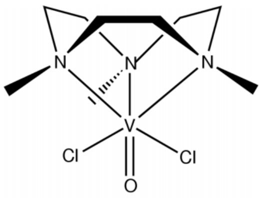
## Comment

This compound, (I), along with other mononuclear complexes of the type $MLX_3$, is used as a starting material for the preparation of binuclear oxo-bridged complexes. In turn, these dimers are used for modeling structural and magnetic properties of biologically relevant compounds (Steinkamp *et al.*, 1981). The title organometallic complex was prepared according to a previously published procedure (Köppen *et al.*, 1988). The authors reported that crystals were formed, but no X-ray analysis was presented.



(I)

The V atom has a distorted octahedral environment (Fig. 1 and Table 1) and is coordinated by the three N atoms of the $Me_3$tacn (tacn = triazacyclononane) ligand, and by two chlorides and an oxo ligand. The V—N bond length *trans* to the oxo ligand is significantly longer than the other two V—N bonds [V1—N3 = 2.349 (4) Å compared with V1—N2 = 2.189 (4) Å and V1—N1 = 2.195 (4) Å]. This is consistent with other vanadium–oxo–tacn complexes described in the literature (Köppen *et al.*, 1988; Knopp *et al.*, 1991). The angle O1—V1—N3 is 165.38 (13)°, which is similar to values observed in analogous $Ti^{IV}$ and $Mo^{IV}$ complexes of 162.54 (1) and 160.55 (6)°, respectively (Jeske *et al.*, 1994; Burger *et al.*, 1993).

## Experimental

The title compound, (I), was synthesized as reported previously (Köppen *et al.*, 1988). Crystals of (I) were prepared by slow evaporation of a dimethylformamide solution.

### *Crystal data*

[$VCl_2O(C_9H_{21}N_3)$]
$M_r = 309.13$
Monoclinic, $P2_1/c$
$a = 13.6069\ (15)$ Å
$b = 7.7759\ (8)$ Å
$c = 12.6586\ (14)$ Å
$\beta = 90.000\ (2)°$
$V = 1339.4\ (3)$ Å$^3$
$Z = 4$

$D_x = 1.533$ Mg m$^{-3}$
Mo $K\alpha$ radiation
Cell parameters from 5166 reflections
$\theta = 1.7$–$25.4°$
$\mu = 1.12$ mm$^{-1}$
$T = 141\ (2)$ K
Plate, blue
0.16 × 0.12 × 0.10 mm

*Data collection*

Siemens SMART CCD diffractometer
$\omega$ scans
Absorption correction: multi-scan (Blessing, 1995)
$T_{\min} = 0.48$, $T_{\max} = 0.92$
5156 measured reflections
2167 independent reflections
1929 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.046$
$\theta_{\max} = 25.4°$
$h = -15 \rightarrow 16$
$k = -6 \rightarrow 9$
$l = -14 \rightarrow 13$

*Refinement*

Refinement on $F^2$
$R[F^2 > 2\sigma(F^2)] = 0.038$
$wR(F^2) = 0.108$
$S = 0.72$
2167 reflections
146 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.1P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.002$
$\Delta\rho_{\max} = 0.30$ e Å$^{-3}$
$\Delta\rho_{\min} = -0.53$ e Å$^{-3}$

**Table 1**
Selected geometric parameters (Å, °).

| | | | |
|---|---|---|---|
| V1—O1 | 1.637 (3) | V1—N3 | 2.349 (4) |
| V1—N2 | 2.189 (4) | V1—Cl1 | 2.3569 (12) |
| V1—N1 | 2.195 (4) | V1—Cl2 | 2.3603 (12) |
| | | | |
| O1—V1—N2 | 91.63 (15) | N1—V1—Cl1 | 163.50 (11) |
| O1—V1—N1 | 93.34 (14) | N3—V1—Cl1 | 87.36 (9) |
| N2—V1—N1 | 80.02 (13) | O1—V1—Cl2 | 100.19 (11) |
| O1—V1—N3 | 165.38 (13) | N2—V1—Cl2 | 165.44 (11) |
| N2—V1—N3 | 76.49 (14) | N1—V1—Cl2 | 90.76 (10) |
| N1—V1—N3 | 76.41 (13) | N3—V1—Cl2 | 90.47 (10) |
| O1—V1—Cl1 | 102.02 (11) | Cl1—V1—Cl2 | 92.40 (4) |
| N2—V1—Cl1 | 93.41 (11) | | |

The crystal had a 40.9% mirror twin perpendicular to the $a$ axis (twin law = [$\bar{1}$00,010,001]). The unique angle $\beta$ therefore was indistinguishable from 90°. H atoms were included in calculated positions and constrained.

Data collection: *SMART* (Bruker, 1999); cell refinement: *SAINT* (Bruker, 1995); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997).

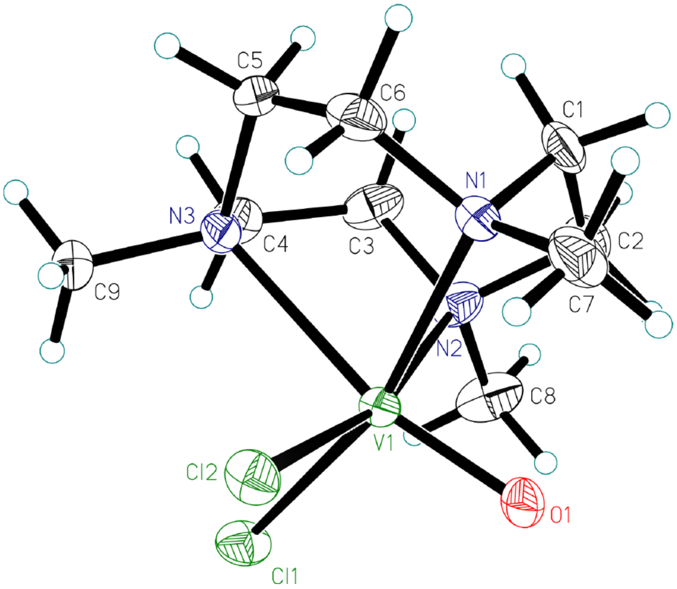


**Figure 1**
The molecular structure of (I), with the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level.

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