

Aqua[*N,N'*-ethylenebis(*o*-vanillylideneiminato- $\kappa^2N,O$ )]-oxovanadium(IV) monohydrate

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## Key indicators

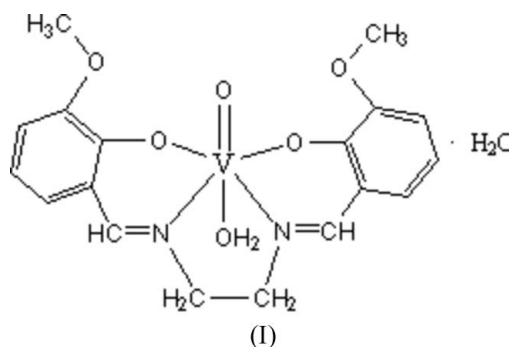
Single-crystal X-ray study  
 $T = 298\text{ K}$   
Mean  $\sigma(\text{C}-\text{C}) = 0.007\text{ \AA}$   
 $R$  factor = 0.049  
 $wR$  factor = 0.126  
Data-to-parameter ratio = 12.5For details of how these key indicators were  
automatically derived from the article, see  
<http://journals.iucr.org/e>.

Reaction of a vanadyl sulfate solution with a tetradentate Schiff base ligand derived from the condensation of *o*-vanillin and ethylenediamine in ethanol leads to the title compound, aqua{6,6'-dimethoxy-2,2'-[1,2-ethanediylbis(nitrilomethylidene)]diphenolato- $\kappa^2N,O$ }oxovanadium(IV) monohydrate,  $[\text{V}(\text{C}_{18}\text{H}_{18}\text{N}_2\text{O}_4)\text{O}(\text{H}_2\text{O})]\cdot\text{H}_2\text{O}$ , in which the  $\text{V}^{\text{IV}}$  atom is six-coordinated by two N atoms and four O atoms in a distorted octahedral configuration. In the crystal structure, molecules are linked through  $\text{O}-\text{H}\cdots\text{O}$  hydrogen bonding [ $\text{O}\cdots\text{O} = 2.788(4)-2.969(4)\text{ \AA}$ ] to form a two-dimensional network.

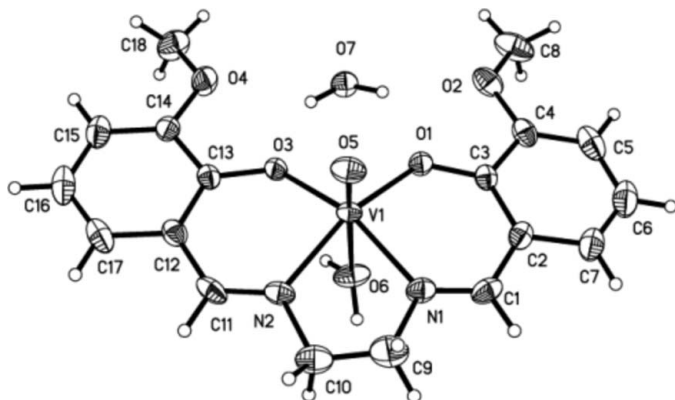
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## Comment

Vanadium is found naturally in soil and water as a trace metal. Its coordination chemistry has received increasing attention due to the fact that vanadium compounds in various oxidation states have insulin-mimetic properties (Diego *et al.*, 2003). Efforts have been made to design and synthesize all kinds of vanadium complexes in place of insulin (Wikksky *et al.*, 2001; Thompson *et al.*, 1999). We report here the synthesis and crystal structure of a new oxovanadium(IV) complex, (I), with a tetradentate Schiff base ligand derived from the condensation of *o*-vanillin and ethylenediamine.



The molecular structure of (I) is shown in Fig. 1. Selected bond angles and distances are given in Table 1. This complex has a distorted octahedral  $\text{VO}^{2+}$  species in which the terminal oxo (O5) and the coordinated water (O6) atoms occupy the axial positions with a *trans* angle of  $176.60(13)^\circ$ . The tetradentate ligand derived from the condensation of *o*-vanillin and ethylenediamine forms the equatorial plane of the molecule. The  $\text{V}^{\text{IV}}$  center is displaced by  $0.2851(12)\text{ \AA}$  from the equatorial plane towards oxo atom O5. The complex forms two six-membered rings (N1/C1–C3/O1/V1 and N2/C11–C13/O3/V1) and a five-membered ring (N1/C9/C10/N2/V1), which increase the stability of the complex. The two six-membered rings subtend different dihedral angles with the five-membered ring [ $14.73(19)$  and  $7.33(23)\text{ \AA}$ , respectively], possibly because of



**Figure 1**  
The structure of the title compound, showing 30% probability displacement ellipsoids and the atom-numbering scheme.

steric effects in the equatorial plane. V1–O5 is a typical V=O double bond with a distance of 1.590 (3) Å, close to that reported previously for VO(salen) {salen is 2,2'-[1,2-ethanediylbis(nitrilomethylidene)]diphenolate} (V=O = 1.588 Å; Riley *et al.*, 1986). The O6 water molecule is weakly coordinated due to the strong *trans* influence of V=O and the steric hindrance of the equatorial coordination positions [V1–O6 = 2.389 (3) Å].

In addition to the vanadium complex, the asymmetric unit contains one molecule of water. Both molecules are involved in O–H···O hydrogen bonding, which links the molecules in a two-dimensional network (Table 2 and Fig. 2).

## Experimental

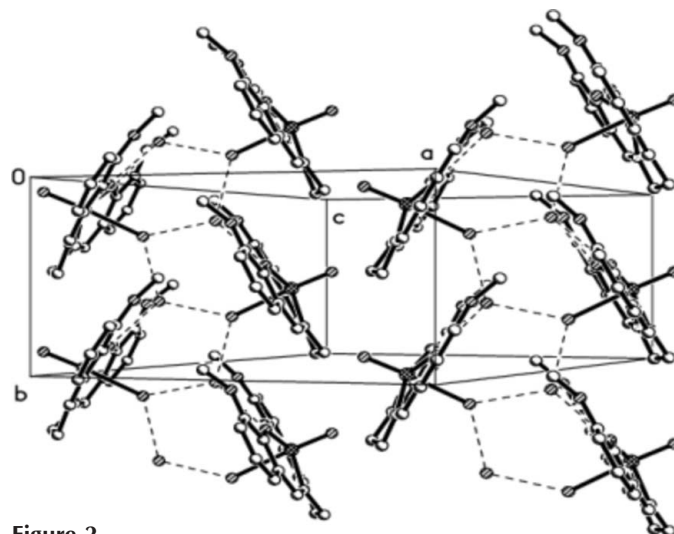
o-Vanillin (2 mmol, 304.4 mg) was dissolved in hot ethanol (10 ml) and added slowly to ethylenediamine (1 mmol, 0.06 ml). A yellow flocculent precipitate appeared immediately. After stirring for 1 h, sodium acetate (2.2 mmol, 0.3 g) was added and the precipitate dissolved after 10 min of stirring. A solution of vanadyl sulfate hydrate (1 mmol, 235 mg) in 4 ml water was added dropwise to the mixture, with stirring, and further refluxed for 4 h. The mixture was then cooled to room temperature and a brown powder was precipitated. It was collected by filtration and washed with ethanol. The precipitate was dissolved in acetonitrile, yielding yellow crystals suitable for X-ray diffraction after two days of slow evaporation.

### Crystal data

|   |   |
|---|---|
| [V(C <sub>18</sub> H <sub>18</sub> N <sub>2</sub> O <sub>4</sub> )O(H <sub>2</sub> O)]·H <sub>2</sub> O | $D_x = 1.511 \text{ Mg m}^{-3}$           |
| $M_r = 429.32$  | Mo K $\alpha$ radiation                   |
| Monoclinic, $P2_1/n$  | Cell parameters from 1765 reflections     |
| $a = 12.613 (7) \text{ \AA}$  | $\theta = 3.2\text{--}23.6^\circ$         |
| $b = 6.701 (4) \text{ \AA}$   | $\mu = 0.57 \text{ mm}^{-1}$              |
| $c = 22.383 (12) \text{ \AA}$   | $T = 298 (2) \text{ K}$                   |
| $\beta = 94.086 (10)^\circ$   | Block, brown                              |
| $V = 1886.9 (18) \text{ \AA}^3$   | $0.39 \times 0.27 \times 0.06 \text{ mm}$ |
| $Z = 4$   |   |

### Data collection

|   |  |
|---|--|
| Bruker SMART CCD area-detector diffractometer               | 3311 independent reflections           |
| $\varphi$ and $\omega$ scans                                | 1977 reflections with $I > 2\sigma(I)$ |
| Absorption correction: multi-scan (SADABS; Sheldrick, 1996) | $R_{\text{int}} = 0.050$               |
| $T_{\text{min}} = 0.808$ , $T_{\text{max}} = 0.967$         | $\theta_{\text{max}} = 25.0^\circ$     |
| 9439 measured reflections                                   | $h = -11 \rightarrow 15$               |
|   | $k = -7 \rightarrow 7$                 |
|   | $l = -26 \rightarrow 24$               |



**Figure 2**  
Packing diagram of the title compound, with the hydrogen-bonding interactions shown as dashed lines.

### Refinement

Refinement on  $F^2$   
 $R[F^2 > 2\sigma(F^2)] = 0.049$   
 $wR(F^2) = 0.126$   
 $S = 1.01$   
 3311 reflections  
 265 parameters  
 H atoms treated by a mixture of independent and constrained refinement

$$w = 1/[\sigma^2(F_o^2) + (0.0499P)^2 + 0.8719P]$$

$$\text{where } P = (F_o^2 + 2F_c^2)/3$$

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

$$\Delta\rho_{\text{max}} = 0.38 \text{ e \AA}^{-3}$$

$$\Delta\rho_{\text{min}} = -0.36 \text{ e \AA}^{-3}$$

**Table 1**

Selected geometric parameters (Å, °).

|          |             |          |             |
|----------|-------------|----------|-------------|
| V1–O5    | 1.590 (3)   | V1–N1    | 2.041 (3)   |
| V1–O3    | 1.944 (3)   | V1–N2    | 2.060 (3)   |
| V1–O1    | 1.952 (3)   | V1–O6    | 2.389 (3)   |
| O5–V1–O3 | 103.30 (14) | O1–V1–N2 | 159.48 (12) |
| O5–V1–O1 | 101.88 (13) | N1–V1–N2 | 80.08 (15)  |
| O3–V1–O1 | 93.49 (11)  | O5–V1–O6 | 176.60 (13) |
| O5–V1–N1 | 98.14 (14)  | O3–V1–O6 | 79.38 (11)  |
| O3–V1–N1 | 157.05 (12) | O1–V1–O6 | 79.93 (11)  |
| O1–V1–N1 | 90.00 (13)  | N1–V1–O6 | 78.92 (12)  |
| O5–V1–N2 | 97.31 (14)  | N2–V1–O6 | 80.55 (12)  |
| O3–V1–N2 | 89.08 (13)  |          |             |

**Table 2**

Hydrogen-bond geometry (Å, °).

| $D\text{--}H\cdots A$     | $D\text{--}H$ | $H\cdots A$ | $D\cdots A$ | $D\text{--}H\cdots A$ |
|---------------------------|---------------|-------------|-------------|-----------------------|
| O6–H19···O7 <sup>i</sup>  | 0.90 (1)      | 2.07 (2)    | 2.915 (4)   | 156 (4)               |
| O6–H20···O7 <sup>ii</sup> | 0.90 (1)      | 1.89 (1)    | 2.788 (4)   | 173 (4)               |
| O7–H21···O3               | 0.89 (1)      | 2.09 (2)    | 2.889 (4)   | 148 (3)               |
| O7–H21···O4               | 0.89 (1)      | 2.21 (3)    | 2.931 (4)   | 138 (4)               |
| O7–H22···O2               | 0.90 (1)      | 2.16 (2)    | 2.969 (4)   | 151 (3)               |
| O7–H22···O1               | 0.90 (1)      | 2.20 (3)    | 2.919 (4)   | 136 (3)               |

Symmetry codes: (i)  $-x + \frac{1}{2}, y - \frac{1}{2}, -z + \frac{1}{2}$ ; (ii)  $x, y - 1, z$ .

Water H atoms were located in difference Fourier maps and during the refinement the O–H distances were restrained to 0.90 (1) Å,

with  $U_{\text{iso}} = 0.08 \text{ \AA}^2$ . All other H atoms were positioned geometrically (C–H = 0.93–0.97 Å) and allowed to ride on their respective parent atoms, with  $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$  or  $1.5U_{\text{eq}}(\text{methyl C})$ .

Data collection: *SMART* (Siemens, 1996); cell refinement: *SMART*; data reduction: *SAINT* (Siemens, 1996); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 2000); software used to prepare material for publication: *SHELXTL*.

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