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Key indicators

Single-crystal X-ray study  
 T = 273 K  
 Mean  $\sigma(\text{C}-\text{C}) = 0.007 \text{ \AA}$   
 R factor = 0.036  
 wR factor = 0.100  
 Data-to-parameter ratio = 13.6

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

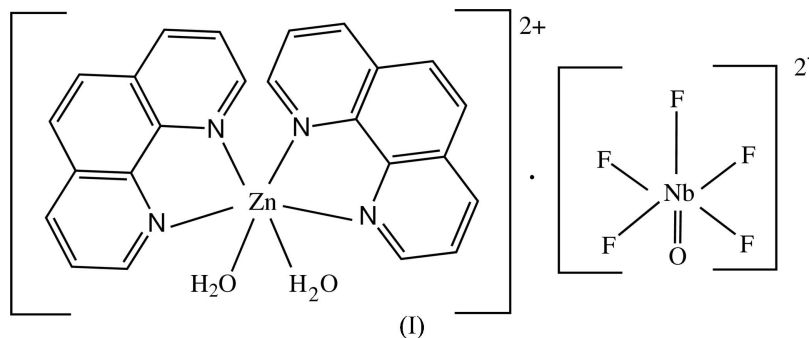
Diaquabis(1,10-phenanthroline- $\kappa^2N,N'$ )zinc(II) pentafluorooxoniate(V)

The crystal structure of the title compound,  $[\text{Zn}(\text{C}_{12}\text{H}_8\text{N}_2)_2(\text{H}_2\text{O})_2][\text{NbF}_5\text{O}]$ , consists of  $\text{Zn}^{\text{II}}$  complex cations and  $\text{Nb}^{\text{V}}$  complex anions. The  $\text{Zn}^{\text{II}}$  and  $\text{Nb}^{\text{V}}$  atoms each have a distorted octahedral coordination geometry. The Nb—F bond in the *trans* position with respect to the Nb=O bond is significantly longer than the other four Nb—F bonds in the same  $[\text{NbOF}_5]^{2-}$  anion.

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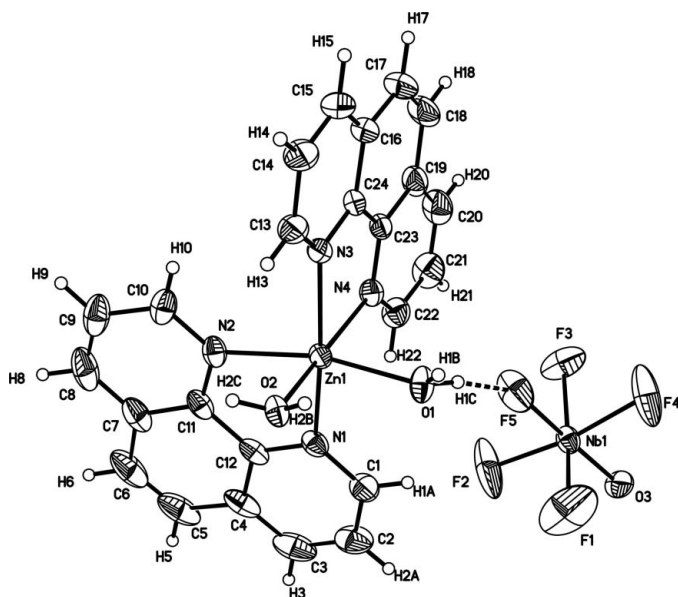
Comment

Out-of-centre 'primary' electronic distortions are inherent in the oxide fluoride anions of the early  $d^0$  transition metals. In the  $[\text{NbOF}_5]^{2-}$  anion, the  $\text{Nb}^{\text{V}}$  atom deviates from the centre of the octahedron toward the oxo ligand to form a short Nb=O bond, with a long Nb—F bond in the *trans* position. The *trans*-directing property of the  $[\text{NbOF}_5]^{2-}$  anion can be used in the design of new materials with specific structure-related properties (Heier *et al.*, 1998). We present here the crystal structure of the title compound, (I), which incorporates the  $[\text{NbOF}_5]^{2-}$  anion.



The crystal structure of (I) consists of  $\text{Zn}^{\text{II}}$  complex cations and  $\text{Nb}^{\text{V}}$  complex anions (Fig. 1). The  $\text{Zn}^{\text{II}}$  atom has an octahedral coordination geometry formed by two 1,10-phenanthroline (phen) ligands and two water molecules. The  $\text{Nb}^{\text{V}}$  atom assumes a distorted octahedral coordination geometry formed by five F atoms and one O atom. The Nb—F bond in the position *trans* to the Nb=O bond is significantly longer than the other four Nb—F bonds in the same anion (Table 1). This agrees with the situation found in the reported structures  $[4\text{-apyH}]_2[\text{Cu}(4\text{-apy})_4(\text{NbOF}_5)_2]$  (4-apy = 4-aminopyridine),  $\text{Cd}(3\text{-apy})_4\text{NbOF}_5$  (3-apy = 3-aminopyridine) and  $\text{Cu}(3\text{-apy})_4\text{NbOF}_5$  (Izumi *et al.*, 2005),  $[\text{pyH}^+]_2[\text{CuNb}_2(\text{py})_4\text{O}_2\text{F}_{10}]^{2-}$  and  $\text{CuNb}(\text{py})_4\text{OF}_5$  (Halasyamani *et al.*, 1996), and  $\text{CdNb}(\text{py})_4\text{OF}_5$  (Halasyamani *et al.*, 1998).

The coordinated water molecules of the  $\text{Zn}^{\text{II}}$  complex cation are hydrogen bonded with the  $[\text{NbOF}_5]^{2-}$  anions. The



**Figure 1**

The molecular structure of (I), shown with 30% probability displacement ellipsoids (arbitrary spheres for H atoms). The dashed line indicates a hydrogen bond.

phen ligands of the Zn<sup>II</sup> complex are also linked to the [NbOF<sub>5</sub>]<sup>2-</sup> anions by C—H···F hydrogen bonding (Table 2).

## Experimental

All reagents used were of analytical grade from commercial sources and were used without further purification. Nb<sub>2</sub>O<sub>5</sub> (0.133 g, 0.5 mmol) was first dissolved in HF solution (1 ml, 42 wt% in H<sub>2</sub>O) at 393 K for 2 h in a Teflon-lined stainless steel vessel. After being cooled to room temperature, ZnSO<sub>4</sub>·7H<sub>2</sub>O (0.288 g, 1 mmol), phen (0.396 g, 2 mmol) and H<sub>2</sub>O (15 ml) were added to the above solution. The pH of the mixture was adjusted to about 6 using NaOH solution. The mixture was then heated under autogenous hydrothermal conditions at 413 K for 4 d. After cooling to room temperature, the mixture was filtered. Colourless single crystals of (I) were obtained from the filtrate after several days.

### Crystal data

[Zn(C <sub>12</sub> H <sub>8</sub> N <sub>2</sub> ) <sub>2</sub> (H <sub>2</sub> O) <sub>2</sub> ][NbF <sub>5</sub> O]	Z = 2
<i>M<sub>r</sub></i> = 665.72	<i>D<sub>x</sub></i> = 1.762 Mg m <sup>-3</sup>
Triclinic, <i>P</i> $\bar{1}$	Mo K $\alpha$ radiation
<i>a</i> = 9.4833 (2) Å	Cell parameters from 2965 reflections
<i>b</i> = 11.1136 (3) Å	$\theta$ = 2.9–25.7°
<i>c</i> = 13.0749 (3) Å	$\mu$ = 1.48 mm <sup>-1</sup>
$\alpha$ = 73.486 (2)°	<i>T</i> = 273 (2) K
$\beta$ = 72.134 (2)°	Block, colourless
$\gamma$ = 81.704 (2)°	0.12 × 0.10 × 0.08 mm
<i>V</i> = 1254.99 (5) Å <sup>3</sup>	

### Data collection

Bruker SMART CCD area-detector diffractometer	4843 independent reflections
$\varphi$ and $\omega$ scans	3773 reflections with <i>I</i> > 2 $\sigma$ ( <i>I</i> )
Absorption correction: multi-scan (SADABS; Sheldrick, 2002)	<i>R</i> <sub>int</sub> = 0.020
<i>T</i> <sub>min</sub> = 0.806, <i>T</i> <sub>max</sub> = 0.875	$\theta$ <sub>max</sub> = 26.0°
9091 measured reflections	<i>h</i> = -11 → 11
	<i>k</i> = -11 → 13
	<i>l</i> = -16 → 16

### Refinement

Refinement on *F*<sup>2</sup>  
*R* [*F*<sup>2</sup> > 2 $\sigma$ (*F*<sup>2</sup>)] = 0.036  
*wR*(*F*<sup>2</sup>) = 0.100  
*S* = 1.08  
 4843 reflections  
 355 parameters  
 H atoms treated by a mixture of independent and constrained refinement

$$w = 1/[\sigma^2(F_o^2) + (0.0527P)^2 + 0.3561P]$$

where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\max} < 0.001$   
 $\Delta\rho_{\max} = 0.69 \text{ e } \text{\AA}^{-3}$   
 $\Delta\rho_{\min} = -0.65 \text{ e } \text{\AA}^{-3}$

**Table 1**

Selected bond lengths (Å).

Nb1—O3	1.790 (2)	Zn1—O1	2.094 (3)
Nb1—F2	1.860 (3)	Zn1—O2	2.095 (3)
Nb1—F1	1.879 (3)	Zn1—N1	2.144 (3)
Nb1—F3	1.897 (3)	Zn1—N2	2.174 (3)
Nb1—F4	1.940 (3)	Zn1—N3	2.149 (3)
Nb1—F5	2.012 (2)	Zn1—N4	2.188 (3)

**Table 2**

Hydrogen-bond geometry (Å, °).

<i>D</i> —H··· <i>A</i>	<i>D</i> —H	H··· <i>A</i>	<i>D</i> ··· <i>A</i>	<i>D</i> —H··· <i>A</i>
O1—H1B···F5 <sup>i</sup>	0.83 (3)	2.35 (3)	3.090 (4)	150 (5)
O1—H1C···F5	0.86 (3)	1.71 (3)	2.558 (3)	167 (5)
O2—H2B···F4 <sup>i</sup>	0.84 (2)	1.76 (2)	2.595 (4)	169 (5)
O2—H2C···O3 <sup>ii</sup>	0.84 (3)	1.81 (3)	2.646 (3)	175 (4)
C17—H17···F5 <sup>iii</sup>	0.93	2.37	3.277 (6)	166
C18—H18···F1 <sup>iv</sup>	0.93	2.37	3.225 (7)	152

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

The water H atoms were located in a difference Fourier map and refined with a restrained O—H bond length of 0.85 (2) Å and fixed isotropic displacement parameters of 0.08 Å<sup>2</sup>. Other H atoms were placed at calculated positions (C—H = 0.93 Å) and refined in riding mode [*U*<sub>iso</sub>(H) = 1.2*U*<sub>eq</sub>(C)].

Data collection: *SMART* (Bruker, 2002); cell refinement: *SAINT* (Bruker, 2002); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1990); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Siemens, 1995); software used to prepare material for publication: *SHELXTL*.

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