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Research Article

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Study on a structure of 2,6-diaminopyridine vanadate hydrate, $C_{20}H_{38}N_{12}O_{15}V_4$

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ABSTRACT

A novel 2,6-diaminopyridine vanadate hydrate $C_{20}H_{38}N_{12}O_{15}V_4$ has been synthesized from hydrothermal reaction and the crystal structure has been determined by means of single-crystal X-ray diffraction. Triclinic, P-1. a=7.9516(6) Å, b =10.2810(10) Å, c =11.7780(11) Å, α= 74.590(3)°, β=72.500(3)°, γ=89.151(5)°. V=883.06(14). Z=1. R_1 =0.0424, wR_2 =0.1378, T=291(2) K. The V atom is four coordinated by four O atoms. The crystal packing is stabilized by O-H...O and O-H...N hydrogen bonding interactions.

Key words: 2,6-diaminopyridine, vanadate, structure analysis, hydrogen bond

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INTRODUCTION

Schiff bases have many applications in food industry, dye industry, analytical chemistry, catalysis, fungicidal, agrochemical, and biological activities. Schiff bases exhibit a broad spectrum of pharmacological and biological properties, such as analgesics, anticancer agents, anti-inflammatory, etc., which may be due to the azomethine linkage [1]. Schiff bases show the ability to complex with many metal ions and such complexes play an important role in coordination chemistry, enzyme reactions, and molecular architectures [2-4]. Metal complexes play an essential role in agriculture, pharmaceutical, and industrial chemistry [5]. Many drugs possess modified toxicological and pharmacological properties when administered as metal complexes [6-8]. Aminopyridines is widely used as the ligands with the 1,3-*N*,*N*-coordination and effectively bound d-metal cations. The metal-oxo-clusters are always high symmetry with combine of VOn units which assemble into either discrete molecular clusters or link together to form supramolecular frameworks. The decavanadate polyanions incorporate with the intercalated metal/organic fragments, displaying charming variety of architectures and topologies [9]. In this paper, the novel 2,6-diaminopyridine vanadate hydrate is reported.

EXPERIMENTAL SECTION

All commercially obtained reagent-grade chemicals were used without further purification. A mixture of $MgCl_2$ (0.1mmol, 0.01g), 2,6'-diaminopyridine (0.1mmol, 0.011g), NH_4VO_3 (0.1 mmol, 0.01g), alanine (0.1mmol, 0.009g) and H_3BO_3 (0.2mmol, 0.012g) were added into 10 mL water with 10% ethanol and heated for 6h at 404K. The solution was obtained by filtration after cooling the reaction to room temperature. Colorless block single crystals suitable for X-ray measurements were obtained after a few weeks.

The crystal data was collected on a Bruker smart CCD Area Detector.

A colorless block single crystal with dimensions of 0.35 x 0.30 x 0.00 mm was selected for measurement. Diffraction data of the single crystal were collected by ψ-ω scan mode using a graphite-monochromatic Mo *K*α rediation (λ=0.71073 Å) at 293(2) K on a Bruker Smart Apex CCD diffractometer.

RESULTS AND DISCUSSION

The title crystal structure (Fig. 1) is built up of 2,6-diaminopyridine cation, vanadate anion and water molecule. The crystal data and structure refinement is shown in Table 1. The V1 atom is four coordinated by O1, O2, O3 and O4 atoms. The V2 atom is four coordinated by O1, O2, O5 and O6 atoms. The distance d (V-O) are in the range of 1.620-1.818 Å. The angles of O-V-O are in the range of 107.76-111.82°. The torsion angles of V1-O1-V2-O2#, O3-V1-O1-V2, O4-V1-O2-V2# are 93.46, 78.7, 67.0°, respectively. Selected bond lengths and bond angles are shown in Table 2.

The crystal packing is stabilized by O-H...O and O-H...N hydrogen bonding interaction.

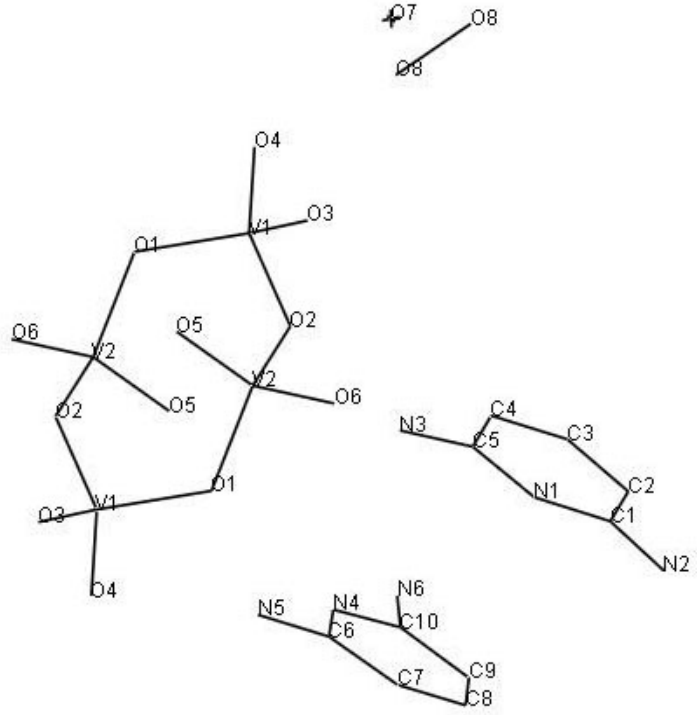

Fig.1 The molecular structure of C20H38N12015V4

Table 1. Crystal data and structure refinement for the title complex

Empirical formula	$C_{20}H_{38}N_{12}O_{15}V_4$
Formula weight	890.38
Temperature	293(2) K
Wavelength	0.71073 Å
Crystal system, space group	Triclinic, P-1
Unit cell dimensions	a = 7.9516(6) Å alpha = 74.590(3)°.
	b = 10.2810(10) Å beta = 72.500(3)°.
	c = 11.7780(11) Å gamma = 89.151(5)°.
Volume	883.06(14) $Å^3$
Z, Calculated density	1, 1.674 Mg/m^3
Absorption coefficient	1.107 mm^{-1}
F(000)	454
Crystal size	0.35 x 0.30 x 0.00 mm
Theta range for data collection	3.25 to 25.02°.
Limiting indices	-9<=h<=9, -12<=k<=12, -14<=l<=14
Reflections collected / unique	6657 / 3071 [R(int) = 0.0352]
Completeness to theta = 25.02	98.5 %
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	1.0000 and 0.6979

Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	3071 / 0 / 235
Goodness-of-fit on F^2	1.113
Final R indices [I>2sigma(I)]	R1 = 0.0424, wR2 = 0.1192
R indices (all data)	R1 = 0.0533, wR2 = 0.1378
Largest diff. peak and hole	0.691 and -0.278 e.$Å^{-3}$

Table 2. Select bond lengths [Å] and angles [°] for the title complex

V(1)-O(4)	1.620(3)
V(1)-O(3)	1.640(3)
V(1)-O(2)	1.799(3)
V(1)-O(1)	1.818(2)
V(2)-O(5)	1.623(3)
V(2)-O(6)	1.638(2)
V(2)-O(2)#1	1.799(3)
V(2)-O(1)	1.814(3)
N(1)-C(1)	1.353(5)
N(1)-C(5)	1.361(4)
N(1)-H(1)	0.8600
N(2)-C(1)	1.339(5)
N(2)-H(2B)	0.8600
N(3)-C(5)	1.325(5)
N(3)-H(3A)	0.8600
N(4)-C(10)	1.349(5)
N(4)-C(6)	1.366(5)
N(4)-H(4)	0.8600
N(5)-C(6)	1.334(5)
N(5)-H(5A)	0.8600
N(5)-H(5B)	0.8600
N(6)-C(10)	1.332(5)
N(6)-H(6A)	0.8600
O(2)-V(2)#1	1.799(3)
O(7)-H(7C)	0.8501
O(7)-H(7D)	0.8500
O(8)-O(8)#2	1.46(2)
O(8)-H(8C)	0.8500
C(1)-C(2)	1.388(5)
C(2)-C(3)	1.373(6)
C(2)-H(2)	0.9300
C(3)-C(4)	1.361(6)
C(3)-H(3)	0.9300
C(4)-C(5)	1.392(5)
C(4)-H(4A)	0.9300
C(6)-C(7)	1.379(5)
C(7)-C(8)	1.371(6)
C(8)-C(9)	1.370(6)
C(9)-C(10)	1.395(6)
C(9)-H(9)	0.9300
O(4)-V(1)-O(3)	110.69(16)
O(4)-V(1)-O(2)	109.93(13)
O(3)-V(1)-O(2)	108.51(14)
O(4)-V(1)-O(1)	107.24(13)
O(3)-V(1)-O(1)	111.82(13)
O(2)-V(1)-O(1)	108.62(12)
O(5)-V(2)-O(6)	109.49(14)
O(5)-V(2)-O(2)#1	110.69(14)
O(6)-V(2)-O(2)#1	109.91(13)
O(5)-V(2)-O(1)	110.18(14)
O(6)-V(2)-O(1)	107.76(13)
O(2)#1-V(2)-O(1)	108.76(12)
C(1)-N(1)-C(5)	123.9(3)
C(1)-N(1)-H(1)	118.0
C(5)-N(1)-H(1)	118.0
C(1)-N(2)-H(2A)	120.0
C(1)-N(2)-H(2B)	120.0
H(2A)-N(2)-H(2B)	120.0
C(5)-N(3)-H(3A)	120.0
C(5)-N(3)-H(3B)	120.0
H(3A)-N(3)-H(3B)	120.0

C(10)-N(4)-C(6)	124.3(3)
C(10)-N(4)-H(4)	117.8
C(6)-N(4)-H(4)	117.8
C(6)-N(5)-H(5A)	120.0
C(6)-N(5)-H(5B)	120.0
H(5A)-N(5)-H(5B)	120.0
C(10)-N(6)-H(6A)	120.0
C(10)-N(6)-H(6B)	120.0
H(6A)-N(6)-H(6B)	120.0
V(2)-O(1)-V(1)	122.57(14)
V(2)#1-O(2)-V(1)	133.54(14)
H(7C)-O(7)-H(7D)	107.9
H(7C)-O(7)-H(7B)	109.3
H(7D)-O(7)-H(7B)	109.3
O(8)#2-O(8)-H(8C)	139.3
O(8)#2-O(8)-H(8D)	90.2
H(8C)-O(8)-H(8D)	108.6
N(2)-C(1)-N(1)	116.3(3)
N(2)-C(1)-C(2)	125.0(4)
N(1)-C(1)-C(2)	118.7(4)
C(3)-C(2)-C(1)	118.2(4)
C(3)-C(2)-H(2)	120.9
C(1)-C(2)-H(2)	120.9
C(4)-C(3)-C(2)	122.5(4)
C(4)-C(3)-H(3)	118.7
C(2)-C(3)-H(3)	118.7
C(3)-C(4)-C(5)	119.2(4)
C(3)-C(4)-H(4A)	120.4
C(5)-C(4)-H(4A)	120.4
N(3)-C(5)-N(1)	117.6(3)
N(3)-C(5)-C(4)	124.9(3)
N(1)-C(5)-C(4)	117.5(3)
N(5)-C(6)-N(4)	117.7(3)
N(5)-C(6)-C(7)	124.2(4)
N(4)-C(6)-C(7)	118.1(4)
C(8)-C(7)-C(6)	118.6(4)
C(8)-C(7)-H(7)	120.7
C(6)-C(7)-H(7)	120.7
C(9)-C(8)-C(7)	122.5(4)
C(9)-C(8)-H(8)	118.7
C(7)-C(8)-H(8)	118.7
C(8)-C(9)-C(10)	118.7(4)
C(8)-C(9)-H(9)	120.6
C(10)-C(9)-H(9)	120.6
N(6)-C(10)-N(4)	118.2(4)
N(6)-C(10)-C(9)	124.1(4)
N(4)-C(10)-C(9)	117.6(4)

CONCLUSION

A novel 2,6-diaminopyridine vanadate hydrate $C_{20}H_{38}N_{12}O_{15}V_4$ has been synthesized from hydrothermal reaction and the crystal structure has been determined by means of single-crystal X-ray diffraction.

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