

Syntheses, Structures and Characterizations of Two New Vanadium(V) Complexes: $[2\text{-MePyH}][\text{V}^{\text{V}}\text{O}_2(\text{C}_{14}\text{H}_9\text{N}_2\text{O}_3\text{Br})]$ and $[2\text{-EtPyH}][\text{V}^{\text{V}}\text{O}_2(\text{C}_{14}\text{H}_9\text{N}_2\text{O}_3\text{Br})]$

ZHANG, Xiu-Tang(张修堂) ZHANG, Quan-Zheng(张全争) YANG, Wen-Bin(杨文斌)

WU, Ding-Ming(吴鼎铭) LU, Can-Zhong*(卢灿忠)

State Key Laboratory of Structural Chemistry, Fujian Institute of Research on the Structure of Matter, Chinese Academy of Sciences, Fuzhou, Fujian 350002, China

Two new oxovanadium(V) complexes, $[2\text{-MePyH}][\text{V}^{\text{V}}\text{O}_2(\text{L})]$ (3) and $[2\text{-EtPyH}][\text{V}^{\text{V}}\text{O}_2(\text{L})]$ (4) (salicylaldehyde 5-bromo salicyloylhydrazone is abbreviated as H_2L ; 2-MePyH is protonated 2-Me-pyridine; 2-EtPyH presents protonated 2-Et-pyridine) were obtained from a reaction of VO_2SO_4 and H_2L in acetonitrile-methanol with small quantity of 2-Me-pyridine or 2-Et-pyridine, and characterized by X-ray diffraction and spectroscopic methods. Crystal data: $[2\text{-MePyH}][\text{VO}_2(\text{L})]$ (3), $\text{C}_{20}\text{H}_{17}\text{N}_3\text{O}_5\text{BrV}$, $M_r = 510.2$, monoclinic, $P2_1/n$, $a = 0.7363(1)$ nm, $b = 0.9514(1)$ nm, $c = 2.8594(2)$ nm, $\beta = 95.305(2)^\circ$, $Z = 4$ and $V = 1.9946(3)$ nm³, $\mu(\text{Mo K}\alpha) = 2.539$ mm⁻¹; $[2\text{-EtPyH}][\text{VO}_2(\text{L})]$ (4), $\text{C}_{21}\text{H}_{19}\text{N}_3\text{O}_5\text{BrV}$, $M_r = 524.2$, triclinic, $P\bar{1}$, $a = 0.8051(1)$ nm, $b = 0.9413(1)$ nm, $c = 1.4648(2)$ nm, $\alpha = 99.1900(10)^\circ$, $\beta = 99.4530(10)^\circ$, $\gamma = 104.6670(10)^\circ$, $Z = 2$ and $V = 1.0355(2)$ nm³, $\mu(\text{Mo K}\alpha) = 2.448$ mm⁻¹. X-Ray analyses revealed that the crystal structures of 3 and 4 have similar packing modes.

Keywords salicylaldehyde 5-bromo salicyloylhydrazone, vanadium(V) complex, crystal structure

Introduction

Complexes of metal-hydrazone continue to attract considerable attention owing to their interesting biological activities and potential applications. For example, Schiff base hydrazones of pyridoxal phosphate and their analogs have been well studied in order to understand the action mechanism of Vitamin B₆-containing enzymes.¹⁻³ On the other hand, the richness and importance of vanadate chemistry have emerged for quite some time.^{4,5} One of our current interests lies in the design, isolation and characterization of novel oxovanadium(V) complexes with tridentate —O—N—O— type ligands. In order to further investigate the coordination modes of aroylhydrazone with transitional metals and to study the chemistry of its complexes, we synthesized the ligand H_2L ⁶ (no M-L complexes have been found so far), and previously reported two

vanadium complexes: $[\text{VO}(\text{L})(\text{OCH}_3)]$ (1) and $[\text{PyH}][\text{VO}_2(\text{L})]$ (2)⁷ that have not been described in detail. We report here other two new vanadium complexes: $[2\text{-MePyH}][\text{V}^{\text{V}}\text{O}_2(\text{L})]$ (3) and $[2\text{-EtPyH}][\text{V}^{\text{V}}\text{O}_2(\text{L})]$ (4). The structures and characterizations of the two oxovanadium(V) complexes 3 and 4 with the same “ONO” tridentate ligand have been described and the differences of hydrogen bonds or van der Waal's effect among these four compounds have been also discussed.

Experimental

Preparation of $[2\text{-MePyH}][\text{V}^{\text{V}}\text{O}_2(\text{L})]$ (3)

To a solution of H_2L (1 mmol) and VO_2SO_4 (1 mmol) in 20 mL of acetonitrile and 5 mL of methanol, 1 mL of 2-Me-pyridine was added. The resultant solution was refluxed in 150 °C oil bath overnight, and then filtered. The filtrate was kept at room temperature to evaporate slowly to give dark block crystals. Yield 0.39 g (77%); IR (KBr) ν : 1000 (s, V = O), 1580 (s, C = N) cm⁻¹. Anal. calcd for $\text{C}_{20}\text{H}_{17}\text{N}_3\text{O}_5\text{BrV}$: C 47.08, H 3.36, N 8.24; found C 47.03, H 3.28, N 8.35.

Preparation of $[2\text{-EtPyH}][\text{V}^{\text{V}}\text{O}_2(\text{L})]$ (4)

To a methanol solution (200 mL) of H_2L (20 mmol) and VO_2SO_4 (20 mmol), 10 mL of 2-Et-pyridine was added and then refluxed for 2 h. The solution was filtered and the filtrate was kept at ambient temperature to evaporate slowly. Red-brown crystals were obtained after one day and recrystallized in methanol for X-ray analysis. Yield 0.37 g (70%); IR (KBr) ν : 965 (s, V = O), 1590 (s, C = N) cm⁻¹. Anal. calcd for $\text{C}_{21}\text{H}_{19}\text{N}_3\text{O}_5\text{BrV}$: C 48.12, H 3.65, N

* E-mail: czlu@ms.fjirsm.ac.cn

Received June 28, 2002; revised and accepted November 9, 2002.

Project supported by Chinese Academy of Sciences, the National Natural Science Foundation of China (No. 20073048) and the Natural Science Foundation of Fujian Province (No. 2002F015).

8.02; found C 47.82, H 3.69, N 8.14.

X-Ray crystallography

The data for **3** and **4** were collected on a Siemens SMART CCD diffractometer, using graphite-monochromated MoK α radiation ($\lambda = 0.071073$ nm) at 293 K. Cell constants and orientation matrix for data collection were obtained from least-squares refinement, using the setting angles in the range of 1.43° to 25.10° for **3** and 1.44° to 25.05° for **4**. The measured reflections, independent reflections (R_{int}) and observed reflections are listed in Table 1. The structure was solved by direct methods and refined by full-matrix least-squares calculation using anisotropic thermal parameters for all non-hydrogen atoms, revealing all the positions of non-hydrogen atoms. Hydrogen atoms were located and added to the structure factor calculations as their positions were fixed during the structure refinement.

Results and discussion

The chemical draw of the ligand H₂L is shown in Fig. 1. The crystal structures of **3** and **4** are shown in Figs. 2 and 3, respectively. The selected bond lengths and angles are

listed in Table 2. Essential atomic coordinates and equivalent isotropic displacement parameters for complexes **3** and **4** are listed in Tables 3 and 4, respectively. Different from complexes **1** and **2** (square pyramid), the coordination geometry around the V(V) in both **3** and **4** can be best described as trigonal bipyramid. For complex **3**, O(3), O(4), N(1) and V(1) [O(3) and O(4) are terminal oxygen atoms] define one satisfactory plane with a mean deviation of 0.0018 nm and corresponding deviations of -0.0010, -0.0015, -0.0011, +0.0036 nm for O(3), O(4), N(1) and V(1), respectively, while O(1) and O(2) are 0.1908 and -0.1814 nm away from the plane, respectively. For complex **4**, similarly, O(3), O(4), N(1) and V(1) [O(3) and O(4) are two terminal oxygen atoms] define the base plane with their deviations of 0.0010, 0.0016, 0.0012 and -0.0038 nm, respectively, and the mean deviation of 0.0019 nm, while O(1) and O(2) are -0.1892 nm and 0.1835 nm away from the corresponding plane, respectively. All double bonds [for complex **3**, V(1) = O(3), 0.1610(4) nm; V(1) = O(4), 0.1648(4) nm; for complex **4**, V(1) = O(3), 0.1590(4) nm; V(1) = O(4), 0.1645(4) nm] are unexceptional compared with reported ones⁸ and consistent with the empirical rule: V = O (oxo) < V = O (phenolate) < V = O (enolic) < V = O (alcoholic).⁹

Table 1 Crystallographic data for complexes **3** and **4**

	3	4
Formula	C ₂₀ H ₁₇ N ₃ O ₅ BrV	C ₂₁ H ₁₉ N ₃ O ₅ BrV
M_r	510.2	524.2
Colour	Brown	Brown
Crystal size (mm)	0.40 × 0.30 × 0.25	0.45 × 0.30 × 0.20
Crystal system	Monoclinic	Triclinic
Space group	$P2_1/n$	$P\bar{1}$
a (nm)	0.73632(6)	0.80512(6)
b (nm)	0.95142(7)	0.94136(7)
c (nm)	2.8594(2)	1.46480(11)
α (°)		99.1900(10)
β (°)	95.305(2)	99.4530(10)
γ (°)		104.6670(0)
V (nm ³)	1.9946(3)	1.03558(13)
Z	4	2
D_c (g/cm ³)	1.699	1.681
μ (mm ⁻¹)	2.539	2.448
$F(000)$	1024	528
Temperature (K)	293(2)	293(2)
θ range for data collections	1.43° to 25.10°	1.44° to 25.05°
Reflections measured	7206	5443
Independent reflections (R_{int})	3505 (0.0363)	3648 (0.0280)
Observed reflections	2538	3056
S	1.102	1.108
Final R indices [$I > 2\sigma(I)$]	$R_1 = 0.0517$, $wR_2 = 0.1369^a$	$R_1 = 0.0557$, $wR_2 = 0.1436^b$
R indices (all data)	$R_1 = 0.0816$, $wR_2 = 0.1593$	$R_1 = 0.0683$, $wR_2 = 0.1603$
$(\Delta/\sigma)_{max}$	0.001	0.000
$(\Delta\rho)_{max}$ (e/nm ³)	475	673
$(\Delta\rho)_{min}$ (e/nm ³)	-814	-868

^a $w = 1/[\sigma^2(F_o)^2 + (0.0825P)^2 + 2.2058P]$, where $P = (F_o^2 + 2F_c^2)/3$ for complex **3**; ^b $w = 1/[\sigma^2(F_o)^2 + (0.0846P)^2 + 1.7808P]$, where $P = (F_o^2 + 2F_c^2)/3$ for complex **4**.

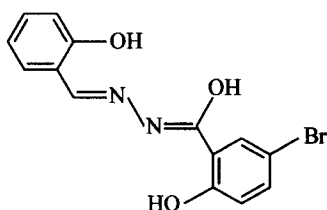


Fig. 1 Molecular structure of ligand H₂L.

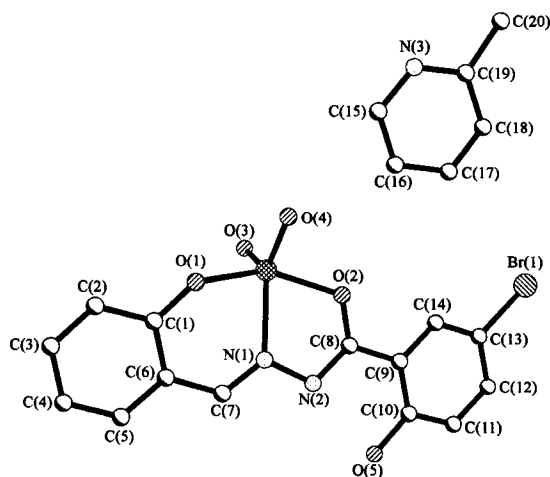


Fig. 2 Perspective view of 3 with atom labeling scheme.

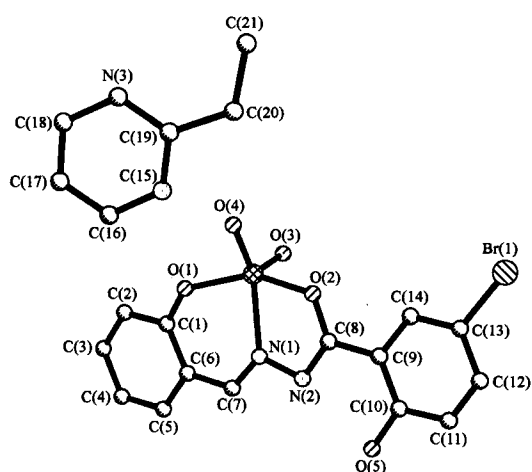


Fig. 3 Perspective view of 4 with atom labeling scheme.

Table 2 Selected bond lengths (nm) and angles (°) for 3 and 4

	3	4
V(1)—O(1)	0.1918(3)	0.1907(3)
V(1)—O(2)	0.1983(3)	0.1973(3)
V(1)—O(3)	0.1610(4)	0.1590(4)
V(1)—O(4)	0.1648(4)	0.1645(4)
V(1)—N(1)	0.2152(4)	0.2144(4)
O(1)—V(1)—O(2)	149.66(16)	148.22(15)
O(1)—V(1)—O(3)	102.18(17)	103.52(18)
O(1)—V(1)—O(4)	94.25(17)	93.94(16)
O(1)—V(1)—N(1)	81.50(15)	81.76(13)

Continued

	3	4
O(2)—V(1)—O(3)	101.79(17)	102.27(17)
O(2)—V(1)—O(4)	94.60(16)	94.37(15)
O(2)—V(1)—N(1)	73.33(14)	73.60(13)
O(3)—V(1)—O(4)	110.3(2)	110.1(2)
O(3)—V(1)—N(1)	108.82(18)	105.67(18)
O(4)—V(1)—N(1)	140.62(19)	143.97(19)

Table 3 Essential atomic coordinates ($\times 10^4$) and equivalent isotropic displacement parameters ($\text{nm}^2 \times 10$) for 3

Atom	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{eq} ^a
V(1)	5384(1)	6589(1)	-1616(1)	36(1)
C(1)	4654(6)	9565(5)	-1914(2)	36(1)
C(2)	3910(7)	10363(6)	-2296(2)	43(1)
C(3)	3344(7)	11721(6)	-2233(2)	48(1)
C(4)	3541(7)	12362(6)	-1795(2)	48(1)
C(5)	4252(7)	11588(6)	-1411(2)	45(1)
C(6)	4792(6)	10192(5)	-1462(2)	36(1)
C(7)	5475(7)	9414(5)	-1055(2)	39(1)
C(8)	6687(7)	6181(5)	-661(2)	35(1)
C(9)	7180(6)	5359(5)	-226(2)	34(1)
C(10)	7443(7)	6010(6)	217(2)	40(1)
C(11)	7826(8)	5176(6)	616(2)	49(1)
C(12)	8008(7)	3759(6)	581(2)	46(1)
C(13)	7744(7)	3125(6)	141(2)	41(1)
C(14)	7314(7)	3907(5)	-256(2)	40(1)
C(17)	9447(8)	1900(8)	-1158(2)	65(2)
C(16)	8794(8)	2890(8)	-1492(2)	62(2)
C(15)	8524(8)	2477(7)	-1943(2)	59(2)
N(3)	8883(6)	1146(5)	-2061(1)	50(1)
C(19)	9497(7)	177(7)	-1755(2)	50(1)
C(20)	9833(10)	-1248(7)	-1932(3)	82(2)
N(1)	5869(5)	8093(4)	-1056(1)	35(1)
N(2)	6472(6)	7527(4)	-621(1)	41(1)
C(18)	9787(7)	567(7)	-1289(2)	51(1)
O(1)	5236(5)	8273(4)	-1986(1)	45(1)
O(2)	6465(5)	5535(4)	-1060(1)	44(1)
O(4)	6488(6)	5697(4)	-1994(1)	56(1)
O(3)	3286(5)	6096(4)	-1643(1)	52(1)
O(5)	7263(6)	7411(4)	271(1)	60(1)
Br(1)	7917(1)	1133(1)	99(1)	67(1)

^a *U*_{eq} is defined as one third of the trace of the orthogonalized *U*_{ij} tensor.

Table 4 Essential atomic coordinates ($\times 10^4$) and equivalent isotropic displacement parameters ($\text{nm}^2 \times 10$) for 4

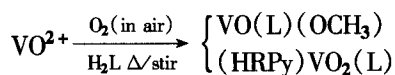
Atom	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{eq} ^a
V(1)	-1719(1)	-7911(1)	-3276(1)	38(1)
C(1)	-1341(6)	-10972(5)	-3858(3)	37(1)
C(2)	-1078(7)	-11928(5)	-4624(3)	45(1)
C(3)	-544(7)	-13178(6)	-4494(4)	47(1)
C(4)	-267(7)	-13530(6)	-3611(4)	51(1)

				Continued
Atom	<i>x</i>	<i>y</i>	<i>z</i>	U_{eq}^a
C(5)	-466(7)	-12591(6)	-2847(4)	45(1)
C(6)	-985(6)	-11296(5)	-2948(3)	36(1)
C(7)	-1183(6)	-10351(5)	-2130(3)	38(1)
C(8)	-2095(6)	-7107(5)	-1399(3)	37(1)
C(9)	-2255(6)	-6096(5)	-551(3)	38(1)
C(10)	-2056(6)	-6461(6)	347(3)	42(1)
C(11)	-2139(7)	-5445(6)	1123(3)	49(1)
C(12)	-2431(7)	-4092(6)	1024(3)	45(1)
C(13)	-2639(6)	-3738(5)	141(3)	41(1)
C(14)	-2536(6)	-4713(5)	-638(3)	41(1)
C(15)	-3565(8)	10286(7)	3742(4)	64(2)
C(16)	-3704(9)	9766(8)	2805(5)	72(2)
C(17)	-4158(8)	10606(8)	2180(4)	68(2)
C(18)	-4461(8)	11962(7)	2505(4)	58(1)
C(19)	-4298(6)	12467(6)	3465(4)	49(1)
C(20)	-4592(8)	13894(7)	3919(4)	62(2)
C(21)	-4601(10)	15061(9)	3320(5)	80(2)
N(1)	-1566(5)	-9105(4)	-2150(2)	35(1)
N(2)	-1765(5)	-8361(4)	-1286(3)	40(1)
N(3)	-3856(6)	11604(6)	4043(3)	54(1)
O(1)	-1962(5)	-9841(4)	-4018(2)	45(1)
O(2)	-2290(5)	-6716(4)	-2200(2)	45(1)
O(3)	250(5)	-6995(4)	-3244(2)	58(1)
O(4)	-3083(6)	-7490(4)	-4083(2)	59(1)
O(5)	-1770(6)	-7770(4)	495(2)	55(1)
Br(1)	-2953(1)	-1835(1)	27(1)	59(1)

^a U_{eq} is defined as one third of the trace of the orthogonalized U_{ij} tensor.

In our previous paper,⁷ the vanadium in starting materials is of V(IV) oxidation state, but it appears to be V(V) in all compounds of **1** to **4**, indicating that it was oxidized by air during reaction procedures (Scheme 1), which was mentioned in the literature.¹⁰ Moreover, we found the phenomenon that the complexes have been coordinated by solvents, as **1** owns the molecular formula of VO(L)(OCH₃), while complexes **2** to **4** form the ones of (HRPy)VO₂(L) (HRPy represents pyridine, 2-Me-pyridine or 2-Et-pyridine), which indicate that the solvents play important roles in the formations of these compounds.

Scheme 1



From the comparison of complexes **1** to **4** (Table 5), different hydrogen bonds or van der Waal's effect can be found. (1) In terms of intramolecular hydrogen bonds, the bond lengths of N(2)⋯O(5) for complexes **1** to **4** are 0.2598, 0.2583, 0.2578 and 0.2580 nm, respectively, and their angles of N(2)⋯H—O(5) are 129.06°, 131.10°, 146.55° and 146.04°, respectively. The angles of N(2)⋯H—O(5) for **3** and **4** are much larger than those of **1** and **2**, which may be attributed to their different coordination environments (square pyramid for **1** and **2**, trigonal bipyramid for **3** or **4**). On the other hand, intramolecular hydrogen bond [N(3)⋯O(4)] between the N atom of pyridine and one terminal oxygen (Fig. 4 for complex **2**, Fig. 5 for complex **3** or **4**) is formed and the corresponding lengths for complexes **2** to **4** are 0.2699, 0.2725 and 0.2659 nm, respectively. (2) In terms of intermolecular hydrogen bonds, compound **1** has a few unique intermolecular hydrogen bonds compared with the latter three complexes, which can bind two adjacent molecules strongly (seen from part of its packing diagram in Fig. 6). The reason for complexes **2** to **4** having no such bonds might be the hindrance effect of R-pyridine (R-pyridine, R = H, CH₃, CH₃CH₂). (3) Furthermore, for complex **2**, there is weak contact between two bromide atoms (Br⋯Br: 0.3618 nm, van der Waal's radii for Br, 0.192 nm¹¹) (shown in Fig. 4) from adjacent molecules. While, for complex **3**, there is almost no contact between two bromide atoms (Br⋯Br 0.3834 nm) from adjacent molecules. The shortest length of Br⋯Br (0.6016 nm) in complex **4** indicates that there is no any contact between two Br atoms.

The latter three complexes **2** to **4** were obtained from the similar reactions of H₂L with VOSO₄ in acetonitrile-methanol with the small quantity of pyridine, 2-Me-pyridine or 2-Et-pyridine. In the meantime, X-ray analyses have shown that VO³⁺ adopts square pyramid coordination environment in both **1** and **2**, while it shows trigonal bipyramidal coordination in both complexes **3** and **4**. On the other hand, though the latter three have the same coordination, the fourth has different space group from complexes **2** and **3** ($P2_1/n$ for **2** and **3**, $P\bar{1}$ for **4**).

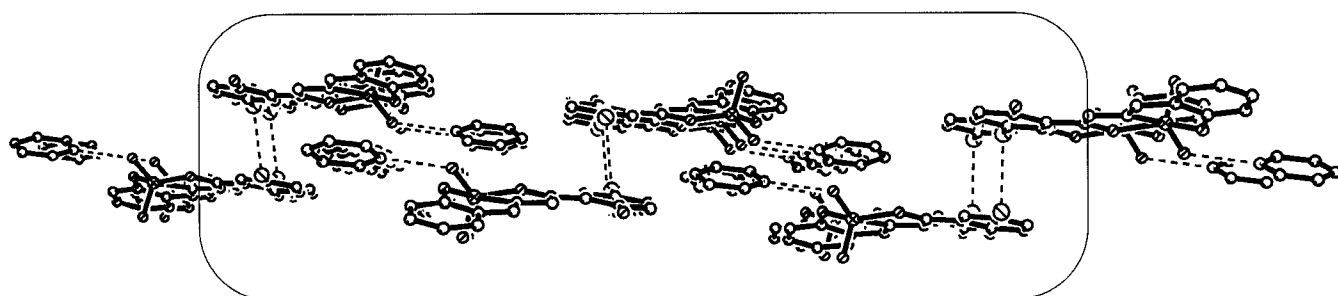


Fig. 4 Packing diagram for complex **2** showing the van der Waal contact between Br⋯Br.

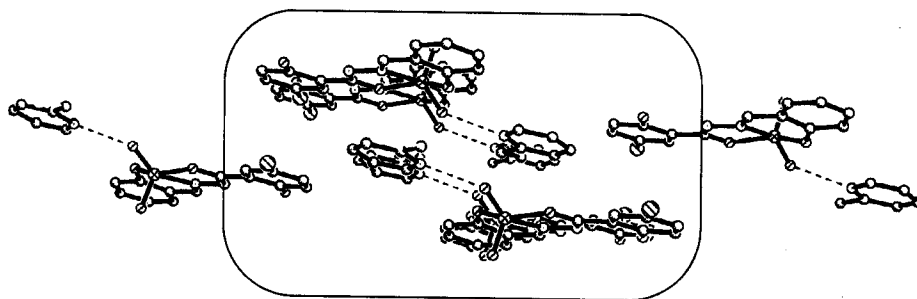


Fig. 5 Packing diagram for complex 3 (complex 4 has the similar structure).

Table 5 Hydrogen bonds and angles, van der Waal's effect for complexes 1 to 4 (A, B represent different molecules)

Compound	Hydrogen bonds (intramolecular, nm)		Hydrogen bonds or van der Waal's effect (intermolecular, nm)	
1 [PyH][V ^V O ₂ (C ₁₄ H ₉ N ₂ O ₃ Br)]	N(2)⋯O(5)	0.2598	O(1A)⋯O(1B)	0.2674
			O(1A)⋯O(2B)	0.2883
2 [V ^V O(C ₁₄ H ₉ N ₂ O ₃ Br)(OCH ₃)]	N(2)⋯O(5)	0.2583	O(1A)⋯O(3B)	0.2831
	O(4A)⋯N(3B)	0.2699	Br(1A)⋯Br(1B)	0.3618
3 [2-MePyH][V ^V O ₂ (C ₁₄ H ₉ N ₂ O ₃ Br)]	N(2)⋯O(5)	0.2578	Br(1A)⋯Br(1B)	0.3834
	O(4A)⋯N(3B)	0.2725		
4 [2-EtPyH][V ^V O ₂ (C ₁₄ H ₉ N ₂ O ₃ Br)]	N(2)⋯O(5)	0.2580		
	O(4A)⋯N(3B)	0.2659		

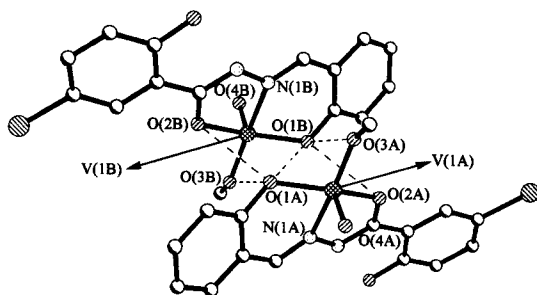


Fig. 6 View of intermolecular hydrogen bonds for complex 1.

Thermal analysis

The crystal samples of the four complexes were heated from 30 °C to 830 °C. For 1, the losses of the ligands (L²⁻ and -OCH₃) were from 320 °C; for 2 to 4, TGA showed that the first weight loss occurred from 120 °C to 195 °C and the estimated weight losses were 16.5%, 17.1% and 20.0%, respectively, which is consistent with the losses of R-pyridine molecules (calcd 16.1% for 2, 18.5% for 3 and 20.6% for 4), while L²⁻ ligands were lost gradually from 295 °C.

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(E0206282 PAN, B. F.; ZHENG, G. C.)