Vanadium(V) complexes of mandelic acid

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SUPPORTING INFORMATION

Contents

Table S1 Crystal structure solution and refinement data

	(**1**)	(**2**)	(**3**)	(**4**)	(**5**)
CCDC Number	1911697	1911693	1911694	1911695	1911696
Chemical formula	$C_{16}H_{12}O_{10}V_2{\cdot}2(C_4H_{12}N)$	$C_{16}H_{12}O_{10}V_2{\cdot}2(C_4H_{12}N){\cdot}H_2O$	$C_{16}H_{26}NO_5V$	$C_{16}H_{12}O_{10}V_2{\cdot}2(C_{24}H_{20}P){\cdot}2(C_3H_6O){\cdot}2(H_2O)$	$C_{16}H_{12}NO_{13}V_3$
*M*r	614.43	632.44	363.32	1297.06	579.09
Crystal system, space group	Monoclinic, $P2_1/c$	Orthorhombic, $P2_12_12_1$	Monoclinic, $P2_1$	Triclinic, *P*-1	Orthorhombic, *Pbcm*
Temperature (K)	120	150	120	150	120
a, b, c (Å)	12.3039 (5), 13.3030 (4), 18.5521 (9)	10.7521 (8), 15.6443 (11), 18.2430 (13)	7.4443 (4), 18.6609 (12), 13.3061 (8)	10.8560 (3), 11.7821 (4), 13.1551 (4)	11.6214 (4), 13.4677 (5), 25.1952 (8)
α, β, γ (°)	β = 109.344 (1)		β = 95.543 (2)	74.534 (2), 82.691 (1), 71.735 (1)	
V (Å^3)	2865.2 (2)	3068.6 (4)	1839.80 (19)	1538.24 (8)	3943.4 (2)
Z	4	4	4	1	4
Radiation type	Mo *K*α	Mo *K*α	Mo *K*α	Mo *K*α	Mo *K*α
μ (mm^{-1})	0.71	0.66	0.56	0.42	0.73
Crystal size (mm)	0.75 × 0.32 × 0.30	0.68 × 0.65 × 0.39	0.54 × 0.29 × 0.20	0.97 × 0.43 × 0.28	0.38 × 0.24 × 0.20
Data collection					
Diffractometer	Bruker D8 VENTURE	Nonius KappaCCD with Bruker APEXII detector	Bruker D8 VENTURE	Nonius KappaCCD with Bruker APEXII detector	Bruker D8 VENTURE
Absorption correction	Multi-scan	Multi-scan	Multi-scan	Multi-scan	Multi-scan
T_{min}, T_{max}	0.72, 0.82	0.56, 0.78	0.67, 0.73	0.76, 0.82	0.76, 0.83
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	48284, 11633, 10139	21743, 7004, 5989	54352, 14165, 11081	75879, 8321, 7496	97181, 5373, 4261
R_{int}	0.030	0.051	0.054	0.023	0.057
(sin θ/λ)max (Å^{-1})	0.814	0.650	0.804	0.687	0.683
Refinement					
$R[F^2 > 2\sigma(F^2)]$, *wR*(F2), *S*	0.039, 0.101, 1.07	0.051, 0.142, 1.04	0.062, 0.176, 1.02	0.028, 0.078, 1.03	0.039, 0.117, 1.05
No. of reflections	11633	7004	14165	8321	5373

No. of parameters	352	367, 15 restraints	424, 7 restraints	399	151
H-atom treatment	H-atom parameters constrained	H atoms treated by a mixture of independent and constrained refinement	H-atom parameters constrained	H-atom parameters constrained	H-atom parameters constrained
Flack parameter	N/A	0.00(4)	0.02(3)	N/A	N/A
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.80, −0.64	1.05, −0.56	2.07, −0.63	0.40, −0.33	0.68, −0.66

Infrared Spectra

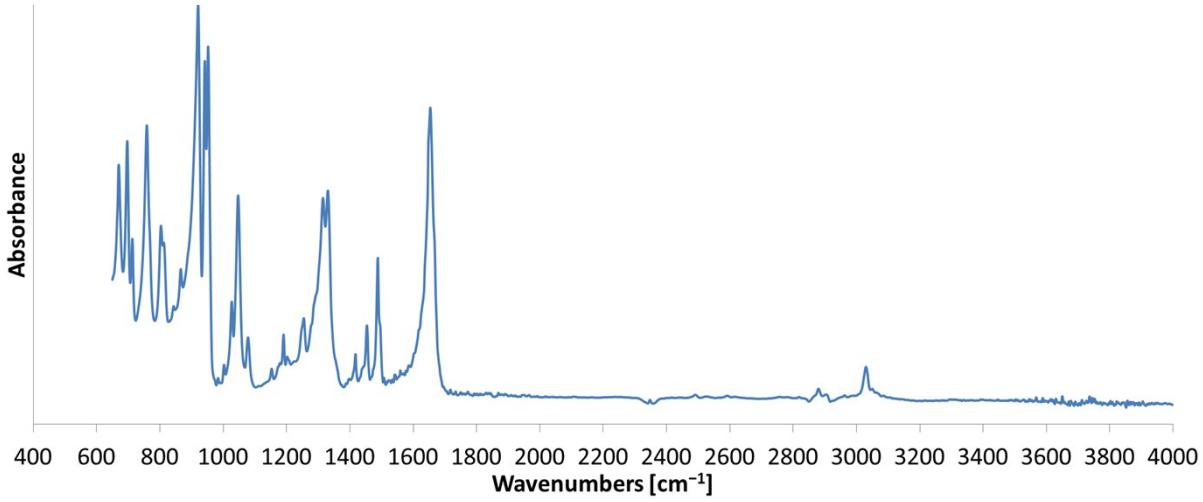

Fig. S1 Infrared spectrum of substance (**1**) measured by ATR technique.

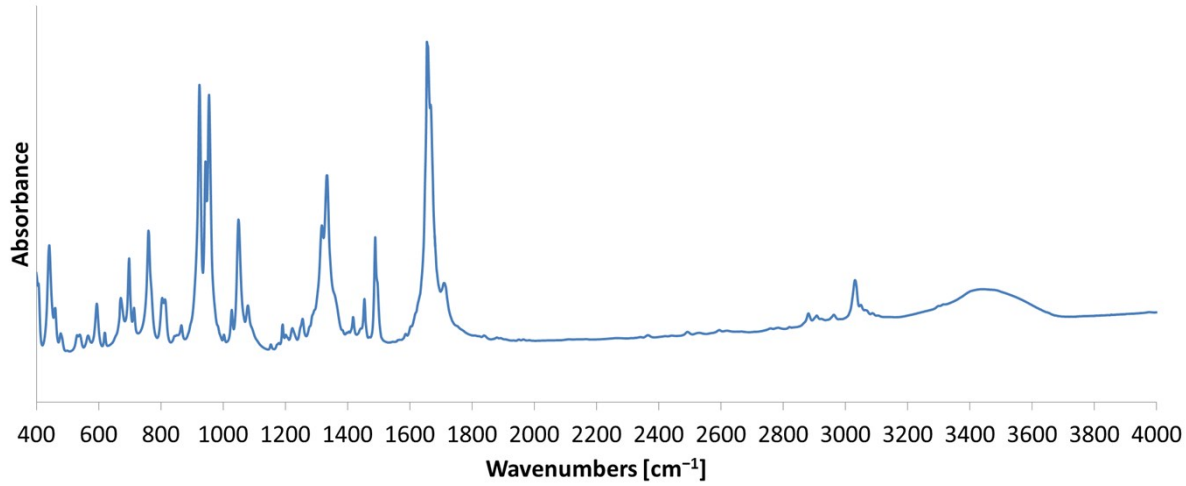

Fig. S2 Infrared spectrum of substance (**1**) measured in KBr pellet.

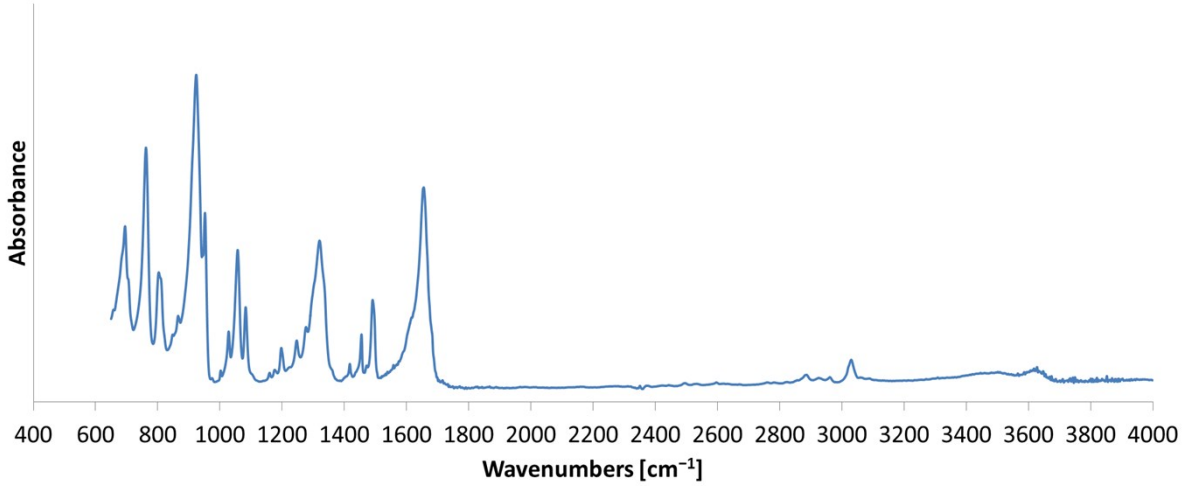

Fig. S3 Infrared spectrum of substance (**2**) measured by ATR technique.

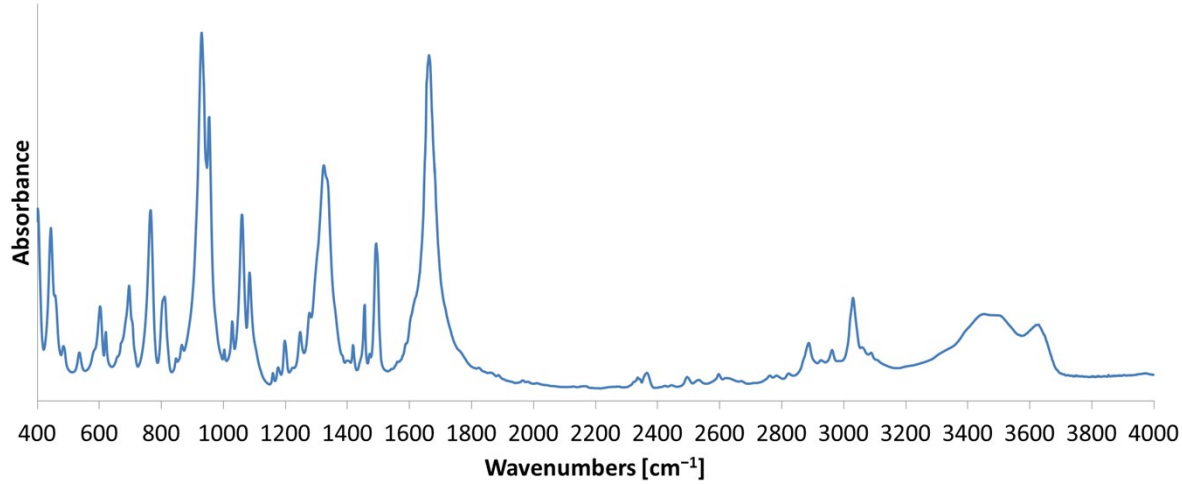

Fig. S4 Infrared spectrum of substance (**2**) measured in KBr pellet.

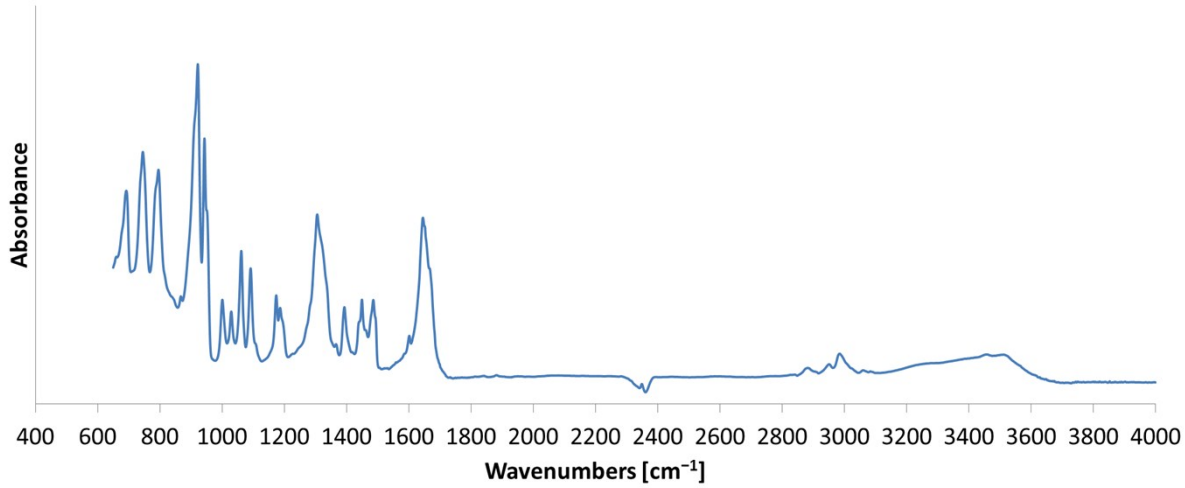

Fig. S5 Infrared spectrum of substance (**3**) measured by ATR technique.

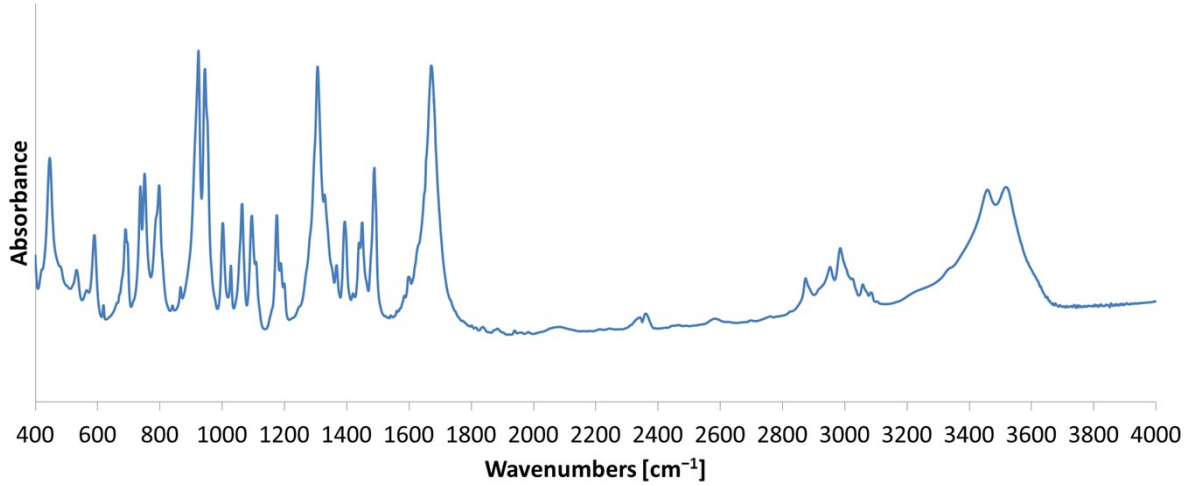

Fig. S6 Infrared spectrum of substance (**3**) measured in KBr pellet.

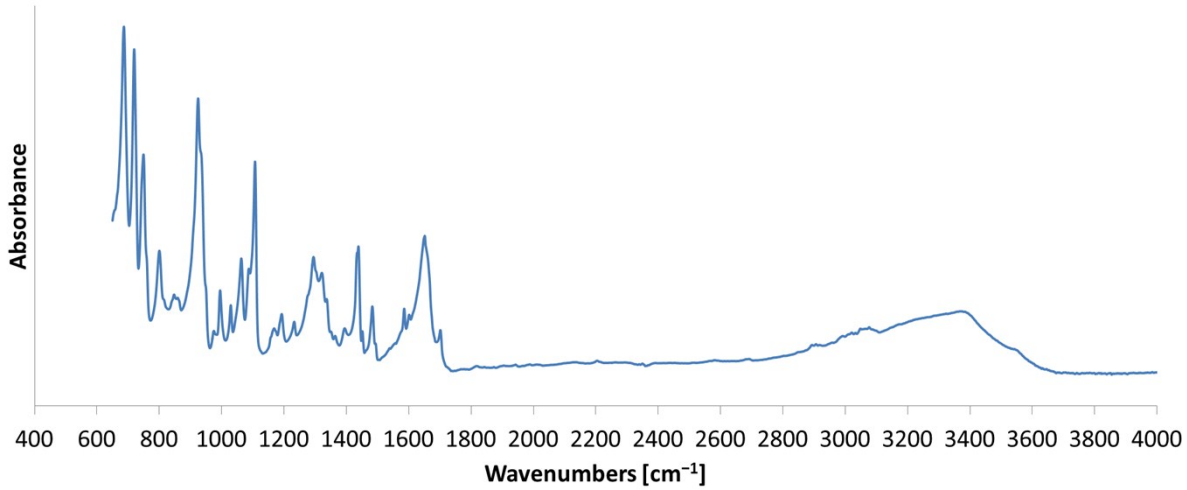

Fig. S7 Infrared spectrum of substance (**4**) measured by ATR technique.

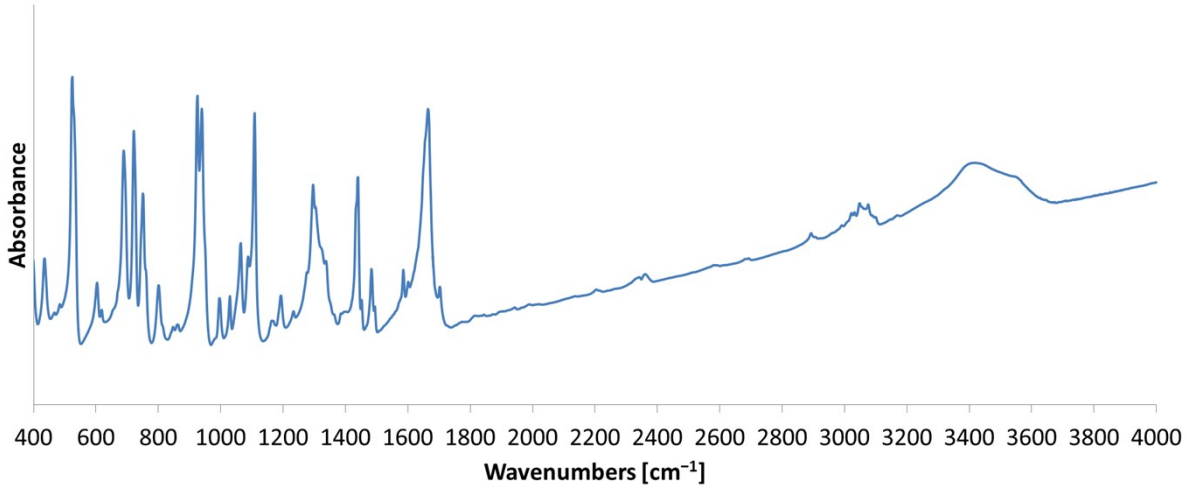

Fig. S8 Infrared spectrum of substance (**4**) measured in KBr pellet.

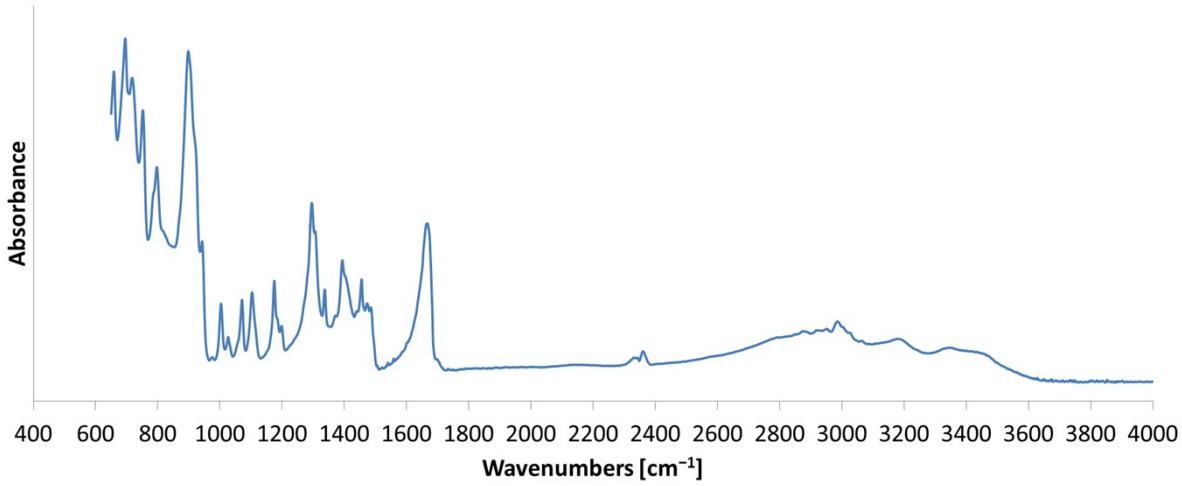

Fig. S9 Infrared spectrum of substance (**5**) measured by ATR technique.

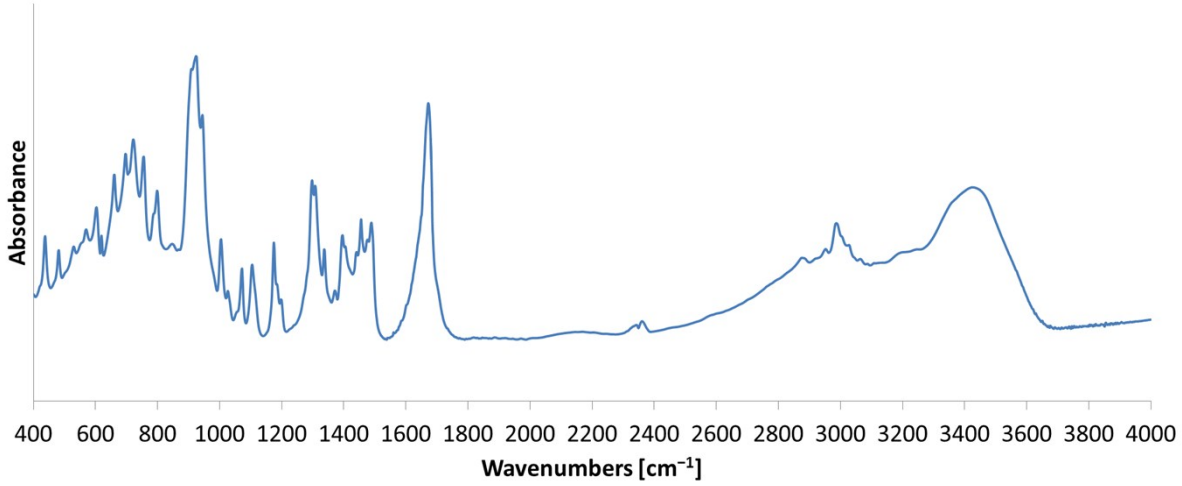

Fig. S10 Infrared spectrum of substance (**5**) measured in KBr pellet.

Table S2 Band assignment of cations and solvent molecules in IR spectra of **1** - **5**

	(**1**)	(**2**)	(**3**)	(**4**)	(**5**)
NMe_4^+	951 (m) 1488 (m)	954 (m) 1491 (m)			
NEt_4^+			798 (s) 1002 (s) 1174 (m-s) 1494 (m-s)		799 (s) 1004 (m) 1175 (m) 1489 (m)
PPh_4^+				689 (s) 722 (s) 751 (m) 996 (m) 1107 (s) 1438 (m)	
NH_4^+					~3200 (m,b)
H_2O		3441 (m) 3614 (m)	3458 (m) 3518 (m)	3377 (m,b)	~3322 (m,b)
Acetone				1702 (m)	

s strong, m medium, b broad

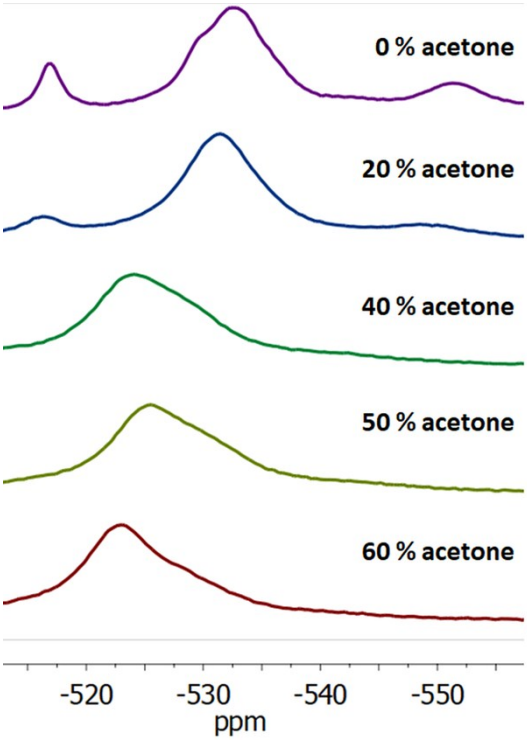

Fig. S11 The ^{51}V NMR spectra of the system N*Me*$_4$VO$_3$ − *rac*-H$_2$*mand* − H$_2$O − acetone. *c*(V) = 0,03 mol/L, *c*(L) = 0,02 mol/L. The assignment of chemical shifts: $\mathbf{V_2L_2}$ −534 ppm (0 % acetone); −532 ppm (20 % acetone); −524 ppm (40 % acetone); −526 ppm (50 % acetone) and −523 ppm (60 % acetone). $\mathbf{V_3L_2}$ −551 ppm (0 % acetone). $\mathbf{V_{10}}$ −517 ppm (0 % acetone).