

Crystal structure of $\text{Na}_4\text{Rb}_2(\text{V}_{10}\text{O}_{28}) \cdot 10\text{H}_2\text{O}$, $\text{H}_{20}\text{Na}_4\text{O}_{38}\text{Rb}_2\text{V}_{10}$

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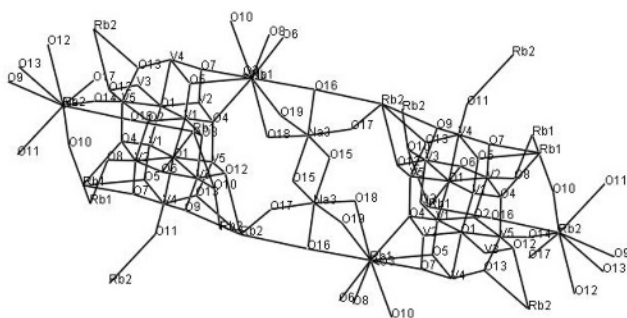
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Abstract

$\text{H}_{20}\text{Na}_4\text{O}_{38}\text{Rb}_2\text{V}_{10}$, triclinic, $P\bar{1}$ (no. 2), $a = 8.5555(9)$ Å, $b = 10.448(1)$ Å, $c = 11.237(2)$ Å, $\alpha = 68.483(1)^\circ$, $\beta = 87.170(2)^\circ$, $\gamma = 67.291(1)^\circ$, $V = 857.0$ Å³, $Z = 1$, $R_{\text{gt}}(F) = 0.0611$, $wR_{\text{ref}}(F^2) = 0.1679$, $T = 293$ K.

Table 1. Data collection and handling.

Crystal:	colourless lumps, size 0.27×0.28×0.30 mm
Wavelength:	Mo K_{α} radiation (0.71073 Å)
μ :	55.91 cm ⁻¹
Diffractometer, scan mode:	CCD area detector, φ and ω
$2\theta_{\text{max}}$:	50.04°
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$:	4439, 2997
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 2458
$N(\text{param})_{\text{refined}}$:	246
Programs:	SHELX [5]

Source of material

All commercially obtained reagent-grade chemicals were used without further purification. A mixture of RbNO_3 (0.01 mmol, 0.002 g), NH_4VO_3 (0.1 mmol, 0.01 g), H_3BO_3 (0.1 mmol, 0.006 g) and NH_4OH (1 mL) were added into 20 mL water with 40% (v/v) methanol and heated for 10 h at 343 K. The solution was obtained by filtration after cooling the reaction to room temperature. Colourless lump single crystals suitable for X-ray measurements were obtained after a few weeks.

Discussion

Polyoxovanadates act as a rich class of inorganic materials, exhibiting a range of molecular and electronic structural versatility, reactivity and relevance to catalysis, biology, medicine, geochemistry, material science and topology [1,2]. Metal oxide clusters contain symmetrical core composed of VO_n units. The decavanadate polyanions $[\text{V}_{10}\text{O}_{28}]^{6-}$ incorporate with the intercalated metal/organic fragments, displaying charming variety of architectures and topologies [3,4]. We obtained a novel decavanadate complex by a simple reaction in aqueous solution.

The title crystal structure is built up of four Na atoms, two Rb atoms and a decavanadate anion fragment. In the cluster, the V atom are coordinated by six O atoms to form a distorted octahedron. There are eight μ_2 -O atoms, nine μ_3 -O atoms and a μ_4 -O atoms. The μ_2 -O8 bridges V2 and Rb1, and μ_2 -O15 bridges two Na atoms, while μ_2 -O16 bridges Rb1 and Rb2. The μ_3 -O3 bridges V1, V3 and Rb1, while μ_3 -O10 bridges V3, Rb1 and Rb2. The μ_4 -O4 bridges V1, V2, V5 and Rb1. The distances of V–O and Rb–O are in the range of 1.603–2.361 and 2.855–3.445 Å. The angle Na–O15–Na is 88.35°. The crystal packing is stabilized by intramolecular O–H...O hydrogen bonds.

Table 2. Atomic coordinates and displacement parameters (in Å²).

Atom	Site	x	y	z	U_{iso}
H(15A)	2i	–0.2089	0.5634	–0.0641	0.047
H(15B)	2i	–0.2698	0.5674	0.0539	0.047
H(16A)	2i	–0.1419	0.2207	0.2957	0.058
H(16B)	2i	–0.0042	0.0927	0.2954	0.058
H(17A)	2i	0.1518	0.0869	0.0596	0.055
H(17B)	2i	0.0841	0.2096	–0.0555	0.055
H(18A)	2i	0.0803	0.5417	0.2231	0.057
H(18B)	2i	–0.0820	0.5403	0.2440	0.057
H(19A)	2i	0.3846	0.0925	0.2497	0.055
H(19B)	2i	0.4137	0.2219	0.2285	0.055

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Table 3. Atomic coordinates and displacement parameters (in Å²).

Atom	Site	Occ.	x	y	z	U ₁₁	U ₂₂	U ₃₃	U ₁₂	U ₁₃	U ₂₃
Rb(1)	2i	0.5	0.1768(1)	0.2050(1)	0.4937(1)	0.0296(6)	0.0205(5)	0.0387(6)	−0.0100(4)	0.0040(4)	−0.0160(4)
Rb(2)	2i	0.5	0.2828(2)	0.8236(1)	0.9267(1)	0.0471(8)	0.0264(6)	0.0352(7)	0.0003(5)	−0.0045(5)	−0.0140(5)
Na(1)	2i	0.5	0.1768(1)	0.2050(1)	0.4937(1)	0.0296(6)	0.0205(5)	0.0387(6)	−0.0100(4)	0.0040(4)	−0.0160(4)
Na(2)	2i	0.5	0.2828(2)	0.8236(1)	0.9267(1)	0.0471(8)	0.0264(6)	0.0352(7)	0.0003(5)	−0.0045(5)	−0.0140(5)
Na(3)	2i		0.0679(4)	0.3327(3)	0.1268(3)	0.040(2)	0.027(1)	0.049(2)	−0.015(1)	0.003(1)	−0.017(1)
V(1)	2i		0.3105(1)	0.4968(1)	0.52043(9)	0.0164(6)	0.0123(5)	0.0214(6)	−0.0055(4)	0.0009(4)	−0.0076(4)
V(2)	2i		0.3781(1)	0.7819(1)	0.4984(1)	0.0237(6)	0.0111(5)	0.0238(6)	−0.0053(4)	0.0006(4)	−0.0088(4)
V(3)	2i		0.1725(1)	0.7839(1)	0.2780(1)	0.0197(6)	0.0159(5)	0.0264(6)	−0.0048(4)	−0.0030(4)	−0.0063(4)
V(4)	2i		0.5282(1)	0.7915(1)	0.2364(1)	0.0229(6)	0.0148(5)	0.0238(6)	−0.0081(4)	0.0009(4)	−0.0057(4)
V(5)	2i		0.5286(1)	0.4942(1)	0.74117(9)	0.0244(6)	0.0147(5)	0.0203(6)	−0.0068(4)	0.0007(4)	−0.0092(4)
O(1)	2i		0.4321(5)	0.6242(4)	0.3944(3)	0.019(2)	0.010(2)	0.016(2)	−0.005(2)	0.003(2)	−0.005(2)
O(2)	2i		0.3169(5)	0.6138(4)	0.6171(4)	0.019(2)	0.017(2)	0.020(2)	−0.007(2)	0.001(2)	−0.009(2)
O(3)	2i		0.1263(5)	0.6194(4)	0.4279(4)	0.022(2)	0.017(2)	0.029(2)	−0.007(2)	0.000(2)	−0.008(2)
O(4)	2i		0.3969(5)	0.3804(4)	0.4158(4)	0.016(2)	0.014(2)	0.020(2)	−0.005(2)	−0.001(2)	−0.007(2)
O(5)	2i		0.2626(5)	0.3650(4)	0.6425(4)	0.017(2)	0.018(2)	0.027(2)	−0.006(2)	−0.001(2)	−0.009(2)
O(6)	2i		0.1715(5)	0.8631(4)	0.4037(4)	0.021(2)	0.015(2)	0.027(2)	−0.003(2)	0.003(2)	−0.009(2)
O(7)	2i		0.4828(5)	0.8689(4)	0.3669(4)	0.024(2)	0.012(2)	0.027(2)	−0.006(2)	−0.000(2)	−0.009(2)
O(8)	2i		0.3438(6)	0.8804(5)	0.5863(4)	0.037(3)	0.020(2)	0.036(3)	−0.010(2)	0.000(2)	−0.018(2)
O(9)	2i		0.2978(5)	0.8814(4)	0.1768(4)	0.023(2)	0.015(2)	0.025(2)	−0.006(2)	−0.003(2)	−0.005(2)
O(10)	2i		−0.0218(6)	0.8819(5)	0.2112(4)	0.028(3)	0.022(2)	0.037(3)	−0.005(2)	−0.007(2)	−0.007(2)
O(11)	2i		0.6093(6)	0.8974(5)	0.1366(4)	0.040(3)	0.018(2)	0.034(3)	−0.014(2)	0.002(2)	−0.005(2)
O(12)	2i		0.7500(5)	0.3643(4)	0.7944(4)	0.021(2)	0.016(2)	0.024(2)	−0.007(2)	−0.004(2)	−0.008(2)
O(13)	2i		0.4406(5)	0.3570(4)	0.8309(4)	0.024(2)	0.016(2)	0.023(2)	−0.006(2)	0.001(2)	−0.009(2)
O(14)	2i		0.4945(6)	0.5955(5)	0.8278(4)	0.035(3)	0.025(2)	0.030(2)	−0.013(2)	0.005(2)	−0.018(2)
O(15)	2i		−0.2096(7)	0.5126(5)	0.0146(5)	0.041(3)	0.042(3)	0.034(3)	−0.014(2)	0.012(2)	−0.017(2)
O(16)	2i		−0.0349(7)	0.1811(6)	0.2936(5)	0.043(3)	0.032(3)	0.069(4)	−0.013(2)	0.029(3)	−0.022(3)
O(17)	2i		0.0738(7)	0.1739(6)	0.0243(5)	0.058(4)	0.036(3)	0.028(3)	−0.001(3)	−0.004(2)	−0.011(2)
O(18)	2i		0.0226(7)	0.4878(6)	0.2441(5)	0.046(3)	0.058(4)	0.070(4)	−0.031(3)	0.026(3)	−0.050(3)
O(19)	2i		0.3424(7)	0.1810(5)	0.2488(5)	0.042(3)	0.023(2)	0.073(4)	−0.011(2)	−0.009(3)	−0.018(3)

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