Electronic Supplementary Information (ESI)

Synthesis and structural characterization of AMV_2O_8 (A = K, Rb, Tl, Cs; M = Nb, Ta) vanadates: Structural comparison of $A^+M^{5+}V_2O_8$ vanadates and $A^+M^{5+}P_2O_8$ phosphates

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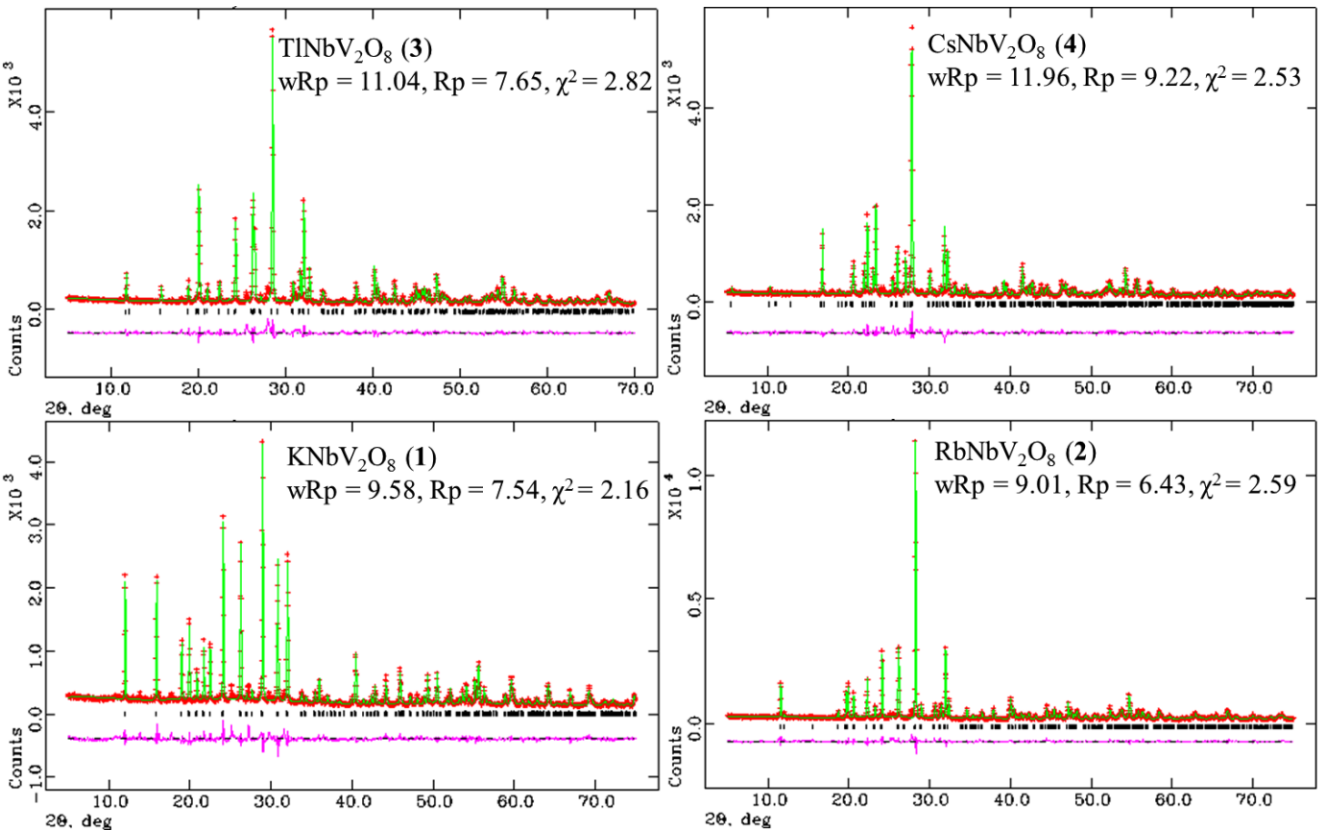

Figure. Final Rietveld XRD data plots of $KNbV_2O_8$ (**1**), $RbNbV_2O_8$ (**2**), $TlNbV_2O_8$ (**3**) and $CsNbV_2O_8$ (**4**) compounds. (red, observed; green, calculated; black, vertical bars- positions of the Bragg reflections; pink, difference between observed and calculated intensities); The values of agreement factors and χ^2 are presented.

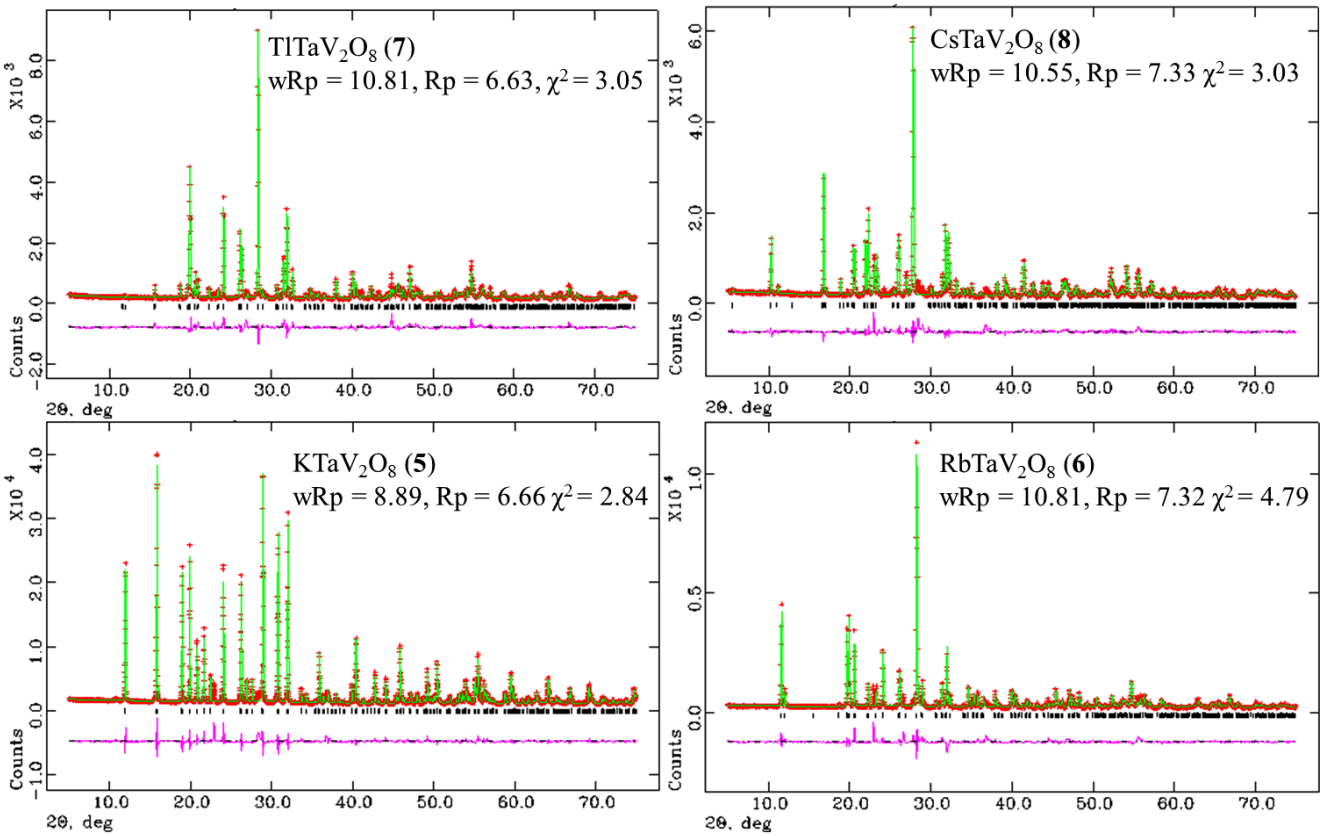

Figure. Final Rietveld XRD data plots of $KTaV_2O_8$ (**5**), $RbTaV_2O_8$ (**6**), $TlTaV_2O_8$ (**7**) and $CsTaV_2O_8$ (**8**) compounds. (red, observed; green, calculated; black, vertical bars- positions of the Bragg reflections; pink, difference between observed and calculated intensities); The values of agreement factors and χ^2 are presented.

Supplementary information

Table S1. Atomic coordinates and thermal parameters (Å^2), obtained from single crystal and Rietveld analysis of powder XRD data (marked with *), of ANbV$_2$O$_8$ (A = K(1), Rb(2), Tl(3), Cs(4)) and ATaV$_2$O$_8$ (A = K(5), Rb(6), Tl(7), Cs(8)) compounds.

Compound		x (× 10^4)	x*	y (× 10^4)	y*	z (× 10^4)	z*	$^{\#}U_{eq}$
KNbV$_2$O$_8$ (1)	Nb		0.1158(4)		0.2500000(0)		0.6914(6)	
	K		0.3754(11)		0.2500000(0)		0.5467(13)	
	V(1)		0.2885(7)		0.2500000(0)		0.9930(11)	
	V(2)		0.0310(6)		0.2500000(0)		0.2845(10)	
	O(1)		0.2024(11)		0.0139(28)		0.5996(21)	
	O(2)		0.0364(13)		-0.0047(25)		0.7768(20)	
	O(3)		0.1863(15)		0.2500000(0)		0.8888(26)	
	O(4)		0.0530(19)		0.2500000(0)		0.4861(20)	
	O(5)		0.3655(17)		0.2500000(0)		0.8546(28)	
	O(6)		0.3749(17)		-0.250000(0)		0.6948(31)	
KTaV$_2$O$_8$ (5)	Ta	1174(1)	0.11717(26)	2500	0.2500000(0)	6885(1)	0.6883(39)	5(1)
	K	3772(1)	0.3747(13)	2500	0.2500000(0)	5478(2)	0.5487(15)	25(1)
	V(1)	2866(1)	0.2885(8)	2500	0.2500000(0)	9891(1)	0.9904(12)	7(1)
	V(2)	281(1)	0.0292(7)	2500	0.2500000(0)	2902(1)	0.2864(11)	5(1)
	O(1)	2013(2)	0.1986(13)	81(5)	-0.0009(33)	6018(4)	0.6088(24)	14(1)
	O(2)	362(2)	0.0381(15)	4(5)	-0.0052(29)	7700(4)	0.7707(22)	11(1)
	O(3)	1837(3)	0.1884(17)	2500	0.25000(0)	8848(6)	0.8826(29)	12(1)
	O(4)	508(3)	0.0512(21)	2500	0.25000(0)	4890(5)	0.4848(23)	13(1)
	O(5)	3660(3)	0.3658(20)	2500	0.25000(0)	8640(7)	0.8554(33)	21(1)
	O(6)	3800(3)	0.3785(20)	-2500	-0.2500(0)	6915(6)	0.6849(35)	18(1)

Compound		x (× 10^4)	x*	y (× 10^4)	y*	z (× 10^4)	z*	$^{\#}U_{eq}$
$RbNbV_2O_8$ (2)	Nb	1603(1)	0.16007(26)	2500	0.2500000(0)	5040(1)	0.5054(5)	9(1)
	Rb	1195(1)	0.12014(29)	2500	0.2500000(0)	484(1)	0.0494(5)	23(1)
	V(1)	3399(1)	0.3412(5)	2500	0.2500000(0)	2536(1)	0.2516(9)	9(1)
	V(2)	-636(1)	-0.0645(6)	2500	0.2500000(0)	6834(1)	0.6823(9)	9(1)
	O(1)	3081(2)	0.3127(10)	-12(4)	-0.0072(22)	1528(3)	0.1575(16)	21(1)
	O(2)	-1278(2)	-0.1259(11)	-40(4)	-0.0019(23)	6416(3)	0.6401(15)	15(1)
	O(3)	2851(2)	0.2853(12)	2500	0.2500000(0)	4296(4)	0.4301(21)	13(1)
	O(4)	358(2)	0.0380(12)	2500	0.2500000(0)	5796(4)	0.5832(24)	17(1)
	O(5)	4470(3)	0.4461(13)	2500	0.2500000(0)	2807(5)	0.2777(30)	25(1)
	O(6)	-412(2)	-0.0395(17)	2500	0.2500000(0)	8591(4)	0.8578(21)	17(1)
$RbTaV_2O_8$ (6)	Ta	1597(1)	0.15983(24	2500	0.2500000(0)	5044(1)	0.5049(6)	7(1)
	Rb	1194(1)	0.1206(4)	2500	0.2500000(0)	486(1)	0.0478(8)	22(1)
	V(1)	3393(1)	0.3434(9)	2500	0.2500000(0)	2537(1)	0.2523(13)	8(1)
	V(2)	-635(1)	-0.0625(9)	2500	0.2500000(0)	6836(1)	0.6840(14)	7(1)
	O(1)	3078(2)	0.3113(16)	-12(5)	0.001(4)	1526(4)	0.1518(26)	18(1)
	O(2)	-1276(2)	-0.1270(16)	-43(5)	0.006(4)	6427(3)	0.6358(27)	13(1)
	O(3)	2845(3)	0.2821(18)	2500	0.2500000(0)	4283(5)	0.4211(32)	14(1)
	O(4)	358(3)	0.0351(18)	2500	0.2500000(0)	5812(5)	0.576(4)	17(1)
	O(5)	4461(3)	0.4493(20)	2500	0.2500000(0)	2815(5)	0.283(5)	22(1)
	O(6)	-418(3)	-0.0380(29)	2500	0.2500000(0)	8587(5)	0.8586(33)	17(1)

Compound		x (× 10^4)	x*	y (× 10^4)	y*	z (× 10^4)	z*	$^{\#}U_{eq}$
$TlNbV_2O_8$ (3)	Nb	1195(1)	0.1602(5)	2500	0.2500000(0)	6881(1)	0.5059(8)	7(1)
	Tl	3807(1)	0.1131(4)	2500	0.2500000(0)	5582(1)	0.0550(4)	25(1)
	V(1)	2848(1)	0.3396(11)	2500	0.2500000(0)	9915(1)	0.2588(18)	8(1)
	V(2)	286(1)	-0.0641(10)	2500	0.2500000(0)	2947(1)	0.6831(15)	7(1)
	O(1)	2043(2)	0.3091(18)	70(5)	0.004(4)	6031(3)	0.1530(28)	13(1)
	O(2)	366(2)	-0.1294(22)	14(4)	0.000(4)	7587(3)	0.6398(24)	12(1)
	O(3)	1838(3)	0.2859(18)	2500	0.2500000(0)	8811(5)	0.436(4)	13(1)
	O(4)	566(3)	0.0353(18)	2500	0.2500000(0)	4856(5)	0.587(5)	14(1)
	O(5)	3668(3)	0.4470(20)	2500	0.2500000(0)	8704(5)	0.276(5)	19(1)
	O(6)	3819(3)	-0.0423(33)	-2500	0.2500000(0)	6891(5)	0.8642(31)	18(1)
$TlTaV_2O_8$ (7)	Ta	1596(1)	0.15957(22)	2500	0.2500000(0)	5049(1)	0.5048(4)	7(1)
	Tl	1125(1)	0.11337(23)	2500	0.2500000(0)	569(1)	0.05655(35)	28(1)
	V(1)	3411(1)	0.3408(9)	2500	0.2500000(0)	2566(2)	0.2552(13)	9(1)
	V(2)	-638(1)	-0.0646(8)	2500	0.2500000(0)	6848(2)	0.6855(14)	8(1)
	O(1)	3111(3)	0.3146(15)	-17(9)	-0.0018(31)	1527(7)	0.1524(22)	18(1)
	O(2)	-1275(3)	-0.1245(18)	-57(8)	0.0033(31)	6464(7)	0.6380(21)	14(1)
	O(3)	2843(4)	0.2855(16)	2500	0.2500000(0)	4311(9)	0.4309(30)	12(2)
	O(4)	330(5)	0.0339(15)	2500	0.2500000(0)	5778(10)	0.582(4)	17(2)
	O(5)	4481(5)	0.4479(19)	2500	0.2500000(0)	2878(10)	0.287(5)	20(2)
	O(6)	-399(5)	-0.0417(26)	2500	0.2500000(0)	8630(9)	0.8669(29)	17(2)

Compound		x (× 10^4)	x*	y (× 10^4)	y*	z (× 10^4)	z*	$^{\#}U_{eq}$
$CsNbV_2O_8$ (4)	Nb(1)	582(1)	0.0636(27)	2500	0.2500000(0)	1504(1)	0.1494(13)	9(1)
	Nb(2)	-752(1)	-0.0772(24)	2500	0.2500000(0)	3470(1)	0.3459(12)	9(1)
	Cs(1)	-5990(1)	-0.5987(22)	2500	0.2500000(0)	3868(1)	0.3894(11)	19(1)
	Cs(2)	-4177(1)	-0.4202(24)	2500	0.2500000(0)	1239(1)	0.1216(13)	28(1)
	V(1)	-1695(1)	-0.163(5)	7500	0.7500000(0)	405(1)	0.0486(23)	8(1)
	V(2)	1530(1)	0.159(5)	7500	0.7500000(0)	4444(1)	0.4489(20)	8(1)
	V(3)	-3223(1)	-0.350(5)	7500	0.7500000(0)	3394(1)	0.3243(23)	11(1)
	V(4)	3104(1)	0.291(4)	7500	0.7500000(0)	2053(1)	0.1962(23)	14(1)
	O(1)	-755(3)	-0.087(6)	4953(5)	0.482(7)	884(2)	0.090(4)	24(1)
	O(2)	-1807(4)	-0.162(11)	7500	0.7500000(0)	-640(2)	-0.053(4)	17(1)
	O(3)	2078(3)	0.209(7)	4939(5)	0.491(8)	2105(1)	0.2133(26)	18(1)
	O(4)	-442(4)	-0.031(11)	2500	0.2500000(0)	2376(2)	0.240(4)	13(1)
	O(5)	-2441(3)	-0.245(7)	4929(5)	0.500(10)	3106(2)	0.3093(33)	18(1)
	O(6)	1199(4)	0.147(14)	7500	0.7500000(0)	5406(2)	0.549(4)	19(1)
	O(7)	755(3)	0.055(7)	4965(4)	0.519(9)	3863(1)	0.388(4)	17(1)
	O(8)	-3431(4)	-0.321(8)	7500	0.7500000(0)	504(2)	0.077(6)	22(1)
	O(9)	3369(4)	0.340(6)	7500	0.7500000(0)	4546(2)	0.453(6)	18(1)
	O(10)	-2978(5)	-0.329(16)	7500	0.7500000(0)	4385(2)	0.4237(29)	30(1)
	O(11)	-5254(4)	-0.552(5)	7500	0.7500000(0)	2942(3)	0.287(4)	33(1)
	O(12)	3656(6)	0.318(16)	7500	0.7500000(0)	1218(3)	0.104(4)	41(1)

Compound		x (× 10^4)	x*	y (× 10^4)	y*	z (× 10^4)	z*	$^{\#}U_{eq}$
$CsTaV_2O_8$ (8)	Ta(1)	587(1)	0.0601(19)	2500	0.2500000(0)	1505(1)	0.1511(11)	8(1)
	Ta(2)	-754(1)	-0.0759(19)	2500	0.2500000(0)	3474(1)	0.3491(10)	8(1)
	Cs(1)	-5994(1)	-0.5968(28)	2500	0.2500000(0)	3867(1)	0.3858(14)	19(1)
	Cs(2)	-4161(1)	-0.4176(31)	2500	0.2500000(0)	1234(1)	0.1197(15)	28(1)
	V(1)	-1692(1)	-0.158(5)	7500	0.7500000(0)	407(1)	0.0440(22)	8(1)
	V(2)	1538(1)	0.165(5)	7500	0.7500000(0)	4446(1)	0.4465(21)	7(1)
	V(3)	-3220(1)	-0.314(4)	7500	0.7500000(0)	3389(1)	0.3295(18)	10(1)
	V(4)	3112(1)	0.320(5)	7500	0.7500000(0)	2049(1)	0.2032(19)	13(1)
	O(1)	-764(3)	-0.094(6)	4954(4)	0.481(9)	893(1)	0.093(5)	22(1)
	O(2)	-1783(4)	-0.170(9)	7500	0.7500000(0)	-633(2)	-0.0598(32)	17(1)
	O(3)	2082(3)	0.190(6)	4930(4)	0.526(8)	2100(1)	0.2037(34)	18(1)
	O(4)	-422(3)	0.021(12)	2500	0.2500000(0)	2383(2)	0.259(4)	14(1)
	O(5)	-2435(2)	-0.239(6)	4934(4)	0.500(8)	3104(1)	0.3062(29)	17(1)
	O(6)	1205(4)	0.134(11)	7500	0.7500000(0)	5414(2)	0.5429(33)	19(1)
	O(7)	768(2)	0.073(8)	4967(4)	0.503(11)	3869(1)	0.390(5)	16(1)
	O(8)	-3441(4)	-0.327(8)	7500	0.7500000(0)	498(2)	0.063(6)	21(1)
	O(9)	3376(3)	0.352(6)	7500	0.7500000(0)	4541(2)	0.466(6)	18(1)
	O(10)	-2974(4)	-0.275(11)	7500	0.7500000(0)	4383(2)	0.4292(24)	30(1)
	O(11)	-5256(4)	-0.520(5)	7500	0.7500000(0)	2945(2)	0.2953(33)	32(1)
	O(12)	3671(5)	0.361(12)	7500	0.7500000(0)	1216(2)	0.1145(34)	43(1)

$^{\#}U_{eq}$ is defined as one-third of the trace of the orthogonalized U_{ij} tensor.

Supplementary information

Table S2. Bond lengths (Å) obtained from single crystal and Rietveld analysis of powder XRD data (marked with *) and Bond valence sums (BVS) for ANbV$_2$O$_8$ (A = K(1), Rb(2), Tl(3), Cs(4)) and ATaV$_2$O$_8$ (A = K(5), Rb(6), Tl(7), Cs(8)) compounds.

Bonds	**1***	**2**	**2***	**3**	**3***	**5**	**5***	**6**	**6***	**7**	**7***
M-O(1) ×2	2.009(12)	1.983(2)	1.973(10)	1.995(3)	1.983(14)	1.983(3)	1.976(13)	1.984(3)	1.975(15)	1.963(4)	1.951(13)
M-O(2) ×2	1.986(12)	1.956(2)	1.978(10)	1.956(2)	1.959(14)	1.971(3)	1.979(13)	1.963(3)	1.974(15)	1.977(3)	1.974(13)
M-O(3)	1.998(17)	1.959(3)	1.976(16)	1.929(4)	1.952(22)	1.945(4)	1.971(19)	1.969(4)	1.964(23)	1.961(5)	1.975(20)
M-O(4)	1.987(16)	1.959(3)	1.939(15)	1.992(4)	1.978(19)	1.971(4)	1.998(18)	1.958(4)	1.958(23)	1.977(5)	1.981(19)
BVS	4.81	5.15	5.10	5.09	5.14	5.22	5.19	5.23	5.29	5.23	5.31
V(1)-O(1) ×2	1.742(12)	1.736(2)	1.728(10)	1.745(3)	1.727(14)	1.746(3)	1.736(13)	1.738(3)	1.733(16)	1.744(3)	1.725(13)
V(1)-O(3)	1.755(16)	1.771(3)	1.805(15)	1.776(4)	1.764(22)	1.766(4)	1.750(18)	1.761(4)	1.767(22)	1.762(5)	1.762(20)
V(1)-O(5)	1.649(18)	1.600(4)	1.573(17)	1.607(4)	1.591(23)	1.591(5)	1.628(20)	1.600(4)	1.597(26)	1.605(5)	1.610(22)
BVS	5.00	5.21	5.29	5.11	5.33	5.20	5.14	5..23	5.32	5.15	5.26
V(2)-O(2)×2	1.779(12)	1.750(2)	1.726(11)	1.771(2)	1.742(14)	1.772(3)	1.767(13)	1.751(3)	1.728(16)	1.750(3)	1.696(13)
V(2)-O(4)	1.753(15)	1.736(4)	1.760(15)	1.711(4)	1.696(19)	1.733(5)	1.730(17)	1.731(4)	1.743(22)	1.721(5)	1.723(18)
V(2)-O(6)	1.589(19)	1.607(4)	1.620(18)	1.611(4)	1.639(23)	1.603(4)	1.621(22)	1.600(5)	1.608(26)	1.622(6)	1.647(22)
BVS	5.10	5.18	5.21	5.14	5.23	5.09	5.04	5.24	5.41	5.17	5.51
A-O(1) ×2	2.923(23)	3.255(3)	3.343(16)	2.970(3)	3.31(12)	2.974(3)	3.011(27)	3.260(3)	3.318(31)	3.368(3)	3.493(2)
A-O(2) ×2	2.988(19)	3.100(3)	3.120(15)	3.131(3)	3.05(24)	3.042(4)	3.037(22)	3.090(3)	3.373(35)	2.978(4)	3.241(2)
A-O(2) ×2	3.165(24)	-	-	3.204(3)	-	3.152(4)	3.212(27)	-	-	-	-
A-O(3) ×2	3.242(14)	3.309(2)	3.318(11)	3.339(2	3.34(32)	3.255(3)	3.279(17)	3.321(2)	3.321(20)	3.381(3)	3.300(1)
A-O(5)	2.636(25)	2.971(4)	3.011(20)	2.723(4)	2.87(11)	2.709(6)	2.629(28)	2.981(4)	3.02(4)	2.796(5)	2.797(3)
A-O(6) ×2	3.075(11)	3.140(2)	3.165(12)	3.027(2)	3.07(41)	3.058(2)	3.036(12)	3.140(2)	3.167(23)	3.085(2)	3.059(1)
A-O(6)	-	2.915(4)	2.926(21)	-	2.84(24)	-	-	2.928(4)	2.92(4)	2.837(7)	2.820(4)
BVS	0.98	0.95	0.85	1.14	0.81	0.95	1.06	0.93	0.76	1.03	0.89

Table S2 (continued)

Bonds	**4**	**4***	**8**	**8***
M (1)-O(1) ×2	1.938(3)	1.951(16)	1.941(2)	1.949(14)
M (1)-O(2)	2.030(3)	2.026(23)	2.021(3)	2.028(21)
M (1)-O(3) ×2	1.996(2)	1.988(14)	1.989(2)	1.980(19)
M (1)-O(4)	1.914(3)	1.923(21)	1.913(3)	1.952(19)
BVS	5.14	5.11	5.32	5.21
M (2)-O(4)	1.912(3)	1.910(21)	1.914(3)	1.939(19)
M (2)-O(5) ×2	2.014(2)	2.040(16)	2.013(2)	2.015(14)
M (2)-O(6)	2.014(3)	2.010(22)	1.997(3)	2.022(19)
M (2)-O(7) ×2	1.929(2)	1.938(16)	1.939(2)	1.929(14)
BVS	5.15	5.02	5.25	5.00
V (1)-O(1) ×2	1.747(3)	1.727(16)	1.747(2)	1.726(14)
V (1)-O(2)	1.712(3)	1.693(24)	1.710(3)	1.697(21)
V (1)-O(8)	1.613(4)	1.620(24)	1.618(3)	1.626(22)
BVS	5.31	5.42	5.24	5.42
V (2)-O(6)	1.703(3)	1.697(23)	1.713(3)	1.714(19)
V (2)-O(7) ×2	1.761(2)	1.759(16)	1.757(2)	1.748(14)
V (2)-O(9)	1.617(4)	1.613(24)	1.617(3)	1.630(22)
BVS	5.39	5.28	5.18	5.18
V (3)-O(5) ×2	1.733(3)	1.752(16)	1.731(2)	1.728(14)
V (3)-O(10)	1.602(4)	1.613(24)	1.609(3)	1.604(22)
V (3)-O(11)	1.779(4)	1.756(23)	1.781(3)	1.761(20)
BVS	5.18	5.09	5.16	5.11
V (4)-O(3) ×2	1.728(3)	1.696(17)	1.734(2)	1.705(15)
V (4)-O(11)	1.788(4)	1.771(23)	1.791(3)	1.789(20)
V (4)-O(12)	1.595(4)	1.617(24)	1.594(4)	1.594(22)
BVS	5.25	5.45	5.19	5.40
Cs(1)-O(3)×2	3.282(2)	3.35(5)	3.286(2)	3.49(7)
Cs(1)-O(7)×2	3.239(2)	3.48(5)	3.227(2)	3.31(8)
Cs(1)-O(9)	3.029(3)	3.091(29)	3.037(3)	2.83(8)
Cs(1)-O(9)×2	3.146(2)	3.04(7)	3.142(1)	3.21(4)
Cs(1)-O(10)	3.277(4)	3.28(4)	3.280(3)	3.56(7)
Cs(1)-O(11)×2	3.362(3)	3.380(27)	3.360(2)	3.353(33)
BVS	1.1	1	1.1	1.01
Cs(2)-O(1)×2	3.564(3)	3.39(5)	3.533(2)	3.32(7)
Cs(2)-O(2)	3.480(4)	3.53(5)	3.514(3)	3.57(8)
Cs(2)-O(4)	3.385(3)	3.29(5)	3.395(3)	4.00(11)
Cs(2)-O(5)×2	3.370(2)	3.41(5)	3.374(2)	3.39(6)
Cs(2)-O(8)	3.111(4)	3.150(24)	3.104(3)	3.150(29)
Cs(2)-O(8)×2	3.214(2)	3.35(8)	3.209(2)	3.29(10)
Cs(2)-O(12)×2	3.423(3)	3.56(6)	3.423(2)	3.44(5)
BVS	0.91	0.7	0.86	0.8

Supplementary information

Table S3. Band assignment in the IR and Raman spectra of ANbV$_2$O$_8$ (A = K(1), Rb(2), Tl(3), Cs(4)) and ATaV$_2$O$_8$ (A = K(5), Rb(6), Tl(7), Cs(8)) compounds.

	1		2		3		4	
	IR	**RAMAN**	**IR**	**RAMAN**	**IR**	**RAMAN**	**IR**	**RAMAN**
VO_4 stretching (ν_s and ν_{as})	984(w)	973(s)	999(w)	998(s)	992(w)	983(m)	1001(w)	991(s)
	965(w)	961(w)	968(m)	964(s)	965(w)	957(m)	974(m)	993(s)
						938(s)	964(w)	
NbO_6 sym. Stretching (ν_1)	868(b)		889(b)	836(m)	858(b)	827(s)	899(b)	852(m)
(ν_2)	698(b)	661(m)	703(b)	665(s)	703(b)	655(s)	704(b)	688(s)
(ν_3)				451(w)		555(w)		554(w)
VO_4 Bending (ν_s and ν_{as})				496(w)		485(w)		493(w)
		416(m)		404(m)		395(m)		398(m)

	5		6		7		8	
	IR	**RAMAN**	**IR**	**RAMAN**	**IR**	**RAMAN**	**IR**	**RAMAN**
VO_4 stretching (ν_s and ν_{as})	990(w)	981(s)	984(m)	1004(m)	1017(w)	983(s)	1024(m)	978(s)
	972(m)	961(w)	964(w)	969(s)	972(s)	957(s)	979(m)	965(s)
NbO_6 sym. Stretching (ν_1)	879(b)	871(m)	858(w)	865(m)	862(w)	854(w)	852(w)	855(m)
(ν_2)	711(b)	688(s)	702(b)	692(s)	712(b)	655(s)	704(b)	670(w)
(ν_3)		557(w)		492(w)		553(w)		495(w)
VO_4 Bending (ν_s and ν_{as})		497(w)		490(w)		445(w)		
		422(m)		414(m)		404(s)		405(m)

Note: s = strong, m = medium, w = weak, b = broad.