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Supporting information for article:

Dehydration of microporous vanadosilicates: the case of VSH-13Na

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Figure S1 EDX analysis of VSH-13Na. The sample was Au-covered and C and Cl in the spectrum are from the surface of the sample holder.

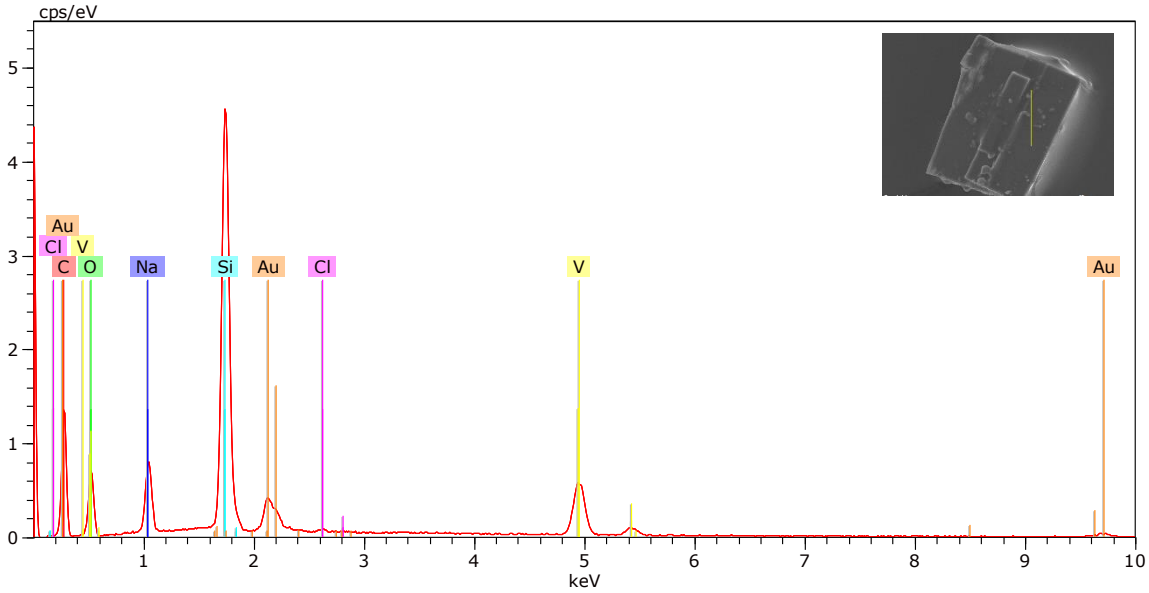

Table S1 Unit-cell parameters of VSH-13Na Crystal Y at different temperatures and after rehydration.

T (K)	*a* (Å)	*b* (Å)	*c* (Å)	*β* (°)	*V* (Å^3)
RT	14.364(4)	9.134(2)	10.373	90.056(7)	1360.9(7)
398	12.584(16)	9.525(13)	9.696(14)	90.10(4)	1162(3)
473	12.564(19)	9.511(15)	9.675(17)	90.11(4)	1156(5)
623	12.557(19)	9.482(15)	9.630(15)	90.11(3)	1147(5)
RT-rehydrated	14.58(5)	9.15(1)	10.20(1)	89.7(2)	1361(6)

Table S2 Selected bond distances for VSH-13Na.

	Sample X	Sample Y	
V coordination		RT	398 K
V1-OV1	1.592(19)	1.59(3)	1.59(4)
V1-O11	2.042(19)	1.95(2)	1.84(4)
V1-O12	1.961(18)	1.95(2)	2.05(4)
V1-O17	1.924(18)	2.02(2)	1.94(4)
V1-O18	2.030(18)	2.06(2)	1.97(4)
V2-V2A	1.174(14)	1.20(2)	0.96(4)
V2-OV2	1.75(3)	1.69(3)	1.64(5)
V2-O16	1.86(2)	1.89(3)	1.89(5)
V2-O14	1.961(19)	1.97(3)	2.01(5)
V2-O15	2.060(19)	2.02(2)	2.05(5)
V2-O13	2.033(18)	2.14(2)	1.98(5)
V2A-OV2/A	1.95(3)	1.95(3)	1.67(8)
V2A-O13	1.94(2)	1.93(3)	1.90(5)
V2A-O15	1.92(2)	1.98(3)	1.85(5)
V2A-O16	1.93(2)	2.01(3)	2.02(5)
V2A-O14	2.15(2)	2.06(3)	2.20(6)
Na coordination		RT	398 K
Na11-O18	2.45(2)	2.32(3)	2.33(6)
Na11-O12	2.34(2)	2.41(3)	2.60(6)
Na11-O17	2.45(2)	2.46(3)	2.44(6)
Na11-O11	2.54(2)	2.39(3)	2.44(6)

	Sample X	Sample Y	
Si coordination		RT	398 K
Si1-O18	1.580(16)	1.554(19)	1.62(3)
Si1-O42	1.548(16)	1.577(18)	1.62(3)
Si1-O23	1.583(17)	1.582(18)	1.62(3)
Si1-O34	1.572(17)	1.627(17)	1.66(3)
Si2-O22	1.624(17)	1.633(18)	1.65(3)
Si2-O17	1.625(16)	1.625(19)	1.62(3)
Si2-O33	1.652(16)	1.669(17)	1.67(3)
Si2-O42	1.615(15)	1.659(18)	1.64(3)
Si3-O22	1.587(17)	1.563(19)	1.58(3)
Si3-O31	1.627(16)	1.583(18)	1.60(3)
Si3-O12	1.578(16)	1.564(19)	1.52(3)
Si3-O43	1.607(17)	1.584(19)	1.58(3)
Si4-O11	1.587(16)	1.599(19)	1.64(3)
Si4-O23	1.599(16)	1.610(17)	1.63(3)
Si4-O32	1.593(17)	1.632(18)	1.66(3)
Si4-O43	1.613(16)	1.639(18)	1.66(3)
Si5-O13	1.590(16)	1.574(19)	1.56(3)
Si5-O21	1.618(17)	1.586(18)	1.56(3)
Si5-O44	1.636(17)	1.593(18)	1.61(3)
Si5-O31	1.649(16)	1.614(18)	1.60(3)
Si6-O14	1.582(16)	1.584(19)	1.62(3)
Si6-O32	1.621(17)	1.616(18)	1.68(3)

Na11-OW1	2.45(4)	2.57(5)	
Na11-O24			2.73(7)
Na12-O16	2.22(2)	2.35(3)	2.33(6)
Na12-O14	2.39(2)	2.33(3)	2.31(6)
Na12-O15	2.45(2)	2.42(3)	2.55(6)
Na12-O13	2.36(2)	2.56(3)	2.45(6)
Na12-OW2	2.36(4)	2.26(4)	
Na12-O22			2.86(7)
Na21-OV1	2.39(2)	2.44(3)	2.15(6)
Na21-O17	2.55(3)	2.37(3)	2.66(6)
Na21-O11	2.43(2)	2.55(3)	2.72(6)
Na21-OW1	2.45(4)	2.51(5)	
Na21-OW5	2.52(4)	2.54(5)	
Na21-OW3	2.47(4)	2.55(6)	
Na21-O21			2.82(7)
Na21-O44			2.56(6)
Na22-O13		2.61(4)	2.70(10)
Na22-OV2	2.42(4)	2.43(5)	2.62(12)
Na22-O15	2.49(3)	2.87(4)	2.47(10)
Na22-OW4	2.19(4)	1.92(5)	
Na22-OW6	2.37(5)	2.43(5)	
Na22-OW2	2.68(4)	2.83(4)	
Na22-O23			2.87(11)
Na22-O43			2.42(10)
Na2A-O15	2.05(5)	2.21(6)	2.79(16)
Na2A-OV2	2.03(5)	2.21(6)	2.60(18)
Na2A-O13	2.47(5)	2.42(6)	2.48(15)

Si6-O44	1.650(17)	1.648(18)	1.67(3)
Si6-O24	1.641(17)	1.650(18)	1.64(3)
Si7-O16	1.635(16)	1.605(19)	1.61(3)
Si7-O21	1.661(17)	1.647(18)	1.64(3)
Si7-O41	1.723(16)	1.625(18)	1.64(3)
Si7-O33	1.686(16)	1.660(17)	1.65(3)
Si8-O15	1.604(17)	1.552(18)	1.58(3)
Si8-O41	1.629(16)	1.600(18)	1.61(3)
Si8-O34	1.580(17)	1.571(18)	1.66(3)
Si8-O24	1.632(17)	1.614(18)	1.63(3)

Na2A-OW6	2.51(6)	2.50(6)	
Na2A-OW4	2.64(5)	2.61(7)	
Na2A-OW2	2.99(6)	2.86(6)	
Na2A-O23			2.81(16)
Na2A-O43			2.19(16)

Table S3 T-O-T angles for VSH-13Na.

T-O-T	RT Cryst. X	RT Cryst. Y	398 K Cryst. Y
Si5-O21-Si7	147.5(13)	152.1(16)	140(2)
Si3-O22-Si2	151.8(15)	154.2(16)	152(2)
Si1-O23-Si4	151.5(14)	151.4(16)	140(2)
Si8-O24-Si6	143.9(13)	146.3(15)	148(2)
Si3-O31-Si5	136.0(13)	137.8(16)	126(2)
Si6-O32-Si4	137.5(14)	137.0(16)	122(2)
Si2-O33-Si7	134.3(12)	134.5(15)	121(2)
Si8-O34-Si1	136.8(13)	136.9(15)	123(2)
Si8-O41-Si7	129.4(11)	129.7(14)	130(2)
Si1-O42-Si2	127.6(11)	130.4(14)	128(2)
Si3-O43-Si4	128.9(12)	130.2(14)	127(2)
Si5-O44-Si6	128.4(11)	132.1(14)	133(2)
Average	**137.8**	**139.4**	**132.5**