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Dehydration reactions in guano-derived minerals: the taranakite-to-francoanellite transformation

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S1. Further details on spectral fitting and data evaluation

The Origin-Pro 2021 software was used for fitting and data evaluation. Each spectrum was baseline-corrected to account for the eventual luminescence background. The Raman scattering intensity I was temperature-reduced according to the Bose-Einstein phonon population factor $n(\omega,T)$ (e.g., Kuzmany, 2009): $I_{reduced} = I_{measured}/[n(\omega,T)+1]$ where $n(\omega,T) = \left(e^{\hbar\omega/k_{\mathrm{B}}T}-1\right)^{-1}$, and $\hbar$, k_B, ω, and T are the reduced Plank's constant, Boltzmann's constant, phonon angular frequency (in s^{-1}) and temperature (in K), respectively. A pseudo-Voigt function ($PV = qL + (1-q)\,G$, where L and G are the Lorentz and Gauss peak-shape functions, respectively, and q is a weight coefficient) was used to obtain the position, full-width at half maximum and integrated intensity of the Raman peaks.

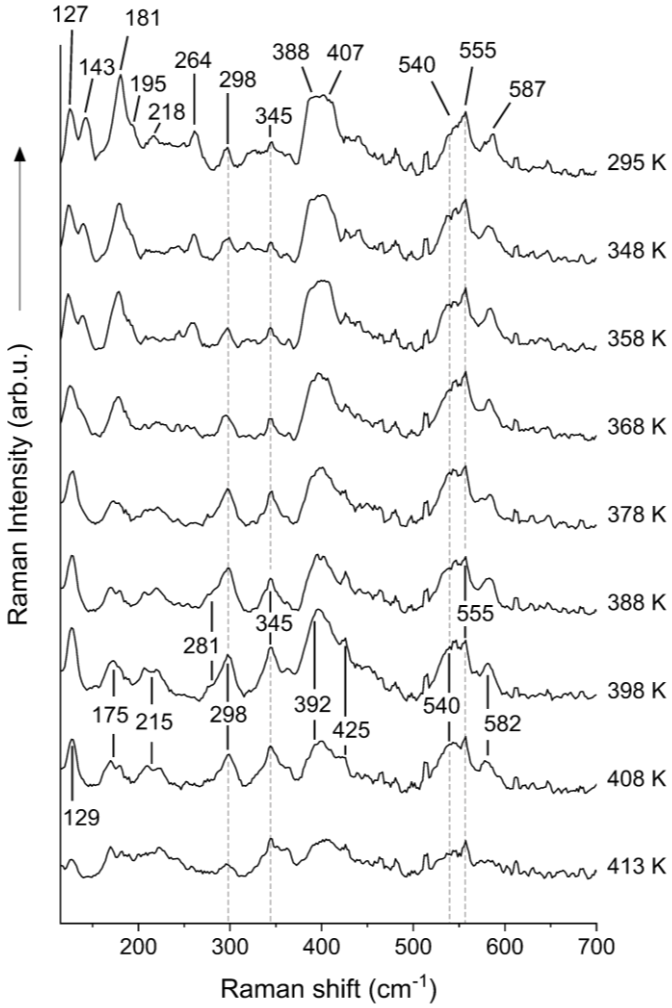

Figure S1. Selected Raman spectra collected in situ at increasing temperatures, displaying the external lattice modes and the phosphate bending modes in the range 100–700 cm^{-1}.

S2. Comment to Figure S1

In the range 100–700 cm^{-1} (**Fig. S1**), at T between 348 and 398 K, the spectra are subject to significant changes in intensity and band position of the lattice modes and phosphate bending modes. In these spectra, collected at HT conditions, only the most intense peaks are visible due to hampening of the Raman scattering intensity of already weak peaks by the borosilicate cover of the HT-stage. The wavenumbers of the observable peaks are marked, showing correspondence with the peak positions from the spectra of taranakite and francoanellite collected at room temperature without the HT-stage (see **Fig. 6a** and **Fig. 6c** in the manuscript). Between 295 K and 388 K, the taranakite Raman peaks near to 143 and 264 cm^{-1} greatly decrease in intensity and are not observed at higher T. The taranakite Raman peaks near to 298, 345, 540 and 555 cm^{-1} do not display significant changes in peak positions, and show an overall modest increase in intensity up to 398 K. At 398 K, the peaks are coincident with those of francoanellite as measured at ambient conditions, although the loss in intensity and overall resolution is noticeable for the reasons mentioned earlier. At T > 413 K, all peaks show a marked decrease in intensity and finally disappear at 478 K, pointing to the complete amorphisation of the material.

S3. Supplementary data to Figure 7.

francoanellite $\nu_1(PO_4)$

T (K)	Wavenumber (cm^{-1})	σ (Wavenumber) (cm^{-1})	FWHM (cm^{-1})	σ (FWHM) (cm^{-1})	Integrated Intensity (arb.u.)	σ (Integrated Intensity) (arb.u.)
358	930.848	3.240	22.322	3.251	46.823	17.535
363	931.314	1.400	13.150	4.494	27.149	13.887
368	933.259	0.800	16.874	1.382	88.405	16.499
373	933.684	0.486	16.929	1.252	112.312	16.904
378	933.358	0.409	16.920	0.886	158.154	15.359
383	933.493	0.289	16.869	1.068	148.855	15.251
388	933.330	0.226	17.086	1.095	174.019	15.241
393	933.375	0.259	18.614	0.620	198.681	10.269
398	933.490	0.230	19.129	0.659	238.885	5.778
403	933.370	0.255	19.575	0.728	200.560	5.121
408	932.758	0.307	21.800	0.885	207.140	5.794

Table S1. Supplementary data to Fig.7 (in the main document). Raman data for francoanellite $\nu_1(PO_4)$ peak. Collected under increasing temperature conditions.

taranakite $\nu_1(PO_4)$

T (K)	Wavenumber (cm^{-1})	σ (Wavenumber) (cm^{-1})	FWHM (cm^{-1})	σ (FWHM) (cm^{-1})	Integrated Intensity (arb.u.)	σ (Integrated Intensity) (arb.u.)
295	950.654	0.347	25.731	1.331	174.666	13.201
343	949.746	0.319	24.997	1.359	201.055	14.455
348	949.350	0.290	26.315	0.797	226.810	4.951
353	949.649	0.364	25.205	1.575	184.916	20.539
358	950.263	0.746	23.714	1.842	166.630	16.474
363	948.774	0.687	27.334	1.641	206.730	13.823
368	950.022	1.085	24.374	2.645	140.994	18.666
373	949.315	2.017	28.762	4.314	98.357	21.318
378	950.250	3.220	23.894	8.962	46.004	20.455
383	948.900	6.166	46.807	8.627	58.101	19.223
388	950.728	7.740	61.652	10.764	53.115	17.902

Table S2. Supplementary data to Fig.7 (in the main document). Raman data for taranakite $\nu_1(PO_4)$ peak. Collected under increasing temperature conditions.

S4. Supplementary data for HT-Raman analysis under isothermal conditions.

francoanellite $\nu_1(PO_4)$ – 353 K

time	Wavenumber (cm^{-1})	σ (Wavenumber) (cm^{-1})	FWHM (cm^{-1})	σ (FWHM) (cm^{-1})	Integrated Intensity (arb.u.)	σ (Integrated Intensity) (arb.u.)
3	*n.o.*	*n.o.*	*n.o.*	*n.o.*	*n.o.*	*n.o.*
8	*n.o.*	*n.o.*	*n.o.*	*n.o.*	*n.o.*	*n.o.*
13	*n.o.*	*n.o.*	*n.o.*	*n.o.*	*n.o.*	*n.o.*
18	935.906	4.448	22.885	8.046	32.812	17.395
23	933.893	1.450	14.817	3.872	32.868	8.475
28	934.659	1.377	14.822	3.460	36.111	8.514
33	934.604	1.378	16.375	3.459	42.962	9.576
38	934.053	0.927	14.060	2.472	48.552	8.217

43	934.209	0.911	15.715	2.357	58.176	8.867
48	935.136	0.869	15.483	2.128	62.474	8.847
53	934.812	0.870	15.431	2.210	64.331	9.345
58	934.105	0.735	14.927	1.909	65.408	8.228
63	934.712	0.784	15.536	1.938	66.213	8.418
68	934.525	0.764	15.275	1.977	68.401	8.802
73	934.939	0.807	16.491	2.008	78.326	10.081
78	934.449	0.665	14.517	1.738	74.559	8.627
83	934.853	0.691	15.061	1.764	75.091	8.705
88	935.099	0.753	16.726	1.895	86.657	10.395
93	934.850	0.713	15.923	1.691	73.729	8.007
98	934.451	0.665	15.937	1.639	78.337	8.320
103	934.884	0.757	16.628	1.757	77.750	8.757
108	934.934	0.606	14.436	1.531	77.313	8.075
113	934.566	0.689	16.099	1.698	78.814	8.593
118	934.797	0.681	15.856	1.710	82.330	9.043
123	935.058	0.828	18.051	1.806	80.688	9.188
128	934.827	0.766	17.735	1.716	84.191	9.095
133	934.459	0.656	16.424	1.659	90.186	9.672
138	934.222	0.659	15.899	1.696	86.446	9.442

Table S3. Raman data for francoanellite $\nu_1(PO_4)$ peak from time-resolved isothermal HT micro-Raman experiment at 353 K. Abbreviations: *n.o.* = not observed.

taranakite $\nu_1(PO_4)$ – 353 K

time	Wavenumber (cm^{-1})	σ (Wavenumber) (cm^{-1})	FWHM (cm^{-1})	σ (FWHM) (cm^{-1})	Integrated Intensity (arb.u.)	σ (Integrated Intensity) (arb.u.)
3	949.267	0.474	26.295	1.334	180.871	6.308
8	949.267	*	28.544	1.390	186.736	6.479
13	949.267	*	29.711	1.477	192.163	6.750
18	949.267	*	27.631	2.374	144.897	17.152
23	949.267	*	27.162	2.100	143.699	11.074

28	949.267	*	28.137	2.346	126.728	10.649
33	949.267	*	30.226	2.633	136.583	12.047
38	949.267	*	28.265	2.640	122.311	11.179
43	949.267	*	30.905	3.078	117.098	11.600
48	949.267	*	33.235	3.612	114.566	11.895
53	949.267	*	34.484	3.758	119.980	12.676
58	949.267	*	31.781	3.490	107.477	11.383
63	949.267	*	33.339	3.639	112.300	11.587
68	949.267	*	37.198	4.285	112.798	12.366
73	949.267	*	36.055	5.286	93.822	13.415
78	949.267	*	37.004	4.698	105.190	12.590
83	949.267	*	40.166	5.342	103.736	12.657
88	949.267	*	40.869	6.666	89.931	13.914
93	949.267	*	42.022	5.949	77.868	10.731
98	949.267	*	30.079	4.897	70.037	11.017
103	949.267	*	36.002	6.164	78.110	12.075
108	949.267	*	32.853	5.663	69.392	11.313
113	949.267	*	33.938	5.294	78.284	11.616
118	949.267	*	40.240	7.299	77.206	12.853
123	949.267	*	39.104	7.936	71.107	12.416
128	949.267	*	37.128	8.544	64.768	13.920
133	949.267	*	28.277	6.745	51.982	12.894
138	949.267	*	30.462	7.289	51.213	12.286

Table S4. Raman data for taranakite $\nu_1(PO_4)$ peak from time-resolved isothermal HT micro-Raman experiment at 353 K. Abbreviations: *n.o.* = not observed.

francoanellite $\nu_1(PO_4)$ – 358 K

time	Wavenumber (cm^{-1})	σ (Wavenumber) (cm^{-1})	FWHM (cm^{-1})	σ (FWHM) (cm^{-1})	Integrated Intensity (arb.u.)	σ (Integrated Intensity) (arb.u.)
3	*n.o.*	*n.o.*	*n.o.*	*n.o.*	*n.o.*	*n.o.*
8	930.565	5.786	32.587	11.040	37.561	16.071

13	936.193	2.554	24.451	4.676	61.439	16.721
18	935.525	1.041	16.907	2.531	60.035	9.471
23	935.554	0.766	16.168	1.826	76.580	8.915
28	936.345	0.704	16.973	1.607	99.760	10.123
33	935.946	0.544	15.871	1.367	103.699	9.098
38	936.344	0.547	16.710	1.288	117.103	9.582
43	936.431	0.514	16.797	1.262	124.629	9.738
48	935.878	0.445	15.991	1.093	131.471	9.080
53	935.866	0.414	16.082	1.048	144.334	9.439
58	935.825	0.384	16.405	0.959	157.284	9.359
63	936.194	0.362	15.844	0.958	158.318	9.445
68	936.039	0.340	15.969	0.888	163.199	8.935
73	936.160	0.345	16.241	0.877	171.879	9.353
78	935.997	0.343	16.304	0.900	174.514	9.517
83	936.047	0.326	15.921	0.841	174.430	9.018
88	936.081	0.292	15.499	0.810	177.197	8.891
93	935.963	0.303	16.011	0.747	171.567	7.904
98	936.045	0.291	15.772	0.787	185.890	8.975
103	936.057	0.280	16.242	0.749	183.637	8.347
108	936.150	0.284	15.657	0.803	190.337	9.434
115	935.967	0.266	15.614	0.704	186.968	8.153
120	935.885	0.278	15.759	0.753	196.520	9.124
125	936.080	0.275	16.216	0.742	188.681	8.531
130	936.081	0.285	16.369	0.774	199.763	9.380
135	935.828	0.258	15.316	0.730	191.669	8.695
140	935.854	0.264	15.922	0.726	186.110	8.256
145	935.977	0.269	16.332	0.738	197.990	8.810
150	935.877	0.261	16.140	0.707	187.871	8.100

Table S5. Raman data for francoanellite $\nu_1(PO_4)$ peak from time-resolved isothermal HT micro-Raman experiment at 358 K. Abbreviations: *n.o.* = not observed.

taranakite $\nu_1(PO_4)$ – 358 K

time	Wavenumber (cm^{-1})	σ (Wavenumber) (cm^{-1})	FWHM (cm^{-1})	σ (FWHM) (cm^{-1})	Integrated Intensity (arb.u.)	σ (Integrated Intensity) (arb.u.)
3	951.130	0.339	29.151	0.875	231.176	5.234
8	951.130	*	28.693	1.172	200.380	12.769
13	951.130	*	30.279	1.669	215.504	15.892
18	951.130	*	29.296	1.686	175.836	10.698
23	951.130	*	30.444	1.853	149.124	9.686
28	951.130	*	33.116	2.413	134.484	10.737
33	951.130	*	32.995	2.494	149.355	11.420
38	951.130	*	33.314	2.831	122.847	10.995
43	951.130	*	39.002	3.212	120.787	10.946
48	951.130	*	36.479	3.253	106.440	10.256
53	951.130	*	39.206	3.539	118.326	11.338
58	951.130	*	38.458	3.612	124.957	11.891
63	951.130	*	45.405	4.332	114.150	11.714
68	951.130	*	43.971	4.052	91.808	9.808
73	951.130	*	42.287	4.932	95.985	11.688
78	951.130	*	44.160	4.561	74.860	9.473
83	951.130	*	42.615	4.474	66.585	8.632
88	951.130	*	48.750	4.577	78.603	9.191
93	951.130	*	43.169	4.348	68.119	8.104
98	951.130	*	46.277	4.586	71.343	8.860
103	951.130	*	49.709	4.442	78.543	8.672
108	951.130	*	51.305	5.957	75.525	11.184
115	951.130	*	47.758	4.894	68.433	8.540
120	951.130	*	46.703	5.388	79.636	10.521
125	951.130	*	50.269	5.308	74.481	9.409
130	951.130	*	48.705	5.870	78.270	10.848
135	951.130	*	52.403	5.338	91.234	10.553

140	951.130	*	53.609	4.955	79.031	8.924
145	951.130	*	52.410	5.331	74.802	9.329
150	951.130	*	54.184	5.338	75.174	8.917

Table S6. Raman data for taranakite $\nu_1(PO_4)$ peak from time-resolved isothermal HT micro-Raman experiment at 358 K. Abbreviations: * = parameter locked.

francoanellite $\nu_1(PO_4)$ – 363 K

time	Wavenumber (cm^{-1})	σ (Wavenumber) (cm^{-1})	FWHM (cm^{-1})	σ (FWHM) (cm^{-1})	Integrated Intensity (arb.u.)	σ (Integrated Intensity) (arb.u.)
3	*n.o.*	*n.o.*	*n.o.*	*n.o.*	*n.o.*	*n.o.*
8	938.934	1.239	15.493	2.977	50.790	10.283
13	938.539	0.733	14.970	1.695	69.763	8.100
18	938.417	0.578	15.466	1.490	98.137	9.595
23	938.525	0.507	16.669	1.300	117.383	9.820
28	938.070	0.465	16.431	1.258	127.847	10.143
33	937.924	0.418	15.979	1.084	126.293	8.801
38	937.680	0.427	15.705	1.050	123.363	8.377
43	937.342	0.455	16.946	1.056	131.030	8.685
49	937.489	0.410	17.140	1.006	129.667	8.122
54	937.525	0.380	16.248	1.021	145.180	9.355

Table S7. Raman data for francoanellite $\nu_1(PO_4)$ peak from time-resolved isothermal HT micro-Raman experiment at 363 K. Abbreviations: *n.o.* = not observed.

taranakite $\nu_1(PO_4)$ – 363 K

time	Wavenumber (cm^{-1})	σ (Wavenumber) (cm^{-1})	FWHM (cm^{-1})	σ (FWHM) (cm^{-1})	Integrated Intensity (arb.u.)	σ (Integrated Intensity) (arb.u.)
3	951.863	0.431	35.143	1.124	242.244	5.867
8	951.863	*	37.390	2.308	194.825	12.442
13	951.863	*	39.430	2.806	156.489	10.742
18	951.863	*	42.518	3.397	124.875	11.156
23	951.863	*	48.659	5.120	120.879	12.667
28	951.863	*	48.891	5.246	111.430	12.695

33	951.863	*	49.702	5.384	110.003	11.785
38	951.863	*	45.068	5.211	100.248	11.328
43	951.863	*	43.949	5.752	107.708	12.450
49	951.863	*	52.083	6.214	99.018	11.215
54	951.863	*	56.663	9.258	101.293	14.120

Table S8. Raman data for taranakite $\nu_1(PO_4)$ peak from time-resolved isothermal HT micro-Raman experiment at 363 K. Abbreviations: * = parameter locked.

francoanellite $\nu_1(PO_4)$ – 373 K

time	Wavenumber (cm^{-1})	σ (Wavenumber) (cm^{-1})	FWHM (cm^{-1})	σ (FWHM) (cm^{-1})	Integrated Intensity (arb.u.)	σ (Integrated Intensity) (arb.u.)
3	934.485	0.671	17.389	2.135	135.412	34.906
8	934.294	0.426	15.894	1.078	129.568	8.735
13	934.484	0.420	17.427	1.069	158.506	10.279
18	934.677	0.365	16.611	1.010	167.854	10.098
23	934.493	0.325	16.238	0.945	166.344	9.361
28	934.568	0.301	16.788	0.915	178.615	9.547
33	934.352	0.299	16.512	0.860	181.297	9.179
38	934.137	0.301	16.560	0.864	175.784	8.921
43	934.265	0.297	16.588	0.895	186.997	9.802
48	934.100	0.299	17.155	0.949	191.051	10.378
53	934.137	0.301	16.560	0.864	175.784	8.921

Table S9. Raman data for francoanellite $\nu_1(PO_4)$ peak from time-resolved isothermal HT micro-Raman experiment at 373 K.

taranakite $\nu_1(PO_4)$ – 373 K

time	Wavenumber (cm^{-1})	σ (Wavenumber) (cm^{-1})	FWHM (cm^{-1})	σ (FWHM) (cm^{-1})	Integrated Intensity (arb.u.)	σ (Integrated Intensity) (arb.u.)
3	950.020	3.284	33.356	5.898	138.318	41.199
8	950.020	*	37.010	3.901	119.668	12.099
13	950.020	*	32.450	5.437	76.979	13.014
18	950.020	*	49.959	7.905	62.173	11.501

23	950.020	*	55.420	6.507	68.058	9.971
28	950.020	*	61.764	7.591	64.632	10.080
33	950.020	*	55.203	7.685	54.112	9.525
38	950.020	*	59.692	8.452	58.420	9.947
43	950.020	*	60.947	10.499	52.133	12.060
48	950.020	*	70.182	12.271	57.485	14.010
53	950.020	*	59.692	8.452	58.420	9.947

Table S10. Raman data for taranakite $\nu_1(PO_4)$ peak from time-resolved isothermal HT micro-Raman experiment at 373 K. Abbreviations: * = parameter locked.

S5. References

Kuzmany, H.: Solid-State Spectroscopy: An Introduction, Springer-Verlag Berlin, Heidelberg, Germany, 554 pp., doi:10.1007/978-3-642-01479-6, 2009.