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Sequential Single–Crystal–to–Single–Crystal Transformations Promoted by Gradual Thermal Dehydration in a Porous Metavanadate Hybrid

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Supporting Information

FIGURES:

TABLES:

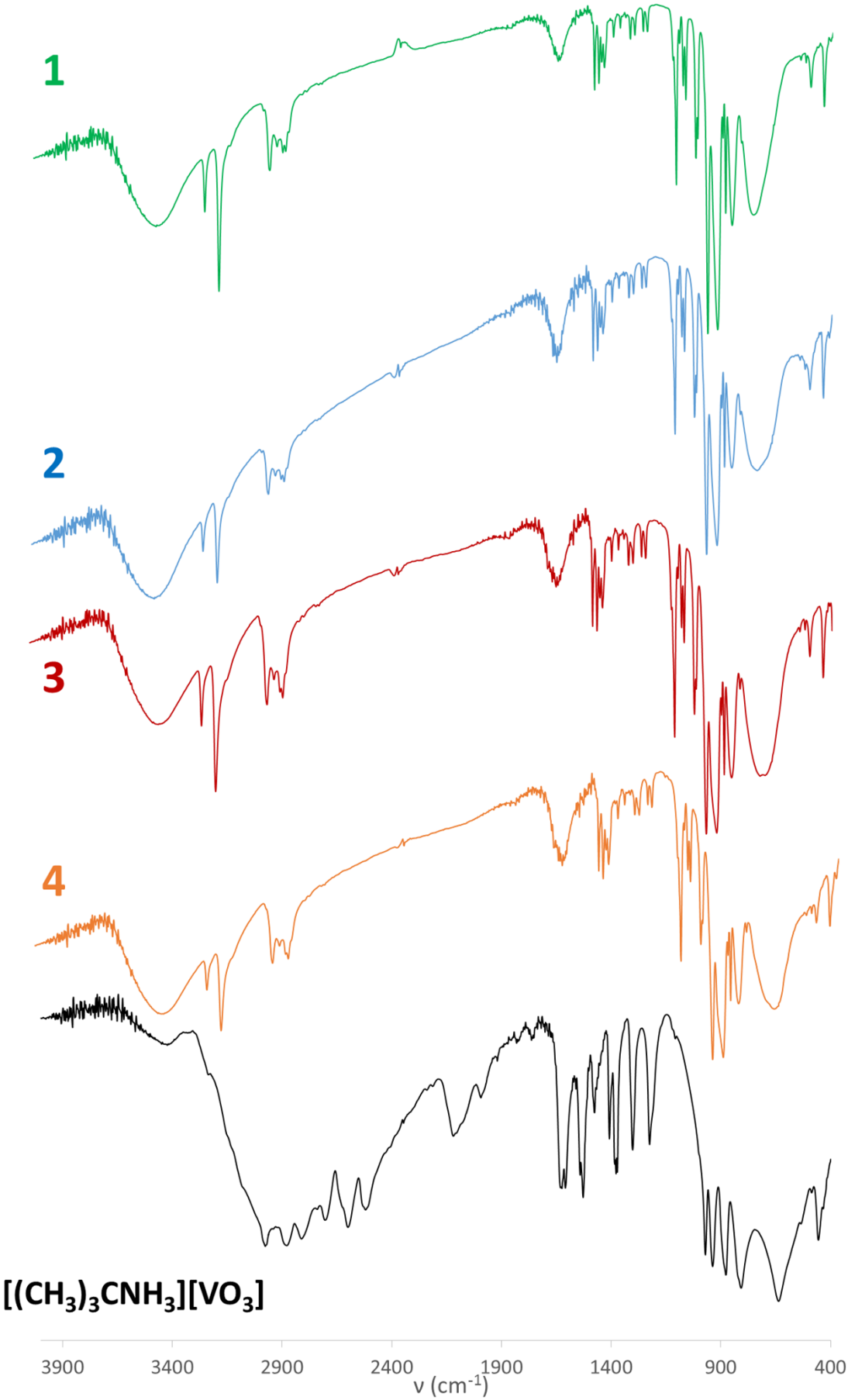

Fig. S1 FT-IR spectra of **1–4** compared to that of the $[(CH_3)_3CNH_3][VO_3]$ precursor.

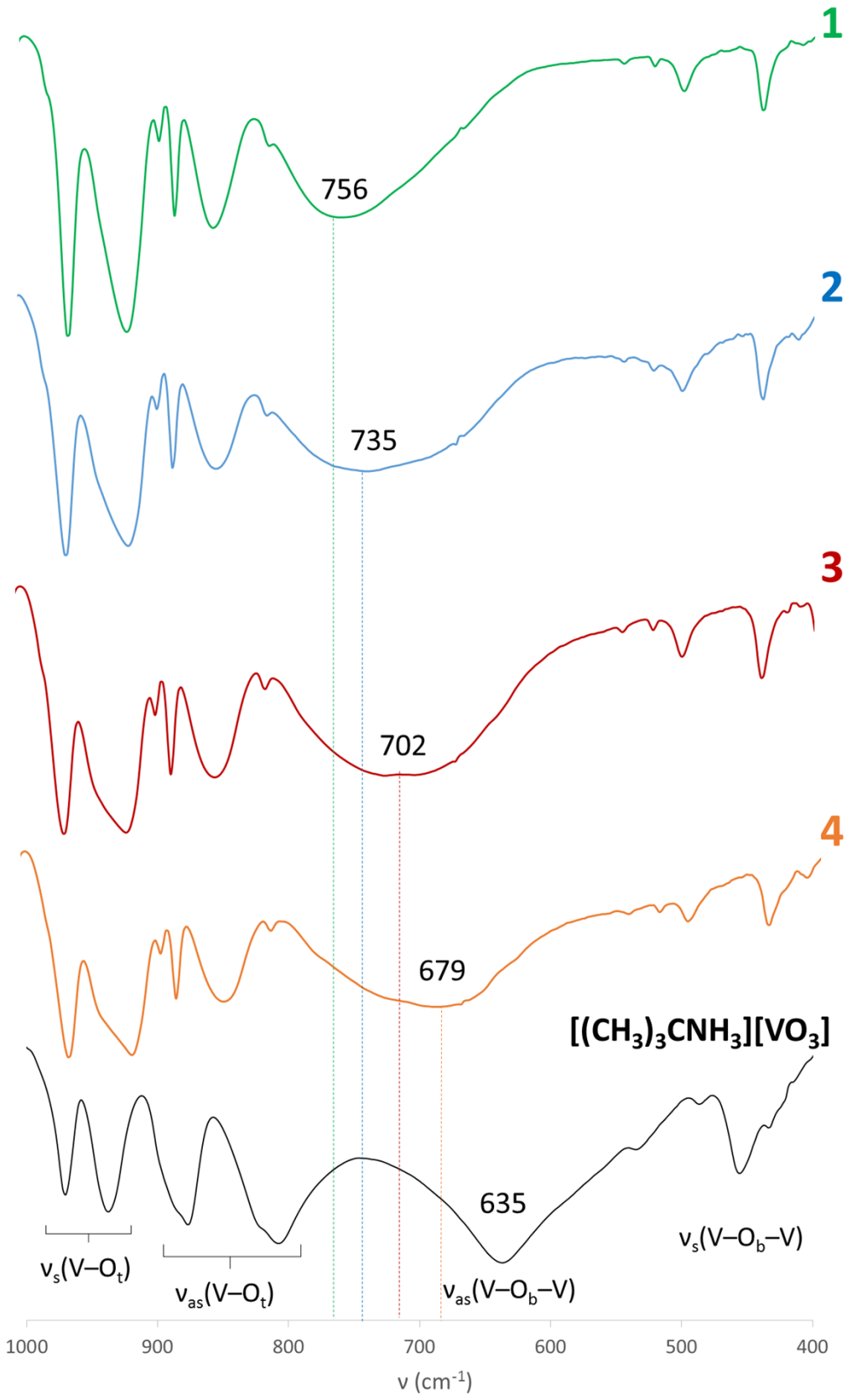

Fig. S2 Details of the inorganic region below 1000 cm^{-1} in the FT-IR spectra of compounds **1**–**4** and the $[(CH_3)_3CNH_3][VO_3]$ precursor. The bands corresponding to the $\nu_s(V–O_t)$ vibration in **1**–**4** are slightly shifted to lower wavenumbers, compared to the metavanadate precursor while those associated with the $\nu_{as}(V–O_t)$ vibration split into four weaker signals. Moreover, the position of the signal of strong intensity originating from the $\nu_{as}(V–O_b–V)$ vibration differs greatly from that of the precursor. This signal migrates to lower wavenumbers as the sample is dehydrated in such a way that it appears at 756 cm^{-1} for **1** while it is centred at 735, 702 and 679 cm^{-1} for **2**, **3** and **4**, respectively.

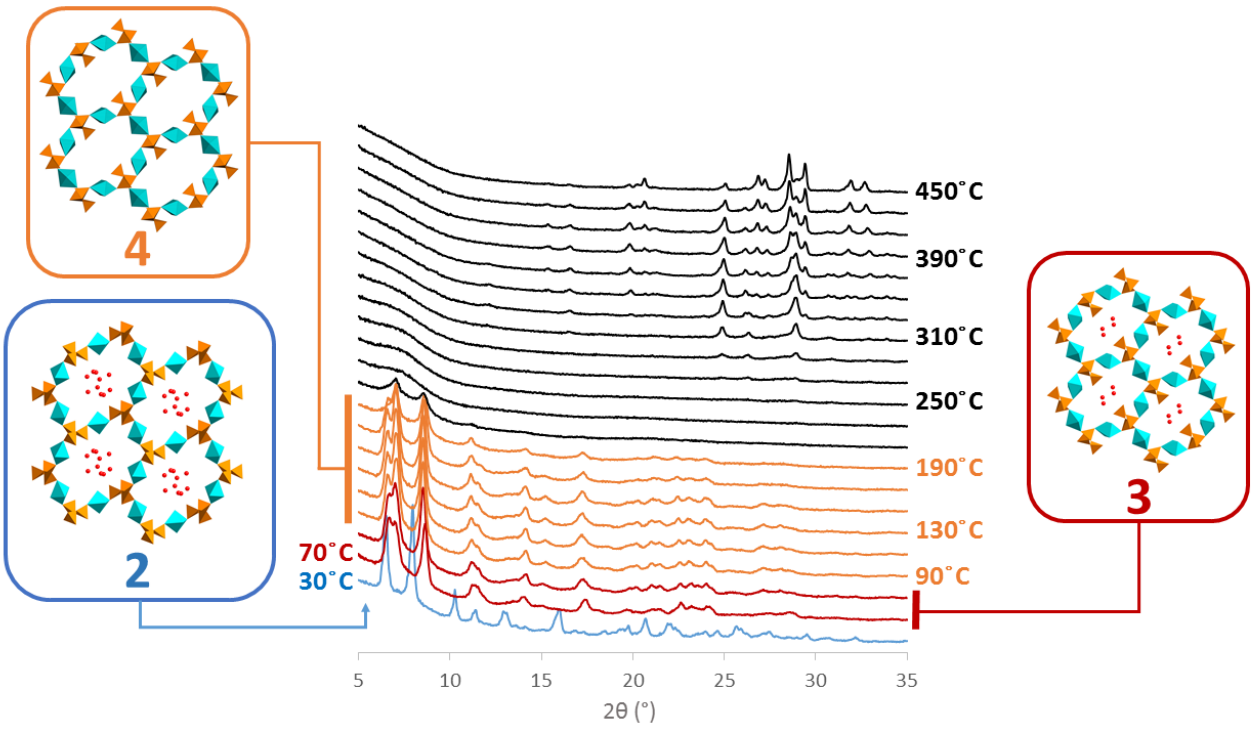

Fig. S3 Variable temperature PXRD patterns of **1** from 30 to 450 °C.

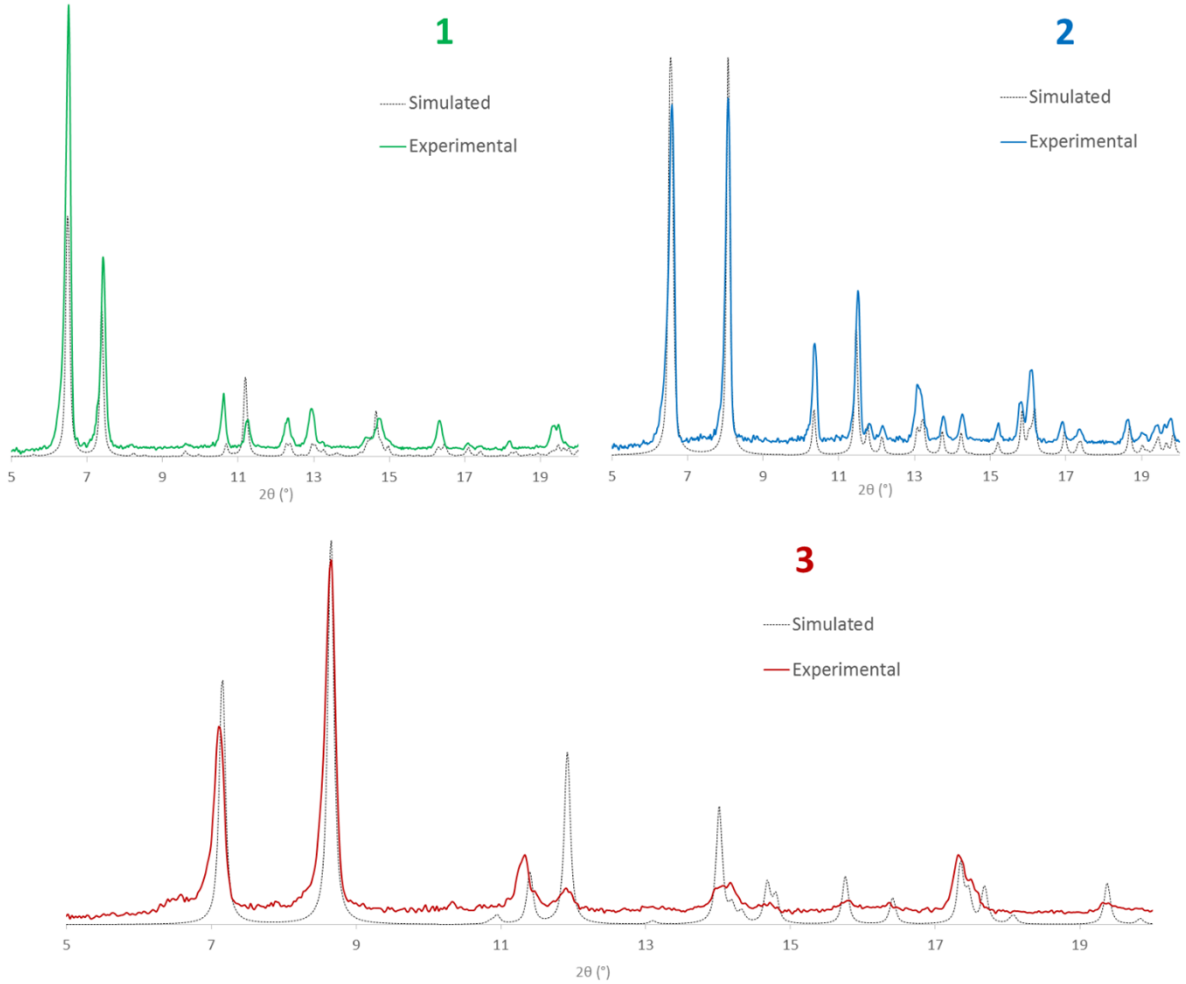

Fig. S4 Comparison between the experimental PXRD patterns of **1–3** and those simulated from single-crystal X-ray diffraction data**.**

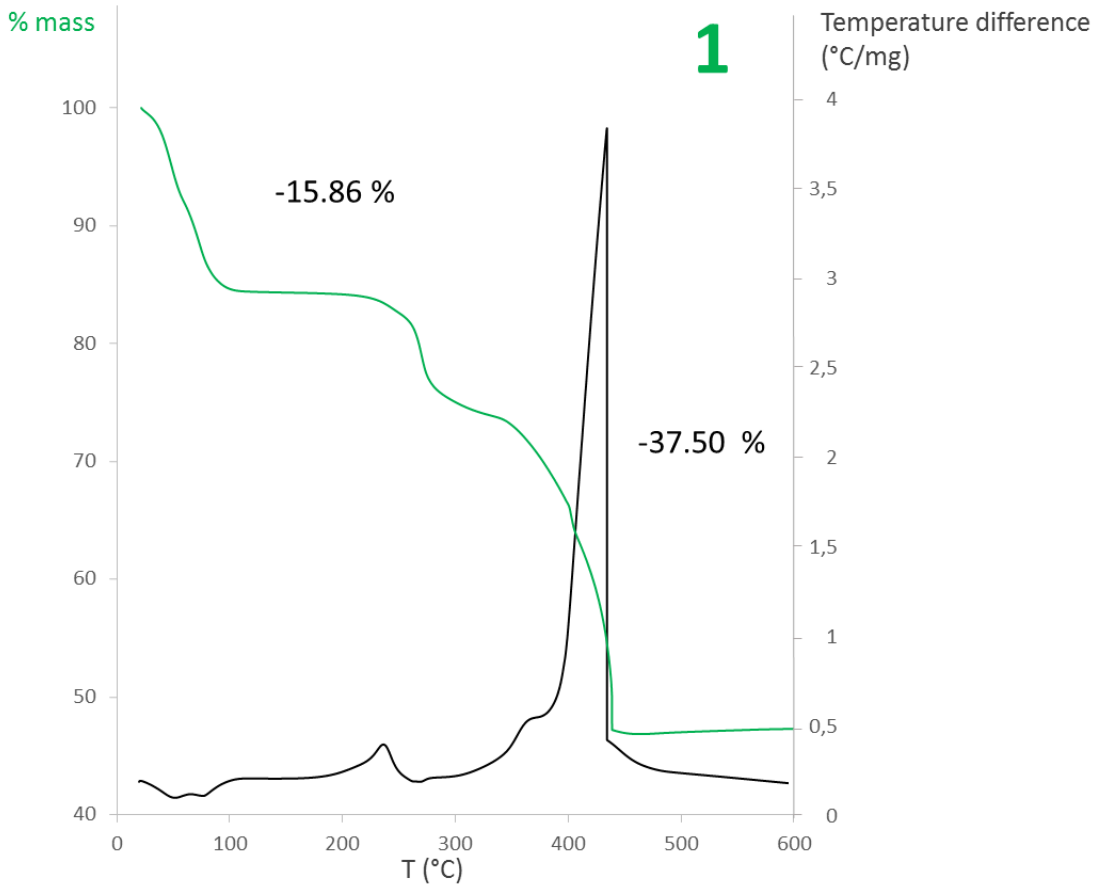

Fig. S5 TGA/DTA curves for **1.**

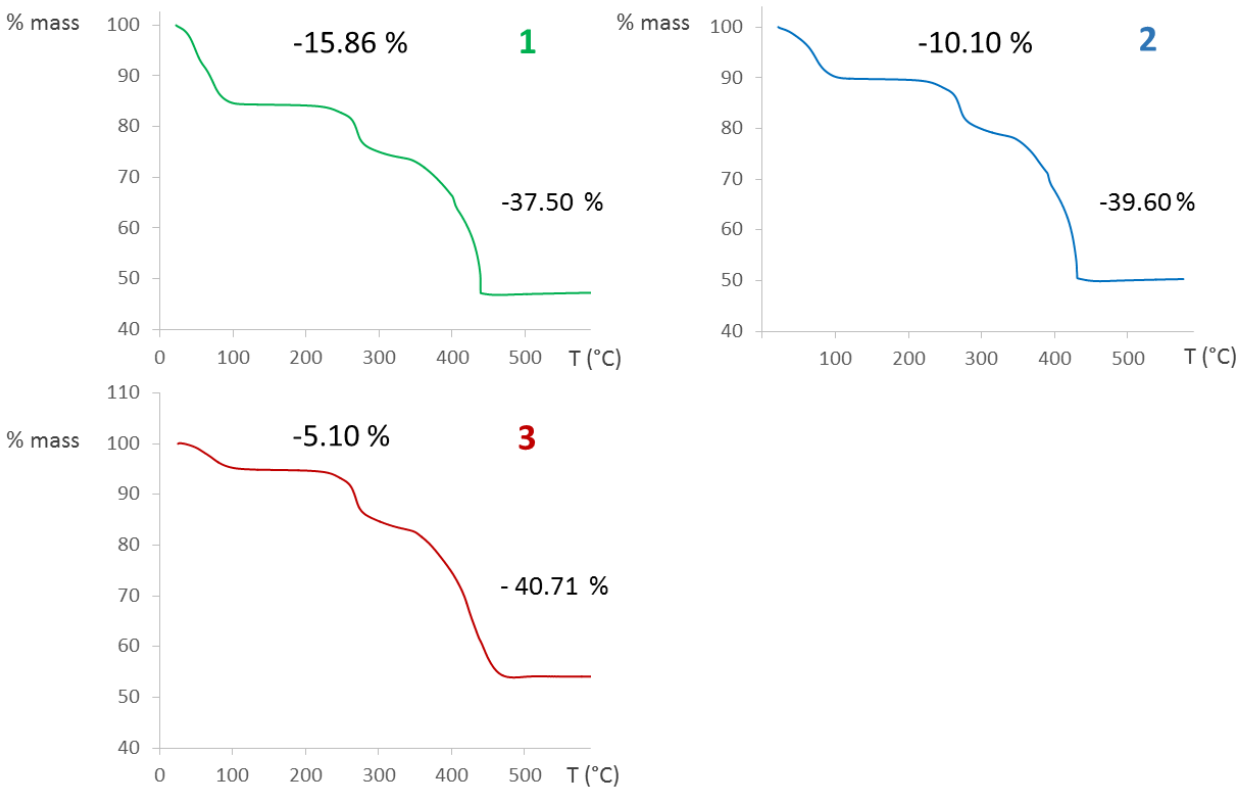

Fig. S6 Comparison between the TGA curves of **1–3.**

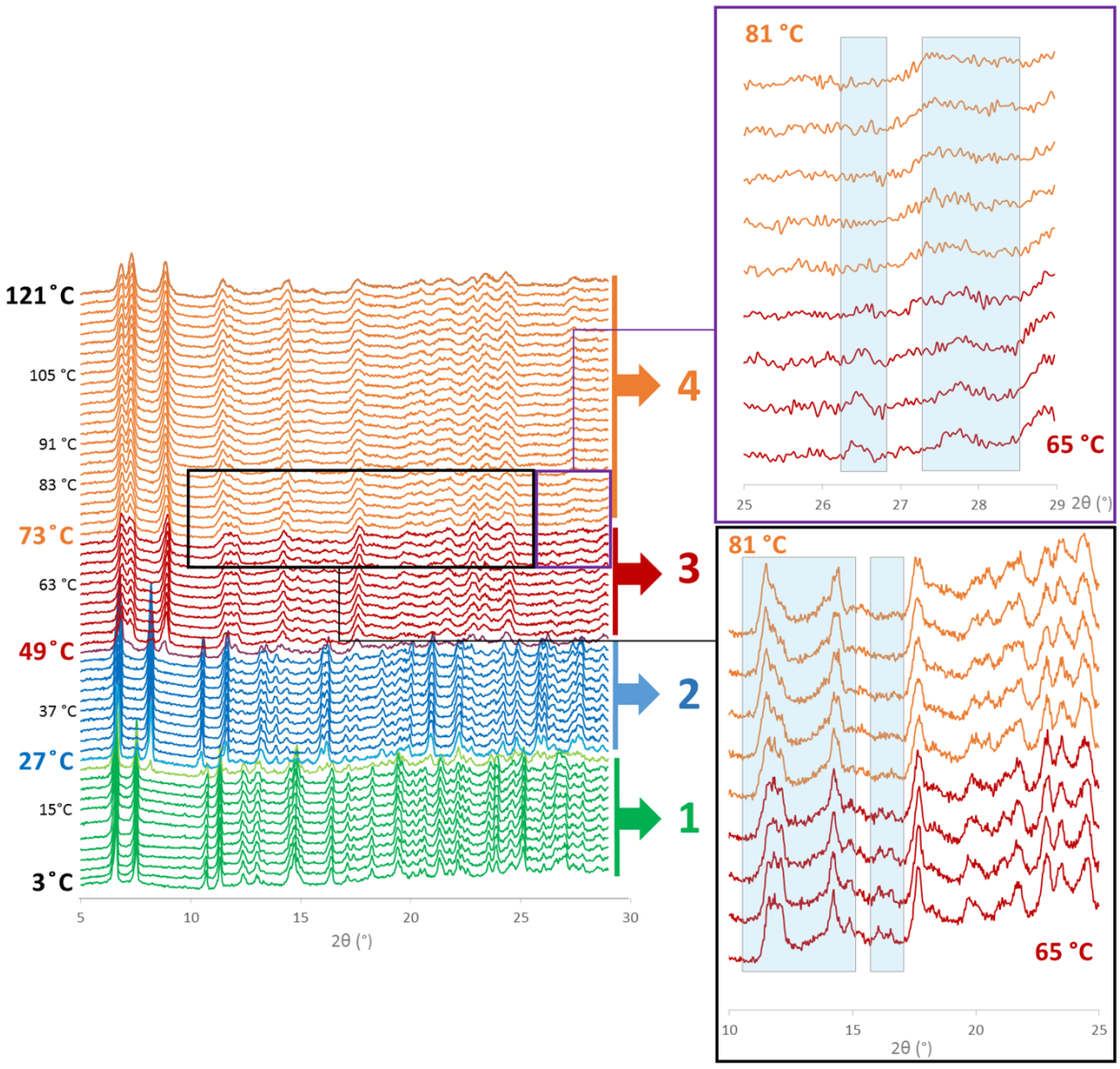

Fig. S7 Details of the variable temperature PXRD patterns in the temperature range 65–81 °C.

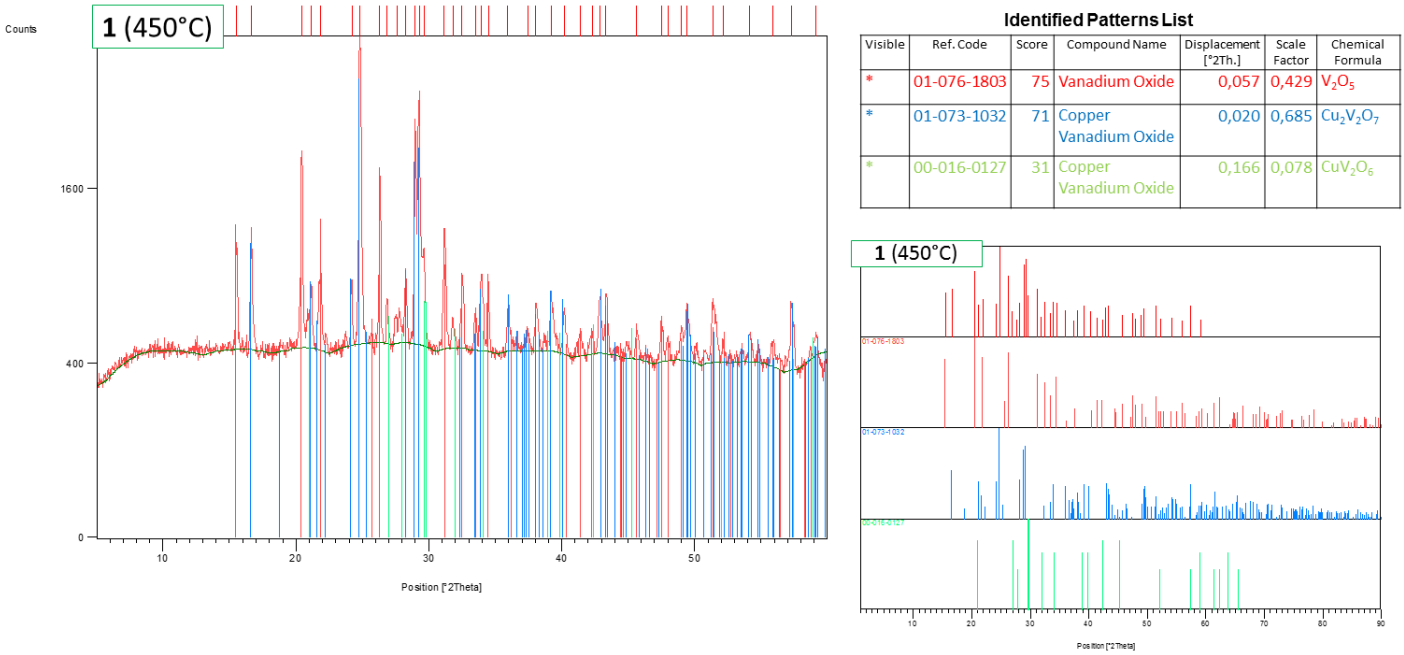

Fig. S8 Identification of the phases that constitute the final residue of the variable temperature PXRD experiments for compound **1.**

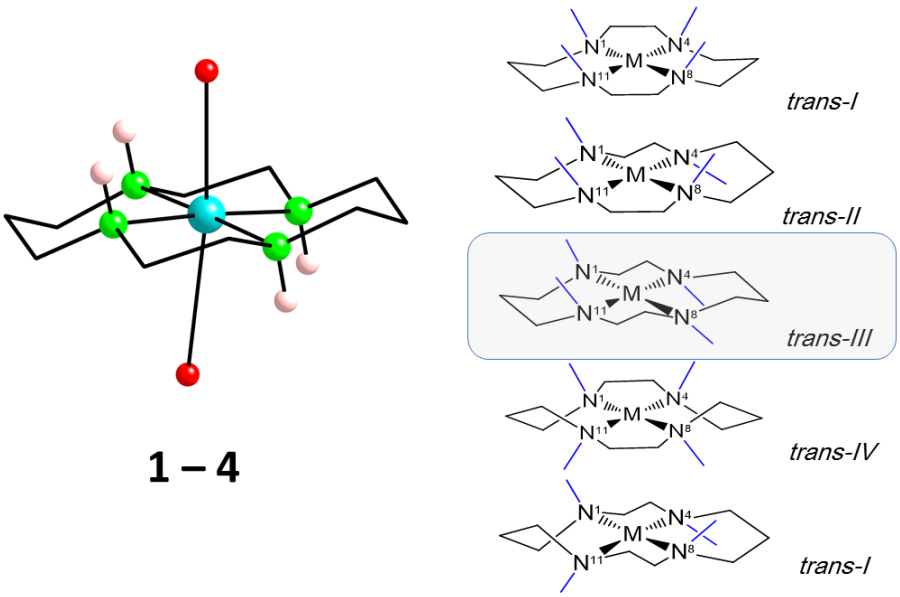

Fig. S9 Left: *trans–III* configuration in all the {Cu(cyclam)} complexes in compounds **1**-**4**; Right: possible configurations of the cyclam ligand.

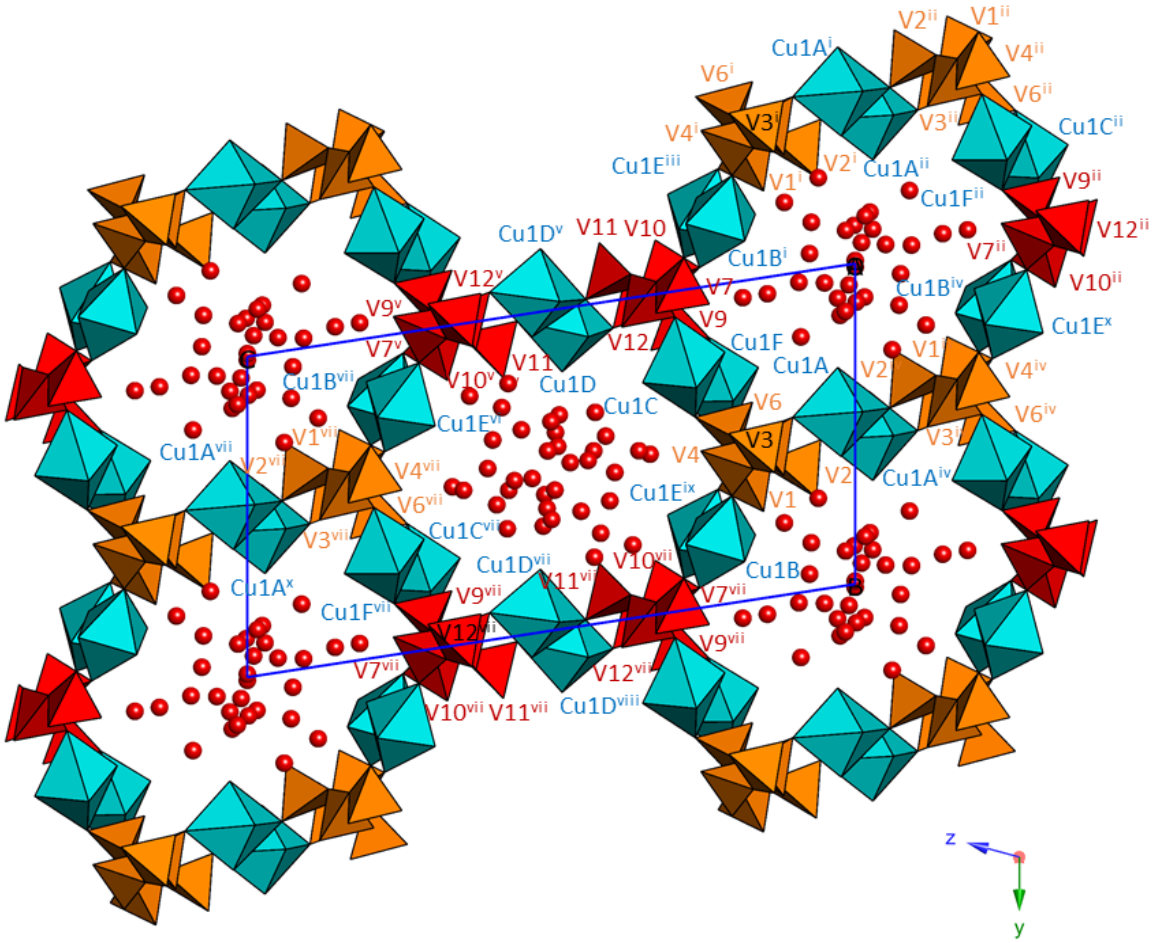

Fig. S10 Crystal packing of **1** with atom labeling. Symmetry codes: i) –1+x, –1+y, z; ii) –x, –y, –z; iii) x, –1+y, z; iv) 1–x, 1–y, –z; v) –x, –y, 1–z; vi) –x, 1–y, 1–z; vii) 1–x, 1–y, 1–z; viii) 1+x, 1+y, z; ix) 1+x, y, z; x) 2–x, 1–y, 1–z.

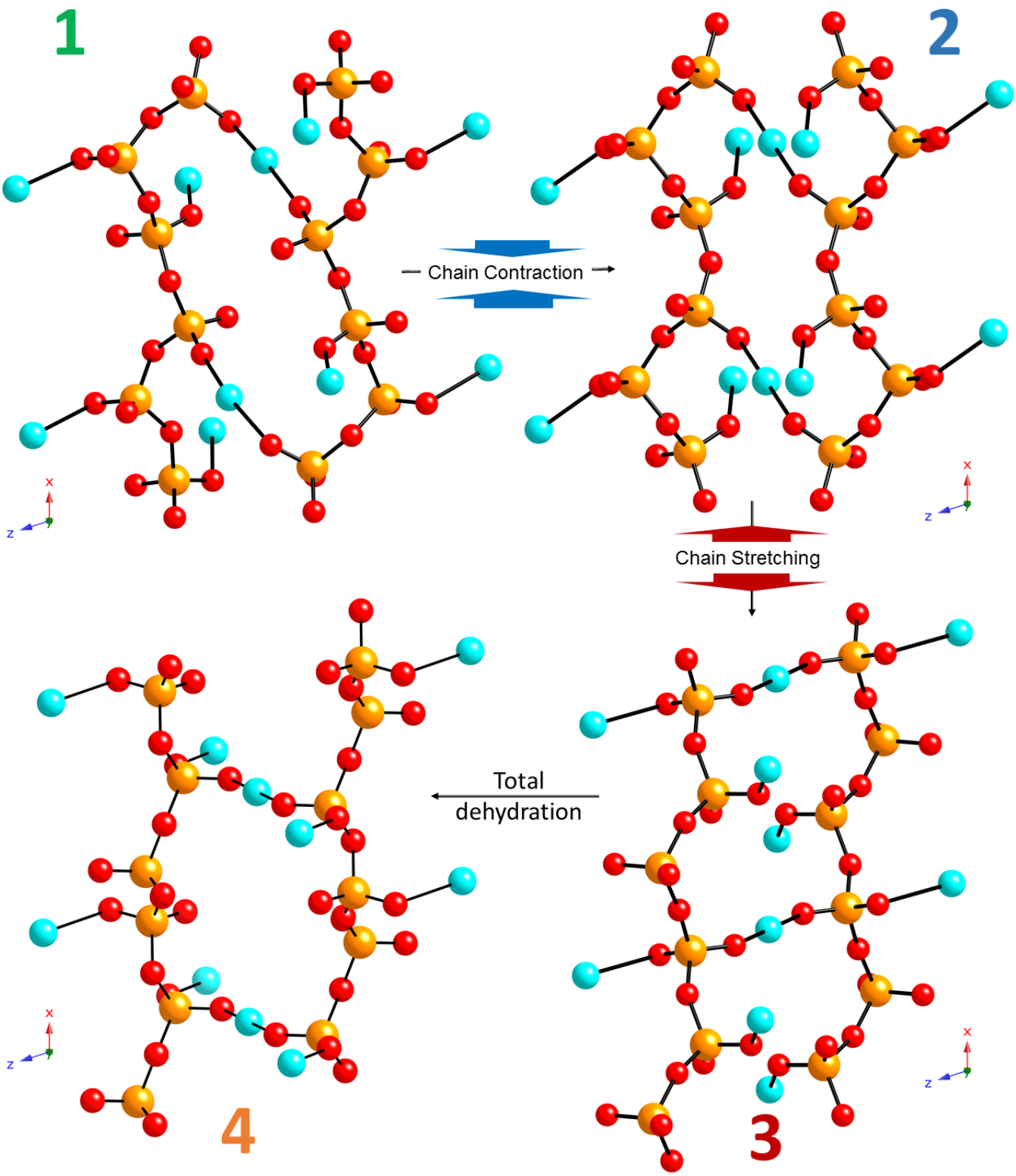

Fig. S11 Comparison between the metavanadate chains in **1**–**4**.

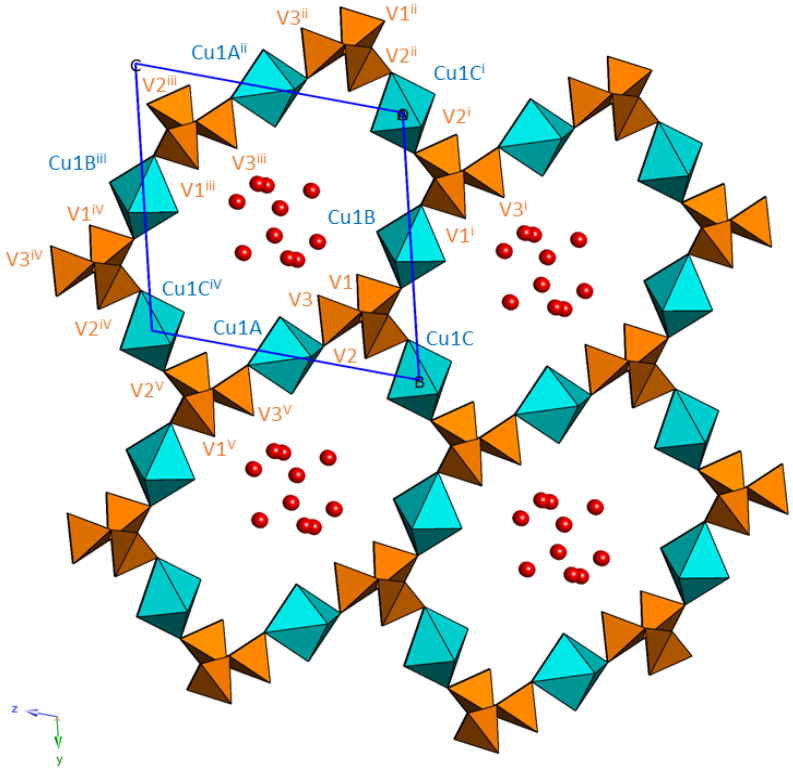

Fig. S12 Crystal packing of **2** with atom labeling. Symmetry codes: i) 1–x, 1–y, z; ii) x, 1+y, z; iii) 1–x, 1–y, 1–z; iv) x, y, 1+z; v) 1–x, 2–y, 1–z.

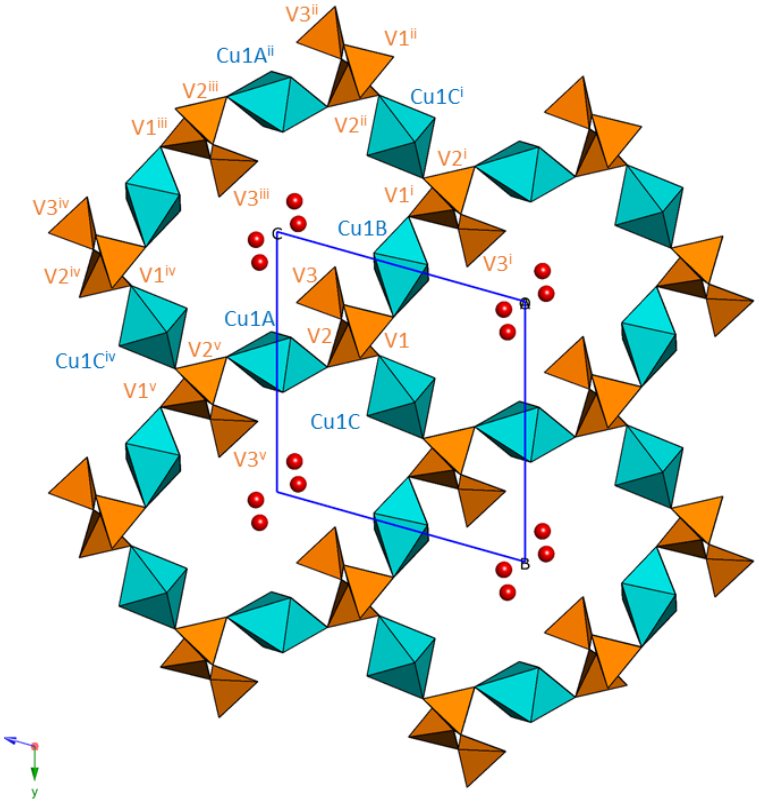

Fig. S13 Crystal packing of **3** with atom labeling. Symetry codes: i) –x, –y, 1–z; ii) –1+x, –1+y, z; iii) –x, –y, 2–z; iv) x, y, 1+z; v) 1–x, 1–y, 2–z.

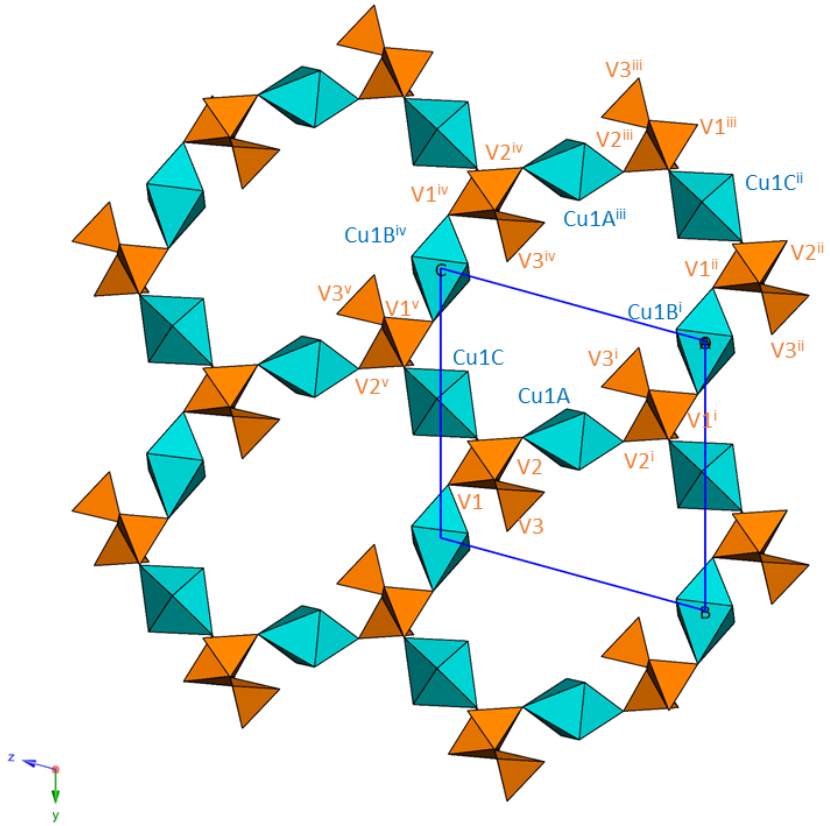

Fig. S14 Crystal packing of **4** with atom labeling. Symetry codes: i) 1–x, 1–y, 1–z; ii) –1+x, –1+y, 1–z; iii) 1–x, –y, 1–z; iv) x, –1+y, z; v) 3–x, 1–y, 2–z.

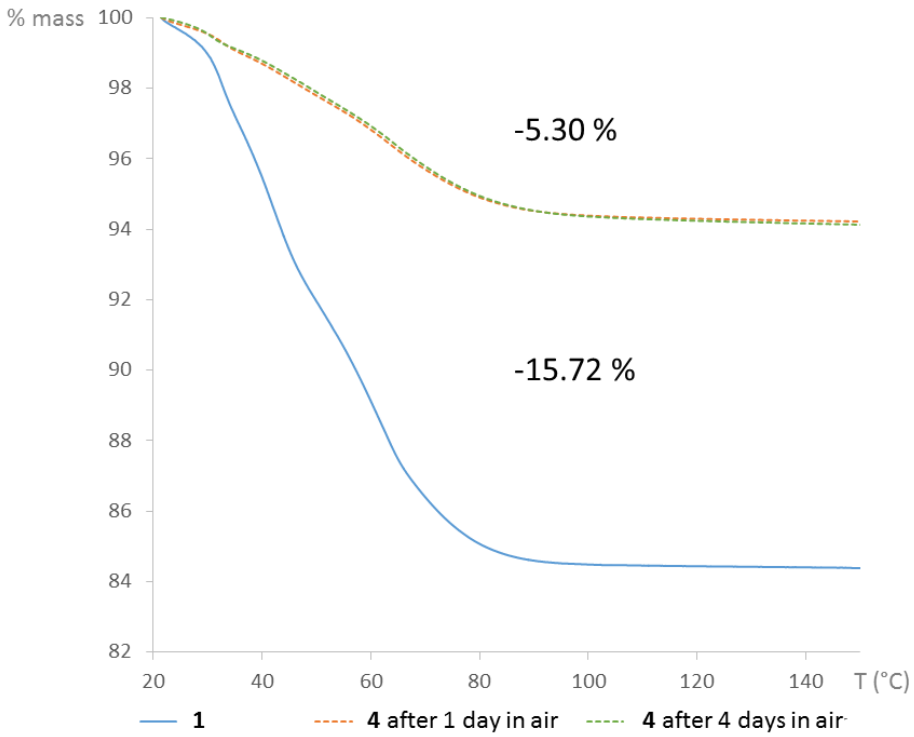

Fig. S15 Comparative TGA curves for the dehydration of **1** and the resulting anhydrous **4** after being exposed to air for 1 and 4 days**.**

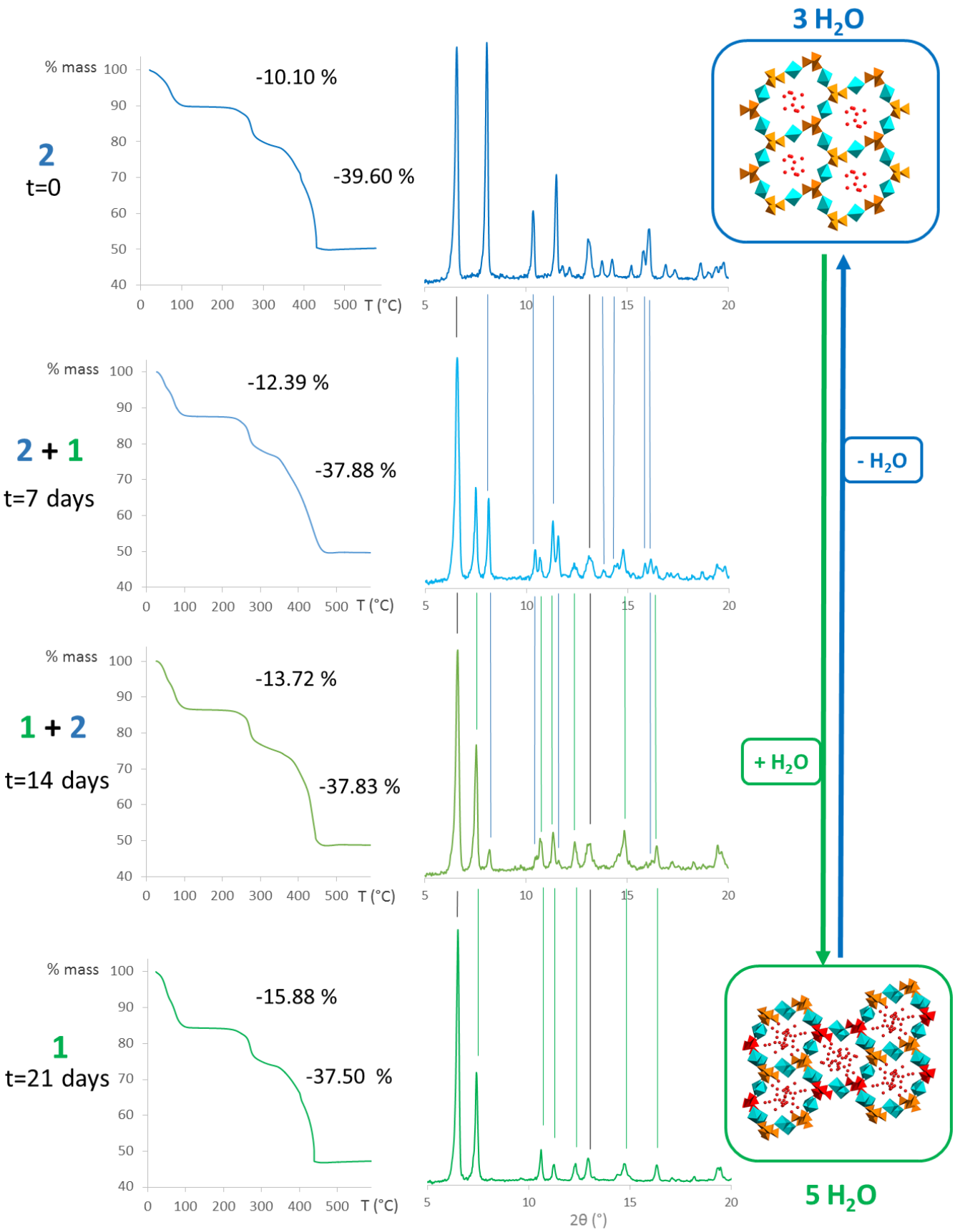

Fig. S16 Monitoring of the reversibility of the transformation of **2** into **1** by TGA and PXRD.

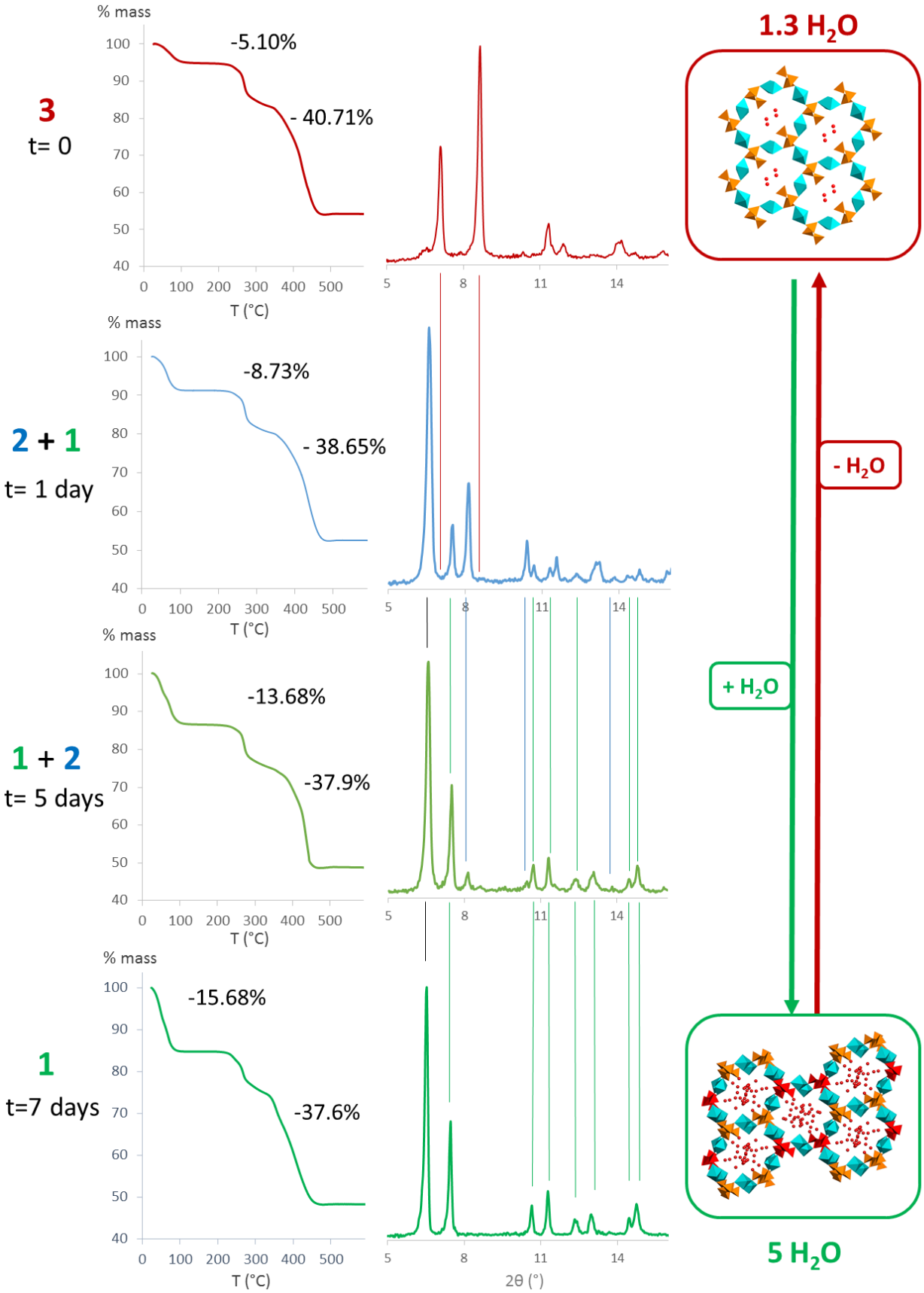

Fig. S17 Monitoring of the reversibility of SCSC transformation between **3** to **1** by TGA and PXRD.

Table S1 Selected bond lengths (Å) of the {Cu(cyclam)} complexes in compounds **1**–**4**.

1				**2**		**3**		**4**	
Cu1A–N1A	2.022(4)	Cu1D–N1D	2.015(4)	Cu1A–N1A	2.024(3)	Cu1A–N1A	2.009(8)	Cu1A–N1A	2.005(12)
Cu1A–N4A	2.007(4)	Cu1D–N4D	2.016(4)	Cu1A–N4A	1.998(2)	Cu1A–N4A	2.005(8)	Cu1A–N4A	2.000(12)
Cu1A–N8A	2.024(4)	Cu1D–N8D	2.014(4)	Cu1A–N1A^{i}	2.024(3)	Cu1A–N1A^{i}	2.009(8)	Cu1A–N1A^{i}	2.005(12)
Cu1A–N11A	2.007(4)	Cu1D–N11D	2.010(4)	Cu1A–N4A^{i}	1.998(2)	Cu1A–N4A^{i}	2.005(8)	Cu1A–N4A^{i}	2.000(12)
Cu1A–O5M	2.431(3)	Cu1D–O8M	2.411(3)	Cu1A–O3M	2.523(3)	Cu1A–O2	2.643(7)	Cu1A–O2M	2.493(10)
Cu1A–O2M^{i}	2.524(3)	Cu1D–O11M^{iv}	2.566(3)	Cu1A–O3M^{i}	2.523(3)	Cu1A–O2^{i}	2.643(7)	Cu1A–O2M^{i}	2.493(10)
Cu1B–N1B	2.012(4)	Cu1E–N1E	2.022(4)	Cu1B–N1B	2.017(3)	Cu1B–N1B	2.023(8)	Cu1B–N1B	1.996(12)
Cu1B–N4B	2.016(4)	Cu1E–N4E	2.011(4)	Cu1B–N4B	2.018(4)	Cu1B–N4B	2.003(8)	Cu1B–N4B	1.996(11)
Cu1B–N8B	2.030(4)	Cu1E–N8E	2.023(4)	Cu1B–N1B^{ii}	2.017(3)	Cu1B–N1B^{ii}	2.023(8)	Cu1B–N1B^{ii}	1.996(12)
Cu1B–N11B	1.994(4)	Cu1E–N11E	2.014(4)	Cu1B–N4B^{ii}	2.017(4)	Cu1B–N4B^{ii}	2.003(8)	Cu1B–N4B^{ii}	1.996(11)
Cu1B–O4M	2.335(3)	Cu1E–O1M	2.479(3)	Cu1B–O1M	2.493(3)	Cu1B–O1M	2.587(8)	Cu1B–O1	2.636(11)
Cu1B–O10M^{ii}	2.770(3)	Cu1E–O7M^{v}	2.450(3)	Cu1B–O1M^{ii}	2.493(3)	Cu1B–O1M^{ii}	2.587(8)	Cu1B–O1^{ii}	2.636(11)
Cu1C–N1C	2.021(3)	Cu1F–N1F	2.012(4)	Cu1C–N1C	2.016(2)	Cu1C–N1C	2.007(8)	Cu1C–N1C	2.003(11)
Cu1C–N4C	2.019(4)	Cu1F–N4F	2.029(4)	Cu1C–N4C	2.025(2)	Cu1C–N4C	2.038(8)	Cu1C–N4C	2.013(12)
Cu1C–N8C	2.016(4)	Cu1F–N8F	2.018(4)	Cu1C–N1C^{iii}	2.016(2)	Cu1C–N1C^{iii}	2.007(8)	Cu1C–N1C^{iii}	2.003(11)
Cu1C–N11C	2.019(3)	Cu1F–N11F	2.027(4)	Cu1C–N4C^{iii}	2.016(2)	Cu1C–N4C^{iii}	2.038(8)	Cu1C–N4C^{iii}	2.013(12)
Cu1C–O6M	2.713(3)	Cu1F–O3M	2.398(3)	Cu1C–O2M	2.523(3)	Cu1C–O2M	2.446(8)	Cu1C–O1M	2.420(10)
Cu1C–O12M^{iii}	2.457(3)	Cu1F–O9M	2.587(3)	Cu1C–O2M^{iii}	2.523(3)	Cu1C–O2M^{iii}	2.446(8)	Cu1C–O1M^{iii}	2.420(10)

Symmetry codes: **1**: i) 1–x, 1–y, –z; ii) 1+x, 1+y, z; iii) 1+x, y, z; iv) –x, –y, 1–z; v) x, 1+y, z. **2**: i) 1–x, 2–y, 1–z; ii) 1–x, 1–y, z; iii) 1–x, 2–y, –z. **3**: i) 1–x, –1–y, 2–z; ii) –x, –y, 1–z; iii) 1–x, 1–y, 1–z. **4**: i) 1–x, 1–y, 1–z; ii) 2–x, 2–y, 2–z; iii) 2–x, 1–y, 2–z.

Table S2 Intermolecular N–H···O and C–H···O interactions in **1.**

Donor–H···Acceptor	D–H (Å)	H···A (Å)	D···A (Å)	D–H···A (°)
N1A–H1A···O2^{i}	0.98	2.05	3.026(5)	170
N1C–H1C···O61	0.98	1.95	2.896(4)	162
N1D–H1D···O8	0.98	2.05	3.005(5)	164
N1E–H1E···O8^{ii}	0.98	2.21	3.148(5)	161
N1F–H1F···O3	0.98	1.99	2.943(5)	163
N4A–H4A···O61	0.98	2.06	2.934(5)	147
N4B–H4B···O15W	0.98	2.08	3.046(5)	169
N4C–H4C···O101^{iii}	0.98	2.33	3.208(5)	148
N4D–H4D···O127^{iv}	0.98	2.21	3.014(5)	138
N4E–H4E···O1	0.98	1.98	2.931(5)	164
N4F–H4F···O89	0.98	2.38	3.346(5)	167
N8A–H8A···O5	0.98	1.98	2.959(5)	179
N8B–H8B···O5	0.98	2.03	2.984(5)	166
N8C–H8C···O127^{iii}	0.98	2.09	3.045(5)	164
N8D–H8D···O11^{iv}	0.98	2.29	3.210(5)	156
N8D–H8D···O14W^{v}	0.98	2.57	3.235(6)	126
N8E–H8E···O23	0.98	2.33	3.196(5)	146
N8F–H8F···O9	0.98	1.87	2.836(5)	170
N11A–H11A···O3^{i}	0.98	2.12	2.976(5)	146
N11B–H11B···O10^{vi}	0.98	1.93	2.903(5)	173
N11C–H11C···O56	0.98	2.23	3.185(4)	165
N11D–H11D···O9	0.98	1.97	2.863(5)	151
N11E–H11E···O25W	0.98	2.22	3.075(5)	145
N11F–H11F···O12	0.98	2.41	3.243(5)	143
C7F–H7FB···O3M	0.97	2.54	3.152(6)	121
C10A–H10B···O30W^{i}	0.97	2.54	3.474(6)	163
C10B–H10E···O56	0.97	2.32	3.282(6)	169
C10C–H10J···O4	0.97	2.57	3.513(6)	164
C12F–H12C···O61^{vii}	0.97	2.60	3.440(5)	146
C12F–H12D···O9M	0.97	2.57	3.184(5)	121
C12C–H12J···O7	0.97	2.59	3.449(5)	148
C13F–H13C···O2M	0.97	2.50	3.441(6)	163
C13C–H13J···O5M	0.97	2.46	3.385(5)	158
C13E–H13K···O29W	0.97	2.63	3.503(6)	150
C9A–H9AA···O3^{i}	0.97	2.65	3.330(5)	127
C5A–H5AB···O12^{iii}	0.97	2.62	3.467(5)	146
C14B–H14F···O21W	0.97	2.65	3.441(7)	139
C14C–H14I···O12M^{iii}	0.97	2.46	3.113(5)	125
C3F–H3FA···O7	0.97	2.43	3.400(6)	173
C3E–H3EB···O89^{viii}	0.97	2.56	3.352(6)	139
C6E–H6EA···O2	0.97	2.59	3.508(7)	166

Symmetry codes: i) 1–x, 1–y, –z; ii) x, 1+y, z; iii) 1+x, y, z; iv) –x, –y, 1–z; v) 1–x, 1–y, 1–z; vi) 1+x, 1+y, z; vii) –1+x, y, z, viii) x, 1+y, z.

Table S3 Selected bond lengths (Å) of the metavanadate chains and the {Cu(cyclam)} complexes in **1**.

Inorganic block: chain A		Inorganic block: chain B	
V1–O1	1.648(3)	V7–O7	1.636(3)
V1–O1M	1.640(3)	V7–O7M	1.637(3)
V1–O12	1.810(3)	V7–O712	1.822(3)
V1–O6^{i}	1.811(3)	V7–O78	1.825(3)
V2–O2	1.643(3)	V8–O8	1.652(3)
V2–O2M	1.643(3)	V8–O8M	1.637(3)
V2–O23	1.821(3)	V8–O89	1.799(3)
V2–O12	1.805(3)	V8–O78	1.801(3)
V3–O3	1.654(3)	V9–O9	1.647(3)
V3–O3M	1.632(3)	V9–O9M	1.644(3)
V3–O34	1.805(3)	V9–O910	1.782(3)
V3–O23	1.793(3)	V9–O89	1.795(3)
V4–O4	1.632(3)	V10–O10	1.640(3)
V4–O4M	1.639(3)	V10–O10M	1.648(3)
V4–O45	1.827(3)	V10–O910	1.787(3)
V4–O34	1.821(3)	V10–O101	1.802(3)
V5–O5	1.653(3)	V11–O11	1.653(3)
V5–O5M	1.641(3)	V11–O11M	1.632(3)
V5–O45	1.803(3)	V11–O112	1.797(3)
V5–O56	1.797(3)	V11–O101	1.808(3)
V6–O6	1.782(3)	V12–O712^{i}	1.799(3)
V6–O6M	1.647(3)	V12–O12M	1.633(3)
V6–O56	1.819(3)	V12–O112	1.798(3)
V6–O61	1.648(3)	V12–O127	1.654(3)

Metal–organic Blocks			
Cu1A–O5M	2.431(3)	Cu1D–O8M	2.410(3)
Cu1A–O2M^{ii}	2.524(3)	Cu1D–O11M^{v}	2.566(3)
Cu1A–N1A	2.022(4)	Cu1D–N1D	2.015(4)
Cu1A–N4A	2.009(3)	Cu1D–N4D	2.016(4)
Cu1A–N8A	2.024(4)	Cu1D–N8D	2.014(4)
Cu1A–N11A	2.007(4)	Cu1D–N11D	2.010(3)
Cu1B–O4M	2.335(3)	Cu1E–O1M	2.479(3)
Cu1B–O10M	2.770(3)^{iii}	Cu1E–O7M^{vi}	2.450(3)
Cu1B–N1B	2.012(4)	Cu1E–N1E	2.022(4)
Cu1B–N4B	2.016(4)	Cu1E–N4E	2.011(4)
Cu1B–N8B	2.030(4)	Cu1E–N8E	2.023(4)
Cu1B–N11B	1.994(4)	Cu1E–N11E	2.014(4)
Cu1C–O6M	2.713(3)	Cu1F–O3M	2.399(3)
Cu1C–O12M	2.457(3)^{iv}	Cu1F–O9M	2.587(3)
Cu1C–N1C	2.021(3)	Cu1F–N1F	2.012(4)
Cu1C–N4C	2.019(4)	Cu1F–N4F	2.029(4)
Cu1C–N8C	2.016(4)	Cu1F–N8F	2.018(4)
Cu1C–N11C	2.019(4)	Cu1F–N11F	2.027(4)

Symmetry codes: i) –1 + x, y, z; ii) 1–x, 1–y, –z; iii) 1+x, 1+y, z; iv) 1+x, y, z; v) –x, –y, 1–z; vi) x, 1+y, z.

Table S4 Selected bond lengths (Å) of the metavanadate chains and the {Cu(cyclam)} complexes in **2.**

Inorganic Block		Metal–organic blocks	
V1–O1	1.6522(19)	Cu1A–O3M	2.523(3)
V1–O1M	1.6422(19)	Cu1A–O3M^{ii}	2.523(3)
V1–O12	1.6422(19)	Cu1A–N1A	2.024(3)
V1–O13	1.8185(19)	Cu1A–N4A	1.998(2)
V2–O2M	1.6424(18)	Cu1A–N1A^{ii}	2.024(5)
V2–O2	1.6452(18)	Cu1A–N4A^{ii}	1.998(2)
V2–O23	1.8096(18)	Cu1B–O1M	2.493(3)
V2–O12^{i}	1.8003(18)	Cu1B–O1M^{iii}	2.493(3)
V3–O3	1.6512(19)	Cu1B–N1B	2.017(3)
V3–O3M	1.6440(19)	Cu1B–N4B	2.018(4)
V3–O13	1.8185(19)	Cu1B–N1B^{iii}	2.017(3)
V3–O23	1.8096(18)	Cu1B–N4B^{iii}	2.018(4)
		Cu1C–O2M	2.523(3)
		Cu1C–O2M^{iv}	2.523(3)
		Cu1C–N1C	2.016(2)
		Cu1C–N4C	2.025(2)
		Cu1C–N1C^{iv}	2.016(2)
		Cu1C–N4C^{iv}	2.025(2)

Symmetry codes: i) –1+x, y, z; ii) 1–x, 2–y, 1–z; iii) 1–x, 1–y, z; iv) 1–x, 2–y, –z.

Table S5 Selected bond lengths (Å) of the metavanadate chains and the {Cu(cyclam)} complexes in **3.**

Inorganic Block		Metal–organic Blocks	
V1–O1	1.622(7)	Cu1A–O2	2.643(7)
V1–O1M	1.630(7)	Cu1A–O2^{ii}	2.643(7)
V1–O12	1.838(6)	Cu1A–N1A	2.009(8)
V1–O13	1.807(7)	Cu1A–N4A	2.005(8)
V2–O2M	1.634(7)	Cu1A–N1A^{ii}	2.009(8)
V2–O2	1.640(7)	Cu1A–N4A^{ii}	2.005(8)
V2–O23	1.770(7)	Cu1B–O1M	2.587(8)
V2–O12	1.821(6)	Cu1B–O1M^{iii}	2.587(8)
V3–O3	1.628(7)	Cu1B–N1B	2.023(8)
V3–O3M	1.633(7)	Cu1B–N4B	2.003(8)
V3–O23	1.799(7)	Cu1B–N1B^{iii}	2.023(8)
V3–O13^{i}	1.811(7)	Cu1B–N4B^{iii}	2.003(8)
		Cu1C–O2M	2.446(8)
		Cu1C–O2M^{iv}	2.446(8)
		Cu1C–N1C	2.007(8)
		Cu1C–N4C	2.038(8)
		Cu1C–N1C^{iv}	2.007(8)
		Cu1C–N4C^{iv}	2.038(8)

Symmetry codes: i) 1+x, y, z; ii) 1–*x*, –1–*y*, 2–*z*; iii) –*x*, –*y*, 1–*z*; iv) 1–*x*, 1–*y*, 1–*z*.

Table S6 Selected bond lengths (Å) of the metavanadate chains and the {Cu(cyclam)} complexes in **4.**

Inorganic Block		Metal–organic Blocks	
V1–O1	1.637(9)	Cu1A–O2M	2.493(10)
V1–O1M	1.636(9)	Cu1A–O2M^{ii}	2.493(10)
V1–O12	1.823(9)	Cu1A–N1A	2.005(12)
V1–O31^{i}	1.781(10)	Cu1A–N4A	2.000(12)
V2–O2M	1.601(10)	Cu1A–N1A^{ii}	2.005(12)
V2–O2	1.602(11)	Cu1A–N4A^{ii}	2.000(12)
V2–O23	1.750(10)	Cu1B–O1	2.636(11)
V2–O12	1.825(9)	Cu1B–O1^{iii}	2.636(11)
V3–O3	1.580(12)	Cu1B–N1B	1.996(12)
V3–O3M	1.625(11)	Cu1B–N4B	1.996(11)
V3–O23	1.825(11)	Cu1B–N1B^{iii}	1.996(12)
V3–O31	1.764(9)	Cu1B–N4B^{iii}	1.996(11)
		Cu1C–O1M	2.420(10)
		Cu1C–O1M^{iv}	2.420(10)
		Cu1C–N1C	2.003(11)
		Cu1C–N4C	2.013(12)
		Cu1C–N1C^{iv}	2.003(11)
		Cu1C–N4C^{iv}	2.013(12)

Symmetry codes: i) 1+x, y, z; ii) 1–*x*, 1–*y*, 1–*z*; iii) 2–*x*, 2–*y*, 2–*z*; iv) 2–*x*, 1–*y*, 2–*z*.

Table S7 Selected V–O_b–V–O_b and O_b–V–O_b–V torsion angles (°) in the metavanadate chains of **1**–**4.**

1				2		3		4	
O6–V1–O12–V2	164.1	O712–V7–O78–V8	114.5	O13–V1–O12–V2	60.7	O13–V1–O12–V2	163.4	O31–V1–O12–V2	161.9
V1–O12–V2–O23	76.7	V7–O78–V8–O89	81.5	V1–O12–V2–O23	61.4	V1–O12–V2–O23	157.8	V1–O12–V2–O23	136.2
O12–V2–O23–V3	85.8	O78–V8–O89–V9	138.4	O12–V2–O23–V3	158.0	O12–V2–O23–V3	110.3	O12–V2–O23–V3	97.2
V2–O23–V3–O34	155.9	V8–O89–V9–O910	93.9	V2–O23–V3–O13	82.5	V2–O23–V3–O13	104.0	V2–O23–V3–O31	121.0
O23–V3–O34–V4	168.1	O89–V9–O910–V10	175.4	V3–O13–V1–O12	170.8	V3–O13–V1–O12	142.3	V3–O31–V1–O12	139.8
V3–O34–V4–O45	72.9	V9–O910–V10–O101	47.9						
O34–V4–O45–V5	125.9	O910–V10–O101–V1	145.6						
V4–O45–V5–O56	77.5	V10–O101–V11–O11	97.1						
O45–V5–O56–V6	157.3	O101–V11–O112–V1	66.2						
V5–O56–V6–O16	79.1	V11–O112–V12–O71	174.9						

Table S8 Intermolecular N–H···O and C–H···O interactions in **2.**

Donor–H···Acceptor	D–H (Å)	H···A (Å)	D···A (Å)	D–H···A (°)
N1A–H1A···O3	0.98	2.14	3.072(3)	159
N1B–H1B···O1^{i}	0.98	1.99	2.948(3)	164
N1C–H1C···O2^{ii}	0.98	2.00	2.931(3)	159
N4A–H4A···O2	0.98	1.98	2.868(3)	150
N4B–H4B···O23	0.98	2.22	3.107(3)	149
N4C–H4C···O13^{iii}	0.98	2.28	3.147(3)	148
C3B–H3BA···O12^{iv}	0.97	2.50	3.439(3)	164
C6B–H6BB···O3	0.97	2.54	3.480(4)	162
C3C–H3CA···O12^{iii}	0.97	2.47	3.408(3)	162
C6C–H6CB···O3M^{iii}	0.97	2.49	3.448(3)	169
C7A–H7AB···O3W	0.97	2.16	2.885(11)	130
C7A–H7AB···O5W	0.97	2.63	3.602(9)	177
C6B–H6BA···O9W^{v}	0.97	2.62	3.442(17)	143
C2C–H2CA···O8W^{vi}	0.97	2.46	3.101(18)	124

Symmetry codes: i) 1–x, 1–y, –z; ii) x, y, 1+z; iii) 1–x, 2–y, 1–z; iv) –1+x, y, z; v) 1–x, 1–y, 1–z; vi) x, y, –1+z.

Table S9 Intermolecular N–H···O and C–H···O interactions in **3.**

Donor–H···Acceptor	D–H (Å)	H···A (Å)	D···A (Å)	D–H···A (°)
N1A–H1A···O3^{i}	0.98	2.29	2.976(10)	126
N1B–H1B···O12	0.98	2.06	3.039(10)	172
N4A–H4A···O12^{ii}	0.98	2.06	3.035(11)	173
N4C–H4C···O2^{iii}	0.98	2.16	3.135(12)	172
N1C–H1C···O1^{iv}	0.98	2.28	2.881(11)	118
N4B–H4B···O3M^{i}	0.98	2.53	3.157(10)	122
N4B–H4B···O13^{v}	0.98	2.04	2.993(10)	163
C3B–H3BA···O3M^{i}	0.97	2.53	3.220(12)	127
C6C–H6CB···O1W^{v}	0.97	2.57	3.499(13)	160
C5B–H5BB···O1M	0.97	2.58	3.205(11)	123
C6B–H6BB···O1W	0.97	2.59	3.328(13)	133
C2A–H2AA···O1^{vi}	0.97	2.48	3.245(13)	135
C2A–H2AB···O3^{i}	0.97	2.52	3.123(13)	120
C6A–H6AB···O2W^{vii}	0.97	2.47	3.364(13)	153
C2C–H2CB···O1^{iv}	0.97	2.39	3.028(13)	123
C3A–H3AB···O3^{viii}	0.97	2.66	3.421(13)	136

Symmetry codes: i) 1–x, –y, 1 –z; ii) x, –1+y, –1+z; iii) 1–x, 1–y, 1–z; iv) –x, 1–y, 1–z; v) –x, –y, 1–z; vi) x, –1+y, –1+z; vii) 1–x, –y, –z; viii) –1+x, –1+y, –1+z.

Table S10 Intermolecular N–H···O and C–H···O interactions in **4.**

Donor–H···Acceptor	D–H (Å)	H···A (Å)	D···A (Å)	D–H···A (°)
N1A–H1A···O12	0.98	2.09	3.073(16)	177
N1B–H1B···O3M^{i}	0.98	2.57	3.198(15)	122
N1B–H1B···O31^{i}	0.98	2.31	3.282(16)	170
N1C–H1C···O2	0.98	1.99	2.956(17)	168
N4A–H4A···O3^{ii}	0.98	2.02	2.830(16)	139
N4B–H4B···O12	0.98	2.11	3.076(13)	169
N4C–H4C···O1^{iii}	0.98	2.03	3.004(15)	174
C3B–H3BA···O3M	0.97	2.58	3.40(2)	142
C3B–H3BA···O23	0.97	2.52	3.32(2)	139
C2B–H2BA···O3M^{i}	0.97	2.50	3.206(18)	129
C2A–H2AA···O3^{iv}	0.97	2.52	3.42(2)	153
C7C–H7CA···O2^{iv}	0.97	2.49	3.27(2)	137
C2C–H2CA···O2^{v}	0.97	2.61	3.30(2)	128

Symmetry codes: i) 1–x, 2–y, 2–z; ii) 1–x, 1–y, 1–z; iii) 2–x, 1–y,2 –z; iv) 1+x, y, z; v) –x, –y, –z; vi) 1–x, 1–y, –z.