Formation of a 2D Supramolecular Water Framework via Metal-Organic Unit Templating

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Experimental Section

Materials and Methods. All of the chemicals were obtained from commercial sources and used without further purification. The determinations of the unit cell and data collection for the crystal of compound **1** were performed on a Siemens SMART CCD APEX II. The data were collected using graphite–monochromatic Mo-Kα radiation (λ = 0.71073 Å) at 296 K. The data sets were corrected by **SADABS** program.[1] The structures were solved by direct methods, and refined by full-matrix least-square method with the **SHELXTL-97** program package.[2] H atoms on C atoms were generated geometrically. The H-atoms of the water molecule were clearly visible in a difference map and were handled in the subsequent refinement with fixed isotropic displacement parameters. The IR spectrum was recorded from KBr pellet on a FTS-40 spectrophotometer. Thermogravimetric analysis (TGA) was carried out under air on a NETZSCH STA 409 PC/PG instrument at a heating rate of 4 ^{o}C/min.

References:

1. G. M. Sheldrick, **SADABS**; *Siemens Analytical X-ray Instrument Division*: Madison, WI, **1995**.
2. G. M. Sheldrick, *Program for Structure Refinement*: University of Göttingen, Germany, **1997**.

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Figures

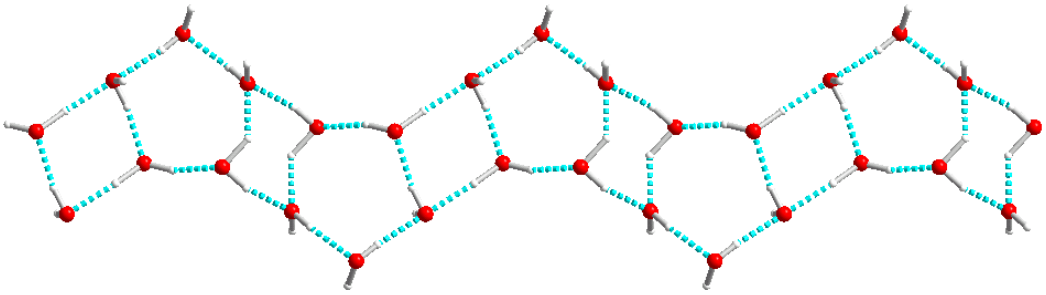

Figure S1. The supramolecular water tape in **1**.

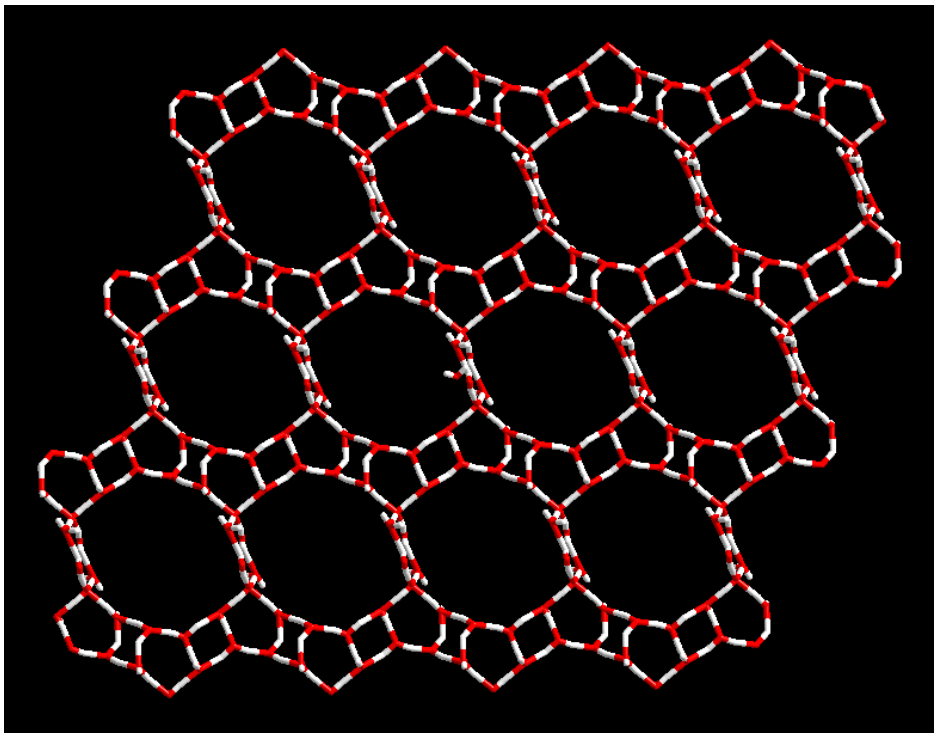

Figure S2. Supramolecular water layer in **1**.

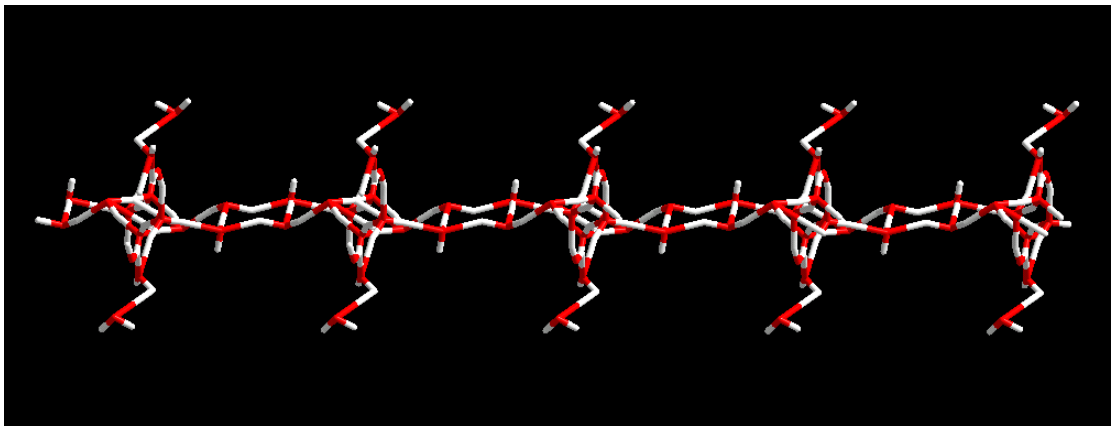

Figure S3. Side view of the supramolecular water layer in **1**.

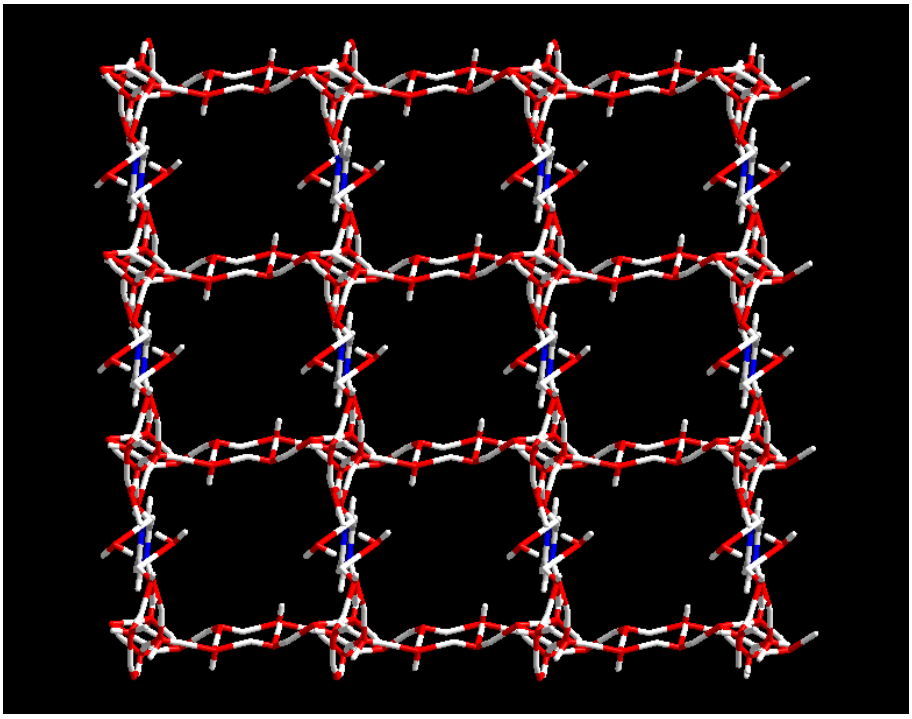

Figure S4. A view of the 3D supramolecular network in **1** along 1 0 1 direction.

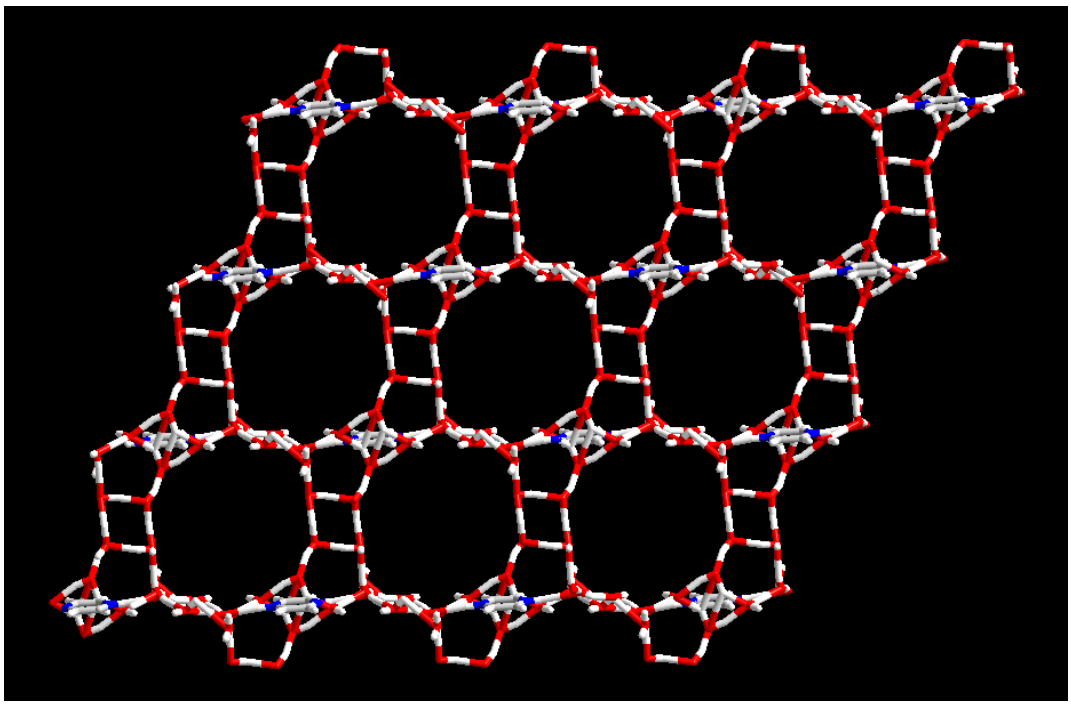

Figure S5. A view of the 3D supramolecular network in **1** along the *a* axis.

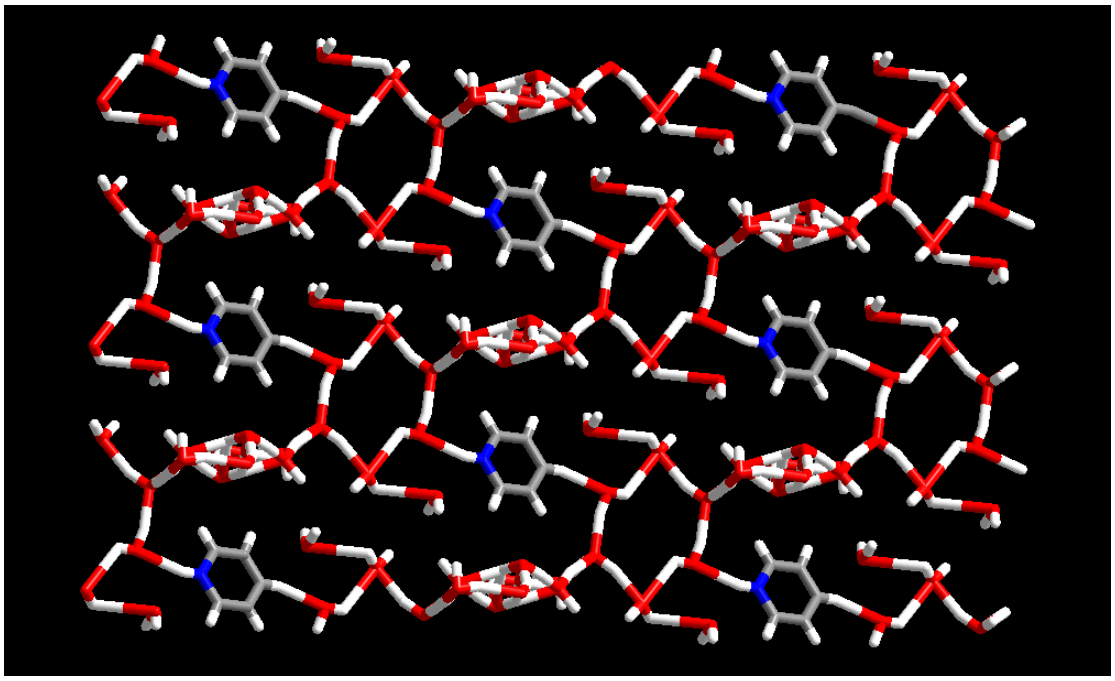

Figure S6. A view of the 3D supramolecular network in **1** along the *b* axis.

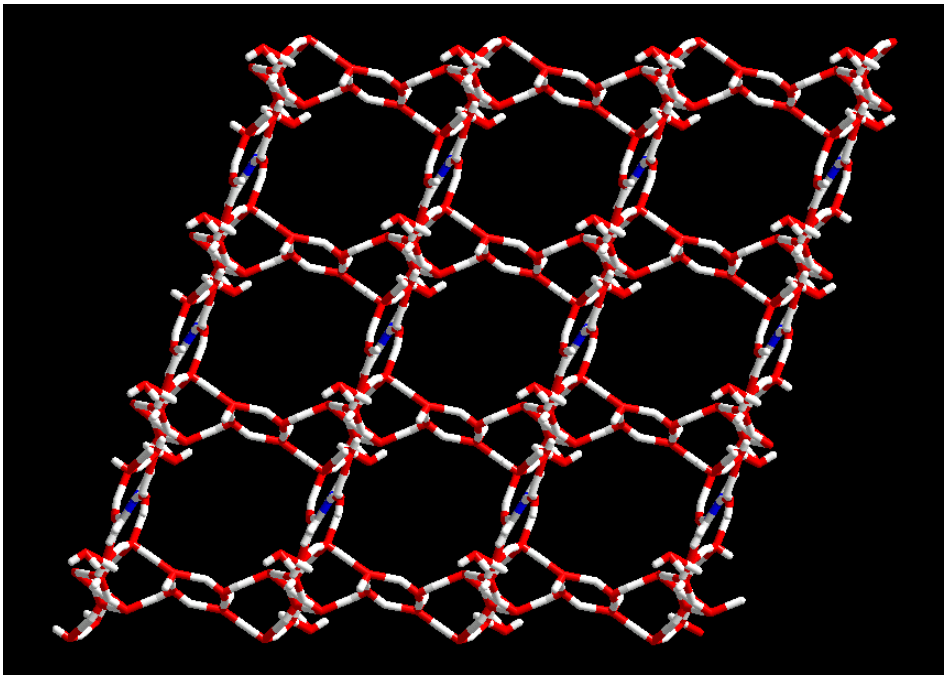

Figure S7. A view of the 3D supramolecular network in **1** along the *c* axis.

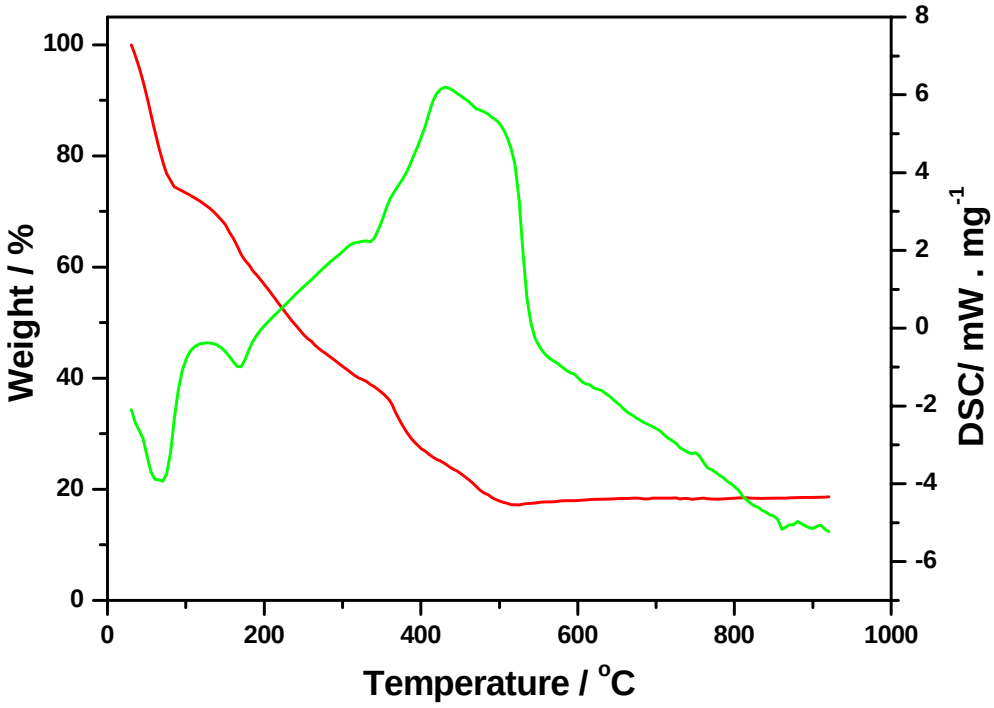

Figure S8. Thermogravimetric analysis curve of **1**.

Tables

Table S1. Crystal data and structure refinements for compound **1**.

	1
Formula	C39H61N5Ni2O24
Formula weight	1101.35
Crystal size (mm^3)	0.32 × 0.17 × 0.12
Crystal color	Green
Crystal system, Space group	triclinic, *P*-1
a (Å)	10.261(1)
b (Å)	11.412(1)
c (Å)	13.314(2)
α (^{o})	110.27(1)
β (^{o})	107.37(1)
γ (^{o})	100.93(1)
Volume ($Å^3$)	1318.9(2)
Z	1
Calculated density ($g·cm^{-3}$)	1.387
F(000)	578
Temperature (K)	296(2)
Wavelength (Å)	0.71073
Absorption coefficient (mm^{-1})	0.796
θ for data collection (^{o})	1.78 to 27.72
Limiting indices	-13≤h≤13, -14≤k≤14, -17≤l≤17
Reflections collected	20611 [R(int) = 0.0651]
Unique reflections used for refinement	6096
Data / parameters	6096 / 316
Goodness-of-fit on F^2	1.017
R1 (wR2) [$I > 2\sigma(I)$] (3837 reflections)	0.0481 (0.1047)
R1 (wR2) (all data) (6096 reflections)	0.0966 (0.1199)
Largest diff. peak and hole ($e·Å^3$)	0.533 and -0.441

$R1 = \sum(|F_o| - |F_c|) / \sum|F_o|$, $wR2 = [\sum w(F_o^2 - F_c^2)^2 / \sum w(F_o^2)^2]^{0.5}$.

Table S2. Selected bond lengths (Å) and angles (°) for **1**.

Bond lengths	(Å)	Bond angles	(°)	Bond angles	(°)
Ni(1)-O(2)	2.047(2)	O(2)-Ni(1)-O(7)	91.87(7)	N(2)-Ni(1)-O(6)	178.20(8)
Ni(1)-O(7)	2.077(2)	O(2)-Ni(1)-N(2)	92.20(9)	N(1)-Ni(1)-O(6)	89.55(9)
Ni(1)-N(2)	2.079(3)	O(7)-Ni(1)-N(2)	88.20(9)	O(2)-Ni(1)-O(5)	172.39(9)
Ni(1)-N(1)	2.092(2)	O(2)-Ni(1)-N(1)	87.73(8)	O(7)-Ni(1)-O(5)	84.48(8)
Ni(1)-O(6)	2.093(2)	O(7)-Ni(1)-N(1)	177.92(9)	N(2)-Ni(1)-O(5)	94.35(10)
Ni(1)-O(5)	2.112(2)	N(2)-Ni(1)-N(1)	89.77(10)	N(1)-Ni(1)-O(5)	96.16(9)
		O(2)-Ni(1)-O(6)	89.44(7)	O(6)-Ni(1)-O(5)	84.07(9)
		O(7)-Ni(1)-O(6)	92.49(7)		

Table S3. Hydrogen bond lengths (Å) and bond angles **(°)** in **1**.

D-H…A	**D-H** (Å)	**H…A** (Å)	**D…A** (Å)	<D-H…A (°)
O5-H5B...O105	0.94	1.83	2.745(3)	164.2
O5-H5C...O6	0.72	2.40	2.816(3)	118.6
O6-H6A...O3	0.89	1.88	2.754(2)	165.6
O6-H6B...O4^{i}	0.99	1.71	2.597(3)	147.0
O7-H7A...O1	0.93	1.74	2.646(3)	164.3
O7-H7B...O3^{i}	0.94	1.83	2.751(3)	168.5
O101-H10D...O104^{ii}	0.85	1.98	2.830(4)	171.9
O101-H10E...O104	0.86	1.96	2.807(4)	166.6
O102-H10J...O101^{iii}	0.84	1.96	2.793(4)	168.4
O102-H10K...O103	0.81	2.22	2.949(4)	149.1
O103-H10H...O1	0.97	1.87	2.742(3)	147.8
O103-H10I...O102^{iv}	0.93	1.92	2.815(4)	160.9
O104-H10F...O3	0.83	1.95	2.766(3)	169.2
O104-H10G...O105^{v}	0.83	2.08	2.903(3)	170.6
O105-H10B...O6^{v}	0.82	2.04	2.851(3)	169.8
O105-H10C...O103^{vi}	0.89	1.93	2.814(4)	173.4
C20-H20A...O5	0.93	2.39	3.301(9)	167.4

Symmetry transformations used to generate equivalent atoms: i) -x+1, -y+1, -z+1; ii) -x+1, -y+1, -z+2; iii) -x+1, -y, -z+1; iv) -x+1, -y, -z; v) -x, -y+1, -z+1; vi) x-1, y, z.