Electronic Supplementary Material (ESI) for CrystEngComm

Electronic Supplementary Information

Combination between metal-ligand coordination and hydrogen bond interaction: a facile route for the construction of 3D coordination networks with inclusion ability to relatively large aromatic molecules

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Contents

Experimental Section

Supporting Figures and Tables

References

Material and instrumentations

All chemicals and solvents were purchased from Kanto Chemical Co., Ltd., Wako Pure Chemical Co., Ltd., and Tokyo Kasei Kogyo Co., Ltd., and were used as received without further purification. Powder X-ray diffraction patterns were measured on a Rigaku Multi-Flex X-ray diffractometer using graphite-monochromatized Cu Kα radiation (λ = 1.5418 Å) with a scanning rate of 0.020 ° sec^{-1} at room temperature. TG/DTA analysis was measured on a SEIKO instrument Inc. EXSTAR 6000 TG/DTA 6200. X-ray crystallographic data for **1** and **2** were collected on a Rigaku RAXIS-RAPID imaging plate area detector using graphite-monochromatized Mo Kα radiation (λ = 0.71073 Å) at 153 K. The crystal structures were solved by direct method using the *SHELXS-97* program[1] attached to *WinGX* ver. 1.64.00[2] and refined by the successive differential Fourier syntheses and full-matrix least-squares procedure using the *SHELXL-97* program[1] attached to *WinGX* ver. 1.64.00. Anisotropic thermal factors were applied to all non-hydrogen atoms. All hydrogen atoms were generated geometrically. Computer graphics of the crystal structures of **1** and **2** (Fig. 2, 5, S3 and S5) were portrayed with *ORTEP-3* for Windows ver. 2.00 program.[3] Computer graphics of the crystal structures of **1** and **2** (Fig. 3, 4, and S4) were portrayed with *Mercury* 1.4.2 program.[4] Simulation of the XRPD patterns of **1** and **2** were carried out by *Mercury* 1.4.2 program.

Supporting Figures and Tables

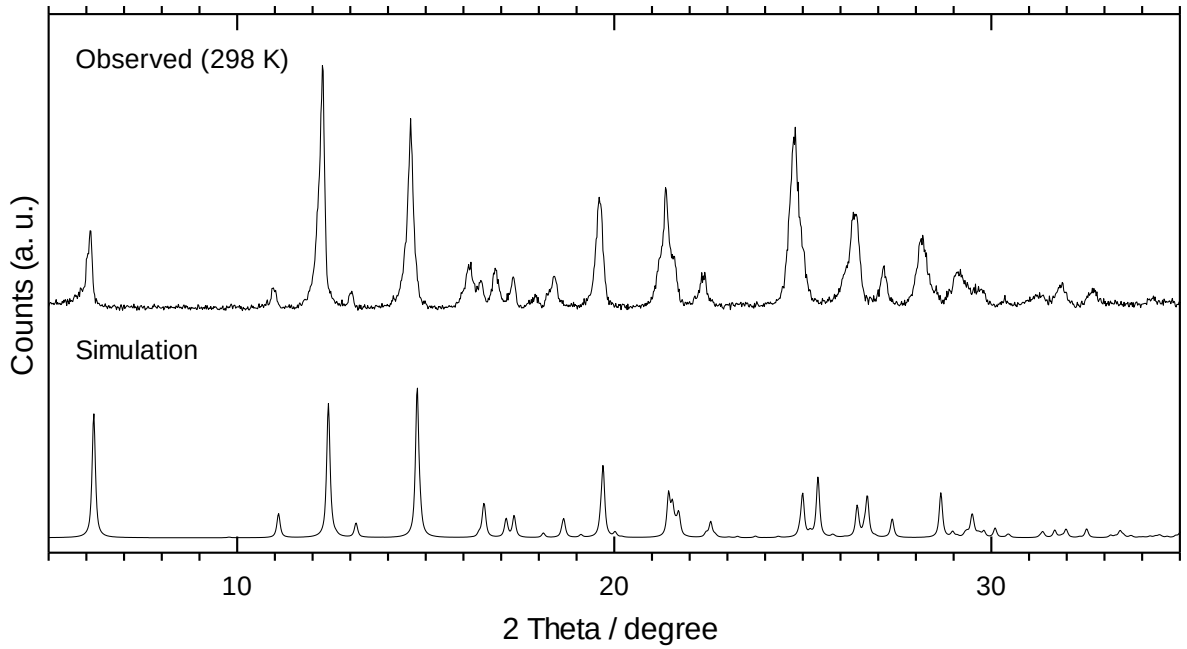

Fig. S1. Observed (top) and calculated (bottom) X-ray powder diffraction patterns of **1** (2θ range: $5° < 2\theta < 35$ 2θ).

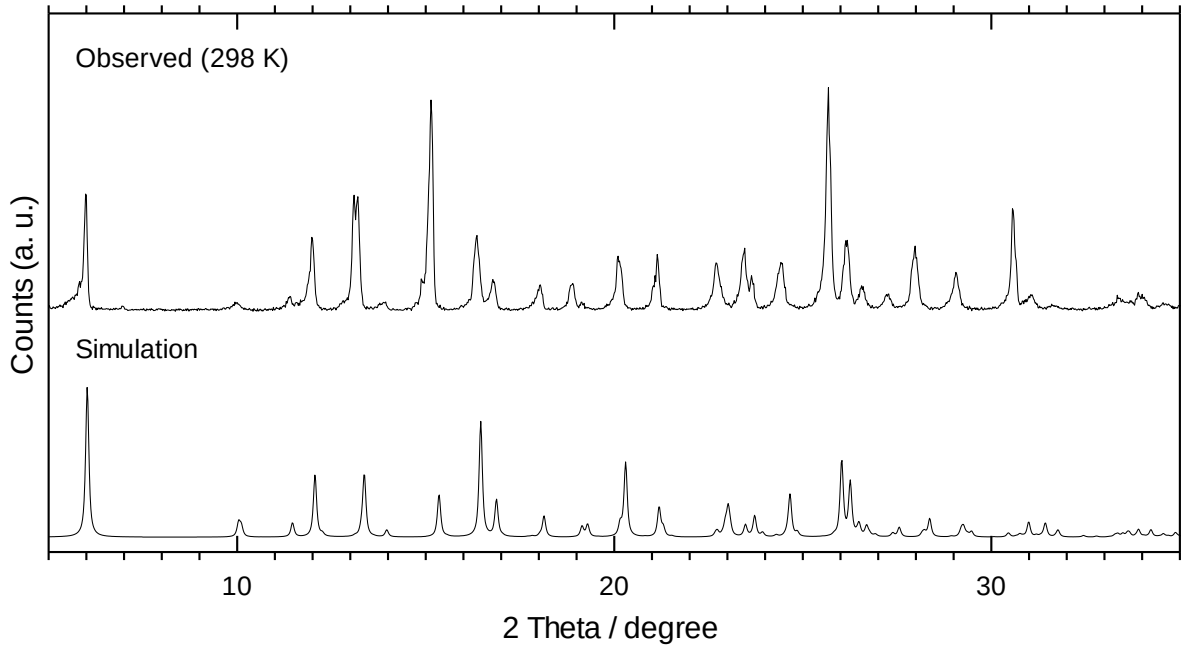

Fig. S2. Observed (top) and calculated (bottom) X-ray powder diffraction patterns of **2** (2θ range: $5° < 2\theta < 35$ 2θ).

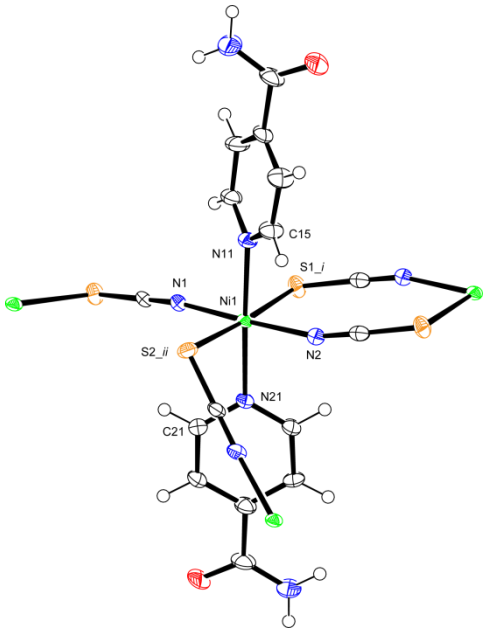

Fig. S3. ORTEP drawing (50 % probability ellipsoids) of the coordination environment around the Ni^{2+} ion of **1**. Colour scheme: black (carbon or hydrogen), blue (nitrogen), red (oxygen), orange (sulfur), green (nickel). _*i* and _*ii* denote symmetry operations (_*i*: $-x + 3/2, -y + 3/2, -z + 1$; _*ii*: $-x + 3/2, y + 1/2, -z + 3/2$).

Table S1. Selected bond lengths, angles and torsion angle of **1**.[a]

	Bond length / Å	Bond angle / °	Torsion angle / °
Ni1 – N1	2.043(4)		
Ni1 – N2	2.040(4)		
Ni1 – N11	2.122(4)		
Ni1 – N21	2.098(4)		
Ni1 – S1_*i*[b]	2.561(1)		
Ni1 – S2_*ii*[b]	2.507(1)		
N1 – Ni1 – N11		92.1(2)	
N1 – Ni1 – N21		89.9(2)	
N1 – Ni1 – N2		174.5(1)	
N1 – Ni1 – S1_*i*		82.8(1)	
N1 – Ni1 – S2_*ii*		93.0(1)	
N11– Ni1 – S1_*i*		88.8(1)	
N11– Ni1 – S2_*ii*		90.0(1)	
N11 – Ni1 – N21		178.0(2)	
S1_*i* – Ni1 – S2_*ii*		175.60(5)	
C15 – N11– N21 – C21			60.7(4)

[a] All bond length, bond angle and torsion angle was calculated with *SHELXL-97* program.[1]

[b] Symmetry operations (_*i* : $-x + 3/2, -y + 3/2, -z + 1$; _*ii*: $-x + 3/2, y + 1/2, -z + 3/2$).

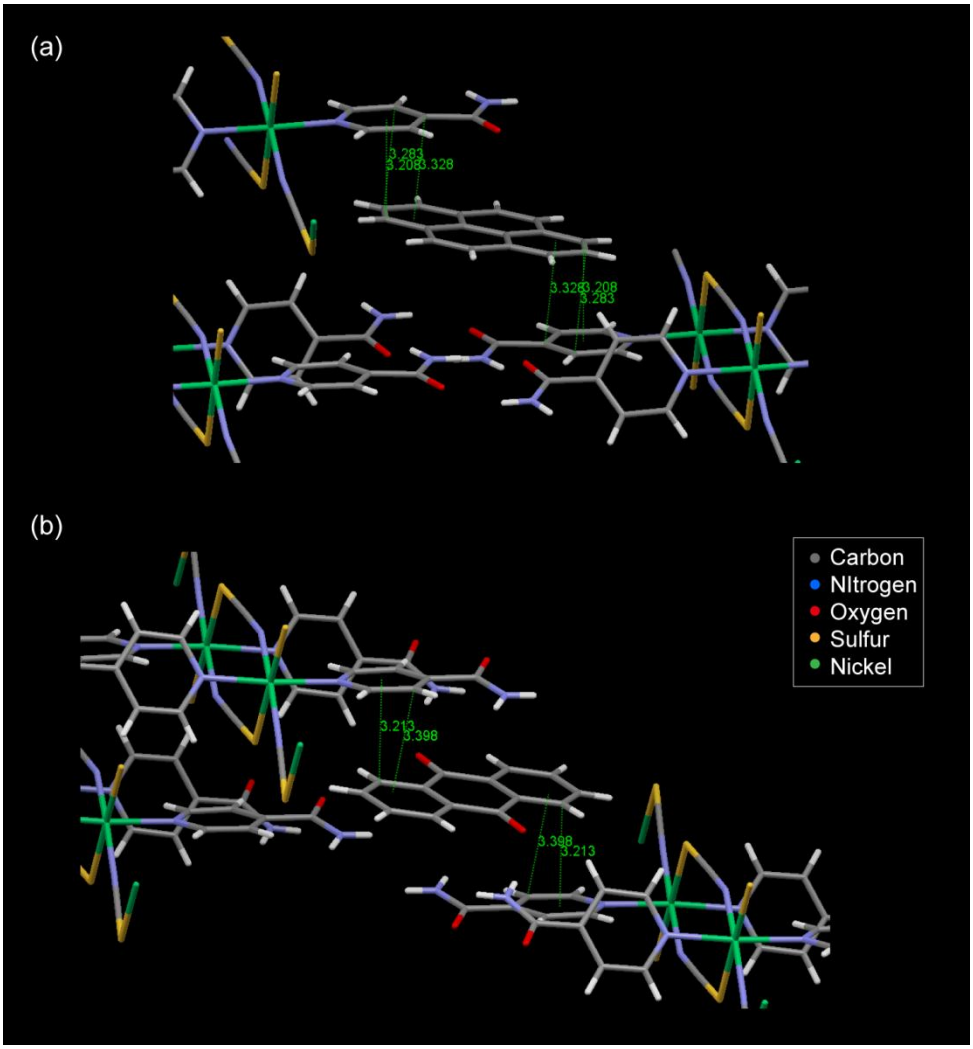

Fig. S4. π···π interaction (a) between the PY guest and the isoNH$_2$ dimers found in **1** and (b) between the AQ guest and the isoNH$_2$ dimers found in **2**.

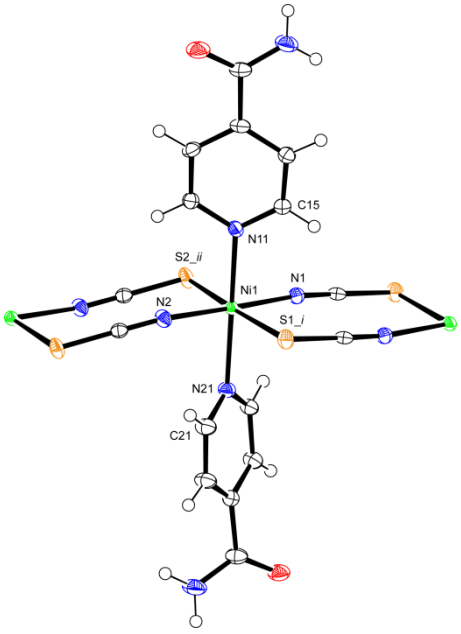

Fig. S5. ORTEP drawing (50 % probability ellipsoids) of the coordination environment around the Ni^{2+} ion of **2**. Colour scheme: black (carbon or hydrogen), blue (nitrogen), red (oxygen), orange (sulfur), green (nickel). _*i* and _*ii* denote symmetry operations (_*i*: −*x*, −*y* + 1, −*z*; _*ii*: −*x*, −*y*, −*z*).

Table S2. Selected bond lengths, angles and torsion angle of **2**.[a]

	Bond length / Å	Bond angle / °	Torsion angle / °
Ni1 – N1	2.018(2)		
Ni1 – N2	2.008(2)		
Ni1 – N11	2.141(2)		
Ni1 – N21	2.120(2)		
Ni1 – S1_*i*[b]	2.5414(9)		
Ni1 – S2_*ii*[b]	2.5160(9)		
N1 – Ni1 – N11		91.75(9)	
N1 – Ni1 – N21		91.22(9)	
N1 – Ni1 – N2		175.1(1)	
N1 – Ni1 – S1_*i*		93.93(7)	
N1 – Ni1 – S2_*ii*		80.48(7)	
N11– Ni1 – S1_*i*		87.15(7)	
N11 – Ni1 – S2_*ii*		91.72(7)	
N11– Ni1 – N21		175.5(1)	
S1_*i* – Ni1 – S2_*ii*		174.26(3)	
C15 – N11– N21 – C21			66.1(3)

[a] All bond length, bond angle and torsion angle was calculated with *SHELXL-97* program.[1]

[b] Symmetry operations (_*i*: −*x*, −*y* + 1, −*z*; _*ii*: −*x*, −*y*, −*z*).

Table S3. Crystallographic parameters of **1** and **2**.

	1	**2**
Formula	$C_{22}H_{17}N_6O_2S_2Ni$	$C_{21}H_{16}N_6O_3S_2Ni$
Formula weight	520.25	523.22
Crystal size /mm^3	0.10 × 0.10 × 0.10	0.15 × 0.15 × 0.10
Crystal habit	Block	Block
Crystal colour	Bluish green	Green
Crystal system	Monoclinic	Triclinic
Space group	*C*2/*c* (#15)	$P\ \bar{1}$ (#2)
Temperature / *K*	153	153
a / Å	32.418(3)	7.9523(6)
b / Å	9.5238(8)	10.9632(6)
c / Å	15.937(2)	16.9567(10)
α / °	90	90.564(4)
β / °	118.442(5)	65.422(3)
γ / °	90	56.997(9)
V / Å^3	4326.6(7)	1071.9(1)
Z	8	2
μ (Mo Kα) / mm^{-1}	1.124	1.138
$D_{calc.}$ / g cm^{-3}	1.60	1.62
h range	0 → 45	0 → 11
k range	0 → 13	−12 → 15
l range	−22 → 19	−21 → 23
No. of reflections measured	24805	11387
No. of reflections unique	6259	5927
No. of reflections observed	2759	4173
R_{int}	0.152	0.034
No. of parameter used	298	298
*R*1 ($I > 2\sigma(I)$)	0.0592	0.0430
*wR*2	0.0997	0.1096
G. O. F.	0.846	1.046
$\Delta\rho_{max}$ / e Å^{-3}	+0.666	+0.964
$\Delta\rho_{min}$ / e Å^{-3}	−0.827	−0.875
CCDC number	727046	727047

References

1. G. M. Sheldrick, *Acta Cryst*, **A64**, 112.
2. L. J. Farrugia, *J. Appl. Cryst*., 1999, **32**, 837.
3. L. J. Farrugia, *J. Appl. Cryst*., 1997, **30**, 565.
4. Mercury ver. 1.4.2 program. See: http://www.ccdc.cam.ac.uk/products/mercury/