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SUPPORTING INFORMATION

Guest Responsivity of a Two-Dimensional Coordination Polymer Incorporating a Cholesterol-Based Co-Ligand

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Physical Measurements

Single-crystal X-ray data were recorded on a Bruker SMART APEX II ULTRA CCD Diffractometer with confocal monochromated Mo-Kα radiation. The structures was solved by a direct method (Sir 97) and refined by full-matrix leastsquares refinement using the SHELXL-97 computer program. The hydrogen atoms were refined geometrically by using a riding model. X-ray powder diffraction was carried out on a Rigaku Ultima IV diffractrometer with Cu-Kα radiation. Synchrotron powder X-ray diffraction patterns were collected using the BL44B2 beamline (λ = 0.799640 Å) at SPring-8, Japan. Elemental analyses of carbon, hydrogen and nitrogen were performed at the Elemental Analysis Service Center of Kyushu University. Thermogravimetric Analysis was carried out on a Perkin Elmer STA6000. X-ray fluorescence analysis was carried out on a Rigaku ZSX-100S. Infrared spectra were measured with a JASCO FT/IR-4200 using ATR method. The magnetic susceptibilities of all samples were measured on Quantum Design MPMS-XL5R SQUID susceptometer in the temperature range of 4-300 K in an applied dc field of 1000 Oe. Solid-state reflectance spectra were measured using an Ocean Optice USB2000. Raman spectra measurements were carried out on a Horiba iHR320 equipped with a Peltier-effect cooled CCD (charge-coupled devise) as detector (Horiba Jobin-Yvon-Synapse). The excitation source was a 532-nm DPSS (diode pumped solid state) laser. NMR spectra were recorded on a JEOL ECA-600 spectrometer. MALDI-TOF mass spectrum was measured on a Bruker Autoflex.

Isolation of co-ligands from 1·H_2O·$CHCl_3$

1·H_2O·$CHCl_3$ (6 mg) was decomposed with an aqueous Na_2EDTA solution (0.05 M, 1.25 ml) and stirred vigorously for 14 h at room temperature. The resulting mixture was extracted with $CHCl_3$. The organic extract was dried by Na_2SO_4, and concentrated under reduced pressure. Column chromatography of the residue using hexane:ethyl acetate = 5:2 afforded the extract (cholest-5-en-7-on2-3-yl isonicotinate) as a colorless amorphous. Rf = 0.44 (hexane:ethyl acetate = 1:1); IR (solid) 1725, 1671 cm^{-1}. ^1H NMR (600 MHz, $CDCl_3$): δ 8.79 (2H, d, *J* = 5.7 Hz), 7.84 (2H, d, *J* = 5.7 Hz), 5.75 (1H, s), 5.00 (1H, dddd, *J* = 13.8, 11.7, 4.8, 4.2 Hz), 2.71 (1H, ddd, *J* = 13.8, 4.8, 1.8 Hz), 2.64 (1H, dd, *J* = 13.8, 11.7 Hz), 2.38-2.43 (1H, m), 2.26 (1H, dd, *J* = 11.4, 11.4 Hz), 2.10-2.17 (1H, m), 2.03-2.06 (m), 1.80-1.94 (3H, m), 1.50-1.62 (m), 1.25-1.39 (m), 1.01-1.20 (m), 0.93 (3H, d, *J* = 6.0 Hz), 0.87 (3H, d, *J* = 2.4 Hz), 0.86 (3H, d, *J* = 3.0 Hz), 0.70 (3H, s). ^{13}C NMR (150 MHz, $CDCl_3$): δ 201.8, 164.3, 163.2, 150.6, 127.0, 122.8, 73.8, 54.8, 50.0, 49.8, 45.5, 43.1, 39.4, 38.7, 38.4, 37.7, 36.2, 36.0, 35.7, 28.5, 28.0, 27.4, 26.3, 23.8, 22.8, 22.5, 21.2, 18.9, 17.3, 12.0.

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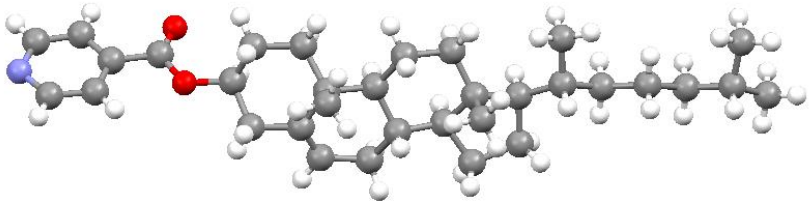

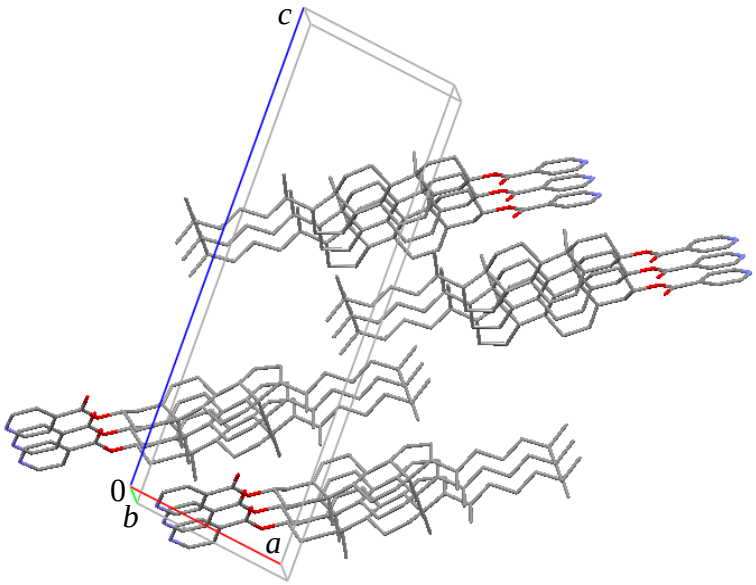

Temperature	100 K
Formula	$C_{33}H_{49}NO_2$
F. W.	491.73
Crystal system	monoclinic
Space group	$P2_1$(#4)
a / Å	9.195(1)
b / Å	5.792(1)
c / Å	27.350(5)
α / °	90
β / °	97.538(2)
γ / °	90
V / Å^3	1444.2(5)
Z	2
D_c / g cm^{-3}	1.131
m / cm^{-1}	0.069
R	0.0484
R_w	0.1233

Fig. S1 Crystal structure of Cholest-5-en-3-yl isonicotinate (Cholpy). (Table: crystal parameters of Cholpy).

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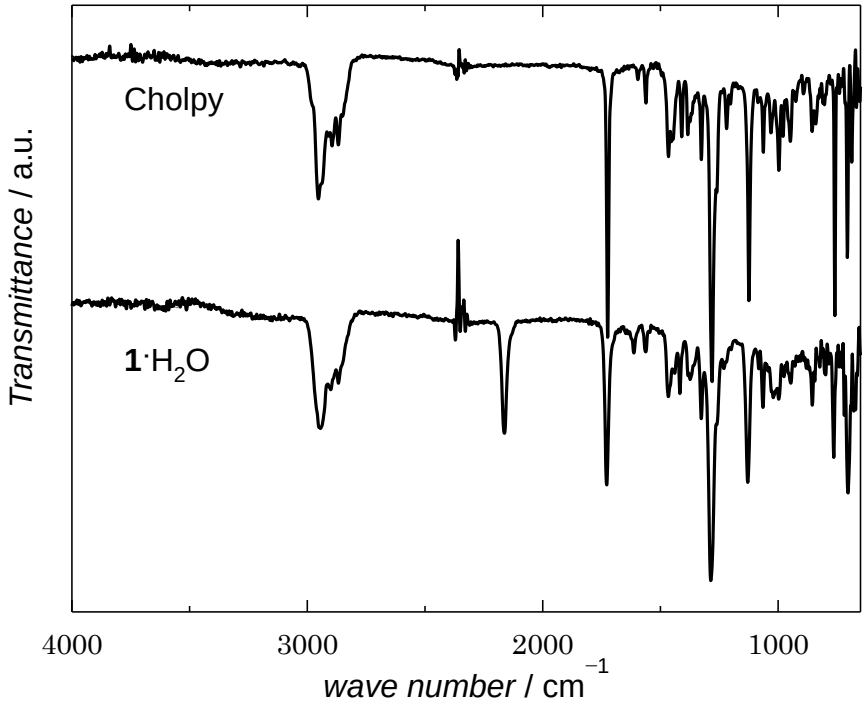

Fig. S2 IR spectra of Cholpy and **1**·H_2O.

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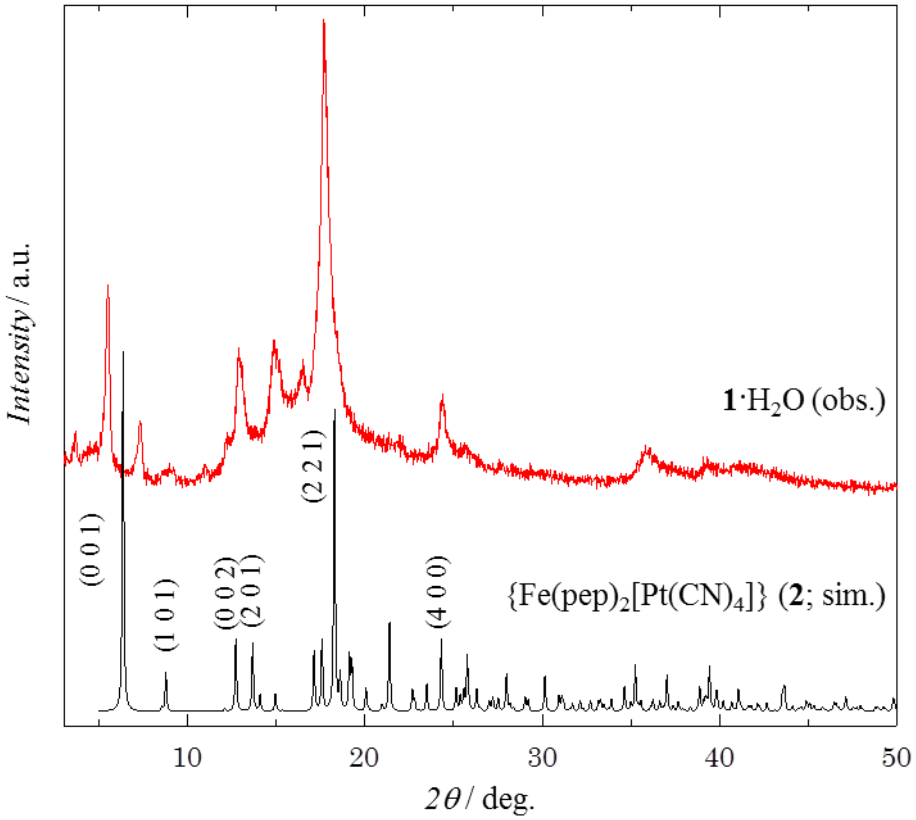

Fig. S3 XRPD patterns of **1**·H_2O and {$Fe(pep)_2[Pt(CN)_4]$} (**2**; simulated).

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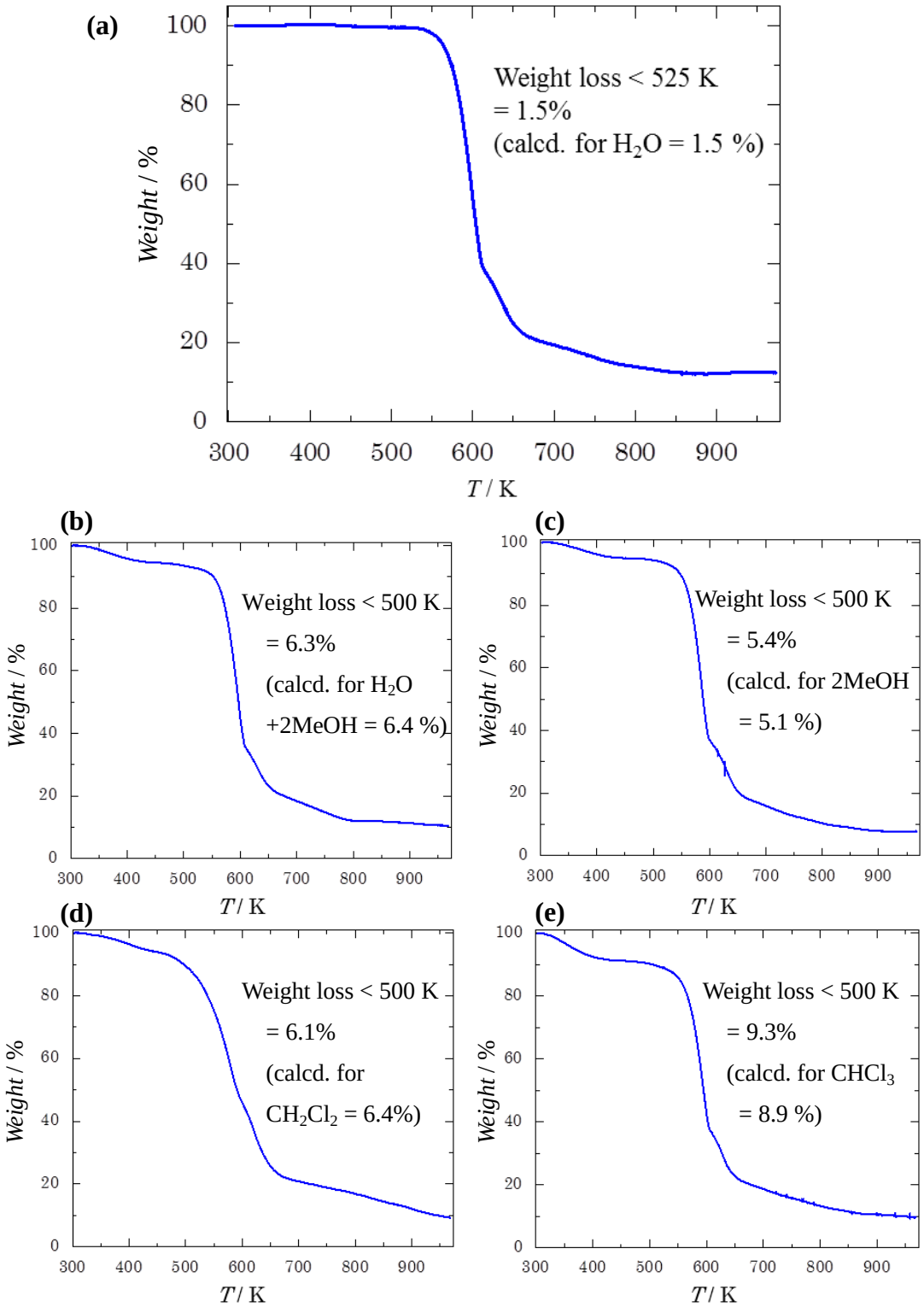

Fig. S4 TGA curves for **1**·H_2O·(a), **1**·H_2O·MeOH (b), **1**·MeOH (c), **1**·H_2O·$CHCl_3$ (d) and **1**·$CHCl_3$ (e). (In the case of (d) **1**·H_2O·$CHCl_3$, the co-ligand Cholpy was oxidized and absorbed $CHCl_3$ was assumed to be changed to CH_2Cl_2.)

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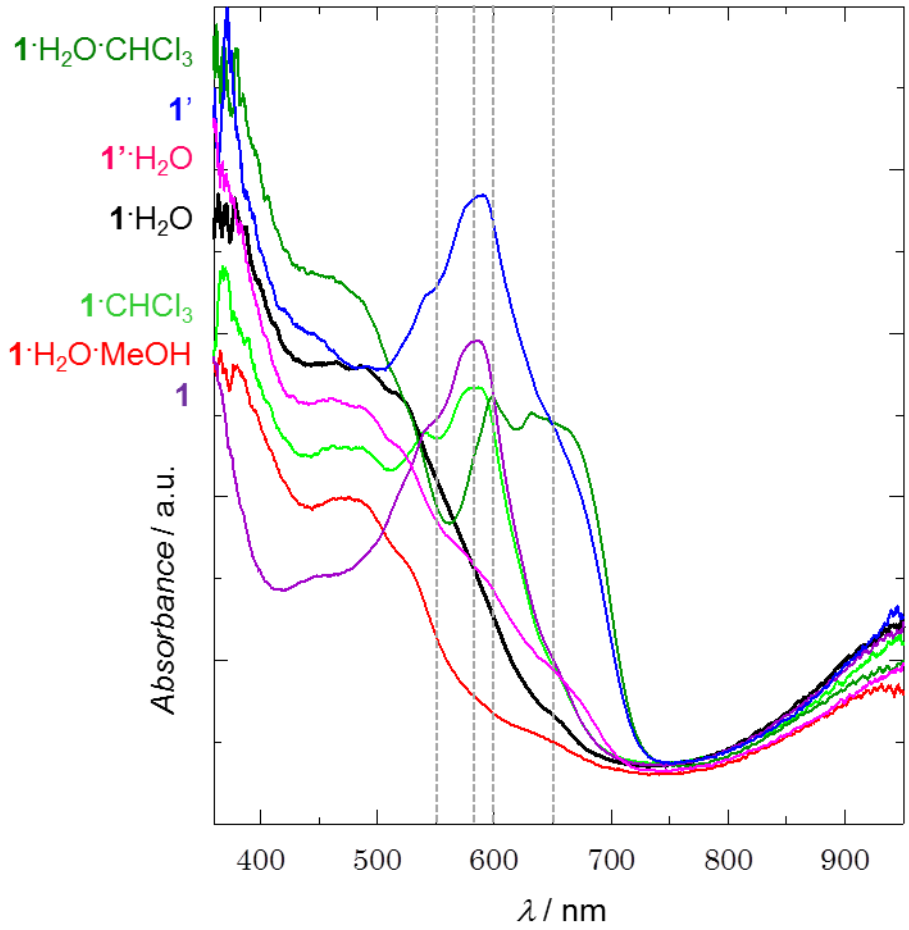

Fig. S5 Solid-state reflectance spectra of **1**·H_2O and the resulting compounds through the guest adsorption and desorption processes shown in Scheme 2.

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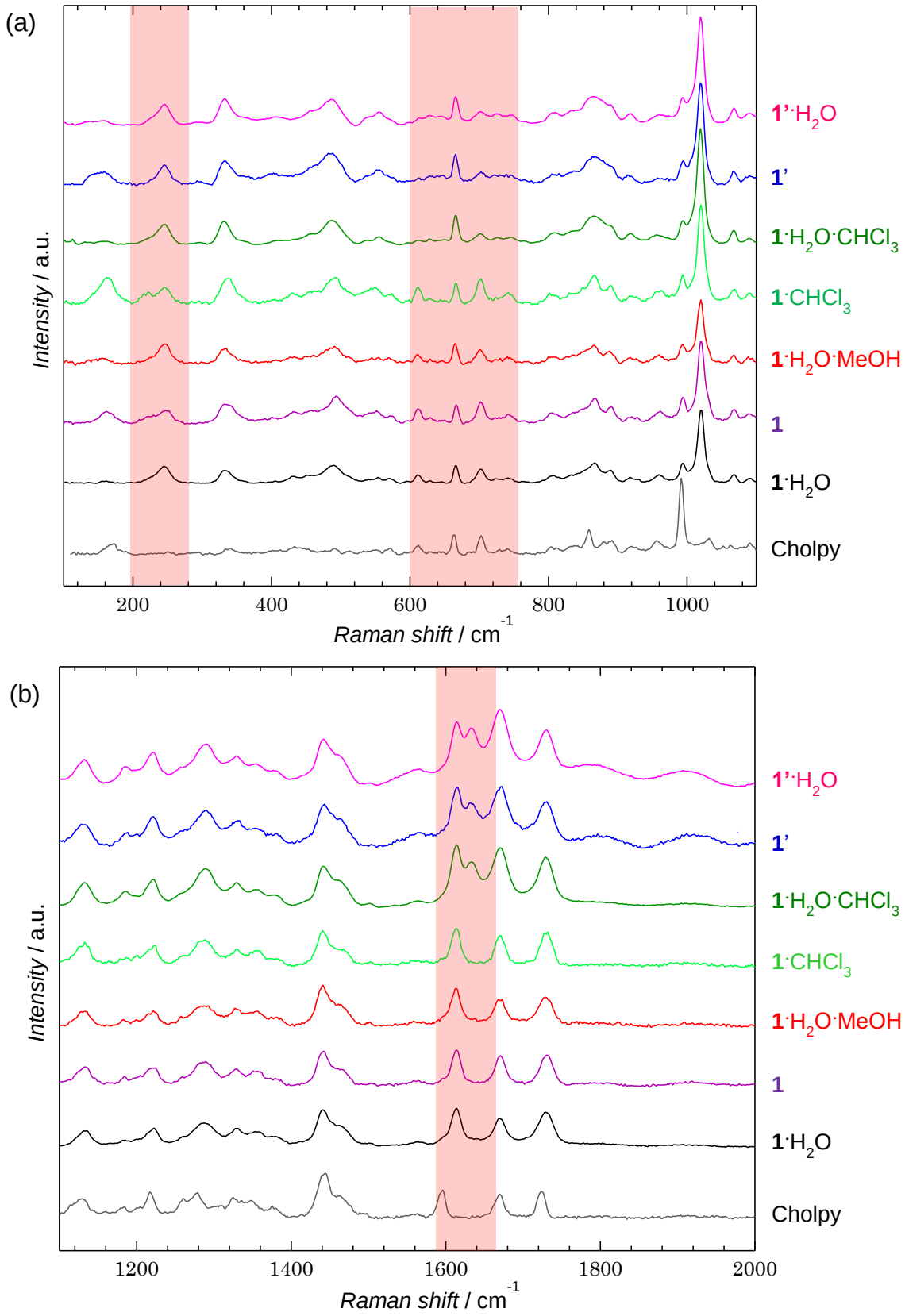

Fig. S6 Raman spectra of **1**·H_2O, Cholpy and the resulting compounds through the guest adsorption and desorption processes, (a) 100−1100 cm^{-1}, (b) 1100−2000 cm^{-1}.

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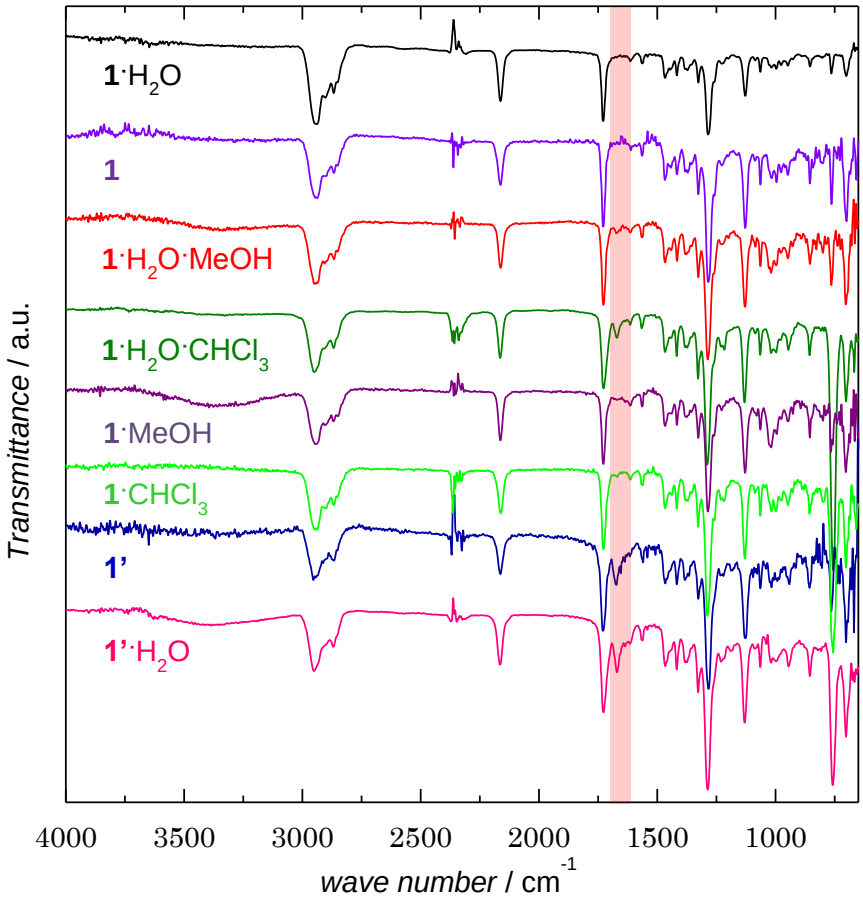

Fig. S7 IR spectra of **1**·H_2O and the resulting compounds through the guest adsorption and desorption processes.

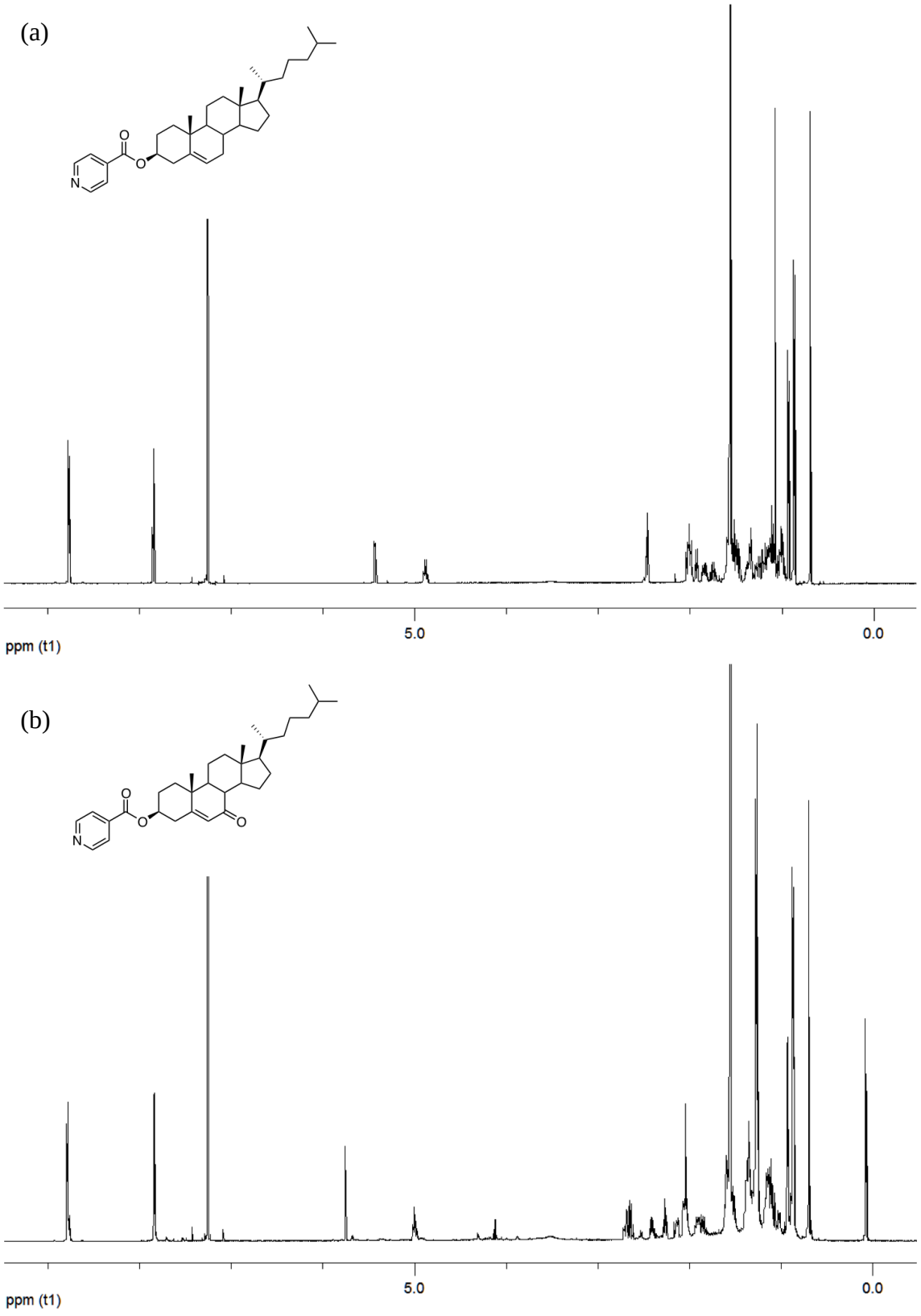

Fig. S8 ^{1}H-NMR spectra of Cholpy (a) and the extracted co-ligand of **1**·H_2O·$CHCl_3$ (b).

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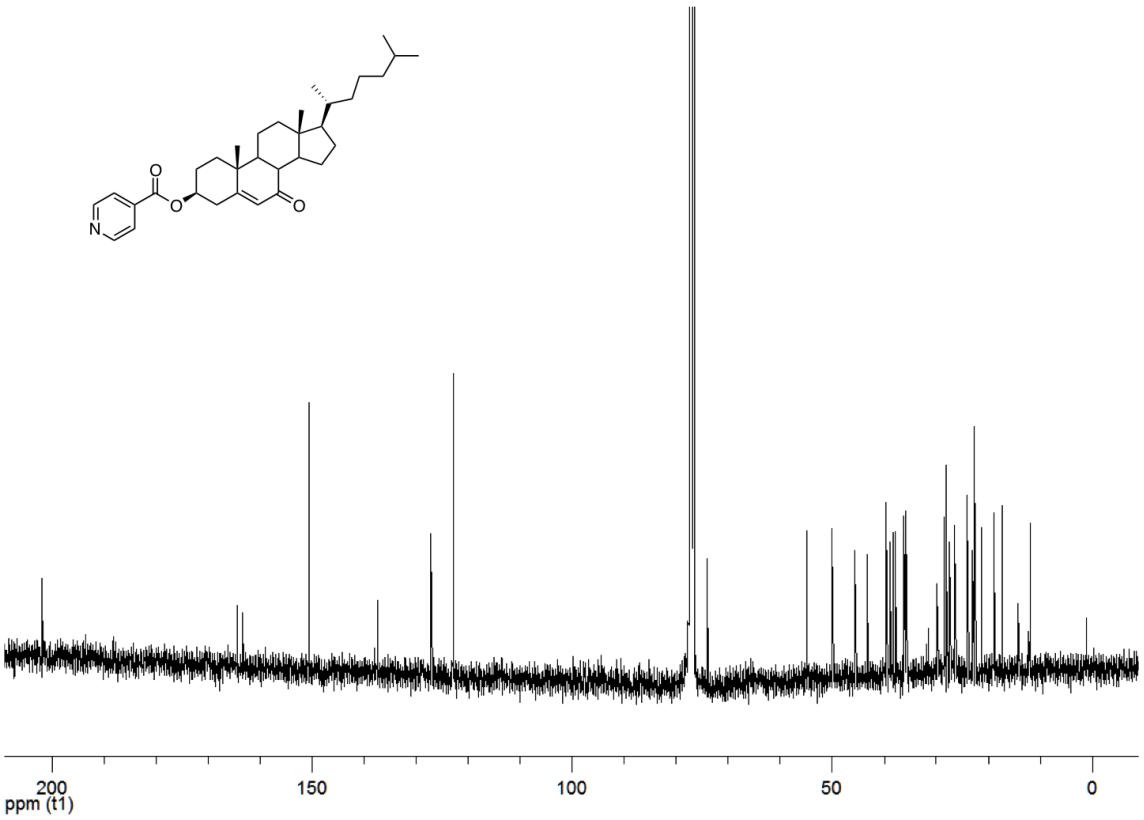

Fig. S9 ^{13}C NMR spectrum of the extracted co-ligand of **1**·H_2O·$CHCl_3$ (Cholest-5-en-7-one-3-yl isonicotinate).

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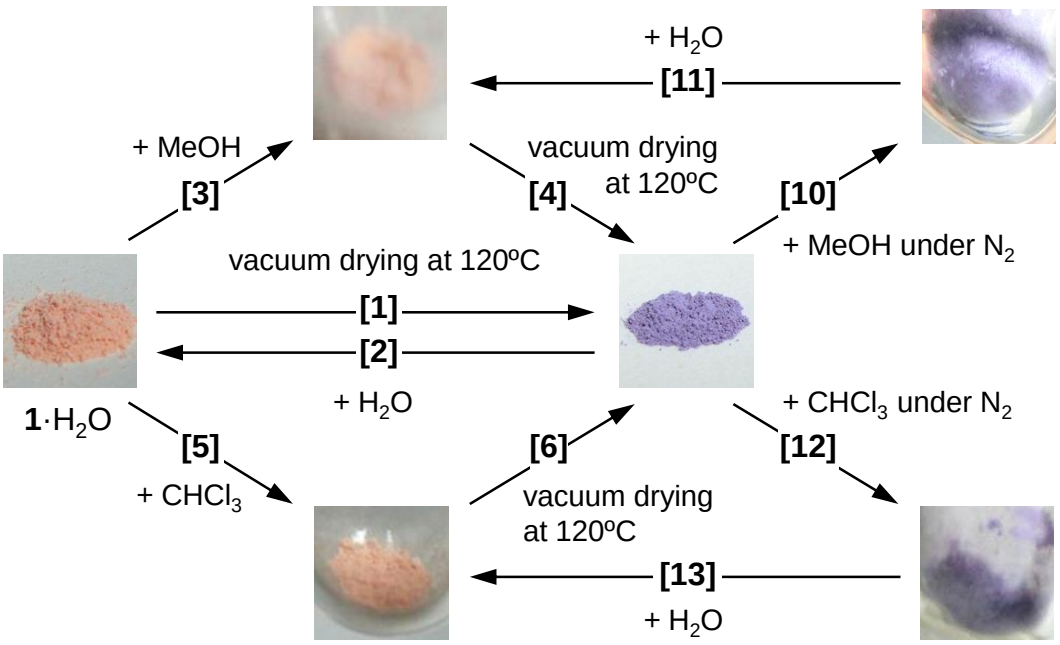

Fig. S10 Guest responsivity of $\{Co(stpy)_2[Ni(CN)_4]\}$.

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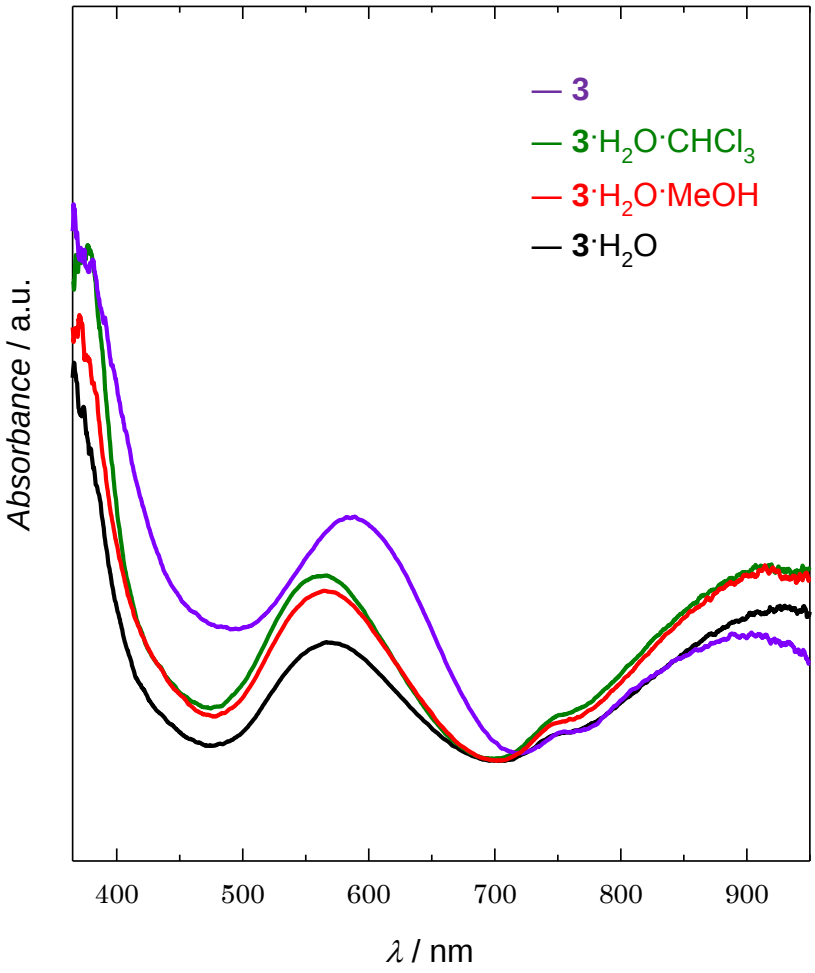

Fig. S11 Solid-state reflectance spectra of {Ni(Cholpy)$_2$[Ni(CN)$_4$]·H$_2$O} (**3**·H$_2$O) and the resulting compounds through the guest adsorption and desorption processes.