Supporting Information for

Pillar-template strategy switching the redox activity and magnetic properties of trisphenylamine-based coordination polymers

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5. References

1. Materials and Measurements

FT-IR spectra were measured on a Thermo Nicolet iS10 spectrometer from 4000 to 400 cm⁻¹. Elemental analyses were recorded using an Elementar Vario MICRO analyzer. ¹H NMR spectra were recorded on a Bruker DPX 400 MHz spectrometer with internal standard tetramethylsilane (TMS) and solvent signals as internal references at room temperature. Mass spectra were recorded on a Bruker Autoflex^{II} TM instrument for MALDI-TOF-MS or on a Varian MAT 311A instrument for ESI-MS. The experimental powder X-ray diffraction (PXRD) patterns were obtained on a Bruker D8 Advance diffractometer (Cu K \langle radiation, λ = 1.5405 Å) in the region of 3-50°. Simulated PXRD spectra were obtained from the single crystal data using the program Mercury 3.8. Magnetic susceptibility measurements for freshly prepared samples of compounds 1-4 were measured on a Quantum Design MPMS-SQUID-VSM magnetometer. The variable-temperature magnetic susceptibilities were carried out in the range of 2-300 K. The field dependent magnetizations were collected with external direct current (dc) field ranging from 0 to 70 kOe. For dynamic magnetic susceptibilities, the oscillating alternating current (ac) field is 2 Oe and the frequency ranges from 1 to 999 Hz. Diamagnetic contributions of sample holder and the constituent atoms were estimated using Pascal's constants.^[S1] Solid state cyclic voltammetry (CV) was performed using a BASi Epsilon Electrochemical Analyzer with ferrocene (Fc) as an internal reference. Measurements were conducted under an inert Ar atmosphere using a conventional 3-electrode cell with a glassy carbon working electrode containing the immobilized solid, a Pt wire auxiliary electrode and an Ag/Ag⁺ quasi reference electrode. A 0.1 M tetrabutylammonium hexafluorophosphate ([n-Bu₄N]PF₆/CH₃CN) electrolyte was employed with a scan rate of 20-500 mV/s. Soloid-state vis-NIR spectra were obtained on the samples at room temperature using a CARY5000 spectrophotometer equipped with a Harrick Praying Mantis accessory over the wavenumber range 5000-40000 cm⁻¹. BaSO₄ was used to acquire the baseline spectrum. Spectra are reported as the Kubelka-Munk transform, where F(R) = (1-R)2/2R (R is the diffuse reflectance of the sample compared to BaSO₄).

2. X-ray Crystallography

The crystal structures were determined from different machines for compounds 1–4. One is Siemens (Bruker) SMART CCD diffractometer using monochromated MoK α radiation (λ = 0.71073 Å). The other is Bruker D8 VENTURE diffractometer with a PHOTON CMOS detector equipped with METALJET-X-ray Source (Ga, λ = 1.34138 Å). The raw frame data were integrated into SHELX-format reflection files and corrected for Lorentz and polarization effects using SAINT.^[S2] Corrections for incident and diffracted beam absorption effects were applied using SADABS.^[S3] No change of diffraction intensity was evidence during data collection. The structures were solved and refined against F² by the full-matrix least-squares using the SHELXL-2016/6 program.^[S4] The positions of the metal atoms and their first coordination spheres were located from direct method E-maps. All non-hydrogen atoms were refined with anisotropic thermal parameters, and hydrogen atoms of the organic ligands were calculated theoretically onto the specific atoms and refined isotropically with fixed thermal factors. The diffuse electron densities resulting from these residual molecules in compounds 1-3 were removed from the data set using the SQUEEZE routine of PLATON and refined further using the data generated. The solvent contents are not represented in the unit cell formula in the crystal data. The final formulas of compounds 1-3 were calculated from the SQUEEZE results and TGA data. More details for the crystal data, data collection parameters, and refinement statistics of compounds 1-4 are listed in Table 1. Selected bond lengths and bond angles are given in Tables S1-S4 (supporting information). CCDC reference numbers: 1959693 (1), 1959694 (2), 1959695 (3), 1959696 (4).

Table S1 Selected bond lengths (Å) and bond	angles (°) for	compound 1
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Compound 1			
Co1—N4 ⁱ	2.150 (3)	N4 ⁱ —Co1—O4	88.0 (2)
Co1—N3 ⁱⁱ	2.155 (3)	N2—Co1—O4	141.19 (15)
Co1—N2	2.162 (3)	O1—Co1—O4	134.34 (15)
Col—Ol	2.208 (3)	N4 ⁱ —Co1—O5	89.61 (18)
Co1—O4	2.209 (5)	N3 ⁱⁱ —Co1—O5	89.51 (17)
Co1—O5	2.232 (4)	N2—Co1—O5	85.64 (14)
Co1—O2	2.333 (3)	O1—Co1—O5	168.56 (16)
N4 ⁱ —Co1—N3 ⁱⁱ	171.76 (14)	O4—Co1—O5	56.04 (16)

N4 ⁱ —Co1—N2	98.65 (12)	N4 ⁱ —Co1—O2	87.47 (13)
N3 ⁱⁱ —Co1—N2	89.45 (12)	N3 ⁱⁱ —Co1—O2	87.34 (12)
N4 ⁱ —Co1—O1	86.36 (15)	N2—Co1—O2	139.36 (14)
N3 ⁱⁱ —Co1—O1	95.98 (13)	01—Co1—O2	55.76 (14)
N2—Co1—O1	84.40 (12)	O4—Co1—O2	78.76 (16)

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

 Table S2 Selected bond lengths (Å) and bond angles (°) for compound 2

Compound 2			
Nil—O4	2.046 (3)	N3—Ni1—N4	170.76 (12)
Nil—N2	2.056 (3)	04—Ni1—O1	176.1 (4)
Ni1—N3	2.100 (3)	N2—Ni1—O1	92.7 (4)
Nil—N4	2.115 (3)	N3—Ni1—O1	84.7 (3)
Nil—Ol	2.189 (14)	N4—Ni1—O1	95.6 (3)
Nil—O2	2.237 (14)	04—Ni1—O2	118.7 (4)
N1—C12 ⁱ	1.397 (4)	N2—Ni1—O2	150.4 (3)
04—Ni1—N2	89.85 (11)	N3—Ni1—O2	80.1 (3)
O4—Ni1—N3	92.22 (12)	N4—Ni1—O2	92.2 (3)
N2—Ni1—N3	91.72 (11)	01—Ni1—O2	58.4 (5)
O4—Ni1—N4	86.99 (13)	01—Ni1—O2	58.4 (5)

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

Table S3 Selected bond lengths (Å) and bond angles (°) for compound 3

Zn1—O1	2.467 (4)	N3—Zn1—O2	86.74 (14)
Zn1—O2	2.192 (3)	N3—Zn1—O4	87.2 (3)
Zn1—O4	2.292 (8)	N3—Zn1—O5	89.3 (2)
Zn1—O5	2.259 (5)	N5 ⁱ —Zn1—O1	86.54 (12)
Zn1—N3	2.128 (4)	N5 ⁱ —Zn1—O2	96.08 (13)
Zn1—N5 ⁱ	2.120 (3)	N5 ⁱ —Zn1—O4	81.6 (3)
Zn1—N6 ⁱⁱ	2.127 (3)	N5 ⁱ —Zn1—O5	88.85 (19)
O2—Zn1—O1	54.46 (12)	N5 ⁱ —Zn1—N3	166.86 (14)
O2—Zn1—O4	133.6 (2)	N5 ⁱ —Zn1—N6 ⁱⁱ	92.37 (12)
02—Zn1—O5	173.71 (18)	N6 ⁱⁱ —Zn1—O1	142.03 (12)
O4—Zn1—O1	79.2 (2)	N6 ⁱⁱ —Zn1—O2	88.06 (12)
05—Zn1—O1	130.04 (17)	N6 ⁱⁱ —Zn1—O4	138.2 (2)
05—Zn1—O4	50.9 (2)	N6 ⁱⁱ —Zn1—O5	87.83 (17)
N3—Zn1—O1	84.68 (13)	N6 ⁱⁱ —Zn1—N3	100.56 (13)

Symmetry codes: (i) -*x*+1/2, -*y*+1/2, -*z*+1; (ii) -*x*+1, *y*-1, -*z*+1/2; (iii) -*x*+1, *y*+1, -*z*+1/2.

Table S4 Selected bond	lengths (Å)	and bond angles	(°) for compound 4
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Compound 4			
Co1—Cl1	2.3181 (11)	O2—Co1—C1	27.62 (12)
Co1—O1	2.105 (3)	N1—Co1—Cl1	105.56 (9)
Co1—O2	2.311 (3)	N1—Co1—O1	154.34 (12)
Co1—N1	2.092 (3)	N1—Co1—O2	95.27 (11)
Co1—N3 ⁱ	2.208 (3)	N1—Co1—N3 ⁱ	93.33 (11)
Co1—N4 ⁱⁱ	2.168 (3)	N1—Co1—N4 ⁱⁱ	87.95 (11)
Cl1—Co1—C1	131.48 (12)	N1—Co1—C1	122.89 (14)

O1—Co1—Cl1	100.11 (9)	N3 ⁱ —Co1—Cl1	91.64 (9)
O1—Co1—O2	59.08 (11)	N3 ⁱ —Co1—O2	84.82 (11)
01—Co1—N3 ⁱ	85.74 (12)	N3 ⁱ —Co1—C1	83.68 (13)
01—Co1—N4 ⁱⁱ	90.23 (12)	N4 ⁱⁱ —Co1—Cl1	94.41 (8)
O1—Co1—C1	31.48 (14)	N4 ⁱⁱ —Co1—O2	88.45 (11)
O2—Co1—Cl1	159.05 (8)	N4 ⁱⁱ —Co1—N3 ⁱ	173.24 (12)

Symmetry codes: (i) -x+1, y-1, -z+3/2; (ii) x-1/2, -y+1/2, z+1/2; (iii) -x+1, y+1, -z+3/2; (iv) x+1/2, -y+1/2, z-1/2; (v) -x+1, -y, -z+2.



Fig. S1 The detailed analysis for the interpenetrated networks of 1.



Fig. S2 The coordination environments of m-NPy₃ and central Ni(II) (a), 1D chain (b), 2D network (c) containing the m-NPy₃ ligand coordinated to Ni(II) centers and the interpenetrated networks along a axis (d) of 2. All of the hydrogen atoms are omitted for clarity, carbon - gray, nitrogen -blue, oxygen - red, nickel - green.



Fig. S3 The coordination environments of m-NPy₃ and central Zn(II) (a), 1D chain (b), 2D network (c) containing the m-NPy₃ ligand coordinated to Zn(II) centers and the interpenetrated networks along a axis (d) of **3**. All of the hydrogen atoms are omitted for clarity, carbon - gray, nitrogen -blue, oxygen - red, zinc - turquiose.



Fig. S4 The coordination environments of m-NPy₃ and central Co(II) of 4. All of the hydrogen atoms are omitted for clarity, carbon - gray, nitrogen -blue, oxygen - red, chlorine - bright green, cobalt - violet.

3. Characterizations



Fig. S5 The PXRD patterns obtained from the as-synthesized solids (black lines) and the simulated ones (red lines) from the single crystal data of **1-4**.



Fig. S6 Observed X-ray powder diffraction patterns of 1 in different solvents for 24 hours.



Fig. S7 Observed X-ray powder diffraction patterns of **1** in different pH solutions (pH = 3-12) for 24 hours.



Fig. S8 Observed X-ray powder diffraction patterns of 4 in different solvents for 24 hours.



Fig. S9 Observed X-ray powder diffraction patterns of **4** in different pH solutions (pH = 3-10) for 24 hours.



Fig. S10 Thermogravimetric analyses of 1-4 were conducted from 30 to 1000 °C under N_2 atmosphere. These four compounds exhibit similar thermal behavior.



Fig. S11 The PXRD patterns (a) and the FT-IR spectra (b) of **1** (black lines), **4** (red lines) and SC-SC solids (blue lines).



Fig. S12 Solid state CV upon cycling anodically (a) and cathodically (b) at 100 mV/s three times of **1**.



Fig. S13 Solid state CV upon cycling cathodically at 100 mV/s to 1440 mV (a) and to 2000 mV (b) three times of **1**.



Fig. S14 Solid state CV upon cycling anodically (a) and cathodically (b) at 100 mV/s three times of **4**.



Fig. S15 Solid state CV upon cycling cathodically at 100 mV/s to 1250 mV (a) and to 2000 mV (b) three times of 4.



Fig. S16 M versus HT^{-1} plots for 1, 2 and 4 at 1.8, 3.0 and 4.0 K, respectively.



Fig. S17 Plot of out-of-phase (χ'') *versus* frequency at 1.8 K for 1 (a) and 4 (b) under the application of variable dc fields.



Fig. S18 Temperature dependence of the in-phase (χ') and out-of-phase (χ'') ac susceptibility signals for **1** (a and b) and **4** (c and d) under zero dc field.



Fig. S19 Temperature dependence of the in-phase (χ') (a) and out-of-phase (χ'') (b) ac susceptibility signals for **1** under 1 kOe dc field.



Fig. S20 Cole-Cole plots for **1** under 1 kOe dc field. The solid lines represent the fit to the generalized Debye model at the indicated temperatures (1.8, 2.2 and 2.6 K). Inset: $\ln \tau vs T^{-1}$ plot for **1**. The solid line represents the fit to the Arrhenius law.



Fig. S21 Temperature dependence of the in-phase (χ') (a) and out-of-phase (χ'') (b) ac susceptibility signals for 4 under 1 kOe dc field.



Fig. S22 Cole-Cole plots for **4** under 1 kOe dc field. The solid lines represent the fit to the generalized Debye model at the indicated temperatures (1.8, 2.2, 2.6 and 3.0 K). Inset: $\ln \tau vs$ T^{-1} plot for **4**. The solid line represents the fit to the Arrhenius law.

 Table S5 Fitting of the Cole-Cole plots for 1 with a generalized Debye model in the temperature range 1.8-2.6 K under 1 kOe dc field.

Τ/	χ_s / cm ³ mol ⁻¹	$\chi_T/\mathrm{cm}^3\mathrm{mol}^{-1}$	α	τ	lnτ
К					
1.8	0.0665	0.856	0.0977	0.000393	-7.84272
2.2	0.0634	0.707	0.0910	0.000239	-8.34072
2.6	0.0678	0.609	0.0865	0.000163	-8.7236

 Table S6 Fitting of the Cole-Cole plots for 4 with a generalized Debye model in the temperature range 1.8-3.0 K under 1 kOe dc field.

Τ/	χ_s / cm ³ mol ⁻¹	$\chi_T/\mathrm{cm}^3\mathrm{mol}^{-1}$	α	τ	lnτ
K					
1.8	0.124	1.015	0.176	0.00167	-6.39289
2.2	0.0925	0.844	0.176	0.000729	-7.22384
2.6	0.0824	0.723	0.172	0.000321	-8.04407
3.0	0.0780	0.627	0.163	0.000147	-8.82508

Molecular Structure	Dimension	Energy	pre-exponential	References
		barrier (E_{a} ,	factor (τ_0, s)	
		K)		
$\{[Co(L1)(2,2'-bipy)]\cdot 0.5DMF\}_n$	1D	13.94	3.47×10^{-6}	S5
${[Co(bimb)(H_2O)_4]\cdot(L2)\cdot 2DMF}_n$	1D	8.82	$2.60 imes 10^{-6}$	S5
[Co(tdmmb)(bpe1)][BF ₄] ₂ ·3CH ₃ CN	1D	19.00	7.50×10 ⁻⁶	S6
[Co(dca) ₂ (bim) ₂]	2D	7.67	1.54×10^{-6}	S7
$[Co(dca)_2(bmim)_2]_n$	2D	19.88	$0.63 imes 10^{-6}$	S7
$[Co(dca)_2(atz)_2]$	2D	7.34	$1.70 imes 10^{-6}$	S8
[Co(azbbpy)(4,4'-bipy) _{0.5} (DMF)(NCS) ₂]	2D	14.00	$1.20 imes 10^{-6}$	S9
[Co(azbbpy)(bpe2) _{0.5} (DMF)(NCS) ₂]	2D	8.35	$1.70 imes 10^{-6}$	S9
$[Co(ppad)_2]_n$	2D	16.37	5.03×10^{-6}	S10
{[Co(bmzbc) ₂]·2DMF}	2D	11.80	1.30×10^{-5}	S11
$\{[Co(HL)(bpy)(H_2O)_2)]$ ·DMF $\}$	2D	13.87	8.98 ×10 ⁻⁶	S12
$\{[Co(HL)(bpe2)] \cdot 0.5bpe\}_n$	2D	8.70	1.43 ×10 ⁻⁵	S12
$Co(NCS)_2)_3(k^3-TPT)_4$	3D	7.00	8.68×10^{-6}	S13

Table S7 Recent reported coordination polymer for Co(II)-based SIMs.

Notes: $H_2L1 = 2,2'$ -[benzene-1,4-diylbis(methanediylsulfanediyl)]dibenzoic acid; 2,2'-bipy = 2,2'-bipyridine; $H_2L2 = 2,2'$ -(1,4- Phenylenebis(methylene))bis(sulfanediyl)dinicotinicacid; bimb = 1,4-bis(benzoimidazo-1-ly)benzene; tdmmb = 1,3,10,12-tetramethyl-1,2,11,12-tetra-aza[3](2,6)pyridino[3](2,9)-1; 10-phenanthrolinophan-2,10-diene; bpe1 = 1,2-di(4-pyridyl)ethane); dca = dicyanamide; bim = 1-benzylimidazole; bmim = 1-benzyl-2-methylimidazole; atz = 2-amino-1,3,5- triazine; azbbpy = 1,3-bis(4- pyridyl)azulene; 4,4'-bipyridyl; azbbpy = 1,3-bis(4- pyridyl)azulene; bpe2 = 1,2-bis(4-pyridyl)ethylene; ppad = N³- (3-pyridoyl)-3-pyridinecarboxamidrazone; bmzbc = 4-(benzimidazole-1-yl)benzoate; $H_3L = 1,3$ -bis(2-carboxylphenoxy)benzoic acid; bpy = 4,4'-bipyridine;; TPT = 2,4,6-tris(4-pyridyl)-1,3,5-triazine.

4. Synthesis and characterizations of ligand

Synthesis of ligand. Tris(4-bromophenyl)amine (5.00 g, 10.4 mmol), 4- pyridylboronic acid (5.08 g, 36.3 mmol), Pd(PPh₃)₄ (0.6 g, 0.520 mmol) and anhydrous potassium carbonate (7.19 g, 52.0 mmol) were suspended in a degassed mixture of toluene (120 mL), ethanol (80.0 mL) and water (40.0 mL) under nitrogen. The reaction mixture was heated at reflux for 48 h upon which a colour change from yellow to dark brown was observed. The solvent was removed under vacuum and the residue dissolved in dichloromethane and water. The product was extracted with dichloromethane and the combined organic layers dried over anhydrous sodium sulfate, filtered and the solvent were removed under the vacuum to yield a brown solid. The ligand was purified by flash column chromatography (dichloromethane, gradient to 5% MeOH with 1% trimethylamine) to yield the product as a crystalline yellow solid (4.0 g, 80%). Elemental analysis calculated for C₃₃H₂₄N₄ (Mr = 476.57): C 83.17, N 11.76, H 5.08%. Found: C 82.98, N 11.58, H 4.90%. IR (KBr, cm⁻¹): 3414 (s), 3035 (m), 1900 (m), 1590 (s), 1510 (m), 1471 (m), 1279 (m), 1180 (w), 1112 (m), 1002 (s), 796 (m). ¹H NMR (CDCl₃, ppm): δ 8.58 (d, 4H, pyridyl), 7.90 (d, 4H, phenyl), 7.54 (d, 4H, pyridyl), 7.31 (d, 4H, phenyl).



Fig. S23 The ¹H NMR spectrum for m-NPy₃ in CDCl₃.

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