Supporting Information

Three unique two-fold interpenetrated three-dimensional networks with PtS-type topology constructed from $[M(CN)_4]^{2-}$ (M = Ni, Pd, Pt) as "square-planar" building blocks

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[†] Electronic supplementary information (ESI) available: Additional crystallographic diagrams,

TGA diagram and adsorption isotherm. See DOI: ?

Section S1: Single crystal X-ray diffraction analysis for 1-3.

The single crystals of **1-3** were very easy to pulverize to powder after they were removed from the mother liquor and exposed in air. Therefore, as soon as the well-shaped single crystal was removed from the mother liquor, it was immediately introduced into a glass capillary having an open end and fixed. Then the capillary was soon filled with the mother liquor, and sealed for single-crystal X-ray structure determination at 291(2) K.

Diffraction data for 1-3 were collected on a Bruker Smart Apex II CCD diffractometer equipped with MoK_{α} ($\lambda = 0.71073$ Å) radiation. Diffraction data analysis and reduction were performed within SMART and SAINT (*SMART, SAINT and XPREP. Area detector and data integration and reduction software*; Bruker Analytical Instruments Inc.: Madison, WI, 1995.). Correction for Lorentz, polarization, and absorption effects were performed within SADABS (G.M. Sheldrick. *SADABS. Empirical adsorption correction program for area detector data*; University of Göttingen: Göttingen, Germany, 1996.). Structures were solved using Patterson method within SHELXL-97 and refined using SHELXL-97 (*SHELXL97. Program for crystal structural solution and refinement*; Bruker Analytical Instruments Inc.: Madison, WI, 1997.).



C1 (b)

Figure S1. ORTEP diagrams of (a) 1, (b) 2, and (c) 3. Displacement ellipsoids are drawn at the 30% probability level.

(c)

Section S2: Thermogravimetric analysis.

Thermogravimetric analysis (TGA) was carried out on a TA Instrument Perkin-Elmer Diamond analyzer to determine the thermal stability of 1. The temperature was ramped at 2 °C/min from 20 to 730 °C under nitrogen atmosphere. After removed from the mother liquor, the samples were immediately performed on a TGA analyzer. Therefore, the negligible weight loss (1.0%) until 100°C can be attributed to the fact that the samples were not dried completely before TGA experiment.



Figure S2. Thermogravimetric curve of 1.

Section S3: Nitrogen adsorption of 1.

A nitrogen adsorption isotherm of **1** was measured volumetrically at 77 K with 99.99% N_2 on a Micromeritics ASAP 2020 M+C. The BET plot is shown in Figure S3, and the parameters presented in Tables S1. Data of N_2 adsorption and desorption for **1** is listed in Table S2.



Figure S3. BET surface area plot for 1.

Table S1. Fitted parameters for the BET model.

BET Surface Area	$8.3243 \pm 0.1023 \text{ m}^2/\text{g}$
Slope	11.014160 ± 0.142692 g/mmol
Y-Intercept	0.707367 ± 0.019808 g/mmol
С	16.570634
Qm	0.08531 mmol/g
Correlation Coefficient	0.9994969
Molecular Cross-Sectional Area	0.1620 nm ²

 Table S2. Nitrogen isotherm data for 1.

Relative pressure (p/p₀)	Absolute pressure (kPa)	Quantity adsorbed (mmol/g)
0.000071887	0.0072809	0.00178
0.000563641	0.0570906	0.00592
0.001087136	0.1101189	0.00795
0.009675765	0.9801024	0.02200
0.030254380	3.0646804	0.03476
0.060169064	6.0950644	0.04743
0.086692976	8.7821279	0.05665
0.100647980	10.1959809	0.06138
0.120494388	12.2067169	0.06693
0.140544459	14.2381604	0.07222
0.160604620	16.2706007	0.07753
0.180303009	18.2665542	0.08254
0.200813611	20.3447427	0.08563
0.244484177	24.7695386	0.09389
0.302186115	30.6160928	0.10242
0.353533168	35.8187830	0.11061
0.400344676	40.5623230	0.11586
0.449895041	45.5835421	0.12127
0.499912915	50.6520026	0.12591
0.550054335	55.7338002	0.13022
0.599816928	60.7767094	0.13471
0.649778049	65.8398603	0.13862
0.700156400	70.9445320	0.14401

0.750286640	76.0240634	0.14982
0.800130179	81.0745444	0.15933
0.820404841	83.1289088	0.16355
0.850150718	86.1429601	0.17163
0.875269082	88.6881209	0.18097
0.900080830	91.2022132	0.19587
0.925312910	93.7588964	0.21121
0.950131645	96.2736967	0.23566
0.974890630	98.7824428	0.29010
0.980418879	99.3426019	0.31913
0.990307536	100.3445868	0.39162
0.994861406	100.8060155	0.46351
0.981270064	99.4288497	0.34317
0.969022518	98.1878464	0.28619
0.944745863	95.7279732	0.23959
0.926045641	93.8331416	0.22184
0.900576733	91.2524613	0.20535
0.875476598	88.7091478	0.19597
0.850391161	86.1673233	0.18825
0.825544559	83.6496994	0.18227
0.800272244	81.0889394	0.17842
0.754471896	76.4481415	0.17106
0.700264816	70.9555174	0.16145
0.650237411	65.8864060	0.15510
0.600400256	60.8365719	0.14939
0.550405656	55.7707845	0.14323
0.500478516	50.7118325	0.13655
0.450478174	45.6454633	0.13026
0.400569826	40.5884155	0.12318
0.350584422	35.5235598	0.11536
0.300326573	30.4310982	0.10555
0.250643710	25.3968980	0.09569
0.200416096	20.3075001	0.08354
0.140712118	14.2578935	0.06718