Supporting Information

Preparation:

$\{[La_4(ox)_3Ni_3(IDA)_6\ (H_2O)_6]\}_n \cdot 3nH_2O$ (**1**), Iminodiacetic acid (0.27 g, 2.0 mmol), oxalic acid (0.25 g, 2.0 mmol), $La(NO_3)_3 \cdot 6H_2O$ (1.0 mmol) and $Ni(NO_3)_2 \cdot 6H_2O$ (0.29 g, 1.0 mmol) were dissolved in 10.0 ml water with stirring at room temperature. When the pH of the solution was adjusted to about 4 by 1.0 mol/L NaOH, the solution was transferred and sealed in a 25 mL Teflon-lined stainless steel container. The container was heated to 180 °C and held at that temperature for 100 hours, then cooled to 100 °C at a rate of 3 $°C \cdot h^{-1}$ and held for 16 hours, followed by further cooling to 30 °C at a rate of 5 $°C \cdot h^{-1}$. Purple needle crystals were collected by filtration and air-dried in 25% yield. IR(KBr, cm^{-1}): 3436 (s), 3322.8 (s), 2964 (w), 2929 (w), 1683 (m), 1640 (s), 1619 (s), 1580 (s), 1458 (m), 1414 (s), 1310 (s), 1110 (m), 1076 (w), 1001 (w), 948 (m), 793 (m), 734 (w), 525 (m); elemental analysis Calcd (%) for $C_{15}H_{24}La_2N_3Ni_{1.5}O_{22.5}$ (972.26): C 18.53, N 4.32, H 2.49; found: C 18.62, N 4.21, H 2.38.

$\{[La_4(ox)_3Ni_3(IDA)_6\ (H_2O)_6]\}_n$ (**1**a) Complex **1**a was prepared by heating crystals of **1** at 230 ^oC in open glass thin tube for 30 minutes. When cooled to 80 ^oC, the crystal was quickly sealed into the tube.

$\{[Nd_4Ni_3(IDA)_6(ox)_3(H_2O)_6]\}_n \cdot 2nH_2O$ (**2**) Complex **2** was prepared in the same way as for **1**, except that $Nd(NO_3)_3$ (1.0 mmol) was used to replace $La(NO_3)_3$. Purple crystals were obtained in 32% yield. IR(KBr, cm^{-1}): 3396 (s), 3325 (s), 3288 (s), 2967 (w), 2930 (w), 1683 (m), 1647 (s), 1622 (s), 1597 (s), 1460 (m), 1415 (s), 1312 (s), 1276 (w), 1112 (m), 1076 (w), 1001 (w), 948 (m), 794 (m), 734 (w), 526 (m); elemental analysis Calcd (%) for $C_{15}H_{23}N_3Nd_2Ni_{1.5}O_{22}$ (973.9): C 18.50, N 4.31, H 2.38; found: C 18.48, N 4.35, H 2.36.

$\{[Eu_4Ni_3(IDA)_6(ox)_3(H_2O)_6]\}_n \cdot 2nH_2O$ (**3**) Complex **3** was prepared in the same way as for **1**, except that $Eu(NO_3)_3$ (1.0 mmol) was used to replace $La(NO_3)_3$. Blue crystals

were obtained in 30% yield. IR(KBr, cm^{-1}): 3435 (s), 3327 (s), 3286 (s), 2968 (w), 2932(w), 1685 (m), 1649 (s), 1586 (s), 1461 (s), 1416 (s), 1383 (m), 1313 (s), 1276 (m), 1232 (w), 1114 (m), 1075 (w), 1002 (m), 949 (m), 927 (m), 795 (m), 734 (s), 527 (m); elemental analysis Calcd(%) for $C_{15}Eu_2H_{23}N_3Ni_{1.5}O_{22}$ (989.4): C 18.21, N 4.25, H 2.34; found: C 18.40, N 4.31, H 2.44.

$\{[Gd_4Ni_3(IDA)_6(ox)_3(H_2O)_6]\}_n \cdot 2nH_2O$ (**4**) Complex **4** was prepared in the same way as for **1**, except that $Gd(NO_3)_3$ (1.0 mmol) was used to replace $La(NO_3)_3$. Blue crystals were obtained in 28% yield. IR(KBr, cm^{-1}): 3430 (s), 3327 (s), 2968 (w), 2932 (w), 1684 (m), 1641 (s), 1619 (s), 1582 (s), 1461 (m), 1415 (s), 1314 (s), 1113 (m), 1075 (w), 1004 (w), 949 (m), 796 (m), 735 (w), 539 (m); elemental analysis Calcd (%) for $C_{15}H_{23}Gd_2N_3Ni_{1.5}O_{22}$ (999.9): C 18.02, N 4.20, H 2.32; found: C 17.78, N 4.16, H 2.54.

$\{[Pr_2Ni_2(IDA)_4(ox)(H_2O)_2]\}_n \cdot 2nH_2O$ (**5**) Iminodiacetic acid (0.266 g, 2.0 mmol), $Pr(NO_3)_3.5H_2O$(0.417g, 1.0 mmol) and $Ni(NO_3)_2 \cdot 6H_2O$ (0.297 g, 1.0 mmol) were dissolved in 10 mL water while stirring at room temperature. When the pH of the mixture was adjusted to about 4 with 1.0 $mol \cdot L^{-1}$ NaOH, the solution was transferred and sealed in a 25 mL Teflon-lined stainless steel container. The container was heated to 180 °C and held at that temperature for 100 hours, then cooled to 100 °C at a rate of 3 $°C \cdot h^{-1}$ and held for 16 hours, followed by further cooling to 30 °C at a rate of 5 $°C \cdot h^{-1}$. Blue needles crystals of **5** were obtained in 36% yield based on $Pr(NO_3)_3.5H_2O$. IR spectra (KBr, cm^{-1}): 3443 (s), 3315 (s), 2970 (w), 2931 (w), 1683 (m), 1641 (s), 1601 (s), 1581 (s), 1458 (m), 1409 (s), 1311 (s), 1114 (m), 1074 (w), 998 (w), 942 (m), 794 (m), 734 (w), 534 (m); elemental analysis Calcd (%) for $C_{18}H_{24}N_4Ni_2O_{24}Pr_2$ (1083.7): C 19.95, N 5.17, H 2.60; found: C 19.80, N 5.09, H 2.66.

$\{[Pr_2Ni_2(IDA)_4(ox)(H_2O)_2]\}_n$ (**5**a) Complex **5**a was prepared by heating crystal of **5** at 230 ^oC in open glass thin tube for 30 minutes. When cooled to 80 ^oC, the crystal was quickly sealed into the tube.

{[$La_2Ni_2(IDA)_4(ox)(H_2O)_2$]}$_n$·2n$H_2O$ (**6**) Complex **6** was prepared in the similar way as for **5**, expect for using $La(NO_3)_3.6H_2O$ (0.433g, 1.0 mmol) to replace $Pr(NO_3)_3.5H_2O$. Pruple crystals of **6** were obtained in a 15% yield based on $La(NO_3)_3.6H_2O$. IR spectra (KBr, cm^{-1}): 3444 (s), 3317 (s), 2969 (w), 2929 (w), 1682 (m), 1641 (s), 1603 (s), 1581 (s), 1457 (m), 1409 (s), 1310 (s), 1113 (m), 1074 (w), 998 (w), 943 (m), 792 (m), 734 (w), 531 (m); elemental analysis Calcd (%) for $C_{18}H_{24}La_2N_4Ni_2O_{24}$ (1079.7): C 20.02, N 5.19, H 2.61; found: C 19.87, N 5.07, H 2.72.

{[$Nd_2Ni_2(IDA)_4(ox)(H_2O)_2$]}$_n$·2n$H_2O$ (**7**) Complex **7** was prepared in the same way as for **5**, expect for using $Nd(NO_3)_3.6H_2O$(0.438g, 1.0 mmol) to replace $Pr(NO_3)_3.5H_2O$. Blue crystals were obtained in 28% yield based on $Nd(NO_3)_3.6H_2O$. IR spectra (KBr, cm^{-1}): 3442 (s), 3315 (s), 2971 (w), 2935 (w), 1684 (s), 1642 (s), 1601 (s), 1579 (s), 1457 (m), 1409 (s), 1385 (s), 1311 (s), 1230 (w), 1113 (m), 1074 (w), 998 (w), 942 (m), 925 (m), 897 (w), 795 (m), 734 (s), 601 (w), 534 (m); elemental analysis Calcd (%) for $C_{18}H_{24}N_4Nd_2Ni_2O_{24}$ (1090.3): C 19.89, N 5.15, H 2.60; fonud: C 19.70, N 5.08, H 2.68.

{[$Eu_2Ni_2(IDA)_4(ox)(H_2O)_2$]}$_n$·2n$H_2O$ (**8**) Complex **8** was prepared in the same way as for **5**, expect for using $Eu(NO_3)_3.6H_2O$(0.443g, 1.0 mmol) to replace $Pr(NO_3)_3.6H_2O$. Blue crystals of **8** were obtained in 24% yield based on $Eu(NO_3)_3.6H_2O$. IR spectra (KBr, cm^{-1}): 3434 (s), 3312 (s), 2972 (w), 2936 (w), 1686 (s), 1641(s), 1602 (s), 1576 (s), 1456 (m), 1409 (s), 1387 (s), 1313 (s), 1232 (w), 1115 (m), 1074 (w), 999 (m), 943 (m), 925 (m), 898 (w), 796 (m), 734 (s), 602 (w), 537 (m); elemental analysis Calcd (%) for $C_{18}Eu_2H_{24}N_4Ni_2O_{24}$ (1105.8): C 19.55, N 5.07, H 2.55; found: C 19.36, N 5.01, H 2.65.

{[$Dy_2Ni(IDA)_2(ox)_2(H_2O)_2$]}$_n$·2n$H_2O$ (**9**) Complex **9** was prepared using the same conditions as for **5**, but with additional oxalic acid (0.25 g, 2.0 mmol) and $Dy(NO_3)_3·5H_2O$ (0.428g, 1.0 mmol) instead of $Pr(NO_3)_3·5H_2O$. The blue crystals were collected by filtration and air-dried (yield 25%). IR spectra (KBr, cm^{-1}): 3440 (s), 3348 (s), 2972 (w), 1693 (s), 1641 (s), 1601 (s), 1581 (s), 1466 (s), 1417 (s), 1360 (m),

1331 (m), 1317 (s), 1278 (w), 1236 (m), 1111 (m), 1077 (m), 1006 (m), 948 (m), 900 (m), 804 (s), 744 (s), 599 (w), 543 (m), 496 (m); elemental analysis Calcd (%) for $C_{12}Dy_2H_{18}N_2NiO_{20}$ (894.0): C 16.12, N 3.13, H 2.03; found: C 16.18, N 3.02, H 2.13.

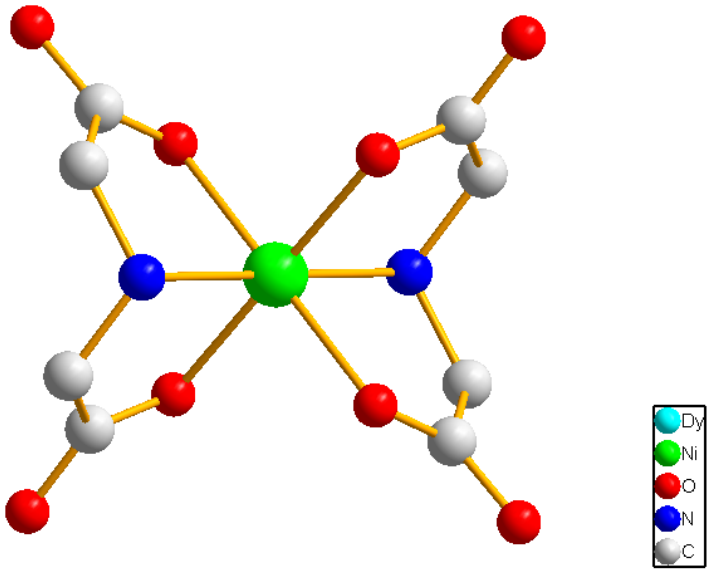

Figure S1 Ball and Stick plot showing the $[Ni(IDA)_2]^{2-}$ metalloligand.

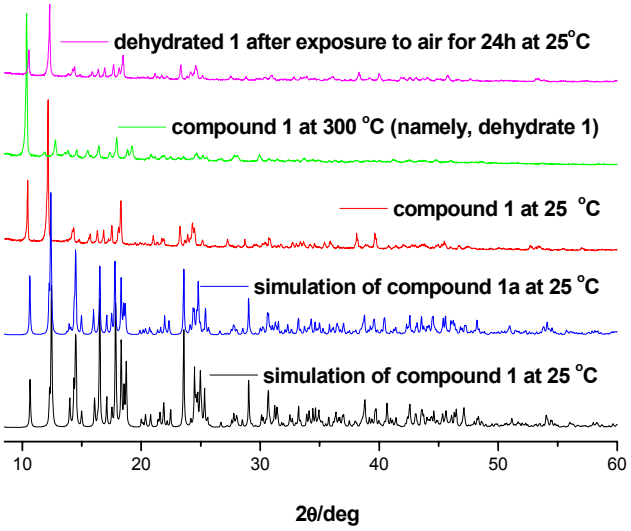

Figure S2 XPRD pattern of **1** at different conditions.

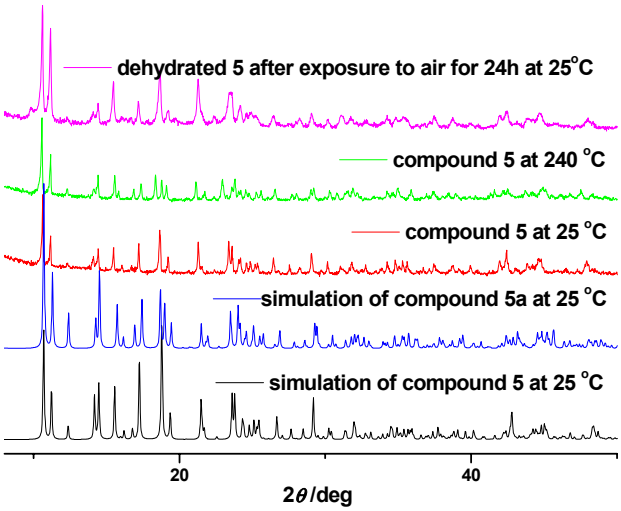

Figure S3 XPRD pattern of **5** at different conditions.