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Supporting Information

Formation of 1D and 3D coordination polymers in the solid state induced by mechanochemical and annealing treatments: bis 3-cyano-pentane-2,4-dionato metal complexes

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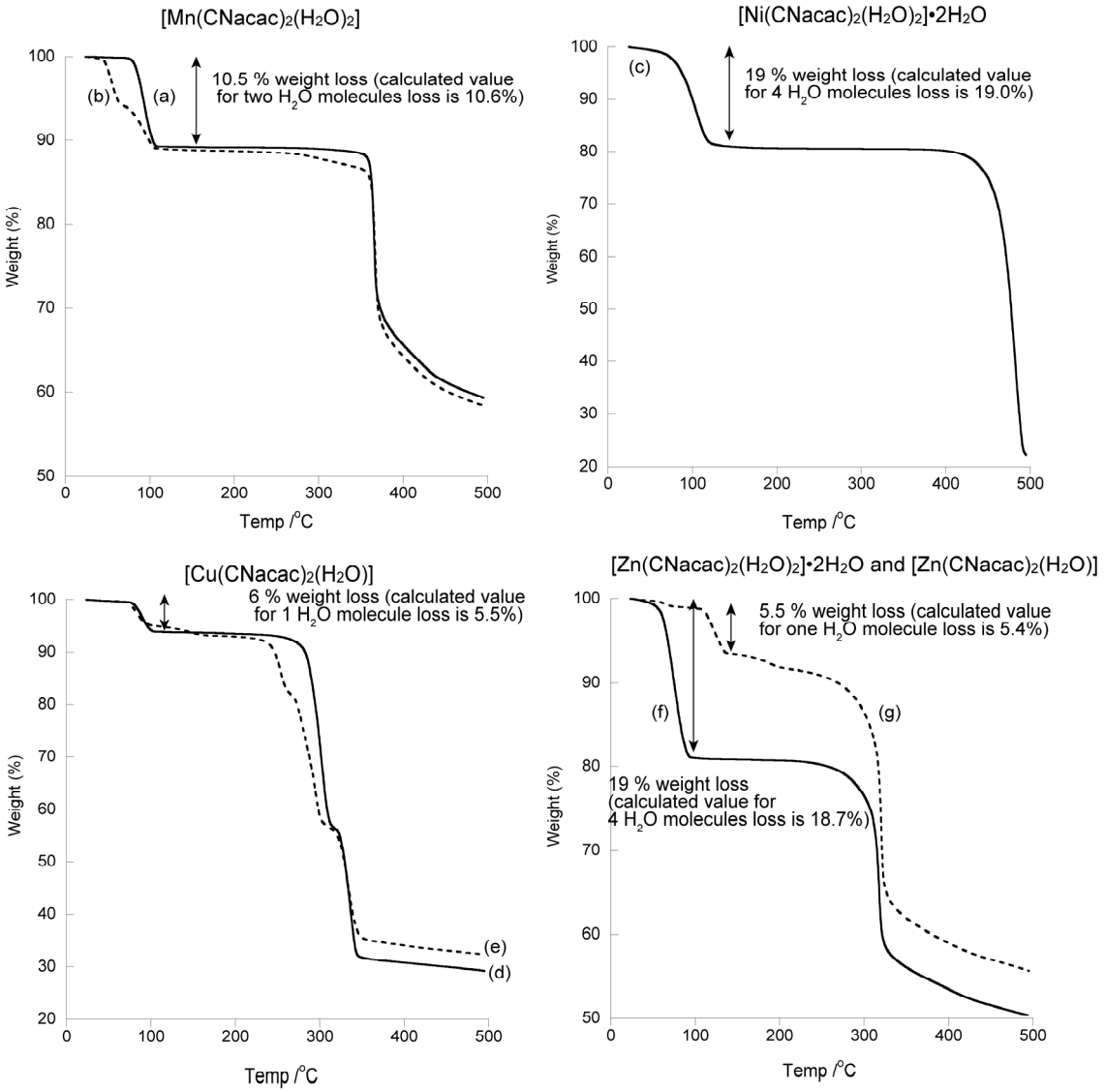

Figure S1. TG curves of (a) $[Mn(CNacac)_2(H_2O)_2]$ (solution reaction), (b) $[Mn(CNacac)_2(H_2O)_2]$ (mechanochemical reaction), (c) $[Ni(CNacac)_2(H_2O)_2]\cdot 2H_2O$ (solution), (d) $[Cu(CNacac)_2(H_2O)]$ (solution), (e) $[Cu(CNacac)_2(H_2O)]$ (mechanochemical), (f) $[Zn(CNacac)_2(H_2O)_2]\cdot 2H_2O$ (solution) and (g) $[Zn(CNacac)_2(H_2O)]$ (mechanochemical).

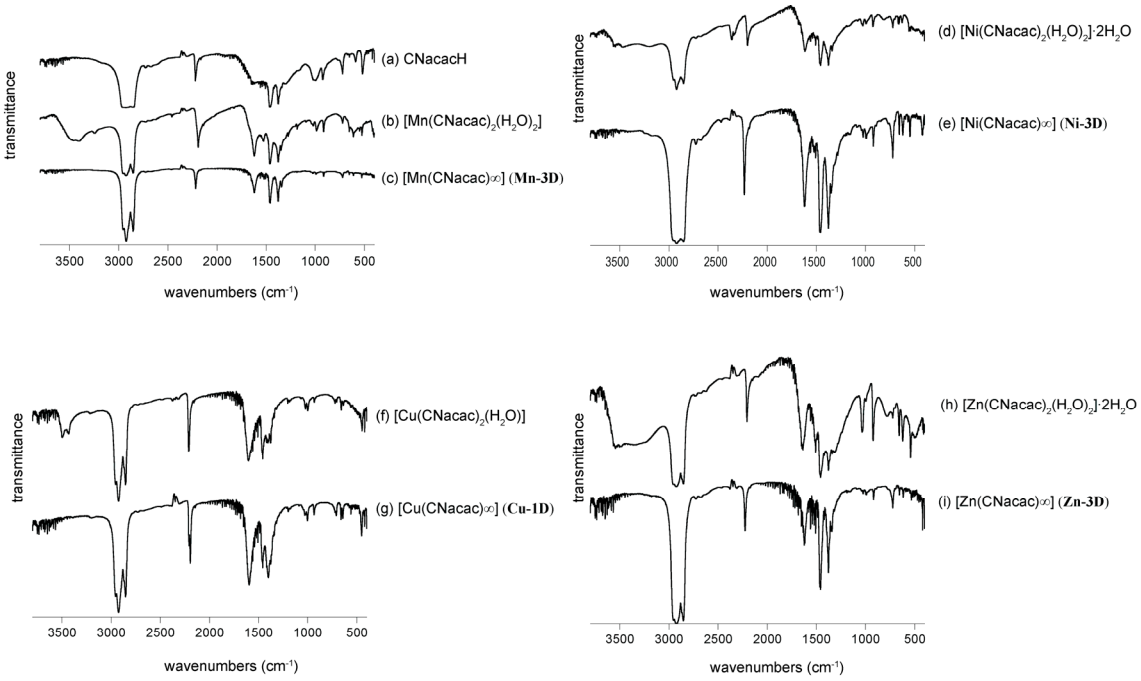

Figure S2. IR spectra of (a) CNacacH, (b) $[Mn(CNacac)_2(H_2O)_2]$, (c) **Mn-3D**, (d) $[Ni(CNacac)_2(H_2O)_2]\cdot 2H_2O$, (e) **Ni-3D**, (f) $[Cu(CNacac)_2(H_2O)]$, (g) **Cu-1D**, (h) $[Zn(CNacac)_2(H_2O)_2]\cdot 2H_2O$ and (i) **Zn-3D,** measured by the Nujol mull method.

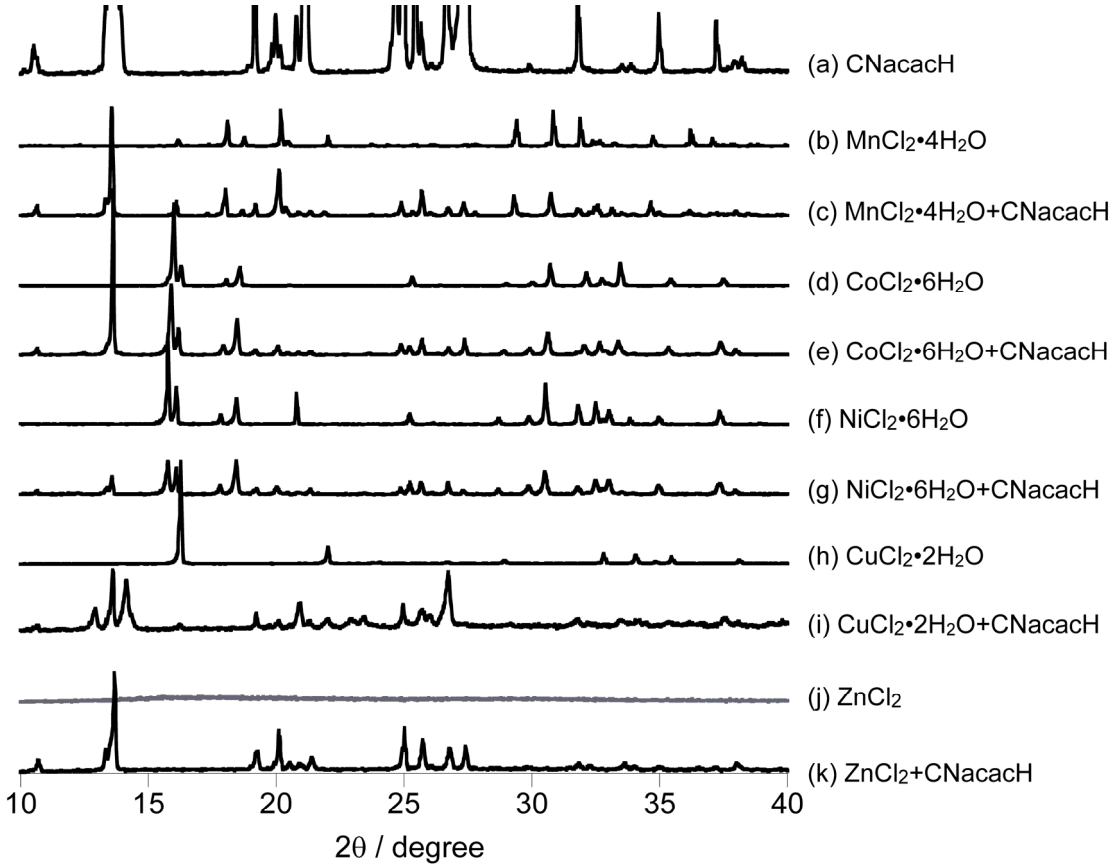

Figure S3. XPRD patterns of (a) CNacacH, (b) $MnCl_2 \cdot 4H_2O$, (c) a mixture of $MnCl_2 \cdot 4H_2O$ and CNacacH after 5 min grinding, (d) $CoCl_2 \cdot 6H_2O$, (e) a mixture of $CoCl_2 \cdot 6H_2O$ and CNacacH after 5 min grinding, (f) $NiCl_2 \cdot 6H_2O$, (g) a mixture of $NiCl_2 \cdot 6H_2O$ and CNacacH after 5 min grinding, (h) $CuCl_2 \cdot 2H_2O$, (i) a mixture of $CuCl_2 \cdot 2H_2O$ and CNacacH after 5 min grinding, (j) $ZnCl_2$ and (k) a mixture of $ZnCl_2$ and CNacacH after 5 min grinding.

Table S1. Crystallographic and experimental data for new complexes synthesized in this study.

Compound	$[Mn(CNacac)_2(H_2O)_2]$	$[Fe_2(\mu\text{-}OMe)_2(CNacac)_4]$	$[Ni(CNacac)_2(H_2O)_2]\cdot 2H_2O$	$[Cu(CNacac)_2(H_2O)]$	$[Zn(CNacac)_2(H_2O)]$
Formula	$C_{12}H_{16}N_2O_6Mn$	$C_{26}H_{30}N_4O_{10}Fe_2$	$C_{12}H_{20}N_2O_8Ni$	$C_{12}H_{14}N_2O_5Cu$	$C_{12}H_{14}N_2O_5Zn$
*M*r	339.21	670.24	379.01	329.79	331.62
Space group	*C*2/c	$P\bar{1}$	*C*2/m	$P\bar{1}$	*F*dd2
a / Å	9.1222(4)	8.016(2)	7.7703(4)	7.971(5)	12.5272(6)
b / Å	12.5848(8)	9.868(2)	21.2435(15)	8.227(5)	27.3148(10)
c / Å	14.0541(8)	10.433(4)	5.1790(3)	12.025(5)	8.4374(3)
α / °	-	84.415(14)	-	77.969(5)	-
β / °	98.495(2)	76.815(14)	101.817(1)	81.610(5)	-
γ / °	-	71.185(11)	-	66.629(5)	-
V / Å^3	1595.72(15)	760.3(4)	836.77(9)	706.2(7)	2887.1(2)
Z	4	1	2	2	8
μ(Mo Kα) / mm^{-1}	0.853	1.013	1.200	1.566	1.720
[a]GOF on F^2	0.929	1.048	1.119	1.099	1.039
[b]*R*1 [on *F*, $I>2\sigma(I)$]	0.0347 (1343)	0.0310 (4545)	0.0342 (945)	0.0281 (4216)	0.0388 (2273)
[c]*wR*2 (on F^2, all data)	0.0849 (1805)	0.0807 (5195)	0.0919 (983)	0.0791 (4850)	0.1223 (2422)

[a] $\mathrm{GOF} = \left\{\Sigma[w(F_o^2 - F_c^2)^2]/(n-p)\right\}^{1/2}$ (*n*; number of reflections, *p*; total number of parameters refined), [b] $R1 = \Sigma(|F_o| - |F_c|)/\Sigma|F_o|$.

[c] $wR2 = \left\{\Sigma[w(F_o^2 - F_c^2)^2]/\Sigma[w(F_o^2)^2]\right\}^{1/2}$