Supplementary Material (ESI) for Dalton Transactions

Electronic Supplementary Information for:

Synthetic, electrochemical and solvent extraction studies of neutral trinuclear cobalt(II), nickel(II), copper(II) and zinc(II) metallocycles and tetrahedral tetranuclear iron(III) species incorporating 1,4-aryl-linked bis-*β*-diketonato ligands

Jack K. Clegg,[a] David J. Bray,[a] Kerstin Gloe,[b] Karsten Gloe,[b] Katrina A. Jolliffe,[c] Geoffrey A. Lawrance,[d] Leonard F. Lindoy,[a*] George V. Meehan,[e] and Marco Wenzel[b]

[a] *Centre for Heavy Metals Research, School of Chemistry, University of Sydney, NSW, 2006, Australia*

[b] *Department of Chemistry and Food Chemistry, Technical University Dresden, 01062 Dresden, Germany*

[c] *School of Chemistry, University of Sydney, NSW, 2006, Australia*

[d] *Discipline of Chemistry, School of Environmental and Life Sciences, The University of Newcastle, Callaghan, NSW 2308, Australia*

[e] *School of Pharmacy and Molecular Sciences, James Cook University, Townsville, Qld, 4811, Australia*

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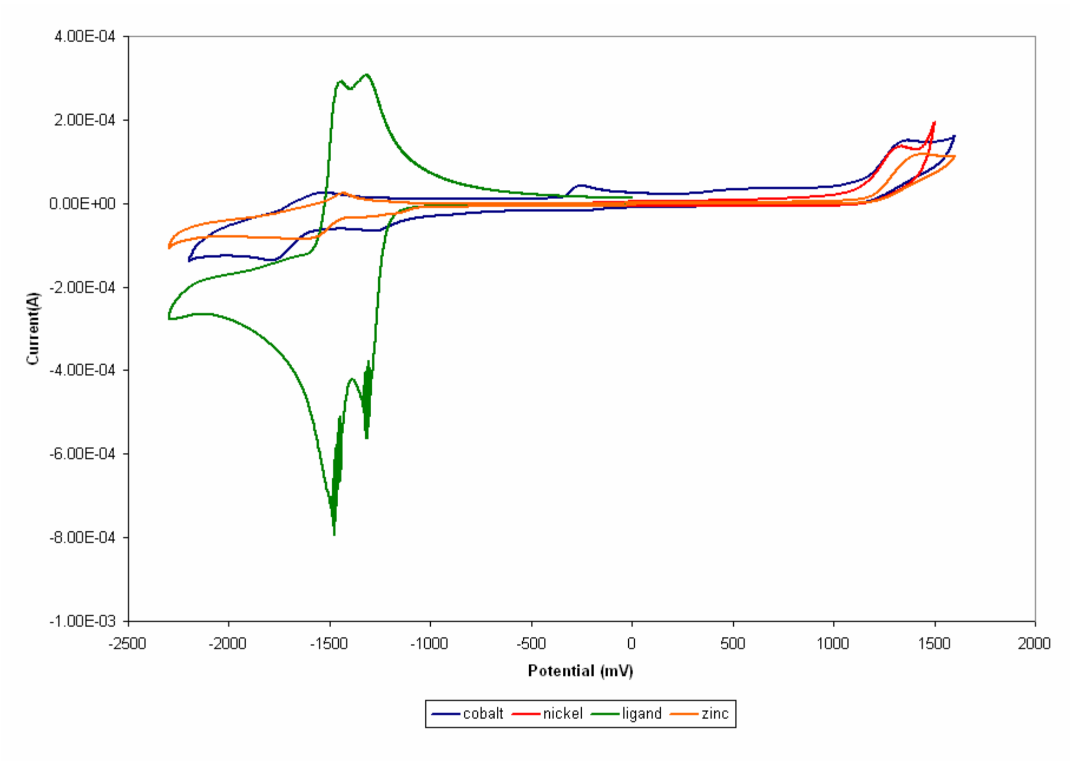

Figure S1. Cyclic voltammograms (reference Ag/AgCl) for H_2L^1 (R = *t*-Bu) for $[Co_3(L^1)_3(Py)_6]$ (R = *t*-Bu), $[Ni_3(L^1)_3(Py)_6]$ (R = *t*-Bu) and $[Zn_3(L^1)_3(Py)_6]$ (R = *t*-Bu).

Representative plots for the extraction experiments are are given in Figs S2 –S4.

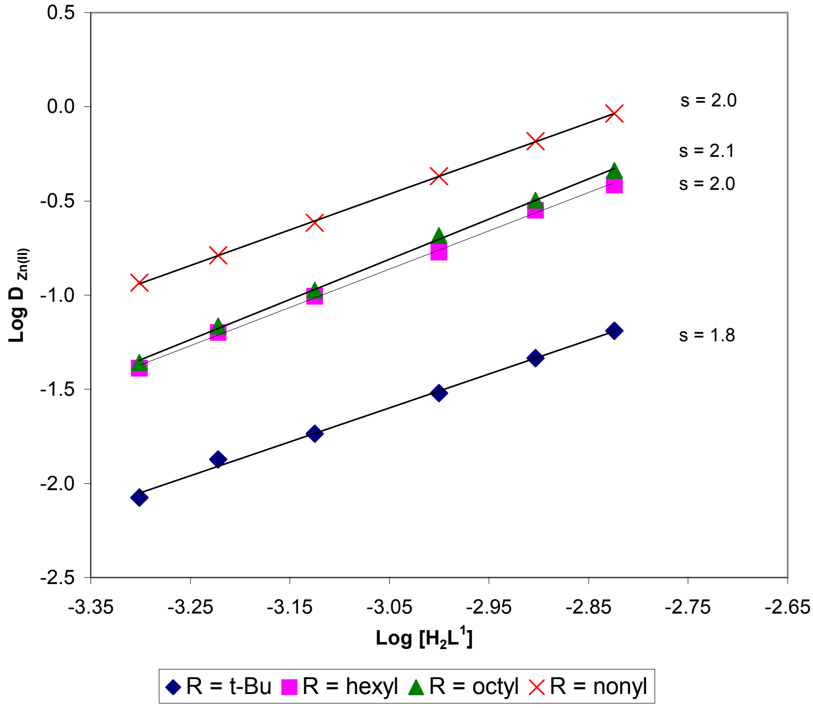

Figure S2. Plots of log $D_{Zn(II)}$ against log $[H_2L^1]$ (R = *t*-Bu, hexyl, octyl and nonyl) in the absence of 4-ethylpyridine showing their corresponding slopes (*s*). $[Zn(ClO_4)_2] = 1 \times 10^{-4}$ M, pH = 8.7 (TAPS/NaOH buffer); $[H_2L^1] = 5 \times 10^{-3}$ to 1.5×10^{-3} M in $CHCl_3$; shaking time 30 min; T = 24 ± 1°C.

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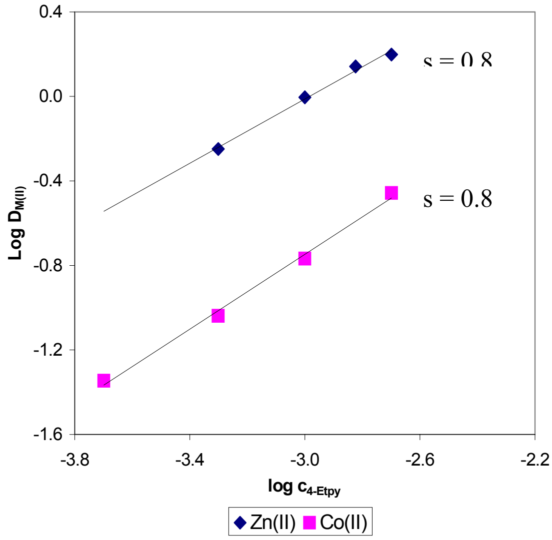

Figure S3. Plots of log $D_{M(II)}$ (M(II) = zinc(II) and cobalt(II)) against log [4-Ethylpyridine] for $[H_2L^1]$ (R = nonyl) showing their corresponding slopes (*s*); $[M(ClO_4)_2]$ = 1×10^{-4} M, pH = 8.7 (TAPS/NaOH buffer); $[H_2L^1] = 1\times 10^{-3}$ M, [4-ethylpyridine] = 2×10^{-4} - 2×10^{-3} M in $CHCl_3$; shaking time 30 min for zinc(II), 3 h for cobalt(II); $T = 24 \pm 1°C$.

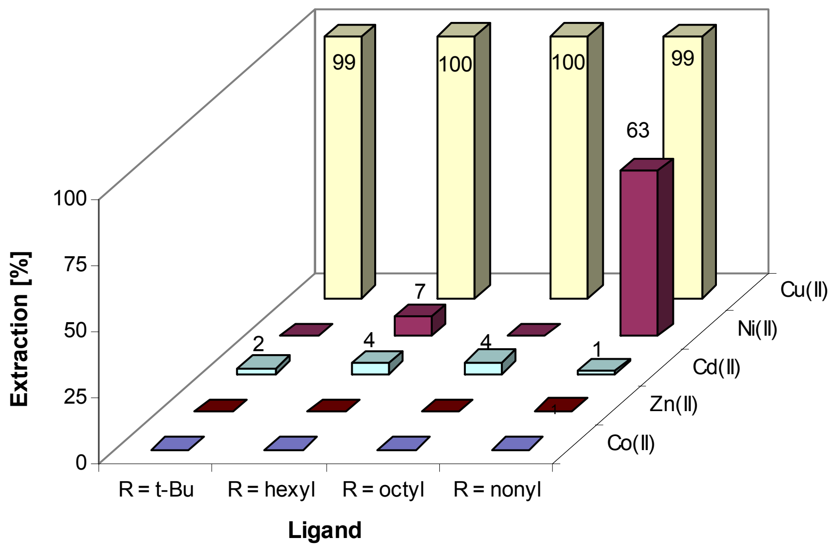

Figure S4**.** Percentage of cobalt(II), zinc(II), cadmium(II), nickel(II) and copper(II) competitive extracted from the aqueous into the organic phase with $H_2\mathbf{L}^1$ (R = *t*-Bu, hexyl, octyl and nonyl) in the presence of 4-ethylpyridine. $[M(ClO_4)_2] = 1 \times 10^{-4}$ M, pH 7.4 (HEPES/NaOH buffer); $[H_2\mathbf{L}^1] = 1 \times 10^{-3}$ M; [4-ethylpyridine] $= 2 \times 10^{-3}$ M in CHCl3; shaking time 24 h; $T = 24 \pm 1$ °C.

Table 1S. Hydrogen bond geometry of H_2L^1 (R = *t*-Bu).

Donor	Hydrogen	Acceptor	D-H(Å)	H-A(Å)	D-A(Å)	DHA (º)
O(1)	H(13)	O(2)	0.913(10)	1.625(13)	2.4790(18)	154(2)

Table 2S. Selected bond lengths (Å) and angles (º) in $[Ni_3(L^2)_3(Py)_6]\cdot 3.5Py$ (R = *t*-Bu).

N(1)	Ni(1)	2.111(7)	N(2)	Ni(1)	2.091(8)
N(3)	Ni(2)	2.119(8)	N(4)	Ni(2)	2.125(8)
N(5)	Ni(3)	2.096(8)	N(6)	Ni(3)	2.105(8)
O(1)	Ni(1)	2.019(6)	O(2)	Ni(1)	2.018(6)
O(3)	Ni(2)	2.015(5)	O(4)	Ni(2)	2.030(6)
O(5)	Ni(2)	2.015(5)	O(6)	Ni(2)	1.997(6)
O(7)	Ni(3)	2.013(6)	O(8)	Ni(3)	2.033(6)
O(9)	Ni(1)	2.025(6)	O(10)	Ni(1)	2.012(6)
O(11)	Ni(3)	2.020(6)	O(12)	Ni(3)	2.043(6)

O(10)	Ni(1)	O(2)	89.9(2)
O(10)	Ni(1)	O(1)	176.9(2)
O(2)	Ni(1)	O(1)	90.4(2)
O(10)	Ni(1)	O(9)	89.6(2)
O(2)	Ni(1)	O(9)	177.8(3)
O(1)	Ni(1)	O(9)	90.3(2)
O(10)	Ni(1)	N(2)	88.1(3)
O(2)	Ni(1)	N(2)	89.5(3)
O(1)	Ni(1)	N(2)	88.8(3)
O(9)	Ni(1)	N(2)	92.6(3)
O(10)	Ni(1)	N(1)	92.6(3)
O(2)	Ni(1)	N(1)	89.5(3)
O(1)	Ni(1)	N(1)	90.5(3)
O(9)	Ni(1)	N(1)	88.4(3)
N(2)	Ni(1)	N(1)	178.8(3)
O(6)	Ni(2)	O(3)	86.8(2)
O(6)	Ni(2)	O(5)	91.3(2)

O(3)	Ni(2)	O(5)	178.1(2)
O(6)	Ni(2)	O(4)	177.0(2)
O(3)	Ni(2)	O(4)	90.6(2)
O(5)	Ni(2)	O(4)	91.3(2)
O(6)	Ni(2)	N(3)	91.4(3)
O(3)	Ni(2)	N(3)	93.0(2)
O(5)	Ni(2)	N(3)	86.7(3)
O(4)	Ni(2)	N(3)	90.4(3)
O(6)	Ni(2)	N(4)	88.7(3)
O(3)	Ni(2)	N(4)	89.8(3)
O(5)	Ni(2)	N(4)	90.5(3)
O(4)	Ni(2)	N(4)	89.7(3)
N(3)	Ni(2)	N(4)	177.2(3)
O(7)	Ni(3)	O(11)	87.9(2)
O(7)	Ni(3)	O(8)	91.6(2)
O(11)	Ni(3)	O(8)	179.4(2)
O(7)	Ni(3)	O(12)	177.8(2)
O(11)	Ni(3)	O(12)	90.2(2)
O(8)	Ni(3)	O(12)	90.4(2)
O(7)	Ni(3)	N(5)	90.8(3)
O(11)	Ni(3)	N(5)	90.4(3)
O(8)	Ni(3)	N(5)	89.8(3)
O(12)	Ni(3)	N(5)	88.3(3)
O(7)	Ni(3)	N(6)	90.0(3)
O(11)	Ni(3)	N(6)	91.7(3)
O(8)	Ni(3)	N(6)	88.2(3)
O(12)	Ni(3)	N(6)	91.0(3)
N(5)	Ni(3)	N(6)	177.9(3)