Electronic supplementary information

Heterometallic Re/Mo and Re/W cubane-type cluster complexes

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Table S1. Crystal data and structure refinement for the compounds **1** – **4**.

Identification code	**1**	**2**	**3**	**4**
Empirical formula	$C_{12}K_6Mo_2N_{12}O_{10}Re_2Se_4$	$C_{14}H_{24}K_7Mo_3N_{12}O_{10}ReSe_4$	$C_{12}K_6N_{12}O_{10}Re_2Se_4W_2$	$C_{13}H_3Cs_6N_{12}O_{6.24}ReSe_4W_3$
Formula weight	1607.08	1584.77	1782.9	2291.66
Temperature/K	150(2)	150(2)	150(2)	150(2)
Crystal system	trigonal	orthorhombic	trigonal	monoclinic
Space group	*P*3	*Pnma*	*P*3	$P2_1/n$
a/Å	33.127(3)	11.8651(2)	33.1303(10)	18.6439(3)
b/Å	33.127(3)	21.2079(4)	33.1303(10)	10.4129(2)
c/Å	9.3387(7)	15.7889(3)	9.3359(5)	22.0131(4)
β/°	90	90	90	108.4950(10)
Volume/Å^3	8875.5(17)	3973.02(13)	8874.4(7)	4052.83(13)
Z	9	4	9	4
ρ_{calc}g/cm^3	2.674	2.619	3.002	3.733
μ/mm^{-1}	11.151	8.446	16.313	20.407
F(000)	6484.0	2897.0	7057.0	3911.0
Crystal size/mm^3	0.25 × 0.08 × 0.05	0.1 × 0.08 × 0.05	0.15 × 0.04 × 0.04	0.3 × 0.08 × 0.08
Radiation	MoKα (λ = 0.71073)	MoKα (λ = 0.71073)	MoKα (λ = 0.71073)	MoKα (λ = 0.71073)
2Θ range for data collection/°	2.84 to 48.812	4.704 to 61.024	2.458 to 51.38	3.458 to 57.392
Index ranges	-38 ≤ h ≤ 32, -35 ≤ k ≤ 25, -10 ≤ l ≤ 8	-16 ≤ h ≤ 14, -30 ≤ k ≤ 30, -20 ≤ l ≤ 22	-38 ≤ h ≤ 40, -40 ≤ k ≤ 40, -11 ≤ l ≤ 11	-21 ≤ h ≤ 25, -14 ≤ k ≤ 14, -29 ≤ l ≤ 29
Reflections collected	34885	54820	159689	64300
Independent reflections	15373 [R_{int} = 0.0641, R_{sigma} = 0.0939]	6215 [R_{int} = 0.0549, R_{sigma} = 0.0291]	22473 [R_{int} = 0.0705, R_{sigma} = 0.0410]	10457 [R_{int} = 0.0568, R_{sigma} = 0.0397]
Data/restraints/parameters	15373/546/1162	6215/3/254	22473/154/1289	10457/26/456
Goodness-of-fit on F^2	1.022	1.181	1.106	1.148
Final R indexes [I>=2σ (I)]	R_1 = 0.0518, wR_2 = 0.0957	R_1 = 0.0420, wR_2 = 0.0863	R_1 = 0.0329, wR_2 = 0.0649	R_1 = 0.0314, wR_2 = 0.0752
Final R indexes [all data]	R_1 = 0.0732, wR_2 = 0.1051	R_1 = 0.0478, wR_2 = 0.0883	R_1 = 0.0359, wR_2 = 0.0661	R_1 = 0.0326, wR_2 = 0.0758
Largest diff. peak/hole / e Å^{-3}	2.35/-1.14	1.52/-1.33	2.60/-1.73	1.95/-2.83
Flack parameter	0.55(2)		0.397(10)	

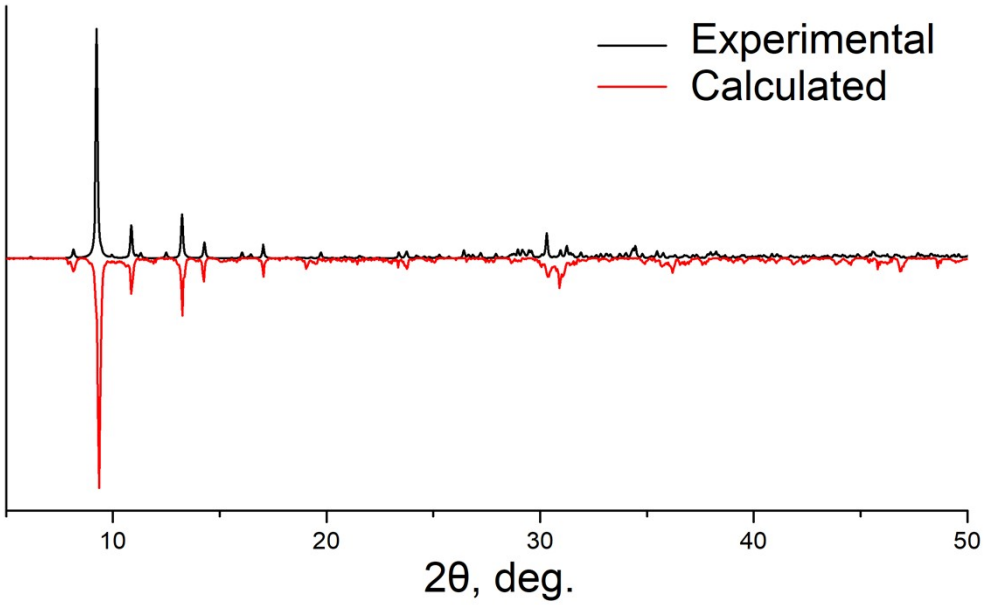

Figure S1. Experimental (top) *vs* calculated (bottom) powder diffraction pattern for compound **1**.

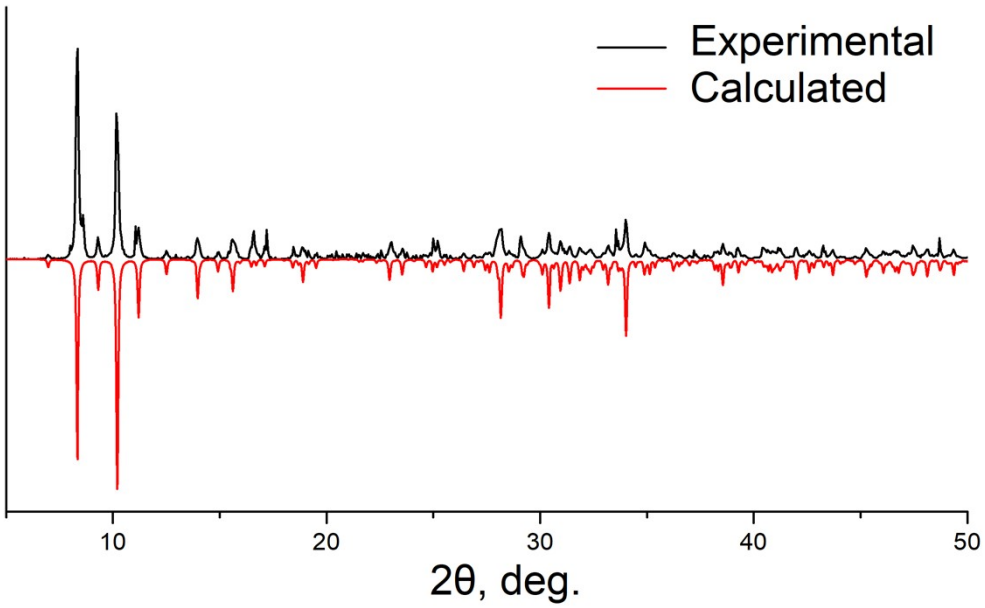

Figure S2. Experimental (top) *vs* calculated (bottom) powder diffraction pattern for compound **2**.

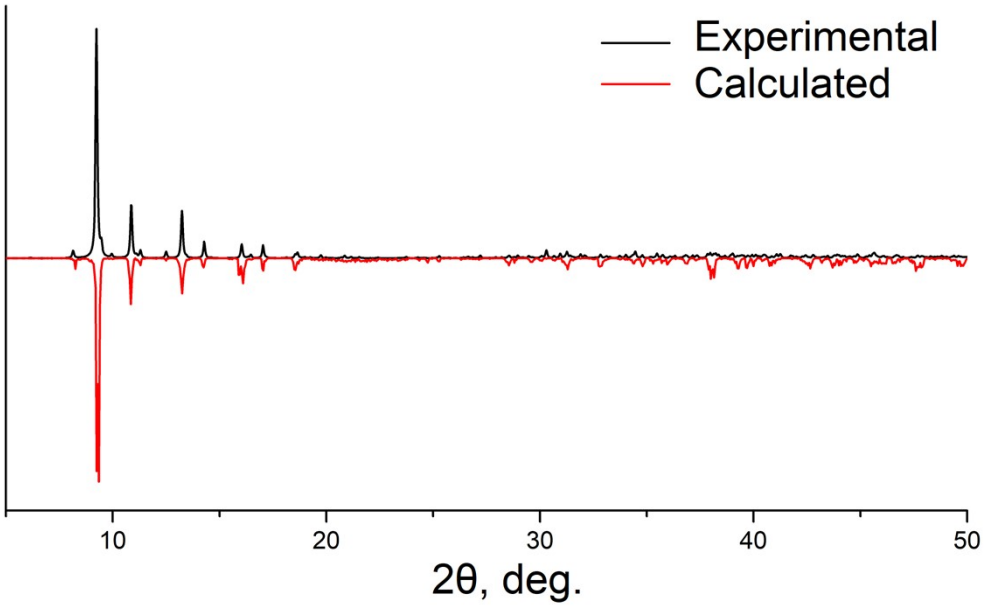

Figure S3. Experimental (top) *vs* calculated (bottom) powder diffraction pattern for compound **3**.

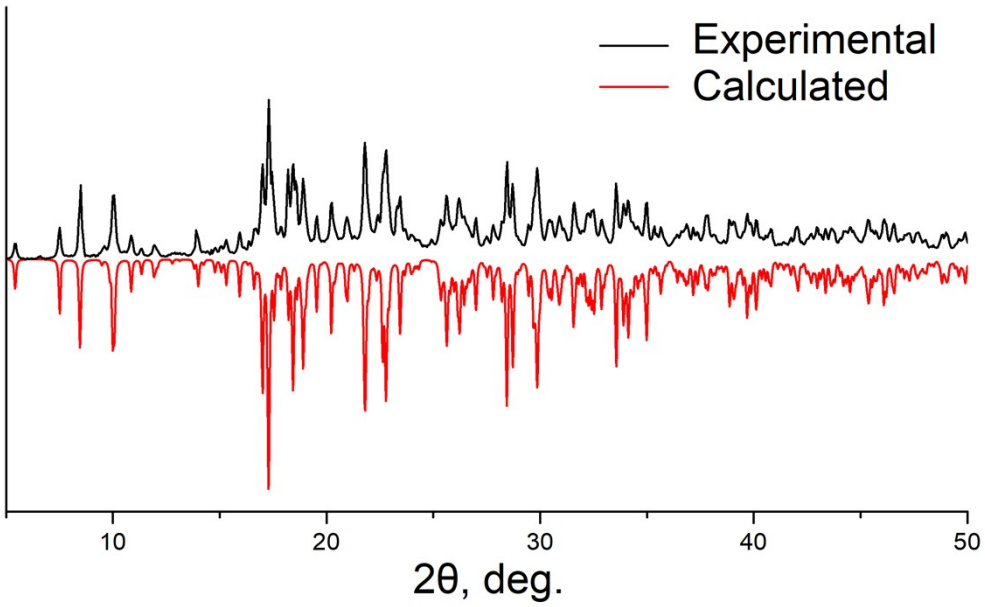

Figure S4. Experimental (top) *vs* calculated (bottom) powder diffraction pattern for compound **4**.

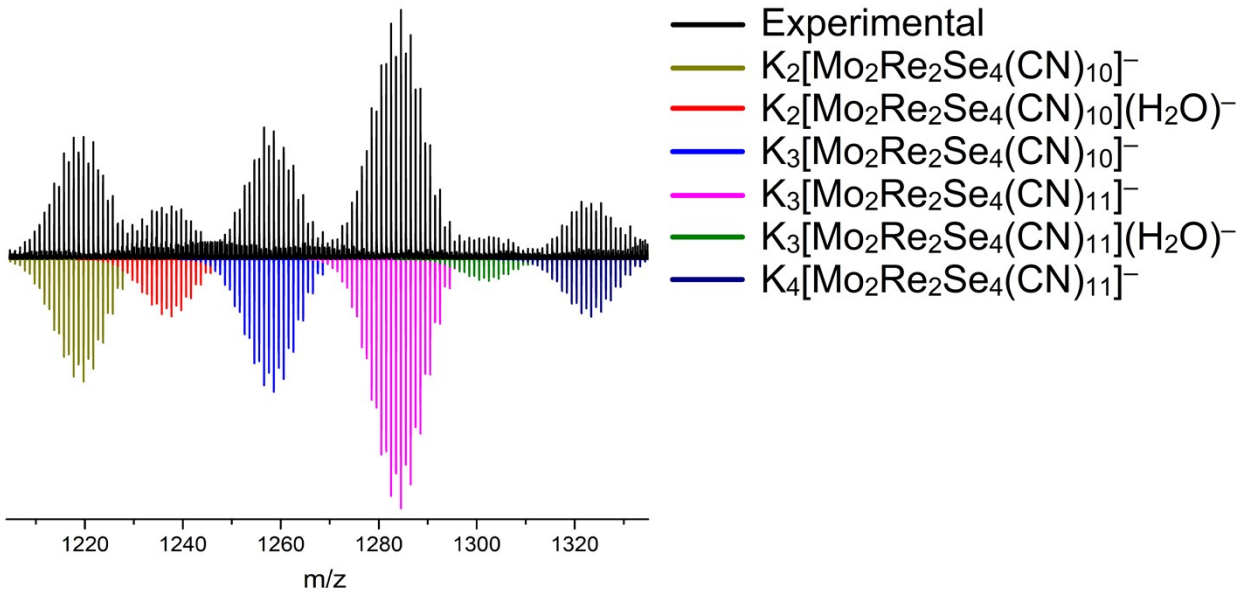

Figure S5. Observed (top) *vs* calculated (bottom) isotopic distribution peak sets correspond to the cluster anionic adducts for compound **1**.

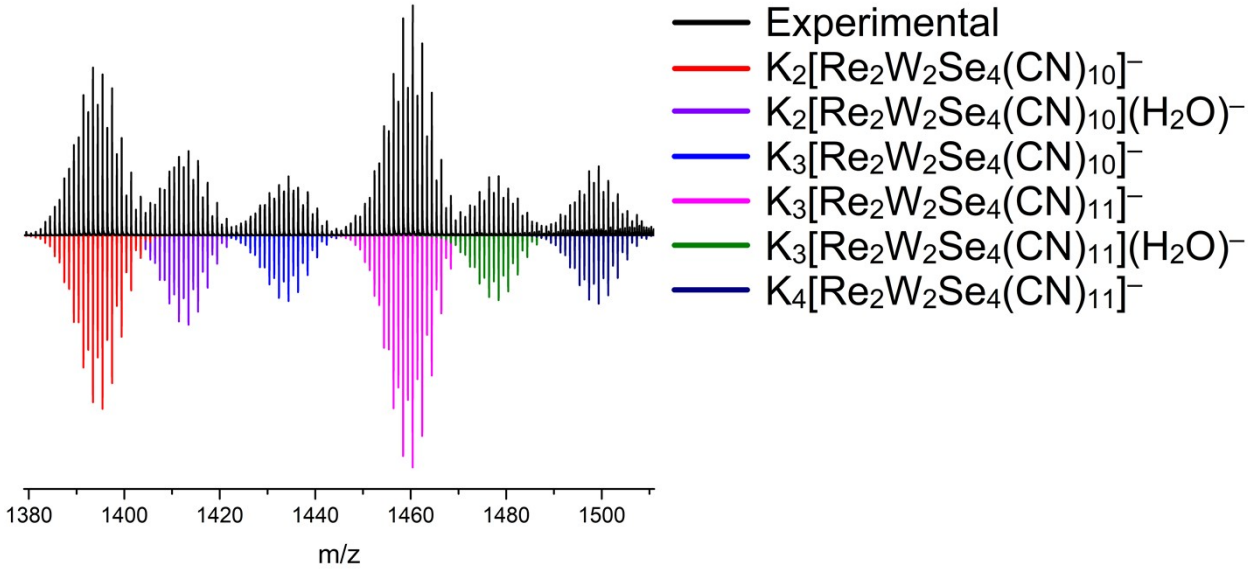

Figure S6. Observed (top) *vs* calculated (bottom) isotopic distribution peak sets correspond to the cluster anionic adducts for compound **3**.

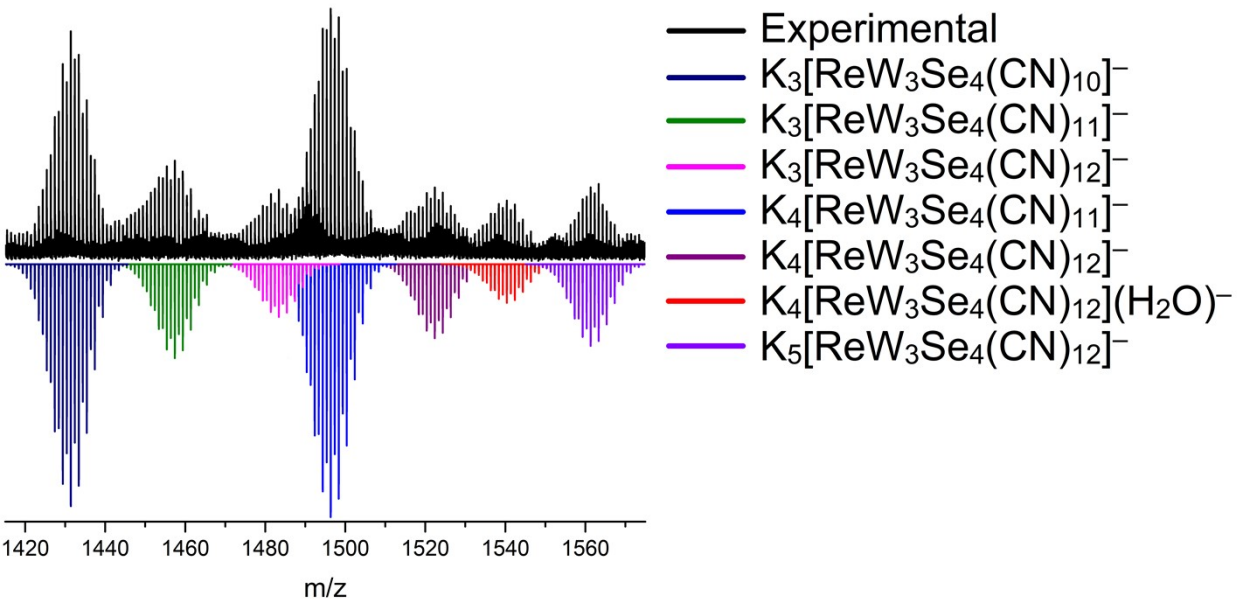

Figure S7. Observed (top) *vs* calculated (bottom) isotopic distribution peak sets correspond to the cluster anionic adducts for compound **4**.

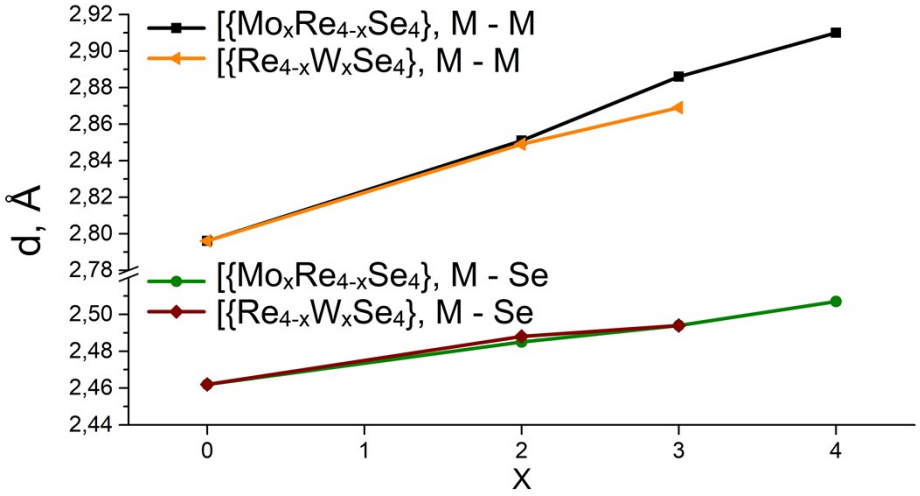

Figure S8. Dependence of M–M and M–Se mean bond lengths for $[\{M_xRe_{4-x}Se_4\}(CN)_{12}]^{n-}$ clusters.