SUPPLEMENTARY INFORMATION

Organic-soluble lacunary $\{M_2(P_2W_{15})_2\}$ polyoxometalate sandwiches with previously unseen $\alpha\beta\beta\alpha$ isomers

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Table of Contents 1
1. General Experimental Section 2
2. Synthesis and Experimental Section 4
3. Thermogravimetric Analysis (TGA) 7
4. Infrared Spectroscopy 10
5. UV/Visible Spectroscopy 12
6. Cyclic Voltammetry 14
7. X-Ray Crystallography 19
    7.1. Crystallographic references 25

1. General Experimental Section

All chemicals and solvents were of analytical grade purchased from Sigma Aldrich and used as supplied, without further purification. $Na_{12}[\alpha\text{-}P_2W_{15}O_{56}]\cdot 24H_2O$ was synthesized by a method reported by a previously reported method found in *Inorganic Syntheses, Vol. 27*, John Wiley & Sons, 1990.

Single crystal X-Ray diffraction: Single crystal datasets were collected at 150(2) K on an Oxford Diffraction Gemini Ultra S using Mo radiation source ($\lambda_{Mo\text{-}K\alpha}$ = 0.71073 Å) or Cu radiation source ($\lambda_{Cu\text{-}K\alpha}$ = 1.54184 Å).

Flame Atomic Absorption Spectroscopy Analysis: FAAS analysis was performed at the Environmental Chemistry Section, School of Chemistry, The University of Glasgow on a Perkin-Elmer 1100B Atomic Absorption Spectrophotometer.

Thermogravimetric Analysis: TGA was performed on a TA Q500 instrument under a nitrogen atmosphere. The initial heating range was from RT to 150 ° C at 2.00 ° C per min followed by a second range from 150 ° to 1000 ° C at 5.00 ° C per min.

Fourier-transform infrared (FT-IR) spectroscopy: The materials were prepared as KBr pellets and FT-IR spectra were collected in transmission mode using a JASCO FT-IR-410 spectrometer. Wavenumbers (ν) are given in cm^{-1}; intensities as denoted as w = weak, m = medium, s = strong, br = broad, sh = sharp.

UV-Vis spectroscopy: UV-Vis spectra were collected using a JASCO V-670 spectrometer equipped with an ISV723 60mm integrating sphere in diffuse reflectance mode.

Microanalysis: Carbon, hydrogen, and nitrogen content was determined by the microanalysis services within the School of Chemistry, University of Glasgow using a EA 1110 CHNS, CE-440 Elemental Analyser.

Cyclic Voltammetry: Voltammograms were obtained using a VersaStat 4 model 200 electro analysis system by Princeton Applied Research. The standard three-electrode arrangement was employed with a Pt mesh auxiliary electrode, 1.5 mm glassy carbon working electrode, and Ag/Ag+ reference electrode at room temperature (19 ± 1 oC). All potentials are quoted relative to the Ag/Ag+ reference electrode. The glassy carbon working electrodes (diameter 1.5 mm) were polished with alumina (3 µm) on polishing pads and then rinsed with distilled water and sonicated in acetone solution before each experiment. Distilled acetonitrile was used to prepare all aqueous solutions. The cell was purged with Ar for at least 10 min before each experiment. The required amount of solid $TBABF_4$ was then added (thereby effecting a

0.1 M solution) along with the desired amount (usually 10-4 M unless otherwise stated) of the compound under investigation. UV-visible spectroscopy showed that the compounds remained stable for a sufficiently long time to allow their electrochemical characterization.

2. Synthesis and Experimental Section

General synthetic procedure for compounds **1-5**:

Into 50 mL of deionised water was dissolved 1.5 mmols of the relevant transition metal sulfate (**1** = $MnSO_4{\cdot}H_2O$, 0.25 g. **2** = $CoSO_4{\cdot}7H_2O$, 0.42 g. **3** = $NiSO_4{\cdot}6H_2O$, 0.39 g. **4** = $FeSO_4{\cdot}7H_2O$, 0.417 g, **5** = $CuSO_4{\cdot}5H_2O$, 0.375 g) followed by $Na_{12}[P_2W_{15}O_{56}]{\cdot}24H_2O$ (1.0 g, 0.23 mmol) and the solution was stirred for 10 minutes. 75 mL of acetonitrile was then added to this reaction mixture giving a cloudy solution which was stirred for a further 5 minutes followed by the addition of tetrabutylammonium bromide (6.0g, 18.612 mmol). The solution was then stirred for another 10 minutes before lowering the pH to 1.5 using aqueous 4.5 M sulfuric acid. The resulting turbid solution was stirred for a further 5 minutes and then set aside for 2-3 hours. During this time an oily material was formed which separated down the beaker, and was collected by centrifugation. The aqueous solution was poured away and the oil was washed three times with 20 mL aliquots of water and once with a 20 mL aliquot of ethanol to dry (take care, the ethanol slightly dissolves the product) eventually resulting in a fine powder which was dried overnight in a desiccator.

Yield for **1** = 0.58 g, 58 µmol = 50.4 % by cluster
Yield for **2** = 0.61 g = 63.4 µmol = 55 % by cluster
Yield for **3** = 0.97 g = 0.1 mmol 87 % by cluster
Yield for **4** = 0.92 g, 95.9 µmol = 83 % by cluster
Yield for **5** = 0.78 g = 77.4 µmol = 67.3% by cluster

Crystals were obtained from this powder by dissolving 0.2 g in 20 mL of acetonitrile and allowing diethyl-ether to diffuse slowly into the solution.

Yield for **1**= 0.171 g = 17.2 µmol = 85.5 %
Yield for **2**= 0.1429 g = 14.8 µmol = 71.45 %
Yield for **3**= only a few crystals were obtained
Yield for **4**= 0.109 g = 11.4 µmol = 54.5 %
Yield for **5**= 0.16 g = 15.9 µmol = 80 %

Elemental analysis for the partially dried crystalline material of **1**, $(TBA)_9Na_1[H_{10}Mn_2(P_2W_{15}O_{56})_2]\cdot6CH_3CN$, $C_{156}H_{352}Mn_2N_{15}NaO_{110}P_4W_{30}$ (**1**), MW = 9970.46 g mol^{-1} (partial solvent lost). Calculated values (found values in brackets): Mn 1.10 (0.97), W 55.32 (55.7), Na 0.23 (0.44), C 18.79 (17.32), H 3.56 (3.35), N 2.10 (1.53). TGA weight loss to 1000 ^{o}C, calculated (found) %: 24.45 (24.31). Characteristic FT-IR-bands: 355 (s, sh), 524 (m, sh), 740 (s, br), 915 (s, sh), 1089 (s, sh), 1377 (w, sh), 1482 (m, sh), 1624 (m), 2868 (m, sh), 2954 (m, sh). UV-bands solid state, nm (Abs): 445(0.87), 1754(0.41).

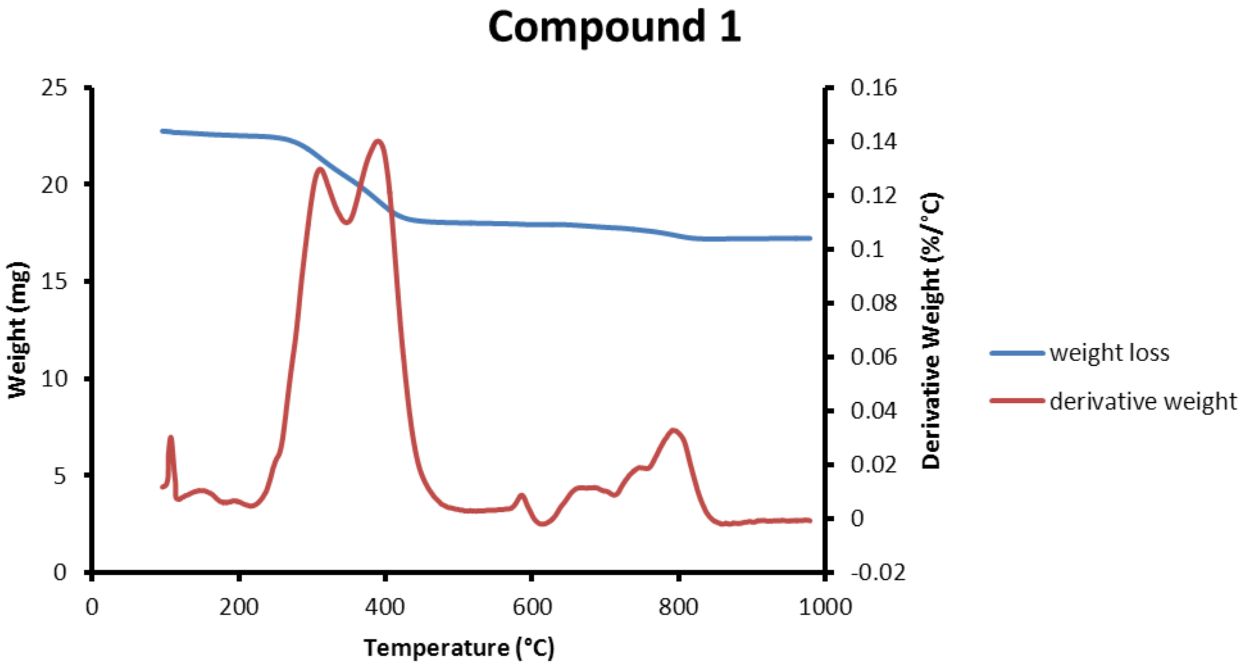
Elemental analysis for the partially dried crystalline material of **2**, $(TBA)_8[H_{12}Co_2(P_2W_{15}O_{56})_2]\cdot3CH_3CN$, $C_{134}H_{309}Co_2N_{11}O_{112}P_4W_{30}$ (**2**), MW = 9623.86 g mol^{-1} (partial solvent lost). Calculated values (found values in brackets): Co 1.22 (1.01), W 57.30 (59.7), C 16.72 (16.48), H 3.24 (3.22) N 1.60 (1.49). TGA water loss to 1000 ^{o}C, calculated (found) %: 21.56 (23.92). Characteristic FT-IR-bands: 349.1 (s, sh), 750 (s, b), 941 (s, b), 1087 (s, sh), 1374 (m, sh), 1480 (m, sh), 1650 (m, b), 2952 (m). UV-bands solid state, nm (abs): 234(0.72), 447(0.93), 1327(0.46), 1750(0.36).

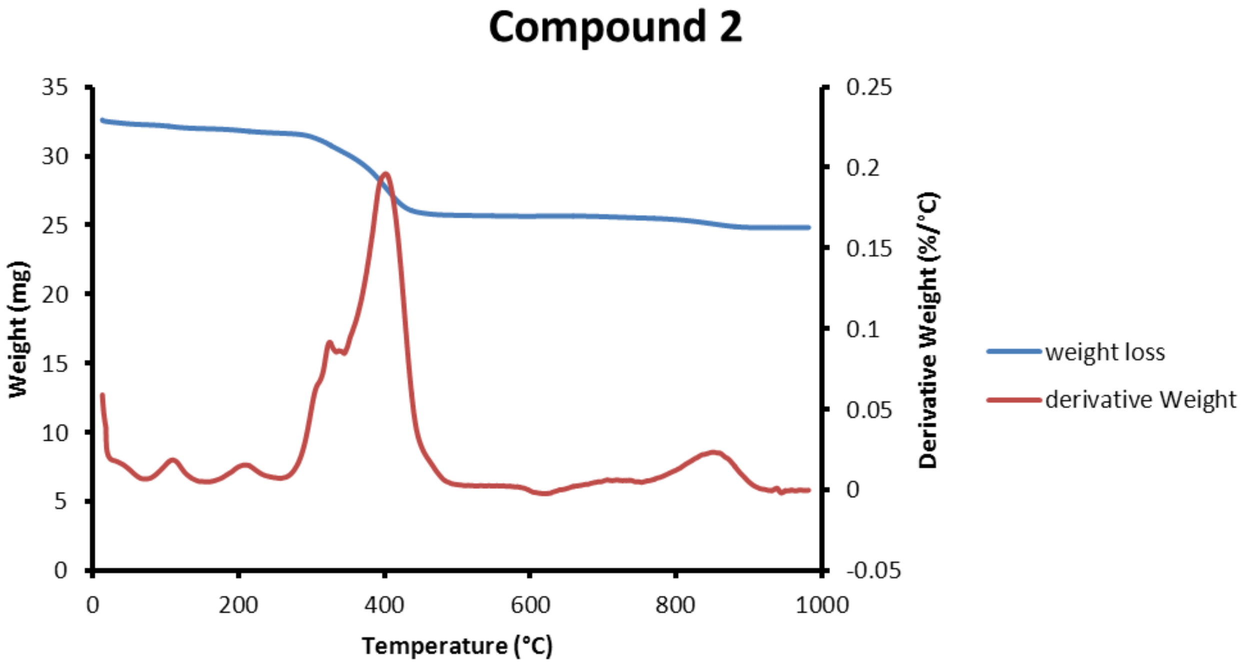
Elemental analysis for the partially dried crystalline material of **3**, $(TBA)_9[H_{11}Ni_2(P_2W_{15}O_{56})_2]\cdot CH_3CN$, $C_{137}H_{338}N_{10}Ni_2O_{112}P_4W_{30}$ (**3**), MW = 9674.62 g mol^{-1} (partial solvent lost). Calculated values (found values in brackets): Ni 1.21 (0.58), W 57.01 (60.45), C 17.01 (18.05), H 3.50 (3.47), N 1.45 (1.68). TGA water loss to 1000 ^{o}C, calculated (found) %: 21.96 (26.34). Characteristic FT-IR-bands: 351 (s, sh), 513 (w, sh), 760 (m, br), 906 (m,sh), 949 (m, sh), 1083 (m, sh), 1372 (w, sh), 1458 (m, sh), 1609 (w, br), 2862 (m, sh), 2951 (m, sh). UV-bands solid state, nm: 377(0.88), 1224(0.34), 1411(0.32), 1747(0.34).

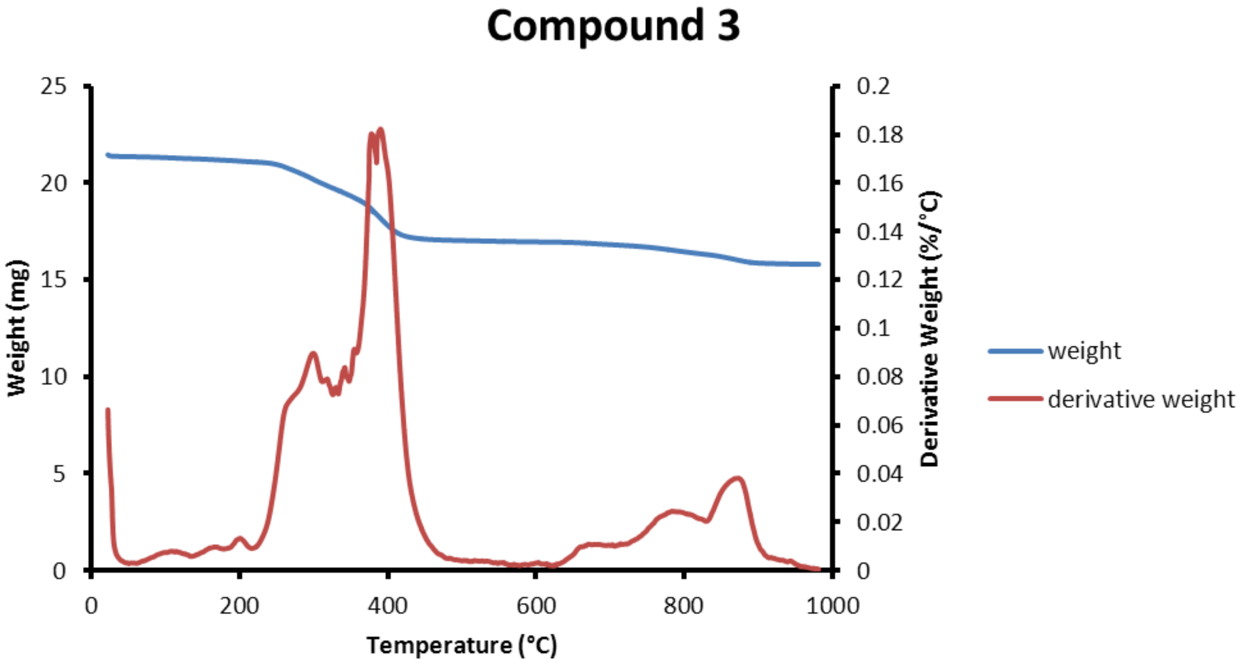
Elemental analysis for the partially dried crystalline material of **4**, $(TBA)_8Na_1$ $[H_9Fe_2(P_2W_{15}O_{56})_2]\cdot2CH_3CN$, $C_{132}H_{303}Fe_2N_{10}NaO_{112}P_4W_{30}$ (**4**), MW = 9596.59 g mol^{-1} (partial solvent lost). Calculated values (found values in brackets): Fe 1.16 (1.65), W 57.47 (60.8), Na 0.24 (0.3) C 16.52 (16.68), H 3.18 (3.22), N 1.45 (1.42). TGA weight loss to 1000 ^{o}C, calculated (found) %: 21.14 (26.67). Characteristic FT-IR-bands: 350 (s, sh), 522 (m, sh), 618 (m, sh), 789 (s, br) 956 (s, sh), 1088 (s, sh), 1375 (w, sh), 1478 (m, sh), 1619 (w, br), 2867 (m, sh), 2955 (m, sh). UV-bands solid state, nm(abs): 241(0.74), 433(0.92), 1222(0.28), 1429(0.30), 1742(0.35).

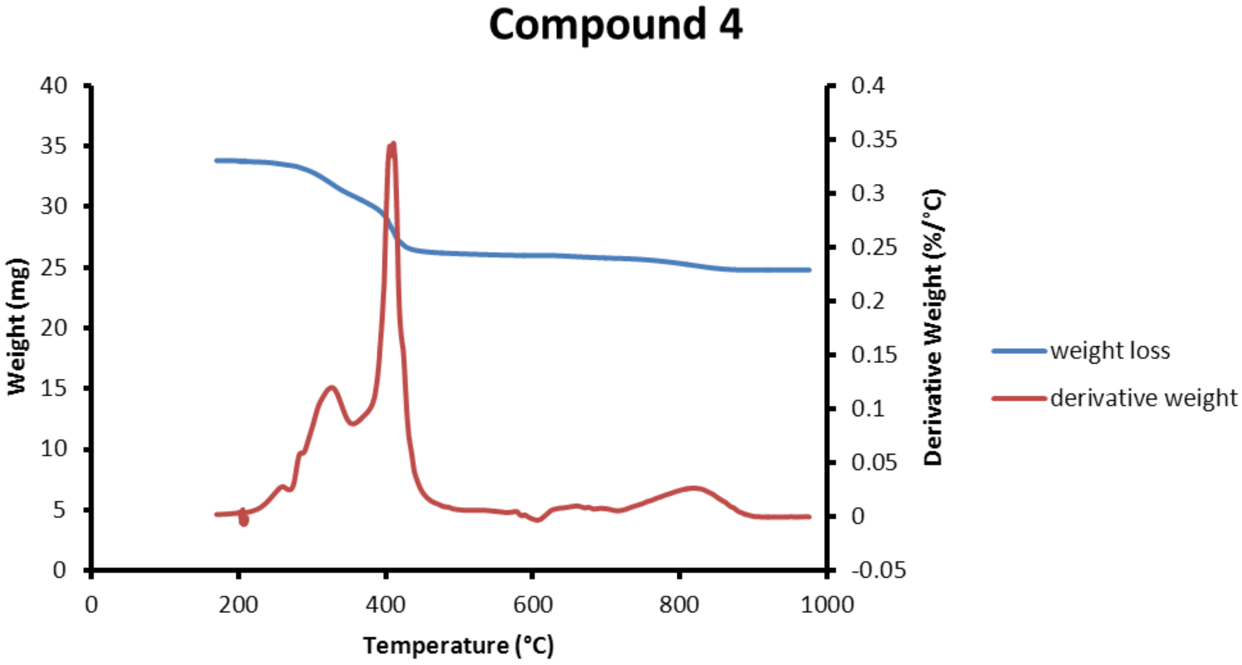
Elemental analysis for the partially dried crystalline material of **5**, $(TBA)_9Na_1[H_6Cu_4(P_2W_{15}O_{56})_2]\cdot 3CH_3CN\cdot 3H_2O$, $C_{150}H_{345}Cu_4N_{12}NaO_{115}P_4W_{30}$ (**5**), MW = 10073.62 g mol^{-1} (partial solvent lost). Calculated values (found values in brackets): Cu 2.52 (1.66), W 54.74 (60.9), Na 0.23 (0), C 17.88 (17.71), H 3.45 (3.42), N 1.67 (1.88). TGA water loss to 1000 ^{o}C, calculated (found) %: 23.00 (25.66). Characteristic FT-IR-bands: 357 (s, sh), 524 (s, sh), 576 (s, sh), 747 (s, br), 1084 (s, sh), 1376 (m, sh), 1481 (m, sh), 1656 (m, sh), 2955 (s, sh), 3437 (m, br) UV-bands solid state, nm: 359(0.82), 796(0.28), 1737(0.15).

3. Thermogravimetric Analysis (TGA)






Compound 3
Weight (mg)
25
20
15
10
5
0
0
200
400
600
800
1000
Temperature (°C)
Derivative Weight (%/°C)
0.2
0.18
0.16
0.14
0.12
0.1
0.08
0.06
0.04
0.02
0
weight
derivative weight


Compound 4
Weight (mg)
40
35
30
25
20
15
10
5
0
0
200
400
600
800
1000
Temperature (°C)
Derivative Weight (%/°C)
0.4
0.35
0.3
0.25
0.2
0.15
0.1
0.05
0
-0.05
weight loss
derivative weight

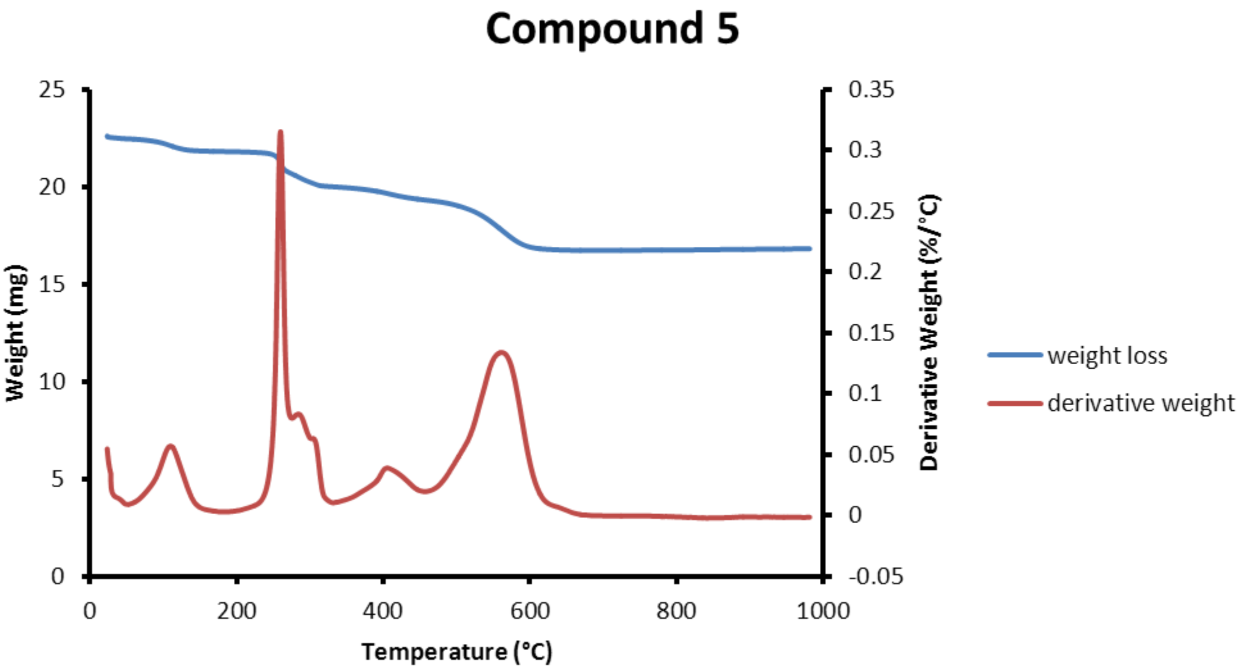
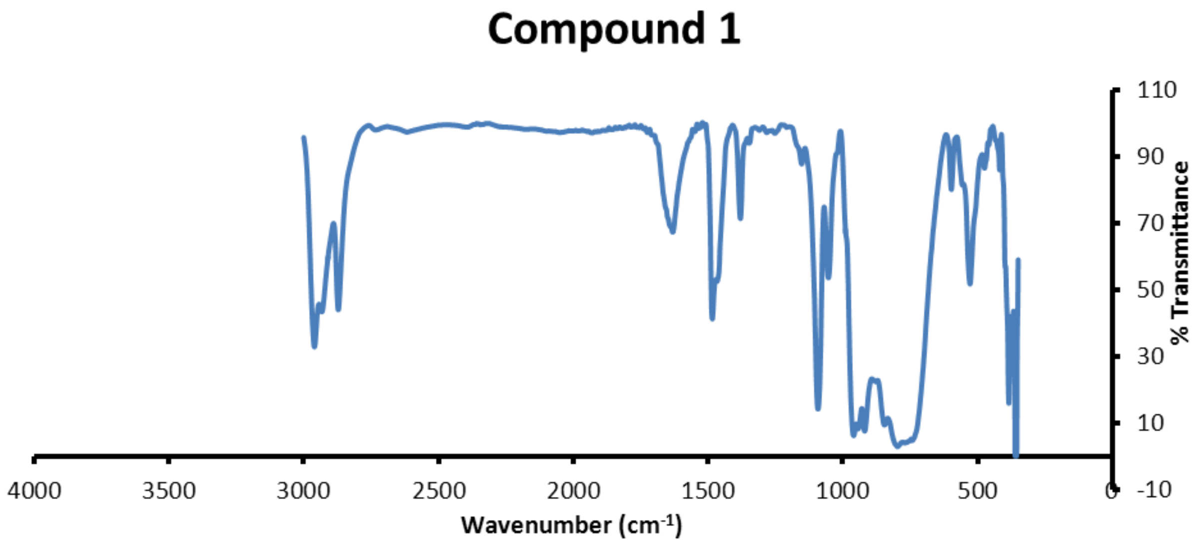
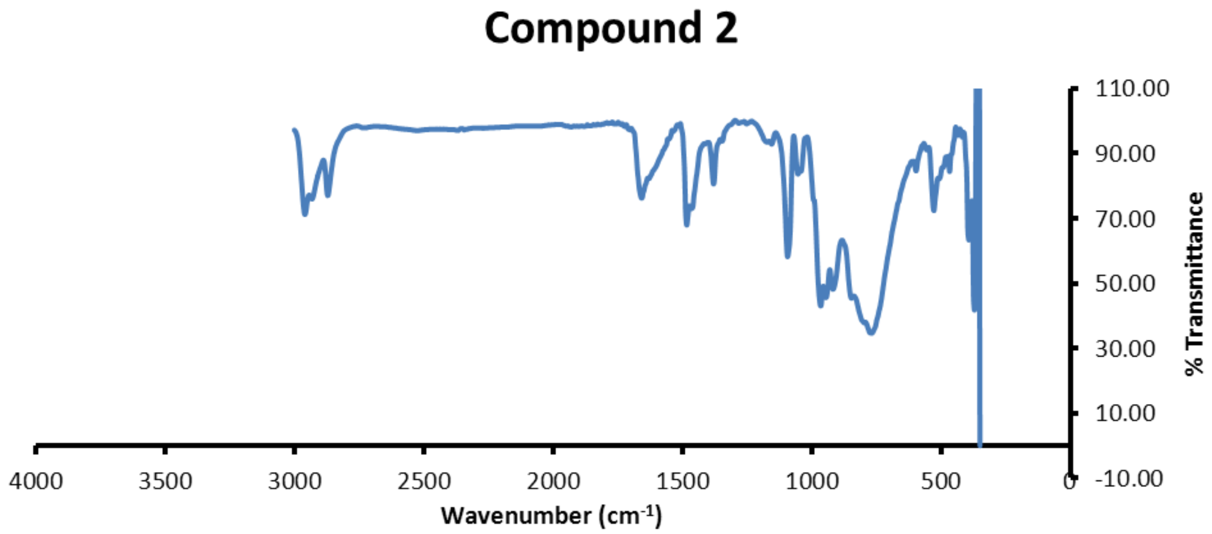
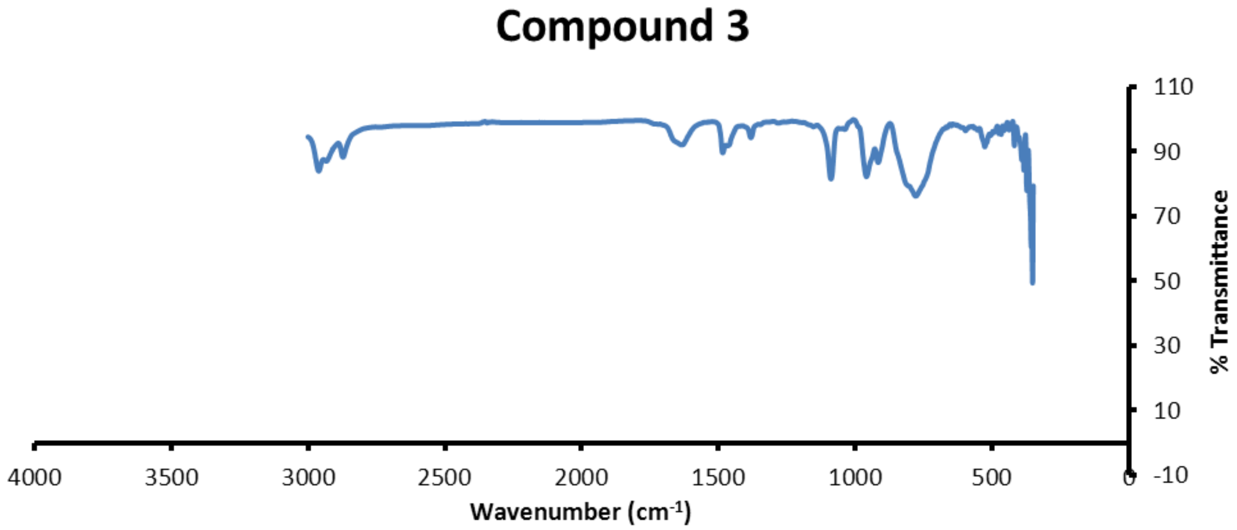


Figure S1a-e: Thermogravimetric analysis of compounds **1-5** (performed under nitrogen) showing the loss of solvent (RT - 250 °C) followed by loss of TBA cations at *ca.* 300 °C.

4. IR spectroscopy







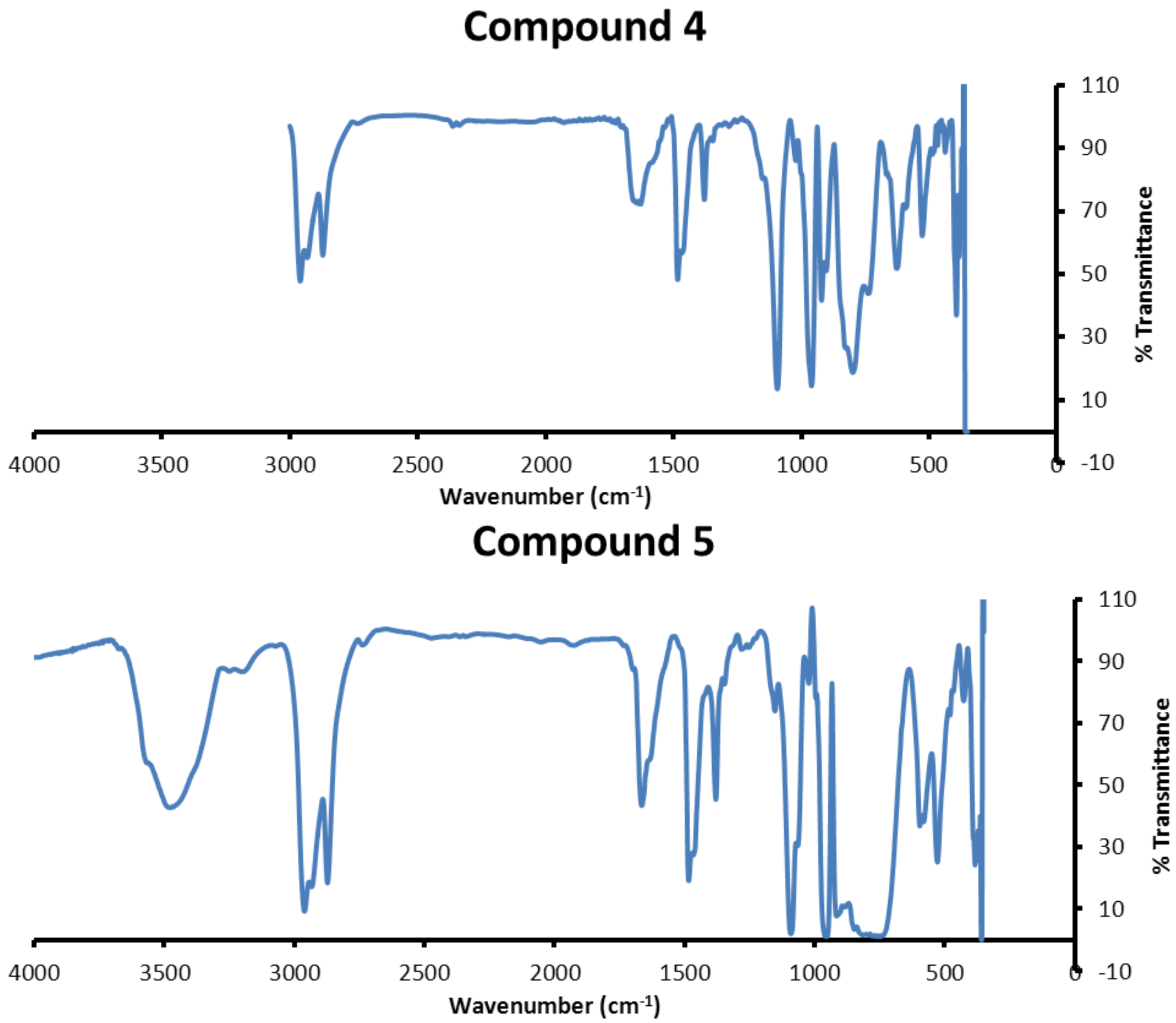
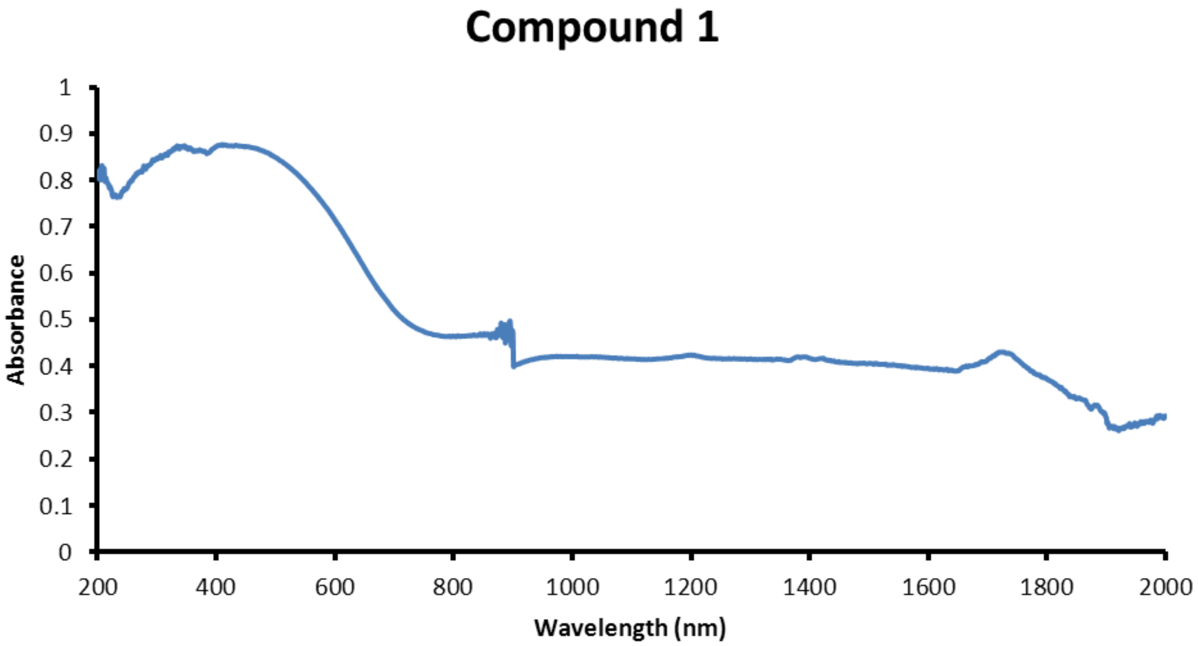


Figure S2a-e: IR scans of compounds **1-5**

5. UV/Visible Spectroscopy



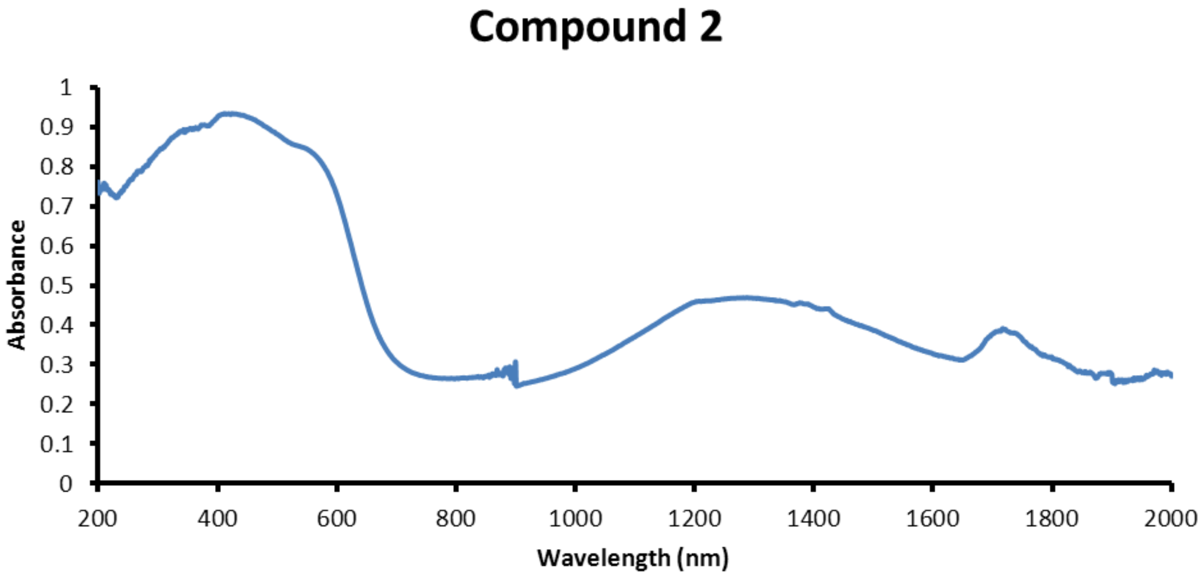
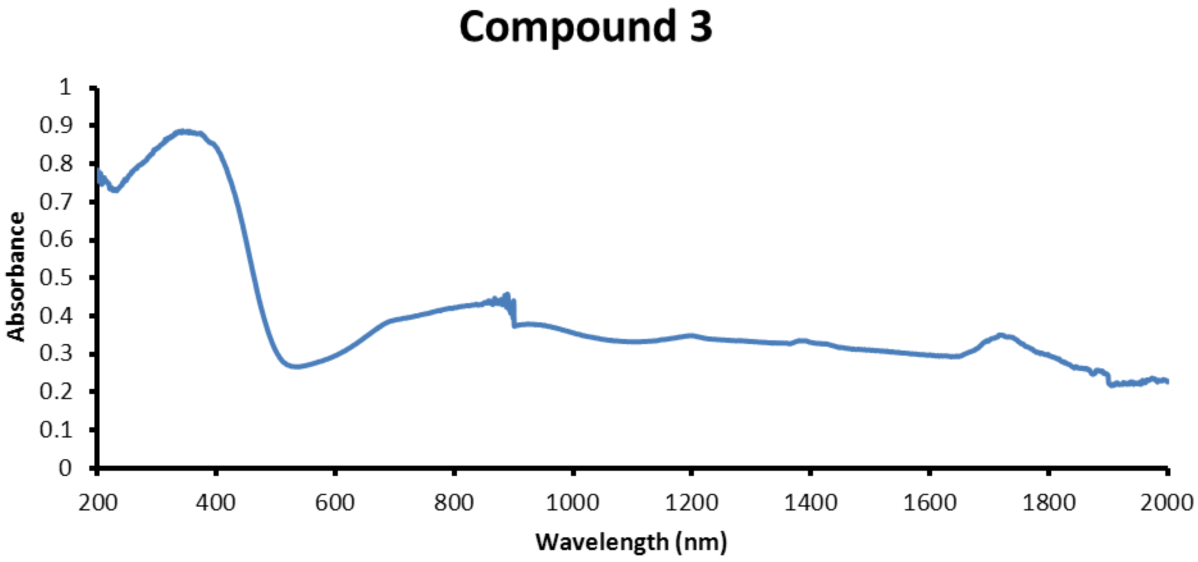
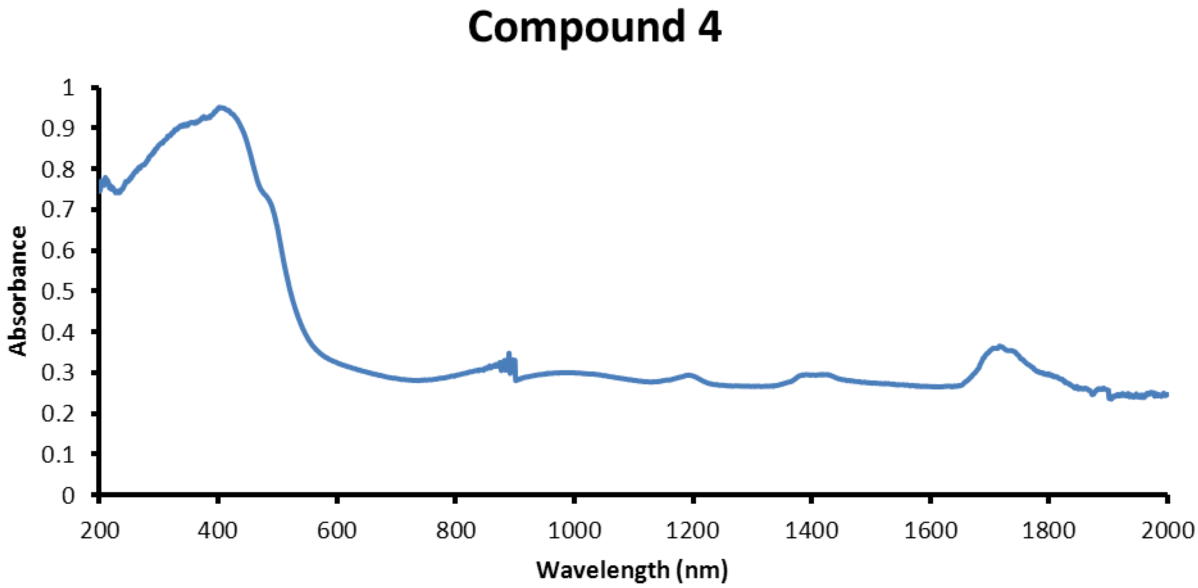
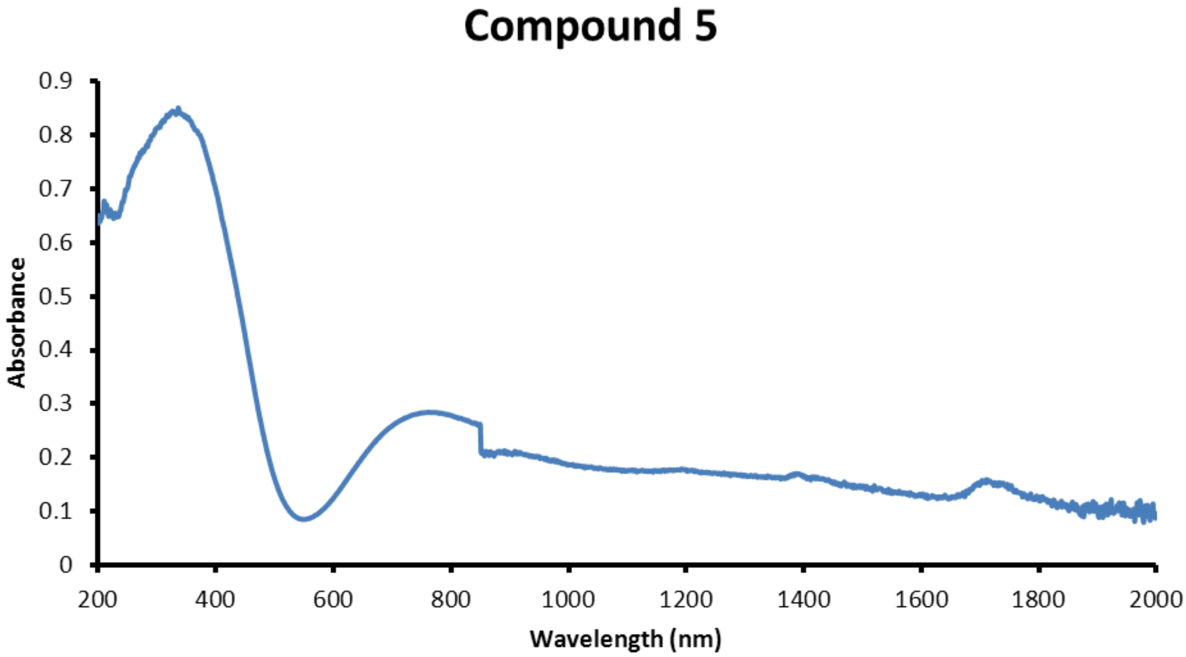


Figure S3a-e: UV/Vis scans of compounds **1-5**


Compound 3
Absorbance
1
0.9
0.8
0.7
0.6
0.5
0.4
0.3
0.2
0.1
0
200
400
600
800
1000
1200
1400
1600
1800
2000
Wavelength (nm)


Compound 4
Absorbance
1
0.9
0.8
0.7
0.6
0.5
0.4
0.3
0.2
0.1
0
200
400
600
800
1000
1200
1400
1600
1800
2000
Wavelength (nm)


Compound 5
Absorbance
0.9
0.8
0.7
0.6
0.5
0.4
0.3
0.2
0.1
0
200
400
600
800
1000
1200
1400
1600
1800
2000
Wavelength (nm)

6. Cyclic Voltammetry

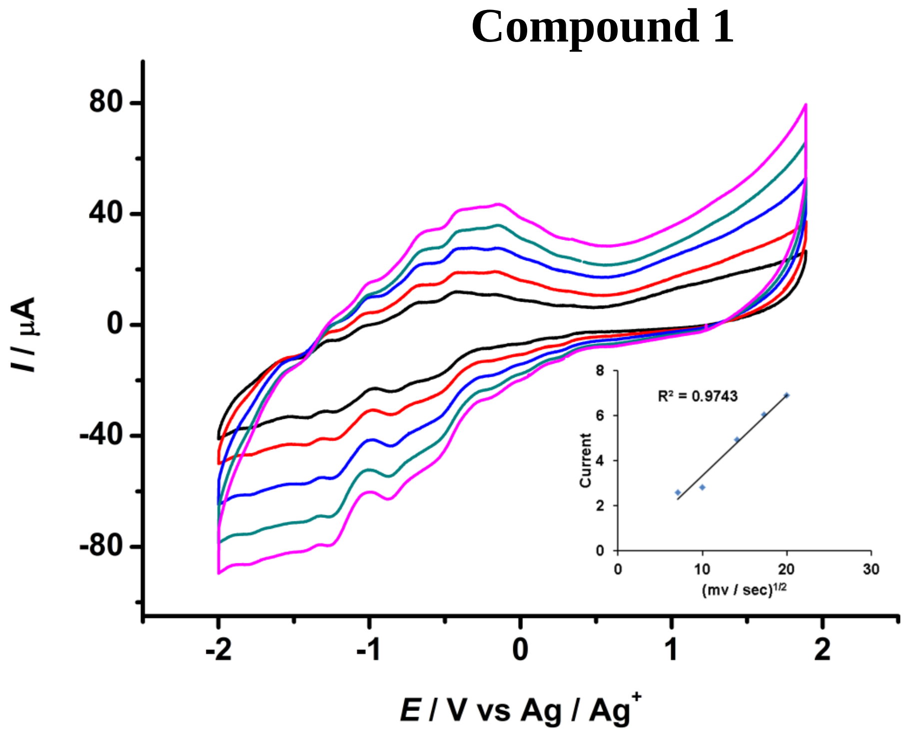


Figure S4: Cyclic voltammograms of compound **1** at scan rates (from inner to outer) 50, 100, 200, 300 and 400 mV/sec. The inset on each graph shows the dependence of the current's intensity as a function of the square root of the scanning rate.

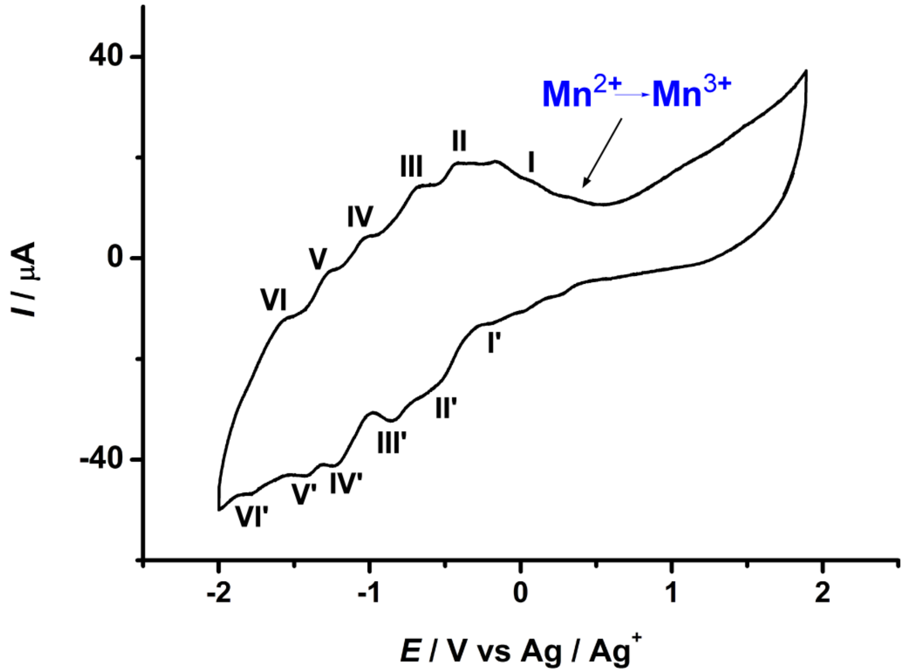


Figure S5: Cyclic voltammogram of **1**, $[Mn_2(P_2W_{15}O_{56})_2]^{20-}$ (10^{-4} M), in 10 mL of 0.1 M $TBAPF_6$ solution. The scan rate was 100 mVs^{-1}, the working electrode was glassy carbon (1.5 mm) and the reference electrode was Ag/Ag^+.

Compound 2

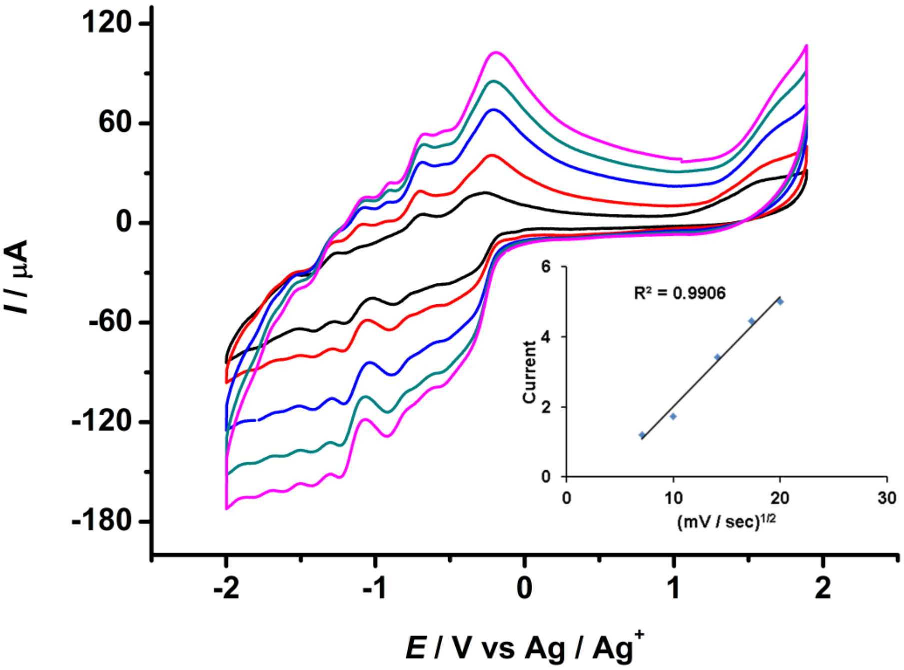
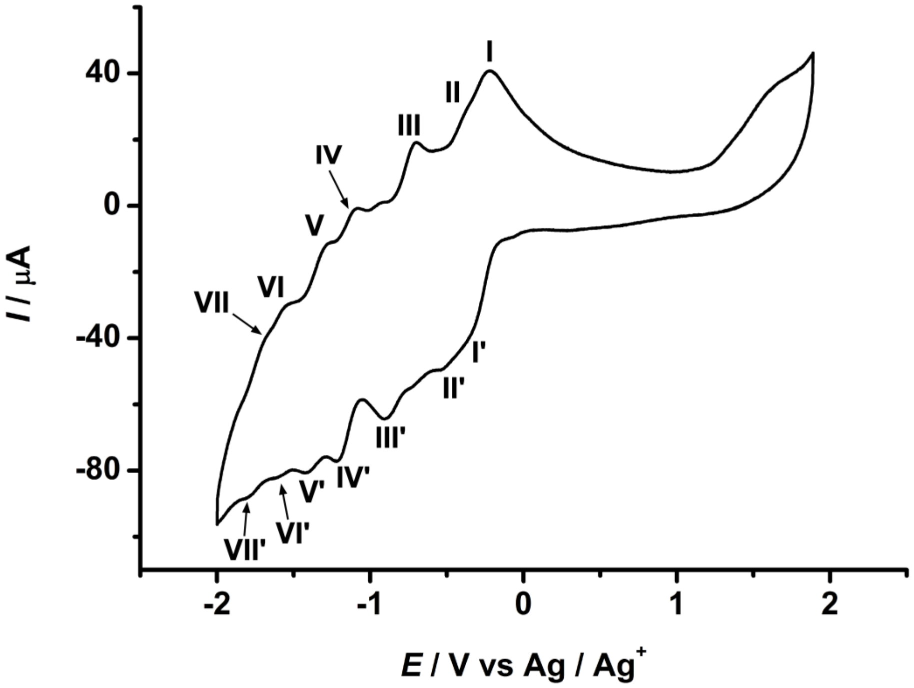


Figure S6: Cyclic voltammograms of compound **2** at scan rates (from inner to outer) 50, 100, 200, 300 and 400 mV/sec. The inset on each graph shows the dependence of the current's intensity as a function of the square root of the scanning rate.



Fugure S7: Cyclic voltammogram of **2**, $[Co_2(P_2W_{15}O_{56})_2]^{20-}$ (10^{-4} M), in 10 mL of 0.1 M $TBAPF_6$ solution. The scan rate was 100 mVs^{-1}, the working electrode was glassy carbon (1.5 mm) and the reference electrode was Ag/Ag^+.

Compound 3

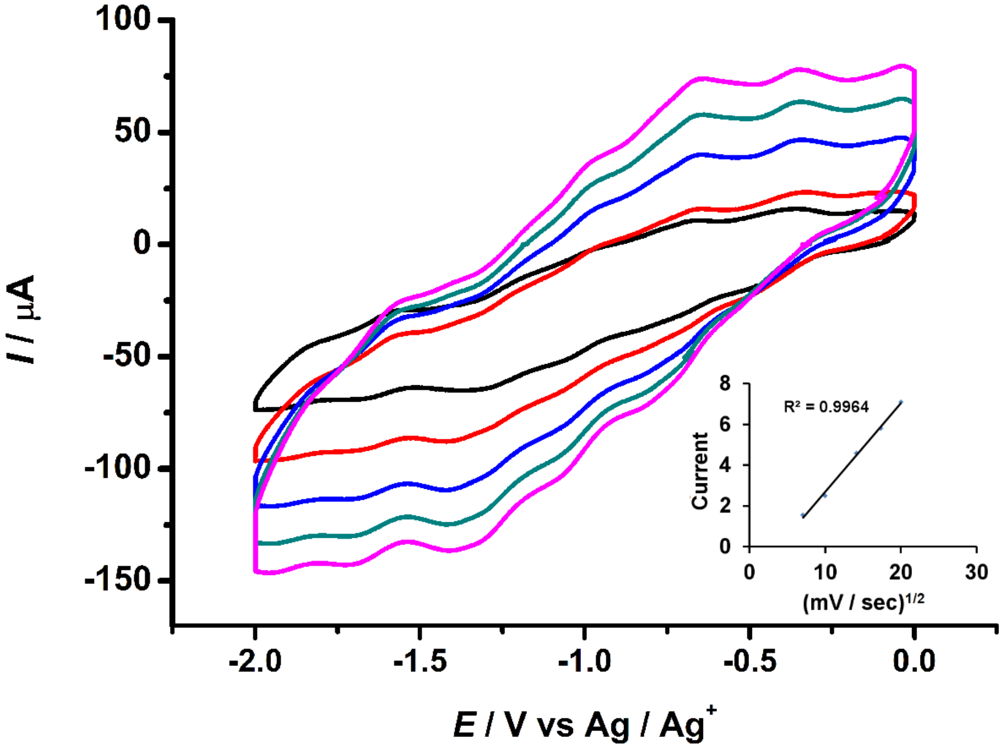


Figure S8: Cyclic voltammograms of compound **3** at scan rates (from inner to outer) 50, 100, 200, 300 and 400 mV/sec.

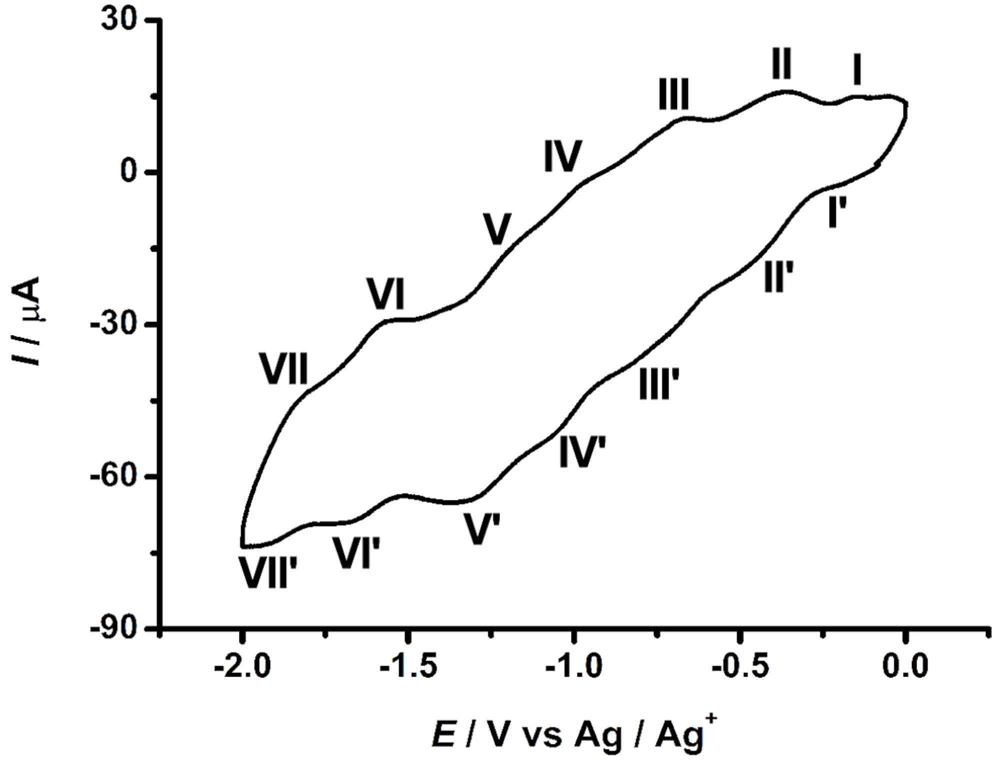


Figure S9: Cyclic voltammogram of **3**, $[Ni_2(P_2W_{15}O_{56})_2]^{20-}$ (10^{-4} M), in 10 mL of 0.1 M $TBAPF_6$ solution. The scan rate was 100 mVs^{-1}, the working electrode was glassy carbon (1.5 mm) and the reference electrode was Ag/Ag^+.

Compound 4

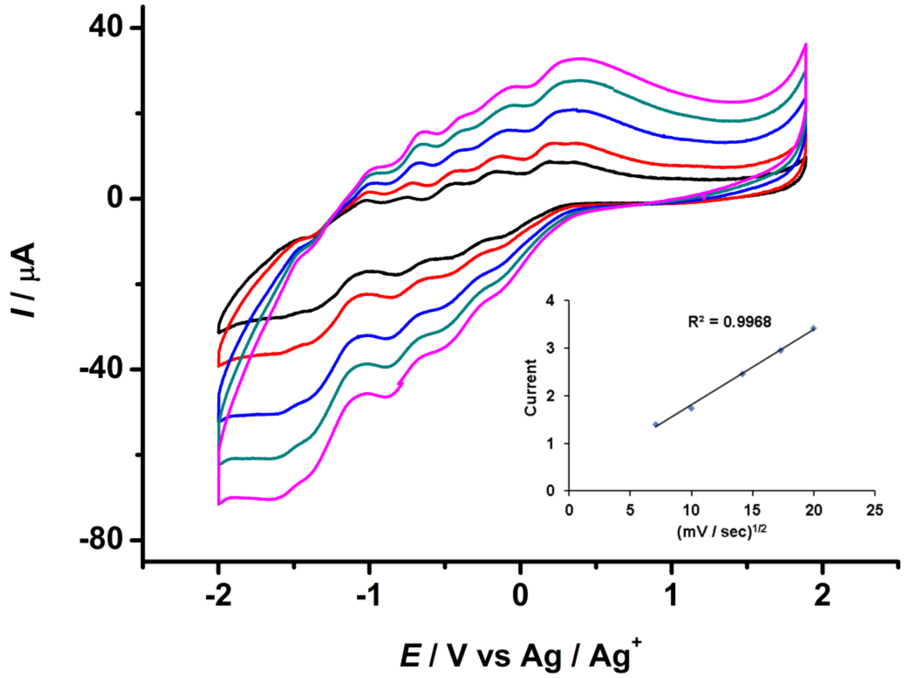


Figure S10: Cyclic voltammograms of compound **4** at scan rates (from inner to outer) 50, 100, 200, 300 and 400 mV/sec. The inset on each graph shows the dependence of the current's intensity as a function of the square root of the scanning rate.

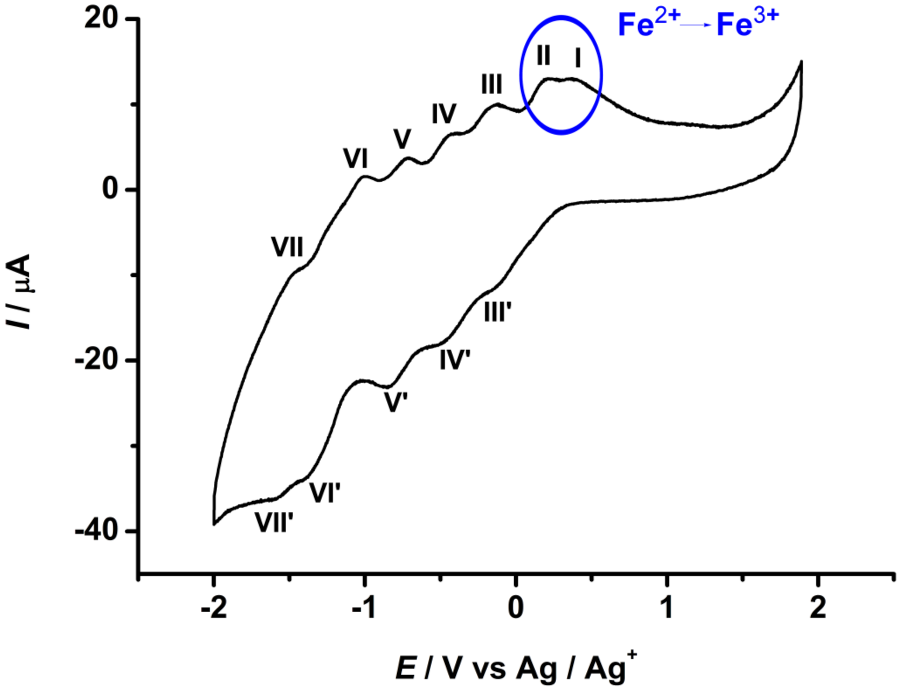


Figure S11: Cyclic voltammogram of **4**, $[Fe_2(P_2W_{15}O_{56})_2]^{18-}$ (10^{-4} M), in 10 mL of 0.1 M $TBAPF_6$ solution. The scan rate was 100 mVs^{-1}, the working electrode was glassy carbon (1.5 mm) and the reference electrode was Ag/Ag^+.

Compound 5

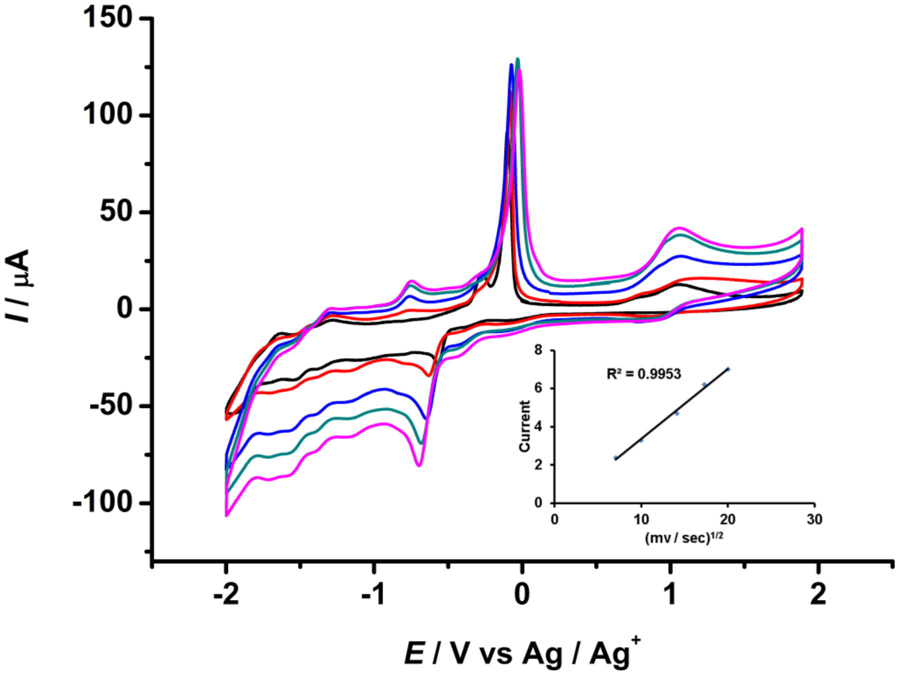


Figure S12: Cyclic voltammograms of compound **5** at scan rates (from inner to outer) 50, 100, 200, 300 and 400 mV/sec. The inset shows the dependence of the current's intensity as a function of the square root of the scanning rate.

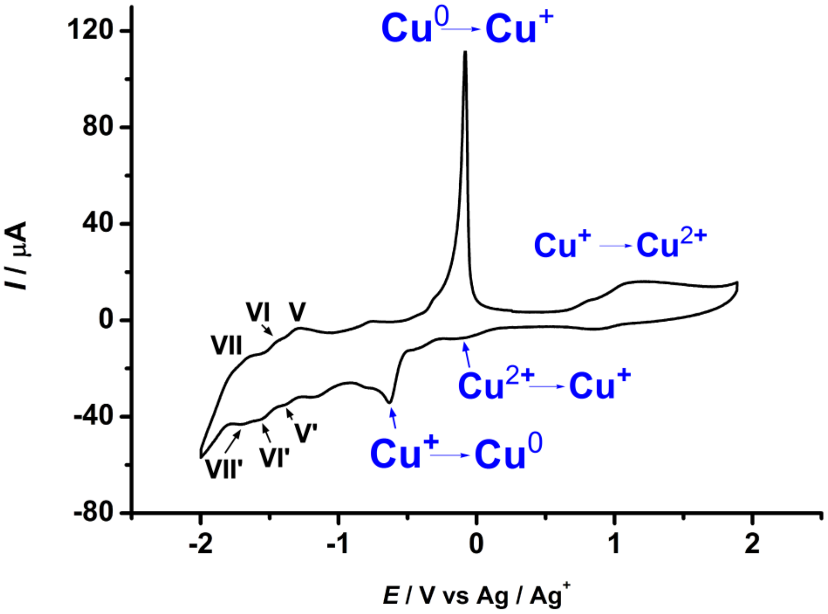


Figure S13: Cyclic voltammogram of **5**, $[Cu_4(H_2O)_2(P_2W_{15}O_{56})_2]^{16-}$ (10^{-4} M), in 10 mL of 0.1 M $TBAPF_6$ solution. The scan rate was 100 mVs^{-1}, the working electrode was glassy carbon (1.5 mm) and the reference electrode was Ag/Ag^+.

7. X-Ray Crystallography

X-ray diffraction structure analysis and crystallographic data: Suitable single crystals of compounds **1-5** were selected and mounted onto the end of a thin glass fibre using Fomblin oil. X-ray diffraction intensity data was measured on an Oxford Diffraction Gemini Ultra with an ATLAS CCD detector using Mo radiation source ($\lambda_{Mo\text{-}K\alpha}$ = 0.71073 Å) or Cu radiation source ($\lambda_{Cu\text{-}K\alpha}$ = 1.54184 Å) at 150 K. Data reduction was performed using the CrysAlis software package and structure solution and refinement was carried out using SHELXS-97[S1] and SHELXL-97[S2] *via* WinGX.[S3] Corrections for incident and diffracted beam absorption effects were applied using analytical numeric absorption correction using a multifaceted crystal model.[S4]

The W, P and O atom positions are crystallographically well defined but the solvent molecules and cations, because of disorder, are difficult to fully analyse by crystallography alone. Therefore, the final molecular formula has been determined by a combination of elemental analysis, thermogravimetric analysis and crystallography. Due to the nature of the crystals obtained by diethylether diffusion into acetonitrile the quality of the reflections from the single crystal were not of high quality. We attempted to collect as many reflections as possible; however, the resulting data are still relatively weak.

Identification code	1	
Empirical formula	$C_{156}H_{352}Mn_2N_{15}NaO_{112}P_4W_{30}$	
Formula weight	10002.78	
Temperature	150(2) K	
Wavelength	0.71073 Å	
Crystal system	Triclinic	
Space group	P-1	
Unit cell dimensions	a = 15.4501(2) Å	α= 70.288(2)°.
	b = 19.6621(4) Å	β= 89.395(2)°.
	c = 28.1389(5) Å	γ = 67.700(2)°.
Volume	7377.3(2) $Å^3$	
Z	1	
Density (calculated)	2.251 Mg/m^3	
Absorption coefficient	11.817 mm^{-1}	
F(000)	4630	
Crystal size	0.46 x 0.21 x 0.10 mm^3	
Theta range for data collection	2.88 to 26.00°.	
Index ranges	-19<=h<=19, -24<=k<=24, -34<=l<=34	
Reflections collected	146141	
Independent reflections	28945 [R(int) = 0.0370]	
Completeness to theta = 26.00°	99.9 %	
Absorption correction	Analytical	
Max. and min. transmission	0.3845 and 0.0740	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	28945 / 87 / 1425	
Goodness-of-fit on F^2	1.081	
Final R indices [I>2sigma(I)]	R1 = 0.0349, wR2 = 0.1029	
R indices (all data)	R1 = 0.0421, wR2 = 0.1075	
Extinction coefficient	none	
Largest diff. peak and hole	3.69 and -1.66 e.$Å^{-3}$	

Identification code	2	
Empirical formula	$C_{134}H_{309}Co_2N_{11}O_{112}P_4W_{30}$	
Formula weight	9624.16	
Temperature	150(2) K	
Wavelength	1.54184 Å	
Crystal system	Monoclinic	
Space group	C2/c	
Unit cell dimensions	a = 29.5229(5) Å	α= 90°.
	b = 29.3096(4) Å	β= 100.074(2)°.
	c = 30.8213(5) Å	γ = 90°.
Volume	26258.6(7) $Å^3$	
Z	4	
Density (calculated)	2.434 Mg/m^3	
Absorption coefficient	25.371 mm^{-1}	
F(000)	17680	
Crystal size	0.35 x 0.28 x 0.23 mm^3	
Theta range for data collection	3.02 to 68.09°.	
Index ranges	-35<=h<=35, -35<=k<=34, -35<=l<=37	
Reflections collected	96962	
Independent reflections	23716 [R(int) = 0.0687]	
Completeness to theta = 68.09°	99.0 %	
Absorption correction	Analytical	
Max. and min. transmission	0.0677 and 0.0408	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	23716 / 40 / 922	
Goodness-of-fit on F^2	1.022	
Final R indices [I>2sigma(I)]	R1 = 0.0807, wR2 = 0.2014	
R indices (all data)	R1 = 0.0886, wR2 = 0.2114	
Extinction coefficient	none	
Largest diff. peak and hole	4.07 and -1.49 e.$Å^{-3}$	

Identification code	3	
Empirical formula	$C_{146}H_{338}N_{10}Ni_2O_{112}P_4W_{30}$	
Formula weight	9783.06	
Temperature	150(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	C2/c	
Unit cell dimensions	a = 45.2790(9) Å	α= 90°.
	b = 21.0927(7) Å	β= 97.190(6)°.
	c = 31.0661(11) Å	γ = 90°.
Volume	29436.6(15) $Å^3$	
Z	4	
Density (calculated)	2.207 Mg/m^3	
Absorption coefficient	11.883 mm^{-1}	
F(000)	18064	
Crystal size	0.35 x 0.19 x 0.07 mm^3	
Theta range for data collection	2.90 to 21.98°.	
Index ranges	-47<=h<=47, -22<=k<=22, -32<=l<=32	
Reflections collected	87247	
Independent reflections	17953 [R(int) = 0.1196]	
Completeness to theta = 21.98°	99.6 %	
Absorption correction	Analytical	
Max. and min. transmission	0.4901 and 0.1033	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	17953 / 48 / 811	
Goodness-of-fit on F^2	0.895	
Final R indices [I>2sigma(I)]	R1 = 0.0783, wR2 = 0.1977	
R indices (all data)	R1 = 0.1807, wR2 = 0.2286	
Extinction coefficient	none	
Largest diff. peak and hole	4.00 and -2.08 e.$Å^{-3}$	

Identification code	4	
Empirical formula	$C_{132}H_{303}Fe_2N_{10}NaO_{112}P_4W_{30}$	
Formula weight	9596.91	
Temperature	150(2) K	
Wavelength	1.54184 Å	
Crystal system	Monoclinic	
Space group	C2/c	
Unit cell dimensions	a = 47.3357(16) Å	α= 90°.
	b = 17.9304(6) Å	β= 114.472(2)°.
	c = 38.5281(11) Å	γ = 90°.
Volume	29762.9(17) Å^3	
Z	4	
Density (calculated)	2.142 Mg/m^3	
Absorption coefficient	22.303 mm^{-1}	
F(000)	17616	
Crystal size	0.27 x 0.26 x 0.24 mm^3	
Theta range for data collection	3.13 to 56.39°.	
Index ranges	-50<=h<=50, -18<=k<=18, -39<=l<=41	
Reflections collected	66530	
Independent reflections	19143 [R(int) = 0.1477]	
Completeness to theta = 56.39°	97.4 %	
Absorption correction	Analytical	
Max. and min. transmission	0.0754 and 0.0651	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	19143 / 63 / 917	
Goodness-of-fit on F^2	1.126	
Final R indices [I>2sigma(I)]	R1 = 0.1065, wR2 = 0.3115	
R indices (all data)	R1 = 0.1368, wR2 = 0.3387	
Extinction coefficient	none	
Largest diff. peak and hole	2.10 and -1.05 e.Å^{-3}	

Identification code	5	
Empirical formula	$C_{150}H_{345}Cu_4N_{12}NaO_{115}P_4W_{30}$	
Formula weight	10073.91	
Temperature	150(2) K	
Wavelength	1.54184 Å	
Crystal system	Monoclinic	
Space group	C2/c	
Unit cell dimensions	a = 35.5081(16) Å	α= 90°.
	b = 15.7651(6) Å	β= 90.826(5)°.
	c = 48.504(3) Å	γ = 90°.
Volume	27149(2) $Å^3$	
Z	4	
Density (calculated)	2.465 Mg/m^3	
Absorption coefficient	23.990 mm^{-1}	
F(000)	18624	
Crystal size	0.17 x 0.07 x 0.06 mm^3	
Theta range for data collection	3.20 to 60.00°.	
Index ranges	-31<=h<=39, -17<=k<=14, -53<=l<=54	
Reflections collected	42551	
Independent reflections	19321 [R(int) = 0.1036]	
Completeness to theta = 60.00°	95.9 %	
Absorption correction	Analytical	
Max. and min. transmission	0.3270 and 0.1058	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	19321 / 23 / 911	
Goodness-of-fit on F^2	0.993	
Final R indices [I>2sigma(I)]	R1 = 0.0928, wR2 = 0.2491	
R indices (all data)	R1 = 0.1503, wR2 = 0.2896	
Extinction coefficient	none	
Largest diff. peak and hole	3.22 and -2.85 e.$Å^{-3}$	

7.1 Crystallographic references

[S1] Sheldrick,G. M. Phase annealing in *SHELX*-90: direct methods for larger structures. *Acta Cryst. A* **46**, 467 - 473 (1990).

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[S3] Farrugia, L. J. *WinGX* suite for small-molecule single-crystal crystallography. *J. Appl. Cryst.*, **32**, 837 - 838 (1999).

[S4] Clark, R. C. & Reid, J. S. *Acta Cryst. A* **51**, 887-897 (1995).