Preparation of [$Pt_2Pb(P_2Phen)_3$](ClO_4)$_2$, **2**.

A 250 mL round bottomed flask was charged with 0.100 g [0.182 mmol] of 2,9-bis(diphenylphosphino)-1,10-phenanthroline dissolved in 20 mL of CH_2Cl_2. $Pb(ClO_4)_2$ 0.084g [0.182 mmol] (dissolved in 40 mL of MeOH/MeCN) was added slowly with swirling. After stirring for 10 minutes a suspension of 0.081g $(dba)_2Pt$ [0.122 mmol] in 50 mL of MeOH was added. After stirring an additional 12 hours, volatiles were removed and residue dissolved in a minimum amount of MeCN. Flash chromatography (alumina) eluting with MeCN followed by precipitation with Et_2O affords 0.110g [0.0450 mmol] of **2** as a brown solid (76%). $C_{114}H_{84}Cl_2N_6O_8P_6PbPt_2$ Calc (%) C 54.34, H 3.36, N 3.33; Found C 54.66, H 2.87, N 3.51. ^{1}H NMR (499.8 MHz, 25°C, acetone-d_6) δ 8.44 (d, J = 8.5 Hz, 6H) 8.069 (s, 6H) 7.66 (d, J = 8.5 Hz, 6H) 7.41 (t, J = 8.0 Hz, 6H) 7.14 (m-br, 12H) 6.97(t, J = 8.5 Hz, 12H) 6.4 (t, J = 7.5 Hz, 6H) 6.35 (m-br, 12H) 6.22 (t, J = 7.5 Hz, 12H). ^{31}P{^{1}H} (121 MHz, $CDCl_3$/DCM/MeOH, 25°C) δ 48.1 (s, $^1J_{Pt\text{-}P}$ = 4085 Hz); ^{195}Pt (107.1 MHz, MeOH/acetone-d_6, ext. ref. H_2PtCl_6 in D_2O, 25°C) δ -2856 (q, $^1J_{P\text{-}Pt}$ = 4085 Hz). UV-Vis (CH_2Cl_2, 0.005495 mM): λ in nm (ε in M^{-1}, cm^{-1}) : 231 (139820), 291 (112720), 319* (99443), 470 (5003). [* denotes shoulder]

MALDI-TOF spectrum for *[$Pt_2Pb(P_2Phen)_3$](ClO_4)$_2$*, **2**.