Supplementary Material (ESI) for Dalton Transactions
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Trigonal prismatic Cu^{II}_6-pyrazolato cages: Structural and Electrochemical Studies

Gellert Mezei, Marlyn Rivera-Carrillo and Raphael G. Raptis*

PPN{[Cu_3(μ$_3$-O)(μ-4-Cl-pz)$_3$]$_2$(μ-3,5-Ph$_2$pz)$_3$} (PPN[4])

Prepared by method B, using $CuCl_2{\cdot}2H_2O$, 4-chloropyrazole, 3,5-diphenylpyrazole, NaOH and PPNCl (83%). Anal.: Cal. for $C_{99}H_{75}Cl_6Cu_6N_{19}O_2P_2$: C, 53.59; H, 3.41; N, 11.99 %. Found: C, 52.08; H, 3.73; N, 11.84 %. δ_H (300 MHz; $CDCl_3$) 29.17 (3H, s, H^4-3,5-Ph$_2$-pz), 21.09 (12H, s, $H^{3,5}$-pz), 11.96 (12H, s, orthoH-Ph).

PPN{[Cu_3(μ$_3$-O)(μ-pz)$_3$]$_2$(μ-4-Cl-3,5-Ph$_2$pz)$_3$} (PPN[5])

Prepared by method B, using $CuCl_2{\cdot}2H_2O$, pyrazole, 4-chloro-3,5-diphenylpyrazole, NaOH and PPNCl. Anal.: Cal. for $C_{99}H_{86}Cl_3Cu_6N_{19}O_6P_2$: C, 54.36; H, 3.96; N, 12.19 %. Found: C, 54.36; H, 3.88; N, 12.17 %. δ_H (300 MHz; $CDCl_3$) 35.57 (6H, s, H^4-pz), 18.56 (12H, s, $H^{3,5}$-pz), 10.83 (12H, s, orthoH-Ph).

PPN{[Cu_3(μ$_3$-O)(μ-4-Cl-pz)$_3$]$_2$(μ-4-Cl-3,5-Ph$_2$pz)$_3$} (PPN[6])

Prepared by method B, using $CuBr_2$, 4-chloropyrazole, 4-chloro-3,5-diphenylpyrazole, NaOH and PPNCl (88%). Anal.: Cal. for $C_{99}H_{72}Cu_6Cl_9N_{19}O_2P_2$: C, 51.21; H, 3.13; N, 11.46 %. Found: C, 50.54; H, 3.31; N, 11.94 %. δ_H (300 MHz; $CDCl_3$ 20.74 (12H, s, $H^{3,5}$-Ph)), 10.74 (12H, s, orthoH-Ph).

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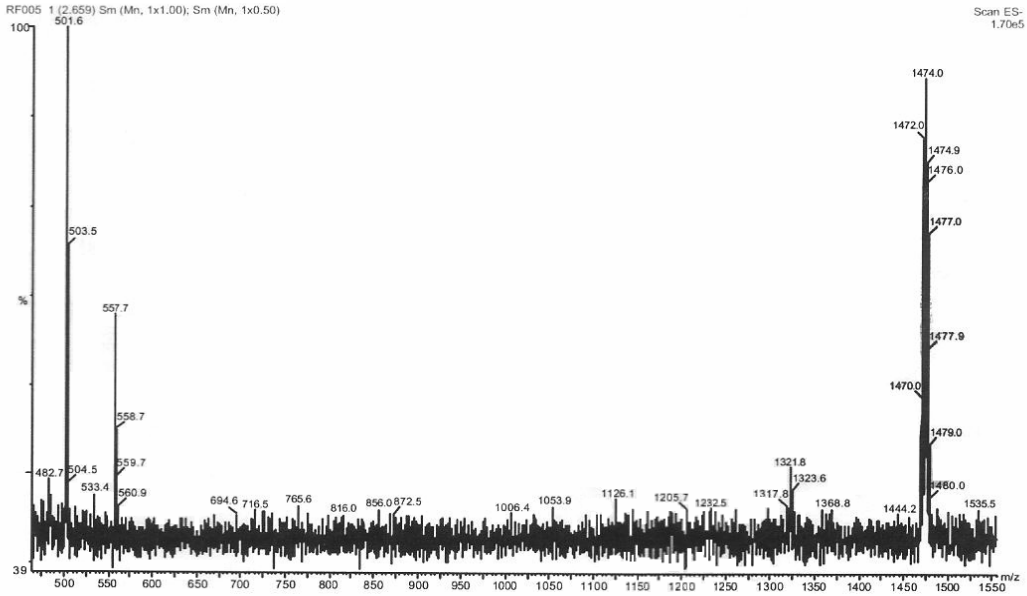
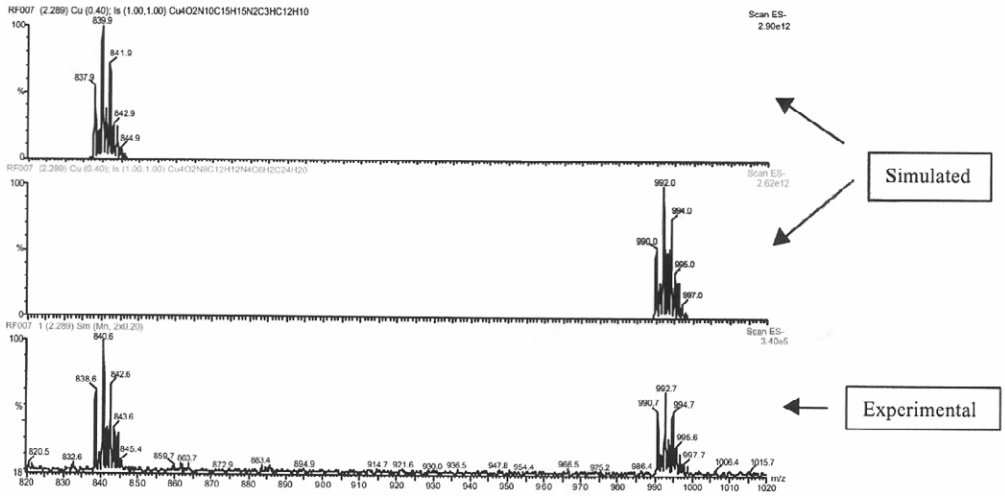
ESI-MS Spectra for PPN[**3**]

3$^-$ in CH_2Cl_2, 25 V, Negative Mode



3$^-$ in CH_2Cl_2, 75 V, Negative Mode



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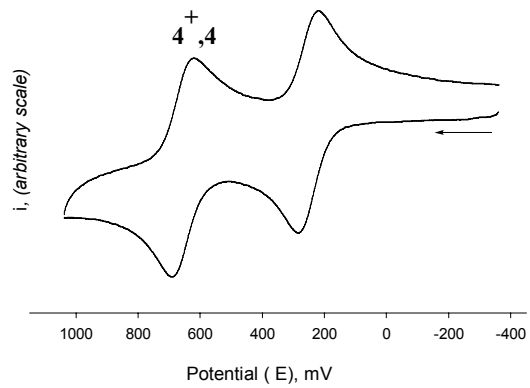
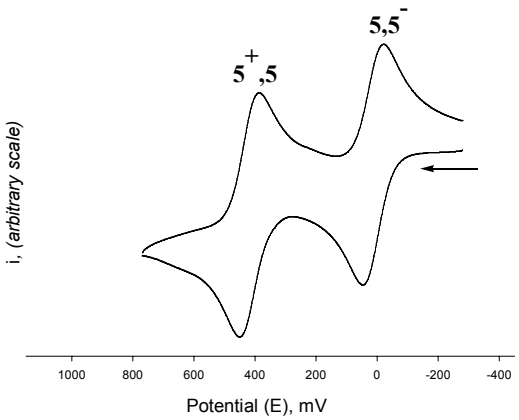
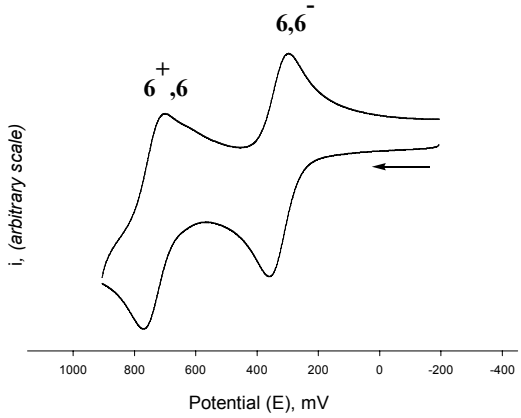
Cyclic voltammograms of **4**$^-$, **5**$^-$ and **6**$^-$; 0.5M Bu_4NPF_6/CH_2Cl_2, Pt working electrode, versus ferrocene/ferricinium.







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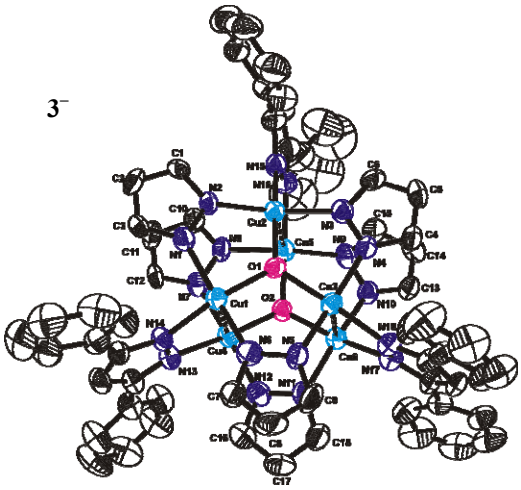
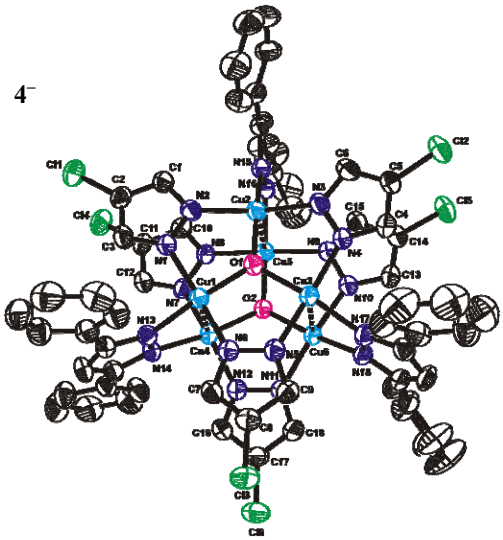
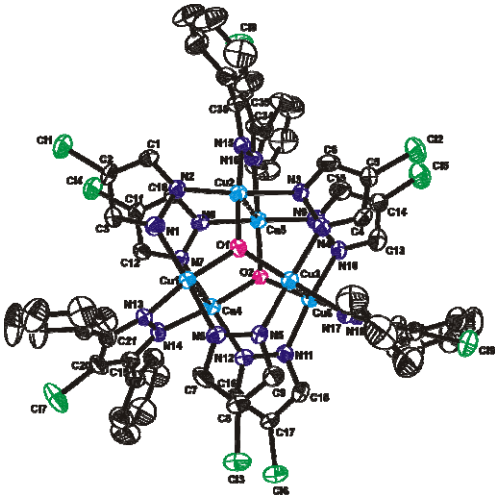
ORTEP drawings for compounds **3**$^-$, **4**$^-$, and **6**$^-$ showing the atom labeling scheme. Thermal ellipsoids at 30% probability level. Hydrogen atoms omitted.

3$^-$



4$^-$



6$^-$