Synthesis of 1 A mixture of $CuCl_2 \cdot 2H_2O$ (0.170 g, 1.00 mmol) and $OP[N(H)^tBu]_3$ (0.263 g, 1.00 mmol) in toluene (10 mL) in a polytetrafluoroethylene-lined Parr acid digestion vessel (23 mL) was heated to 80°C for 38 h, and then cooled to room temperature over a period of 24h. Yellow block-like crystals of **1** were obtained (0.093g, 0.149 mmol, 45%). Anal. Calcd for $C_8H_{27}Cl_7Cu_3N_2O_2$: C, 15.45; H, 4.38; N, 4.50. Found: C, 16.12; H, 4.10; N, 4.86. IR (cm^{-1}): 3515 [υ(O-H)], 3123 [υ(N-H)].

Synthesis of 2 Compound **2** was synthesized by employing the same procedure except for the use of

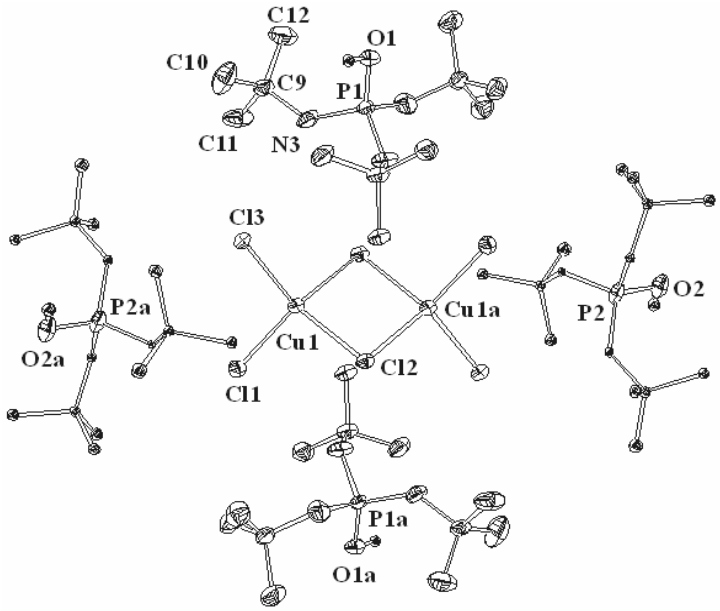

Fig ORTEP drawing of the $[P(OH)(NH^tBu)_3]_4[Cu_2Cl_6]$ structure showing the labeling scheme of the atoms.

$[^tBuNH_3]Cl$ instead of $OP[N(H)^tBu]_3$. Red needle-like crystals were isolated (0.129g, 0.530mmol, 53%). Anal. Calcd for $C_4H_{12}Cl_3CuN$: C, 19.69; H, 4.96; N, 5.74. Found: C, 19.52; H, 4.83; N, 5.59. IR (cm^{-1}): 3102 [υ(N-H)].

Synthesis of 3 Pale yellow crystals of **3** were isolated from the mother liquor in the synthesis of **1.** IR (cm^{-1}): 3504 [υ(O-H)], 3381 [υ(N-H)].