Crystal data for **1**: $C_4H_{13.5}Cl_{3.5}Cu_{1.5}NO$, M = 311.04, orthorhombic, *Pnma*, a = 9.1349(18), b = 26.780(5), c = 8.5459(17) Å, V = 2090.6(7) Å^3, Z = 8, μ(Mo-K$_\alpha$) = 3.922 mm^{-1}, $\theta_{max.}$ = 25.03^o, 6759 reflections measured, 1873 unique (R_{int} = 0.0624) and used to refine 119 parameters. $R_1(2\sigma)$ = 0.0338. For **2**: $C_4H_{12}Cl_3CuN$, M = 244.04, monoclinic, $P2_1/n$, a = 6.1048(12), b = 16.946(3), c = 9.1044(18) Å, β = 90.63(3)^o, V = 941.8(3) Å^3, Z = 4, μ(Mo-K$_\alpha$) = 3.095 mm^{-1}, $\theta_{max.}$ = 26.37^o, 3476 reflections measured, 1926 unique (R_{int} = 0.0519) and used to refine 82 parameters. $R_1(2\sigma)$ = 0.0461. For **3**: $C_{27.5}H_{66}Cl_3CuN_6O_2P_2$, M = 744.69, triclinic, P-1, a = 10.241(2), b = 13.790(3), c = 16.238(3) Å, α = 103.15(3)^o, β = 94.85(3)^o, γ = 103.03(3)^o, V = 2153.1(7) Å^3, Z = 2, μ(Mo-K$_\alpha$) = 0.797 mm^{-1}, $\theta_{max.}$ = 23.25^o, 10118 reflections measured, 5976 unique (R_{int} = 0.0392) and used to refine 352 parameters. $R_1(2\sigma)$ = 0.0915. Data sets were collected at 173K using a Nonius Kappa CCD diffractometer. The structure was solved by direct methods[1] and refined by full-matrix least-squares methods with SHELXL-97.[2]

1 A. Altomare, M. Cascarano, C. Giacovazzo and A. Guagliardi, *J. Appl. Crystallogr.*, 1993, **26**, 343.

2 G. M. Sheldrick, *SHELXL-97, Program for the Solution of Crystal Structures*; University of Göttingen: Göttingen, Germany, 1997.