

Copper(I) iodide complexes derived from *N*-alkyl-1,3,5-triaza-7-phosphaadamantanes; synthesis, crystal structures, photoluminescence, and identification of the unprecedented $\{\text{Cu}_3\text{I}_5\}^{2-}$ cluster.

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The new luminescent mononuclear $[\text{CuI}(\text{PTA-Me})_3](\text{I})_3$ (**1**) and $[\text{CuI}(\text{PTA-Et})_3](\text{I})_3$ (**2**) and trinuclear $[\text{Cu}_3\text{I}_2(\mu\text{-I})(\mu_3\text{-I})_2(\text{PTA-Pr})_2]$ (**3**) copper(I) complexes have been easily prepared, in aqueous medium and at ambient conditions, from copper(II) nitrate and *N*-m e t h y l - , *N*-ethyl-, and *N*-propyl-1,3,5-triaza-7-phosphaadamantane iodides $[\text{PTA-R}]\text{I}$ ($\text{R} = \text{Me}, \text{Et}, n\text{Pr}$), respectively. They have been fully characterized by IR, ^1H and ^{31}P NMR spectroscopies, FAB- MS^+ , and elemental and single-crystal X-ray diffraction analyses, the latter featuring a novel type of tricopper iodide $\{\text{Cu}_3\text{I}_2(\mu\text{-I})(\mu_3\text{-I})_2\}^{2-}$ cluster. Compounds **1**, **2**, and **3** exhibit distinct photoluminescence in the solid state, DMSO solution, and frozen glass, thus extending to copper centers the application (previously limited to only Au complexes) of 1,3,5-triaza-7-phosphaadamantane (PTA) or any of its derivatives toward the preparation of luminescent organometallic materials. In addition, complex **3** widens the cluster chemistry of PTA, providing the first example of a metal halide cluster bearing a cage-like PTA core.

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