

Supporting Information

[Ag₇₀(PW₉O₃₄)₂(^tBuC≡C)₄₄(H₂O)₂]⁸⁺: Ionothermal Synthesis of a Silver Cluster Encapsulating Lacunary Polyoxometalate Ions

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1. Materials and Methods.

For the synthesis of AgC≡CBu^t: Ag₂O (3.00g, 12.9mmol) was dissolved in ammonia (60 ml) under stirring. HC≡CBu^t (1.50g, 18.3mmol) in ethanol (5 ml) was dropwise added to the silver ammonia solution, white precipitate was generated immediately, which was filtered and washed by water, ethanol and ethyl ether. Yield: 3.25 g, 94.7%.

Na₉[A-α-PW₉O₃₄] was synthesized according to the literature.¹ Other reagents and solvents for synthesis were obtained from commercial sources and used without further purification.

C, H, N elemental analyses were carried out with a Vario El III microanalyzer. P, W and Ag were analyzed by energy dispersive X-ray spectroscopy with a SEM, Hitachi S4800 scanning electron microscopy. X-ray powder diffractometry study of the compound **1** was performed on a Panalytical X-Pert pro diffractometer with Cu K_α radiation. FT-IR spectrum was recorded with KBr pellets in the range 4000-400 cm⁻¹ with a Nicolet AVATAR FT-IR380 spectrometer. Electrochemical measurements were performed on a CHI-660C electrochemical workstation (CH Instruments, Inc.).

Reference:

1. Tézé, A.; Hervé, G. *Inorganic Syntheses*; John Wiley & Sons: New York, **1990**, 27, 87.

2. Characterization

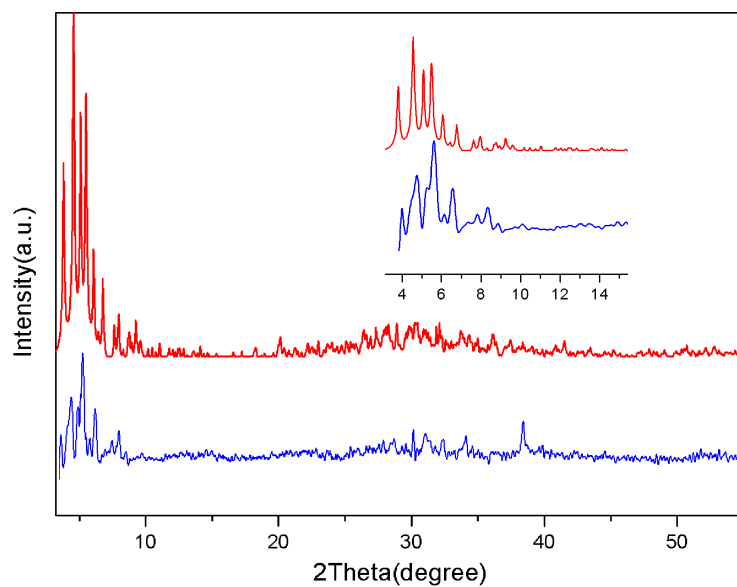


Figure S1. The measured (blue) and simulated (red) X-ray powder diffraction patterns of **1**.

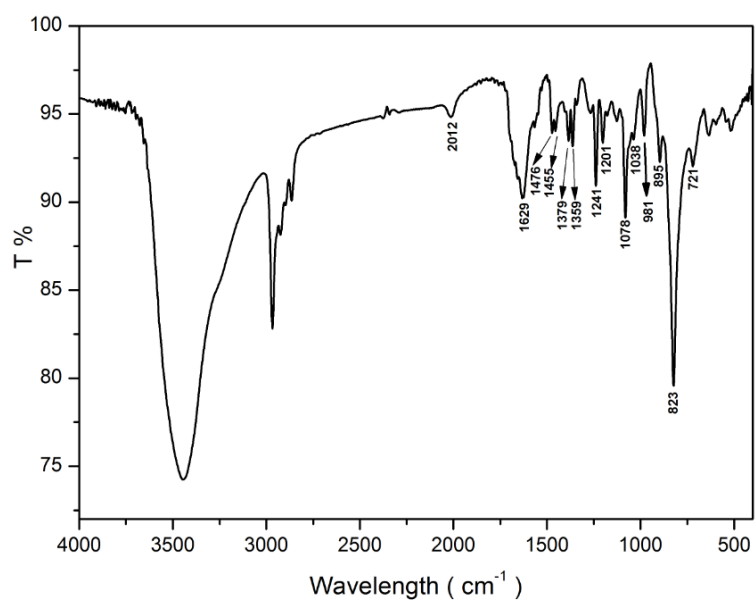


Figure S2. IR spectrum of compound **1**.

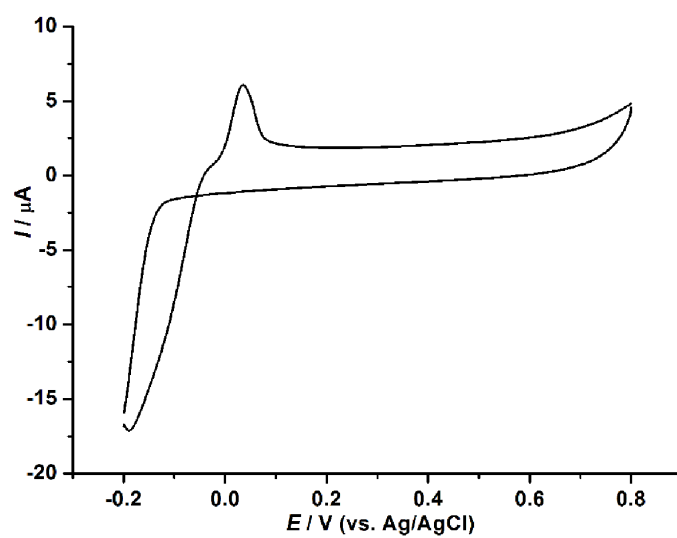


Figure S3. Cyclic voltammogram of AgBF_4 in deoxygenated $[\text{BMIm}]\text{BF}_4$ electrolyte in N_2 stream at a scan rate of 0.02 V s^{-1} .