

Supporting Information

Pyrovanadate-templated Silver(I)-ethynide Cluster Circumscribed by Macrocyclic Polyoxovanadate(V)

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1. Experimental details

Materials and Methods

All chemicals were commercially purchased and used as supplied. Elemental analyses for C, H and N were performed with a Perkin-Elmer 2400 CHN elemental analyzer. IR spectra were recorded in the range 400–4000 cm^{-1} on a Nicolet Impact 420 FTIR spectrometer with pressed KBr pellets. The morphology of the samples was studied by transmission electron microscopy (TEM) using a Hitachi model H-800 transmission electron microscope at an accelerating voltage of 200 kV. Electrochemical Experiments. The electrochemical properties were investigated with a Potentiostat/Galvanostat Model 263A instrument (Princeton Applied Research) driven by a PC with proprietary software. A conventional three-electrode system was used. The concentrations of **1**, $(\text{Et}_4\text{N})\text{VO}_3$ and AgBF_4 in the measurement of cyclic voltammetric behavior were in DMF. The solutions were de-aerated thoroughly for at least 20 min. with pure nitrogen before experimental measurement.

X-ray Crystallography

Crystal data were collected on a Bruker Smart Apex II CCD diffractometer with $\text{Mo-K}\alpha$ radiation ($\lambda = 0.71073 \text{ \AA}$). The structures were solved by direct methods with SHELXS-97^[1] and refined by full-matrix least-squares techniques using the SHELXL-97 program^[2] within WINGX.^[2]

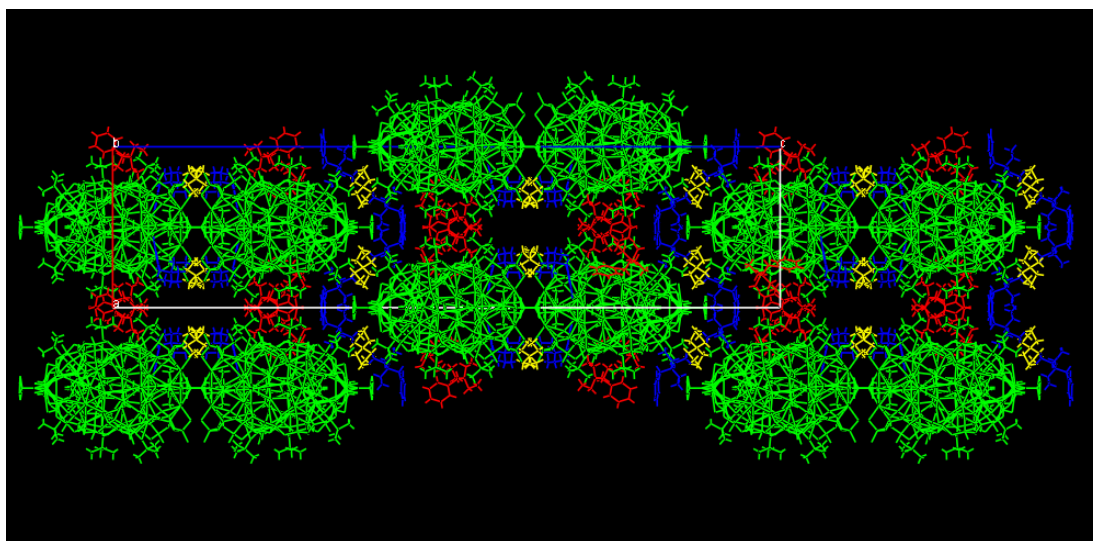


Figure S1. Packing diagram of complex **1** with eight BzEt_3N^+ and two Et_4N^+ counter cations, viewed along the b -axis. The $[(\text{V}_2\text{O}_7)_2@ \text{Ag}_{44}(\text{C}\equiv\text{C}^t\text{Bu})_{14}@(\text{V}_{32}\text{O}_{96})]^{10-}$ cluster is shown in green color, the Et_4N^+ cation in yellow, and the independent BzEt_3N^+ cations in red and blue.

2. TEM results for complex **1**.

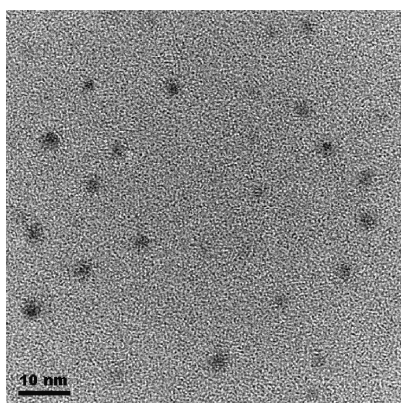


Figure S2. TEM image of complex **1**.

3. Cyclic voltammogram for **1**.

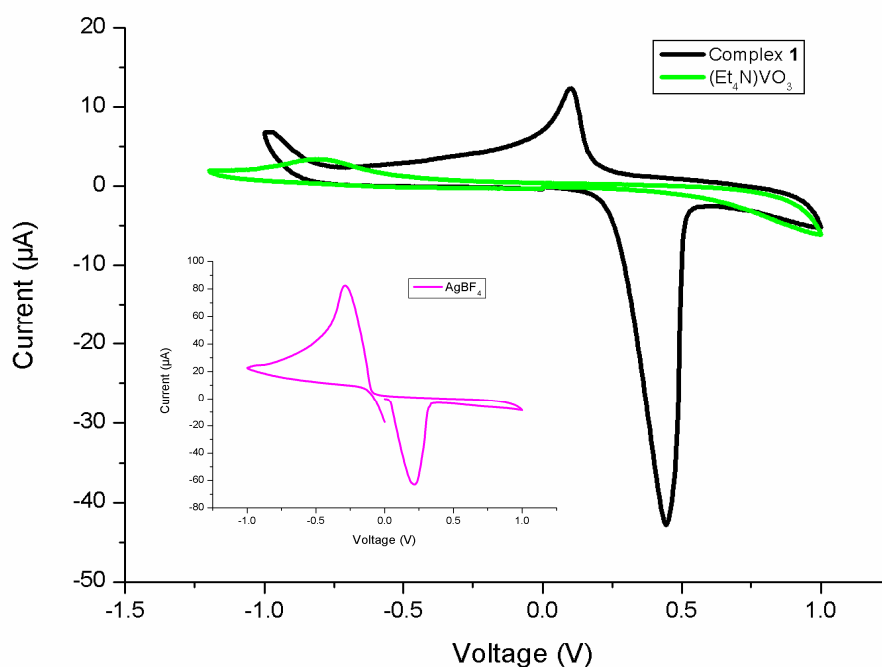


Figure S3. Cyclic voltammograms for complex **1**, $(\text{Et}_4\text{N})\text{VO}_3$ and AgBF_4 in DMF.

References

- [1] Sheldrick, G. M. *SHELX-97, Program for Crystal Structure Refinement*; University of Göttingen: Göttingen, Germany, 1997.
- [2] Sheldrick, G. M. *SHELXL-97, Program for Crystal Structure Solution*; University of Göttingen: Göttingen, Germany, 1997.
- [3] Farrugia, L. J. *WINGX, A Windows Program for Crystal Structure Analysis*, University of Glasgow, UK, 1988.