

**Supporting Information for**

**Silver-Ethynide Clusters Constructed with Fluorinated  $\beta$ -  
Diketonate ligands**

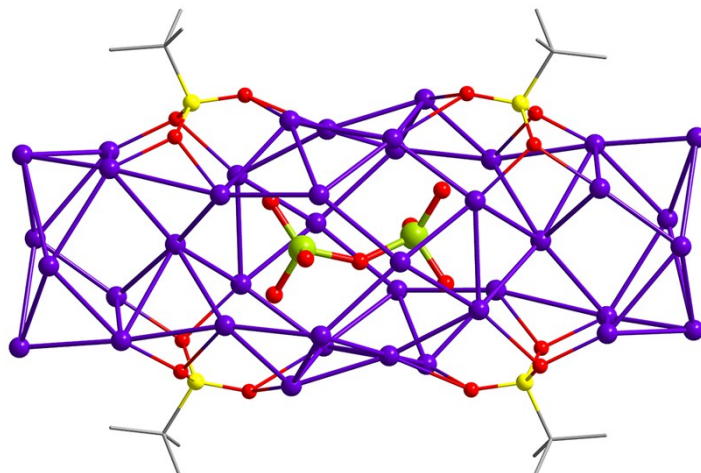
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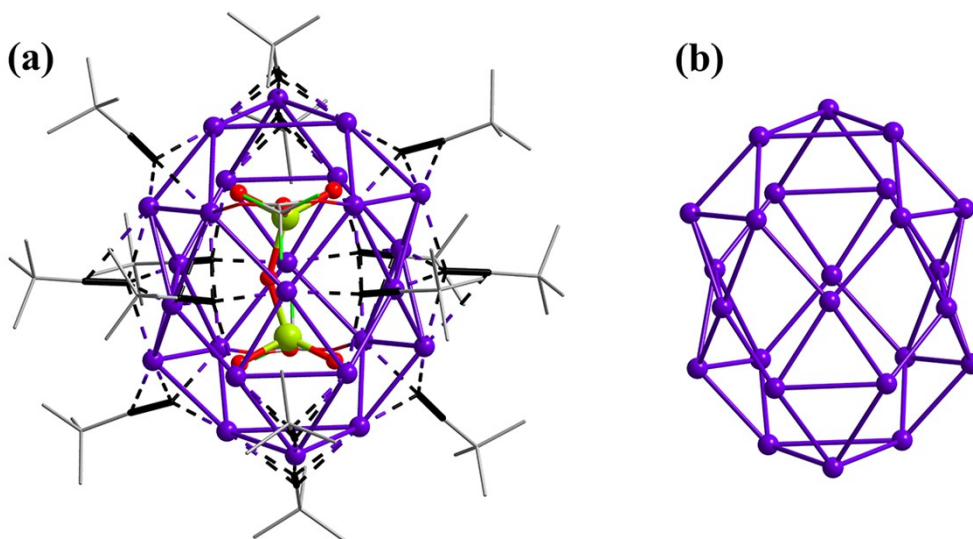
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### X-ray Crystallography.

Single crystal X-ray data were collected at 173 K with Mo/K $\alpha$  radiation ( $\lambda = 0.71073$  Å) on a Bruker D8 Quest diffractometer equipped with a Photon 100 CMOS detector. A multi-scan method (SADABS) was used for absorption corrections. The structures were solved by direct methods and refined with SHELXL-2014.<sup>[S1]</sup>



**Fig. S1** Perspective view of V<sub>2</sub>O<sub>7</sub>@Ag<sub>40</sub>(<sup>t</sup>BuPO<sub>3</sub>)<sub>4</sub> cluster in compound **8**. All peripheral <sup>t</sup>BuC≡C<sup>−</sup>, hfac<sup>−</sup>, TFA and two aqua molecules are omitted for clarity. Color mode: Ag, violet; V, lime; P, yellow; O, red; C, gray.



**Fig. S2** (a) The view of [V<sub>2</sub>O<sub>7</sub>@Ag<sub>24</sub>(<sup>t</sup>BuC≡C)<sub>14</sub>(CF<sub>3</sub>COO)<sub>2</sub>]<sup>4+</sup> cluster in complex **8**. (b) The silver arrangement of the Ag<sub>24</sub> cluster. The C≡C triple bonds are represented by thick black rods and Ag...C bonds are indicated by broken lines. Color mode: Ag, violet; V, lime; O, red; C, gray.

## FT-IR spectra

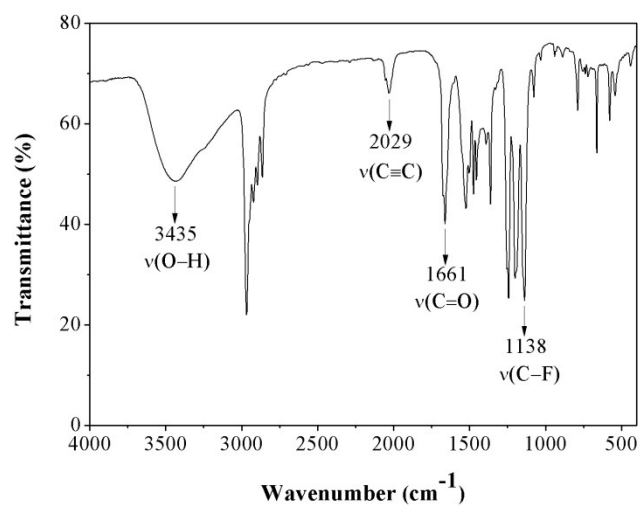


Fig. S3 IR spectrum of compound 1

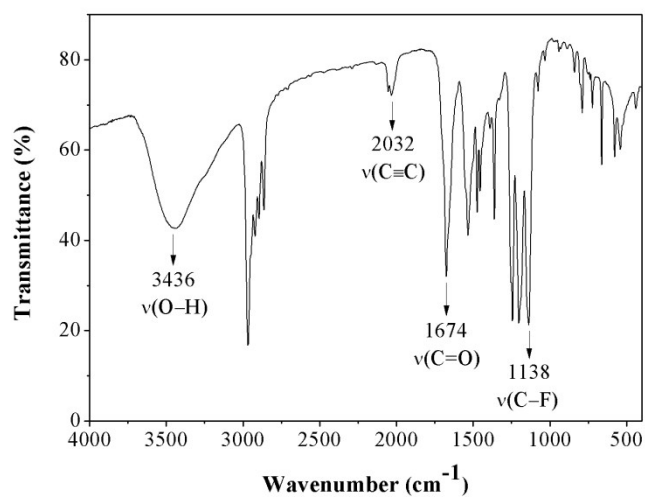


Fig. S4 IR spectrum of compound 2

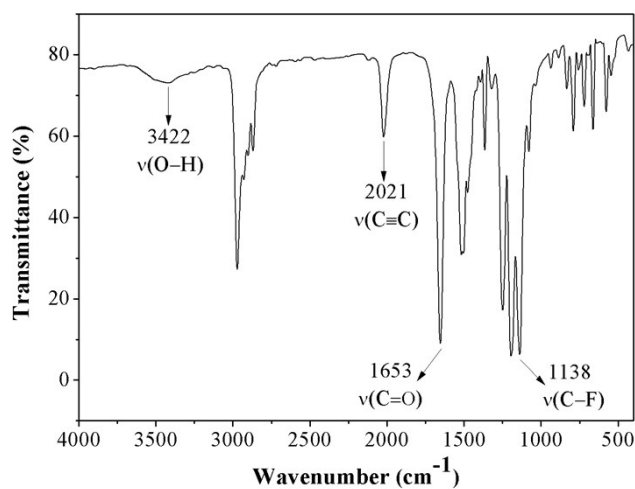
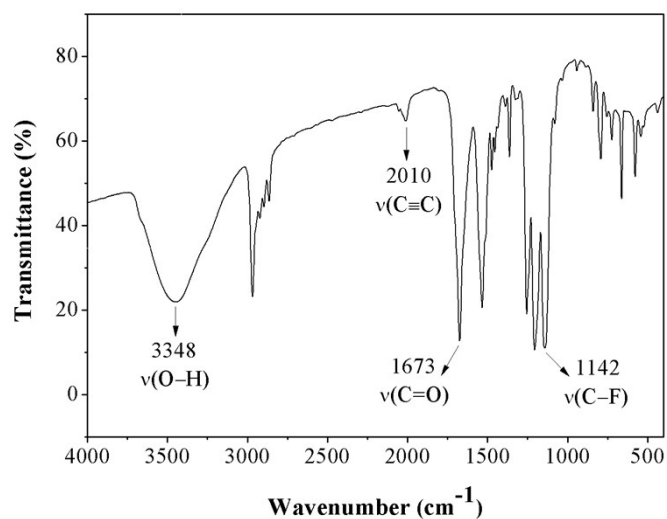
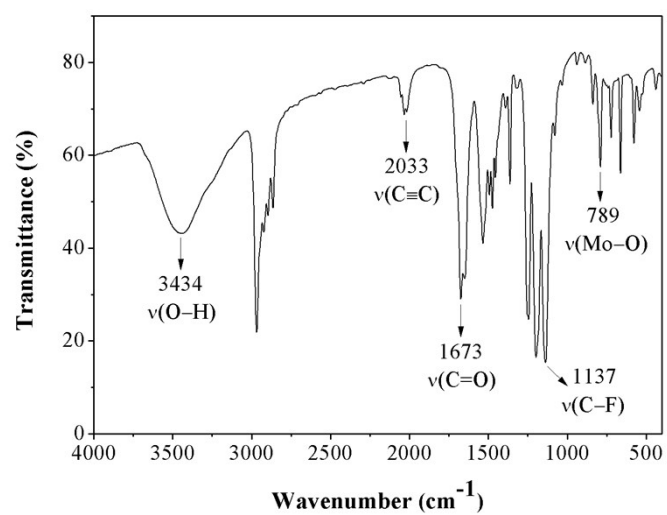


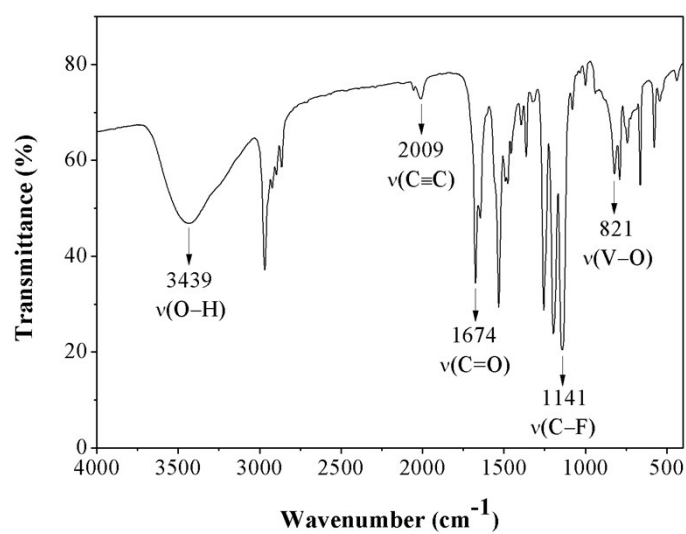
Fig. S5 IR spectrum of compound 3



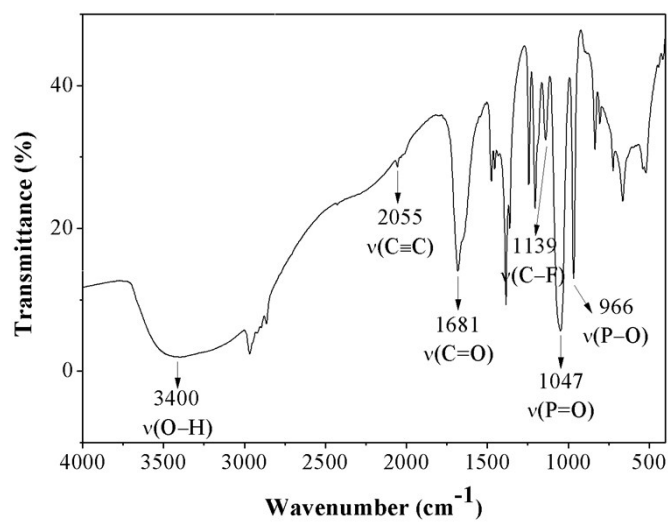
**Fig. S6** IR spectrum of compound 4



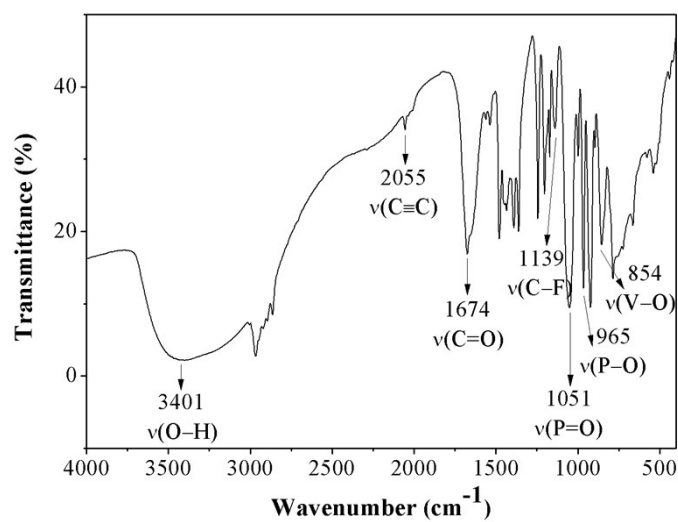
**Fig. S7** IR spectrum of compound 5



**Fig. S8** IR spectrum of compound 6



**Fig. S9** IR spectrum of compound **7**



**Fig. S10** IR spectrum of compound **8**

## References

[S1] Sheldrick, G. M. *Acta Crystallogr. Sect. C Struct. Chem.*, 2015, **71**, 3–8.