#Supplementary Material (ESI) for Chemical Communications

$[\{(Mo)Mo_5O_{21}(H_2O)_3(SO_4)\}_{12}(VO)_{30}(H_2O)_{20}]^{36-}$: A molecular quantum spin icosidodecahedron

Bogdan Botar,*[a] Paul Kögerler*[b] and Craig L. Hill*[a]

[a] *Department of Chemistry, Emory University, Atlanta, GA, USA 30322. E-mail: bbotar@emory.edu; chill@emory.edu*
[b] *Ames Laboratory, Iowa State University, Ames, IA 50011, USA. E-mail: kogerler@ameslab.gov*

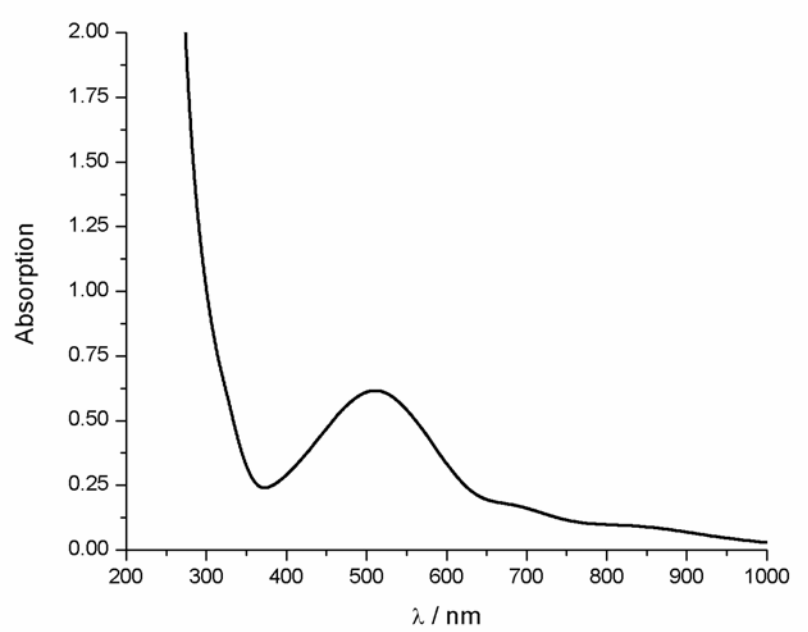

Fig. S1 Electronic absorption spectrum of **1** in water (pH 2.5).

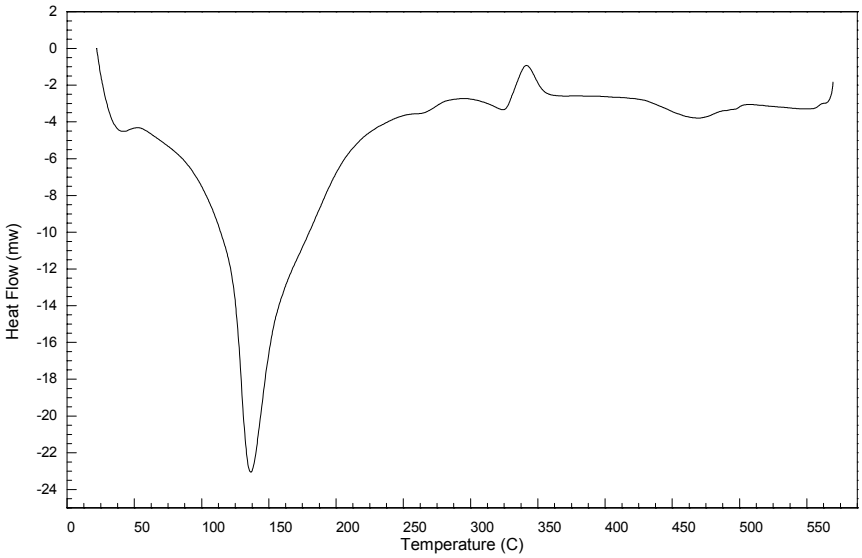

Fig. S2 Differential scanning calorimetry (DSC) data of **1**. Crystalline samples (10 mg) heated from 25 °C to 570 °C at a rate of 20 °C/min.

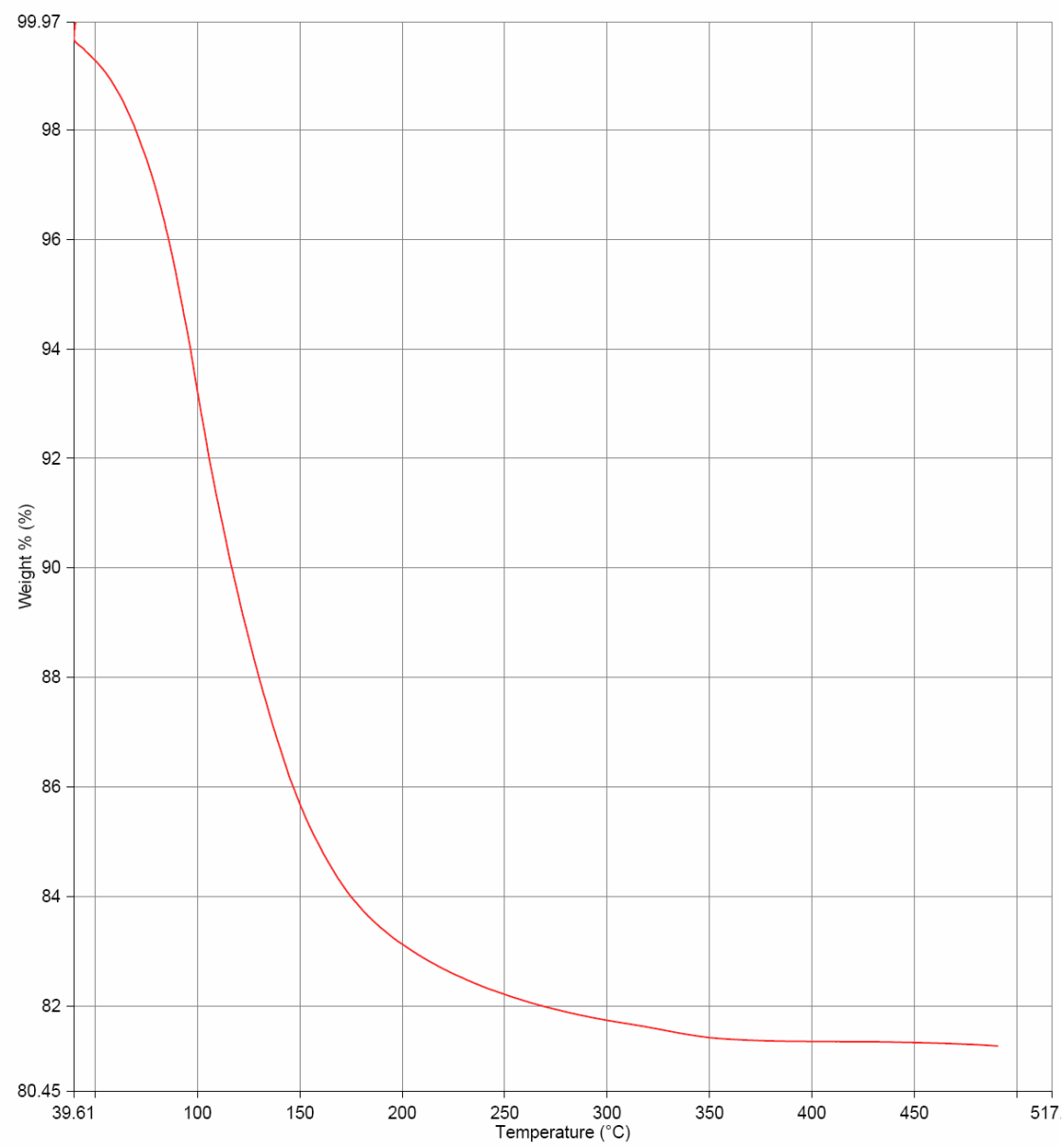

Fig. S3 Thermogravimetric data for **1** (28.193 mg, heating rate: 10 °C/min, N_2).

#Supplementary Material (ESI) for Chemical Communications

Potentiometric titrations:

Fifteen samples were dissolved in 0.5 M H_2SO_4 containing excess $(NH_4)_2Ce^{IV}(NO_3)_6$ at 60-70 °C and back-titrated with 0.1 M $NaNO_2$. This resulted in a sharp drop in the potential (typically from 1250 to 850 mV) at the equivalence point.

Crystallographic data:

One S atom was found to be distributed over three sites (S6, S7 and S8) with the total occupancy of 1.0. Some Mo atoms show the commonly observed O=Mo-OH_2 ↔ H_2O-Mo=O disorder, present in nearly all giant polyoxomolybdate species. Some K atoms and water molecules of crystallization were disordered and were refined with partial occupancies based on their large thermal parameters.