SUPPLEMENTARY INFORMATION

A new class of hybrid super-supertetrahedral cluster and its assembly into a five-fold interpenetrating network

P. Vaqueiro,[a*] S. Makin,[a] Y. Tong[b] and S. J. Ewing[b]

a. Department of Chemistry, University of Reading, Reading RG6 6AD, UK.

b. Institute of Chemical Sciences, Heriot-Watt University, Edinburgh EH14 4AS, UK.

Experimental details:

1. *Powder X-ray diffraction:*

Finely ground bulk samples were scanned on a Bruker D8 Discover diffractometer, equipped with a Lynx Eye linear detector and using nickel-filtered Cu Kα radiation (λ=1.54178 Å). This instrument, which is equipped with a Goebel mirror, operates in transmission mode.
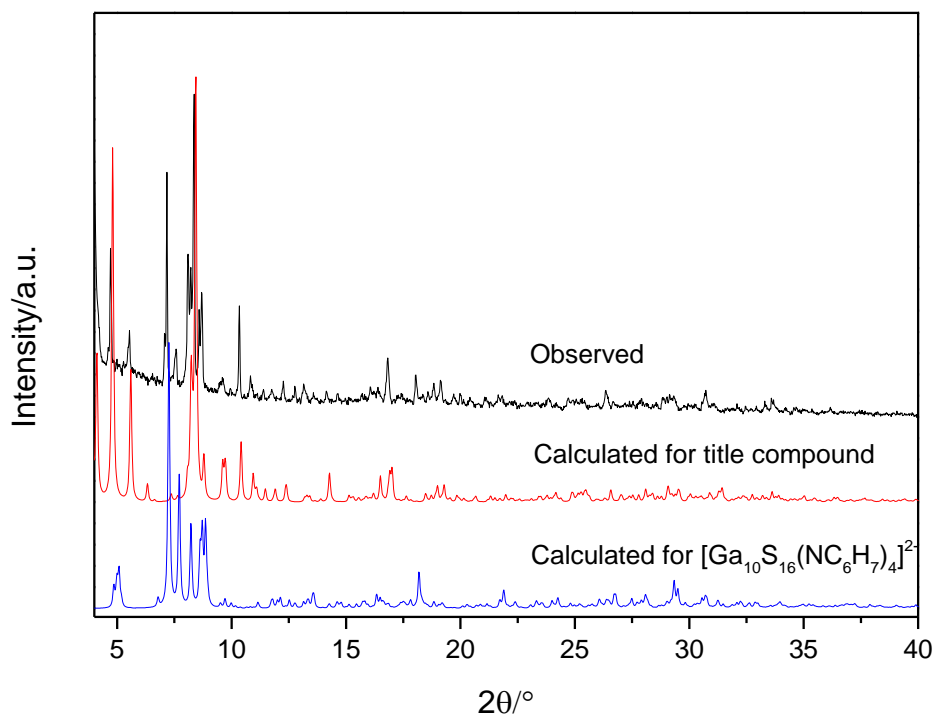
2. *FT-IR:*

Infrared measurements were carried out using a Perkin Elmer Spectrum 100 ATR spectrometer. Data were collected on ground single crystals.

3. *Thermogravimetric analysis*:

Thermogravimetric analysis was performed using a TA Instruments SDT Q600 DSC-TGA on ground, hand-picked crystals, under an atmosphere of air. The sample was heated from room temperature up to 800 °C, using a heating rate of 5 °C min^{-1}, and held at 800 °C for 30 minutes.

4. *UV-Vis diffuse reflectance:*

Diffuse-reflectance measurements were collected using a Perkin Elmer Lambda 35 UV-vis spectrometer. $BaSO_4$ powder was used as a reference, and the absorption data were calculated from the reflectance data using the Kubelka-Munk function.

Figure S1. Powder X-ray data on a ground bulk sample (black line) and calculated powder diffraction pattern for the title compound (red line) and for $[C_{12}H_{10}N_2]_2[C_{12}H_{12}N_2]_2$ $[C_6H_8N]_6[Ga_{10}S_{16}(NC_6H_7)_4]_4$.[1]
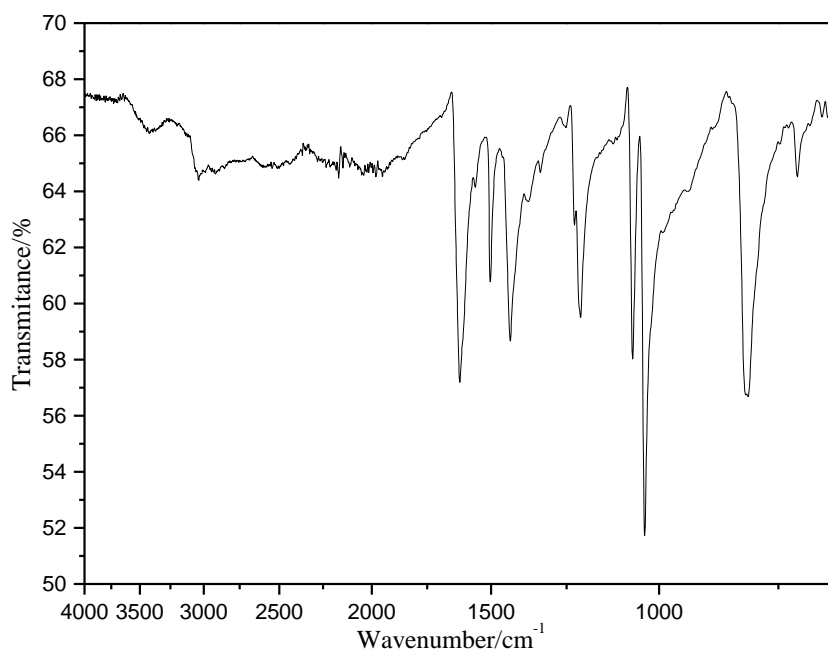
Figure S2. FTIR spectrum collected on ground single-crystals of the title compound.

[1] P. Vaqueiro and M. L. Romero, *Mater. Res. Soc. Symp. Proc.* 2009, 1148E, 1148-PP10-07.