SUPPLEMENTARY INFORMATION

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

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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. BaSO₄ 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}][C_{12}H_{12}N_{2}]_{2} [C_{6}H_{8}N][Ga_{10}S_{16}(NC_{6}H_{7})_{4}]_{4}.\textsuperscript{1}

Figure S2. FTIR spectrum collected on ground single-crystals of the title compound.