Supporting Information

Synthesis of [(CH₃)₄N]₄[Re₆Te₆(CN)₆]

[(CH₃)₄N]₄[Re₆Te₆(CN)₆] was prepared as described previously [Ref. 7]. Re₆Te₁₅ (1.45 g, 0.478 mmol) and KCN (0.559 g, 8.58 mmol) powders were placed in a quartz tube. The tube was sealed under vacuum and heated at 600 °C (ramping rate = 0.2 °C/min) for 48 h and then cooled with cooling rate of 0.3 °C/min. The material was dissolved in methanol and filtered. The red filtrate was evaporated to get the powder of K₄[Re₆Te₆(CN)₆]. Excess amount of NMe₄Cl was added into the aqueous solution of K₄[Re₆Te₆(CN)₆] to exchange the cations. The powder phase of [(CH₃)₄N]₄[Re₆Te₆(CN)₆] was obtained after filtration and washed with cold water.

Physical Measurements

Powder XRD patterns were obtained on a Rigaku X-ray Diffractometer using Cu-K radiation (λ = 1.5418Å) at 40 kV and 30 mA. HR-TEM images were acquired with a JEOL JEM-3010 instrument (300 kV). TG/DTA was carried out on a TGA Q5000 (TA Instruments) under nitrogen. FT-IR spectra were collected at room temperature on a JASCO FT/IR-6100. UV-Vis spectrum was recorded on a JASCO V-550. Re, Te and Mn analyses were performed by ICP-AES (OPTIMA 4300DV, Perkin-Elmer). The source was argon plasma and resolution was better than 0.006 nm at 200 nm. Elemental analyses for C, H, and N were obtained on an EA 1110 elemental analyzer. The EDX results were performed using LEO SUPRA 55 (JEOL) equipped with an EDAX Genesis 2000 X-ray Microanalysis System.
Figure S1. EDX spectrum of the H-Re/Te/Mn. Copper is from the Cu grid.