**Amino Acids as Highly Efficient Modulators for Single Crystals of Zirconium and Hafnium Metal-Organic Frameworks**

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The data comprise experimental synthesis and characterisation of a number of Zr and Hf metal-organic frameworks, specifically using different amino acids as crystallisation promoters. Properties such as stability and porosity are reported.

Data can be accessed by Bruker TopSpin v3.2 NMR software (NMR data) or by OriginPro v8.5 (all other data). Images can be opened by any imaging software.

**Data Collection Protocols:**

All chemicals and solvents were purchased from Alfa Aesar, Fisher Scientific, Fluorochem, Merck, Sigma-Aldrich, Strem Chemicals and VWR and used without further purification.

**Powder X-ray Diffraction Experiments:** PXRD measurements were carried out at 298 K using a PANalytical X’Pert PRO diffractometer (λ (CuKα) = 1.4505 Å) on a mounted bracket sample stage. Data were collected over the range 2Ѳ = 3 – 45 ° or 5 – 45 °. When comparison of the PXRD patterns was required, identical step size / scan speed parameters were used. (University of Glasgow)

**Microwave Synthesis:** Microwave reactions were performed in 35 ml pressure vials using a CEM Discover SP microwave, equipped with an Explorer 12 Hybrid autosampler. The power was allowed to fluctuate to maintain a constant temperature of 100 °C throughout the reaction. (University of Glasgow)

**Single Crystal X-ray Diffraction:** Data for **Hf-L6** were collected on Agilent Technologies SuperNova diffractometer using CuKα radiation, and data for **Hf-L7** were collected on a Bruker Apex II (λ (MoKα = 0.71073 Å) diffractometer (\*University of Edinburgh).

**Scanning Electron Microscopy**: Powder samples were deposited onto conductive carbon tabs mounted on an aluminium stub and coated with Pd for 150 seconds using a Polaron SC7640 sputter coater. The prepared samples were transferred to and imaged using a Philips XL30 ESEM tungsten filament electron microscope, operating at an acceleration voltage of 20 Kv. (University of Glasgow)

**Gas Uptake:** N2 adsorption isotherms were carried out at 77 K on a Quantachrome Autosorb iQ gas sorption analyser. Samples were degassed under vacuum at 120 °C for 20 hours using the internal turbo pump. BET surface areas were calculated from the isotherms using the Micropore BET Assistant and pore-size distribution analysis was carried out using QSDFT (N2 on carbon at 77 K, slit/cylindrical pore model) both in the Quantachrome ASiQwin operating software. (University of Glasgow)

**Thermal Gravimetric Analysis (TGA):** Measurements were carried out using a TA Instruments Q500 Thermogravimetric Analyser. Measurements were collected from room temperature to 1000 °C with a heating rate of 10 °C / min under an N2 atmosphere. (University of Glasgow)

**Nuclear Magnetic Resonance (NMR):** NMR spectra were recorded on either a Bruker AVIII 400 MHz spectrometer or a Bruker AVI 500 MHz Spectrometer and referenced to residual solvent peaks. (University of Glasgow)