Supplementary Material (ESI) for Chemical Communications

Isoreticular synthesis and modification of frameworks with the UiO-66 topology

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SUPPORTING INFORMATION

Experimental Methods

General

Starting materials and solvents were purchased and used without further purification from commercial suppliers (Sigma-Aldrich, Alfa Aesar, EMD, TCI, Cambridge Isotope Laboratories, Inc., and others).

Characterization of UiO-66 functionalized frameworks

^{1}H NMR Digestion and Analysis. Approximately 10 mg of microcrystalline UiO-66 was digested by sonication in 570 μL of d_6-DMSO and 30 μL of HF. After complete dissolution of the material, the solution was used to collect a ^{1}H NMR spectrum. ^{1}H NMR spectra were recorded on a JEOL ECA spectrometer (500 MHz).

ESI-MS Analysis. Electrospray ionization mass spectrometry (ESI-MS) was performed using a ThermoFinnigan LCQ-DECA mass spectrometer and the data were analyzed using the Xcalibur software suite in negative ion mode. UiO-66 samples were digested by sonicating the materials in a mixture of 10 μL of HF and 1.0 mL of CH_3CN.

Thermal Gravimetric Analysis. Approximately 10-20 mg of modified BET analyzed UiO-66 samples were used for TGA measurements. Samples were analyzed under a stream of dinitrogen using a TA Instrument Q600 SDT running from room temperature to 800 °C with a scan rate of 5 °C/min.

PXRD Analysis. PXRD data were collected at ambient temperature on a Bruker Advance D8 diffractometer at 40 kV, 40 mA for *K*α (λ = 1.5418 Å) with a scan speed of 3°/min, a step size of 0.02° in 2θ, and a 2θ range of 5-45°. Approximately 15 mg of microcrystalline UiO-66 samples were dried at 150 °C for at least 2 h before PXRD

analysis. The experimental backgrounds were corrected using the Jade 5.0 software package.

FT-IR Analysis. Approximately 5-10 mg of modified UiO-66 was dried at 150 °C for at least 2 h before FT-IR analysis. FT-IR spectra were collected using a Bruker ALPHA-P FT-IR spectrometer with a diamond ATR.

BET Surface Area Analysis. BET surface area (m^2/g) measurements were collected at 77 K using dinitrogen on an ASAP 2020 using the volumetric technique. Approximately 40-60 mg of activated UiO-66 samples were evacuated on a vacuum line for 5-18 h. The sample was then transferred to a preweighed sample tube and degassed at 105 °C for approximately 24 h or until the outgas rate was <5 μmHg. The sample tube was re-weighed to obtain a consistent mass for the degassed UiO-66 samples.

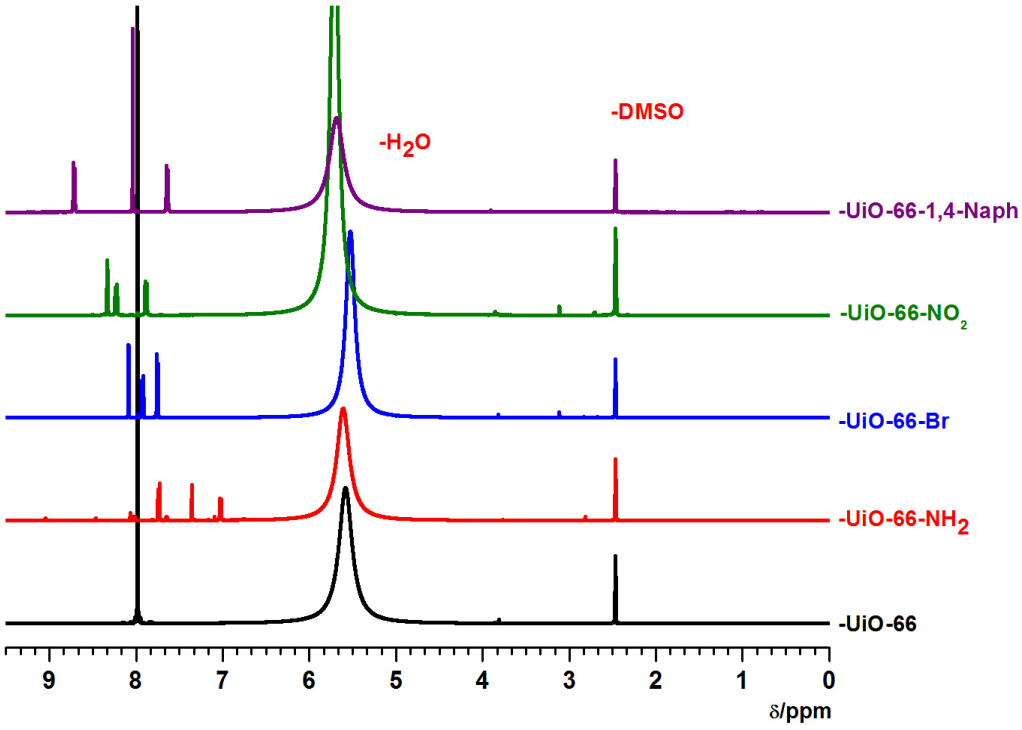

Figure S1. ^{1}HNMR Spectra of digested UiO-66 and UiO-66 functionalized samples.

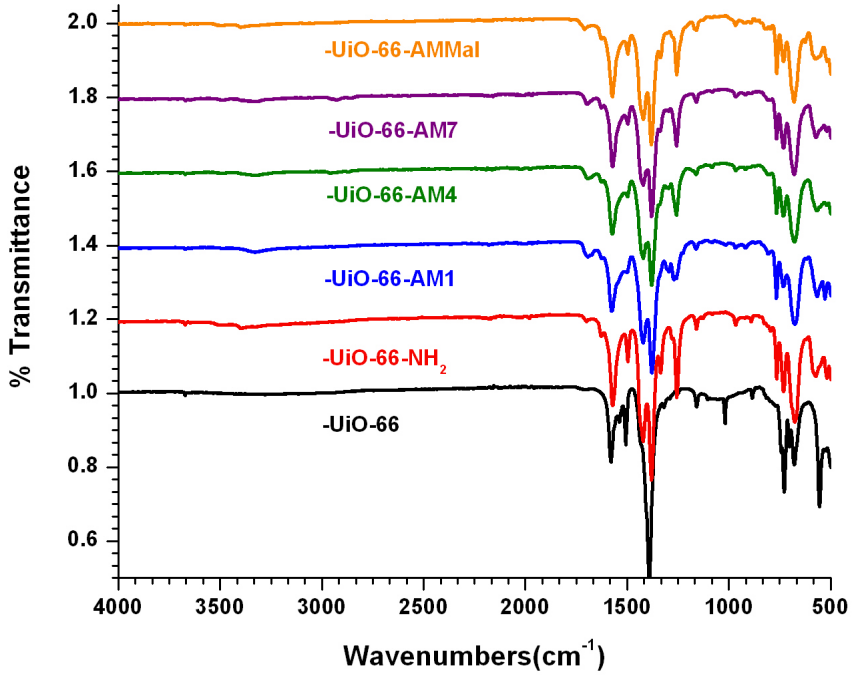

Figure S2. FTIR spectra of modified UiO-66-NH_2 samples.

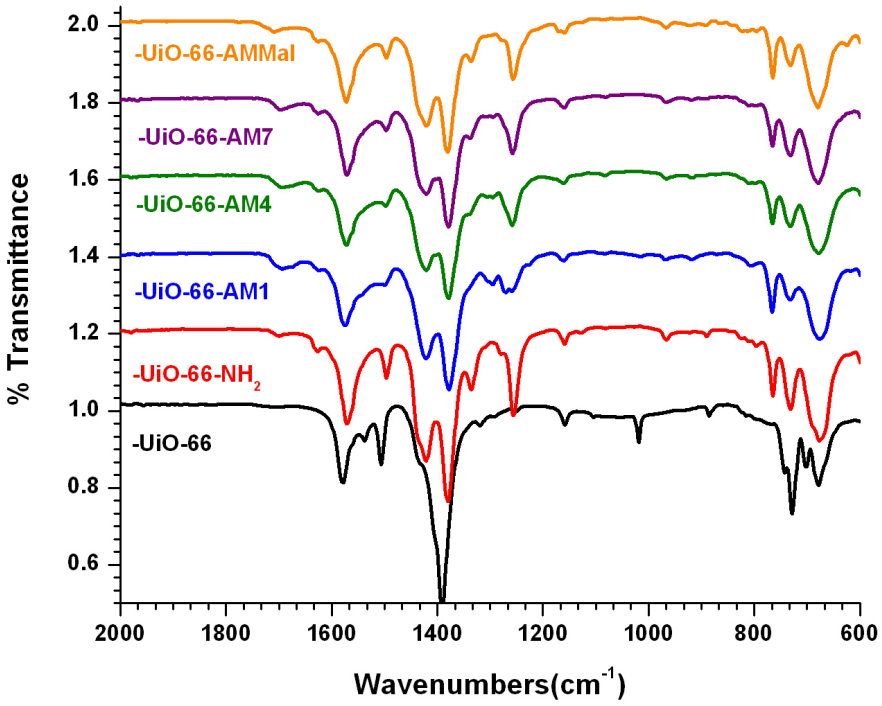

Figure S3. Magnification of the FTIR spectra of modified UiO-66-NH_2 samples.

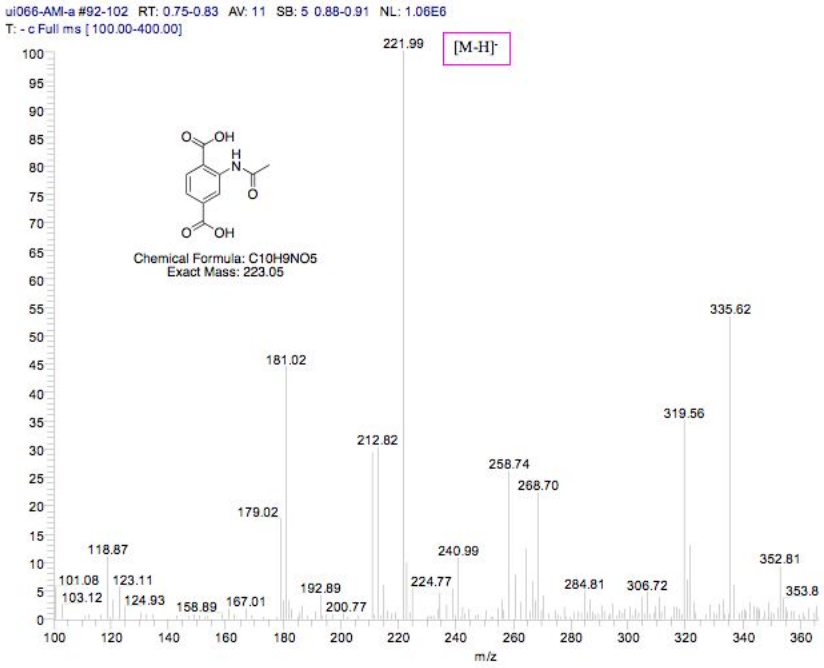

Figure S4. ESI-MS of digested UiO-66-AM1.

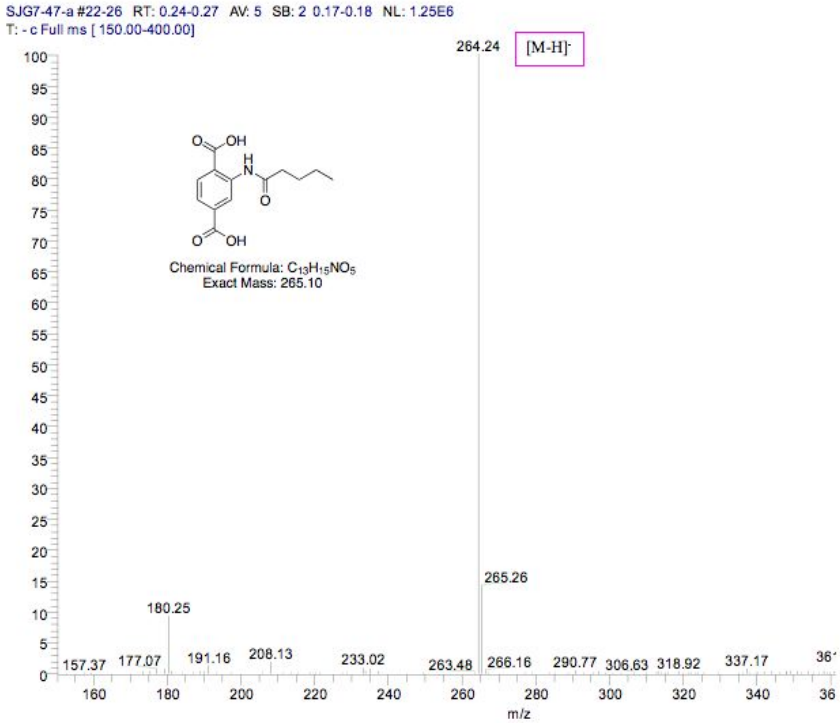

Figure S5. ESI-MS of digested UiO-66-AM4.

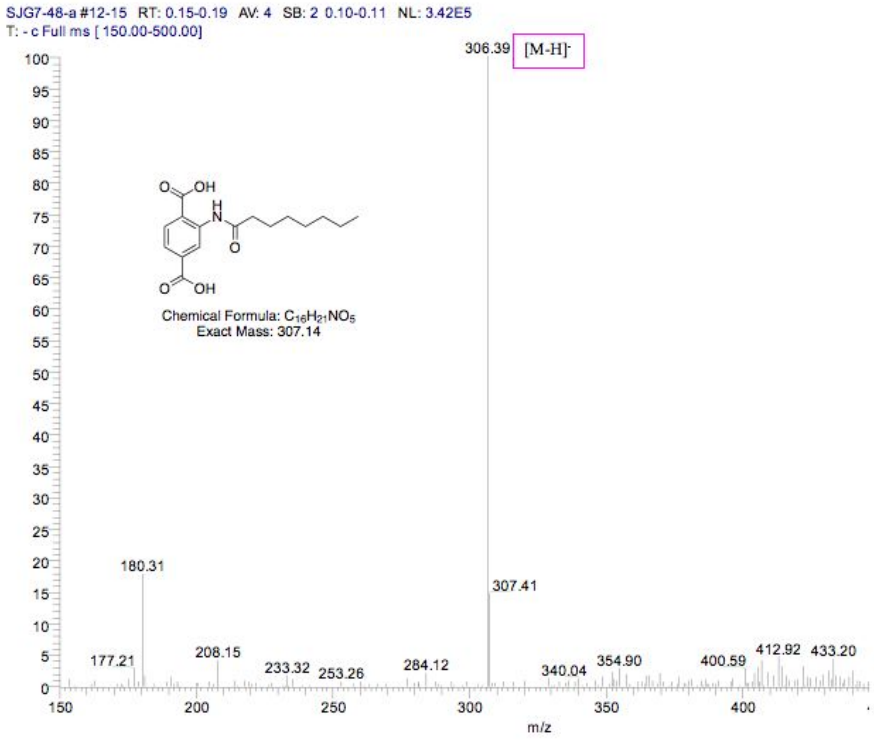

Figure S6. ESI-MS of digested UiO-66-AM7.

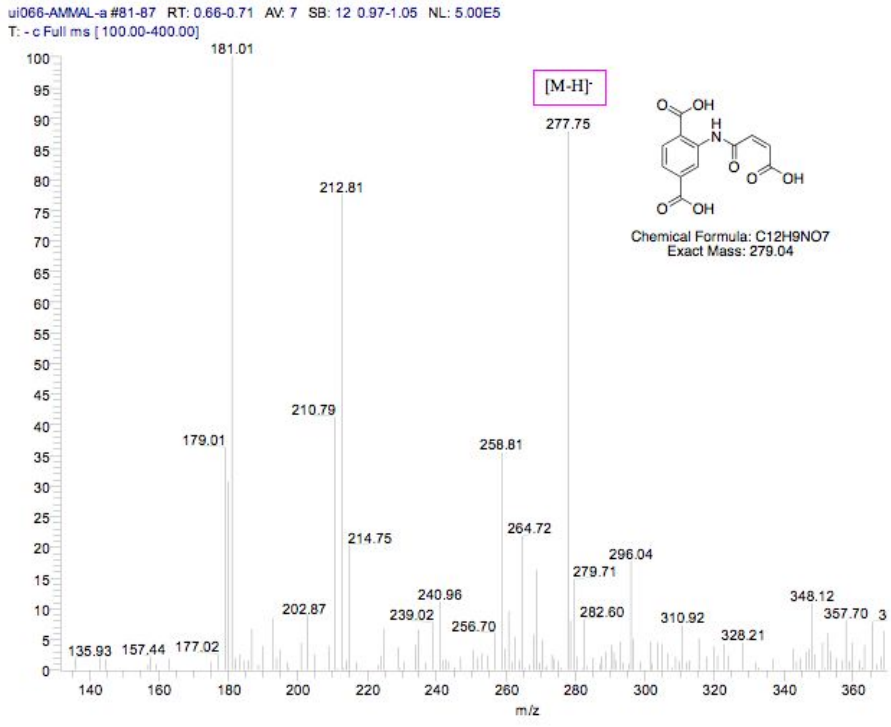

Figure S7. ESI-MS of digested UiO-66-AMMal.