Electronic Supplementary Information

A zirconium metal-organic framework with exceptionally high volumetric surface area

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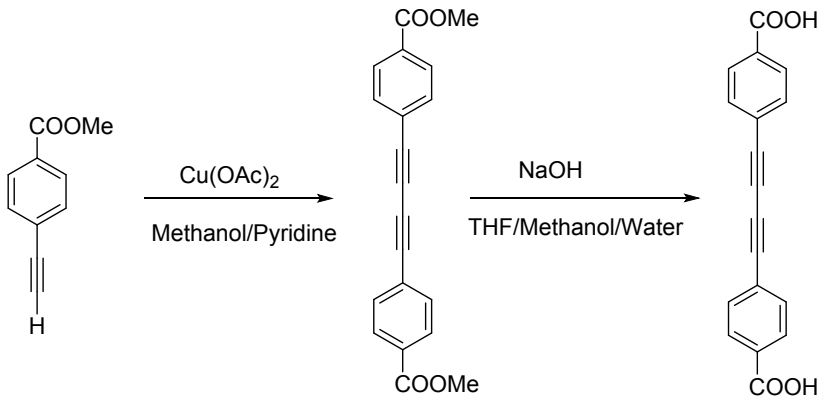

Figure S1. Synthetic procedure of diacid of organic linker

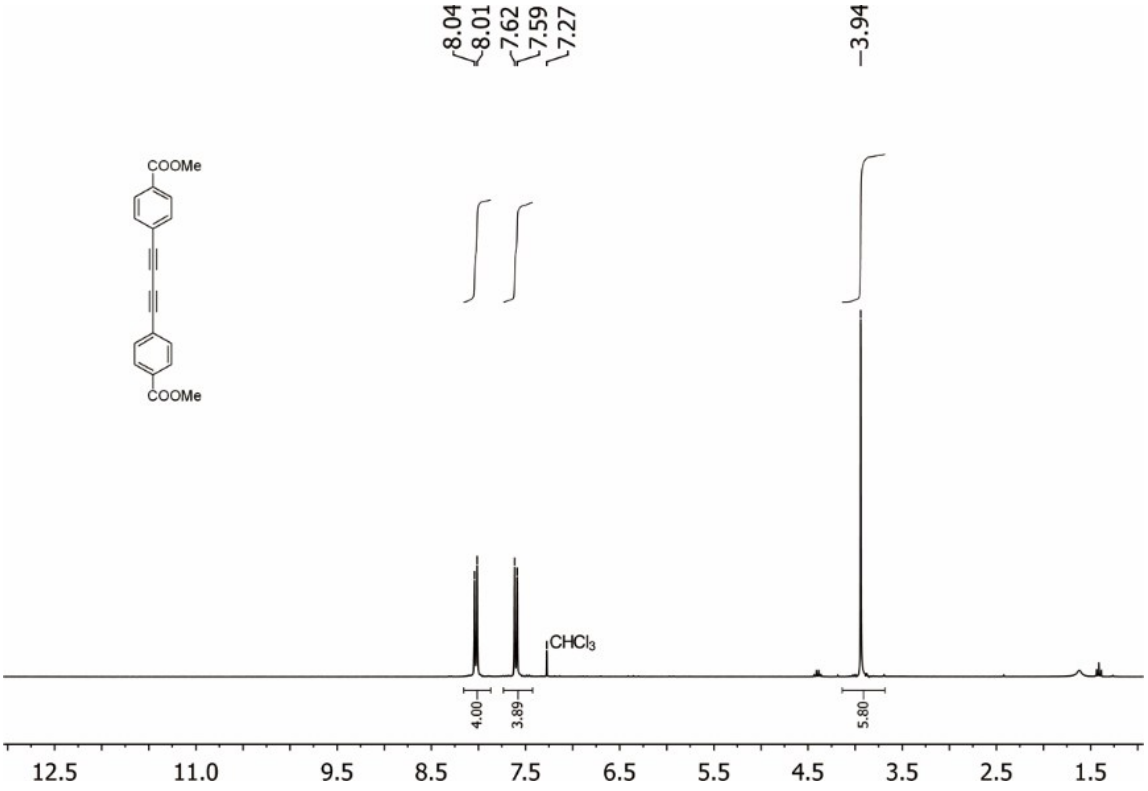

Figure S2. ^{1}H-NMR (300 MHz, $CDCl_3$) for methyl 4,4'-(buta-1,3-diyne-1,4-diyl)dibenzoate

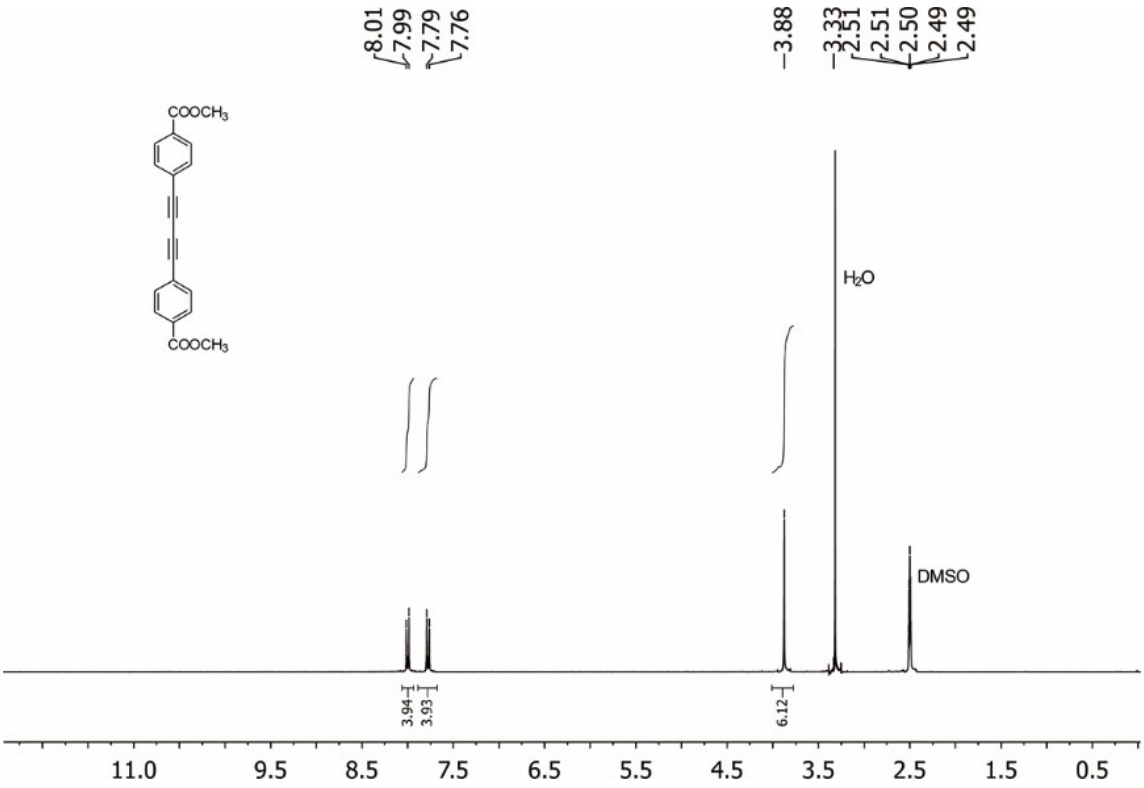

Figure S3. ^{1}H-NMR (300 MHz, DMSO-d_6) for methyl 4,4'-(buta-1,3-diyne-1,4-diyl)dibenzoate

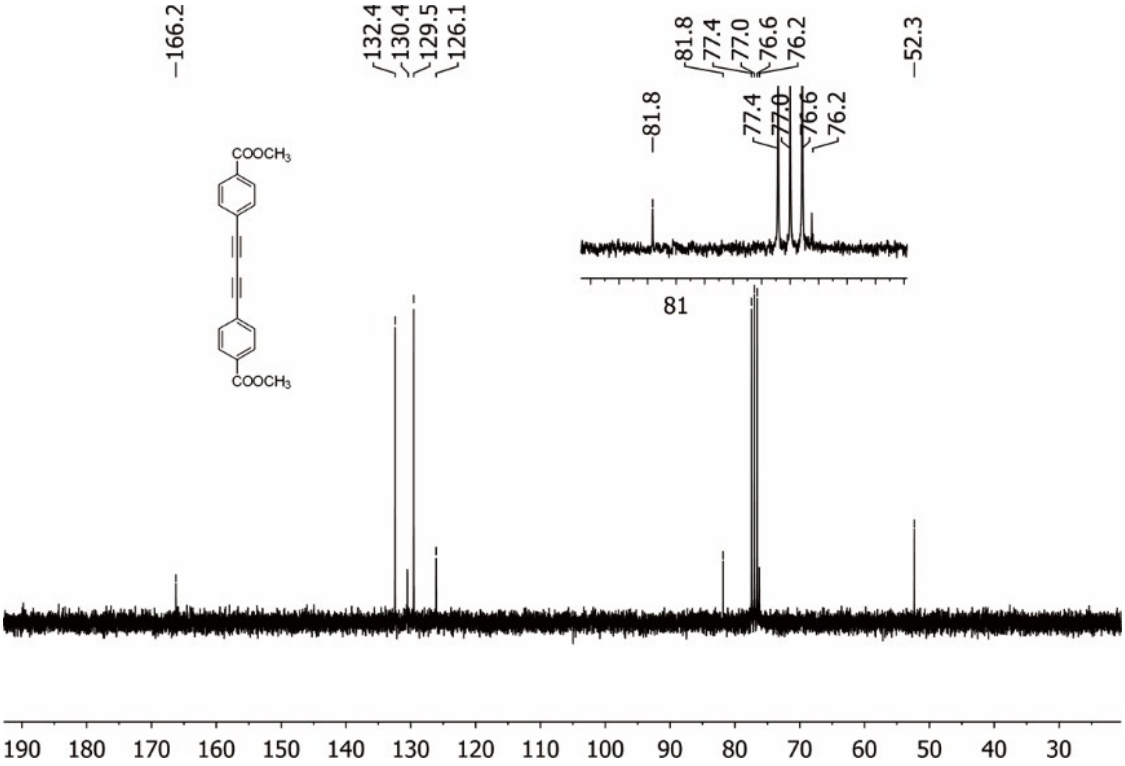

Figure S4. ^{13}C-NMR (75 MHz, $CDCl_3$) for methyl 4,4'-(buta-1,3-diyne-1,4-diyl)dibenzoate

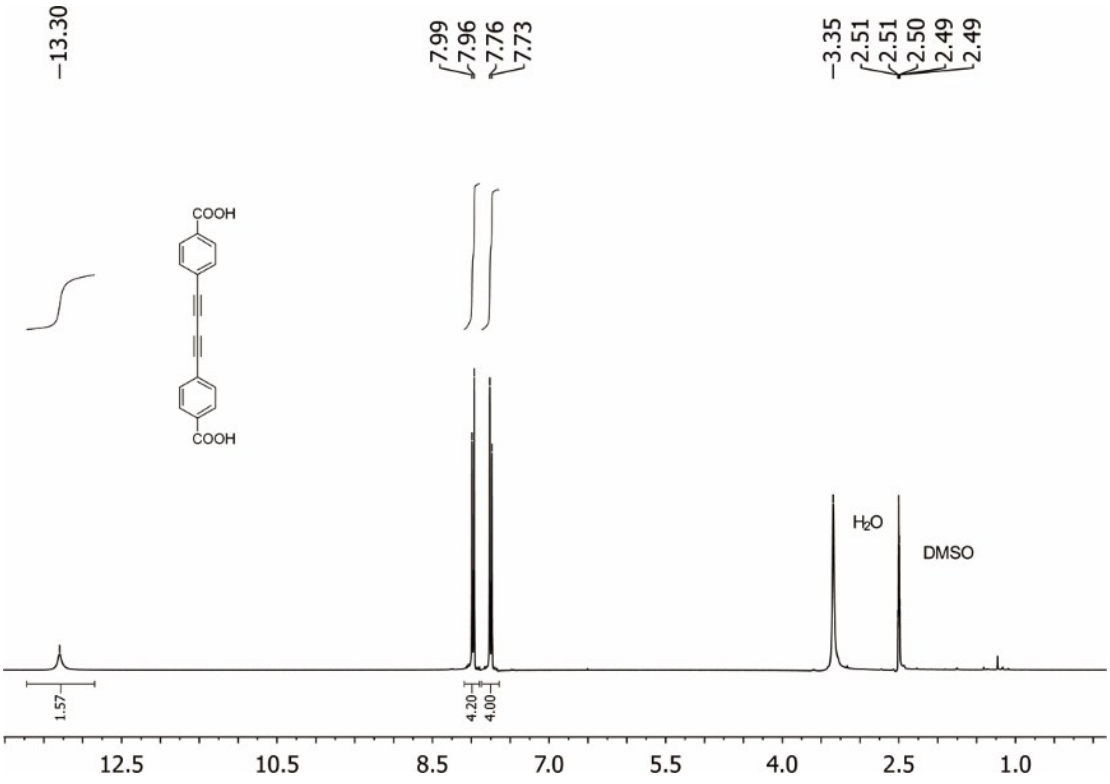

Figure S5. ^{1}H-NMR (300 MHz, DMSO-d_6) for 4,4'-(buta-1,3-diyne-1,4-diyl)dibenzoic acid

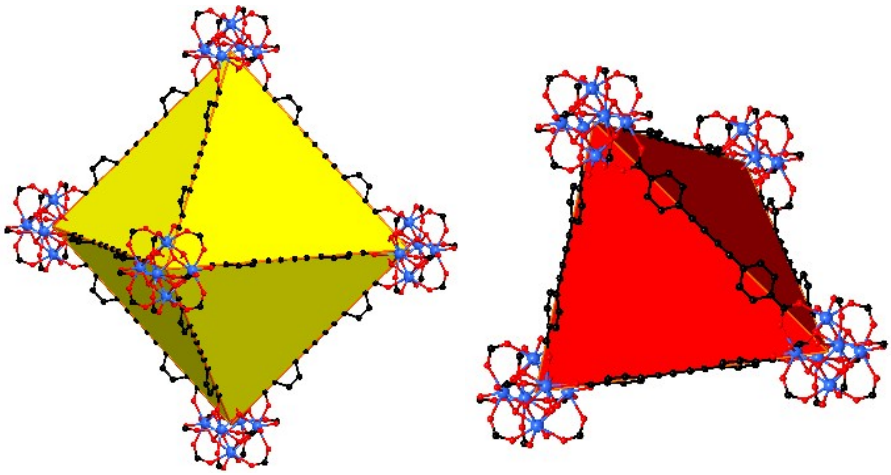

Figure S6. Two polyhedral in PCN-111 structure, octahedron (yellow) and tetrahedron (red).

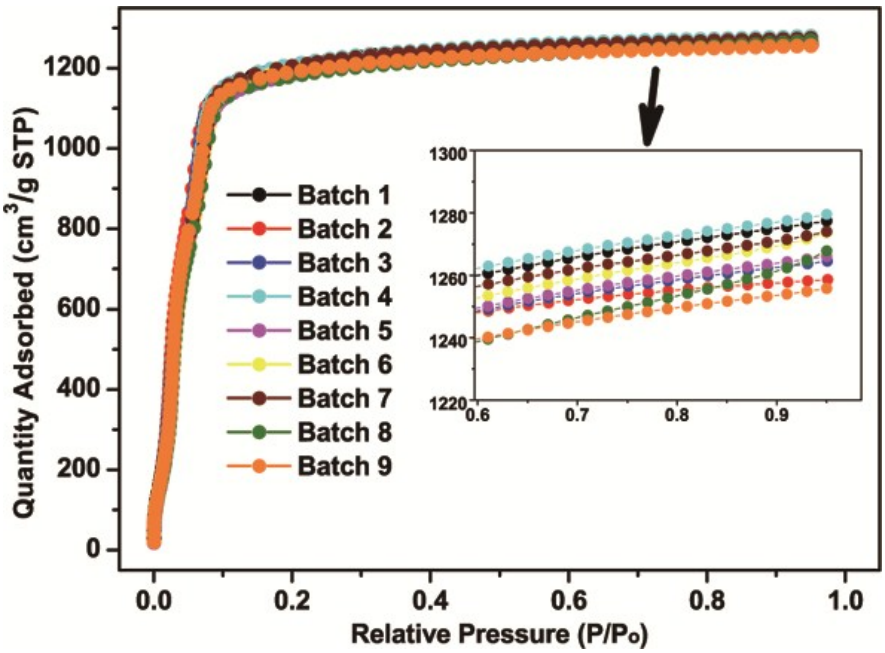

Figure S7. N_2 adsorption isotherms at 77 K for nine batches of PCN-111 samples; sample amount varied between 50 mg and 150 mg, inset: a zoom at high relative pressure.

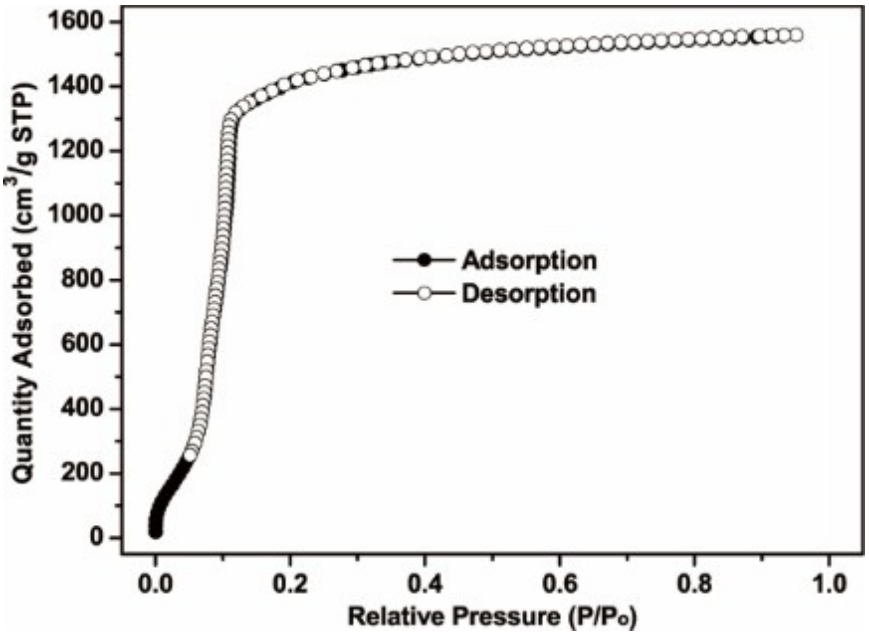

Figure S8. Ar sorption isotherms for PCN-111 at 87 K, adsorption (●)/desorption (○).

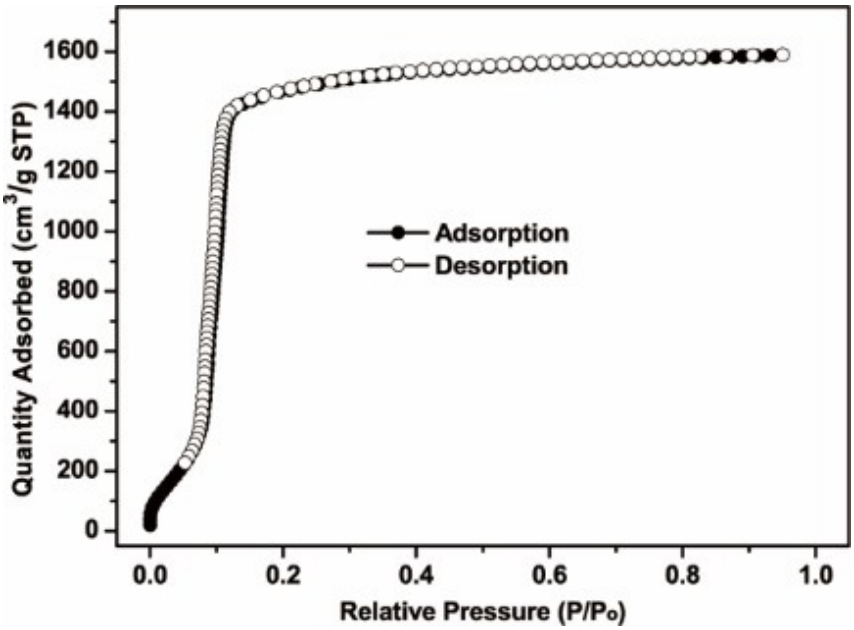

Figure S9. O_2 sorption isotherms for PCN-111 at 87 K, adsorption (●)/desorption (○).

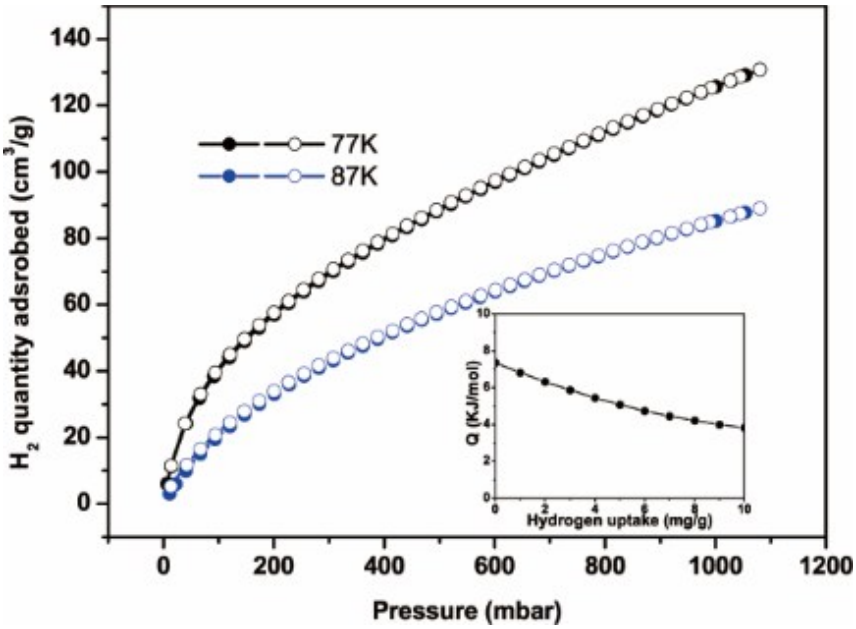

Figure S10. H_2 sorption isotherms for PCN-111 at 77 K (black) and 87 K (blue); inset: H_2 heat of adsorption (kJ/mol) vs. hydrogen uptake (mg/g).

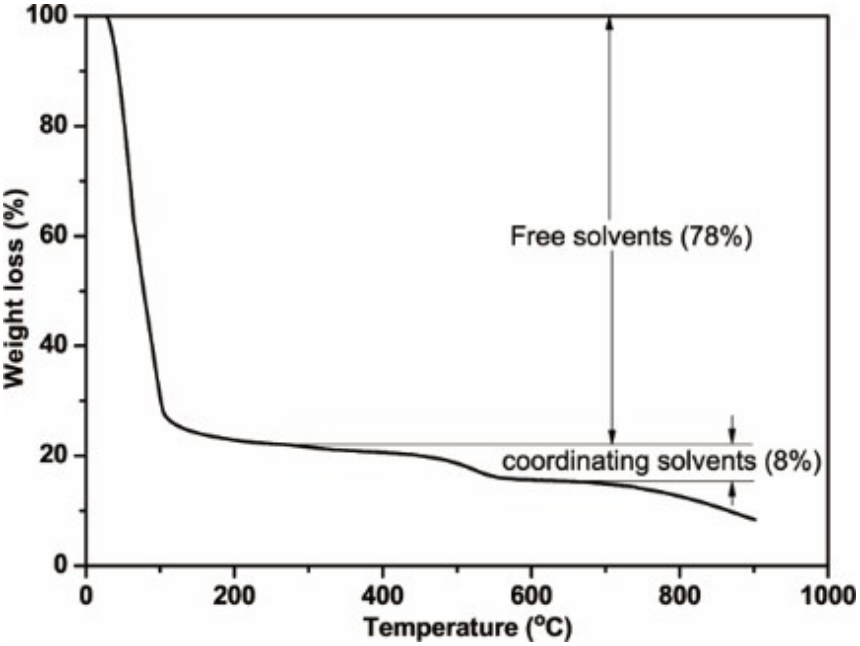

Figure S11. Thermogravimetric analysis of PCN-111; crystals were scooped out of DMF solution and dried on a paper towel for *ca.* 5 min before loaded for measurement.

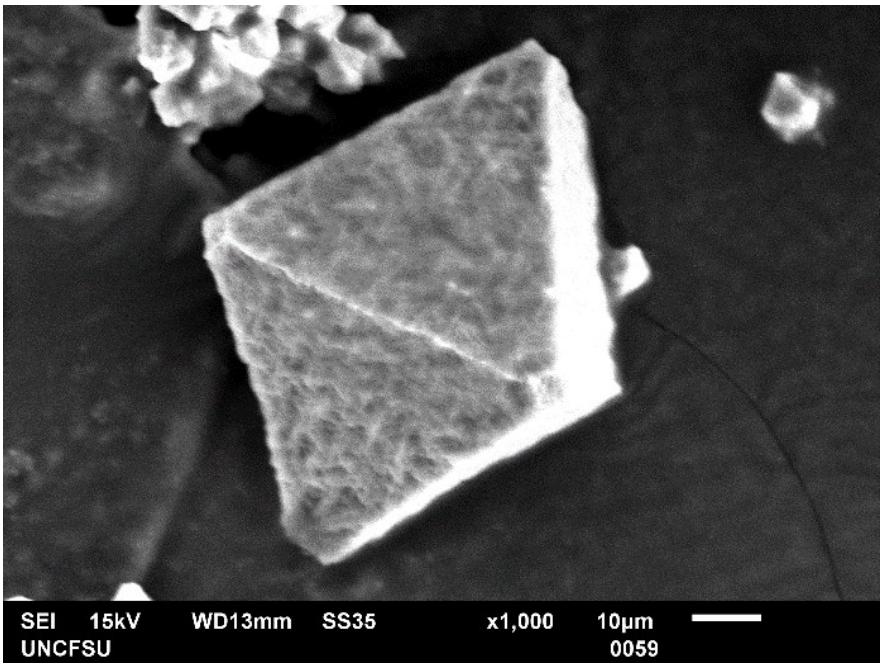

Figure S12. SEM imaging of octahedral shaped PCN-111 crystal.

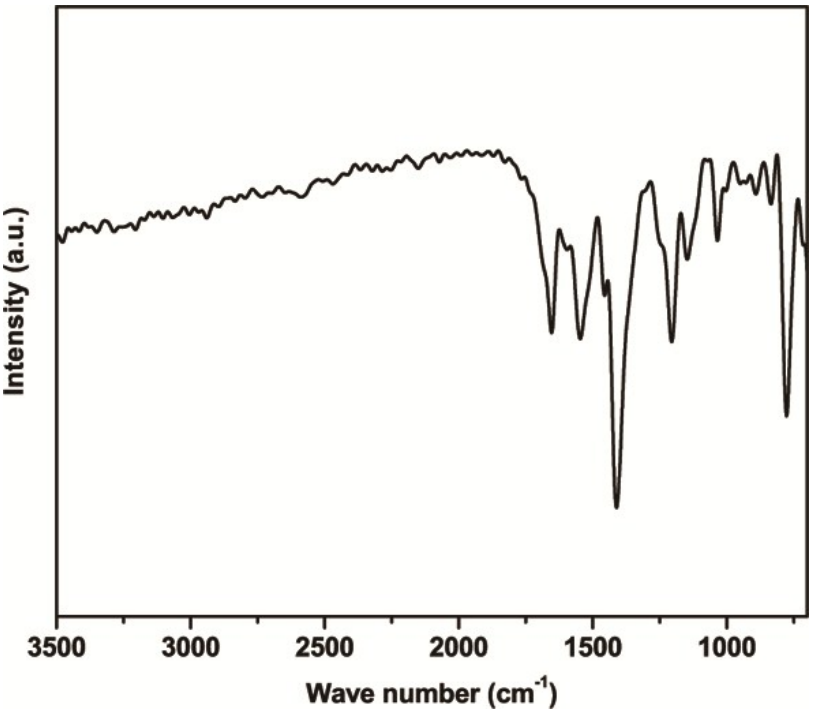

Figure S13. FT-IR spectrum of PCN-111 after solvent exchange and dried in air.