Supporting Information

Rapid Microwave Synthesis and Purification of Porous Covalent Organic Frameworks

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FT-IR Analysis: Samples were prepared as a compressed IR-transparent KBr disc for analysis and scanned in transmission mode by a Bruker Optics Tensor 27 FT-IR spectrophotometer

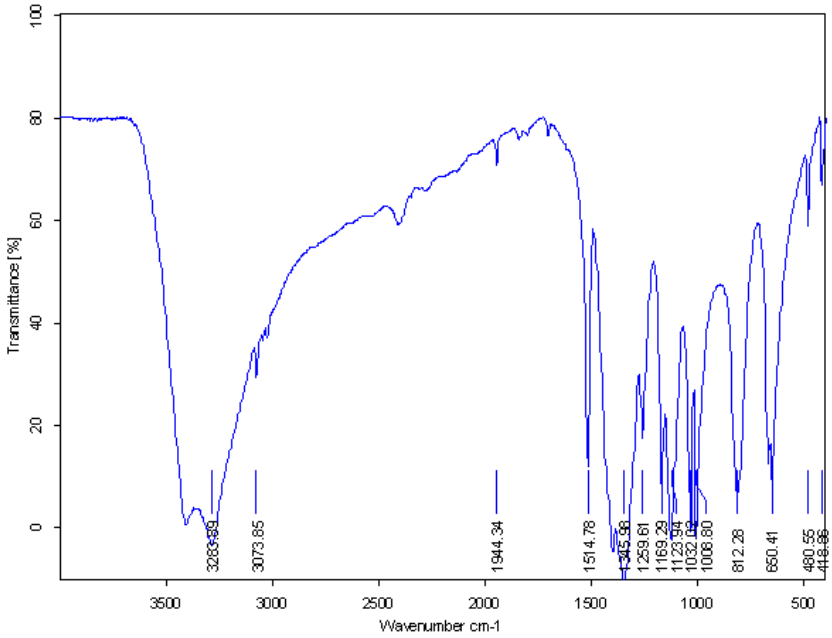

Figure S1. FTIR of benzene diboronic acid (BDBA)

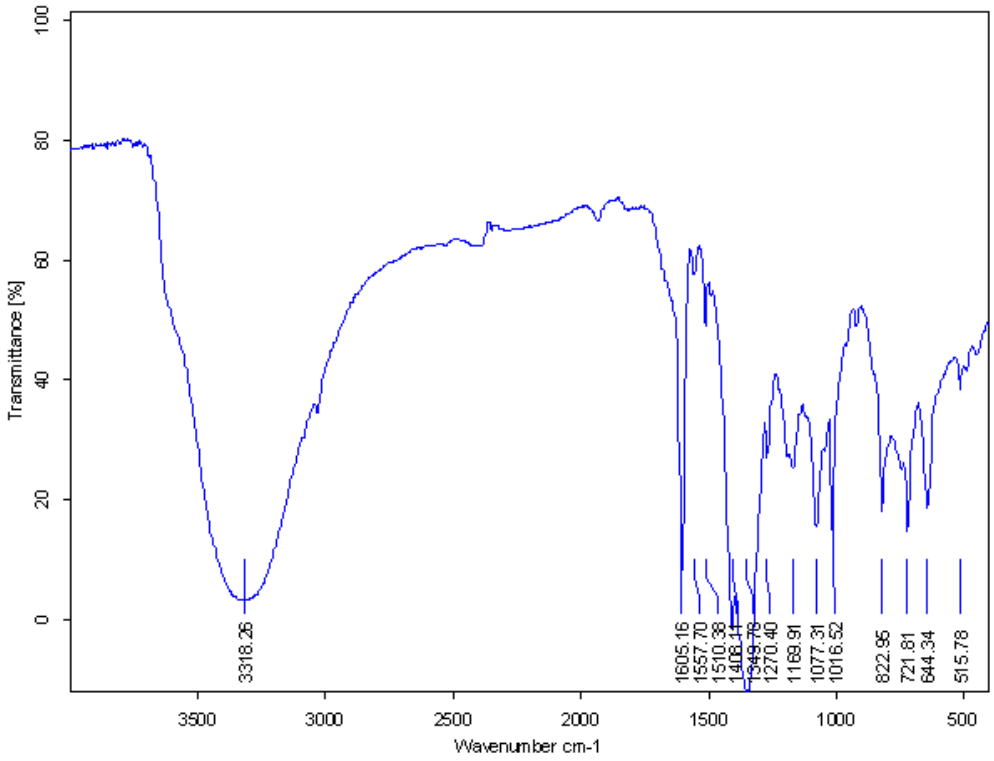

Figure S2. FTIR of tetraborophenylmethane (TBPM)

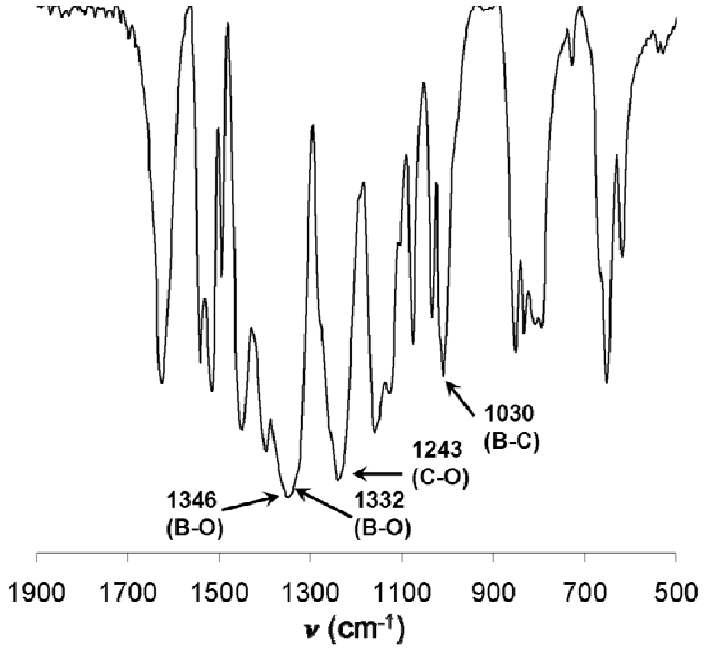

Figure S3. FTIR of COF-5 synthesized in sealed vessel

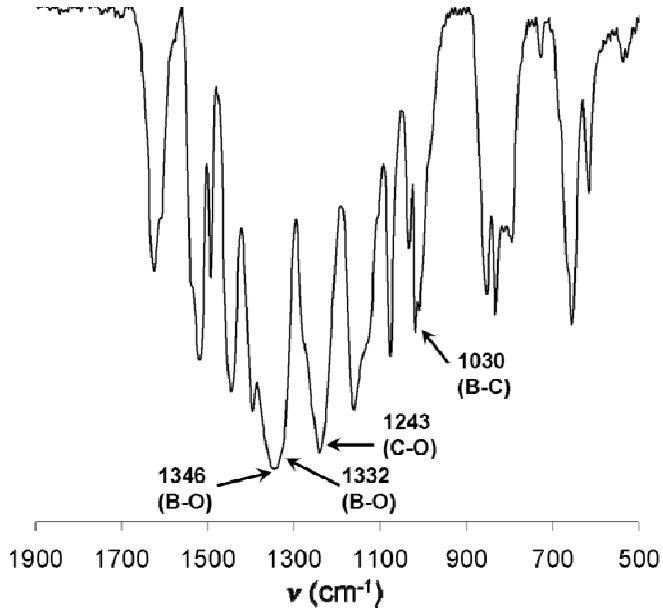

Figure S4. FTIR of COF-5 synthesized in open vessel

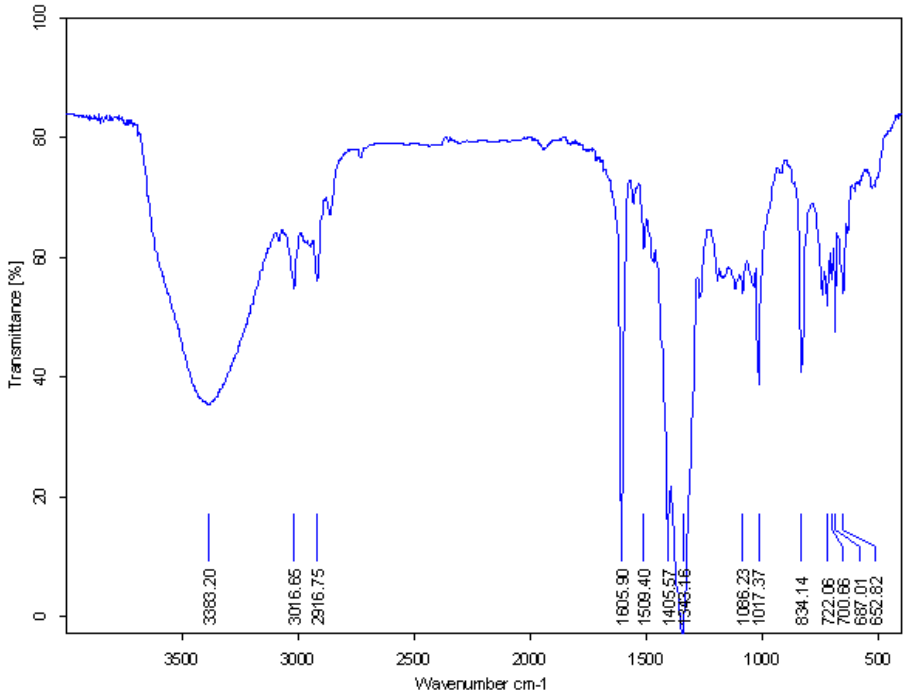

Figure S5. FTIR of COF 102

FE-SEM Analysis: All samples were mounted onto an M3 Hitachi 15mm stub using silver paste and then coated with platinum film to avoid charge effects, which would reduce image quality during electron microscopy operation. The samples were observed using a mix of upper and lower secondary electron detectors of a Hitachi S4800 Type II Cold Field Emission Scanning Electron Microscope (FE-SEM) operating at an acceleration voltage of 3 kV, working distance of 8.0 mm.

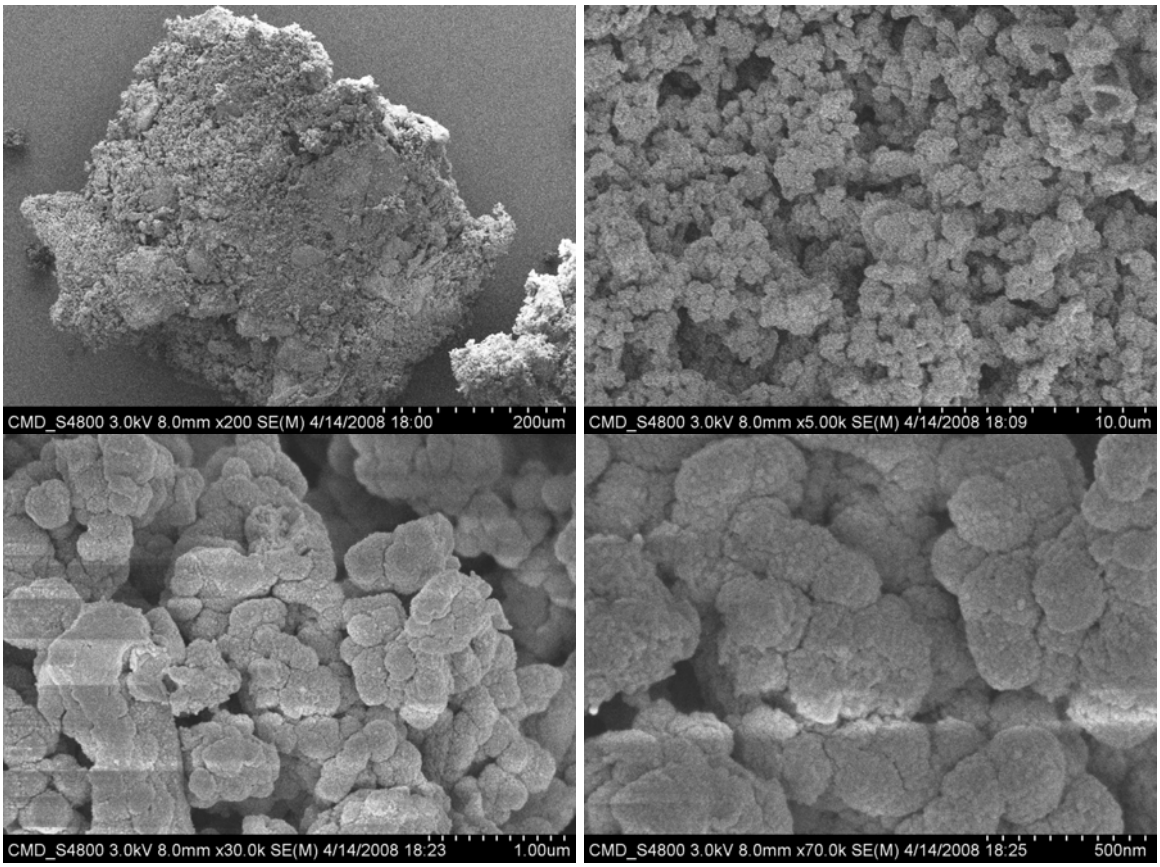

Figure S6. Scanning electron micrograph of COF-5 synthesized in a sealed microwave tube

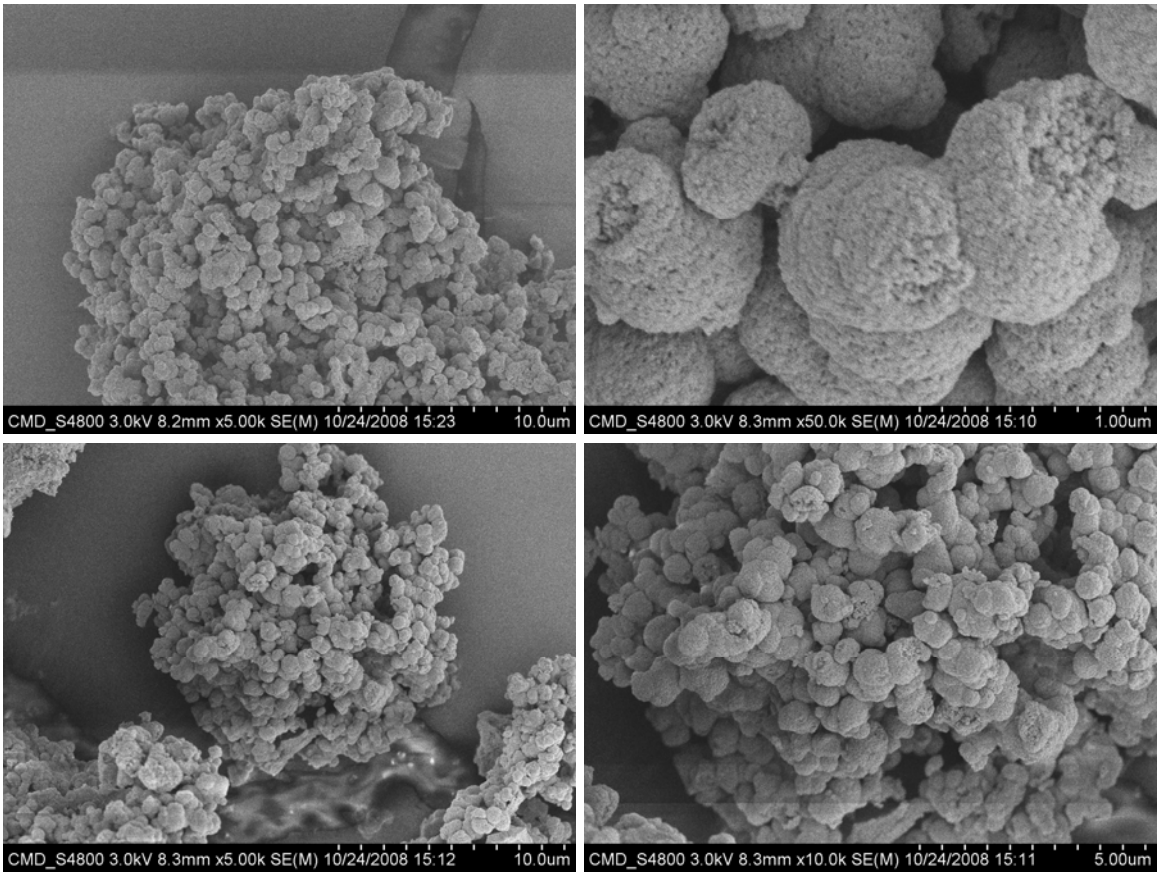

Figure S7. Scanning electron micrograph of COF-102 synthesized in a sealed microwave tube

Powder XRD Analysis: Samples were prepared as a loose powder on a zero background holder and measured in Bragg-Brantano geometry PANalytical MPD diffractometer**.**

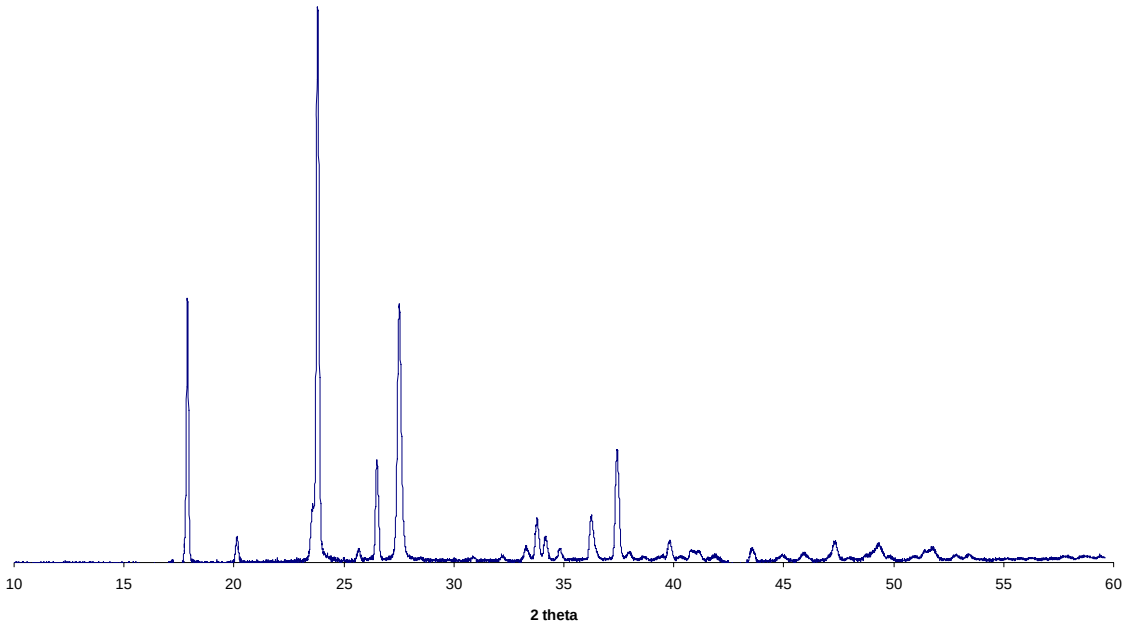

Figure S8. Powder X-ray diffraction pattern for benzene diboronic acid (BDBA)

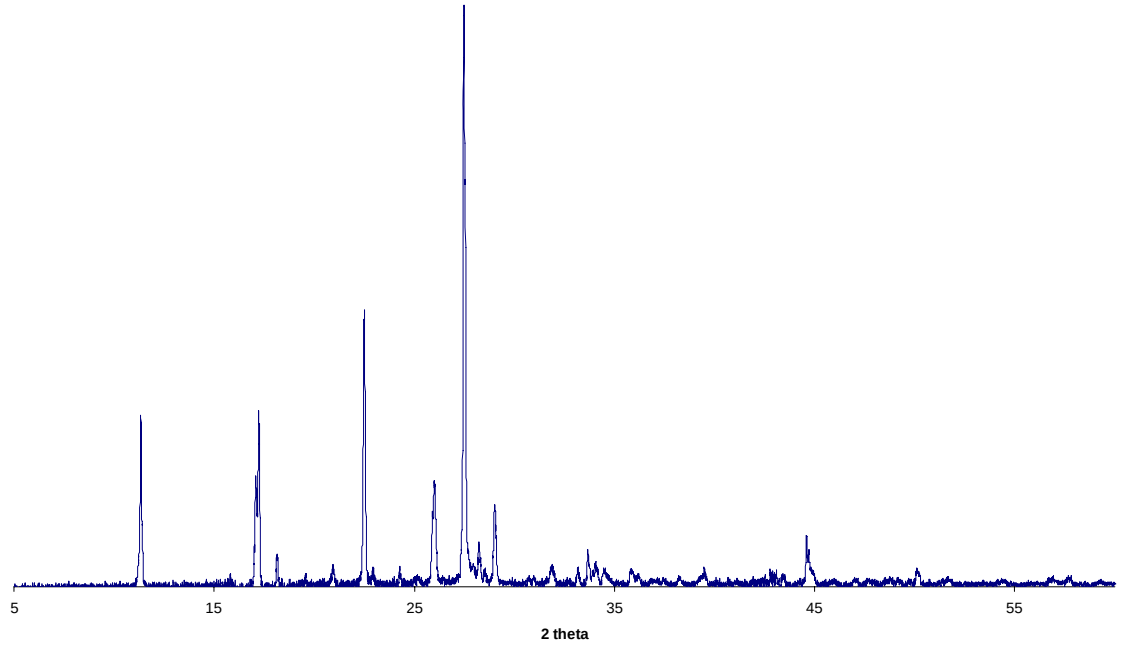

Figure S9. Powder X-ray diffraction pattern for hexahydroxytriphenylene (HHTP)

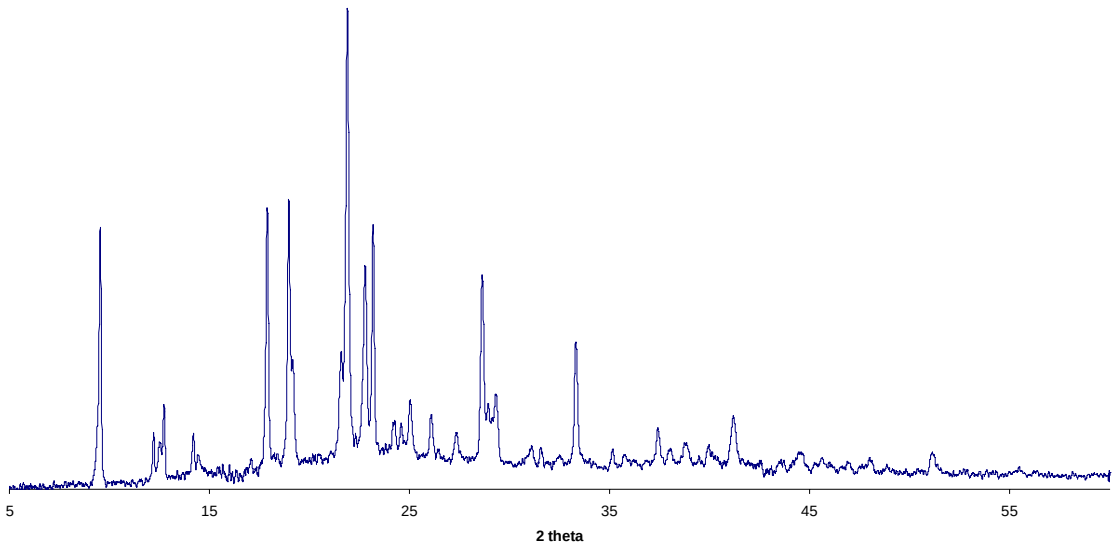

Figure S10. Powder X-ray diffraction pattern for tetraborophenylmethane (TBPM)

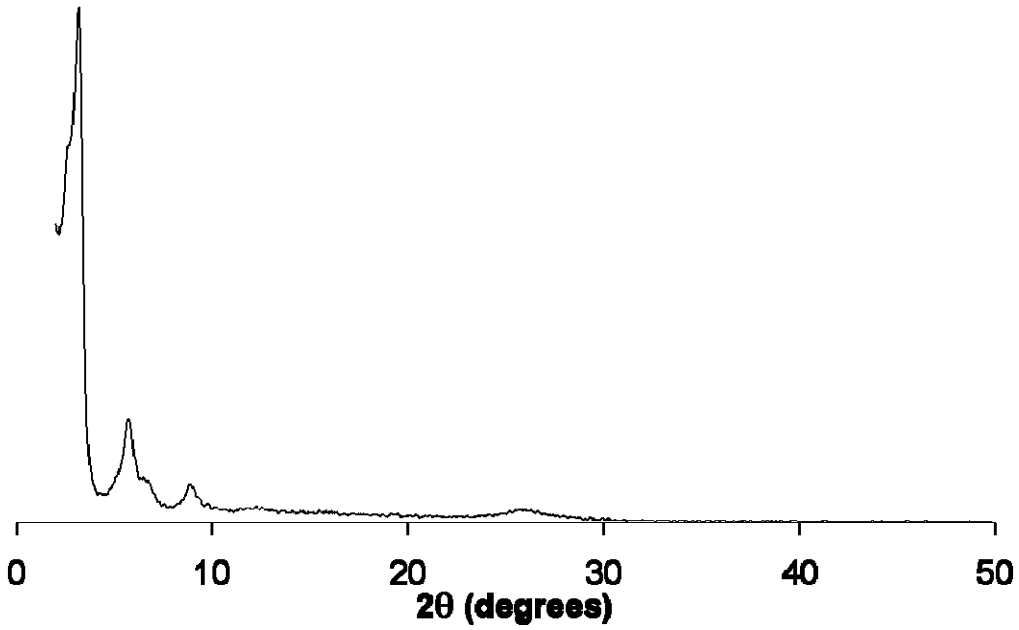

Figure S11. Powder X-ray diffraction pattern for COF-5 synthesized in sealed vessel

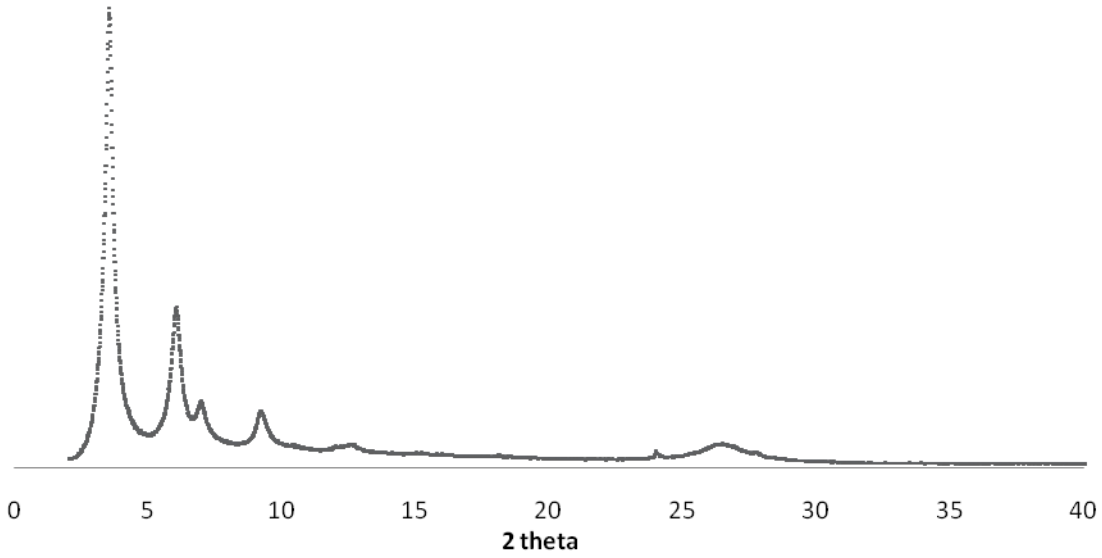

Figure S12. Powder X-ray diffraction pattern for COF-5 synthesized in open vessel

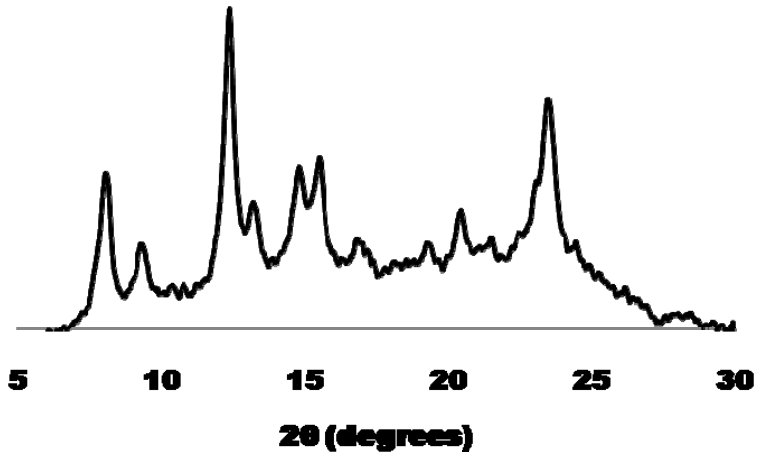

Figure S13. Powder X-ray diffraction pattern for COF-102 synthesized in sealed vessel

Gas Adsorption Analysis: The samples were dried in a vacuum desiccator (24 h) and then transferred to a standard gas sorption sample holder for outgassing at 90 °C under dynamic vacuum (10^{-5} bar). High levels of porosity were confirmed by measuring the N_2 gas adsorption / desorption isotherm for the desolvated material at 77.3 K using a Micromeritics ASAP 2020 sorption analyzer.

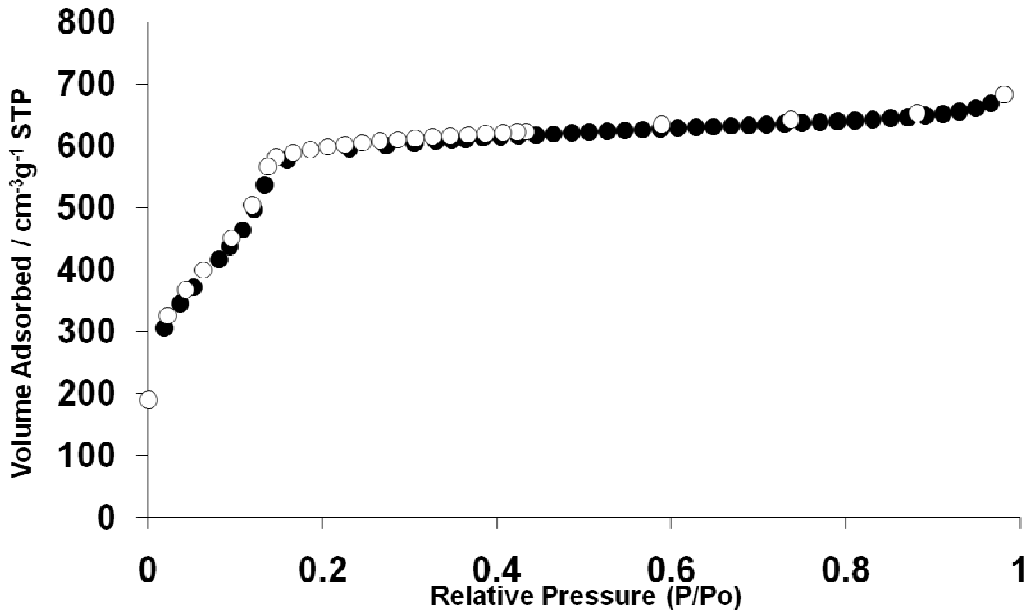

Figure S14. N_2 gas adsorption isotherms for COF-5 (sealed vessel) measured at 77.3 K (adsorption = filled symbols)

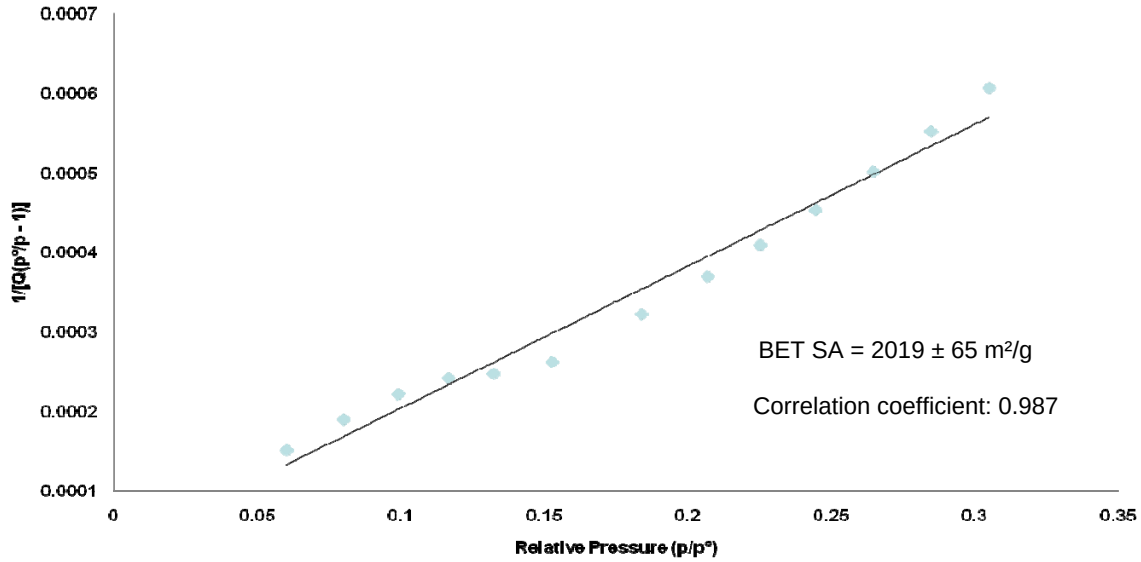

Figure S15. BET surface area plot for COF 5 (sealed vessel). Surface area shown was calculated over relative pressure range P/P_o = 0.05–0.1

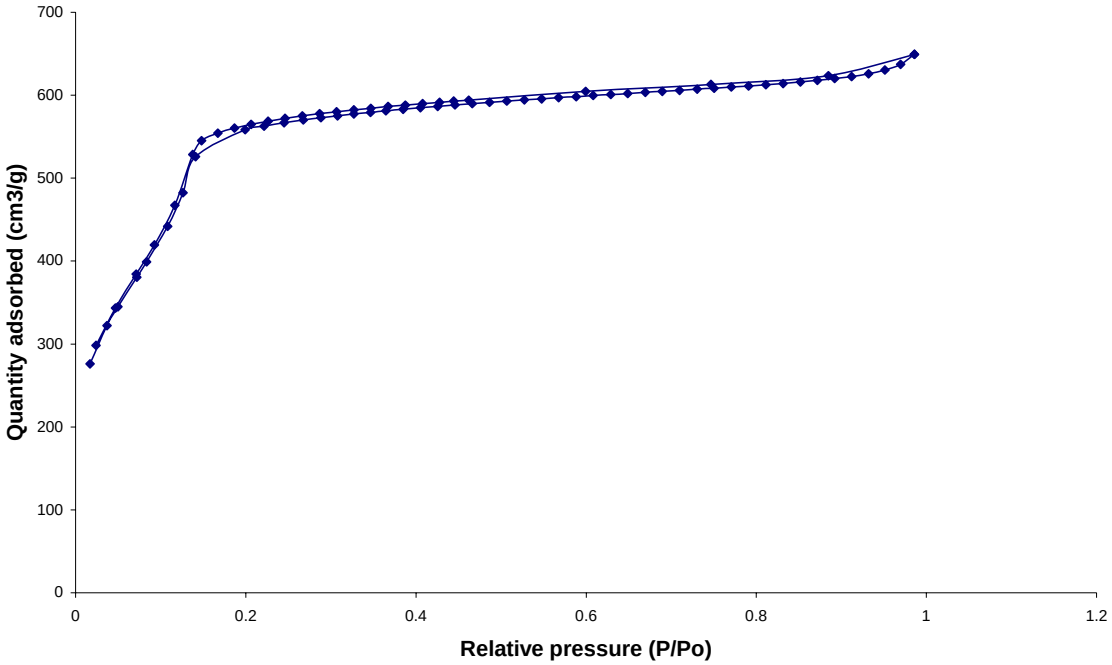

Figure S16. N_2 gas adsorption / desorption isotherms for COF-5 (open vessel) measured at 77.3 K

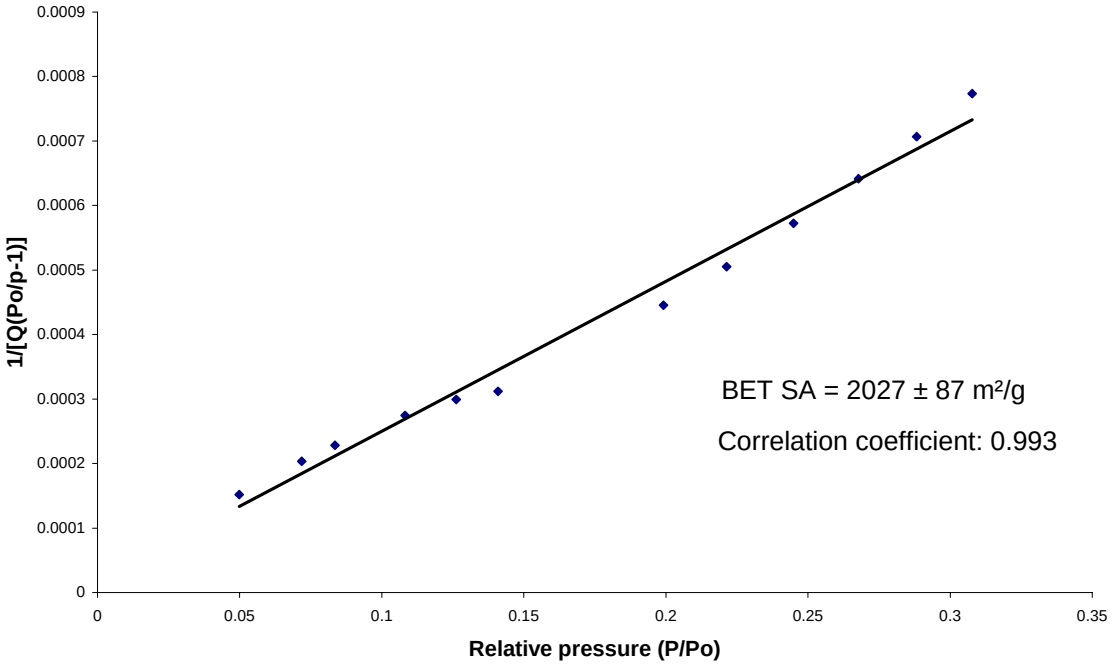

Figure S17. BET surface area plot for COF 5 (open vessel). Surface area shown was calculated over pressure range P/P_o = 0.05–0.1

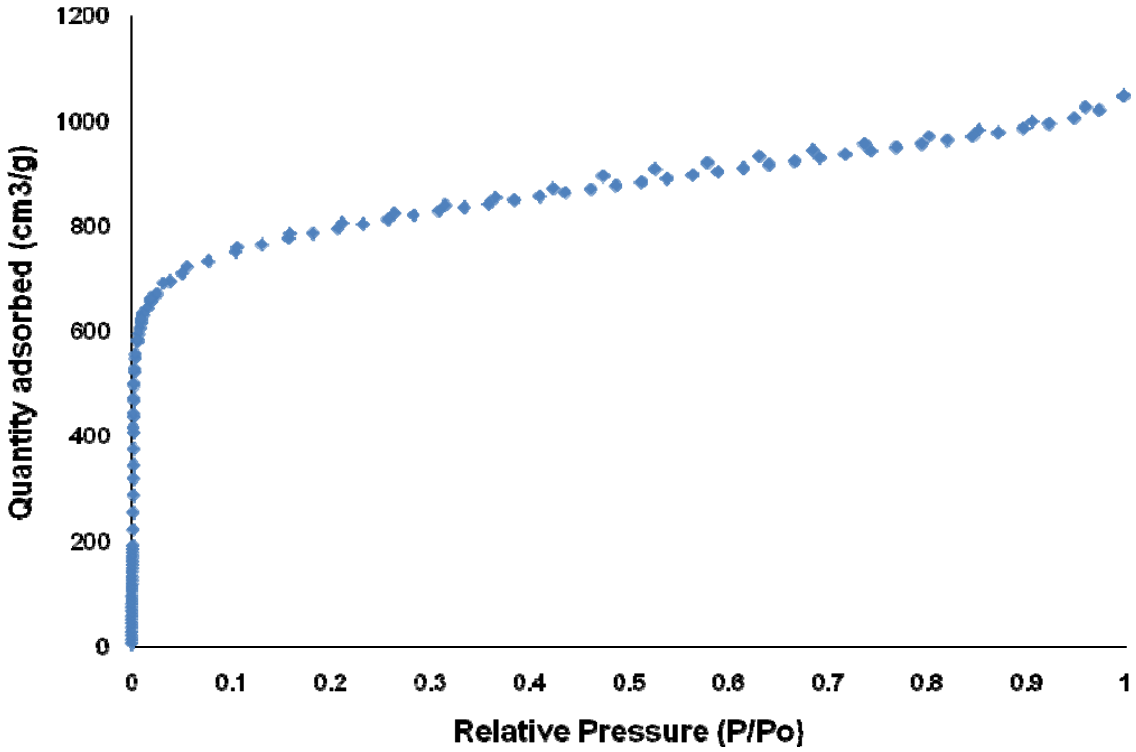

Figure S18. N_2 gas adsorption / desorption isotherms for COF-102 measured at 77.3 K

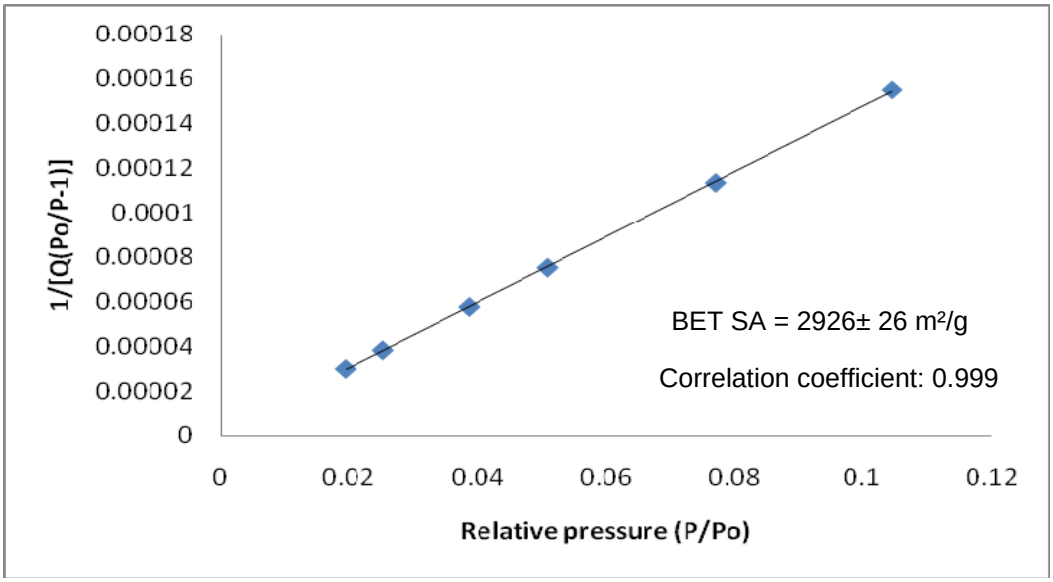

Figure S19. BET surface area plot for COF 102. Surface area shown was calculated over the relative pressure range P/P_o = 0.05–0.1

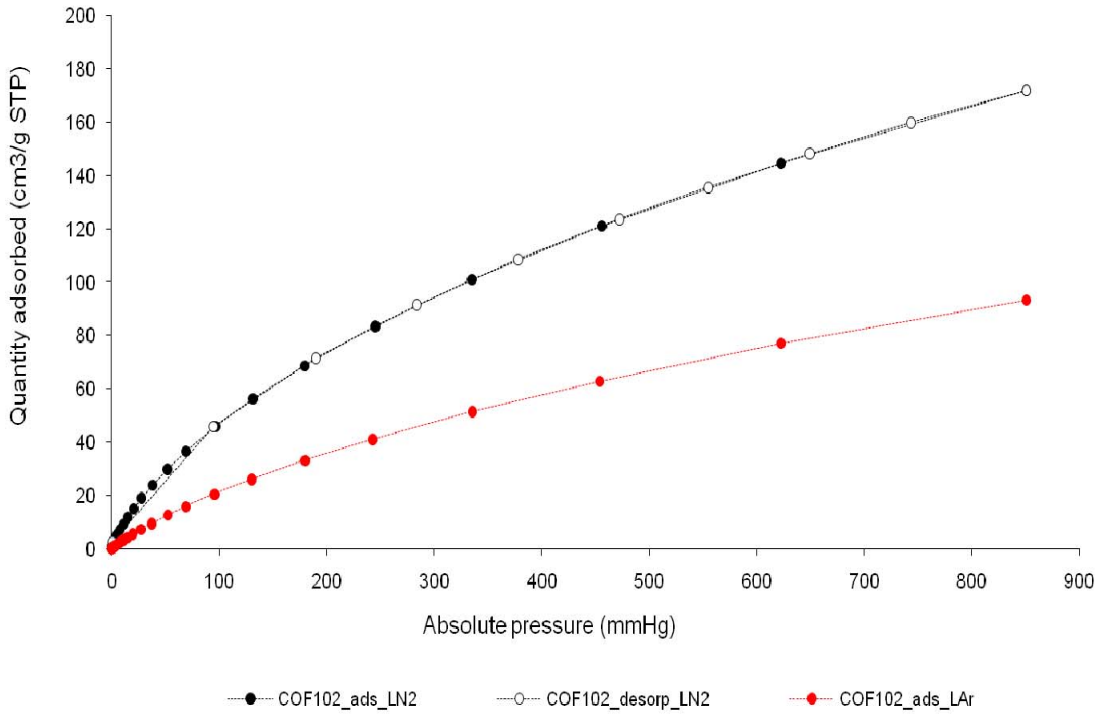

Figure S20. Hydrogen adsorption for COF 102 as measured at liquid N_2 and liquid Ar temperatures (77.3 and 87.2 K, respectively)

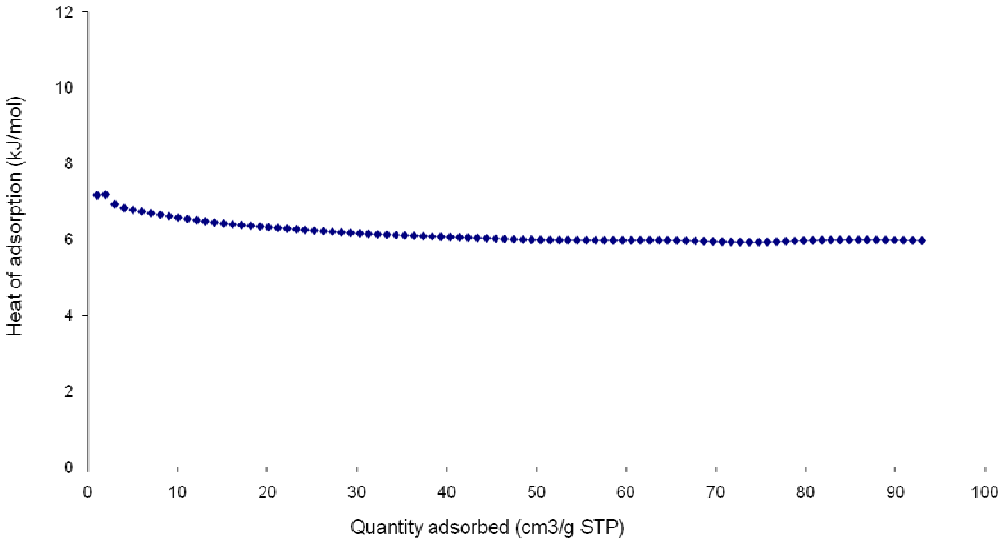

Figure S21. Isosteric heat of sorption for H_2 on COF 102 as calculated from adsorption isotherms shown in S16; average value approx.– 6.5 kJ/mol/

Sample	P/P_0 range	Surface area, m^2/g (error +/- m^2/g)	Correlation coefficient	Published Values[1,2]
COF 5 open vessel	0.017-0.3	1870 (53)	0.995	
COF 5 open vessel	0.05-0.3	1858 (68)	0.993	
COF 5 open vessel	0.05-0.2	2236 (94)	0.995	
COF 5 open vessel	0.05-0.1	2027 (87)	0.998	1590[1]
COF 5 open vessel	0.017-0.1	1849 (74)	0.996	
COF 5 open vessel	0.017-0.1	2117 (81)	0.995	
COF 5 sealed vessel	0.05-0.1	2019 (65)	0.998	1590[1]
COF 5 sealed vessel	0.05-0.2	2234 (120)	0.991	
COF 5 sealed vessel	0.05-0.3	1945 (85)	0.992	
COF 102	0.02-0.1	2964 (15)	0.999	
COF 102	0.02-0.2	2781 (36)	0.999	
COF 102	0.02-0.3	2559 (53)	0.997	
COF 102	0.05-0.1	2926 (26)	0.999	3472 (Ar)[2]
COF 102	0.05-0.2	2718 (44)	0.999	
COF 102	0.05-0.3	2483 (59)	0.997	

Figure S22. Table showing sensitivity of using different P/P_o relative pressure ranges in the calculation of apparent BET surface areas for COF-5 and COF-102 produced by microwave heating. Figures shown in red are closest to matching previously published analysis conditions although it should be noted that *argon*, not N_2, was used as the analysis gas (87.2 K) to characterize COF-102 in *Science*, **2007**, *316*, 268.

[1] Côté *et al.*, *Science*, **2005**, *310*, 1166

[2] El-Kaderi *et. al.*, *Science*, **2007**, *316*, 268