

**Table S1** Mo K-edge EXAFS-derived structural parameters for

[Mo<sub>2</sub>(AA)<sub>2</sub>(AN)<sub>4</sub>](BF<sub>4</sub>)<sub>2</sub> (**2**) and [Mo<sub>2</sub>(AA)<sub>2</sub>(AN)<sub>4</sub>](BF<sub>4</sub>)<sub>2</sub>/MCM-41 (AA =  $\mu$ -CH<sub>3</sub>CONH)

Sample	Atom	CN	$r/\text{\AA}$	$2\sigma^2/\text{\AA}^2$	$E_f/\text{eV}$	$R$ (%)
<b>2</b> (3)	O	2.0(2)	2.076(9)	0.0063(19)	-3.3(6)	22.4
	Mo	1.0(1)	2.132(2)	0.0031(2)		
	N	2	2.12	0.007		
	C	2	3.232(13)	0.0116(24)		
<b>2</b> /MCM-41 (6)	O	3.0(2)	2.070(6)	0.0049(11)	-12.2(6)	29.5
	Mo	1.0(1)	2.109(3)	0.0049(3)		
<b>2</b> /MCM-41/air (3)	O	1.5(1)	1.683(3)	0.0063(5)	1.4(5)	30.0
	O	3.0(3)	1.964(4)	0.0113(9)		
	O	1.0(1)	2.128(11)	0.0099(27)		
	Mo	1.0(1)	2.585(2)	0.0080(3)		
	Si	1.0(4)	3.730(18)	0.0085(33)		

**Figure S1** Mo K-edge EXAFS and Fourier transforms for (a)  $[\text{Mo}_2(\text{AA})_2(\text{AN})_4](\text{BF}_4)_2$  (**2**), (b) **2**/MCM-41 and (c) **2**/MCM-41/air. The solid line represents the experimental data and the dashed line shows the best fit using parameters given in Table S1.

