Electronic Supplementary Material (ESI) for Dalton Transactions.

Efficient Microwave Assisted Synthesis of Metal-Organic Framework UiO-66: Optimization and Scale Up

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ELECTRONIC SUPPLEMENTARY INFORMATION

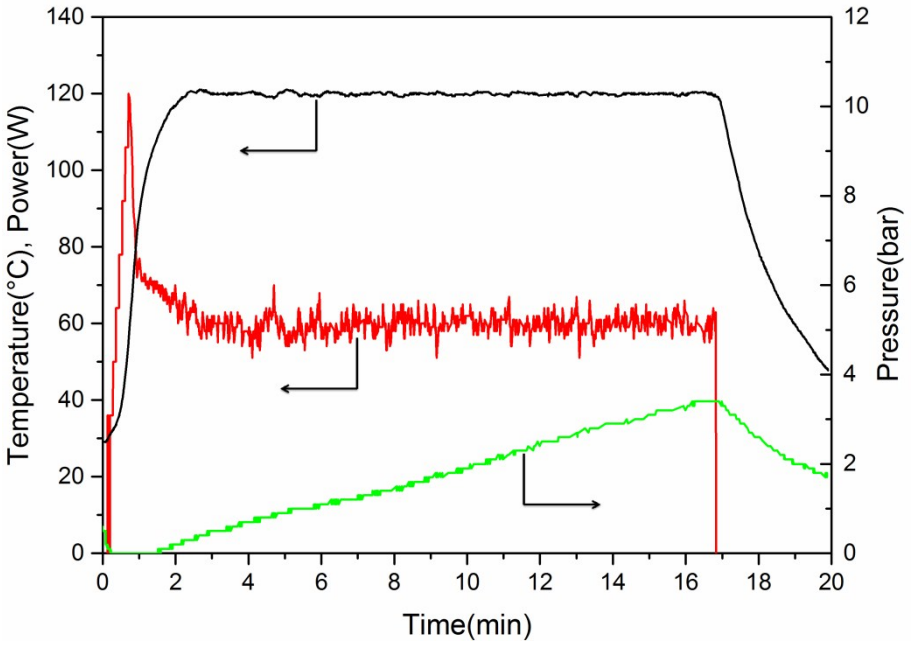

Figure S1. The evolution of power (red line), temperature (black line), and pressure (green line) as functions of time in experiment **22** (see Table S1).

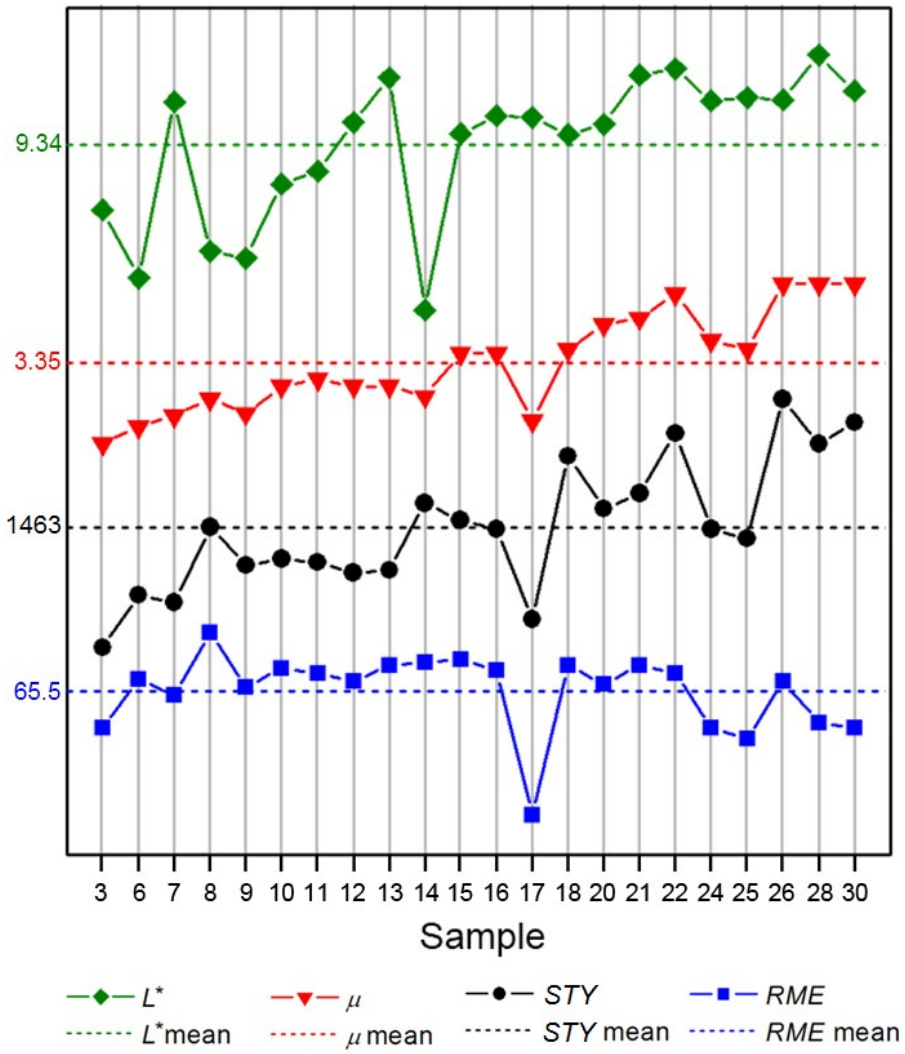

Figure S2. Graphical summary of the values of *RME*, *STY*, μ, and *L** for each experiment conducted with MW heating, compared to the mean values calculated over all of the experiments. Sample numbers refer to Table S1. Samples **1**, **2**, **4**, and **5** were not included in the calculation of the mean value and were not reported in the graph, due to presence of amorphous matter.

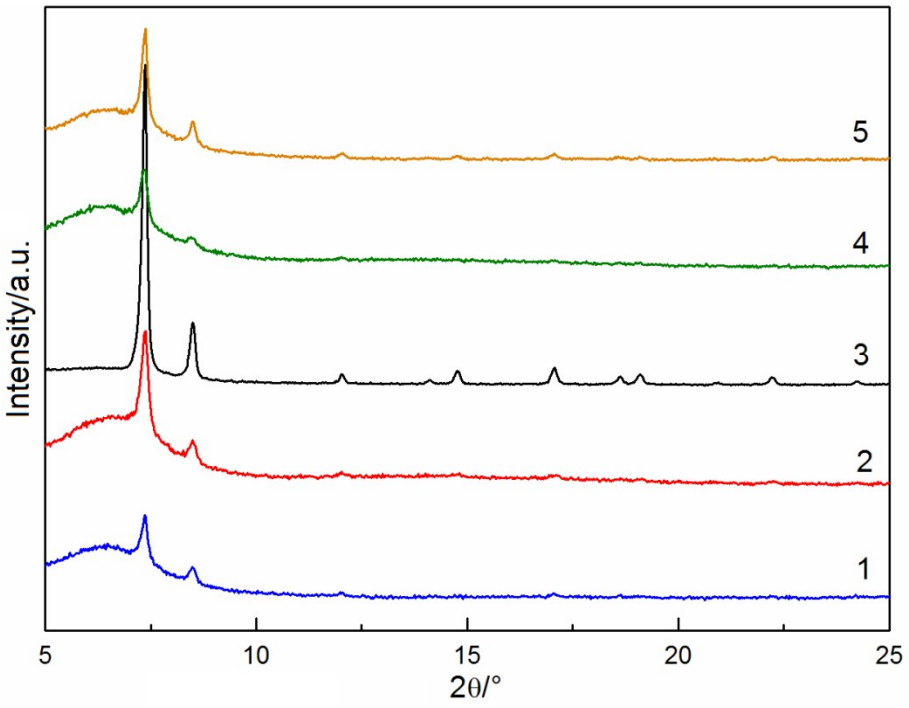

Figure S3. PXRD patterns of samples **1**÷**5**.

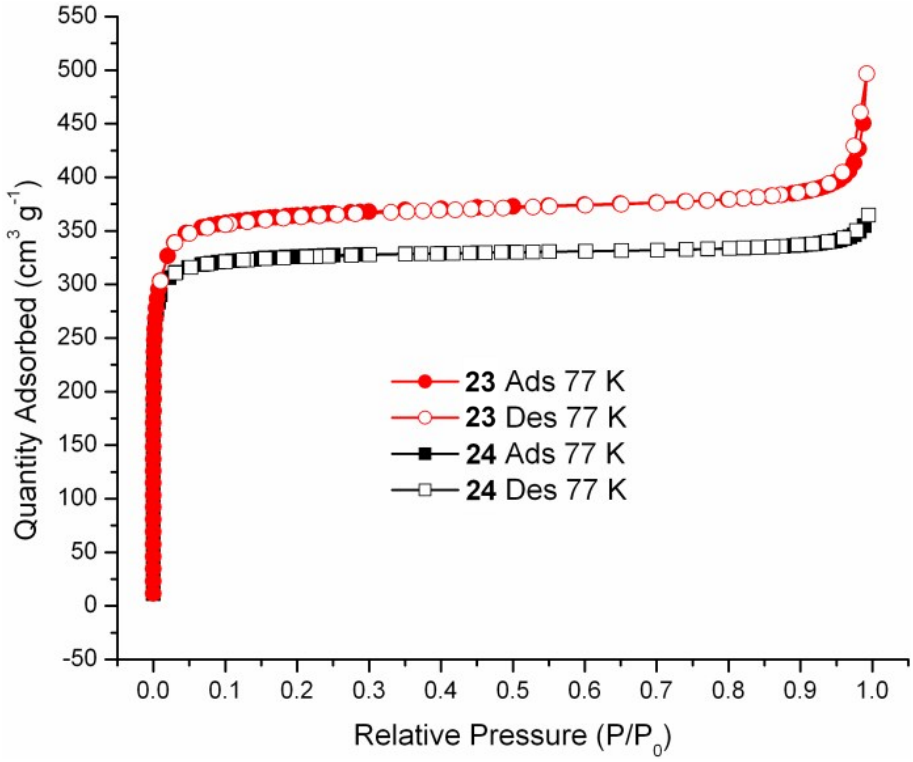

Figure S4. N_2 isotherms at 77 K of samples **23** and **24**.

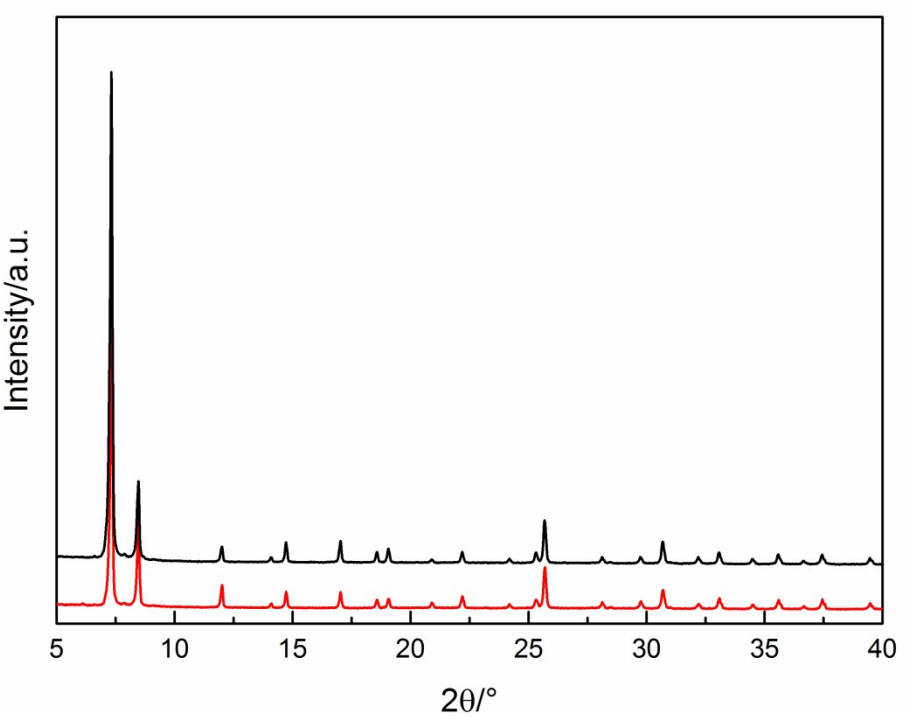

Figure S5. Comparison between the PXRD patterns of **22** (red line) and **22su** (black line).

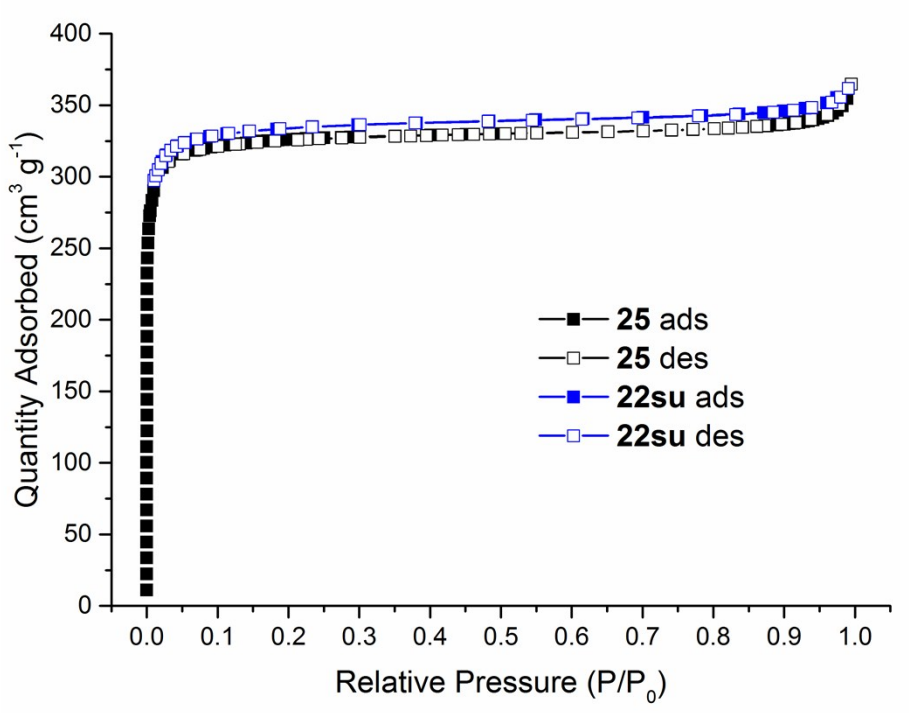

Figure S6. N_2 adsorption isotherms for **22** and **22su**.

Table S1. List of all of the experiments performed in this work.

Entry	n_{Zr}, n_{bdc} (mmol)	[Zr], [H_2bdc] (mol/L)	AcOH (eq)	*T* (°C)	Irradiation Time (s)	Mean Power (W)	*E* (kJ)	Absolute Yield (mg)	*FWHM* 004 (°2θ)	*RME* (%)	*STY* (kg/m^{3}d)	μ (mg/kJ)	*L**
1	0.25	0.016	70	120	523	149	77.9	48	-[c]	48	493	0.62	-
2	0.25	0.016	70	120	649	105	68.1	55	-[c]	55	455	0.81	-
3	0.25	0.016	70	120	831	151	125.5	52	0.144	52	336	0.41	6.94
4	0.25	0.016	70	120	951	133	126.5	52	-[c]	52	293	0.41	-
5	0.25	0.016	70	120	1113	126	140.2	48	-[c]	48	231	0.34	-
6	0.50	0.034	0	120	848	161	136.5	140	0.225	70	951	1.03	4.45
7	0.50	0.029	70	120	1053	85	89.5	127	0.092	64	606	1.42	10.89
8	0.75	0.050	0	120	1115	115	128.2	260	0.184	87	1343	2.03	5.44
9	0.75	0.048	10	120	1116	118	131.7	200	0.193	67	999	1.52	5.19
10	0.75	0.046	30	100	1052	85	89.4	223	0.127	74	1117	2.49	7.88
11	0.75	0.046	30	120	1040	75	78.0	217	0.120	72	1099	2.78	8.36
12	0.75	0.044	50	120	1047	80	83.8	208	0.099	69	992	2.48	10.15
13	0.75	0.042	70	120	1049	86	90.2	225	0.085	75	1015	2.49	11.78
14	1.00	0.067	0	120	1110	130	144.3	304	0.305	76	1578	2.11	3.28
15	1.00	0.060	30	100	1048	82	84.0	308	0.103	77	1502	3.67	9.74
16	1.00	0.060	30	120	1032	75	77.4	290	0.096	73	1437	3.75	10.38
17[a]	1.00	0.085	30	100	1017	64	65.1	80	0.097	20	571	1.23	10.33
18[a]	1.00	0.085	30	120	1043	75	78.2	300	0.103	75	2088	3.84	9.71
19[b]	1.00	0.060	30	120	24h	-	-	240	0.092	60	14	-	10.93
20	1.25	0.073	30	100	1032	70	72.2	342	0.099	68	1655	4.74	10.08
21	1.25	0.073	30	120	1037	74	76.7	377	0.084	75	1816	4.92	11.88
22[a]	1.25	0.103	30	120	1010	61	61.6	360	0.083	72	2504	5.90	12.11
23[b]	1.25	0.073	30	120	24h	-	-	400	0.109	80	23	-	9.16
24	1.50	0.085	30	90	1018	73	74.3	310	0.092	52	1478	4.17	10.93
25	1.50	0.085	30	100	1031	73	75.3	290	0.090	48	1365	3.85	11.06
26[a]	1.50	0.119	30	100	1017	66	67.1	416	0.091	69	2761	6.20	10.98
27[a,b]	1.50	0.119	30	120	24h	-	-	538	0.115	90	42	-	8.71
28[a]	1.75	0.135	30	100	1013	60	60.8	375	0.079	54	2414	6.17	12.63
29[a,b]	1.75	0.135	30	120	24h	-	-	244	0.076	35	18	-	13.21
30[a]	2.00	0.149	30	100	1020	66	67.3	414	0.089	52	2560	6.15	11.30
31[a,b]	2.00	0.149	30	120	24h	-	-	100	0.073	13	7	-	13.77

All of the experiments were carried out using 15 mL of DMF in 30 mL vial in static conditions, except where differently noted. The Zr/H_2bdc/H_2O ratio was 1:1:6. The irradiation times are the sum of the heating ramp and the isothermal phase.

[a] 10 mL DMF; [b] Conventional Heating; [c] Presence of amorphous matter