

Article type: Full paper

Supporting Information for

Developing a sustainable route to environmentally relevant metal-organic frameworks: ultra-rapid synthesis of MFM-300(Al) using microwave heating

Ieuan Thomas-Hillman,^a Lee A. Stevens,^a Marcus Lange,^b Jens Möllmer,^b William Lewis,^{c,d} Chris Dodds,^a Samuel W. Kingman,^a Andrea Laybourn^{a}*

- a. Faculty of Engineering, University of Nottingham, Nottingham, NG7 2RD, UK
- b. Institute of Non-Classical Chemistry, Permoserstrasse 15, 04318 Leipzig, Germany,
- c. School of Chemistry, University of Nottingham, Nottingham NG7 2RD, UK
- d. School of Chemistry, The University of Sydney, New South Wales 2006, Australia

*Andrea.Laybourn@nottingham.ac.uk

Contents

Further experimental details	2
------------------------------	---

Starting from the conventional solvothermal route,¹⁻³ a reproducible microwave-assisted synthetic procedure to MFM-300(Al) was developed. The aims of developing the new route were to reduce the reaction time, negate the requirement for additional chemicals (piperazine and nitric acid), and to minimise the amount of unwanted side products (*i.e.* γ -Al(O)OH) and unreacted starting materials. A summary of the method development steps is given in **Table S1** below.

Table S1. Summary of method development for a microwave-assisted route to MFM-300(Al).

All reactions performed in deionised water without piperazine or nitric acid.

Synthetic modification	Aluminium chloride source	Reactant ratio Aluminium: H ₄ BPTC	Reaction time (minutes)	Synthetic outcome
1	Anhydrous	2:1	120	γ -Al(O)OH produced.
2	Anhydrous	2:1	30	γ -Al(O)OH produced.
3	Hexahydrate	2:1	10	Unreacted linker, <i>ca.</i> 5.4 – 10.4% from TGA.
4*	Hexahydrate	4:1	10	Unreacted linker present in PXRD pattern but none detected by TGA.
5	Hexahydrate	> 4:1	10	High intensity peak corresponding to unreacted linker in PXRD pattern.

*Desired synthetic outcome achieved; results presented in main paper.

References

- (1) Han, X. ; Godfrey, H. G. W.; Briggs, L.; Davies, A. J.; Cheng, Y.; Daemen, L. L.; Sheveleva, A. M.; Tuna, F.; McInnes, E. J. L.; Sun, J.; Drathen, C.; George, M. W.; Ramirez-Cuesta, A. J.; Thomas, K. M.; Yang, S.; Schröder, M. Reversible adsorption of nitrogen dioxide within a robust porous metal–organic framework. *Nature Materials* **2018**, *17*, 691-696.
- (2) Yang, S.; Ramirez-Cuesta, A. J.; Newby, R.; Garcia-Sakai, V.; Manuel, P.; Callear, S. K.; Campbell, S. I.; Tang, C. C.; Schröder, M. Supramolecular binding and separation of hydrocarbons within a functionalized porous metal–organic framework. *Nature Chemistry* **2015**, *7*, 121-129.
- (3) Yang, S.; Sun, J.; Ramirez-Cuesta, A. J.; Callear, S. K.; David, W. I. F.; Anderson, D. P.; Newby, R.; Blake, A. J.; Parker, J. E.; Tang, C. C.; Schröder, M. Selectivity and direct visualization of carbon dioxide and sulfur dioxide in a decorated porous host. *Nature Chemistry* **2012**, *4*, 887-894.

