A "green" method for isolation of cucurbit[7]uril via a solid state metathesis reaction

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Supporting Information

Both 20% DCl and the NH_4PF_6 salt are from Alfa Aesar. [$C_n$mim]Br was synthesised from methyl imidazole (Aldrich) and the appropriate 1-bromo alkyl (Aldrich). NMR spectra were recorded on a Bruker DRX-400, and chemical shifts are quoted in parts per million relative to residual proteo species. The starting CB[5]/[7] mixture used in these experiments was obtained from a standard CB[n] synthesis.[1, 2] The overall yield of CB[7] and CB[5] is 19.9% and 6.4%, respectively, using the procedure disclosed in this paper, produces both greater isolated yield and higher purity of both CB[7] and CB[5] than in previously published routes for all preparing all CB[n] species in a single reaction.

The waste from each step has also been carefully controlled. The non-recycled salt (NH_4Br) was filtered from the solution after the ion exchange process. The waste solvents (DCM and methanol) were collected from the isolation process and 90% of both of these solvents could be recycled for further use in this procedure after distillation. Finally, 80% of the [$C_n$mim]PF_6 was isolated from the solvent and was reused after a simple ion exchange form PF_6 back to the original Br counterion.

References

[1] J. Kim, I.-S. Jung, S.-Y. Kim, E. Lee, J.-K. Kang, S. Sakamoto, K. Yamaguchi and K. Kim, *J. Am. Chem. Soc.*, 2000, **122**, 540-541.

[2] A. I. Day, A. P. Arnold, R. J. Blanch and B. Snushall, *J. Org. Chem.*, 2001, **66**, 8094-8100.

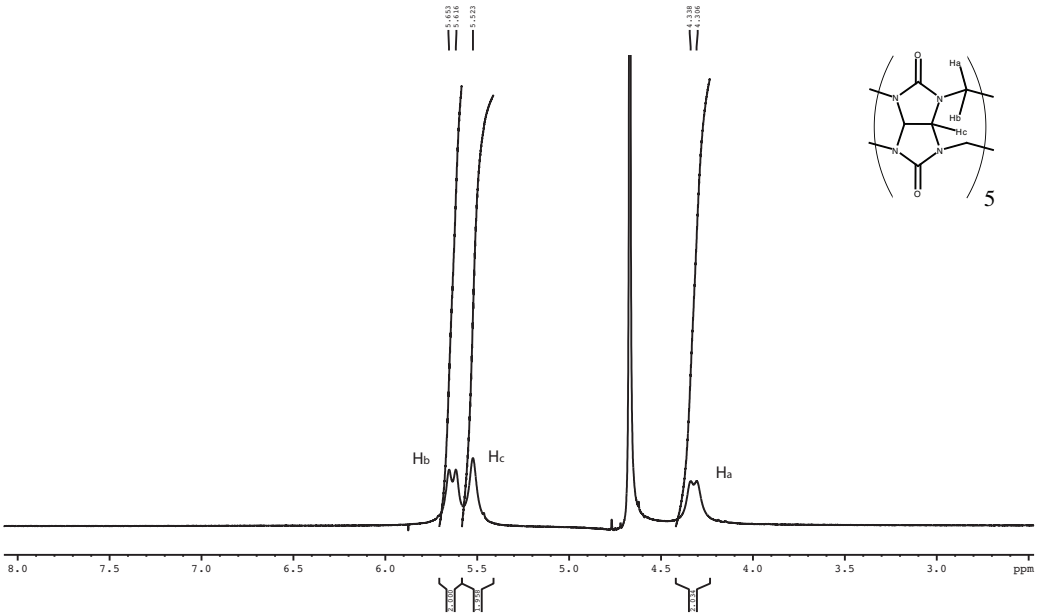

Figure 1: ^{1}H NMR spectrum (in D_2O) of CB[5]

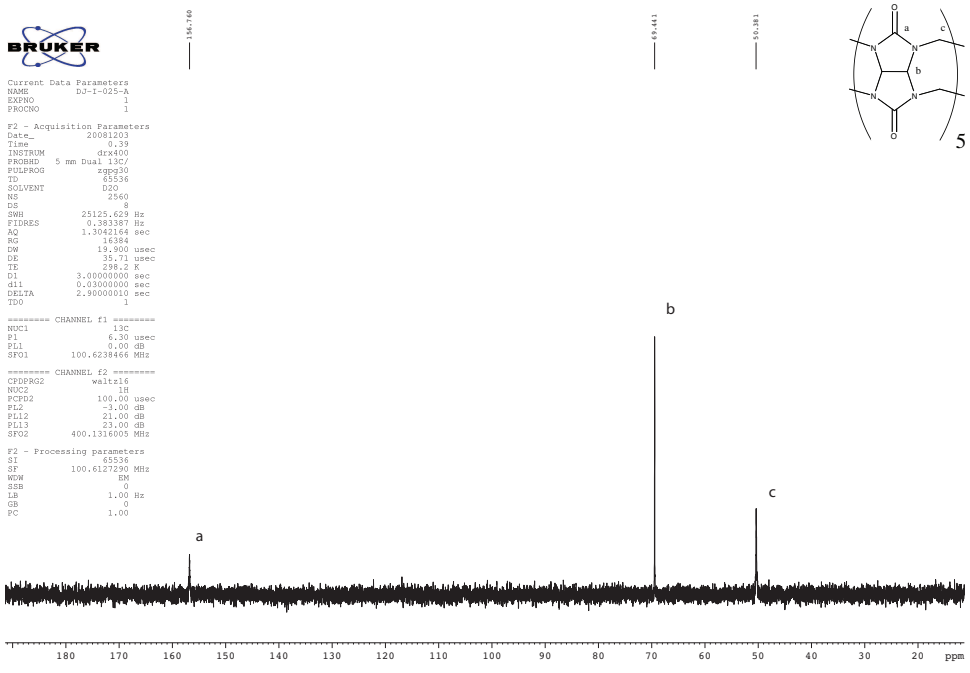

Figure 2: ^{1}3C NMR spectrum (in D_2O) of CB[5]

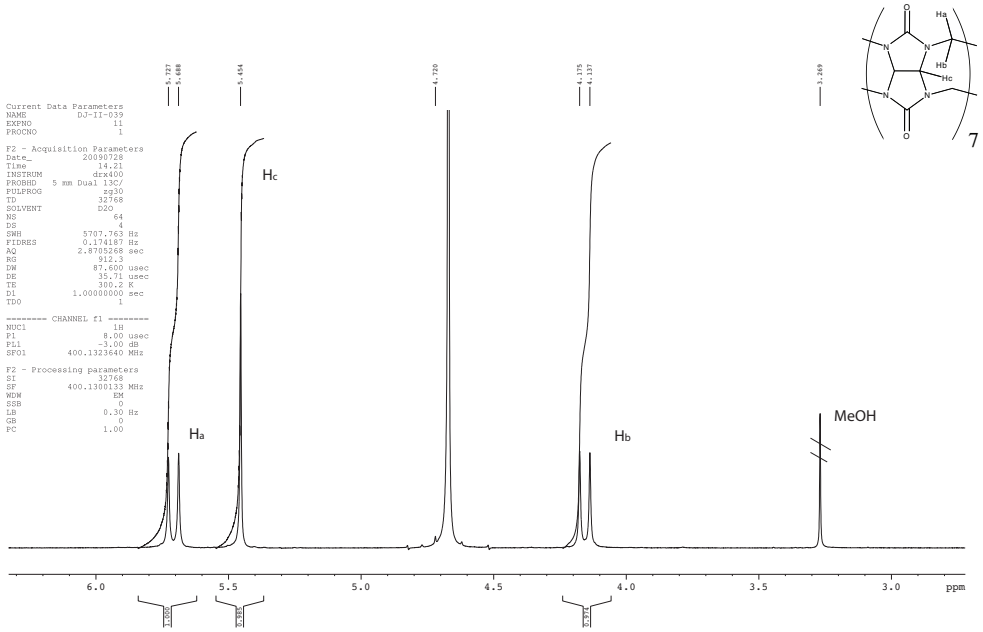

Figure 3: ^{1}H NMR spectrum (in D_2O) of CB[7]

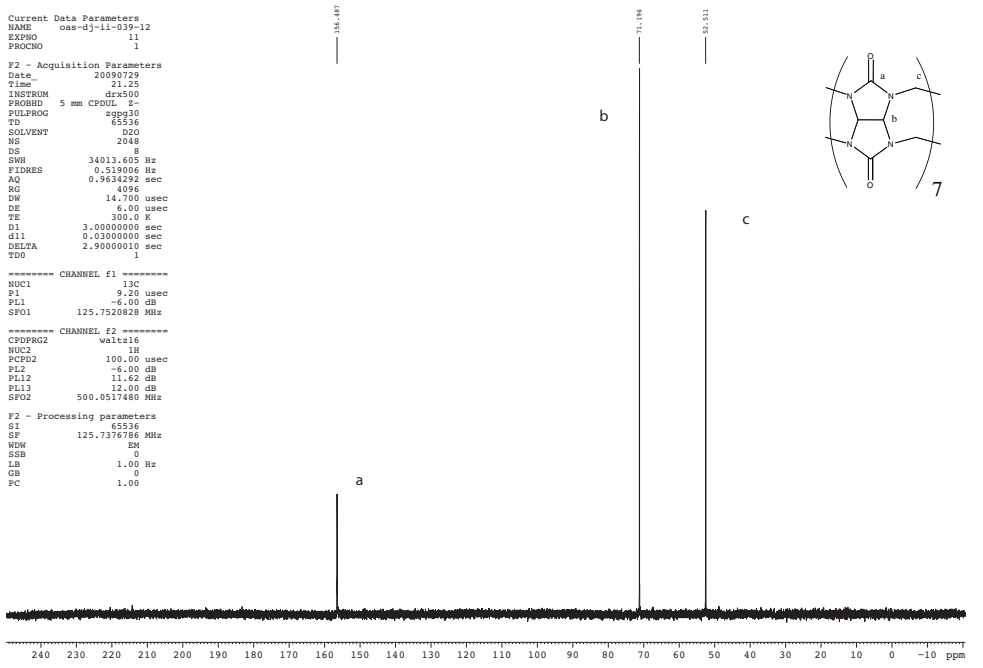

Figure 4: ^{1}3C NMR spectrum (in D_2O) of CB[7]

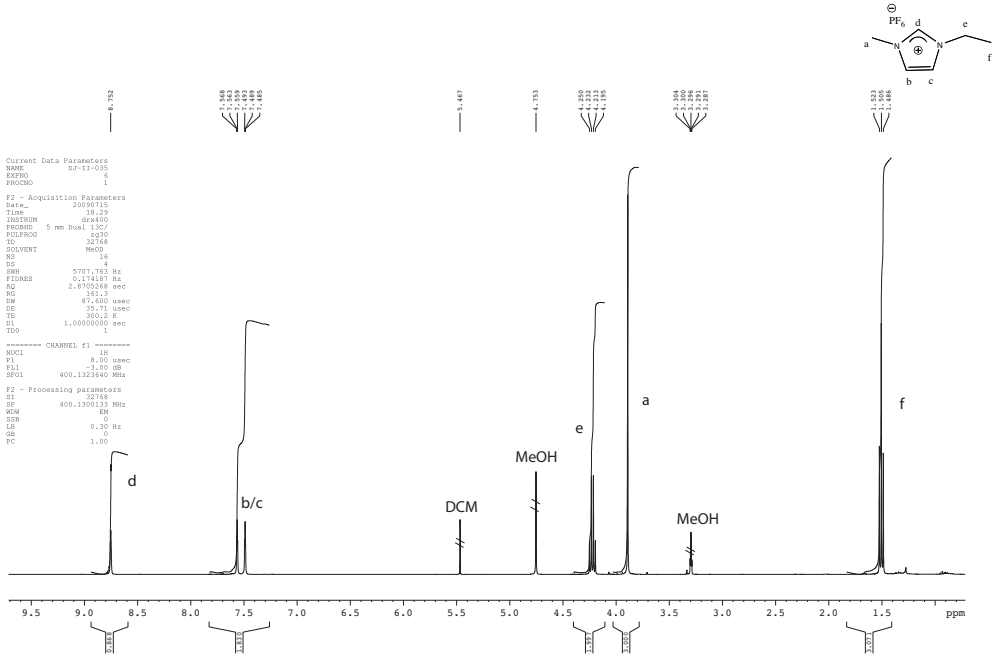

Figure 5: ^{1}H NMR spectrum of recycled [C_2mim]PF_6 (in MeOD) by extraction with DCM from water.