

**Supplemental Information for Manuscript Entitled: "Calix[5]arene: A versatile
sublimate that displays gas sorption properties"**

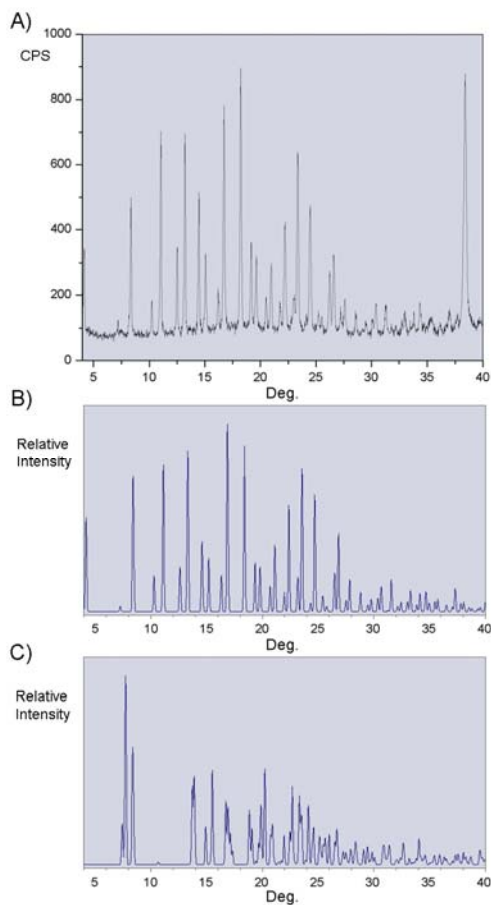


Figure S1. Experimental and simulated (from crystal structure calculation) X-ray powder patterns of the structures found for sublimed **3**. A) Experimental XRPD of pure β obtained at 290 °C. B) Simulated XRPD of β . C) Simulated XRPD of α for comparison.

Large versions of Figures 2 and 3:

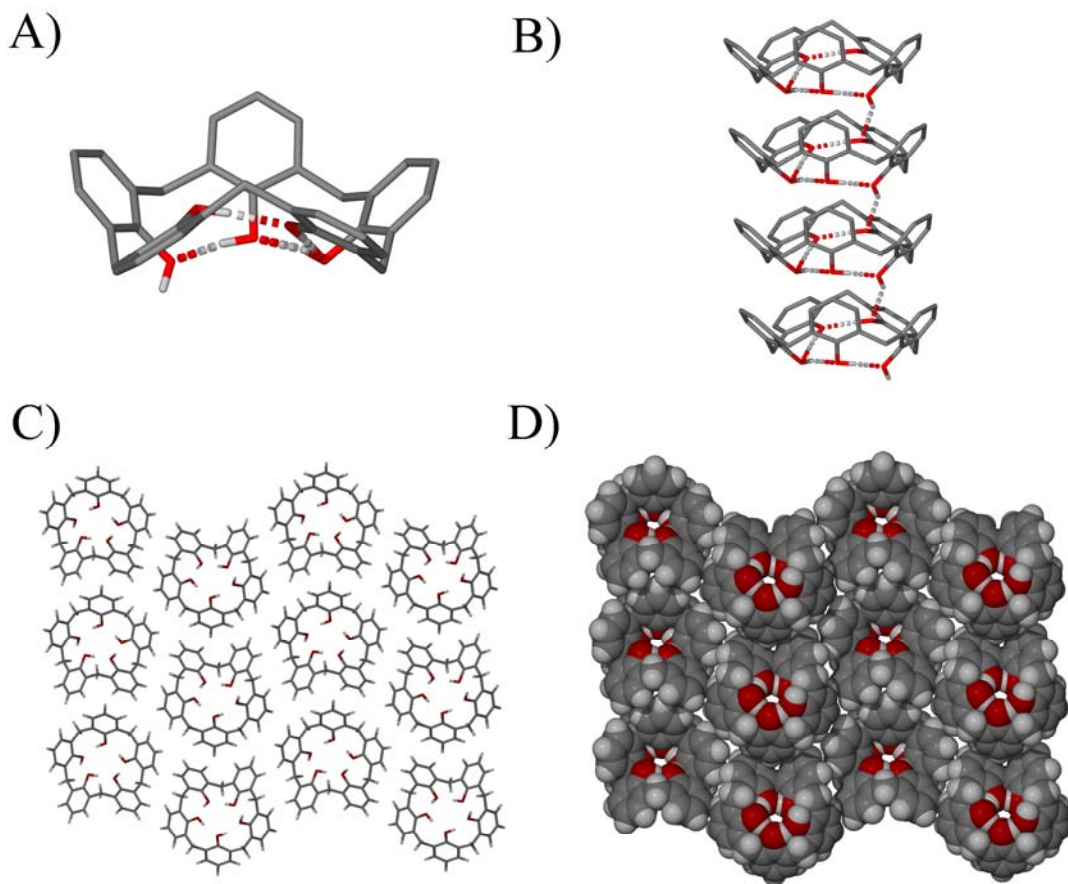


Figure S2. Different views (not to scale) of polymorph α . A) The asymmetric unit showing the distortion to the cone conformation of **3**. B) A stack of **3** showing the hydrogen-bond helix running through the core of the stack. C) Packing of stacks of **3** along the *a* axis: layers of stacks running in alternating directions form a wave-like arrangement along the *c* axis.. D) Space filling representation of the packing diagram in C. In A and B some hydrogen atoms have been omitted for clarity.

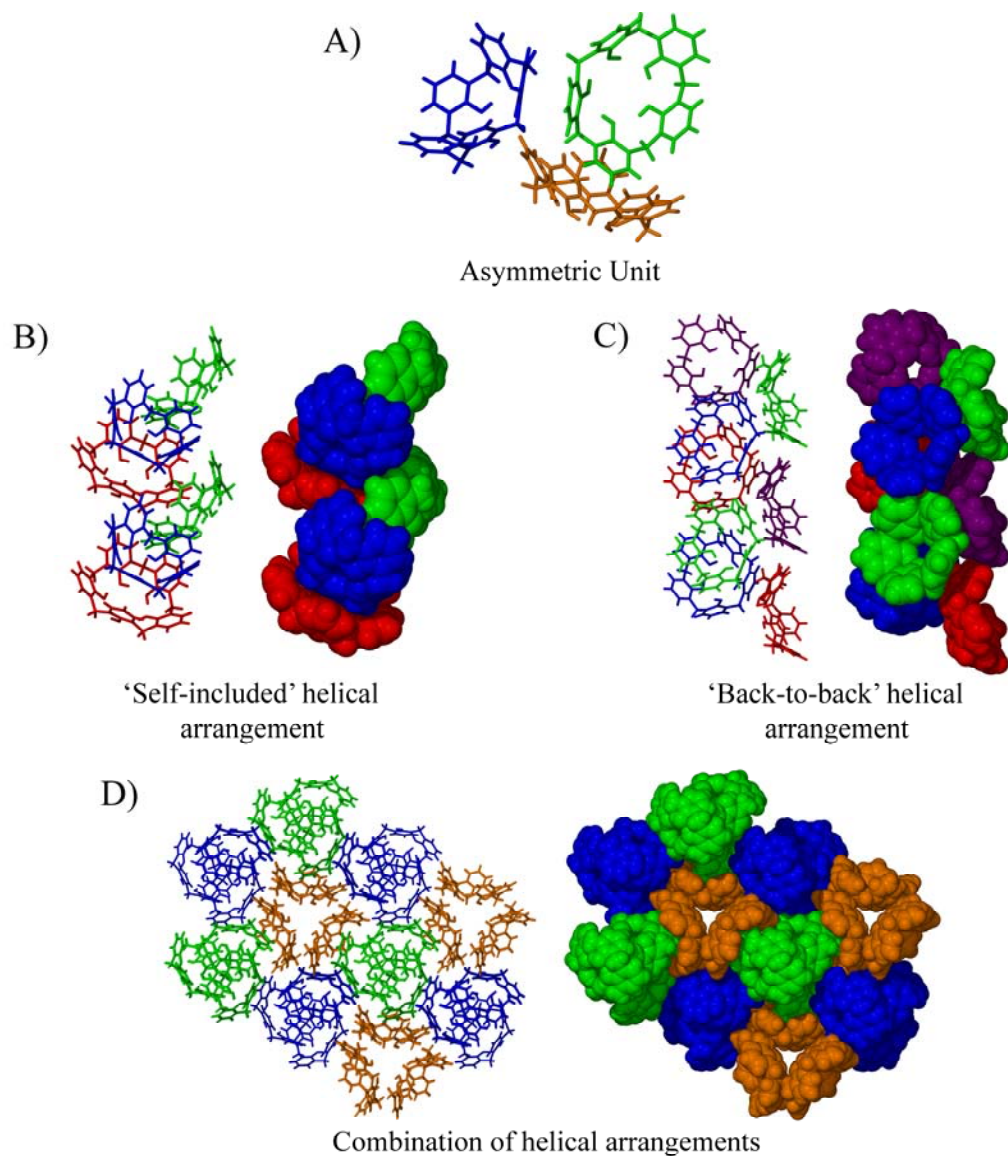


Figure S3. Stick and space filling views (not to scale) of crystal structure β . A) The asymmetric unit showing three independent molecules of **3** (colored individually). B) A 'self-included' helical arrangement. C) The 'back-to-back' helical arrangement. D) The extended structure showing the packing of 'self-included' (green and blue) and 'back-to-back' (orange) helical arrangements, as well as the small pores present in the latter. Colors of **3** in (B) and (C) are not relative to (A) but have been used to show the helical nature of the arrangements. Colors of molecules of **3** in (D) are relative to those in (A).