Electronic Supplementary Material (ESI) for RSC Advances.

Synthesis, structure and conformational mobility of tetra-substituted cyanomethoxy *p*-*tert*-butylcalix[4]arenes

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Supplementary Information

Conformational changes in DMSO solution measured by ^{1}H NMR integration.

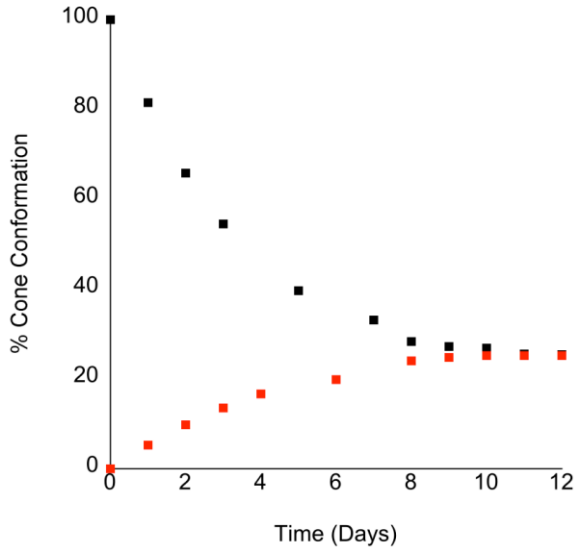

Figure S1. Percentage of cone conformation of 5,11,17,23-tetra-*tert*-butyl-25,26,27,28-tetrakis(cyanomethoxy)calix[4]arene present over time while heating in d_6-DMSO solution at 150˚ C, commencing with the cone conformer **2** (black) or partial cone **3** (red).

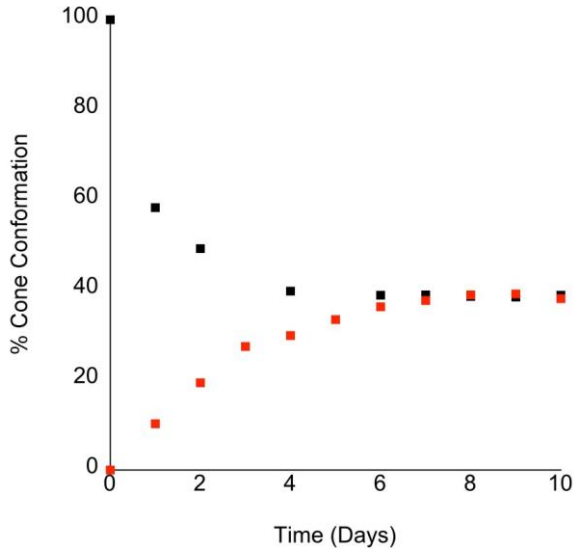

Figure S2. Percentage of cone conformation of 5,11,17,23-tetra-*tert*-butyl-25,27-bis(diethylcarbamoylmethoxy)-26,28-bis(cyanomethoxy)calix[4]arene present over time while heating in d_6-DMSO solution at 150˚ C, commencing with the cone conformer **4** (black) or partial cone **5** (red).

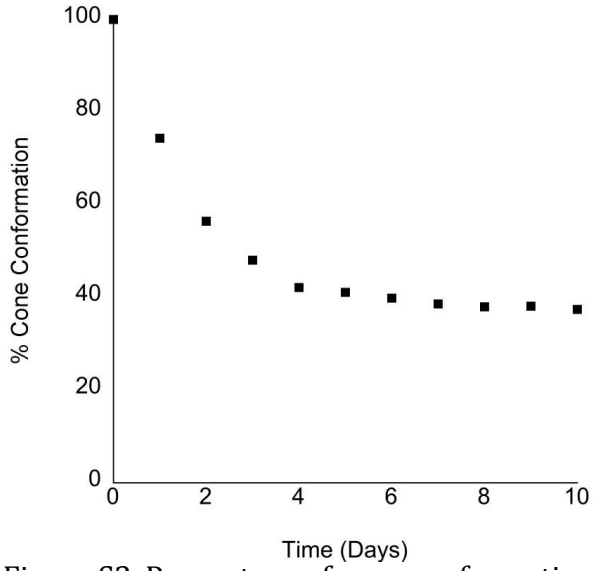

Figure S3. Percentage of cone conformation of 5,11,17,23-tetra-*tert*-butyl-25-diethylcarbamoylmethoxy-26,27,28-tris(cyanomethoxy)calix[4]arene present over time while heating in d_6-DMSO solution at 150° C, commencing with the cone conformer **7**.

Figures for Structures of Calixarenes 3 – 7.

Ellipsoids have been drawn at the 50% probability level

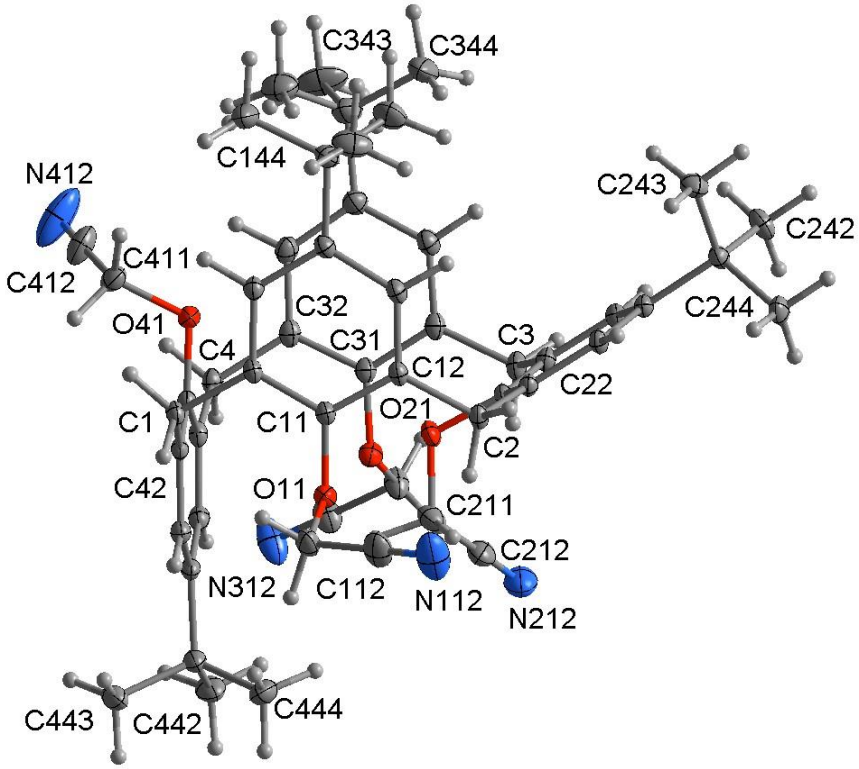

Figure S4 Molecular structure of **3**. One set of atoms of the disordered groups have been omitted for clarity.

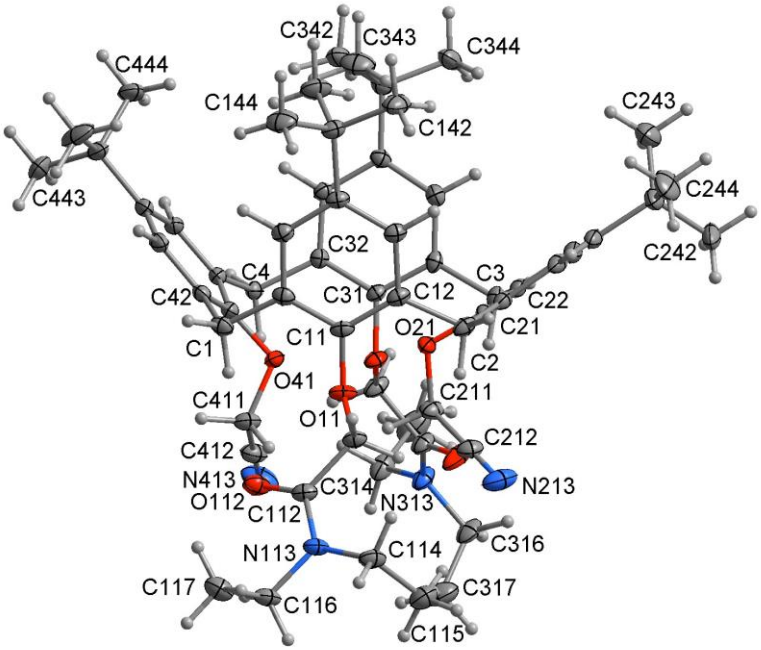

Figure S5. Molecular structure of molecule 1 of **4**. The minor components of the disorder have been omitted.

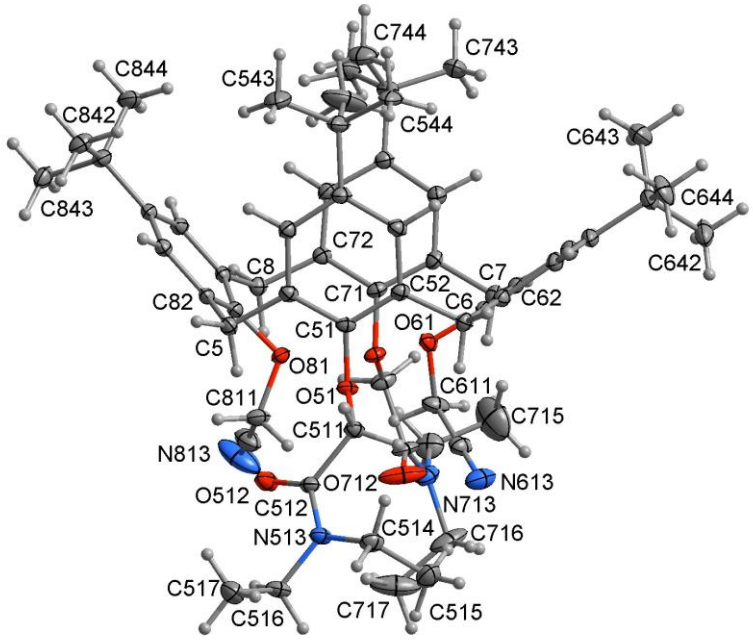

Figure S6. Molecular structure of molecule 2 of **4**. The minor components of the disorder have been omitted.

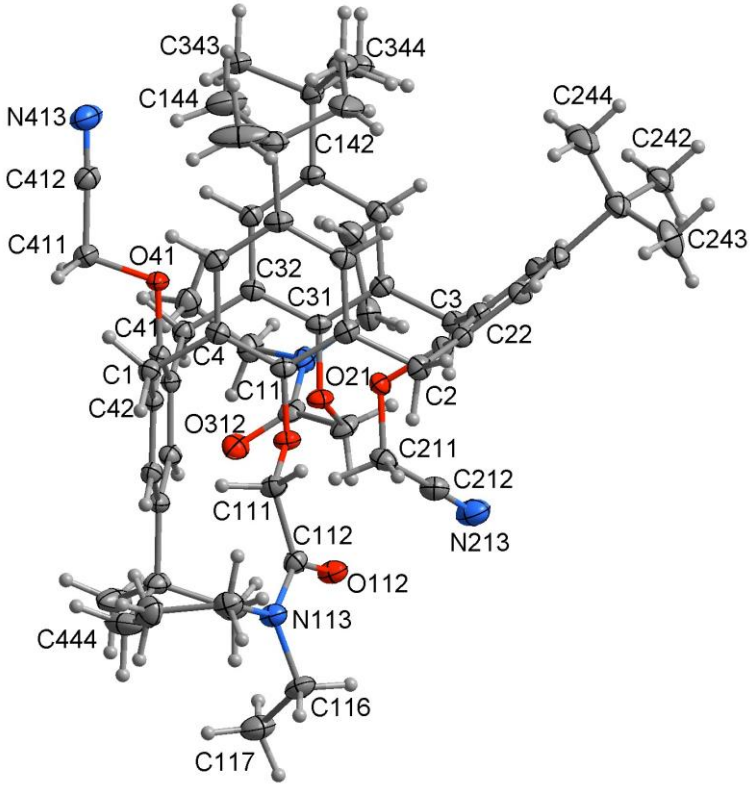

Figure S7. Molecular structure of **5** in **5**·EtOH.

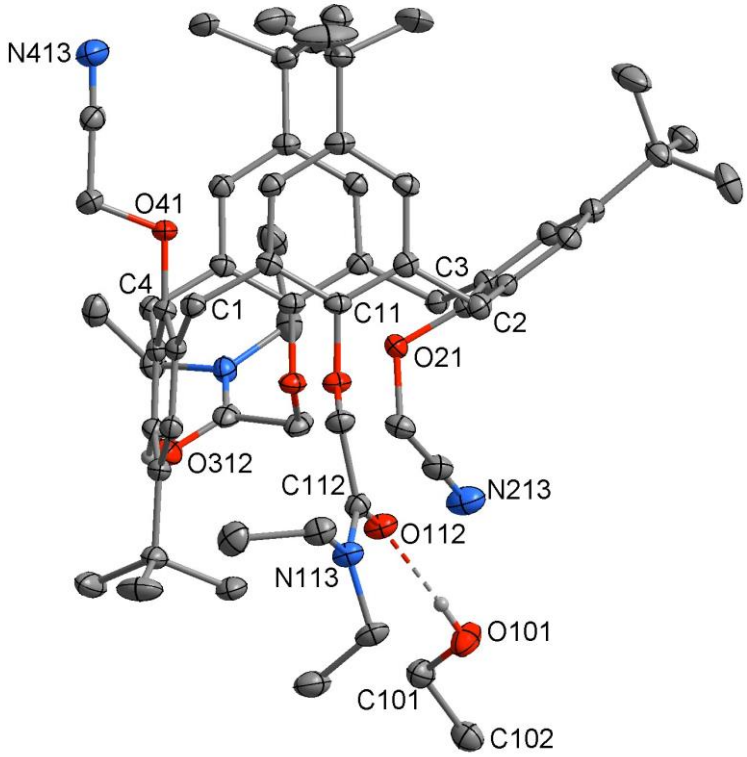

Figure S8. Structure of **5**·EtOH showing the hydrogen bonding to the ethanol solvent molecule.

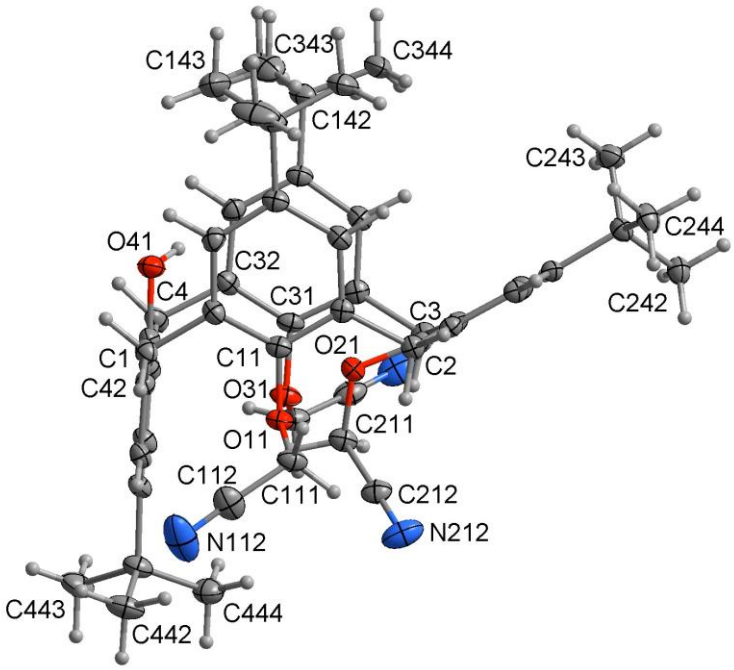

Figure S9. Molecular structure of **6**. The minor components of the disorder have been omitted.

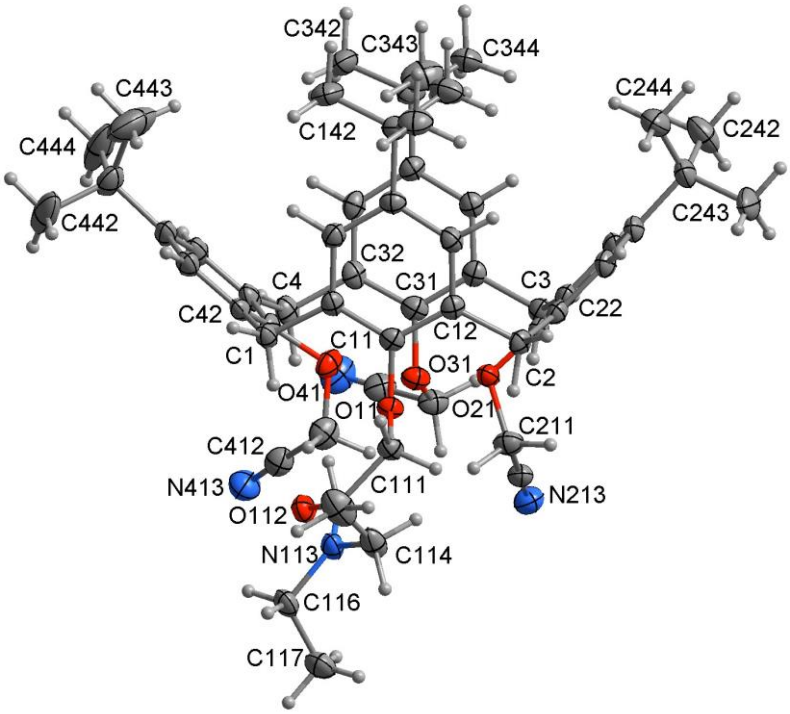

Figure S10. Molecular structure of **7** in **7**·EtOH.

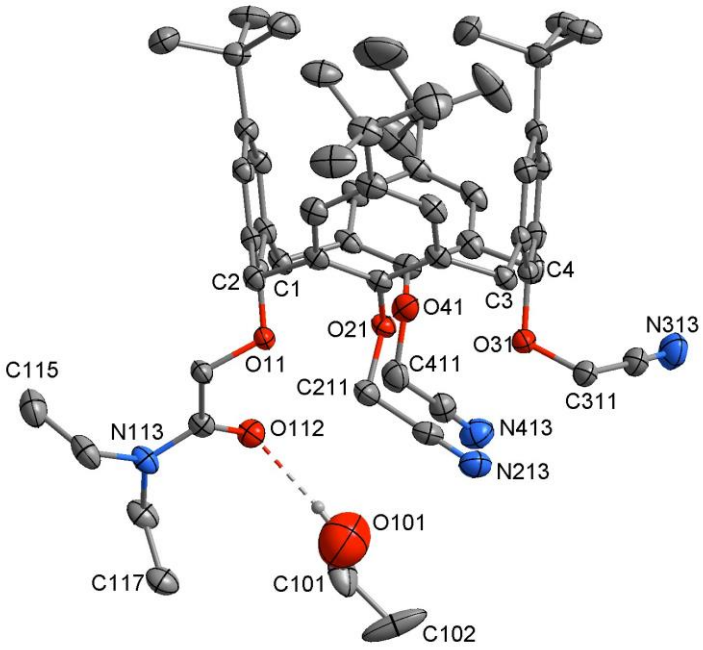

Figure S11. Structure of **7**·EtOH showing the hydrogen bonding to the ethanol solvent molecule.

Hydrogen Bond Geometries

Table S1. Hydrogen bonds for **5**·EtOH [Å and °].

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)
O(101)-H(101)...O(112)	0.84	1.94	2.7821(18)	177.2

Table S2. Hydrogen bonds for **7**·EtOH [Å and °].

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)
O(101)-H(101)...O(112)	0.84	2.00	2.840(14)	175.4
O(201)-H(201)...O(112)	0.84	2.04	2.812(5)	152.7