Supplementary Material (ESI) for CrystEngComm

Supporting Information

A solid-state molecular capsule based on *p*-Sulfonatocalix[7]arene and dicationic Diquat guest

Loredana Erra,*[a,c] Consiglia Tedesco,[a,b] Gavin Vaughan[c], Michela Brunelli[d], Francesco Troisi,[a] Carmine Gaeta,*[a,b] and Placido Neri[a,b]

e-mail: erra@esrf.fr *(L.R.);* cgaeta@unisa.it *(C.G.)*

Fax: (+39)089969603

[a] *Dipartimento di Chimica, Università di Salerno, via Ponte don Melillo, I-84084 Fisciano (Salerno), Italy.* [b] *NANO_MATES, Research Centre for NANOMAterials and nanoTEchnology at Salerno University, Università di Salerno, Via Ponte don Melillo, I-84084 Fisciano (SA), Italy.* [c] *ESRF (European Synchrotron Radiation Facility), 6 rue Jules Horowitz, BP220, 38043 Grenoble, France.* [d] *ILL Institut Laue-Langevin, BP 156, 38042 Grenoble Cedex 9, France.*

General Comments. ESI(+)-MS measurements were performed on a Micromass Bio-Q triple quadrupole mass spectrometer equipped with electrospray ion source, using a mixture of H_2O/CH_3CN (1:1) and 5% HCOOH as solvent. All chemicals were reagent grade and were used without further purification.

Reaction temperatures were measured externally; reactions were monitored by TLC on Merck silica gel plates (0.25 mm) and visualized by UV light and sprying with H_2SO_4-$Ce(SO_4)_2$. *p-tert*-butylcalix[7]arene was prepared following the procedure reported by Gutsche. [1] *p*-sulfonatocalix[7]arene was prepared following the procedure reported recently by us. [2] NMR spectra were recorded on Bruker Avance-400 spectrometer [400 (^{1}H) and 100.57 MHz (^{13}C)].

---

[1] D. R. Stewart, C. D. Gutsche, *J. Am. Chem. Soc.* 1999, **121**, 4136.

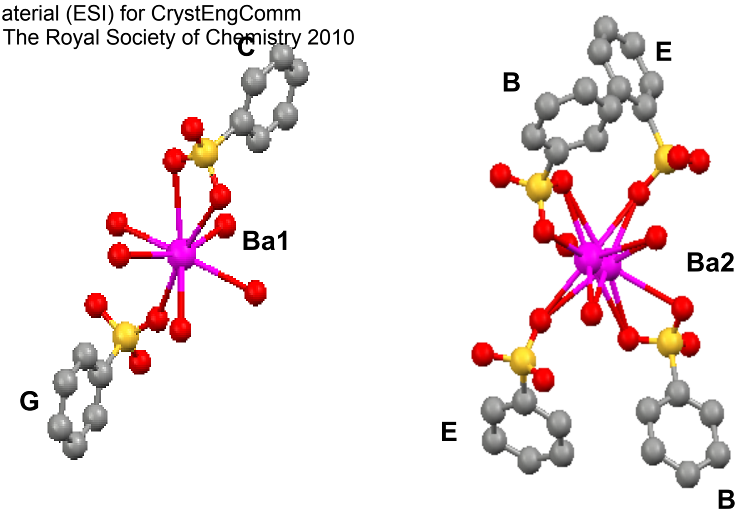
[2] C. Gaeta, T. Caruso, M. Mincolelli, F. Troisi, E. Vasca, P. Neri, *Tetrahedron* 2008, **64**, 5370.

Table S1. ϕ and χ torsion angles (°) for *p*-sulfonatocalix[7]arene **1** (distorted double pinched conformation) and *p*-tert-butylcalix[7]arene (double pinched conformation).[3]

Torsion angles	**1**	***p*-tert-butylcalix[7]arene**
C2 - C1 - C49 - C47	101.8(1.7)	98.5
C46 - C47 - C49 - C1	-86.8(1.8)	-89.0
C40 - C42 - C43 - C44	20(2)	-90.8
C39 - C40 - C42 - C43	66(2)	92.6
C33 - C35 - C36 - C37	89.6(1.9)	98.9
C32 - C33 - C35 - C36	-102.1(1.6)	-78.5
C26 - C28 - C29 - C30	81.8(1.8)	72.2
C25 - C26 - C28 - C29	-124.8(1.5)	-70.4
C19 - C21 - C22 - C23	108.0(1.6)	77.5
C18 - C19 - C21 - C22	-55.4(1.8)	-94.2
C12 - C14 - C15 - C16	85.9(1.7)	-94.4
C11 - C12 - C14 - C15	91.7(1.8)	91.1
C5 - C7 - C8 - C9	107.0(1.7)	89.2
C4 - C5 - C7 - C8	-95.35(1.8)	-98.4

---

[3] T. Harada, S. Shinkai, *J. Chem. Soc., Perkin Trans. 2*, 1995, **12**, 2231.



(a) **(b)**

Figure S1. (a) Ba1 and (b) Ba2 coordination in compound **1•2**. The aromatic rings belong to *p*-sulfonatocalix[7]arene molecules.

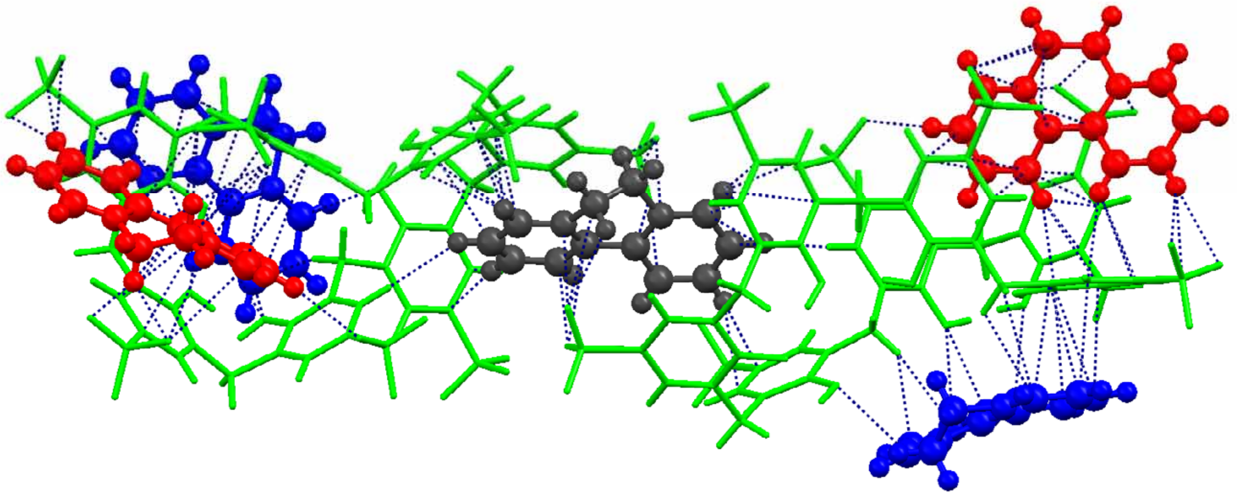


Figure S2. Closest contacts between *p*-sulfonatocalix[7]arene dimers and the three independent Diquat guest molecular cations.
The grey one is located on the crystallographic two-fold axis and is encapsulated between two ¾ cone trimeric subunits of two different *p*-sulfonatocalix[7]arene molecules forming a dimer.
The red one is enclosed in the tetrameric unit by electrostatic interactions, the blue one lies externally and with one pyridinium moiety almost parallel to the phenolic ring of the *p*-sulfonatocalix[7]arene molecule.