

Electronic supplementary information

Nonregular structure-property relationships for inclusion parameters of *tert*-butylcalix[5]arene

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**Supplemental TG data for pure *tert*-butylcalix[4]arene and *tert*-butylcalix[5]arene and
their clathrates, XRPD data for pure *tert*-butylcalix[5]arene**

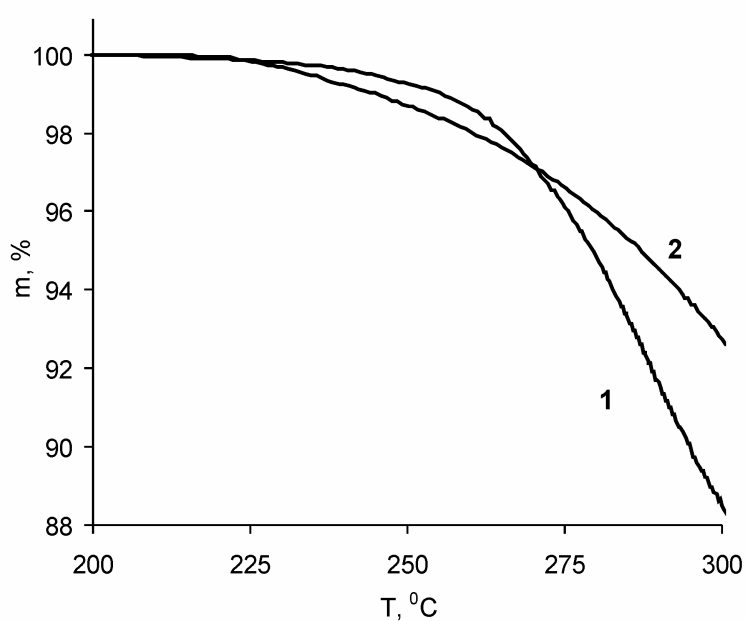


Fig. S1 TG curves determined under the static air conditions for pure hosts: *tert*-butylcalix[4]arene (1) and *tert*-butylcalix[5]arene (2).

Table S1 Supplemental TG data of clathrate decomposition*

№	Guest	$T_{0}^{\text{b.p.}} / ^\circ\text{C}$	$T_1 / ^\circ\text{C}$		$T_{\text{end}} / ^\circ\text{C}$	
			1	2	1	2
1	MeOH	64.7	32	-	152	-
2	MeCN	81.6	53	30	196	124
3	EtOH	78.4	66	33	189	121
4	EtCN	97	103	44	207	131
5	Acetone	56.2	94	43	173	163
6	CH ₂ Cl ₂	40.1	90	36 ^a	193	167
7	n-PrOH	97.2	93	42	171	163
8	i-PrOH	82.2	97	38	164	153
9	DMF ^b	153	133	42	220	156
10	1,2-C ₂ H ₄ Cl ₂	83.7	114	58 ^a	198	168
11	CHCl ₃	61.2	91	43 ^a	166	177
12	1,4-Dioxane	101.3	131	61	197	151
13	n-BuOH	117.7	120	51	158	156
14	Pyridine ^b	115.6	115	52	203	162
15	C ₂ HCl ₃	89	113	53 ^a	176	174
16	C ₆ H ₆ ^b	80.1	97	43	173	80
17	CCl ₄	76.8	93	42	161	130
18	c-Hexane ^b	81	99	79	164	159
19	n-Hexane	68.7	118	44	179	163
20	C ₂ Cl ₄	121	88	53 ^a	159	156
21	Toluene ^b	110.6	70.7	67	195.8	139
22	n-Heptane	98.4	123	47	191	147
23	n-Octane	124.7	108	43	198	168
24	i-Octane	99.3	107	102	158	132
25	n-Decane	195.8	60	59	112	170

* T_1 is onset temperature and T_{end} is the end point of decomposition; $T_{\text{b.p.}}$ is the boiling point of guest; ^adata for initially saturated clathrates after guest loss on the air at room temperature (20°C); ^bdata of simultaneous TG/DSC experiment under dynamic argon atmosphere.

TG data determined under static air conditions for clathrates stable on the air at room temperature (20°C):

2 with pyridine: $T_1 = 48^\circ\text{C}$, $T_{\text{end}} = 155^\circ\text{C}$, $T_{\text{max1}} (T_{\text{max2}}) = 61 (139)^\circ\text{C}$, $\Delta m_1 = 1.9\%$, $\Delta m = 15.8\%$;
 2 with benzene: $T_1 = 41^\circ\text{C}$, $T_{\text{end}} = 144^\circ\text{C}$, $T_{\text{max}} = 129^\circ\text{C}$, $\Delta m = 15.01\%$;
 2 with c-hexane: $T_1 = 50^\circ\text{C}$, $T_{\text{end}} = 158^\circ\text{C}$, $T_{\text{max}} = 87 (138)^\circ\text{C}$, $\Delta m_1 = 11.18\%$, $\Delta m = 24.51\%$,
 2 with toluene: $T_1 = 78^\circ\text{C}$, $T_{\text{end}} = 166^\circ\text{C}$, $T_{\text{max}} = 123^\circ\text{C}$, $\Delta m = 15.51\%$;
 1 with toluene: $T_1 = 81^\circ\text{C}$, $T_{\text{end}} = 193^\circ\text{C}$, $T_{\text{max}} = 119 (161)^\circ\text{C}$, $\Delta m_1 = 6.30\%$, $\Delta m = 12.72\%$.

where T_{max1} , T_{max2} – temperatures of DTG peaks for the first and second decomposition steps, respectively; Δm_1 – mass loss at the first decomposition step.

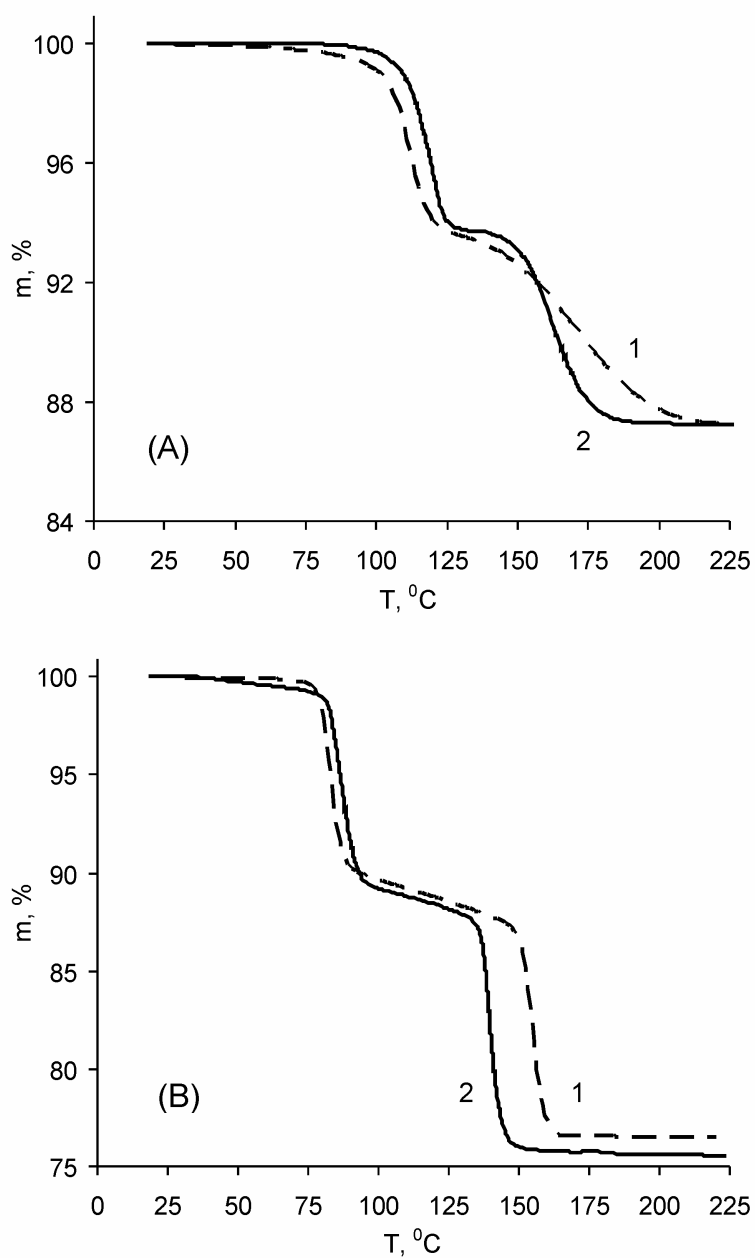


Fig. S2 Comparison of TG curves obtained for clathrates of host **1** with toluene (A) and of host **2** with cyclohexane (B) under dynamic argon (1) and static air (2) conditions.

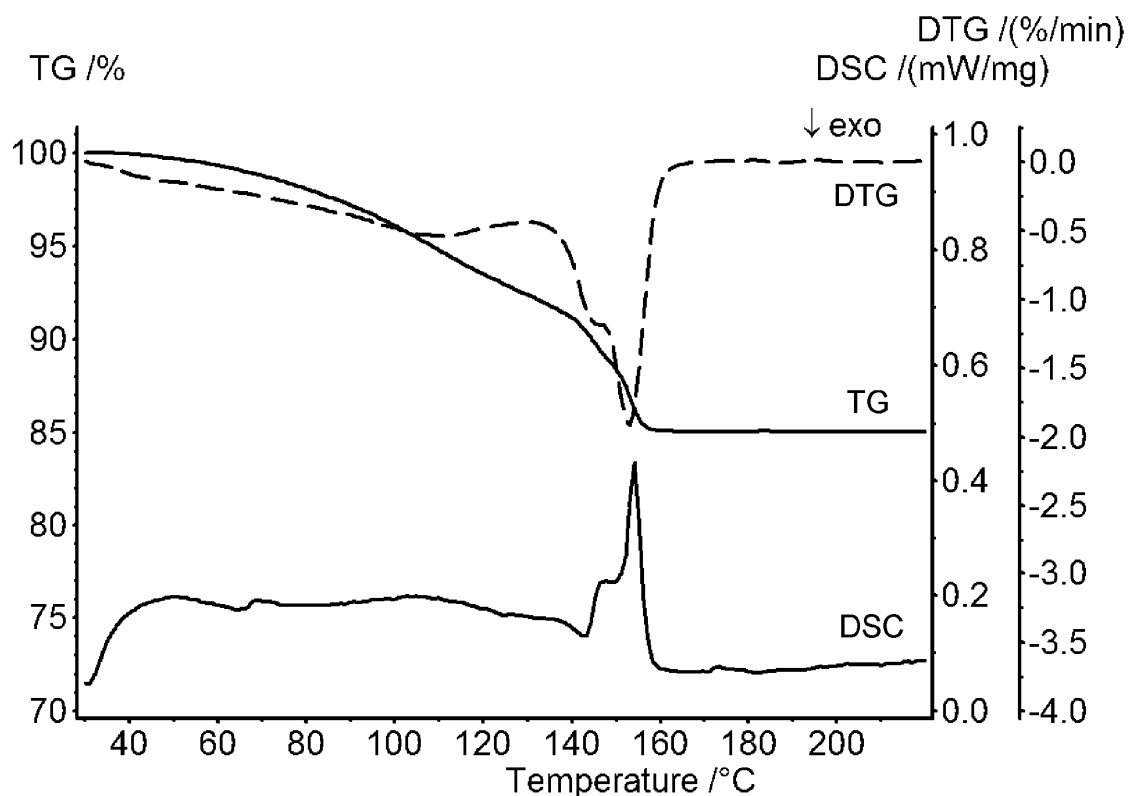


Fig. S3 The data of simultaneous TG/DSC experiment for saturated clathrate of *tert*-butylcalix[5]arene (**2**) with DMF.

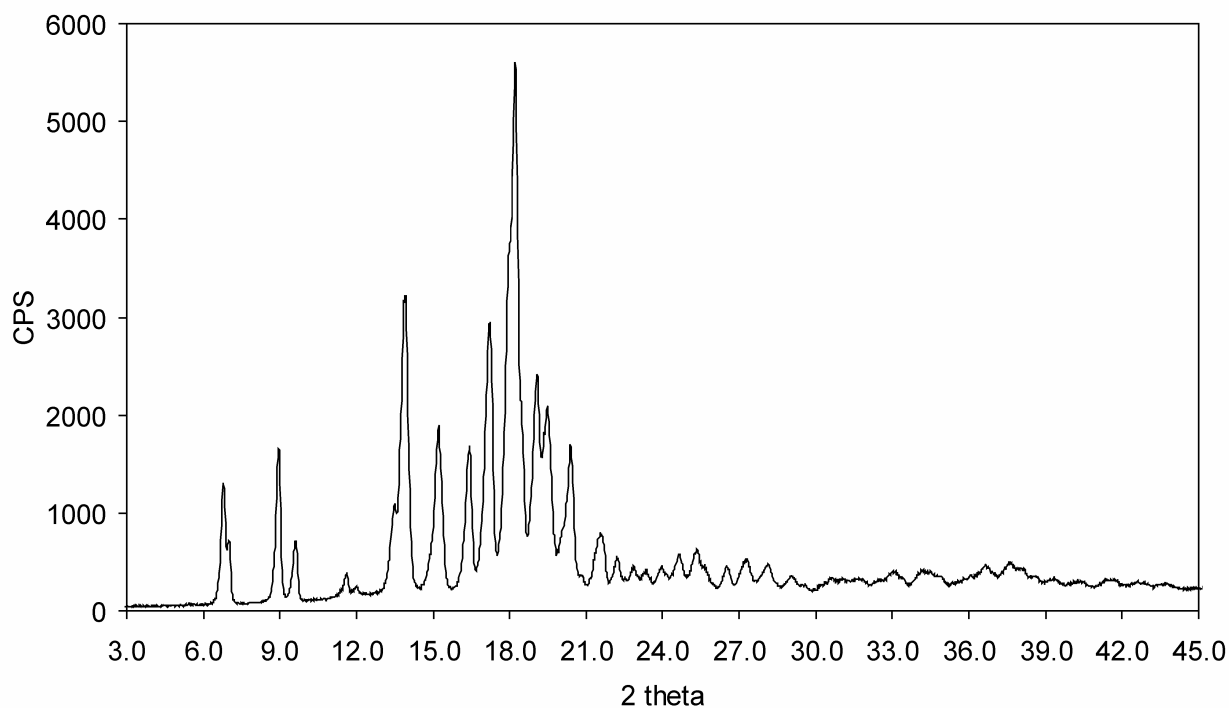


Fig. S4 X-ray powder diffraction patterns (XRPD) from *tert*-butylcalix[5]arene powder previously heated during 8 h at 200°C in vacuum (100 Pa). The powder sample was not subject of further other treatment. XRPD data were collected on a Bruker D8 Advance (Bruker AXS)

with Cu K α (40 kV, 30 mA) radiation, 2θ step size 0.02°, scan speed 1 step per 3 sec, sample rotation rate of 30 rpm, X-ray tube with fixed slit set of 1 mm* variable V20.