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Supporting Information

X-ray diffraction, FT-IR and ^{13}C CP/MAS NMR structural studies of solvated and desolvated C-methylcalix[4]resorcinarene

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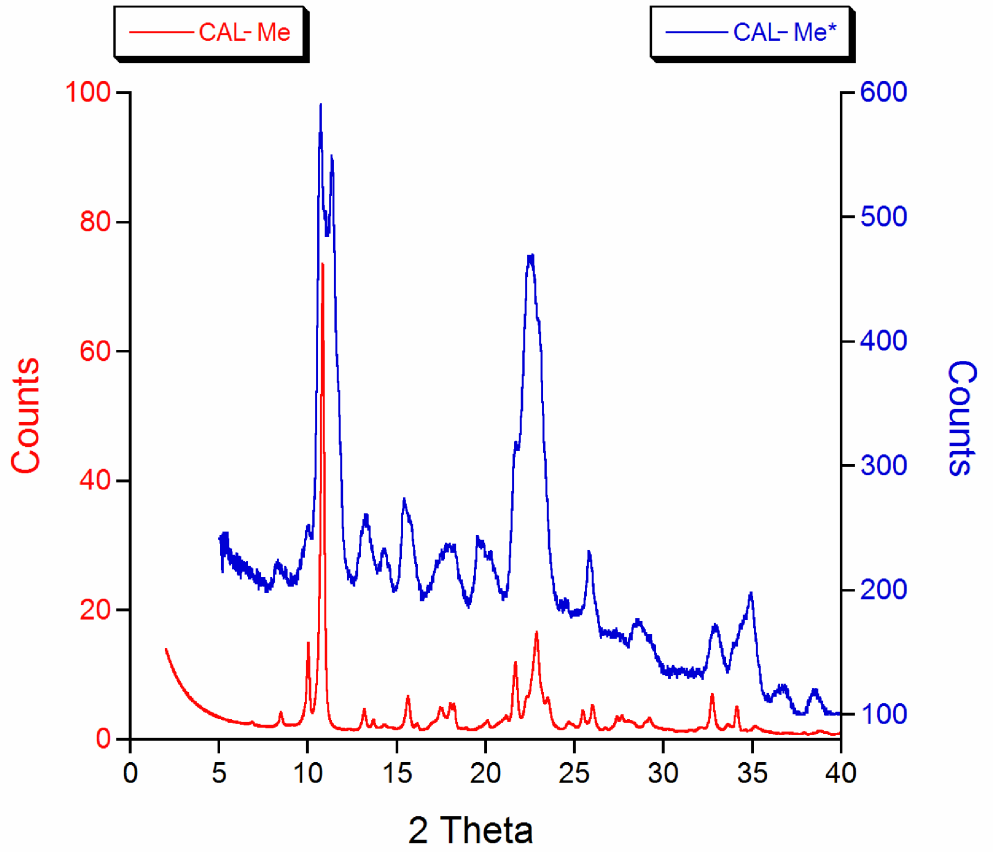

Figure 1S. Powder X-ray diffractograms of solvated CAL-Me† (red) and desolvated CAL-Me* (blue) forms C-methylcalix[4]resorcinarene.

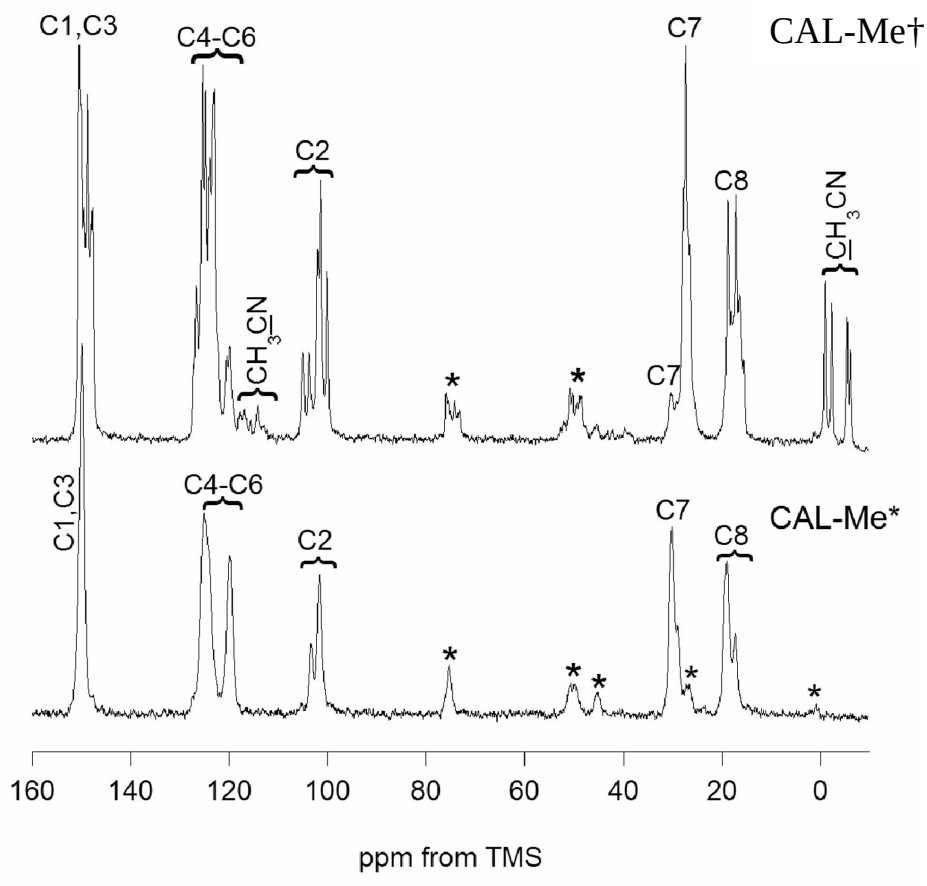

Figure 2S. ^{13}C CP/MAS NMR spectra of solvated (CAL-Me†) and desolvated (CAL-Me*) forms of C-methylcalix[4]resorcinarene recorded with the contact time of 8 ms.

Table 1S. Parameters of the $^{1}H \rightarrow {}^{13}C$ CP NMR kinetics for the solvated CAL-Me† form of C-methylcalix[4]resorcinarene, obtained using the KaleidaGraph program and the non-classical CP model. The calculation was performed only for prominent peaks.

δ / ppm	Assignment	λ	T_2 / μs	T_{df} / ms	$T_{1\rho}^{H}$ / ms
		CH_3			
-4.7	CH_3CN	0.51 ± 0.02	46 ± 3	4.3 ± 0.6	120 ± 20
-4.0	CH_3CN	0.42 ± 0.02	45 ± 3	3.6 ± 0.6	120 ± 20
-0.9	CH_3CN	0.45 ± 0.01	50 ± 3	2.7 ± 0.2	130 ± 20
0.5	CH_3CN	0.45 ± 0.01	48 ± 3	2.3 ± 0.2	110 ± 10

17.9	C8	0.39 ± 0.01	51 ± 2	1.9 ± 0.1	92 ± 9
18.5	C8	0.38 ± 0.02	50 ± 2	1.4 ± 0.1	120 ± 10
20.2	C8	0.37 ± 0.02	52 ± 2	0.79 ± 0.07	90 ± 8
			CH		
28.8	C7	0.42 ± 0.01	19 ± 1	1.56 ± 0.09	70 ± 5
31.8	C7	0.42 ± 0.02	18 ± 1	1.1 ± 0.2	8.5 ± 0.5
101.6	C2	0.47 ± 0.01	17.6 ± 0.9	1.85 ± 0.08	60 ± 6
102.9	C2	0.39 ± 0.01	17 ± 1	1.2 ± 0.2	67 ± 9
103.5	C2	0.38 ± 0.02	17 ± 1	1.1 ± 0.2	61 ± 8
105.3	C2	0.35 ± 0.01	16 ± 1	1.4 ± 0.1	110 ± 10
106.5	C2	0.44 ± 0.02	18 ± 2	1.6 ± 0.1	140 ± 20
121.3	C5	0.65 ± 0.02	20 ± 3	0.46 ± 0.03	57 ± 5
			CH + carbons without adjacent hydrogens[a]		
124.5	C4, C5, C6	0.56 ± 0.03	68 ± 8	1.5 ± 0.2	[b]
125.7	C4, C5, C6	0.43 ± 0.03	27 ± 3	0.6 ± 0.1	[b]
126.3	C4, C5, C6	0.47 ± 0.04	55 ± 6	1.0 ± 0.1	~ 70
126.9	C4, C5, C6	0.53 ± 0.03	55 ± 6	1.0 ± 0.1	73 ± 3
128.2	C4, C5, C6	0.49 ± 0.03	35 ± 4	0.56 ± 0.09	~ 70
			Carbons without adjacent hydrogens		
149.2	C1, C3	0.50 ± 0.02	88 ± 5	1.6 ± 0.1	130 ± 20
150.2	C1, C3	0.49 ± 0.01	132 ± 5	2.0 ± 0.1	115 ± 4
152.0	C1, C3	0.42 ± 0.02	140 ± 6	1.5 ± 0.1	120 ± 10

[a]Overlapped peaks. [b]Too large fitting errors.