Supporting Information

Stuffed Pumpkins: Mechanochemical Synthesis of Host-Guest Complexes with Cucurbit[7]uril

Martin Dračínský,^a Carina Santos Hurtado,^a Eric Masson,^{b*} and Jiří Kaleta^{a*}

 ^a Institute of Organic Chemistry and Biochemistry of the Czech Academy of Sciences, Flemingovo nám. 2, 160 00 Prague 6, Czech Republic.
^b Department of Chemistry and Biochemistry, Ohio University, Athens, Ohio 45701, United States.

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EXPERIMENTAL SECTION

Chemicals. Guest molecules 1–4 were purchased and used without additional purification. The CB[7] sample was prepared and purified using a method thoroughly described by Isaacs and coworkers.¹ Acetone diffusion into an aqueous solution of mainly CB[7] (with traces of other macrocycles) afforded CB[7] as an amorphous white solid, that was filtered and dried under high vacuum. This compound is obtained as solvate with variable amounts of H₂O and HCl. Its apparent molecular weight (sample-dependent; 1400–1700 g/mol) was determined by ¹H NMR titration with an aqueous solution of adamantylammonium chloride (**2**) with a precise concentration.

Ball milling. The setup used in this study consisted of a NARVA Brand-Erbisdorf Vibrator containing 220V, 30W, 50 Hz engine (Figure S1), which was equipped with a stainless-steel spherical chamber (27 mm diameter) carrying two stainless-steel balls (8 mm diameter, 4 g each). Neat CB[7] (40 mg, ~0.025 mmol) was ball-milled using this vibrator for 5 min at room temperature (no temperature control was applied during milling). Subsequently, neat guest 1–4 was added and the mixture was ball-milled 4×5 min. The milling chamber was opened after each cycle and the glassy solid material was thoroughly scratched from the mill walls. Each scratching usually lasts ca 5 min and the whole ball-milling procedure thus did not exceed 60 min.



Figure S1. Stainless-steel milling chamber with two balls: closed (A), opened (B), and the complete ball-mill used in this study (C).

Solution phase NMR experiments. Solution NMR spectra were obtained using a Bruker Avance IIITM HD 500 MHz spectrometer operating at 125.7 MHz for ¹³C and 500 MHz for ¹H. ¹H spectra were referenced to the solvent residual peak (HDO, $\delta = 4.8$ ppm). The assignment of hydrogen and carbon spectra was based on a combination of 1D and 2D experiments (¹H, ¹³C-APT, ¹H,¹H-COSY, ¹H,¹³C-HMBC, ¹H,¹³C-HSQC and ¹H,¹³C-HMBC). All spectra were measured at 25 °C.

Solid-state NMR experiments. High-resolution ¹H and ¹³C solid-state NMR spectra were obtained using a JEOL ECZ600R spectrometer operating at 150.9 MHz for ¹³C and 600.2 MHz for ¹H. Samples were packed into 1 and 3.2 mm magic angle spinning rotors (MAS) and measurements were taken at MAS rates of 70 and 18 kHz, respectively. ¹³C spectra of compounds

1–3, CB[7] and their host-guest complexes were measured using cross polarization (CP). ¹³C spectra of adamantane (4) and its complexes were measured with direct carbon excitation. Note that the CP technique relies on dipolar interactions, which are suppressed in adamantane due to its fast reorientation in its solid form. Proton and carbon chemical shifts were referenced to the signal of sodium trimethylsilylpropanesulfonate (DSS), which was used as an internal or external standard ($\delta({}^{1}H) = \delta({}^{13}C) = 0$ ppm). The ramped amplitude shape pulse was used during the cross-polarization. The contact time for CP was 5 ms and the relaxation delays were estimated from ¹H saturation recovery experiments and ranged from 0.5 s for the inclusion complexes to 27 s for neat toluidine hydrochloride (3). The assignment of the signals was done with the help of a 2D double-CP experiment and 2D CP-INEPT experiment showing C–H correlations via dipolar coupling or *J*-coupling, respectively. These 2D experiments were taken at MAS rates of 70 kHz. ¹H–¹H DQ–SQ MAS spectra were recorded using the rotor-synchronized BABA (back-to-back) recoupling pulse sequence at MAS rates of 70 kHz.²

Elemental analysis. CB[7] samples were analyzed with a PE 2400 Series II CHNS/O instrument (Perkin Elmer, USA). The driest sample returned C 34.58%, H 4.79%, N 26.38%, Cl 7.38% (theoretical values for anhydrous CB[7] C₄₂H₄₂N₂₈O₁₄: C 43.38%, H 3.64%, N 33.72%). The sample thus contains approximately 11 water and 3 HCl molecules per CB[7] unit (theoretical values for C₄₂H₆₇N₂₈O₂₅Cl₃: C 34.31%, H 4.59%, N 26.67%, Cl 7.23%). The apparent molecular weight of this sample, as determined by elemental analysis, is 1471 g/mol, in excellent agreement with the molecular weight obtained by NMR titration (1485 g/mol).

NMR SPECTROSCOPY



(1) NMR Experiments in Solution





Figure S3. ¹H NMR spectra of D₂O solutions of (a) free guest **2**, (b) a 1:2 mixture of CB[7] and **2**, (c) a 2:1 mixture of CB[7] and **2**, and (d) free CB[7].



Figure S4. Part of ¹³C NMR spectra in D_2O of (a) free guest **2**, (b) a 1:2 mixture of CB[7] and **2**, (c) a 2:1 mixture of CB[7] and **2**, and (d) free CB[7].



Figure S5. ¹H NMR spectra in D_2O of (a) free guest **3**, (b) a 1:1 mixture of **3** and CB[7], and (c) free CB[7].



Figure S6. ¹³C attached-proton-test (APT) NMR spectra in D_2O of (a) free guest **3**, (b) a 1:1 mixture of **3** and CB[7], and (c) free CB[7]. *Tert*-butyl alcohol was used as an internal standard.



Figure S7. ¹H NMR spectra in D_2O of (a) free guest **4**, (b) a 1:1 mixture of **4** and CB[7], and (c) free CB[7].



Figure S8. ¹³C NMR spectra in D_2O of (a) free guest **4**, (b) a 1:1 mixture of **4** and CB[7], and (c) free CB[7].

(2) Solid-State NMR (ssNMR) Experiments

The most common ssNMR experiments employ the magic-angle spinning (MAS) technique with spinning speed of 10–30 kHz to suppress the chemical-shift anisotropy, and high-power decoupling to suppress dipolar heteronuclear interactions between the measured nuclei and protons. This technique enables detecting, for example, ¹³C ssNMR spectra with relatively narrow signals, which resemble those obtained for solutions. However, the detection of proton ssNMR spectra is much more challenging because of the presence of very strong homonuclear dipolar couplings in proton-rich organic molecules. Therefore, we measured ssNMR experiments with ultra-fast MAS speed of 70 kHz, which leads to a significant narrowing of proton signals. This signal narrowing has also been exploited in proton-detected two-dimensional (2D) ssNMR experiments, which were necessary for the detection of proton signals of guest molecules overlapped with signals of the host and for an unequivocal confirmation of the host-guest complex formation.

SS-NMR experiments can also serve for an estimation of intermolecular distances. There are several approaches to this task, for example, measurements of heteronuclear dipolar couplings or detection of homo- or heteronuclear correlations based on dipolar interactions,^{3, 4} proton-detected ¹H-¹⁴N correlations,⁵ dynamics of cross-polarization,⁶ or comparison of calculated and experimental chemical shifts.⁷ Some of these approaches have been also used for structural characterization of host-guest inclusion complexes.^{6, 8, 9} These techniques might be also applied in the future for an insight into the geometries of the CB[7] inclusions studied in this work.

(a) Adamantane (1). Ball milling of crystalline compound 1 does not lead to any change of its ssNMR carbon chemical shifts (Figure S9). This indicates that the ball milling does not induce a polymorphic change. Intermolecular interactions, and hence spatial proximity, between CB[7] and

guest molecules can be observed in spectra that exploit dipolar (through-space) coupling. Figures S10 and S11 show ¹H homonuclear double-quantum (DQ) – single-quantum (SQ) correlation spectra, where correlations between the adamantane (**1**) and CB[7] hydrogen atoms (overlapping $H_a + H_c$) are clearly visible. The experiment shown in Figure S10 was carried out in the presence of 1 equiv. CB[7] leading to observations of encapsulated **1** only. On the other hand, the experiment shown in Figure S11 was carried out in the presence of 0.5 equiv. CB[7] that clearly differentiates between the signals of free and CB[7]-bound adamantane (**1**). An additional strong autocorrelation signal of the free adamantane nuclei indicates larger domains of neat **1**. Table S1 summarizes proton and carbon atoms chemical shifts of free compound **1** and its inclusion in CB[7] both in solution and in the solid state.



Figure S9. ¹³C CP-MAS spectra of (a) crystalline **1** and (b) ball-milled **1**.



Figure S10. ¹H⁻¹H DQ–SQ MAS spectrum of a milled 1:1 mixture of **1** and CB[7] obtained using a rotor-synchronized BABA (back-to-back) recoupling at a MAS rate of 70 kHz.



Figure S11. ¹H⁻¹H DQ–SQ MAS spectrum of a milled 2:1 mixture of **1** and CB[7] obtained using a rotor-synchronized BABA (back-to-back) recoupling at a MAS rate of 70 kHz.

Cmpd.	Nucleus		H1 (ppm)	H2 (ppm)
1	$^{1}\mathrm{H}$	Solution	-	-
	$^{1}\mathrm{H}$	Solid	1.6	1.6
1 ⊂CB[7]	$^{1}\mathrm{H}$	Solution	1.2	1.1
	$^{1}\mathrm{H}$	Solid	1.0	1.0
Cmpd.	Nucleus		C1 (ppm)	C2 (ppm)
Cmpd. 1	Nucleus ¹³ C	Solution	C1 (ppm)	C2 (ppm)
Cmpd. 1	Nucleus ¹³ C ¹³ C	Solution Solid	C1 (ppm) - 28.8	C2 (ppm) - 37.9
Cmpd. 1 1⊂CB[7]	Nucleus ¹³ C ¹³ C ¹³ C ¹³ C	Solution Solid Solution	C1 (ppm) - 28.8 27.4	C2 (ppm) - 37.9 37.1

Table S1. ¹H and ¹³C chemical shifts of free compound **1** and its CB[7] inclusion complex.

(b) 1-Adamantylammonium chloride (2). Ball milling of neat compound 2 does not induce any polymorphic change (Figure S12). The ¹H spectra of ball-milled 2 and of a 1:2 and 2:1 mixtures of 2 with CB[7] are shown in Figure S13. All proton signals of 2 are shifted upfield upon milling with CB[7]. The chemical-shift change is close to that observed in solution. The ¹³C ssNMR spectra of neat 2, CB[7] and their mixtures are shown in Figure S14. Like in solution, the chemical shift changes upon complexation are small (within 1 ppm). The assignment of the proton and carbon signals is based on a 2D CP-INEPT experiment showing C–H correlations via heteronuclear *J*-coupling (Figure S15). Table S2 summarizes proton and carbon chemical shifts of free compound 2 and its inclusion in CB[7] both in solution and in the solid state.

Intermolecular interactions between CB[7] and guest molecules can be observed in spectra that exploit dipolar (through-space) coupling. Figure S16 shows a ¹H homonuclear double-quantum (DQ) – single-quantum (SQ) correlation spectrum, where correlations between the adamantyl hydrogens and hydrogens of CB[7] are clearly visible, which undoubtedly confirms a spatial proximity of the two species. Apart from these intermolecular interactions, intramolecular autocorrelation signals are also present in the spectrum. Similarly, a proton-detected heteronuclear 2D double-CP experiment showing C–H correlations via dipolar coupling shows correlations between the adamantyl protons and CB[7] methylene and carbonyl carbons (highlighted by red circles in Figure S17). The spectrum shown in Figure S17 was obtained with contact times for cross-polarization of 3 ms; the intermolecular correlations are not apparent in spectra obtained with shorter contact times (1 or 2 ms).



Figure S12. ¹³C CP-MAS spectra of (a) crystalline 2, and (b) ball-milled 2.



Figure S13. ¹H ssNMR spectra of ball-milled (a) **2**, (b) a 1:2 mixture of CB[7] and **2**, (c) a 2:1 mixture of CB[7] and **2**, and (d) neat CB[7].



Figure S14. ¹³C CP-MAS spectra of ball-milled (a) **2**, (b) a 1:2 mixture of CB[7] and **2**, (c) a 2:1 mixture of CB[7] and **2**, and (d) neat CB[7].



Figure S15. 2D CP-INEPT ssNMR spectrum of a ball-milled mixture of CB[7] and **2** in 2:1 ratio showing C–H correlations via heteronuclear *J*-coupling, measured at MAS speed of 70 kHz.



Figure S16. ¹H⁻¹H DQ–SQ MAS spectrum of a mixture of **2** and CB[7] obtained using a rotor-synchronized BABA (back-to-back) recoupling at MAS rate of 70 kHz.



Figure S17. ¹H-detected heteronuclear 2D double-CP spectrum of a mixture of CB[7] and **2** showing C–H correlations via dipolar coupling. Intermolecular correlations are highlighted by red circles.

Cmpd.	Nucleus		H2	H3	H4a	H4b	NH3
			(ppm)	(ppm)	(ppm)	(ppm)	(ppm)
2	$^{1}\mathrm{H}$	Solution	1.9	2.2	1.8	1.7	-
	$^{1}\mathrm{H}$	Solid	2.1	2.5	1.7	1.7	8.6
2 ⊂CB[7]	¹ H	Solution	1.2	1.5	1.2	0.9	-
	$^{1}\mathrm{H}$	Solid	1.4	1.7	1.2	1.2	7.8
Cmpd.	Nucleus		C1	C2	C3	C4	
			(ppm)	(ppm)	(ppm)	(ppm)	
2	¹³ C	Solution	52.2	39.9	28.7	34.7	
	¹³ C	Solid	54.2	40.8	29.6	36.9	
2 ⊂CB[7]	¹³ C	Solution	52.2	40.3	28.4	34.4	
	¹³ C	Solid	_a	41.0	29.5	35.9	

Table S2. ¹H and ¹³C chemical shifts of free compound 2 and its CB[7] inclusion complex.

^aOverlapped with CB[7] signal.

To confirm that ball milling leads to the same host-guest inclusion complex as the one prepared in solution, the ¹³C ssNMR spectrum of the sample obtained after ball milling is compared in Figure S18 with the spectrum of the mixture obtained after evaporation of the water solution.



Figure S18. Comparison of ¹³C ssNMR spectra of (a) a 2:1 mixture of CB[7] and **2** after ball milling with (b) a mixture of CB[7] and **2** after drying the aqueous solution.

(c) Toluidine hydrochloride (3). Similarly to guests 1 and 2, ball-milling of crystalline compound 3 does not lead to any change of its ssNMR carbon chemical shifts (Figure S19). This indicates that the ball milling does not induce a polymorphic change. Due to intermolecular interactions in solid 3, the proton chemical shift of the methyl group (1.0 ppm) differs significantly from that obtained in D₂O solution (2.3 ppm). The chemical shifts of solid 3 change significantly upon milling with CB[7] (Figure S20). The signal of NH₃ and the aromatic protons are shifted upfield by ca 1.6 ppm and 0.7 ppm, respectively. The methyl shifts downfield from 1.0 to 1.6 ppm, i.e. close to the chemical shift measured in an aqueous solution of $3 \subset CB[7]$. Carbon chemical shifts of compound 3 also change upon ball milling (Figure S21). The assignment of the proton and carbon signals is confirmed by a 2D heteronuclear experiment showing C–H correlations via heteronuclear *J*-coupling (Figure S22). Table S3 summarizes proton and carbon chemical shifts of free compound 3 and its inclusion in CB[7] both in solution and in the solid state.



Figure S19. ¹³C CP-MAS spectra of (a) crystalline 3 and (b) ball-milled 3.



Figure S20. ¹H ssNMR spectra of ball-milled (a) neat **3**, (b) a 1:2 mixture of CB[7] and **3**, (c) a 2:1 mixture of CB[7] and **3**, and (d) CB[7].



Figure S21. ¹³C ssNMR spectra of ball-milled (a) **3**, (b) a 1:2 mixture of CB[7] and **3**, (c) a 2:1 mixture of CB[7] and **3**, and (d) neat CB[7].



Figure S22. 2D CP-INEPT ssNMR spectrum of a 1:2 mixture of CB[7] and **3** after ball milling showing C–H correlations via heteronuclear *J*-coupling, measured at MAS speed of 70 kHz.

Cmpd.	Nucleus		H2	H3	CH ₃	NH ₃	
			(ppm)	(ppm)	(ppm)	(ppm)	
3	$^{1}\mathrm{H}$	Solution	7.2	7.3	2.3	-	
	$^{1}\mathrm{H}$	Solid	7.3	7.3	1.0	10.4	
3 ⊂CB[7]	$^{1}\mathrm{H}$	Solution	6.5	6.6	1.8	-	
	$^{1}\mathrm{H}$	Solid	6.8	6.4	1.6	8.8	
Cmpd.	Nucleus		C1	C2	C3	C4	CH ₃
			(ppm)	(ppm)	(ppm)	(ppm)	(ppm)
3	¹³ C	Solution	126.8	122.5	130.4	139.6	20.0
	^{13}C	Solid	126.9	124.0	131.6	141.7	19.6
3 ⊂CB[7]	$^{13}\mathrm{C}$	Solution	125.7	121.3	128.6	140.3	20.0
	^{13}C	Solid	_a	121.8	128.9	139.5	20.3

Table S3. ¹H and ¹³C chemical shifts of free compound 3 and its CB[7] inclusion complex.

^{*a*}Overlapped by C-1 or C-2.

To confirm that the ball milling leads to the same host-guest inclusion complex as the one in solution, the ¹³C ssNMR spectrum of the sample obtained after the ball milling is compared in Figure S23 with the spectrum of the mixture obtained after evaporation of the aqueous solution.



Figure S23. Comparison of ¹³C ssNMR spectra of (a) a 2:1 mixture of CB[7] and **3** after ball milling with (b) a mixture of CB[7] and **3** after drying the aqueous solution.

(d) Benzene-1,4-diammonium dichloride (4). Similarly, the chemical shifts of 4 change upon milling with CB[7], thereby indicating that an inclusion complex is formed. However, an overlap between the signals of guest 4 and CB[7] are observed, and correlation experiments are not possible. The assignment of the proton signals is confirmed by a 2D heteronuclear experiment showing C–H correlations via heteronuclear *J*-coupling (Figure S27). Table S4 summarizes proton and carbon chemical shifts of free compound 4 and its inclusion in CB[7] both in solution and in the solid state. Surprisingly, another set of signals of 4 (apart from those obtained after ball milling) can be observed in a sample obtained after evaporation of a 1:1 mixture of 4 and CB[7]. We speculate that these signals might correspond to deprotonated compound 4 or to a product of oxidation (1,4-diiminobenzene).



Figure S24. ¹³C CP-MAS spectra of (a) crystalline 4 and (b) ball-milled 4.



Figure S25. Comparison of ¹H ssNMR spectra of ball-milled (a) **4**, (b) a 1:2 mixture of CB[7] and **4**, and (c) a 2:1 mixture of CB[7] and **4**; (d) a 1:1 mixture of CB[7] and **4** after drying the aqueous solution, and (e) neat CB[7] after ball milling.



Figure S26. Comparison of ¹³C ssNMR spectra of ball-milled (a) **4**, (b) a 1:2 mixture of CB[7] and **4**, and (c) a 2:1 mixture of CB[7] and **4**; (d) a 1:1 mixture of CB[7] and **4** after drying the aqueous solution, and (e) neat CB[7] after ball milling.



Figure S27. 2D CP-INEPT ssNMR spectrum of a mixture of 4 and CB[7] after ball milling, showing C-H correlations via heteronuclear J-coupling, measured at MAS speed of 70 kHz.

Table S4. ¹	^I H and	¹³ C che	emica	l shifts	of free comp	pound 3	and its	CB[7]	inclu	sion	comp	olex.
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Cmpd.	Nucleus		H-arom (ppm)	NH ₃ (ppm)
4	$^{1}\mathrm{H}$	Solution	7.5	-
	$^{1}\mathrm{H}$	Solid	8.1 and 6.8	9.9
4 ⊂CB[7]	$^{1}\mathrm{H}$	Solution	6.7	-
	$^{1}\mathrm{H}$	Solid	6.6	8.5
Cmpd.	Nucleus		C1 (ppm)	C2 (ppm)
Cmpd. 4	Nucleus ¹³ C	Solution	C1 (ppm) 131.2	C2 (ppm) 124.3
Cmpd. 4	Nucleus ¹³ C ¹³ C	Solution Solid	C1 (ppm) 131.2 131.9	C2 (ppm) 124.3 126.0
Cmpd. 4 4⊂CB[7]	Nucleus ¹³ C ¹³ C ¹³ C	Solution Solid Solution	C1 (ppm) 131.2 131.9 131.0	C2 (ppm) 124.3 126.0 123.4

MASS SPECTROMETRY

High-resolution mass spectra of supramolecular complexes $2-4 \subset CB[7]$ in H₂O solutions were determined by the ESI(+) MS technique (Table S5). Complex $1 \subset CB[7]$ fragmented during ionization and only individual components were detected.

	1 0	I
Cmpd.	Calcd. Mass (m/z)	Exp. Mass (m/z)
1 ⊂CB[7]	1300.2255 (z = 1)	Not detected
2 ⊂CB[7]	1314.4869 (z = 1)	1314.4874
3 ⊂CB[7]	1270.4243 (z = 1)	1270.4248
4 ⊂CB[7]	636.2134 (z = 2)	636.2132

Table S5. Calculated and experimental masses of host-guest complexes.



Figure S28. HR-ESIMS spectrum of 1 \subset CB[7]. The highest peak corresponds to free CB[7], calculated mass C₄₂H₄₄O₁₄N₂₈²⁺ = 582.17904 (z = 2). The mass of the host-guest complex was not observed.



Figure S29. HR-ESIMS spectrum of 2 \subset CB[7]. The highest peak corresponds to the host-guest complex, calculated mass $C_{52}H_{60}O_{14}N_{29}^+ = 1314.4869$.



Figure S30. HR-ESIMS spectrum of **3** \subset CB[7]. The highest peak corresponds to the host-guest complex, calculated mass $C_{49}H_{52}O_{14}N_{29}^+ = 1270.4243$.



Figure S31. HR-ESIMS spectrum of 4 \subset CB[7]. The highest peak corresponds to the host-guest complex, calculated mass $C_{48}H_{52}O_{14}N_{30}^{2+} = 636.2134$ (z = 2).

DIFFERENTIAL SCANNING CALORIMETRY



Figure S32. Differential scanning calorimetry traces from -90 °C to 450 °C: (A) neat CB[7], (B) neat adamantane (1), and (C) complex 1 \subset CB[7]. Endotherms are negative peaks.



Figure S33. Differential scanning calorimetry traces from $-90 \degree C$ to 450 $\degree C$: (A) neat CB[7], (B) neat adamantyl-1-amine hydrochloride (2), and (C) complex 2 \subset CB[7]. Endotherms are negative peaks.



Figure S34. Differential scanning calorimetry traces from −90 °C to 450 °C: (A) neat CB[7], (B) neat adamantane (3), and (C) complex 3⊂CB[7]. Endotherms are negative peaks.



Figure S35. Differential scanning calorimetry traces from -90 °C to 450 °C: (A) neat CB[7], (B) neat adamantane (4), and (C) complex 4 \subset CB[7]. Endotherms are negative peaks.

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