Electronic Supplementary Information

Solution-mediated and single-crystal to single-crystal transformations of the cucurbit[6]uril host-guest complexes with dopamine

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Crystallization

The dopamine was purchased from Sigma Aldrich. CB6 was synthesized according to a literature procedure [A. Day et al., J. Org. Chem., 2001, 66, 8094]. CB6·10H2O (10 mg, 8.5 μmol) and MgCl2·6H2O (173 mg, 0.85 mmol) were dissolved in distilled water (1 ml) upon gentle heating. The solution of dopamine guest (2 equivalents) in 1 ml of water was layered over the solution of the macrocycle. The diffraction quality crystals of complexes I (prisms) and II (needles) were formed concomitantly after one day. After several days only crystals of complex III were present in the crystallization vial. The crystals of complex II undergo single-crystal to single-crystal desolvation when out of mother solution. Despite some cracking and higher mosaicity the dried crystals III gave a satisfactory diffraction pattern.

Single crystal X-ray diffraction

The crystals were selected under Paratone-N oil, mounted on the nylon loops and positioned in the cold stream on the diffractometer. Uniformity of the samples was checked by unit cell determination of several crystals for each sample. The X-ray data for complexes I-III were collected on a SuperNova Agilent diffractometer using CuKα radiation (λ = 1.54184 Å). The data were processed with CrysAlisPro. Structures were solved by direct methods and refined using SHELXL.2 The figures were prepared using Chimera.3

Crystal data for I: (C50H30N22O12)·(C6H2NO3)·Cl·13.5(H2O), Mr =1429.7, colorless prisms, orthorhombic, space group P212121, a = 12.6209(6), b = 15.8200(4), c = 30.3571(1) Å, V = 6061.2(4) Å³, Z = 4, ρcalc = 1.57 g cm⁻³, μ(CuKα) = 1.51 mm⁻¹, θmax = 68.2°, 19840 reflections measured, 11066 unique, 1072 parameters, R = 0.094, wR = 0.256 (R = 0.113, wR = 0.281 for all data), Goof = 1.02. CCDC 1954457.

Crystal data for II: (C50H30N22O12)·(C6H2NO3)·Cl·14(H2O), Mr =1438.7, colorless needles, trigonal, space group R-3, a = 50.711(2), c = 12.5325(8) Å, V = 27911(3) Å³, Z = 18, ρcalc = 1.54 g cm⁻³, μ(CuKα) = 1.49 mm⁻¹, θmax = 70.1°, 33038 reflections measured, 11712 unique, 1096 parameters, R = 0.094, wR = 0.279 (R = 0.110, wR = 0.306 for all data), Goof = 1.10. CCDC 1954458.

Crystal data for III: (C50H30N22O12)·(C6H2NO3)·Cl·7.333(H2O), Mr = 1318.6, colorless needles, trigonal, space group R-3, a = 48.442(2), c = 12.4117(4) Å, V = 25223(3) Å³, Z = 18, ρcalc = 1.56 g cm⁻³, μ(CuKα) = 1.50 mm⁻¹, θmax = 68.3°, 59616 reflections measured, 10226 unique, 964 parameters, R = 0.116, wR = 0.319 (R = 0.138, wR = 0.342 for all data), Goof = 1.12. CCDC 1954459.
**Thermal analysis**

The TG measurements were performed using STA 449 F1 Jupiter analyzer (Netzsch) coupled with quadrupole mass spectrometry QMS 403C Aéolos (Netzsch) and FT-IR spectrofotometer TENSOR 27 (Bruker) in helium flow. The heating rate was equal to 5 K/min.

The differential scanning calorimetry (DSC) curves were recorded using a Netzsch DSC 204 F1 Phoenix apparatus in argon flow. Aluminium crucible with a hole in a lid was used. The heating rate was equal to 5 K/min.

![TG and DSC curves](image)

**Fig. ESI-1** TG and DSC curves of cucurbit[6]uril (a and d), dopamine (b and e) and cucurbit[6]uril-dopamine complex III (c and f). The crystals of complex III were dried for 24 hours in air at ambient conditions prior to measurements.