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Isolation of the Saddle and Crown Conformers of Cyclotriveratrylene (CTV) Oxime

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Abstract
The individual crown and saddle conformers of cyclotriveratrylene (CTV) oxime have been isolated and characterized, and the equilibrium ratio determined in $CDCl_3$ and d_6-DMSO.

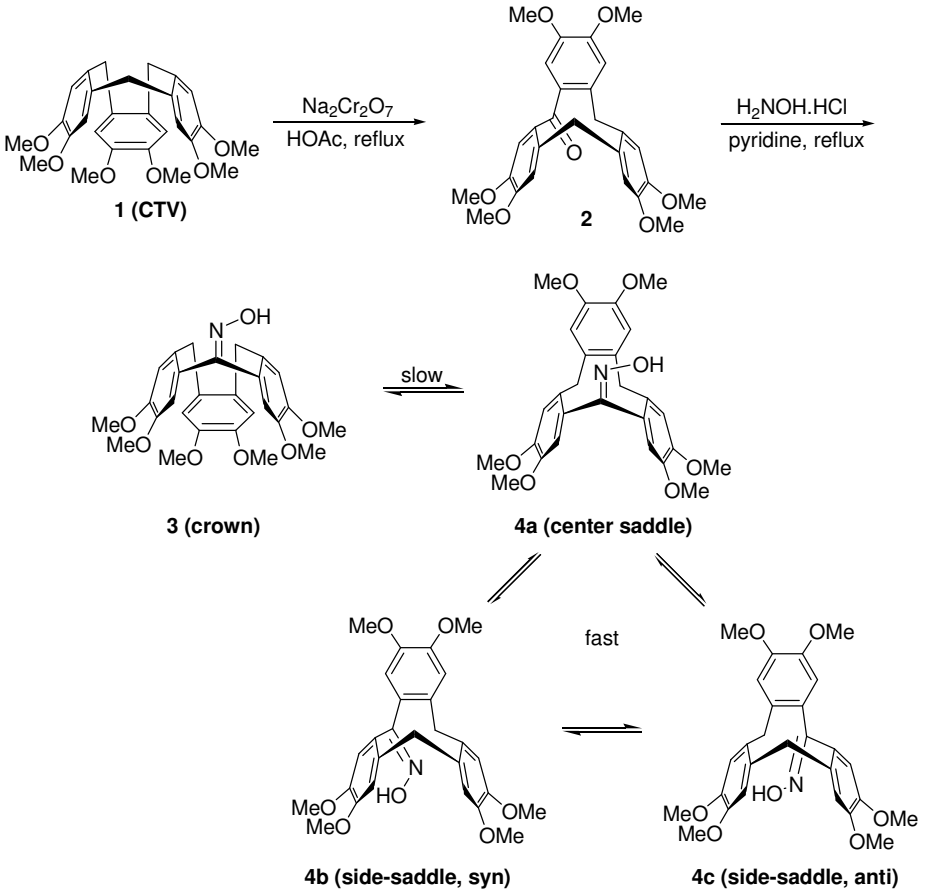
Supramolecular chemistry involves the formation of complex molecular entities that have the capacity to participate in specific molecular recognition of guest molecules,[1, 2] Cyclophanes are supramolecular structures comprised of aromatic units with bridging chains forming cage-like structures[3] and have found applications in synthetic receptors and molecular recognition, as models for intercalation, as building blocks for organic catalysts, in the preparation of crown ethers and cryptands[4] and as molecular scaffolds and as specific host molecules in the design of new pharmaceuticals[5-8] including cholesterol shuttles to modulate cholesterol metabolism[9] as potent human choline kinase (ChoK) inhibitors[10] and as inhibitors of HIV protease.[11]

An archetypal cyclophane scaffold commonly employed in supramolecular chemistry is the trimeric crown-shaped molecule cyclotriveratrylene (CTV, **1,** hexamethoxy tribenzocyclononene,),[12] a [1.1.1]orthocyclophane that is readily prepared from the trimerization of veratryl alcohol in acid. CTV has been extensively studied for its capability to bind a variety of small organic and organometallic guests within its bowl-shaped cleft.[13-15] Many clathrates of CTV have been structurally characterized, including DMSO and ethanol[16], chlorinated organics[17], xenon[18], lanthanides[19] organometallic complexes,[20] C60,[21] and anionic C70 dimers.[22] CTV has also been used for selective anion sensing,[23] and thioether derivatives of CTV have recently been employed as hosts to immobilize C60 onto gold surfaces.[24, 25] CTV has been employed as a supramolecular building block to construct more complex cage-like cryptophanes.[26] CTV derivatives have also been studied for their mesomorphic properties[27] and are capable of forming liquid crystals by themselves[28] and as complexes with C60.[28] CTV has also been studied as a crystal engineering tecton giving network structures of unusual topology.[29]

We are interested in the preparation of novel apex-functionalized derivatives of CTV. Toward this end we synthesized the new oxime derivative of CTV via the CTV ketone **2** (Scheme 1). Ketone **2** was prepared through a modification of Steven's method[30] through the oxidation of CTV with sodium dichromate in acetic acid under reflux, and we found that the ketone **2** may be purified by sublimation. The conversion of **2** to the corresponding oxime was quite sluggish, apparently due to the aryl hydrogens ortho to the ketone hindering the approach of the nucleophilic hydroxylamine. The ortho hydrogen atoms are almost at contact distance, with 2.5 between their centers.[12] Complete conversion to the oxime required overnight heating under reflux in neat pyridine, which afforded two major products by thin-layer chromatography. Chromatographic separation yielded the desired oxime as two distinct conformers which we have now characterized. Indeed, the first clue to the conformational relationship was the fact that the individual components equilibrated back to a mixture of the two conformers upon standing in solution at room temperature over a period of 1-2 days. The first-eluting crown conformer **3** was obtained in 70% yield, followed by 27% of the slower-eluting saddle conformation **4**. This isolated ratio reflects some conversion during workup and chromatography at room temperature from an initial saddle to crown ratio of 56:44, based on a proton NMR spectrum of the crude reaction mixture after 16 hours in pyridine under reflux (115°C). The methylene protons of crown oxime **3** are two AX quartets due to the asymmetry of the oxime [δ 4.77 (1H, d, J = 13.8 Hz), 4.38 (1H, d, J = 13.5 Hz), 3.58 (1H, d, J = 13.8 Hz), and 3.50 (1H, d, J = 13.5 Hz).], the axial (internal) protons resonating 1.2 ppm and

0.8 ppm downfield, respectively. For the saddle conformation, the two methylenes are either syn or anti to the oxime hydroxyl and are thus at two separate chemical shifts, [δ 4.02 (2H, s), 3.54 (2H, s)], and there is no geminal coupling due to the equivalence resulting from rapid interconversion of conformers, as illustrated by the equilibrium between **3a**, **3b** and **3c**, and bearing in mind that each conformer shown may exist as two twist conformers as for the parent CTV saddle conformation.[31] The CTV ketone is known to exist exclusively as the flexible saddle conformation, due to greater conjugation of the carbonyl with the phenyl rings, and as evidenced by the equivalence of its four methylene protons [δ 3.77 (4H, br s)] due to pseudorotation that is rapid on the NMR time scale. Similar to the oxime, CTV 5-methylene and 5-isopropylidene derivatives are also equilibrium mixtures of saddle and crown conformations.[30] The absorbances for the C=N stretches of the crown and saddle oximes were essentially identical at 1606 and 1607 cm^{-1}, respectively, although we expected to see some difference due to greater conjugation in the saddle.

The symmetry successively decreases from CTV, which has C_{3v} symmetry, to the ketone **2**, which has C_s symmetry, to the oxime **4**, which lacks symmetry elements except for identity. Curiously, the *syn* and *anti* oxime saddle isomers **4b** and **4b** are interconverted through the process of pseudorotation. The oxime is a structurally chiral molecule, although lacking chiral tetrahedral carbon atoms.



Scheme 1

Despite the fact that CTV and its derivatives have been studied for decades, the saddle conformation of the parent CTV was only recently isolated and characterized after preparation by high temperature melt

and quench by Zimmerman who also studied the thermodynamic and kinetic interconversion of the crown and saddle conformers.[31] The intermediacy of the more flexible saddle conformation had been proposed by as an intermediate in CTV crown umbrella inversion by Collet, who elegantly demonstrated the barrier to crown-to-crown inversion via racemization of chiral d9-CTV to be 26.5 kcal/mol[32] which most likely proceeds via the saddle conformation.[12] Zimmerman also studied the mesomorphic properties of nonasubstituted tribenzocyclononene derivatives[27] wherein the crown conformation is destabilized relative to the saddle form due to steric interactions with neighboring aryl rings.[12] For CTV, the saddle conformation is thermodynamically disfavored due to the negative steric crowding of proton H_a (Figure 1) with the face of the central aromatic ring at the back of the saddle.[31] This destabilizing steric interaction is absent in oxime conformation **4a**, and although there may be a stabilizing pi-pi interaction between the central aromatic ring and the oxime in **4a**, the side-saddle conformations may be lower in energy due to greater conjugation.

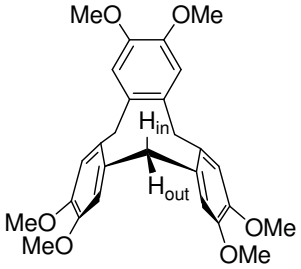


Figure 1

We observed that he rigid oxime crown conformer **3** and the more flexible saddle conformer **4** slowly interconvert at room temperature over 1-2 days and equilibrate at room temperature (20°C) predominantly to the crown conformer. Specifically a 1:6 mixture of saddle/crown is obtained in $CDCl_3$, (Keq = [saddle]/[crown] = 0.14) and to a 1:14 ratio in d_6-DMSO (Keq = 0.07). The crown conformer, having the higher dipole moment, is favored even more in the more polar solvent, although differences in H-bonding between the oxime functionality in the two conformers and the DMSO solvent certainly may also play a role. Zimmerman found that for CTV the equilibrium constant K = [saddle]/[crown] = 0.008 in DMF, and K = 0.1 in chloroform, which is very close to the value of K = 0.14 for the $CDCl_3$equilibrium mixture of oxime conformers. We expected a greater preference for oxime saddle relative to CTV based on the steric arguments provided above, and the fact that the ketone exists in the saddle conformation. The fact that the oxime prefers the crown conformation impacts the potential host-guest chemistry of the oxime, since the CTV nucleus interacts with guests within the bowl-shaped crown structure, not the saddle.

Kinetic and thermodynamic measurements of the dynamic interconversion of the oxime saddle and crown conformers are underway utilizing temperature-dependent NMR and will be reported in due course.

Experimental Section

All solvents and reagents were used without further purification unless otherwise noted. Reactions were performed under an atmosphere of nitrogen. Merck silica gel 60 (230-400 mesh) was used for flash chromatography. Merck Kieselgel 60 F254 DC-Fertigplatten (0.25 mm, Art. 5719) were used for TLC. ^{1}H NMR spectra were obtained from either a Varian INOVA 300 or Varian Gemini 2000 300 MHz spectrometer with tetramethylsilane (TMS) as an internal standard. Noise-decoupled and ^{13}C NMR spectra were recorded at 75 MHz on either the Varian INOVA 300 or Varian Gemini 2000 spectrometer. IR spectra were recorded on a Thermo Nicolet Nexus 670 FT-IR using an Alfa Aesar NaCl crystal polished optic disc, (25mm x 4mm). Mass spectra were run on a Thermo Finnigan LCQ Advantage instrument. UV-Vis spectra were

obtained from an Agilent 8452 Value Analysis UV-Vis Spectrometer and using Agilent UV-Vis Chemstation version 8.2 software. Melting points were obtained using an Electrothermal Mel-Temp®. CTV was prepared from veratryl alcohol in formic acid according to the procedure of AUTHOR [REF] and was recrystallized from dry toluene affording guest-free crystals.[33]

10,15-Dihydro-2,3,7,8,12,13-hexamethoxy-5H-tribenzo[a,d,g]cyclononen-5-one, CTV Ketone – (2). A modification of the method of Stevens[30] was employed. To a solution of cyclotriveratrylene (4.44 mmoL, 1.0 eq) in glacial acetic acid (14 mL) was added sodium dichromate (7.10 mmoL, 1.6 eq) and the dark orange solution was heated under reflux for 12-16 h. Proton NMR indicated complete consumption of starting material. The dark green solution was washed with sodium bicarbonate (1X 100 mL) and extracted with methylene chloride (3 X 60 mL). The combined organic layers were successively washed with water (2 X 50 mL), and brine. The organic layer was then dried over Na_2SO_4 and concentrated to give a brown foam which was chromatographed on silica gel (180 g) eluting with EA/CH_2Cl_2 (20/80) to afford the desired ketone (0.802 g, 39%) as a pale yellow crystalline solid: mp 207-208ºC (lit 213-214 ºC;[30] Sublimation afforded ***a fine white powder, mp *** [the ketone has not previously been sublimed.] UV-Vis (EtOH): λ_{max} = 204 (log ε = 6.50), 284 (log ε = 6.56), 326 (log ε = 6.70) nm; IR (thin film from CH_2Cl_2) 3057, 3002, 2935, 2850, 1586 (str, C=O), 1514, 1463, 1285, 1262, 1215, 1095 (s), 735 cm^{-1}. ^1H NMR (300 MHz, $CDCl_3$) δ 7.43 (2H, s), 6.76 (2H, s), 6.49 (2H, s), 3.96 (6H, s), 3.92 (6H, s), 3.81 (6H, s), 3.77 (4H, br s). ^{13}C NMR (75 MHz, $CDCl_3$) δ 194.3, 152.8, 148.0, 147.7, 133.2, 133.0, 132.2, 114.5, 112.9, 111.7, 56.4, 56.2, 56.0, 37.1.

10,15-Dihydro-2,3,7,8,12,13-hexamethoxy-5H-tribenzo[a,d,g]cyclononen-5-oxime, CTV Oxime Crown (3) and CTV Oxime Saddle (4). To a solution of CTV ketone **2** (194 mg, 0.430 mmol, 1.0 eq) in pyridine (2.0 mL) was added hydroxylamine hydrochloride (4.30 mmol, 10.0 eq) and the resulting solution was heated under reflux for 16 h. The reaction mixture was concentrated in vacuo and the residue was dissolved in methylene chloride (12 mL) and washed successively with 1N hydrochloric acid (2 X 25 mL), water (1 X 20 mL), and brine. The organic layer was then dried over Na_2SO_4 and concentrated to give an off white foam which was chromatographed on silica gel (18 g) eluting with EA/CH_2Cl_2 (20/80) to afford the oxime crown conformer **3** (0.144 g, 70%) as a colorless solid that was crystallized from MeOH to afford colorless needles: mp139-141°C; UV-Vis (EtOH): λ_{max} 208 (log ε = 7.15), 290 (log ε = 7.30) 235 (log ε = 7.21); IR (thin film from CH_2Cl_2) 3441 , 3288, 3203, 3057, 3001, 2934, 2846, 1606 (str, C=N), 1514, 1464, 1345, 1263, 1223, 1127, 1081, 734 cm^{-1}. ^1H NMR (300 MHz, $CDCl_3$) δ 9.41 (1H, br s), 6.96 (1H, s), 6.90 (1H, s), 6.86 (1H, s), 6.81 (2H, s), 6.71 (1H, s), 4.77 (1H, d, J = 13.8 Hz), 4.38 (1H, d, J = 13.5 Hz), 3.89 (3H, s), 3.87 (3H, s), 3.83 (12 H, s), 3.58 (1H, d, J = 13.8 Hz), 3.50 (1H, d, J = 13.5 Hz). ^{13}C NMR (75 MHz, $CDCl_3$) δ 158.4, 149.2, 149.1, 147.7, 147.5, 147.4, 147.2, 133.1, 131.2, 131.1, 131.0, 128.0, 127.0, 112.6, 112.5, 112.4, 110.9, 108.6, 56.0, 55.9, 55.8, 55.7, 36.7, 36.0. MS MH^+ calcd for $C_{27}H_{29}NO_7$ 480.20, found 480.20. The sample was stored at -80°C immediately after chromatographic isolation and concentration.

Further elution afforded saddle oxime **4** (0.055 g, 27%) as a colorless glass: mp xx-xx°C; UV-Vis (EtOH): λ_{max} 208 (log ε = 7.40), 237 (log ε = 7.45), and 286 (log ε = 7.54) nm. IR (thin film from CH_2Cl_2) 3442, 3296, 3057, 3001, 2933, 2851, 1607 (str, C=N), 1514, 1464,1347, 1263, 1214, 1215, 1081, 735 cm^{-1}. ^1H NMR (300 MHz, CDCl3) δ 8.88 (1H, br), 7.32 (1H, s), 6.70 (2H, s), 6.67 (1H, s), 6.64 (1H, s), 6.53 (1H, s), 4.02 (2H, s), 3.89 (3H, s), 3.86 (3H, s), 3.85 (3H,

s), 3.84 (6H, s), 3.80 (3H, s), 3.54 (2H, s). ^{13}C NMR (75 MHz, $CDCl_3$) δ 157.3, 149.6, 149.4, 147.4, 147.2, 147.0, 146.9, 132.8, 132.1, 130.4, 130.3, 127.2, 125.1, 113.9,113.7, 113.6, 112.2, 110.8, 110.5, 60.3, 55.8, 38.7, 36.9 ppm. MS MH^+ calcd for $C_{27}H_{29}NO_7$ 480.20, found 480.20. The sample was stored at -80°C immediately after chromatographic isolation and concentration.

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Supplementary Material

CTV Ketone Spectra
1H NMR
13C NMR
IR – but not reported in paper
MS– but not reported in paper
UV– but not reported in paper

CTV Oxime Crown Spectra
1H NMR
13C NMR
IR
MS
CHN

CTV Oxime Saddle Spectra
1H NMR
13C NMR
IR
MS
CHN

*****Chromatography elution profile of saddle and crown conformers of CTV oxime after baseline separation NOT—that profile would have vacant fractions in the middle! Delete or replace?*