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Accessing highly electron-rich calix[n]arene (n = 4 and 8) derivatives from acid-catalyzed condensation of 1,3,5-tripropoxybenzene

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Introduction

Calixarenes have been widely used as a synthetic framework in many aspects including functional materials, supramolecular chemistry and molecular sensors [1]. Base-catalyzed condensation of 4-tert-butylphenol/formaldehyde allows the preparation of calix [*n*] arenes with varied ring sizes (where n = 4, 5, 6, 7 and 8) [2]. Thermodynamically stable calix[4]arene can be easily isolated and it remains the most celebrated and explored member of this macrocyclic arene family [3]. However, high yield synthesis, isolation and purification of higher homologues remained a challenge for synthetic chemists [2a,b]. Beyond the difficulty of synthesis and purification, modulation of the electronic properties is another challenge for calixarene materials. In this regard, the development of new electron-rich calixarenes can potentially improve the binding affinity towards electron acceptors in host-guest chemistry; increase the stability of its oxidized product (cation radical species) and promote the development and implementation as molecular sensors [4]. Therefore, our group has been interested in synthesizing new types of calixarene derivatives with highly electron-rich property.

It has been shown that methyl ether of p-*tert*-butylcalix[4] arene (1) is conformationally mobile at ambient temperatures. Modest cooling (-30 °C) of a solution of methyl ether of p-*tert*-butylcalix[4]arene (1) in CH₂Cl₂ shows the presence of four distinct conformations by NMR spectroscopy [5]. The presence of all four conformers in the same solution suggests that energy differences

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ABSTRACT

Synthesizing novel electron-rich calix[*n*]arene derivatives to alter the electronic properties of calixarenebased materials has been one of the long-standing interests. Herein, two new electron-rich calix[*n*]arenes (*n* = 4 and 8) with different sizes were synthesized by acid-catalyzed condensation reaction of 1,3,5tripropoxybenzene and paraformaldehyde. Both derivatives were fully characterized with ¹H and ¹³C NMR, mass spectrometry and X-ray crystallography. Furthermore, their electrochemical properties and oxidized product (cation radicals) have also been investigated.

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among them must be small (i.e., in the range of 1–3 kcal/mol) [6]. Higher alkyl ethers of p-*tert*-butylcalix[4]arene (e.g., propyl ethers, **2**) are conformationally rigid and they do not interconvert due to prevention of flipping the aromatic rings (Fig. 1A). These different rigid conformers (i.e., 2_{paco} , 2_{cone} , $2_{1,3-alt}$, and $2_{1,2-alt}$) of propyl ethers of p-*tert*-butylcalix[4]arene can be synthesized and isolated, albeit via multistep syntheses [2a,b].

Recently, Ogoshi and coworkers reported that an electron-rich calix[4]arene derivative $\mathbf{3}$ can be readily accessed via a simple acid-catalyzed condensation of 1,3,5-trimethoxybenzene and paraformaldehyde in excellent yield (Fig. 1B) [7]. Interestingly, however, this condensation reaction produces exclusively only one conformation $(\mathbf{3}_{paco})$ as confirmed by NMR spectroscopy and X-ray crystallography [7]. Our group further studied the electronic properties of this particular calixarene and the application of its cation radical as a NO sensor. The results revealed that the cation radical of $\mathbf{3}_{paco}$ have an unprecedented binding affinity for nitric oxide $(k > 2 \times 10^{12})$ [8]. As the energy differences amongst the four conformers of 3 are not prohibitively large, their synthesis should be feasible as rigid conformers by employing 1,3,5-tripropoxybenzene as the reactant instead of 1,3,5-trimethoxybenzene (Fig. 1B). On the other hand, the larger steric of propoxy groups have possibility to produce larger sizes of highly electron rich calix[n]arene (n > 4) derivatives.

Accordingly, herein we report that the reaction of readily-available 1,3,5-tripropoxybenzene with paraformaldehyde in CH_2Cl_2 in the presence of an acid as the catalyst (such as $BF_3 \cdot OEt_2$ or CH_3SO_3 -H) produces rigid conformers of calix[4]arene ($\mathbf{4}_{1,3-alt}$) together with decent amounts (>45% yield) of calix[8]arene ($\mathbf{5}$). We will also

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Fig. 1. A) Four interconvertible conformations of 4-*tert*-butylcalix[4]arene methyl ethers (1) and their rigidification in the corresponding propyl ethers (2). B) The preparation of electron-rich calix[4]arene derivative $\mathbf{3}_{\text{paco}}$.

show that the composition of $\mathbf{4}_{1,3-\text{alt}}$ and higher macrocycles calix [8]arene (5) can, in part, be controlled by choice of acid/amount of acid catalyst. The structures of these, hitherto unknown, highly-electron-rich calixarene derivative were established by NMR spectroscopy, mass spectroscopy and X-ray crystallography and further evaluated by DFT calculations. Also, we have studied their electrochemical and spectral properties of one-electron oxidized products (cation radical). The details of these findings are described herein.

Results and discussion

Synthesis

In order to synthesize highly electron-rich calixarene derivatives, we chose 1,3,5-tripropoxybenzene with three more bulky and more electron-rich propoxy groups as a starting material. Firstly, the starting material (1,3,5-tripropoxybenzene) was synthesized according to literature with the alkylation of phloroglucinol (Scheme 1) [9]. The calixarene derivative was then prepared by the acid catalyzed condensation reaction of 1,3,5-tripropoxybenzene with paraformaldehyde according to the reported method by Ogoshi and coworkers [7]. Interestingly, we found that this reaction resulted in a more complicated product in comparison to the methoxy substituted analog which leads to the one dominant product $(\mathbf{3}_{paco})$. After the purification with column chromatography, two new compounds, $calix[4]arene (\mathbf{4}_{1,3-alter})$ and calix[8]arene (5) were obtained with the yield 12% and 45%, respectively. In order to alter the product distribution and synthesize calix[4]arene more efficiently, we systematically studied the



Scheme 1. Synthesis of highly electron rich calix[*n*]arene (*n* = 4 or 8) derivatives.

effects of several factors including the influence of different catalyst, the amount of catalyst, reaction solvent, temperature and reaction time.

First, in order to investigate the influence of catalyst, both Lewis (entries 1-3) and Brønsted acids (entries 4-8) have been screened. As listed in Table 1, $BF_3 \cdot Et_2O$ showed the best results with the yield 12% of calix[4]arene and 45% of calix[8]arene among the investigated Lewis acids. The total yield is less than 10% when FeCl₃ or AlCl₃ was applied as catalyst. Entries 4 and 5 illustrated that the weak Brønsted acids (CH₃CO₂H and H₃PO₄) have very poor or no catalytic effect toward this transformation. To the contrary of weak Brønsted acids, the stronger ones (entries 6-8) showed generally better catalytic effects. Among them, methanesulfonic acid (MeSO₃H, entry 8) showed the best result with 21% of calix[4]arene and 41% of calix[8]arene. Although, the resulting product is dominated by calix[8]arene, methanesulfonic acid has the best catalytic effect to produce the smaller macrocycle molecule (calix[4]arene). This acid showed promising results for synthesis of several different macrocyclic arenes (e.g., cyclotetraveratrylene and pillarenes) [10]. In order to selectively make calix[4]arene, methanesulfonic acid was applied as a catalyst in the subsequent condition optimization reactions.

Entries 1–4 in Table 2 revealed that the amount of catalyst at 20% and 50% have comparable product composition and are slightly better than 5% or 100% of catalyst. For solvent screening (entries 5–7), dichloromethane and chloroform indicated better results, the reaction did not happen in the solvents such as THF and toluene; while produced insoluble by-products (may be higher oligomer or polymer molecules) in more polar solvent acetonitrile. Entries 8–9 showed that the reaction works better at room

Table 1		
The catalyst screen in	the synthesis of	f calix[n]arene ($n = 4$ or 8).

Entry	Cat.	%Yield of OPr-calix[n]arene	
		<i>n</i> = 4	<i>n</i> = 8
1	BF ₃ ·Et ₂ O	12	45
2	FeCl ₃	2	3
3	AlCl ₃	2	6
4	CH ₃ COOH	0	0
5	H_3PO_4	1	3
6	H_2SO_4	4	12
7	CF ₃ CO ₂ H	7	20
8	MeSO ₃ H	21	41
9	none	0	0

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Table 2
The condition optimization in the synthesis of $calix[n]$ arene ($n = 4$ or 8).

Entry	Amount of catalyst	Solvent	Temp	% Yield of OPr-calix[n]arene	
				<i>n</i> = 4	<i>n</i> = 8
1	5%	CH ₂ Cl ₂	20	10	29
2	20%	CH ₂ Cl ₂	20	21	41
3	50%	CH ₂ Cl ₂	20	20	43
4	100%	CH ₂ Cl ₂	20	12	34
5	20%	CHCl ₃	20	7	21
6	20%	MeCN	20	0	4
7	20%	THF	20	0	0
8	20%	CH ₂ Cl ₂	0	0	0
9	20%	CH ₂ Cl ₂	35	2	8
10	20%	CH ₂ Cl ₂	20	5 ^a	38 ^a
11	20%	CH_2Cl_2	20	10 ^b	31 ^b

Note: ^areaction time 2 h; ^breaction time 6 h.

temperature. However, decreasing the temperature (entry 8) halts the reaction and the starting materials remain untouched. To the contrary of low temperatures, a large amount of white insoluble by-product is formed at high temperature, which greatly reduces the reaction yield. The reaction time was also screened, and 4 h has given the better yield (entries 10–11). In summary, both Lewis (BF₃·Et₂O) and Brønsted (MeSO₃H) acids produce calix[*n*]arenes (*n* = 4 and 8) with high total yield. Additionally, methanesulfonic acid as catalyst can slightly alter the product distribution and increase the yield of calix[4]arene to 21%.

Both calix[*n*]arenes (*n* = 4 and 8) can be recrystallized from a mixed solvent of dichloromethane and acetonitrile to obtain high quality single crystals for X-ray diffraction experiments. The results of X-ray diffraction revealed that this novel calix[4]arene hold with 1,3-alter conformer noted as $4_{1,3-alter}$. This result is different from the structure of 3_{paco} , which contain methoxy instead of propoxy groups. To better comprehend this change, we utilized the DFT level calculation to simulate the energy profile of four possible conformations (4_{paco} , 4_{cone} , $4_{1,2-alter}$ and $4_{1,3-alter}$ at Fig. S4). It was found that $4_{1,3-alter}$ has the lowest energy and is around 6 kcal/mol lower than the second stable 4_{paco} . This is in agreement to our experimental results from both NMR and solid-state structure (X-ray).

Crystal structures

In the structure of $\mathbf{4}_{1,3-\text{alter}}$, there are two sets of facial interacted aromatic rings, and the dihedral angles are 47.7° and 56.4°, respectively (Fig. 2). Such a large dihedral angle is mainly due to the steric effect of propoxy groups. The distance between two sets of facial aromatic rings is \sim 6.42 Å, in contrast to \sim 5.60 Å in **2**_{1,3-alter}; therefore, only weak π - π interaction can be observed in the structure of 4. This orientation also shortens the distance between inner oxygen atoms on the opposite rings with \sim 3.85 Å in average. Fig. 2C shows the packing of crystal structures which indicates weak intermolecular CH... π interactions. Variable temperature ¹H NMR experiment were performed on both calix[4]arene (4) and calix[8]arene (5) in the temperature range from -40 to 55 °C (Figs. S1 and S2). In the case of calix[4]arene (4), the aromatic region only display one sharp signal (6.14 ppm) in the whole temperature range and the bridged-methylene proton showed at 3.90 ppm as a sharp peak, while the peaks raised from two kinds of propoxy group slightly shifted to lower field and became broad at lower temperature. The VT-NMR result revealed that $\mathbf{4}_{1,3-\text{alt}}$ is not conformationally flexible in the range from -40 to 55 °C. The variable temperature NMR characterization data of 5 revealed only one sharp signal in the aromatic region, however, there are three kinds of propoxy group signal were observed at a lower tempera-



Fig. 2. Side view (A), top view (B), and packing (C) of the crystal structures of $(\mathbf{4}_{1,3\text{-alter}})$. Hydrogens and solvent molecules were omitted for clarity; oxygen atoms are red; phenyl rings are gold; alkyl groups are bright gray and bridge carbon are dark green. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

ture (-20 and -40 °C) and two of propoxy signal merged at room temperature. Which indicate that the molecule is interconversion of conformers at room temperature and can be frozen at -20 °C or below. In the solid state, the octameric 1,3,5-tripropoxybenzene forms a molecule resembling through the bonding of methylene groups as bridge, which looks like a garland structure (Fig. 3A). The bridging methylene groups are alternately oriented up and down relative to its mean plane (Fig. 3B). All internal (positioned

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Fig. 3. Top view (A), and side view (B) of the crystal structures of new calix[8]arene (**5**). Hydrogens and solvent molecules were omitted for clarity; oxygen atoms are red; phenyl rings are gold; alkyl groups are bright gray and bridge carbon are dark green; and all alkyl groups are omitted in the side view mode. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

between methylene bridges) propoxy groups are oriented inwards (also alternating up and down) and ordered.

Electrochemical property

The electrochemical properties of $\mathbf{4}_{1,3-\text{alter}}$ and $\mathbf{5}$ were investigated by cyclic voltammograms (CV) and square waves voltammograms (SWV) and the results are summarized in Fig. 4. The CV of $\mathbf{4}_{1,3-\text{alter}}$ shows two reversible oxidation peaks at 0.42 and 0.68 V vs. Fc/Fc⁺. The first oxidation potential is lower than the analog $\mathbf{3}_{paco}$ by 0.11 V [8]. This difference can be attributed to two factors: 1) propoxy group is slightly more electron donating than methoxy group (Fig. S7); and each molecule contain twelve propoxy groups which can significantly reduce the oxidation potential; 2) the structure distinction in the stable conformation. The structure of $\mathbf{4}_{1,3-alter}$ contains two sets of facial interacted aromatic rings, which can stabilize the generated cation radicals by one-electron oxidation. However, the structure of $\mathbf{3}_{\text{paco}}$ has only a pair of parallel aromatic rings with an oxidation potential of 0.53 V. Similar stabilization effect was observed in $\mathbf{2}_{\text{paco}}$ and $\mathbf{2}_{\text{1,3-alter}}$ system. The redox potential of $\mathbf{2}_{1,3-\text{alter}}$ is lower than $\mathbf{2}_{\text{paco}}$ by 0.12 V [11]. For the calix[8]arene (5) molecule, the CV shows three sets of reversible couples (0.54, 0.65 and 0.76 V). No facial interacted aromatic structure was observed in 5. Indeed, the first oxidation potential is higher than $\mathbf{4}_{1,3-\text{alter}}$ by 0.12 V, which provides more evidence



Fig. 4. Cyclic voltammograms (CVs, solid lines, blue color for $\mathbf{4}_{1,3\text{-alter}}$, red for **5**) and overlaid square waves (SW, dashed lines) of 2 mM $\mathbf{4}_{1,3\text{-alter}}$ (top) and **5** (bottom) in CH₂Cl₂ (0.1 M nBu₄NPF₆) at a scan rate of 100 mVs⁻¹. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

for the effects of the facially-interacted aromatic rings on the stabilization of the cation-radical species.

Cation radical generation

We further generated the cation radical of $\mathbf{4}_{1,3-alter}$ in solution by using THEO⁺·SbCl₆ as one electron oxidant (THEO = 1,2,3,4,5, 6,7,8-octahydro-9,10-dimethoxy-1,4,5,8-dime-thano-anthracene, $E_{red} = 0.67 \text{ V}$ vs. Fc/Fc^+ , $\lambda_{max} = 518 \text{ nm}$, $\varepsilon_{max} = 7300 \text{ cm}^{-1} \text{ mol}^{-1}$) [12]. The Fig. 5 illustrates the electronic spectra obtained upon an sub-stoichiometric addition of a solution of neutral $\mathbf{4}_{1,3-\text{alter}}$ in CH₂Cl₂ to a solution of THEO⁺SbCl₆ as oxidant. After the addition of 1 equiv of neutral $\mathbf{4}_{1,3\text{-alter}}$, the oxidant THEO⁺·SbCl₆ was completely converted to $\mathbf{4}_{1,3-\text{alter}}^{+}$ which was determined by a quantitative deconvolution of the component spectra of various species present at each titration point (Fig. 5). It was found that a broad near-infrared absorption peak on $4^+_{1,3-alter}$ spectrum, started from 800 nm and all the way up to 3000 nm. This charge transfer band is mainly derived from two pairs of facial interacted aromatic rings. The extinction coefficient of $\mathbf{4}_{1,3-\text{alter}}^+$ (1250 cm⁻¹mol⁻¹) is slightly lower than $\mathbf{3}_{\text{paco}}^+$ (1700 cm⁻¹mol⁻¹) due to the larger distance between two pairs of facial aromatic rings as well as more electron-rich nature in the structure [8].

Conclusion

In conclusion, we report the preparation of two novel highly electron-rich calix[n]arenes (n = 4 and 8) by the acid-catalyzed condensation reaction of 1,3,5-tripropoxybenzene and paraformaldehyde. After screening the reaction condition, we found both $BF_3 \cdot Et_2O$ and methanesulfonic acids produce calix[n]arenes (n = 4 and 8) with high total yield. Additionally, methanesulfonic acid as catalyst can slightly alter the product distribution and increase the yield of calix[4]arene. Both calixarene derivatives are fully characterized by NMR spectra, mass spectroscopy and X-ray diffraction. In addition, the electrochemical properties and cation radical of $\mathbf{4}_{1,3-alter}$ and $\mathbf{5}$ were characterized. Studies on the application of such electron-rich calixarene derivatives as sensors and the interaction with metal cations and small molecules (e.g., NO) will be reported in due course.

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Fig. 5. Left: Spectral changes observed upon the reduction of 0.074 mmol THEO^{+.} in CH₂Cl₂ (3 mL) by adding 2.13 mmol solution of **4**_{1,3-alter} in CH₂Cl₂. Right: Plot of the mole fractions of THE0⁺. (red) and $\mathbf{4}_{1,3-alter}^+$ (black) against added equivalents of neutral $\mathbf{4}_{1,3-alter}$. Symbols represent experimental points, whereas the solid lines show best-fit to experimental points using $\Delta G = E_{ox} (4^+_{1,3-alter} - E_{red} (THEO^*) = -246 \text{ mV}$. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

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Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.tetlet.2019.151215.

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