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Enantioselective synthesis of inherently chiral calix[4]arenes *via* asymmetric C-H bromination of phenols

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ABSTRACT

Inherently chiral calix[4]arenes (ICCs) represent a unique class of chiral macrocyclic arenes, which hold significant potentials in chiral recognition, sensing, and asymmetric catalysis. However, accessing these distinct chiral molecules poses notable challenges, with their catalytic enantioselective synthesis being largely underdeveloped. Herein, we report an efficient enantioselective synthesis of ICCs using an asymmetric C-H brominative desymmetrization strategy. Utilizing the chiral phosphoric acid-catalyzed asymmetric electrophilic aromatic *ortho*-bromination reaction of phenol, we successfully broke the symmetry of prochiral calix[4]arenes, which resulted in the formation of various 1,3-bis-phenyl rings-substituted ICCs with good yields and high enantioselectivities. Moreover, the asymmetric di-bromination of bisphenol-containing prochiral calix[4]arenes was demonstrated, yielding ICCs featuring three or even four rings displaying various modifications with high enantioselectivity. Leveraging the versatility of the introduced bromide handle and the phenolic hydroxyl group in the ICC products, a range of derivatizations were performed to produce diverse ICC derivatives, one of which exhibited notable chiroptical properties, highlighting the significance of this method.

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Chiral molecules, referring to three-dimensional molecules that are non-superimposable on their mirror images, have found significant applications in pharmaceuticals, agrochemicals, asymmetric synthesis and material science. Molecular chirality is generally classified into four categories of chiral elements: Central chirality, axial chirality, planar chirality and helical chirality (Fig. 1A). Calix[4]arenes are a type of privileged macrocyclic compounds, which have been extensively explored in supramolecular chemistry over recent decades, particularly for their applications in sensors, separations and catalysis [1–8]. The rigidly curved structure of calix[4]arenes engenders chirality through the asymmetric distribution of achiral substitutions within their frameworks, a concept coined as "inherent chirality" by Böhmer *et al.* in 1994 [9–11], which distinguishes it from the conventional four chiral elements. For instance, modifying of one phenyl ring of calix[4]arene with a *meta*-substitution [12], or altering two adjacent phenyl rings with different *para*-substitutions, would result in the unique inherent chirality (Fig. 1B) [13].

Given the significant potential of inherently chiral calix[4]arenes (ICCs) in chiral recognition, asymmetric catalysis and chiral material science [13–16], there is a pressing demand to develop efficient catalytic asymmetric methods for accessing these chiral compounds [17–21]. However, due to the unique scaffolds and relatively large size of calix[4]arenes, the early development of catalytic enantioselective methods met with limited success [22–23]. In 2020, Tong, Wang and co-workers reported an elegant enantioselective Pd-catalyzed intramolecular C-N coupling method for the asymmetric synthesis of heterocalix[4]arenes [24–26]. However, due to the requirement of forming C-heteroatom bonds, this method is not applicable for conventional methylene-linked calix[4]arenes. On the other hand, enantioselective desymmetrization [27–29] has proven to be the most effective method for asymmetric synthesis of ICCs [30]. Notable advancements have been made in recent years, particularly through the directing group-enabled asymmetric *meta*-functionalization of the upper-rim, including approaches of C-H cyclization desymmetrization and C-H substitution desymmetrization (Fig. 1C). In 2022, both the Cai group [31] and the Tong group [32] independently reported the enantioselective synthesis of ICCs through a Pd-catalyzed intramolecular C-H arylation-enabled desymmetrization, albeit producing different cyclization products. Later, our group [33]

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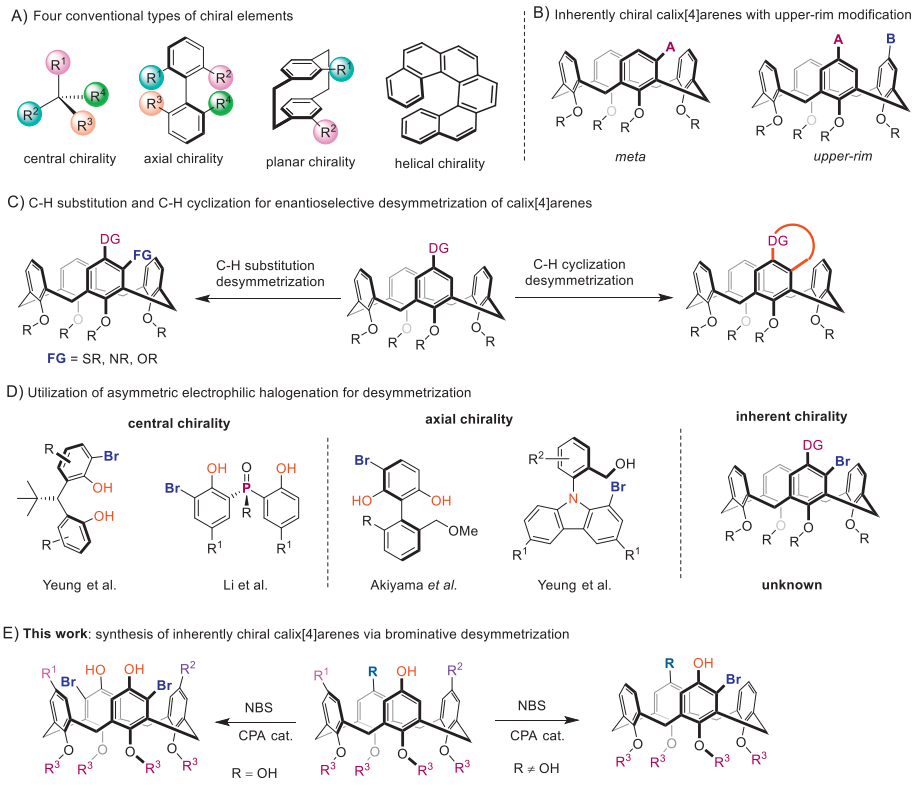


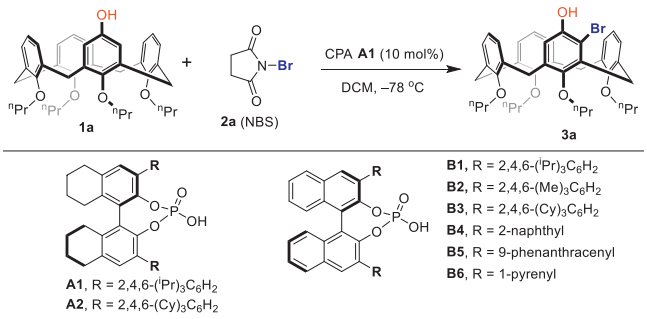
Fig. 1. Enantioselective synthesis of inherently chiral calix[4]arenes and asymmetric brominative desymmetrization.

and the Liu group [34] respectively presented the asymmetric synthesis of ICCs by desymmetrizing *para*-amino-substituted prochiral calix[4]arenes using sequential asymmetric Povarov reaction and aromatization. Recently, the Shi group [35] and the Niu group [36] detailed the asymmetric synthesis of ICCs using cobalt-catalyzed C-H annulation with alkynes in the presence of a directing group, leading to products exhibiting both inherent chirality and axial chirality. On the other hand, the enantioselective synthesis of ICCs through C-H substitution desymmetrization has received relatively less attention, as exemplified by recent demonstrations involving asymmetric electrophilic sulfenylation [37] and amination [38] of electron-rich arenes, along with directing group-enabled C-H acyloxylation [39]. Although numerous enantioselective desymmetrization methods have been developed to achieve high enantioselectivity, approaches that can facilitate the generation of ICCs with multiple modifications of phenyl rings and easy subsequent derivatization remain relatively limited.

Aryl halides play a crucial role in modern organic synthesis, which have been widely employed for synthesizing various functional molecules through cross-coupling reactions [40–42]. Given that electrophilic aromatic halogenation reaction represents the most classic and convenient way to produce aryl halides, the development of a catalytic enantioselective version holds significant importance, as the resulting chiral aryl halides would have broad applications in synthesizing chiral molecules through various cross-coupling reactions. Pioneered by Miller and co-workers, peptide-catalyzed enantioselective aromatic bromination of phenols has been extensively explored in the synthesis of axially chiral molecules through a dynamic kinetic resolution strategy, including biaryl [43–45], benzamides [46] and C-N atropoisomers [47–48]. Moreover, asymmetric aromatic bromination has been widely utilized for desymmetrizing prochiral molecules, resulting in the formation of carbon/heteroatom central chirality [49–52] and C-C/C-N axial chirality (Fig. 1D) [53–55]. However, while aryl bromide-containing ICCs hold promise as valuable precursors for straightforward derivatizations, the asymmetric synthesis of ICCs through aromatic C-H bromination reactions has yet to be revealed. In this work, we present the application of a chiral phosphoric acid (CPA) [56–62] catalyzed aromatic bromination reaction for the enantioselective synthesis of valuable aryl bromide-containing ICCs by desymmetrizing phenol-containing prochiral calix[4]arenes (Fig. 1E). Moreover, asymmetric di-bromination and even multi-bromination were also viable, which afford ICCs with three or even four phenyl rings precisely modified, a structure rarely attained by other known catalytic asymmetric methods.

We commenced our study by choosing the 4-phenol-containing calix[4]arene **1a** as the model substrate, which can be readily prepared from unsubstituted calix[4]arene through sequential bromination, boronation and oxidation steps (Table 1). Utilization *N*-bromosuccinimide (NBS, **2a**) as the electrophilic bromination reagent, and after extensive optimizations of reaction conditions, we were delighted to find that the reaction between **1a** and NBS (1.05 equiv.) in the presence of CPA **A1** (10 mol%) yielded the inherently chiral C-H brominated calix[4]arene **3a** in 93 % yield with 93 % *ee* in DCM at −78 °C (entry 1). Our screening of the CPA catalysts suggested that both the 3,3'-substitutions and the chiral scaffolds of the CPA played a critical role in achieving high enantioselectivity in this reaction (entries 2-8). Notably, CPAs featuring 3,3'-bis-(2,4,6-(iPr)$_3$C$_6$H$_2$) groups exhibited notably superior enantioselectivity, with H8-BINOL-derived CPAs also surpassing the performance of the BINOL-derived CPAs. Examination of the solvents revealed that this reaction could proceed in various solvents; however, none of them could yield superior or comparable enantioselectivity (entries 9-12). Increasing the reaction temperature from −78 °C to −50 °C resulted in the formation of **3a** with a decreased 85 % *ee* (entry 13). Increasing the quantity of **2a** from 1.05 equiv. to

Table 1
Optimization of reaction conditions.[a]



Entry	Variation from the standard conditions	Yield (%)[b]	*ee* (%)[c]
1	None	93	93
2	CPA **A2** instead of **A1**	89	85
3	CPA **B1** instead of **A1**	94	76
4	CPA **B2** instead of **A1**	85	6
5	CPA **B3** instead of **A1**	59	44
6	CPA **B4** instead of **A1**	75	5
7	CPA **B5** instead of **A1**	78	12
8	CPA **B6** instead of **A1**	69	7
9	Toluene instead of DCM	61	32
10	THF instead of DCM	57	14
11	EtOAc instead of DCM	85	27
12	MeOH instead of DCM	78	3
13	−50 °C instead of −78 °C	93	85
14	NBS (1.2 equiv.) instead of 1.05 equiv.	83	94

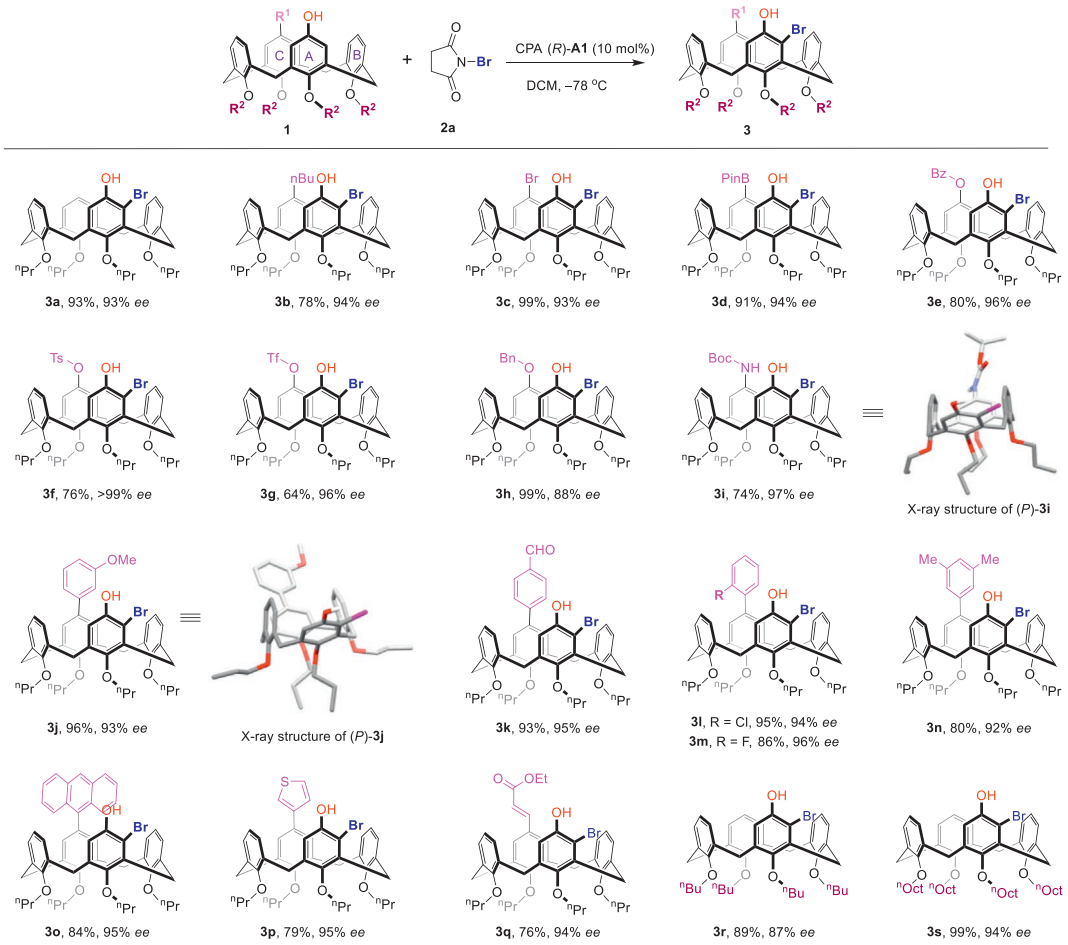
[a] Unless otherwise noted, the reactions were performed with **1a** (0.01 mmol), **2a** (0.0105 mmol) with CPA (0.001 mmol) in DCM (0.3 mL) at -78 °C for 12 h.
[b] Isolated yield.
[c] *ee* values were determined by chiral HPLC analysis.

1.2 equiv. resulted in slightly improved ee value but reduced yield due to the increased formation of the di-bromination product (entry 14).

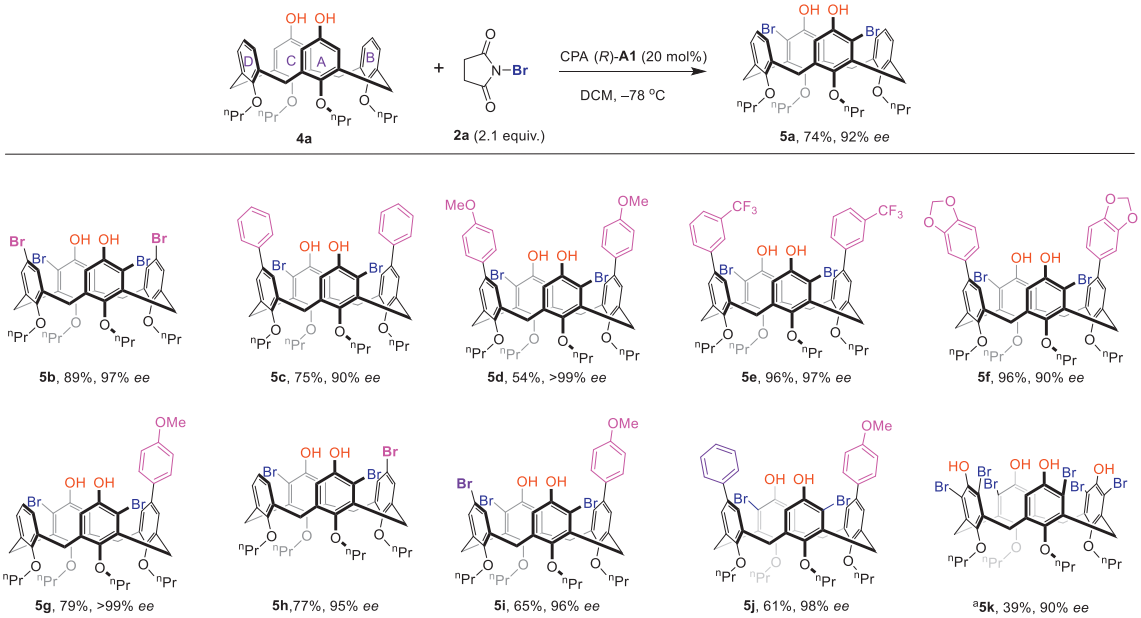
With the optimal reaction conditions established, we set out to explore the scope of this method for the enantioselective synthesis of ICCs (Scheme 1). A series of C-ring modified prochiral phenol-containing calix[4]arenes were studied using this method. We were pleased to find various substitutions (**3b-3d**), including the functionalizable groups such as halides (**3c**) and boronic ester group (**3d**), were well tolerated by the method. Moreover, other heteroatom substitution on the C-ring were examined, such as the acyloxy (**3e**), sulfonyloxy (**3f** and **3g**), alkoxy (**3h**) and amido (**3i**) groups, all of which exclusively yielded the desired phenol ring bromination products with high enantioselectivities, even though these electrondonating groups may potentially enhance the reactivity of the C-ring. Furthermore, a series of substituted phenyl rings at the *para*-position of the C-ring demonstrated compatibility with this enantioselective desymmetrization method, regardless of their substitution positions and electronic nature (**3j-3n**), including the sterically demanding *ortho*-substituted ones (**3l-3m**). The absolute configuration of these ICCs was assigned as (*P*) by analogy to the configuration of products **3i** and **3j**, whose structures were unambiguously determined by X-ray crystallography analysis. In addition, introducing 9-anthracenyl (**3o**), heteroaryl (**3p**) and acrylate (**3q**) substitution on the C-ring proved successful with this approach, yielding ICCs with good yield and high enantioselectivity. Modifications of the alkoxyl groups at the lower rim of calix[4]arenes were also studied, which indicated that various alkoxyl groups were feasible with the current method (**3r**-**3s**).

After successfully demonstrating the excellent compatibility for catalytic enantioselective synthesis of ICCs through asymmetric mono-bromination, our focus shifted to asymmetric synthesis of ICCs through asymmetric di-bromination and multi-bromination (Scheme 2). Encouragingly, the reaction between the A-ring and C-ring bis-phenol-containing calix[4]arene **4a** with NBS (2.1 equiv.) enabled by CPA **A1** (20 mol%) yielded the di-brominated ICC **5a** in 74% yield with 92% *ee*, featuring C-2 symmetry. Moreover, the prochiral bis-phenol-containing calix[4]arenes with B-ring and D-ring identical substitutions were explored with this method, resulting in the formation of ICCs where all four phenyl rings were modified (**5b-5f**). Furthermore, the calix[4]arenes with a single B-ring substitution (**5g-5h**) or B-ring and D-ring featuring different substitutions (**5i-5j**) proved to be viable substrates under these conditions, yielding the chiral calix[4]arenes with C-1 symmetry in good yield and high enantioselectivity. Notably, the tetra-phenol-containing calix[4]arene **4k** was also investigated under the standard condition. However, due to its poor solubility, the reaction at -78 °C could not yield a major product. Interestingly, conducting this reaction at room temperature led to the isolation of a hexa-brominated ICC **5k** in 39% yield with 90% *ee*.

To provide further insight into this method, a series of control experiments were performed. The reaction between **1a** and NBS in the absence of CPA catalyst yield the brominated **3a** in 85% yield at room temperature, with a yield of 47% achieved even when the reaction was performed at -78 °C (Fig. 2A). These results suggest a strong background reaction without chiral catalyst, thus highlighting the challenges to achieve high enantioselectivity control. Employing an O-Me protected calix[4]arene **6a** as the substrate under the standard conditions failed to produce the expected bromination product **7a**, which indicated that the free OH group is essential for both reactivity and enantioselectivity in this reaction, potentially engaging in hydrogen bonding with the CPA catalyst (Fig. 2B). Additionally, the use of hydrogen-bond-competing solvents, such as THF and MeOH, resulted in the formation of products with low enantioselectivities (see results in Table 1), further underscoring the critical role of hydrogen bonding interactions. Moreover, other electrophilic halogenation reagents were also explored using this method, and we were delighted to find that *N*-bromophthalimide could also yield ICC **3a** with 93% *ee* (Fig. 2C). However, the use of NIS or NCS proved unsuccessful, resulting in either the formation of the product in nearly racemic form (**3a'**) or a failure to yield the product. These results can be attributed to their inappropriate reactivities under these conditions: NCS exhibits insufficient reactivity, while NIS displays overly high reactivity, making it challenging to control stereoselectivity with the CPA catalyst. Given the observation of di-brominated byproducts for certain substrates, the potential involvement of kinetic resolution in the second bromination step was investigated (Fig. 2D). Treatment of racemic **3a** with NBS (0.5 equiv.) under the standard conditions resulted in the achiral di-bromination calix[4]arene **8a** and the recovered **3a** in 47% yield with 68% *ee* ($s = 8.0$), with the same configuration as achieved through the desymmetrization method. This result indicated that the high enantiopurity of the obtained ICCs might arise from the synergistic effect of the enantioselective desymmetrization (the first bromination step) and kinetic resolution (the second di-bromination step) (Fig. 2E). In this process, the minor enantiomer (*ent*-**3**) formed during the initial desymmetrization step may be consumed more rapidly in the subsequent kinetic resolution step, ultimately enhancing the enantioselectivity of the products (**3**). Based on the results of the control experiments and previous studies [55], a plausible mechanism and the origin of enantioselectivity for inherent chirality were proposed (Fig. 2F). Through the dual-hydrogen bonding activation facilitated by the CPA catalyst on the phenol moiety of **1a** and NBS, the *ortho*-selective electrophilic addition would lead to the formation of the dearomatized intermediate **INT-A**, which serves as both the rate-determining and enantio-determining step. Following that, the facile tautomerization step led to rearomatization, yielding the desired ICC **3a**. Given the intrinsic cone-shaped structure of



Scheme 1. Scope for enantioselective synthesis of inherently chiral calix[4]arenes *via* asymmetric mono-bromination. Reaction conditions: The reactions were performed with **1** (0.05 mmol), NBS (0.0525 mmol), CPA (*R*)-**A1** (0.005 mmol) in DCM (1.5 mL) at −78 °C for 24 h. Yields refer to isolated yields, and the *ee* values were determined by chiral HPLC analysis.



Scheme 2. Scope for enantioselective synthesis of inherently chiral calix[4]arenes *via* asymmetric di/multi-bromination. The reactions were performed with **4** (0.05 mmol), NBS (0.105 mmol), CPA (*R*)-**A1** (0.01 mmol) in DCM (1.5 mL) at −78 °C for 24 h. Yields refer to isolated yields, and the *ee* values were determined by chiral HPLC analysis. [a] The reaction was performed with **4k** (0.02 mmol), NBS (0.12 mmol), CPA (*R*)-**A1** (0.008 mmol) in DCM (1.5 mL) at room temperature for 24 h.

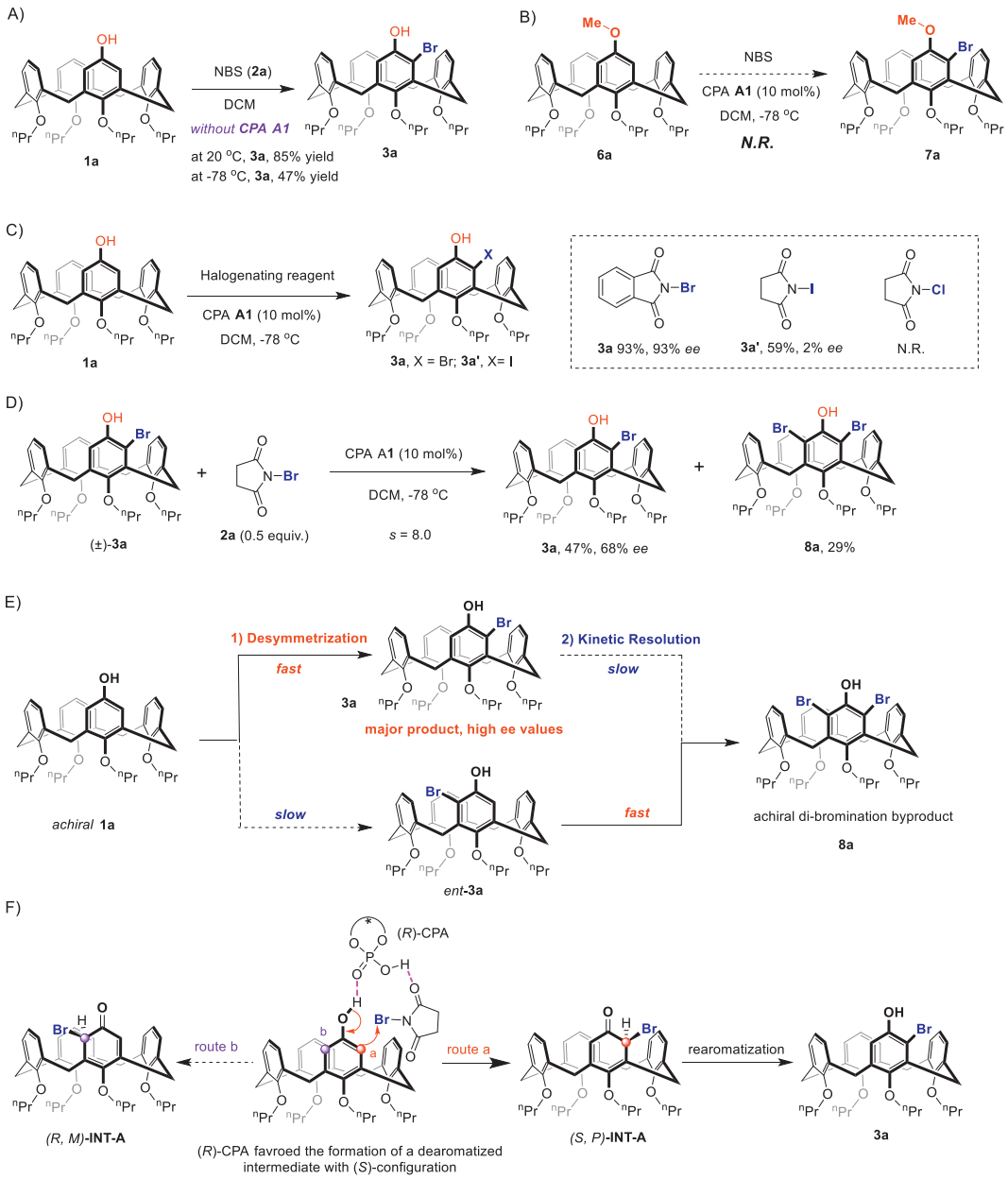
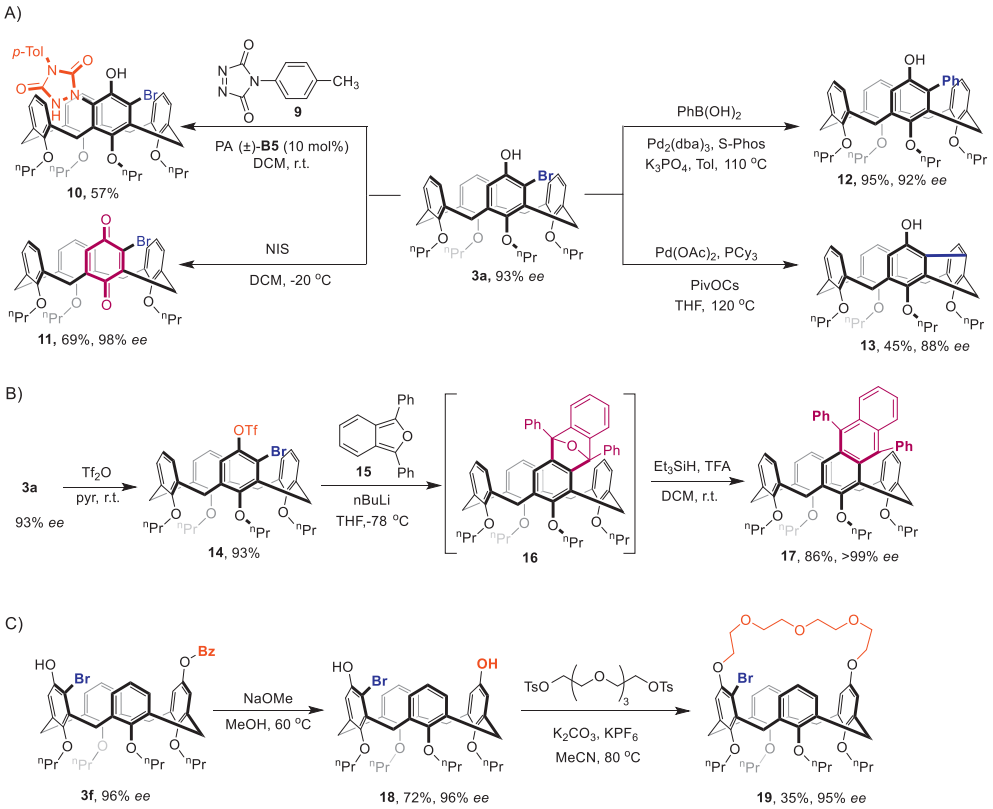


Fig. 2. Control experiments and proposed reaction mechanism. (A) Control experiments in the absence of CPA catalyst. (B) Control experiment of the OH-substituted substrate. (C) Asymmetric halogenation using other electrophilic halogenation reagents. (D) Kinetic resolution through di-bromination. (E) Proposed synergistic effect of the enantioselective desymmetrization and kinetic resolution. (F) Proposed reaction mechanism and origin of inherent chirality of the reaction.

calix[4]arene, we presume that NBS would approach the phenol ring exclusively from the outer face. Accordingly, the addition of *ortho*-position **a** (route a) would lead to the formation of intermediate (*S,P*)-**INT-A**, whereas the addition using *ortho*-position **b** (route b) would yield intermediate (*R,M*)-**INT-A**. As demonstrated in the previous DFT calculation studies by Akiyama and co-workers [55], the (*R*)-CPA favors the formation of a brominative dearomatized intermediate with (*S*)-configuration. Consequently, the formation of the (*S,P*)-**INT-A** would be more favorable as (*R*)-CPA **A1** was used in our reaction, which effectively explains the configuration of inherent chirality observed in these reactions.

To showcase the utilities of this method, a variety of derivatizations of the ICC products were performed (Scheme 3). Electrophilic aromatic substitution of **3a** with diazo reagent **9** afforded the *ortho*-amination product **10** in 57% yield (Scheme 3A). Interestingly, treatment of **3a** with NIS did not yield the expected iodinated product; instead, the oxidative quinone-containing calix[4]arene **11** was obtained. Moreover, Suzuki coupling of **3a** with phenylboronic acid in the presence of a palladium catalyst led to the cross-coupling product **12**. Notably, treatment of **3a** with a Pd catalyst without adding other coupling component resulted in the formation of the transannular arene–arene coupling product **13**. Moreover, the trifluoromethanesulfonylation of **3a** produced **14**, featuring two cross-coupling handles for further derivatizations (Scheme 3B). Treatment of **14** with *n*-BuLi generated the active benzyne intermediate, which underwent a Diels-Alder reaction with isobenzofuran derivatives **15** to give the corresponding cycloaddition product **16** [63]. Subsequent deoxygenative aromatization [64] of **16** led to the formation of the anthracene-containing ICCs **17** in 86% yield with 99% *ee*. The derivatizations



Scheme 3. Derivatizations of the inherently chiral calix[4]arenes.

of the 1,3-bis-phenyl ring modified ICC products were also investigated (Scheme 3C). Starting with the C-ring benzoyloxy-substituted chiral calix[4]arene **3f**, the removal of the O-Bz group under basic conditions afforded diphenol **18**, which then underwent the bis-alkylative macrocyclization to give the upper rim-bridged inherently chiral calix[4]arene **19**, potentially creating a rigidified chiral cavity [65].

While the brominative ICC products did not exhibit notable photophysical properties, we were thrilled to find that the anthracene-containing ICC derivative **17** demonstrates potent photophysical and chiroptical properties, which have been extensively studied (Fig. 3). The UV-vis absorption spectrum of **17** revealed a maximum absorption band at 273 nm (Fig. 3A). The fluorescence spectrum of ICC **17** displayed its strongest emission band at 465 nm, with the compound exhibiting a light-blue fluorescence in solution upon excitation (Fig. 3B). To investigate the chiroptical properties of this compound, both enantiomers of **17** were synthesized through the same route from the pair of enantiomers of **3a**, which were derived by employing the CPA **A1** catalyst with opposite configurations. The circular dichroism (CD) spectrum of *(P)/(M)*-**17** displayed a clear mirror image relationship, exhibiting remarkable Cotton effects (Fig. 3C). Moreover, the circularly polarized luminescence (CPL) analysis of *(P)/(M)*-**17** also revealed a mirror image spectrum, indicating its CPL activity (Fig. 3D). The luminescence dissymmetry factor $|g_{lum}|$ value of this compound was measured to be 1.72×10^{-3} (at 465 nm), suggesting its promising applications in chiroptical materials.

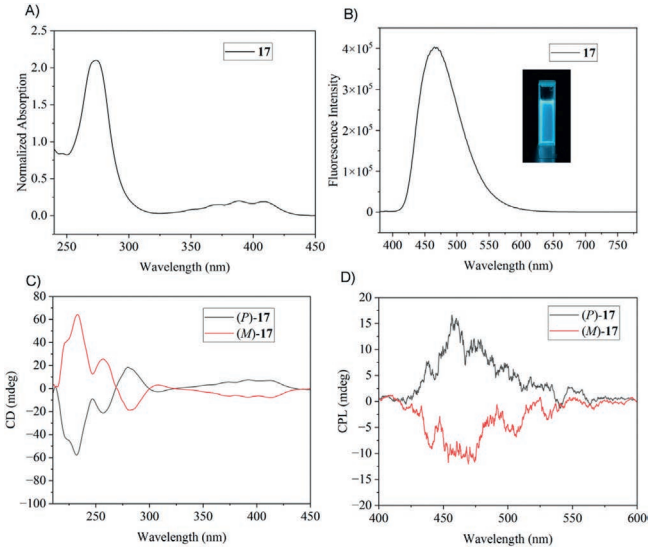


Fig. 3. Studies of the photophysical and chiroptical properties of the calix[4]arene **17**. (A) UV-vis absorption spectra of calix[4]arene **17**. (B) Fluorescence spectra of calix[4]arene **17** and its picture upon excitation. (C) CD spectra of the two enantiomers of calix[4]arene **17**. (D) CPL spectra of the two enantiomers of calix[4]arene **17**.

In conclusion, we have developed an efficient method for asymmetric synthesis of inherently chiral calix[4]arenes through an organocatalyzed enantioselective desymmetrization protocol. Leveraging the chiral phosphoric acid-catalyzed asymmetric *ortho*-bromination of phenols, the symmetry of a series of prochiral calix[4]arenes were successfully broken, which led to the formation of various 1,3-bis-phenyl ring-modified ICCs with high enantioselectivities. Moreover, this method is applicable for bis-phenol-containing prochiral calix[4]arene substrates, resulting in ICCs with three or even four phenyl rings precisely decorated. The versatile reactivity of the introduced bromide handle and the intrinsic phenolic OH group in products allowed for a variety of derivatizations, producing a diverse range of ICC derivatives. Notably, one ICC derivative containing anthracene moiety exhibited promising pho-

tophysical and chiroptical properties, highlighting the potential of this method.

CRediT authorship contribution statement

Mengyao Yuan: Writing – original draft, Methodology, Investigation. **Wansen Xie:** Investigation. **Xiaoyu Yang:** Writing – review & editing, Supervision, Funding acquisition, Conceptualization.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Supplementary materials

Supplementary material associated with this article can be found, in the online version, at doi:10.1016/j.cclet.2025.112157.

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