

# Biaryl cross-coupling reactions: convenient routes to new functionalized calix[8]arenes

Roselyne Baudry,<sup>a</sup> Caroline Felix,<sup>\*a</sup> Claude Bavoux,<sup>b</sup> Monique Perrin,<sup>b</sup> Francis Vocanson,<sup>a</sup> Isabelle Dumazet-Bonnamour<sup>a</sup> and Roger Lamartine<sup>a</sup>

<sup>a</sup> Laboratoire d'Application de la Chimie à l'Environnement, Université Lyon 1, 43 bd du 11 Nov. 1918, F-69622, Villeurbanne cedex. E-mail: c.felix@cdlyon.univ-lyon1.fr

<sup>b</sup> Laboratoire de Cristallographie, Université Lyon 1, 43 bd du 11 Nov. 1918, F-69622, Villeurbanne cedex

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Calix[8]arene substituted by styryl groups was prepared by Suzuki and Negishi type cross-coupling reactions respectively *via* the fully *para* brominated- and iodinated-octamethoxycalix[8]arene as intermediates. Single X-ray structure of the octabromo-octamethoxycalix[8]arene has been resolved.

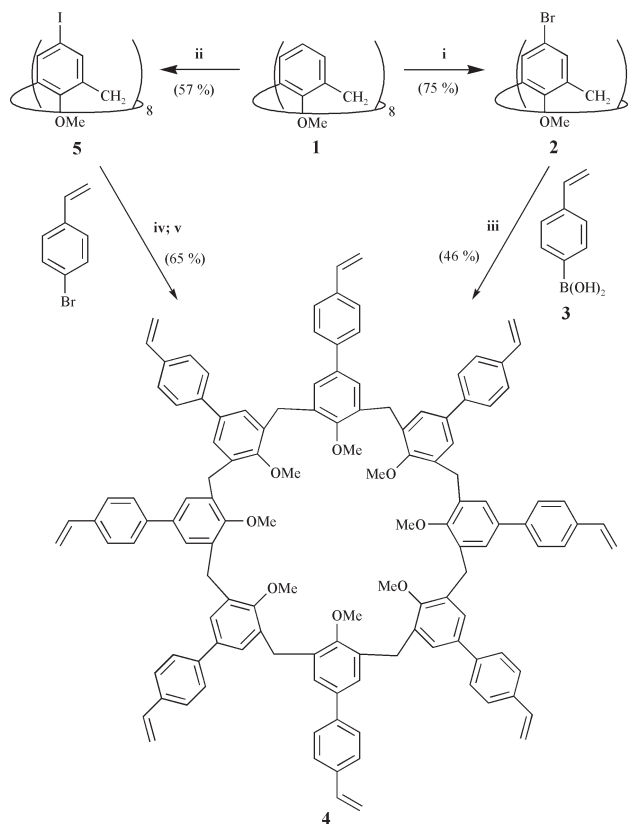
## Introduction

Calixarenes are an important class of host molecules. It has been demonstrated that they are powerful building blocks for the recognition of ions and neutral organic molecules.<sup>1</sup> Their chemistry has been extensively investigated using essentially the smallest macrocycle of this class, the calix[4]arene. Alkylation or acylation of the phenolic hydroxy groups are the simplest way to introduce functional groups in the *lower rim* of these molecules. Electrophilic substitutions can take place at the *upper rim* to introduce different functionalities. However, the small dimension of the cavity and its moderate flexibility sets a limit to their possible applications in the host–guest chemistry. To increase the depth of the cavity calix[4]arenes have been derivatized at the *upper rim* with aromatic moieties.<sup>2</sup> Calix[6]arenes and calix[8]arenes seem to be more promising candidates for the complexation of large organic molecules.<sup>3</sup> Previous studies in our laboratory<sup>4</sup> have shown that the cavity of the calix[8]arene presents the more suitable size and enough flexibility for the binding of PAHs (Polycyclic Aromatic Hydrocarbons) and thus have confirmed previously reported results.<sup>3</sup>

Calix[8]arenes can be prepared directly from the corresponding *p*-alkylphenol.<sup>2a,5</sup> Nevertheless, the drastic reaction conditions (basic catalysis and high temperature) have limited this procedure. Synthetic efforts directed towards the functionalization of the calix[8]arene have been made both on the *lower*<sup>3b,6</sup> or *upper rim*.<sup>3a,7</sup> During the course of our investigations, we were interested in the functionalization of the calix[8]arene by styryl groups. Some authors have described the introduction of aromatic moieties at the *upper rim* of calix[4]arene by Suzuki<sup>8</sup> or Negishi<sup>9</sup> biaryl cross-coupling reaction. Recently, Böhmer *et al.* described a Sonagashira cross-coupling reaction on calix[8]arene.<sup>10</sup> Nevertheless, still little is known about calix[8]arene.<sup>11</sup>

## Results and discussion

Compound **2** (Scheme 1) was synthesized in three steps from the easily available *p*-*tert*-butylcalix[8]arene.<sup>12,13</sup> The calix[8]arene was obtained with a good yield. Böhmer *et al.*<sup>10</sup> described the synthesis of compound **2** *via* an exhaustive



**Scheme 1** (i) Br<sub>2</sub>, Fe<sup>0</sup>, AcOH, CHCl<sub>3</sub>; (ii) CF<sub>3</sub>COOAg, I<sub>2</sub>, CH<sub>2</sub>Cl<sub>2</sub>; (iii) Pd(PPh<sub>3</sub>)<sub>4</sub>, Benz., MeOH, Na<sub>2</sub>CO<sub>3</sub>; (iv) BuLi, THF; (v) Pd(PPh<sub>3</sub>)<sub>4</sub>, THF.

bromination step followed by the methylation of the hydroxy groups with an overall yield of 52%. We chose to proceed to the methylation reaction before the bromination step leading to an overall yield of 69%. Thus, the free hydroxy groups were subjected to an exhaustive methylation (NaH/THF–DMF) to afford **1**. Finally, **1** was brominated with Fe<sup>0</sup>/AcOH as catalyst to give the fully *para* brominated calix[8]arene derivative **2** with a good and reproducible yield (75%)<sup>2a</sup> compared to a

recently described procedure.<sup>14</sup> **2** provides a versatile intermediate to further modifications at the *upper rim*.

The key step of the Suzuki route is the coupling reaction between the boronic acid **3** and the brominated calixarene derivative **2**. The boronic acid derivative **3** was synthesized according to the reported procedures from the commercially available *para*-bromostyrene.<sup>8e</sup> The coupling reaction occurred in the presence of palladium (0) with sodium carbonate as the base. In order to optimize the reaction, especially by choice of reaction solvent, the reaction was carried out in different conditions.

DMSO was known to be a good solvent for the Suzuki reaction,<sup>15</sup> but in our case no interesting result was obtained with this solvent. Assays conducted in a mixture of methanol and toluene led to the formation of a mixture of partially substituted calixarene derivatives. The constitution of this mixture was confirmed by mass spectra. Finally, we achieved the synthesis of the fully substituted calixarene derivative **4** in a mixture of methanol and benzene. This reaction illustrates the first example of the Suzuki type cross-coupling reaction occurring on all of the eight units of a calix[8]arene. The styryl derivative **4** was obtained with an acceptable yield after purification (46%).

In order to improve the yield of the reaction, we attempted the cross-coupling step under Negishi conditions.<sup>9</sup> The intermediate was the iodinated derivative **5** which was obtained from **1** as described in Scheme 1. The cross-coupling reaction was achieved with the zinc derivative of 4-bromostyrene. This derivative was generated *in situ* and then coupled to **5** in the presence of Pd(0). **4** was obtained with 65% yield after purification.

The structure of all compounds was determined by <sup>1</sup>H, <sup>13</sup>C NMR and mass spectrometry.

The fully substituted calix[8]arene derivatives give simple <sup>1</sup>H NMR spectra. The <sup>1</sup>H NMR spectrum of the derivative **1** is in accordance with those previously reported.<sup>13</sup> The <sup>1</sup>H NMR spectra of compounds **2** and **5** show the presence of only one phenyl ring according to the symmetry of the molecule. The signal of the Ar–CH<sub>2</sub>–Ar appears as a singlet according to the flexible conformation of calix[8]arenes in solution.

The structural identity of derivative **4** was confirmed from its HMQC NMR sequence.

As shown in this sequence, **4** exhibits a perfect symmetry of the signals. The Ar–CH<sub>2</sub>–Ar appears as a singlet in <sup>1</sup>H NMR (one signal for the <sup>13</sup>C NMR). The vinylic protons are clearly identified.

The structure of compound **2** was confirmed by its mass spectrum (MALDI-TOF).

Single crystal X-ray structures of **2** crystallized from tetrachloromethane have been obtained. A summary of the crystallographic data for this compound is listed in Table 1.<sup>†</sup>

The asymmetric unit of the calix[8]arene **2** consists of half a calixarene; the center of inversion gives the whole macrocycle. The complex is composed of one calix[8]arene derivative for two molecules of CCl<sub>4</sub> arranged in a flattened bipyramidal shape in which the two CCl<sub>4</sub> are the summits and the calixarene the base of this bipyramid; the shortest distances between the molecules correspond to Van der Waals interactions. The usual pairs of torsion angles around the Ar–CH<sub>2</sub> bonds as defined by Uguzzoli *et al.*<sup>16</sup> give the sequence: Ci (–+, +–, +–, –+). The sequence differs with the one of a pleated loop conformation Ci (–+, +–, –+, +–). Fig. 1 shows that two methoxy groups point outside the cavity and the six others are inside. This fact was found previously.<sup>16,17</sup>

Aromatic rings bearing O(25), O(26), O(27) and O(28) are called respectively R25, R26, R27 and R28. The same ones deduced by the inversion center are called R25S, R26S,

**Table 1** Selected crystallographic data for compound **2**

Chemical formula	C <sub>64</sub> H <sub>56</sub> Br <sub>8</sub> O <sub>8</sub> ·2CCl <sub>4</sub>
Formula weight	1899.98
Temperature/K	293(2)
Wavelength	0.71073 Å
Crystal system	monoclinic
Space group	C2/c
a/Å	27.894(6)
b/Å	14.665(3)
c/Å	21.659(4)
α/°	90
β/°	126.01(3)
γ/°	90
Volume/Å <sup>3</sup>	7167(2)
Calculated density/Mg m <sup>3</sup>	1.761
Z	4
Absorption coefficient/mm <sup>–1</sup>	4.827
Final R1 [ <i>I</i> > 2σ( <i>I</i> )]	0.0740
Final wR2 [ <i>I</i> > 2σ( <i>I</i> )]	0.1470
R1(all data)	0.1806
R2(all data)	0.2033
Crystal size/mm	0.22 × 0.18 × 0.04
Absorption correction	none

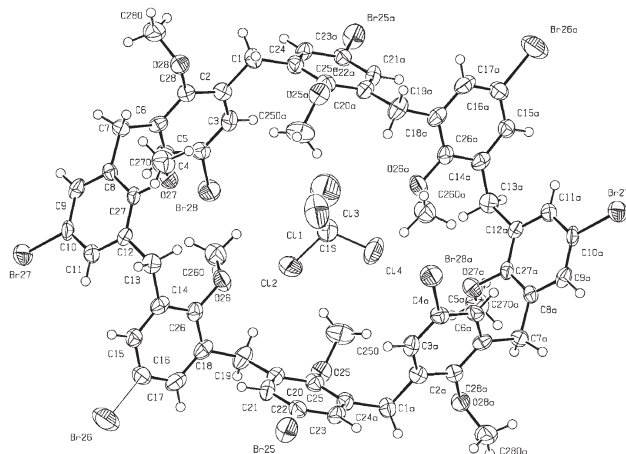
R27S and R28S (Fig. 1). The shape of the cavity is rather circular and the distances between centroids of the aromatic rings are 11.26 Å for R25, R25S, R28 and R28S, 12.73 Å for R26 and R26S and 14.59 Å for R27 and R27S, in good agreement with the values of Atwood *et al.*<sup>17</sup> Angles between the neighbouring rings R25 and R26, R26 and R27, R27 and R28, R28 and R25S are respectively 76.7(3)°, 74.9(3)°, 83.6(3)° and 83.2(3)°.

Compound **5** crystallized with two molecules of CCl<sub>4</sub> for one macrocycle. The CCl<sub>4</sub> molecule is disordered: three site occupation factors were found at 0.63, 0.25 and 0.12.

Recently, a crystal of the iodine derivative **5** was obtained but it is of poor quality. However a preliminary structure shows a very near conformation. This compound crystallizes with pyridine molecules.

## Conclusion

The work reviewed in this paper demonstrates that the calix[8]arene can be fully derivatized at the *upper rim* by a biaryl cross-coupling reaction. It is noteworthy that the Negishi conditions gave a better result than the Suzuki conditions. Nevertheless, both reactions provide convenient routes to obtain large calixarenes with suitable groups for further polymerization.



**Fig. 1** X-ray crystal structure of **2**.

<sup>†</sup> CCDC reference number 201017. See <http://www.rsc.org/suppdata/nj/b3/b302613b/> for crystallographic data in .cif or other electronic format.

Work is currently being undertaken to prepare highly cross-linked polymer resins derived from **4**.

## Experimental section

### General methods

Solvents were purified and dried by standard methods prior to use. All reactions were carried out under nitrogen. Column chromatography was performed on basic alumina from Macherey-Nagel. Reactions and column eluents were monitored by TLC on silica gel (SIL G/UV<sub>254</sub>) or alumina (ALOX N/UV<sub>254</sub>). Melting points were determined on an Electrothermal-9100 Capillary apparatus and are uncorrected. <sup>1</sup>H NMR and <sup>13</sup>C NMR spectra were obtained on a Bruker AM-300 (300.13 and 75 MHz), TMS as internal standard, chemical shifts in ppm. MALDI-TOF mass spectra were acquired using 1,8,9-anthracenetriol-NaI as the matrix. UV measurements were recorded on a Shimadzu UV-2401 PC spectrophotometer, λ<sub>max</sub> in nm, ε in M<sup>-1</sup>.cm<sup>-1</sup> (CH<sub>2</sub>Cl<sub>2</sub> as solvent).

As verified by other authors<sup>18</sup> elemental analyses of calixarenes are very often incorrect because of the inclusion of solvent molecules and thus cannot be considered as an appropriate criterion of purity. Nevertheless, the identities of the reported compounds have been confirmed by their structural data.

### Syntheses

**5,11,17,23,29,35,41,47-Octabromo-49,50,51,52,53,54,55,56-octamethoxycalix[8]arene (2).** To a cooled (0 °C) solution of the octamethoxycalix[8]arene<sup>13,17</sup> (1 g, 1.04 mmol) in 100 mL of CHCl<sub>3</sub> containing a catalytic amount of Fe metal, was added, with stirring, 1.1 mL (20.8 mmol) of Br<sub>2</sub> in 20 mL of acetic acid over a period of 30 min. The mixture was stirred at 0 °C for 1 h and then at rt until TLC analysis (SiO<sub>2</sub>, heptane/AcOEt 7:3) indicated the disappearance of the starting material (R<sub>f</sub> = 0.53). Then, the reaction was quenched by pouring it into 50 mL of 10% NaHSO<sub>3</sub>. The organic layer was separated, concentrated by steam distillation and then stirred again in 100 mL of 10% NaHSO<sub>3</sub>. The precipitate was filtered off, washed 3 times with water and with 30 mL of acetone to afford 1.24 g of white powder, which was found to be pure by <sup>1</sup>H NMR spectroscopy. Yield: 75 %. Mp = 300–302 °C. <sup>1</sup>H NMR (Pyridine-d<sub>5</sub>, 90 °C) δ 3.69 (s, 24 H, CH<sub>3</sub>), 4.11 (s, 16 H, Ar-CH<sub>2</sub>-Ar), 7.31 (s, 16 H, Ar); <sup>13</sup>C NMR (Pyridine-d<sub>5</sub>, 90 °C) δ 30.3 (Ar-CH<sub>2</sub>-Ar), 61.1 (CH<sub>3</sub>), 117.5 (Br-C, Ar), 132.3 (CH, Ar), 136.7 (C-CH<sub>2</sub>, Ar), 156.4 (MeO-C, Ar). UV: 231 (91 000). MALDI-TOF MS for C<sub>64</sub>H<sub>56</sub>O<sub>8</sub>Br<sub>8</sub> (1591.2) *m/z* = 1614.9 [M + Na<sup>+</sup>].

**5, 11, 17, 23, 29, 35, 41, 47-Octaiodo-49, 50, 51, 52, 53, 54, 55, 56-octamethoxycalix[8]arene (5).** To a suspension of CF<sub>3</sub>COOAg (1.36 g, 6.2 mmol) in refluxing dichloromethane (150 mL) were added the octamethoxycalix[8]arene (371 mg, 0.386 mmol) and iodine (3.13 g, 12.3 mmol). The heterogeneous mixture was refluxed until starting material was not detected by TLC (R<sub>f</sub> = 0.53; heptane/AcOEt 7/3; SiO<sub>2</sub>), then the yellow precipitate was removed by filtration. The organic solution was washed with a solution of Na<sub>2</sub>S<sub>2</sub>O<sub>5</sub> (10% v/v) until organic phase was permanently colourless. It is then washed twice with water, dried over Na<sub>2</sub>SO<sub>4</sub>, and then the solvent was evaporated *in vacuo* to dryness. Recrystallization from pyridine afforded 431 mg (57% yield) of **5** as a white solid. Mp: decomposed. The product was insoluble in common organic solvents. <sup>1</sup>H NMR (Pyridine-d<sub>5</sub>, 90 °C) δ 3.61 (s, 24 H, CH<sub>3</sub>), 3.77 (s, 16 H, Ar-CH<sub>2</sub>-Ar), 7.48 (s, 24H, Ar); <sup>13</sup>C NMR (Pyridine-d<sub>5</sub>) δ 30.1 (Ar-CH<sub>2</sub>-Ar), 61.2 (CH<sub>3</sub>), 89.4 (I-C, Ar), 136 (CH, Ar), 138.4 (C-CH<sub>2</sub>, Ar), 157.2 (MeO-C, Ar). ES-MS (+) for C<sub>64</sub>H<sub>56</sub>O<sub>8</sub>I<sub>8</sub> (1968.3) *m/z* = 1990.3 [M + Na]<sup>+</sup>.

**5, 11, 17, 23, 29, 35, 41, 47-Octa(4-styryl)-49, 50, 51, 52, 53, 54, 55, 56-octamethoxycalix[8]arene (4).** *Suzuki procedure.* A mixture of calixarene **2** (332 mg, 0.208 mmol), 4-styrylboronic acid (500 mg, 3.4 mmol), tetrakis(triphenylphosphine)palladium(0) (120 mg, 0.104 mmol), anhydrous benzene (5 mL), and anhydrous methanol (1 mL) was stirred at 60 °C for 30 min and then treated with 2 M aqueous Na<sub>2</sub>CO<sub>3</sub> (1.5 mL). The suspension was stirred at 60 °C for 48 h, cooled to rt, diluted with CHCl<sub>3</sub> (100 mL) and washed with 2 M aqueous Na<sub>2</sub>CO<sub>3</sub> (10 mL) containing 1 mL of concentrated NH<sub>3</sub>. The organic layer was separated, filtered off on celite and concentrated by rotary evaporation to give a dark brown oil as a crude product which was stirred in a large portion of methanol. The grey precipitate was filtered off, dried under vacuum, eluted from a column of alumina with toluene/CH<sub>2</sub>Cl<sub>2</sub> (5/4) and recrystallized from CH<sub>2</sub>Cl<sub>2</sub>/hexane to give 169 mg of a white solid. Yield: 46 %.

*Negishi procedure.* To a stirred solution of *p*-bromostyrene (680 mg, 3.6 mmol) in dry THF (3 mL) at -78 °C was added 1.6 M *n*-BuLi/hexane (2.9 mL, 4.6 mmol). The yellow solution was stirred at -78 °C for 1 h, cannulated into a solution of anhydrous ZnCl<sub>2</sub> (624 mg, 4.6 mmol) in dry THF (5 mL), and stirred for 30 min at 25 °C. The organozinc reagent was cannulated into a solution of **5** (300 mg, 0.15 mmol) and Pd(PPh<sub>3</sub>)<sub>4</sub> (90 mg, 0.076 mmol) in dry THF and stirred for 18 h at 25 °C in the dark. The orange mixture was hydrolyzed with 5M hydrochloric acid (60 mL) and extracted with CHCl<sub>3</sub> (3 × 100 mL). The organic phase was washed with brine (100 mL) and 10% Na<sub>2</sub>SO<sub>3</sub> (100 mL) and dried with Na<sub>2</sub>SO<sub>4</sub>. The solvent was removed *in vacuo* to give a brown oil which was diluted with a small amount of chloroform. A large portion of methanol is added under stirring and the resulting orange precipitate was collected by filtration. It was then refluxed in acetone (100 mL) and the insoluble materials were collected by filtration, and recrystallized from CHCl<sub>3</sub>/MeOH to give 175 mg (65 %) of **4** as a white powder.

Mp > 400 °C. <sup>1</sup>H NMR (CD<sub>2</sub>Cl<sub>2</sub>) δ 3.52 (s, 24 H, CH<sub>3</sub>), 4.13 (s, 16 H, Ar-CH<sub>2</sub>-Ar), 5.20 (d, 8 H, *J* = 10.9 Hz, *H cis*), 5.68 (d, 8 H, *J* = 17.5 Hz, *H trans*), 6.65 (dd, 8 H, *J* = 17.5 Hz and 10.9 Hz, *H gem*), 7.24–7.33 (m, 48 H, Ar); <sup>13</sup>C NMR (CD<sub>2</sub>Cl<sub>2</sub>) δ 30.8 (Ar-CH<sub>2</sub>-Ar), 61.05 (O-CH<sub>3</sub>), 113.67 (CH<sub>2</sub>=CH-Ar), 136.71 (CH<sub>2</sub>=CH-Ar), 126.79, 127.08, 127.77, 134.86, 136.34, 136.44, 140.8, 156.73 (C, Ar). UV: 231 (163 000), 280 (165 000). MALDI-TOF MS for C<sub>128</sub>H<sub>112</sub>O<sub>8</sub> (1776) *m/z* = 1800.8 [M<sup>+</sup> + Na<sup>+</sup>].

### X-ray analysis of 2

Single crystals of **2** were obtained from a saturated tetrachloromethane solution. Data were collected at 293 K on a Nonius Kappa CCD with Mo-Kα radiation (λ = 0.71073 Å). Final unit cell parameters were obtained by means of a least-squares refinement. The structure has been solved by direct methods using SHELXS97.<sup>19</sup> Usual non-H atoms were refined isotropically except for some disordered solvents. Hydrogen were calculate reported on Table 1.†

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