Organic LETTERS

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# Syntheses and Structures of Functionalized [9]Cycloparaphenylenes as Carbon Nanohoops Bearing Carbomethoxy and *N*-Phenylphthalimido Groups

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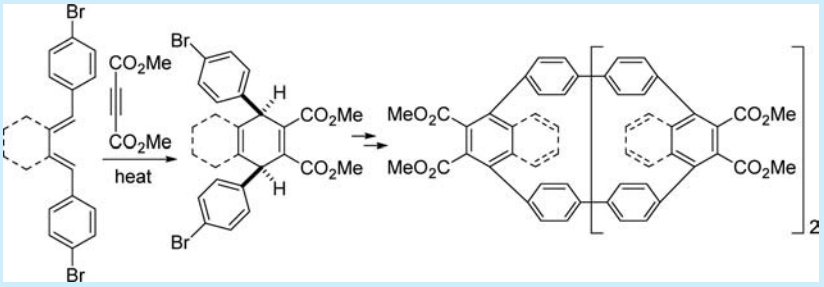
Supporting Information




**ABSTRACT:** Functionalized [9]cycloparaphenylenes ([9]CPPs) bearing nine aromatic units in the macrocyclic structures were synthesized. The macrocyclic structures were substituted with carbomethoxy or *N*-phenylphthalimido groups. The Diels–Alder reaction of (*E*,*E*)-1,4-bis(4-bromophenyl)-1,3-butadiene or a related diene with dimethyl acetylenedicarboxylate followed by the nickel-mediated homocoupling reactions and oxidative aromatization produced the functionalized [9]CPPs. Treatment of a resultant [9]CPP with aniline or 1,4-diaminobenzene gave the corresponding *N*-phenylphthalimides. The X-ray structure of a [9]CPP bearing six carbomethoxy groups was obtained.

Connecting benzene units in *para* positions in a cyclic array produces cycloparaphenylenes (CPPs), which are the shortest repeating hooplike structures of the corresponding armchair carbon nanotubes.[1] It was envisioned that CPPs could serve as templates for growing carbon nanotubes of a single chirality and diameter for nanotechnology applications.[2] In addition, CPPs are inherently interesting because they have been shown to exhibit unique size-dependent optoelectronic and redox properties.[3] The presence of well-defined cavity space provides opportunity to investigate host–guest interactions.[4] Furthermore, the unusual alignments of the radially $\pi$-conjugated systems in these molecules present a platform for the study of the effect of pyramidalization of $sp^2$-hybridized carbons on aromaticity[5] and ring size on strain energy.[6]

Several synthetic pathways to CPPs have been developed,[7] allowing the construction of various ring sizes ranging from the smallest with five benzene units, [5]CPP,[7l,m,q] to the largest with 18 units.[7a] We recently reported the use of the Diels–Alder reaction as a key step in producing a CPP precursor for the synthesis of a functionalized [9]CPP[7o] and thiophene-containing CPPs.[8] We now have further extended this strategy to the construction of other functionalized [9]CPPs by using different combinations of dienes and dienophiles for the Diels–Alder reaction to form CPP precursors.

Condensation between (*E*,*E*)-1,4-bis(4-bromophenyl)-1,3-butadiene (**1**) and dimethyl acetylenedicarboxylate (**2**) at 140 °C produced the Diels–Alder adduct **3** in 85% yield (Scheme 1).[9] It is worth noting that due to the high stereoselectivity of the Diels–Alder reaction the two 4-bromophenyl groups in **3** are *cis* to each other exclusively, which is essential for the subsequent macrocyclic ring formation. The X-ray structure of **3** showed that the included angle between the two 4-bromophenyl groups is 73.5° in the crystal lattice. Treatment of **3** with $Ni(cod)_2$ (cod: 1,5-cyclooctadiene) in the presence of 2,2′-bipyridyl (bipy) promoted the homocoupling reactions[7e] to give the macrocyclic dimers, *syn*-**4** and *anti*-**4** (1:1), and the macrocyclic trimers, *syn*-**5** and *anti*-**5** (1:3). Small fractions of the four macrocyclic products were isolated for structural elucidation. The ratio between the dimers and trimers was determined by NMR spectroscopy to be 1:10. This is in contrast to the earlier report that the homocoupling reactions of the precursor derived from **1** and 1,4-benzoquinone followed by methylation gave the corresponding macrocyclic dimers predominantly.[7o] We found that it was operationally simpler to treat the mixture of the dimers and the trimers, without further purification, with 2,3-dichloro-5,6-dicyano-1,4-benzoquinone (DDQ) to produce the fully aromatized [9]CPP **6** bearing six carbomethoxy groups in 14% yield from **3** over two steps. The corresponding fully aromatized [6]CPP was not obtained. Oxidative aromatization of the

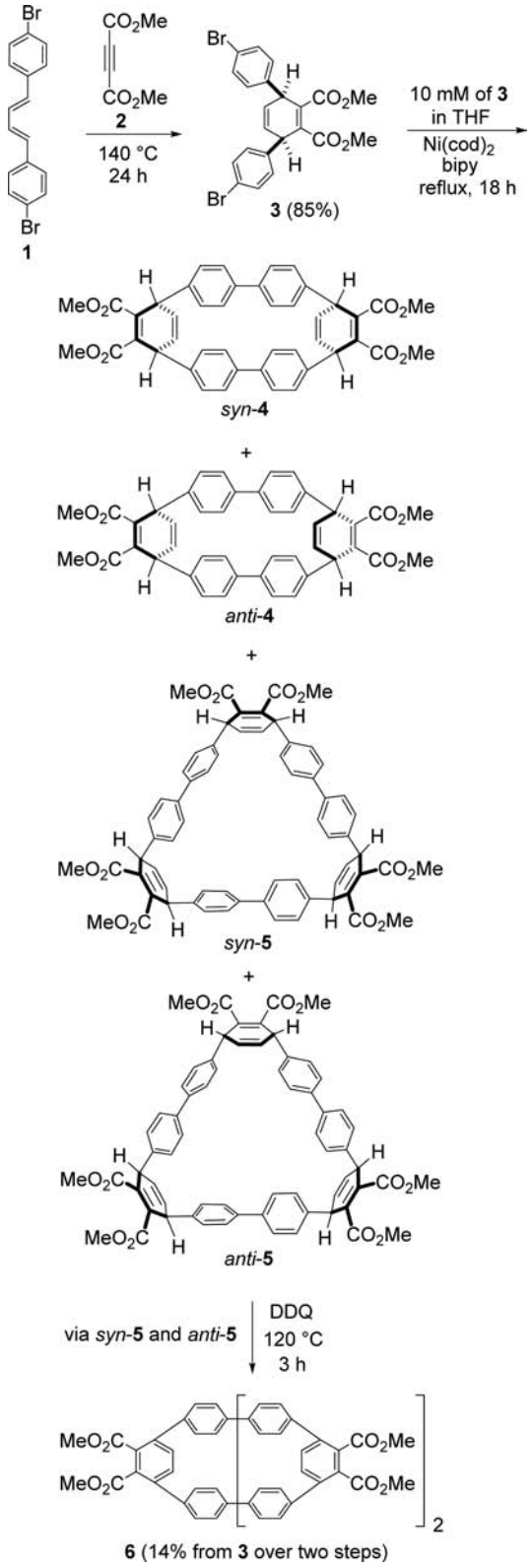
**Received:** March 29, 2016
**Published:** April 21, 2016





DOI: 10.1021/acs.orglett.6b00904
*Org. Lett.* 2016, 18, 2268–2271

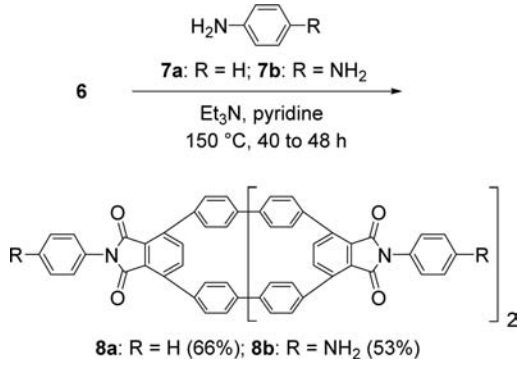
**Scheme 1. Synthesis of the Functionalized [9]CPP 6 Bearing Six Carbomethoxy Groups**




isolated *anti*-**5** with DDQ gave **6** in 80% yield. A similar result was also obtained from the isolated *syn*-**5** in producing **6**.

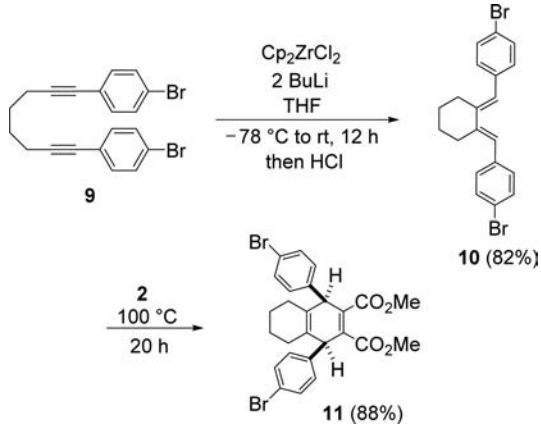
In a recent report, a [12]CPP-hexacarboxylate was synthesized by the rhodium-catalyzed cross-cyclotrimerization followed by reductive aromatization.[7s] The structure of **6** also contains six carbomethoxy groups, which on treatment with aniline (**7a**) and 1,4-diaminobenzene (**7b**) gave [9]CPPs **8a** and **8b** bearing three *N*-phenylphthalimido groups, respectively (Scheme 2).

**Scheme 2. Synthesis of [9]CPPs 8a and 8b Bearing Three *N*-Phenylphthalimido Groups**




Diene **10**, readily prepared from 1,7-octadiyne **9** and $Cp_2ZrBu_2$ followed by hydrolysis,[10] underwent the Diels–Alder reaction with **2** to give **11** in 88% yield (Scheme 3). Compared to **1**, the fixed s-*cis* configuration of the 1,3-butadienyl structure in **10** facilitates its reaction with **2**.

**Scheme 3. Synthesis of the CPP Precursor 11**



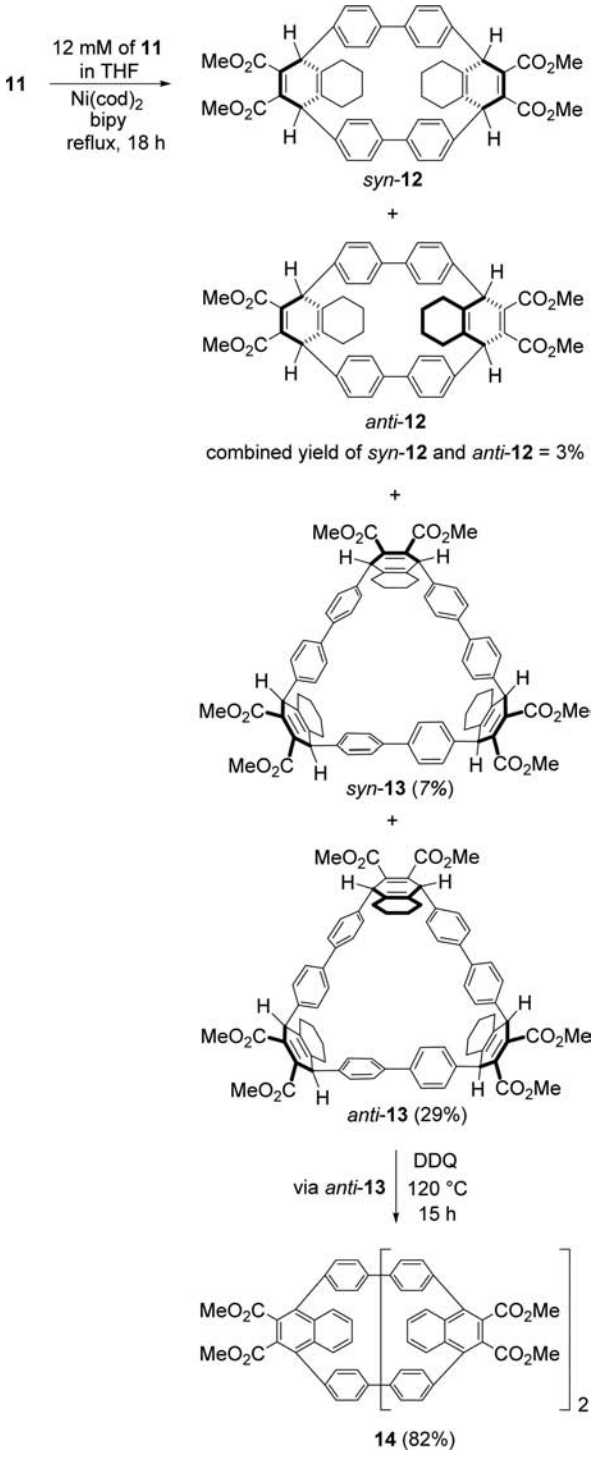

The $Ni(cod)_2$-mediated homocoupling reactions of **11** produced the macrocyclic dimers, *syn*-**12** and *anti*-**12** (1:1), in 3% combined yield, and the macrocyclic trimers, *syn*-**13** in 7% yield and *anti*-**13** in 29% yield (Scheme 4). Treatment of *anti*-**13** with DDQ then produced the fully aromatized **14** in 82% yield. Similarly, **14** was also obtained from *syn*-**13**. It is worth noting that the tetramethylene moieties were also oxidized to form part of the naphthyl systems in **14**.

The UV–vis spectra of **6** and **14** in DMSO showed the absorption maxima ($\lambda_{abs}$) at 327 and 346 nm, respectively. In comparison, the absorption maximum of the parent [9]CPP occurred at 340 nm[7a] and [9]cyclo-1,4-naphthalene occurred at 378 nm.[11] The fluorescence maxima ($\lambda_{em}$) of **6** and **14** were observed at 464 and 445 nm, respectively. They are blue-shifted from those of the parent [9]CPP at 494 nm and [9]cyclo-1,4-naphthalene at 491 nm. For **8a** and **8b**, the UV–vis absorption maxima occurred at 342 and 340 nm, respectively, and the fluorescence maxima occurred at 525 and 440 nm, respectively. It



DOI: 10.1021/acs.orglett.6b00904
*Org. Lett.* 2016, 18, 2268–2271

**Scheme 4. Synthesis of the Functionalized [9]CPP 14**




is worth noting that the fluorescence maximum of **8a** showed a significant red-shift from those of the other [9]CPPs.

A single crystal of **14** suitable for X-ray structure analysis was obtained by recrystallization from a mixture of dichloromethane and cyclohexane. The X-ray structure of **14** (Figure 1) indicates that the ester groups of the 2,3-dimethyl 2,3-naphthalenedicarboxylate units all cant toward the inner plane of the [9]CPP circle with two of them tilting above the ring while the third was tilting below the ring. This arrangement is different from that of the [12]CPP-hexacarboxylate, where the ester groups all cant away from the inner plane and are pointed toward the same side.[7s] In the crystalline state, molecules of **14** align to form a linear carbon nanotube-like structure with columnar assemblies all packed parallel to one another.

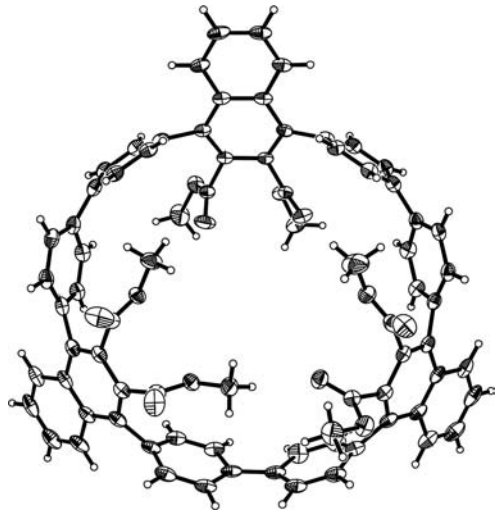


**Figure 1.** ORTEP drawing (solvents, $CH_2Cl_2$ and cyclohexane, are omitted for clarity) of [9]CPP **14**. The thermal ellipsoids are scaled to enclose 30% probability.

In summary, functionalized [9]CPPs bearing carbomethoxy or *N*-phenylphthalimido groups have been synthesized. The high stereoselectivity of the Diels–Alder reaction provides easy access to precursors **3** and **11** having two 4-bromophenyl groups *cis* to each other, which is essential for the macrocyclic ring formation. Treatment of [9]CPP **6** with aniline or 1,4-diaminobenzene produces **8a** and **8b** bearing three *N*-phenylphthalimido groups, respectively. The X-ray structure of **14** indicates an interesting tubular arrangement in the crystalline state resembling that of a carbon nanotube. The presence of six carbomethoxy groups in **6** and **14** provides the potential opportunity for them to serve as hinges to connect multiple units of CPPs together in a nanotube-like arrangement. The results of this research will be presented in due course.

## ■ ASSOCIATED CONTENT

### Supporting Information

The Supporting Information is available free of charge on the ACS Publications website at DOI: 10.1021/acs.orglett.6b00904.

Complete experimental details of new compounds; spectral data (PDF)

Crystallographic data (CIF)

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### Notes

The authors declare no competing financial interest.

## ■ ACKNOWLEDGMENTS

This material is based upon work supported by the National Science Foundation under CHE-1464026. The NMR spectrometer on which this work was carried out was supported by the Major Research Instrumentation (MRI) Program of the NSF (CHE-1228336). J.L.P. acknowledges the financial support provided by the MRI Program of the NSF (CHE-1336071) for



DOI: 10.1021/acs.orglett.6b00904
*Org. Lett.* 2016, 18, 2268–2271

the acquisition of a Bruker AXS D8 Venture X-ray diffractometer. B.V.P. acknowledges the financial support by the NSF/MRI Program (CHE-1427136) for the acquisition of an in situ IR spectroscopy system. We thank Gregory C. Donohoe and Jessica A. Rogers of West Virginia University for their assistance in obtaining the high resolution mass spectra and the operation of the in situ IR spectroscopy system, respectively.

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DOI: 10.1021/acs.orglett.6b00904
*Org. Lett.* 2016, 18, 2268−2271