

Letter

Tandem Oxidative Ring Expansion for Synthesis of Dibenzocyclooctaphenanthrenes

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Cite This: https://dx.doi.org/10.1021/acs.orglett.0c01725 **Read Online** ACCESS III Metrics & More Article Recommendations **SUPPORTING Information** Tandem Oxidative Ring Expansion ABSTRACT: A novel tandem single-electron oxidative ring expansion reaction has been developed for the construction of the saddle-shaped polycyclic arenes DDQ / Cu(OTf)₂ fused with cyclooctatetraene, that is, dibenzo[3,4:7,8]cycloocta[1,2-l]-

phenanthrenes (dbCOTPs). The combination of Cu(OTf)₂ catalyst with DDQ triggered the selective oxidation of *o*-biphenyl-tethered methylenecirculenes fused with a seven-membered ring, giving rise to the formation of the corresponding eight-membered ring fused dbCOTPs. The present tandem ring expansion of a



seven- to an eight-membered ring takes place via the selective single-electron oxidation of the benzylidene moiety, intramolecular spirocyclization, and 1,2-aryl migration sequence.

Polycyclic arene units with negative curvature are prevalent in various curved nanographenes and hypothetical toroidal carbon nanotubes, which have attracted increasing attention in the field of computation and synthetic chemistry as well as materials science in terms of their negatively curved structural features and electronic properties.¹⁷² Embedding seven- or eight-membered carbon rings into the polycyclic aromatic hydrocarbon (PAH) frameworks has been regarded as an intriguing strategy for the construction of the negatively curved polycyclic arenes.³ Particularly, polycyclic arenes fused with an eight-membered cyclooctatetraene (COT) and benzenoid rings, such as tetraphenylene,⁴ dibenzo [a,e] [8]annulene (dbCOT),⁵ tribenzo[a,c,e][8]annulene (tribCOT),⁶ and dibenzo[3,4:7,8]cycloocta[1,2-l]phenanthrene (dbCOTP),⁷ displayed negatively bent structures owing to the tub-shaped geometry of COT (Scheme 1a). They not only can serve as important key segments of negatively curved nanographenes¹⁻³ and Mackay crystals⁸ but also can act as COT ligands for transition metal complexes^{5e} and host molecules for dynamic molecular recognition.⁹ In this context, the development of efficient synthetic methods for COTembedded polycyclic arenes is of great significance and highly demanded. However, in contrast to the well-studied tetraphenylene and dbCOT,^{4,5} synthetic methods to tribCOT and dbCOTP are significantly rare. Nonetheless, tribCOT can be synthesized by Diels-Alder cycloaddition of a strained acetylene of 5,6-didehydrodibenzo[a,e]cyclooctene with furan and subsequent deoxygenation,⁶ but only a single example was reported for dimethyl-substituted dbCOTP, through cycloaddition of dienediyne followed by an intramolecular homocoupling reaction (Scheme 1b).

Recently, we are interested in developing new synthetic methodologies for π -extended polycycles using single-electron oxidation of functionalized methylene fluorenes.¹⁰ Particularly, we have discovered that under the single-electron oxidation

Scheme 1. Representative COT-Embedded Polycyclic Arene Units and Synthetic Methods

(a) Representative eight-membered ring fused polycyclic arene units



(b) Reported single example for synthesis of dimethyl-substituted dbCOTP



(c) This study: new and general synthetic method for dbCOTPs:oxidative ring expansion of seven- to eight-membered ring



conditions, a single-electron oxidation took place at the alkene moiety of the o-biphenyl-tethered methylene fluorene, giving rise to the ring expansion of a five- to a six-membered ring to

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construct various π -extended PAHs.^{10b} In light of this fact, we envisioned that if the oxidative ring expansion takes place for the *o*-biphenyl-tethered methylene dibenzo[7]annulene **1b**, a new class of polycyclic arene **2b** fused with an eight-membered ring and benzenoid rings could be formed (Scheme 1c). This method may provide a promising and general synthetic approach to the eight-membered ring embedded polycyclic arenes, although it is predicted to suffer from problems with regard to varied redox potentials and two alkene moieties of the **1b** structure for the selective single-electron oxidation. Herein, we describe a novel single-electron oxidative ring expansion of a seven- to an eight-membered ring for the construction of a new series of saddle-shaped dbCOTPs fused with an eight-membered ring and benzenoid rings.

First, the readily available substrate 5-((4'-methyl-biphenyl-2-yl)methylene)-5*H*-dibenzo[a,d][7]annulene (BMDBA) (1a) was subjected to various oxidation conditions to develop a new ring expansion reaction to access desired product 2a (Table 1).

Table 1. Screening of Oxidation Conditions for Ring Expansion Reaction of $1a^a$



^{*a*}Reaction conditions: **1a** (0.1 mmol), oxidant (1.5 equiv or 2.5 equiv), acid, CH_2Cl_2 (0.2 M), at 40 °C for 12 h. ^{*b*}Yields were determined by ¹H NMR spectroscopy using CH_2Br_2 as an internal standard. ^{*c*}The reaction was performed in *o*-xylene (0.2 M) at 80 °C. ^{*d*}The reaction was performed in 1,2-dichloroethane at 80 °C. ^{*e*}An unknown product was obtained in 20% yield. ^{*f*}Yield of isolated product is shown after silica gel chromatography.

Unfortunately, the reactions with our previously developed two oxidation systems,^{10b} CuCl/PhCO₃^tBu and DDQ (2,3-dichloro-5,6-dicyanobenzoquinone)/CF₃CO₂H, did not produce the desired product **2a** and **1a** was recovered in 30% and 44% yield, respectively (entries 1 and 2). The use of a common single-electron oxidant of FeCl₃^{10a} did not give the desired product **2a**, but instead, an unknown structural isomer of **2a** (not Scholl reaction product) was obtained in 20% yield (entry 3). Moreover, the use of DDQ oxidant without any additives could not trigger the desired tandem oxidation reaction (entry 4). It was reported that the combination of DDQ with strong

acids may improve the single-electron oxidation ability, enabling the formation of radical cation species.¹¹ After an extensive survey of the reaction conditions, we found that the combination of DDQ (1.5 equiv) with a catalytic amount of the strong acid, namely, trifluoromethanesulfonic acid (TfOH), was able to induce oxidative tandem ring expansion transforming compound 1a into 2a in 36% yield along with the decomposition of 1a (entry 5). To our delight, the use of Lewis acidic Cu(OTf)₂ catalyst instead of TfOH could further increase the yield of 2a to 52% (entry 6). It was also noted that the use of $Cu(OTf)_2$ catalyst without DDQ led to no reaction. Finally, we were able to obtain 2a in a good yield of 71% by using an excess of DDQ (2.5 equiv) combined with $Cu(OTf)_2$ (30 mol %) (entry 7). The use of other oxidants such as pchloranil and PhCO₃^tBu in conjunction with the Cu(OTf)₂ catalyst resulted in no reaction (entries 8 and 9). Other nontriflate copper salts such as $Cu(NTf_2)_2$, $Cu(BF_4)_2$, and CuCl₂ combined with DDQ were examined to be totally ineffective for the formation of 2a, resulting in decomposition or recovery of 1a (entries 10-12). Further screening of other metal triflates revealed that $Fe(OTf)_3$ and $In(OTf)_3$ were also effective in achieving moderate yields of 2a (entries 13 and 14), indicating the important role of metal triflates for the successful implementation of the present tandem ring expansion reaction.

With the optimized $DDQ/Cu(OTf)_2$ oxidation system in hand, the substrate scope has been investigated to understand the influence of substituent effect on the oxidation reactivity (Scheme 2). The reaction of 1b without any substituents on the aromatic rings afforded the corresponding dbCOTP 2b in a good yield of 65%. The reactions of 1c and 1d with electronrich substituents such as n-heptyl and tert-butyl at the 4'position of the o-biphenyl moiety (R^1) favor the present oxidation, giving the corresponding dbCOTPs 2c and 2d in 68% and 80% yields, respectively. The BMDBA 1e bearing an electron-withdrawing ester group at R¹ showed low efficiency, producing the corresponding product 2e in 38% yield. The electronic effect on the reaction efficiency implied the involvement of a Friedel-Crafts-type cyclization process. The reaction of 1f having a phenyl substituent at R¹ proceeded uneventfully to afford the desired product **2f** in 72% yield. The substrate 1g bearing a methyl substituent (R^1) at the 3'position of the o-biphenyl moiety showed steric effect on the reaction outcome, affording the corresponding product 2g in 37% yield. The introduction of a strong electron-withdrawing nitro group (R^2) to the 5-position of *o*-biphenyl lowered the reactivity of 1h and 1i, but the corresponding products 2h and 2i could be obtained in good yields at the elevated temperature of 60 °C using excess amounts of DDQ (4 equiv) and Cu(OTf) (60 mol %). Likewise, the substrates 1j and 1k bearing an electron-withdrawing F or Cl at the 4-position and an electron-donating *tert*-butyl at the 4'-positions of *o*-biphenyl individually worked well at 60 °C with excess amounts of DDQ and $Cu(OTf)_{2}$, yielding the corresponding product 2j and 2k in good to high yields. The substrates 1l and 1m containing electron-withdrawing monobromo- or dibromosubstituents on the dibenzo [a,d] [7] annulene moiety also underwent the present tandem oxidative ring expansion reaction, affording the corresponding products 2l and 2m in moderate yields, which can be further functionalized by various coupling reactions. The introduction of an electron-donating methoxy group into the 5*H*-dibenzo[a,d][7]annulene moiety $(1n, R^3 = OMe)$ showed higher reactivity compared to 11 and

Scheme 2. Substrate Scope of Oxidative Ring Expansion of o-Biphenyl-tethered Methylene Dibenzo[a,d][7]annulenes^a



^{*a*}Reaction conditions: 1 (0.2 mmol), DDQ (0.5 mmol, 2.5 equiv), Cu(OTf)₂ (0.06 mmol, 30 mol %), CH₂Cl₂ (0.2 M), at 40 °C for 12 h. ^{*b*}DDQ (4 equiv) and Cu(OTf)₂ (60 mol %) were used at 60 °C for 12 h. ^{*c*}DDQ (4 equiv) and Cu(OTf)₂ (30 mol %) were used at 60 °C for 12 h. ^{*d*}The reaction time was 6 h.

1m having electron-withdrawing groups, affording the corresponding product 2n in 57% yield under the standard conditions within 6 h.

The structure of **2b** was determined unambiguously by X-ray crystallography (Figure 1a). The X-ray structure indicated that



Figure 1. (a) ORTEP drawing of the dbCOTP **2b**. Thermal ellipsoids are shown at 50% probability. (b) The NICS(0) values were calculated using Gaussian09 at the B3LYP/6-31G(d,p) level.

2b is a saddle-shaped molecule consisting of peripheral benzenoid rings, a planar phenanthrene moiety, and a tub-shaped COT ring. The C16–C18 bond length (1.336 Å) corresponding to the nonsubstituted double bond of COT is shortest compared to C2–C9 (1.401 Å) and C3–C5 (1.359 Å) adjacent to the benzene and phenanthrene moieties,

respectively. The unidentical bond angles in COT are measured to be 125.1° for C10-C18-C16, 122.2° for C4-C10-C18, 120.9° for C3-C4-C10, and 121.1° for C4-C3-C5. The torsion angles of the COT in 2b are 59.2° for C4-C10-C18-C16 and 73.7° for C5-C3-C4-C10, respectively, which are larger than that of the parent COT (56°) .¹² The aromaticity of 2b was investigated with the nucleusindependent chemical shift (NICS) by the calculation at the B3LYP/6-31G(d,p) level (Figure 1b). The NICS(0) values of -8.7 ppm and -9.9 ppm corresponding to the peripheral benzene A and the phenanthrene benzene D, respectively, indicate their remarkably high aromaticity compared to the moderate aromatic character of the peripheral benzene C (-5.7 ppm). The NICS(0) value of the COT ring B was calculated to be 2.4 ppm, indicating its nonaromatic character. The UV/vis absorption and fluorescence spectra of 2b were measured in diluted chloroform solution (Figure S1 in the Supporting Information). A strong absorption maximum (λ_{abs}) value at 261 nm and two weak λ_{abs} values at 297 and 308 nm were observed, which are relatively short compared to planar PAHs probably due to the highly nonplanar structure of 2b.^{10b} The fluorescence spectra of **2b** showed emission maxima (λ_{em}) values of 361, 375, and 395 nm at the excited wavelength of 300 nm with a low fluorescence quantum yield of 5.2%.

In light of the structural analysis and the nonaromaticity, we thought that the bared double bond of COT in **2b** may exhibit typical olefin reactivity. Thus, to demonstrate the synthetic utility of the COT-embedded products, we carried out the additional functionalization of dbCOTPs. Oxidation of the bared alkene moiety of COTs in 2b and 2d with benzeneseleninic anhydride produced the corresponding diketone 3b and 3d in good yields. The condensation of 3b and 3d with 1,2-diaminobenzene, respectively, in the presence of *p*-toluenesulfonic acid (*p*-TSA) catalyst afforded a new class of quinoxaline-fused dbCOTPs 4b and 4d in quantitative yields (Scheme 3a).¹³ In addition, the selective bromination of 2b and the subsequent elimination of the resulting 9,10dibrominated product 5b with t-BuOK produced a strained cyclic alkyne **5b**' (Scheme 3b). **5b**' was subjected directly with 1,3-diphenylisobenzofuran to the Diels-Alder cycloaddition, affording the oxygen-bridged cycloadduct 6b. Subsequently,

Scheme 3. Synthetic Applications of dbCOTPs for π -Extended Polycyclic Arenes



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Scheme 4. Proposed Oxidative Ring Expansion Reaction Mechanism



the deoxygenation and aromatization of **6b** using the Wong's low-valent titanium gave the corresponding π -extended polycyclic arene **7b** in 85% yield.¹⁴

It was found that two alkene moieties in 1b were calculated to possess high electron density by frontier molecular orbital calculation (Figure S2 in the Supporting Information). Among them, the benzylidene alkene was predicted to undergo singleelectron oxidation preferentially compared to the alkene in the seven-membered ring, owing to the formation of a rather stable radical cation species E of the former (Scheme 4).^{1d} In light of the electronic substituent effect, indispensable role of oxidant, and electron density distribution, the proposed reaction mechanism of the present tandem oxidative ring expansion reaction is outlined in Scheme 4. First, the single-electron oxidation occurs by the DDQ/Cu(OTf)₂ oxidation system at the electron-rich alkene moiety of 1b to form a radical cation species E. Subsequent intramolecular Friedel-Crafts reaction affords a spirocyclic radical species F, which will be further oxidized to give a spirocyclic cation G. Next, the intermediate G undergoes the 1,2-aryl migration through the formation of an arenium cation **H** (path I) or the direct migration (path II), affording the cation I. The rapid deprotonation takes place in the cation I to give the desired product 2b. To support the reaction mechanism, the spirocyclic compound 1a' was subjected to our standard conditions (eq 1). As predicted,



the reaction of 1a' did produce the desired 1,2-aryl migration product 2a, even though the yield was low, suggesting the involvement of the intermediate F and G in the present tandem oxidation. In addition, the reactions of 1a under the standard conditions were completely hampered by adding radical scavengers of 2,2,6,6-tetramethylpiperidine 1-oxyl (TEMPO) or 2,6-di-*tert*-butyl-4-methylphenol (BHT), implying that the single-electron oxidation takes place in the initial step (Scheme S5 in the Supporting Information).

In conclusion, we have developed a novel and general synthetic method for constructing a new class of polycyclic

arenes embedded with a COT ring by the tandem oxidative ring expansion. The *o*-biphenyl-tethered methylene dibenzo[7]annulenes fused with a seven-membered ring could be oxidized by the DDQ/Cu(OTf)₂ oxidation system efficiently to the corresponding saddle-shaped dibenzo-[3,4:7,8]cycloocta[1,2-*l*]phenanthrenes fused with an eightmembered ring through a single-electron oxidation, spirocyclization, and 1,2-aryl migration tandem process. The present protocol provides a new synthetic approach to polycyclic arenes fused with an eight-membered ring, which is expected to be highly applicable for the synthesis of diverse negatively curved nanocarbons.

ASSOCIATED CONTENT

Supporting Information

The Supporting Information is available free of charge at https://pubs.acs.org/doi/10.1021/acs.orglett.0c01725.

Experimental procedures and characterization of related compounds (PDF)

X-ray structure report for 2b (CCDC 1994572) (PDF)

Accession Codes

CCDC 1994572 contains the supplementary crystallographic data for this paper. These data can be obtained free of charge via www.ccdc.cam.ac.uk/data_request/cif, or by emailing data_request@ccdc.cam.ac.uk, or by contacting The Cambridge Crystallographic Data Centre, 12 Union Road, Cambridge CB2 1EZ, UK; fax: +44 1223 336033.

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Notes

The authors declare no competing financial interest.

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