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Ring Closing Metathesis Approach for the Synthesis of *o*-Terphenyl Derivatives

Shilpi Karmakar,[a] Tirtha Mandal[a] and Jyotirmayee Dash*[a]

Dedication ((optional))

Abstract: A linear synthesis of *o*-terphenyls derivatives has been delineated using ring closing metathesis (RCM) as the key step. In this approach, benzil derivatives upon allyl Grignard addition provides diphenyl-1,2-diallyl dihydroxy derivatives which undergo ring closing metathesis to afford tetrahydro terphenyl derivatives. Aromatization-driven dehydration then leads to a diverse set of electron rich and electron deficient *o*-terphenyls. Furthermore, oxidative coupling of electron rich *o*-terphenyls provides the corresponding triphenylene derivatives.

Introduction

Polycyclic aromatic hydrocarbons (PAHs)[1] are a ubiquitous group of several hundred chemically related compounds having two or more single or fused aromatic rings. Terphenyls and triphenylene systems are important PAHs owing to their structural diversity and wide applications in various fields such as supramolecular and organic material chemistry.[2-5] Terphenyls serves as leading components in organic and optoelectronic devices. They are often exploited for designing organic electroluminescent devices and liquid crystalline materials.[4] Synthetic terphenyls also show significant biological activities like immunosuppressant, neuroprotective, antithrombotic, anticoagulant, specific 5-lipoxygenase inhibitory and cytotoxic activities.[3]

Several methods have been developed for the synthesis of *o*-terphenyls.[6] A rational route for the synthesis of substituted *o*-terphenyl intermediate is palladium catalyzed cross-coupling (Suzuki-Miyaura) reactions involving aryl boronic acids (Scheme 1, a). Considerable efforts have been made for the synthesis of functionalized Pd ligands in order to carry out the reaction under mild conditions as well as to improve the yield of o-terphenyls. Gu *et al*. synthesized symmetrical *o*-terphenyls by developing acid-based ligand.[6i] Lu *et al*. developed preligand for facile synthesis of sterically hindered *o*-terphenyls.[6k] Miguez *et al*. designed synthesis of unsymmetrical *o*-terphenyls at room temperature using ligandless palladium catalyst.[6a] Besides Pd catalysis, other routes have been developed for the synthesis of *o*-terphenyls;[6] for instance, synthesis involving 6π electrocyclization of *cis*-triene intermediate, developed by Lim *et al*. (Scheme 1, b).[6f]

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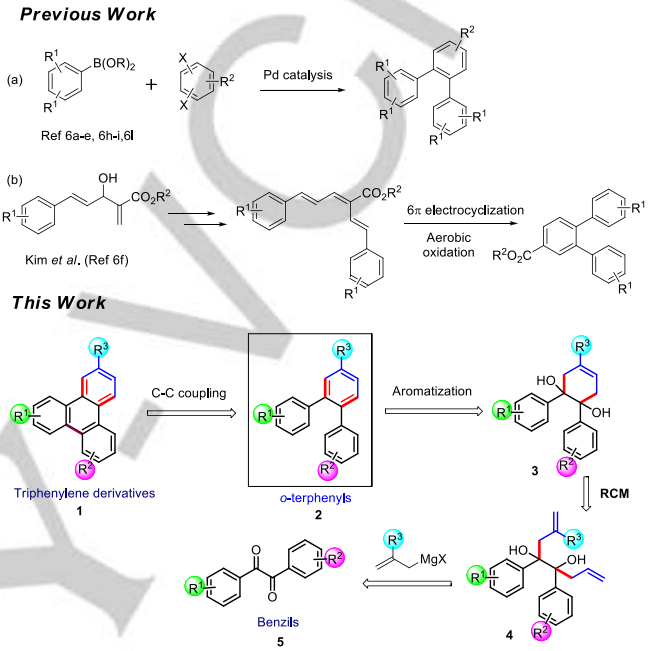


Scheme 1. Reported methods and our strategy for the synthesis of *o*-terphenyls and triphenylene derivatives.

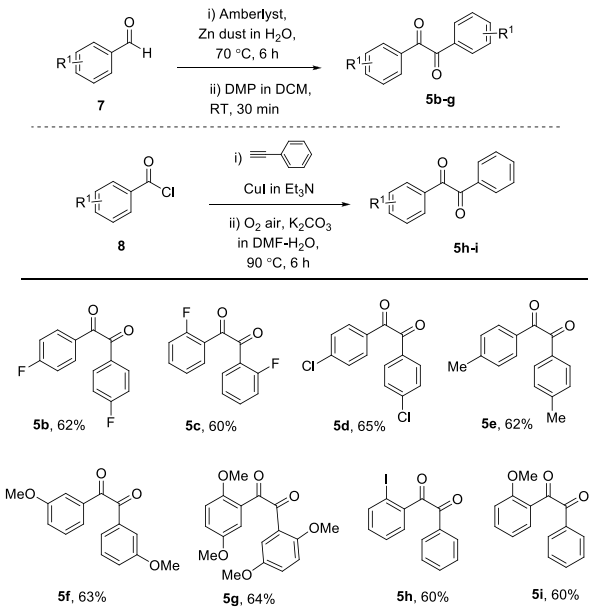
Our interest is to develop an alternate and convenient protocol for the synthesis of *o*-terphenyls from readily available starting materials by using ring closing metathesis as the key step.

Results and Discussion

Olefin metathesis based approaches have received considerable attention for creating aromatic and heteroaromatic compounds.[7-10] In continuation of our work on RCM based approaches for the synthesis of biologically significant compounds,[9,10] we herein disclose the synthesis of *o*-terphenyl scaffolds using ring closing metathesis[8] (RCM) as the key step. We have synthesized both symmetrical and unsymmetrical *o*-terphenyls using 1,2-diphenylethane-1,2-dione (benzil) derivatives[11] as our synthetic precursors. In our approach, triphenylenes **1** could be obtained via C-C coupling[12] of *o*-terphenyls **2**, which could be acquired by dehydration of RCM products **3**. The allyl metal addition (Grignard reaction)[13] of benzils **5** could provide the diallylated products **4** as RCM precursors for constructing the tricyclic diols **3**.

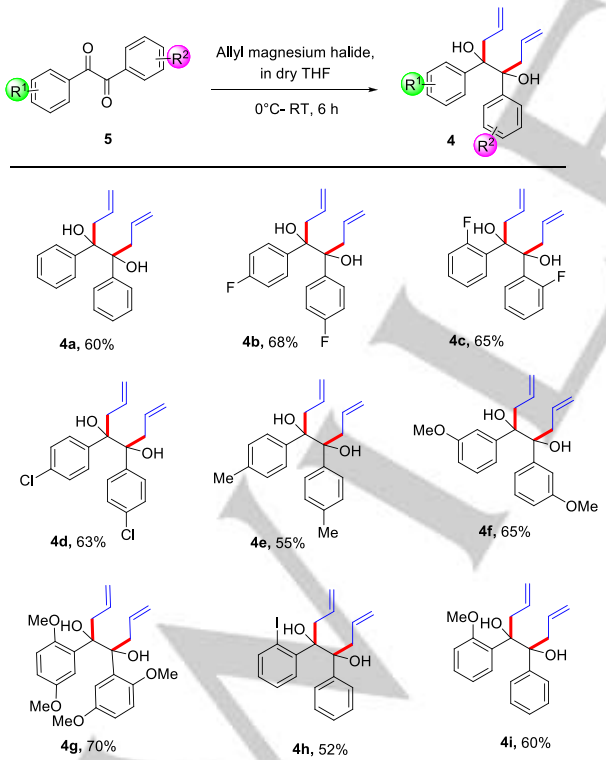
As benzil derivatives **5b-i** are not commercially available, they were prepared from the corresponding aromatic aldehydes using previously reported procedures (Scheme 2). Symmetrical benzil derivatives **5b-g** were prepared from the corresponding

aromatic aldehydes by pinacol coupling followed by Dess-Martin periodinane (DMP) mediated oxidation.[14] Unsymmetrical benzils **5h-i** were prepared from corresponding acid chlorides by phenylacetylene addition forming alkynone intermediates followed by cleavage of the C-C bond using molecular oxygen.[11g,15]



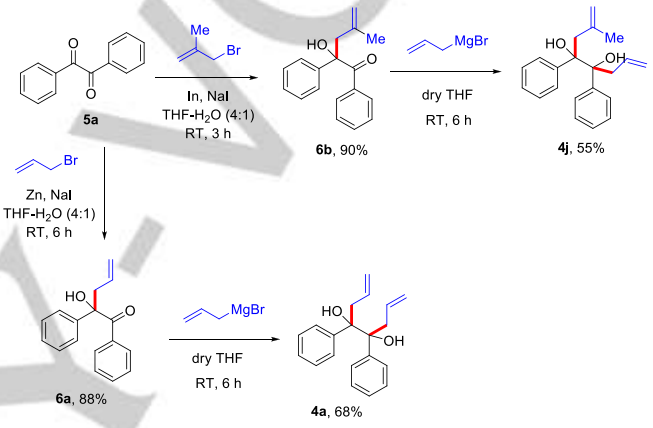
Scheme 2. Synthesis of symmetrical and unsymmetrical benzil derivatives.

Table 1. Synthesis of bisallylated diol derivatives[a]



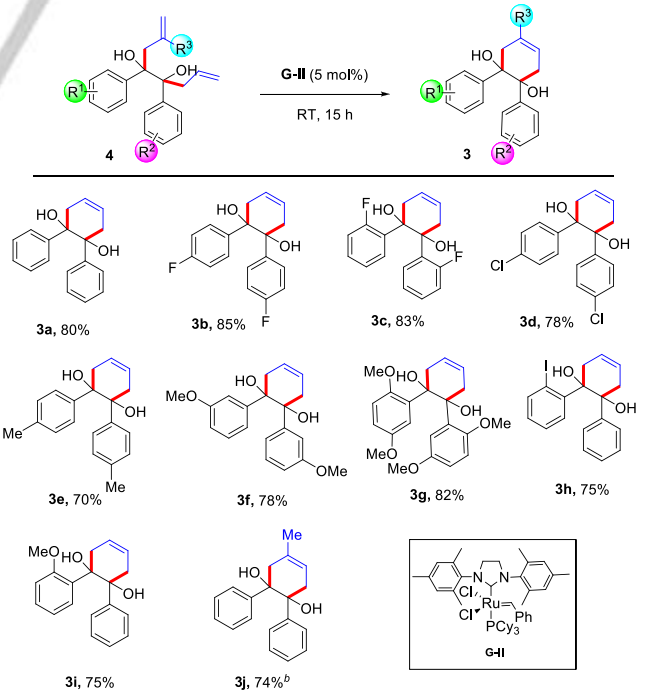
[a]The addition of allylmagnesium halide (3.0 equiv, 1 M in THF) to **5a-i** (2.0 mmol, 1.0 equiv) was carried out in THF (10 mL) at 0 °C and stirred at rt for 6 h.

To examine the feasibility of our approach (Scheme 1), benzil **5a** was treated with allylmagnesium bromide in THF at room temperature providing diallylated compound **4a** in 60% yield. The reaction of allylzinc bromide with benzil **5a** provided exclusively monoallyl compound **6a**. Compound **6a** further reacted with allyl Grignard reagent to obtain the diallyl compound **4a**. We then explored the generality of the reaction by allyl Grignard addition to electron rich and electron deficient benzils. Symmetrical benzil derivatives **5a-g** afforded the corresponding symmetrical diallylated diols **4a-g** in 55-70% yield whereas the unsymmetrical benzils **5h-i** produced the corresponding unsymmetrical diallylated products **4h-i** in 52-60% yield (Table 1). The diol **4j** containing a substituted allyl group was synthesized from benzil **5a** by sequential allyl indium and Grignard reactions. First, 2-methylallyl group was incorporated using indium-mediated allylation with 2-methylallyl bromide to obtain homoallylic alcohol **6b**, which was treated with allyl Grignard reagent to give diol **4j** (Scheme 3).



Scheme 3. Allyl metal addition to benzil **5a**.

Table 2. Ring closing metathesis (RCM) of the dialkene precursors[a]

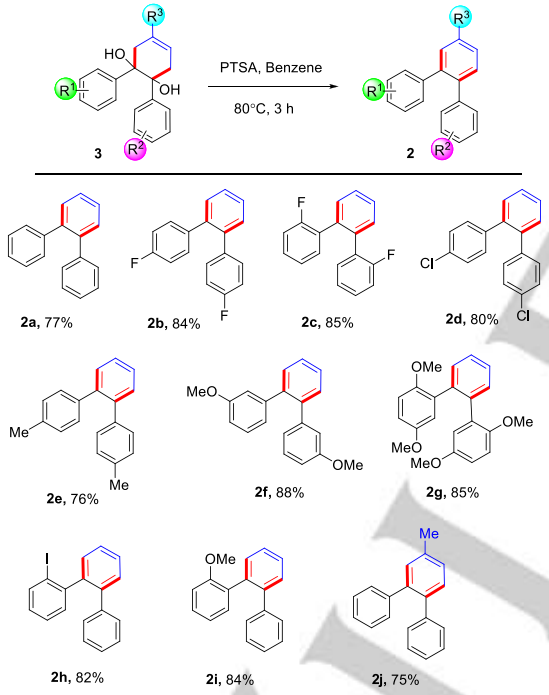


[a]The reactions were performed using **4a-i** (0.3 mmol, 1.0 equiv) with **G-II** catalyst (5 mol %) in CH_2Cl_2 (8 mL) at rt for 15 h. [b]The reaction was performed using **4j** (0.3 mmol, 1.0 equiv) with **G-II** catalyst (5 mol %) in CH_2Cl_2 (8 mL) at 50 °C for 3 h.

Using the synthesized bis-allylated diol precursors **4**, the ring closing metathesis (RCM) was next explored. In this step, 1,2-diallyl derivatives **4a-j** were subjected to RCM with Grubbs' catalyst in dry CH_2Cl_2 to obtain the desired tricyclic diols **3a-j** in excellent yields (Table 3). RCM of the dialkene **4a** with 10 mol% of Grubbs' first-generation catalyst (**G-I**)[15] in dry CH_2Cl_2 at room temperature provided the desired tetrahydro terphenyl derivative **3a** in 78% yield, after separation by column chromatography. Treating **4b** with 10 mol% **G-I** provided **3b** in poor yield. However, the yield was improved by employing 5 mol% Grubbs' second-generation catalyst (**G-II**)[16]. Thus, RCM of **4b-i** was performed with 5 mol% **G-II** providing **3b-i** in high to excellent yields (Table 2). Again, treating **4j** with 5 mol% **G-II** at room temperature provided **3j** with poor yield. However, the yield could be improved by carrying out the reaction at 50 °C for 3 h.

Next, we synthesized the corresponding *o*-terphenyls **2** by dehydration of the RCM products **3** (Table 3). We observed that dehydration of tetrahydro terphenyl derivative **3a** using 30 mol% *p*-toluenesulfonic acid (PTSA) in benzene at 80 °C for 3 h provided *o*-terphenyl **2a** in 77% yield. Subsequently, *p*-toluenesulfonic acid (PTSA) assisted dehydration of tricyclic diols **3b-j** under similar reaction conditions led to aromatization, providing the desired *o*-terphenyls **2b-j** in excellent yields.

Table 3. Aromatization driven dehydration of the tricyclic diol compounds[a]

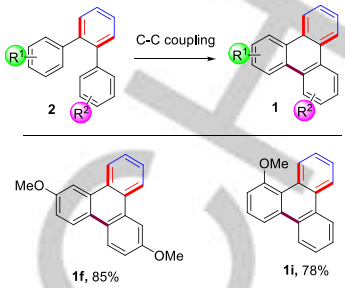


[a]The reactions were performed using **3** (0.3 mmol, 1.0 equiv), PTSA (0.3 equiv) in C_6H_6 (8 mL) at 80 °C for 3 h.

It is intriguing to find out that the reaction time or yields is not influenced by the electronic character of the substrates and in all cases, the corresponding *o*-terphenyls **2b-j** were isolated in 75-88% yield. The *o*-terphenyls containing halogen substituents such as **2b**, **2c**, **2d**, **2h** are important precursors which can be further employed as coupling partners in different organometallic transformations.

Furthermore, we envisioned that C-C coupling of *o*-terphenyl derivatives could afford triphenylene systems. Following literature procedure, Scholl reaction was performed by treating *o*-terphenyl with $FeCl_3$ in CH_2Cl_2/CH_3NO_2. We observed that the electron rich *o*-terphenyls containing methoxy group (s) **2f** and **2i** underwent cyclization to form triphenylene derivatives **1f** and **1i** whereas the electron deficient and unsubstituted *o*-terphenyls remained inert under the reaction conditions (Table 4).

Table 4. Synthesis of triphenylene derivatives from *o*-terphenyls[a]



[a]The reactions were performed using **2f**, **2i** (0.3 mmol, 1.0 equiv.) in CH_2Cl_2 (8 mL) and $FeCl_3$ (10.0 equiv.) in CH_3NO_2 (120 µL) at rt for 24 h.

Conclusions

In conclusion, the synthesis of *o*-terphenyls has been delineated from easily accessible starting materials using simple organic transformations. Allyl Grignard addition to substituted benzil derivatives provides diallylated benzil derivatives suitable for RCM. RCM has been employed effectively for the synthesis tricyclic skeleton of the *o*-terphenyls. Aromatization driven dehydration of the RCM products enable the synthesis of desired symmetrical and unsymmetrical *o*-terphenyls containing electron rich and electron deficient groups. Moreover, electron rich *o*-terphenyls have been used to obtain triphenylene derivatives via oxidative cyclization. The scope of this method with the aim of evaluating its application for the synthesis of diverse aromatic and heteroaromtic framework is currently under study.

Experimental Section

All experiments were carried out in flame-dried reaction vials. Solvents were dried using standard procedures. All starting materials were obtained from commercial suppliers and used as received. Products were purified by flash chromatography on silica gel (100-200 mesh). Unless otherwise stated, yield refers to analytical pure samples. Melting point is measured using melting point apparatus. IR spectra were recorded using FT-IR Spectrometer. NMR spectra were recorded in $CDCl_3$. ^1H NMR spectra were recorded using 500 MHz and 400 MHz instruments at 278 K. Signals are quoted as δ values in ppm using residual protonated solvent signals as internal standard ($CDCl_3$: δ 7.26 ppm). Data is reported as follows: chemical shift, integration, multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet, br = broad) and coupling constants (Hz). ^{13}C NMR spectra were recorded on either a 100 MHz or a 125 MHz with complete proton decoupling. Chemical shifts (δ) are reported in ppm downfield from tetramethylsilane with the solvent as the internal reference ($CDCl_3$: δ 77.26 ppm). HRMS analyses were performed with Q-TOF YA263 high resolution (Water Corporation) instruments by +ve mode electrospray ionization. Elemental analysis (CHN) is performed with CHNS/O Analyzer.

General procedure for the synthesis of symmetrical benzil derivatives (GP-1): Aromatic aldehyde (1.0 equiv.) was added to Amberlyst 15 (2.0 equiv.) and zinc dust (3.0 equiv.) in water and the mixture was heated with constant stirring at 70 °C for 6h. Then, the reaction mixture was filtered, extracted with EtOAc and the pinacol product was purified by column chromatography with EtOAC-hexane (20/80). In the next step, the pinacol product in dry DCM was added to the solution Dess-Martin Periodinane (DMP) (1.2 equiv.) in dry DCM and the reaction was allowed to stir for 30 min. Finally, DMP was filtered out and purification of the crude product by column chromatography on silica gel with EtOAc–hexane (2/98 to 5/95) yielded the symmetrical benzil derivative **5b-g**. Compounds **5b-f** were previously reported.[11]

General procedure for the synthesis of unsymmetrical benzil derivatives (GP-2): In a round- bottomed flask, CuI (0.02 equiv.), TMEDA (0.05 equiv.) and acid chloride (1.2 equiv.) were added sequentially to phenylacetylene (1.0 equiv.) in Et_3N. The reaction mixture was stirred for 1 h under N_2 atmosphere. After completion, the reaction was quenched with $NaHCO_3$. The organic layer was extracted with EtOAc and purified by column chromatography on silica gel with EtOAc–hexane (5/95 to 10/90) to give corresponding alkynone intermediates. In the next step, K_2CO_3 (1.0 equiv.) was added to alkynones in DMSO-water (50:1) and heated at 70 °C for 8 h under oxygen atmosphere. After reaction was completed, water was added and extracted with EtOAc. The organic phase was dried over Na_2SO_4, concentrated and purified by column chromatography on silica gel with EtOAc–hexane (2/98 to 5/95) to provide the unsymmetrical benzil derivatives **5h** and **5i**. **5i** is previously reported.[11,15]

1,2-Bis(4-fluorophenyl)ethane-1,2-dione (5b): Using the general procedure **GP-1**, 4-fluorobenzaldehyde **7a** (3.0 g, 24.1 mmol), Amberlyst 15 (10.17 g, 48.2 mmol) and zinc (4.74 g, 72.3 mmol) followed by oxidation with DMP (3.5 g, 10.9 mmol) yielded compound **5b** (1.69 g, 58%) as a yellow solid. Benzil **5b** is previously reported.[11c,11g-i]

1,2-Bis(2-fluorophenyl)ethane-1,2-dione (5c): Using the general procedure **GP-1**, 2-fluorobenzaldehyde **7b** (3.0 g, 24.1 mmol), Amberlyst 15 (10.17 g, 48.2 mmol) and zinc (4.74 g, 72.3 mmol) followed by oxidation with DMP (3.7 g, 11.5 mmol) yielded compound **5c** (1.75 g, 60%) as a light yellow solid. Benzil **5c** is previously reported.[11c,11g]

1,2-Bis(4-chlorophenyl)ethane-1,2-dione (5d): Using the general procedure **GP-1**, 4-chlorobenzaldehyde **7c** (3.0 g, 21.3 mmol), Amberlyst 15 (9.0 g, 42.7 mmol) and zinc (4.18 g, 63.9 mmol) followed by oxidation with DMP (3.34 g, 10.3 mmol) yielded compound **5d** (1.9 g, 65%) as a yellow solid. Benzil **5d** is previously reported.[11i]

1,2-Bis(4-methylphenyl)ethane-1,2-dione (5e): Using the general procedure **GP-1**, 4-methylbenzaldehyde **7d** (3.0 g, 25.0 mmol), Amberlyst 15 (10.5 g, 50.0 mmol) and zinc (4.9 g, 75.0 mmol) followed by oxidation with DMP (3.4 g, 10.3 mmol) yielded compound **5e** (1.84 g, 62%) as a white solid. Benzil **5e** is previously reported.[11g-i]

1,2-Bis(3-methoxyphenyl)ethane-1,2-dione (5f): Using the general procedure **GP-1**, 3-methoxybenzaldehyde **7e** (3.0 g, 22.0 mmol), Amberlyst 15 (9.28 g, 44.0 mmol) and zinc (4.32 g, 66.0 mmol) followed by oxidation with DMP (4.27 g, 11.2 mmol) yielded compound **5f** (1.87 g, 63%) as a yellow solid. Benzil **5f** is previously reported.[11h]

1,2-Bis(2,5-dimethoxyphenyl)ethane-1,2-dione (5g): ^{1}H NMR (400 MHz, $CDCl_3$): Using the general procedure **GP-1**, 2,5-dimethoxybenzaldehyde **7f** (3.0 g, 18.0 mmol), Amberlyst 15 (7.6 g, 36.0 mmol) and zinc (3.55 g, 54.2 mmol) followed by oxidation with DMP (3.5 g, 10.8 mmol) yielded compound **5g** (1.9 g, 64%) as a yellowish white solid; mp 150-153 °C; IR (ATR) ν. 3006, 2943, 2838, 1658, 1609, 1495, 1417, 1274, 1221, 1158, 1041, 1016, 950, 817, 715 cm^{-1}; ^{1}H NMR (500 MHz, $CDCl_3$): 7.57 (2H, d, J = 4.0 Hz), 7.15 (2H, dd, J = 11.2, 4.0 Hz), 6.92 (2H, d, J = 11.2 Hz), 3.85 (6H, s), 3.55 (6H, s); ^{13}C (100 MHz, $CDCl_3$): 192.2, 155.2, 155.4, 124.1, 123.3, 114.9, 112.4, 56.9, 56.0; HRMS (ESI) calcd for $C_{18}H_{18}NaO_6$ [M+Na]$^+$: 353.1001; Found: 353.1000. Anal calcd for $C_{18}H_{18}O_6$ (330.33): calcd. C 65.45, H 5.49; found C 65.49, H 5.44.

1-(2-Iodophenyl)-2-phenylethane-1,2-dione (5h): Using the general procedure **GP-2**, 2-iodobenzoyl chloride **8a** (3.0 g, 11.3 mmol), CuI (35.8 mg, 0.19 mmol) and TMEDA (70 µL, 0.47 mmol) followed by addition of phenylacetylene (958 mg, 9.4 mmol) yielded the intermediate alkynone (2.75 g, 88%) as a white solid. In the next step, the alkynone (2.0 g, 6.0 mmol) and K_2CO_3 (830 mg, 6.0 mmol) in O_2 air gave corresponding benzil **5h** (1.2 g, 60%) as a light yellow solid; mp 180-182 °C; IR (ATR) ν. 2923, 2851, 1709, 1675, 1580, 150, 1291, 1249, 1022, 758, 701 cm^{-1}; ^{1}H NMR (500 MHz, $CDCl_3$): 8.07 (2H, d, J = 8.4 Hz), 8.00 (1H, d, J = 7.0 Hz), 7.67 (2H, t, J = 5.0 Hz), 7.54 (2H, t, J = 8.0 Hz), 7.48 (1H, t, J = 7.5 Hz), 7.26 (1H, d, J = 7.5); ^{13}C (125 MHz, $CDCl_3$): 194.8, 191.4, 141.3, 138.5, 134.7, 134.1, 133.4, 133.0, 130.5, 130.0, 129.1, 129.0, 128.4, 93.3; HRMS (ESI) calcd for $C_{14}H_9INaO_2$ [M+Na]$^+$: 358.9545; Found: 358.9547. Anal calcd for $C_{14}H_9IO_2$ (336.12): calcd. C 50.03, H 2.70; found C 50.00, H 2.78.

1-(2-Methoxyphenyl)-2-phenylethane-1,2-dione (5i): Using the general procedure **GP-2**, 2-iodobenzoyl chloride **8b** (3.0 g, 17.6 mmol), CuI (55.6 mg, 0.29 mmol) and TMEDA (108 µL g, 0.73 mmol) followed by addition of phenylacetylene (1.79 g, 14.6 mmol) yielded the intermediate alkynone (3.03 g, 88%) as a white solid. In the next step, the alkynone (3.0 g, 12.7 mmol) and K_2CO_3 (1.75 g, 12.7 mmol) in O_2 air gave corresponding benzil **5i** (1.58 g, 52%) as yellow solid. Benzil **5i** is previously reported.[11e,11g]

General procedure for the synthesis of diallylated products from benzils (GP-3): To a solution of benzil **5** (1.0 equiv.) in dry THF, allyl magnesium halide solution (3.0 equiv.) was added dropwise at 0 °C and stirred at room temperature for 6 h. The reaction mixture was quenched with saturated aqueous NH_4Cl solution and extracted with ethyl acetate (3 x 20 mL). The combined organic phase was washed with brine, dried over anhydrous Na_2SO_4, filtered and concentrated in vacuum. The crude residue was then purified by column chromatography on silica gel with EtOAc–hexane (10/90 to 15/85) to give compound **4**.

4,5-Diphenylocta-1,7-diene-4,5 diol (4a): Using the general procedure **GP-3**, benzil **5a** (1.0 g, 4.75 mmol) and allyl magnesium bromide (14.25 mL, 14.25 mmol) yielded compound **4a** (845 mg, 60%) as a colorless oil; ^{1}H NMR (500 MHz, $CDCl_3$): 7.39 (4H, d, J = 7.1 Hz), 7.30-7.23 (6H, m), 5.35-5.27 (2H, m), 4.97-4.93 (4H, m), 3.18 (2H, dd, J = 14.2, 5.4 Hz), 2.31 (2H, s), 2.22 (2H, dd, J = 14.2, 8.9 Hz); ^{13}C (125 MHz, $CDCl_3$): 142.1,

134.0, 128.0, 127.5, 127.0, 119.6, 79.7, 41.0; HRMS (ESI) calcd for $C_{20}H_{23}O_2$ $[M+H]^+$: 295.1698; Found: 295.1694. Compound **4a** is previously reported.[18]

4,5-Bis(4-fluorophenyl)octa-1,7-diene-4,5-diol (4b): Using the general procedure **GP-3**, benzil **5b** (1.0 g, 4.06 mmol) and allyl magnesium bromide (12.18 mL, 12.18 mmol) yielded compound **4b** (910 mg, 68%) as a colorless oil; ^{1}H NMR (400 MHz, $CDCl_3$): 7.09 (4H, s $_{br}$), 6.93 (4H, t, *J* = 8.5 Hz), 5.42-5.32 (2H, m), 5.11 (2H, d, *J* = 17.0 Hz), 5.04 (2H, d, *J* = 10.0 Hz), 2.84 (2H, s), 2.80 (2H, dd, *J* = 14.3, 8.8 Hz), 2.52 (2H, dd, *J* = 14.2, 5.1 Hz), ^{13}C (100 MHz, $CDCl_3$): 162.1 (d, J_{C-F} = 150.0 Hz), 136.2 (d, J_{C-F} = 3.2 Hz), 133.8, 130.0 (d, J_{C-F} = 7.9 Hz), 120.1, 114.1 (d, J_{C-F} = 20.9 Hz), 79.6, 40.2; HRMS (ESI) calcd for $C_{20}H_{20}F_2NaO_2$ $[M+Na]^+$: 353.1329; Found: 353.1327. Compound **4b** is previously reported.[18]

4,5-Bis(2-fluorophenyl)octa-1,7-diene-4,5-diol (4c): Using the general procedure **GP-3**, benzil **5c** (1.0 g, 4.06 mmol) and allyl magnesium bromide (12.18 mL, 12.18 mmol) yielded compound **4c** (865 mg, 65%) as a colorless liquid; IR (ATR) *ν*. 3539, 3077, 1639, 1613, 1578, 1448, 1442, 1269, 1211, 994, 917, 820, 753 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$): 7.54 (1H, s $_{br}$), 7.28-7.17 (3H, m), 7.10 (1H, t, *J* = 7.5 Hz), 6.97-6.82 (3H, m), 5.60-5.47 (2H, m), 5.12 (2H, t, *J* = 16.5 Hz), 5.00 (2H, t, *J* = 10.0 Hz), 3.47 (1H, s $_{br}$), 3.18 (1H, d, *J* = 9.4 Hz), 2.97 (2H, s $_{br}$), 2.62-2.55 (2H, m); ^{13}C (100 MHz, $CDCl_3$): 134.2 (d, J_{C-F} = 10.0 Hz), 131.3 (dd, J_{C-F} = 14.5, 4.0 Hz), 129.6 (dd, J_{C-F} = 21.3, 9.0 Hz), 127.8 (d, J_{C-F} = 12.2 Hz), 123.7 (d, J_{C-F} = 3.0 Hz), 123.3 (d, J_{C-F} = 3.0 Hz), 119.3 (d, J_{C-F} = 10.1 Hz), 115.9 (d, J_{C-F} = 25.6 Hz), 80.6, 39.8, 39.7; HRMS (ESI) calcd for $C_{20}H_{20}F_2NaO_2$ $[M+Na]^+$: 353.1329; Found: 353.1328.

4,5-Bis(4-chlorophenyl)octa-1,7-diene-4,5-diol (4d): Using the general procedure **GP-3**, benzil **5d** (1.0 g, 3.58 mmol) and allyl magnesium bromide (10.75 mL, 10.75 mmol) yielded compound **4d** (820 mg, 63%) as a colorless liquid; IR (ATR) *ν*. 3501, 3022, 1638, 1601, 1583, 1443, 1438, 1257, 1188, 977, 907, 822, 761 cm^{-1}; ^{1}H NMR (500 MHz, $CDCl_3$): 7.21 (4H, d, *J* = 8.9 Hz), 7.07 (4H, s $_{br}$), 5.40-5.31 (2H, m), 5.10 (2H, d, *J* = 16.7 Hz), 5.04 (2H, d, *J* = 10.1 Hz), 2.85 (2H, s $_{br}$), 2.78 (2H, dd, *J* = 8.8, 5.6 Hz), 2.52 (2H, dd, *J* = 8.8, 5.6 Hz); ^{13}C (125 MHz, $CDCl_3$): 139.1, 133.6, 133.5, 129.8, 127.5, 120.2, 79.7, 40.2; HRMS (ESI) calcd for $C_{20}H_{21}Cl_2O_2$ $[M+H]^+$: 363.0919; Found: 363.0914.

4,5-Di-*p*-tolylocta-1,7-diene-4,5-diol (4e): Using the general procedure **GP-3**, benzil **5e** (1.0 g, 4.2 mmol) and allyl magnesium bromide (12.6 mL, 12.6 mmol) yielded compound **4e** (745 mg, 55%) as a white solid; ^{1}H NMR (400 MHz, $CDCl_3$): 7.27 (4H, d, *J* = 7.8 Hz), 7.10 (4H, d, *J* = 7.8 Hz), 5.37-5.26 (2H, m), 4.97-4.91 (4H, m), 3.14 (2H, dd, *J* = 14.1, 5.2 Hz), 2.34 (6H, s), 2.22 (2H, s), 2.19-2.16 (2H, m); ^{13}C (100 MHz, $CDCl_3$): 139.1, 136.4, 134.2, 128.3, 127.9, 119.3, 79.7, 41.0, 21.1; HRMS (ESI) calcd for $C_{22}H_{26}NaO_2$ $[M+Na]^+$: 345.1830; Found: 345.1830. Compound **4e** is previously reported.[18]

4,5-Bis(3-methoxyphenyl)octa-1,7-diene-4,5-diol (4f): Using the general procedure **GP-3**, benzil **5f** (1.0 g, 3.7 mmol) and allyl magnesium bromide (11.1 mL, 11.1 mmol) yielded compound **4f** (855 mg, 65%) as a colorless liquid; IR (ATR) *ν*. 3533, 2938, 1677, 1637, 1599, 1581, 1486, 1430, 1244, 1041, 995, 835, 758 cm^{-1}; ^{1}H NMR (500 MHz, $CDCl_3$): 7.16 (2H, t, *J* = 8.0 Hz), 6.80-6.75 (6H, m), 5.45-5.37 (2H, m), 5.10 (2H, d, *J* = 16.8 Hz), 5.02 (2H, d, *J* = 10.1 Hz), 3.73 (6H, s), 2.90 (2H, s), 2.79 (2H, dd, *J* = 14.4, 9.0 Hz), 2.54 (2H, dd, *J* = 14.4, 5.4 Hz); ^{13}C (100 MHz, $CDCl_3$): 158.9, 142.3, 134.3, 128.1, 121.1, 119.7, 114.3, 112.9, 80.0, 55.2, 40.5; HRMS (ESI) calcd for $C_{22}H_{26}NaO_4$ $[M+Na]^+$: 377.1729; Found: 377.1729.

4,5-Bis(2,5-dimethoxyphenyl)octa-1,7-diene-4,5-diol (4g): Using the general procedure **GP-3,** benzil **5g** (1.0 g, 3.02 mmol) and allyl magnesium bromide (9.0 mL, 9.0 mmol) yielded the compound **4g** (880 mg, 70%) as colourless liquid; IR (ATR) *ν*. 3372, 2957, 2926, 2857, 1742, 1666, 1618, 1495, 1466, 1184, 1081, 967, 837, 772 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$): 6.71 (4H, m), 6.33 (1H, s), 5.93 (1H, s), 5.70-5.45 (2H, m), 5.04 (2H, d, *J* = 16.9 Hz), 4.94 (2H, d, *J* = 9.6 Hz), 3.77 (2H, s $_{br}$), 3.60 (6H, s), 3.42 (6H, s), 2.86-2.81 (4H,m); ^{13}C (100 MHz, $CDCl_3$): 153.8, 152.8, 135.5, 129.6, 117.2, 117.0, 113.0, 112.9, 85.5, 56.6, 55.7, 39.6; HRMS (ESI) calcd for $C_{24}H_{30}NaO_6$ $[M+Na]^+$: 437.1940; Found: 437.1942.

4-(2-Iodophenyl)-5-phenylocta-1,7-diene-4,5-diol (4h): Using the general procedure **GP-3**, benzil **5h** (1.0 g, 2.97 mmol) and allyl magnesium bromide (8.92 mL, 8.92 mmol) yielded the compound **4h** (750 mg, 60%) as a colourless liquid; IR (ATR) *ν*. 3493, 3010, 1635, 1610, 1572, 1450, 1433, 1262, 1201, 962, 820, 783 cm^{-1}; ^{1}H NMR (500 MHz, $CDCl_3$): 7.39 (3H, d, *J* = 7.1 Hz), 7.30-7.27 (4H, m), 7.25-7.23 (2H, m), 5.35-5.27 (2H, m), 4.97-4.93 (4H, m), 3.18 (2H, dd, *J* = 8.5, 5.5 Hz), 2.31 (2H, s), 2.23 (2H, dd, *J* = 8.5, 5.5 Hz); ^{13}C (125 MHz, $CDCl_3$): 142.3, 134.0, 128.5, 128.1, 127.5, 127.2, 127.0, 119.5, 79.8, 41.1; HRMS (ESI) calcd for $C_{20}H_{21}INaO_2$ $[M+Na]^+$: 443.0484; Found: 443.0482.

4-(2-Methoxyphenyl)-5-phenylocta-1,7-diene-4,5-diol (4i): Using the general procedure **GP-3**, benzil **5i** (1.0 g, 4.16 mmol) and allyl magnesium bromide (12.5 mL, 12.5 mmol) yielded compound **4i** (810 mg, 60%) as a colorless liquid; IR (ATR) *ν*. 3465, 3072, 3009, 2976, 1638, 1581, 1489, 1435, 1232, 1180, 1050, 1022, 910, 752, 702 cm^{-1}; ^{1}H NMR (500 MHz, $CDCl_3$): 7.69 (1H, dd, *J* = 7.7, 1.6 Hz), 7.49-7.47 (2H, m), 7.31-7.30 (3H, m), 7.00-6.97 (3H, m), 6.00-5.91 (1H, m), 5.47-5.38 (1H, m), 5.13 (2H, d, *J* = 13.6 Hz), 4.90 (2H, s $_{br}$), 3.95 (3H, s), 3.37 (2H, s $_{br}$), 3.04-2.95 (4H, m); ^{13}C (125 MHz, $CDCl_3$): 156.8, 142.3, 133.3, 131.9, 131.0, 129.2, 128.9, 128.3, 128.0, 127.1, 126.7, 121.1, 118.5, 111.9, 86.1, 81.3, 55.6, 47.2, 40.0; HRMS (ESI) calcd for $C_{21}H_{24}NaO_3$ $[M+Na]^+$: 347.1623; Found: 347.1622.

Zinc mediated allylation of benzil 5a: To a solution of benzil **5a** (2.0 g, 9.5 mmol) in THF-H_2O (4 mL-1mL) was added zinc (2.0 g, 30.4 mmol), allyl bromide (3.3 mL, 38.0 mmol) and NaI (3.4 g, 22.8 mmol). The reaction was stirred vigorously at room temperature for 6 h. After completion of the reaction, the aqueous phase was extracted with CH_2Cl_2, concentrated and the product was purified by column chromatography on silica gel with EtOAc–hexane (5/95) to give exclusively the monoallyl compound **6a** (2.11 g, 88%) as a white solid; ^{1}H NMR (500 MHz, $CDCl_3$): 7.72 (2H, dd, *J* = 8.3, 1.0 Hz), 7.51-7.49 (2H, m), 7.43 (1H, t, *J* = 9.8 Hz), 7.38 (2H, t, *J* = 7.6 Hz), 7.32-7.27 (3H, m), 5.77-5.69 (1H, m), 5.12 (1H, d, *J* = 10.9 Hz), 5.02 (1H, dd, *J* = 17.2, 1.5 Hz), 3.13 (1H, dd, *J* = 13.7, 7.5 Hz), 2.97 (1H, dd, *J* = 13.7, 6.9 Hz); ^{13}C (100 MHz, $CDCl_3$): 200.9, 141.9, 134.8, 132.8, 132.4, 130.2, 129.0, 128.2, 128.1, 125.8, 120.4, 81.6, 44.1; HRMS (ESI) calcd for $C_{17}H_{17}O_2$ $[M+H]^+$: 253.1229; Found: 253.1225.

Synthesis of compound 4a from 6a: To a solution of monoallyl compound **6a** (1.0 g, 3.96 mmol) in dry THF, allyl

magnesium halide solution (5.94 mL, 5.94 mmol) was added dropwise at 0 °C and stirred at room temperature for 6 h. The reaction mixture was quenched with saturated aqueous NH_4Cl solution and extracted with ethyl acetate (3 x 20 mL). The combined organic phase was washed with brine, dried over anhydrous Na_2SO_4, filtered and concentrated in vacuum. The crude residue was then purified by column chromatography on silica gel with EtOAc–hexane (5/95) to give compound **4a** (793 mg, 68%) as a colorless oil.

Indium promoted allylation of benzil 5a: To a solution of benzil **5a** (2.0 g, 9.51 mmol) in THF-H_2O (4 mL-1mL) was added indium ingots (1.53 g, 13.3 mmol), 2-methylallyl bromide (1.9 mL, 19.0 mmol) and NaI (1.7 g, 11.4 mmol). The reaction was stirred vigorously at room temperature for 3 h. After completion of the reaction, the aqueous phase was extracted with CH_2Cl_2, concentrated and the product was purified by column chromatography on silica gel with EtOAc–hexane (5/95) to give benzoin derivative **6b** (2.53 g, 90%) as a white solid. ^{1}H NMR (500 MHz, $CDCl_3$): 7.80 (2H, dd, *J* = 8.5, 1.3 Hz), 7.55-7.53 (2H, m), 7.44-7.41 (1H, m), 7.38-7.35 (2H, m), 7.31-7.28 (3H, m), 4.89 (1H, s), 4.63 (1H, s), 3.24 (1H, d, *J* = 14.3 Hz), 2.94 (1H, d, *J* = 13.6 Hz), 1.54 (3H, s); ^{13}C (125 MHz, $CDCl_3$): 201.1, 142.5, 141.6, 132.7, 130.4, 128.9, 128.1, 127.9, 127.8, 127.5, 127.3, 125.6, 116.7, 81.4, 47.6, 24.3; HRMS (ESI) calcd for $C_{18}H_{19}O_2$ $[M+H]^+$: 267.1385; Found: 267.1387.

Synthesis of compound 4j from 6b: To a solution of benzoin derivative **6b** (1.0 g, 3.76 mmol) in dry THF, allyl magnesium bromide solution (5.6 mL, 5.6 mmol) was added dropwise at 0 °C and stirred at room temperature for 6 h. The reaction mixture was quenched with saturated aqueous NH_4Cl solution and extracted with ethyl acetate (3 x 20 mL). The combined organic phase was washed with brine, dried over anhydrous Na_2SO_4, filtered and concentrated in vacuum. The crude residue was then purified by column chromatography on silica gel with EtOAc–hexane (5/95) to give compound **4j** (753 mg, 65%) as a colorless liquid; IR (ATR) *ν*. 3522, 3071, 3026, 2975, 1637, 1492, 1445, 1374, 1327, 1222, 1181, 1059, 995, 907, 763, 717, 701 cm^{-1}; ^{1}H NMR (500 MHz, $CDCl_3$): 7.22 (10H, s $_{br}$), 5.48-5.40 (1H, m), 5.04 (1H, d, *J* = 17.1 Hz), 4.97 (1H, d. *J* = 10.2 Hz), 4.83 (1H, s), 4.71 (1H, s), 2.99 (1H, d, *J* = 14.0 Hz), 2.78 (1H, dd, *J* = 14.6, 8.3 Hz), 2.46 (1H, dd, *J* = 14.6, 5.8 Hz), 2.38 (1H, d, *J* = 14.0 Hz), 1.18 (3H, s); ^{13}C (125 MHz, $CDCl_3$): 143.0, 141.3, 140.3, 134.5, 128.8, 128.4, 127.2, 127.1, 127.0, 118.9, 116.4, 80.1, 79.1, 43.4, 40.3, 24.4; HRMS (ESI) calcd for $C_{21}H_{24}NaO_2$ $[M+Na]^+$: 331.1674; Found: 331.1672.

General procedure for ring closing metathesis (GP-4): To a stirred solution of **4** (1.0 equiv.) in dry dichloromethane, Grubbs' second generation catalyst (**G-II**) (5 mol %) was added and stirred at room temperature for 15 h under an argon atmosphere. The reaction mixture was then concentrated and purified by column chromatography using silica gel with EtOAc–hexane (15/85 to 20/80) to yield the tricyclic compound **3**.

1,2-Diphenylcyclohex-4-ene-1,2-diol (3a): Using general procedure **GP-4**, compound **4a** (800 mg, 2.7 mmol) and Grubbs' second generation catalyst (**G-II**) (11.2 mg, 0.13 mmol) provided compound **3a** (560 mg, 78%) as a colorless liquid; IR (ATR) *ν*. 3560, 3026, 2924, 2850, 1493, 1446, 1068, 1029, 886, 725, 698 cm^{-1}; ^{1}H NMR (500 MHz, $CDCl_3$): 7.33-7.30 (4H, m), 7.12-7.10 (6H, m), 5.95 (2H, m), 3.34 (2H, d, *J* = 20.9 Hz), 2.27 (2H, dd, *J* = 18.3, 3.3 Hz), 2.17 (2H, s); ^{13}C (100 MHz, $CDCl_3$): 143.0, 127.3, 126.8, 126.5, 125.3, 76.6, 38.7; HRMS (ESI) calcd for $C_{18}H_{18}NaO_2$ $[M+Na]^+$: 289.1204; Found: 289.1206.

1,2-Bis(4-fluorophenyl)cyclohex-4-ene-1,2-diol (3b): Using general procedure **GP-4**, compound **4b** (800 mg, 2.4 mmol) and Grubbs' second generation catalyst (**G-II**) (98.7 mg, 0.12 mmol) provided compound **3b** (617 mg, 85%) as a colorless liquid; IR (ATR) ν. 3460, 3081, 2932, 2864, 1771, 1684, 1600, 1508, 1450, 1411, 1230, 1160, 1093, 1032, 1019, 889, 834, 814, 580 cm^{-1}; ^{1}H NMR (500 MHz, $CDCl_3$): 7.29-7.26 (4H, m), 6.79 (4H, t, *J* = 8.7 Hz), 5.93 (2H, m), 3.29 (2H, d, *J* = 17.0 Hz), 2.24 (2H, d, *J* = 17.0 Hz), 2.15 (2H, s); ^{13}C (125 MHz, $CDCl_3$): 162.0 (d, $J_{C\text{-}F}$ = 243.9 Hz), 138.8 (d, $J_{C\text{-}F}$ = 3.3 Hz), 128.3 (d, $J_{C\text{-}F}$ = 7.9 Hz), 125.3, 114.1 (d, $J_{C\text{-}F}$ = 20.8 Hz), 76.3, 39.0; HRMS (ESI) calcd for $C_{18}H_{16}F_2NaO_2$ $[M+Na]^+$: 325.1016; Found: 325.1014.

1,2-Bis(2-fluorophenyl)cyclohex-4-ene-1,2-diol (3c): Using general procedure **GP-4**, compound **4c** (800 mg, 2.4 mmol) and Grubbs' second generation catalyst (**G-II**) (98.7 mg, 0.12 mmol) provided compound **3c** (602 mg, 83%) as a colorless liquid; IR (ATR) *ν*. 3593, 3033, 2915, 1613, 1577, 1497, 1447, 1331, 1273, 1210, 1034, 883, 748, 654 cm^{-1}; ^{1}H NMR (500 MHz, $CDCl_3$): 7.36 (2H, t, *J* = 10.0 Hz), 7.24-7.21 (2H, m), 7.05 (2H, t, *J* = 9.5 Hz), 6.81 (2H, dd, *J* = 17.0, 10.2 Hz), 5.93 (2H, s), 2.92 (2H, d, *J* = 21.3 Hz), 2.64 (2H, d, *J* = 22.5 Hz); ^{13}C (100 MHz, $CDCl_3$): 160.2 (d, $J_{C\text{-}F}$ = 242.8 Hz), 129.8 (d, $J_{C\text{-}F}$ = 9.5 Hz), 129.4 (d, $J_{C\text{-}F}$ = 3.7 Hz), 129.2 (d, $J_{C\text{-}F}$ = 6.9 Hz), 125.4, 124.0 (d, $J_{C\text{-}F}$ = 3.1 Hz), 116.4 (d, $J_{C\text{-}F}$ = 26.2 Hz), 78.5 (d, $J_{C\text{-}F}$ = 3.6 Hz), 37.4; HRMS (ESI) calcd for $C_{18}H_{16}F_2NaO_2$ $[M+Na]^+$: 325.1016; Found: 325.1014.

1,2-Bis(4-chlorophenyl)cyclohex-4-ene-1,2-diol (3d): Using general procedure **GP-4**, compound **4d** (800 mg, 2.2 mmol) and Grubbs' second generation catalyst (**G-II**) (90.5 mg, 0.11 mmol) provided compound **3d** (575 mg, 78%) as a colorless liquid; IR (ATR) *ν*. 3574, 3038, 2925, 1596, 1490, 1400, 1304, 1174, 1093, 1031, 988, 890, 826, 757, 731, 670, 569, 480 cm^{-1}; ^{1}H NMR (500 MHz, $CDCl_3$): 7.27 (4H, d, *J* = 8.5 Hz), 7.06 (4H, d, *J* = 8.5 Hz), 5.92 (2H, s), 3.25 (2H, d, *J* = 16.9 Hz), 2.23 (2H, dd, *J* .= 15.0, 2.5 Hz); ^{13}C (100 MHz, $CDCl_3$): 141.5, 132.9, 128.0, 127.4, 125.2, 76.3, 39.0; HRMS (ESI) calcd for $C_{18}H_{16}Cl_2NaO_2$ $[M+Na]^+$: 357.0425; Found: 357.0426.

1,2-Di-*p*-tolylcyclohex-4-ene-1,2-diol (3e): Using general procedure **GP-4**, compound **4e** (700 mg, 2.2 mmol) and Grubbs' second generation catalyst (**G-II**) (90.5 mg, 0.11 mmol) provided compound **3e** (453 mg, 70%) as a colorless liquid; IR (ATR) *ν*. 3584, 3026, 2921, 2857, 1709, 1657, 1511, 1444, 1416, 1363, 1183, 1080, 1031, 984, 888, 816, 729, 698, 661, 582, 465 cm^{-1}; ^{1}H NMR (500 MHz, $CDCl_3$): 7.38 (4H, d, *J* = 8.2 Hz), 7.16 (4H, d, *J* = 7.9 Hz), 5.81 (2H, s), 2.93 (2H, d, *J* = 16.5 Hz), 2.73 (2H, d, *J* = 16.5 Hz), 2.34 (6H, s); ^{13}C (125 MHz, $CDCl_3$): 140.3, 136.3, 128.1, 126.5, 125.4, 76.5, 39.0, 21.0; HRMS (ESI) calcd for $C_{20}H_{23}O_2$ $[M+H]^+$: 295.1698; Found: 295.1693.

1,2-Bis(3-methoxyphenyl)cyclohex-4-ene-1,2-diol (3f): Using general procedure **GP-4**, compound **4f** (800 mg, 2.2) and Grubbs' second generation catalyst (**G-II**) (90.5 mg, 0.11 mmol) provided compound **3f** (560 mg, 78%) as a colorless liquid; IR (ATR) *ν*. 3475, 3035, 2936, 2834, 1687, 1600, 1582, 1464, 1429, 1289, 1252, 1158, 1032, 972, 894, 870, 782, 697, 662, 608 cm^{-1}; ^{1}H NMR (500 MHz, $CDCl_3$): 7.07 (2H, t, *J* = 7.9 Hz), 6.97 (2H, d, *J* = 7.8 Hz), 6.88 (2H, s), 6.67 (2H, d, *J* = 8.0 Hz), 5.93 (2H, s), 3.63 (6H, s), 3.30 (2H, d, *J* = 16.8 Hz), 2.26 (2H, d, *J* = 17.1 Hz),

2.19 (2H, s); ^{13}C (125 MHz, $CDCl_3$): 159.0, 144.8, 128.2, 125.3, 118.9, 112.7, 112.5, 76.0, 55.3, 38.7; HRMS (ESI) calcd for $C_{20}H_{23}O_4$ $[M+H]^+$: 327.1596; Found: 327.1591.

1,2-Bis(2,5-dimethoxyphenyl)cyclohex-4-ene-1,2-diol (3g): Using general procedure **GP-4**, compound **4g** (800 mg, 1.9 mmol) and Grubbs' second generation catalyst (**G-II**) (80.0 mg, 0.1 mmol) provided compound **3g** (605 mg, 82%) as a colorless liquid; IR (ATR) *ν*. 3460, 3078, 2941, 2838, 1593, 1496, 1463, 1408, 1281, 1220, 1178, 1045, 905, 814, 730 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$): 7.21 (2H, s), 6.67 (4H, m), 6.01 (2H, s), 5.87 (2H, s), 3.67 (12H, s), 3.13 (2H, d, *J* = 16.8 Hz), 2.28 (2H, d, *J* = 17.1 Hz); ^{13}C (100 MHz, $CDCl_3$): 153.8, 151.3, 133.6, 125.3, 115.7, 112.6, 112.5, 79.8, 56.4, 55.8, 37.9; HRMS (ESI) calcd for $C_{22}H_{26}NaO_6$ $[M+Na]^+$: 409.1627; Found: 409.1625.

1-(2-Iodophenyl)-2-phenylcyclohex-4-ene-1,2-diol (3h): Using general procedure **GP-4**, compound **4h** (700 mg, 1.6 mmol) and Grubbs' second generation catalyst (**G-II**) (68.5 mg, 0.08 mmol) provided compound **3h** (490 mg, 75%) as a colorless liquid; IR (ATR) *ν*. 3534, 3061, 3027, 2923, 2847, 1601, 1495, 1446, 1362, 1177, 1053, 986, 920, 876, 740, 700, 662 cm^{-1}; ^{1}H NMR (500 MHz, $CDCl_3$): 7.20 (2H, t, *J* = 7.2 Hz), 7.16 (4H, t, *J* = 7.5 Hz), 7.01 (3H, d, *J* = 7.8 Hz), 6.00 (2H, m), 2.89 (2H, d, *J* = 17.1 Hz), 2.82 (2H, s), 2.58 (2H, d, *J* = 18.2 Hz); ^{13}C (100 MHz, $CDCl_3$): 142.3, 127.3, 127.1, 126.9, 126.4, 77.08, 39.4; HRMS (ESI) calcd for $C_{18}H_{18}IO_2$ $[M+H]^+$: 393.0351; Found: 393.0344.

1-(2-Methoxyphenyl)-2-phenylcyclohex-4-ene-1,2-diol (3i): Using general procedure **GP-4**, compound **4i** (700 mg, 2.2 mmol) and Grubbs' second generation catalyst (**G-II**) (90.5 mg, 0.11 mmol) provided compound **3i** (490 mg 75%) as a colorless liquid; IR (ATR) *ν*. 3488, 3058, 2932, 2844, 1598, 1477, 1443, 1400, 1278, 1223, 1166, 1035, 911, 828, 703 cm^{-1}; ^{1}H NMR (500 MHz, $CDCl_3$): 7.54 (1H, dd, *J* = 7.8, 1.6 Hz), 7.22 (1H, dt, *J* = 8.1, 1.6 Hz), 7.16-7.15 (3H, m), 6.98-6.93 (3H, m), 6.57 (1H, d, *J* = 8.9 Hz), 6.05-6.02 (1H, m), 5.85-5.82 (1H, m), 3.03 (3H, s), 2.76 (2H, m), 2.70 (1H, d, *J* = 18.7 Hz), 2.41 (1H, dd, *J* = 17.8, 4.5 Hz); ^{13}C (100 MHz, $CDCl_3$): 157.2, 143.7, 130.6, 129.1, 127.9, 127.1, 127.0, 126.9, 126.8, 125.0, 121.3, 110.9, 79.7, 77.2, 55.0, 40.5, 35.9; HRMS (ESI) calcd for $C_{21}H_{24}NaO_3$ $[M+Na]^+$: 347.1623; Found: 347.1622.

4-Methyl-1,2-diphenylcyclohex-4-ene-1,2-diol (3j): Using general procedure **GP-4**, compound **4j** (700 mg, 2.3 mmol) and Grubbs' second generation catalyst (**G-II**) (90.5 mg, 0.11 mmol) provided compound **3j** (477 mg, 74%) as a colorless liquid; ^{1}H NMR (400 MHz, $CDCl_3$): 7.31 (4H, s_{br}), 7.10 (6H, s_{br}), 5.63 (1H, s), 3.28 (2H, t, *J* = 14.6 Hz), 2.18 (2H, d, *J* = 7.6 Hz), 1.86 (3H, s); ^{13}C (100 MHz, $CDCl_3$): 143.0, 132.8, 127.3, 126.8, 126.6, 126.5, 118.9, 76.3, 43.5, 38.7, 23.6; HRMS (ESI) calcd for $C_{19}H_{20}KO_2$ $[M+K]^+$: 319.1100; Found: 319.1101. Diol **3j** is previously reported.[8d]

General procedure for dehydrative aromatization (GP-5): PTSA (30 mol %) was added to a solution of compound **3** in benzene and the resulting mixture was stirred at 80 °C for 3 h. The reaction mixture was concentrated and extracted with EtOAc (3 x 20 mL). The combined organic extracts were dried over Na_2SO_4, filtered and concentrated. Purification by column chromatography on silica gel with EtOAc–hexane (2/98 to 5/95) afforded compound **2**.

***o*-Terphenyl (2a):** Using the general procedure **GP-5**, compound **3a** (500 mg, 1.9 mmol) and PTSA (96 mg, 0.56 mmol) provided compound **2a** (337 mg, 77%) as a viscous colorless liquid; ^{1}H NMR (500 MHz, $CDCl_3$): 7.42 (4H, m), 7.21-7.19 (6H, m), 7.15-7.12 (4H, m); ^{13}C (125 MHz, $CDCl_3$): 141.7, 140.7, 130.7, 130.0, 127.9, 127.6, 126.6; HRMS (ESI) calcd for $C_{18}H_{15}$ $[M+H]^+$: 231.1174; Found: 231.1169. *o*-Terphenyl **2a** is previously reported. [11f]

4,4''-Difluoro-*o*-terphenyl (2b): Using the general procedure **GP-5**, compound **3b** (500 mg, 1.6 mmol) and PTSA (82.6 mg, 0.48 mmol) provided compound **2b** (355 mg, 84%) as a colorless solid; ^{1}H NMR (500 MHz, $CDCl_3$): 7.42-7.37 (4H, m), 7.07 (4H, dd, *J* = 8.6, 3.2 Hz), 6.91 (4H, t, *J* = 8.6 Hz); ^{13}C (125 MHz, $CDCl_3$): 161.9 (d, $J_{C\text{-}F}$ = 244.4 Hz), 139.7, 137.5, 131.54 (d, $J_{C\text{-}F}$ = 7.7 Hz), 130.6, 127.8, 115.0 (d, $J_{C\text{-}F}$ = 21.2 Hz); HRMS (ESI) calcd for $C_{18}H_{12}NaF_2$ $[M+Na]^+$: 289.0805; Found: 289.0800. *o*-Terphenyl **2b** is previously reported. [6h]

2,2''-Difluoro-*o*-terphenyl (2c): Using the general procedure **GP-5**, compound **3c** (500 mg, 1.6 mmol) and PTSA (82.6 mg, 0.48 mmol) provided compound **2c** (360 mg, 85%) as a colorless solid; mp 90-94 °C; IR (ATR) *ν*. 2923, 2853, 1730, 1493, 1469, 1239, 1216, 753 cm^{-1}; ^{1}H NMR (500 MHz, $CDCl_3$): 7.47-7.42 (4H, m), 7.18 (2H, m), 7.11 (2H, t, *J* = 7.0 Hz), 6.99 (2H, t, *J* = 7.4 Hz), 6.91 (2H, t, *J* = 9.1 Hz); ^{13}C (125 MHz, $CDCl_3$): 159.7 (d, $J_{C\text{-}F}$ = 250.0 Hz), 135.8, 131.9, 130.8, 129.0 (d, $J_{C\text{-}F}$ = 8.7 Hz), 127.9, 123.6, 115.4 (d, $J_{C\text{-}F}$ = 22.2 Hz); HRMS (ESI) calcd for $C_{18}H_{12}NaF_2$ $[M+Na]^+$: 289.0805; Found: 289.0802. Anal calcd for $C_{18}H_{12}F_2$ (266.28): calcd. C 81.19, H 4.54; found C 81.17, H 4.59.

4,4''-Dichloro-*o*-terphenyl (2d): Using the general procedure **GP-5**, compound **3d** (500 mg, 1.5 mmol) and PTSA (77.5 mg, 0.45 mmol) provided compound **2d** (360 mg, 80%) as a white solid; mp 108-112 °C; IR (ATR) *ν*. 2951, 2922, 2852, 1602, 1465, 1376, 1091, 1006, 830, 758 cm^{-1}; ^{1}H NMR (500 MHz, $CDCl_3$): 7.42-7.37 (4H, m), 7.24 (4H, d, *J* = 8.4 Hz), 7.05 (4H, d, *J* = 8.4 Hz); ^{13}C (100 MHz, $CDCl_3$): 139.8, 139.4, 133.0, 131.2, 130.6, 128.4, 128.0; HRMS (ESI) calcd for $C_{18}H_{13}Cl_2$ $[M+H]^+$: 299.0394; Found: 299.0350. Anal calcd for $C_{18}H_{12}Cl_2$ (299.19): calcd. C 72.26, H 4.04; found C 72.20, H 4.00.

4,4''-Dimethyl-*o*-terphenyl (2e): Using the general procedure **GP-5**, compound **3e** (400 mg, 1.3 mmol) and PTSA (67.2 mg, 0.39 mmol) provided compound **2e** (255 mg, 76%) as a colorless solid; ^{1}H NMR (500 MHz, $CDCl_3$): 7.40-7.36 (4H, m), 7.05-7.01 (8H, m), 2.31 (6H, s); (100 MHz, $CDCl_3$): 140.6, 138.9, 136.1, 130.7, 129.8, 128.7, 127.3, 21.2; HRMS (ESI) calcd for $C_{20}H_{18}Na$ $[M+Na]^+$: 281.1306; Found: 281.1300. *o*-Terphenyl **2e** is previously reported. [6i]

3,3''-Dimethoxy-*o*-terphenyl (2f): Using the general procedure **GP-5**, compound **3f** (500 mg, 1.5 mmol) and PTSA (77.5 mg, 0.45 mmol) provided compound **2f** (384 mg, 88%) as a colorless solid; ^{1}H NMR (500 MHz, $CDCl_3$): 7.46-7.40 (4H, m), 7.14 (2H, t, *J* = 7.9 Hz), 6.79-6.74 (4H, m), 6.69-6.68 (2H, m), 3.62 (6H, s); ^{13}C (100 MHz, $CDCl_3$): 159.2, 143.0, 140.6, 130.5, 129.0, 127.6, 122.4, 115.3, 112.8, 55.2; HRMS (ESI) calcd for $C_{20}H_{18}NaO_2$ $[M+Na]^+$: 313.1204; Found: 313.1207. *o*-Terphenyl **2f** is previously reported. [12e]

2,2'',5,5''-Tetramethoxy-*o*-terphenyl (2g): Using the general procedure **GP-5,** compound **3g** (500 mg, 1.29 mmol) and PTSA (66.8 mg, 0.39 mmol) provided compound **2g** (384 mg, 85%)) as a colorless solid; mp 75-78 °C; IR (ATR) *ν*. 2960, 2923, 2853, 1733, 1648, 1612, 1462, 1260, 1086, 1018, 842,

799 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$): 7.42-7.38 (4H, m), 6.71-6.65 (6H, m), 3.62 (6H, s), 3.47 (6H, s); ^{13}C (125 MHz, $CDCl_3$): 153.0, 150.8, 138.1, 131.7, 130.6, 127.3, 117.0, 113.5, 111.5, 55.8, 55.7; HRMS (ESI) calcd for $C_{22}H_{23}O_4$ $[M+H]^+$: 351.1596; Found: 351.1590. Anal calcd for $C_{22}H_{22}O_4$ (350.40): calcd. C 75.41, H 6.33; found C 75.45, H 6.39.

2-Iodo-*o*-terphenyl (2h): Using the general procedure **GP-5**, compound **3h** (400 mg, 1.0 mmol) and PTSA (51.6 mg, 0.3 mmol) provided compound **2h** (292 mg, 82%) as a colorless solid; mp 80-83 °C; IR (ATR) *ν*. 2957, 2921, 2852, 1712, 1462, 1377, 1260, 1103, 1021, 802, 759, 746, 699 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$): 7.45-7.40 (4H, m), 7.21-7.18 (5H, m), 7.14-7.12 (4H, m); ^{13}C (100 MHz, $CDCl_3$): 141.7, 140.7, 130.7, 130.0, 127.9, 127.6, 126.5; HRMS (ESI) calcd for $C_{18}H_{14}I$ $[M+H]^+$: 357.0140; Found: 357.0156. Anal calcd for $C_{18}H_{13}I$ (356.20): calcd. C 60.69, H 3.68; found C 60.61, H 3.64.

2-Methoxy-*o*-terphenyl (2i): Using the general procedure **GP-5**, compound **3i** (400 mg, 1.3 mmol) and PTSA (67.2 mg, 0.39 mmol) provided compound **2i** (284 mg, 84%) as a colorless oil; ^1H NMR (400 MHz, $CDCl_3$): 7.42-7.37 (4H, m), 7.19-7.12 (6H, m), 6.91 (1H, t, *J* = 7.4 Hz), 6.69 (1H, d, *J* = 8.2 Hz), 3.33 (3H, s); ^{13}C (100 MHz, $CDCl_3$): 142.3, 141.2, 131.7, 131.0, 130.9, 129.8, 129.0, 128.6, 127.7, 127.5, 127.3, 126.3, 120.5, 110.8, 55.0; HRMS (ESI) calcd for $C_{19}H_{17}O$ $[M+H]^+$: 261.1279; Found: 261.1272. *o*-Terphenyl **2i** is previously reported.[6a]

4'-Methyl-*o*-terphenyl (2j): Using the general procedure **GP-5**, compound **3j** (400 mg, 1.4 mmol) and PTSA (72.3 mg, 0.42 mmol) provided compound **2j** (256 mg, 75%) as a colorless solid; mp 80-85 °C; IR (ATR) *ν*. 2951, 2923, 2553, 1463, 699 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$): 7.32 (1H, d, *J* = 7.6 Hz), 7.26-7.25 (2H, m), 7.20-7.14 (6 H, m), 7.13-7.11 (4H, m), 2.44 (3H, s); ^{13}C (100 MHz, $CDCl_3$): 141.8, 141.6, 140.6, 137.9, 137.3, 131.5, 130.7, 130.1, 128.8, 128.3, 127.9, 126.5, 126.3, 22.8; HRMS (ESI) calcd for $C_{19}H_{17}$ $[M+H]^+$: 245.1330; Found: 245.1334. Anal calcd for $C_{19}H_{16}$ (244.33): calcd. C 93.40, H 6.60; found C 93.43, H 6.57.

General procedure for C-C bond formation (GP-6): To a stirred solution of **2** (1.0 equiv.) in dry CH_2Cl_2 (8 mL), a solution of $FeCl_3$ (10.0 equiv.) in nitromethane (120 µL) was added and allowed to stir at room temperature for 24 h. The solvent was evaporated and the crude product was extracted with EtOAc (3 x 20 mL). The combined organic extracts were dried over Na_2SO_4, filtered and concentrated. Purification was done by column chromatography on silica gel with EtOAc–hexane (2/98 to 5/95) to obtain desired triphenylene derivative **1**.

2,7-Dimethoxytriphenylene (1f): Using the general procedure **GP-6**, compound **2f** (100 mg, 0.34 mmol) and $FeCl_3$ (558 mg, 3.4 mmol) provided compound **1f** (85 mg, 87%) as a colorless oil; ^1H NMR (500 MHz, $CDCl_3$): 8.58 (2H, dd, *J* = 6.1, 3.3 Hz), 8.47 (2H, d, *J* = 9.0 Hz), 8.04 (2H, d, *J* = 2.5 Hz), 7.65 (2H, dd, *J* = 6.2, 3.3 Hz), 7.27 (2H, d, *J* = 2.5 Hz), 4.03 (6H, s); ^{13}C (100 MHz, $CDCl_3$): 158.4, 130.3, 130.1, 127.3, 124.5, 124.1, 123.5, 116.1, 106.0, 55.6; HRMS (ESI) calcd for $C_{20}H_{16}NaO_2$ $[M+Na]^+$: 311.1048; Found: 311.1054. Triphenylene **1f** is previously reported.[12e]

1-Methoxytriphenylene (1i): Using the general procedure **GP-6**, compound **2i** (100 mg, 0.38 mmol) and $FeCl_3$ (623 mg, 3.8 mmol) provided compound **1i** (84 mg, 85%) as a colorless solid; ^1H NMR (500 MHz, $CDCl_3$): 7.40-7.35 (3H, m), 7.07 (5H, m), 6.88 (2H, d, *J* = 8.5 Hz), 6.58 (1H, d, *J* = 8.1 Hz), 3.33 (3H, s); ^{13}C (100 MHz, $CDCl_3$): 154.7, 142.3, 141.8, 137.5, 133.5, 132.3, 131.0, 130.6, 129.8, 129.1, 128.8, 127.6, 127.5, 127.2, 126.2, 110.9, 55.2; HRMS (ESI) calcd for $C_{19}H_{14}NaO$ $[M+Na]^+$: 281.0942; Found: 281.0944. Triphenylene **1i** is previously reported.[6g]

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Keywords: Aromatization · Benzil · Ring closing metathesis · *o*-Terphenyl · Triphenylene

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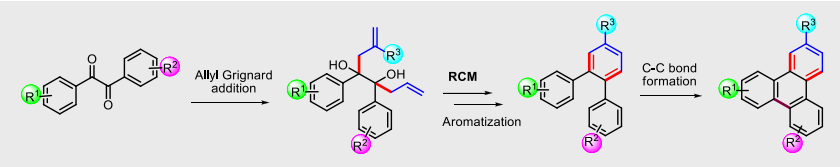

A synthetic method for *o*-terphenyls from benzils is reported using ring closing metathesis (RCM) as the key step. *o*-Terphenyls with halogen substituents are synthesized that can be further functionalized by cross coupling reactions. The synthesis of triphenylenes demonstrates the synthetic utility of the approach.

RCM Approach to *o*-Terphenyls*

*Shilpi Karmakar, Tirtha Mandal and Jyotirmayee Dash**

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Title

Ring Closing Metathesis Approach for the Synthesis of *o*-Terphenyl Derivatives