SUPPORTING INFORMATION

FOR

Copper-catalyzed enantioselective arylalkynylation of alkenes

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1. General Experimental Information

NMR spectra were recorded at room temperature on the following spectrometers: Agilent (400 MHz) and Bruker (400 MHz). EPR spectra were recorded at room temperature on the following spectrometer: Bruker E500 10/12. $^1$H spectra were calibrated in relation to the reference measurement of TMS (0.00 ppm). $^{13}$C spectra were calibrated in relation to deuterated solvents, namely CDCl$_3$ (77.00 ppm). The following abbreviations were used for $^1$H NMR spectra to indicate the signal multiplicity: s (singlet), d (doublet), t (triplet), q (quartet) and m (multiplet) as well as combinations of them. When combinations of multiplicities are given the first character noted refers to the largest coupling constant. ESI -HR and EI-HR (GC-TOF) spectrometer was applied. The method is denoted in brackets. For the most significant bands the wave number $\tilde{\nu}$ (cm$^{-1}$) is given.

Chemicals were purchased from commercial suppliers. Unless stated otherwise, all the substrates and solvents were purified and dried according to standard methods prior to use. Reactions requiring inert conditions were carried out in glove box.
2. Experimental detail

2.1 General Procedure

In a dried sealed tube, Ligand 9 (0.037 mmol, 5 mol %), K₂CO₃ (0.4 mmol, 2.0 equiv.) and Cu(MeCN)₄PF₆ (0.037 mmol, 5 mol %) were dissolved in anhydrous CH₃CN (2.0 mL) under a N₂ atmosphere, and the mixture was stirred for 10 min. Then diaryliodonium salts (0.25 mmol, 1.25 equiv.), terminal olefin (0.2 mmol, 1.0 equiv.) and terminal alkyne (0.4 mmol, 2.0 equiv.) were added sequentially. The tube was sealed with Teflon septum, the reaction mixture was stirred at room temperature for 24 h. After completion (monitored by TLC plate), the desired product was purified by column chromatography on silica gel with a gradient eluent of petroleum ether and ethyl acetate.

2.2 Synthesis and Characterization of Ligand

Ligands (L₁ to L₉) was synthesized according to reference¹.

2.3 Screening of Reaction Conditions
### Table S1. Ligand screening

<table>
<thead>
<tr>
<th>Entry</th>
<th>Ligand</th>
<th>Yield (%)&lt;sup&gt;a&lt;/sup&gt;</th>
<th>ee (%)&lt;sup&gt;b&lt;/sup&gt;</th>
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<td>12</td>
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<sup>a</sup>The reactions were carried out at room temperature.<br><sup>b</sup>Yields determined by <sup>1</sup>H NMR analysis with internal standard diethyl phthalate.<br><sup>c</sup>Determined by chiral HPLC analysis.

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### Table S2. Catalyst screening.

<table>
<thead>
<tr>
<th>Entry</th>
<th>Catalyst</th>
<th>Yield (%)&lt;sup&gt;a&lt;/sup&gt;</th>
<th>ee (%)&lt;sup&gt;b&lt;/sup&gt;</th>
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<td>CuBr</td>
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<td>CuCl</td>
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<td>Cu(MeCN)4PF&lt;sub&gt;6&lt;/sub&gt;</td>
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<tr>
<td>6</td>
<td>CuCN</td>
<td>68</td>
<td>85</td>
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</tbody>
</table>

<sup>a</sup>The reactions were carried out at room temperature.<br><sup>b</sup>Yields determined by <sup>1</sup>H NMR analysis with internal standard diethyl phthalate.<br><sup>c</sup>Determined by chiral HPLC analysis.
Table S3. Solvent screening

<table>
<thead>
<tr>
<th>Entry&lt;sup&gt;a&lt;/sup&gt;</th>
<th>Solvent</th>
<th>Yield (%)&lt;sup&gt;b&lt;/sup&gt;</th>
<th>ee (%)&lt;sup&gt;c&lt;/sup&gt;</th>
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<tr>
<td>4</td>
<td>MeCN</td>
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<tr>
<td>5</td>
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<tr>
<td>7</td>
<td>dioxane</td>
<td>trace</td>
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<sup>a</sup>The reactions were carried out at room temperature. <sup>b</sup>Yields determined by <sup>1</sup>HNMR analysis with internal standard diethyl phthalate. <sup>c</sup>Determined by chiral HPLC analysis.

Table S4. Base screening

<table>
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<tr>
<th>Entry&lt;sup&gt;a&lt;/sup&gt;</th>
<th>Base</th>
<th>Yield (%)&lt;sup&gt;b&lt;/sup&gt;</th>
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<tr>
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<td>86</td>
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<td>K&lt;sub&gt;2&lt;/sub&gt;HPO&lt;sub&gt;4&lt;/sub&gt;</td>
<td>34</td>
<td>85</td>
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<sup>a</sup>The reactions were carried out at room temperature. <sup>b</sup>Yields determined by <sup>1</sup>HNMR analysis with internal standard diethyl phthalate. <sup>c</sup>Determined by chiral HPLC analysis.
2.4 Synthesis and Characterization of products

\((R)-(3-(p\text{-tolyl})\text{but-1-yne-1,4-diyl})\text{dibenzene (4aa)}\)

According to general procedure, from 0.2 mmol of alkene, the desired product 4aa (46.2 mg, 0.16 mmol) was obtained as colorless oil in 78% yield.

\(^1\text{H NMR (400 MHz, CDCl}_3\) δ 7.45 – 7.38 (m, 2H), 7.35 – 7.27 (m, 8H), 7.25 (d, \(J = 7.7\) Hz, 2H), 7.18 (d, \(J = 7.7\) Hz, 2H), 4.09 (t, \(J = 7.3\) Hz, 1H), 3.14 (d, \(J = 7.3\) Hz, 2H), 2.39 (s, 3H). \(^{13}\text{C NMR (100 MHz, CDCl}_3\) δ 139.0, 138.3, 136.4, 131.5, 129.5, 129.1, 128.1, 128.0, 127.7, 127.5, 126.4, 123.7, 91.2, 84.1, 45.1, 40.4, 21.1. IR (neat) cm\(^{-1}\) ν: 3059, 2920, 1690, 1601, 1492, 1450, 1316, 1177, 1023, 807, 755, 593, 559, 524. HRMS: m/z (EI) calculated [M]\(^+\): 296.1565, found: 296.1562. HPLC (Chiralcel OD-H column, hexanes:i-PrOH = 100:0, 0.8 mL/min, 210 nm), \(t_{\text{minor}}\) = 25.9 min, \(t_{\text{major}}\) = 29.4 min, ee = 86%.

\([\alpha]_D^{25} = 5.4, (c = 0.47, \text{CHCl}_3)\).

Methyl \((R)-4-(1,4\text{-diphenylbut-3-yn-2-yl})\text{benzoate (4a)}\)

According to general procedure, from 0.2 mmol of alkene, the desired product 4a (56.4 mg, 0.17 mmol) was obtained as colorless oil in 83% yield.

\(^1\text{H NMR (400 MHz, CDCl}_3\) δ 7.90 (d, \(J = 7.9\) Hz, 2H), 7.35 – 7.27 (m, 4H), 7.23 – 7.19 (m, 3H), 7.18 – 7.12 (m, 3H), 7.05 (d, \(J = 6.9\) Hz, 2H), 4.05 (t, \(J = 7.1\) Hz, 1H), 3.82 (s, 3H), 3.10 – 2.97 (m, 2H). \(^{13}\text{C NMR (100 MHz, CDCl}_3\) δ 166.9, 146.4, 138.2, 131.5, 129.7, 129.5, 128.8, 128.2, 128.1, 128.0, 127.8, 126.6, 123.3, 90.0, 84.7, 52.0, 44.7, 40.7. IR (neat) cm\(^{-1}\) ν: 3060, 2949, 1718, 1691, 1605, 1491, 1435, 1275, 1178, 1106, 1018, 936, 857, 756, 694, 559, 485. HRMS : m/z (ESI) calculated [M+Na]\(^+\): 363.1361, found: 363.1355. HPLC (Chiralcel OD-H column, hexanes:i-PrOH = 98:2, 0.8 mL/min, 210 nm), \(t_{\text{minor}}\) = 8.9 min, \(t_{\text{major}}\) = 9.9 min, ee = 89%.  

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[α]D25 = 8.1, (c =0.15, CHCl3).

(R)-(3-(4-(trifluoromethyl)phenyl)but-1-yn-1,4-diyl)dibenzene (4b)

According to general procedure, from 0.2 mmol of alkene, the desired product 4b (55.3 mg, 0.16 mmol) was obtained as colorless oil in 79% yield.

1H NMR (400 MHz, CDCl3) δ 7.56 (d, J = 7.9 Hz, 2H), 7.44 (d, J = 8.0 Hz, 2H), 7.41 - 7.35 (m, 2H), 7.32 - 7.19 (m, 6H), 7.14 (d, J = 7.1 Hz, 2H), 4.13 (t, J = 7.2 Hz, 1H), 3.19 - 3.05 (m, 2H).

13C NMR (100 MHz, CDCl3) δ 140.2, 138.1, 131.5, 129.5, 129.2 (q, J = 32.3 Hz), 128.3, 128.1, 128.1, 128.0, 126.7, 125.4 (q, J = 3.0 Hz), 124.2 (q, J = 270.1 Hz), 123.2, 89.9, 84.8, 44.8, 40.1.

19F NMR (376 MHz, CDCl3) δ -62.2 (s, 3F). IR (neat) cm⁻¹ ν: 3063, 2924, 1692, 1600, 1492, 1450, 1413, 1321, 1118, 1065, 1016, 833, 754, 693, 603, 530. HRMS: m/z (EI) calculated [M]+: 350.1282, found: 350.1287. HPLC (Chiralcel OD-H column, hexanes:i-PrOH = 99.2:0.8, 0.35 mL/min, 210 nm), tminor = 15.2 min, tmajor = 15.9 min, ee = 86%.

[α]D25 = 4.9, (c =0.28, CHCl3).

(R)-but-3-yn-1,2,4-triyltribenzene (4c)

According to general procedure, from 0.2 mmol of alkene, the desired product 4c (40.6 mg, 0.14 mmol) was obtained as colorless oil in 72% yield.

1H NMR (400 MHz, CDCl3) δ 7.49 - 7.42 (m, 4H), 7.41 - 7.28 (m, 9H), 7.26 (d, J = 7.2 Hz, 2H), 4.15 (t, J = 7.2 Hz, 1H), 3.19 (d, J = 7.2 Hz, 2H). 13C NMR (100 MHz, CDCl3) δ 141.2, 138.8, 131.5, 129.5, 128.4, 128.1, 128.0, 127.8, 127.7, 126.8, 126.4, 123.6, 90.9, 84.3, 45.1, 40.8. IR (neat) cm⁻¹ ν: 3059, 2921, 1688, 1597, 1490, 1449, 1271, 1175, 1070 1026, 913, 843, 754, 691, 559, 513. HRMS: m/z (EI) calculated [M]+: 282.1409, found: 282.1411. HPLC (Chiralcel OD-H
column, hexanes:i-PrOH = 100:0, 0.8 mL/min, t_{\text{minor}} = 34.0 min, t_{\text{major}} = 30.7 min, ee = 87%.

\[ [\alpha]_D^{25} = 103.8 \text{ (c =0.05, CHCl}_3). \]

(4d)

(R)-4-(1,4-diphenylbut-3-yn-2-yl)-1,1'-biphenyl

According to general procedure, from 0.2 mmol of alkene, the desired product 4d (53.7 mg, 0.15 mmol) was obtained as colorless oil in 75% yield.

\(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 7.56 (dd, \(J = 16.7, 7.7\) Hz, 4H), 7.45 – 7.37 (m, 5H), 7.36 – 7.15 (m, 9H), 4.11 (t, \(J = 7.2\) Hz, 2H). \(^{13}\)C NMR (100 MHz, CDCl\(_3\)) \(\delta\) 140.8, 140.4, 139.7, 138.8, 131.5, 129.5, 128.7, 128.2, 128.1, 128.0, 127.8, 127.2, 127.1, 127.0, 126.5, 123.6, 90.8, 84.4, 45.0, 40.5. IR (neat) cm\(^{-1}\) \(\tilde{\nu}\): 3057, 2921, 1688, 1599, 1516, 1486, 1448, 1405, 1284, 1111, 912, 833, 756, 691, 569, 509. HRMS: m/z (EI) calculated [M]+: 358.1722, found: 358.1732. HPLC (Chiralcel OD-H column, hexanes:i-PrOH = 99.5:0.5, 0.8 mL/min, 210 nm),

\(t_{\text{minor}} = 13.7\) min, \(t_{\text{major}} = 15.7\) min, ee = 89%.

\[ [\alpha]_D^{25} = 7.0, \text{ (c =0.33, CHCl}_3). \]

(4e)

(R)-4-(1,4-diphenylbut-3-yn-2-yl)phenyl acetate

According to general procedure, from 0.2 mmol of alkene, the desired product 4e (59.8 mg, 0.18 mmol) was obtained as colorless oil in 88% yield.

\(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 7.45 – 7.38 (m, 4H), 7.36 – 7.30 (m, 5H), 7.28 (d, \(J = 6.9\) Hz, 1H), 7.23 (d, \(J = 7.4\) Hz, 2H), 7.09 (d, \(J = 8.3\) Hz, 2H), 4.13 (t, \(J = 7.2\) Hz, 1H), 3.15 (d, \(J = 7.3\) Hz, 2H), 2.34 (s, 3H). \(^{13}\)C NMR (100 MHz, CDCl\(_3\)) \(\delta\) 169.5, 149.4, 138.8, 138.6, 131.5, 129.5, 128.7,
According to general procedure, from 0.2 mmol of alkene, the desired product 4f (50.7 mg, 0.15 mmol) was obtained as colorless solid in 75% yield. Melting Point = 53 °C – 54 °C. 

\[^1\text{H}\] NMR (400 MHz, CDCl\(_3\)) \(\delta\) 7.46 – 7.39 (m, 6H), 7.37 – 7.26 (m, 8H), 4.12 (t, \(J = 7.1\) Hz, 1H), 3.24 – 3.05 (m, 2H), 1.39 (s, 9H). 

\[^{13}\text{C}\] NMR (100 MHz, CDCl\(_3\)) \(\delta\) 149.7, 139.2, 138.4, 131.5, 129.5, 128.1, 128.0, 127.7, 127.2, 126.4, 125.4, 123.7, 91.1, 84.2, 45.1, 40.4, 34.4, 31.4. IR (neat) cm\(^{-1}\) \(\tilde{\nu}\): 3053, 2922, 1690, 1598, 1508, 1360, 1334, 1288, 1149, 1069, 1021, 969, 860, 753, 692, 568, 522. HRMS: m/z (ESI) calculated [M+H]: 339.2113, found: 339.2112. HPLC (Chiralcel OD-H column, hexanes:i-PrOH = 99.7:0.3, 0.8 mL/min, 210 nm), \(t_{\text{minor}} = 14.3\) min, \(t_{\text{major}} = 16.5\) min, ee = 88%. 

\([\alpha]_D^{25} = 28.1, (c = 0.35, \text{CHCl}_3)\).

\((R)-(3-(4-(tert-butyl)phenyl)but-1-yne-1,4-diyl)dibenzene (4f)\)

\((R)-(3-(4-bromophenyl)but-1-yne-1,4-diyl)dibenzene (4g)\)

According to general procedure, from 0.2 mmol of alkene, the desired product 4g (61.2 mg, 0.17 mmol) was obtained as colorless oil in 85% yield.
$^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.42 (d, $J = 8.1$ Hz, 2H), 7.40 – 7.35 (m, 4H), 7.26 (dd, $J = 12.4$, 5.3 Hz, 4H), 7.20 (d, $J = 8.1$ Hz, 2H), 7.14 (d, $J = 7.3$ Hz, 2H), 4.04 (t, $J = 7.2$ Hz, 1H), 3.16 – 3.00 (m, 2H). $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 140.2, 138.3, 131.5, 131.4, 129.5, 129.5, 128.2, 128.1, 128.0, 126.6, 123.3, 120.7, 90.3, 84.6, 44.8, 40.2. IR (neat) cm$^{-1}$ $\tilde{\nu}$: 3059, 2922, 1689, 1584, 1485, 1450, 1402, 1318, 1285, 1176, 1070, 815, 753, 692, 556, 515. HRMS: m/z (El) calculated [M$^+$]: 360.0514, found: 360.0508. HPLC (Chiralcel OD-H column, hexanes:i-PrOH = 99.5:0.5, 0.8 mL/min, 210 nm), $t_{\text{minor}}$ = 7.5 min, $t_{\text{major}}$ = 7.9 min, ee = 87%.

$[\alpha]_D^{25} = 0.51$, (c =0.23, CHCl$_3$).

(R)-(3-(4-chlorophenyl)but-1-yne-1,4-diyl)dibenzene (4h)

According to general procedure, from 0.2 mmol of alkene, the desired product 4h (51.8 mg, 0.16 mmol) was obtained as colorless oil in 82% yield.

$^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.44 – 7.39 (m, 2H), 7.35 – 7.24 (m, 10H), 7.18 (d, $J = 7.5$ Hz, 2H), 4.09 (t, $J = 7.1$ Hz, 1H), 3.19 – 3.05 (m, 2H). $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 139.7, 138.3, 132.6, 131.5, 129.5, 129.1, 128.5, 128.2, 128.1, 127.9, 126.6, 123.4, 90.4, 84.5, 44.9, 40.1. IR (neat) cm$^{-1}$ $\tilde{\nu}$: 3061, 2922, 1689, 1589, 1488, 1450, 1317, 1175, 1090, 1013, 819, 754, 693, 557, 541, 524. HRMS: m/z (El) calculated [M$^+$]: 316.1019, found: 316.1007. HPLC (Chiralcel OD-H column, hexanes:i-PrOH = 99:1, 0.5 mL/min, 210 nm), $t_{\text{minor}}$ = 10.8 min, $t_{\text{major}}$ = 11.3 min, ee = 86%.

$[\alpha]_D^{25} = 1.3$, (c =0.42, CHCl$_3$).

(R)-(3-(4-fluorophenyl)but-1-yne-1,4-diyl)dibenzene (4i)

According to general procedure, from 0.2 mmol of alkene, the desired product 4i (48.1 mg, 0.16
mmol) was obtained as colorless oil in 80% yield.

$^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.41 – 7.34 (m, 2H), 7.31 – 7.17 (m, 8H), 7.13 (d, $J = 7.2$ Hz, 2H), 6.97 (t, $J = 8.8$ Hz, 2H), 4.05 (t, $J = 7.2$ Hz, 1H), 3.08 (qd, $J = 13.1, 7.1$ Hz, 2H). $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 161.7 (d, $J = 244$ Hz), 138.5, 136.8 (d, $J = 3$ Hz), 131.5, 129.5, 129.2 (d, $J = 8$ Hz), 128.2, 128.0, 127.9, 126.5, 123.4, 115.2 (d, $J = 21$ Hz), 90.7, 84.4, 45.1, 40.0. $^{19}$F NMR (376 MHz, CDCl$_3$) $\delta$ -116.0 – 115.9 (m, 1F). IR (neat) cm$^{-1}$: 3061, 2922, 1690, 1599, 1506, 1507, 1450, 1222, 1156, 1092, 1096, 1071, 1015, 913, 832, 755, 692, 557, 524. HRMS: m/z (EI) calculated [M]$^+$: 300.1314, found: 300.1323. HPLC (Chiralcel OD-H column, hexanes:i-PrOH = 99:1, 0.5 mL/min, 210 nm), $t_{\text{minor}}$ = 9.9 min, $t_{\text{major}}$ = 10.3 min, ee = 85%.

$\left[\alpha\right]_{D}^{25} = 3.4$, (c =0.23, CHCl$_3$).

(R)-(3-(4-(trifluoromethoxy)phenyl)but-1-yne-1,4-diyl)dibenzene (4j)

According to general procedure, from 0.2 mmol of alkene, the desired product 4j (56.4 mg, 0.15 mmol) was obtained as colorless oil in 77% yield.

$^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.51 – 7.46 (m, 2H), 7.43 (d, $J = 8.3$ Hz, 2H), 7.40 – 7.30 (m, 5H), 7.27 – 7.21 (m, 5H), 4.18 (t, $J = 7.2$ Hz, 1H), 3.26 – 3.12 (m, 2H). $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 148.0, 139.9, 138.3, 131.5, 129.5, 129.0, 128.2, 128.1, 128.0, 126.6, 123.3, 120.9, 120.5 (q, 257.6 Hz), 90.2, 84.7, 44.9, 40.1. $^{19}$F NMR (376 MHz, CDCl$_3$) $\delta$ -57.78 (s, 3F). IR (neat) cm$^{-1}$: 3062, 2923, 1692, 1599, 1504, 1450, 1416, 1253, 1210, 1158, 1018, 920, 847, 754, 692, 560, 527. HRMS: m/z (EI) calculated [M]$^+$: 366.1232, found: 366.1234. HPLC (Chiralcel OD-H column, hexanes:i-PrOH = 99.7:0.3, 0.8 mL/min, 210 nm), $t_{\text{minor}}$ = 23.3 min, $t_{\text{major}}$ = 24.4 min, ee = 91%.

$\left[\alpha\right]_{D}^{25} = 8.2$, (c =0.25, CHCl$_3$).

(R)-(3-(4-(difluoromethoxy)phenyl)but-1-yne-1,4-diyl)dibenzene (4k)
According to general procedure, from 0.2 mmol of alkene, the desired product 4k (51.5 mg, 0.15 mmol) was obtained as colorless oil in 74% yield.

$^1$H NMR (400 MHz, CDCl$_3$) δ 7.41 – 7.35 (m, 2H), 7.32 (d, $J = 8.4$ Hz, 2H), 7.29 – 7.19 (m, 6H), 7.15 (d, $J = 8.1$ Hz, 2H), 7.05 (d, $J = 8.3$ Hz, 2H), 6.47 (t, $J = 74.4$ Hz, 1H), 4.07 (t, $J = 7.2$ Hz, 1H), 3.15 – 3.02 (m, 2H). $^{13}$C NMR (100 MHz, CDCl$_3$) δ 150.0, 138.4, 131.5, 129.5, 129.1, 128.2, 128.1, 127.9, 126.5, 123.4, 119.4, 116.0 (t, $J = 260.6$ Hz), 90.5, 84.5, 45.0, 40.1. $^{19}$F NMR (376 MHz, CDCl$_3$) δ -80.53 (d, 2F). IR (neat) cm$^{-1}$: 3062, 2923, 1690, 1600, 1504, 1506, 1451, 1381, 1217, 1120, 1039, 830, 755, 692, 561, 525. HRMS: m/z (EI) calculated [M]$^+$: 348.1326, found: 348.1329. HPLC (Chiralcel OD-H column, hexanes:i-PrOH = 99.5:0.5, 0.8 mL/min, 210 nm), $t_{\text{minor}} = 10.0$ min, $t_{\text{major}} = 10.9$ min, ee = 86%.

$[\alpha]_D^{25} = 12.5$, (c =0.25, CHCl$_3$).

$(R)$-(3-(3-bromophenyl)but-1-yn-1,4-diyl)dibenzene (4l)

According to general procedure, from 0.2 mmol of alkene, the desired product 4l (55.4 mg, 0.15 mmol) was obtained as colorless oil in 77% yield.

$^1$H NMR (400 MHz, CDCl$_3$) δ 7.54 – 7.50 (m, 1H), 7.41 – 7.35 (m, 3H), 7.32 – 7.21 (m, 7H), 7.19 – 7.13 (m, 3H), 4.04 (t, $J = 6.7$ Hz, 1H), 3.15 – 3.03 (m, 2H). $^{13}$C NMR (100 MHz, CDCl$_3$) δ 143.5, 138.3, 131.5, 130.7, 130.0, 129.9, 129.5, 128.20, 128.10, 128.0, 126.6, 126.4, 90.0, 84.8, 44.9, 40.4. IR (neat) cm$^{-1}$: 3060, 2922, 1689, 1592, 1566, 1507, 1490, 1450, 1420, 1284, 1093, 1069, 1025, 995, 912, 878, 781, 753, 689, 527, 480. HRMS: m/z (EI) calculated [M]$^+$: 360.0514, found: 360.0510. HPLC (Chiralcel OD-H column, hexanes:i-PrOH = 100:0, 0.8 mL/min, 210 nm), $t_{\text{minor}} = 34.8$ min, $t_{\text{major}} = 32.1$ min, ee = 88%.

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$[\alpha]_D^{25} = 15.1$, (c =0.25, CHCl$_3$).

$(R)$-(3-(3-fluorophenyl)but-1-yn-1,4-diyl)dibenzene (4m)

According to general procedure, from 0.2 mmol of alkene, the desired product 4m (48.6 mg, 0.16 mmol) was obtained as colorless oil in 81% yield.

$^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.43 – 7.35 (m, 2H), 7.32 – 7.22 (m, 7H), 7.16 (d, $J = 6.7$ Hz, 2H), 7.09 (d, $J = 8.9$ Hz, 2H), 6.98 – 6.89 (m, 1H), 4.07 (t, $J = 7.2$ Hz, 1H), 3.16 – 3.03 (m, 2H). $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 162.8 (d, $J = 45.5$ Hz), 143.8 (d, $J = 7.1$ Hz), 138.4, 131.5, 129.8 (d, $J = 8.1$ Hz), 129.5, 128.2, 128.1, 128.0, 126.6, 123.4 (d, $J = 3.0$ Hz), 123.3, 114.7 (d, $J = 22.2$ Hz), 113.8 (d, $J = 21.2$ Hz), 90.1, 84.6, 44.8, 40.5, 40.3 (d, $J = 2.0$ Hz). $^{19}$F NMR (376 MHz, CDCl$_3$) $\delta$ -112.99 – -113.09 (m, 1F). IR (neat) $\tilde{\nu}$: 3061, 2923, 1691, 1587, 1487, 1445, 1246, 1071, 1025, 913, 872, 783, 755, 689, 521, 486. HRMS: m/z (EI) calculated [M]$^+$: 300.1314, found: 300.1309.

HPLC (Chiralcel OD-H column, hexanes:i-PrOH = 99.9:0.1, 0.8 mL/min, 210 nm), $t_{\text{minor}}$ = 29.0 min, $t_{\text{major}}$ = 26.0 min, ee = 87%.

$[\alpha]_D^{25} = 13.3$, (c =0.25, CHCl$_3$).

$(R)$-(3-(3,5-bis(trifluoromethyl)phenyl)but-1-yn-1,4-diyl)dibenzene (4n)

According to general procedure, from 0.2 mmol of alkene, the desired product 4n (71.1 mg, 0.17 mmol) was obtained as colorless oil in 85% yield.

$^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.77 (s, 1H), 7.72 (s, 2H), 7.45 – 7.37 (m, 2H), 7.35 – 7.25 (m, 6H), 7.11 (d, $J = 7.9$ Hz, 2H), 4.22 (t, $J = 7.2$ Hz, 1H), 3.19 (dd, $J = 13.2, 7.8$ Hz, 1H), 3.09 (dd, $J = 13.2, 6.7$ Hz, 1H). $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 143.6, 137.4, 131.6, 131.5 (q, $J = 33.3$Hz), 129.4, 128.3, 128.3, 128.0 (q, $J = 2.0$ Hz), 127.0, 123.7 (q, $J = 273.7$ Hz), 122.8, 121.0
According to general procedure, from 0.2 mmol of alkene, the desired product 4o (67.3 mg, 0.18 mmol) was obtained as colorless oil in 89% yield.

\[ ^1H \text{NMR (400 MHz, CDCl}_3 \delta 7.44 \text{ (dd, } J = 8.2, 7.1 \text{ Hz, 1H}), 7.38 \text{ (dd, } J = 6.6, 3.1 \text{ Hz, 1H}), 7.30 - 7.21 \text{ (m, 6H)}, 7.15 - 7.11 \text{ (m, 3H)}, 6.96 \text{ (dd, } J = 8.3, 2.1 \text{ Hz, 1H}), 4.04 \text{ (t, } J = 7.1 \text{ Hz, 1H}), 3.17 - 3.01 \text{ (m, 2H)}. \]

\[ ^13C \text{NMR (100 MHz, CDCl}_3 \delta 158.9 \text{ (d, } J = 248.5 \text{ Hz}), 143.0 \text{ (d, } J = 6.1 \text{ Hz}), 137.9, 133.2, 131.5, 129.4, 128.2, 128.1, 126.7, 124.6 \text{ (d, } J = 3.0 \text{ Hz}), 123.1, 115.9 \text{ (d, } J = 23.2 \text{ Hz}), 107.1 \text{ (d, } J = 21.2 \text{ Hz}), 89.6, 84.9, 44.6, 40.1. \]

\[ ^19F \text{NMR (376 MHz, CDCl}_3 \delta -107.15 \text{ (dd, } J = 9.6, 7.2 \text{ Hz, 1F}). \]

IR (neat) cm\(^{-1}\) \(\tilde{\nu}\): 3061, 2922, 1690, 1598, 1480, 1450, 1414, 1281, 1239, 1041, 913, 871, 811, 755, 692, 621, 572, 496. HRMS: m/z (EI) calculated [M]\(^+\): 378.0419, found: 308.0416. HPLC (Chiralcel AD-H column, hexanes:i-PrOH = 99.5:0.5, 0.8 mL/min, 210 nm), \(t_{\text{minor}} = 10.0 \text{ min, } t_{\text{major}} = 9.1 \text{ min, ee = 88\%}. \]

\[ [\alpha]_D^{25} = 1.3, (c =0.39, \text{CHCl}_3). \]

\((S)-2-(1,4\text{-diphenylbut-3-yn-2-yl})\text{thiophene (4p)}\)

\[
\begin{align*}
\text{According to general procedure, from 0.2 mmol of alkene, the desired product 4p (50.3 mg, 0.17} & \end{align*}
\]
mmol) was obtained as colorless oil in 87% yield.

\(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta 7.41 – 7.36 (m, 2H), 7.31 – 7.22 (m, 8H), 7.19 (dd, \(J = 4.4, 1.9\) Hz, 1H), 6.96 – 6.91 (m, 2H), 4.36 (t, \(J = 7.2\) Hz, 1H), 3.26 – 3.16 (m, 2H). \(^{13}\)C NMR (100 MHz, CDCl\(_3\)) \(\delta 144.7, 138.4, 131.5, 129.5, 128.2, 128.1, 127.9, 126.6, 126.6, 124.70, 124.0, 123.3, 99.2, 84.0, 45.1, 35.8.\) IR (neat) cm\(^{-1}\) \(\tilde{\nu}: 3027, 2921, 1690, 1597, 1490, 1440, 1226, 1071, 1029, 913, 847, 824, 754, 689, 543, 510.\) HRMS: m/z (ESI) calculated \([M+H]^+\): 289.1051, found: 289.1046.

HPLC (Chiralcel OD-H column, hexanes: \(i\)-PrOH = 99.6:0.4, 0.5 mL/min, 250 nm), \(t_{\text{minor}} = 15.0\) min, \(t_{\text{major}} = 14.5\) min, ee = 89%.

\([\alpha]_D^{25} = 38.2, (c =0.41, \text{CHCl}_3).\)

\((S)-2-(1,4\text{-diphenylbut-3-yn-2-yl})-3\text{-methylthiophene} (4q)\)

According to general procedure, from 0.2 mmol of alkene, the desired product 4q (43.6 mg, 0.14 mmol) was obtained as colorless oil in 72% yield.

\(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta 7.50 – 7.44 (m, 2H), 7.39 – 7.29 (m, 6H), 7.28 – 7.22 (m, 2H), 7.17 (d, \(J = 5.1\) Hz, 1H), 6.82 (d, \(J = 5.1\) Hz, 1H), 4.38 (t, \(J = 7.3\) Hz, 1H), 3.23 (ddd, \(J = 40.9, 13.1, 7.4\) Hz, 2H), 2.09 (s, 3H). \(^{13}\)C NMR (100 MHz, CDCl\(_3\)) 138.6, 137.9, 133.2, 131.5, 129.9, 129.4, 128.1, 128.1, 127.9, 126.6, 123.4, 122.3, 90.7, 83.3, 44.6, 34.2, 13.5. IR (neat) cm\(^{-1}\) \(\tilde{\nu}: 3026, 2922, 1673, 1594, 1489, 1443, 1071, 1028, 914, 833, 749, 693, 606, 546, 507.\) HRMS: m/z (EI) calculated \([M]^+\): 302.1129, found: 302.1131. HPLC (Chiralcel AD-H column, hexanes: \(i\)-PrOH = 99.6:0.4, 0.5 mL/min, 250 nm), \(t_{\text{minor}} = 7.9\) min, \(t_{\text{major}} = 8.5\) min, ee = 97%.

\([\alpha]_D^{25} = 22.5, (c =0.05, \text{CHCl}_3).\)

\((S)-5-(1,4\text{-diphenylbut-3-yn-2-yl})\text{thiazole} (4r)\)

According to general procedure, from 0.2 mmol of alkene, the desired product 4r (42.2 mg, 0.15 mmol) was obtained as orange oil in 73% yield.
\[ ^1 \text{H NMR (400 MHz, CDCl}_3 \] \( \delta \) 8.66 (s, 1H), 7.69 (s, 1H), 7.38 (dd, \( J = 6.7 \), 3.0 Hz, 2H), 7.33 – 7.24 (m, 6H), 7.21 – 7.17 (m, 2H), 4.40 (t, \( J = 7.2 \) Hz, 1H), 3.28 – 3.09 (m, 2H).\[ ^{13} \text{C NMR (100 MHz, CDCl}_3 \] \( \delta \) 152.3, 140.9, 139.5, 137.7, 131.6, 129.5, 128.4, 128.3, 127.0, 122.9, 89.3, 84.3, 44.9, 33.2.\[ \text{IR (neat) cm}^{-1} \tilde{\nu} : 3027, 2920, 1668, 1490, 1442, 1402, 1332, 1248, 1106, 1071, 1028, 912, 868, 794, 753, 691, 606, 557, 526.\[ \text{HRMS: m/z (ESI)} \] calculated [M+H]\(^+\): 290.1003, found: 290.1010. HPLC (Chiralcel AD-H column, hexanes:i-PrOH = 95:5, 0.8 mL/min, 250 nm), \( t_{\text{minor}} = 13.3 \) min, \( t_{\text{major}} = 13.9 \) min, ee = 89%. 

\[ [\alpha]_D^{25} = 55.7, (c =0.05, \text{CHCl}_3). \]

\((S)-5-(1,4\text{-diphenylbut-3-yn-2-yl})-4\text{-methylthiazole} (4s)\)

\[
\begin{align*}
&\text{According to general procedure, from 0.2 mmol of alkene, the desired product } 4s \text{ (31.5 mg, 0.10 mmol) was obtained as orange oil in 52\% yield.} \\
&\text{\[ ^1 \text{H NMR (400 MHz, CDCl}_3 \] \( \delta \) 8.64 (s, 1H), 7.46 – 7.39 (m, 2H), 7.37 – 7.25 (m, 7H), 7.17 (d, \( J = 7.2 \) Hz, 2H), 4.34 (t, \( J = 7.2 \) Hz, 1H), 3.28 (dd, \( J = 13.1 \), 7.2 Hz, 1H), 3.10 (dd, \( J = 13.0 \), 7.3 Hz, 1H), 2.22 (s, 3H).} \\
&\text{\[ ^{13} \text{C NMR (100 MHz, CDCl}_3 \] \( \delta \) 150.1, 148.8, 137.7, 132.2, 131.5, 129.3, 128.3, 128.2, 126.8, 122.9, 90.0, 83.5, 44.5, 32.8, 14.8.} \\
&\text{IR (neat) cm}^{-1} \tilde{\nu} : 3058, 2924, 1668, 1597, 1543, 1488, 1443, 1411, 1310, 1264, 1173, 1069, 932, 842, 797, 755, 692, 580, 512, 480.} \\
&\text{HRMS: m/z (ESI)} \] calculated [M+H]\(^+\): 304.1160, found: 304.1158. HPLC (Chiralcel OD-H column, hexanes:i-PrOH = 98:2, 0.8 mL/min, 210 nm), \( t_{\text{minor}} = 11.3 \) min, \( t_{\text{major}} = 11.9 \) min, ee = 97%. \\
&\[ [\alpha]_D^{25} = -10.6, (c =0.47, \text{CHCl}_3). \]
\]

\((S)-4-(3\text{-benzyl-4-(4-methoxyphenyl)but-1-yn-1-yl)}-1,1\text{-biphenyl} (4t)\)

\[
\begin{align*}
&\text{According to general procedure, from 0.2 mmol of alkene, the desired product } 4t \text{ (58.0 mg, 0.14}\]

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mmol) was obtained as yellow solid in 72% yield. Melting Point = 55 °C – 56 °C.

$^1$H NMR (400 MHz, CDCl$_3$) δ 7.62 (d, $J = 7.4$ Hz, 2H), 7.55 (d, $J = 8.2$ Hz, 2H), 7.48 (t, $J = 7.5$ Hz, 2H), 7.44 – 7.3 3 (m, 7H), 7.32 – 7.25 (m, 3H), 6.92 (d, $J = 8.5$ Hz, 2H), 3.84 (s, 3H), 3.17 – 3.08 (m, 1H), 2.99 – 2.82 (m, 4H). $^{13}$C NMR (101 MHz, CDCl$_3$) δ 158.1, 140.4, 140.3, 139.5, 131.8, 131.5, 130.3, 129.4, 128.8, 128.2, 127.4, 126.9, 126.8, 126.3, 122.8, 113.6, 93.2, 83.4, 55.2, 40.8, 40.0, 36.7. IR (neat) cm$^{-1}$ $\tilde{\nu}$: 3029, 2911, 2834, 1608, 1509, 1484, 1441, 1403, 1298, 1249, 1177, 1109,1034, 967, 836, 811, 759, 731, 692, 604, 570, 552, 531. HRMS: m/z (EI) calculated [M]$^+$: 402.1984, found: 402.1982. HPLC (Chiralcel OD-H column, hexanes:i-PrOH = 99:0.7, 0.7 mL/min, 210 nm), $t_{\text{minor}} = 17.2$ min, $t_{\text{major}} = 18.0$ min, ee = 0%.

$(R)$-2-(4-([1,1′-biphenyl]-4-yl)-2-benzylbut-3-yn-1-yl)isoindoline-1,3-dione(4u)

According to general procedure, from 0.2 mmol of alkene, the desired product 4u (69.8 mg, 0.16 mmol) was obtained as orange solid in 79% yield. Melting Point = 79 °C – 80 °C.

$^1$H NMR (400 MHz, CDCl$_3$) δ 7.85 (dd, $J = 5.5$, 3.0 Hz, 2H), 7.71 (dd, $J = 5.4$, 3.1 Hz, 2H), 7.56 – 7.52 (m, 2H), 7.45 (dd, $J = 9.2$, 2.6 Hz, 2H), 7.41 (d, $J = 7.9$ Hz, 2H), 7.36 – 7.26 (m, 7H), 7.22 – 7.17 (m, 1H), 4.01 (dd, $J = 13.4$, 8.6 Hz, 1H), 3.85 (dd, $J = 13.4$, 6.7 Hz, 1H), 3.53 (p, $J = 7.9$, 7.4 Hz, 1H), 3.03 – 2.87 (m, 2H). $^{13}$C NMR (101 MHz, CDCl$_3$) δ 168.2, 140.6, 140.4, 138.3, 134.0, 132.0, 131.9, 129.2, 128.8, 128.3, 127.5, 127.0, 126.8, 126.5, 123.3, 122.2, 89.8, 83.7, 41.8, 39.1, 33.7. IR (neat) cm$^{-1}$ $\tilde{\nu}$: 3028, 2929, 1771, 1713, 1603, 1485, 1393, 1355, 1306, 1188, 1109,1073, 994, 901, 842, 761, 720, 692, 607, 487. HRMS: m/z (ESI) calculated [M+H]$^+$: 442.1807, found: 442.1803. HPLC (Chiralcel OD-H column, hexanes:i-PrOH = 99:1, 0.8 mL/min, 210 nm), $t_{\text{minor}} = 21.2$ min, $t_{\text{major}} = 25.1$ min, ee = 22%.

$[\alpha]_D^{25} = 0.3$, (c = 0.8, CHCl$_3$).

$(S)$-4-(3-cyclohexyl-4-phenylbut-1-yn-1-yl)-1,1′-biphenyl(4v)
According to general procedure, from 0.2 mmol of alkene, the desired product 4v (64.2 mg, 0.18 mmol) was obtained as colorless oil in 88% yield.

\[ ^1H\text{ NMR (400 MHz, CDCl}_3\text{)} \delta 7.59 - 7.54 (m, 2H), 7.52 - 7.47 (m, 2H), 7.45 - 7.38 (m, 4H), 7.36 - 7.27 (m, 5H), 7.25 - 7.19 (m, 1H), 2.94 - 2.79 (m, 2H), 2.76 - 2.64 (m, 1H), 1.97 (d, \text{ } J = 11.2 \text{ Hz, 1H}), 1.85 - 1.73 (m, 3H), 1.68 (d, \text{ } J = 9.8 \text{ Hz, 1H}), 1.48 - 1.22 (m, 6H). \]

\[ ^{13}C\text{ NMR (101 MHz, CDCl}_3\text{)} \delta 140.6, 140.3, 140.2, 131.9, 129.2, 128.8, 128.1, 127.4, 126.9, 126.8, 126.1, 123.1, 92.6, 83.5, 41.1, 40.9, 38.8, 31.9, 28.89, 26.47, 26.5, 26.4, 26.3. \]

IR (neat) cm\(^{-1}\): 3028, 2921, 2850, 1601, 1485, 1447, 1073, 1007, 837, 760, 695, 558, 499. HRMS: m/z (EI) calculated [M]\(^+\): 364.2191, found: 364.2196. HPLC (Chiralcel OD-H column, hexanes:i-PrOH = 99:1, 0.8 mL/min, 250 nm), \( t_{\text{minor}} = 5.9 \text{ min, } t_{\text{major}} = 6.4 \text{ min, ee = 22%}. \)

\[ [\alpha]_D^{25} = 4.8, (c = 0.4, \text{ CHCl}_3). \]

**Methyl (S)-6-([1,1'-biphenyl]-4-yl)-4-benzyl-3,3-dimethylhex-5-ynoate(4w)**

According to general procedure, from 0.2 mmol of alkene, the desired product 4w (55.5 mg, 0.14 mmol) was obtained as yellow solid in 70% yield. Melting point = 50 °C - 51 °C.

\[ ^1H\text{ NMR (400 MHz, CDCl}_3\text{)} \delta 7.60 (d, \text{ } J = 8.1 \text{ Hz, 2H}), 7.53 (d, \text{ } J = 8.1 \text{ Hz, 2H}), 7.47 (t, \text{ } J = 7.7 \text{ Hz, 2H}), 7.43 - 7.34 (m, 7H), 7.30 - 7.25 (m, 1H), 3.73 (s, 3H), 3.08 (dd, \text{ } J = 12.6, 3.2 \text{ Hz, 1H}), 2.91 (dd, \text{ } J = 11.6, 3.1 \text{ Hz, 1H}), 2.70 - 2.53 (m, 3H), 1.30 (s, 6H). \]

\[ ^{13}C\text{ NMR (101 MHz, CDCl}_3\text{)} \delta 172.5, 140.5, 140.4, 140.3, 131.8, 129.4, 128.8, 128.1, 127.5, 127.0, 126.8, 126.2, 122.8, 91.4, 84.7, 51.3, 45.6, 44.4, 36.6, 36.0, 25.2, 25.1. \]

IR (neat) cm\(^{-1}\): 3028, 2961, 1731, 1601, 1485, 1435, 1324, 1216, 1148, 1111, 1073, 1007, 913, 842, 761, 733, 557, 500. HRMS: m/z (EI) calculated [M+H]\(^+\):
397.2168, found: 397.2258. HPLC (Chiralcel OD-H column, hexanes:i-PrOH = 99:1, 0.8 mL/min, 250 nm), t\textsubscript{minor} = 9.7 min, t\textsubscript{major} = 11.5 min, ee = 40%.

$\left[\alpha\right]_D^{25} = 73.3$, (c = 0.4, CHCl\textsubscript{3}).

\((R)-(4-[[1,1'-biphenyl]-4-yl]-1-phenylbut-3-yn-2-yl)trimethylsilane(4x)\)

According to general procedure, from 0.2 mmol of alkene, the desired product 4x (57.4 mg, 0.16 mmol) was obtained as colorless oil in 81% yield.

$^1$H NMR (400 MHz, CDCl\textsubscript{3}) $\delta$ 7.62 (d, $J = 7.1$ Hz, 2H), 7.54 (d, $J = 8.4$ Hz, 2H), 7.51 – 7.35 (m, 9H), 7.30 – 7.26 (m, 1H), 2.94 (dd, $J = 13.6$, 4.2 Hz, 1H), 2.82 (dd, $J = 13.6$, 11.0 Hz, 1H), 2.21 (dd, $J = 10.9$, 4.2 Hz, 1H), 0.24 (s, 9H). $^{13}$C NMR (101 MHz, CDCl\textsubscript{3}) $\delta$ 141.7, 140.6, 139.8, 131.8, 128.8, 128.7, 128.2, 127.3, 126.9, 126.8, 126.1, 123.8, 92.8, 82.1, 35.6, 23.5, -3.0. IR (neat) cm$^{-1}$ $\tilde{\nu}$: 3028, 2953, 2209, 1600, 1516, 1485, 1450, 1404, 1247, 1108, 1028, 835, 760, 693, 620, 557, 497. HRMS: m/z (EI) calculated [M]$^+$: 354.1804, found: 354.1805. HPLC (Chiralcel OD-H column, hexanes:i-PrOH = 99:1, 0.8 mL/min, 210 nm), t\textsubscript{minor} = 5.4 min, t\textsubscript{major} = 5.1 min, ee = 32%.

$\left[\alpha\right]_D^{25} = -60.4$, (c =0.4, CHCl\textsubscript{3}).

\((S)-1-(tert-butyl)-4-(3-(4-fluorobenzyl)-4,4-dimethylpent-1-yn-1-yl)benzene(4y)\)

According to general procedure, from 0.2 mmol of alkene, the desired product 4y (55.9 mg, 0.17 mmol) was obtained as colorless oil in 83% yield.

$^1$H NMR (400 MHz, CDCl\textsubscript{3}) $\delta$ 7.36 – 7.24 (m, 6H), 7.02 (t, $J = 8.3$ Hz, 2H), 2.98 (dd, $J = 12.3$, 2.3 Hz, 1H), 2.64 – 2.46 (m, 2H), 1.33 (s, 9H), 1.16 (s, 9H). $^{19}$F NMR (376 MHz, CDCl\textsubscript{3}) $\delta$ -115.62 – 115.69 (m, 1F). $^{13}$C NMR (101 MHz, CDCl\textsubscript{3}) $\delta$ 161.5 (d, $J = 244.0$ Hz), 150.6, 136.7, 131.1, 130.6 (d, $J = 7.9$ Hz), 125.1, 121.1, 114.8, 114.6, 90.7, 84.2, 47.3, 35.6, 34.6, 34.0, 31.2, 27.7. IR
(neat) cm$^{-1}$ v: 2959, 2866, 1692, 1364, 1268, 1221, 1156, 1110, 1093, 1017, 829, 784, 740, 563, 528, 499. HRMS: m/z (EI) calculated [M]$^+$: 336.2253, found: 336.2251.

HPLC (Chiralcel OD-H column, hexanes:i-PrOH = 99.8:0.2, 0.7 mL/min, 210 nm), $t_{\text{minor}} = 8.3$ min, $t_{\text{major}} = 6.8$ min, ee = 47%.

$[\alpha]_D^{25} = 112.2$, (c =0.4, CHCl$_3$).

 According to general procedure, from 0.2 mmol of alkene, the desired product 4z (45.8 mg, 0.14 mmol) was obtained as white solid in 68% yield.$^3$

$^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.53 (d, $J = 7.4$ Hz, 2H), 7.45 – 7.37 (m, 4H), 7.36 – 7.28 (m, 5H), 7.25 – 7.18 (m, 3H), 2.70 – 2.56 (m, 2H), 2.24 (d, $J = 15.3$ Hz, 1H), 1.94 (d, $J = 13.3$ Hz, 1H), 1.86 (d, $J = 9.1$ Hz, 2H), 1.70 – 1.51 (m, 2H), 1.47 – 1.36 (m, 2H). $^{13}$C NMR (101 MHz, CDCl$_3$) $\delta$ 145.4, 140.5, 140.0, 131.8, 128.8, 128.1, 127.6, 126.9, 126.7, 126.2, 122.9, 94.0, 81.6, 50.2, 37.1, 34.1, 33.6, 26.3, 25.8. IR (neat) cm$^{-1}$ v: 3029, 2924, 2851, 1599, 1484, 1444, 1300, 1158, 1112, 1004, 837, 757, 696, 554, 510, 483. HRMS: m/z (EI) calculated [M]$^+$: 336.1878, found: 336.1888. HPLC (Chiralcel OD-H column, hexanes:i-PrOH = 100:0, 0.6 mL/min, 210 nm), $t_{\text{minor}} = 44.4$ min, $t_{\text{major}} = 47.7$ min, ee = 16%.

$[\alpha]_D^{25} = 7.2$, (c =0.5, CHCl$_3$).

(1S,2R,3R,4R)-2-phenyl-3-(phenylethynyl)bicyclo[2.2.1]heptane(4bb)

According to general procedure, from 0.2 mmol of alkene, the desired product 4bb (40.9 mg, 0.15 mmol) was obtained as colorless oil in 75% yield.$^3$

$^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.53 – 7.47 (m, 2H), 7.43 – 7.31 (m, 7H), 7.30 – 7.23 (m, 1H), 2.92 (m, 1H), 2.85 (m, 1H), 2.63 – 2.52 (m, 2H), 2.11 (m, 1H), 1.83 – 1.68 (m, 2H), 1.67 – 1.51 (m,
2H), 1.48 (dq, \( J = 10.0, 1.5 \text{ Hz}, 1 \text{H} \)). \(^{13}\text{C} \text{ NMR} (101 \text{ MHz}, \text{CDCl}_3) \delta 145.6, 131.6, 128.4, 128.2, 127.5, 126.6, 125.9, 124.0, 92.7, 82.0, 55.4, 42.9, 42.4, 42.3, 37.5, 30.7, 23.6. IR (neat) cm\(^{-1}\): 3057, 2953, 2871, 2218, 1598, 1489, 1446, 1329, 1070, 1030, 908, 839, 752, 732, 696, 548. HRMS: m/z (EI) calculated [M]\(^+\): 272.1565, found: 272.1569. HPLC (Chiralcel OD-H column, hexanes:i-PrOH = 100:0, 0.7 mL/min, 210 nm), \( t_{\text{minor}} = 15.3 \text{ min}, t_{\text{major}} = 14.6 \text{ min}, \text{ ee} = 53\% \).

\([\alpha]_D^{25} = -86.3, (c =0.3, \text{CHCl}_3)\).

\((R)\)-but-3-yne-1,1,2,4-tetrayltetrabenzene (4cc)

According to general procedure, from 0.2 mmol of alkene, the desired product 4cc (32.9 mg, 0.09 mmol) was obtained as colorless oil in 46\% yield.

\(^1\text{H} \text{ NMR} (400 \text{ MHz}, \text{CDCl}_3) \delta 7.42 – 7.20 (m, 20H), 4.73 (d, \( J = 8.1 \text{ Hz}, 1 \text{H} \)), 4.43 (d, \( J = 8.1 \text{ Hz}, 1 \text{H} \)). \(^{13}\text{C} \text{ NMR} (100 \text{ MHz}, \text{CDCl}_3) \delta 142.5, 141.9, 140.4, 131.4, 129.3, 128.5, 128.4, 128.2, 128.1, 127.8, 127.7, 126.8, 126.5, 126.3, 90.6, 85.6, 58.9, 43.6. IR (neat) cm\(^{-1}\): 3027, 2919, 1804, 1751, 1666, 1592, 1487, 1445, 1347, 1239, 1148, 1072, 1025, 1071, 1015, 910, 830, 747, 691, 618, 515. HRMS: m/z (EI) calculated [M]\(^+\): 358.1722, found: 358.1720. HPLC (Chiralcel AD-H column, hexanes:i-PrOH = 99.5:0.5, 0.5 mL/min, 254 nm), \( t_{\text{minor}} = 11.9 \text{ min}, t_{\text{major}} = 12.9 \text{ min}, \text{ ee} = 74\% \).

\([\alpha]_D^{25} = 13.4, (c =0.25, \text{CHCl}_3)\).

\((S)\)-9-(1-(4-fluorophenyl)-4-phenylbut-3-yn-2-yl)-9H-carbazole (4dd)

According to general procedure, from 0.2 mmol of alkene, the desired product 4dd (45.2 mg, 0.12 mmol) was obtained as colorless oil in 58\% yield.

\(^1\text{H} \text{ NMR} (400 \text{ MHz}, \text{CDCl}_3) \delta 8.13 (d, \( J = 7.7 \text{ Hz}, 2 \text{H} \)), 7.69 – 7.59 (m, 2H), 7.51 – 7.41 (m, 4H), 7.37 – 7.33 (m, 3H), 7.31 – 7.25 (m, 2H), 7.06 (dd, \( J = 8.5, 5.4 \text{ Hz}, 2 \text{H} \)), 6.87 (t, \( J = 8.7 \text{ Hz}, 2 \text{H} \)), 5.82 (t, \( J = 7.4 \text{ Hz}, 1 \text{H} \)), 3.64 – 3.39 (m, 2H)). \(^{13}\text{C} \text{ NMR} (100 \text{ MHz}, \text{CDCl}_3) \delta 161.96 (d, \( J = 243.8 \text{ Hz} \)), 139.3, 132.6, 131.6, 130.8 (d, \( J = 7.9 \text{ Hz} \)), 128.6, 128.3, 125.6, 123.3, 122.3, 120.3, 119.3,
115.2 (d, J = 21.1 Hz), 110.0, 86.6, 85.7, 49.0, 40.3. \(^{19}\)F NMR (376 MHz, CDCl\(_3\)) \(\delta -115.70 \ldots -115.78\) (m, 1F).

IR (neat) cm\(^{-1}\): 3054, 2922, 2854, 1594, 1445, 1327, 1222, 1158, 1024, 920, 827, 737, 686, 522. HRMS: m/z (EI) calculated \([M]^+\): 389.1580, found: 389.1586. HPLC (Chiralcel AD-H column, hexanes:i-PrOH = 99.7:0.3, 0.6 mL/min, 254 nm), \(t_{\text{major}} = 16.5\) min, \(t_{\text{minor}} = 22.1\) min, ee = 11%.

\([\alpha]_D^{25} = 14.8, (c =0.25, \text{CHCl}_3)\)

IR (neat) cm\(^{-1}\): 3026, 2922, 1673, 1594, 1489 1443, 1416, 1317, 1172, 1024, 914, 838, 750, 693, 546, 507. HRMS: m/z (EI) calculated \([M]^+\): 302.1129, found: 302.1126. HPLC (Chiralcel AD-H column, hexanes:i-PrOH = 99.6:0.4, 0.7 mL/min, 210 nm), \(t_{\text{minor}} = 7.9\) min, \(t_{\text{major}} = 8.5\) min, ee = 97%.

\([\alpha]_D^{25} = 10.5, (c =0.4, \text{CHCl}_3)\).

(8R,9S,13S,14S)-3-(R)-1,4-diphenylbut-3-yn-2-yl)-13-methyl-6,7,8,9,11,12,13,14,15,16-decahydro-17H-cyclopenta[a]phenanthren-17-one (4ee)

According to general procedure, from 0.2 mmol of alkene, the desired product 4ee (55.0 mg, 0.12 mmol) was obtained as colorless oil in 60% yield.

\(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta 7.46 \ldots 7.28\) (m, 1H), 7.38 – 7.28 (m, 10H), 7.24 – 7.16 (m, 1H), 4.12 – 4.04 (m, 1H), 3.22 – 3.10 (m, 2H), 3.05 – 2.92 (m, 2H), 2.63 – 2.45 (m, 2H), 2.42 – 2.31 (m, 1H), 2.28 – 2.01 (m, 5H), 1.80 – 1.45 (m, 8H), 0.98 (s, 3H). \(^{13}\)C NMR (100 MHz, CDCl\(_3\)) \(\delta 220.8, 139.1, 138.9, 136.5, 131.5, 129.4, 128.1, 128.1, 128.0, 127.7, 126.4, 125.4, 125.0, 123.7, 91.1, 84.1, 50.4, 47.9, 45.0, 44.2, 40.4, 38.1, 35.8, 31.5, 29.4, 26.4, 25.6, 21.5, 13.8.

IR (neat) cm\(^{-1}\): 2923, 2860, 1731, 1599, 1490, 1446, 1253, 1077, 1007, 907, 822, 727, 695, 576, 522, 443. HRMS: m/z (EI) calculated \([M]^+\): 458.2610, found: 458.2618. HPLC (Chiralcel OD-H column, hexanes:i-PrOH = 99:1, 0.7 mL/min, 210 nm), \(t_{\text{minor}} = 24.2\) min, \(t_{\text{major}} = 27.0\) min, ee = 80%.

\([\alpha]_D^{25} = 5.6, (c =0.40, \text{CHCl}_3)\).

(S)-5-(4-([1,1'-biphenyl]-4-yl)-1-phenylbut-3-yn-2-yl)-4-methylthiazole (5a)
According to general procedure, from 0.2 mmol of alkene, the desired product 5a (43.2 mg, 0.11 mmol) was obtained as orange solid in 57% yield. Melting Point = 76 °C – 77 °C.

$^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 8.57 (s, 1H), 7.52 (dd, $J = 15.8$, 7.8 Hz, 4H), 7.46 – 7.36 (m, 4H), 7.31 (t, $J = 7.4$ Hz, 1H), 7.23 (t, $J = 8.1$ Hz, 3H), 7.11 (d, $J = 6.8$ Hz, 2H), 4.30 (t, $J = 7.3$ Hz, 1H), 3.22 (dd, $J = 13.2$, 7.3 Hz, 1H), 3.04 (dd, $J = 13.2$, 7.3 Hz, 1H), 2.16 (s, 3H). $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 150.0, 148.8, 140.9, 140.3, 137.7, 132.2, 131.9, 129.3, 128.8, 128.3, 127.6, 126.9, 126.9, 126.8, 121.8, 90.7, 83.4, 44.5, 32.9, 14.8. IR (neat) cm$^{-1}$ $\tilde{\nu}$: 3061, 2918, 1533, 1484, 1450, 1405, 1320, 1110, 973, 830, 760, 692, 557, 493. HRMS: m/z (ESI) calculated [M+H$^+$]: 380.1473, found: 380.1465. HPLC (Chiralcel AD-H column, hexanes:i-PrOH = 95:5, 0.8 mL/min, 210 nm), $t_{\text{minor}}$ = 18.6 min, $t_{\text{major}}$ = 31.5 min, ee = 98%.

$[^{\alpha}]_{D^{25}}$ = -18.8, (c =0.40, CHCl$_3$).

(S)-5-(4-(4-methoxyphenyl)-1-phenylbut-3-yn-2-yl)-4-methylthiazole (5b)

According to general procedure, from 0.2 mmol of alkene, the desired product 5b (40.6 mg, 0.12 mmol) was obtained as orange oil in 61% yield.

$^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 8.58 (s, 1H), 7.33 (d, $J = 7.6$ Hz, 2H), 7.29 – 7.19 (m, 3H), 7.12 (d, $J = 6.9$ Hz, 2H), 6.82 (d, $J = 8.4$ Hz, 2H), 4.28 (t, $J = 7.3$ Hz, 1H), 3.78 (s, 3H), 3.22 (dd, $J = 13.4$, 7.2 Hz, 1H), 3.04 (dd, $J = 13.3$, 7.4 Hz, 1H), 2.17 (s, 3H). $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 159.4, 149.9, 148.7, 137.8, 132.9, 132.5, 129.3, 128.2, 126.8, 115.0, 113.8, 88.6, 83.4, 55.2, 44.6, 32.8, 14.8. IR (neat) cm$^{-1}$ $\tilde{\nu}$: 3061, 2918, 1698, 1603, 1540, 1453, 1440, 1287, 1244, 1169, 1105, 1028, 938, 830, 752, 563, 487. HRMS: m/z (ESI) calculated [M+H$^+$]: 334.1266, found: 334.1266. HPLC (Chiralcel AD-H column, hexanes:i-PrOH = 95:5, 0.8 mL/min, 210 nm), $t_{\text{minor}}$ = 16.9 min, $t_{\text{major}}$ = 26.6 min, ee = 97%.
$[\alpha]_D^{25} = -7.6$, (c = 0.30, CHCl$_3$).

(R)-1-methoxy-4-(4-phenyl-3-(4-trifluoromethyl)phenyl)but-1-yn-1-yl)benzene (5c)

According to general procedure, from 0.2 mmol of alkene, the desired product 5c (57.0 mg, 0.15 mmol) was obtained as colorless oil in 75% yield.

$^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.55 (d, $J = 8.0$ Hz, 2H), 7.43 (d, $J = 8.0$ Hz, 2H), 7.32 (d, $J = 8.2$ Hz, 2H), 7.30 – 7.20 (m, 3H), 7.14 (d, $J = 7.2$ Hz, 2H), 4.12 (t, $J = 7.2$ Hz, 1H), 3.79 (s, 3H), 3.10 (qd, $J = 13.2$, 7.1 Hz, 2H). $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 159.4, 145.4, 138.2, 132.9, 129.5, 129.1 (q, $J = 32.1$ Hz), 128.1, 126.6, 125.3 (q, $J = 3.8$ Hz), 124.2 (q, $J = 270.3$ Hz), 115.3, 113.8, 88.3, 84.6, 55.2, 44.8, 40.6. $^{19}$F NMR (376 MHz, CDCl$_3$) $\delta$ -62.29 (s, 3F). IR (neat) cm$^{-1}$ $\tilde{\nu}$: 3029, 2931, 1689, 1602, 1508, 1455, 1416, 1322, 1246, 1163, 1109, 1065, 1017, 830, 731, 698, 602, 534.

HRMS: m/z (ESI) calculated [M+H]$^+$: 381.1466, found: 381.1448. HPLC (Chiralcel OD-H column, hexanes:i-PrOH = 99.5:0.5, 0.8 mL/min, 210 nm), $t_{\text{minor}} = 9.3$ min, $t_{\text{major}} = 9.6$ min, ee = 89%.

$[\alpha]_D^{25} = 8.5$, (c = 0.30, CHCl$_3$).

(R)-4-(4-(4-(tert-butyl)phenyl)-1-phenylbut-3-yn-2-yl)phenyl acetate (5d)

According to general procedure, from 0.2 mmol of alkene, the desired product 5d (61.1 mg, 0.15 mmol) was obtained as colorless oil in 73% yield.

$^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.41 (d, $J = 8.5$ Hz, 2H), 7.39 – 7.26 (m, 7H), 7.24 (d, $J = 7.9$ Hz, 2H), 7.08 (d, $J = 8.4$ Hz, 2H), 4.12 (t, $J = 7.2$ Hz, 1H), 3.14 (d, $J = 7.2$ Hz, 2H), 2.33 (s, 3H), 1.35 (s, 9H). $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 169.5, 151.0, 149.4, 138.9, 138.6, 131.2, 129.5, 128.7,
128.0, 126.4, 125.2, 121.4, 120.4, 89.8, 84.5, 45.1, 40.2, 34.6, 31.1, 21.1. IR (neat) cm\(^{-1}\) \(\tilde{\nu}\): 3030, 2960, 1758, 1687, 1502, 1366, 1192, 1163, 1013, 910, 834, 698, 560, 522. HRMS: m/z (ESI) calculated [M+H\(^+\)]\(^+\): 419.1987, found: 419.1993. HPLC (Chiralcel AD-H column, hexanes:i-PrOH = 97:3, 0.8 mL/min, 250 nm), \(t_{\text{minor}}\) = 12.1 min, \(t_{\text{major}}\) = 9.9 min, ee = 90%.
\([\alpha]_D^{25}\) = 68.0, (c =0.25, CHCl\(_3\)).

\(\left(R\right)-4-(4-(4\text{-bromophenyl})-1\text{-phenylbut-3-yn-2-yl})\text{phenyl acetate (5e)}\)

According to general procedure, from 0.2 mmol of alkene, the desired product 5e (66.9 mg, 0.16 mmol) was obtained as yellow solid in 80% yield. Melting Point = 56 °C – 57 °C.

\(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 7.42 – 7.37 (m, 2H), 7.36 – 7.31 (m, 2H), 7.29 – 7.19 (m, 5H), 7.18 – 7.13 (m, 2H), 7.06 – 7.01 (m, 2H), 4.05 (t, \(J = 7.3\) Hz, 1H), 3.08 (d, \(J = 7.3\) Hz, 2H), 2.28 (s, 3H). \(^{13}\)C NMR (100 MHz, CDCl\(_3\)) \(\delta\) 169.5, 149.5, 138.5, 132.9, 131.4, 129.4, 128.6, 128.1, 126.5, 122.4, 122.0, 121.5, 91.9, 83.4, 44.8, 40.2, 21.1. IR (neat) cm\(^{-1}\) \(\tilde{\nu}\): 3032, 2922, 1751, 1601, 1503, 1483, 1367, 1218, 1164, 1100, 1008, 915, 819, 701, 671, 557, 517, 472. HRMS: m/z (ESI) calculated [M+Na\(^+\)]\(^+\): 441.0466, found: 441.0465. HPLC (Chiralcel AD-H column, hexanes:i-PrOH = 97:3, 0.8 mL/min, 250 nm), \(t_{\text{minor}}\) = 16.5 min, \(t_{\text{major}}\) = 14.3 min, ee = 85%.
\([\alpha]_D^{25}\) = 16.9, (c =0.40, CHCl\(_3\)).

\(\left(R\right)-4-(4\text{-fluorophenyl})-1\text{-phenylbut-3-yn-2-yl})\text{phenyl acetate (5f)}\)

According to general procedure, from 0.2 mmol of alkene, the desired product 5f (55.2 mg, 0.15 mmol) was obtained as colorless oil in 77% yield.
^{1}H NMR (400 MHz, CDCl\textsubscript{3}) \( \delta 7.37 - 7.29 \) (m, 4H), 7.29 - 7.19 (m, 3H), 7.19 - 7.14 (m, 2H), 7.06 - 7.01 (m, 2H), 6.99 - 6.92 (m, 2H), 4.05 (t, \( J = 7.3 \) Hz, 1H), 3.08 (d, \( J = 7.3 \) Hz, 2H), 2.28 (s, 3H). ^{13}C NMR (100 MHz, CDCl\textsubscript{3}) \( \delta 169.5, 162.2 \) (d, \( J = 247.2 \) Hz), 149.4, 138.6 (d, \( J = 10.5 \) Hz), 133.3 (d, \( J = 8.3 \) Hz), 129.4, 128.6, 128.0, 126.5, 121.4, 119.5 (d, \( J = 3.5 \) Hz), 115.5, 115.3, 90.2, 83.3, 44.9, 40.1, 21.1. ^{19}F NMR (376 MHz, CDCl\textsubscript{3}) \( \delta -115.51 - 115.60 \) (m, 1F). IR (neat) cm\textsuperscript{-1} \( \tilde{\nu} \): 3029, 2924, 1755, 1680, 1597, 1502, 1368, 1191, 1160, 1012, 943, 835, 739, 698, 631, 613, 592, 528. HRMS: m/z (ESI) calculated [M+Na]\textsuperscript{+}: 381.1267, found: 381.1248. HPLC (Chiralcel AD-H column, hexanes:i-PrOH = 97:3, 0.8 mL/min, 250 nm), \( t_{\text{minor}} = 12.6 \) min, \( t_{\text{major}} = 11.8 \) min, \( ee = 86\% \).

\([\alpha]_{D}^{25} = 22.3, (c = 0.30, \text{CHCl}_{3}).\)

Methyl (\textit{R})-4-(3-(4-acetoxyphenyl)-4-phenylbut-1-yn-1-yl)benzoate (5\textit{g})

\[
\begin{array}{c}
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\text{O} \\
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\text{O}
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\]

According to general procedure, from 0.2 mmol of alkene, the desired product 5\textit{g} (56.5 mg, 0.14 mmol) was obtained as colorless oil in 71% yield.

^{1}H NMR (400 MHz, CDCl\textsubscript{3}) \( \delta 7.95 \) (d, \( J = 8.4 \) Hz, 2H), 7.41 (d, \( J = 8.4 \) Hz, 2H), 7.38 - 7.32 (m, 2H), 7.31 - 7.21 (m, 3H), 7.20 - 7.14 (m, 2H), 7.08 - 7.03 (m, 2H), 4.10 (t, \( J = 7.3 \) Hz, 1H), 3.89 (s, 3H), 3.10 (d, \( J = 7.3 \) Hz, 2H), 2.29 (s, 3H). ^{13}C NMR (100 MHz, CDCl\textsubscript{3}) \( \delta 169.4, 149.4, 138.6, 138.5, 129.8, 129.4, 128.6, 128.0, 128.0, 126.4, 125.0, 122.4, 121.4, 90.1, 79.5, 44.9, 40.1, 21.0. \) IR (neat) cm\textsuperscript{-1} \( \tilde{\nu} \): 3028, 2924, 1759, 1602, 1502, 1433, 1368, 1273, 1193, 1105, 1046, 910, 855, 768, 729, 696, 559, 527. HRMS: m/z (ESI) calculated [M+Na]\textsuperscript{+}: 421.1416, found: 421.1424. HPLC (Chiralcel AD-H column, hexanes:i-PrOH = 97:3, 0.8 mL/min, 250 nm), \( t_{\text{major}} = 27.7 \) min, \( ee = 86\% \).

\([\alpha]_{D}^{25} = 20.1, (c = 0.22, \text{CHCl}_{3}).\)

(\textit{R})-4-(1-phenyl-4-(thiophen-3-yl)but-3-yn-2-yl)phenyl acetate (5\textit{h})
According to general procedure, from 0.2 mmol of alkene, the desired product 5h (48.5 mg, 0.14 mmol) was obtained as colorless oil in 70% yield.

\[ \text{H NMR (400 MHz, CDCl}_3\text{)} \delta 7.35 - 7.30 (m, 3H), 7.26 - 7.18 (m, 4H), 7.15 (d, J = 6.8 Hz, 2H), 7.06 - 6.98 (m, 3H), 4.04 (t, J = 7.3 Hz, 1H), 3.14 - 3.00 (m, 2H), 2.26 (s, 3H). \]

\[ \text{C NMR (100 MHz, CDCl}_3\text{)} \delta 169.4, 149.4, 138.6, 138.5, 129.8, 129.4, 128.6, 128.0, 128.0, 126.4, 125.0, 122.4, 121.4, 90.1, 79.5, 44.9, 40.1, 21.0. \]

IR (neat) cm\(^{-1}\): 3027, 2922, 1755, 1599, 1501, 1366, 1191, 1164, 1013, 940, 864, 780, 697, 558, 517. HRMS : m/z (ESI) calculated [M+Na]\(^+\): 369.0925, found: 369.0919. HPLC (Chiralcel AD-H column, hexanes:i-PrOH = 97:3, 0.8 mL/min, 250 nm), \(t_{\text{minor}} = 14.4\) min, \(t_{\text{major}} = 13.7\) min, ee = 87%.

\( [\alpha]_D^{25} = 24.1, (c = 0.40, \text{CHCl}_3). \)

(R)-(3-([1,1'-biphenyl]-4-yl)-4-phenylbut-1-yn-1-yl)trimethylsilane (5i)

According to general procedure, from 0.2 mmol of alkene, the desired product 5i (44.6 mg, 0.13 mmol) was obtained as yellow solid in 63% yield. Melting Point = 51 °C – 52 °C.

\[ \text{H NMR (400 MHz, CDCl}_3\text{)} \delta 7.60 (d, J = 7.7 Hz, 2H), 7.54 (d, J = 7.8 Hz, 2H), 7.45 (t, J = 7.6 Hz, 2H), 7.36 (d, J = 7.6 Hz, 3H), 7.28 - 7.22 (m, 3H), 7.16 (d, J = 7.2 Hz, 2H), 3.93 (t, J = 7.2 Hz, 1H), 3.06 (d, J = 7.2 Hz, 2H), 0.18 (s, 9H). \]

\[ \text{C NMR (100 MHz, CDCl}_3\text{)} \delta 140.8, 140.0, 139.7, 138.6, 129.6, 128.7, 128.1, 127.9, 127.2, 127.1, 127.0, 126.4, 107.5, 88.6, 45.0, 40.9, 0.1. \]

IR (neat) cm\(^{-1}\): 3026, 2955, 2167, 1600, 1486, 1451, 1404, 1248, 1057, 835, 760, 728, 693, 568, 496. HRMS : m/z (ESI) calculated [M+Na]\(^+\): 377.1701, found: 377.1689. HPLC (Chiralcel OD-H
column, hexanes:i-PrOH = 99.7:0.3, 0.8 mL/min, 250 nm), t\textsubscript{minor} = 27.5 min, t\textsubscript{major} = 19.7 min, ee = 88%.
\[\alpha\]\textsubscript{D}\textsuperscript{25} = 1.5, (c = 0.16, CHCl\textsubscript{3}).

(3-((R)-3-(4-acetoxyphenyl)-4-phenylbut-1-yn-1-yl)cyclopenta-2,4-dien-1-yl)(cyclopenta-2,4-dien-1-yl)iron (5j)

\[
\begin{align*}
\text{O} & \\
\text{Fe} & \\
\end{align*}
\]

According to general procedure, from 0.2 mmol of alkene, the desired product 5j (73.5 mg, 0.16 mmol) was obtained as orange solid in 82% yield. Melting Point = 87 °C – 88 °C.

\(^1\)H NMR (400 MHz, CDCl\textsubscript{3}) \(\delta\) 7.31 – 7.20 (m, 7H), 7.13 (d, \(J = 7.9\) Hz, 2H), 4.35 (d, \(J = 3.5\) Hz, 2H), 4.14 – 4.09 (m, 7H), 3.96 (dd, \(J = 8.6, 6.1\) Hz, 1H), 3.13 – 2.98 (m, 2H), 2.34 (s, 3H).

\(^1^3\)C NMR (100 MHz, CDCl\textsubscript{3}) \(\delta\) 139.2, 138.7, 136.3, 129.5, 129.1, 128.0, 127.5, 126.3, 87.1, 82.1, 71.2 (d, \(J = 4.6\) Hz), 69.7, 68.2, 66.1, 45.2, 40.4, 21.1. IR (neat) cm\(^{-1}\) \(\tilde{\nu}\): 3028, 2919, 2111, 1602, 1509, 1452, 1409, 1103, 1025, 997, 816, 727, 694, 590, 554, 489, 471. MALDI-TOFMS: m/z calculated [M-H-C\textsubscript{2}H\textsubscript{3}O]\textsuperscript{+}: 404.0858, found: 404.1229. HPLC (Chiralcel AD-H column, hexanes:i-PrOH = 97:3, 0.8 mL/min, 250 nm), t\textsubscript{minor} = 7.7 min, t\textsubscript{major} = 6.6 min, ee = 86%.

\[\alpha\]\textsubscript{D}\textsuperscript{25} = 15.7, (c =0.21, CHCl\textsubscript{3}).

(S)-5-(4-cyclopropyl-1-phenylbut-3-yn-2-yl)-4-methylthiazole (5k)

\[
\begin{align*}
\text{S} & \\
\text{N} & \\
\end{align*}
\]

According to general procedure, from 0.2 mmol of alkene, the desired product 5k (35.3 mg, 0.13 mmol) was obtained as orange oil in 66% yield.

\(^1\)H NMR (400 MHz, CDCl\textsubscript{3}) \(\delta\) 8.58 (s, 1H), 7.30 – 7.22 (m, 3H), 7.07 (d, \(J = 7.2\) Hz, 2H), 4.04 (t, \(J = 7.3\) Hz, 1H), 3.12 (dd, \(J = 13.1, 7.1\) Hz, 1H), 2.94 (dd, \(J = 13.1, 7.5\) Hz, 1H), 2.11 (s, 3H), 1.30 –
1.21 (m, 1H), 0.83 – 0.72 (m, 2H), 0.70 – 0.58 (m, 2H). $^{13}$C NMR (100 MHz, CDCl$_3$) δ 149.8, 148.5, 138.0, 133.0, 129.3, 128.1, 126.7, 86.8, 76.0, 44.9, 32.2, 14.7, 8.0, 0.6. IR (neat) cm$^{-1}$: 3084, 2922, 1696, 1601, 1493, 1450, 1413, 1377, 1312, 1199, 1027, 931, 811, 561, 489.

HRMS: m/z (ESI) calculated [M+H]$^+$: 268.1160, found: 268.1160. HPLC (Chiralcel OD-H column, hexanes:i-PrOH = 99.5:0.5, 0.6 mL/min, 210 nm), $t_{\text{minor}}$ = 18.8 min, $t_{\text{major}}$ = 19.5 min, ee = 89%.

$[\alpha]_D^{25} = -1.7$, (c =0.15, CHCl$_3$).

(R)-4-(2-(4-chlorophenyl)-4-phenylbut-3-yn-1-yl)benzonitrile (5l)

![Chemical structure of 5l](image)

According to general procedure, from 0.2 mmol of alkene, the desired product 5l (53.2 mg, 0.16 mmol) was obtained as colorless oil in 78% yield.

$^1$H NMR (400 MHz, CDCl$_3$) δ 7.55 (d, $J$ = 8.3 Hz, 2H), 7.38 – 7.33 (m, 2H), 7.32 – 7.26 (m, 5H), 7.26 – 7.20 (m, 4H), 4.10 (t, $J$ = 7.0 Hz, 1H), 3.19 – 3.06 (m, 2H). $^{13}$C NMR (100 MHz, CDCl$_3$) δ 143.7, 138.7, 133.0, 131.8, 131.4, 130.3, 128.9, 128.7, 128.3, 128.2, 122.8, 118.9, 110.5, 89.1, 85.3, 44.6, 39.4. IR (neat) cm$^{-1}$: 3058, 2927, 2226, 1689, 1604, 1590, 1488, 1444, 1409, 1286, 1091, 1013, 990, 910, 826, 756, 690, 613, 566, 547. HRMS: m/z (ESI) calculated [M+Na]$^+$: 364.0869, found: 364.0874. HPLC (Chiralcel OD-H column, hexanes:i-PrOH = 99:1, 0.8 mL/min, 250 nm), $t_{\text{minor}}$ = 13.2 min, $t_{\text{major}}$ = 14.0 min, ee = 90%.

$[\alpha]_D^{25} = 29.7$, (c =0.49, CHCl$_3$).

(R)-1-(tert-butyl)-4-(2-(4-chlorophenyl)-4-phenylbut-3-yn-1-yl)benzene (5m)

![Chemical structure of 5m](image)

According to general procedure, from 0.2 mmol of alkene, the desired product 5m (63.2 mg, 0.17 mmol) was obtained as colorless oil in 85% yield.
$^1$H NMR (400 MHz, CDCl$_3$) δ 7.46 – 7.41 (m, 2H), 7.37 – 7.31 (m, 9H), 7.19 – 7.15 (m, 2H), 4.09 (dd, J = 8.1, 6.4 Hz, 1H), 3.20 – 3.03 (m, 2H), 1.37 (s, 9H). $^{13}$C NMR (100 MHz, CDCl$_3$) δ 149.4, 140.0, 135.5, 132.6, 131.6, 129.2, 129.1, 128.6, 128.3, 128.0, 125.1, 123.5, 90.7, 84.6, 44.5, 40.3, 34.5, 31.5. IR (neat) cm$^{-1}$ v: 3025, 2959, 1597, 1511, 1487, 1441, 1407, 1266, 1089, 1014, 910, 825, 754, 689, 570, 506, 477. HRMS: m/z (EI) calculated [M]$^+$: 372.1645, found: 372.1633. HPLC (Chiralcel OD-H column, hexanes:i-PrOH = 100:0, 0.5 mL/min, 254 nm), $t_{\text{minor}}$ = 33.6 min, $t_{\text{major}}$ = 39.4 min, ee = 91%.

$[$α$]_D^{25}$ = 26.3, (c = 0.40, CHCl$_3$).

(R)-1-chloro-4-(1-(4-fluorophenyl)-4-phenylbut-3-yn-2-yl)benzene (5n)

According to general procedure, from 0.2 mmol of alkene, the desired product 5n (60.1 mg, 0.18 mmol) was obtained as colorless oil in 90% yield.

$^1$H NMR (400 MHz, CDCl$_3$) δ 7.45 – 7.38 (m, 2H), 7.36 – 7.25 (m, 7H), 7.11 (dd, J = 8.3, 5.6 Hz, 2H), 6.98 (t, J = 8.6 Hz, 2H), 4.07 (t, J = 7.0 Hz, 1H), 3.15 – 3.04 (m, 2H). $^{13}$C NMR (100 MHz, CDCl$_3$) δ 161.7 (d, J = 242.8 Hz), 139.4, 133.9 (d, J = 3.2 Hz), 132.7, 131.5, 131.0 (d, J = 7.9 Hz), 129.1, 128.5, 128.3, 128.0, 123.2, 114.9 (d, J = 21.0 Hz), 90.0, 84.8, 44.0, 40.1. $^{19}$F NMR (376 MHz, CDCl$_3$) δ -116.36 – -116.48 (m, 1F). IR (neat) cm$^{-1}$ v: 3037, 2925, 1690, 1598, 1507, 1488, 1443, 1408, 1220, 1156, 1091, 1013, 909, 824, 755, 689, 546, 517, 460, 422. HRMS: m/z (EI) calculated [M]$^+$: 334.0925, found: 334.0930. HPLC (Chiralcel OD-H column, hexanes:i-PrOH = 100:0, 0.5 mL/min, 254 nm), $t_{\text{minor}}$ = 52.1 min, $t_{\text{major}}$ = 56.5 min, ee = 92%. $[$α$]_D^{25}$ = 4.6, (c =0.40, CHCl$_3$).

(R)-1-chloro-4-(4-phenyl-1-(p-tolyl)but-3-yn-2-yl)benzene (5o)
According to general procedure, from 0.2 mmol of alkene, the desired product 5o (55.4 mg, 0.17 mmol) was obtained as colorless oil in 84% yield.

$^1$H NMR (400 MHz, CDCl$_3$) δ 7.40 – 7.35 (m, 2H), 7.29 – 7.22 (m, 7H), 7.10 – 7.00 (m, 4H), 4.01 (dd, $J = 7.6, 6.6$ Hz, 1H), 3.10 – 2.95 (m, 2H), 2.31 (s, 3H). $^{13}$C NMR (100 MHz, CDCl$_3$) δ 139.8, 136.0, 135.3, 132.5, 131.5, 129.4, 129.1, 128.8, 128.5, 128.2, 127.9, 123.4, 90.5, 84.4, 44.5, 40.2, 21.1. IR (neat) cm$^{-1}$ $\tilde{\nu}$: 3022, 2920, 1690, 1595, 1487, 1443, 1407, 1318, 1089, 1013, 911, 822, 754, 689, 616, 545, 517, 481. HRMS: m/z (EI) calculated [M]$^+$: 330.1175, found: 330.1173

HPLC (Chiralcel OD-H column, hexanes:i-PrOH = 99.6:0.4, 0.8 mL/min, 250 nm), $t_{\text{minor}} = 10.5$ min, $t_{\text{major}} = 11.5$ min, ee = 89%.

$[\alpha]_{D}^{25} = 9.3$, (c =0.41, CHCl$_3$).

(R)-4,4′-(4-phenylbut-3-yne-1,2-diyl)bis(chlorobenzene) (5p)

According to general procedure, from 0.2 mmol of alkene, the desired product 5p (58.1 mg, 0.17 mmol) was obtained as colorless oil in 83% yield.

$^1$H NMR (400 MHz, CDCl$_3$) δ 7.40 – 7.35 (m, 2H), 7.32 – 7.20 (m, 9H), 7.04 (d, $J = 8.3$ Hz, 2H), 4.04 (t, $J = 7.0$ Hz, 1H), 3.10 – 2.99 (m, 2H). $^{13}$C NMR (100 MHz, CDCl$_3$) δ 139.2, 136.7, 132.4, 131.5, 130.9, 129.1, 128.6, 128.3, 128.2, 128.1, 123.1, 89.8, 84.8, 44.0, 39.9. IR (neat) cm$^{-1}$ $\tilde{\nu}$: 3029, 2924, 1689, 1591, 1488, 1444, 1406, 1284, 1217, 1090, 1013, 823, 805, 754, 689, 543, 513. HRMS: m/z (EI) calculated [M]$^+$: 350.0692, found: 350.0634. HPLC (Chiralcel OD-H column, hexanes:i-PrOH = 100:0, 0.8 mL/min, 254 nm), $t_{\text{minor}} = 38.9$ min, $t_{\text{major}} = 44.9$ min, ee = 88%.

$[\alpha]_{D}^{25} = 10.6$, (c =0.41, CHCl$_3$).

(R)-4-(4-phenyl-1-(4-(trifluoromethyl)phenyl)but-3-yn-2-yl)phenyl acetate (5q)
According to general procedure, from 0.2 mmol of alkene, the desired product 5q (71.8 mg, 0.18 mmol) was obtained as colorless oil in 88% yield.

\[ ^1H \text{NMR (400 MHz, CDCl}_3 \delta 7.56 (d, } J = 8.0 \text{ Hz, 2H), 7.43 – 7.36 (m, 4H), 7.35 – 7.29 (m, 5H), 7.09 (d, } J = 8.6 \text{ Hz, 2H), 4.14 (dd, } J = 7.8, 6.5 \text{ Hz, 1H), 3.25 – 3.11 (m, 2H), 2.33 (s, 3H).} \]
\[ ^13C \text{NMR (100 MHz, CDCl}_3 \delta 169.5, 149.6, 142.6, 138.2, 131.5, 129.8, 128.9 (q, } J = 32.1 \text{ Hz), 128.6, 128.3, 128.1, 125.0 (q, } J = 3.7 \text{ Hz), 124.3 (q, } J = 270.2 \text{ Hz), 123.1, 121.6, 89.8, 84.9, 44.6, 39.8, 21.1.} \]
\[ ^19F \text{NMR (376 MHz, CDCl}_3 \delta -62.23 (s, 3F).} \]
\[ \text{IR (neat) cm}^{-1} \delta: 3051, 2928, 1756, 1616, 1504, 1417, 1369, 1322, 1254, 1162, 1119, 1065, 1016, 908, 847, 822, 754, 730, 692, 595, 558, 525.} \]
\[ \text{HRMS: m/z (ESI) calculated [M+Na]^+: 431.1235, found: 431.1235.} \]
\[ \text{HPLC (Chiralcel AD-H column, hexanes:i-PrOH = 99:1, 0.4 mL/min, 254 nm), } t_{\text{minor}} = 26.4 \text{ min, } t_{\text{major}} = 29.0 \text{ min, ee = 86%.} \]
\[ [\alpha]_{D}^{25} = 23.4, (c =0.49, \text{CHCl}_3). \]

(R)-4-(1-((1,1'-biphenyl)-4-yl)-4-phenylbut-3-yn-2-yl)phenyl acetate (5r)

According to general procedure, from 0.2 mmol of alkene, the desired product 5r (71.6 mg, 0.17 mmol) was obtained as yellow solid in 86% yield. Melting Point = 103 °C – 104 °C.

\[ ^1H \text{NMR (400 MHz, CDCl}_3 \delta 7.65 (d, } J = 7.1 \text{ Hz, 2H), 7.57 (d, } J = 8.2 \text{ Hz, 2H), 7.52 – 7.42 (m, 6H), 7.39 (d, } J = 7.4 \text{ Hz, 1H), 7.36 – 7.28 (m, 5H), 7.11 (d, } J = 8.6 \text{ Hz, 2H), 4.17 (td, } J = 7.2, 4.0 \text{ Hz, 1H), 3.19 (d, } J = 7.2 \text{ Hz, 2H), 2.35 (s, 3H).} \]
\[ ^13C \text{NMR (100 MHz, CDCl}_3 \delta 169.5, 149.5, 140.9, 139.3, 138.8, 137.7, 131.5, 129.9, 128.7, 128.2, 127.9, 127.1, 127.0, 126.7, 123.4, 121.5, 90.6, 84.6, 44.7, 40.2, 21.1.} \]
\[ \text{IR (neat) cm}^{-1} \delta: 3037, 2922, 1756, 1596, 1503, 1487, 1441, 1406, 1371, 1194, 1177, 1014, 941, 905, 851, 822, 756, 689, 664, 595, 561, 522, 481.} \]
\[ \text{HRMS: m/z} \]
(ESI) calculated [M+Na]+: 439.1674, found: 439.1690. HPLC (Chiralcel AD-H column, hexanes:i-PrOH = 99:1, 0.4 mL/min, 254 nm), t_minor = 26.4 min, t_major = 29.0 min, ee = 87%.

[α]D25 = 38.5, (c =0.20, CHCl3).

(R)-3-(2-(4-chlorophenyl)-4-phenylbut-3-yn-1-yl)pyridine (5s)

According to general procedure, from 0.2 mmol of alkene, the desired product 5s (50.9 mg, 0.16 mmol) was obtained as colorless oil in 80% yield.

1H NMR (400 MHz, CDCl3) δ 8.55 – 8.42 (m, 2H), 7.48 – 7.39 (m, 3H), 7.34 – 7.26 (m, 7H), 7.21 (dd, J = 7.7, 4.8 Hz, 1H), 4.12 (t, J = 6.9 Hz, 1H), 3.16 – 3.05 (m, 2H). 13C NMR (100 MHz, CDCl3) δ 150.7, 148.0, 138.8, 136.9, 133.5, 132.9, 131.5, 129.0, 128.6, 128.2, 128.1, 122.9, 122.9, 89.2, 85.2, 41.6, 39.6. IR (neat) cm⁻¹: 3032, 2920, 2852, 1681, 1584, 1483, 1415, 1416, 1272, 1089, 1015, 913, 820, 750, 700, 638, 528. HRMS: m/z (EI) calculated [M]+: 317.0971, found: 317.0979. HPLC (Chiralcel OD-H column, hexanes:i-PrOH = 98:2, 0.7 mL/min, 210 nm), t_minor = 41.7 min, t_major = 60.0 min, ee = 77 %.

[α]D25 = 42.3, (c =0.25, CHCl3).

(R)-1-methoxy-4-(5,5,5-trifluoro-1-phenylpent-1-yn-3-yl)benzene (5t)

According to general procedure, from 0.2 mmol of alkene, the desired product 5t (10.3 mg, 0.03 mmol) was obtained as colorless oil in 17% yield.

1H NMR (400 MHz, CDCl3) δ 7.49 – 7.44 (m, 2H), δ 7.41 – 7.37 (m, 2H), 7.36 – 7.31 (m, 3H), 4.19 (dd, J = 9.1, 5.4 Hz, 1H), 3.84 (s, 3H), 2.79 – 2.46 (m, 2H). 13C NMR (100 MHz, CDCl3) δ 158.9, 131.7, 131.6, 128.4, 128.2, 128.1, 125.6 (q, J = 251 Hz), 123.0, 114.2, 88.8, 83.9, 55.3, 42.3 (q, J = 27.2 Hz), 31.8. 19F NMR (376 MHz, CDCl3) δ -64.29 (t, 3F). HPLC (Chiralcel OD-H column, hexanes:i-PrOH = 100:0, 0.8 mL/min, 210 nm), t_minor = 25.4 min, t_major = 33.2 min, ee = 67 %.
3. Transformations of diarylated propargylic compounds

(S)-4-(1-phenylbut-3-ylnyl)-1,1'-biphenyl (8)

The 5i (40 mg, 0.11 mmol) was added to the solvent MeOH (10 mL), then K$_2$CO$_3$ (47 mg, 0.33 mmol) was added to the solution, the mixture was stirred at room temperature for 24 h. The mixture was concentrated in vacuo, the residue was purified by silica gel column chromatography (PE) to yield the desired product 8 (28.7 mg, 0.10 mmol) as orange solid in 90% yield, Melting Point = 56 °C – 57 °C.

$^1$H NMR (400 MHz, CDCl$_3$) δ 7.58 (d, $J$ = 7.1 Hz, 2H), 7.53 (d, $J$ = 8.3 Hz, 2H), 7.42 (t, $J$ = 7.5 Hz, 2H), 7.36 (d, $J$ = 8.1 Hz, 2H), 7.34 – 7.19 (m, 4H), 7.16 (d, $J$ = 6.5 Hz, 2H), 3.92 (ddd, $J$ = 8.3, 6.6, 2.5 Hz, 1H), 3.14 – 3.01 (m, 2H), 2.28 (d, $J$ = 2.5 Hz, 1H). $^{13}$C NMR (100 MHz, CDCl$_3$) δ 140.7, 139.8, 139.8, 138.5, 129.4, 128.7, 128.1, 128.0, 127.2, 127.1, 127.0, 126.5, 85.2, 72.1, 44.6, 39.5. IR (neat) cm$^{-1}$ ν: 3281, 3027, 2917, 1599, 1487, 1451, 1404, 1331, 1120, 1127, 1074, 909, 838, 762, 724, 692, 657, 559, 486. HRMS: m/z (EI) calculated [M]$^+$: 282.1409, found: 282.1414. HPLC (Chiralcel OD-H column, hexanes:i-PrOH = 100:0, 1.0 mL/min, 250 nm), $t_{\text{minor}}$ = 40.2 min, $t_{\text{major}}$ = 47.2 min, ee = 88%. [α]$_D^{25}$ = -22.3, (c =0.1, CHCl$_3$).

(R)-butane-1,2,4-triyltribenzene (9)

The 4c (40 mg, 0.14 mmol) was added to the solvent MeOH (10 mL), Pd/C (10 mg) was added to the solution, then stirred under an H$_2$ atmosphere (balloon) at room temperature for 24 h. the mixture was filtered through Celite, and the filtrate was concentrated in vacuo to give the corresponding desired product 9 (38.6 mg, 0.14 mmol) as a colorless oil in 95% yield.
$^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.29 – 7.06 (m, 11H), 7.03 (d, $J$ = 7.4 Hz, 2H), 6.97 (d, $J$ = 7.2 Hz, 2H), 2.91 – 2.78 (m, 3H), 2.51 – 2.33 (m, 2H), 2.07 – 1.87 (m, 2H). $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 144.7, 142.3, 140.4, 129.1, 128.3, 128.3, 128.2, 128.0, 127.8, 126.1, 125.8, 125.6, 47.4, 43.8, 37.0, 33.7. IR (neat) cm$^{-1}$ $\tilde{\nu}$: 3025, 2921, 1600, 1493, 1451, 1072, 1028, 907, 753, 732, 695, 614, 590, 545, 510, 487. HRMS: m/z (EI) calculated [M]$^+$: 286.1722, found: 286.1728. HPLC (Chiralcel OD-H column, hexanes:i-PrOH = 100:0, 0.8 mL/min, 254 nm), $t_{\text{minor}}$ = 25.0 min, $t_{\text{major}}$ = 32.5 min, ee = 86%.

$[\alpha]_D^{25} = -7.0$, (c = 0.21, CHCl$_3$).

4. References

5. Spectrogram information

(R)-(3-(p-tolyl)but-1-yn-1,4-diyl)dibenzene (4aa) $^1$H NMR

(R)-(3-(p-tolyl)but-1-yn-1,4-diyl)dibenzene (4aa) $^{13}$C NMR
4aa-HPLC (racemic)

4aa-HPLC (86%)
Methyl (R)-4-(1,4-diphenylbut-3-yn-2-yl)benzoate (4a) $^1$H NMR

Methyl (R)-4-(1,4-diphenylbut-3-yn-2-yl)benzoate (4a) $^{13}$C NMR
4a-HPLC (racemic)

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4a-HPLC (89%)

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Totals: 4610.28484 337.17507
(R)-(3-(4-(trifluoromethyl)phenyl)but-1-yne-1,4-diyl)dibenzene (4b) $^1$H NMR

(4b)

$^1$H NMR spectrum

(R)-(3-(4-(trifluoromethyl)phenyl)but-1-yne-1,4-diyl)dibenzene (4b) $^{13}$C NMR

(4b)

$^{13}$C NMR spectrum
(R)-(3-(4-(trifluoromethyl)phenyl)but-1-yne-1,4-diyl)dibenzene (4b) $^{19}$F NMR

4b-HPLC (racemic)
**4b-HPLC (86%)**

| Peak RetTime Type Width Area Height Area % |
|-----------------------------------------|---------------------------------|---------------------|-------------------|---------------------|-------------------|
| 1 15.230 MF 0.3006 191.07643 10.59255 7.0643 | 2 15.892 FM 0.2923 2513.72266 143.32932 92.9357 |

Totals: 2704.79909 153.92186

(R)-but-3-yne-1,2,4-tribenzene (4c) $^1$H NMR
(R)-but-3-yne-1,2,4-triyltribenzene (4c) $^{13}$C NMR

4c-HPLC (racemic)
4c-HPLC (87%)

(R)-4-(1,4-diphenylbut-3-yn-2-yl)-1,1'-biphenyl (4d) ^1^H NMR
(R)-4-(1,4-diphenylbut-3-yn-2-yl)-1,1'-biphenyl (4d) $^{13}$C NMR

4d-HPLC (racemic)

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Totals: 2.9772e4 1275.89862
4d-HPLC (89%)
(R)-4-(1,4-diphenylbut-3-yn-2-yl)phenyl acetate (4e) $^{13}$C NMR

4e-HPLC (racemic)
4e-HPLC (87%)
(R)-(3-(4-(tert-butyl)phenyl)but-1-yn-1,4-diyl)dibenzene (4f) $^{13}$C NMR

4f-HPLC (racemic)
4f-HPLC (88%)

(R)-(3-(4-bromophenyl)but-1-yne-1,4-diyl)dibenzene (4g) $^1$H NMR
(R)-(3-(4-bromophenyl)but-1-yn-1,4-diyl)dibenzene (4g) \(^{13}\text{C}\) NMR

4g - HPLC (racemic)
4g-HPLC (87%)

(R)-(3-(4-chlorophenyl)but-1-yne-1,4-diyl)dibenzene (4h) $^1$H NMR
(R)-(3-(4-chlorophenyl)but-1-yne-1,4-diyl)dibenzene (4h) $^{13}$C NMR

4h-HPLC (racemic)
4h-HPLC (86%)

(\(\text{R}\)-(3-(4-fluorophenyl)but-1-yn-1,4-diyl)dibenzene (4i) \(\text{\textsuperscript{1}H NMR}\)

\begin{center}
\begin{tabular}{llllll}
\hline
\# & RetTime & Type & Width [min] & Area [mAU's] & Height [mAU] & Area [%] \\
\hline
1 & 18.764 & MF & 0.1929 & 428.86860 & 36.98890 & 7.0937 \\
2 & 11.341 & FM & 0.2121 & 5666.42529 & 448.60681 & 92.9063 \\
\hline
\multicolumn{5}{|l|}{\textbf{Totals}}: & 6034.49390 & 477.59571 \\
\end{tabular}
\end{center}
(R)-(3-(4-fluorophenyl)but-1-yne-1,4-diyl)dibenzene (4i) $^{13}$C NMR

(5R)-(3-(4-fluorophenyl)but-1-yne-1,4-diyl)dibenzene (4i) $^{19}$F NMR
4i-HPLC (racemic)

4i-HPLC (85%)
(R)-(3-(4-(trifluoromethoxy)phenyl)but-1-yne-1,4-diyl)dibenzene (4j) \(^1\)H NMR

\(\text{\[\text{Diagram of molecule}\]}\)

(R)-(3-(4-(trifluoromethoxy)phenyl)but-1-yne-1,4-diyl)dibenzene (4j) \(^13\)C NMR

\(\text{\[\text{Diagram of molecule}\]}\)
(R)-(3-(4-(trifluoromethoxy)phenyl)but-1-yn-1,4-diyl)dibenzene (4j) $^{19}$F NMR

4j-HPLC (racemic)

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Totals: 1.56051e4 454.13455
**4j-HPLC (91%)**

(R)-(3-(4-(difluoromethoxy)phenyl)but-1-yne-1,4-diyl)dibenzene (4k) \(^1\)H NMR
(R)-(3-(4-(difluoromethoxy)phenyl)but-1-yne-1,4-diyl)dibenzene (4k) $^{13}$C NMR

(R)-(3-(4-(difluoromethoxy)phenyl)but-1-yne-1,4-diyl)dibenzene (4k) $^{19}$F NMR
4k-HPLC (racemic)

Peak RetTime Type Width Area Height Area
# [min] [min] [mAU*s] [mAU] %
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1 10.180 MM 0.1863 3720.31201 332.89948 50.3711
2 10.941 MM 0.2175 3665.49878 280.83096 49.6289

Totals : 7385.81079 613.73044

4k-HPLC (86%)

Peak RetTime Type Width Area Height Area
# [min] [min] [mAU*s] [mAU] %
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1 9.969 MM 0.2319 557.17188 48.04671 7.2427
2 10.890 MM 0.2004 7135.66309 593.36810 92.7573

Totals : 7692.83496 633.41481
(R)-(3-(3-bromophenyl)but-1-yne-1,4-diyl)dibenzene (4l) \( ^1\)H NMR

(\(R\))-(3-(3-bromophenyl)but-1-yne-1,4-diyl)dibenzene (4l) \( ^13\)C NMR
**41-HPLC (racemic)**

![HPLC Chromatogram](image)

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Totals :  
4.11947e4 766.11670

**41-HPLC (88%)**

![HPLC Chromatogram](image)

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Totals : 1.32442e4 214.21753
(R)-(3-(3-fluorophenyl)but-1-yne-1,4-diyl)dibenzene (4m) \(^1\)H NMR

(\(R\))-(3-(3-fluorophenyl)but-1-yne-1,4-diyl)dibenzene (4m) \(^{13}\)C NMR
(R)-(3-(3-fluorophenyl)but-1-yne-1,4-diyl)dibenzene (4m) $^1$F NMR

4m-HPLC (racemic)

<table>
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Totals: 2.66882e4 624.35876
4m-HPLC (87%)

(R)-(3-(3,5-bis(trifluoromethyl)phenyl)but-1-yne-1,4-diyl)dibenzene (4n) 1H NMR
(R)-(3-(3,5-bis(trifluoromethyl)phenyl)but-1-yne-1,4-diyl)dibenzene (4n) $^{13}$C NMR

(\textsuperscript{19}F NMR)

(R)-(3-(3,5-bis(trifluoromethyl)phenyl)but-1-yne-1,4-diyl)dibenzene (4n) $^{19}$F NMR
4n-HPLC (racemic)

<table>
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Totals : 8986.45850 354.98964

4n-HPLC (90%)

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Totals : 5160.58112 239.16447
(R)-(3-(4-bromo-3-fluorophenyl)but-1-yne-1,4-diyl)dibenzene (40) $^1$H NMR

(\(R\))-(3-(4-bromo-3-fluorophenyl)but-1-yne-1,4-diyl)dibenzene (40) $^{13}$C NMR
$(R)$-(3-(4-bromo-3-fluorophenyl)but-1-yne-1,4-diyl)dibenzene (4o) $^{19}$F NMR

$^{19}$F NMR spectrum of 4o

$4o$-HPLC (racemic)

HPLC profile with retention times and areas:

<table>
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<tr>
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Totals: 2.31626e4 2161.94556
4o-HPLC (88%)

(S)-2-(1,4-diphenylbut-3-yn-2-yl)thiophene (4p) H NMR
(S)-2-(1,4-diphenylbut-3-yn-2-yl)thiophene (4p) $^{13}$C NMR

4p-HPLC (racemic)
4p-HPLC (89%)

Peak RetTime Type Width Area Height Area %
--- | ------- | ------- | ------- |------- | ------- | ------- |
1  53.913 MM 1.7942 6.93555e4 644.24097 94.7857
2  77.258 BB 1.6133 3815.36938 33.76534 5.2143

(S)- (S)-2-(1,4-diphenylbut-3-yn-2-yl)-3-methylthiophene (4q) $^1$H NMR

![NMR spectrum of (S)- (S)-2-(1,4-diphenylbut-3-yn-2-yl)-3-methylthiophene (4q)]
(S)-2-(1,4-diphenylbut-3-yn-2-yl)-3-methylthiophene (4q) $^{13}$C NMR

4q - HPLC (racemic)
4q - HPLC (97%)

5-(1,4-diphenylbut-3-yn-2-yl)thiazole (4r) $^1$H NMR
(S)-5-(1,4-diphenylbut-3-yn-2-yl)thiazole (4r) $^{13}$C NMR

4r-HPLC (racemic)
4r-HPLC (89%)
(S)-5-(1,4-diphenylbut-3-yn-2-yl)-4-methylthiazole (4s) \(^{13}\)C NMR

4s-HPLC (racemic)

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Totals : 1.58658e4 1009.14142
4s-HPLC (97%)

(S)-4-(3-benzyl-4-(4-methoxyphenyl)but-1-yn-1-yl)-1,1'-biphenyl(4t) $^1$H NMR
(S)-4-(3-benzyl-4-(4-methoxyphenyl)but-1-yn-1-yl)-1,1'-biphenyl(4t) $^{13}$C NMR

4t-HPLC (racemic)
(R)-2-(4-((1,1'-biphenyl)-4-yl)-2-benzylbut-3-yn-1-yl)isoindoline-1,3-dione (4u) H NMR
$(R)$-2-(4-([1,1'-biphenyl]-4-yl)-2-benzylbut-3-yn-1-yl)isoindoline-1,3-dione(4u) $^{13}$C NMR

4u-HPLC (racemic)
4u-HPLC (22%)

(S)-4-(3-cyclohexyl-4-phenylbut-1-yn-1-yl)-1,1'-biphenyl(4v) $^1$H NMR
(S)-4-(3-cyclohexyl-4-phenylbut-1-yn-1-yl)-1,1'-biphenyl(4v) $^{13}$C NMR

4v - HPLC (racemic)
4v - HPLC (22%)

Methyl (S)-6-([1,1'-biphenyl]-4-yl)-4-benzyl-3,3-dimethylhex-5-ynoate(4w) $^1$H NMR
Methyl (S)-6-([1,1'-biphenyl]-4-yl)-4-benzyl-3,3-dimethylhex-5-ynoate(4w) $^{13}$C NMR

4w - HPLC (racemic)

Peak RetTime Type Width Area Height Area %
# [min] [min] [mAU*s] [mAU] | | | |
1 9.752 BV R 0.2454 2671.92676 168.11839 50.5226 |
2 11.546 BB 0.2864 2616.65479 142.41815 49.4774 |

Totals : 5288.58154 310.53654
4w - HPLC (40%)

(R)-(4-[[1,1'-biphenyl]-4-yl]-1-phenylbut-3-yn-2-yl)trimethylsilane (4x) $^1$H NMR
(R)-(4-([1,1'-biphenyl]-4-yl)-1-phenylbut-3-yn-2-yl)trimethylsilane(4x) $^{13}$C NMR

4x - HPLC (racemic)
4x - HPLC (32%)

(S)-1-(tert-butyl)-4-(3-(4-fluorobenzyl)-4,4-dimethylpent-1-yn-1-yl)benzene (4y)

NMR
(S)-1-(tert-butyl)-4-(3-(4-fluorobenzyl)-4,4-dimethylpent-1-yn-1-yl)benzene (4y) $^{13}$C NMR

(S)-1-(tert-butyl)-4-(3-(4-fluorobenzyl)-4,4-dimethylpent-1-yn-1-yl)benzene (4y) $^{19}$F NMR
### 4y - HPLC (racemic)

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**Totals:**

|           | 1.36875e4 | 1381.60590 |

### 4y - HPLC (47%)

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**Totals:**

|           | 8730.20239 | 971.36467 |
4-(((1S,2S)-2-phenylcyclohexyl)ethynyl)-1,1'-biphenyl(4z) \textsuperscript{1}H NMR

4-(((1S,2S)-2-phenylcyclohexyl)ethynyl)-1,1'-biphenyl(4z) \textsuperscript{13}C NMR
**4z - HPLC (racemic)**

![HPLC Racemic Graph]

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- Area: 377.24821

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**4z - HPLC (16%)**

![HPLC 16% Graph]

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- Area: 188.88799
(1S,2R,3R,4R)-2-phenyl-3-(phenylethynyl)bicyclo[2.2.1]heptane (4bb) $^1$H NMR

(1S,2R,3R,4R)-2-phenyl-3-(phenylethynyl)bicyclo[2.2.1]heptane (4bb) $^{13}$C NMR
4bb - HPLC (racemic)

- HPLC (53%)
(R)-but-3-yne-1,1,2,4-tetrayltetraphenylene (4cc) $^1$H NMR

(R)-but-3-yne-1,1,2,4-tetrayltetraphenylene (4cc) $^{13}$C NMR
4cc - HPLC (racemic)

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4cc - HPLC (74%)

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(S)-9-(1-(4-fluorophenyl)-4-phenylbut-3-yn-2-yl)-9H-carbazole (4dd) $^1$H NMR

(S)-9-(1-(4-fluorophenyl)-4-phenylbut-3-yn-2-yl)-9H-carbazole (4dd) $^{13}$C NMR
(S)-9-(1-(4-fluorophenyl)-4-phenylbut-3-yn-2-yl)-9H-carbazole (4dd) $^1$F NMR

4dd - HPLC (racemic)
(8R,9S,13S,14S)-3-((R)-1,4-diphenylbut-3-yn-2-yl)-13-methyl-
6,7,8,9,11,12,13,14,15,16-decahydro-17H-cyclopenta[a]phenanthren-17-one (4ee) ^1H NMR
(8R,9S,13S,14S)-3-((R)-1,4-diphenylbut-3-yn-2-yl)-13-methyl-6,7,8,9,11,12,13,14,15,16-decahydro-17H-cyclopenta[a]phenanthren-17-one (4ee) $^{13}$C NMR

4ee - HPLC (racemic)
4ee - HPLC (80%)
(S)-5-((1,1'-biphenyl)-4-yl)-1-phenylbut-3-yn-2-yl)-4-methylthiazole (5a) 

\(^{13}\)C NMR

5a-HPLC (racemic)

<table>
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Totals: 1.69167e4 563.27542
5a-HPLC (98%)
(S)-5-(4-(4-methoxyphenyl)-1-phenylbut-3-yn-2-yl)-4-methylthiazole (5b) $^{13}$C NMR

5b-HPLC (racemic)
5b-HPLC (97%)

(R)-1-methoxy-4-(4-phenyl-3-(4-(trifluoromethyl)phenyl)but-1-yn-1-yl)benzene (5c)

$^1$H NMR
($R$)-1-methoxy-4-(4-phenyl-3-(4-(trifluoromethyl)phenyl)but-1-yn-1-yl)benzene (5c)

$^{13}$C NMR

![$^{13}$C NMR spectrum](image1)

$^{19}$F NMR

![$^{19}$F NMR spectrum](image2)
5c-HPLC (racemic)

Peak RetTime Type Width Area Height Area
# [min] [min] [mAU*s] [mAU] %
1 9.293 BV 0.1750 2878.46045 183.05428 47.8262
2 9.628 VB 0.1968 2258.67676 174.75003 52.1738

Totals : 4329.13721 357.80431

5c-HPLC(89%)

Peak RetTime Type Width Area Height Area
# [min] [min] [mAU*s] [mAU] %
1 9.280 MF 0.1710 55.69259 5.42655 5.7177
2 9.605 FM 0.1987 918.34875 77.04473 94.2823

Totals : 974.04134 82.47128
(R)-4-(4-(4-(tert-butyl)phenyl)-1-phenylbut-3-yn-2-yl)phenyl acetate (5d) $^1$H NMR

(5d) $^{13}$C NMR
**5d-HPLC (racemic)**

![Graph showing chromatogram for 5d-HPLC (racemic)]

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Totals: 5462.70264 439.48892

**5d-HPLC (90%)**

![Graph showing chromatogram for 5d-HPLC (90%)]

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Totals: 2638.30896 232.52743
(R)-4-(4-(4-bromophenyl)-1-phenylbut-3-yn-2-yl)phenyl acetate (5e) $^1$H NMR

(R)-4-(4-(4-bromophenyl)-1-phenylbut-3-yn-2-yl)phenyl acetate (5e) $^{13}$C NMR
5e-HPLC (racemic)

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Totals: 2581.24109 147.04922

5e-HPLC (85%)

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Totals: 2594.89774 161.35341
(R)-4-(4-(4-fluorophenyl)-1-phenylbut-3-yn-2-yl)phenyl acetate (5f) $^1$H NMR

(R)-4-(4-(4-fluorophenyl)-1-phenylbut-3-yn-2-yl)phenyl acetate (5f) $^{13}$C NMR
(R)-4-(4-(4-fluorophenyl)-1-phenylbut-3-yn-2-yl)phenyl acetate (5f) $^{19}$F NMR

5f-HPLC (racemic)

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Totals: 7027.12036 524.93565
Methyl (R)-4-(3-(4-acetoxyphenyl)-4-phenylbut-1-yn-1-yl)benzoate (5g) $^1$H NMR
Methyl (R)-4-(3-(4-acetoxyphenyl)-4-phenylbut-1-yn-1-yl)benzoate (5g) $^{13}$C NMR

5g-HPLC (racemic)

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<td>65.94847</td>
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Totals: 4712.02271 139.02829
5g-HPLC (86%)

(R)-4-(1-phenyl-4-(thiophen-3-yl)but-3-yn-2-yl)phenyl acetate (5h) \(^{1}\text{H} \text{NMR} \)
(R)-4-(1-phenyl-4-(thiophen-3-yl)but-3-yn-2-yl)phenyl acetate (5h) $^{13}$C NMR

5h-HPLC (racemic)
5h-HPLC (87%)

(R)-(3-[(1,1’-biphenyl]-4-yl)-4-phenylbut-1-yn-1-yl)trimethylsilane (5i) $^1$H NMR
$(R)$-$(3-[[1,1’\text{-biphenyl}]-4-yl)-4-phenylbut-1-yn-1-yl]trimethylsilane (5i) $^{13}$C NMR

5i-HPLC (racemic)

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Totals: 3749.76624 102.05680
5i-HPLC (88%)

(3-((R)-3-(4-acetoxyphenyl)-4-phenylbut-1-yn-1-yl)cyclopenta-2,4-dien-1-yl)(cyclopenta-2,4-dien-1-yl)iron (5j) $^1$H NMR
(3-((R)-3-(4-acetoxyphenyl)-4-phenylbut-1-yn-1-yl)cyclopenta-2,4-dien-1-yl)(cyclopenta-2,4-dien-1-yl)iron (5j) $^{13}$C NMR

$^{5j}$-HPLC (racemic)

<table>
<thead>
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<th>Area [mAU's]</th>
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<td>2 7.748 BB</td>
<td>0.1312</td>
<td>142.39235</td>
<td>16.57516</td>
<td>50.4764</td>
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Totals : 282.89702 35.91291
**5j-HPLC (86%)**

(S)-5-(4-cyclopropyl-1-phenylbut-3-yn-2-yl)-4-methylthiazole (5k) $^1$H NMR
(S)-5-(4-cyclopropyl-1-phenylbut-3-yn-2-yl)-4-methylthiazole (5k) $^{13}$C NMR

5k-HPLC (racemic)
5k-HPLC (89%)

(R)-4-(2-(4-chlorophenyl)-4-phenylbut-3-yn-1-yl)benzonitrile (5l) \(^1\)H NMR
$(R)$-4-(2-(4-chlorophenyl)-4-phenylbut-3-yn-1-yl)benzonitrile (5l) $^{13}$C NMR

5l-HPLC (racemic)

<table>
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Total: 1.30524e4 705.75870
HPLC (90%)

(R)-1-(tert-butyl)-4-(2-(4-chlorophenyl)-4-phenylbut-3-yn-1-yl)benzene (5m)

¹H NMR
(R)-1-(tert-butyl)-4-(2-(4-chlorophenyl)-4-phenylbut-3-yn-1-yl)benzene (5m)

$^{13}$C NMR

5m-HPLC (racemic)
**5m-HPLC (91%)**

![HPLC chromatogram](image)

<table>
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<tr>
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**Totals:** 1.7727e4   306.64734

**(R)-1-chloro-4-(1-(4-fluorophenyl)-4-phenylbut-3-yn-2-yl)benzene (5n)** ¹H NMR

![NMR spectrum](image)
(R)-1-chloro-4-(1-(4-fluorophenyl)-4-phenylbut-3-yn-2-yl)benzene (5n) $^{13}$C NMR

(R)-1-chloro-4-(1-(4-fluorophenyl)-4-phenylbut-3-yn-2-yl)benzene (5n) $^{19}$F NMR
(R)-1-chloro-4-(4-phenyl-1-(p-tolyl)but-3-yn-2-yl)benzene (5o) $^1$H NMR

(R)-1-chloro-4-(4-phenyl-1-(p-tolyl)but-3-yn-2-yl)benzene (5o) $^{13}$C NMR
50-HPLC (racemic)

```
Peak RetTime Type Width  Area  Height  Area  %
#  [min]  [min]  [mAU*s]  [mAU]  %
1 9.721 BV  0.1972  5335.59668  417.19208  47.9889
2 10.495 MM  0.3025  5881.36523  319.65601  52.0111

Totals :    1.1137e4  736.84808
```

50-HPLC (89%)

```
Peak RetTime Type Width  Area  Height  Area  %
#  [min]  [min]  [mAU*s]  [mAU]  %
1 10.506 MF  0.2655  61.93018  3.88757  5.5172
2 11.521 FM  0.3797  1060.55469  46.55320  94.4828

Totals :    1122.48487  50.44077
```
(R)-4,4'-((4-phenylbut-3-yn-1,2-diyl)bis(chlorobenzene) (5p) $^1$H NMR

(R)-4,4'-((4-phenylbut-3-yn-1,2-diyl)bis(chlorobenzene) (5p) $^{13}$C NMR
**5p-HPLC (racemic)**

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**Totals:**

- 4.13643e4
- 472.41519

**5p-HPLC (88%)**

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**Totals:**

- 1.57638e4
- 183.90742
$(R)$-4-(4-phenyl-1-(4-(trifluoromethyl)phenyl)but-3-yn-2-yl)phenyl acetate (5q)

$^1$H NMR

$^{13}$C NMR
**(R)-4-(4-phenyl-1-(4-(trifluoromethyl)phenyl)but-3-yn-2-yl)phenyl acetate (5q)**

$^{19}$F NMR

**5q-HPLC (racemic)**
5q-HPLC (86%)

(R)-4-((1,1'-biphenyl)-4-y1)-4-phenylbut-3-yn-2-yl)phenyl acetate (5r) $^1$H NMR
(R)-4-(1-[(1,1'-biphenyl]-4-yl)-4-phenylbut-3-yn-2-yl)phenyl acetate (5r) $^{13}$C NMR

5r-HPLC (racemic)
5r-HPLC (87%)

(R)-3-(2-(4-chlorophenyl)-4-phenylbut-3-yn-1-yl)pyridine (5s) ¹H NMR
(R)-3-(2-(4-chlorophenyl)-4-phenylbut-3-yn-1-yl)pyridine (5s) $^{13}$C NMR

5s - HPLC (racemic)
5s - HPLC (77%)

(R)-1-methoxy-4-(5,5,5-trifluoro-1-phenylpent-1-yn-3-yl)benzene (5t) $^1$H NMR
(R)-1-methoxy-4-(5,5,5-trifluoro-1-phenylpent-1-yn-3-yl)benzene (5t) $^{13}$C NMR

(R)-1-methoxy-4-(5,5,5-trifluoro-1-phenylpent-1-yn-3-yl)benzene (5t) $^{19}$F NMR
5t-HPLC (racemic)

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5t-HPLC (67%)

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(S)-4-(1-phenylbut-3-yn-2-yl)-1,1'-biphenyl (8) $^1$H NMR

(8)

(S)-4-(1-phenylbut-3-yn-2-yl)-1,1'-biphenyl (8) $^{13}$C NMR

(8)
8-HPLC (racemic)

<table>
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<th>Area</th>
<th>Height</th>
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8-HPLC (88%)

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(R)-butane-1,2,4-triyltribenzene (9) $^1$H NMR

(9)-butane-1,2,4-triyltribenzene (9) $^{13}$C NMR
**9-HPLC (racemic)**

![Graph and Table]

**9-HPLC (86%)**

![Graph and Table]
6. The NOESY, H-H COSY and $^1$H Spectra
7. Single Crystal X-Ray Diffraction Data (4f)

A crystal structure of the compound 4f was obtained by recrystallization from PE/DCM.

Table S5. Crystal data and structure refinement for mo_d8v19247_0m.

<table>
<thead>
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<th>Identification code</th>
<th>mo_d8v19247_0m</th>
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<tbody>
<tr>
<td>Empirical formula</td>
<td>C26 H26</td>
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<tr>
<td>Formula weight</td>
<td>338.47</td>
</tr>
<tr>
<td>Temperature</td>
<td>293(2) K</td>
</tr>
<tr>
<td>Wavelength</td>
<td>0.71073 Å</td>
</tr>
<tr>
<td>Crystal system</td>
<td>Monoclinic</td>
</tr>
<tr>
<td>Space group</td>
<td>P 21</td>
</tr>
<tr>
<td>Unit cell dimensions</td>
<td>a = 9.423(7) Å</td>
</tr>
<tr>
<td></td>
<td>b = 10.032(6) Å</td>
</tr>
<tr>
<td></td>
<td>c = 11.391(7) Å</td>
</tr>
<tr>
<td>Volume</td>
<td>1042.1(12) Å³</td>
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<tr>
<td>Z</td>
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<tr>
<td>Density (calculated)</td>
<td>1.079 Mg/m³</td>
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<tr>
<td>Absorption coefficient</td>
<td>0.061 mm⁻¹</td>
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<tr>
<td>F(000)</td>
<td>364</td>
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<tr>
<td>Crystal size</td>
<td>0.200 x 0.150 x 0.100 mm³</td>
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<tr>
<td>Theta range for data collection</td>
<td>2.514 to 24.996°.</td>
</tr>
<tr>
<td>Index ranges</td>
<td>-11&lt;=h&lt;=11, -11&lt;=k&lt;=11, -13&lt;=l&lt;=13</td>
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<tr>
<td>Reflections collected</td>
<td>9901</td>
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<tr>
<td>Independent reflections</td>
<td>3551 [R(int) = 0.0520]</td>
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<tr>
<td>Completeness to theta</td>
<td>25.242°</td>
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<tr>
<td>Absorption correction</td>
<td>Semi-empirical from equivalents</td>
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<tr>
<td>Max. and min. transmission</td>
<td>0.7456 and 0.5795</td>
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<tr>
<td>Description</td>
<td>Value</td>
</tr>
<tr>
<td>-------------------------------------</td>
<td>------------------------</td>
</tr>
<tr>
<td>Refinement method</td>
<td>Full-matrix least-squares on $F^2$</td>
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<tr>
<td>Data / restraints / parameters</td>
<td>3551 / 55 / 269</td>
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<tr>
<td>Goodness-of-fit on $F^2$</td>
<td>1.046</td>
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<tr>
<td>Final R indices [I&gt;2σ(I)]</td>
<td>R1 = 0.0628, wR2 = 0.1624</td>
</tr>
<tr>
<td>R indices (all data)</td>
<td>R1 = 0.1028, wR2 = 0.1939</td>
</tr>
<tr>
<td>Absolute structure parameter</td>
<td>2.3(10)</td>
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<tr>
<td>Largest diff. peak and hole</td>
<td>0.208 and -0.155 e Å$^{-3}$</td>
</tr>
</tbody>
</table>
8. EPR study

General Procedure for the ERP Analysis. After 1 h, the solution sample was taken out into a small tube and then analyzed by EPR. EPR spectra was recorded at room temperature on a EPR spectrometer operated at 9.870 GHz. Typical spectrometer parameters are shown as follows, sweep width: 3000 G; center field sets: 3500 G; time constant: 163.84 ms; sweep time: 41.943 s; modulation amplitude: 4.0 G; modulation frequency: 100 kHz; receiver gain: 7.1 × 10^4; microwave power: 2.016 mW.

The EPR spectrum of control experiment showed the existence of carbon radical and Cu^{II}.

Figure 1. EPR spectrum of the reaction

The EPR spectrum of control experiment showed the existence of carbon radical and Cu^{II}. 

\[
g = 2.00583 \\
A_H = 14.3776 \\
A_H = 21.1275 
\]
9. Cyclic Voltammetry Analysis

Preparation of copper(I) phenylacetylide: according to reference procedure (Angew. Chem. Int. Ed. 2015, 54, 13896 –13901) CuI (1.0 g, 5.0 mmol) was dissolved in ammonium hydroxide to form a blue solution. While stirring, this solution was added drop wise to the solution of phenylacetylene (0.5g, 5.1 mmol) in 50 mL of ethanol. The system was allowed to stand for 15 min to form a yellow precipitate suspension. The precipitate was filtered out and washed with water, ethanol, and diethyl ether, three times each. The solid was vacuum-dried, a bright yellow solid was obtained.

Cyclic voltammogram of Cu(I)-phenylacetylide measured in acetonitrile, scan rate 0.1 V/s, electrolyte: 0.1 M [TBA][PF₆]; auxiliary electrode: a 20 × 20 mm² platinum thin film; working electrode: glassycarbon; reference electrode: Ag/AgCl. CV data collected in a dry-N₂ atmosphere using three electrode system measured by Zennium Electrochemical Workstation.