Supporting Information

An Aqueous Medium-Controlled Stereospecific Oxidative Iodination of Alkynes: An Efficient Access to (E)-Diiodoalkene Derivatives

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1. General Information

All chemicals used were reagent grade and used as received without further purification. \(^1\)H NMR spectra were recorded at 300 or 500 MHz and Proton-decoupled \(^1\)\(^3\)C NMR spectra were obtained at 75 or 125 MHz in CDCl\(_3\). The chemical shifts (\(\delta\)) are reported in ppm units relative to TMS as an internal standard for \(^1\)H NMR and CDCl\(_3\) for \(^1\)\(^3\)C NMR spectra. Coupling constants (\(J\)) are reported in hertz (Hz) and multiplicities are indicated as follows: s (singlet), br s (broad singlet), d (doublet), dd (doublet of doublet), m (multiplet). TLC inspections were performed on Silica gel 60 F\(_{254}\) plates. Column chromatography was performed on silica gel (100-200 mesh) using \(n\)-hexane-EtOAc as eluent.

2. Reaction Optimization

Table S1. Optimization of reaction conditions.\(^a\),\(^b\)

\[
\begin{array}{cccc}
\text{Entry} & \text{Solvent} & \text{Time (min)} & \text{NH}_4\text{I (equiv.)} & \text{Oxidant (equiv.)} & \text{Yield (%)} \\
1 & H_2O & 20 & 2 & \text{oxone (1)} & 88 \\
2 & H_2O & 20 & 3 & \text{oxone (1)} & 97 \\
3 & H_2O & 10 & 3 & \text{oxone (1)} & 61 \\
4 & H_2O & 20 & 3 & \text{oxone (1.5)} & 97 \\
5 & H_2O & 20 & 3 & \text{oxone (0.75)} & 69 \\
6 & H_2O & 20 & 4 & \text{oxone (1)} & 97 \\
7 & H_2O & 20 & 3 & K_2S_2O_8 (1) & 30 (93)\(^c\) \\
8 & H_2O & 20 & 3 & mCPBA (1) & 13 (32)\(^d\) \\
9 & H_2O & 20 & 3 & aq. H_2O_2 (1) & 00 (04)\(^d\) \\
10 & H_2O & 20 & 3 & aq. TBHP (1) & 00 (40)\(^d\) \\
\end{array}
\]

\(^a\)Reaction conditions: phenyl acetylene (1a) (1 mmol), NH\(_4\)I and oxidant in solvent (5 mL), rt. \(^b\) Isolated yields after column chromatography purification. \(^c\) The values shown in paranthesis refers to the yield of the reaction performed for 1 h. \(^d\) The values shown in paranthesis refers to the yield of the reaction performed for 24 h.
3. General Procedure

General procedure for the stereo selective vicinal diiodination of alkynes using NH$_4$I and oxone in aqueous media

\[
\begin{align*}
\text{PhCHCH}_2 & \quad \xrightarrow{\text{NH}_4\text{I (3 equiv.)}} \quad \text{PhCHCH}_2\text{I} \\
\text{H}_2\text{O (5 mL)} & \quad \xrightarrow{\text{oxone (1 equiv.)}} \quad \text{PhCHCH}_2\text{I} \\
& \quad \xrightarrow{\text{RT}} \quad \text{PhCHCH}_2\text{I} \\
\end{align*}
\]

In an oven dried double necked round bottom flask equipped with a magnetic stirring bar, alkyne 1 (1 mmol, 1 equiv.), NH$_4$I (3 mmol, 3 equiv.) and water (5 mL) were taken at room temperature and stirred for some time. Then, oxone (1 mmol, 1 equiv.) was added slowly and the resulting solution was allowed to stir at room temperature for the time indicated in the tables. After completion of the reaction, as indicated by the TLC, the reaction was quenched with aqueous Na$_2$S$_2$O$_3$ and stirred vigorously for few minutes. The reaction mixture was extracted with DCM (15 x 3 mL) and the organic phase was washed with water (2 x 5 mL) and dried over anhydrous Na$_2$SO$_4$. After evaporation of the solvent under reduced pressure, the crude reaction mixture was purified by column chromatography (silica gel, hexanes or ethyl acetate/hexanes mixture) to give the corresponding \textit{trans}-diiodoalkene 2.
4. Spectroscopic data of all compounds:

$(E)$-(1,2-diiodovinyl)benzene (2a)

![Diagram of 2a]

The compound 2a was prepared following general procedure and purified by column chromatography using hexanes as an eluent.

$^1$H NMR (400 MHz, CDCl$_3$): $\delta$ (ppm) = 7.37-7.30 (m, 5H, Ar-H), 7.25 (s, 1H, vinylic-H); $^{13}$C NMR (100 MHz, CDCl$_3$): $\delta$ (ppm) = 143.03, 128.92, 128.45, 128.38, 96.13, 80.75.

$(E)$-1-(1,2-diiodovinyl)-2-methylbenzene (2b)

![Diagram of 2b]

The compound 2b was prepared following general procedure and purified by column chromatography using hexanes as an eluent.

Yellow solid; mp = 48-50 °C; IR (KBr): $\nu_{\text{max}}$ 3060, 2933, 1712, 1598, 1450, 1371, 1233, 1147, 1100, 1030, 845, 779, 749, 717, 655, and 593 cm$^{-1}$; $^1$H NMR (400 MHz, CDCl$_3$): $\delta$ (ppm) = 7.25 (s, 1H, vinylic-H), 7.24-7.23 (m, 1H, Ar-H), 7.22-7.17 (m, 2H, Ar-H), 7.10-7.08 (m, 1H, Ar-H), 2.23 (s, 3H, Ar-CH$_3$); $^{13}$C NMR (100 MHz, CDCl$_3$): $\delta$ (ppm) = 142.83, 134.43, 130.52, 129.00, 127.54, 126.22, 96.18, 82.83, 19.44; HR-MS (EI) m/z calcd for C$_9$H$_8$I$_2$: 369.86800; Found 369.86645; Anal. Calcd for C$_9$H$_8$I$_2$: C, 29.22; H, 2.18. Found: C, 29.59; H, 2.51.

$(E)$-1-(1,2-diiodovinyl)-3-methylbenzene (2c)

![Diagram of 2c]

The compound 2c was prepared following general procedure and purified by column chromatography using hexanes as an eluent.
\(^1\)H NMR (500 MHz, CDCl\(_3\)): \(\delta\) (ppm) = 7.26-7.23 (m, 1H, Ar-\(H\)), 7.22 (s, 1H, vinylic-\(H\)), 7.16-7.12 (m, 3H, Ar-\(H\)), 2.36 (s, 3H, Ar-CH\(_3\)); \(^1\)C NMR (100 MHz, CDCl\(_3\)): \(\delta\) (ppm) = 142.91, 138.12, 129.70, 128.92, 128.23, 125.45, 96.40, 80.45, 21.34.

\((E)-(1,2\text{-diiodovinyl})-4\text{-methylbenzene (2d)}\)

\[
\begin{array}{c}
\text{2d} \\
\end{array}
\]

The compound 2d was prepared following general procedure and purified by column chromatography using hexanes as an eluent.

\(^1\)H NMR (400 MHz, CDCl\(_3\)): \(\delta\) (ppm) = 7.26 (d, \(J = 8.19\) Hz, 2H, Ar-\(H\)), 7.21 (s, 1H, vinylic-\(H\)), 7.15 (d, \(J = 7.94\) Hz, 2H, Ar-\(H\)), 2.35 (s, 3H, Ar-CH\(_3\)); \(^1\)C NMR (100 MHz, CDCl\(_3\)): \(\delta\) (ppm) = 140.10, 139.01, 129.03, 128.42, 96.57, 80.19, 21.41.

\((E)-(1,2\text{-diiodovinyl})-4\text{-pentylbenzene (2e)}\)

\[
\begin{array}{c}
\text{2e} \\
\end{array}
\]

The compound 2e was prepared following general procedure and purified by column chromatography using hexanes as an eluent.

\(^1\)H NMR (500 MHz, CDCl\(_3\)): \(\delta\) (ppm) = 7.28 (d, \(J = 8.24\) Hz, 2H, Ar-\(H\)), 7.21 (s, 1H, vinylic-\(H\)), 7.15 (d, \(J = 8.24\) Hz, 2H, Ar-\(H\)), 2.60 (t, \(J = 7.93\) Hz, 1 x 2H, benzylic-\(H\)), 1.66-1.59 (m, 1 x 2H, aliphatic-\(H\)), 1.36-1.31 (m, 2 x 2H, aliphatic-\(H\)), 0.90 (t, \(J = 7.17\) Hz, 1 x 3H, aliphatic-\(H\)); \(^1\)C NMR (100 MHz, CDCl\(_3\)): \(\delta\) (ppm) = 144.02, 140.12, 128.47, 128.30, 96.73, 80.03, 35.78, 31.53, 30.78, 22.50, 14.02.

\((E)-(1\text{-}t\text{ert-buty}l)-4\text{-}(1,2\text{-diiodovinyl})\text{benzene (2f)}\)

\[
\begin{array}{c}
\text{2f} \\
\end{array}
\]

The compound 2f was prepared following general procedure and purified by column chromatography using hexanes as an eluent.
IR (Neat): $\nu_{\text{max}}$ 3065, 2961, 2903, 2867, 1607, 1499, 1398, 1363, 1267, 1198, 1156, 1106, 1019, 856, 838, 820, 777, 705 and 600 cm$^{-1}$; $^1$H NMR (400 MHz, CDCl$_3$): $\delta$ (ppm) = 7.37-7.34 (m, 2H, Ar-H), 7.33-7.29 (m, 2H, Ar-H), 7.22 (s, 1H, vinylic-H), 1.33 (s, 9H, Ar-C(CH$_3$)$_3$); $^{13}$C NMR (100 MHz, CDCl$_3$): $\delta$ (ppm) = 152.04, 139.73, 128.34, 125.21, 96.77, 79.86, 34.76, 31.19; HR-MS (EI) m/z calcd for C$_{12}$H$_{14}$I$_2$, 411.92000; Found 411.91849; Anal. Caled for C$_{12}$H$_{14}$I$_2$: C, 34.98; H, 3.42. Found: C, 34.60; H, 3.37.

$^{(E)}$-1-(1,2-diiodovinyl)-2-methoxybenzene (2g)

The compound 2g was prepared following general procedure and purified by column chromatography using EtOAc/hexanes (0:10 to 0.1:9.9) as an eluent.

IR (Neat): $\nu_{\text{max}}$ 3064, 2904, 2833, 1582, 1482, 1459, 1434, 1288, 1251, 1155, 1108, 1023, 849, 785, 751, 660 and 601 cm$^{-1}$; $^1$H NMR (400 MHz, CDCl$_3$): $\delta$ (ppm) = 7.34 (t, $J$ = 8.80 Hz, 1H, Ar-H), 7.25 (s, 1H, vinylic-H), 7.14 (d, $J$ = 7.58 Hz, 1H, Ar-H), 6.97 (t, $J$ = 7.45 Hz, 1H, Ar-H), 6.90 (d, $J$ = 8.31 Hz, 1H, Ar-H), 3.90 (s, 3H, OC$_3$H$_3$); $^{13}$C NMR (100 MHz, CDCl$_3$): $\delta$ (ppm) = 155.17, 131.94, 130.47, 129.34, 120.54, 111.54, 92.53, 83.13, 55.66; HR-MS (EI) m/z calcd for C$_9$H$_8$I$_2$O, 385.86600; Found 385.86645; Anal. Caled for C$_9$H$_8$I$_2$O: C, 28.01; H, 2.09. Found: C, 28.26; H, 2.10.

$^{(E)}$-1-(1,2-diiodovinyl)-4-methoxybenzene (2h)

The compound 2h was prepared following general procedure and purified by column chromatography using EtOAc/hexanes (0:10 to 0.1:9.9) as an eluent.

$^1$H NMR (400 MHz, CDCl$_3$): $\delta$ (ppm) = 7.33 (d, $J$ = 8.80 Hz, 2H, Ar-H), 7.19 (s, 1H, vinylic-H), 6.87 (d, $J$ = 8.92 Hz, 2H, Ar-H), 3.83 (s, 3H, OCH$_3$); $^{13}$C NMR (100 MHz, CDCl$_3$): $\delta$ (ppm) = 159.75, 135.22, 130.19, 113.63, 96.53, 79.82, 55.31.

$^{(Z)}$-1-(1,2-diiodovinyl)-4-methoxybenzene (2h')
The compound $2h'$ was prepared following general procedure and purified by column chromatography using EtOAc/hexanes (0:10 to 0.1:9.9) as an eluent.

IR (Neat): $\nu_{\text{max}}$ 3027, 2955, 2834, 1601, 1501, 1457, 1294, 1252, 1176, 1030, 865, 831, 771, 679, and 542 cm$^{-1}$; $^{1}$H NMR (400 MHz, CDCl$_3$): $\delta$ (ppm) = 7.43 (s, 1H, vinylic-$H$), 7.39 (d, $J = 8.68$ Hz, 2H, Ar-$H$), 6.81 (d, $J = 8.68$ Hz, 2H, Ar-$H$), 3.81 (s, 3H, OCH$_3$); $^{13}$C NMR (100 MHz, CDCl$_3$): $\delta$ (ppm) = 160.08, 136.40, 129.72, 119.63, 113.63, 90.59, 55.40; HR-MS (EI) m/z calcd for C$_9$H$_8$I$_2$O, 385.86611; Found 385.86645; Anal. Calcd for C$_9$H$_8$I$_2$O: C, 28.01; H, 2.09. Found: C, 28.23; H, 2.15.

($E$)-1-(1,2-diiodovinyl)-4-phenoxybenzene ($2i$)

The compound $2i$ was prepared following general procedure and purified by column chromatography using EtOAc/hexanes (0:10 to 0.1:9.9) as an eluent.

Pale yellow solid; mp = 56-58 °C; IR (KBr): $\nu_{\text{max}}$ 3060, 1610, 1582, 1486, 1280, 1244, 1197, 1148, 1100, 1063, 1018, 869, 823, 778, 744, 687, and 593 cm$^{-1}$; $^{1}$H NMR (400 MHz, CDCl$_3$): $\delta$ (ppm) = 7.39-7.32 (m, 4H, Ar-$H$), 7.23 (s, 1H, vinylic-$H$), 7.15 (t, $J = 7.45$ Hz, 1H, Ar-$H$), 7.06 (d, $J = 7.82$ Hz, 2H, Ar-$H$), 6.94 (d, $J = 8.68$ Hz, 2H, Ar-$H$); $^{13}$C NMR (100 MHz, CDCl$_3$): $\delta$ (ppm) = 157.92, 156.03, 137.29, 130.31, 129.87, 124.02, 119.72, 117.70, 95.87, 80.46; HR-MS (EI) m/z calcd for C$_{14}$H$_{10}$I$_2$O, 447.88100; Found 447.88210; Anal. Calcd for C$_{14}$H$_{10}$I$_2$O: C, 37.53; H, 2.25. Found: C, 38.29; H, 2.25.

($E$)-1-(1,2-diiodovinyl)-2-fluorobenzene ($2j$)
The compound 2j was prepared following general procedure and purified by column chromatography using hexanes as an eluent.

IR (Neat): $\nu_{\text{max}}$ 3064, 2922, 1616, 1577, 1481, 1448, 1269, 1242, 1221, 1151, 1098, 862, 796, 755, 656, and 605 cm$^{-1}$; $^1$H NMR (500 MHz, CDCl$_3$): $\delta$ (ppm) = 7.40 (s, 1H, vinylic-$H$), 7.39-7.34 (m, 1H, Ar-$H$), 7.22 (ddd, $J = 1.83$, 7.47, 14.64 Hz, 1H, Ar-$H$), 7.17 (ddd, $J = 1.06$, 7.32, 14.95 Hz, 1H, Ar-$H$), 7.09-7.05 (m, 1H, Ar-$H$); $^{13}$C NMR (100 MHz, CDCl$_3$): $\delta$ (ppm) = 130.94 (d, $J = 8.06$ Hz), 129.97, 124.25 (d, $J = 3.66$ Hz), 116.28 (d, $J = 20.54$ Hz), 87.88, 84.91; HR-MS (EI) m/z calcd for C$_8$H$_5$FI$_2$, 373.8450; Found 373.84646; Anal. Calcd for C$_8$H$_5$FI$_2$: C, 25.70; H, 1.35. Found: C, 25.65; H, 1.31.

(E)-1-(1,2-diiodovinyl)-3-fluorobenzene (2k)

![2k](image)

The compound 2k was prepared following general procedure and purified by column chromatography using hexanes as an eluent.

IR (Neat): $\nu_{\text{max}}$ 3064, 2924, 1609, 1582, 1477, 1429, 1263, 1205, 1157, 1120, 1073, 941, 874, 789, 771, 694, and 611 cm$^{-1}$; $^1$H NMR (500 MHz, CDCl$_3$): $\delta$ (ppm) = 7.37-7.32 (m, 1H, Ar-$H$), 7.30 (s, 1H, vinylic-$H$), 7.15-7.12 (m, 1H, Ar-$H$), 7.07-7.01 (m, 2H, Ar-$H$); $^{13}$C NMR (100 MHz, CDCl$_3$): $\delta$ (ppm) = 162.16 (d, $J = 247.94$ Hz), 145.00 (d, $J = 8.06$ Hz), 130.07 (d, $J = 8.06$ Hz), 124.23 (d, $J = 2.20$ Hz), 116.00 (d, $J = 21.27$ Hz), 115.61 (d, $J = 22.74$ Hz), 93.94, 81.86; HR-MS (EI) m/z calcd for C$_8$H$_5$FI$_2$, 373.84450; Found 373.84646; Anal. Calcd for C$_8$H$_5$FI$_2$: C, 25.70; H, 1.35. Found: C, 24.66; H, 1.45.

(E)-1-(1,2-diiodovinyl)-4-fluorobenzene (2l)

![2l](image)

The compound 2l was prepared following general procedure and purified by column chromatography using hexanes as an eluent.
$^1$H NMR (400 MHz, CDCl$_3$): δ (ppm) = 7.38-7.32 (m, 2H, Ar-H), 7.27 (s, 1H, vinylic-H), 7.08-7.02 (m, 2H, Ar-H); $^{13}$C NMR (100 MHz, CDCl$_3$): δ (ppm) = 162.48 (d, $J = 250.14$ Hz), 139.11 (d, $J = 2.93$ Hz), 130.55 (d, $J = 8.80$ Hz), 115.50 (d, $J = 22.00$ Hz), 94.78, 81.40.

$(E)$-1-chloro-3-(1,2-diiodovinyl)benzene (2m)

![Image of compound 2m]

The compound 2m was prepared following general procedure and purified by column chromatography using hexanes as an eluent.

IR (Neat): $\nu_{\text{max}}$ 3064, 2925, 1564, 1465, 1408, 1237, 1158, 1080, 877, 787, 726, 685, and 605 cm$^{-1}$; $^1$H NMR (400 MHz, CDCl$_3$): δ (ppm) = 7.34-7.32 (m, 1H, Ar-H), 7.31-7.29 (m, 2H, Ar-H), 7.30 (s, 1H, vinylic-H) 7.26-7.21 (m, 1H, Ar-H); $^{13}$C NMR (100 MHz, CDCl$_3$): δ (ppm) = 144.66, 134.09, 129.38 (d, $J = 69.69$ Hz), 127.57 (d, $J = 184.86$ Hz), 93.76, 82.09.

HR-MS (EI) m/z calcd for C$_8$H$_5$ClI$_2$, 389.82000; Found 389.81691; Anal. Calcd for C$_8$H$_5$ClI$_2$: C, 24.61; H, 1.29. Found: C, 24.83; H, 1.33.

$(E)$-1-bromo-4-(1,2-diiodovinyl)benzene (2n)$^1$

![Image of compound 2n]

The compound 2n was prepared following general procedure and purified by column chromatography using hexanes as an eluent.

$^1$H NMR (400 MHz, CDCl$_3$): δ (ppm) = 7.52-7.49 (m, 2H, Ar-H), 7.29 (s, 1H, vinylic-H), 7.25-7.21 (m, 2H, Ar-H); $^{13}$C NMR (125 MHz, CDCl$_3$): δ (ppm) = 141.96, 131.67, 130.12, 123.08, 94.44, 81.57.

$(E)$-1-(1,2-diiodovinyl)-4-(trifluoromethyl)benzene (2o)$^2$

![Image of compound 2o]
The compound 2o was prepared following general procedure and purified by column chromatography using hexanes as an eluent.

$^1$H NMR (400 MHz, CDCl$_3$): $\delta$ (ppm) = 7.63 (d, $J = 8.19$ Hz, 2H, Ar-H), 7.46 (d, $J = 8.07$ Hz, 2H, Ar-H), 7.36 (s, 1H, vinylic-H); $^{13}$C NMR (100 MHz, CDCl$_3$): $\delta$ (ppm) = 146.54, 130.74 (d, $J = 33.01$ Hz), 128.90, 125.53 (d, $J = 3.66$ Hz), 123.71 (d, $J = 272.88$ Hz), 93.64, 82.38.

**methyl (E)-4-(1,2-diiodovinyl)benzoate(2p)**

![Methyl (E)-4-(1,2-diiodovinyl)benzoate](image)

The compound 2p was prepared following general procedure and purified by column chromatography using EtOAc/hexanes (0:10 to 0.2:9.8) as an eluent.

$^1$H NMR (400 MHz, CDCl$_3$): $\delta$ (ppm) = 8.05-8.02 (m, 2H, Ar-H), 7.43-7.39 (m, 2H, Ar-H), 7.33 (s, 1H, vinylic-H), 3.92 (s, 3H, OCH$_3$); $^{13}$C NMR (100 MHz, CDCl$_3$): $\delta$ (ppm) = 166.21, 147.30, 130.26, 129.70, 128.49, 94.32, 81.99, 52.24

**(E)-1-(1,2-diiodovinyl)-4-nitrobenzene (2q)**

![Methyl (E)-4-(1,2-diiodovinyl)benzoate](image)

The compound 2q was prepared following general procedure and purified by column chromatography using EtOAc/hexanes (0:10 to 0.1:9.9) as an eluent.

IR (Neat): $\nu_{\text{max}}$ 3068, 2850, 1599, 1518, 1344, 1315, 1152, 1107, 857, 822, 787, 705, and 598 cm$^{-1}$; $^1$H NMR (400 MHz, CDCl$_3$): $\delta$ (ppm) = 8.24 (d, $J = 8.68$ Hz, 2H, Ar-H), 7.51 (d, $J = 8.80$ Hz, 2H, Ar-H), 7.42 (s, 1H, vinylic-H); $^{13}$C NMR (100 MHz, CDCl$_3$): $\delta$ (ppm) = 149.31, 147.51, 129.57, 123.84, 92.26, 83.41; HR-MS (EI) m/z calcd for C$_8$H$_5$I$_2$NO$_2$, 400.84011; Found 400.84096; Anal. Calcd for C$_8$H$_5$I$_2$NO$_2$: C, 23.97; H, 1.26; N, 3.49. Found: C, 24.04; H, 1.23; N; 4.78.
(E)-(1,2-diiodoprop-1-en-1-yl)benzene (2r)

![Structure of 2r]

The compound 2r was prepared following general procedure and purified by column chromatography using hexanes as an eluent. 

$^1$H NMR (500 MHz, CDCl$_3$): $\delta$ (ppm) = 7.34 (t, $J = 7.01$ Hz, 2H, Ar-H), 7.28 (t, $J = 7.17$ Hz, 1H, Ar-H), 7.22 (d, $J = 7.01$ Hz, 2H, Ar-H), 2.79 (s, 1 x 3H, allylic-H); $^{13}$C NMR (100 MHz, CDCl$_3$): $\delta$ (ppm) = 148.04, 128.40, 128.35, 128.20, 96.30, 95.47, 40.17.

(E)-(1,2-diiodobut-1-en-1-yl)benzene (2s)

![Structure of 2s]

The compound 2s was prepared following general procedure and purified by column chromatography using hexanes as an eluent.

$^1$H NMR (400 MHz, CDCl$_3$): $\delta$ (ppm) = 7.37-7.32 (m, 2H, Ar-H), 7.30-7.25 (m, 1H, Ar-H), 7.21-7.18 (m, 2H, Ar-H), 2.87 (q, $J = 7.33$ Hz, 1 x 2H, allylic-H), 1.17 (t, $J = 7.45$ Hz, 1 x 3H, aliphatic-H); $^{13}$C NMR (125 MHz, CDCl$_3$): $\delta$ (ppm) = 148.00, 128.44, 128.35, 128.15, 106.49, 93.61, 44.84, 12.90.

(E)-(1,2-diiodohex-1-en-1-yl)benzene (2t)

![Structure of 2t]

The compound 2t was prepared following general procedure and purified by column chromatography using hexanes as an eluent.

$^1$H NMR (400 MHz, CDCl$_3$): $\delta$ (ppm) = 7.37-7.32 (m, 2H, Ar-H), 7.30-7.27 (m, 1H, Ar-H), 7.21-7.18 (m, 2H, Ar-H), 2.85 (t, $J = 7.58$ Hz, 1 x 2H, allylic-H), 1.70-1.62 (m, 1 x 2H, aliphatic-H), 1.51-1.42 (m, 1 x 2H, aliphatic-H), 1.00 (t, $J = 7.33$ Hz, 1 x 3H, aliphatic-H);
$^{13}$C NMR (100 MHz, CDCl$_3$): $\delta$ (ppm) = 148.18, 128.44, 128.35, 128.13, 105.28, 94.26, 50.26, 30.52, 21.61, 14.09.

**(E)**-$^2$-(1,2-diiodovinyl)pyridine (2u)

![Structure of 2u](image)

The compound 2u was prepared following general procedure and purified by column chromatography using EtOAc/hexanes (0.2:9.8 to 0.5:9.5) as an eluent.

IR (Neat): $\nu$max 3046, 2923, 1571, 1454, 1279, 1160, 989, 793, 744, 682 and 598 cm$^{-1}$; $^1$H NMR (400 MHz, CDCl$_3$): $\delta$ (ppm) = 8.68 (d, $J = 4.15$ Hz, 1H, Ar-$H$), 7.56 (ddd, $J = 1.83$, 7.82, 15.52 Hz, 1H, Ar-$H$), 7.47 (d, $J = 7.94$ Hz, 1H, Ar-$H$), 7.43 (s, 1H, vinylic-$H$), 7.27-7.23 (m, 1H, Ar-$H$); $^{13}$C NMR (100 MHz, CDCl$_3$): $\delta$ (ppm) = 158.94, 149.74, 136.45, 123.53, 123.40, 96.53, 81.73; HR-MS (EI) m/z calcd for C$_7$H$_5$I$_2$N, 356.85400; Found 356.85113; Anal. Calcd for C$_7$H$_5$I$_2$N: C, 23.56; H, 1.41; N, 3.92. Found: C, 23.68; H, 1.71; N, 5.12.

**(E)**-$^3$-(1,2-diiodovinyl)thiophene (2v)$^6$

![Structure of 2v](image)

The compound 2v was prepared following general procedure and purified by column chromatography using EtOAc/hexanes (0.2:9.8 to 0.5:9.5) as an eluent.

$^1$H NMR (500 MHz, CDCl$_3$): $\delta$ (ppm) = 7.58-7.56 (m, 1H, Ar-$H$), 7.33-7.30 (m, 1H, Ar-$H$), 7.29-7.25 (m, 1H, Ar-$H$), 7.24 (s, 1H, vinylic-$H$); $^{13}$C NMR (100 MHz, CDCl$_3$): $\delta$ (ppm) = 142.17, 128.42, 126.46, 125.19, 90.27, 79.67.

**(E)**-$^4$-(3,4-diiodobut-3-en-1-yl)benzene (2w)

![Structure of 2w](image)

The compound 2w was prepared following general procedure and purified by column chromatography using hexanes as an eluent.
IR (Neat): $\nu_{\text{max}}$ 3067, 3026, 2854, 1598, 1494, 1260, 1216, 1161, 1076, 1007, 903, 772, 747, 698, and 557 cm$^{-1}$; $^1$H NMR (400 MHz, CDCl$_3$): $\delta$ (ppm) = 7.32-7.19 (m, 5H, Ar-H), 6.84 (s, 1H, vinylic-H), 2.82 (s, 2 x 2H, benzylic-H + allylic-H); $^{13}$C NMR (100 MHz, CDCl$_3$): $\delta$ (ppm) = 139.52, 128.73, 128.40, 126.34, 102.35, 80.09, 46.81, 34.10; HR-MS (EI) m/z calcd for C$_{10}$H$_{10}$I$_2$, 383.88343; Found 383.88719; Anal. Calcd for C$_{10}$H$_{10}$I$_2$: C, 31.28; H, 2.63. Found: C, 31.96; H, 2.44.

$(E)$-1,2-diiodohept-1-ene (2x)$^3$

\begin{center}
\includegraphics{2x.png}
\end{center}

The compound 2x was prepared following general procedure and purified by column chromatography using hexanes as an eluent.

$^1$H NMR (400 MHz, CDCl$_3$): $\delta$ (ppm) = 6.80 (s, 1H, vinylic-H), 2.50 (t, $J = 7.21$ Hz, 1 x 2H, allylic-H), 1.54 (quint, $J = 7.45$ Hz, 1 x 2H, aliphatic-H), 1.40-1.28 (m, 2 x 2H, aliphatic-H), 0.91 (t, $J = 6.96$ Hz, 1 x 3H, aliphatic-H); $^{13}$C NMR (100 MHz, CDCl$_3$): $\delta$ (ppm) = 104.44, 78.90, 44.60, 30.29, 27.81, 22.47, 13.97.

$(E)$-1,2-diiodooct-1-ene (2y)$^4$

\begin{center}
\includegraphics{2y.png}
\end{center}

The compound 2y was prepared following general procedure and purified by column chromatography using hexanes as an eluent.

$^1$H NMR (500 MHz, CDCl$_3$): $\delta$ (ppm) = 6.80 (s, 1H, vinylic-H), 2.50 (t, $J = 7.32$ Hz, 1 x 2H, allylic-H), 1.53 (quint, $J = 7.32$ Hz, 1 x 2H, aliphatic-H), 1.37-1.30 (m, 3 x 2H, aliphatic-H), 0.90 (t, $J = 6.86$ Hz, 1x 3H, aliphatic-H); $^{13}$C NMR (100 MHz, CDCl$_3$): $\delta$ (ppm) = 104.43, 78.86, 44.65, 31.59, 28.11, 27.82, 22.52, 14.05.

$(E)$-1,2-diiododec-1-ene (2z)$^5$

\begin{center}
\includegraphics{2z.png}
\end{center}
The compound $2z$ was prepared following general procedure and purified by column chromatography using hexanes as an eluent.

$^1$H NMR (400 MHz, CDCl$_3$): $\delta$ (ppm) = 6.80 (s, 1H, vinylic-$H$), 2.50 (t, $J = 7.21$ Hz, 1 x 2H, allylic-$H$), 1.53 (quint, $J = 7.45$ Hz, 1 x 2H, aliphatic-$H$), 1.34-1.26 (m, 5 x 2H, aliphatic-$H$), 0.88 (t, $J = 6.72$ Hz, 1 x 3H, aliphatic-$H$);

$^{13}$C NMR (125 MHz, CDCl$_3$): $\delta$ (ppm) = 104.44, 78.86, 44.63, 31.82, 29.36, 29.15, 28.16, 28.14, 22.65, 14.12.

\((E)-(1,2\text{-diiodovinyl})\text{cyclohexane (2aa)}\)

\begin{center}
\includegraphics[width=0.2\textwidth]{2aa.png}
\end{center}

The compound $2aa$ was prepared following general procedure and purified by column chromatography using hexanes as an eluent.

IR (Neat): $\nu_{\text{max}}$ 3067, 2925, 2851, 2663, 1533, 1446, 1349, 1222, 1133, 1057, 949, 892, 767, 655, and 574 cm$^{-1}$; $^1$H NMR (400 MHz, CDCl$_3$): $\delta$ (ppm) = 6.78 (s, 1H, vinylic-$H$), 2.12-2.05 (m, 1H, allylic-$H$), 1.83-1.55 (m, 5H, cyclohexyl-$H$), 1.49-1.29 (m, 4H, cyclohexyl-$H$), 1.21-1.13 (m, 1H, cyclohexyl-$H$);

$^{13}$C NMR (100 MHz, CDCl$_3$): $\delta$ (ppm) = 114.34, 76.37, 48.78, 32.28, 25.44, 25.18; HR-MS (El) m/z calcd for C$_8$H$_{12}$I$_2$, 361.90100; Found 361.90284; Anal. Calcd for C$_8$H$_{12}$I$_2$: C, 26.54; H, 3.34. Found: C, 26.15; H, 3.15.

\((E)-2,3\text{-diiodooct-2-ene (2ab)}\)

\begin{center}
\includegraphics[width=0.2\textwidth]{2ab.png}
\end{center}

The compound $2ab$ was prepared following general procedure and purified by column chromatography using hexanes as an eluent.

IR (Neat): $\nu_{\text{max}}$ 3025, 2925, 2858, 1706, 1457, 1375, 1058, 980, 728, and 543 cm$^{-1}$; $^1$H NMR (400 MHz, CDCl$_3$): $\delta$ (ppm) = 2.68-2.63 (m, 1 x 2H, allylic-$H$), 2.60 (s, 1 x 3H, allylic-$H$), 1.54 (quint, $J = 5.01$ Hz, 1 x 2H, aliphatic-$H$), 1.42-1.25 (m, 2 x 2H, aliphatic-$H$), 0.91 (t, $J = 6.84$ Hz).
Hz, 1 x 3H, aliphatic-\( H \)); \(^{13}\)C NMR (125 MHz, CDCl\(_3\)): \( \delta \) (ppm) = 103.06, 92.85, 50.76, 40.34, 30.49, 27.90, 22.51, 13.99; HR-MS (EI) \( m/z \) calcd for C\(_8\)H\(_{14}\)I\(_2\), 363.91700; Found 363.91849; Anal. Calcd for C\(_8\)H\(_{14}\)I\(_2\): C, 26.40; H, 3.88. Found: C, 26.92; H, 4.03.

\((E)-4,5\)-diiodooct-4-ene (2ac)\(^1\)

![Diagram of 2ac]

The compound 2ac was prepared following general procedure and purified by column chromatography using hexanes as an eluent.

\(^1\)H NMR (400 MHz, CDCl\(_3\)): \( \delta \) (ppm) = 2.68 (t, \( J = 7.33 \) Hz, 2 x 2H, allylic-\( H \)), 1.64-1.54 (m, 2 x 2H, aliphatic-\( H \)), 0.96 (t, \( J = 7.33 \) Hz, 2 x 3H, aliphatic-\( H \)); \(^{13}\)C NMR (125 MHz, CDCl\(_3\)): \( \delta \) (ppm) = 101.96, 52.53, 21.61, 12.80.
5. Copies of $^1$H and $^{13}$C NMR spectra of all products:
2ac
6. References: