Supporting Information

Observation of Solvent Enantio-Isotope Effect in Asymmetric Ring-Opening of Cyclic Diaryliodoniums with Selenocyanate

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Contents

1. General information					S3
2. Synthesis of chiral ligands					S3
3. Copper-catalyzed asymme	tric coupling	between	cyclic	diaryliodonium	and
KSeCN					S6
4. Synthetic applications					S16
5. Kinetic Studies					S25
6. Crystal structures and structur	e refinement				. S31
7. References			•••••		. S34
8. Copies of NMR spectra					. S35
9. Copies of HPLC traces					S 81

1. General Information and Materials

All reactions were carried out under a nitrogen atmosphere, unless the reaction procedure states otherwise. ¹H and ¹³C NMR spectra were recorded on Bruker AC-400 FT spectrometer and Bruker AC-500 FT spectrometer using solvent residue as an internal reference (7.26 and 77.00 ppm for CDCl₃, 2.50 and 39.00 ppm for DMSO-*d*₆, respectively). Chemical shifts (δ) are reported in ppm, and coupling constants (*J*) are in Hertz (Hz). The following abbreviations explained the multiplicities: s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet, br = broad. High resolution mass spectra (HRMS (ESI)) was recorded on a high-resolution mass spectrometer with a Q-TOF analyzer (Waters XEVO-G2 Q-TOF). Single crystal data were collected at 100 K on a Rigaku Oxford Diffraction Supernova Dual Source, Cu at Zero equipped with an AtlasS2 CCD using Cu K α radiation. Reaction heats were measured using Omnical SuperCRC. Reaction heats were normalized to 100% conversion, and the sensible heat of dosing has been removed unless otherwise stated. All reagents were used as received from commercial sources and used without further purification. Flash column chromatography was performed using 200-300 mesh silica gel as the stationary phase. Room temperature refers 25°C-30°C.

2. Synthesis of Chiral Ligands

General Procedure for the Synthesis of Chiral Ligands (L7-L11)¹⁻⁴

Synthesis of bisoxazoline L7¹



A mixture of dimethyl 4-chloropyridine-2,6-dicarboxylate (459 mg, 2.0 mmol) and (1*S*,2*R*)aminoindanol (895 mg, 6.0 mmol, 3.0 equiv) was stirred at 120 °C for 16 h. The solid obtained was dissolved in hot CH₂Cl₂ (25 mL) and it was subjected to column chromatography using CH₂Cl₂/EtOAc (20/1, v/v) as the elution to afford **I** in 99% yield. To a flame-fried 50 mL Schlenk tube was added **I** (463 mg, 1.0 mmol), followed by the addition of 20 mL of CH₂Cl₂. The resulting solution was cooled to -20 °C and was charged with DAST (diethylaminosulfur trifluoride) (396 uL, 3.0 mmol). The reaction was allowed to proceed at -20 °C for 24 h and then was quenched with aqueous NH₄OH, diluted with H₂O. The aqueous layer was extracted with CH₂Cl₂ (20 mL×3), the combined organic phase was dried with Na₂SO₄, filtrated, and then filtrate was concentrated under reduced pressure. The residue was subjected to column chromatography using CH₂Cl₂/EtOAc (5/1, v/v) as the eluent to afford L7 as a white solid (319 mg, 74% yield). [α]²⁰_D = -115.8 (c 0.50, CH₂Cl₂). ¹H NMR (400 MHz, CDCl₃) δ 8.11 (s, 2H), 7.79 – 7.46 (m, 2H), 7.44 – 7.10 (m, 6H), 5.79 (d, *J* = 8.0 Hz, 2H), 5.70 – 5.45 (m, 2H), 3.57 – 3.41 (m, 4H). ¹³C NMR (101 MHz, CDCl₃) δ 162.0, 148.0, 145.3, 141.1, 139.7, 128.7, 127.5, 126.1, 125.6, 125.3, 84.6, 76.9, 39.6. HRMS (ESI) m/z: calcd. for C₂₅H₁₉ClN₃O₂ [M+H]⁺: 428.1160; Found: 428.1172.

Synthesis of bisoxazoline L8²



To a flame-dried round-bottom flask under nitrogen was added L7 (85.6 mg, 0.20 mmol, 1.0 equiv), phenol (22.6 mg, 0.24 mmol, 1.2 equiv), Cs₂CO₃ (130 mg, 0.40 mmol, 2.0 equiv) and dry dimethyl sulfoxide (2.0 mL). The mixture was stirred at 80 °C for 12 h before it was cooled to room temparature. The crude reaction mixture was quenched with H₂O (5.0 mL). The aqueous layer was extracted with EtOAc (5.0 mL×3), the combined organic phase was dried with Na₂SO₄, filtrated, and then the filtrate was concentrated under reduced pressure. The residue was subjected to column chromatography using CH₂Cl₂/EtOAc (5/1, v/v) as the eluent to afford L8 as a white solid (63.0 mg, 65% yield). [α]²⁰_D = -25.6 (c 0.50, CH₂Cl₂). ¹H NMR (600 MHz, CDCl₃) δ 7.62 (s, 2H), 7.56 – 7.50 (m, 2H), 7.41 (t, *J* = 8.4 Hz, 2H), 7.26 – 7.21 (m, 7H), 7.04 (d, *J* = 7.8 Hz, 2H), 5.74 (d, *J* = 7.8 Hz, 2H), 5.65 – 5.47 (m, 2H), 3.59 – 3.36 (m, 4H). ¹³C NMR (126 MHz, CDCl₃) δ 165.5, 162.6, 153.6, 148.8, 141.3, 139.9, 130.4, 128.6, 127.4, 125.7, 125.6, 125.3, 120.6, 114.3, 84.3, 39.6. HRMS (ESI) m/z: calcd. for C₃₁H₂₄N₃O₃ [M+H]⁺: 486.1812; Found: 486.1819.

Synthesis of bisoxazoline L9³



To a flame-dried round-bottom flask under nitrogen was added A1 (168.8 mg, 0.60 mmol, 3.0 equiv), A2 (53.0 mg, 0.20 mmol, 1.0 equiv), and dry THF (5.0 mL). The mixture was stirred at 50 °C for 20 h before it was cooled to room temperature. The crude reaction mixture was filtrated through a pad of celite and concentrated under vacuum. The residue was subjected to column chromatography on silica gel using CH₂Cl₂/EtOAc (5/1, v/v) as the eluent to afford L9 as a white solid (53.7 mg, 41% yield). $[\alpha]_D^{20} = +52.2$ (c 0.25, CH₂Cl₂). ¹H NMR (500 MHz, CDCl₃) δ 8.12 (d, J = 8.0 Hz, 2H), 7.79 (d, J = 2.0 Hz, 3H), 7.56 (d, J = 8.5 Hz, 4H), 7.50 (dd, J = 8.0, 2.0 Hz, 2H), 7.46 (d, J = 8.5 Hz, 4H), 7.30 (d, J = 8.0 Hz, 2H), 5.83 (d, J = 8.0 Hz, 2H), 5.68 – 5.61 (m, 2H), 3.52 (d, J = 4.0 Hz, 4H), 1.37 (s, 18H). ¹³C NMR (126 MHz, CDCl₃) δ 162.8, 150.1, 146.9, 142.0, 140.5, 138.7,

138.0, 137.2, 127.6, 126.7, 126.0, 125.7, 125.5, 124.1, 84.5, 39.4, 34.5, 31.3. **HRMS (ESI)** m/z: calcd. for C₄₅H₄₄N₃O₂ [M+H]⁺: 658.3428; Found: 658.3442.

Synthesis of bisoxazoline L10



A reported procedure was followed with some modifications.³⁻⁴ To a nitrogen-filled round-bottom flask were added **A3** (254.1 mg, 1.0 mmol, 1.0 equiv), Pd(dppf)Cl₂ (21.9 mg, 0.03 mmol, 3.0 mol%), Cs₂CO₃ (651.6 mg, 2.0 mmol, 2.0 equiv.) and (3,5-di-*tert*-butylphenyl)boronic acid (280.9 mg, 1.2 mmol, 1.2 equiv) in THF (5.0 mL) and H₂O (1.0 mL). The mixture was heated to 80 °C and stirred for 12 h before it was cooled to room temperature. The reaction mixture was quenched with water (5 mL), extracted with EtOAc (10 mL×3). The combined organic layers were washed with brine, dried over Na₂SO₄, and filtered. The solvent was removed under reduced pressure, and the residue was purified by silica gel column chromatography (CH₂Cl₂/EtOAc = 5/1) to afford compound **A4** as a white solid (253.4 mg, 70% yield). $[\alpha]_D^{20} = +21.0$ (c 0.50, CH₂Cl₂). ¹H NMR (500 MHz, CDCl₃) δ 7.43 (t, J = 2.0 Hz, 1H), 7.39 – 7.33 (m, 2H), 7.31 – 7.24 (m, 1H), 7.22 (d, J = 2.0 Hz, 2H), 6.61 (s, 1H), 5.50 – 5.35 (m, 1H), 5.24 (d, J = 7.5 Hz, 1H), 3.54 – 3.20 (m, 2H), 1.36 (s, 18H). ¹³C NMR (126 MHz, CDCl₃) δ 159.6, 150.8, 141.0, 140.7, 139.3, 137.6, 129.9, 128.5, 123.3, 122.7, 121.3, 80.3, 61.3, 38.9, 34.9, 31.5. HRMS (ESI) m/z: calcd. for C₂₄H₃₀NO₂ [M+H]⁺: 364.2271; Found: 364.2219.

A 50-mL round bottom flask equipped with a condenser was charged with compound A4 (253.4 mg, 0.70 mmol, 1.0 equiv.), KOH (117.8 mg, 3.6 mmol, 3.6 equiv.), EtOH (10 mL) and water (10 mL). The mixture was stirred at 100 °C for 6 h. After being cooled to room temperature, the mixture was extracted with EtOAc (3 x 5 mL) and the organic phase was extracted with aqueous HCl (2 M, 5 mL). The aqueous phase was brought to pH 14 by adding solid NaOH and was extracted again with EtOAc (5 mL×3). The combined EtOAc layers were dried over MgSO₄ and concentrated to obtain the title compound A5 as a colorless solid without further purification (270.0 mg, 80 % yield). ¹H NMR (500 MHz, CDCl₃) δ 8.34 (s, 2H), 7.98 (s, 1H), 7.48 (dd, *J* = 8.0, 1.5 Hz, 1H), 7.40 (d, *J* = 1.5 Hz, 2H), 7.35 (t, *J* = 1.5 Hz, 1H), 7.13 (d, *J* = 8.0 Hz, 1H), 5.50 (s, 1H), 4.75 (d, *J* = 6.0 Hz, 1H), 4.68 – 4.47 (m, 1H), 3.29 – 2.70 (m, 2H), 1.30 (s, 18H). ¹³C NMR (126 MHz, CDCl₃) δ 151.1, 141.9, 139.8, 139.5, 136.2, 129.1, 125.5, 125.2, 121.6, 121.4, 71.2, 57.5, 38.1, 34.9, 31.5, 31.4. [α]²⁰_D = -19.7 (c 0.50, CH₂Cl₂). HRMS (ESI) m/z: calcd. for C₂₃H₃₂NO [M+H]⁺: 338.2478; Found: 338.2480.



To a flame-dried round-bottom flask under argon was added **A5** (253.1 mg, 0.7 mmol, 2.8 equiv.), **A2** (66.3 mg, 0.25 mmol, 1.0 equiv.), and dry THF (5.0 mL). The mixture was stirred at 50 °C for 20 h before it was cooled to room temperature. The crude reaction mixture was filtrated with a pad of celite and concentrated under vacuum. The residue was subjected to column chromatography using CH₂Cl₂/EtOAc (5/1, v/v) as the eluent to afford **L10** (74.1 mg, 38% yield). $[\alpha]_D^{20} = +230.4$ (c 0.50, CH₂Cl₂). ¹H NMR (500 MHz, CDCl₃) δ 8.14 (dd, J = 8.0, 2.0 Hz, 2H), 7.83 – 7.79 (m, 2H), 7.55 – 7.50 (m, 3H), 7.46 – 7.42 (m, 6H), 7.33 (dd, J = 8.0, 2.0 Hz, 2H), 5.86 (dd, J = 8.0, 2.5 Hz, 2H), 5.67 (dt, J = 7.0, 3.5 Hz, 2H), 3.56 – 3.49 (m, 4H), 1.41 – 1.38 (m, 36H). ¹³C NMR (126 MHz, CDCl₃) δ 162.9, 151.0, 147.0, 141.9, 140.3, 138.7, 137.2, 128.0, 126.0, 125.5, 124.5, 121.7, 121.3, 84.5, 39.4, 34.9, 31.5. HRMS (ESI) m/z: calcd. for C₅₃H₆₀N₃O₂ [M+H]⁺: 770.4680; Found: 770.4670.

Synthesis of bisoxazoline L11³



To a flame-dried round-bottom flask under argon was added **A6** (126.7 mg, 0.6 mmol, 3.0 equiv.), **A2** (53.0 mg, 0.2 mmol, 1.0 equiv.), and dry THF (5 mL). The mixture was stirred at 50 °C for 20 h before it was cooled to room temperature. The crude reaction mixture was filtrated with a pad of celite and concentrated under vacuum. The residue was subjected to column chromatography using CH₂Cl₂/EtOAc (5/1, v/v) as the elution to afford **L11** as a white solid (58.5 mg, 58% yield). $[\alpha]_D^{20}$ = +45.1 (c 0.25, CH₂Cl₂). ¹H NMR (500 MHz, CDCl₃) δ 8.10 (d, *J* = 8.0 Hz, 2H), 7.84 – 7.75 (m, 2H), 7.59 (d, *J* = 2.0 Hz, 2H), 7.39 – 7.28 (m, 2H), 7.18 (d, *J* = 8.0 Hz, 2H), 5.76 (d, *J* = 8.0 Hz, 2H), 5.65 – 5.56 (m, 2H), 3.50 – 3.38 (m, 4H), 1.33 (s, 18H). ¹³C NMR (126 MHz, CDCl₃) δ 162.7, 150.7, 147.1, 141.3, 137.0, 136.9, 126.1, 126.0, 124.8, 122.3, 84.5, 39.4, 34.7, 31.5. HRMS (ESI) m/z: calcd. for C₃₃H₃₆N₃O₂ [M+H]⁺: 506.2802; Found: 506.2805.

3. Copper-catalyzed asymmetric coupling between cyclic diaryliodonium and KSeCN

General procedure for Cu-catalyzed coupling of cyclic iodonium, and potassium

selenocyanate in CH₂Cl₂/D₂O (General Procedure A)



Under nitrogen atmosphere, a Schlenk tube containing a stirring bar was charged with CuI (1.9 mg, 0.01 mmol), L11 (10.1 mg, 0.02 mmol), and cyclic diaryliodonium salt 1 (0.20 mmol, 1.0 equiv.), in anhydrous dichloromethane (5.0 mL), the resulting solution was stirred for 30 min. A solution of potassium selenocyanate (0.40 mmol, 2.0 equiv.) in deuterium oxide (1.0 mL) was added. The reaction mixture was stirred at room temperature for 12 h. The reaction mixture was directly extracted with CH_2Cl_2 , and the combined organic phase was dried with Na_2SO_4 . The solid was filtered, and the filtrate was concentrated under reduced pressure. The residue was subjected to column chromatography using PE/EtOAc (50/1, v/v) as the eluent to afford product 3.

General procedure for Cu-catalyzed coupling of cyclic iodonium, and potassium selenocyanate in CH₂Cl₂/H₂O (General Procedure A')



Under nitrogen atmosphere, a Schlenk tube containing a stirring bar was charged with CuI (1.0 mg, 0.005 mmol), L11 (5.0 mg, 0.01 mmol), and cyclic diaryliodonium salt 1 (0.10 mmol, 1.0 equiv.), in anhydrous dichloromethane (2.5 mL), the resulting solution was stirred for 30 min. A solution of potassium selenocyanate (0.20 mmol, 2.0 equiv.) in water (0.5 mL) was added. The reaction mixture was stirred at room temperature for 12 h. The reaction mixture was directly extracted with CH₂Cl₂, and the combined organic phase was dried with Na₂SO₄. The solid was filtered, and the filtrate was concentrated under reduced pressure. The residue was subjected to column chromatography using PE/EtOAc (50/1, v/v) as the eluent to afford product **3**.



3a was prepared following the General Procedure A

The reaction of **1a** (91.2 mg, 0.20 mmol, 1.0 equiv), CuI (1.9 mg, 0.01 mmol) and **L11** (10.1 mg, 0.02 mmol), potassium selenocyanate (57.6 mg, 0.40 mmol, 2.0 equiv) in anhydrous dichloromethane (5.0 mL) and deuterium oxide (1.0 mL) at room temperature for 12 h afforded **3a** as a pale-yellow solid (68.0 mg, 91%, 94% ee) (eluent for column chromatography PE/EtOAc = 50:1). **HPLC conditions**: Chiralpak AD-H, isopropanol/hexane = 0.5:99.5, flow: 1.0 mL/min, λ = 254 nm, t_r = 8.937 min (major), 11.521 min (minor). [α]²⁰_D = -14.2 (c 0.50, CH₂Cl₂). ¹H NMR (500 MHz, CDCl₃) δ 7.82 (d, *J* = 8.0 Hz, 1H), 7.73 (d, *J* = 7.5 Hz, 1H), 7.36 (t, *J* = 7.5 Hz, 1H), 7.35 – 7.29 (m, 2H), 7.06 (t, *J* = 8.0 Hz, 1H), 2.07 (s, 3H), 1.99 (s, 3H). ¹³C NMR (126 MHz, CDCl₃) δ 142.6, 141.8, 138.3, 137.8, 137.3, 130.63, 130.60, 130.5, 129.9, 127.9, 124.8, 101.8, 100.7, 21.2, 19.6. HRMS (ESI)

m/z: calcd. for $C_{15}H_{12}IN^{80}$ SeNa [M+Na]⁺: 435.9072; Found: 435.9071.

3a was prepared following the General Procedure A'

The reaction of **1a** (45.6 mg, 0.10 mmol, 1.0 equiv), CuI (1.0 mg, 0.005 mmol) and L**11** (5.0 mg, 0.01 mmol), potassium selenocyanate (28.8 mg, 0.20 mmol, 2.0 equiv) in anhydrous dichloromethane (2.5 mL) and water (0.5 mL) at room temperature for 12 h afforded **3a** as a pale-yellow solid (37.2 mg, 90%, 92% ee) (eluent for column chromatography PE/EtOAc = 50:1).



3b was prepared following the General Procedure A

The reaction of **1b** (48.4 mg, 0.10 mmol, 1.0 equiv), CuI (1.0 mg, 0.005 mmol) and L**11** (5.0 mg, 0.01 mmol), potassium selenocyanate (28.8 mg, 0.20 mmol, 2.0 equiv) in anhydrous dichloromethane (2.5 mL) and deuterium oxide (0.5 mL) at room temperature for 12 h afforded **3b** as a pale-yellow solid (43.8 mg, 99%, 88% ee) (eluent for column chromatography PE/EtOAc = 50:1). **HPLC conditions**: Chiralpak AD-H, isopropanol/hexane = 0.2:99.8, flow: 1.0 mL/min, λ = 230 nm, t_r = 9.936 min (minor), 11.408 min (major). [α]²⁰_D = -1.4 (c 0.50, CH₂Cl₂). ¹H **NMR (400 MHz, CDCl₃)** δ 7.85 – 7.79 (m, 1H), 7.78 – 7.70 (m, 1H), 7.43 (t, *J* = 7.6 Hz, 1H), 7.40 – 7.36 (m, 2H), 7.13 (t, *J* = 7.6 Hz, 1H), 2.41 – 2.17 (m, 4H), 1.16 (t, *J* = 7.6 Hz, 3H), 1.12 (t, *J* = 7.6 Hz, 3H). ¹³C **NMR (126 MHz, CDCl₃)** δ 144.4, 143.5, 141.9, 141.2, 137.3, 130.8, 130.1, 128.4, 128.2, 127.6, 125.5, 102.1, 101.6, 27.3, 25.9, 14.2, 13.8. **HRMS (ESI)** m/z: calcd. for C₁₇H₁₇IN⁸⁰Se [M+H]⁺: 441.9565; Found: 441.9571.



3c was prepared following the General Procedure A

The reaction of **1c** (99.4 mg, 0.20 mmol, 1.0 equiv), CuI (1.9 mg, 0.01 mmol) and **L11** (10.1 mg, 0.02 mmol), potassium selenocyanate (57.6 mg, 0.40 mmol, 2.0 equiv) in anhydrous dichloromethane (5.0 mL) and deuterium oxide (1.0 mL) at room temperature for 12 h afforded **3c** as a white solid (87.9 mg, 97%, 96% ee) (eluent for column chromatography PE/EtOAc = 50:1). **HPLC conditions**: Chiralpak OD-3, isopropanol/hexane = 1:99, flow: 0.4 mL/min, λ = 230 nm, t_r = 72.797 min (major), 75.472 min (minor). [α]_D²⁰ = -11.1 (c 0.50, CH₂Cl₂). ¹H NMR (500 MHz, **CDCl**₃) δ 7.91 (dd, *J* = 8.0, 1.0 Hz, 1H), 7.83 (dd, *J* = 8.0, 1.0 Hz, 1H), 7.55 (td, *J* = 8.0, 6.5, 1.0 Hz, 2H), 7.44 (t, *J* = 8.0 Hz, 1H), 7.14 (t, *J* = 8.0 Hz, 1H). ¹³C NMR (126 MHz, CDCl₃) δ 140.7, 139.8, 138.1, 135.1, 134.0, 132.0, 131.3, 130.1, 129.9, 129.7, 126.3, 100.7, 100.2. HRMS (ESI) m/z: calcd. for C₁₃H₆Cl₂IN⁸⁰SeNa [M+Na]⁺: 475.7979; Found: 475.7971.



3d was prepared following the General Procedure A

The reaction of 1d (117.2 mg, 0.20 mmol, 1.0 equiv), CuI (1.9 mg, 0.01 mmol) and L11 (10.1 mg, 0.02 mmol), potassium selenocyanate (57.6 mg, 0.40 mmol, 2.0 equiv) in anhydrous dichloromethane (5.0 mL) and deuterium oxide (1.0 mL) at room temperature for 12 h afforded 3d a white solid (86.7 mg, 80%, 90% ee) (eluent for column chromatography PE/EtOAc = 50:1). HPLC conditions: Chiralpak AD-H, isopropanol/hexane = 1.5:98.5, flow: 0.4 mL/min, λ = 230 nm, t_r = 78.143 min (major), 81.087 min (minor). [α]²⁰_D = -6.3 (c 0.50, CH₂Cl₂). ¹H NMR (500 MHz, CDCl₃) δ 7.95 (dd, *J* = 8.0, 1.0 Hz, 1H), 7.87 (dd, *J* = 8.0, 1.0 Hz, 1H), 7.74 (dd, *J* = 3.5, 1.0 Hz, 1H), 7.72 (dd, *J* = 3.5, 1.0 Hz, 1H), 7.35 (t, *J* = 8.0 Hz, 1H), 7.04 (t, *J* = 8.0 Hz, 1H). ¹³C NMR (101 MHz, CDCl₃) δ 143.9, 142.9, 138.9, 133.27, 133.25, 132.3, 131.6, 130.1, 126.3, 125.0, 123.4, 101.1, 99.9. HRMS (ESI) m/z: calcd. for C₁₃H₆Br₂IN⁸⁰SeNa [M+Na]⁺: 563.6969; Found: 563.6975.



3e was prepared following the General Procedure A

The reaction of **1e** (153.7 mg, 0.20 mmol, 1.0 equiv), CuI (1.9 mg, 0.01 mmol) and **L11** (10.1 mg, 0.02 mmol), potassium selenocyanate (57.6 mg, 0.40 mmol, 2.0 equiv) in anhydrous dichloromethane (5.0 mL) and deuterium oxide (1.0 mL) at room temperature for 12 h afforded **3e** as a white solid (138.4 mg, 96%, 96% ee) (eluent for column chromatography PE/EtOAc = 10:1). **HPLC conditions**: Chiralpak AD-H, isopropanol/hexane = 30:70, flow: 0.8 mL/min, λ = 230 nm, t_r = 17.498 min (minor), 21.854 min (major). [α]²⁰_D = -47.5 (c 0.50, CH₂Cl₂). ¹H NMR (**500 MHz**, **CDCl**₃) δ 7.82 - 7.75 (m, 2H), 7.51 - 7.44 (m, 4H), 7.42 - 7.37 (m, 2H), 7.22 (d, *J* = 8.0 Hz, 2H), 7.20 - 7.14 (m, 4H), 7.08 (t, *J* = 8.0 Hz, 2H), 2.43 (s, 3H), 2.40 (s, 3H). ¹³C NMR (**126 MHz, CDCl**₃) δ 147.9, 147.0, 145.8, 145.4, 137.8, 133.53, 133.45, 132.9, 132.4, 131.5, 131.4, 131.2, 129.81, 129.79, 129.7, 128.0, 127.92, 127.86, 122.4, 122.1, 103.1, 101.6, 21.72, 21.68. HRMS (ESI) m/z: calcd. for C₂₇H₂₀INO₆S₂⁸⁰SeNa [M+Na]⁺: 747.8834; Found: 747.8845.

3e was prepared following the General Procedure A'

The reaction of **1e** (72.4 mg, 0.10 mmol, 1.0 equiv), CuI (1.0 mg, 0.005 mmol) and **L11** (5.0 mg, 0.01 mmol), potassium selenocyanate (28.8 mg, 0.20 mmol, 2.0 equiv) in anhydrous dichloromethane (2.5 mL) and water (0.5 mL) at room temperature for 12 h afforded **3e** as a white solid (66.0 mg, 91%, 88% ee) (eluent for column chromatography PE/EtOAc = 10:1).



3f was prepared following the General Procedure A

The reaction of **1f** (66.8 mg, 0.10 mmol, 1.0 equiv), CuI (1.0 mg, 0.005 mmol) and **L11** (5.0 mg, 0.01 mmol), potassium selenocyanate (28.8 mg, 0.20 mmol, 2.0 equiv) in anhydrous dichloromethane (2.5 mL) and deuterium oxide (0.5 mL) at room temperature for 12 h afforded **3f** a white solid (55.4 mg, 89%, 97% ee) (eluent for column chromatography PE/EtOAc = 10:1). **HPLC conditions**: Chiralpak OD-3, isopropanol/hexane = 25:75, flow: 0.4 mL/min, λ = 230 nm, t_r = 20.400 min (minor), 24.224 min (major). [α]²⁰_D = -45.8 (c 0.50, CH₂Cl₂). ¹H **NMR (500 MHz, CDCl**₃) δ 7.89 (dd, *J* = 8.0, 1.0 Hz, 1H), 7.86 – 7.81 (m, 4H), 7.78 (dd, *J* = 6.0, 3.0 Hz, 1H), 7.59 – 7.53 (m, 2H), 7.49 – 7.43 (m, 2H), 7.43 – 7.37 (m, 4H), 7.33 (dd, *J* = 8.0, 1.0 Hz, 1H), 7.20 (t, *J* = 8.0 Hz, 1H). ¹³C **NMR (126 MHz, CDCl**₃) δ 164.5, 163.6, 149.12, 149.06, 136.8, 133.9, 133.72, 133.71, 133.4, 131.6, 131.0, 130.1, 130.0, 129.8, 128.7, 128.53, 128.52, 128.3, 126.3, 123.8, 122.9, 102.0, 100.4. **HRMS (ESI)** m/z: calcd. for C₂₇H₁₆INO₄⁸⁰SeNa [M+Na]⁺: 647.9181; Found: 647.9185.



3g was prepared following the General Procedure A

The reaction of **1g** (52.8 mg, 0.10 mmol, 1.0 equiv), CuI (1.0 mg, 0.005 mmol) and **L11** (5.0 mg, 0.01 mmol), potassium selenocyanate (28.8 mg, 0.20 mmol, 2.0 equiv) in anhydrous dichloromethane (2.5 mL) and deuterium oxide (0.5 mL) at room temperature for 12 h afforded **3g** as a white solid (47.8 mg, 99%, 98% ee) (eluent for column chromatography PE/EtOAc = 20:1). **HPLC conditions**: Chiralpak OD-H, isopropanol/hexane = 5:95, flow: 1.0 mL/min, λ = 254 nm, t_r = 13.436 min (major), 17.715 min (minor). [α]²⁰_D = -34.6 (c 0.50, CH₂Cl₂). ¹H **NMR (500 MHz, CDCl₃)** δ 8.06 (dd, *J* = 8.5, 3.0 Hz, 2H), 8.02 (d, *J* = 8.5 Hz, 1H), 8.00 (d, *J* = 8.5 Hz, 1H), 7.95 (d, *J* = 8.0 Hz, 1H), 7.76 (d, *J* = 8.5 Hz, 1H), 7.59 – 7.51 (m, 2H), 7.37 (ddd, *J* = 8.0, 7.0, 1.0 Hz, 1H), 7.06 (t, *J* = 9.0 Hz, 2H). ¹³C **NMR (126 MHz, CDCl₃)** δ 140.0, 139.2, 135.67, 133.1, 133.0, 132.7, 132.4, 130.9, 130.7, 128.5, 128.4, 128.1, 127.9, 127.13, 127.10, 126.9, 125.9, 125.2, 123.5, 101.7, 100.2. **HRMS (ESI)** m/z: calcd. for C₂₁H₁₂IN⁸⁰SeNa [M+Na]⁺: 507.9072; Found: 507.9054.

3g was prepared following the General Procedure A'

The reaction of **1g** (52.8 mg, 0.10 mmol, 1.0 equiv), CuI (1.0 mg, 0.005 mmol) and **L11** (5.0 mg, 0.01 mmol), potassium selenocyanate (28.8 mg, 0.20 mmol, 2.0 equiv) in anhydrous dichloromethane (2.5 mL) and water (0.5 mL) at room temperature for 12 h afforded **3g** as a white solid (43.3 mg, 89%, 94% ee) (eluent for column chromatography PE/EtOAc = 20:1).



3h was prepared following the General Procedure A

The reaction of **1h** (96.9 mg, 0.20 mmol, 1.0 equiv), CuI (1.9 mg, 0.01 mmol) and **L11** (10.1 mg, 0.02 mmol), potassium selenocyanate (57.6 mg, 0.40 mmol, 2.0 equiv) in anhydrous dichloromethane (5.0 mL) and deuterium oxide (1.0 mL) at room temperature for 12 h afforded **3h** as a white solid (79.1 mg, 90%, 94% ee) (eluent for column chromatography PE/EtOAc = 50:1). **HPLC conditions**: Chiralpak AD-H, isopropanol/hexane = 0.5:99.5, flow: 1.0 mL/min, λ = 230 nm, t_r = 8.402 min (major), 9.129 min (minor). [α]²⁰_D = -21.8 (c 0.50, CH₂Cl₂). ¹H **NMR (400 MHz, CDCl₃)** δ 7.70 (d, *J* = 8.0 Hz, 1H), 7.61 (d, *J* = 8.0 Hz, 1H), 7.25 (d, *J* = 8.0 Hz, 1H), 6.96 (d, *J* = 8.0 Hz, 1H), 2.35 (s, 3H), 2.30 (s, 3H), 1.97 (s, 3H), 1.87 (s, 3H). ¹³C **NMR (101 MHz, CDCl₃)** δ 143.4, 142.3, 138.0, 137.9, 137.1, 136.6, 136.5, 132.0, 131.2, 127.7, 121.7, 102.3, 97.4, 20.4, 20.3, 17.8, 16.3. **HRMS (ESI)** m/z: calcd. for C₁₇H₁₆IN⁸⁰SeNa [M+Na]⁺: 463.9385; Found: 463.9383.

3h was prepared following the General Procedure A'

The reaction of **1h** (48.4 mg, 0.10 mmol, 1.0 equiv), CuI (1.0 mg, 0.005 mmol) and L**11** (5.0 mg, 0.01 mmol), potassium selenocyanate (28.8 mg, 0.20 mmol, 2.0 equiv) in anhydrous dichloromethane (2.5 mL) and water (0.5 mL) at room temperature for 12 h afforded **3h** as a white solid (41.0 mg, 93%, 86% ee) (eluent for column chromatography PE/EtOAc = 50:1).



3i was prepared following the General Procedure A

The reaction of **1i** (96.8 mg, 0.20 mmol, 1.0 equiv), CuI (1.9 mg, 0.01 mmol) and **L11** (10.1 mg, 0.02 mmol), potassium selenocyanate (57.6 mg, 0.40 mmol, 2.0 equiv) in anhydrous dichloromethane (5.0 mL) and deuterium oxide (1.0 mL) at room temperature for 12 h afforded **3i** as a white solid (74.5 mg, 85%, 90% ee) (eluent for column chromatography PE/EtOAc = 50:1). **HPLC conditions**: Chiralpak AD-H, isopropanol/hexane = 0.5:99.5, flow: 1.0 mL/min, λ = 230 nm, t_r = 6.547 min (minor), 8.493 min (major). [α]²⁰_D = -17.0 (c 0.50, CH₂Cl₂). ¹H **NMR (500 MHz, CDCl₃)** δ 7.66 – 7.62 (m, 1H), 7.53 – 7.49 (m, 1H), 7.15 – 7.02 (m, 2H), 2.41 (s, 3H), 2.33 (s, 3H), 2.02 (s, 3H), 1.93 (s, 3H). ¹³C **NMR (126 MHz, CDCl₃)** δ 140.7, 140.0, 139.5, 138.8, 138.1, 137.7, 137.6, 131.5, 131.3, 128.0, 124.8, 102.3, 101.1, 21.3, 21.2, 20.7, 19.6. **HRMS (ESI)** m/z: calcd. for C₁₇H₁₇IN⁸⁰Se [M+H]⁺: 441.9565; Found: 441.9565.

3i was prepared following the General Procedure A'

The reaction of **1i** (48.4 mg, 0.10 mmol, 1.0 equiv), CuI (1.0 mg, 0.005 mmol) and **L11** (5.0 mg, 0.01 mmol), potassium selenocyanate (28.8 mg, 0.20 mmol, 2.0 equiv) in anhydrous dichloromethane (2.5 mL) and water (0.5 mL) at room temperature for 12 h afforded **3i** as a white solid (39.5 mg, 90%, 86% ee) (eluent for column chromatography PE/EtOAc = 50:1).



3j was prepared following the General Procedure A

The reaction of **1j** (52.5 mg, 0.10 mmol, 1.0 equiv), CuI (1.0 mg, 0.005 mmol) and **L11** (5.0 mg, 0.01 mmol), potassium selenocyanate (28.8 mg, 0.20 mmol, 2.0 equiv) in anhydrous dichloromethane (2.5 mL) and deuterium oxide (0.5 mL) at room temperature for 12 h afforded **3j** as a white solid (40.1 mg, 83%, 96% ee) (eluent for column chromatography PE/EtOAc = 50:1). **HPLC conditions**: Chiralpak AD-H, isopropanol/hexane = 0.5:99.5, flow: 1.0 mL/min, λ = 230 nm, t_r = 8.696 min (minor), 16.332 min (major). [α]²⁰_D = -14.8 (c 0.50, CH₂Cl₂). ¹H **NMR (500 MHz, CDCl₃)** δ 7.83 (d, *J* = 2.0 Hz, 1H), 7.72 (d, *J* = 2.0 Hz, 1H), 7.36 – 7.31 (m, 2H), 2.05 (s, 3H), 1.96 (s, 3H). ¹³C **NMR (126 MHz, CDCl₃)** δ 139.9, 139.6, 139.43, 139.36, 136.9, 135.84, 135.81, 130.9, 130.8, 127.7, 126.2, 100.8, 100.6, 21.2, 19.6. **HRMS (ESI)** m/z: calcd. for C₁₅H₁₁Cl₂IN⁸⁰Se [M+H]⁺: 481.8473; Found: 481.8496.



3k was prepared following the General Procedure A

The reaction of **1k** (98.2 mg, 0.20 mmol, 1.0 equiv), CuI (1.9 mg, 0.01 mmol) and **L11** (10.1 mg, 0.02 mmol), potassium selenocyanate (57.6 mg, 0.40 mmol, 2.0 equiv) in anhydrous dichloromethane (5.0 mL) and deuterium oxide (1.0 mL) at room temperature for 12 h afforded **3k** as a white solid (73.5 mg, 82%, 92% ee) (eluent for column chromatography PE/EtOAc = 50:1). **HPLC conditions**: Chiralpak AD-H, isopropanol/hexane = 0.5:99.5, flow: 1.0 mL/min, λ = 254 nm, t_r = 10.303 min (minor), 14.402 min (major). [α]²⁰_D = -8.1 (c 0.50, CH₂Cl₂). ¹H **NMR (500 MHz, CDCl₃)** δ 7.56 (dd, *J* = 7.5, 2.5 Hz, 1H), 7.48 (dd, *J* = 8.0, 2.5 Hz, 1H), 7.06 (ddd, *J* = 13.5, 9.0, 2.5 Hz, 2H), 2.07 (s, 3H), 1.97 (s, 3H). ¹⁹F **NMR (471 MHz, CDCl₃)** δ -110.13, -110.43. ¹³C **NMR (126 MHz, CDCl₃)** δ 162.8 (d, *J* = 253.1 Hz), 162.2 (d, *J* = 254.8 Hz), 140.5 (d, *J* = 8.3 Hz), 140.3 (d, *J* = 8.2 Hz), 137.3 (d, *J* = 3.5 Hz), 136.9 (d, *J* = 3.3 Hz), 126.8 (d, *J* = 8.7 Hz), 124.64(d, *J* = 23.7 Hz), 117.9 (d, *J* = 20.9 Hz), 117.5 (d, *J* = 21.2 Hz), 114.9 (d, *J* = 25.5 Hz), 101.0, 100. 8 (d, *J* = 8.5 Hz) 21.52, 21.51, 19.83, 19.82. **HRMS (ESI)** m/z: calcd. for C₁₅H₁₁F₂IN⁸⁰Se [M+H]⁺: 449.9064; Found: 449.9066.



31 and 31' was prepared following the General Procedure A

The reaction of **11** (98.1 mg, 0.20 mmol, 1.0 equiv), CuI (1.9 mg, 0.01 mmol) and **L11** (10.1 mg, 0.01 mmol), potassium selenocyanate (57.6 mg, 0.40 mmol, 2.0 equiv) in anhydrous dichloromethane (5.0 mL) and deuterium oxide (1.0 mL) at room temperature for 12 h afforded **31** and **31**' as a pale-yellow solid (78.2 mg, 88%, 1:5 dr) (eluent for column chromatography PE/EtOAc = 50:1). A small amount of pure compound can be obtained by re-crystallization from CH₂Cl₂. Date for major isomer: ¹H NMR (500 MHz, CDCl₃) δ 7.77 – 7.72 (m, 1H), 7.45 (d, *J* = 8.0 Hz, 1H), 7.37 (t, *J* = 7.5 Hz, 1H), 7.35 – 7.32 (m, 1H), 7.27 (d, *J* = 7.5 Hz, 1H), 2.04 (s, 3H), 1.97 (s, 3H). ¹³C NMR (126 MHz, CDCl₃) δ 144.7, 143.4, 137.6, 137.5, 136.2, 131.7, 130.8, 130.1, 129.5, 128.6, 124.3, 104.9, 101.5, 20.9, 19.6. HRMS (ESI) m/z: calcd. for C₁₅H₁₁CIIN⁸⁰SeNa [M+Na]⁺: 469.8682; Found: 469.8676.

General procedure for Cu-catalyzed coupling of cyclic dianyliodonium, and potassium selenocyanate in CH₂Cl₂ (General Procedure B)



Under nitrogen atmosphere, a Schlenk tube containing a stirring bar was charged with CuI (1.9 mg, 0.01 mmol), L2 (7.2 mg, 0.02 mmol), and cyclic diaryliodonium salt 1 (0.20 mmol, 1.0 equiv), in anhydrous dichloromethane (5.0 mL), the resulting solution was stirred for 30 min. Potassium selenocyanate 2 (0.40 mmol, 2.0 equiv) was added and the resulting mixture was stirred at room temperature for 12 h. After completing the consumption of starting materials, the mixture was filtered through a plug of celite with ethyl acetate. The filtrate was concentrated under reduced pressure, and the residue was purified by column chromatography on silica gel to deliver the corresponding products 4 and 3.



4a was prepared following the General Procedure A

The reaction of **1a** (91.2 mg, 0.20 mmol, 1.0 equiv), CuI (1.9 mg, 0.01 mmol) and **L2** (7.2 mg, 0.02 mmol), potassium selenocyanate (57.6 mg, 0.40 mmol, 2.0 equiv), in dry CH₂Cl₂ (5.0 mL) at room temperature for 12 h afforded **4a** as a white solid (45.3 mg, 55%, 94% ee) and **3a** as a pale-yellow solid (30.0 mg, 36%) (eluent for column chromatography PE/EtOAc = 100:1). Analytic Data for **4a**: **HPLC conditions**: Chiralpak OD-3, isopropanol/hexane = 0.1:99.9, flow: 0.4 mL/min, λ = 230 nm, t_r = 18.366 min (minor), 19.937 min (major). [α]²⁰_D = +35.8 (c 0.125, CH₂Cl₂). ¹**H NMR (500 MHz, CDCl₃)** δ 7.81 (d, *J* = 8.0 Hz, 1H), 7.35 – 7.26 (m, 3H), 7.19 (dd, *J* = 7.5, 2.0 Hz, 1H), 7.05 (t, *J* = 7.5 Hz, 1H), 2.02 (d, *J* = 5.0 Hz, 6H). ¹³**C NMR (126 MHz, CDCl₃)** δ 141.8, 140.9, 138.0, 137.9, 136.9, 130.2, 130.1, 129.9, 128.7, 123.1, 100.4, 21.2, 19.8. **HRMS (ESI)** m/z: calcd. for C₁₅H₁₃IN⁸⁰Se [M+H]⁺: 413.9252; Found: 413.9268.



4b was prepared following the General Procedure B

The reaction of **1b** (48.4 mg, 0.10 mmol, 1.0 equiv), CuI (1.9 mg, 0.01 mmol) and **L2** (7.2 mg, 0.02 mmol), potassium selenocyanate (28.8 mg, 0.20 mmol, 2.0 equiv), in dry CH₂Cl₂ (2.5 mL) at room temperature for 12 h afforded **3b** as a pale-yellow solid (18.0 mg, 41%, 80% ee) and **3b** as a pale-yellow solid (19.8 mg, 45%) (eluent for column chromatography PE/EtOAc = 100:1). Analytic Data for **4b**: **HPLC conditions**: Chiralpak OD-3, isopropanol/hexane = 0.1:99.9, flow: 0.4 mL/min, $\lambda = 230$ nm, t_r = 22.661 min (minor), 23.639 min (major). [α]²⁰_D = +11.8 (c 0.50, CH₂Cl₂). ¹H NMR (**600 MHz, CDCl₃**) δ 7.83 (d, *J* = 7.8 Hz, 1H), 7.41 – 7.32 (m, 3H), 7.20 (d, *J* = 7.2 Hz, 1H), 7.11 (t, *J* = 7.8 Hz, 1H), 2.37 – 2.22 (m, 4H), 1.14 (t, *J* = 7.8 Hz, 3H), 1.08 (t, *J* = 7.8 Hz, 3H). ¹³C NMR (**151 MHz, CDCl₃**) δ 144.2, 143.5, 141.3, 140.0, 136.9, 131.6, 130.3, 129.4, 128.9, 128.4, 127.8, 123.0, 101.4, 27.6, 26.1, 14.5, 13.9. HRMS (**ESI**) m/z: calcd. for C₁₇H₁₆IN⁸⁰SeNa [M+Na]⁺: 463.9385; Found: 463.9346.



4c was prepared following the General Procedure B

The reaction of **1c** (99.4 mg, 0.20 mmol, 1.0 equiv), CuI (1.9 mg, 0.01 mmol) and **L2** (7.2 mg, 0.02 mmol), potassium selenocyanate (57.6 mg, 0.40 mmol, 2.0 equiv), in dry CH₂Cl₂ (5.0 mL) at room temperature for 12 h afforded **4c** as a white solid (45.4 mg, 50%, 96% ee) and **3c** as a white solid (33.6 mg, 37%) (eluent for column chromatography PE/EtOAc = 100:1). There is no separation of **4c** for HPLC on various chiral columns. The ee value of compound **4c** was determined by transferring **4c** to its corresponding selenourea (see below). Analytic Data for **4c**: $[\alpha]_D^{20} = +9.5$ (c 0.50, CH₂Cl₂). ¹H NMR (**500 MHz, CDCl₃**) δ 7.90 (dd, J = 8.0, 1.0 Hz, 1H), 7.54 (dd, J = 8.0, 1.0 Hz, 1H), 7.49 (dd, J = 8.0, 1.0 Hz, 1H), 7.39 (t, J = 8.0 Hz, 1H), 7.28 (d, J = 1.0 Hz, 1H), 7.12 (t, J = 8.0 Hz, 1H). ¹³C NMR (**126 MHz, CDCl₃**) δ 139.6, 138.8, 137.7, 134.8, 134.1, 131.6, 130.2, 129.6, 129.0, 123.9, 100.2. HRMS (ESI) m/z: calcd. for C₁₃H₆Cl₂IN⁸⁰SeNa [M+Na]⁺: 475.7979; Found: 475.7971.



Under nitrogen atmosphere, a Schlenk tube containing a stirring bar was charged with 4c (11.4 mg, 0.025 mmol) in anhydrous CH₂Cl₂ (1.0 mL) followed by the addition of benzylamine (0.050 mmol, 2.0 equiv). The solution was stirred at room temperature for 2 h. After completing the

consumption of starting materials, the solution was concentrated under reduced pressure, and the residue was purified by column chromatography on silica gel to deliver the corresponding product **6d** as a white solid (9.4 mg, 67%, 96% ee) (eluent for column chromatography PE/EtOAc = 5:1). Analytic Data for **6d**: **HPLC conditions**: Chiralpak AD-H, isopropanol/hexane = 25:75, flow: 1.0 mL/min, $\lambda = 254$ nm, t_r = 8.491 min (major), 9.314 min (minor). [α]²⁰_D = +15.2 (c 0.50, CH₂Cl₂). ¹H **NMR (500 MHz, CDCl₃)** δ 7.87 (d, *J* = 8.0 Hz, 1H), 7.51 (dd, *J* = 8.0, 2.5 Hz, 2H), 7.43 (t, *J* = 7.9 Hz, 2H), 7.37 – 7.23 (m, 6H), 7.11 (t, *J* = 8.0 Hz, 1H), 6.49 (s, 1H), 4.90 (s, 2H). ¹³C **NMR (126 MHz, CDCl₃)** δ 138.8, 138.4, 138.1, 136.3, 133.2, 131.6, 130.9, 129.9, 129.5, 128.8, 128.0, 127.9, 125.2, 99.8, 52.6. **HRMS (ESI)** m/z: calcd. for C₂₀H₁₆Cl₂IN₂⁸⁰Se [M+H]⁺: 560.8895; Found: 560.8886.



4d was prepared following the General Procedure B

The reaction of **1d** (117.2 mg, 0.20 mmol, 1.0 equiv), CuI (1.9 mg, 0.01 mmol) and **L2** (7.2 mg, 0.02 mmol), potassium selenocyanate (57.6 mg, 0.40 mmol, 2.0 equiv), in dry CH₂Cl₂ (5.0 mL) at room temperature for 12 h afforded **4d** as a white solid (47.6 mg, 44%, 98% ee) and **3d** as a white solid (50.8 mg, 47%) (eluent for column chromatography PE/EtOAc = 100:1). Analytic Data for **4d**: **HPLC conditions**: Chiralpak AD-3, isopropanol/hexane = 0.5:99.5, flow: 0.5 mL/min, λ = 230 nm, t_r = 18.336 min (major), 19.908 min (minor). [α]²⁰_D = -9.7 (c 0.50, CH₂Cl₂). ¹H NMR (500 MHz, CDCl₃) δ 7.94 (dd, *J* = 8.0, 1.0 Hz, 1H), 7.72 (dd, *J* = 8.0, 1.0 Hz, 1H), 7.67 (dd, *J* = 6.5, 3.0 Hz, 1H), 7.35 - 7.26 (m, 2H), 7.03 (t, *J* = 8.0 Hz, 1H). ¹³C NMR (101 MHz, CDCl₃) δ 142.0, 138.4, 132.8, 132.1, 131.8, 130.4, 124.5, 124.4, 123.4, 99.8. HRMS (ESI) m/z: calcd. for C₁₃H₇Br₂IN⁸⁰Se [M+H]⁺: 541.7150; Found: 541.7184.



4e was prepared following the General Procedure B

The reaction of **1e** (52.8 mg, 0.10 mmol, 1.0 equiv), CuI (1.0 mg, 0.005 mmol) and **L2** (3.6 mg, 0.01 mmol), potassium selenocyanate (28.8 mg, 0.20 mmol, 2.0 equiv), in dry CH₂Cl₂ (2.5 mL) at room temperature for 12 h afforded **4e** as a white solid (25.0 mg, 52%, 99% ee) and **3e** as a white solid (16.3 mg, 34%) (eluent for column chromatography PE/EtOAc = 100:1). Analytic Data for **4e**: **HPLC conditions**: Chiralpak AD-H, isopropanol/hexane = 1:99, flow: 1.0 mL/min, λ = 254 nm, t_r = 8.954 min (major), 11.419 min (minor). [α]²⁰_D = -7.9 (c 0.50, CH₂Cl₂). ¹H NMR (400 MHz, CDCl₃) δ 8.07 (d, *J* = 8.8 Hz, 1H), 8.01 (d, *J* = 8.8 Hz, 1H), 7.95 (dd, *J* = 8.0, 5.2 Hz, 2H), 7.76 (d, *J* = 8.4 Hz, 1H), 7.58 – 7.46 (m, 3H), 7.41 – 7.33 (m, 1H), 7.33 – 7.25 (m, 1H), 7.15 (d, *J* = 8.4, 1H), 7.05 (d, *J* = 8.4, 1H), 1³C NMR (101 MHz, CDCl₃) δ 138.4, 138.1, 135.5, 133.4, 132.8, 132.3, 132.1, 130.4, 130.0, 128.5, 128.3, 127.9, 127.5, 127.2, 126.7, 125.90, 125.87, 123.1, 99.8. HRMS (ESI) m/z: calcd. for C₂₁H₁₃IN⁸⁰Se [M+H]⁺: 485.9252; Found: 485.9272.



4f was prepared following the General Procedure B

The reaction of **1f** (48.4 mg, 0.1 mmol, 1.0 equiv), CuI (1.0 mg, 0.005 mmol) and **L2** (3.6 mg, 0.01 mmol), potassium selenocyanate (28.8 mg, 0.20 mmol, 2.0 equiv), in dry CH₂Cl₂ (2.5 mL) at room temperature for 12 h afforded **4f** as a white solid (23.1 mg, 48%, 98% ee) and **3f** as a white solid (21.2 mg, 44%) (eluent for column chromatography PE/EtOAc = 100:1). Analytic Data for **4f**: **HPLC conditions**: Chiralpak OD-3, isopropanol/hexane = 0.1:99.9, flow: 0.4 mL/min, λ = 230 nm, t_r = 18.992 min (minor), 19.910 min (major). [α]²⁰_D = -15.2 (c 0.50, CH₂Cl₂). ¹H NMR (400 MHz, **CDCl₃**) δ 7.70 (d, *J* = 8.0 Hz, 1H), 7.18 (d, *J* = 8.0 Hz, 1H), 7.10 (d, *J* = 8.0 Hz, 1H), 6.94 (d, *J* = 8.0 Hz, 1H), 2.34 (s, 3H), 2.30 (s, 3H), 1.93 (s, 3H), 1.91 (s, 3H). ¹³C NMR (101 MHz, CDCl₃) δ 142.4, 141.0, 137.6, 137.5, 136.7, 136.4, 136.2, 131.5, 129.8, 122.6, 97.0, 20.6, 20.4, 17.8, 16.5. HRMS (ESI) m/z: calcd. for C₁₇H₁₇IN⁸⁰Se [M+H]⁺: 441.9565; Found: 441.9579.

4. Synthetic applications

General procedure of selenocyanate with Grignard reagent (General Procedure C)



Under nitrogen atmosphere, a Schlenk tube containing a stirring bar was charged with **3a** (41.2 mg, 0.10 mmol) in anhydrous tetrahydrofuran (2.0 mL), Grignard reagent (0.20 mmol, 2.0 equiv) was added and the resulting mixture was stirred at room temperature for 2 h. After completing the consumption of starting materials, the mixture was filtered through a plug of celite with ethyl acetate. The filtrate was concentrated under reduced pressure, and the residue was purified by column chromatography on silica gel to deliver the corresponding product **5**.



5a was prepared following the General Procedure C

The reaction of **3a** (90% ee, 41.2 mg, 0.10 mmol, 1.0 equiv) and phenylmagnesium bromide (1.0 mol/L in THF, 0.20 mL, 2.0 equiv) in anhydrous tetrahydrofuran (2.0 mL) at room temperature for 2 h afforded **5a** as a pale-yellow solid (45.8 mg, 99%, 90% ee) (eluent for column chromatography PE/EtOAc = 100:1). **HPLC conditions**: Chiralpak OD-3, isopropanol/hexane =0.5:99.5, flow: 0.6

mL/min, $\lambda = 254$ nm, t_r = 10.457 min (major), 11.240 min (minor). $[\alpha]_D^{20} = -23.3$ (c 0.50, CH₂Cl₂). ¹H NMR (500 MHz, CDCl₃) δ 7.84 (d, J = 8.0 Hz, 1H), 7.69 – 7.58 (m, 2H), 7.40 – 7.27 (m, 4H), 7.17 – 7.08 (m, 2H), 7.02 (t, J = 8.0 Hz, 1H), 6.96 (dd, J = 7.5, 1.5 Hz, 1H), 2.10 (s, 3H), 1.97 (s, 3H). ¹³C NMR (126 MHz, CDCl₃) δ 144.0, 143.1, 138.2, 136.9, 136.4, 135.80, 135.75, 134.3, 130.1, 129.5, 129.4, 129.2, 128.5, 128.2, 127.9, 127.8, 101.3, 21.4, 19.9. HRMS (ESI) m/z: calcd. for C₂₀H₁₇I⁸⁰SeNa [M+Na]⁺: 486.9432; Found: 486.9448.



5b was prepared following the General Procedure C

The reaction of **3a** (90% ee, 41.2 mg, 0.10 mmol, 1.0 equiv) and *p*-tolylmagnesium bromide (1.0 mol/L in THF, 0.20 mL, 2.0 equiv) in anhydrous tetrahydrofuran (2.0 mL) at room temperature for 2 h afforded **5b** as a pale-yellow solid (41.5 mg, 87%, 90% ee) (eluent for column chromatography PE/EtOAc = 100:1). **HPLC conditions**: Chiralpak OD-3, isopropanol/hexane = 0.5:99.5, flow: 0.6 mL/min, $\lambda = 230$ nm, t_r = 31.937 min (major), 44.652 min (minor). [α]_D²⁰ = -17.3 (c 0.50, CH₂Cl₂). ¹H **NMR (500 MHz, CDCl₃)** δ 7.83 (d, *J* = 8.0 Hz, 1H), 7.58 – 7.43 (m, 2H), 7.29 (dt, *J* = 7.5, 1.0 Hz, 1H), 7.19 – 7.09 (m, 2H), 7.11 – 7.04 (m, 2H), 7.02 (t, *J* = 7.5 Hz, 1H), 6.89 (dd, *J* = 7.0, 2.0 Hz, 1H), 2.36 (s, 3H), 2.11 (s, 3H), 1.96 (s, 3H). ¹³C **NMR (126 MHz, CDCl₃)** δ 144.0, 142.7, 138.4, 138.2, 136.9, 136.24, 136.18, 134.9, 130.3, 130.1, 129.5, 128.4, 127.6, 127.2, 125.2, 101.3, 21.4, 21.3, 19.9. **HRMS (ESI)** m/z: calcd. for C₂₁H₂₀I⁸⁰Se [M+H]⁺: 478.9769; Found: 478.9759.



5c was prepared following the General Procedure C

The reaction of **3a** (90% ee, 41.2 mg, 0.10 mmol, 1.0 equiv) and *p*-methoxyphenylmagnesium bromide (1.0 mol/L in THF, 0.20 mL, 2.0 equiv) in anhydrous tetrahydrofuran (2.0 mL) at room temperature for 2 h afforded **5c** as a pale-yellow solid (44.4 mg, 90%, 90% ee) (eluent for column chromatography PE/EtOAc = 100:1). **HPLC conditions**: Chiralpak OD-3, isopropanol/hexane = 0.5:99.5, flow: 0.6 mL/min, $\lambda = 230$ nm, t_r = 18.457 min (major), 22.670 min (minor). $[\alpha]_D^{20} = -14.6$ (c 0.50, CH₂Cl₂). ¹**H NMR (500 MHz, CDCl₃)** δ 8.02 – 7.73 (m, 1H), 7.58 – 7.52 (m, 2H), 7.33 – 7.28 (m, 1H), 7.13 – 7.05 (m, 2H), 7.02 (t, *J* = 8.0 Hz, 1H), 6.96 – 6.66 (m, 2H), 6.85 – 6.81 (m, 1H), 3.83 (s, 3H), 2.12 (s, 3H), 1.96 (s, 3H). ¹³**C NMR (126 MHz, CDCl₃)** δ 160.1, 143.9, 142.3, 138.21, 138.15, 136.9, 136.2, 135.3, 130.1, 129.5, 128.4, 127.4, 126.7, 118.8, 115.1, 101.4, 55.3, 21.4, 19.8. **HRMS (ESI)** m/z: calcd. for C₂₁H₁₉OI⁸⁰SeNa [M+ Na]⁺: 516.9538; Found: 516.9579.



5d was prepared following the General Procedure C

The reaction of **3a** (90% ee, 41.2 mg, 0.10 mmol, 1.0 equiv) and 4-fluorophenylmagnesium bromide (1.0 mol/L in THF, 0.20 mL, 2.0 equiv) in anhydrous tetrahydrofuran (2.0 mL) at room temperature for 2 h afforded **5d** as a pale-yellow solid (46.9 mg, 98%, 90% ee) (eluent for column chromatography PE/EtOAc = 100:1). **HPLC conditions**: Chiralpak OD-3, isopropanol/hexane = 0.5:99.5, flow: 0.6 mL/min, $\lambda = 254$ nm, t_r = 11.127 min (major), 12.479 min (minor). $[\alpha]_D^{20} = -15.8$ (c 0.50, CH₂Cl₂). ¹**H NMR (500 MHz, CDCl₃)** δ 7.83 (d, J = 8.0 Hz, 1H), 7.66 – 7.51 (m, 2H), 7.30 (d, J = 7.4 Hz, 1H), 7.17 – 7.06 (m, 2H), 7.06 – 6.99 (m, 3H), 6.88 (dd, J = 7.5, 1.5 Hz, 1H), 2.09 (s, 3H), 1.97 (s, 3H). ¹⁹**F NMR (471 MHz, CDCl₃)** δ -112.80. ¹³**C NMR (126 MHz, CDCl₃)** δ 163.0 (d, J = 248.8 Hz), 143.8, 142.8, 138.2, 138.1, 136.9, 136.5, 134.3, 130.1, 129.5, 128.5, 127.9, 127.3, 123.62, 123.60, 116.7, 116.6, 101.3, 21.4, 19.9. **HRMS (ESI)** m/z: calcd. for C₂₀H₁₆IF⁸⁰SeNa [M+Na]⁺: 504.9338; Found: 504.9318.



5e was prepared following the General Procedure C

The reaction of **3a** (90% ee, 41.2 mg, 0.10 mmol, 1.0 equiv) and *p*-chlorophenylmagnesium bromide (1.0 mol/L in THF, 0.20 mL, 2.0 equiv) in anhydrous tetrahydrofuran (2.0 mL) at room temperature for 2 h afforded **5e** as a pale-yellow solid (46.8 mg, 95%, 90% ee) (eluent for column chromatography PE/EtOAc = 100:1). **HPLC conditions**: Chiralpak OD-3, isopropanol/hexane = 0.5:99.5, flow: 0.6 mL/min, $\lambda = 254$ nm, t_r = 10.660 min (major), 13.282 min (minor). $[\alpha]_D^{20} = -19.2$ (c 0.50, CH₂Cl₂). ¹**H NMR (500 MHz, CDCl₃)** δ 7.80 (d, J = 8.0 Hz, 1H), 7.52 – 7.44 (m, 2H), 7.28 – 7.20 (m, 3H), 7.14 – 7.06 (m, 2H), 6.99 (t, J = 8.0 Hz, 1H), 6.92 (dd, J = 7.5, 1.5 Hz, 1H), 2.05 (s, 3H), 1.94 (s, 3H). ¹³**C NMR (126 MHz, CDCl₃)** δ 143.9, 143.2, 138.1, 137.0, 136.90, 136.89, 136.6, 134.5, 133.8, 130.1, 129.6, 129.5, 128.6, 128.2, 127.9, 127.5, 101.3, 21.4, 19.9. **HRMS (ESI)** m/z: calcd. for C₂₀H₁₆ICl⁸⁰SeNa [M+Na]⁺: 520.9043; Found: 520.9055.



5f was prepared following the General Procedure C

The reaction of **3a** (90% ee, 41.2 mg, 0.1 mmol, 1.0 equiv) and *p*-tertbutylphenylmagnesium bromide (1.0 mol/L in THF, 0.2 mL, 2.0 equiv) in anhydrous tetrahydrofuran (2.0 mL) at room temperature for 2 h afforded **5f** as a pale-yellow solid (41.5 mg, 80%, 90% ee) (eluent for column

chromatography PE/EtOAc = 100:1). HPLC conditions: Chiralpak OD-3, isopropanol/hexane = 0.3:99.7, flow: 0.4 mL/min, $\lambda = 230$ nm, t_r = 24.145 min (major), 31.098 min (minor). [α]²⁰_D = -14.9 (c 0.50, CH₂Cl₂). ¹H NMR (500 MHz, CDCl₃) δ 7.84 (d, J = 8.0 Hz, 1H), 7.59 – 7.50 (m, 2H), 7.39 – 7.31 (m, 2H), 7.33 – 7.27 (m, 1H), 7.15 – 7.07 (m, 2H), 7.03 (t, J = 8.0 Hz, 1H), 7.00 – 6.95 (m, 1H), 2.11 (s, 3H), 1.97 (s, 3H), 1.34 (s, 9H). ¹³C NMR (126 MHz, CDCl₃) δ 151.5, 144.0, 142.9, 138.2, 136.9, 136.2, 135.7, 134.6, 130.0, 129.4, 128.4, 127.7, 127.6, 126.5, 125.5, 101.4, 34.6, 31.2, 21.4, 19.9. HRMS (ESI) m/z: calcd. for C₂₄H₂₆I⁸⁰Se [M+H]⁺: 521.0239; Found: 521.0250.



5g was prepared following the General Procedure C

The reaction of **3a** (90% ee, 41.2 mg, 0.10 mmol, 1.0 equiv) and *m*-methoxyphenylmagnesium bromide (1.0 mol/L in THF, 0.20 mL, 2.0 equiv) in anhydrous tetrahydrofuran (2.0 mL) at room temperature for 2 h afforded **5g** as a pale-yellow solid (47.3 mg, 96%, 90% ee) (eluent for column chromatography PE/EtOAc = 50:1). **HPLC conditions**: Chiralpak OD-3, isopropanol/hexane = 0.5:99.5, flow: 0.6 mL/min, $\lambda = 254$ nm, t_r = 13.721 min (major), 15.710 min (minor). $[\alpha]_D^{20} = +34.0$ (c 0.50, CH₂Cl₂). ¹H NMR (**500 MHz, CDCl**₃) δ 7.80 (d, J = 8.0 Hz, 1H), 7.26 (d, J = 7.5 Hz, 1H), 7.23 – 7.10 (m, 3H), 7.12 – 7.02 (m, 2H), 7.04 – 6.90 (m, 2H), 6.88 – 6.82 (m, 1H), 3.74 (s, 3H), 2.07 (s, 3H), 1.94 (s, 3H). ¹³C NMR (126 MHz, CDCl₃) δ 159.8, 144.0, 143.1, 138.1, 136.8, 136.3, 134.0, 130.11, 130.06, 130.0, 129.4, 128.5, 128.1, 128.0, 127.8, 120.6, 114.2, 101.3, 55.3, 21.4, 19.9. HRMS (ESI) m/z: calcd. for C₂₁H₁₉OI⁸⁰SeNa [M+Na]⁺: 516.9538; Found: 516.9519.



5h was prepared following the General Procedure C

The reaction of **3a** (90% ee, 41.2 mg, 0.10 mmol, 1.0 equiv) and *o*-methoxyphenylmagnesium bromide (1.0 mol/L in THF, 0.20 mL, 2.0 equiv) in anhydrous tetrahydrofuran (2.0 mL) at room temperature for 2 h afforded **5h** as a pale-yellow solid (44.4 mg, 90%, 90% ee) (eluent for column chromatography PE/EtOAc = 50:1). **HPLC conditions**: Chiralpak AD-H, isopropanol/hexane = 0.5:99.5, flow: 1.0 mL/min, $\lambda = 230$ nm, t_r = 7.480 min (minor), 8.326 min (major). [α]_D²⁰ = -23.7 (c 0.50, CH₂Cl₂). ¹H NMR (**500 MHz, Chloroform**-*d*) δ 7.81 (d, *J* = 8.0 Hz, 1H), 7.51 (dd, *J* = 8.0, 2.0 Hz, 1H), 7.35 – 7.23 (m, 2H), 7.15 (dd, *J* = 8.0, 6.5 Hz, 1H), 7.11 (d, *J* = 7.5 Hz, 1H), 7.07 (dd, *J* = 8.0, 1.5 Hz, 1H), 6.99 (t, *J* = 7.5 Hz, 1H), 6.89 – 6.79 (m, 2H), 3.78 (s, 3H), 2.11 (s, 3H), 1.98 (s, 3H). ¹³C NMR (126 MHz, CDCl₃) δ 158.6, 144.4, 144.2, 138.1, 136.8, 136.4, 135.5, 132.5, 130.0, 129.4, 129.3, 129.1, 128.3, 128.2, 121.4, 119.2, 110.7, 101.3, 55.8, 21.4, 20.0. HRMS (ESI) m/z: calcd. for C₂₁H₂₀OI⁸⁰Se [M+H]⁺: 494.9719; Found: 494.9741.



5i was prepared following the General Procedure C

The reaction of **3a** (90% ee, 41.2 mg, 0.10 mmol, 1.0 equiv) and vinylmagnesium bromide (1.0 mol/L in THF, 0.20 mL, 2.0 equiv) in anhydrous tetrahydrofuran (2.0 mL) at room temperature for 2 h afforded **5i** as a pale-yellow solid (37.2 mg, 90%, 90% ee) (eluent for column chromatography PE). **HPLC conditions**: Chiralpak OD-3, isopropanol/hexane = 0.2:99.8, flow: 0.6 mL/min, λ = 254 nm, t_r = 12.130 min (major), 13.377 min (minor). $[\alpha]_D^{20}$ = -39.3 (c 0.50, CH₂Cl₂). ¹H **NMR** (**500 MHz, CDCl₃**) δ 7.81 (d, *J* = 8.0 Hz, 1H), 7.39 (d, *J* = 8.0 Hz, 1H), 7.34 – 7.25 (m, 2H), 7.21 (d, *J* = 7.5 Hz, 1H), 7.02 (t, *J* = 8.0 Hz, 1H), 6.88 (dd, *J* = 17.0, 9.5 Hz, 1H), 5.85 (d, *J* = 9.5 Hz, 1H), 5.71 (d, *J* = 17.0 Hz, 1H), 2.06 (s, 3H), 1.98 (s, 3H). ¹³C **NMR (126 MHz, CDCl₃)** δ 144.1, 143.8, 138.0, 136.8, 136.6, 131.9, 130.0, 129.4, 128.5, 128.4, 127.9, 126.6, 122.0, 101.2, 21.4, 20.0. **HRMS (ESI)** m/z: calcd. for C₁₆H₁₆I⁸⁰Se [M+H]⁺: 414.9456; Found: 414.9451.



5j was prepared following the General Procedure C

The reaction of **3a** (90% ee, 41.2 mg, 0.10 mmol, 1.0 equiv) and prop-1-ea-2-ylmagnesium bromide (1.0 mol/L in THF, 0.20 mL, 2.0 equiv) in anhydrous tetrahydrofuran (2.0 mL) at room temperature for 2 h afforded **5j** as a pale-yellow solid (29.0 mg, 68%, 90% ee) (eluent for column chromatography PE). **HPLC conditions**: Chiralpak OD-3, isopropanol/hexane = 0:100, flow: 0.4 mL/min, $\lambda = 230$ nm, t_r = 22.831 min (major), 24.599 min (minor). [α]_D²⁰ = -21.9 (c 2.00, CH₂Cl₂). ¹**H NMR (500 MHz, CDCl₃)** δ 7.82 – 7.77 (m, 1H), 7.38 (dd, J = 7.5, 1.5 Hz, 1H), 7.29 – 7.21 (m, 2H), 7.21 – 7.16 (m, 1H), 6.99 (t, J = 7.5 Hz, 1H), 5.62 (d, J = 1.5 Hz, 1H), 5.45 (d, J = 1.0 Hz, 1H), 2.15 (t, J = 1.0 Hz, 3H), 2.05 (s, 3H), 1.96 (s, 3H). ¹³**C NMR (126 MHz, CDCl₃)** δ 144.50, 144.47, 137.9, 136.7, 136.6, 135.9, 131.3, 129.9, 129.30, 129.26, 128.4, 128.3, 122.8, 101.2, 26.1, 21.4, 20.1. **HRMS (ESI)** m/z: calcd. for C₁₇H₁₈I⁸⁰Se [M+H]⁺: 428.9613; Found: 428.9620.



5k was prepared following the General Procedure C

The reaction of **3a** (90% ee, 41.2 mg, 0.10 mmol, 1.0 equiv) and ethynylmagnesium chloride (1.0 mol/L in THF, 0.20 mL, 2.0 equiv) in anhydrous tetrahydrofuran (2.0 mL) at room temperature for 2 h afforded **5k** as a colorless oil (26.7 mg, 65%, 90% ee) (eluent for column chromatography PE). **HPLC conditions**: Chiralpak OD-3, isopropanol/hexane = 0.2:99.8, flow: 0.6 mL/min, λ = 230 nm,

t_r = 25.834 min (major), 30.195 min (minor). $[\alpha]_D^{20}$ = -21.5 (c 0.50, CH₂Cl₂). ¹H NMR (500 MHz, CDCl₃) δ 7.84 – 7.79 (m, 1H), 7.79 – 7.75 (m, 1H), 7.32 (t, *J* = 8.0 Hz, 1H), 7.29 (dt, *J* = 7.5, 1.0 Hz, 1H), 7.21 (dt, *J* = 7.5, 1.0 Hz, 1H), 7.03 (t, *J* = 8.0 Hz, 1H), 3.25 (s, 1H), 2.07 (s, 3H), 1.97 (s, 3H). ¹³C NMR (126 MHz, CDCl₃) δ 142.2, 141.5, 138.4, 137.2, 136.6, 130.3, 130.1, 129.1, 128.9, 128.6, 125.8, 100.9, 92.8, 64.9, 21.2, 19.5. HRMS (ESI) m/z: calcd. for C₁₆H₁₄I⁸⁰Se [M+H]⁺: 412.9300; Found: 412.9294.



51 was prepared following the General Procedure C

The reaction of **3a** (90% ee, 41.2 mg, 0.10 mmol, 1.0 equiv) and cydopropylmagnesium bromide (1.0 mol/L in THF, 0.20 mL, 2.0 equiv) in anhydrous tetrahydrofuran (2.0 mL) at room temperature for 2 h afforded **5l** as a colorless oil (41.2 mg, 96%, 90% ee) (eluent for column chromatography PE). **HPLC conditions**: Chiralpak OD-3, isopropanol/hexane = 0.5:99.5, flow: 0.6 mL/min, λ = 254 nm, t_r = 9.721 min (minor), 10.297 min (major). [α]²⁰_D = -31.6 (c 0.50, CH₂Cl₂). ¹H **NMR (500 MHz, CDCl₃)** δ 7.83 – 7.72 (m, 1H), 7.60 (d, *J* = 7.5 Hz, 1H), 7.32 – 7.20 (m, 2H), 7.13 (dd, *J* = 7.5, 1.0 Hz, 1H), 6.99 (t, *J* = 8.0 Hz, 1H), 2.27 – 2.10 (m, 1H), 2.04 (s, 3H), 1.94 (s, 3H), 1.17 – 1.07 (m, 2H), 0.74 – 0.58 (m, 2H). ¹³C **NMR (126 MHz, CDCl₃)** δ 143.8, 142.5, 138.2, 136.9, 136.1, 134.3, 130.1, 129.5, 128.4, 127.1, 125.9, 101.3, 21.3, 19.8, 7.9, 7.7, 5.3. **HRMS (ESI)** m/z: calcd. for C₁₇H₁₈I⁸⁰Se [M+H]⁺: 428.9613; Found: 428.9623.



A reported procedure was followed with some modifications.⁵ In a 25 mL round-bottom flask, a mixture of **3a** (90% ee, 41.2 mg, 0.10 mmol, 1.0 equiv) and Cs₂CO₃ (130.3 mg, 0.40 mmol, 4.0 equiv) was dissolved in CH₃CN (2.0 ml) and cooled to 0 °C. Then TMSCF₃ (56.9 mg, 0.40 mmol, 4.0 equiv) was added at once via syringe and the mixture was then stirred at ambient temperature for 2 h. The resulting mixture was filtered through a short pad of celite and extracted with CH₂Cl₂ (3×5.0 mL). The resulting organic solution was washed with water (10 mL) and brine (10 mL). The organic layer was dried over anhydrous MgSO₄, filtered and concentrated. The residue was purified by column chromatography to give the corresponding products **5m** a colorless oil (30.2 mg, 66%, 88% ee) (eluent for column chromatography PE). **HPLC conditions**: Chiralpak OD-3, isopropanol/hexane = 0:100, flow: 0.6 mL/min, $\lambda = 254$ nm, t_r = 10.606 min (major), 11.678 min (minor). [**α**]²⁰_D = -45.3 (c 0.50, CH₂Cl₂). ¹H **NMR (500 MHz, CDCl₃)** δ 7.78 (d, *J* = 8.0 Hz, 1H), 7.74 – 7.69 (m, 1H), 7.38 – 7.33 (m, 2H), 7.30 – 7.25 (m, 1H), 7.02 (t, *J* = 8.0 Hz, 1H), 2.02 (s, 3H), 1.99 (s, 3H). ¹⁹F **NMR (471 MHz, CDCl₃)** δ -34.06. ¹³C **NMR (126 MHz, CDCl₃)** δ 146.5, 144.0, 137.8, 137.5, 136.6, 132.7, 131.3, 130.1, 129.7, 128.9, 125.4, 122.6 (q, *J* = 333.4 Hz) 101.2, 21.1, 20.3. **HRMS (ESI)** m/z: calcd. for C₁₅H₁₃F₃I⁸⁰Se [M+H]⁺: 456.9174; Found: 456.9172.



A reported procedure was followed with some modifications.⁶ To a solution of **3a** (90% ee, 206.1 mg, 0.50 mmol, 1.0 equiv) in MeOH (5.0 mL) was added NaBH₄ (37.8 mg, 1.0 mmol, 2.0 equiv) at 0 °C. After the mixture had stirred at 0 °C for 30 min, water (10 mL) was added in one portion. Subsequently, the mixture was stirred at room temperature for 2 h under air; a white solid formed. **DS** as a pale-yellow solid (143.0 mg, 93%, dr = 1:9) was collected by filtration and no further purification was carried out. $[\alpha]_D^{20} = -24.1$ (c 0.50, CH₂Cl₂). ¹H NMR (500 MHz, CDCl₃) δ 7.84 (d, J = 8.0 Hz, 1H), 7.64 (dd, J = 8.0, 1.5 Hz, 1H), 7.29 (d, J = 7.5 Hz, 1H), 7.18 – 7.12 (m, 2H), 7.03 (t, J = 7.5 Hz, 1H), 2.06 (s, 3H), 1.96 (s, 3H). ¹³C NMR (126 MHz, CDCl₃) δ 143.1, 142.12, 142.08, 138.54, 138.51, 137.12, 137.09, 136.12, 136.06, 130.29, 130.26, 129.97, 129.91, 129.85, 128.86, 128.82, 128.33, 128.30, 127.01, 126.98, 101.55, 101.51, 21.44, 21.40, 19.38, 19.36. HRMS (ESI) m/z: calcd. for C₂₈H₂₅I⁸⁰Se₂ [M+H]⁺: 774.8371; Found: 774.8372.



A reported procedure was followed with some modifications.⁷ DS (38.6 mg, 0.05 mmol, 1.0 equiv) was added to a round bottom flask followed by addition of dry MeCN (1.0 mL). To this solution, selectfluor (35.4 mg, 0.10 mmol, 2.0 equiv) was added under N₂ atmosphere. After 5 min a solution of 1-methoxy-2-phenylethynyl-benzene (20.8 mg, 0.10 mmol 2.0 equiv) in dry MeCN (1.0 mL) was added to the reaction mixture. The reaction mixture was stirred at room temperature for 12 h. After reaction completion EtOAc (5 mL) and distilled water (5 mL) were added and the aquous layer was washed with EtOAc (3×5 mL), the combined organic phase was dried with Na₂SO₄, filtrated, and then filtrate was concentrated under reduced pressure. The residue was subjected to column chromatography using PE/EtOAc (50/1, v/v) as the eluent to afford **5n** as a pale-yellow solid (45.8 mg, 67%, 86% ee) (eluent for column chromatography PE/EtOAc = 100:1). HPLC conditions: Chiralpak OD-3, isopropanol/hexane = 0.3:99.7, flow: 0.6 mL/min, λ = 254 nm, t_r = 14.579 min (minor), 16.911 min (major). $[\alpha]_D^{20} = -28.4$ (c 0.50, CH₂Cl₂). ¹H NMR (500 MHz, CDCl₃) δ 8.24 – 8.16 (m, 2H), 7.89 (d, J = 8.0 Hz, 1H), 7.62 - 7.55 (m, 2H), 7.46 - 7.34 (m, 5H), 7.29 - 7.20 (m, 1H), 7.11 (d, J = 7.5 Hz, 1H), 7.06 (t, J = 7.5 Hz, 1H), 7.00 (t, J = 7.5 Hz, 1H), 6.81 (d, J = 8.0 Hz, 1H), 2.26 (s, 3H), 2.01 (s, 3H). ¹³C NMR (126 MHz, CDCl₃) δ 158.0, 154.2, 143.2, 141.9, 138.5, 137.2, 136.6, 132.5, 132.1, 130.4, 129.9, 129.8, 129.2, 128.7, 128.4, 127.9, 127.4, 125.2, 124.7, 123.4, 121.3, 111.1, 101.5, 98.7, 21.4, 19.6. **HRMS (ESI)** m/z: calcd. for C₂₈H₂₁IO⁸⁰SeNa [M+Na]⁺: 602.9695; Found: 602.9678.

General reaction of isoselenate with amines (General Procedure D)



Under nitrogen atmosphere, a Schlenk tube containing a stirring bar was charged with **4a** (94% ee, 41.2 mg, 0.10 mmol) in anhydrous CH_2Cl_2 (2.0 mL) followed by the addition of amines (0.20 mmol, 2.0 equiv). The solution was stirred at room temperature for 2 h. After completing the consumption of starting materials, the solution was concentrated under reduced pressure, and the residue was purified by column chromatography on silica gel to deliver the corresponding product **6**.



6a was prepared following the General Procedure D

The reaction of **4a** (94% ee, 41.2 mg, 0.10 mmol, 1.0 equiv), methylamine (40 % in H₂O) (15.5 uL, 0.20 mmol, 2.0 equiv) in anhydrous CH₂Cl₂ (2.0 mL) at room temperature for 2 h afforded **6a** as a white solid (36.1 mg, 82%, 94% ee) (eluent for column chromatography PE/EtOAc = 5:1). **HPLC conditions**: Chiralpak ID, isopropanol/hexane = 20:80, flow: 1.0 mL/min, λ = 254 nm, t_r = 7.443 min (major), 8.828 min (minor). $[\alpha]_D^{20}$ = +15.1 (c 0.50, CH₂Cl₂). ¹H NMR (500 MHz, CDCl₃) δ 7.78 (d, *J* = 8.0 Hz, 1H), 7.38 (t, *J* = 8.0 Hz, 1H), 7.29 (d, *J* = 7.5 Hz, 2H), 7.22 (d, *J* = 8.0 Hz, 2H), 7.01 (t, *J* = 8.0 Hz, 1H), 6.53 (s, 1H), 3.17 (d, *J* = 4.5 Hz, 3H), 1.99 (s, 3H), 1.97 (s, 3H). ¹³C NMR (126 MHz, CDCl₃) δ 180.1, 140.3, 139.9, 140.0, 137.3, 137.2, 133.3, 130.6, 130.1, 129.6, 129.2, 123.1, 100.3, 34.9, 21.3, 19.8. HRMS (ESI) m/z: calcd. for C₁₆H₁₈IN₂⁸⁰Se [M+H]⁺: 444.9674; Found: 444.9669.



6b was prepared following the General Procedure D

The reaction of **4a** (94% ee, 41.2 mg, 0.10 mmol, 1.0 equiv) and 4-methoxyaniline (24.6 mg, 0.20 mmol, 2.0 equiv) in anhydrous CH₂Cl₂ (2.0 mL) at room temperature for 2 h afforded **6b** as a white solid (52.3 mg, 80%, 94% ee) (eluent for column chromatography PE/EtOAc = 20:1). **HPLC conditions**: Chiralpak AD-H, isopropanol/hexane = 10:90, flow: 1.0 mL/min, $\lambda = 254$ nm, t_r = 16.087 min (major), 17.424 min (minor). [α]_D²⁰ = +18.8 (c 0.50, CH₂Cl₂). ¹H **NMR (500 MHz, CDCl**₃) δ 8.07 (s, 1H), 8.00 (s, 1H), 7.60 (d, *J* = 8.0 Hz, 1H), 7.31 (t, *J* = 8.0 Hz, 1H), 7.20 (d, *J* = 7.5 Hz, 1H), 7.17 – 7.09 (m, 2H), 6.92 (t, *J* = 7.5 Hz, 1H), 6.70 (d, *J* = 8.0 Hz, 2H), 6.64 (s, 2H), 3.76 (s, 3H), 2.01 (s, 3H), 1.85 (s, 3H). ¹³C **NMR (126 MHz, CDCl**₃) δ 159.1, 141.2, 136.8, 130.6, 129.7, 128.5, 128.0, 127.4, 125.0, 124.6, 115.3, 114.2, 100.6, 55.5, 21.8, 19.5. **HRMS (ESI)** m/z: calcd. for C₂₂H₂₂IN₂O⁸⁰Se [M+H]⁺: 536.9937; Found: 536.9946.



6c was prepared following the General Procedure D

The reaction of **4a** (94% ee, 41.2 mg, 0.10 mmol, 1.0 equiv) and N-ethylaniline (24.2 mg, 0.20 mmol, 2.0 equiv) at room temperature for 2 h afforded **6c** as a white solid (53.7 mg, 95%, 94% ee) (eluent for column chromatography PE/EtOAc = 20:1). **HPLC conditions**: Chiralpak ID, isopropanol/hexane = 7:93, flow: 1.0 mL/min, $\lambda = 254$ nm, t_r = 9.582 min (minor), 10.353 min (major). [α]²⁰_D = +58.6 (c 0.50, CH₂Cl₂). ¹H NMR (500 MHz, CDCl₃) δ 7.85 (d, *J* = 8.0 Hz, 1H), 7.53 (d, *J* = 8.0 Hz, 1H), 7.35 – 7.19 (m, 6H), 7.08 (d, *J* = 7.5 Hz, 1H), 6.94 (t, *J* = 7.5 Hz, 1H), 6.54 (s, 1H), 6.45 (s, 1H), 4.49 – 3.87 (m, 2H), 2.04 (s, 3H), 1.80 (s, 3H), 1.08 (t, *J* = 7.0 Hz, 3H). ¹³C NMR (126 MHz, CDCl₃) δ 179.0, 141.8, 139.7, 139.5, 138.5, 136.7, 136.6, 135.8, 130.7, 130.4, 129.2, 128.9, 127.9, 127.4, 126.5, 100.6, 52.3, 22.0, 19.4, 12.9. HRMS (ESI) m/z: calcd. for C₂₃H₂₄IN₂⁸⁰Se [M+H]⁺: 535.0144; Found: 535.0156.

5. Kinetic Studies

General Procedure for Reactions in Omnical Heat Flow Calorimeter

Heat flow calorimetric data were acquired on Omnical Super CRC, which allows continuous monitoring of the instantaneous heat flow around the reaction vessel. In the absence of side reactions, the isothermal reaction heat flow, \mathbf{q} , is proportional to the reaction rate, \mathbf{r} , where $\Delta \mathbf{H}_{rxn}$ is the heat of the reaction and \mathbf{V} is the volume.

$$q = \Delta H_{rxn} \cdot V \cdot r$$

The enthalpy change of the reaction, ΔH_{rxn} , can be calculated by the integration of the total heat flow curve over time and divide the integral sum by the mole (n) amount of the limiting reagent consumed.

$$\Delta H_{rxn} = \frac{\int_{t=0}^{t=\infty} \mathbf{q} \cdot \mathbf{dt}}{n}$$

The observed heat profile can also be used to determine the fraction conversion by calculation of the fractional area under the temporal heat flow curve where the numerator denotes the area under the heat flow curve to time (t) and the denominator denotes the total area under the heat flow curve.

Fraction Conversion (FC) =
$$\frac{\int_{t=0}^{t} q \cdot dt}{\int_{t=0}^{t=\infty} q \cdot dt}$$

Unless otherwise indicated, all reactions were carried out in a 15 mL septum-capped vial equipped with a magnetic stir bar. The stir rate of the calorimeter was set at 900 rpm (rotations per minute) and the reaction mixture was allowed to equilibrate inside of the calorimeter at 30 °C for 60 minutes prior to the initiation of the reaction by injection of potassium selenocyanate in H_2O or D_2O or the copper iodide and **L6** catalyst. All liquid reagents were performed using plastic syringes.

(1) H₂O or D₂O effect on the reaction rate



A 15 mL septum-capped vial equipped with a magnetic stir bar was charged with CuI (1.0 mg, 0.005 mmol), L6 (5.0 mg, 0.01 mmol), cyclic diaryliodonium salt 1a (45.6 mg, 0.10 mmol, 1.0 equiv). 2.5 mL of dry CH_2Cl_2 was added to the vial, and it was placed into the calorimeter and allowed to thermally equilibrate to 30 °C for ~60 minutes. While this vial was equilibrating, potassium selenocyanate (0.20 mmol, 2.0 equiv) dissolved in water (0.5 mL) was added, and the solution was drawn into a 1 mL plastic syringe; the needle was capped with a small piece of Teflon, and placed into the thermostated syringe block. After the system reached thermal equilibrium, the reaction was initiated by the injection of the KSeCN solution.



A 15 mL septum-capped vial equipped with a magnetic stir bar was charged with CuI (1.0 mg, 0.005 mmol), L6 (5.0 mg, 0.01 mmol), cyclic diaryliodonium salt 1a (45.6 mg, 0.10 mmol, 1.0 equiv). 2.5 mL of dry CH_2Cl_2 was added to the vial, and it was placed into the calorimeter and allowed to thermally equilibrate to 30 °C for ~60 minutes. While this vial was equilibrating, potassium selenocyanate (0.20 mmol, 2.0 equiv) dissolved in deuterium oxide (0.5 mL) was added, and the solution was drawn into a 1 mL plastic syringe; the needle was capped with a small piece of Teflon, and placed into the thermostated syringe block. After the system reached thermal equilibrium, the reaction was initiated by the injection of the KSeCN solution.



A 15 mL septum-capped vial equipped with a magnetic stir bar was charged with CuI (1.0 mg, 0.005 mmol), L6 (5.0 mg, 0.01 mmol), cyclic diaryliodonium salt 1a (45.6 mg, 0.10 mmol, 1.0 equiv) and potassium selenocyanate (0.20 mmol, 2.0 equiv). It was placed into the calorimeter and allowed to thermally equilibrate to 30 °C for ~60 minutes. While this vial was equilibrating, 2.5 mL of dry CH₂Cl₂ was drawn into a 2.5 mL plastic syringe; the needle was capped with a small piece of Teflon, and placed into the thermostated syringe block. After the system reached thermal equilibrium, the reaction was initiated by the injection of the CH₂Cl₂ solution.



Figure S1. Heat Flow vs time at different solvent



Figure S2. Heat Flow integration vs time at different solvent



Figure S3. Conversion rate vs time at different solvent

(2) The effect of the concentration of CuI(L6)₂.



A 15 mL septum-capped vial equipped with a magnetic stir bar was charged with $CuI(L6)_2$ (X mol%), cyclic diaryliodonium salt **1a** (45.6 mg, 0.1 mmol, 1.0 equiv). 2.5 mL of dry CH_2Cl_2 was added to the vial, and it was placed into the calorimeter and allowed to thermally equilibrate to 30 °C for ~60 minutes. While this vial was equilibrating, potassium selenocyanate (0.20 mmol, 2.0 equiv) dissolved in water (0.5 mL) was added, and the solution was drawn into a 1 mL plastic syringe; the needle was capped with a small piece of Teflon, and placed into the thermostated syringe block. After the system reached thermal equilibrium, the reaction was initiated by the injection of the KSeCN solution.



Figure S4. Heat Flow vs time at different concentrations of CuI(L6)₂



Figure S5. Heat Flow Integration vs time at different concentrations of CuI(L6)₂



Figure S6. Reaction rate of different concentrations of CuI(L6)₂

(3) The effect of the concentration of KSeCN.



A 15 mL septum-capped vial equipped with a magnetic stir bar was charged with $CuI(L6)_2$ (10 mol%), cyclic diaryliodonium salt 1a (45.6 mg, 0.1 mmol, 1.0 equiv). 2.5 mL of dry CH_2Cl_2 was added to the vial, and it was placed into the calorimeter and allowed to thermally equilibrate to 30 °C for ~60 minutes. While this vial was equilibrating, potassium selenocyanate (x equiv) dissolved in water (0.1 mL) was added, and the solution was drawn into a 1 mL plastic syringe; the needle was capped with a small piece of Teflon, and placed into the thermostated syringe block. After the system reached thermal equilibrium, the reaction was initiated by the injection of the KSeCN solution.



Figure S7. Heat Flow vs time at different concentrations of KSeCN



Figure S8. Heat Flow Integration vs time at different concentrations of KSeCN



Figure S9. Reaction rate of different concentrations of KSeCN

6. Crystal structures and structure refinement

X-ray Structure of **3g**



	Table S1	Crystal	data and	l structure	refinement	for	3g.
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Identification code	6-20-2 (3 g)	
Empirical formula	C ₂₁ H ₁₂ INSe	
Formula weight	484.18	
Temperature/K	292.96(13)	
Crystal system	tetragonal	
Space group	P41	
a/Å	10.94350(10)	
b/Å	10.94350(10)	
c/Å	14.74820(10)	
$\alpha/^{\circ}$	90	
β/°	90	
γ/°	90	
Volume/Å ³	1766.25(3)	
Z	4	
$\rho_{calc}g/cm^3$	1.821	
μ/mm^{-1}	16.593	
F(000)	928.0	
Crystal size/mm ³	$0.15 \times 0.13 \times 0.12$	
Radiation	Cu Kα (λ = 1.54184)	
20 range for data collection/° 8.08 to 146.798		
Index ranges	$\text{-9} \le h \le 13, \text{-13} \le k \le 13, \text{-18} \le \text{1} \le 18$	
Reflections collected	13914	
Independent reflections	3436 [$R_{int} = 0.0435$, $R_{sigma} = 0.0314$]	

Data/restraints/parameters	3436/1/218		
Goodness-of-fit on F ²	1.027		
Final R indexes [I>= 2σ (I)]	$R_1 = 0.0373, wR_2 = 0.0962$		
Final R indexes [all data]	$R_1 = 0.0379, wR_2 = 0.0969$		
Largest diff. peak/hole / e Å ⁻³ 0.53/-0.32			
Flack/Hooft parameter	-0.005(6)/-0.021(3)		

X-ray Structure of 6b



Table S2 Crystal data and structure refinement for 6b.

Identification code	6-98-1 (6b)
Empirical formula	$C_{22}H_{21}IN_2OSe$
Formula weight	535.27
Temperature/K	293.3(5)
Crystal system	triclinic
Space group	P1
a/Å	9.3735(5)
b/Å	11.1993(6)
c/Å	11.3482(5)
α/°	63.903(5)
β/°	80.905(4)
$\gamma/^{\circ}$	86.542(4)
Volume/Å ³	1056.34(10)
Z	2
$\rho_{calc}g/cm^3$	1.683
µ/mm ⁻¹	13.981
F(000)	524.0
Crystal size/mm ³	$0.13 \times 0.11 \times 0.08$
Radiation	Cu Ka ($\lambda = 1.54184$)

2Θ range for data collection/°	^o 8.772 to 146.49
Index ranges	$\textbf{-11} \leq h \leq 11, \textbf{-13} \leq k \leq 13, \textbf{-14} \leq l \leq 12$
Reflections collected	14604
Independent reflections	6247 [$R_{int} = 0.1189, R_{sigma} = 0.0745$]
Data/restraints/parameters	6247/413/493
Goodness-of-fit on F ²	1.049
Final R indexes [I>= 2σ (I)]	$R_1 = 0.0917, wR_2 = 0.2514$
Final R indexes [all data]	$R_1 = 0.1086, wR_2 = 0.2709$
Largest diff. peak/hole / e Å-3	3 1.09/-0.92
Flack/Hooft parameter	0.007(14)/0.092(5)

7. References

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8. Copies of NMR spectra



¹³C NMR spectra of L7



¹³C NMR spectra of L8


¹³C NMR spectra of L9



¹³C NMR spectra of A4



¹³C NMR spectra of A5



/¹³C NMR spectra of L10



¹³C NMR spectra of L11



¹³C NMR spectra of **3a**





LYY-5-178-2.2.fi



 ^{13}C NMR spectra of 3c

lyy5-26-2.1.fi

7,960 7,942 7,942 7,863 7,863 7,863 7,863 7,863 7,863 7,863 7,734 7,734 7,734 7,734 7,734 7,734 7,734 7,734 7,734 7,734 7,734 7,735 6 7,734 6 7,733 7,755 7,755 7,705 6 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,705 7,7050



¹³C NMR spectra of **3d**



¹³C NMR spectra of **3e**



¹³C NMR spectra of **3f**



 $^{13}\mathrm{C}$ NMR spectra of $3\mathrm{g}$



¹³C NMR spectra of **3h**



¹³C NMR spectra of **3i**



¹³C NMR spectra of **3j**



¹⁹F NMR spectra of **3**k



¹H NMR spectra of **3**l'







¹H NMR spectra of **4b**



¹H NMR spectra of 4c



¹H NMR spectra of **6d**







¹H NMR spectra of **4e**



¹H NMR spectra of 4f







¹H NMR spectra of **5b**



¹H NMR spectra of **5**c



¹H NMR spectra of 5d



¹³C NMR spectra of **5d**



¹³C NMR spectra of **5e**



¹³C NMR spectra of **5**f



¹³C NMR spectra of **5g**

7,817 7,801 7,516 6,516 7,515 7,516 7,516 7,501 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203 7,203



¹³C NMR spectra of **5h**



¹³C NMR spectra of **5i**



¹³C NMR spectra of **5**j



¹³C NMR spectra of 5k


¹³C NMR spectra of **5**l



 $^{19}\mathrm{F}$ NMR spectra of $5\mathrm{m}$



¹H NMR spectra of **DS**







¹H NMR spectra of **6a**











¹³C NMR spectra of **6c**

9. Copies of HPLC traces



	Area Percent Report										
Peak #	RetTime [min]	Туре	Width [min]	Area [mAU*s]	Height [mAU]	Area ۶					
1	8.908	VB	0.3556	1.31113e4	589.24768	49.7652					
2	11.144	MM	0.3505	1.32350e4	629.34705	50.2348					

HPLC spectra of 3a-rac



				Area Percen	t Report	
Peak #	RetTime [min]	Туре	Width [min]	Area [mAU*s]	Height [mAU]	Area ۶
1	8.937	BB	0.4496	5795.02832	183.93570	96.7591
2	11.521	MM	0.5223	194.10034	6.19353	3.2409





			1	Area Percent	t Report		
Peak	RetTime	Туре	Width	Area	Height	Area	
#	[min]		[min]	[mAU*s]	[mAU]	20	
1	9.593	BB	0.4020	1.09644e4	402.25760	50.3583	
2	11.491	BB	0.5938	1.08083e4	268.38559	49.6417	

HPLC spectra of 3b-rac



	Area Percent Report										
Peak	RetTime [min]	Туре	Width [min]	Area [mAU*s]	Height [mAU]	Area %					
1	9.936	MM	0.3058	1519.90540	82.82503	6.2077					
2	11.408	VB	0.4683	2.29643e4	747.65234	93.7923					

HPLC spectra of 3b



	Area Percent Report											
Peak #	RetTime [min]	Туре	Width [min]	Area [mAU*s]	Height [mAU]	Area %						
1	72.797	MF	1.0323	2.26845e4	366.22885	97.6540						
2	75.472	FM	1.0125	544.97473	8.97083	2.3460						

HPLC spectra of 3c



====		=====					
Peak	RetTime	Туре	Width	Area	Height	Area	
#	[min]		[min]	[mAU*s]	[mAU]	ele ele	
1	78.831	MF	1.0782	4223.05664	65.27996	49.4567	
2	81.186	FM	1.1800	4315.84033	60.95868	50.5433	





			-	101 10100	o nopere		
Peak	RetTime	Туре	Width	Area	Height	Area	
#	[min]		[min]	[mAU*s]	[mAU]	ह ।	
1	78 143	ME	1 1 9 9 1	2 15424e4	299 41440	94 7505	
2	81 087	FM	1 2/71	1103 51526	15 95043	5 2/05	
2	01.007	1.14	1.24/1	1193.31320	10.90045	5.2495	





	1	Area Percer	nt Report	
Peak RetTime Type	Width	Area [mAU*s]	Height	Area ۶
		[IIIA0 - 5]		
1 17.278 VB 2 21.549 BB	0.7278 0.8563	6.34728e4 6.61145e4	1295.07837 1133.86572	48.9807 51.0193

HPLC spectra of 3e-rac



			1	Area Percent	t Report		
=====							
Peak	RetTime	Туре	Width	Area	Height	Area	
#	[min]		[min]	[mAU*s]	[mAU]	8	
1	17.498	MM	0.6571	651.21869	16.51740	1.9551	
2	21.854	MM	0.9304	3.26578e4	585.03436	98.0449	

HPLC spectra of 3e



Peak RetTime Type Width Area Height Area # [min] [min] [mAU*s] [mAU] % 1 20.389 MM 0.4120 1.86534e4 754.50677 50.6092				1	Area Percent	t Report	
 1 20.389 MM 0.4120 1.86534e4 754.50677 50.6092	Peak #	RetTime [min]	Туре	Width [min]	Area [mAU*s]	Height [mAU]	Area %
		20.389	MM	0.4120	1.86534e4	754.50677	50.6092





	Area Percent Report										
Peak #	RetTime [min]	Туре	Width [min]	Area [mAU*s]	Height [mAU]	Area %					
1	20.400	MM	0.3832	203.70009	8.85909	1.5299					
2	24.224	MM	0.4975	1.31105e4	439.22388	98.4701					

HPLC spectra of $\mathbf{3f}$



			I	Area Percent	t Report	
===== Peak #	RetTime [min]	Туре	Width [min]	Area [mAU*s]	Height [mAU]	Area %
 1 2	13.420 17.505	BB BB	0.3865	1.05301e4 1.07534e4	414.27277 317.43173	49.4754 50.5246

HPLC spectra of 3g-rac



			1	Area Percent	Report		
Peak #	RetTime [min]	Туре	Width [min]	Area [mAU*s]	Height [mAU]	Area %	
1	13.436	MM	0.4182	7960.60938	317.24777	98.6042	
2	17.715	MM	0.5431	112.69091	3.45821	1.3958	

HPLC spectra of 3g



			1	Area Percen	t Report		
Peak	RetTime	Туре	Width	Area	Height	Area	
#	[min]		[min]	[mAU*s]	[mAU]	ele .	
1	8.340	VV	0.3186	1.56762e4	779.72083	50.2815	
2	9.154	VV	0.2406	1.55007e4	1012.51062	49.7185	

HPLC spectra of 3h-rac



	Area Percent	Report	
Peak RetTime Type Wi # [min] [n	idth Area nin] [mAU*s]	Height [mAU]	Area %
	-		
1 8.402 VV 0.	.3021 9802.07520	519.60944 9	6.6400
2 9.129 MM 0.	.1958 340.80350	29.01172	3.3600

HPLC spectra of $\mathbf{3h}$



			1	Area Percen	t Report		
Peak	RetTime	Туре	Width	Area	Height	Area	
#	[min]		[min]	[mAU*s]	[mAU]	90	
1	6.303	BV	0.2351	1.66794e4	1018.47943	49.5195	
2	8.482	BB	0.3748	1.70031e4	754.79767	50.4805	





			1	Area Percent	Report	
Peak #	RetTime [min]	Туре	Width [min]	Area [mAU*s]	Height [mAU]	Area १
1	6.547	 ММ	0.2402	355.35434	24.65356	5.1917
2	8.493	BB	0.3715	6489.24561	261.50790	94.8083





			1	Area Percent	t Report	
Peak	RetTime	Туре	Width	Area	Height	Area
#	[min]		[min]	[mAU*s]	[mAU]	용
1	8.643	VV	0.2533	7712.78809	451.46548	50.3804
2	16.511	BB	0.5648	7596.33154	196.60860	49.6196

HPLC spectra of 3j-rac



			1	Area Percent	t Report		
Peak	RetTime	Tvpe	Width	Area	Height	Area	
#	[min]		[min]	[mAU*s]	[mAU]	00	
1	8.696	MM	0.2421	363.74374	25.04235	2.2021	
2	16.332	MM	0.6537	1.61541e4	411.87308	97.7979	

HPLC spectra of 3j





min

0.4692 4186.54736 132.99893 95.7766

				Area Percen	t Report		
Peak #	RetTime [min]	Туре	Width [min]	Area [mAU*s]	Height [mAU]	Area ۶	
1	10.303	MM	0.3428	184.61133	8.97687	4.2234	

2 14.402 BB

HPLC spectra of 3k







Area Percent Report

				Area Percen			
Peak #	RetTime [min]	Туре	Width	Area [mAU*s]	Height [mAU]	Area ۶	
 1	18.366	 MM	0.3809	147.25562	6.44348	2.7956	
2	19.937	BB	0.3839	5120.20215	206.02350	97.2044	





			I	Area Percer	nt Report	
Peak	RetTime	Туре	Width	Area	Height	Area
#	[min]		[min]	[mAU*s]	[mAU]	8
1	20.212	BV	0.3985	4.55149e4	1766.90076	49.0814
2	21.471	VB	0.4799	4.72187e4	1473.26477	50.9186

HPLC spectra of 4b-rac





	Area Percent Report										
Peak	RetTime [min]	Туре	Width [min]	Area [mAU*s]	Height [mAU]	Area %					
1	22.661	MM	0.4099	585.25153	23.79885	9.7481					
2	23.639	MM	0.5000	5418.51611	180.60495	90.2519					

HPLC spectra of 4b



<Peak Table>

Detect	or A Chann	el 1 254nm					
Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	8.499	505797	29721	49.516			
2	9.319	515685	27876	50.484		V	
Total		1021482	57597				

HPLC spectra of 6d-rac



<Peak Table>

Detec	tor A Chann	el 1 254nm					
Peak	# Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	8.491	4717647	287262	98.212			
2	9.314	85889	3733	1.788		SV	
Tota	al	4803536	290995				

HPLC spectra of 6d



HPLC spectra	of 4d-rac
--------------	-----------



			1	Area Percen	t Report		
Peak	RetTime	Туре	Width	Area	Height	Area	
#	[min]		[min]	[mAU*s]	[mAU]	8	
1	18.336	MM	0.6895	1.21913e4	294.69824	98.8470	
2	19.908	MM	0.5276	142.20572	4.49226	1.1530	

HPLC spectra of 4d



			1	Area Percent	t Report		
Peak #	RetTime [min]	Туре	Width [min]	Area [mAU*s]	Height [mAU]	Area %	
1 2	8.956 11.427	VB BB	0.2665	2625.16211 2601.20264	145.49535 119.27790	50.2292 49.7708	

HPLC spectra of 4e-rac



			1	Area Percent	t Report		
Peak	RetTime	Туре	Width	Area	Height	Area	
#	[min]		[min]	[mAU*s]	[mAU]	90	
1	8.954	BB	0.2712	2004.41370	109.68398	99.4467	
2	11.419	MM	0.2738	11.15209	6.78901e-1	0.5533	

HPLC spectra of 4e



Area Percent Report						
Peak #	RetTime [min]	Туре	Width [min]	Area [mAU*s]	Height [mAU]	Area १
1	18.300	MF	0.3618	409.77390	18.87851	50.6531
2	19.267	FM	0.3723	399.20679	17.87318	49.3469

HPLC spectra of 4f-rac



				Area Percen	t Report		
==== Peak #	RetTime [min]	Туре	Width [min]	Area [mAU*s]	Height [mAU]	Area १	
1	18.992	MM	0.3436	471.23044	22.86027	1.3243	
2	19.910	VV	0.3693	3.51120e4	1466.47217	98.6757	

HPLC spectra of 4f



			1	Area Percent	Report		
Peak #	RetTime [min]	Туре	Width [min]	Area [mAU*s]	Height [mAU]	Area १	
1	11.837	BB	0.1988	3131.15649	242.21458	50.0193	
2	12.937	BV	0.2004	3128.73560	245.94547	49.9807	
# 1 2	[min] [11.837 12.937	I ype BB BV	[min] 0.1988 0.2004	[mAU*s] [] 3131.15649 3128.73560	[mAU] [242.21458 245.94547	Riea % 50.0193 49.9807	

HPLC spectra of 5a-rac



			1	Area Percent	t Report		
==== Peak #	RetTime [min]	Туре	Width [min]	Area [mAU*s]	Height [mAU]	Area ۴	
1	10.457	VV	0.1923	1.36449e4	1103.44958	94.8701	
2	11.240	MM	0.1927	737.81860	63.81617	5.1299	

HPLC spectra of 5a





1.1579 4.36518e4 542.99207 50.2063

2 44.225 BB



			1	Area Percent	t Report		
Peak	RetTime	Туре	Width	Area	Height	Area	
#	[min]		[min]	[mAU*s]	[mAU]	8	
1	31.937	MM	0.8821	5.36390e4	1013.43005	94.0762	
2	44.652	MM	0.9174	3377.53638	61.35929	5.9238	

HPLC spectra of 5b



				Area Bercon	t Doport		
Alea Felcent Report							
====					===========		
Peak	RetTime	Type	Width	Area	Height	Area	
ICar	ICCCI INC	Type	Withour	ALCa	nergne	ALCU	
#	[min]		[min]	[mAU*s]	[mAU]	00	
	1				I		
1	19.332	BB	0.3568	5445.79492	232.92320	49.6245	
2	23.625	BB	0.4575	5528.20898	184.52164	50.3755	







			1	Area Percen	t Report		
Peak #	RetTime	Туре	Width [min]	Area [mAU*s]	Height [mAU]	Area مع	
	[I I		[[]		
1	18.457	BB	0.3993	1.68869e4	641.11707	95.0570	
2	22.670	MM	0.4326	878.12396	33.82783	4.9430	





				Area Percen	t Report	
Peak #	RetTime [min]	Туре	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	11.341	VB VB	0.2244	4385.31836	303.84277 336.99298	49.9246

HPLC spectra of 5d-rac



			7	Area Percen	t Report		
==== Peak #	RetTime [min]	Туре	Width [min]	Area [mAU*s]	Height [mAU]	Area १	
1	11.127	MM	0.2681	8044.78467	500.03421	95.0039	
2	12.479	MM	0.2424	423.05902	29.09138	4.9961	





Peak #	RetTime [min]	Туре	Width [min]	Area [mAU*s]	Height [mAU]	Area ۶	
1	10.904	BB	0.2193	2827.78882	197.25467	50.4195	
2	12.778	BV	0.2802	2780.73462	165.32210	49.5805	

HPLC spectra of 5e-rac



			1	Area Percen	t Report		
Peak	RetTime	Type	Width	Area	Height	Area	
#	[min]	1100	[min]	[mAU*s]	[mAU]	8	
1	10.660	VB	0.2320	1.55022e4	1004.57373	95.2820	
2	13.282	MM	0.4874	767.60883	26.24605	4.7180	

HPLC spectra of 5e



				Area Percen	it Report	
Peal	RetTime	Type	Width	Area	Height	Area
#	[min]		[min]	[mAU*s]	[mAU]	20
1	23.280	VB	0.7063	1.01307e4	213.21255	54.3197
2	2 31.083	BB	0.3635	8519.45313	380.03293	45.6803

HPLC spectra of 5f-rac



				Area Percen	t Report	
Peak #	RetTime [min]	Туре	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	24.145	BB	0.8003	5.43259e4	1002.42822	94.7586
2	31.098	MM	0.4844	3004.93970	103.38733	5.2414





				Area Percen	t Report	
Peak #	RetTime [min]	Туре	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	13.450	BB	0.2248	4388.46338	303.23022	49.4294
2	15.783	MM	0.3402	4489.78711	219.94670	50.5706

HPLC spectra of 5g-rac





			1	Area Percent	t Report	
Peak #	RetTime [min]	Туре	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	13.721	VB	0.2663	2.10780e4	1278.55042	95.0362
2	15.710	MM	0.3079	1100.91455	59.59592	4.9638

HPLC spectra of 5g



				Area Percen	t Report		
Peak #	RetTime [min]	Туре	Width [min]	Area [mAU*s]	Height [mAU]	Area %	
1	7.231	VV	0.2298	7583.20117	481.25137	49.0572	
2	8.005	VB	0.2634	7874.68604	438.66830	50.9428	

HPLC spectra of 5h-rac



				Area Percen	t Report		
Peak #	RetTime [min]	Туре	Width [min]	Area [mAU*s]	Height [mAU]	Area %	
1	7.480	MM	0.2169	1253.75720	96.35194	4.5051	
2	8.326	VB	0.3178	2.65761e4	1251.01343	95.4949	

HPLC spectra of 5h



			1	Area Percent	t Report	
Peak	RetTime	Туре	Width	Area	Height	Area
#	[min]		[min]	[mAU*s]	[mAU]	۶
1	11.673	BB	0.2166	2261.01221	160.28281	50.5009
2	12.858	BB		2216.15869	140.33112	49.4991

HPLC spectra of 5i-rac



			ž	Area Percent	t Report		
Peak #	RetTime [min]	Туре	Width [min]	Area [mAU*s]	Height [mAU]	Area ۴	
1	12.130	BB	0.2205	1.01287e4	701.26959	95.0328	
2	13.377	MM	0.2443	529.41034	36.11733	4.9672	





	Area Percent Report								
==== Peak #	RetTime [min]	Туре	Width [min]	Area [mAU*s]	Height [mAU]	Area %			
1	23.259	MF	0.5558	5913.73730	177.34207	53.9383			
2	24.845	FM	0.5509	5050.14893	152.78676	46.0617			

HPLC spectra of 5j-rac



=====							
				Area Percent	t Report		
Peak	RetTime	Туре	Width	Area	Height	Area	
#	[min]		[min]	[mAU*s]	[mAU]	8	
1	22.831	MF	0.5621	2.98465e4	884.89301	94.8372	
2	24.599	FM	0.5244	1624.79602	51.64217	5.1628	

HPLC spectra of 5j



Peak RetTime Type Width Area Height Area # [min] [min] [mAU*s] [mAU] %					Area Percer	nt Report	
# [min] [min] [mAU*s] [mAU] % 	Peak	RetTime	Туре	Width	Area	Height	Area
	#	[min]		[min]	[mAU*s]	[mAU]	80
1 24.972 BB 0.3922 6012.13818 226.11945 50.3819 2 28 807 BB 0.6036 5921 00293 143 42139 49 6181							
2 28 807 BB 0 6036 5921 00293 143 42139 49 6181	1	24.972	BB	0.3922	6012.13818	226.11945	50.3819
2 20.007 BB 0.0000 002100200 140.42100 40.0101	2	28.807	BB	0.6036	5921.00293	143.42139	49.6181

HPLC spectra of 5k-rac



				Area Percen	t Report	
Peak #	RetTime [min]	Туре	Width [min]	Area [mAU*s]	Height [mAU]	Area १
1	25.834	BB	0.9697	2.48714e4	426.90152	95.1099
2	30.195	MM	0.5156	1278.76111	41.33352	4.8901

HPLC spectra of 5k


			1	Area Percent	Report	
Peak #	RetTime [min]	Туре	Width [min]	Area [mAU*s]	Height [mAU]	Area ۴
1	10.397	VV	0.2010	7151.31152	552.51837	49.2268
2	11.159	VB	0.2317	7375.94678	495.28275	50.7732

HPLC spectra of $\mathbf{5I}$



	Area Percent Report							
Peak #	RetTime [min]	Туре	Width [min]	Area [mAU*s]	Height [mAU]	Area %		
						I		
1	9.721	MM	0.1778	926.07727	86.79826	5.1350		
2	10.297	VB	0.2149	1.71087e4	1225.09619	94.8650		

HPLC spectra of 51



Area Percent Report							
Peak RetTime : # [min]	Type Width [min]	Area [mAU*s]	Height	Area %			
-							
1 10.008 1 2 10.839 1	BB 0.1741 BB 0.1996	4429.84863 4530.36914	391.35587 348.74747	49.4391 50.5609			

HPLC spectra of 5m-rac





HPLC spectra of 5m



Area Percent Report							
Peak #	RetTime [min]	Туре	Width [min]	Area [mAU*s]	Height [mAU]	Area ۶	
1	14.440	MM	0.4093	2.40892e4	980.92352	50.1900	
2	1/.121	Iviivi	0.3190	2.3900/04	1245./0992	49.0100	

HPLC spectra of 5n-rac



				Area Percer	nt Report	
Peak #	RetTime [min]	Туре	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	14.579	MM	0.3436	1927.10999	93.46421	7.2389
2	16.911	MM	0.9012	2.46946e4	456.71765	92.7611

HPLC spectra of 5n

<Chromatogram>



<Peak Table>

Detector A Channel 1 254nm							
Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	7.798	406613	22569	48.036			
2	9.299	439857	20418	51.964			
Total		846470	42987				





<Peak Table>

Detector A Channel 1 254nm								
Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name	
1	7.443	3292517	212602	96.963				
2	8.828	103115	5067	3.037		VΜ		
Total		3395632	217670					

HPLC spectra of 6a



<Peak Table>

Detector A Channel 1 254nm								
Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name	
1	17.539	1137788	39327	50.007				
2	18.948	1137472	36882	49.993		V		
Total		2275260	76209					

HPLC spectra of	of 6b-rac
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<Peak Table>

Detector A Channel 1 254nm								
Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name	
1	16.087	1091861	41982	97.011		Μ		
2	17.424	33638	1196	2.989		VM		
Total		1125499	43178					

HPLC spectra of 6b

<Chromatogram>



<Peak Table>

Detector A Channel 1 254nm								
Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name	
1	10.091	4594223	260047	49.897		V		
2	10.929	4613185	218531	50.103		V		
Total		9207408	478578					

HPLC	spectra	of	6c-rac
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<Peak Table>

Detector A Channel 1 254nm							
Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	9.582	227642	13948	2.844		V	
2	10.353	7777707	431030	97.156		SV	
Total		8005349	444978				

HPLC spectra of 6c