Supporting Information

Synthesis of α -Quaternary β -Lactams via Copper-Catalyzed Enantioconvergent Radical C(sp³)–C(sp²) Cross-Coupling with Organoboronate Esters

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General information

Reactions were carried out under an argon atmosphere using Schlenk techniques. Reagents were purchased at the highest commercial quality and used without further purification, unless otherwise stated. CuBr·Me2S was purchased from Aladdin. Anhydrous THF and 1.4-dioxane were purchased from J&K Chemical Ltd. Lithium tert-butoxide (99%) was purchased from Macklin, which was dry at 90 °C for 3 h under reduced pressure. Analytical thin layer chromatography (TLC) was performed on precoated silica gel 60 GF254 plates. Flash column chromatography was performed using Tsingtao silica gel (60, particle size 0.040–0.063 mm). Visualization on TLC was achieved by use of UV light (254 nm), iodine or basic KMnO4 indicator. NMR spectra were recorded on Bruker DRX-400 spectrometer at 400 MHz for ¹H NMR, 101 MHz for ¹³C NMR and 376 MHz for ¹⁹F NMR, respectively, in CDCl₃ with tetramethylsilane (TMS) as internal standard. The chemical shifts were expressed in ppm and coupling constants were given in Hz. Data for ¹H NMR are recorded as follows: chemical shift (ppm), multiplicity (s, singlet; d, doublet; t, triplet; q, quartet; p, pentet, m, multiplet; br, broad), coupling constant (Hz), integration. Data for ¹³C NMR were reported in terms of chemical shift (δ , ppm). High-resolution mass spectroscopy (HRMS) was obtained on Thermo Scientific Q Exactive using ESI ion source. Enantiomeric excess (ee) was determined using SHIMADZU LC-20AD with SPD-20AV detector or Agilent high-performance liquid chromatography (HPLC) with Hatachi detector (at appropriate wavelength). Column conditions were reported in the experimental section below. Xray diffraction was measured on a 'Bruker APEX-II CCD' diffractometer with Cu-Ka radiation.



Figure S1. Crude ¹H NMR analysis of E1 under LiO'Bu/DMF conditions: E1 decomposed in the absence of the catalyst under the LiO'Bu/DMF conditions (E1 (0.05 mmol), LiO'Bu (3.0 equiv.) and H₂O (1.0 equiv.) in DMF (1.0 mL) at rt for 20 h under argon).





Figure S2. The X-ray structure of 19.



Table S1. Evaluating the reaction of tertiary α-bromo-β-lactam with alkyl chain^[a]

[a] Reaction conditions: **E18** (0.1 mmol), **A1** (0.12 mmol), CuBr·SMe₂ (10 mol%), L* (15 mol%), LiO'Bu (3.0 equiv.) and H₂O (1.0 equiv.) in solvent (2.0 mL) at rt for 24 h under argon (Ar). Yields were based on ¹H NMR analysis using 1,3,5-trimethoxybenzene as an internal standard. Isolated yield in parenthesis. Ee values were based on HPLC analysis.

F ₃ C	-N Ph +	CuBr-Mez O_B-O <u>L* (1</u> LiO ⁶ Bu H ₂ O (1.0 Ph	2S (10 mol%) 5 mol%) (3.0 equiv.), THF F ₃ C	Ph OMe	
	E1	A18	34	L*3, R = Me	e; L*4, R = [/] Pr
Entry	L*	Temp. (°C)	Conversion of E1 (%)	Yield of 34 (%)	Ee (%)
1	L*3	0	>95	26	81
2	L*4	0	>95	30	90
3 ^[b]	L*4	-20	>95	51	93
4 ^[c]	L*4	-40	>95	75	98

Table S2. Effect of ligands in the reaction of alkenylboronate esters^[a]

[a] Reaction conditions: E1 (0.15 mmol), A18 (0.10 mmol), CuBr·Me₂S (10 mol %), L* (15 mol %), LiO'Bu (3.0 equiv.) and H₂O (1.0 equiv.) in THF (2.0 mL) at 0 °C for 45 h under argon. Conversion of E1 and yield of 34 was based on ¹H NMR analysis of the crude product using 1,3,5-trimethoxybenzene as an internal standard. Yield of 34 was based on A18. Ee values were based on chiral HPLC analysis. [b] Conducted at -20 °C for 3 d. [c] Conducted at -40 °C for 5 d.

The synthesis of ligands, α -bromo- β -lactams and boron reagents

The synthesis of chiral ligands

Chiral ligand L*3 was synthesized according to the reported literature.^[1]



2-isopropyl-*N*-((*S*)-(6-methoxyquinolin-4-yl)((*1S*,*2S*,*4S*,*5R*)-5-vinylquinuclidin-2-yl)methyl)quinoline-8-sulfonamide (L*4)



Step A: Under an argon atmosphere, **SSL4** (3.59 g, 21 mmol) was added slowly to the chlorosulfonic acid (32 mL) while stirring in an ice bath. the reaction mixture was heated to 130 °C for 6 h. Then the mixture was cooled to room temperature with continuous stirring and poured gradually on crushed ice. The mixture was extracted with CH₂Cl₂ for three times. The combined organic phase was dried over Na₂SO₄, filtered and concentrated under reduced pressure. The residue was purified by flash chromatography on silica gel (eluent: petroleum ether/ethyl acetate 5/1) to give **SL4-1** as a white solid (1.98 g, 35% yield).

Step B: Quinine-derived chiral amine **SL4-2** was prepared from quinine according to the previously reported literature procedure.^[1] Under an argon atmosphere, chiral amine **SL4-2** (1.05 g, 3.25 mmol) was dissolved in CH₂Cl₂ (20 mL), followed by the addition of triethylamine (1.36 mL, 9.75 mmol) and **SL4-1** (1.05 g, 3.90 mmol) at ice water bath. The reaction mixture was allowed to warm up to room temperature and stirred overnight. After completion, water was poured to above mixture, and the mixture was extracted with CH₂Cl₂. The combined organic phase was dried over Na₂SO₄, filtered and concentrated under reduced pressure. The crude product was purified by flash chromatography on silica gel (gradient eluent: petroleum ether/ethyl acetate 1/1 to ethyl acetate) to provide the product **L*4** as a white solid (1.31 g, 76% yield).

¹**H NMR (400 MHz, CDCl₃)** δ 8.67 (d, J = 4.5 Hz, 1H), 8.13 (t, J = 7.1 Hz, 2H), 8.00 (d, J = 9.2 Hz, 1H), 7.94 (d, J = 8.2 Hz, 1H), 7.74 – 7.50 (m, 1H), 7.45 (t, J = 8.7 Hz, 2H), 7.39 – 7.31 (m, 1H), 7.18 (m, 1H), 5.76 – 5.24 (m, 1H), 4.86 (d, J = 17.2 Hz, 2H), 4.76 (d, J = 10.6 Hz, 1H), 3.88 (s, 3H), 3.34 (p, J = 6.9 Hz, 1H), 3.04 – 2.75 (m, 2H), 2.48 (d, J = 14.1 Hz, 1H), 2.15 – 1.87 (m, 1H), 1.84 (d, J = 17.5 Hz, 1H), 1.51 (m, 8H), 1.30 – 0.90 (m, 3H), 0.86 – 0.47 (m, 1H).

¹³C NMR (101 MHz, CDCl₃) δ 168.21, 157.63, 147.46, 144.86, 144.26, 142.62, 141.09, 136.51, 135.32, 132.86, 131.62, 130.24, 127.03, 123.96, 120.98, 119.64, 114.39, 100.94, 60.94, 55.37, 55.29, 53.19, 36.70, 27.50, 27.04, 25.04, 21.99.

HRMS (ESI) *m/z* calcd. for C₃₂H₃₇N₄O₃S [M+H]⁺ 557.2581, found 557.2577.

The synthesis of α-bromo-β-lactams

Substrates E1-E18 were prepared according to the previously reported literature procedure.^[2]



3-bromo-3-phenyl-1-(4-(trifluoromethyl)benzyl)azetidin-2-one (E1)



¹H NMR (400 MHz, CDCl₃) δ 7.67 – 7.56 (m, 4H), 7.43 – 7.31 (m, 5H), 4.62 (d, J = 15.5 Hz, 1H), 4.43 (d, J = 15.5 Hz, 1H), 3.92 (ABq, J_{AB} = 6.0 Hz, Δv_{AB} = 13.3 Hz, 2H). ¹³C NMR (101 MHz, CDCl₃) δ 165.40, 138.62, 137.42, 130.60 (q, J = 32.6 Hz), 129.41, 129.12, 128.41, 127.25, 126.17 (q, J = 3.8 Hz), 124.03 (q, J = 272.2 Hz), 60.75, 57.99, 45.85.

¹⁹F NMR (376 MHz, CDCl₃) δ -62.65.

HRMS (ESI) *m/z* calcd. for C₁₇H₁₄BrF₃NO [M+H]⁺ 384.0205, found 384.0203.

3-bromo-1-(4-bromobenzyl)-3-phenylazetidin-2-one (E3)



¹**H NMR (400 MHz, CDCl₃)** δ 7.63 – 7.55 (m, 2H), 7.52 – 7.45 (m, 2H), 7.41 – 7.30 (m, 3H), 7.15 – 7.08 (m, 2H), 4.51 (d, *J* = 15.2 Hz, 1H), 4.32 (d, *J* = 15.3 Hz, 1H), 3.87 (ABq, $J_{AB} = 6.0$ Hz, $\Delta v_{AB} = 12.4$ Hz, 2H).

¹³C NMR (101 MHz, CDCl₃) δ 165.28, 137.49, 133.50, 132.33, 129.88, 129.35, 129.08, 127.24, 122.31, 60.66, 57.76, 45.74.

HRMS (ESI) *m/z* calcd. for C₁₆H₁₄Br₂NO [M+H]⁺ 393.9437, found 393.9436.

3-bromo-1-(4-(tert-butyl)benzyl)-3-phenylazetidin-2-one (E4)



¹H NMR (400 MHz, CDCl₃) δ 7.65 – 7.57 (m, 2H), 7.42 – 7.30 (m, 5H), 7.17 (d, J = 8.2 Hz, 2H), 4.52 (d, J = 15.1 Hz, 1H), 4.37 (d, J = 15.1 Hz, 1H), 3.89 (ABq, $J_{AB} = 6.1$ Hz, $\Delta v_{AB} = 4.0$ Hz, 2H), 1.32 (s, 9H).

¹³C NMR (101 MHz, CDCl₃) δ 165.21, 151.24, 137.80, 131.30, 129.20, 129.01, 127.92, 127.29, 126.03, 60.66, 57.77, 45.97, 34.71, 31.43.

HRMS (ESI) *m/z* calcd. for C₂₀H₂₃BrNO [M+H]⁺ 372.0958, found 372.0956.

3-bromo-1-(4-methoxybenzyl)-3-phenylazetidin-2-one (E5)



¹H NMR (400 MHz, CDCl₃) δ 7.63 – 7.52 (m, 2H), 7.42 – 7.29 (m, 3H), 7.20 – 7.08 (m, 2H), 6.92 – 6.82 (m, 2H), 4.48 (d, *J* = 14.9 Hz, 1H), 4.31 (d, *J* = 14.9 Hz, 1H), 3.84 (ABq, *J* = 6.0 Hz, Δv_{AB} = 9.1 Hz, 2H), 3.79 (s, 3H).

¹³C NMR (101 MHz, CDCl₃) δ 165.00, 159.42, 137.60, 129.49, 129.10, 128.89, 127.14, 126.20, 114.37, 60.44, 57.37, 55.29, 45.66.

HRMS (ESI) *m/z* calcd. for C₁₇H₁₇BrNO₂ [M+H]⁺ 346.0437, found 346.0435.

3-bromo-1-phenethyl-3-phenylazetidin-2-one (E6)



¹**H NMR (400 MHz, CDCl₃)** δ 7.54 – 7.47 (m, 2H), 7.38 – 7.28 (m, 3H), 7.25 – 7.18 (m, 3H), 7.18 – 7.13 (m, 2H), 3.78 (ABq, J = 5.8 Hz, Δv_{AB} = 9.9 Hz, 2H), 3.55 (t, J = 7.1 Hz, 2H), 2.89 (t, J = 7.1 Hz, 2H).

¹³C NMR (101 MHz, CDCl₃) δ 164.91, 137.79, 137.56, 128.98, 128.77, 128.69, 128.60, 127.07, 126.75, 60.18, 58.66, 43.43, 33.92.

HRMS (ESI) *m/z* calcd. for C₁₇H₁₇BrNO [M+H]⁺ 330.0488, found 330.0487.

3-bromo-3-(3-methoxyphenyl)-1-(4-(trifluoromethyl)benzyl)azetidin-2-one (E12)



¹**H NMR (400 MHz, CDCl₃)** δ 7.62 (d, *J* = 8.0 Hz, 2H), 7.36 (d, *J* = 8.0 Hz, 2H), 7.29 (t, *J* = 8.2 Hz, 1H), 7.18 – 7.12 (m, 1H), 6.88 (dd, *J* = 8.3, 2.6 Hz, 1H), 4.61 (d, *J* = 15.5 Hz, 1H), 4.44 (d, *J* = 15.5 Hz, 1H), 3.90 (ABq, *J* = 5.9 Hz, Δv_{AB} = 16.6 Hz, 2H), 3.82 (s, 3H).

¹³C NMR (101 MHz, CDCl₃) δ 165.19, 159.76, 138.58, 138.46 (d, J = 1.5 Hz), 130.41 (q, J = 32.8 Hz), 130.02, 128.27, 126.02 (q, J = 3.8 Hz), 123.89 (q, J = 272.2 Hz), 119.25, 115.07, 112.57, 60.44, 57.88, 55.36, 45.68.

¹⁹F NMR (376 MHz, CDCl₃) δ -62.61.

HRMS (ESI) *m/z* calcd. for C₁₈H₁₆BrF₃NO₂ [M+H]⁺ 414.0311, found 414.0309.

3-bromo-3-(4-fluorophenyl)-1-(4-(trifluoromethyl)benzyl)azetidin-2-one (E14)



¹H NMR (400 MHz, CDCl₃) δ 7.70 – 7.51 (m, 4H), 7.36 (d, J = 7.9 Hz, 2H), 7.12 – 7.01 (m, 2H), 4.62 (d, J = 15.5 Hz, 1H), 4.43 (d, J = 15.5 Hz, 1H), 3.89 (s, 2H). ¹³C NMR (101 MHz, CDCl₃) δ 165.17, 163.05 (d, J = 250.1 Hz), 138.51, 133.47 (d, J = 3.4 Hz), 130.68 (q, J = 32.7 Hz), 129.29 (d, J = 8.5 Hz), 128.42, 126.21 (q, J = 3.8

(q, J = 3.4 Hz), 150.08 (q, J = 32.7 Hz), 129.29 (q, J = 8.5 Hz), 128.42, 120.21 (q, J = 42.1), 124.02 (q, J = 272.3 Hz), 116.22 (d, J = 21.9 Hz), 59.86, 58.05, 45.88.

¹⁹F NMR (376 MHz, CDCl₃) δ -62.66, -111.02.

HRMS (ESI) *m/z* calcd. for C₁₇H₁₂BrF₄NO [M+H]⁺ 402.0111, found 402.0109.

3-bromo-3-(4-bromophenyl)-1-(4-(trifluoromethyl)benzyl)azetidin-2-one (E16)



¹**H NMR (400 MHz, CDCl₃)** δ 7.63 (d, *J* = 7.9 Hz, 2H), 7.51 (d, *J* = 8.3 Hz, 2H), 7.47 (d, *J* = 8.3 Hz, 2H), 7.36 (d, *J* = 7.9 Hz, 2H), 4.61 (d, *J* = 15.5 Hz, 1H), 4.44 (d, *J* = 15.5 Hz, 1H), 3.89 (s, 2H).

¹³C NMR (101 MHz, CDCl₃) δ 164.80, 138.32, 136.33, 132.22, 130.52 (q, *J* = 32.6 Hz), 128.76, 128.29, 126.09 (q, *J* = 3.8 Hz), 123.88 (q, *J* = 272.2 Hz), 123.60, 59.56, 57.66, 45.77.

¹⁹F NMR (376 MHz, CDCl₃) δ -62.61.

HRMS (ESI) m/z calcd. for C₁₇H₁₃Br₂F₃NO [M+H]⁺ 461.9311, found 461.9308.

3-([1,1'-biphenyl]-4-yl)-3-bromo-1-(4-(trifluoromethyl)benzyl)azetidin-2-one (E17)



¹H NMR (400 MHz, CDCl₃) δ 7.73 – 7.55 (m, 8H), 7.46 (t, *J* = 7.5 Hz, 2H), 7.41 – 7.34 (m, 3H), 4.64 (d, *J* = 15.4 Hz, 1H), 4.45 (d, *J* = 15.5 Hz, 1H), 3.96 (ABq, *J* = 6.0 Hz, $\Delta v_{AB} = 17.8$ Hz, 2H).

¹³C NMR (101 MHz, CDCl₃) δ 165.35, 142.34, 140.17, 138.63, 136.24, 130.61 (q, *J* = 32.5 Hz), 129.03, 128.43, 127.97, 127.79, 127.71, 127.24, 126.18 (q, *J* = 3.7 Hz), 124.04 (q, *J* = 272.5 Hz), 60.66, 57.98, 45.88.

¹⁹F NMR (**376** MHz, CDCl₃) δ -62.61.

HRMS (ESI) m/z calcd. for C₂₃H₁₇BrF₃NO [M+H]⁺ 460.0518, found 460.0516.

General procedure A for the synthesis of (hetero)arylboronates



The boron reagents (A1, A3–A11, A13, A15 and A17) were synthesized according to the modified reported procedure.^[3] To a 50-mL flask equipped with a stir bar was added (hetero)aryl boronic acid (5 mmol, 1.0 equiv.), neopentyl glycol (0.55 g, 1.05 equiv.) and magnesium sulfate (1.20 g). Then dry CH₂Cl₂ (20 mL) was added. The reaction mixture was stirred at room temperature overnight. After completion of the reaction, the reaction mixture was filtered, and concentrated under reduced pressure. The crude product was purified by silica gel chromatography to afford the desired product. A12, A14 and A16 were prepared according to the reported procedure.^[4]

2-(3,5-dimethoxyphenyl)-5,5-dimethyl-1,3,2-dioxaborinane (A4)



According to the **general procedure A** starting from 3,5-dimethoxyphenylboronic acid (5 mmol), the product was obtained as a white solid (1.15 g, 4.6 mmol, 92% yield). ¹H NMR (400 MHz, CDCl₃) δ 6.96 (d, J = 2.4 Hz, 2H), 6.55 (t, J = 2.4 Hz, 1H), 3.81

(s, 6H), 3.77 (s, 4H), 1.03 (s, 6H).

¹³C NMR (101 MHz, CDCl₃) δ 160.50, 110.93, 104.08, 72.48, 55.47, 32.01, 22.04.

HRMS (ESI) m/z calcd. for C₁₃H₂₀BO₄ [M+H]⁺ 251.1449, found 251.1447.

2-(dibenzo[b,d]furan-2-yl)-5,5-dimethyl-1,3,2-dioxaborinane (A10)





According to the general procedure A starting from dibenzo[b,d]furan-2-ylboronic acid (5 mmol), the product was obtained as a white solid (1.20 g, 4.3 mmol, 86% yield).

¹H NMR (400 MHz, CDCl₃) δ 8.52 – 8.43 (m, 1H), 8.04 – 7.93 (m, 2H), 7.63 – 7.54 (m, 2H), 7.46 (t, *J* = 7.7 Hz, 1H), 7.36 (t, *J* = 7.5 Hz, 1H), 3.85 (s, 4H), 1.07 (s, 6H). ¹³C NMR (101 MHz, CDCl₃) δ 158.33, 156.33, 133.16, 127.05, 126.86, 124.45, 123.91, 122.91, 120.81, 111.71, 111.01, 72.50, 32.05, 22.04. **HRMS** (ESI) *m/z* calcd. for C₁₇H₁₈BO₃ [M+H]⁺ 281.1344, found 281.1339.

2-chloro-5-(5,5-dimethyl-1,3,2-dioxaborinan-2-yl)pyridine (A13)



A13

According to the general procedure A starting from 2-chloropyridine-5-boronic acid (5 mmol), the product was obtained as a white solid (0.92 g, 4.1 mmol, 82% yield).

¹H NMR (400 MHz, CDCl₃) δ 8.69 (s, 1H), 7.97 (d, J = 7.9 Hz, 1H), 7.29 (d, J = 7.9Hz, 1H), 3.76 (s, 4H), 1.01 (s, 6H).

¹³C NMR (101 MHz, CDCl₃) δ 155.29, 153.81, 144.21, 123.70, 72.47, 32.08, 21.98. HRMS (ESI) *m/z* calcd. for C₁₀H₁₄BClNO₂ [M+H]⁺ 226.0801, found 226.0799.

General procedure B for the synthesis of alkenylboronate esters



The boron reagents (A2, A18, A19, A20, A21, A24)^[5] and (A23, A26, A27)^[6] were synthesized according to the modified reported procedure. In a Schlenk tube were placed alkyne (2 mmol) and catecholborane (1 M in THF, 3 mL, 3 mmol). The resulting mixture was stirred at 75 °C for 16 h and then allowed to cool to room temperature. The reaction was quenched with water (10 mL). The precipitate was isolated by filtration and washed with water. The crude product was placed in a two-neck round flask, and neopentyl glycol (208.3 mg, 2 mmol) and Et₂O (25 mL) were added. The resulting mixture was stirred at room temperature for 12 h, concentrated under reduced pressure, and purified by flash column chromatography on silica gel.

(*E*)-2-(2-([1,1'-biphenyl]-4-yl)vinyl)-5,5-dimethyl-1,3,2-dioxaborinane (A22)



According to the **general procedure B** starting from 4-ethynyl-1,1'-biphenyl (2 mmol), the product was obtained as a white solid (455.2 mg, 1.56 mmol, 78% yield).

¹H NMR (400 MHz, CDCl₃) δ 7.65 – 7.50 (m, 6H), 7.48 – 7.37 (m, 3H), 7.37 – 7.31 (m, 1H), 6.15 (dd, J = 18.4, 2.3 Hz, 1H), 3.70 (s, 4H), 1.00 (s, 6H).
¹³C NMR (101 MHz, CDCl₃) δ 146.63, 141.25, 140.68, 136.83, 128.80, 127.74, 127.48, 127.40, 127.24, 126.99, 72.22, 31.88, 21.90.
HRMS (ESI) *m*/*z* calcd. for C₁₉H₂₂BO₂ [M+H]⁺ 293.1707, found 293.1705.

(E)-5,5-dimethyl-2-(2-(thiophen-3-yl)vinyl)-1,3,2-dioxaborinane (A25)



According to the **general procedure B** starting from 3-ethynylthiophene (2 mmol), the product **A25** was obtained as a white solid (310.9 mg, 1.40 mmol, 70% yield).

¹**H NMR (400 MHz, CDCl₃)** δ 7.52 – 6.91 (m, 4H), 5.88 (d, *J* = 18.2 Hz, 1H), 3.68 (s, 4H), 0.99 (s, 6H).

¹³C NMR (101 MHz, CDCl₃) δ 141.50, 140.83, 125.98, 125.17, 124.23, 72.19, 31.87, 21.88.

HRMS (ESI) *m/z* calcd. for C₁₁H₁₆BO₂S [M+H]⁺ 223.0959, found 223.0957.

(E)-5,5-dimethyl-2-(3-(phenylthio)prop-1-en-1-yl)-1,3,2-dioxaborinane (A28)



A28

According to the **general procedure B** starting from phenyl(prop-2-yn-1-yl)sulfane (2 mmol), the product was obtained as a colorless oil (314.3 mg, 1.20 mmol, 60% yield).

¹**H NMR (400 MHz, CDCl₃)** δ 7.29 – 7.21 (m, 2H), 7.17 (dd, *J* = 8.6, 6.8 Hz, 2H), 7.11 – 7.04 (m, 1H), 6.46 (dt, *J* = 17.5, 6.7 Hz, 1H), 5.62 – 5.29 (m, 1H), 3.53 (d, *J* = 7.1 Hz, 2H), 3.52 (s, 4H), 0.86 (s, 6H).

¹³C NMR (101 MHz, CDCl₃) δ 144.95, 136.22, 129.23, 128.76, 125.93, 72.01, 38.32, 31.72, 21.81.

HRMS (ESI) *m/z* calcd. for C₁₄H₂₀BO₂S [M+H]⁺ 263.1272, found 263.1270.

Experimental procedures

General procedure C for enantioconvergent $C(sp^3)-C(sp^2)$ coupling with (hetero)arylboronate esters.



An oven-dried Schlenk tube equipped with a magnetic stirring bar was charged with CuBr·Me₂S (2.06 mg, 10 mol%), chiral ligand L*3 (7.90 mg, 15 mol%), α -bromo- β -lactam E (0.12 mmol, 1.2 equiv.), (hetero)arylboronate ester A (0.1 mmol, 1.0 equiv.), LiO'Bu (24.0 mg, 0.3 mmol, 3.0 equiv.). The tube was evacuated and backfilled with argon three times. Then 1,4-dioxane/THF (v/v = 4/1, 2.0 mL) and H₂O (1.8 mg, 1.0 equiv.) were sequentially added into the mixture under argon. The tube was sealed and the reaction mixture was allowed to stir at 0 °C for 45 h. Upon completion of the reaction (monitored by TLC), the mixture was then filtered through a pad of celite and rinsed with EtOAc. The filtrate was evaporated and the residue was purified by column chromatography on silica gel to afford the desired product.

General procedure D for enantioconvergent $C(sp^3)-C(sp^2)$ coupling with alkenylboronate esters.



An oven-dried Schlenk tube equipped with a magnetic stirring bar was charged with CuBr·Me₂S (2.06 mg, 10 mol%), chiral ligand L*4 (8.40 mg, 15 mol%), α -bromo- β -lactam E1 (0.15 mmol, 1.5 equiv.), alkenylboronate ester A (0.1 mmol, 1.0 equiv.), LiO'Bu (24.0 mg, 0.3 mmol, 3.0 equiv.). The tube was evacuated and backfilled with argon three times. Then THF (2.0 mL) and H₂O (1.8 mg, 1.0 equiv.) were added under argon. The tube was sealed and the reaction mixture was allowed to stir at -40 °C for 5 d. Upon completion of the reaction (monitored by TLC), After the reaction, the mixture was then filtered through a pad of celite and rinsed with EtOAc. The filtrate was evaporated and the residue was purified by column chromatography on silica gel to afford the desired product.

The synthesis of 18.



An oven-dried Schlenk tube equipped with a magnetic stirring bar was charged with CuBr·Me₂S (2.06 mg, 10 mol%), chiral ligand L*5 (3.71 mg, 15 mol%), α -bromo- β -lactam E18 (34.7 mg, 0.1 mmol, 1.0 equiv.), arylboronate ester A1 (23.2 mg, 0.12 mmol, 1.2 equiv.), LiO'Bu (24.0 mg, 0.3 mmol, 3.0 equiv.). The tube was evacuated and backfilled with argon three times. Then dry DMF (2.0 mL) and H₂O (1.8 mg, 1.0 equiv.) were sequentially added into the mixture under argon. The tube was sealed and the reaction mixture was allowed to stir at rt for 24 h. Upon completion of the reaction (monitored by TLC), the mixture was then filtered through a pad of celite and rinsed with EtOAc. The filtrate was evaporated and the residue was purified by column chromatography on silica gel (eluent: petroleum ether/ethyl acetate 5/1) to yield the product **18** as a white solid (30.9 mg, 0.08 mmol, 80% yield).

General procedure E for the synthesis of racemates 1–17 and 19–33.



An oven-dried Schlenk tube equipped with a magnetic stirring bar was charged with CuBr·Me₂S (2.06 mg, 10 mol%), ligand Lrac (4.19 mg, 15 mol%), α -bromo- β -lactam E (0.12 mmol, 1.2 equiv.), (hetero)arylboronate ester A (0.1 mmol, 1.0 equiv.), LiO'Bu (24.0 mg, 0.3 mmol, 3.0 equiv.). The tube was evacuated and backfilled with argon three times. Then 1,4-dioxane (2.0 mL) and H₂O (1.8 mg, 1.0 equiv.) were sequentially added into the mixture under argon. The tube was sealed and the reaction mixture was allowed to stir at rt for 12 h. Upon completion of the reaction (monitored by TLC), the mixture was then filtered through a pad of celite and rinsed with EtOAc. The filtrate was evaporated and the residue was purified by column chromatography on silica gel to afford the desired product.

General procedure F for the synthesis of racemates 34-45.



An oven-dried Schlenk tube equipped with a magnetic stirring bar was charged with

CuBr·Me₂S (2.06 mg, 10 mol%), ligand Lrac (4.19 mg, 15 mol%), α -bromo- β -lactam E1 (0.15 mmol, 1.5 equiv.), alkenylboronate ester A (0.1 mmol, 1.0 equiv.), LiO'Bu (24.0 mg, 0.3 mmol, 3.0 equiv.). The tube was evacuated and backfilled with argon three times. Then 1,4-dioxane (2.0 mL) and H₂O (1.8 mg, 1.0 equiv.) were added under argon. The tube was sealed and the reaction mixture was allowed to stir at rt for 12 h. Upon completion of the reaction (monitored by TLC), After the reaction, the mixture was then filtered through a pad of celite and rinsed with EtOAc. The filtrate was evaporated and the residue was purified by column chromatography on silica gel to afford the desired product.

Analytical data

3-phenyl-1-(4-(trifluoromethyl)benzyl)azetidin-2-one (1a)



¹**H NMR (400 MHz, CDCl₃)** δ 7.64 (d, J = 7.9 Hz, 2H), 7.42 (d, J = 7.8 Hz, 2H), 7.38 - 7.31 (m, 2H), 7.31 - 7.27 (m, 2H), 4.53 (ABq, $J_{AB} = 15.4$ Hz, $\Delta v_{AB} = 23.7$ Hz, 2H), 4.43 (dd, J = 5.9, 2.5 Hz, 1H), 3.61 (t, J = 5.5 Hz, 1H), 3.24 (dd, J = 5.4, 2.6 Hz, 1H). ¹³C NMR (101 MHz, CDCl₃) δ 168.61, 139.81, 135.43, 130.31 (d, J = 32.7 Hz), 129.04, 128.58, 127.73, 127.34, 126.03 (q, J = 3.7 Hz), 124.10 (d, J = 272.2 Hz), 54.94, 47.52, 45.85.

¹⁹F NMR (376 MHz, CDCl₃) δ -62.56.

HRMS (ESI) *m/z* calcd. for C₁₇H₁₄F₃NO [M+H]⁺ 306.1100, found 306.1092.

3-(3,5-di-*tert*-butyl-1-methyl-4-oxocyclohexa-2,5-dien-1-yl)-3-phenyl-1-(4-(trifluoromethyl)benzyl)azetidin-2-one (1b)



¹**H NMR (400 MHz, CDCl₃)** δ 7.58 (d, *J* = 8.0 Hz, 2H), 7.42 – 7.28 (m, 7H), 6.56 (s, 2H), 4.42 (s, 2H), 3.30 (ABq, *J*_{AB} = 5.6 Hz, Δv_{AB} = 21.1 Hz, 2H), 1.32 (s, 3H), 1.16 (s, 18H).

¹³C NMR (101 MHz, CDCl₃) δ 185.77, 168.91, 148.86, 147.13, 143.00, 141.99, 139.33, 136.05, 130.41 (q, J = 32.6 Hz), 129.19, 128.79, 127.97, 127.71, 126.07 (q, J = 3.8 Hz), 124.04 (q, J = 272.4 Hz), 67.95, 49.49, 45.73, 42.66, 35.03, 35.00, 29.44, 29.40, 21.62. ¹⁹F NMR (376 MHz, CDCl₃) δ -62.67.

HRMS (ESI) *m/z* calcd. for C₃₂H₃₇F₃NO₂ [M+H]⁺ 524.2771, found 524.2772.

(S)-3-(4-acetylphenyl)-3-phenyl-1-(4-(trifluoromethyl)benzyl)azetidin-2-one (1)



F₃C

According to the **general procedure** C with E1 (46.1 mg, 0.12 mmol) and 1-(4-(5,5-dimethyl-1,3,2-dioxaborinan-2-yl)phenyl)ethan-1-one A1 (23.2 mg, 0.10 mmol), the reaction mixture was purified by column chromatography on silica gel (eluent: petroleum ether/ethyl acetate 5/1) to yield the product as a colorless oil (32.6 mg, 0.077 mmol, 77% yield).

¹**H NMR (400 MHz, CDCl₃)** δ 7.92 (d, *J* = 8.2 Hz, 2H), 7.59 (d, *J* = 8.0 Hz, 2H), 7.51 (d, *J* = 8.2 Hz, 2H), 7.42 - 7.27 (m, 7H), 4.56 (ABq, *J*_{AB} = 15.4 Hz, Δv_{AB} = 12.4 Hz, 2H), 3.83 (ABq, *J*_{AB} = 5.6 Hz, Δv_{AB} = 5.7 Hz, 2H), 2.57 (s, 3H).

¹³C NMR (101 MHz, CDCl₃) δ 197.68, 169.02, 145.30, 139.48, 139.30, 136.23, 130.36 (q, *J* = 32.6 Hz), 129.04, 128.92, 128.47, 127.83, 127.36, 127.01, 126.06 (q, *J* = 3.8 Hz), 124.02 (q, *J* = 272.1 Hz), 66.99, 54.03, 45.80, 26.74.

¹⁹F NMR (376 MHz, CDCl₃) δ -62.59.

HPLC conditions: Nu-Analytical Solutions INA, *n*-hexane/*i*-PrOH = 85/15, flow rate 0.8 mL/min. $\lambda = 254$ nm, t(major) = 30.7 min, t(minor) = 41.7 min, 98% ee. **HRMS** (ESI) *m*/*z* calcd. for C₂₅H₂₁F₃NO₂ [M+H]⁺ 424.1519, found 424.1515.

(S)-3-(4-acetylphenyl)-1-benzyl-3-phenylazetidin-2-one (2)



According to the **general procedure** C with E2 (37.9 mg, 0.12 mmol) and 1-(4-(5,5-dimethyl-1,3,2-dioxaborinan-2-yl)phenyl)ethan-1-one A1 (23.2 mg, 0.10 mmol), the reaction mixture was purified by column chromatography on silica gel (eluent: petroleum ether/ethyl acetate 5/1) to yield the product as a colorless oil (27.0 mg, 0.076 mmol, 76% yield).

¹**H NMR (400 MHz, CDCl₃)** δ 7.93 (d, *J* = 8.2 Hz, 2H), 7.52 (d, *J* = 8.1 Hz, 2H), 7.45 – 7.19 (m, 10H), 4.53 (ABq, *J*_{AB} = 15.0 Hz, Δv_{AB} = 25.4 Hz, 2H), 3.88 – 3.70 (m, 2H), 2.59 (s, 3H).

¹³C NMR (101 MHz, CDCl₃) δ 197.54, 168.72, 145.41, 139.52, 135.98, 134.99, 128.14, 127.89, 127.51, 127.28, 126.96, 66.43, 53.69, 46.12, 26.54.

HPLC conditions: Nu-Analytical Solutions INA, *n*-hexane/*i*-PrOH = 85/15, flow rate

1.0 mL/min. $\lambda = 254$ nm, t(major) = 23.6 min, t(minor) = 28.4 min, 99% ee. HRMS (ESI) *m*/*z* calcd. for C₂₄H₂₂NO₂ [M+H]⁺ 356.1645, found 356.1643.

(S)-3-(4-acetylphenyl)-1-(4-bromobenzyl)-3-phenylazetidin-2-one (3)



According to the **general procedure C** with **E3** (47.4 mg, 0.12 mmol) and 1-(4-(5,5-dimethyl-1,3,2-dioxaborinan-2-yl)phenyl)ethan-1-one **A1** (23.2 mg, 0.10 mmol), the reaction mixture was purified by column chromatography on silica gel (eluent: petroleum ether/ethyl acetate 5/1) to yield the product as a colorless oil (31.3 mg, 0.072 mmol, 72% yield).

¹H NMR (400 MHz, CDCl₃) δ 7.97 – 7.89 (m, 2H), 7.51 (d, J = 8.3 Hz, 2H), 7.49 – 7.45 (m, 2H), 7.42 – 7.26 (m, 5H), 7.13 (d, J = 8.3 Hz, 2H), 4.47 (ABq, $J_{AB} = 15.1$ Hz, $\Delta v_{AB} = 16.9$ Hz, 2H), 3.80 (ABq, $J_{AB} = 5.5$ Hz, $\Delta v_{AB} = 6.2$ Hz, 2H). ¹³C NMR (101 MHz, CDCl₃) δ 197.64, 168.87, 145.36, 139.52, 136.18, 134.23, 132.19, 129.94, 128.99, 128.87, 127.75, 127.36, 127.03, 122.06, 66.77, 53.84, 45.65, 26.71. HPLC conditions: Nu-Analytical Solutions INA, *n*-hexane/*i*-PrOH = 85/15, flow rate 1.0 mL/min. $\lambda = 254$ nm, t(major) = 28.6 min, t(minor) = 34.7 min, 98% ee. HRMS (ESI) *m*/*z* calcd. for C₂₄H₂₁BrNO₂ [M+H]⁺ 434.0750, found 434.0749.

(S)-3-(4-acetylphenyl)-1-(4-(tert-butyl)benzyl)-3-phenylazetidin-2-one (4)



According to the **general procedure C** with **E4** (44.7 mg, 0.12 mmol) and 1-(4-(5,5-dimethyl-1,3,2-dioxaborinan-2-yl)phenyl)ethan-1-one **A1** (23.2 mg, 0.10 mmol), the reaction mixture was purified by column chromatography on silica gel (eluent: petroleum ether/ethyl acetate 6/1) to yield the product as a colorless oil (33.7 mg, 0.082 mmol, 82% yield).

¹**H NMR (400 MHz, CDCl₃)** δ 7.91 (d, *J* = 8.2 Hz, 2H), 7.50 (d, *J* = 8.2 Hz, 2H), 7.41 – 7.30 (m, 6H), 7.29 – 7.23 (m, 1H), 7.16 (d, *J* = 8.1 Hz, 2H), 4.47 (ABq, *J*_{AB} = 15.0 Hz, $\Delta v_{AB} = 24.3$ Hz, 2H), 3.82 – 3.75 (m, 2H), 2.57 (s, 3H), 1.30 (s, 9H).

¹³C NMR (101 MHz, CDCl₃) δ 197.73, 168.84, 151.02, 145.65, 139.75, 136.08, 132.01, 128.92, 128.82, 127.96, 127.63, 127.45, 127.12, 125.92, 66.46, 53.88, 45.85, 34.66, 31.40, 26.73.

HPLC conditions: Nu-Analytical Solutions INA, *n*-hexane/*i*-PrOH = 85/15, flow rate 1.0 mL/min. $\lambda = 254$ nm, t(major) = 15.9 min, t(minor) = 25.5 min, 98% ee. **HRMS** (ESI) *m/z* calcd. for C₂₈H₃₀NO₂ [M+H]⁺ 412.2271, found 412.2267.

(S)-3-([1,1'-biphenyl]-4-yl)-1-(4-methoxybenzyl)-3-phenylazetidin-2-one (5)



According to the **general procedure C** with **E5** (41.4 mg, 0.12 mmol) and 2-([1,1'-biphenyl]-4-yl)-5,5-dimethyl-1,3,2-dioxaborinane **A17** (26.6 mg, 0.10 mmol), the reaction mixture was purified by column chromatography on silica gel (eluent: petroleum ether/ethyl acetate 5/1) to yield the product as a colorless oil (28.4 mg, 0.068 mmol, 68% yield).

¹H NMR (400 MHz, CDCl₃) δ 7.62 – 7.49 (m, 4H), 7.48 – 7.39 (m, 6H), 7.37 – 7.31 (m, 3H), 7.30 – 7.22 (m, 1H), 7.23 – 7.12 (m, 2H), 6.95 – 6.79 (m, 2H), 4.45 (s, 2H), 3.79 (s, 3H), 3.76 (ABq, $J_{AB} = 5.4$ Hz, $\Delta v_{AB} = 4.9$ Hz, 2H).

¹³C NMR (101 MHz, CDCl₃) δ 169.39, 159.27, 140.63, 140.30, 140.16, 139.39, 129.62, 128.80, 128.74, 127.54, 127.40, 127.38, 127.33, 127.29, 127.18, 127.08, 114.29, 66.12, 55.32, 53.98, 45.56.

HPLC conditions: Nu-Analytical Solutions INB, *n*-hexane/*i*-PrOH = 80/20, flow rate 0.4 mL/min. $\lambda = 254$ nm, t(major) = 21.1 min, t(minor) = 29.0 min, 93% ee. HRMS (ESI) *m*/*z* calcd. for C₂₉H₂₆NO₂ [M+H]⁺ 420.1958, found 420.1955.

(S)-3-(4-acetylphenyl)-1-phenethyl-3-phenylazetidin-2-one (6)



According to the **general procedure C** with **E6** (39.6 mg, 0.12 mmol) and 1-(4-(5,5-dimethyl-1,3,2-dioxaborinan-2-yl)phenyl)ethan-1-one **A1** (23.2 mg, 0.10 mmol), the reaction mixture was purified by column chromatography on silica gel (eluent: petroleum ether/ethyl acetate 5/1) to yield the product as a colorless oil (28.1 mg, 0.076 mmol, 76% yield).

¹**H NMR (400 MHz, CDCl₃)** δ 7.87 (d, *J* = 8.3 Hz, 2H), 7.37 (d, *J* = 8.3 Hz, 2H), 7.33 – 7.19 (m, 8H), 7.18 – 7.13 (m, 2H), 3.69 (ABq, *J*_{AB} = 5.4 Hz, Δv_{AB} = 9.1 Hz, 2H), 3.66 – 3.55 (m, 2H), 2.91 (t, *J* = 7.1 Hz, 2H).

¹³C NMR (101 MHz, CDCl₃) δ 197.74, 168.73, 145.51, 139.60, 138.17, 136.03, 128.84, 128.78, 128.75, 128.73, 127.57, 127.46, 127.15, 126.80, 66.36, 54.92, 43.13, 34.18, 26.71.

HPLC conditions: Nu-Analytical Solutions INA, *n*-hexane/*i*-PrOH = 85/15, flow rate 1.0 mL/min. $\lambda = 254$ nm, t(major) = 26.7 min, t(minor) = 30.3 min, 99% ee. **HRMS** (ESI) *m/z* calcd. for C₂₅H₂₄NO₂ [M+H]⁺ 370.1802, found 370.1800.

(S)-3-(4-acetylphenyl)-1-ethyl-3-phenylazetidin-2-one (7)



According to the **general procedure C** with E7 (30.5 mg, 0.12 mmol) and 1-(4-(5,5-dimethyl-1,3,2-dioxaborinan-2-yl)phenyl)ethan-1-one A1 (23.2 mg, 0.10 mmol), the reaction mixture was purified by column chromatography on silica gel (eluent: petroleum ether/ethyl acetate 6/1) to yield the product as a colorless oil (27.0 mg, 0.092 mmol, 92% yield).

¹H NMR (400 MHz, CDCl₃) δ 7.92 (d, J = 8.3 Hz, 2H), 7.52 (d, J = 8.3 Hz, 2H), 7.42 - 7.37 (m, 2H), 7.37 - 7.31 (m, 2H), 7.30 - 7.24 (m, 1H), 3.88 (ABq, $J_{AB} = 5.4$ Hz, $\Delta v_{AB} = 4.2$ Hz, 2H), 3.38 (q, J = 7.3 Hz, 2H), 2.57 (s, 3H), 1.20 (t, J = 7.3 Hz, 3H). ¹³C NMR (101 MHz, CDCl₃) δ 197.75, 168.49, 145.72, 139.82, 136.11, 128.94, 128.83, 127.64, 127.47, 127.12, 65.99, 53.66, 36.70, 26.73, 12.83. HPLC conditions: Nu-Analytical Solutions INA, *n*-hexane/*i*-PrOH = 85/15, flow rate 1.0 mL/min. $\lambda = 254$ nm, t(major) = 14.3 min, t(minor) = 17.2 min, 99% ee. HRMS (ESI) *m*/z calcd. for C₁₉H₂₀NO₂ [M+H]⁺ 294.1489, found 294.1486.

(S)-3-(4-acetylphenyl)-1-cyclopropyl-3-phenylazetidin-2-one (8)



According to the **general procedure** C with **E8** (31.9 mg, 0.12 mmol) and 1-(4-(5,5-dimethyl-1,3,2-dioxaborinan-2-yl)phenyl)ethan-1-one **A1** (23.2 mg, 0.10 mmol), the reaction mixture was purified by column chromatography on silica gel (eluent: petroleum ether/ethyl acetate 6/1) to yield the product as a colorless oil (24.1 mg, 0.079 mmol, 79% yield).

¹**H NMR (400 MHz, CDCl₃)** δ 7.91 (d, *J* = 8.4 Hz, 2H), 7.49 (d, *J* = 8.4 Hz, 2H), 7.39 – 7.30 (m, 4H), 7.29 – 7.24 (m, 1H), 3.83 (ABq, *J*_{AB} = 5.5 Hz, Δv_{AB} = 4.0 Hz, 2H), 2.69 – 2.61 (m, 1H), 2.57 (s, 3H), 0.87 – 0.81 (m, 2H), 0.80 – 0.73 (m, 2H).

¹³C NMR (101 MHz, CDCl₃) δ 197.72, 169.21, 145.63, 139.74, 136.10, 128.93, 128.82, 127.64, 127.41, 127.08, 65.09, 54.72, 26.72, 24.43, 5.43.

HPLC conditions: Nu-Analytical Solutions INA, *n*-hexane/*i*-PrOH = 85/15, flow rate 1.0 mL/min. $\lambda = 254$ nm, t(major) = 20.6 min, t(minor) = 22.5 min, 98% ee. **HRMS** (ESI) *m/z* calcd. for C₂₀H₂₀NO₂ [M+H]⁺ 306.1489, found 306.1486.

(S)-3-(4-acetylphenyl)-1-cyclopentyl-3-phenylazetidin-2-one (9)



According to the **general procedure** C with **E9** (35.3 mg, 0.12 mmol) and 1-(4-(5,5dimethyl-1,3,2-dioxaborinan-2-yl)phenyl)ethan-1-one **A1** (23.2 mg, 0.10 mmol), the reaction mixture was purified by column chromatography on silica gel (eluent: petroleum ether/ethyl acetate 5/1) to yield the product as a colorless oil (22.0 mg, 0.066 mmol, 66% yield).

¹H NMR (400 MHz, CDCl₃) δ 7.96 – 7.89 (m, 2H), 7.54 – 7.47 (m, 2H), 7.41 – 7.31 (m, 4H), 7.30 – 7.24 (m, 2H), 4.23 – 4.12 (m, 1H), 3.84 (ABq, $J_{AB} = 5.4$ Hz, $\Delta v_{AB} = 4.3$ Hz, 2H), 2.57 (s, 3H), 1.96 – 1.83 (m, 2H), 1.76 – 1.57 (m, 6H). ¹³C NMR (101 MHz, CDCl₃) δ 197.76, 168.31, 145.80, 139.87, 136.08, 128.92, 128.83, 127.59, 127.49, 127.15, 64.72, 53.39, 52.33, 30.31, 30.29, 26.73, 24.15. HPLC conditions: Nu-Analytical Solutions INA, *n*-hexane/*i*-PrOH = 85/15, flow rate 1.0 mL/min. $\lambda = 254$ nm, t(major) = 17.6 min, t(minor) = 22.9 min, 98% ee. HRMS (ESI) *m*/z calcd. for C₂₀H₂₄NO₂ [M+H]⁺ 334.1802, found 334.1799.

(S)-3-(4-acetylphenyl)-1-cyclohexyl-3-phenylazetidin-2-one (10)



According to the **general procedure C** with **E10** (37.0 mg, 0.12 mmol) and 1-(4-(5,5dimethyl-1,3,2-dioxaborinan-2-yl)phenyl)ethan-1-one **A1** (23.2 mg, 0.10 mmol), the reaction mixture was purified by column chromatography on silica gel (eluent: petroleum ether/ethyl acetate 6/1) to yield the product as a colorless oil (21.9 mg, 0.063 mmol, 63% yield). ¹**H NMR (400 MHz, CDCl₃)** δ 7.92 (d, *J* = 8.3 Hz, 2H), 7.50 (d, *J* = 8.3 Hz, 2H), 7.41 – 7.30 (m, 4H), 7.30 – 7.24 (m, 1H), 3.92 – 3.79 (m, 2H), 3.73 – 3.60 (m, 1H), 2.58 (s, 3H), 1.98 – 1.57 (m, 6H), 1.48 – 1.18 (m, 4H).

¹³C NMR (101 MHz, CDCl₃) δ 197.76, 168.03, 145.83, 139.90, 136.09, 128.92, 128.82, 127.58, 127.53, 127.18, 64.76, 51.93, 51.30, 30.83, 26.72, 25.38, 24.92.

HPLC conditions: Nu-Analytical Solutions INA, *n*-hexane/*i*-PrOH = 90/10, flow rate 1.0 mL/min. $\lambda = 254$ nm, t(major) = 33.9 min, t(minor) = 51.2 min, 97% ee. **HRMS** (ESI) *m*/*z* calcd. for C₂₃H₂₆NO₂ [M+H]⁺ 348.1958, found 348.1954.

(S)-3-(4-acetylphenyl)-1-cycloheptyl-3-phenylazetidin-2-one (11)



According to the **general procedure C** with **E11** (38.7 mg, 0.12 mmol) and 1-(4-(5,5-dimethyl-1,3,2-dioxaborinan-2-yl)phenyl)ethan-1-one **A1** (23.2 mg, 0.10 mmol), the reaction mixture was purified by column chromatography on silica gel (eluent: petroleum ether/ethyl acetate 6/1) to yield the product as a colorless oil (19.9 mg, 0.055 mmol, 55% yield).

¹**H NMR (400 MHz, CDCl₃)** δ 7.97 – 7.87 (m, 2H), 7.51 (dd, *J* = 8.6, 2.9 Hz, 2H), 7.41 – 7.24 (m, 5H), 3.95 – 3.78 (m, 3H), 2.58 (s, 3H), 2.03 – 1.89 (m, 2H), 1.75 – 1.42 (m, 12H).

¹³C NMR (101 MHz, CDCl₃) δ 197.75, 167.58, 145.81, 139.88, 136.08, 128.92, 128.82, 127.58, 127.53, 127.18, 64.91, 53.29, 52.06, 32.94, 32.92, 28.04, 26.72, 24.36.

HPLC conditions: Nu-Analytical Solutions INA, *n*-hexane/*i*-PrOH = 90/10, flow rate 1.0 mL/min. $\lambda = 254$ nm, t(major) = 33.6 min, t(minor) = 45.7 min, 97% ee. **HRMS** (ESI) *m/z* calcd. for C₂₄H₂₈NO₂ [M+H]⁺ 362.2115, found 362.2112.

(*R*)-3-(4-acetylphenyl)-3-(3-methoxyphenyl)-1-(4-(trifluoromethyl)benzyl)azetidin-2-one (12)



According to the **general procedure C** with **E12** (49.7 mg, 0.12 mmol) and 1-(4-(5,5-dimethyl-1,3,2-dioxaborinan-2-yl)phenyl)ethan-1-one **A1** (23.2 mg, 0.10 mmol), the reaction mixture was purified by column chromatography on silica gel (eluent:

petroleum ether/ethyl acetate 5/1) to yield the product as a colorless oil (35.8 mg, 0.079 mmol, 79% yield).

¹**H NMR (400 MHz, CDCl₃)** δ 7.92 (d, *J* = 8.3 Hz, 2H), 7.59 (d, *J* = 8.1 Hz, 2H), 7.51 (d, *J* = 8.3 Hz, 2H), 7.35 (d, *J* = 8.0 Hz, 2H), 7.26 (t, *J* = 7.9 Hz, 1H), 6.98 – 6.90 (m, 2H), 6.82 (dd, *J* = 8.2, 2.5 Hz, 1H), 4.55 (s, 2H), 3.84 (ABq, *J*_{AB} = 5.5 Hz, Δv_{AB} = 9.3 Hz, 2H), 3.76 (s, 3H), 2.57 (s, 3H).

¹³C NMR (101 MHz, CDCl₃) δ 197.66, 168.87, 159.95, 145.16, 140.94, 139.31, 136.22, 130.32 (q, J = 32.7 Hz), 130.04, 128.89, 128.47, 127.32, 126.04 (q, J = 3.7 Hz), 124.01 (q, J = 272.1 Hz),119.27, 113.09, 112.93, 66.89, 55.33, 54.07, 45.78, 26.71. ¹⁹F NMR (376 MHz, CDCl₃) δ -62.59.

HPLC conditions: Nu-Analytical Solutions INA, *n*-hexane/*i*-PrOH = 85/15, flow rate 1.0 mL/min. $\lambda = 254$ nm, t(major) = 27.2 min, t(minor) = 46.1 min, 99% ee. **HRMS** (ESI) *m*/*z* calcd. for C₂₆H₂₃F₃NO₃ [M+H]⁺ 454.1625, found 454.1621.

(S)-3-(4-acetylphenyl)-1-benzyl-3-(4-(tert-butyl)phenyl)azetidin-2-one (13)



According to the **general procedure C** with **E13** (44.6 mg, 0.12 mmol) and 1-(4-(5,5-dimethyl-1,3,2-dioxaborinan-2-yl)phenyl)ethan-1-one **A1** (23.2 mg, 0.10 mmol), the reaction mixture was purified by column chromatography on silica gel (eluent: petroleum ether/ethyl acetate 6/1) to yield the product as a white solid (32.9 mg, 0.080 mmol, 80% yield).

¹**H NMR (400 MHz, CDCl₃)** δ 7.94 (d, *J* = 8.1 Hz, 2H), 7.53 (d, *J* = 8.0 Hz, 2H), 7.41 – 7.23 (m, 9H), 4.52 (ABq, *J*_{AB} = 15.0 Hz, Δv_{AB} = 41.9 Hz, 2H), 3.80 (ABq, *J*_{AB} = 5.5 Hz, Δv_{AB} = 13.4 Hz, 2H), 2.60 (s, 3H), 1.32 (s, 9H).

¹³C NMR (101 MHz, CDCl₃) δ 197.66, 169.01, 150.49, 145.69, 136.57, 136.01, 135.17, 128.96, 128.74, 128.29, 127.96, 127.44, 126.65, 125.78, 66.23, 53.89, 46.22, 34.52, 31.30, 26.64.

HPLC conditions: Nu-Analytical Solutions INA, *n*-hexane/*i*-PrOH = 85/15, flow rate 1.0 mL/min. $\lambda = 254$ nm, t(major) = 15.5 min, t(minor) = 18.1 min, 98% ee. **HRMS** (ESI) *m/z* calcd. for C₂₈H₃₀NO₂ [M+H]⁺ 412.2271, found 412.2268.

(*R*)-3-(4-acetylphenyl)-3-(4-fluorophenyl)-1-(4-(trifluoromethyl)benzyl)azetidin-2-one (14)



According to the **general procedure C** with **E14** (48.3 mg, 0.12 mmol) and 1-(4-(5,5-dimethyl-1,3,2-dioxaborinan-2-yl)phenyl)ethan-1-one **A1** (23.2 mg, 0.10 mmol), the reaction mixture was purified by column chromatography on silica gel (eluent: petroleum ether/ethyl acetate 5/1) to yield the product as a colorless oil (37.1 mg, 0.084 mmol, 84% yield).

¹**H NMR (400 MHz, CDCl₃)** δ 7.92 (d, J = 8.4 Hz, 2H), 7.59 (d, J = 8.1 Hz, 2H), 7.47 (d, J = 8.4 Hz, 2H), 7.39 – 7.30 (m, 4H), 7.07 – 6.97 (m, 2H), 4.56 (ABq, $J_{AB} = 15.8$ Hz, $\Delta v_{AB} = 6.9$ Hz, 2H), 3.80 (ABq, $J_{AB} = 5.6$ Hz, $\Delta v_{AB} = 10.6$ Hz, 2H), 2.57 (s, 3H). ¹³**C NMR (101 MHz, CDCl₃)** δ 197.56, 168.81, 162.23 (d, J = 247.4 Hz), 145.08, 139.23, 136.39, 135.34 (d, J = 3.3 Hz), 130.46 (q, J = 32.6 Hz), 128.99, 128.77 (d, J = 8.0 Hz), 128.47, 127.27, 126.10 (q, J = 3.8 Hz), 124.02 (q, J = 272.1 Hz), 115.97 (d, J = 21.6 Hz), 66.35, 54.13, 45.85, 26.71.

¹⁹**F NMR** (376 MHz, CDCl₃) δ -62.62, -114.17.

HPLC conditions: Nu-Analytical Solutions INA, *n*-hexane/*i*-PrOH = 85/15, flow rate 1.0 mL/min. $\lambda = 254$ nm, t(major) = 20.8 min, t(minor) = 29.5 min, 98% ee. **HRMS** (ESI) *m/z* calcd. for C₂₅H₂₀F₄NO₂ [M+H]⁺ 442.1425, found 442.1423.

(R)-3-(4-acetylphenyl)-1-benzyl-3-(4-chlorophenyl)azetidin-2-one (15)



According to the **general procedure C** with **E15** (42.1 mg, 0.12 mmol) and 1-(4-(5,5-dimethyl-1,3,2-dioxaborinan-2-yl)phenyl)ethan-1-one **A1** (23.2 mg, 0.10 mmol), the reaction mixture was purified by column chromatography on silica gel (eluent: petroleum ether/ethyl acetate 5/1) to yield the product as a colorless oil (33.1 mg, 0.085 mmol, 85% yield).

¹**H NMR (400 MHz, CDCl₃)** δ 7.91 (d, *J* = 8.2 Hz, 2H), 7.46 (d, *J* = 8.2 Hz, 2H), 7.37 – 7.25 (m, 7H), 7.22 (d, *J* = 7.4 Hz, 2H), 4.49 (s, 2H), 3.75 (ABq, *J*_{AB} = 5.7 Hz, Δ v_{AB} = 11.9 Hz, 2H), 2.57 (s, 3H).

¹³C NMR (101 MHz, CDCl₃) δ 197.62, 168.42, 145.02, 138.17, 136.30, 134.99, 133.68, 129.12, 129.09, 128.94, 128.52, 128.28, 128.14, 127.34, 66.00, 53.73, 46.33, 26.72. HPLC conditions: Nu-Analytical Solutions INA, *n*-hexane/*i*-PrOH = 85/15, flow rate 1.0 mL/min. λ = 254 nm, t(major) = 26.7 min, t(minor) = 31.1 min, 99% ee. HRMS (ESI) *m*/*z* calcd. for C₂₄H₂₁ClNO₂ [M+H]⁺ 390.1255, found 390.1252.

(*R*)-3-(4-acetylphenyl)-3-(4-bromophenyl)-1-(4-(trifluoromethyl)benzyl)azetidin-2-one (16)



According to the **general procedure C** with **E16** (55.6 mg, 0.12 mmol) and 1-(4-(5,5-dimethyl-1,3,2-dioxaborinan-2-yl)phenyl)ethan-1-one **A1** (23.2 mg, 0.10 mmol), the reaction mixture was purified by column chromatography on silica gel (eluent: petroleum ether/ethyl acetate 6/1) to yield the product as a colorless oil (38.2 mg, 0.076 mmol, 76% yield).

¹**H NMR (400 MHz, CDCl₃)** δ 7.92 (d, *J* = 8.3 Hz, 2H), 7.59 (d, *J* = 8.1 Hz, 2H), 7.51 – 7.42 (m, 4H), 7.33 (d, *J* = 8.0 Hz, 2H), 7.29 – 7.23 (m, 2H), 4.55 (s, 2H), 3.79 (ABq, *J*_{AB} = 5.6 Hz, Δv_{AB} = 12.0 Hz, 2H), 2.57 (s, 3H).

¹³C NMR (101 MHz, CDCl₃) δ 197.55, 168.47, 144.70, 139.13, 138.50, 136.42, 132.17, 130.46 (q, *J* = 32.6 Hz), 129.02, 128.75, 128.46, 127.25, 126.11 (q, *J* = 3.8 Hz), 123.99 (q, *J* = 272.2 Hz), 121.97, 66.46, 53.86, 45.86, 26.73.

¹⁹F NMR (**376** MHz, CDCl₃) δ -62.60.

HPLC conditions: Nu-Analytical Solutions INA, *n*-hexane/*i*-PrOH = 85/15, flow rate 1.0 mL/min. $\lambda = 254$ nm, t(major) = 28.1 min, t(minor) = 36.9 min, 98% ee. **HRMS** (ESI) *m/z* calcd. for C₂₅H₂₀BrF₃NO₂ [M+H]⁺ 502.0624, found 502.0623.

(S)-3-([1,1'-biphenyl]-4-yl)-3-(4-acetylphenyl)-1-(4-(trifluoromethyl)benzyl)azetidin-2-one (17)



According to the general procedure C with E17 (55.2 mg, 0.12 mmol) and 1-(4-(5,5-

dimethyl-1,3,2-dioxaborinan-2-yl)phenyl)ethan-1-one A1 (23.2 mg, 0.10 mmol), the reaction mixture was purified by column chromatography on silica gel (eluent: petroleum ether/ethyl acetate 5/1) to yield the product as a white solid (39.9 mg, 0.080 mmol, 80% yield).

¹**H NMR (400 MHz, CDCl₃)** δ 7.95 (d, J = 8.4 Hz, 2H), 7.64 – 7.51 (m, 8H), 7.48 – 7.40 (m, 4H), 7.39 – 7.32 (m, 3H), 4.58 (ABq, $J_{AB} = 15.4$ Hz, $\Delta v_{AB} = 12.4$ Hz, 2H), 3.86 (ABq, $J_{AB} = 5.5$ Hz, $\Delta v_{AB} = 11.6$ Hz, 2H), 2.59 (s, 3H).

¹³C NMR (101 MHz, CDCl₃) δ 197.66, 168.98, 145.25, 140.76, 140.41, 139.30, 138.46, 136.31, 130.40 (q, J = 32.6 Hz), 128.98, 128.95, 128.50, 127.73, 127.65, 127.43, 127.39, 127.15, 126.09 (q, J = 3.8 Hz), 124.03 (q, J = 272.2 Hz), 66.79, 54.06, 45.85, 26.74. ¹⁹F NMR (376 MHz, CDCl₃) δ -62.56.

HPLC conditions: Nu-Analytical Solutions INA, *n*-hexane/*i*-PrOH = 85/15, flow rate 1.0 mL/min. $\lambda = 254$ nm, t(major) = 39.2 min, t(minor) = 45.6 min, 99% ee. **HRMS** (ESI) *m/z* calcd. for C₃₁H₂₅F₃NO₂ [M+H]⁺ 500.1832, found 500.1831.

(S)-3-(4-acetylphenyl)-1-(4-bromophenyl)-3-isopropylazetidin-2-one (18)



¹**H** NMR (400 MHz, CDCl₃) δ 7.96 (d, J = 7.9 Hz, 2H), 7.57 (d, J = 8.0 Hz, 2H), 7.48 – 7.40 (m, 2H), 7.31 – 7.22 (m, 2H), 3.86 (ABq, $J_{AB} = 5.9$ Hz, $\Delta v_{AB} = 7.7$ Hz, 2H), 2.61 (s, 3H), 2.38 – 2.23 (m, 1H), 1.01 (d, J = 6.8 Hz, 3H), 0.99 (d, J = 6.7 Hz, 3H). ¹³C NMR (101 MHz, CDCl₃) δ 197.71, 167.27, 143.08, 136.95, 136.06, 132.14, 128.17, 127.90, 117.83, 116.56, 66.28, 48.50, 34.57, 26.64, 18.47, 17.73. HPLC conditions: Chiralcel ODH, *n*-hexane/*i*-PrOH = 90/10, flow rate 0.8 mL/min. λ = 254 nm, t(major) = 13.3 min, t(minor) = 18.9 min, 56% ee. HRMS (ESI) *m*/*z* calcd. for C₂₀H₂₁BrNO₂ [M+H]⁺ 386.0750, found 386.0746.

(S)-3-(4-methoxyphenyl)-3-phenyl-1-(4-(trifluoromethyl)benzyl)azetidin-2-one (19)



According to the general procedure C with E1 (46.1 mg, 0.12 mmol) and 2-(4-methoxyphenyl)-5,5-dimethyl-1,3,2-dioxaborinane A3 (22.0 mg, 0.10 mmol), the

reaction mixture was purified by column chromatography on silica gel (eluent: petroleum ether/ethyl acetate 7/1) to yield the product as a white solid (18.9 mg, 0.046 mmol, 46% yield).

¹**H NMR (400 MHz, CDCl₃)** δ 7.59 (d, *J* = 7.9 Hz, 2H), 7.42 – 7.21 (m, 9H), 6.86 (d, *J* = 8.6 Hz, 2H), 4.55 (s, 2H), 3.83 – 3.70 (m, 5H).

¹³C NMR (101 MHz, CDCl₃) δ 170.06, 158.93, 140.50, 139.64, 132.37, 130.29 (q, J = 32.5 Hz), 128.85, 128.50, 128.30, 127.45, 127.11, 126.02 (q, J = 3.7 Hz), 124.10 (q, J = 272.0 Hz), 114.24, 66.49, 55.41, 54.66, 45.72.

¹⁹F NMR (376 MHz, CDCl₃) δ -62.56.

HPLC conditions: Chiralcel IH, *n*-hexane/*i*-PrOH = 90/10, flow rate 0.8 mL/min. λ = 230 nm, t(minor) = 22.7 min, t(major) = 33.4 min, 95% ee.

HRMS (ESI) m/z calcd. for C₂₄H₂₁F₃NO₂ [M+H]⁺ 412.1519, found 412.1517.

(S)-3-(3,5-dimethoxyphenyl)-3-phenyl-1-(4-(trifluoromethyl)benzyl)azetidin-2one (20)



According to the **general procedure C** with **E1** (46.1 mg, 0.12 mmol) and 2-(3,5dimethoxyphenyl)-5,5-dimethyl-1,3,2-dioxaborinane **A4** (25.0 mg, 0.10 mmol), the reaction mixture was purified by column chromatography on silica gel (eluent: petroleum ether/ethyl acetate 7/1) to yield the product as a white solid (29.1 mg, 0.066 mmol, 66% yield).

¹H NMR (400 MHz, CDCl₃) δ 7.59 (d, J = 7.1 Hz, 2H), 7.46 – 7.19 (m, 7H), 6.54 (s, 2H), 6.36 (s, 1H), 4.54 (ABq, $J_{AB} = 15.4$ Hz, $\Delta v_{AB} = 22.2$ Hz, 2H), 3.85 – 3.62 (m, 8H). ¹³C NMR (101 MHz, CDCl₃) δ 169.56, 161.04, 142.43, 139.95, 139.58, 130.28 (q, J = 32.6 Hz), 128.84, 128.53, 127.57, 127.07, 126.02 (q, J = 3.7 Hz), 124.08 (q, J = 272.0 Hz), 105.51, 99.21, 67.05, 55.44, 54.54, 45.74.

¹⁹F NMR (**376** MHz, CDCl₃) δ -62.58.

HPLC conditions: Nu-Analytical Solutions INA, *n*-hexane/*i*-PrOH = 85/15, flow rate 0.8 mL/min. $\lambda = 214$ nm, t(minor) = 13.9 min, t(major) = 22.2 min, 98% ee. **HRMS** (ESI) *m*/*z* calcd. for C₂₅H₂₃F₃NO₃ [M+H]⁺ 442.1625, found 442.1621.

(S)-3-(4-bromophenyl)-3-phenyl-1-(4-(trifluoromethyl)benzyl)azetidin-2-one (21)



According to the **general procedure C** with **E1** (46.1 mg, 0.12 mmol) and 2-(4bromophenyl)-5,5-dimethyl-1,3,2-dioxaborinane **A5** (26.9 mg, 0.10 mmol), the reaction mixture was purified by column chromatography on silica gel (eluent: petroleum ether/ethyl acetate 10/1) to yield the product as a colorless oil (33.1 mg, 0.072 mmol, 72% yield).

¹**H NMR (400 MHz, CDCl₃)** δ 7.59 (d, *J* = 8.1 Hz, 2H), 7.45 (d, *J* = 8.4 Hz, 2H), 7.38 – 7.30 (m, 6H), 7.29 – 7.23 (m, 3H), 4.54 (ABq, *J*_{AB} = 15.5 Hz, Δv_{AB} = 9.5 Hz, 2H), 3.77 (ABq, *J*_{AB} = 5.5 Hz, Δv_{AB} = 18.4 Hz, 2H).

¹³C NMR (101 MHz, CDCl₃) δ 169.29, 139.68, 139.37, 139.25, 132.01, 130.41 (q, J = 32.6 Hz), 129.01, 128.88, 128.49, 127.76, 127.02, 126.08 (q, J = 3.7 Hz), 124.06 (q, J = 272.1 Hz), 121.65, 66.55, 54.25, 45.81.

¹⁹F NMR (**376** MHz, CDCl₃) δ -62.58.

HPLC conditions: Nu-Analytical Solutions INA, *n*-hexane/*i*-PrOH = 95/05, flow rate 0.8 mL/min. λ = 214 nm, t(minor) = 30.3 min, t(major) = 32.3 min, 98% ee. HRMS (ESI) *m*/*z* calcd. for C₂₃H₁₈BrF₃NO [M+H]⁺ 460.0518, found 460.0515.

(S)-3-(3-acetylphenyl)-3-phenyl-1-(4-(trifluoromethyl)benzyl)azetidin-2-one (22)



According to the **general procedure** C with E1 (46.1 mg, 0.12 mmol) and 1-(3-(5,5-dimethyl-1,3,2-dioxaborinan-2-yl)phenyl)ethan-1-one A6 (23.2 mg, 0.10 mmol), the reaction mixture was purified by column chromatography on silica gel (eluent: petroleum ether/ethyl acetate 5/1) to yield the product as a colorless oil (30.1 mg, 0.071 mmol, 71% yield).

¹**H NMR (400 MHz, CDCl₃)** δ 7.98 (t, *J* = 1.8 Hz, 1H), 7.85 (dt, *J* = 7.8, 1.4 Hz, 1H), 7.68 – 7.63 (m, 1H), 7.59 (d, *J* = 8.1 Hz, 2H), 7.44 (t, *J* = 7.8 Hz, 1H), 7.41 – 7.31 (m, 6H), 7.31 – 7.24 (m, 1H), 4.56 (s, 2H), 3.84 (ABq, *J*_{AB} = 5.5 Hz, Δv_{AB} = 15.1 Hz, 2H), 2.57 (s, 3H).

¹³C NMR (101 MHz, CDCl₃) δ 197.96, 169.26, 140.89, 139.76, 139.36, 137.63, 132.00, 130.36 (q, J = 32.5 Hz), 129.20, 129.04, 128.51, 127.77, 127.65, 127.02, 126.57, 126.07 (q, J = 3.7 Hz), 124.04 (q, J = 272.2 Hz), 66.77, 54.27, 45.81, 26.77. ¹⁹F NMR (376 MHz, CDCl₃) δ -62.59.

HPLC conditions: Nu-Analytical Solutions INB, *n*-hexane/*i*-PrOH = 85/15, flow rate 0.8 mL/min. λ = 230 nm, t(major) = 16.3 min, t(minor) = 17.7 min, 99% ee. **HRMS** (ESI) *m*/*z* calcd. for C₂₅H₂₁F₃NO₂ [M+H]⁺ 424.1519, found 424.1516.

Methyl (S)-4-(2-oxo-3-phenyl-1-(4-(trifluoromethyl)benzyl)azetidin-3-yl)benzoate (23)



According to the **general procedure** C with E1 (46.1 mg, 0.12 mmol) and methyl 4- (5,5-dimethyl-1,3,2-dioxaborinan-2-yl)benzoate A7 (24.8 mg, 0.10 mmol), the reaction mixture was purified by column chromatography on silica gel (eluent: petroleum ether/ethyl acetate 7/1) to yield the product as a colorless oil (35.2 mg, 0.080 mmol, 80% yield).

¹**H NMR (400 MHz, CDCl₃)** δ 8.00 (d, *J* = 8.3 Hz, 2H), 7.59 (d, *J* = 7.9 Hz, 2H), 7.48 (d, *J* = 8.3 Hz, 2H), 7.41 – 7.27 (m, 7H), 4.62 – 4.49 (m, 2H), 3.90 (s, 3H), 3.82 (ABq, *J*_{AB} = 5.6 Hz, Δv_{AB} = 7.1 Hz, 2H).

¹³C NMR (101 MHz, CDCl₃) δ 169.08, 166.77, 145.11, 139.52, 139.33, 130.35 (q, *J* = 32.5 Hz), 130.15, 129.36, 129.02, 128.46, 127.79, 127.18, 127.03, 126.05 (q, *J* = 3.8 Hz), 124.03 (q, *J* = 272.2 Hz), 67.01, 54.10, 52.28, 45.78.

¹⁹F NMR (376 MHz, CDCl₃) δ -62.58.

HPLC conditions: Nu-Analytical Solutions INA, *n*-hexane/*i*-PrOH = 85/15, flow rate 0.8 mL/min. $\lambda = 214$ nm, t(major) = 21.8 min, t(minor) = 30.2 min, 98% ee. **HRMS** (ESI) *m/z* calcd. for C₂₅H₂₁F₃NO₃ [M+H]⁺ 440.1468, found 440.1465.

(S)-4-(2-oxo-3-phenyl-1-(4-(trifluoromethyl)benzyl)azetidin-3-yl)benzonitrile (24)



According to the general procedure C with E1 (46.1 mg, 0.12 mmol) and 4-(5,5-dimethyl-1,3,2-dioxaborinan-2-yl)benzonitrile A8 (24.8 mg, 0.10 mmol), the reaction

mixture was purified by column chromatography on silica gel (eluent: petroleum ether/ethyl acetate 7/1) to yield the product as a white solid (36.2 mg, 0.089 mmol, 89% yield).

¹**H NMR (400 MHz, CDCl₃)** δ 7.61 (t, *J* = 8.5 Hz, 4H), 7.52 (d, *J* = 8.2 Hz, 2H), 7.40 - 7.27 (m, 7H), 4.56 (s, 2H), 3.81 (ABq, *J*_{AB} = 5.6 Hz, Δv_{AB} = 19.3 Hz, 2H).

¹³C NMR (101 MHz, CDCl₃) δ 168.56, 145.34, 139.12, 138.96, 132.69, 130.47 (q, J = 32.5 Hz), 129.18, 128.49, 128.07, 127.91, 126.98, 126.10 (q, J = 3.7 Hz), 123.99 (q, J = 272.0 Hz), 118.58, 111.52, 66.83, 53.88, 45.87.

¹⁹F NMR (376 MHz, CDCl₃) δ -62.59.

HPLC conditions: Chiralcel IH, *n*-hexane/*i*-PrOH = 85/15, flow rate 0.8 mL/min. λ = 230 nm, t(major) = 32.8 min, t(minor) = 38.2 min, 99% ee.

HRMS (ESI) *m/z* calcd. for C₂₄H₁₈F₃NO [M+H]⁺ 407.1366, found 407.1364.

(S)-3-([1,1'-biphenyl]-4-yl)-3-phenyl-1-(4-(trifluoromethyl)benzyl)azetidin-2-one (25)



According to the **general procedure C** with **E1** (46.1 mg, 0.12 mmol) and 2-([1,1'biphenyl]-4-yl)-5,5-dimethyl-1,3,2-dioxaborinane **A17** (26.6 mg, 0.10 mmol), the reaction mixture was purified by column chromatography on silica gel (eluent: petroleum ether/ethyl acetate 10/1) to yield the product as a white solid (27.9 mg, 0.061 mmol, 61% yield).

¹**H NMR (400 MHz, CDCl₃)** δ 7.61 (d, *J* = 7.9 Hz, 2H), 7.57 (d, *J* = 7.8 Hz, 4H), 7.50 – 7.41 (m, 6H), 7.40 – 7.32 (m, 5H), 7.32 – 7.27 (m, 1H), 4.58 (s, 2H), 3.89 – 3.78 (m, 2H).

¹³C NMR (101 MHz, CDCl₃) δ 169.74, 140.60, 140.43, 140.16, 139.54, 139.22, 130.30 (q, *J* = 32.7 Hz), 128.94, 128.92, 128.51, 127.60, 127.58, 127.54, 127.17, 126.04 (q, *J* = 3.8 Hz), 124.09 (q, *J* = 272.2 Hz), 66.85, 54.44, 45.78.

¹⁹F NMR (376 MHz, CDCl₃) δ -62.54.

HPLC conditions: Nu-Analytical Solutions INA, *n*-hexane/*i*-PrOH = 85/15, flow rate 0.8 mL/min. $\lambda = 254$ nm, t(major) = 15.9 min, t(minor) = 17.8 min, 97% ee. **HRMS** (ESI) *m/z* calcd. for C₂₉H₂₃F₃NO [M+H]⁺ 458.1726, found 458.1723.

(S)-3-(benzo[d][1,3]dioxol-5-yl)-3-phenyl-1-(4-(trifluoromethyl)benzyl)azetidin-2one (26)



According to the general procedure C with E1 (46.1 mg, 0.12 mmol) and 2-(benzo[d][1,3]dioxol-5-yl)-5,5-dimethyl-1,3,2-dioxaborinane A9 (23.4 mg, 0.10 mmol), the reaction mixture was purified by column chromatography on silica gel (eluent: petroleum ether/ethyl acetate 6/1) to yield the product as a white solid (25.9 mg, 0.061 mmol, 61% yield).

¹**H** NMR (400 MHz, CDCl₃) δ 7.59 (d, J = 8.0 Hz, 2H), 7.42 – 7.29 (m, 6H), 7.29 – 7.21 (m, 1H), 6.91 – 6.81 (m, 2H), 6.76 (d, J = 8.6 Hz, 1H), 5.93 (s, 2H), 4.54 (ABq, $J_{AB} = 15.4$ Hz, $\Delta v_{AB} = 13.2$ Hz, 2H), 3.75 (ABq, $J_{AB} = 5.4$ Hz, $\Delta v_{AB} = 14.3$ Hz, 2H). ¹³C NMR (101 MHz, CDCl₃) δ 169.77, 148.08, 146.98, 140.31, 139.54, 134.11, 130.30 (q, J = 32.6 Hz), 128.88, 128.48, 127.55, 127.05, 126.03 (q, J = 3.7 Hz), 124.09 (q, J = 272.2 Hz), 120.32, 108.42, 107.93, 101.29, 66.72, 54.62, 45.72.

¹⁹F NMR (376 MHz, CDCl₃) δ -62.56.

HPLC conditions: Nu-Analytical Solutions INA, *n*-hexane/*i*-PrOH = 90/10, flow rate 0.8 mL/min. $\lambda = 230$ nm, t(minor) = 20.5 min, t(major) = 23.3 min, 97% ee. **HRMS** (ESI) *m/z* calcd. for C₂₄H₁₉F₃NO₃ [M+H]⁺ 426.1312, found 426.1308.

(S)-3-(dibenzo[b,d]furan-2-yl)-3-phenyl-1-(4-(trifluoromethyl)benzyl)azetidin-2one (27)



According to the **general procedure C** with **E1** (46.1 mg, 0.12 mmol) and 2-(dibenzo[b,d]furan-2-yl)-5,5-dimethyl-1,3,2-dioxaborinane **A10** (28.0 mg, 0.10 mmol), the reaction mixture was purified by column chromatography on silica gel (eluent: petroleum ether/ethyl acetate 8/1) to yield the product as a white solid (37.2 mg, 0.079 mmol, 79% yield).

¹**H NMR (400 MHz, CDCl₃)** δ 8.05 (d, *J* = 1.6 Hz, 1H), 7.92 (d, *J* = 7.7 Hz, 1H), 7.60 (d, *J* = 8.0 Hz, 2H), 7.58 – 7.51 (m, 2H), 7.48 (d, *J* = 1.9 Hz, 1H), 7.47 – 7.42 (m, 3H),

7.41 – 7.33 (m, 5H), 7.33 – 7.26 (m, 1H), 4.60 (ABq, $J_{AB} = 15.5$ Hz, $\Delta v_{AB} = 8.8$ Hz, 2H), 3.95 - 3.86 (m, 2H).

¹³**C NMR (101 MHz, CDCl₃)** δ 169.95, 156.72, 155.42, 140.49, 139.53, 134.91, 130.31 (q, *J* = 32.5 Hz), 128.95, 128.51, 127.59, 127.56, 127.08, 126.42, 126.04 (q, *J* = 3.8 Hz), 124.68, 124.07 (q, *J* = 272.0 Hz), 124.04, 122.95, 120.96, 119.30, 112.00, 111.83, 66.95, 54.86, 45.77.

¹⁹F NMR (**376** MHz, CDCl₃) δ -62.53.

HPLC conditions: Nu-Analytical Solutions INA, *n*-hexane/*i*-PrOH = 85/15, flow rate 1.0 mL/min. $\lambda = 254$ nm, t(minor) = 13.7 min, t(major) = 15.9 min, 98% ee. **HRMS** (ESI) *m/z* calcd. for C₂₉H₂₁F₃NO₂ [M+H]⁺ 472.1519, found 472.1518.

(S)-3-phenyl-3-(quinolin-6-yl)-1-(4-(trifluoromethyl)benzyl)azetidin-2-one (28)



According to the **general procedure** C with E1 (46.1 mg, 0.12 mmol) and 6-(5,5-dimethyl-1,3,2-dioxaborinan-2-yl)quinoline A11 (24.1 mg, 0.10 mmol), the reaction mixture was purified by column chromatography on silica gel (eluent: petroleum ether/ethyl acetate 3/1) to yield the product as a colorless oil (29.8 mg, 0.069 mmol, 69% yield).

¹**H** NMR (400 MHz, CDCl₃) δ 8.90 (dd, J = 4.3, 1.7 Hz, 1H), 8.16 – 8.11 (m, 1H), 8.08 (d, J = 8.8 Hz, 1H), 7.93 (d, J = 2.0 Hz, 1H), 7.67 (dd, J = 8.8, 2.1 Hz, 1H), 7.59 (d, J = 8.0 Hz, 2H), 7.47 – 7.32 (m, 7H), 7.32 – 7.26 (m, 1H), 4.59 (s, 2H), 3.90 (ABq, $J_{AB} = 5.5$ Hz, $\Delta v_{AB} = 15.5$ Hz, 2H).

¹³C NMR (101 MHz, CDCl₃) δ 169.40, 150.79, 147.54, 139.62, 139.37, 138.44, 136.38, 130.35 (q, J = 32.7 Hz), 130.24, 129.02, 128.86, 128.49, 128.21, 127.78, 127.13, 126.05 (q, J = 3.6 Hz), 125.65, 124.03 (q, J = 272.3 Hz), 121.69, 66.94, 54.18, 45.81. ¹⁹F NMR (376 MHz, CDCl₃) δ -62.57.

HPLC conditions: Nu-Analytical Solutions INA, *n*-hexane/*i*-PrOH = 85/15, flow rate 0.8 mL/min. $\lambda = 254$ nm, t(major) = 31.3 min, t(minor) = 43.6 min, 98% ee. **HRMS** (ESI) *m/z* calcd. for C₂₆H₂₀F₃N₂O [M+H]⁺ 433.1522, found 433.1520.

(S)-3-phenyl-3-(pyridin-3-yl)-1-(4-(trifluoromethyl)benzyl)azetidin-2-one (29)



According to the **general procedure** C with E1 (46.1 mg, 0.12 mmol) and 3-(5,5-dimethyl-1,3,2-dioxaborinan-2-yl)pyridine A12 (19.1 mg, 0.10 mmol), the reaction mixture was purified by column chromatography on silica gel (eluent: petroleum ether/ethyl acetate 3/1) to yield the product as a colorless oil (12.2 mg, 0.032 mmol, 32% yield).

¹**H NMR (400 MHz, CDCl₃)** δ 8.63 (s, 1H), 8.53 (s, 1H), 7.75 (dt, *J* = 8.0, 2.0 Hz, 1H), 7.60 (d, *J* = 8.1 Hz, 2H), 7.42 – 7.32 (m, 6H), 7.32 – 7.23 (m, 2H), 4.56 (s, 2H), 3.83 (s, 2H).

¹³C NMR (101 MHz, CDCl₃) δ 168.76, 148.87, 148.30, 139.22, 139.18, 136.01, 134.96, 130.50 (q, *J* = 32.7 Hz), 129.16, 128.52, 127.97, 127.07, 126.14 (q, *J* = 3.8 Hz), 124.03 (q, *J* = 272.1 Hz), 123.74, 65.11, 53.89, 45.90.

¹⁹F NMR (376 MHz, CDCl₃) δ -62.64.

HPLC conditions: Nu-Analytical Solutions INA, *n*-hexane/*i*-PrOH = 85/15, flow rate 1.0 mL/min. λ = 214 nm, t(major) = 14.4 min, t(minor) = 25.3 min, 94% ee. **HRMS** (ESI) *m*/*z* calcd. for C₂₂H₁₈F₃N₂O [M+H]⁺ 383.1366, found 383.1363.

(S)-3-(6-chloropyridin-3-yl)-3-phenyl-1-(4-(trifluoromethyl)benzyl)azetidin-2-one (30)



According to the **general procedure C** with **E1** (46.1 mg, 0.12 mmol) and 2-chloro-5- (5,5-dimethyl-1,3,2-dioxaborinan-2-yl)pyridine **A13** (22.5 mg, 0.10 mmol), the reaction mixture was purified by column chromatography on silica gel (eluent: petroleum ether/ethyl acetate 3/1) to yield the product as a colorless oil (17.9 mg, 0.043 mmol, 43% yield).

¹H NMR (400 MHz, CDCl₃) δ 8.39 (d, J = 2.6 Hz, 1H), 7.71 (dd, J = 8.3, 2.5 Hz, 1H), 7.60 (d, J = 8.0 Hz, 2H), 7.40 – 7.27 (m, 8H), 4.56 (ABq, $J_{AB} = 15.4$ Hz, $\Delta v_{AB} = 14.0$ Hz, 2H), 3.81 (ABq, $J_{AB} = 5.7$ Hz, $\Delta v_{AB} = 11.5$ Hz, 2H). ¹³C NMR (101 MHz, CDCl₃) δ 168.29, 150.77, 148.09, 139.07, 138.79, 137.84, 135.00, 130.56 (q, J = 32.7 Hz), 129.28, 128.52, 128.16, 126.94, 126.17 (q, J = 3.8 Hz), 124.49, 124.01 (d, J = 272.1 Hz), 64.47, 53.75, 45.93.
¹⁹F NMR (**376** MHz, CDCl₃) δ -62.63.

HPLC conditions: Nu-Analytical Solutions INA, *n*-hexane/*i*-PrOH = 85/15, flow rate 0.8 mL/min. λ = 225 nm, t(major) = 18.8 min, t(minor) = 38.2 min, 98% ee. **HRMS** (ESI) *m/z* calcd. for C₂₂H₁₇ClF₃N₂O [M+H]⁺ 417.0976, found 417.0973.

(S)-3-phenyl-3-(pyridin-4-yl)-1-(4-(trifluoromethyl)benzyl)azetidin-2-one (31)



According to the **general procedure** C with E1 (46.1 mg, 0.12 mmol) and 4-(5,5-dimethyl-1,3,2-dioxaborinan-2-yl)pyridine A14 (19.1 mg, 0.10 mmol), the reaction mixture was purified by column chromatography on silica gel (eluent: petroleum ether/ethyl acetate 2/1) to yield the product as a colorless oil (16.2 mg, 0.042 mmol, 42% yield).

¹H NMR (400 MHz, CDCl₃) δ 8.59 – 8.53 (m, 2H), 7.60 (d, *J* = 8.0 Hz, 2H), 7.41 – 7.31 (m, 7H), 7.31 – 7.28 (m, 2H), 4.56 (ABq, *J*_{AB} = 15.4 Hz, Δv_{AB} = 9.0 Hz, 2H), 3.80 (ABq, *J*_{AB} = 5.6 Hz, Δv_{AB} = 9.3 Hz, 2H).

¹³C NMR (101 MHz, CDCl₃) δ 168.34, 150.34, 148.85, 139.16, 138.55, 130.53 (q, *J* = 32.8 Hz), 129.18, 128.51, 128.13, 127.10, 126.14 (q, *J* = 3.8 Hz), 122.12, 124.02 (q, *J* = 272.1 Hz), 66.36, 53.61, 45.91.

¹⁹F NMR (376 MHz, CDCl₃) δ -62.64.

HPLC conditions: Nu-Analytical Solutions INA, *n*-hexane/*i*-PrOH = 85/15, flow rate 1.0 mL/min. $\lambda = 214$ nm, t(major) = 16.6 min, t(minor) = 22.3 min, 98% ee. **HRMS** (ESI) *m/z* calcd. for C₂₂H₁₈F₃N₂O [M+H]⁺ 383.1366, found 383.1362.

(S)-3-phenyl-3-(thiophen-3-yl)-1-(4-(trifluoromethyl)benzyl)azetidin-2-one (32)



According to the **general procedure** C with E1 (46.1 mg, 0.12 mmol) and 5,5dimethyl-2-(thiophen-3-yl)-1,3,2-dioxaborinane A15 (19.6 mg, 0.10 mmol), the reaction mixture was purified by column chromatography on silica gel (eluent: petroleum ether/ethyl acetate 8/1) to yield the product as a colorless oil (12.0 mg, 0.031 mmol, 31% yield).

¹**H NMR (400 MHz, CDCl₃)** δ 7.60 (d, *J* = 8.0 Hz, 2H), 7.42 – 7.28 (m, 9H), 7.11 (dd, *J* = 3.0, 1.4 Hz, 1H), 6.99 (dd, *J* = 5.0, 1.4 Hz, 1H), 4.55 (ABq, *J*_{AB} = 15.4 Hz, Δv_{AB} = 22.2 Hz, 2H), 3.74 (ABq, *J*_{AB} = 5.3 Hz, Δv_{AB} = 13.2 Hz, 2H).

¹³C NMR (101 MHz, CDCl₃) δ 169.48, 140.78, 139.58, 130.38 (q, *J* = 32.9 Hz), 128.89, 128.53, 127.69, 127.07, 126.75, 126.52, 126.07 (q, *J* = 3.8 Hz), 124.10 (q, *J* = 272.3 Hz), 122.23, 64.28, 54.74, 45.81.

¹⁹F NMR (376 MHz, CDCl₃) δ -62.60.

HPLC conditions: Nu-Analytical Solutions INB, *n*-hexane/*i*-PrOH = 90/10, flow rate 0.8 mL/min. $\lambda = 230$ nm, t(major) = 17.9 min, t(minor) = 20.0 min, 92% ee. **HRMS** (ESI) *m/z* calcd. for C₂₁H₁₇F₃NOS [M+H]⁺ 388.0977, found 388.0973.

(S)-3-phenyl-3-(pyrimidin-5-yl)-1-(4-(trifluoromethyl)benzyl)azetidin-2-one (33)



According to the **general procedure** C with E1 (46.1 mg, 0.12 mmol) and 5-(5,5-dimethyl-1,3,2-dioxaborinan-2-yl)pyrimidine A16 (19.2 mg, 0.10 mmol), the reaction mixture was purified by column chromatography on silica gel (eluent: petroleum ether/ethyl acetate 3/1) to yield the product as a colorless oil (16.9 mg, 0.044 mmol, 44% yield).

¹**H NMR (400 MHz, CDCl₃)** δ 9.14 (s, 1H), 8.75 (s, 2H), 7.61 (d, *J* = 8.0 Hz, 2H), 7.43 – 7.29 (m, 7H), 4.57 (ABq, *J*_{AB} = 15.5 Hz, Δv_{AB} = 9.3 Hz, 2H), 3.83 (ABq, *J*_{AB} = 5.8 Hz, Δv_{AB} = 22.3 Hz, 2H).

¹³C NMR (101 MHz, CDCl₃) δ 167.64, 157.89, 155.53, 138.90, 137.95, 134.01, 130.65 (q, *J* = 32.5 Hz), 129.46, 128.57, 128.44, 127.04, 126.24 (q, *J* = 3.7 Hz), 123.98 (q, *J* = 272.5 Hz), 63.30, 53.33, 46.04.

¹⁹F NMR (376 MHz, CDCl₃) δ -62.66.

HPLC conditions: Nu-Analytical Solutions INA, *n*-hexane/*i*-PrOH = 85/15, flow rate 1.0 mL/min. λ = 254 nm, t(major) = 16.8 min, t(minor) = 36.4 min, 97% ee. HRMS (ESI) *m*/*z* calcd. for C₂₁H₁₇F₃N₃O [M+H]⁺ 384.1318, found 384.1314.

(*R*,*E*)-3-phenyl-3-styryl-1-(4-(trifluoromethyl)benzyl)azetidin-2-one (34)



According to the **general procedure D** with E1 (57.4 mg, 0.15 mmol) and (*E*)-5,5dimethyl-2-styryl-1,3,2-dioxaborinane A18 (21.6 mg, 0.10 mmol) at -40 °C for 5 d, the reaction mixture was purified by column chromatography on silica gel (eluent: petroleum ether/ethyl acetate 10/1) to yield the product as a colorless oil (28.8 mg, 0.071 mmol, 71% yield). ¹**H NMR (400 MHz, CDCl₃)** δ 7.60 (d, *J* = 8.1 Hz, 2H), 7.49 – 7.43 (m, 2H), 7.41 – 7.26 (m, 9H), 7.25 – 7.21 (m, 1H), 6.51 (s, 2H), 4.54 (ABq, *J*_{AB} = 15.4 Hz, Δv_{AB} = 28.9 Hz, 2H), 3.61 (s, 2H).

¹³C NMR (101 MHz, CDCl₃) δ 169.28, 136.33, 131.77, 130.16 (q, *J* = 32.5 Hz), 128.82, 128.61, 128.59, 128.35, 127.93, 127.60, 127.06, 126.50, 125.92 (q, *J* = 3.8 Hz), 123.98 (q, *J* = 272.1 Hz), 65.47, 53.52, 45.57.

¹⁹F NMR (**376** MHz, CDCl₃) δ -62.55.

HPLC conditions: Nu-Analytical Solutions INB, *n*-hexane/*i*-PrOH = 95/5, flow rate 0.7 mL/min. $\lambda = 254$ nm, t(major) = 24.3 min, t(minor) = 30.6 min, 98% ee. **HRMS** (ESI) *m*/*z* calcd. for C₂₅H₂₁F₃NO [M+H]⁺ 408.1570, found 408.1567.

(*R*,*E*)-3-(3-methoxystyryl)-3-phenyl-1-(4-(trifluoromethyl)benzyl)azetidin-2-one (35)



According to the **general procedure D** with **E1** (57.4 mg, 0.15 mmol) and (*E*)-2-(3-methoxystyryl)-5,5-dimethyl-1,3,2-dioxaborinane **A2** (24.6 mg, 0.10 mmol) at -40 °C for 5 d, the reaction mixture was purified by column chromatography on silica gel (eluent: petroleum ether/ethyl acetate 10/1) to yield the product as a colorless oil (30.5 mg, 0.070 mmol, 70% yield).

¹**H NMR (400 MHz, CDCl₃)** δ 7.59 (d, *J* = 8.0 Hz, 2H), 7.46 (d, *J* = 7.5 Hz, 2H), 7.42 – 7.34 (m, 4H), 7.34 – 7.26 (m, 1H), 7.25 – 7.13 (m, 1H), 6.93 (d, *J* = 7.7 Hz, 1H), 6.87 (t, *J* = 2.0 Hz, 1H), 6.78 (dd, *J* = 8.2, 2.5 Hz, 1H), 6.50 (s, 2H), 4.53 (ABq, *J*_{AB} = 15.4 Hz, $\Delta v_{AB} = 23.8$ Hz, 2H), 3.77 (s, 3H), 3.60 (s, 2H).

¹³C NMR (101 MHz, CDCl₃) δ 169.22, 159.80, 139.45, 138.46, 137.76, 131.64, 130.13 (q, J = 32.6 Hz), 129.55, 128.91, 128.81, 128.32, 127.92, 127.63, 127.59, 125.89 (q, J = 3.8 Hz), 123.97 (q, J = 272.1 Hz), 119.11, 113.74, 111.66, 65.42, 55.19, 53.47, 45.54. ¹⁹F NMR (376 MHz, CDCl₃) δ -62.55.

HPLC conditions: Nu-Analytical Solutions INB, *n*-hexane/*i*-PrOH = 95/5, flow rate 1.0 mL/min. $\lambda = 254$ nm, t(minor) = 24.5 min, t(major) = 46.4 min, 94% ee. **HRMS** (ESI) *m/z* calcd. for C₂₆H₂₃F₃NO [M+H]⁺ 438.1675, found 438.1673.

(*R*,*E*)-3-(4-methylstyryl)-3-phenyl-1-(4-(trifluoromethyl)benzyl)azetidin-2-one (36)



According to the **general procedure D** with **E1** (57.4 mg, 0.15 mmol) and (*E*)-5,5-dimethyl-2-(4-methylstyryl)-1,3,2-dioxaborinane **A19** (23.0 mg, 0.10 mmol) at -40 °C for 5 d, the reaction mixture was purified by column chromatography on silica gel (eluent: petroleum ether/ethyl acetate 10/1) to yield the product as a colorless oil (30.2 mg, 0.072 mmol, 72% yield).

¹**H NMR (400 MHz, CDCl₃)** δ 7.60 (d, *J* = 8.0 Hz, 2H), 7.45 (d, *J* = 7.3 Hz, 2H), 7.41 – 7.35 (m, 4H), 7.31 (d, *J* = 7.3 Hz, 1H), 7.27 – 7.19 (m, 2H), 7.10 (d, *J* = 7.9 Hz, 2H), 6.47 (ABq, *J*_{AB} = 16.0 Hz, Δv_{AB} = 9.8 Hz, 2H), 4.54 (ABq, *J*_{AB} = 15.4 Hz, Δv_{AB} = 30.5 Hz, 2H), 3.60 (s, 2H), 2.32 (s, 3H).

¹³C NMR (101 MHz, CDCl₃) δ 169.44, 139.52, 138.64, 137.83, 133.57, 131.64, 130.18 (q, *J* = 32.4 Hz), 129.30, 128.81, 128.36, 127.56, 127.08, 126.42, 125.93 (q, *J* = 3.8 Hz), 124.00 (q, *J* = 272.1 Hz), 65.50, 53.59, 45.58, 21.21.

¹⁹F NMR (376 MHz, CDCl₃) δ -62.55.

HPLC conditions: Nu-Analytical Solutions INB, *n*-hexane/*i*-PrOH = 95/5, flow rate 0.5 mL/min. $\lambda = 254$ nm, t(major) = 27.3 min, t(minor) = 28.5 min, 98% ee. **HRMS** (ESI) *m/z* calcd. for C₂₆H₂₃F₃NO [M+H]⁺ 422.1726, found 422.1723.

(R,E)-3-(4-chlorostyryl)-3-phenyl-1-(4-(trifluoromethyl)benzyl)azetidin-2-one (37)



According to the **general procedure D** with **E1** (57.4 mg, 0.15 mmol) and (*E*)-2-(4-chlorostyryl)-5,5-dimethyl-1,3,2-dioxaborinane **A20** (25.0 mg, 0.10 mmol) at -40 °C for 5 d, the reaction mixture was purified by column chromatography on silica gel (eluent: petroleum ether/dichloromethane 2/1) to yield the product as a colorless oil (30.0 mg, 0.068 mmol, 68% yield).

¹**H NMR (400 MHz, CDCl₃)** δ 7.60 (d, *J* = 8.1 Hz, 2H), 7.45 (dd, *J* = 8.2, 1.5 Hz, 2H), 7.41 - 7.35 (m, 4H), 7.34 - 7.29 (m, 1H), 7.25 (s, 4H), 6.46 (ABq, *J*_{AB} = 15.7 Hz,

 $\Delta v_{AB} = 14.0$ Hz, 2H), 4.54 (ABq, $J_{AB} = 15.4$ Hz, $\Delta v_{AB} = 18.2$ Hz, 2H), 3.60 (ABq, $J_{AB} = 5.4$ Hz, $\Delta v_{AB} = 8.0$ Hz, 2H).

¹³C NMR (101 MHz, CDCl₃) δ 169.07, 139.40, 138.27, 134.84, 133.56, 130.66, 130.22 (q, *J* = 32.6 Hz), 129.36, 128.88, 128.75, 128.37, 127.73, 127.70, 127.07, 125.94 (q, *J* = 3.7 Hz), 123.97 (q, *J* = 272.1 Hz), 65.42, 53.49, 45.62.

¹⁹F NMR (376 MHz, CDCl₃) δ -62.56.

HPLC conditions: Nu-Analytical Solutions INB, *n*-hexane/*i*-PrOH = 95/5, flow rate 0.7 mL/min. $\lambda = 254$ nm, t(minor) = 36.6 min, t(major) = 37.9 min, 90% ee. **HRMS** (ESI) *m/z* calcd. for C₂₅H₂₀ClF₃NO [M+H]⁺ 442.1180, found 442.1179.

(R,E)-3-(4-bromostyryl)-3-phenyl-1-(4-(trifluoromethyl)benzyl)azetidin-2-one (38)



According to the **general procedure D** with **E1** (57.4 mg, 0.15 mmol) and (*E*)-2-(4-bromostyryl)-5,5-dimethyl-1,3,2-dioxaborinane **A21** (29.5 mg, 0.10 mmol) at -40 °C for 5 d, the reaction mixture was purified by column chromatography on silica gel (eluent: petroleum ether/dichloromethane 2/1) to yield the product as a colorless oil (35.0 mg, 0.072 mmol, 72% yield).

¹**H** NMR (400 MHz, CDCl₃) δ 7.60 (d, *J* = 8.0 Hz, 2H), 7.48 – 7.42 (m, 2H), 7.42 – 7.34 (m, 6H), 7.34 – 7.27 (m, 1H), 7.19 (d, *J* = 8.5 Hz, 2H), 6.46 (ABq, *J*_{AB} = 16.0 Hz, $\Delta v_{AB} = 26.5$ Hz, 2H), 4.53 (ABq, *J*_{AB} = 15.4 Hz, $\Delta v_{AB} = 17.4$ Hz, 2H), 3.60 (ABq, *J*_{AB} = 5.4 Hz, $\Delta v_{AB} = 7.5$ Hz, 2H).

¹³C NMR (101 MHz, CDCl₃) δ 169.01, 139.39, 139.37, 138.21, 135.27, 131.67, 130.70, 130.18 (q, *J* = 32.6 Hz), 129.48, 128.86, 128.35, 128.03, 127.69, 127.06, 125.92 (q, *J* = 3.7 Hz), 123.95 (q, *J* = 272.1 Hz), 121.70, 65.41, 53.44, 45.60.

¹⁹F NMR (**376** MHz, CDCl₃) δ -62.51.

HPLC conditions: Nu-Analytical Solutions INB, *n*-hexane/*i*-PrOH = 95/5, flow rate 0.7 mL/min. $\lambda = 254$ nm, t(minor) = 26.0 min, t(major) = 27.9 min, 94% ee. **HRMS** (ESI) *m*/*z* calcd. for C₂₅H₂₀BrF₃NO [M+H]⁺ 486.0675, found 486.0673.

(*R*,*E*)-3-(2-([1,1'-biphenyl]-4-yl)vinyl)-3-phenyl-1-(4-(trifluoromethyl)benzyl)azetidin-2-one (39)



According to the **general procedure D** with **E1** (57.4 mg, 0.15 mmol) and (*E*)-2-(2-([1,1'-biphenyl]-4-yl)vinyl)-5,5-dimethyl-1,3,2-dioxaborinane **A22** (29.2 mg, 0.10 mmol) at -40 °C for 5 d, the reaction mixture was purified by column chromatography on silica gel (eluent: petroleum ether/dichloromethane 2/1) to yield the product as a colorless oil (30.8 mg, 0.064 mmol, 64% yield).

¹H NMR (400 MHz, CDCl₃) δ 7.57 – 7.42 (m, 6H), 7.42 – 7.36 (m, 2H), 7.38 – 7.27 (m, 8H), 7.27 – 7.20 (m, 2H), 6.47 (s, 2H), 4.45 (ABq, J_{AB} = 15.4 Hz, Δv_{AB} = 29.5 Hz, 2H), 3.53 (s, 2H).

¹³C NMR (101 MHz, CDCl₃) δ 169.26, 140.66, 140.54, 139.48, 138.50, 135.37, 131.32, 130.18 (q, J = 32.6 Hz), 128.85, 128.81, 128.71, 128.36, 127.63, 127.41, 127.26, 127.09, 126.96, 126.93, 125.94 (q, J = 3.7 Hz), 123.99 (q, J = 272.0 Hz), 65.54, 53.54, 45.59. ¹⁹F NMR (376 MHz, CDCl₃) δ -62.49.

HPLC conditions: Nu-Analytical Solutions INB, *n*-hexane/*i*-PrOH = 95/5, flow rate 0.7 mL/min. $\lambda = 254$ nm, t(major) = 40.9 min, t(minor) = 61.0 min, 94% ee. **HRMS** (ESI) *m/z* calcd. for C₃₁H₂₅F₃NO [M+H]⁺ 484.1883, found 484.1881.

(*R*,*E*)-3-(2-(naphthalen-2-yl)vinyl)-3-phenyl-1-(4-(trifluoromethyl)benzyl)azetidin-2-one (40)



 F_3C

According to the **general procedure D** with **E1** (57.4 mg, 0.15 mmol) and (*E*)-5,5dimethyl-2-(2-(naphthalen-2-yl)vinyl)-1,3,2-dioxaborinane **A23** (26.6 mg, 0.10 mmol) at -40 °C for 5 d, the reaction mixture was purified by column chromatography on silica gel (eluent: petroleum ether/dichloromethane 2/1) to yield the product as a colorless oil (27.8 mg, 0.061 mmol, 61% yield).

¹**H NMR (400 MHz, CDCl₃)** δ 7.77 (dd, *J* = 9.0, 6.7 Hz, 3H), 7.68 (d, *J* = 1.7 Hz, 1H), 7.61 (d, *J* = 8.0 Hz, 2H), 7.55 (dd, *J* = 8.6, 1.8 Hz, 1H), 7.53 – 7.47 (m, 2H), 7.47 – 7.36

(m, 6H), 7.35 - 7.29 (m, 1H), 6.65 (ABq, $J_{AB} = 16.0$ Hz, $\Delta v_{AB} = 10.7$ Hz, 2H), 4.55 $(ABq, J_{AB} = 15.5 \text{ Hz}, \Delta v_{AB} = 22.1 \text{ Hz}, 2\text{H}), 3.64 (ABq, J_{AB} = 5.4 \text{ Hz}, \Delta v_{AB} = 3.9 \text{ Hz},$ 2H).

¹³C NMR (101 MHz, CDCl₃) δ 169.32, 139.48, 138.52, 133.78, 133.52, 133.10, 131.96, 130.21 (q, J = 32.4 Hz), 129.00, 128.88, 128.39, 128.26, 128.03, 127.68, 127.15, 126.80, 126.37, 126.07, 125.96 (q, J = 3.7 Hz), 123.99 (q, J = 272.1 Hz), 123.39, 65.61, 53.59, 45.63.

¹⁹F NMR (376 MHz, CDCl₃) δ -62.54.

HPLC conditions: Nu-Analytical Solutions INB, *n*-hexane/*i*-PrOH = 95/5, flow rate 0.5 mL/min. $\lambda = 280 \text{ nm}$, t(minor) = 44.3 min, t(major) = 49.6 min, 96% ee. HRMS (ESI) *m/z* calcd. for C₂₉H₂₃F₃NO [M+H]⁺ 458.1726, found 458.1723.

(R,E)-3-phenyl-3-(2-(thiophen-2-yl)vinyl)-1-(4-(trifluoromethyl)benzyl)azetidin-2-one (41)



According to the general procedure D with E1 (57.4 mg, 0.15 mmol) and (E)-5,5dimethyl-2-(2-(thiophen-2-yl)vinyl)-1,3,2-dioxaborinane A24 (22.2 mg, 0.10 mmol) at -40 °C for 5 d, the reaction mixture was purified by column chromatography on silica gel (eluent: petroleum ether/ethyl acetate 10/1) to yield the product as a colorless oil (23.0 mg, 0.056 mmol, 56% yield).

¹H NMR (400 MHz, CDCl₃) δ 7.60 (d, J = 8.0 Hz, 2H), 7.50 – 7.42 (m, 2H), 7.42 – 7.28 (m, 6H), 7.14 (dd, J = 4.9, 1.3 Hz, 1H), 6.97 – 6.89 (m, 2H), 6.61 (d, J = 15.7 Hz, 1H), 6.34 (d, J = 15.8 Hz, 1H), 4.53 (ABq, $J_{AB} = 15.4$ Hz, $\Delta v_{AB} = 11.6$ Hz, 2H), 3.58 $(ABq, J_{AB} = 5.4 \text{ Hz}, \Delta v_{AB} = 12.0 \text{ Hz}, 2\text{H}).$

¹³C NMR (101 MHz, CDCl₃) δ 169.05, 141.40, 139.43, 138.26, 130.22 (q, J = 32.5 Hz), 129.16 (d, J = 29.3 Hz), 128.86, 128.37, 128.31, 128.11, 127.68, 127.45, 127.12, 126.45, 125.95 (q, J = 3.7 Hz), 125.32, 124.62, 123.99 (q, J = 272.1 Hz), 65.35, 53.63, 53.46, 45.63.

¹⁹F NMR (376 MHz, CDCl₃) δ -62.56.

HPLC conditions: Nu-Analytical Solutions INB, n-hexane/i-PrOH = 95/5, flow rate 0.7 mL/min. $\lambda = 254 \text{ nm}$, t(major) = 23.7 min, t(minor) = 40.3 min, 95% ee. **HRMS** (ESI) m/z calcd. for C₂₃H₁₉F₃NOS [M+H]⁺ 414.1134, found 414.1130.

(R,E)-3-phenyl-3-(2-(thiophen-3-yl)vinyl)-1-(4-(trifluoromethyl)benzyl)azetidin-2-one (42)



According to the **general procedure D** with **E1** (57.4 mg, 0.15 mmol) and (*E*)-5,5dimethyl-2-(2-(thiophen-3-yl)vinyl)-1,3,2-dioxaborinane **A25** (22.2 mg, 0.10 mmol) at $-40 \,^{\circ}$ C for 5 d, the reaction mixture was purified by column chromatography on silica gel (eluent: petroleum ether/ethyl acetate 10/1) to yield the product as a colorless oil (19.2 mg, 0.047 mmol, 47% yield).

¹**H NMR (400 MHz, CDCl₃)** δ 7.60 (d, *J* = 8.0 Hz, 2H), 7.45 (d, *J* = 7.2 Hz, 2H), 7.38 (t, *J* = 7.9 Hz, 4H), 7.34 – 7.28 (m, 1H), 7.28 – 7.22 (m, 1H), 7.18 (d, *J* = 5.1 Hz, 1H), 7.12 (d, *J* = 2.9 Hz, 1H), 6.51 (d, *J* = 15.9 Hz, 1H), 4.54 (ABq, *J*_{AB} = 15.4 Hz, $\Delta v_{AB} = 21.3$ Hz, 2H), 3.59 (ABq, *J*_{AB} = 5.3 Hz, $\Delta v_{AB} = 7.8$ Hz, 2H).

¹³C NMR (101 MHz, CDCl₃) δ 169.30, 139.47, 138.97, 138.50, 130.21 (q, *J* = 32.4 Hz), 128.84, 128.49, 128.37, 127.61, 127.10, 126.10, 125.95 (q, *J* = 3.8 Hz), 124.80, 123.99 (q, *J* = 272.1 Hz), 122.86, 65.43, 53.56, 45.61.

¹⁹F NMR (376 MHz, CDCl₃) δ -62.55.

HPLC conditions: Nu-Analytical Solutions INB, *n*-hexane/*i*-PrOH = 95/5, flow rate 0.7 mL/min. $\lambda = 254$ nm, t(major) = 27.8 min, t(minor) = 45.5 min, 95% ee. **HRMS** (ESI) *m*/*z* calcd. for C₂₃H₁₉F₃NOS [M+H]⁺ 414.1134, found 414.1130.

(*R*,*E*)-3-(2-(cyclohex-1-en-1-yl)vinyl)-3-phenyl-1-(4-(trifluoromethyl)benzyl)azetidin-2-one (43)



According to the **general procedure D** with **E1** (57.4 mg, 0.15 mmol) and (*E*)-2-(2-(cyclohex-1-en-1-yl)vinyl)-5,5-dimethyl-1,3,2-dioxaborinane **A26** (22.0 mg, 0.10 mmol) at -40 °C for 5 d, the reaction mixture was purified by column chromatography on silica gel (eluent: petroleum ether/ethyl acetate 10/1) to yield the product as a colorless oil (18.8 mg, 0.046 mmol, 46% yield).

¹**H** NMR (400 MHz, CDCl₃) δ 7.60 (d, *J* = 7.9 Hz, 2H), 7.53 – 7.29 (m, 5H), 7.31 – 7.02 (m, 1H), 6.11 (d, *J* = 15.8 Hz, 1H), 5.82 (d, *J* = 15.9 Hz, 1H), 5.71 (d, *J* = 3.7 Hz, 1H), 4.51 (ABq, *J*_{AB} = 15.4 Hz, $\Delta v_{AB} = 8.5$ Hz, 2H), 3.53 (ABq, *J*_{AB} = 5.3 Hz, $\Delta v_{AB} = 8.5$ Hz,

5.3 Hz, 2H), 2.19 – 2.00 (m, 4H), 1.93 – 1.34 (m, 4H). ¹³C NMR (101 MHz, CDCl₃) δ 169.83, 139.59, 139.09, 135.42, 134.82, 131.00, 130.11 (q, *J* = 32.6 Hz), 128.70, 128.35, 127.37, 127.03, 125.88 (q, *J* = 3.8 Hz), 124.36, 124.01 (q, *J* = 272.1 Hz), 65.37, 53.83, 45.53, 25.90, 24.43, 22.42, 22.36. ¹⁹F NMR (376 MHz, CDCl₃) δ -62.55.

HPLC conditions: Nu-Analytical Solutions INB, *n*-hexane/*i*-PrOH = 95/5, flow rate 0.7 mL/min. $\lambda = 254$ nm, t(minor) = 13.4 min, t(major) = 15.0 min, 90% ee. HRMS (ESI) *m*/*z* calcd. for C₂₅H₂₅F₃NO [M+H]⁺ 412.1883, found 412.1880.

(*R*,*E*)-3-(2-cyclopropylvinyl)-3-phenyl-1-(4-(trifluoromethyl)benzyl)azetidin-2one (44)



According to the **general procedure D** with **E1** (57.4 mg, 0.15 mmol) and (*E*)-2-(2-cyclopropylvinyl)-5,5-dimethyl-1,3,2-dioxaborinane **A27** (18.0 mg, 0.10 mmol) at -40 °C for 5 d, the reaction mixture was purified by column chromatography on silica gel (eluent: petroleum ether/ethyl acetate 10/1) to yield the product as a colorless oil (15.0 mg, 0.040 mmol, 40% yield).

¹**H NMR (400 MHz, CDCl₃)** δ 7.60 (d, J = 7.9 Hz, 2H), 7.43 – 7.31 (m, 6H), 7.31 – 7.25 (m, 1H), 5.85 (d, J = 15.4 Hz, 1H), 5.07 (dd, J = 15.4, 9.0 Hz, 1H), 4.50 (ABq, J_{AB} = 15.4 Hz, Δv_{AB} = 41.0 Hz, 2H), 3.48 (ABq, J_{AB} = 5.2 Hz, Δv_{AB} = 21.0 Hz, 2H), 1.45 – 1.34 (m, 1H), 0.77 – 0.63 (m, 2H), 0.42 – 0.27 (m, 2H).

¹³C NMR (101 MHz, CDCl₃) δ 169.84, 139.65, 138.93, 136.99, 130.09 (q, J = 32.6 Hz), 128.64, 128.30, 127.30, 126.95, 126.62, 125.84 (q, J = 3.7 Hz), 124.00 (q, J = 272.1 Hz), 65.12, 53.73, 45.42, 13.94, 6.86, 6.84.

¹⁹F NMR (376 MHz, CDCl₃) δ -62.54.

HPLC conditions: Nu-Analytical Solutions INB, *n*-hexane/*i*-PrOH = 95/5, flow rate 0.7 mL/min. $\lambda = 254$ nm, t(minor) = 22.4 min, t(major) = 26.9 min, 92% ee. **HRMS** (ESI) *m/z* calcd. for C₂₂H₂₁F₃NO [M+H]⁺ 372.1570, found 372.1566.

(*R*,*E*)-3-phenyl-3-(3-(phenylthio)prop-1-en-1-yl)-1-(4-(trifluoromethyl)benzyl)azetidin-2-one (45)



According to the **general procedure D** with **E1** (57.4 mg, 0.15 mmol) and (*E*)-5,5dimethyl-2-(3-(phenylthio)prop-1-en-1-yl)-1,3,2-dioxaborinane **A28** (26.2 mg, 0.10 mmol) at -40 °C for 5 d, the reaction mixture was purified by column chromatography on silica gel (eluent: petroleum ether/ethyl acetate 10/1) to yield the product as a colorless oil (21.7 mg, 0.048 mmol, 48% yield).

¹**H NMR (400 MHz, CDCl₃)** δ 7.58 (d, J = 8.0 Hz, 2H), 7.40 – 7.10 (m, 12H), 5.87 – 5.72 (m, 1H), 5.67 (dt, J = 15.4, 6.8 Hz, 1H), 4.54 (d, J = 15.4 Hz, 1H), 4.38 (d, J = 15.4 Hz, 1H), 3.51 (dt, J = 6.9, 1.3 Hz, 2H), 3.42 (d, J = 5.3 Hz, 1H), 3.29 (d, J = 5.3 Hz, 1H).

¹³C NMR (101 MHz, CDCl₃) δ 169.06, 139.47, 138.22, 135.15, 132.29, 130.97, 130.14 (q, *J* = 32.6 Hz), 128.80, 128.71, 128.28, 127.92, 127.48, 126.85, 126.66, 125.89 (q, *J* = 3.8 Hz), 123.99 (q, *J* = 272.2 Hz), 64.93, 53.25, 45.45, 36.53.

¹⁹F NMR (376 MHz, CDCl₃) δ -62.54.

HPLC conditions: Nu-Analytical Solutions INB, *n*-hexane/*i*-PrOH = 95/5, flow rate 0.7 mL/min. $\lambda = 254$ nm, t(minor) = 30.4 min, t(major) = 32.0 min, 96% ee.

HRMS (ESI) *m*/*z* calcd. for C₂₆H₂₃F₃NOS [M+H]⁺ 454.1447, found 454.1444.

One-mmol scale reaction



Large Scale: according to the **general procedure C** with **E5** (345 mg, 1.0 mmol) and 2-([1,1]-biphenyl]-4-yl)-5,5-dimethyl-1,3,2-dioxaborinane (266 mg, 1.0 mmol) at 0 °C for 45 h, the reaction mixture was purified by column chromatography on silica gel (eluent: petroleum ether/ethyl acetate 5/1) to yield the product as a colorless oil (272 mg, 0.65 mmol, 65% yield).

Derivatizations of α -quaternary β -lactams



(S)-3-([1,1'-biphenyl]-4-yl)-3-phenylazetidin-2-one (46)



To a stirred solution of **5** (154.5 mg, 0.369 mmol, 1.0 equiv.) in a mixture solvent of CH₂Cl₂ (6 mL) and H₂O (0.6 mL) was added 2,3-dichloro-5,6-dicyano-*para*benzoquinone (DDQ, 251 mg, 1.11 mmol, 3.0 equiv.) at room temperature. The reaction mixture was stirred at that temperature for 24 h. Upon completion (monitored by TLC), the reaction mixture was diluted with CH₂Cl₂, quenched with saturated NaHCO₃ solution, and extracted with CH₂Cl₂ three times (3×10 mL). The combined organic layer was washed with saturated NaHCO₃ solution, dried with Na₂SO₄, filtered and concentrated in vacuo. The residue was purified by column chromatography on silica gel (eluent: petroleum ether/ethyl acetate 4/1) to give product **46** as a colorless oil (77.3 mg, 70% yield).

¹**H NMR (400 MHz, CDCl₃)** δ 7.61 – 7.53 (m, 4H), 7.52 – 7.40 (m, 6H), 7.39 – 7.32 (m, 3H), 7.32 – 7.23 (m, 1H), 6.04 (s, 1H), 3.98 (ABq, J_{AB} = 5.3 Hz, Δv_{AB} = 4.4 Hz, 2H).

¹³C NMR (101 MHz, CDCl₃) δ 170.54, 140.60, 140.31, 140.07, 139.17, 128.87, 128.82, 128.79, 128.71, 127.47, 127.45, 127.41, 127.10, 68.05, 51.27.

HPLC conditions: Nu-Analytical Solutions INB, *n*-hexane/*i*-PrOH = 80/20, flow rate 0.5 mL/min. $\lambda = 254$ nm, t(major) = 17.5 min, t(minor) = 19.8 min, 93% ee. **HRMS** (ESI) *m*/*z* calcd. for C₂₁H₁₈NO [M+H]⁺ 300.1383, found 300.1381.

[11,11] [10,150] [1

tert-butyl (S)-3-([1,1'-biphenyl]-4-yl)-2-oxo-3-phenylazetidine-1-carboxylate (47)



To a solution of **46** (29.9 mg, 0.1 mmol, 1.0 equiv.) and 4-dimethylaminopyridine (DMAP, 24.4 mg, 0.20 mmol, 2.0 equiv.) in anhydrous CH₂Cl₂ (2.0 mL) was added Boc₂O (115 μ L, 0.50 mmol, 5.0 equiv.) under argon atmosphere. The resulting mixture was stirred at room temperature for 1 h. Upon completion (monitored by TLC), the reaction mixture was quenched by HCl (0.5 M, 5.0 mL). The mixture was extracted with CH₂Cl₂ three times (3 × 10 mL). The combined organic layer was washed with water and brine, dried over anhydrous Na₂SO₄, filtered and concentrated under reduced pressure. The residue was purified by column chromatography on silica gel (eluent: petroleum ether/ethyl acetate 10/1) to give product **47** as a colorless oil (36.4 mg, 91% yield).

¹**H** NMR (400 MHz, CDCl₃) δ 7.56 (t, J = 8.4 Hz, 4H), 7.51 – 7.40 (m, 6H), 7.40 – 7.33 (m, 3H), 7.33 – 7.26 (m, 1H), 4.25 (ABq, $J_{AB} = 6.6$ Hz, $\Delta v_{AB} = 4.8$ Hz, 2H), 1.54 (s, 9H).

¹³C NMR (101 MHz, CDCl₃) δ 166.66, 148.19, 140.70, 140.42, 139.27, 138.29, 128.96, 128.85, 127.79, 127.61, 127.53, 127.32, 127.11, 126.99, 126.92, 83.81, 65.23, 52.90, 28.06.

HPLC conditions: Nu-Analytical Solutions INB, *n*-hexane/*i*-PrOH = 90/10, flow rate 0.5 mL/min. $\lambda = 254$ nm, t(minor) = 12.9 min, t(major) = 14.7 min, 93% ee. **HRMS** (ESI) *m*/*z* calcd. for C₂₆H₂₆NO₃ [M+Na]⁺ 422.1727, found 422.1721.

tert-butyl (S)-(2-([1,1'-biphenyl]-4-yl)-3-hydroxy-2-phenylpropyl)carbamate (48)



To a solution of 47 (20.0 mg, 0.05 mmol, 1.0 equiv.) in anhydrous THF (1.0 mL) was added LiAlH₄ (5.7 mg, 0.15 mmol, 3.0 equiv.) at 0 °C under argon atmosphere. The reaction mixture was stirred at that temperature for 4 h. Upon completion (monitored by TLC), the reaction mixture was quenched by saturated NH₄Cl (2.0 mL), filtered through a short pad of celite and rinsed with EtOAc. The filtrate was extracted with EtOAc three times (3×10 mL). The combined organic layer was washed with water and brine, dried over anhydrous Na₂SO₄, filtered and concentrated under reduced pressure. The residue was purified by column chromatography on silica gel (eluent: petroleum ether/ethyl acetate 3/1) to give **48** as a colorless oil (18.5 mg, 92% yield).

¹H NMR (400 MHz, CDCl₃) δ 7.70 – 7.47 (m, 4H), 7.42 (t, *J* = 7.5 Hz, 2H), 7.33 (t, *J* = 7.4 Hz, 3H), 7.28 – 7.18 (m, 5H), 4.59 (t, *J* = 6.6 Hz, 1H), 4.21 (s, 2H), 4.11 – 3.95 (m, 2H), 3.58 (s, 1H), 1.43 (s, 9H).

¹³C NMR (101 MHz, CDCl₃) δ 157.48, 144.13, 143.31, 140.60, 139.42, 128.81, 128.57, 128.32, 127.91, 127.33, 127.13, 127.03, 126.75, 80.29, 66.36, 52.23, 45.50, 28.35. HPLC conditions: Nu-Analytical Solutions INB, *n*-hexane/*i*-PrOH = 80/20, flow rate 0.5 mL/min. λ = 254 nm, t(minor) = 14.8 min, t(major) = 20.0 min, 92% ee. HRMS (ESI) *m*/z calcd. for C₂₆H₃₀NO₃ [M+H]⁺ 404.2220, found 404.2215.

tert-butyl (S)-(2-([1,1'-biphenyl]-4-yl)-3-oxo-2-phenylpropyl)carbamate (49)



To a solution of **47** (20.0 mg, 0.05 mmol, 1.0 equiv.) in anhydrous CH₂Cl₂ (1.0 mL) was added DIBALH (0.1 mL, 0.10 mmol, 1 M in hexane, 2.0 equiv.) at -78 °C under argon atmosphere. The reaction mixture was stirred at that temperature for 2 h. Upon completion (monitored by TLC), the reaction mixture was quenched by saturated NH₄Cl (2.0 mL) at -78 °C, filtered through a short pad of celite and rinsed with EtOAc. The filtrate was extracted with EtOAc (3 × 10 mL). The combined organic layer was washed with water and brine, dried over anhydrous Na₂SO₄, filtered and concentrated under reduced pressure. The residue was purified by column chromatography on silica gel (eluent: petroleum ether/ethyl acetate 10/1) to give **49** as a colorless oil (14.2 mg, 71% yield).

¹**H NMR (400 MHz, CDCl₃)** δ 9.91 (s, 1H), 7.72 – 7.52 (m, 4H), 7.52 – 7.31 (m, 6H), 7.32 – 7.03 (m, 4H), 4.80 (s, 1H), 4.11 (d, *J* = 6.5 Hz, 2H), 1.29 (s, 9H).

¹³C NMR (101 MHz, CDCl₃) δ 199.23, 155.51, 140.68, 140.32, 138.26, 137.16, 129.36, 129.05, 128.93, 128.89, 127.89, 127.62, 127.11, 64.14, 53.47, 43.96, 28.28. HPLC conditions: Nu-Analytical Solutions INB, *n*-hexane/*i*-PrOH = 95/5, flow rate 0.7 mL/min. λ = 254 nm, t(minor) = 13.3 min, t(major) = 16.3 min, 93% ee. HRMS (ESI) *m*/*z* calcd. for C₂₆H₂₈NO₃ [M+H]⁺ 402.2064, found 402.2060.

Methyl (S)-2-([1,1'-biphenyl]-4-yl)-3-((tert-butoxycarbonyl)amino)-2-phenylpropanoate (50)



To a solution of 47 (20.0 mg, 0.05 mmol, 1.0 equiv.) in anhydrous MeOH (1.0 mL) was added LiOH (6.0 mg, 0.25 mmol, 5.0 equiv.) at room temperature under argon atmosphere. The resulting mixture was stirred at that temperature for 2 h. Upon completion (monitored by TLC), the reaction mixture was quenched by saturated, filtered through a short pad of celite, and rinsed with EtOAc. The filtrate was extracted with EtOAc three times (3×10 mL). The combined organic layer was washed with water and brine, dried over anhydrous Na₂SO₄, filtered and concentrated under reduced pressure. The residue was purified by column chromatography on silica gel (eluent: petroleum ether/ethyl acetate 10/1) to give **50** as a white solid (12.9 mg, 60% yield).

¹H NMR (400 MHz, CDCl₃) δ 7.57 – 7.41 (m, 4H), 7.36 (t, J = 7.6 Hz, 2H), 7.32 – 7.10 (m, 8H), 4.93 (s, 1H), 4.07 (d, J = 6.4 Hz, 2H), 3.71 (s, 3H), 1.21 (s, 9H). ¹³C NMR (101 MHz, CDCl₃) δ 174.70, 155.50, 140.54, 140.52, 140.14, 139.50, 129.27, 128.81, 128.77, 128.27, 127.42, 127.08, 126.84, 61.15, 52.76, 46.98, 29.74, 28.29. HPLC conditions: Nu-Analytical Solutions ND, *n*-hexane/*i*-PrOH = 95/5, flow rate 0.7 mL/min. $\lambda = 254$ nm, t(minor) = 19.4 min, t(major) = 22.1 min, 92% ee. HRMS (ESI) *m*/*z* calcd. for C₂₇H₃₀NO4 [M+H]⁺ 432.2169, found 432.2167.



The procedure followed the **general procedure D**, except the following changes: After completion of the reaction, the reaction mixture was filtered by a short pad of silica gel (eluent: EtOAc) and concentrated under reduced pressure. Then crude product was dissolved in MeOH (3 mL), followed by the addition of 4 mg Pd/C as solid (5 wt.%, wetted with ca. 55% water). Then a hydrogen-filled balloon was attached. The reaction

was stirred at 50 °C for 8 h. The resulting mixture was filtered and concentrated, and the residue was purified by column chromatography on silica gel (eluent: petroleum ether/ethyl acetate 10/1) to give **51** as a colorless oil (27.2 mg, 63% yield).

(*R*)-3-(3-methoxyphenethyl)-3-phenyl-1-(4-(trifluoromethyl)benzyl)azetidin-2one (51)



¹**H** NMR (400 MHz, CDCl₃) δ 7.59 (d, J = 8.1 Hz, 2H), 7.51 – 7.42 (m, 2H), 7.42 – 7.27 (m, 5H), 7.15 (t, J = 7.9 Hz, 1H), 6.74 – 6.66 (m, 2H), 6.64 (t, J = 2.1 Hz, 1H), 4.47 (ABq, $J_{AB} = 15.3$ Hz, $\Delta v_{AB} = 37.8$ Hz, 2H), 3.75 (s, 3H), 3.45 (d, J = 5.4 Hz, 1H), 3.32 (d, J = 5.4 Hz, 1H), 2.77 – 2.66 (m, 1H), 2.53 – 2.41 (m, 1H), 2.35 – 2.19 (m, 2H). ¹³C NMR (101 MHz, CDCl₃) δ 170.89, 159.71, 142.96, 139.67, 139.37, 130.15 (q, J = 32.5 Hz), 129.43, 128.70, 128.41, 127.90 (d, J = 4.8 Hz), 127.35, 126.69, 125.88 (q, J = 3.7 Hz), 124.00 (q, J = 272.2 Hz), 120.60, 114.01, 111.40, 63.08, 55.16, 51.92, 45.43, 39.33, 31.37.

¹⁹F NMR (376 MHz, CDCl₃) δ -62.56.

HPLC conditions: Nu-Analytical Solutions INB, *n*-hexane/*i*-PrOH = 90/10, flow rate 1.0 mL/min. $\lambda = 254$ nm, t(minor) = 15.7 min, t(major) = 26.6 min, 92% ee. **HRMS** (ESI) *m/z* calcd. for C₃₄H₂₈N₂O₂P [M+H]⁺ 440.1832, found 440.1829.

Radical trap experiments



An oven-dried Schlenk tube equipped with a magnetic stirring bar was charged with CuBr·Me₂S (2.06 mg, 10 mol%), chiral ligand L*3 (7.90 mg, 15 mol%), α -bromo- β -lactam E1 (46.1 mg, 0.12 mmol, 1.2 equiv.), A1 (23.2 mg, 0.1 mmol, 1.0 equiv.), LiO'Bu (24.0 mg, 0.3 mmol, 3.0 equiv.) and BHT (44.1 mg, 0.2 mmol, 2.0 equiv.). The tube was evacuated and backfilled with argon three times. Then 1,4-dioxane/THF (v/v = 3/1, 2.0 mL) and H₂O (1.8 mg, 1.0 equiv.) were sequentially added into the mixture under argon. The tube was sealed and the reaction mixture was allowed to stir at -10 °C for 45 h. Upon completion, the mixture was filtered through a pad of silica gel and rinsed with EtOAc. The filtrate was evaporated and the residue was purified by column chromatography on silica gel (gradient eluent: petroleum ether/ethyl acetate 10/1–5/1) to give 1b as colorless oil (42.0 mg, 67% yield based on E1) and the coupling product 1 as a colorless oil (6.6 mg, 13% yield based on E1).



An oven-dried Schlenk tube equipped with a magnetic stirring bar was charged with CuBr·Me₂S (2.06 mg, 10 mol%), chiral ligand L*3 (7.90 mg, 15 mol%), α -bromo- β -lactam E1 (46.1 mg, 0.12 mmol, 1.2 equiv.), LiO'Bu (24.0 mg, 0.3 mmol, 3.0 equiv.) and BHT (44.1 mg, 0.2 mmol, 2.0 equiv.). The tube was evacuated and backfilled with argon three times. Then 1,4-dioxane/THF (v/v = 3/1, 2.0 mL) and H₂O (1.8 mg, 1.0 equiv.) were sequentially added into the mixture under argon. The tube was sealed and the reaction mixture was allowed to stir at -10 °C for 45 h. Upon completion, the mixture was filtered through a pad of silica gel and rinsed with EtOAc. The filtrate was evaporated and the residue was purified by column chromatography on silica gel (eluent: petroleum ether/ethyl acetate 10/1) to give 1b as colorless oil (46.5 mg, 74% yield based on E1).

Kinetic experiments



CuBr•Me ₂ S (10 mol%), L*3 (15 mol%)		
LiO ^t Bu (3.0 equiv.), H ₂ O (1.0 equiv.)		
BHT (2.0 equiv.)		
1,4-dioxane/THF (v/v = 3/1), Ar, –10 °C		



Reaction time	Yield of 1b
15 min	3%
30 min	6%
45 min	12%
60 min	18%
75 min	21%

An oven-dried Schlenk tube equipped with a magnetic stirring bar was charged with CuBr·Me₂S (2.06 mg, 10 mol%), chiral ligand L*3 (7.90 mg, 15 mol%), α -bromo- β -lactam E1 (46.1 mg, 0.12 mmol, 1.2 equiv.), LiO'Bu (24.0 mg, 0.3 mmol, 3.0 equiv.) and BHT (44.1 mg, 0.2 mmol, 2.0 equiv.). The tube was evacuated and backfilled with argon three times. Then 1,4-dioxane/THF (v/v = 3/1, 2.0 mL) and H₂O (1.8 mg, 1.0 equiv.) were sequentially added into the mixture under argon. The tube was sealed and the reaction mixture was allowed to stir at -10 °C for the indicated time. Upon completion, the mixture was then filtered through a pad of silica gel eluting with EtOAc. The filtrate was evaporated and the yield of 1b (based on E1) was determined by ¹H NMR spectra with 1,3,5-trimethoxybenzene as an internal standard.



Reaction time	Yield of 1b
15 min	4%
30 min	6%
45 min	13%
60 min	17%
75 min	20%

An oven-dried Schlenk tube equipped with a magnetic stirring bar was charged with CuBr·Me₂S (2.06 mg, 10 mol%), chiral ligand L*3 (7.90 mg, 15 mol%), α -bromo- β -lactams E1 (46.1 mg, 0.12 mmol, 1.2 equiv.), A1 (23.2 mg, 0.1 mmol, 1.0 equiv.), LiO'Bu (24.0 mg, 0.3 mmol, 3.0 equiv.) and BHT (44.1 mg, 0.2 mmol, 2.0 equiv.). The tube was evacuated and backfilled with argon three times. Then 1,4-dioxane/THF (v/v

= 3/1, 2.0 mL) and H₂O (1.8 mg, 1.0 equiv.) were sequentially added into the mixture under argon. The tube was sealed and the reaction mixture was allowed to stir at -10 °C for the indicated time. Upon completion, the mixture was then filtered through a pad of silica gel and rinsed with EtOAc. The filtrate was evaporated and the yield of **1b** (based on **E1**) was determined by ¹H NMR spectra with 1,3,5-trimethoxybenzene as an internal standard.

References

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^{200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0} fl (ppm)



























20 10 0 -10 -20 -30 -40 -50 -50 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 -210 -2 f1 (ppm)











10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 -210 f1 (ppm)







10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 -210 -2 f1 (ppm)


























1a









210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 fl (ppm)





 F_3







10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 -210 f1 (ppm)







200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 f1 (ppm)



210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 fl (ppm)





210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 f1 (ppm)







210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 f1 (ppm)







210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 f1 (ppm)



210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 f1 (ppm)



S88















0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 f1 (ppm)



S95



- 170.064 - 188.930 - 188.930 - 188.930 - 188.960 - 188.960 - 128.980



200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 f1 (ppm)





0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 f1 (ppm)







10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 f1 (ppm)



190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 f1 (ppm)



S103



0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 -21 f1 (ppm)





S106



0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 -21(f1 (ppm)






0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 f1 (ppm)





S112





10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 -210 f1 (ppm)



200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 f1 (ppm)

S114





0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 f1 (ppm)



200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -1(f1 (ppm)







20 10 0 -10 -20 -30 -40 -50 -50 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 -210 -2 f1 (ppm)









20 10 0 -10 -20 -30 -40 -50 -50 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 -210 -2 f1 (ppm)





38

0 -10

-20 -30 -40 -50







20 10 0 -10 -20 -30 -40 -50 -50 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 -210 -2 f1 (ppm)





-70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 -210 -2 f1 (ppm) 10 0 -10 -20 -30 -40-50 -60









-25 -30 -35 -40 -45 -50 -55 -60 -65 -70 -75 -80 -85 -90 -95 -100 -105 -110 -115 -120 -125 -130 -135 -140 f1 (ppm)





-70

-80

42

10

6 -10 -20 -30 -40 -50 -60







zo 10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 -210 -2 f1 (ppm)















 F_3



7,7590 7,7590 7,339 7,339 7,339 7,339 7,339 7,339 7,339 7,339 7,339 7,339 7,339 7,339 7,339 7,339 7,339 7,339 7,339 7,238 7,248 7,24























S141





10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 -210 f1 (ppm) **HPLC** spectra





Signal 1: DAD1 A, Sig=254,4 Ref=360,100



Totals :

1.16892e4 146.25598



Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak	RetTime	Туре	Width	Area	Height	Area
#	[min]		[min]	[mAU*s]	[mAU]	%
1	30.693	MM R	1.1943	2.14763e4	299.70462	98.8018
2	41.698	MM	1.5764	260.45026	2.75362	1.1982
Totals :				2.17367e4	302.45824	


Signal 2: DAD1 B, Sig=254,4 Ref=360,100

Peak	RetTime	Туре	Width	Area	Height	Area	
#	[min]		[min]	[mAU*s]	[mAU]	%	
1	23.795	BB	0.7522	2583.58276	51.24211	50.5549	
2	28.235	BB	0.8380	2526.87061	42.47932	49.4451	



5110.45337 93.72142



Signal 2: DAD1 B, Sig=254,4 Ref=360,100

Peak	RetTime	Туре	Width	Area	Height	Area
#	[min]		[min]	[mAU*s]	[mAU]	%
1	23.629	BB	0.7601	9001.26563	179.18869	99.2820
2	28.365	MM	0.9288	65.09454	1.16811	0.7180
Total	ls :			9066.36017	180.35680	



Signal 2: DAD1 B, Sig=254,4 Ref=360,100

Peak	RetTime	Туре	Width	Area	Height	Area	
#	[min]		[min]	[mAU*s]	[mAU]	%	
1	29.115	BB	0.9599	3335.54395	49.38788	50.7647	
2	34.751	BB	0.9817	3235.05273	40.24904	49.2353	

```
Totals :
```

6570.59668 89.63692



Signal 2: DAD1 B, Sig=254,4 Ref=360,100

Peak	RetTime	Туре	Width	Area	Height	Area
#	[min]		[min]	[mAU*s]	[mAU]	%
1	28.550	BB	0.9629	2.55803e4	397.45813	98.9344
2	34.684	MM	1.2898	275.51050	3.56008	1.0656
Total	ls :			2.58558e4	401.01821	



Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak	RetTime	Туре	Width	Area	Height	Area
#	[min]		[min]	[mAU*s]	[mAU]	%
1	15.861	BB	0.5223	1.64123e4	480.75385	98.8352
2	25.461	BB	0.6674	193.41756	3.41160	1.1648

S146

Totals : 1.66058e4 484.16544



Peak Table

PDA Ch	1 254nm		
Peak#	Ret. Time	Area	Area%
1	20.172	27886958	49.815
2	27.221	28093941	50.185



S147

PDA Ch1 254nm									
Peak#	Ret. Time	Area	Area%						
1	21.056	6592824	96.658						
2	28.975	227969	3.342						





Peak	RetTime	Туре	Width	Area	Height	Area	
#	[min]		[min]	[mAU*s]	[mAU]	%	
1	14.368	BB	0.4509	3221.16846	107.71793	50.2814	
2	17.098	BB	0.5602	3185.11646	85.98223	49.7186	



6406.28491 193.70016



Signal 2: DAD1 B, Sig=254,4 Ref=360,100

Peak	RetTime	Туре	Width	Area	Height	Area
#	[min]		[min]	[mAU*s]	[mAU]	%
1	14.257	BB	0.4478	5384.79639	181.67242	99.2502
2	17.224	MM	0.5602	40.68226	1.21034	0.7498

Totals :

5425.47865 182.88277



Signal 2: DAD1 B, Sig=254,4 Ref=360,100



Totals :

3553.53381 82.27156



Signal 2: DAD1 B, Sig=254,4 Ref=360,100

Area Peak RetTime Type Width Height Area [mAU] % # [min] [min] [mAU*s] 1 20.568 MM 0.6982 5952.60938 142.09669 99.1852 2 22.562 MM 0.8728 48.89840 9.33705e-1 0.8148

Totals :

6001.50777 143.03040



Signal 2: DAD1 B, Sig=254,4 Ref=360,100

Peak	RetTime	Туре	Width	Area	Height	Area
#	[min]		[min]	[mAU*s]	[mAU]	%
1	17.591	BB	0.5546	3188.25757	86.78456	50.4456
2	22.564	BB	0.7177	3131.93481	64.36694	49.5544



6320.19238 151.15150



Signal 2: DAD1 B, Sig=254,4 Ref=360,100

Peak	RetTime	Туре	Width	Area	Height	Area	
#	[min]		[min]	[mAU*s]	[mAU]	%	
1	17.641	BB	0.5547	3865.55786	104.69955	98.8184	
2	22.864	MM	0.7420	46.22134	1.03820	1.1816	

Totals :

3911.77920 105.73775



Signal 2: DAD1 B, Sig=254,4 Ref=360,100

Peak	RetTime	Туре	Width	Area	Height	Area
#	[min]		[min]	[mAU*s]	[mAU]	%
1	34.294	BB	0.9794	2067.60864	29.01498	50.7125
2	51.100	MM R	1.8390	2009.50952	18.21189	49.2875



4077.11816 47.22686

=254,4 Ref=360,100 (D:\CHEM32\...220721-FL-26-151EI-INA-9010-1 2022-07-21 16-39-47\26-151-I.D)



Signal 2: DAD1 B, Sig=254,4 Ref=360,100

Peak RetTime Type Width Area Height Area % # [min] [min] [mAU*s] [mAU] 1 33.872 BB 1.0700 6663.96826 91.89967 98.5065 2 51.154 MM 1.8354 101.03861 9.17474e-1 1.4935

Totals :

6765.00687 92.81714

10



Signal 2: DAD1 B, Sig=254,4 Ref=360,100

Peak	RetTime	Туре	Width	Area	Height	Area	
#	[min]		[min]	[mAU*s]	[mAU]	%	
1	34.051	BB	0.9591	1858.44470	26.02610	50.4398	
2	45.643	MM R	1.6438	1826.03821	18.51455	49.5602	
# 1 2	34.051 45.643	 BB MM R	0.9591 1.6438	1858.44470 1826.03821	26.02610 18.51455	50.439 49.560	- 8

Totals :

3684.48291 44.54065





```
Signal 2: DAD1 B, Sig=254,4 Ref=360,100
```

Peak	RetTime	Туре	Width	Area	Height	Area
#	[min]		[min]	[mAU*s]	[mAU]	%
1	33.629	BB	1.0570	6089.19482	84.27390	98.4325
2	45.684	MM	1.5848	96.96959	1.01980	1.5675
Total	ls :			6186.16441	85.29371	





Signal 2: DAD1 B, Sig=254,4 Ref=360,100

Peak	RetTime	Туре	Width	Area	Height	Area
#	[min]		[min]	[mAU*s]	[mAU]	%
1	28.315	BB	0.9898	1.21344e4	181.53654	99.3087
2	46.142	MM	1.6397	84.46295	8.58495e-1	0.6913

Totals: 1.22189e4 182.39504



Signal 2: DAD1 B, Sig=254,4 Ref=360,100

Peak	RetTime	Туре	Width	Area	Height	Area
#	[min]		[min]	[mAU*s]	[mAU]	%
1	15.760	BB	0.5965	2536.95166	63.44510	50.0725
2	18.008	BB	0.5980	2529.60498	64.15504	49.9275

Totals :

5066.55664 127.60014



FEak	Veritile	Type	with	Alea	nergit	Area	
#	[min]		[min]	[mAU*s]	[mAU]	%	
1	15.480	BB	0.6179	8235.60840	194.53886	99.2437	
2	18.146	MM	0.5748	62.75905	1.81964	0.7563	

Totals :

8298.36745 196.35851







Signal 2: DAD1 B, Sig=254,4 Ref=360,100

Peak	RetTime	Туре	Width	Area	Height	Area
#	[min]		[min]	[mAU*s]	[mAU]	%
1	26.742	BB	0.8926	8853.08496	148.70734	99.3268
2	31.128	MM	1.0817	60.00172	9.24464e-1	0.6732

Totals : 8913.08669 149.63180

S157



Signal 2: DAD1 B, Sig=254,4 Ref=360,100

Peak	RetTime	Туре	Width	Area	Height	Area	
#	[min]		[min]	[mAU*s]	[mAU]	%	
1	27.985	BB	0.9754	5615.32861	83.20028	50.3084	
2	35.295	BB	1.2171	5546.47412	64.38098	49.6916	



1.11618e4 147.58126



Signal 2: DAD1 B, Sig=254,4 Ref=360,100



Totals :

4.93222e4 669.86321





1.10583e4 104.96040



Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak RetTime Type Width Area Height Area [min] [min] [mAU*s] [mAU] % # 1 39.232 BB 1.5050 2.45758e4 239.52751 99.6866 2 45.636 MM 1.5159 77.25607 8.49409e-1 0.3134

Totals : 2.46530e4 240.37692



Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak RetTime		Туре	Width	Area	Height	Area
# [min]			[min]	[mAU*s]	[mAU]	%
1	13.439	MM R	0.2907	3776.43628	216.53685	50.0192
2	19.230	MM R	0.6857	3773.53906	91.71773	49.9808



7549.97534 308.25458



Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak	RetTime	Туре	Width	Area	Height	Area
#	[min]		[min]	[mAU*s]	[mAU]	%
1	13.254	BB	0.2781	1.60378e4	890.75775	78.2390
2	18.912	BB	0.5785	4460.67578	112.04576	21.7610
Total	ls :			2.04985e4	1002.80351	



Signal 4: DAD1 D, Sig=230,4 Ref=360,100

Peak	RetTime	Туре	Width	Area	Height	Area
#	[min]		[min]	[mAU*s]	[mAU]	%
1	22.493	MM	0.9629	1.75595e4	303.93359	50.5541
2	32.404	MM	1.8854	1.71745e4	151.82225	49.4459

Totals :

3.47340e4 455.75584



Totals :

3512.81853 34.12981





PDA Ch3 214nm								
Peak#	Ret. Time	Area	Area%					
1	13.667	32720852	49.639					
2	21.728	33196165	50.361					





PDA Ch3 214nm										
Peak#	Ret. Time	Area	Area%							
1	13.887	221046	1.173							
2	22.209	18629440	98.827							



Signal 2: DAD1 B, Sig=214,4 Ref=360,100

Peak	RetTime	Туре	Width	Area	Height	Area
#	[min]		[min]	[mAU*s]	[mAU]	%
1	30.059	VB R	0.6162	1.60771e4	314.61142	49.3848
2	32.225	VV R	0.6708	1.64776e4	292.29239	50.6152

Totals :

3.25546e4 606.90381



Signal 2: DAD1 B, Sig=214,4 Ref=360,100





methyl (S)-4-(2-oxo-3-phenyl-1-(4-(trifluoromethyl)benzyl)azetidin-3-yl)benzoate

Signal 2: DAD1 B, Sig=230,4 Ref=360,100

Peak	RetTime	Туре	Width	Area	Height	Area
#	[min]		[min]	[mAU*s]	[mAU]	%
1	16.263	BV	0.3605	6783.08887	288.25833	49.8735
2	17.550	VB	0.3959	6817.49170	266.99896	50.1265

Totals :

1.36006e4

555.25729



Signal 2: DAD1 B, Sig=230,4 Ref=360,100

Peak	RetTime	Туре	Width	Area	Height	Area
#	[min]		[min]	[mAU*s]	[mAU]	%
		-				
1	16.306	BB	0.3590	4699.31592	200.86104	99.3281
2	17.665	MM	0.3873	31.78858	1.36809	0.6719
Total	s:			4731.10450	202.22913	



Peak Table

Peak#	Ret. Time	Area	Area%
1	21.799	11476243	50.216
2	30.030	11377531	49.784

mAU



PDA Ch	12 214nm		
Peak#	Ret. Time	Area	Area%
1	21.779	11207166	99.177
2	30.169	93007	0.823



Peak Table

PDA Ch3 230nm									
Peak#	Ret. Time	Area	Area%						
1	33.092	12327428	49.709						
2	39.086	12471958	50.291						



PDA Ch	13 230nm		
Peak#	Ret. Time	Area	Area%
1	32.794	9805200	99.902
2	38.240	9627	0.098

mAU



Peak Table

PDA Ch1 254nm								
Peak#	Ret. Time	Area	Area%					
1	15.848	15269777	49.800					
2	17.680	15392732	50.200					



PDA Ch	1 254nm		
Peak#	Ret. Time	Area	Area%
1	15.872 17087001		98.355
2	17.827	285833	1.645



Signal 4: DAD1 D, Sig=230,4 Ref=360,100

Peak	RetTime	Туре	Width	Area	Height	Area
#	[min]		[min]	[mAU*s]	[mAU]	%
1	20.272	BB	0.4576	3761.28345	126.25066	50.0509
2	23.178	BB	0.5184	3753.62939	111.02550	49.9491

Totals :

7514.91284 237.27615



Signal 4: DAD1 D, Sig=230,4 Ref=360,100

Peak	RetTime	Туре	Width	Area	Height	Area	
#	[min]		[min]	[mAU*s]	[mAU]	%	
1	20.488	BB	0.3688	84.57470	2.71949	1.7094	
2	23.312	BB	0.5310	4863.09961	141.50256	98.2906	

Totals :

4947.67431 144.22206



Signal 2: DAD1 B, Sig=254,4 Ref=360,100

Peak	RetTime	Туре	Width	Area	Height	Area
#	[min]		[min]	[mAU*s]	[mAU]	%
1	13.624	BB	0.4335	3585.18311	126.94472	49.9618
2	15.872	BB	0.5060	3590.66284	109.66668	50.0382

Totals :

7175.84595 236.61140



Signal 2: DAD1 B, Sig=254,4 Ref=360,100

Peak	RetTime	Туре	Width	Area	Height	Area
#	[min]		[min]	[mAU*s]	[mAU]	%
		-				
1	13.703	MM	0.4654	21.38318	7.65778e-1	1.0918
2	15.888	BB	0.5002	1937.14575	59.12378	98.9082
Total	s :			1958.52893	59.88956	







Peak Table

PDA Ch	1 254nm		
Peak#	Ret. Time	Area	Area%
1	31.144	2759963	49.787
2	42.672	2783547	50.213



Peak#	Ret. Time	e Area	Area%
1	31.346	2825799	99.240
2	43.577	21635	0.760



Signal 3: DAD1 C, Sig=214,4 Ref=360,100

Peak	RetTime	Туре	Width	Area	Height	Area
#	[min]		[min]	[mAU*s]	[mAU]	%
1	14.801	MM R	0.5331	6339.51318	198.21106	50.7741
2	24.823	MM R	1.3100	6146.19775	78.19623	49.2259

Totals :

1.24857e4 276.40729



Signal 3: DAD1 C, Sig=214,4 Ref=360,100

Peak	RetTime	Туре	Width	Area	Height	Area
#	[min]		[min]	[mAU*s]	[mAU]	%
		-				
1	14.379	VV R	0.4496	3.38161e4	905.03296	96.9422
2	25.251	MM	0.9768	1066.65369	18.20057	3.0578
Tota]	s:			3.48827e4	923.23353	

mAU



Peak Table

PDA Ch	12 225nm		
Peak#	Ret. Time	Area	Area%
1	18.670	8548344	50.165
2	34.132	8492210	49.835





Peak Table

Peak#	Ret. Time	Area	Area%
1	18.795	3858629	99.082
2	38.164	35743	0.918



Signal 3: DAD1 C, Sig=214,4 Ref=360,100

Peak	RetTime	Туре	Width	Area	Height	Area	
#	[min]		[min]	[mAU*s]	[mAU]	%	
1	16.961	MM	0.6552	5953.33105	151.43237	49.7867	
2	22.460	MM	0.8185	6004.33740	122.25704	50.2133	

Totals :

1.19577e4 273.68941



Signal 3: DAD1 C, Sig=214,4 Ref=360,100

Peak	RetTime	Туре	Width	Area	Height	Area
#	[min]		[min]	[mAU*s]	[mAU]	%
1	16.575	BV R	0.4835	3.27101e4	809.27460	99.2092
2	22.305	MM	0.6294	260.74268	6.90459	0.7908
Total	s:			3.29708e4	816.17918	





Signal 4: DAD1 D, Sig=230,4 Ref=360,100

Peak RetTime Type Width Area Height Area [min] [min] [mAU*s] [mAU] % # 1 17.931 BV R 0.3680 4678.41406 194.91843 95.8975 2 20.056 MM 0.4365 200.14212 7.64223 4.1025

Totals	:
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4878.55618 202.56065



Signal 2: DAD1 B, Sig=254,4 Ref=360,100

Peak	RetTime	Туре	Width	Area	Height	Area
#	[min]		[min]	[mAU*s]	[mAU]	%
1	16.854	MM R	0.6592	429.04758	10.84750	50.2920
2	34.403	MM R	1.9928	424.06506	3.54662	49.7080

Totals :

853.11264 14.39412





Signal 2: DAD1 B, Sig=254,4 Ref=360,100

Peak	RetTime	Туре	Width	Area	Height	Area
#	[min]		[min]	[mAU*s]	[mAU]	%
1	16.798	BB	0.5820	938.88367	23.91457	98.5057
2	36.443	MM	1.5001	14.24299	1.58243e-1	1.4943

Totals :

953.12665 24.07282



Signal 2: DAD1 B, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Туре	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	23.971	BB	0.4541	6379.45605	217.54422	49.7476
2	31.136	BB	0.7674	6444.19189	123.37260	50.2524



Signal 2: DAD1 B, Sig=254,4 Ref=360,100

Peak RetTime Type Width Area Height Area # [min] [min] [mAU*s] [mAU] % 0.5028 1.59656e4 489.14960 99.2687 1 24.283 BB 2 30.558 BB 0.4576 117.61275 3.09334 0.7313 Totals : 1.60832e4 492.24294



Signal 2: DAD1 B, Sig=254,4 Ref=360,100

Peak	RetTime	Туре	Width	Area	Height	Area
#	[min]		[min]	[mAU*s]	[mAU]	%
1	24.457	VB	0.6361	1.08603e4	260.65082	50.4388
2	46.821	BB	1.0525	1.06713e4	150.99754	49.5612
Tatal				2 15216-1	111 64026	



2.15316e4 411.64836



Signal 2: DAD1 B, Sig=254,4 Ref=360,100

Peak	RetTime	Туре	Width	Area	Height	Area
#	[min]		[min]	[mAU*s]	[mAU]	%
1	24.467	BB	0.5134	350.63693	9.75339	2.9943
2	46.368	BB	1.0558	1.13597e4	163.25108	97.0057
Total	ls :			1.17103e4	173.00447	



Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Туре	Width [min]	Area [mAU*s]	Height [mAU]	Area %	
1	27.511	BV	0.5143	5460.81641	164.06172	48.6187	
2	28.522	VB	0.5348	5771.11035	159.25868	51.3813	



1.12319e4 323.32040



Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Total	C		
IUCal	. >	•	

5.62868e4 1433.17461



Signal 2: DAD1 B, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Туре	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	34.135	BV	0.7007	3.48702e4	769.42883	49.2672
2	35.848	VB	0.7411	3.59074e4	736.13171	50.7328



Signal 2: DAD1 B, Sig=254,4 Ref=360,100

Peak	RetTime	Туре	Width	Area	Height	Area	
#	[min]		[min]	[mAU*s]	[mAU]	%	
1	36.572	MM	0.7658	2587.69727	56.32151	5.0286	
2	37.873	MM	0.8618	4.88717e4	945.16632	94.9714	

otals	:	
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5.14594e4 1001.48783


Signal 2: DAD1 B, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Туре	Width [min]	Area [mAU*s]	Height [mAU]	Area %	
1	26.481	BV	0.6935	8.55050e4	1905.64050	49.6414	
2	28.586	VB	0.7630	8.67403e4	1753.86169	50.3586	



1.72245e5 3659.50220



Signal 2: DAD1 B, Sig=254,4 Ref=360,100

Peak RetTime Type Width Area Height Area # [min] [min] [mAU*s] [mAU] % 1 26.015 BB 0.5383 905.49200 25.25428 2.8918 2 27.868 BB 0.6442 3.04072e4 726.60645 97.1082

Totals : 3.13127e4 751.86073



Signal 2: DAD1 B, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Туре	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	38.421	BB	1.4432	1.29516e4	124.54884	49.6708
2	56.835	BB	1.6907	1.31233e4	104.38615	50.3292



2.60749e4 228.93499



Signal 2: DAD1 B, Sig=254,4 Ref=360,100

Peak	RetTime	Туре	Width	Area	Height	Area
#	[min]		[min]	[mAU*s]	[mAU]	%
1	40.887	BB	1.0612	6844.25879	97.00379	97.0259
2	61.035	MM	1.3868	209.79242	2.52130	2.9741
Total	ls :			7054.05121	99.52509	



Signal 8: DAD1 H, Sig=280,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	44.707	BB	0.7247	2658.06616	43.75533	49.7237
2	49.684	BB	0.8184	2687.60474	40.07709	50.2763



5345.67090 83.83242



Signal 8: DAD1 H, Sig=280,4 Ref=360,100

Peak	RetTime	Туре	Width	Area	Height	Area
#	[min]		[min]	[mAU*s]	[mAU]	%
1	44.327	BB	0.9608	7425.79053	112.38922	97.8736
2	49.576	BB	0.5249	161.33397	3.66988	2.1264

Totals : 7587.12450 116.05910



Signal 2: DAD1 B, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Туре	Width [min]	Area [mAU*s]	Height [mAU]	Area %	
1	23.778	BB	0.4961	1.37298e4	428.20868	49.5087	
2	38.925	BB	0.9297	1.40023e4	230.18011	50.4913	
1 2	23.778 38.925	BB BB	0.4961 0.9297	1.37298e4 1.40023e4	428.20868 230.18011	49.5087 50.4913	3



2.77321e4 658.38879



Signal 2: DAD1 B, Sig=254,4 Ref=360,100

Peak	RetTime	Туре	Width	Area	Height	Area
#	[min]		[min]	[mAU*s]	[mAU]	%
1	23.718	BB	0.5071	7898.31250	241.82326	97.4181
2	40.327	BB	0.6459	209.33113	3.85280	2.5819

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Totals :
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8107.64363 245.67606



Signal 2: DAD1 B, Sig=254,4 Ref=360,100

Peak	RetTime	Туре	Width	Area	Height	Area
#	[min]		[min]	[mAU*s]	[mAU]	%
1	28.871	BB	0.6319	2.27318e4	552.64990	50.0859
2	47.902	BB	1.0245	2.26538e4	339.49582	49.9141



4.53856e4 892.14572



Signal 2: DAD1 B, Sig=254,4 Ref=360,100

Peak	RetTime	Туре	Width	Area	Height	Area
#	[min]		[min]	[mAU*s]	[mAU]	%
1	27.833	BB	0.5897	2.12690e4	554.20416	97.3701
2	45.498	BB	0.6722	574.46356	10.18212	2.6299
Total	ls :			2.18435e4	564.38628	

S184



Signal 2: DAD1 B, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Туре	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	13.394	BB	0.3502	5.10920e4	2308.82935	49.3289
2	15.016	BB	0.3838	5.24821e4	2157.73169	50.6711



1.03574e5 4466.56104



Signal 2: DAD1 B, Sig=254,4 Ref=360,100

Peak	RetTime	Туре	Width	Area	Height	Area
#	[min]		[min]	[mAU*s]	[mAU]	%
1	13.437	BB	0.2887	473.63528	24.81087	4.8110
2	14.990	BB	0.3092	9371.19824	468.80698	95.1890
Total	ls :			9844.83353	493.61785	





Totals :

7.95001e4 1741.40368



Signal 6: DAD1 F, Sig=225,4 Ref=360,100

 Peak RetTime Type
 Width
 Area
 Height
 Area

 # [min]
 [min]
 [mAU*s]
 [mAU]
 %

----	-----
 ----|
 ----|

 1
 22.389
 BB
 0.4300
 237.10852
 6.52483
 3.9937

 2
 26.874
 BB
 0.6425
 5700.02686
 130.76941
 96.0063

 Totals :
 5937.13538
 137.29423



Signal 2: DAD1 B, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Туре	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	29.471	BV	0.7753	1.96977e4	391.28668	48.9368
2	31.646	MF	1.1068	2.05536e4	309.49512	51.0632



4.02513e4 700.78180



Signal 2: DAD1 B, Sig=254,4 Ref=360,100

Peak	RetTime	Туре	Width	Area	Height	Area
#	[min]		[min]	[mAU*s]	[mAU]	%
1	30.437	BB	0.5130	342.84610	9.87607	1.9820
2	32.019	BB	0.8869	1.69550e4	283.01810	98.0180
Total	ls :			1.72978e4	292.89416	



Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Туре	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	17.600	BV	0.4497	1.83537e4	612.23517	50.0941
2	19.840	VB	0.5253	1.82847e4	534.26135	49.9059
Tota	ls :			3.66384e4	1146.49652	



Signal 5: DAD1 E, Sig=260,4 Ref=360,100

Peak	RetTime	Туре	Width	Area	Height	Area
#	[min]		[min]	[mAU*s]	[mAU]	%
1	17.456	BB	0.5063	5.56042e4	1697.05603	96.3734
2	19.750	MM	0.5067	2092.40430	68.82820	3.6266
Total	s:			5.76966e4	1765.88423	



Signal 8: DAD1 H, Sig=280,4 Ref=360,100

Peak	RetTime	Туре	Width	Area	Height	Area
#	[min]		[min]	[mAU*s]	[mAU]	%
1	12.710	BB	0.2664	6776.86035	390.68729	50.0474
2	14.609	BB	0.3124	6764.03711	330.99664	49.9526



Signal 8: DAD1 H, Sig=280,4 Ref=360,100

Peak RetTime Type Width Height Area Area # [min] [min] [mAU*s] [mAU] % 1 12.894 MM 0.2405 1059.30017 73.39728 3.1591 2 14.695 BB 0.3607 3.24719e4 1409.70300 96.8409 Totals : 3.35312e4 1483.10028



Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak	RetTime	Туре	Width	Area	Height	Area	
#	[min]		[min]	[mAU*s]	[mAU]	%	
1	15.501	BB	0.3516	2.69694e4	1193.44373	49.5821	
2	21.154	BB	0.6223	2.74241e4	683.18335	50.4179	

5.43935e4 1876.62708



Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak	RetTime	Туре	Width	Area	Height	Area
#	[min]		[min]	[mAU*s]	[mAU]	%
1	14.789	BB	0.3100	890.34546	43.64949	4.0727
2	19.972	BB	0.6135	2.09709e4	530.08606	95.9273
Total	ls:			2.18612e4	573.73555	



Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak	RetTime	Туре	Width	Area	Height	Area
#	[min]		[min]	[mAU*s]	[mAU]	%
1	13.237	BB	0.3141	1.60573e4	786.85956	50.4349
2	16.182	VB R	0.4157	1.57804e4	584.95282	49.5651

Totals :

3.18377e4 1371.81238



Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak	RetTime	Туре	Width	Area	Height	Area
#	[min]		[min]	[mAU*s]	[mAU]	%
1	13.330	BB	0.3019	337.81970	17.14351	3.6321
2	16.265	BB	0.4155	8963.14746	331.46527	96.3679
Tota]	ls :			9300.96716	348.60878	



PDA Ch	1 254nm		
Peak#	Ret. Time	Area	Area%
1	19.388	5089493	50.730
2	22.171	4943011	49.270

Peak Table

PDA Ch	1 254nm		
Peak#	Ret. Time	Area	Area%
1	19.351	316264	4.161
2	22.132	7285119	95.839

mAU

Signal 4: DAD1 D, Sig=230,4 Ref=360,100

Peak	RetTime	Туре	Width	Area	Height	Area
#	[min]		[min]	[mAU*s]	[mAU]	%
1	16.004	BB	0.4168	1.93772e4	727.38300	48.5816
2	27.159	BB	0.7015	2.05087e4	415.77646	51.4184

3.98859e4 1143.15945

Signal 4: DAD1 D, Sig=230,4 Ref=360,100

Peak	RetTime Ty	pe Width	Area	Height	Area
#	[min]	[min]	[mAU*s]	[mAU]	%
1	15.659 BB	0.3329	296.55893	13.36415	3.7787
2	26.581 VB	R 0.6334	7551.70801	185.31050	96.2213
Total	ls :		7848.26694	198.67465	