Supplemental Information

Discrimination of Enantiomers of Amides with Two Stereogenic Centers Enabled by Chiral Bisthiourea Derivatives as Chiral Solvating Agents Using ^{1}H NMR Spectroscopy

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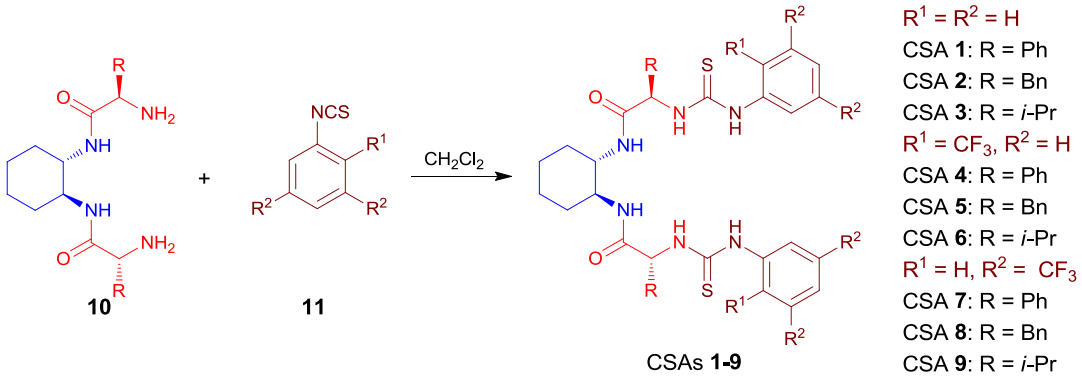
Synthesis of chiral bisthiourea derivatives 1–9 as CSAs.



General procedure of synthesis of chiral bisthiourea derivatives 1–9: Chiral diamines **10** (R = Ph, Bn and ^{i}Pr) were prepared starting from (1*S*,2*S*)-(+)-1,2-diaminocyclohexane, *D*-α-amino acids (phenylglycine, phenylalanine and valine) according to reported procrdures, respectively. Phenylsothiocyanate or its respective derivatives **11** (4 mmol) in CH_2Cl_2 (5 mL) was added into a solution of the corresponding chiral diamines **10** (2 mmol) in CH_2Cl_2 (10 mL). And then, the mixture was stirred under a nitrogen atmosphere at room temperature and monitored by TLC. After the reaction was carried out, and the solvent was removed under reduced pressure. The residue was purified by column chromatography on silica gel to CSAs **1**–**9** in 55–67% yields.

(2*R*,2'*R*)-*N*,*N*'-((1*S*,2*S*)-cyclohexane-1,2-diyl)bis(2-phenyl-2-(3-phenylthioureido)acetamide) (CSA **1**): 58% yield; R_f = 0.3 (ethyl acetate / petroleum ether = 1/1); mp.168–170 ℃; $[\alpha]_D^{25}$ -8.9 (*c* 2.5, THF); ^{1}H NMR (400 MHz, $CDCl_3$) *δ*: 1.06-1.11 (m, 2H), 1.15-1.20 (m. 2H), 1.61-1.66 (m, 2H), 1.82-1.85 (m, 2H), 3.63 (br, 2H), 5.95 (d, *J* = 6.96 Hz, 2H), 6.58 (d, *J* = 4.08 Hz, 2H), 7.23-7.42 (m, 20H), 7.48 (d, *J* = 6.92 Hz, 2H), 8.33 (s, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) *δ*: 24.3, 31.7, 54.4, 62.4, 124.9, 126.9, 127.5, 128.4, 129.0, 129.9, 136.6, 137.1, 171.1, 180.2; HRMS (ESI$^+$-TOF) *m*/*z*: calcd for $C_{36}H_{39}N_6O_2S_2$ $(M+H)^+$: 651.2570, found: 651.2571; IR (KBr): 3285, 2928, 2853, 1649, 1520, 1495, 756, 694 cm^{-1}.

(2*R*,2'*R*)-*N*,*N*'-((1*S*,2*S*)-cyclohexane-1,2-diyl)bis(3-phenyl-2-(3-phenylthioureido)propanamide) (CSA **2**): 63% yield; R_f = 0.3 (ethyl acetate / petroleum ether = 1/1); mp. 171–173 ℃; $[\alpha]_D^{25}$ -37.8 (*c* 2.5, THF); ^{1}H NMR (400 MHz, $CDCl_3$) *δ*: 0.89-0.97 (m, 2H), 1.14-1.19 (m, 2H), 1.61-1.64 (m, 2H), 1.75-1.78 (m, 2H), 2.99 (dd, *J* = 8.48 Hz, *J* = 13.68 Hz, 2H), 3.23 (dd, *J* = 6.32 Hz, *J* = 13.64 Hz, 2H), 3.41-3.45 (m, 2H), 5.03-5.09 (m, 2H), 6.19 (br, 2H), 6.92 (d, *J* = 7.52 Hz, 2H), 7.09-7.11 (m, 4H), 7.19-7.25 (m, 9H), 7.27-7.36 (m, 7H), 8.04 (br, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) *δ*: 24.1, 31.5, 38.0, 53.5, 59.4, 124.7, 126.6, 126.8, 128.4, 129.1, 129.6, 136.1, 136.2, 171.0, 179.6; HRMS (ESI$^+$-TOF) *m*/*z*: calcd for $C_{38}H_{43}N_6O_2S_2$ $(M+H)^+$: 679.2883, found: 679.2880; IR (KBr): 3310, 2926, 2855, 1655, 1524, 1499, 1354, 748, 698 cm^{-1}.

(2*R*,2'*R*)-*N*,*N*'-((1*S*,2*S*)-cyclohexane-1,2-diyl)bis(3-methyl-2-(3-phenylthioureido)butanamide) (CSA **3**): 55% yield; R_f = 0.25 (ethyl acetate / petroleum ether = 2/1); mp. 167–168 ℃; $[\alpha]_D^{25}$ -11.9 (*c* 2.5, THF); ^{1}H NMR (400 MHz, $CDCl_3$) *δ*: 0.96 (d, *J* = 6.56 Hz, 6H), 0.95-0.97 (m, 12H), 1.25-1.26 (m, 4H), 1.70 (br, 2H), 2.02-2.04 (m, 2H), 2.15-2.25 (m, 2H), 3.70 (br, 2H), 4.75-4.79 (m, 2H), 7.03-7.06 (m, 4H), 7.14-7.16 (m, 4H), 7.20-7.24 (m, 2H), 7.31-7.35 (m, 4H), 8.23 (br, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) *δ*: 18.7, 19.0, 24.4, 30.5, 32.0, 54.4, 63.6, 125,5, 126.9, 129.6, 136.9, 171.9, 180.9; HRMS (ESI$^+$-TOF) *m*/*z*: calcd for $C_{30}H_{43}N_6O_2S_2$ $(M+H)^+$: 583.2883, found: 583.2886; IR (KBr): 3287, 2930, 2857, 1643, 1603, 1528, 1350, 1319, 756, 698 cm^{-1}.

(2*R*,2'*R*)-*N*,*N*'-((1*S*,2*S*)-cyclohexane-1,2-diyl)bis(2-phenyl-2-(3-(2-(trifluoromethyl)phenyl)thioureido)aceta mide) (CSA **4**): 62% yield; R_f = 0.3 (ethyl acetate / petroleum ether = 1/2); mp. 227–229 ℃; $[\alpha]_D^{25}$ -32.7 (*c*

2.5, THF); ^{1}H NMR (400 MHz, $CDCl_3$) δ: 1.03-1.05 (m, 4H), 1.55 (br, 2H), 1.78-1.80 (m, 2H), 3.59 (br, 2H), 5.95 (d, J = 5.24 Hz, 2H), 6.71 (br, 2H), 7.29-7.32 (m, 10H), 7.39-7.43 (m, 4H), 7.64-7.70 (m, 4H), 7.74-7.76 (m, 2H), 8.11 (br, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 24.0, 31.6, 54.3, 62.5, 123.2 (q, J = 271.2 Hz), 126.7 (q, J = 29.3 Hz), 127.0, 127.2, 127.7, 128.4, 129.0, 130.3, 133.1, 135.0, 137.0, 171.0, 181.6; ^{19}F NMR (376 MHz, $CDCl_3$) δ: -61.4; HRMS (ESI$^+$-TOF) m/z: calcd for $C_{38}H_{37}N_6O_2F_6S_2$ $(M+H)^+$: 787.2318, found: 787.2320; IR (KBr): 3283, 2936, 1647, 1543, 1321, 1173, 1126, 766, 701 cm^{-1}.

(2*R*,2'*R*)-*N*,*N*'-((1*S*,2*S*)-cyclohexane-1,2-diyl)bis(3-phenyl-2-(3-(2-(trifluoromethyl)phenyl)thioureido)propa namide) (CSA **5**): 62% yield; R_f = 0.3 (ethyl acetate / petroleum ether = 1/1); mp. 129–130 ℃; $[\alpha]_D^{25}$ -62.5 (*c* 2.5, THF); ^{1}H NMR (400 MHz, $CDCl_3$) δ: 0.93-1.02 (m, 2H), 1.11-1.17 (m, 2H), 1.60-1.64 (m, 2H), 1.77-1.81 (m, 2H), 3.04 (dd, J = 7.94 Hz, J = 13.70 Hz, 2H), 3.18 (dd, J = 6.02 Hz, J = 13.78 Hz, 2H), 3.42-3.46 (m, 2H), 5.08-5.13 (m, 2H), 6.38 (d, J = 5.08 Hz, 2H), 6.83 (d, J = 7.48 Hz, 2H), 7.14-7.16 (m, 4H), 7.21-7.26 (m, 6H), 7.32 (d, J = 7.88 Hz, 2H), 7.38 (dd, J = 7.56 Hz, J = 7.60 Hz,2H), 7.55 (dd, J = 7.68 Hz, J = 7.56 Hz,2H), 7.66 (d, J = 7.76 Hz, 2H), 7.77 (br, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 24.2, 31.6, 38.1, 53.9, 59.6, 123.2 (q, J = 271.8 Hz), 126.5 (q, J = 29.8 Hz), 127.0, 127.3, 127.6, 128.6, 129.3, 130.0, 133.2, 134.5, 136.2, 171.2, 181.1; ^{19}F NMR (376 MHz, $CDCl_3$) δ: -61.5; HRMS (ESI$^+$-TOF) m/z: calcd for $C_{40}H_{41}N_6O_2F_6S_2$ $(M+H)^+$: 815.2631, found: 815.2633; IR (KBr): 3318, 2934, 2859, 1726, 1651, 1391, 1319, 1196, 1171, 1032, 766 cm^{-1}.

(2*R*,2'*R*)-*N*,*N*'-((1*S*,2*S*)-cyclohexane-1,2-diyl)bis(3-methyl-2-(3-(2-(trifluoromethyl)phenyl)thioureido)butana mide) (CSA **6**): 61% yield; R_f = 0.25 (ethyl acetate / petroleum ether = 3/2); mp. 215–216 ℃; $[\alpha]_D^{25}$ -5.0 (*c* 2.5, THF); ^{1}H NMR (400 MHz, $CDCl_3$) δ: 0.90 (d, J = 6.80 Hz, 6H), 0.94 (d, J = 6.72 Hz, 6H), 1.27-1.29 (m, 4H), 1.73 (br, 2H), 2.03-2.05 (m, 2H), 2.15-2.25 (m, 2H), 3.71 (br, 2H), 4.80 (m, 2H), 7.06-7.08 (d, 2H), 7.11-7.12 (m, 2H), 7.38 (dd, J = 7.16 Hz, J = 7.32 Hz, 2H), 7.56 (dd, J = 7.48 Hz, J = 7.32 Hz, 2H), 7.66 (d, J = 7.68 Hz, 2H), 7.94 (br, 2H); ^{13}C NMR (100 MHz, DMSO-d_6) δ: 18.3, 19.2, 24.4, 32.2, 32.4, 51.5, 62.0, 122.7, 125.2 (q, J = 28.7 Hz), 125.4, 126.2 (q, J = 4.78 Hz), 126.8, 132.6, 132.7, 137.9, 170.6, 183.0; ^{19}F NMR (376 MHz, $CDCl_3$) δ: -61.5; HRMS (ESI$^+$-TOF) m/z: calcd for $C_{32}H_{41}N_6O_2F_6S_2$ $(M+H)^+$: 719.2631, found: 719.2629; IR (KBr): 3304, 2965, 2934, 1639, 1531, 1321, 1171, 1130, 1059, 764, 711 cm^{-1}.

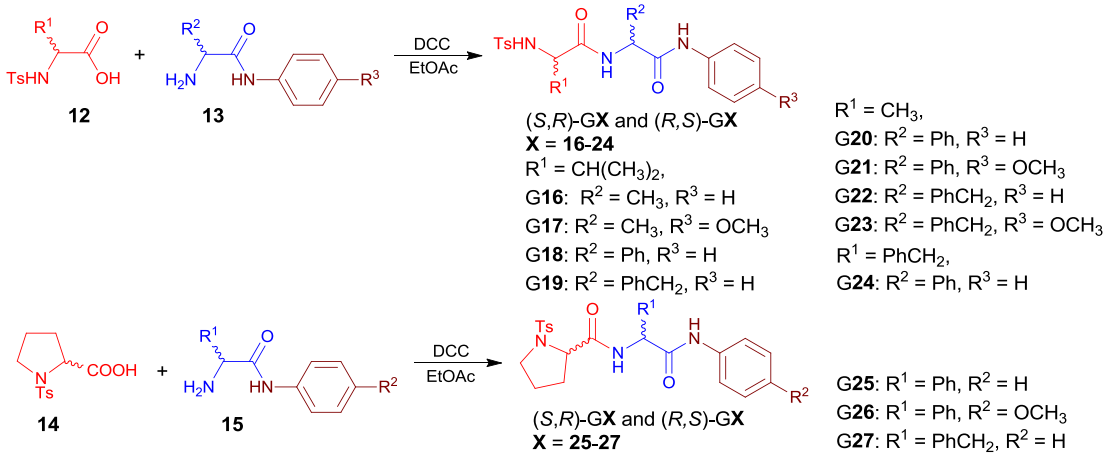
(2*R*,2'*R*)-*N*,*N*'-((1*S*,2*S*)-cyclohexane-1,2-diyl)bis(2-(3-(3,5-bis(trifluoromethyl)phenyl)thioureido)-2-phenyla cetamide) (CSA **7**): 67% yield; R_f = 0.3 (ethyl acetate / petroleum ether = 2/3); mp. 153–155 ℃; $[\alpha]_D^{25}$ -52.0 (*c* 2.5, THF); ^{1}H NMR (400 MHz, $CDCl_3$) δ: 0.98-1.06 (m, 4H), 1.55-1.56 (m, 2H), 1.77-1.80 (m, 2H), 3.62 (br, 2H), 6.04 (br, 2H), 6.78 (br, 2H), 7.31-7.35 (m, 10H), 7.57 (s, 2H), 7.88 (s, 6H), 9.01 (br, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 24.0, 31.6, 54.4, 62.2, 118.6, 122.9 (q, J = 271.2 Hz), 123.3, 127.3, 128.9, 129.2, 132.0 (q, J = 33.9 Hz), 136.2, 139.8, 171.7, 180.6; ^{19}F NMR (376 MHz, $CDCl_3$) δ: -63.0; HRMS (ESI$^+$-TOF) m/z: calcd for $C_{40}H_{35}N_6O_2F_{12}S_2$ $(M+H)^+$: 923.2065, found: 923.2063; IR (KBr): 3428, 2934, 1653, 1516, 1383, 1279, 1180, 1134, 887, 700 cm^{-1}.

(2*R*,2'*R*)-*N*,*N*'-((1*S*,2*S*)-cyclohexane-1,2-diyl)bis(2-(3-(3,5-bis(trifluoromethyl)phenyl)thioureido)-3-phenylpr opanamide) (CSA **8**): 59% yield; R_f = 0.3 (ethyl acetate / petroleum ether = 1/1); mp. 199–200 ℃; $[\alpha]_D^{25}$ -47.3 (*c* 25, THF); ^{1}H NMR (400 MHz, $CDCl_3$) δ: 0.76 (br, 2H), 0.86-0.89 (m, 2H), 1.46 (br, 2H), 1.52-1.55 (m, 2H), 3.05-3.10 (m, 2H), 3.30-3.37 (m, 4H), 5.05 (br, 2H), 6.37 (br, 2H), 7.16-7.26 (m, 4H), 7.22 (br, 6H), 7.54-7.61 (m, 4H), 7.88 (s, 4H), 8.97 (br, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 23.8, 31.4, 38.9, 53.9, 60.1, 118.8, 122.9 (q, J = 271.2 Hz), 123.5, 127.4, 128.8, 129.2, 132.2 (q, J = 33.8 Hz), 135. 7, 139.7, 171.6, 180.4; ^{19}F NMR (376 MHz, $CDCl_3$) δ: -63.0; HRMS (ESI$^+$-TOF) m/z: calcd for $C_{42}H_{39}N_6O_2F_{12}S_2$ $(M+H)^+$: 951.2378, found: 951.2381; IR (KBr): 3480, 3306, 3063, 1655, 1584, 1383, 1329, 1279, 1184, 1130, 849, 707 cm^{-1}.

(2*R*,2'*R*)-*N*,*N*'-((1*S*,2*S*)-cyclohexane-1,2-diyl)bis(2-(3-(3,5-bis(trifluoromethyl)phenyl)thioureido)-3-methylb

utanamide) (CSA **9**): 64% yield; R_f = 0.25 (ethyl acetate / petroleum ether = 2/1); mp. 214–216 ℃; $[\alpha]_D^{25}$ -79.6 (*c* 2.5, THF); ^{1}H NMR (400 MHz, $CDCl_3$) δ: 1.00-1.02 (m, 12H), 1.16-1.22 (m, 4H), 1.67 (br, 2H), 2.01-2.03 (m, 2H), 2.23 (br, 2H), 3.65 (br, 2H), 4.79 (br, 2H), 6.94 (br, 2H), 7.36 (br, 2H), 7.60 (br, 2H), 7.83 (br, 4H), 8.84 (br, 2H); ^{13}C NMR (100 MHz, DMSO-d_6) δ: 22.4, 22.9, 28.2, 35.6, 35.7, 55.7, 65.4, 120.1, 125.4, 127.4 (q, *J* = 273.4 Hz), 134.3 (q, *J* = 31.5 Hz), 146.1, 173.9, 184.2; ^{19}F NMR (376 MHz, $CDCl_3$) δ: -63.1; HRMS (ESI$^+$-TOF) *m/z*: calcd for $C_{34}H_{39}N_6O_2F_{12}S_2$ $(M+H)^+$: 855.2378, found: 855.2381; IR (KBr): 3296, 2968, 2938, 2860, 1634, 1470, 1385, 1279, 1190, 1138, 1101, 885, 704 cm^{-1}.

Synthesis of enantiomers of amides with two stereogenic centers as guests.



General procedure of enantiomers of amides 16–27: *N*-Ts-*L*- and *D*-α-amino acids **12** and **14**, and chiral amines (*R*)- and (*S*)-**13** and **15** were derived from corresponding α-amino acids, respectively. *N*-Ts-*L*- or *D*-α-amino acids **12** or **14** (1.0 mmol) was added into a solution of the corresponding (*R*)- and (*S*)-**13** or **15** (1.0 mmol) in dried EtOAc (10 mL) at room temperature, respectively. DCC (1.05 mmol) dissolved in dried EtOAc (5 mL) was dropped into the stirred mixture at ice bath under nitrogen atmosphere. After the reaction was carried out, the reaction mixture was filtered. The filtrate was concentrated under reduced pressure, and the crude product was purified by column chromatography on silica gel to afford corresponding (*S*,*R*)-G**X** and (*R*,*S*)-G**X** (**X** = **16**–**27**) in 50–68 % yields.

(*S*)-3-methyl-2-(4-methylphenylsulfonamido)-*N*-((*R*)-1-oxo-1-(phenylamino)propan-2-yl)butanamide ((*S*,*R*)-G**16**): 57% yield; R_f = 0.3 (ethyl acetate / petroleum ether = 1/1); mp. 221–223 ℃; $[\alpha]_D^{25}$ +54.1 (*c* 1.67, THF); ^{1}H NMR (400 MHz, $CDCl_3$) δ: 0.80 (d, *J* = 6.44 Hz, 3H), 0.81 (d, *J* = 6.56 Hz, 3H), 1.39 (d, *J* = 6.92 Hz, 3H), 2.04-2.11 (m, 1H), 2.27 (s, 3H), 3.59 (dd, *J* = 7.02 Hz, *J* = 6.04 Hz, 2H), 4.57-4.64 (m, 1H), 6.09 (d, *J* = 8.24 Hz, 1H), 7.07-7.11 (m, 2H), 7.19 (d, *J* = 8.00 Hz, 2H), 7.27 (t, *J* = 7.74 Hz, 2H), 7.48 (d, *J* = 7.88 Hz, 2H), 7.74 (d, *J* = 8.08 Hz, 2H), 8.60 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 17.5, 17.6, 19.1, 21.4, 31.4, 49.7, 62.5, 120.1, 124.5, 127.2, 128.9, 129.7, 136.6, 137.7, 143.9, 170.1, 171.7; HRMS (ESI$^+$-TOF) *m/z*: calcd for $C_{21}H_{28}N_3O_4S$ $(M+H)^+$: 418.1795, found: 418.1792; IR (KBr): 3300, 3252, 2965, 1672, 1641, 1599, 1541, 1447, 1161, 1089, 754, 694 cm^{-1}.

(*R*)-3-methyl-2-(4-methylphenylsulfonamido)-*N*-((*S*)-1-oxo-1-(phenylamino)propan-2-yl)butanamide ((*R*,*S*)-G**16**): 62% yield; R_f = 0.3 (ethyl acetate / petroleum ether = 1/1); mp. 221–223 ℃; $[\alpha]_D^{25}$ -53.8 (*c* 1.67, THF); ^{1}H NMR (400 MHz, $CDCl_3$) δ: 0.81 (d, *J* = 8.00 Hz, 6H), 1.38 (d, *J* = 6.68 Hz, 3H), 2.03-2.08 (m, 1H), 2.27 (s, 3H), 3.60 (dd, *J* = 6.56 Hz, *J* = 6.24 Hz, 1H), 4.59-4.62 (m, 1H), 6.08 (d, *J* = 7.20 Hz, 1H), 7.07-7.10 (m, 2H), 7.18 (d, *J* = 7.64 Hz, 2H), 7.24-7.28 (m, 2H), 7.48 (d, *J* = 7.64 Hz, 2H), 7.73 (d, *J* = 7.76 Hz, 2H), 8.58 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 17.5, 17.6, 19.1, 21.4, 31.4, 49.7, 62.5, 120.1, 124.5, 127.2, 128.9, 129.7, 136.7, 137.7, 143.9, 170.0, 171.7; HRMS (ESI$^+$-TOF) *m/z*: calcd for $C_{21}H_{28}N_3O_4S$

$(M+H)^+$: 418.1795, found: 418.1798; IR (KBr): 3300, 3254, 2965, 1667, 1641, 1541, 1447, 1329, 1159, 1088, 694 cm^{-1}.

(*S*)-*N*-((*R*)-1-((4-methoxyphenyl)amino)-1-oxopropan-2-yl)-3-methyl-2-(4-methylphenylsulfonamido)butanamide ((*S*,*R*)-G**17**): 55% yield; R_f = 0.3 (ethyl acetate / petroleum ether = 1/1); mp. 225–227 ℃; $[\alpha]_D^{25}$ +177.8 (*c* 1.67, DMSO); ^{1}H NMR (400 MHz, $CDCl_3$) δ: 0.81 (d, *J* = 4.72 Hz, 6H), 1.38 (d, *J* = 6.24 Hz, 3H), 2.06 (br, 1H), 2.30 (s, 3H), 3.54 (br, 1H), 3.77 (s, 3H), 4.54 (br, 1H), 5.85 (br, 1H), 6.81 (d, *J* = 8.16 Hz, 2H), 6.89 (d, *J* = 5.88 Hz, 1H), 7.20 (d, *J* = 7.12 Hz, 2H), 7.37 (d, *J* = 8.12 Hz, 2H), 7.73 (d, *J* = 7.40 Hz, 2H), 8.34 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 17.5, 17.6, 19.1, 21.4, 31.4, 49.5, 55.5, 62.5, 114.1, 121.9, 127.2, 129.7, 130.7, 136.7, 143.8, 156.6, 169.8, 171.6; HRMS (ESI^+-TOF) *m*/*z*: calcd for $C_{22}H_{30}N_3O_5S$ $(M+H)^+$: 448.1900, found: 448.1905; IR (KBr): 3294, 3258, 2963, 1641, 1541, 1514, 1447, 1242, 1159, 1088, 694 cm^{-1}.

(*R*)-*N*-((*S*)-1-((4-methoxyphenyl)amino)-1-oxopropan-2-yl)-3-methyl-2-(4-methylphenylsulfonamido)butanamide ((*R*,*S*)-G**17**): 61% yield; R_f = 0.3 (ethyl acetate / petroleum ether = 1/1); mp. 229–231 ℃; $[\alpha]_D^{25}$ -177.6 (*c* 1.67, DMSO); ^{1}H NMR (400 MHz, $CDCl_3$) δ: 0.81 (d, *J* = 6.72 Hz, 6H), 1.38 (d, *J* = 6.96 Hz, 3H), 2.02-2.08 (m, 1H), 2.30 (s, 3H), 3.56-3.59 (m, 1H), 3.76 (s, 3H), 4.54-4.61 (m, 1H), 6.03 (d, *J* = 8.44 Hz, 2H), 6.79 (d, *J* = 8.88 Hz, 2H), 7.00 d, *J* = 7.68 Hz, 1H), 7.20 (d, *J* = 8.04 Hz, 2H), 7.36 d, *J* = 8.88 Hz, 2H), 7.74 (d, *J* = 8.12 Hz, 2H), 8.41 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 17.5, 19.1, 21.5, 29.7, 31.4, 49.6, 55,5, 62.5, 114.2, 121.8, 127.3, 129.8, 130.8, 130.8, 144.0, 156.7, 169.5, 171.4; HRMS (ESI^+-TOF) *m*/*z*: calcd for $C_{22}H_{30}N_3O_5NaS$ $(M+Na)^+$: 470.1720, found: 470.1720; IR (KBr): 3296, 3267, 2965, 1643, 1541, 1514, 1449, 1244, 1159, 1090, 826, 694 cm^{-1}.

(*S*)-3-methyl-2-(4-methylphenylsulfonamido)-*N*-((*R*)-2-oxo-1-phenyl-2-(phenylamino)ethyl)butanamide ((*S*,*R*)-G**18**): 55% yield; R_f = 0.25 (ethyl acetate / petroleum ether = 3/2); mp. 256–258 ℃; $[\alpha]_D^{25}$ -15.5 (*c* 1.67, DMSO); ^{1}H NMR (400 MHz, $CDCl_3$) δ: 0.79 (2d, *J* = 7.56 Hz, *J* = 7.32 Hz, 6H), 1.97-2.04 (m, 1H), 2.29 (s, 3H), 3.54 (dd, *J* = 8.36 Hz, *J* = 6.28 Hz, 1H), 5.26 (d, *J* = 6.20 Hz, 2H), 7.01 (d, *J* = 6.64 Hz, 1H), 7.11-7.15 (m, 1H), 7.23 (d, *J* = 8.16 Hz, 2H), 7.30-7.34 (m, 8H), 7.45 (d, *J* = 7.68 Hz, 2H), 7.73 (d, *J* = 8.04 Hz, 2H); ^{13}C NMR (100 MHz, DMSO-d_6) δ: 18.8, 19.3, 21.2, 31.5, 56.3, 61.8, 119.3, 124.1, 126.9, 127.3, 128.1, 128.8, 129.3, 129.4, 138.8, 138.9, 139.2, 142.5, 168.6, 170.1; HRMS (ESI^+-TOF) *m*/*z*: calcd for $C_{26}H_{30}N_3O_4S$ $(M+H)^+$: 480.1951, found: 480.1950; IR (KBr): 3301, 3258, 2962, 1643, 1539, 1447, 1331, 1161, 1090, 752, 710 cm^{-1}.

(*R*)-3-methyl-2-(4-methylphenylsulfonamido)-*N*-((*S*)-2-oxo-1-phenyl-2-(phenylamino)ethyl)butanamide ((*R*,*S*)-G**18**): 61% yield; R_f = 0.25 (ethyl acetate / petroleum ether = 3/2); mp. 259–261 ℃; $[\alpha]_D^{25}$ +15.1 (*c* 1.67, DMSO); ^{1}H NMR (400 MHz, $CDCl_3$) δ: 0.79 (2d, *J* = 7.36 Hz, *J* = 7.36 Hz, 6H), 1.96-2.02 (m, 1H), 2.29 (s,3H), 3.54 (dd, *J* = 7.48 Hz, *J* = 7.56 Hz, 1H), 5.26 (d, *J* = 6.28 Hz, 1H), 7.02-7.03 (m, 1H), 7.11-7.15 (m, 1H), 7.22 (d, *J* = 8.04 Hz, 1H), 7.30-7.34 (m, 9H), 7.45 (d, *J* = 8.00 Hz, 2H), 7.73 (d, *J* = 8.20 Hz, 2H); ^{13}C NMR (100 MHz, DMSO-d_6) δ: 19.6, 20.2, 22.1, 32.4, 57.4, 62.8, 120.3, 125.0, 127.8, 128.3, 129.0, 129.7, 130.2, 130.4, 139.7, 140.1, 143.4, 169.5, 171.1; HRMS (ESI^+-TOF) *m*/*z*: calcd for $C_{26}H_{30}N_3O_4S$ $(M+H)^+$: 480.1951, found: 480.1957; IR (KBr): 3306, 3253, 2965, 1643, 1539, 1447, 1329, 1161, 1090, 752, 712 cm^{-1}.

(*S*)-3-methyl-2-(4-methylphenylsulfonamido)-*N*-((*R*)-1-oxo-3-phenyl-1-(phenylamino)propan-2-yl)butanamide ((*S*,*R*)-G**19**): 66% yield; R_f = 0.? (ethyl acetate / petroleum ether = 2/1); mp. 258–260 ℃; $[\alpha]_D^{25}$ +11.7 (*c* 1.67, DMSO); ^{1}H NMR (400 MHz, $CDCl_3$) δ: 0.69 (2d, *J* = 7.76 Hz, *J* = 7.52 Hz, 6H), 1.81-1.88 (m, 1H), 2.23 (s, 3H), 3.07 (dd, *J* = 13.68 Hz, *J* = 7.64 Hz, 1H), 3.15 (dd, *J* = 13.90 Hz, *J* = 7.26 Hz, 1H), 3.39 (dd, *J* = 7.14 Hz, *J* = 6.64 Hz, 1H), 4.63-4.69 (m, 1H), 5.42 (d, *J* = 8.16 Hz, 1H), 6.44 (d, *J* = 7.16 Hz, 1H), 7.09-7.12 (m, 1H), 7.16 (d, *J* = 7.88 Hz, 2H), 7.23 (d, *J* = 7.32 Hz,, 2H), 7.29-7.31 (m, 5H), 7.39 (d, *J* = 7.96 Hz, 2H), 7.69 (d, *J* = 7.88 Hz, 2H), 7.77 (br, 1H); ^{13}C NMR (100 MHz, DMSO-d_6) δ: 18.9, 20.3, 22.0, 32.1,

39.5, 55.7, 62.9, 120.6, 124.8, 127.6, 127.7, 129.3, 130.1, 130.4, 130.5, 138.7, 139.8, 140.2, 143.3, 171.1, 171.2; HRMS (ESI^+-TOF) *m/z*: calcd for $C_{27}H_{32}N_3O_4S$ $(M+H)^+$: 494.2108, found: 494.2103; IR (KBr): 3298, 3255, 1674, 1645, 1537, 1323, 1155, 1090, 758, 696 cm^{-1}.

(*R*)-3-methyl-2-(4-methylphenylsulfonamido)-*N*-((*S*)-1-oxo-3-phenyl-1-(phenylamino)propan-2-yl)butanamide ((*R*,*S*)-G**19**): 58% yield; R_f = 0.3 (ethyl acetate / petroleum ether = 2/1); mp. 256–258 ℃; $[\alpha]_D^{25}$ -11.9 (*c* 1.67, DMSO); ^1H NMR (400 MHz, $CDCl_3$) δ: 0.69 (2d, *J* = 7.80 Hz, *J* = 7.20 Hz, 6H), 1.82-1.90 (m, 1H), 2.23 (s, 3H), 3.07 (dd, *J* = 14.0 Hz, *J* = 7.34 Hz, 1H), 3.15 (dd, *J* = 13.7 Hz, *J* = 7.14 Hz, 1H), 3.39 (dd, *J* = 7.00 Hz, *J* = 7.16 Hz, 1H), 4.63-4.69 (m, 1H), 5.40 (br, 1H), 6.42 (br, 1H), 7.11 (m, 1H), 7.16 (d, *J* = 7.88 Hz, 2H), 7.23 (d, *J* = 7.36 Hz, 2H), 7.29-7.31 (m, 5H), 7.39 (d, *J* = 7.64 Hz, 2H), 7.69 (d, *J* = 8.00 Hz, 2H), 7.76 (br, 1H); ^{13}C NMR (100 MHz, DMSO-d_6) δ: 18.9, 20.3, 22.0, 32.1, 39.5, 55.6, 62.9, 120.5, 124.8, 127.6, 127.7, 129.3, 130.1, 130.4, 130.5, 138.7, 139.8, 140.2, 143.3, 171.0, 171.2; HRMS (ESI^+-TOF) *m/z*: calcd for $C_{27}H_{32}N_3O_4S$ $(M+H)^+$: 494.2108, found: 494.2103; IR (KBr): 3296, 3254, 2963, 1672, 1645, 1533, 1449, 1323, 1157, 1090, 756, 696 cm^{-1}.

(*S*)-2-(4-methylphenylsulfonamido)-*N*-((*R*)-2-oxo-1-phenyl-2-(phenylamino)ethyl)propanamide ((*S*,*R*)-G**20**): 50% yield; R_f = 0.3 (ethyl acetate / petroleum ether = 1/1); mp. 227–229 ℃; $[\alpha]_D^{25}$ -50.4 (*c* 1.67, THF); ^1H NMR (400 MHz, $CDCl_3$) δ: 1.24 (d, *J* = 7.52 Hz, 3H), 2.34 (s, 3H), 3.89-3.96 (m, 1H), 5.49 (d, *J* = 7.04 Hz, 1H), 5.70 (d, *J* = 7.96 Hz, 1H), 7.09-7.11 (m, 1H), 7.22-7.29 (m, 5H), 7.34-7.35 (m, 5H), 7.43 (d, *J* = 7.80 Hz, 2H), 7.51 (d, *J* = 6.88 Hz, 1H), 7.73 (d, *J* = 8.00 Hz, 2H), 7.82 (s, 2H); ^{13}C NMR (100 MHz, DMSO-d_6) δ: 20.4, 22.2, 52.9, 57.4, 120.5, 125.1, 127.9, 128.1, 129.1, 129.8, 130.2, 130.7, 139.5, 139.7, 140.0, 143.8, 169.6, 172.2; HRMS (ESI^+-TOF) *m/z*: calcd for $C_{24}H_{26}N_3O_4S$ $(M+H)^+$: 452.1638, found: 452.1640; IR (KBr): 3302, 3262, 1645, 1539, 1341, 1163, 1091, 758, 696 cm^{-1}.

(*R*)-2-(4-methylphenylsulfonamido)-*N*-((*S*)-2-oxo-1-phenyl-2-(phenylamino)ethyl)propanamide ((*R*,*S*)-G**20**): 51% yield; R_f = 0.3 (ethyl acetate / petroleum ether = 1/1); mp. 226–228 ℃; $[\alpha]_D^{25}$ +50.9 (*c* 1.67, THF); ^1H NMR (400 MHz, $CDCl_3$) δ: 1.24 (d, *J* = 7.32 Hz, 3H), 2.33 (s, 3H), 3.90-3.97 (m, 1H), 5.51 (d, *J* = 7.00 Hz, 1H), 5.70 (d, *J* = 7.60 Hz, 1H), 7.07-7.11 (m, 1H), 7.22-7.28 (m, 5H), 7.33-7.34 (m, 5H), 7.43 (d, *J* = 7.92 Hz, 2H), 7.52 (d, *J* = 6.68 Hz, 1H), 7.73 (d, *J* = 8.00 Hz, 2H), 7.85 (s, 1H); ^{13}C NMR (100 MHz, DMSO-d_6) δ: 20.4, 22.2, 53.0, 57.5, 120.5, 125.1, 127.9, 128.1, 129.1, 129.8, 130.2, 130.7, 139.5, 139.7, 140.0, 143.8, 169.6, 172.2; HRMS (ESI^+-TOF) *m/z*: calcd for $C_{24}H_{26}N_3O_4S$ $(M+H)^+$: 452.1638, found: 452.1645; IR (KBr): 3310, 3254, 1647, 1541, 1443, 1337, 1163, 1092, 754, 696 cm^{-1}.

(*S*)-*N*-((*R*)-2-((4-methoxyphenyl)amino)-2-oxo-1-phenylethyl)-2-(4-methylphenylsulfonamido)propanamide ((*S*,*R*)-G**21**): 59% yield; R_f = 0.3 (ethyl acetate / petroleum ether = 1/1); mp. 213–215 ℃; $[\alpha]_D^{25}$ -8.5 (*c* 1.67, DMSO); ^1H NMR (400 MHz, $CDCl_3$) δ: 1.24 (d, *J* = 7.00 Hz, 3H), 2.34 (s, 3H), 3.76 (s, 3H), 3.91-3.94 (m, 1H), 5.49 (d, *J* = 6.48 Hz, 1H), 5.72-5.73 (m, 1H), 6.79 (d, *J* = 8.48 Hz, 2H), 7.22-7.24 (m, 3H), 7.33 (br, 6H), 7.53-7.54 (m, 1H), 7.71-7.77 (m, 3H); ^{13}C NMR (100 MHz, DMSO-d_6) δ: 20.3, 22.2, 52.9, 56.6, 57.4, 115.4, 121.9, 127.9, 128.0, 129.0, 129.8, 130.7, 133.2, 139.5, 139.9, 143.8, 156.9, 169.0, 172.1; HRMS (ESI^+-TOF) *m/z*: calcd for $C_{25}H_{28}N_3O_5S$ $(M+H)^+$: 482.1744, found: 482.1748; IR (KBr): 3423, 3271, 1645, 1543, 1516, 1248, 1163, 1092, 700 cm^{-1}.

(*R*)-*N*-((*S*)-2-((4-methoxyphenyl)amino)-2-oxo-1-phenylethyl)-2-(4-methylphenylsulfonamido)propanamide ((*R*,*S*)-G**21**): 62% yield; R_f = 0.3 (ethyl acetate / petroleum ether = 1/1); mp. 203–205 ℃; $[\alpha]_D^{25}$ +8.4 (*c* 1.67, DMSO); ^1H NMR (400 MHz, $CDCl_3$) δ: 1.24 (d, *J* = 7.00 Hz, 3H), 2.34 (s, 3H), 3.76 (s, 3H), 3.90-3.94 (m, 1H), 5.48 (d, *J* = 6.96 Hz, 1H), 5.67 (d, *J* = 7.40 Hz, 1H), 6.80 (d, *J* = 8.80 Hz, 2H), 7.22-7.33 (m, 10H), 7.50 (d, *J* = 6.72 Hz, 1H), 7.70-7.74 (m, 3H); ^{13}C NMR (100 MHz, DMSO-d_6) δ: 20.4, 22.2, 53.0, 57.5, 120.5, 125.1, 127.9, 128.1, 129.1, 129.8, 130.2, 130.7, 139.5, 139.7, 140.0, 143.8, 169.6, 172.2; HRMS (ESI^+-TOF) *m/z*: calcd for $C_{25}H_{28}N_3O_5S$ $(M+H)^+$: 482.1744, found: 482.1747; IR (KBr): 3298, 3269, 1645,

1601, 1543, 1337, 1242, 1163, 833, 700 cm^{-1}.

(*R*)-2-((*S*)-2-(4-methylphenylsulfonamido)propanamido)-*N*,3-diphenylpropanamide ((*S*,*R*)-G**22**): 55% yield; R_f = 0.25 (ethyl acetate / petroleum ether = 1/1); mp. 199–201 ℃; $[\alpha]_D^{25}$ -5.0 (*c* 1.67, THF); ^{1}H NMR (400 MHz, $CDCl_3$) δ: 1.10 (d, *J* = 6.88 Hz, 3H), 2.30 (s, 3H), 3.05 (dd, *J* = 13.70 Hz, *J* = 7.38 Hz, 1H), 3.14 (dd, *J* = 13.33 Hz, *J* = 7.22 Hz, 1H), 3,80-3.83 (m, 1H), 4.66-4.71 (m, 1H), 5.85 (d, *J* = 7.56 Hz, 1H), 7.04-7.10 (m, 2H), 7.20-7.25 (m, 9H), 7.37 (d, *J* = 7.84 Hz, 2H), 7.71 (d, *J* = 7.72 Hz, 2H), 7.98 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 19.0, 21.4, 38.1, 52.7, 55.5, 120.2, 124.6, 127.1, 128.7, 128.9, 129.3, 129.8, 136.4, 136.8, 137.3, 143.9, 169.0, 172.2; HRMS (ESI^+-TOF) *m*/*z*: calcd for $C_{25}H_{28}N_3O_4S$ $(M+H)^+$: 466.1795, found: 466.1794; IR (KBr): 3310, 3242, 1651, 1537, 1443, 1333, 1163, 1090, 752, 700 cm^{-1}.

(*S*)-2-((*R*)-2-(4-methylphenylsulfonamido)propanamido)-*N*,3-diphenylpropanamide ((*R*,*S*)-G**22**): 60% yield; R_f = 0.25 (ethyl acetate / petroleum ether = 1/1); mp. 198–200 ℃; $[\alpha]_D^{25}$ +5.9 (*c* 1.67, THF); ^{1}H NMR (400 MHz, $CDCl_3$) δ: 1.07 (d, *J* = 6.92 Hz, 3H), 2.27 (s, 3H), 3.01 (dd, *J* = 13.58 Hz, *J* = 7.62 Hz, 1H), 3.15 (dd, *J* = 13.62 Hz, *J* = 7.02 Hz, 1H), 3.84-3.88 (m, 1H), 4.70-4.76 (m, 1H), 6.20 (d, *J* = 7.92 Hz, 1H), 7.05-7.09 (m, 1H), 7.17-7.28 (m, 10H), 7.37 (d, *J* = 7.84 Hz, 2H), 7.70 (d, *J* = 7.96 Hz, 2H), 8.21 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 19.0, 21.4, 38.1, 52.7, 55.5, 120.2, 124.6, 127.1, 128.7, 128.9, 129.3, 129.8, 136.4, 136.8, 137.3, 143.9, 169.0, 172.3; HRMS (ESI^+-TOF) *m*/*z*: calcd for $C_{25}H_{28}N_3O_4S$ $(M+H)^+$: 466.1795, found: 466.1804; IR (KBr): 3300, 3273, 1649, 1543, 1441, 1319, 1159, 1084, 748, 698 cm^{-1}.

(*R*)-*N*-(4-methoxyphenyl)-2-((*S*)-2-(4-methylphenylsulfonamido)propanamido)-3-phenylpropan-amide ((*S*,*R*)-G**23**): 57% yield; R_f = 0.25 (ethyl acetate / petroleum ether = 1/1); mp. 217–219 ℃; $[\alpha]_D^{25}$ -14.3 (*c* 1.67, DMSO); ^{1}H NMR (400 MHz, $CDCl_3$) δ: 1.15 (d, *J* = 7.04 Hz, 3H), 2.33 (s, 3H), 3.10 (d, *J* = 7.24 Hz, 2H), 3.77 (s, 3H), 4.58-4.63 (m, 1H), 5.37 (d, *J* = 7.40 Hz, 1H), 6.78-6.82 (m, 3H), 7.21-7.25 (m, 5H), 7.27-7.32 (m, 4H), 7.55 (s, 1H), 7.72 (d, *J* = 8.12 Hz, 2H); ^{13}C NMR (100 MHz, DMSO-d_6) δ: 20.8, 22.7, 40.2, 53.5, 55.8, 57.1, 115.8, 122.6, 128.2, 128.3, 129.8, 131.1, 133.8, 139.2, 140.2, 144.2, 157.3, 171.0, 172.7; HRMS (ESI^+-TOF) *m*/*z*: calcd for $C_{26}H_{30}N_3O_5S$ $(M+H)^+$: 496.1900, found: 496.1898; IR (KBr): 3453, 3269, 1649, 1599, 1514, 1236, 1161, 1094, 976, 818, 702 cm^{-1}.

(*S*)-*N*-(4-methoxyphenyl)-2-((*R*)-2-(4-methylphenylsulfon-amido)propanamido)-3-phenylpropanamide ((*R*,*S*)-G**23**): 60% yield; R_f = 0.25 (ethyl acetate / petroleum ether = 1/1); mp. 215–217 ℃; $[\alpha]_D^{25}$ +14.1 (*c* 1.67, DMSO); ^{1}H NMR (400 MHz, $CDCl_3$) δ: 1.14 (d, *J* = 7.00 Hz, 3H), 2.33 (s, 3H), 3.05-3.14 (m, 2H), 3.77 (s, 3H), 4.60-4.65 (m, 1H), 5.51 (d, *J* = 7.32 Hz, 1H), 6.80 (d, *J* = 8.92 Hz, 2H), 6.86 (d, *J* = 8.00 Hz, 1H), 7.21-7.25 (m, 5H), 7.27-7.30 (m, 4H), 7.63 (s, 1H), 7.71 (d, *J* = 8.12 Hz, 2H); ^{13}C NMR (100 MHz, DMSO-d_6) δ: 20.3, 22.1, 39.6, 53.0, 55.3, 56.5, 115.3, 122.1, 127.7, 127.8, 129.3, 130.6, 133.3, 138.7, 139.7, 143.6, 156.7, 170.5, 172.1; HRMS (ESI^+-TOF) *m*/*z*: calcd for $C_{26}H_{30}N_3O_5S$ $(M+H)^+$: 496.1900, found: 496.1897; IR (KBr): 3258, 2924, 1649, 1537, 1516, 1333, 1240, 1163, 1092, 822, 702 cm^{-1}.

(*S*)-2-(4-methylphenylsulfonamido)-*N*-((*R*)-2-oxo-1-phenyl-2-(phenylamino)ethyl)-3-phenylpropanamide ((*S*,*R*)-G**24**): 61% yield; R_f = 0.25 (ethyl acetate / petroleum ether = 2/1); mp. 241–243 ℃; $[\alpha]_D^{25}$ -11.4 (*c* 1.67, THF); ^{1}H NMR (400 MHz, $CDCl_3$) δ: 2.31 (s, 3H), 2.91-3.00 (m, 2H), 3.97-4.02 (m, 1H), 5.44 (d, *J* = 7.08 Hz, 1H), 5.46 (d, *J* = 8.16 Hz, 1H), 6.88 (d, *J* = 7.32 Hz, 2H), 7.05-7.10 (m, 2H), 7.12-7.15 (m, 3H), 7.24-7.28 (m, 6H), 7.30-7.33 (m, 3H), 7.44 (d, *J* = 8.04 Hz, 2H), 7.56 (d, *J* = 8.12 Hz, 2H), 7.70 (s, 1H); ^{13}C NMR (100 MHz, DMSO-d_6) δ: 22.1, 39.9, 57.4, 58.5, 120.4, 125.0, 127.6, 127.7, 128.0, 128.9, 129.2, 129.8, 130.3, 130.4, 130.7, 138.3, 139.6, 140.1, 143.4, 169.6, 171.3; HRMS (ESI^+-TOF) *m*/*z*: calcd for $C_{30}H_{30}N_3O_4S$ $(M+H)^+$: 528.1951, found: 528.1950; IR (KBr): 3314, 3250, 1672, 1641, 1539, 1447, 1333, 1161, 1089, 746, 696 cm^{-1}.

(*R*)-2-(4-methylphenylsulfonamido)-*N*-((*S*)-2-oxo-1-phenyl-2-(phenylamino)ethyl)-3-phenylpropanamide ((*R*,*S*)-G**24**): 58% yield; R_f = 0.25 (ethyl acetate / petroleum ether = 2/1); mp. 241–243 ℃; $[\alpha]_D^{25}$ +11.3 (*c*

1.67, THF); ^{1}H NMR (400 MHz, $CDCl_3$) δ: 2.30 (s, 3H), 2.95 (d, J = 6.92 Hz, 2H), 3.98-4.03 (m, 1H), 5.45 (d, J = 7.00 Hz, 1H), 5.50 (d, J = 7.52 Hz, 1H), 6.88 (d, J = 7.24 Hz, 2H), 7.07-7.15 (m, 6H), 7.23-7.26 (m, 3H), 7.27-7.32 (m, 5H), 7.44 (d, J = 7.88 Hz, 2H), 7.56 (d, J = 8.20 Hz, 2H), 7.73 (s, 1H); ^{13}C NMR (100 MHz, DMSO-d_6) δ: 22.1, 39.9, 57.4, 58.5, 120.4, 125.0, 127.6, 127.7, 128.0, 128.9, 129.2, 129.8, 130.2, 130.4, 130.7, 138.3, 139.6, 140.1, 143.4, 169.6, 171.3; HRMS (ESI$^+$-TOF) m/z: calcd for $C_{30}H_{30}N_3O_4S$ $(M+H)^+$: 528.1951, found: 528.1957; IR (KBr): 3306, 3252, 3032, 1670, 1643, 1537, 1447, 1333, 1161, 1088, 696 cm^{-1}.

(*S*)-*N*-((*R*)-2-oxo-1-phenyl-2-(phenylamino)ethyl)-1-tosylpyrrolidine-2-carboxamide ((*S*,*R*)-G**25**): 68% yield; R_f = 0.25 (ethyl acetate / petroleum ether = 1/2); mp. 216–218 ℃; $[\alpha]_D^{25}$ -142.4 (*c* 1.67, THF); ^{1}H NMR (400 MHz, $CDCl_3$) δ: 1.66-1.73 (m, 2H), 1.88 (br, 1H), 2.14 (br, 1H), 2.43 (s, 3H), 3.23-3.25 (m, 1H), 3,61 (br, 1H), 4.18 (d, J = 6.76 Hz, 1H), 5.76 (d, J = 6.96 Hz, 1H), 7.06-7.08 (m, 1H), 7.25-7.26 (m, 2H), 7.30-7.37 (m, 5H), 7.49 (br, 4H), 7.73 (d, J = 7.40 Hz, 2H), 7.96 (d, J = 6.44 Hz, 1H), 8.38 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 21.6, 24.6, 30.4, 49.8, 58.1, 62.4, 119.9, 124.5, 127.4, 127.9, 128.6, 128.9, 129.2, 130.0, 133.1, 136.5, 137.6, 144.4, 167.9, 171.3; HRMS (ESI$^+$-TOF) m/z: calcd for $C_{26}H_{28}N_3O_4S$ $(M+H)^+$: 478.1795, found: 478.1793; IR (KBr): 3300, 3273, 1694, 1657, 1601, 1530, 1356, 1161, 1098, 758, 696 cm^{-1}.

(*R*)-*N*-((*S*)-2-oxo-1-phenyl-2-(phenylamino)ethyl)-1-tosylpyrrolidine-2-carboxamide ((*R*,*S*)-G**25**): 59% yield; R_f = 0.25 (ethyl acetate / petroleum ether = 1/2); mp. 215–217 ℃; $[\alpha]_D^{25}$ +142.7 (*c* 1.67, THF); ^{1}H NMR (400 MHz, $CDCl_3$) δ: 1.66-1.70 (m, 2H), 1.86-1.90 (m, 1H), 2.14-2.17 (m, 1H), 2.43 (s, 3H), 3.23-3.27 (m, 1H), 3.60-3.62 (m, 1H), 4.18 (d, J = 6.32 Hz, 1H), 5.76 (d, J = 7.12 Hz, 1H), 7.04-7.08 (m, 1H), 7.23-7.26 (m, 2H), 7.30-7.37 (m, 5H), 7.50 (br, 4H), 7.73 (d, J = 7.52 Hz, 2H), 7.97 (d, J = 6.68 Hz, 1H), 8.40 (s, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 21.6, 24.6, 30.4, 49.8, 58.1, 62.4, 119.9, 124.4, 127.4, 127.9, 128.6, 128.9, 129.2, 130.0, 133.1, 136.6, 137.6, 144.4, 167.9, 171.3; HRMS (ESI$^+$-TOF) m/z: calcd for $C_{26}H_{28}N_3O_4S$ $(M+H)^+$: 478.1795, found: 478.1798; IR (KBr): 3300, 1692, 1655, 1601, 1530, 1445, 1356, 1161, 1099, 758, 692 cm^{-1}.

(*S*)-*N*-((*R*)-2-((4-methoxyphenyl)amino)-2-oxo-1-phenylethyl)-1-tosylpyrrolidine-2-carboxamide ((*S*,*R*)-G**26**): 63% yield; R_f = 0.25 (ethyl acetate / petroleum ether = 2/1); mp. 171–173 ℃; $[\alpha]_D^{25}$ -122.1 (*c* 1.67, THF); ^{1}H NMR (400 MHz, $CDCl_3$) δ: 1.61-1.71 (m, 2H), 1.82-1.94 (m, 1H), 2.12-2.16 (m, 1H), 2.42 (s, 3H), 3.20-3.28 (m, 1H), 3.58-3.63 (m, 1H), 3.75 (s, 3H), 4.15-4.18 (m, 1H), 5.72 (d, J = 7.52 Hz, 1H), 6.78 (d, J = 8.96 Hz, 2H), 7.29-7.39 (m, 7H), 7.49 (d, J = 6.92 Hz, 2H), 7.73 (d, J = 8.16 Hz, 2H), 7.95 (d, J = 7.52 Hz, 1H), 8.27 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 21.6, 24.6, 30.4, 49.8, 55.4, 58.0, 62.4, 114.0, 121.7, 127.4, 127.9 128.6, 129.2, 129.4, 130.7, 133.2, 136.8, 144.3, 156.5, 167.7, 171.3; HRMS (ESI$^+$-TOF) m/z: calcd for $C_{27}H_{30}N_3O_5S$ $(M+H)^+$: 508.1900, found: 508.1904; IR (KBr): 3310, 1653, 1609, 1514, 1338, 1159, 1028, 839, 694 cm^{-1}.

(*R*)-*N*-((*S*)-2-((4-methoxyphenyl)amino)-2-oxo-1-phenylethyl)-1-tosylpyrrolidine-2-carboxamide ((*R*,*S*)-G**26**): 65% yield; R_f = 0.25 (ethyl acetate / petroleum ether = 2/1); mp. 171–173 ℃; $[\alpha]_D^{25}$ +121.8 (*c* 1.67, THF); ^{1}H NMR (400 MHz, $CDCl_3$) δ: 1.62-1.71 (m, 2H), 1.83-1.89 (m, 1H), 2.12-2.16 (m, 1H), 2.43 (s, 3H), 3.20-3.26 (m, 1H), 3.58-3.63 (m, 1H), 3.75 (s, 3H), 4.16 (d, J = 6.00 Hz, 1H), 5.71 (d, J = 7.44 Hz, 1H), 6.78 (d, J = 8.88 Hz, 2H), 7.29-7.39 (m, 7H), 7.49 (d, J = 7.00 Hz, 2H), 7.72 (d, J = 8.04 Hz, 1H), 8.21 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 21.6, 24.6, 30.4, 49.8, 55.4, 58.0, 62.4, 114.0, 121.7, 127.4, 127.9, 128.6, 129.2, 130.0, 130.7, 133.2, 136.7, 144.3, 156.5, 167.7, 171.3; HRMS (ESI$^+$-TOF) m/z: calcd for $C_{27}H_{30}N_3O_5S$ $(M+H)^+$: 508.1900, found: 508.1898; IR (KBr): 3308, 1686, 1653, 1609,1514, 1339, 1250, 1159, 1028, 823, 698 cm^{-1}.

(*S*)-*N*-((*R*)-1-oxo-3-phenyl-1-(phenylamino)propan-2-yl)-1-tosylpyrrolidine-2-carboxamide ((*S*,*R*)-G**27**): 65%

yield; R_f = 0.3 (ethyl acetate / petroleum ether = 1/2); mp. 181−183 ℃; $[\alpha]_D^{25}$ -59.4 (*c* 1.67, THF); ^{1}H NMR (400 MHz, $CDCl_3$) *δ*: 1.54-1.58 (m, 1H), 1.62-1.69 (m, 2H), 2.02-2.04 (m, 1H), 2.44 (s, 3H), 3.10-3.16 (m, 1H), 3.31 (d, *J* = 7.24 Hz, 2H), 3.46-3.51 (m, 1H), 4.02-4.04 (m, 1H), 4.62-4.67 (m, 1H), 7.05-7.09 (m, 1H), 7.25-7.29 (m, 3H), 7.30-7.35 (m, 7H), 7.44 (d, *J* = 7.64 Hz, 2H), 7.72 (d, *J* = 8.24 Hz, 2H), 7.99 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) *δ*: 21.6, 24.4, 30.4, 37.0, 49.7, 56.1, 62.4, 120.0, 124.3, 127.0, 127.9, 128.8, 128.9, 129.5, 130.0, 132.7, 136.9, 137.6, 144.6, 168.6, 171.2; HRMS (ESI$^+$-TOF) *m*/*z*: calcd for $C_{27}H_{30}N_3O_4S$ $(M+H)^+$: 492.1951, found: 492.1950; IR (KBr): 3345, 3310, 2924, 2864, 1695, 1653, 1524, 1335, 1155, 1095, 698, 662 cm^{-1}.

(*R*)-*N*-((*S*)-1-oxo-3-phenyl-1-(phenylamino)propan-2-yl)-1-tosylpyrrolidine-2-carboxamide ((*R*,*S*)-G**27**): 57% yield; R_f = 0.3 (ethyl acetate / petroleum ether = 1/2); mp. 180−182 ℃; $[\alpha]_D^{25}$ +59.2 (*c* 1.67, THF); ^{1}H NMR (400 MHz, $CDCl_3$) *δ*: 1.54-1.58 (m, 1H), 1.61-1.69 (m, 2H), 1.98-2.04 (m, 1H), 2.43 (s, 3H), 3.09-3.15 (m, 1H), 3.30 (d, *J* = 7.20 Hz, 2H), 3.47-3.52 (m, 1H), 4.02-4.05 (m, 1H), 4.65-4.71 (m, 1H), 7.04-7.08 (m, 1H), 7.24-7.28 (m, 3H), 7.30-7.36 (m, 7H), 7.44 (d, *J* = 7.68 Hz, 2H), 7.72 (d, *J* = 8.24 Hz, 2H), 8.18 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) *δ*: 21.6, 24.4, 30.4, 37.1, 49.7, 56.0, 62.4, 120.0, 124.3, 127.0, 127.9, 128.8, 128.8, 129.5, 130.0, 132.7, 136.9, 137.7, 144.6, 168.6, 171.8; HRMS (ESI$^+$-TOF) *m*/*z*: calcd for $C_{27}H_{30}N_3O_4S$ $(M+H)^+$: 492.1951, found: 492.1960; IR (KBr): 3343, 3310, 2924, 2864, 1695, 1653, 1518, 1447, 1335, 1155, 1098, 748, 698, 662 cm^{-1}.

NMR and HRMS spectra of chiral bisthiourea derivatives 1–9 as CSAs.

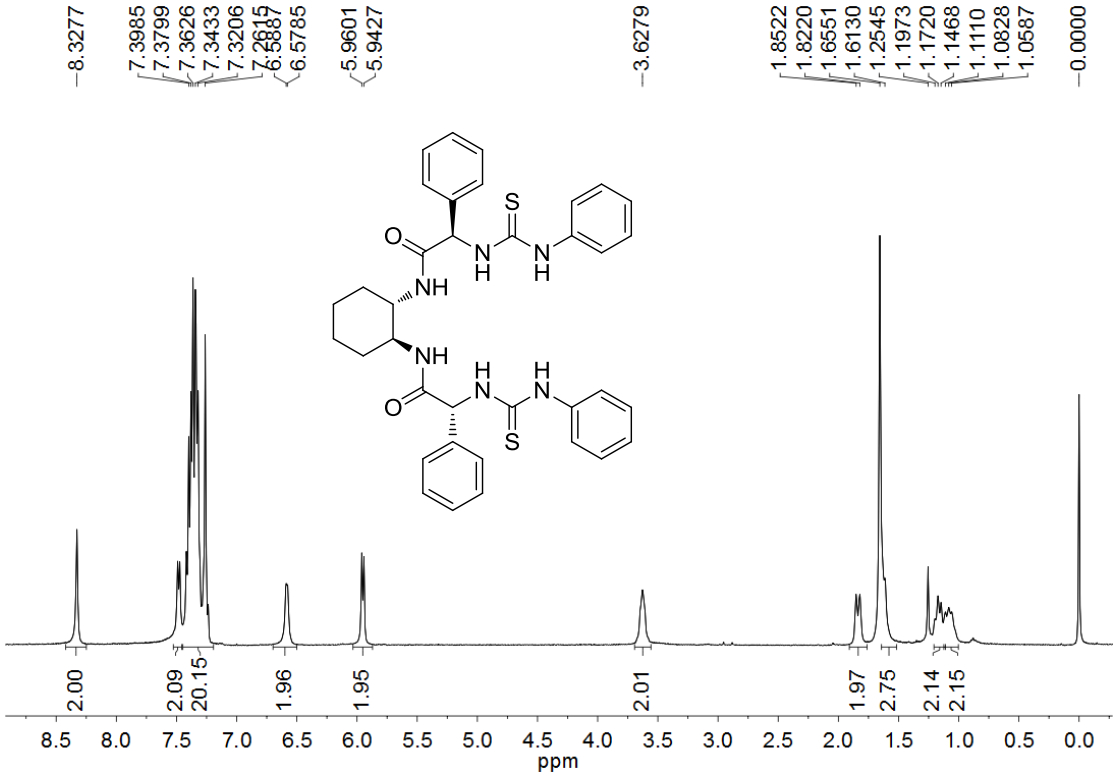


Figure S1. ^{1}H NMR Spectrum of CSA **1** in $CDCl_3$ (400 MHz).

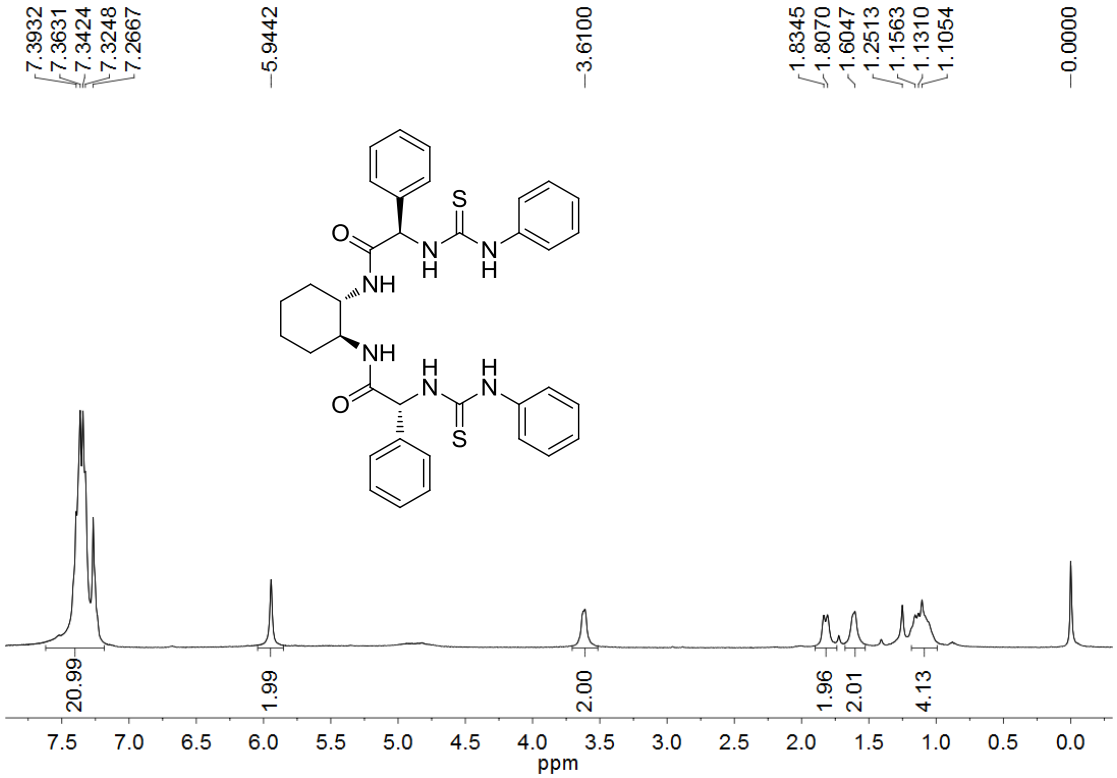


Figure S2. ^{1}H NMR spectrum of CSA **1** in $CDCl_3$ (5% D_2O) (400 MHz).

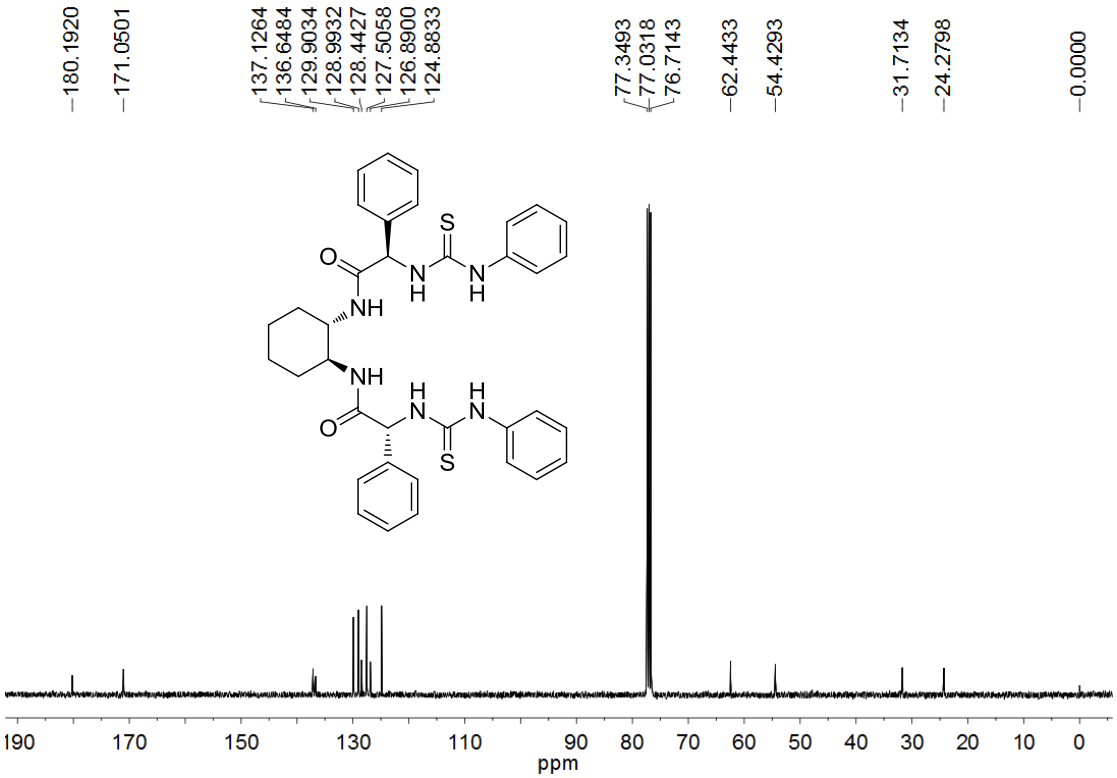


Figure S3. ^{13}C NMR spectrum of CSA **1** in $CDCl_3$ (100 MHz).

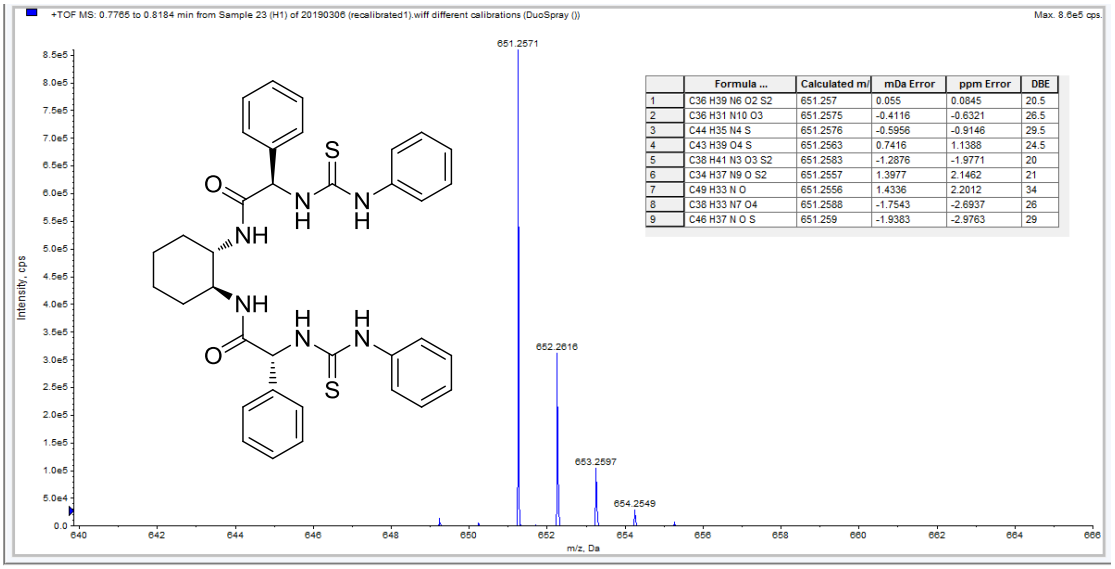


Figure S4. HRMS spectrum of CSA **1**.

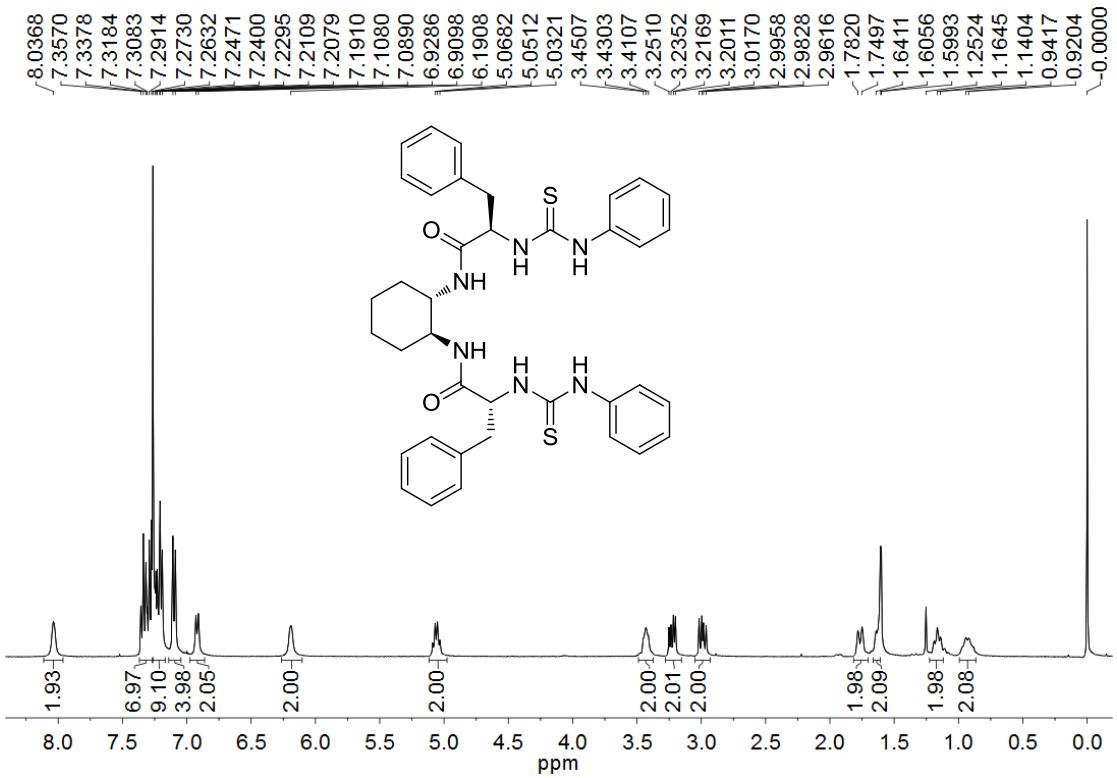


Figure S5. ^{1}H NMR spectrum of CSA **2** in $CDCl_3$ (400 MHz).

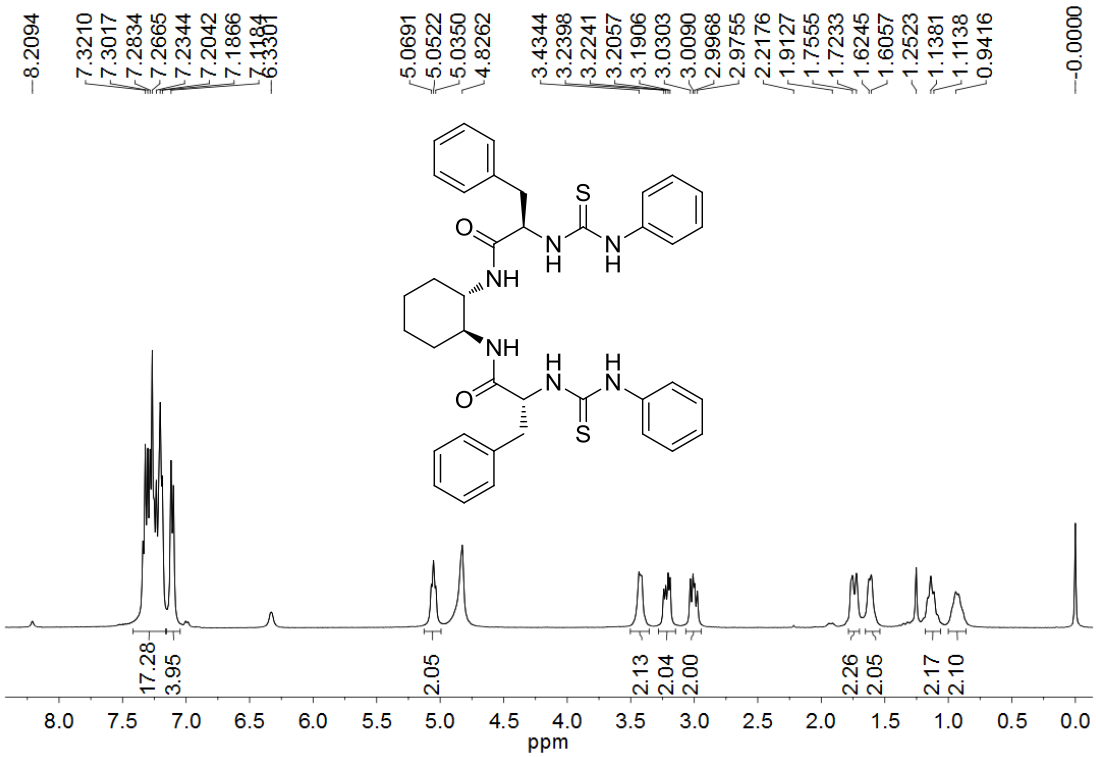


Figure S6. ^{1}H NMR spectrum of CSA **2** in $CDCl_3$ (5% D_2O) (400 MHz).

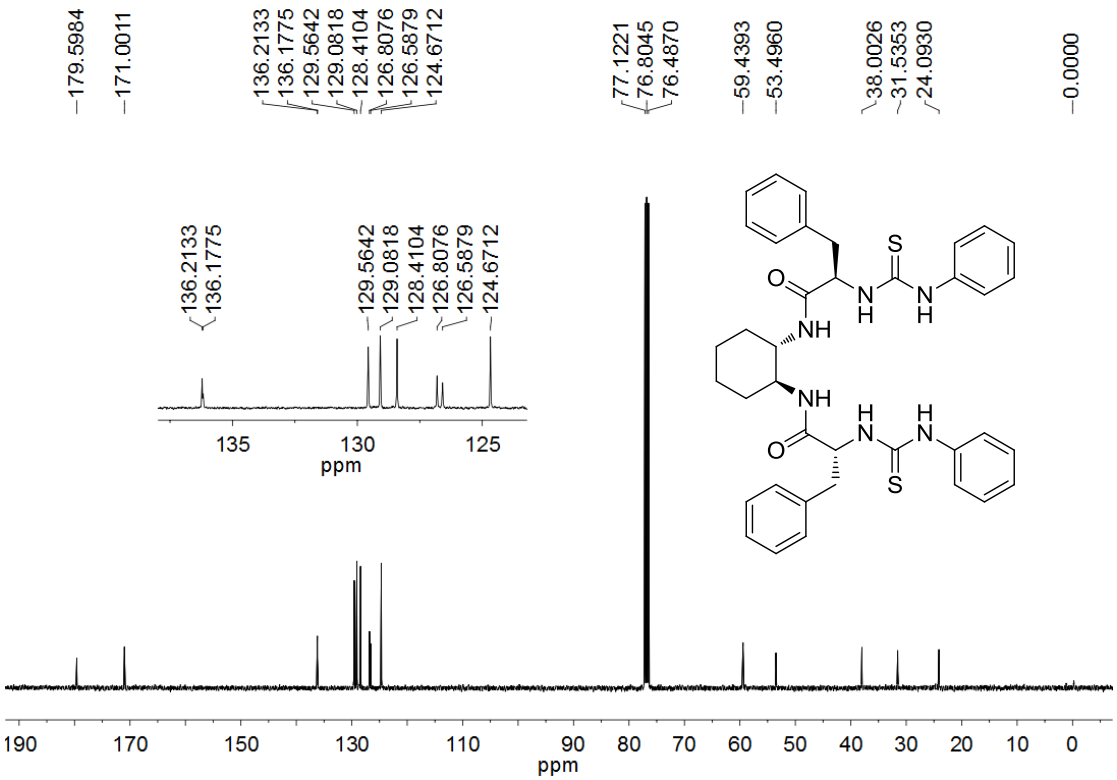


Figure S7. ^{13}C NMR spectrum of CSA **2** in $CDCl_3$ (100 MHz).

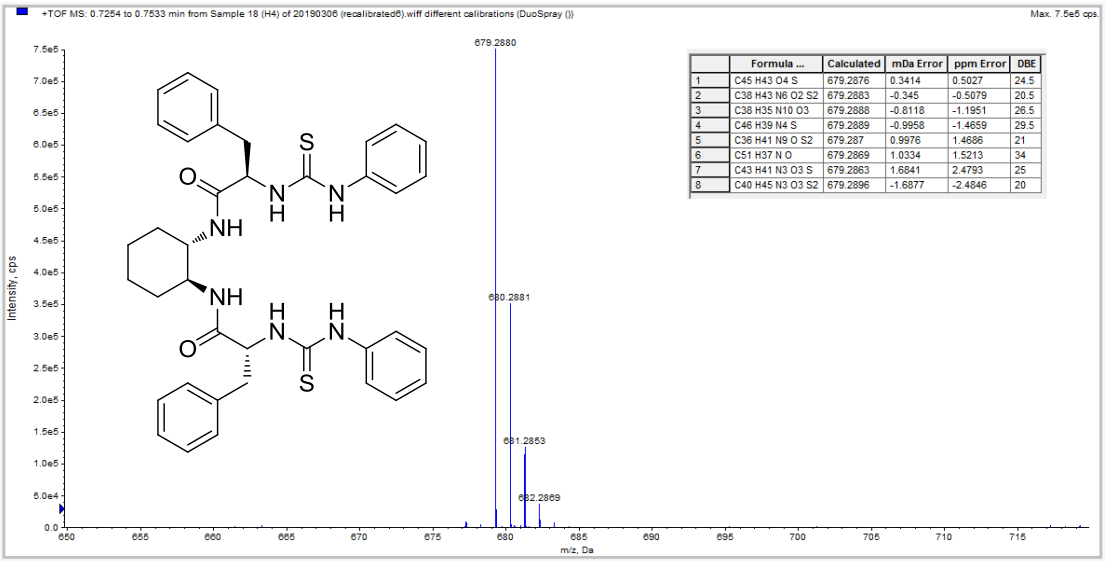


Figure S8. HRMS spectrum of CSA **2**.

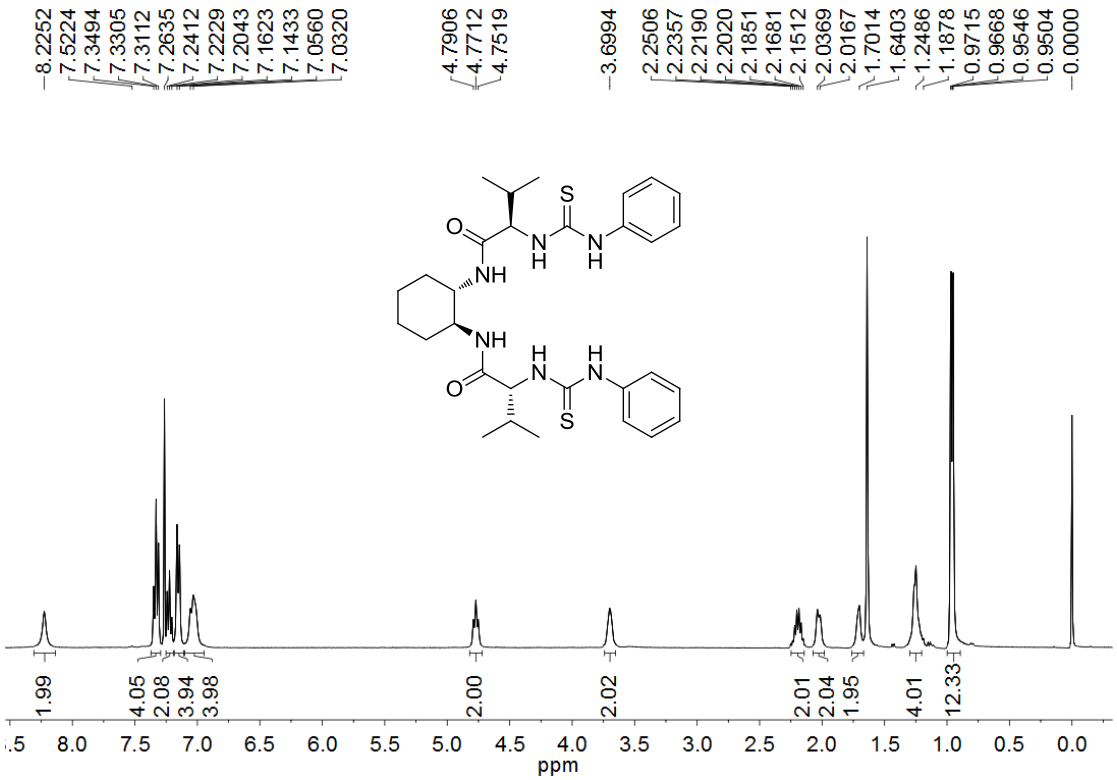


Figure S9. ^{1}H NMR spectrum of CSA **3** in $CDCl_3$ (400 MHz).

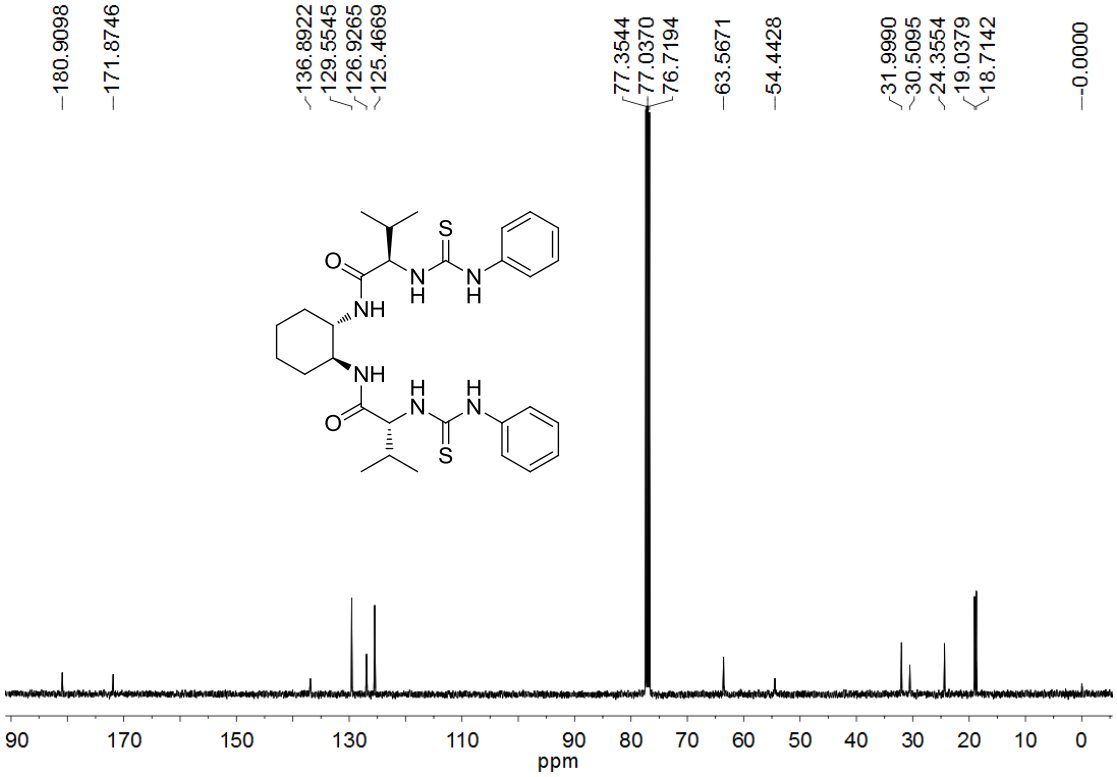


Figure S10. ^{13}C NMR spectrum of CSA **3** in $CDCl_3$ (100 MHz).

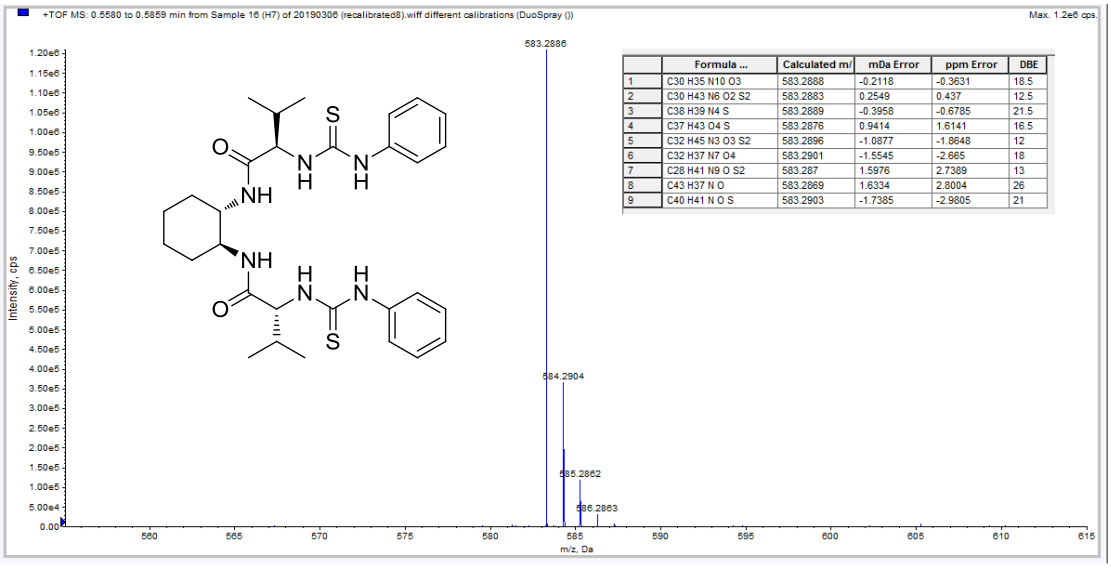


Figure S11. HRMS spectrum of CSA **3**.

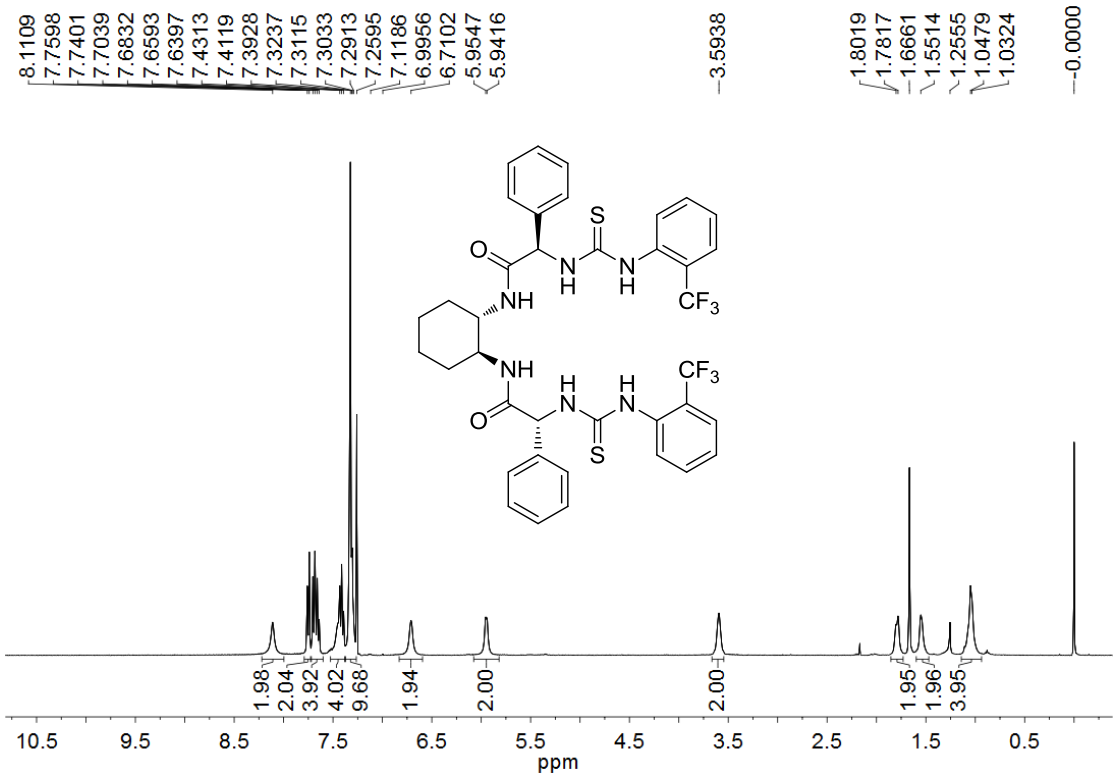


Figure S12. ^{1}H NMR spectrum of CSA **4** in $CDCl_3$ (400 MHz).

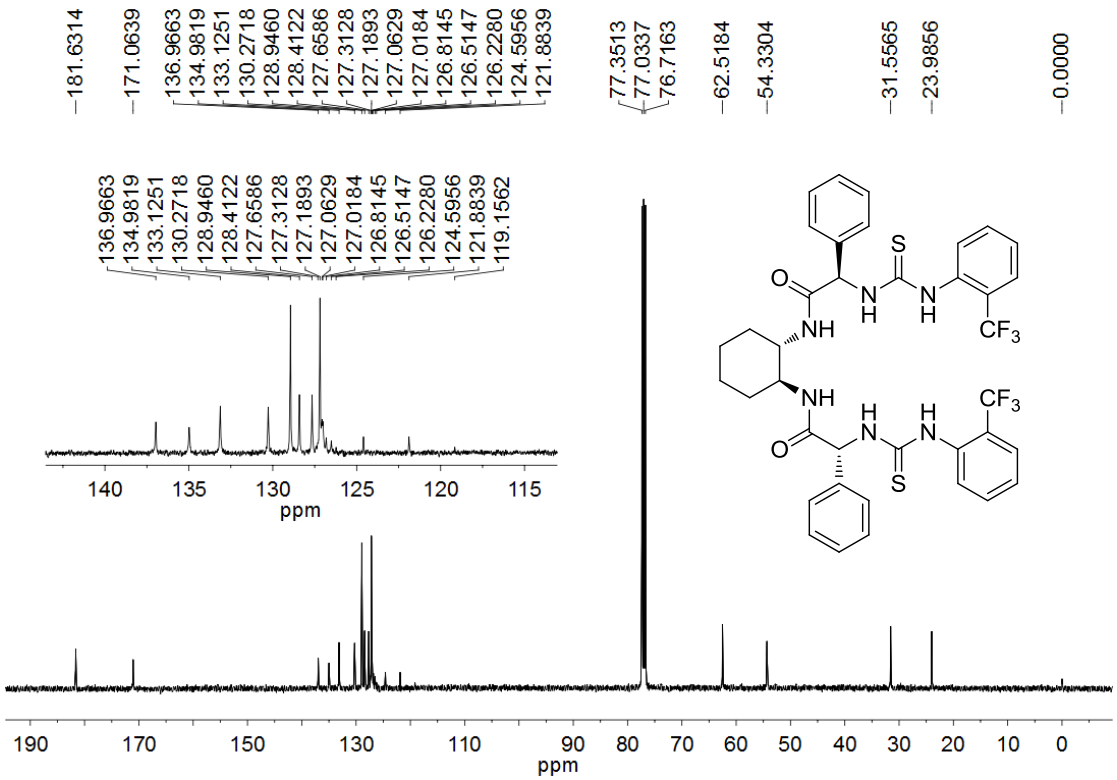
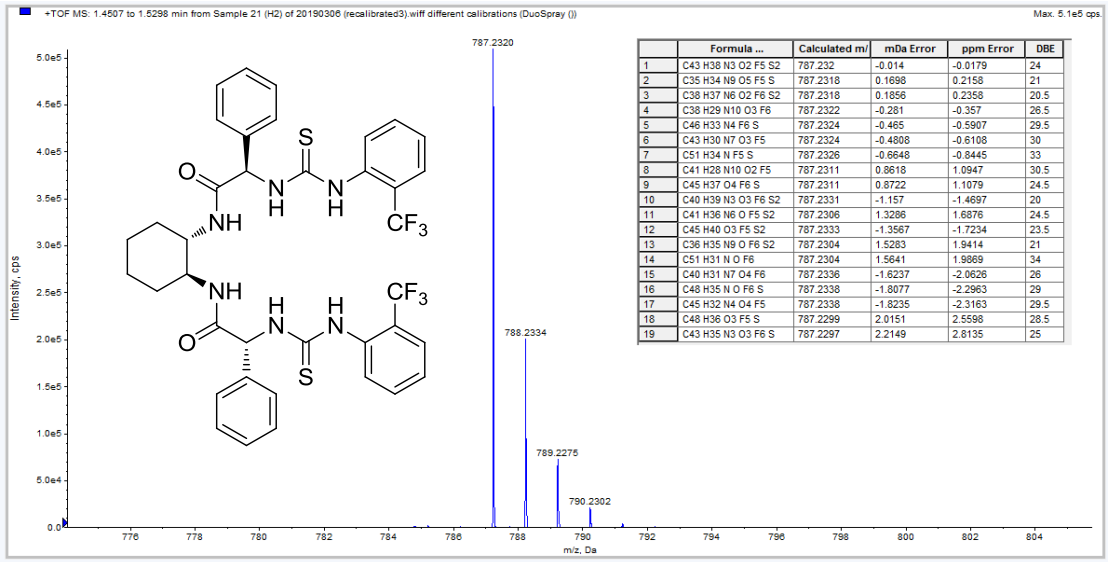


Figure S13. ^{13}C NMR spectrum of CSA **4** in $CDCl_3$ (100 MHz).

Figure S14. ^{19}F NMR spectrum of CSA **4** in $CDCl_3$ (376 MHz).



	Formula ...	Calculated m/	mDa Error	ppm Error	DBE
1	C43 H38 N3 O2 F5 S2	787.232	-0.014	-0.0179	24
2	C35 H34 N9 O5 F5 S	787.2318	0.1698	0.2158	21
3	C38 H37 N6 O2 F6 S2	787.2318	0.1656	0.2358	20.5
4	C38 H29 N10 O3 F6	787.2322	-0.281	-0.357	26.5
5	C46 H33 N4 F6 S	787.2324	-0.465	-0.5907	29.5
6	C43 H30 N7 O3 F5	787.2324	-0.4808	-0.6108	30
7	C51 H34 N F5 S	787.2326	-0.6648	-0.8445	33
8	C41 H28 N10 O2 F5	787.2311	0.8618	1.0947	30.5
9	C45 H37 O4 F6 S	787.2311	0.8722	1.1079	24.5
10	C40 H39 N3 O3 F6 S2	787.2331	-1.157	-1.4697	20
11	C41 H36 N6 O F5 S2	787.2306	1.3286	1.6876	24.5
12	C45 H40 O3 F5 S2	787.2333	-1.3567	-1.7234	23.5
13	C36 H35 N9 O F6 S2	787.2304	1.5283	1.9414	21
14	C51 H31 N O F6	787.2304	1.5641	1.9869	34
15	C40 H31 N7 O4 F6	787.2336	-1.6237	-2.0626	26
16	C48 H35 N O F6 S	787.2338	-1.8077	-2.2963	29
17	C45 H32 N4 O4 F5	787.2338	-1.8235	-2.3163	29.5
18	C48 H36 O3 F5 S	787.2299	2.0151	2.5598	28.5
19	C43 H35 N3 O3 F6 S	787.2297	2.2149	2.8135	25

Figure S15. HRMS spectrum of CSA **4**.

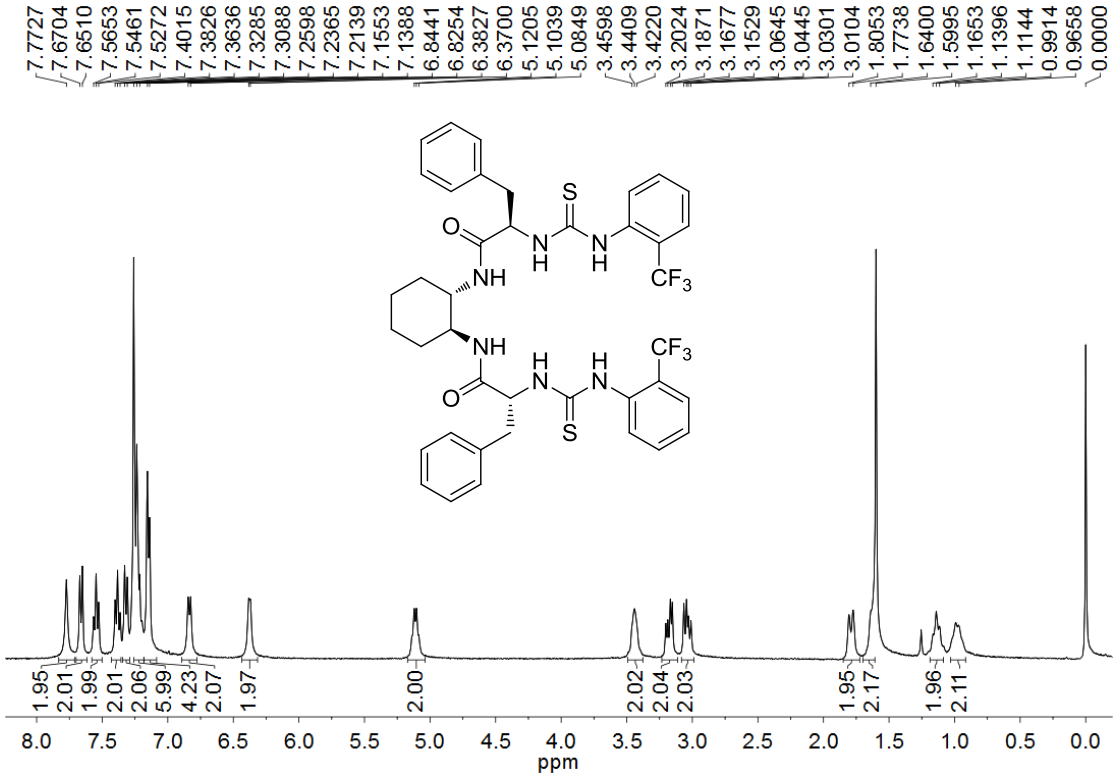


Figure S16. ^{1}H NMR spectrum of CSA **5** in $CDCl_3$ (400 MHz).

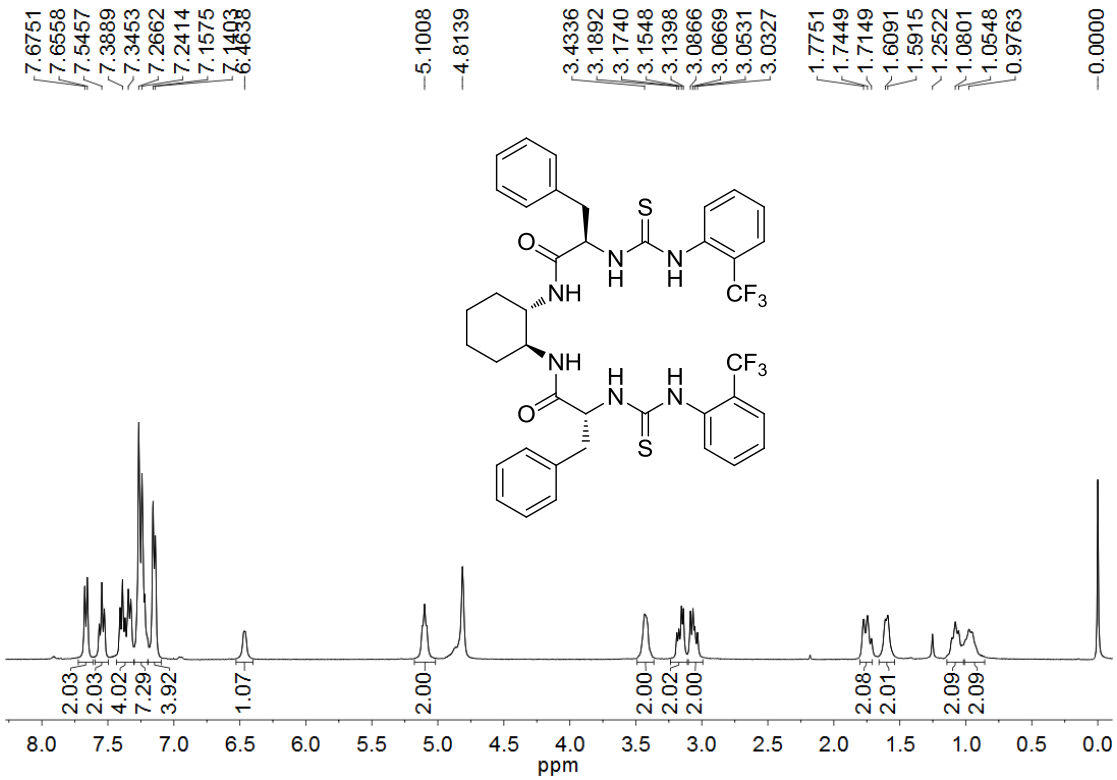


Figure S17. ^{1}H NMR spectrum of CSA **5** in $CDCl_3$ (5% D_2O) (400 MHz).

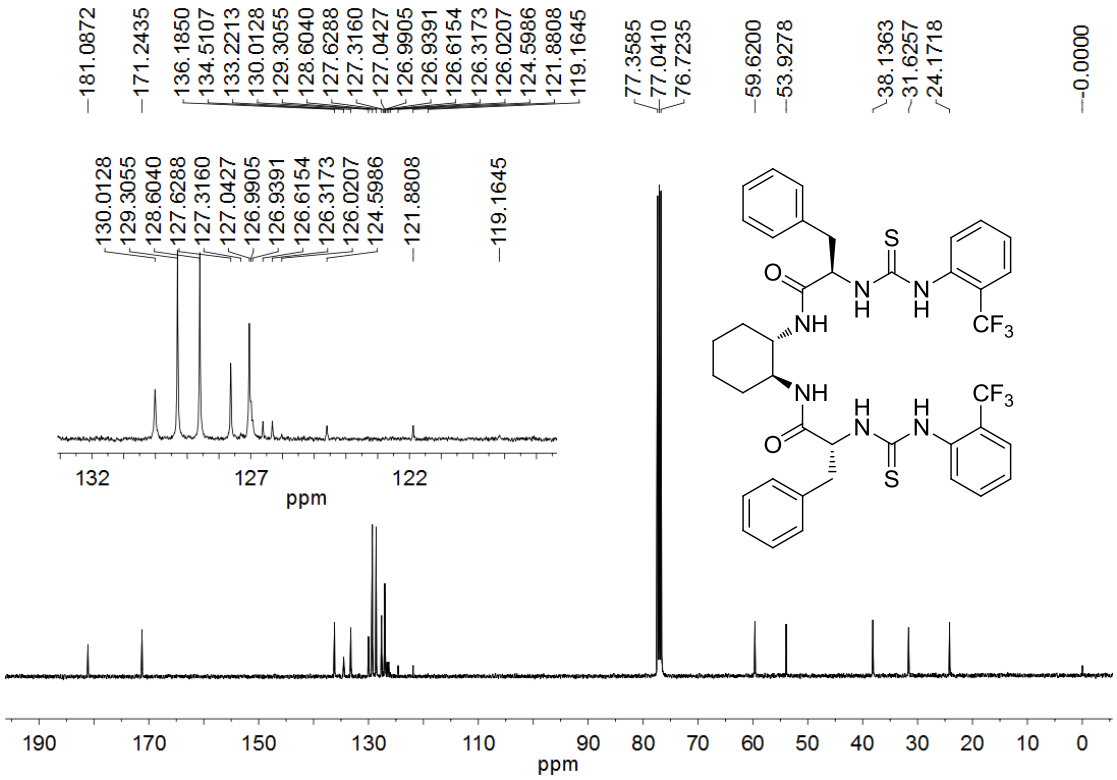


Figure S18. ^{13}C NMR spectrum of CSA **5** in $CDCl_3$ (100 MHz).

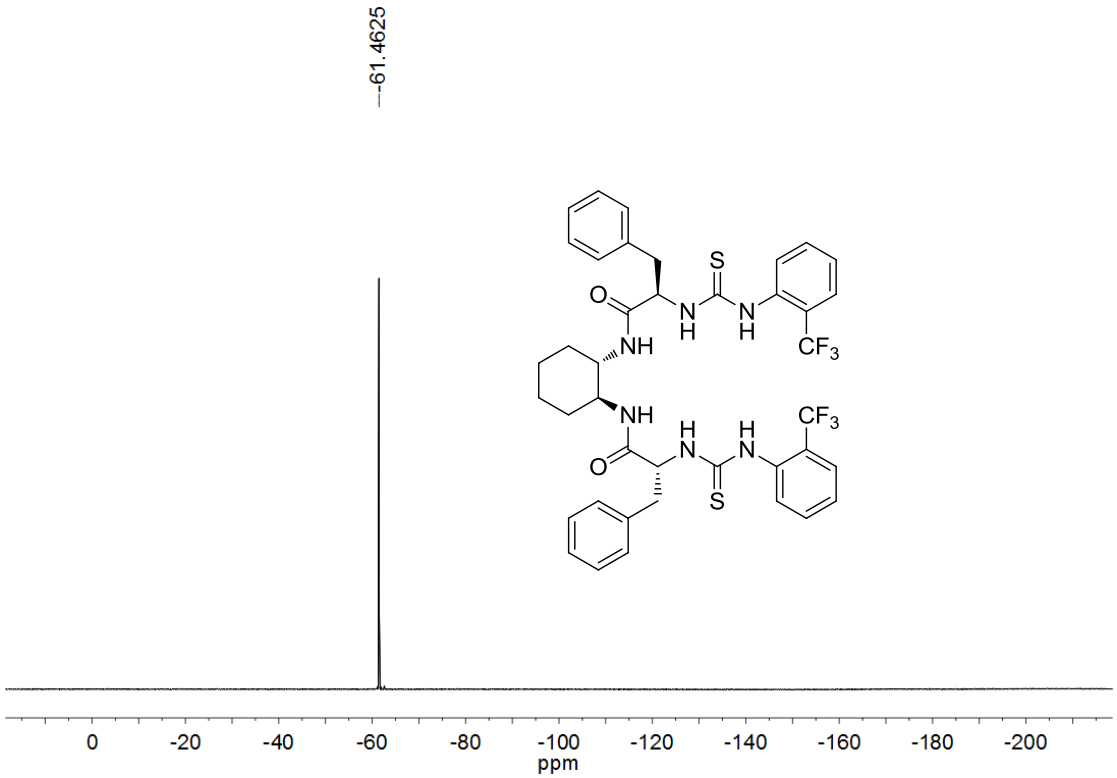
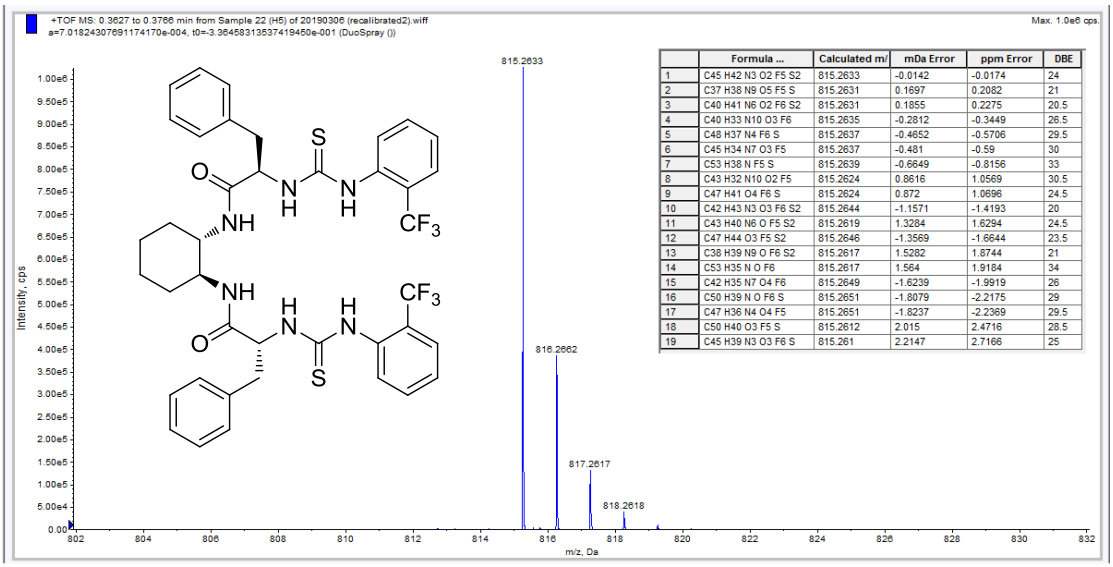


Figure S19. ^{19}F NMR spectrum of CSA **5** in $CDCl_3$ (376 MHz).



	Formula ...	Calculated m/	mDa Error	ppm Error	DBE
1	C45 H42 N3 O2 F5 S2	815.2633	-0.0142	-0.0174	24
2	C37 H38 N9 O5 F5 S	815.2631	0.1697	0.2082	21
3	C40 H41 N6 O2 F6 S2	815.2631	0.1855	0.2275	20.5
4	C40 H33 N10 O3 F6	815.2635	-0.2812	-0.3449	26.5
5	C48 H37 N4 F6 S	815.2637	-0.4652	-0.5706	29.5
6	C45 H34 N7 O3 F5	815.2637	-0.481	-0.59	30
7	C53 H38 N F5 S	815.2639	-0.6649	-0.8156	33
8	C43 H32 N10 O2 F5	815.2624	0.8616	1.0569	30.5
9	C47 H41 O4 F6 S	815.2624	0.872	1.0696	24.5
10	C42 H43 N3 O3 F6 S2	815.2644	-1.1571	-1.4193	20
11	C43 H40 N6 O F5 S2	815.2619	1.3284	1.6294	24.5
12	C47 H44 O3 F5 S2	815.2646	-1.3569	-1.6644	23.5
13	C38 H39 N9 O F6 S2	815.2617	1.5282	1.8744	21
14	C53 H35 N O F6	815.2617	1.564	1.9184	34
15	C42 H35 N7 O4 F6	815.2649	-1.6239	-1.9919	26
16	C50 H39 N O F6 S	815.2651	-1.8079	-2.2175	29
17	C47 H36 N4 O4 F5	815.2651	-1.8237	-2.2369	29.5
18	C50 H40 O3 F5 S	815.2612	2.015	2.4716	28.5
19	C45 H39 N3 O3 F6 S	815.261	2.2147	2.7166	25

Figure S20. HRMS spectrum of CSA **5**.

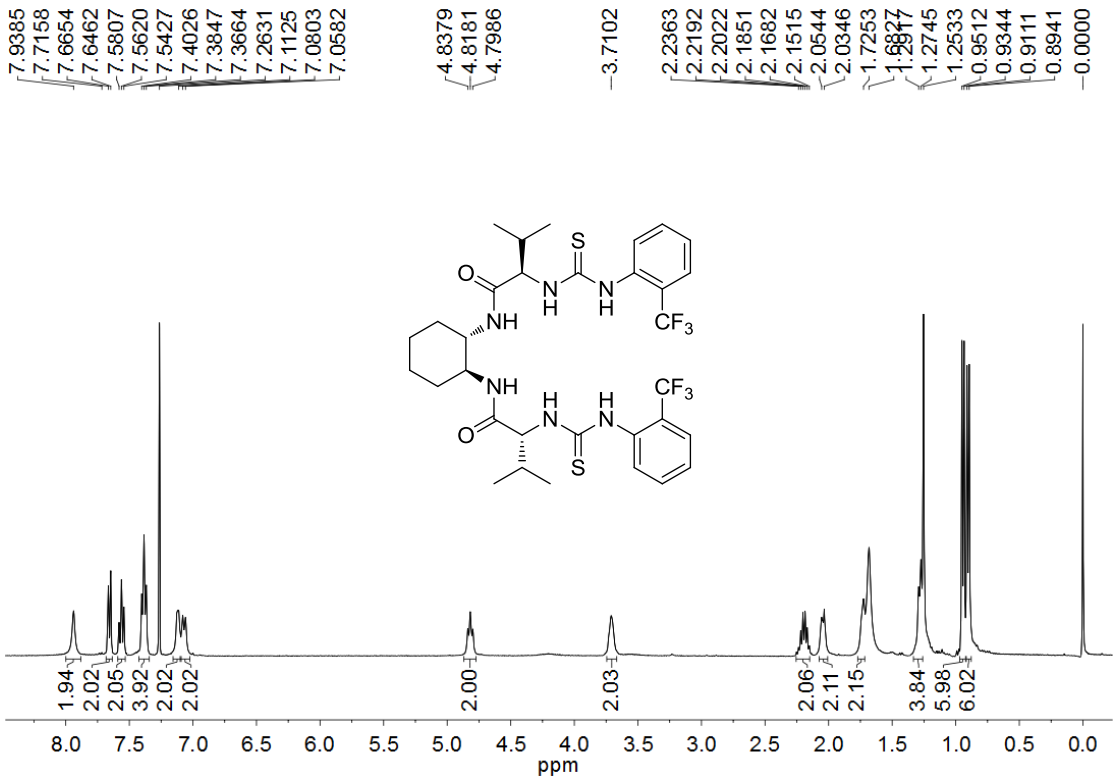


Figure S21. ^{1}H NMR spectrum of CSA **6** in $CDCl_3$ (400 MHz).

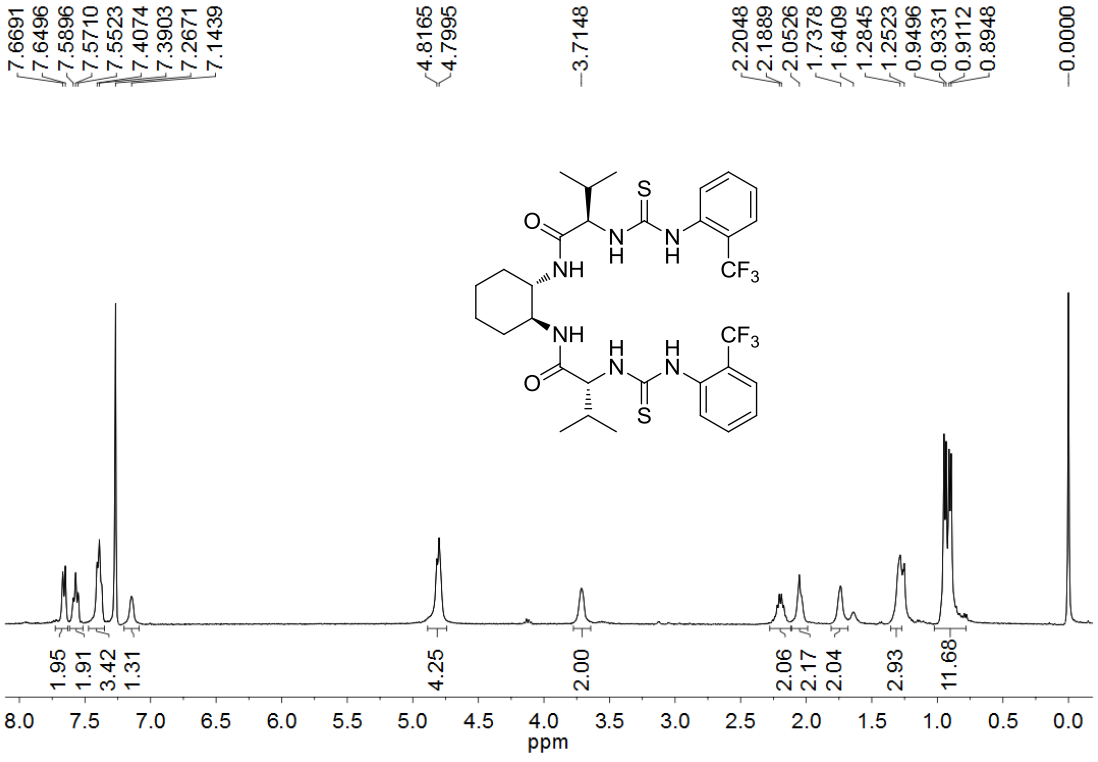


Figure S22. ^{1}H NMR spectrum of CSA **6** in $CDCl_3$ (5% D_2O) (400 MHz).

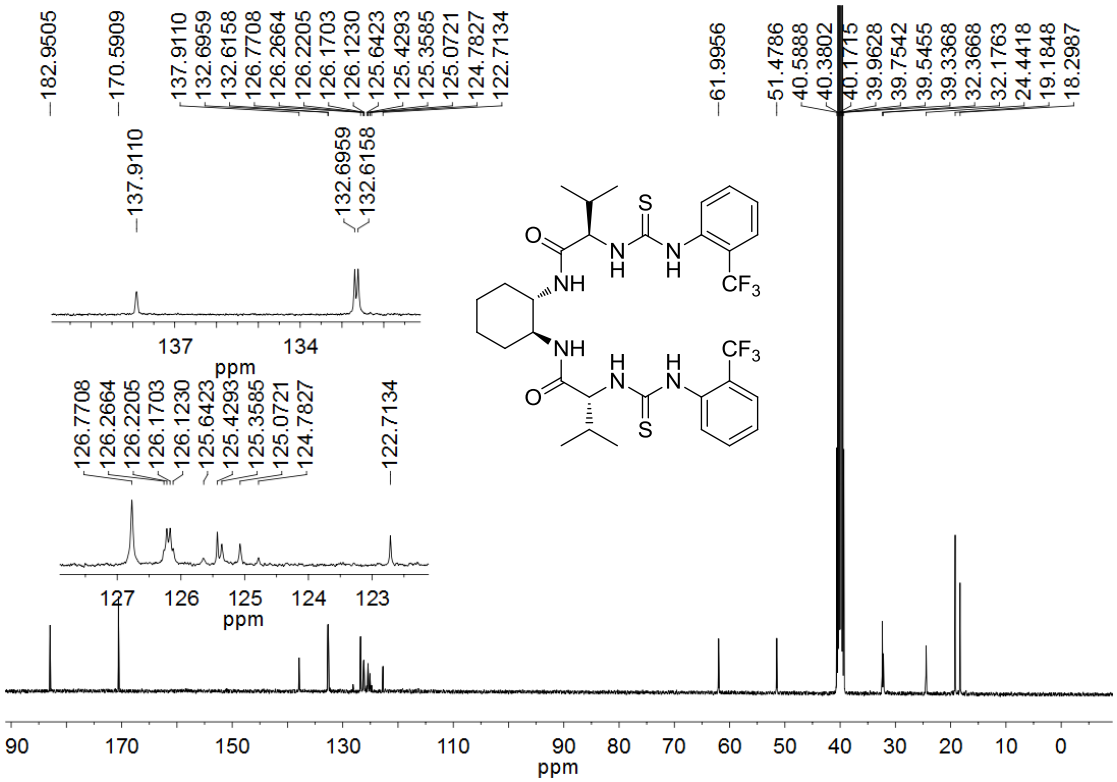


Figure S23. ^{13}C NMR spectrum of CSA **6** in DMSO-d_6 (100 MHz).

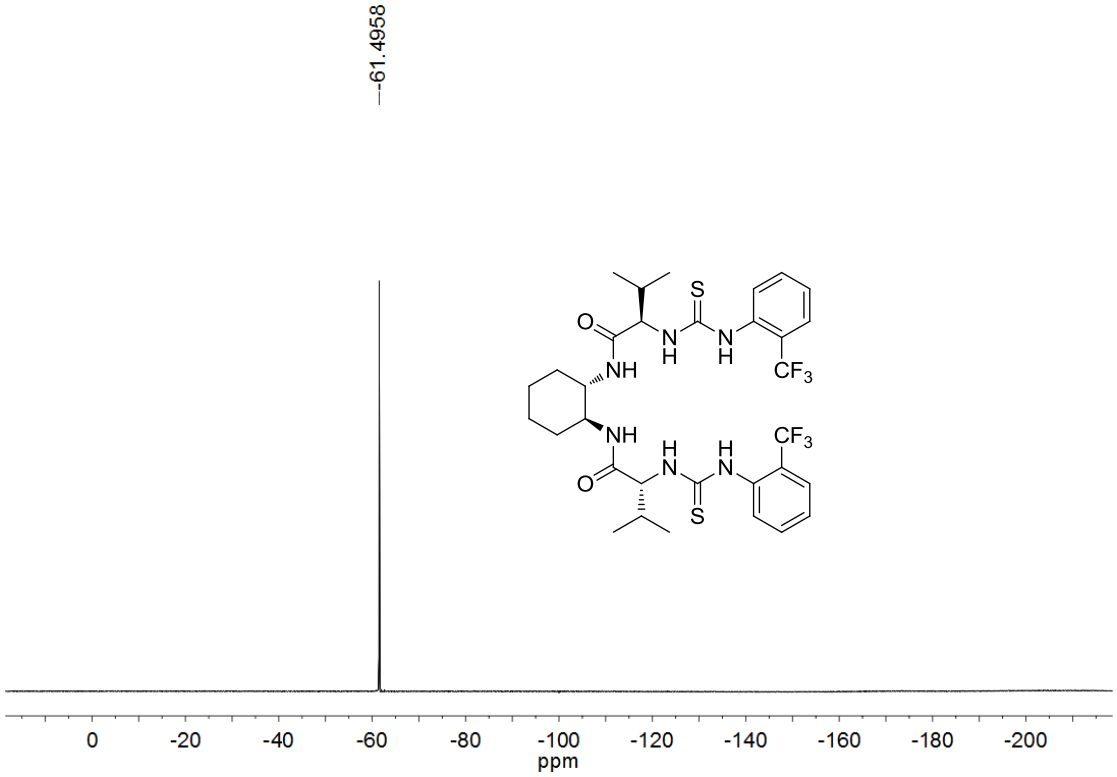
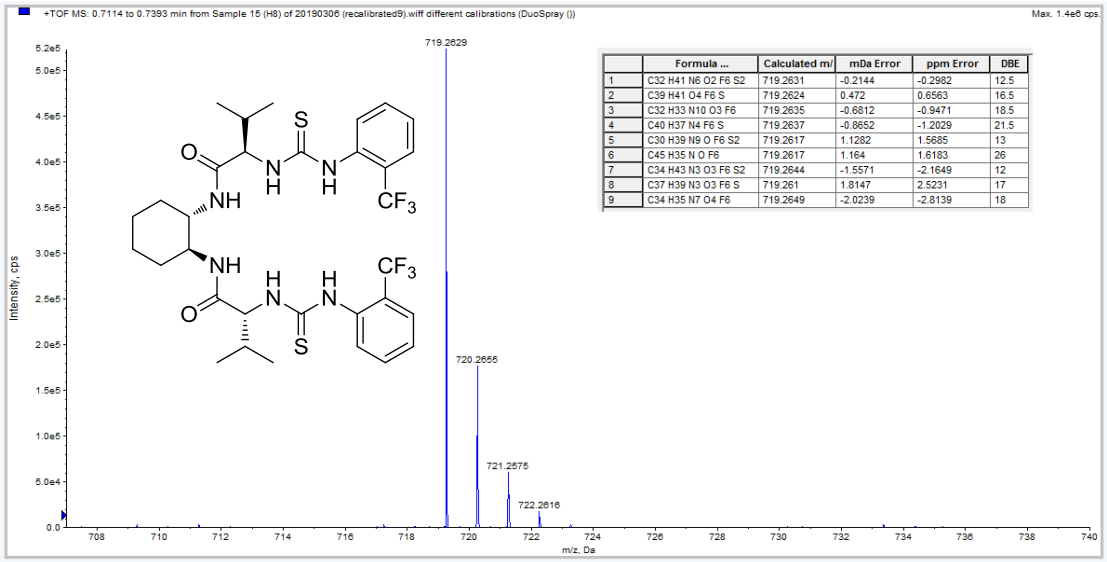


Figure S24. ^{19}F NMR spectrum of CSA **6** in $CDCl_3$ (376 MHz).



	Formula ...	Calculated m/	mDa Error	ppm Error	DBE
1	C32 H41 N6 O2 F6 S2	719.2631	-0.2144	-0.2982	12.5
2	C39 H41 O4 F6 S	719.2624	0.472	0.6563	16.5
3	C32 H33 N10 O3 F6	719.2635	-0.6812	-0.9471	18.5
4	C40 H37 N4 F6 S	719.2637	-0.8652	-1.2029	21.5
5	C30 H39 N9 O F6 S2	719.2617	1.1282	1.5685	13
6	C45 H35 N O F6	719.2617	1.164	1.6183	26
7	C34 H43 N3 O3 F6 S2	719.2644	-1.5571	-2.1649	12
8	C37 H39 N3 O3 F6 S	719.261	1.8147	2.5231	17
9	C34 H35 N7 O4 F6	719.2649	-2.0239	-2.8139	18

Figure S25. HRMS spectrum of CSA **6**.

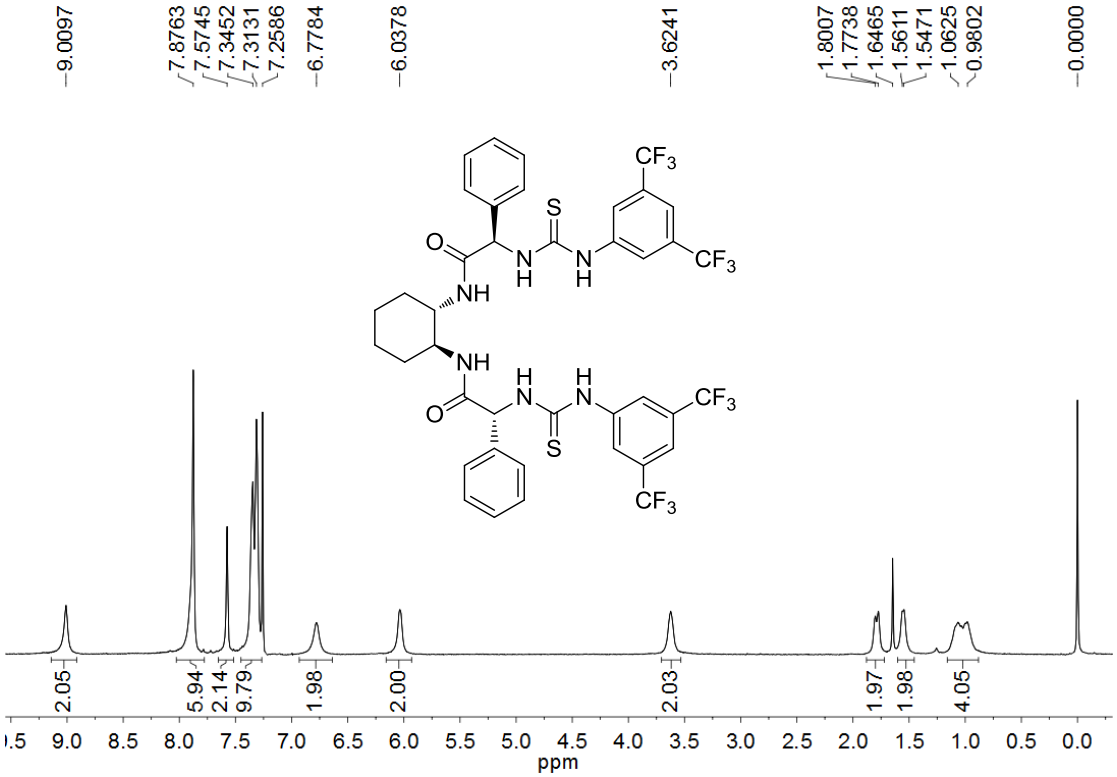


Figure S26. ^{1}H NMR spectrum of CSA **7** in $CDCl_3$ (400 MHz).

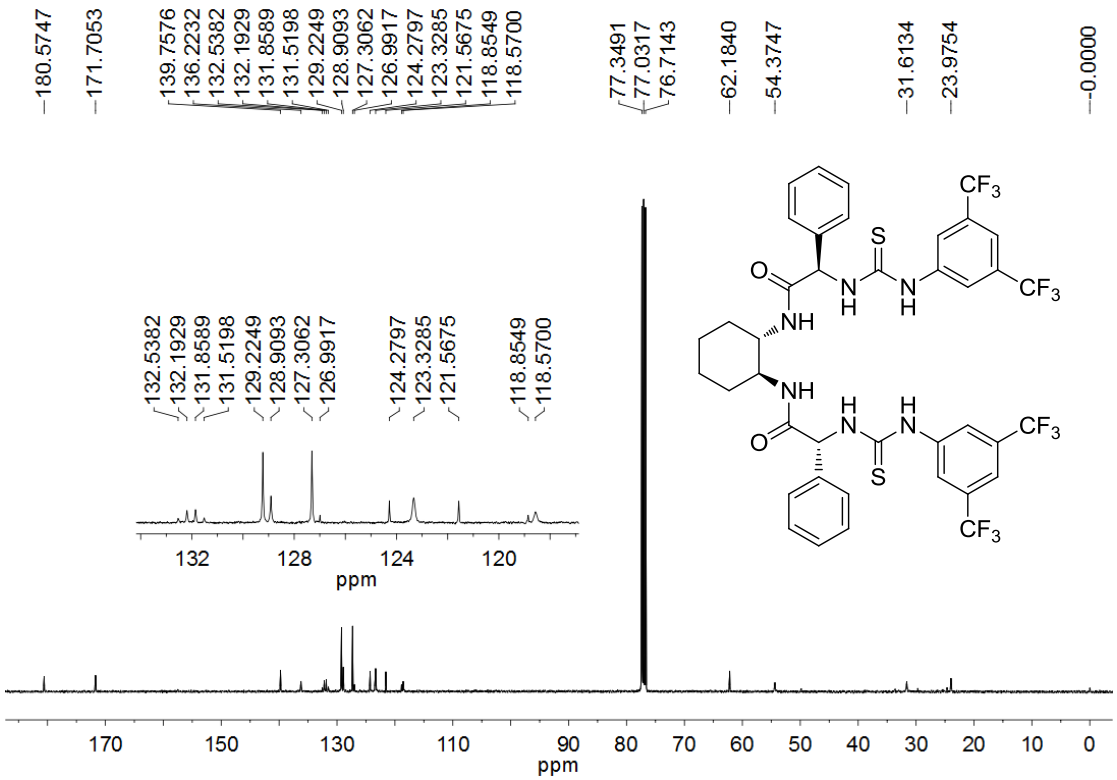


Figure S27. ^{13}C NMR spectrum of CSA **7** in $CDCl_3$ (100 MHz).

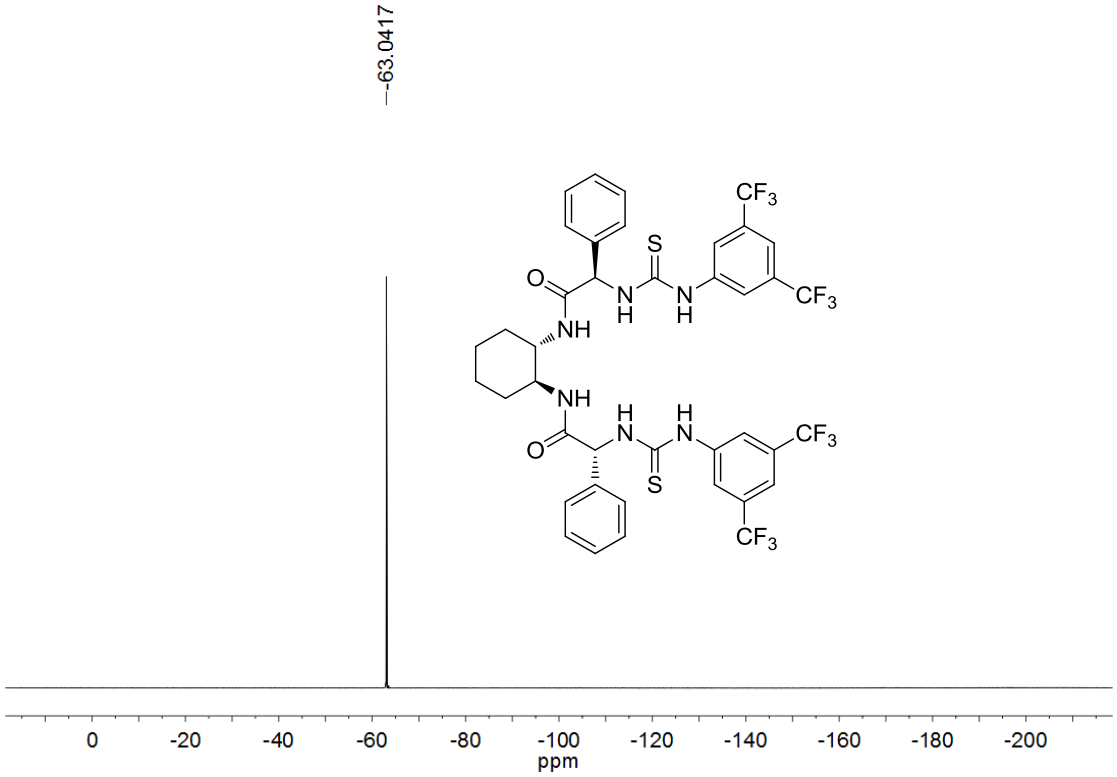
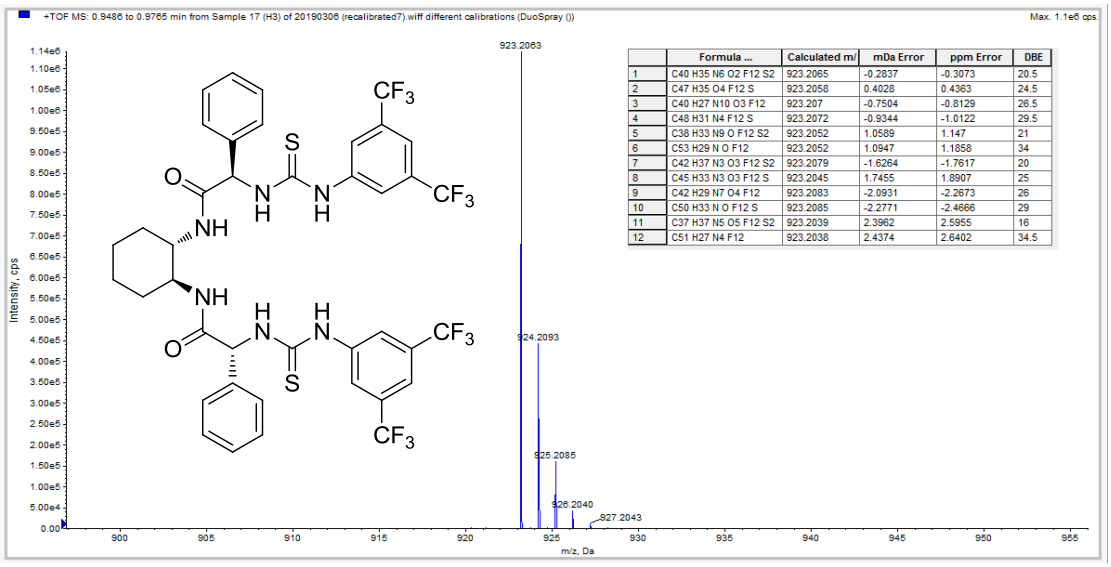


Figure S28. ^{19}F NMR spectrum of CSA **7** in $CDCl_3$ (376 MHz).



	Formula ...	Calculated m/	mDa Error	ppm Error	DBE
1	C40 H35 N6 O2 F12 S2	923.2065	-0.2837	-0.3073	20.5
2	C47 H35 O4 F12 S	923.2058	0.4028	0.4363	24.5
3	C40 H27 N10 O3 F12	923.207	-0.7504	-0.8129	26.5
4	C48 H31 N4 F12 S	923.2072	-0.9344	-1.0122	29.5
5	C38 H33 N9 O F12 S2	923.2052	1.0589	1.147	21
6	C53 H29 N O F12	923.2052	1.0947	1.1858	34
7	C42 H37 N3 O3 F12 S2	923.2079	-1.6264	-1.7617	20
8	C45 H33 N3 O3 F12 S	923.2045	1.7455	1.8907	25
9	C42 H29 N7 O4 F12	923.2083	-2.0931	-2.2673	26
10	C50 H33 N O F12 S	923.2085	-2.2771	-2.4666	29
11	C37 H37 N5 O5 F12 S2	923.2039	2.3962	2.5955	16
12	C51 H27 N4 F12	923.2038	2.4374	2.6402	34.5

Figure S29. HRMS spectrum of CSA **7**.

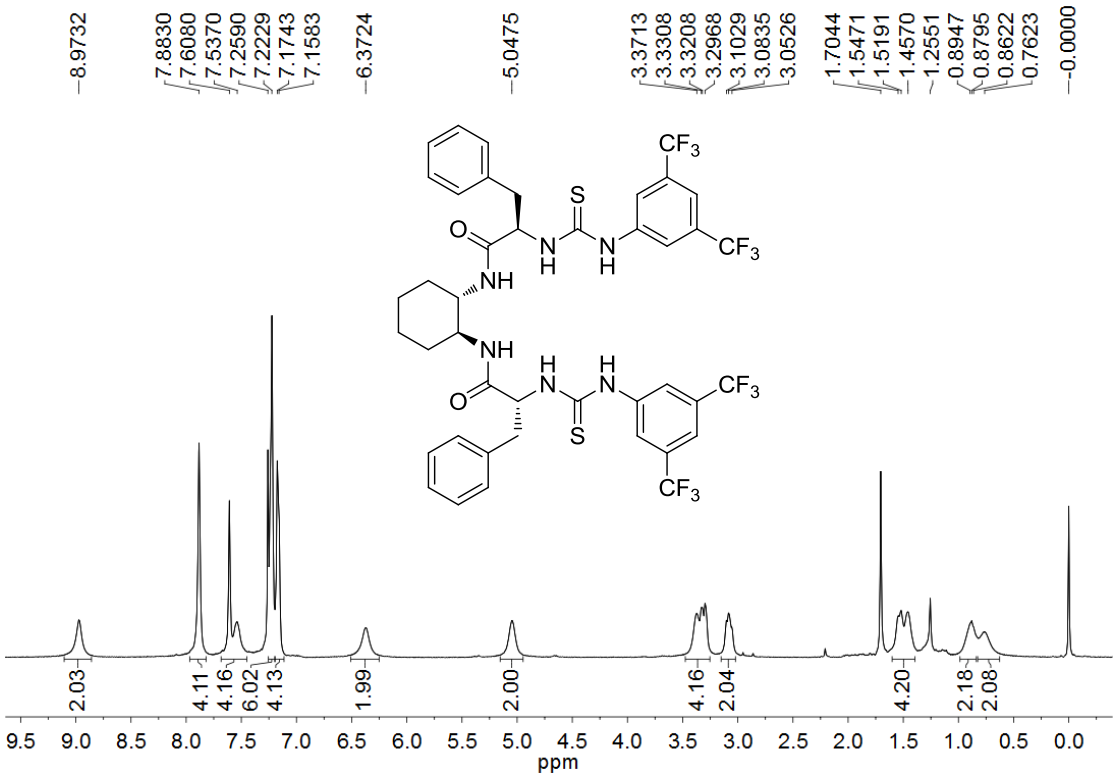


Figure S30. ^{1}H NMR spectrum of CSA **8** in $CDCl_3$ (400 MHz).

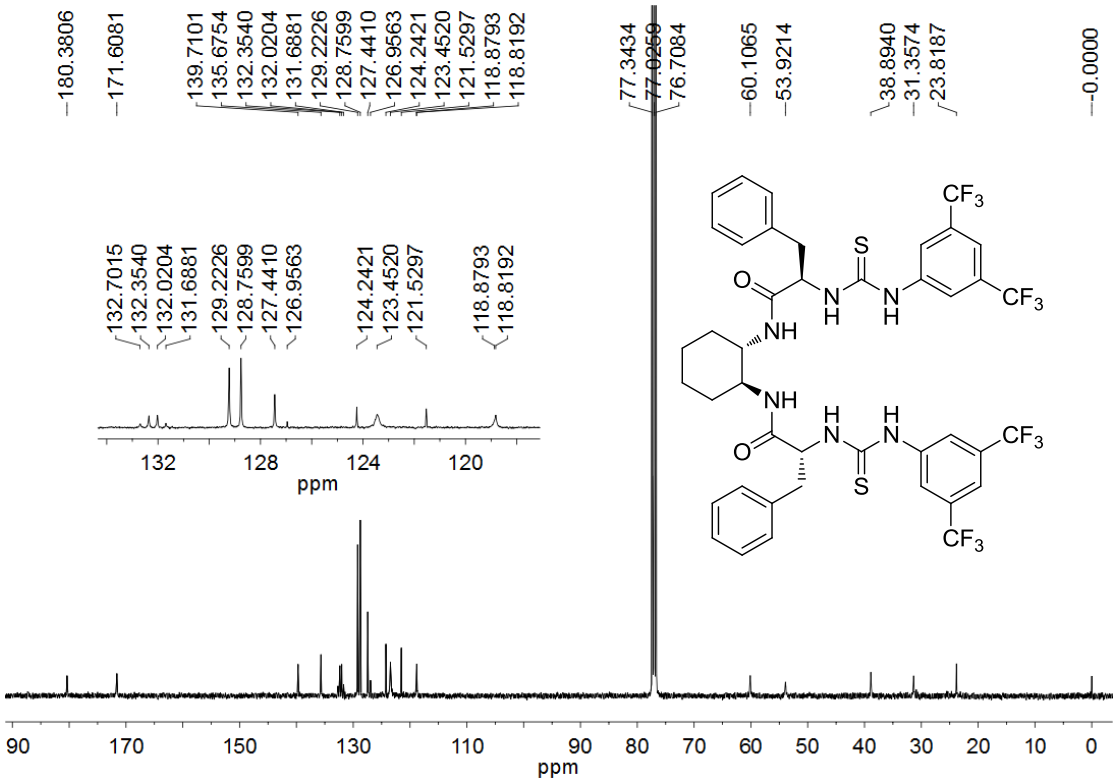


Figure S31. ^{13}C NMR spectrum of CSA **8** in $CDCl_3$ (100 MHz).

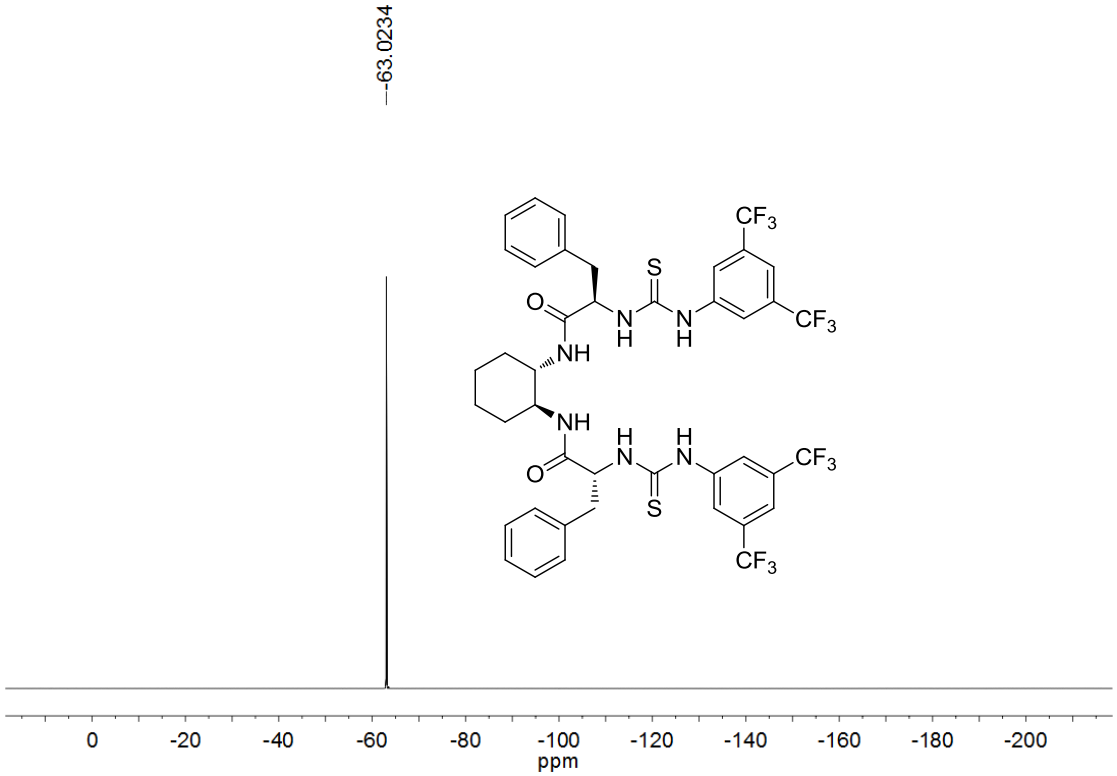
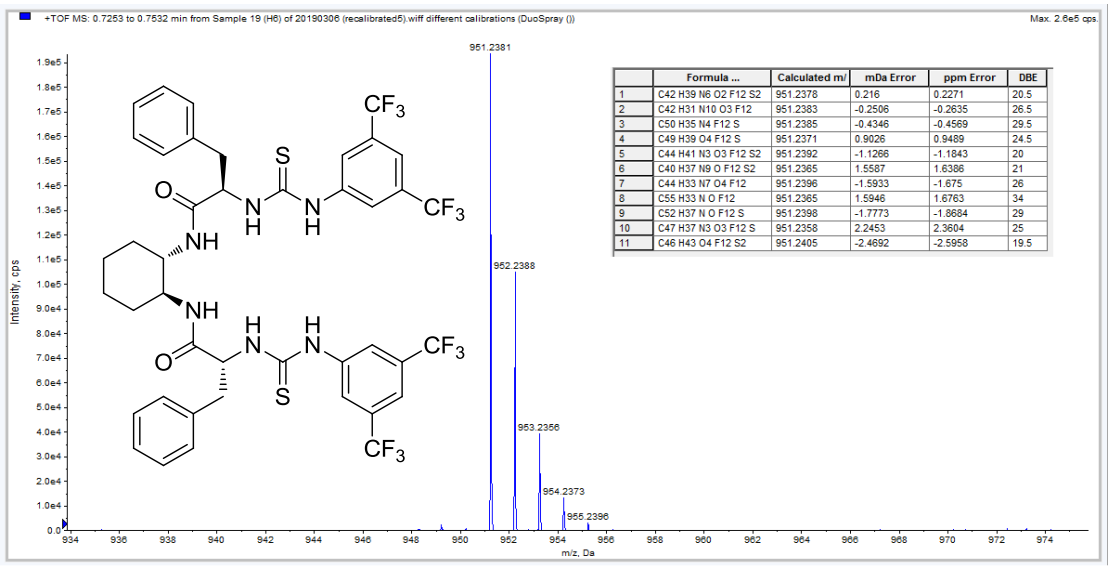


Figure S32. ^{19}F NMR spectrum of CSA **8** in $CDCl_3$ (376 MHz).



	Formula ...	Calculated m/	mDa Error	ppm Error	DBE
1	C42 H39 N6 O2 F12 S2	951.2378	0.216	0.2271	20.5
2	C42 H31 N10 O3 F12	951.2383	-0.2506	-0.2635	26.5
3	C50 H35 N4 F12 S	951.2385	-0.4346	-0.4569	29.5
4	C49 H39 O4 F12 S	951.2371	0.9026	0.9489	24.5
5	C44 H41 N3 O3 F12 S2	951.2392	-1.1266	-1.1843	20
6	C40 H37 N9 O F12 S2	951.2365	1.5587	1.6386	21
7	C44 H33 N7 O4 F12	951.2396	-1.5933	-1.675	26
8	C55 H33 N O F12	951.2365	1.5946	1.6763	34
9	C52 H37 N O F12 S	951.2398	-1.7773	-1.8684	29
10	C47 H37 N3 O3 F12 S	951.2358	2.2453	2.3604	25
11	C46 H43 O4 F12 S2	951.2405	-2.4692	-2.5958	19.5

Figure S33. HRMS spectrum of CSA **8**.

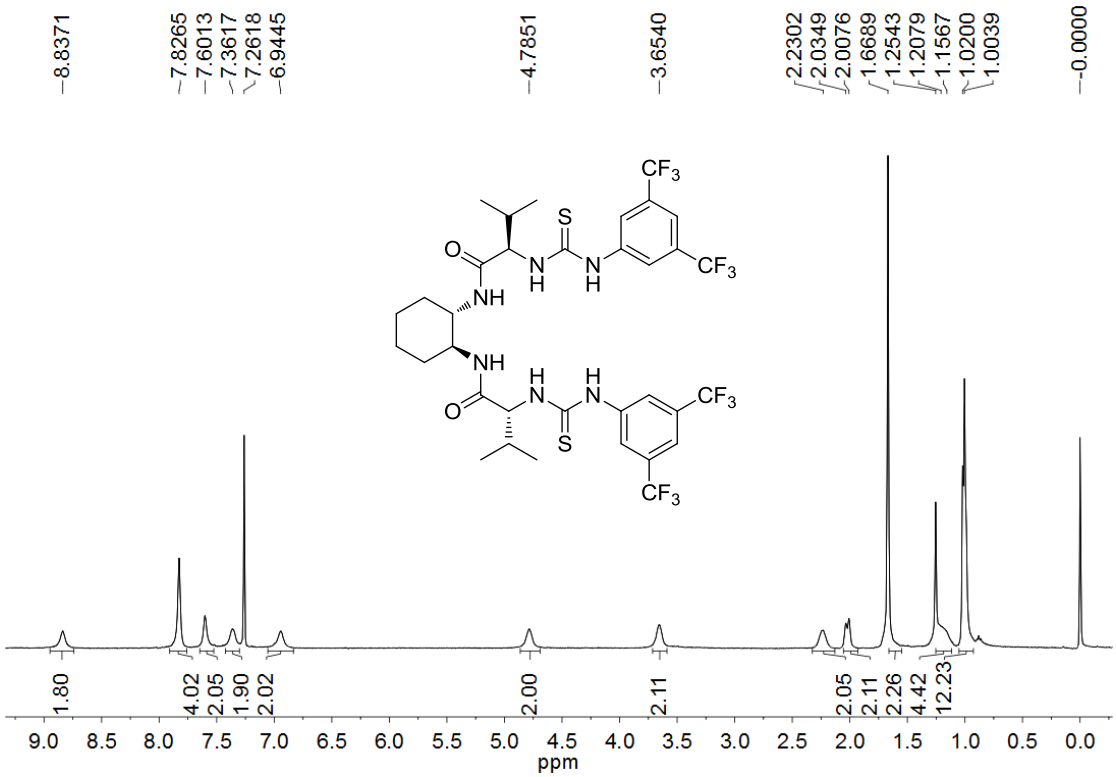


Figure S34. ^{1}H NMR spectrum of CSA **9** in $CDCl_3$ (400 MHz).

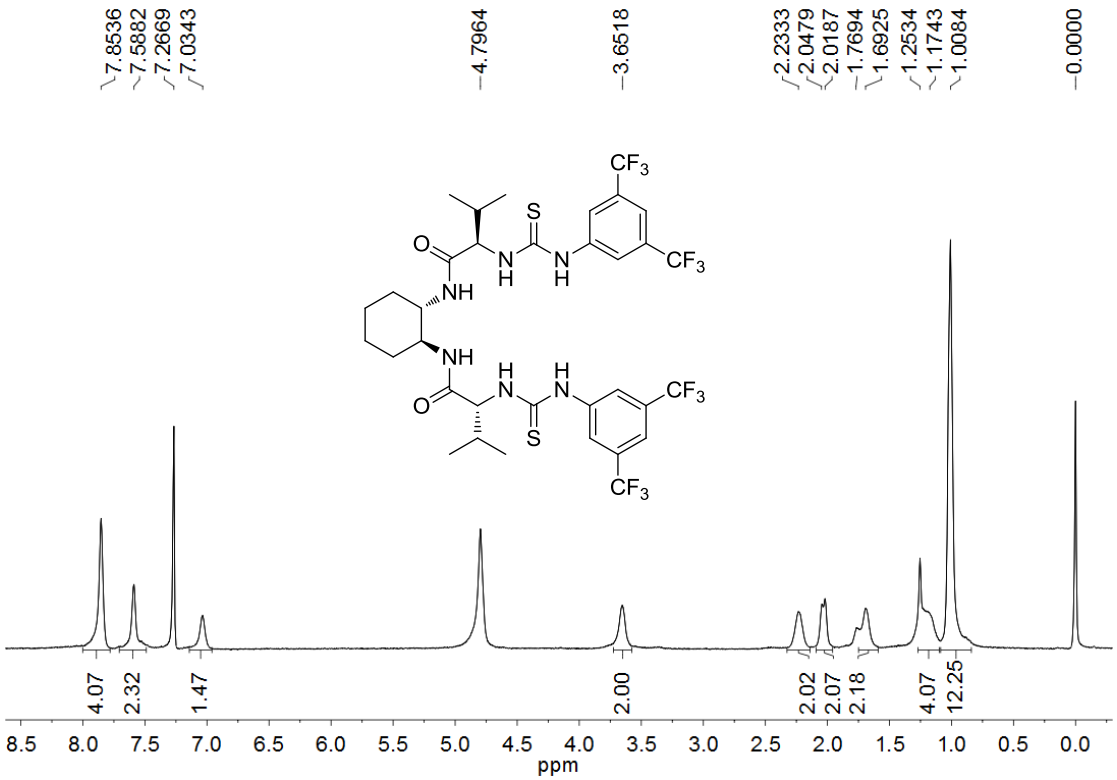


Figure S35. ^{1}H NMR spectrum of CSA **9** in $CDCl_3$ (5% D_2O) (400 MHz).

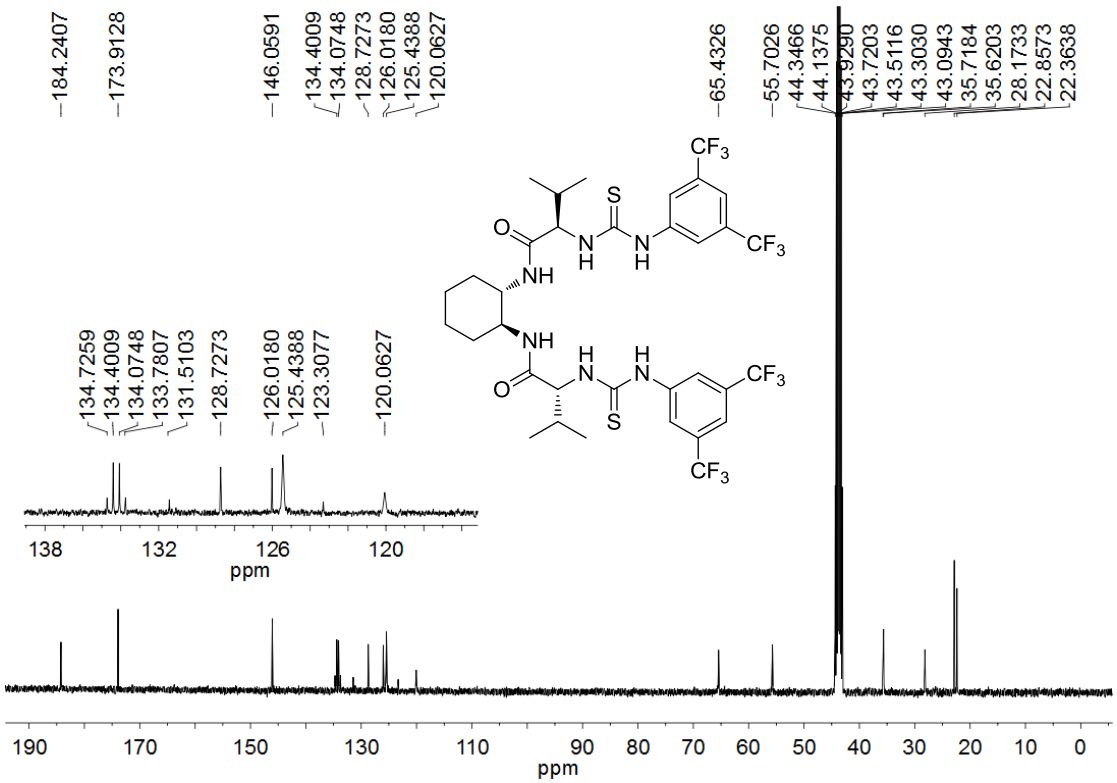


Figure S36. ^{13}C NMR spectrum of CSA **9** in DMSO-d_6 (100 MHz).

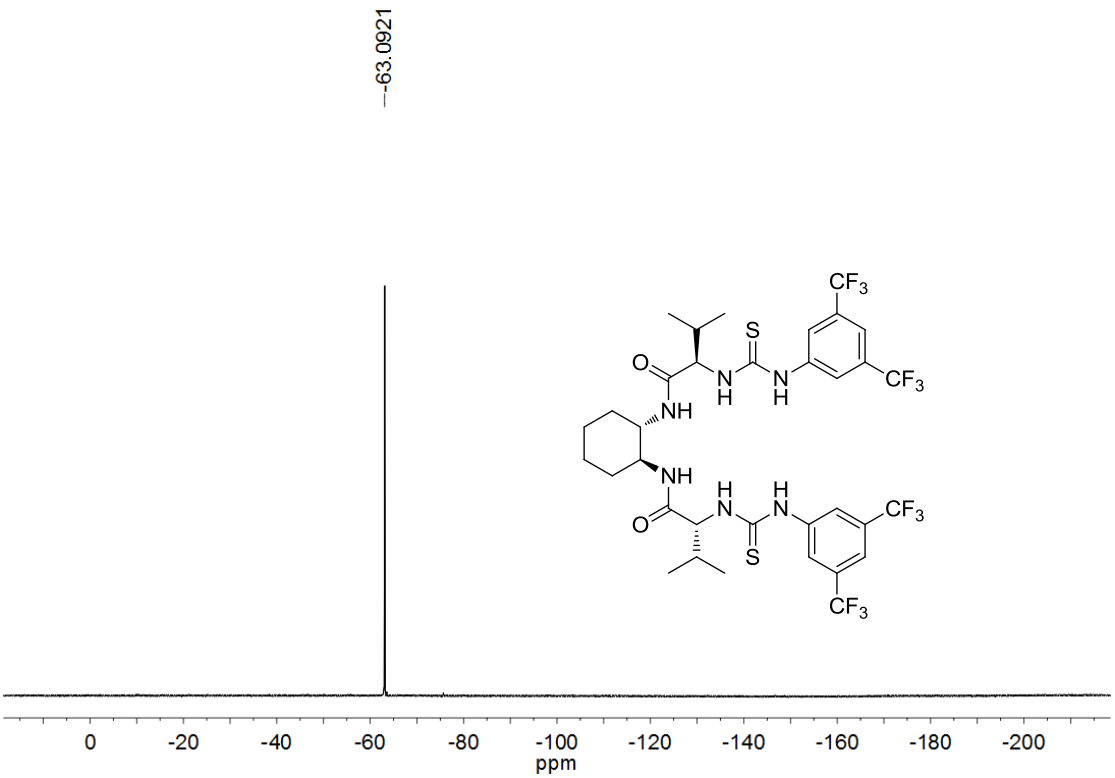
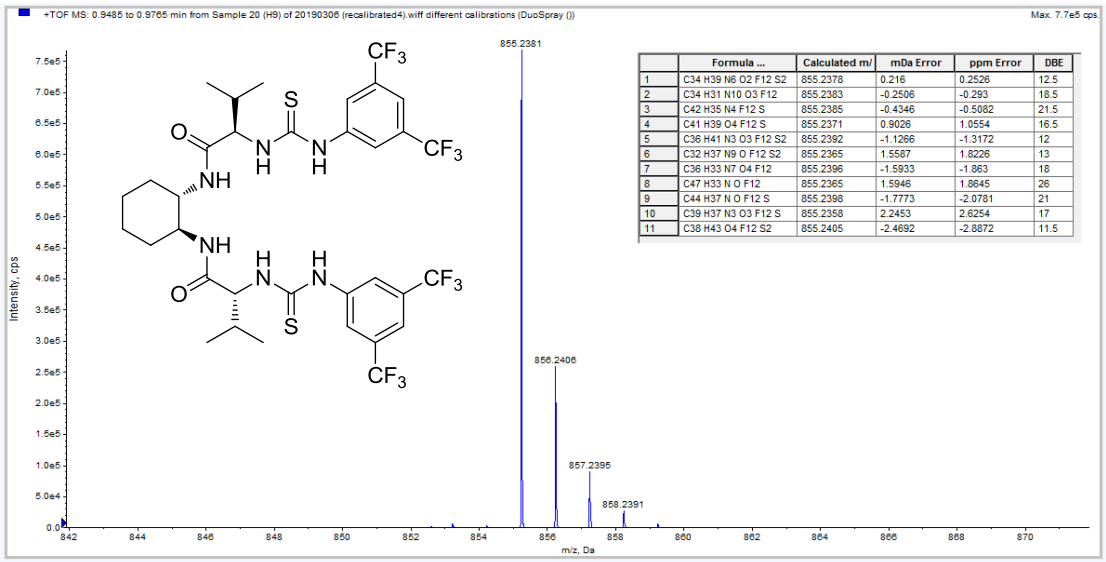


Figure S37. ^{19}F NMR spectrum of CSA **9** in $CDCl_3$ (376 MHz).



	Formula ...	Calculated m/	mDa Error	ppm Error	DBE
1	C34 H39 N6 O2 F12 S2	855.2378	0.216	0.2526	12.5
2	C34 H31 N10 O3 F12	855.2383	-0.2506	-0.293	18.5
3	C42 H35 N4 F12 S	855.2385	-0.4346	-0.5082	21.5
4	C41 H39 O4 F12 S	855.2371	0.9026	1.0554	16.5
5	C36 H41 N3 O3 F12 S2	855.2392	-1.1266	-1.3172	12
6	C32 H37 N9 O F12 S2	855.2365	1.5587	1.8226	13
7	C36 H33 N7 O4 F12	855.2396	-1.5933	-1.863	18
8	C47 H33 N O F12	855.2365	1.5946	1.8645	26
9	C44 H37 N O F12 S	855.2398	-1.7773	-2.0781	21
10	C39 H37 N3 O3 F12 S	855.2358	2.2453	2.6254	17
11	C38 H43 O4 F12 S2	855.2405	-2.4692	-2.8872	11.5

Figure S38. HRMS spectrum of CSA **9**.

NMR and HRMS spectra of enantiomers of amides G16–27 as guests.

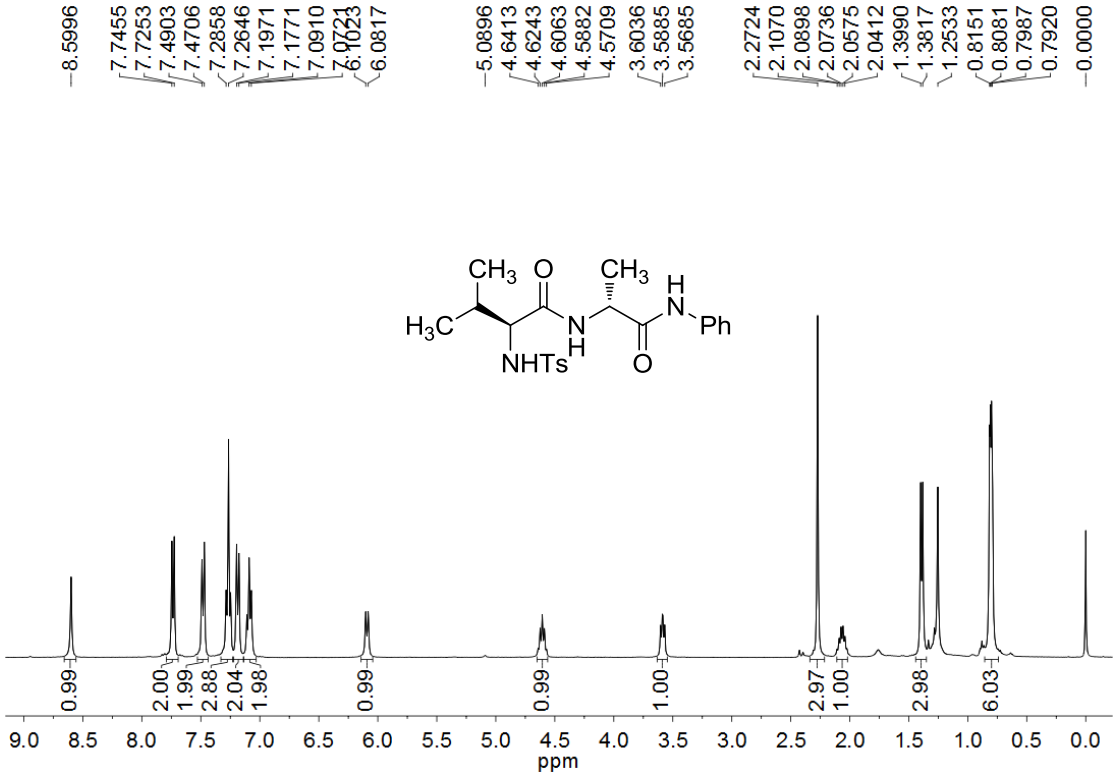


Figure S39. ^{1}H NMR spectrum of (*S*,*R*)-G**16** in $CDCl_3$ (400 MHz).

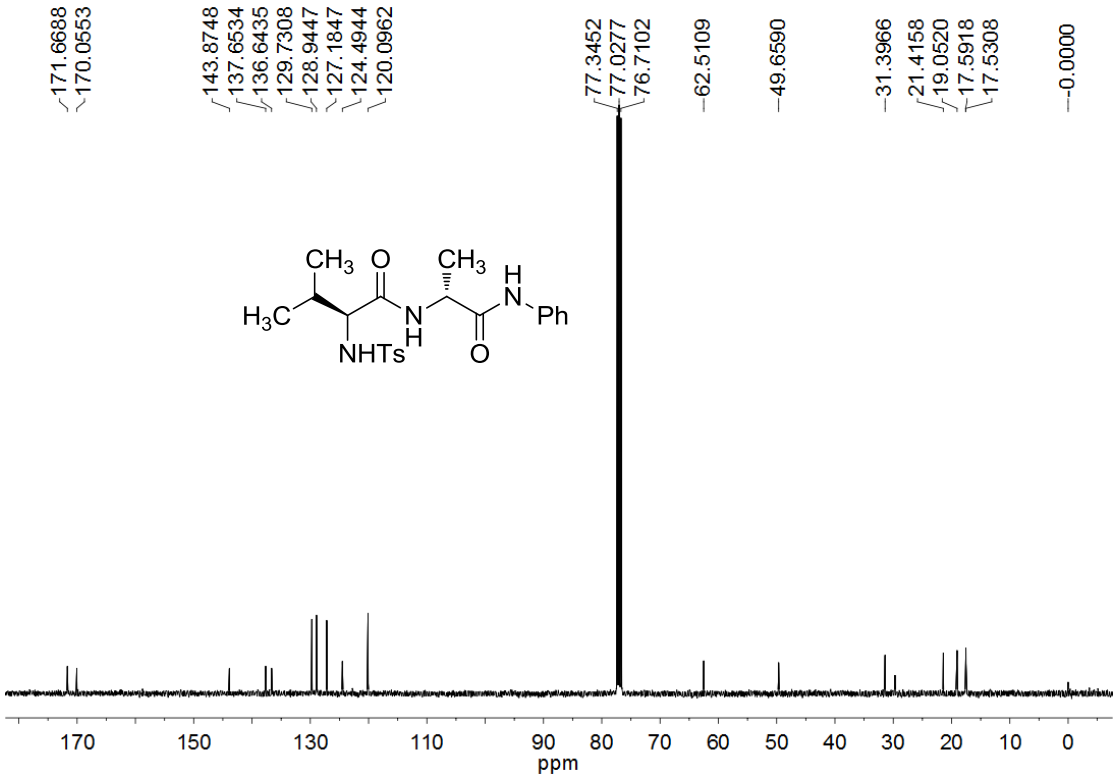


Figure S40. ^{13}C NMR spectrum of (*S*,*R*)-G**16** in $CDCl_3$ (100 MHz).

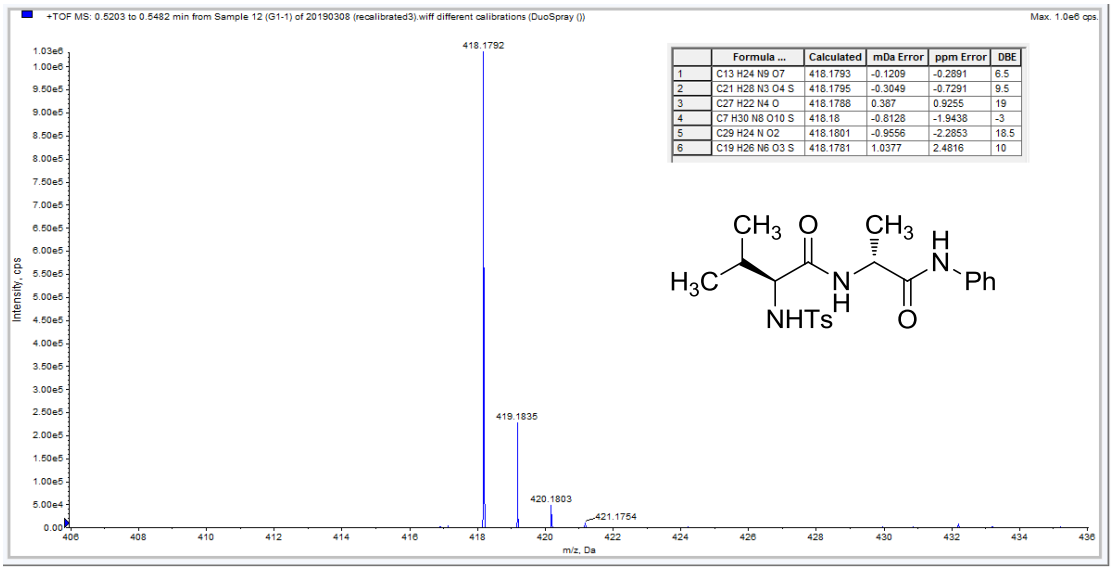


Figure S41. HRMS spectrum of (*S*,*R*)-G**16**.

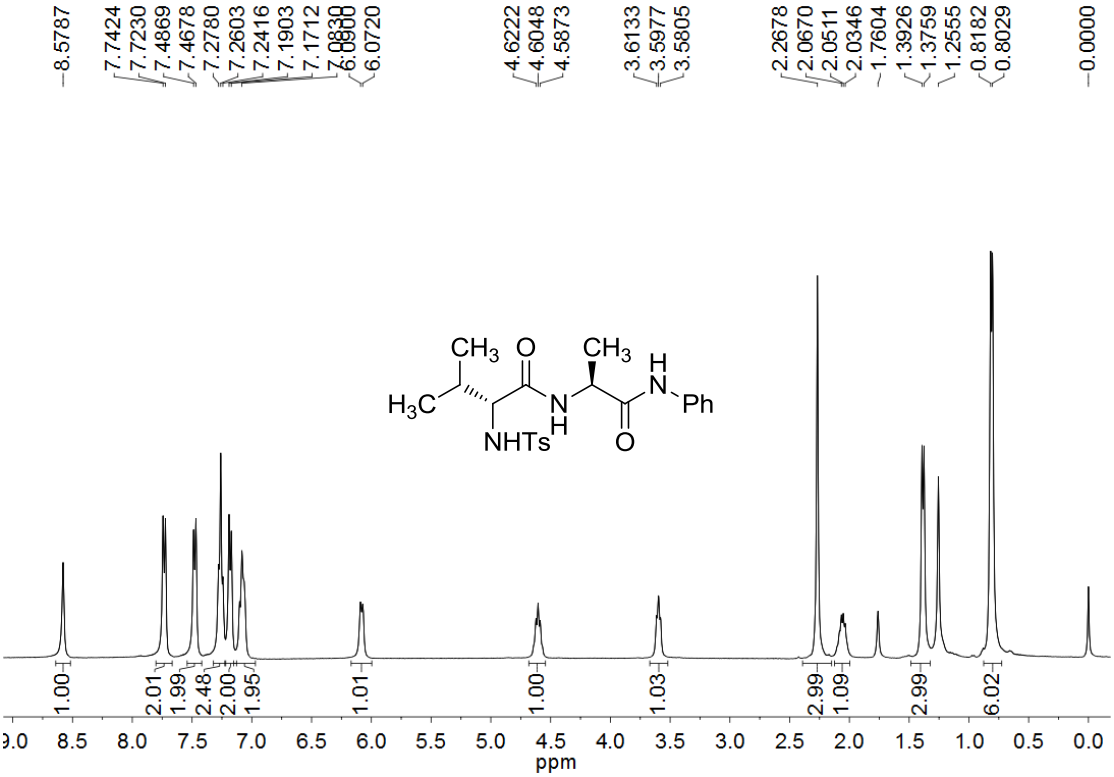


Figure S42. ^{1}H NMR spectrum of (*R*,*S*)-G**16** in $CDCl_3$ (400 MHz).

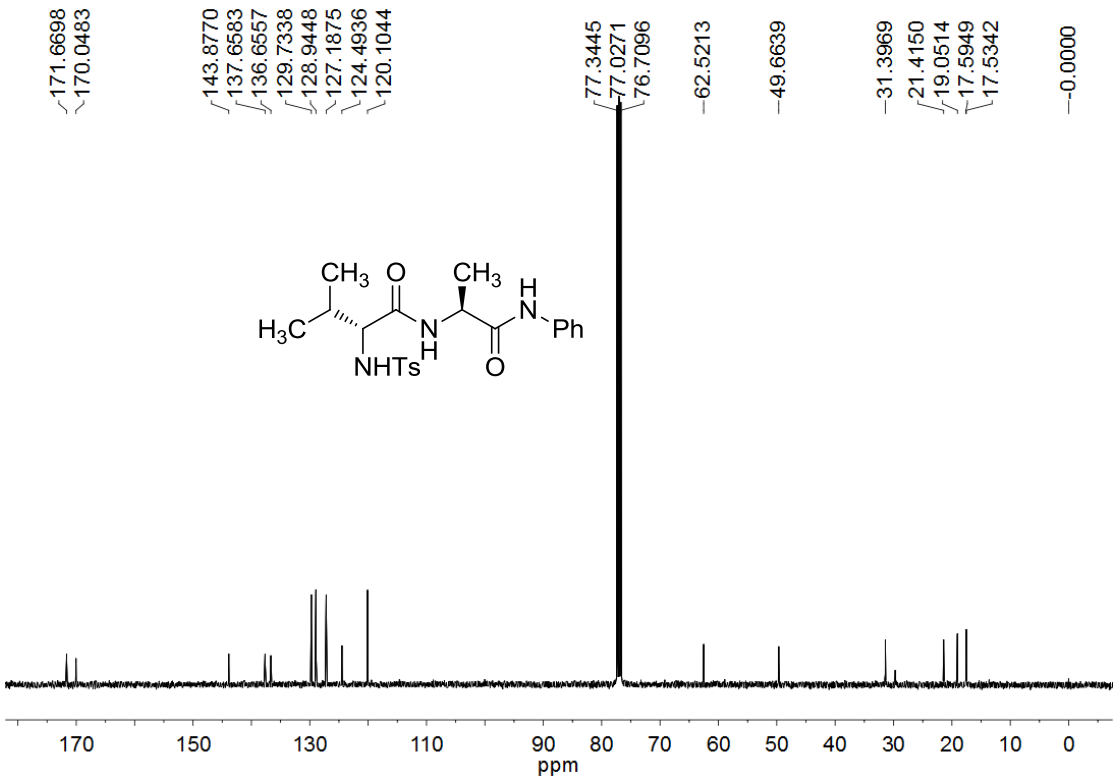


Figure S43. ^{13}C NMR spectrum of (*R*,*S*)-G**16** in $CDCl_3$ (100 MHz).

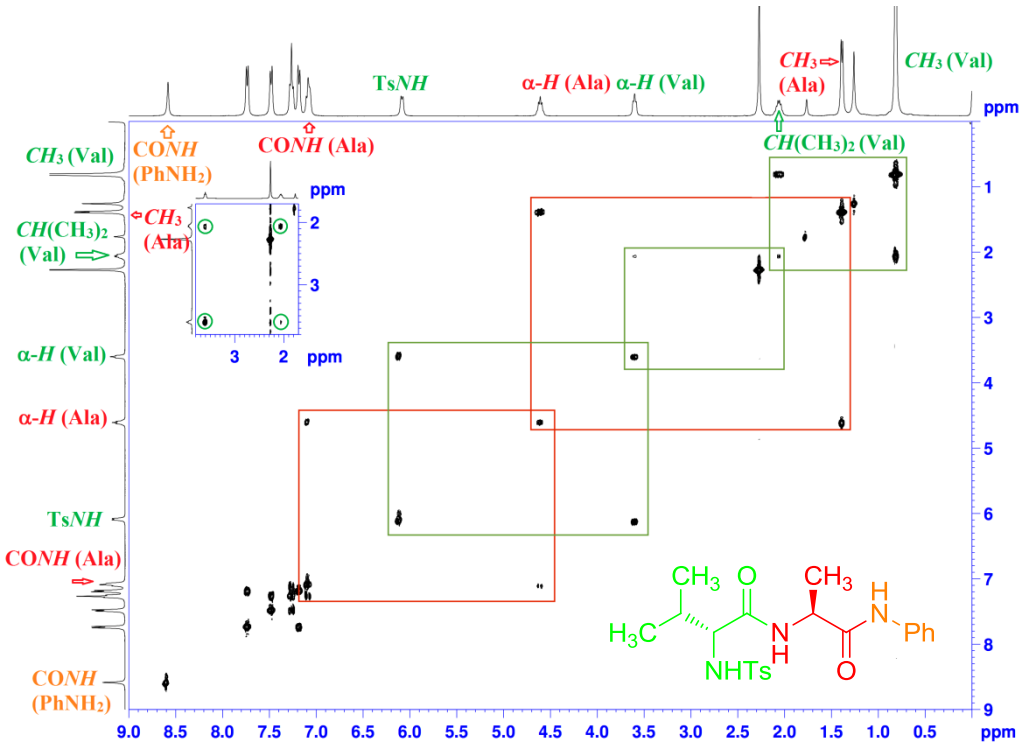


Figure S44. ^{1}H–^{1}H COSY spectrum of (*R*,*S*)-G**16** in $CDCl_3$ (400 MHz).

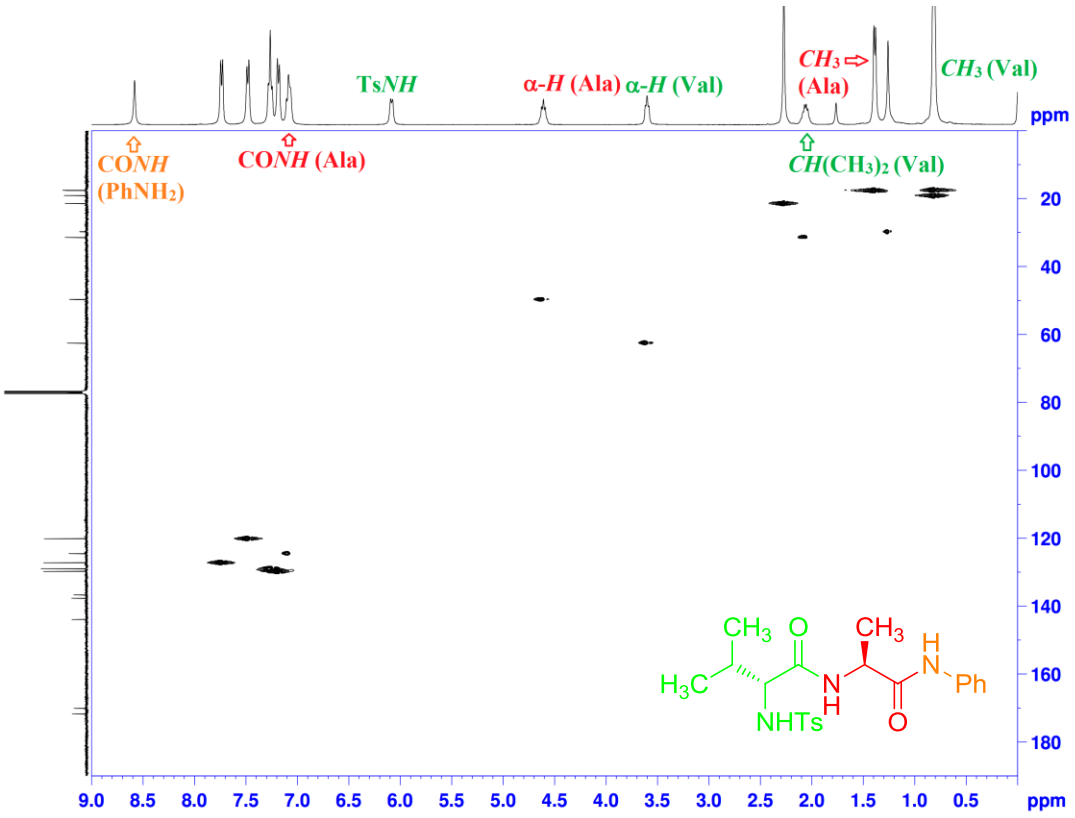


Figure S45. HSQC spectrum of (*R*,*S*)-G**16** in $CDCl_3$.

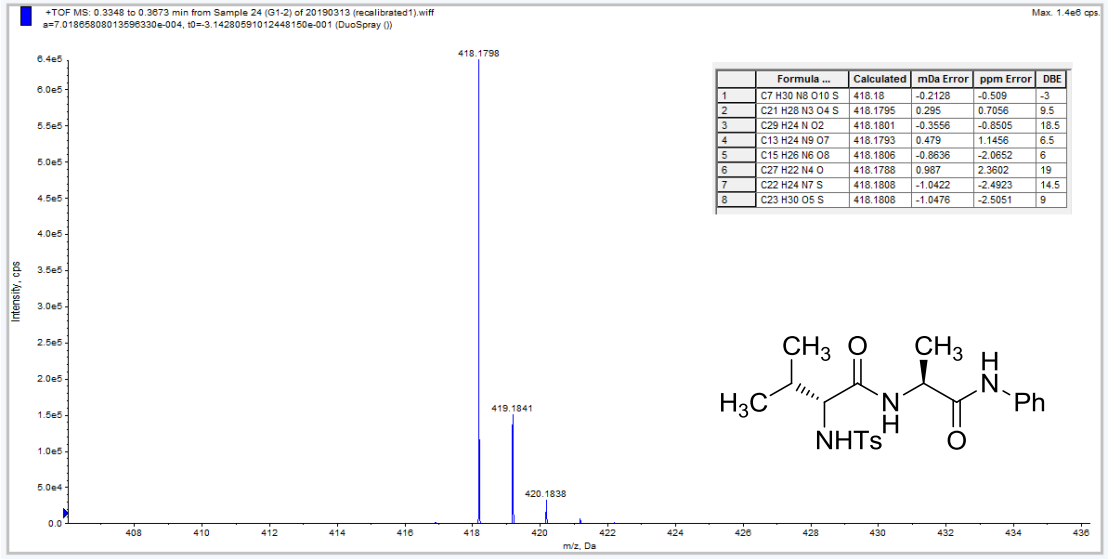


Figure S46. HRMS spectrum of (*R*,*S*)-G**16**.

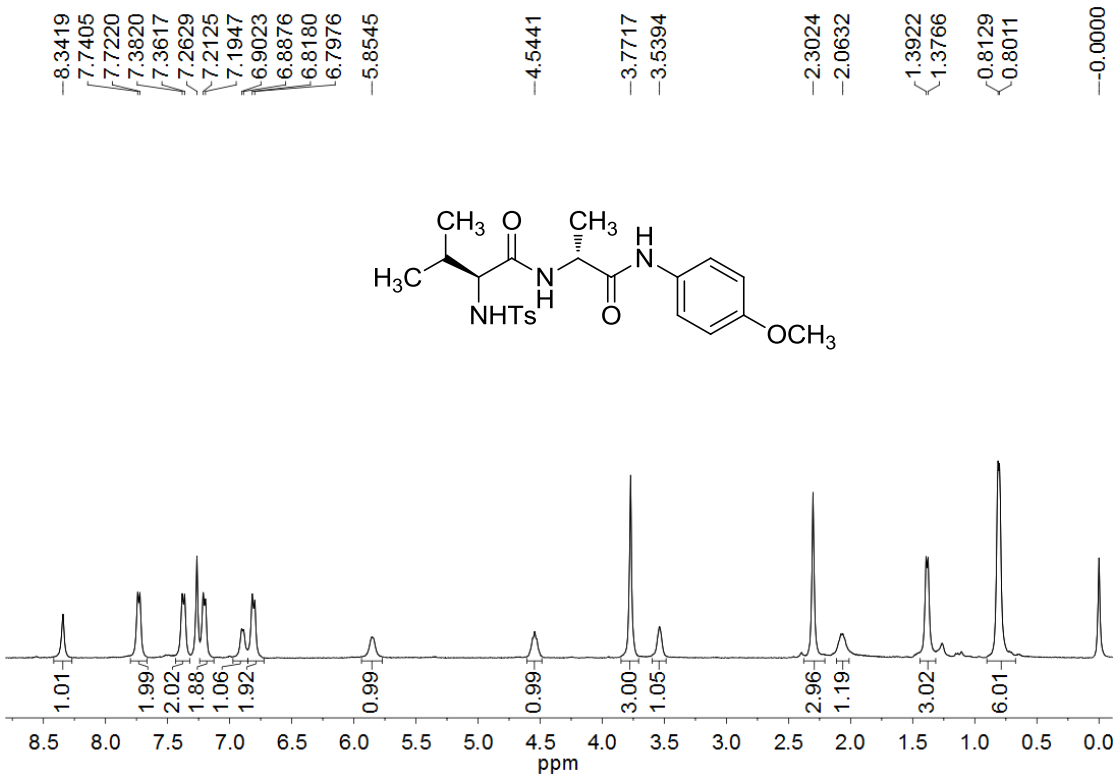


Figure S47. ^{1}H NMR spectrum of (*S*,*R*)-G**17** in $CDCl_3$ (400 MHz).

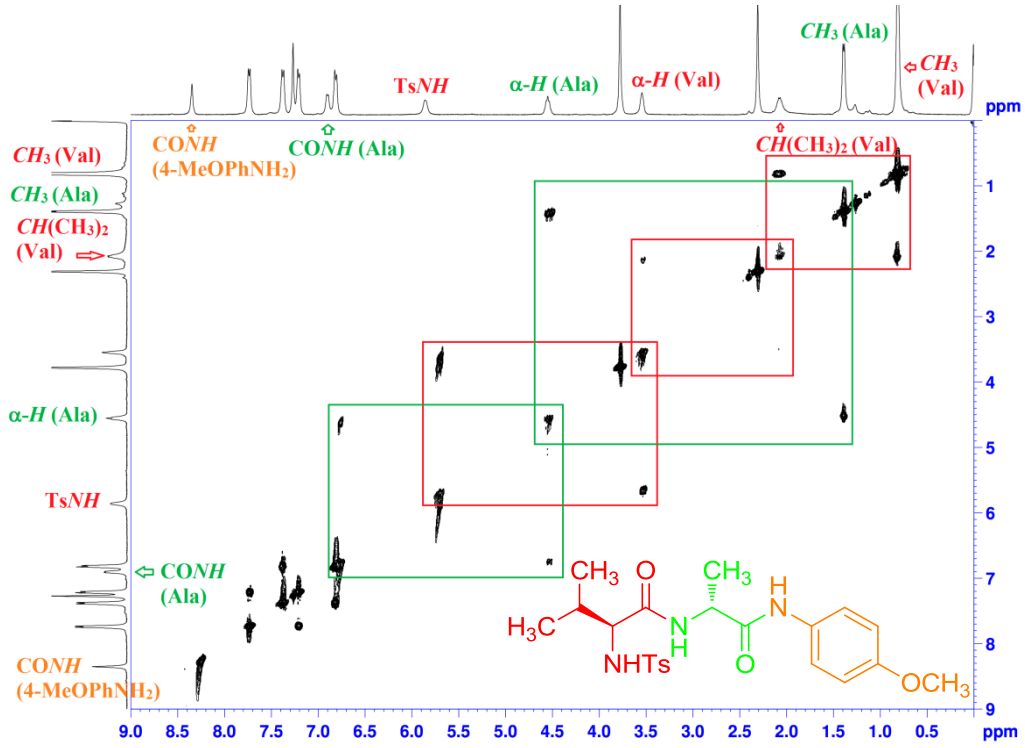


Figure S48. ^{1}H–^{1}H COSY spectrum of (*S*,*R*)-G**17** in $CDCl_3$ (400 MHz).

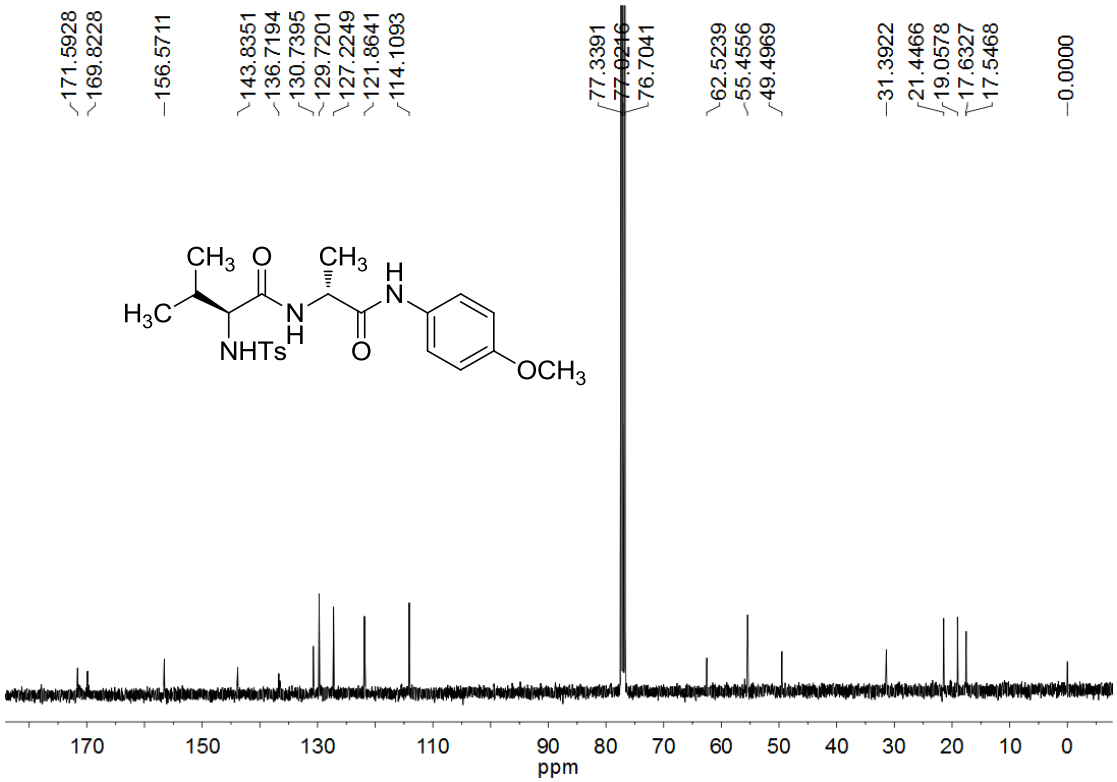


Figure S49. ^{13}C NMR spectrum of (*S*,*R*)-G**17** in $CDCl_3$ (100 MHz).

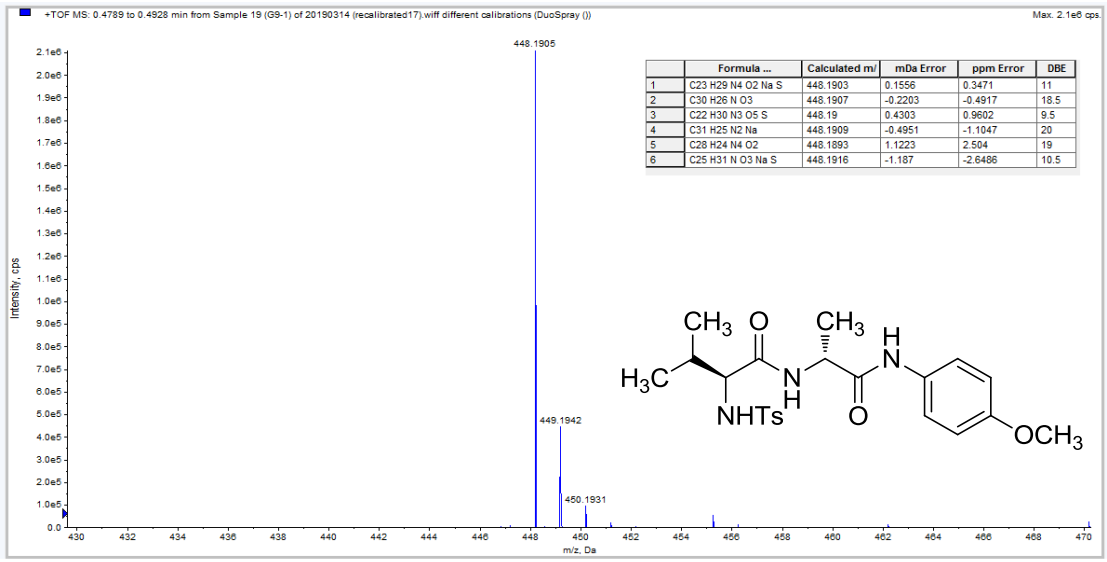


Figure S50. HRMS spectrum of (*S*,*R*)-G**17**.

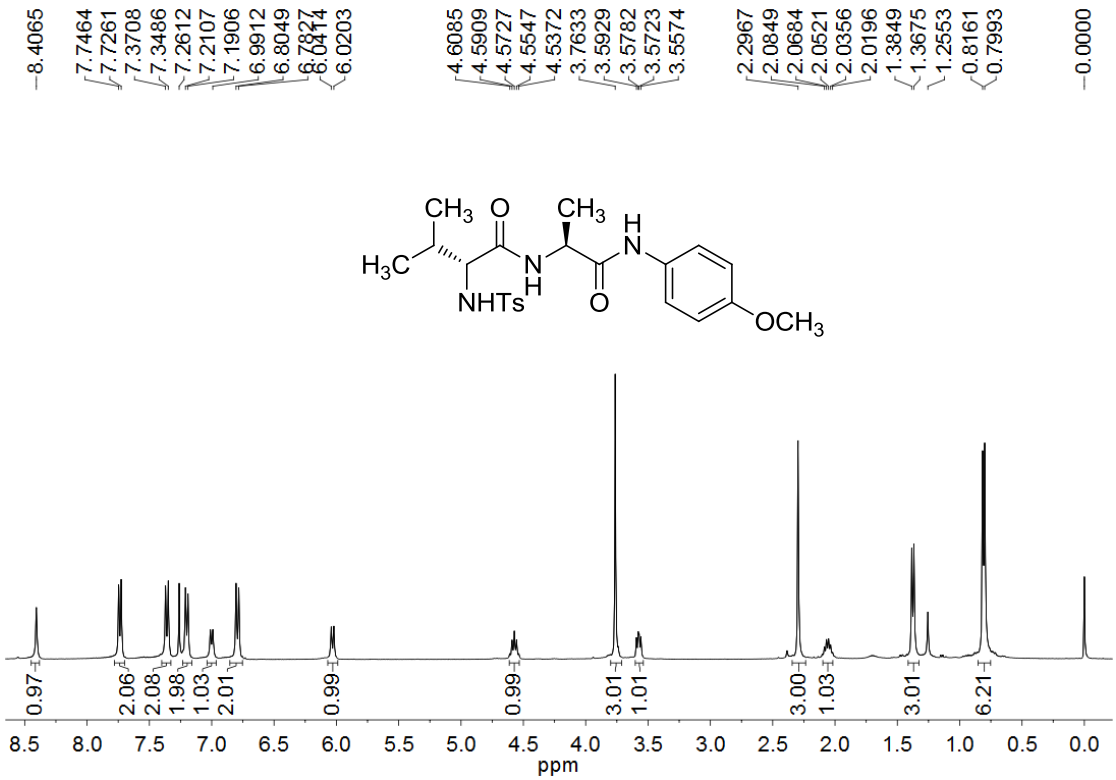


Figure S51. ^{1}H NMR spectrum of (*R*,*S*)-G**17** in $CDCl_3$ (400 MHz).

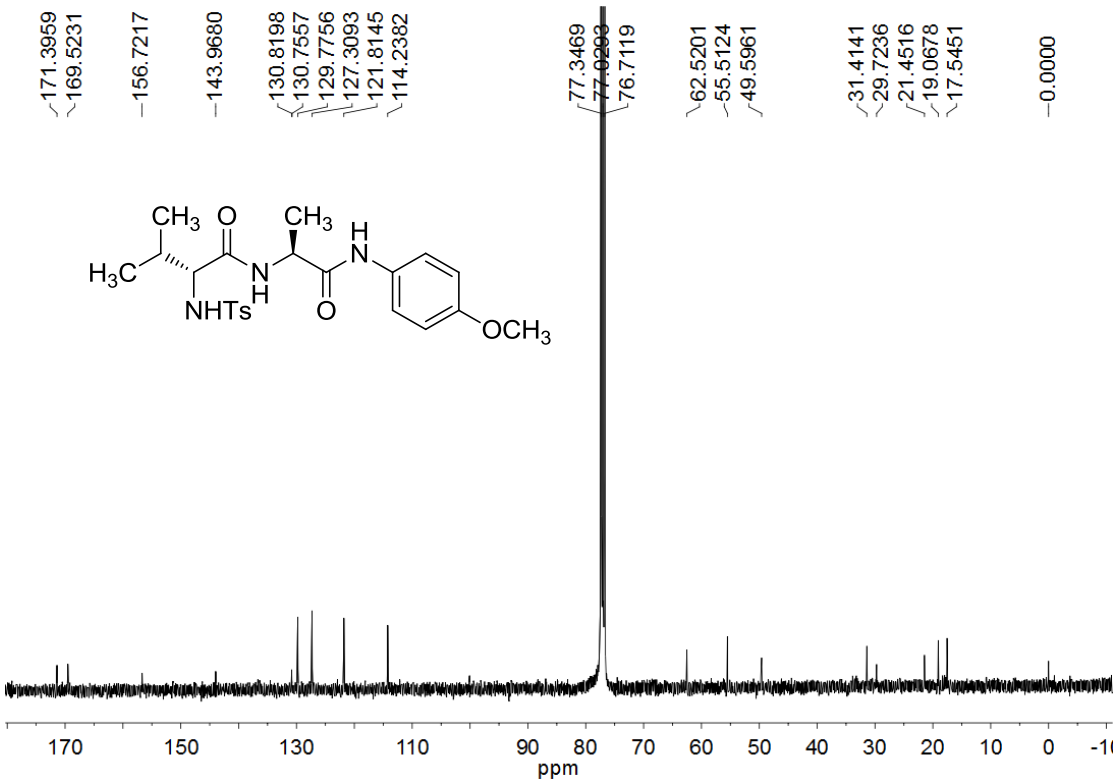


Figure S52. ^{13}C NMR spectrum of (*R*,*S*)-G**17** in $CDCl_3$ (100 MHz).

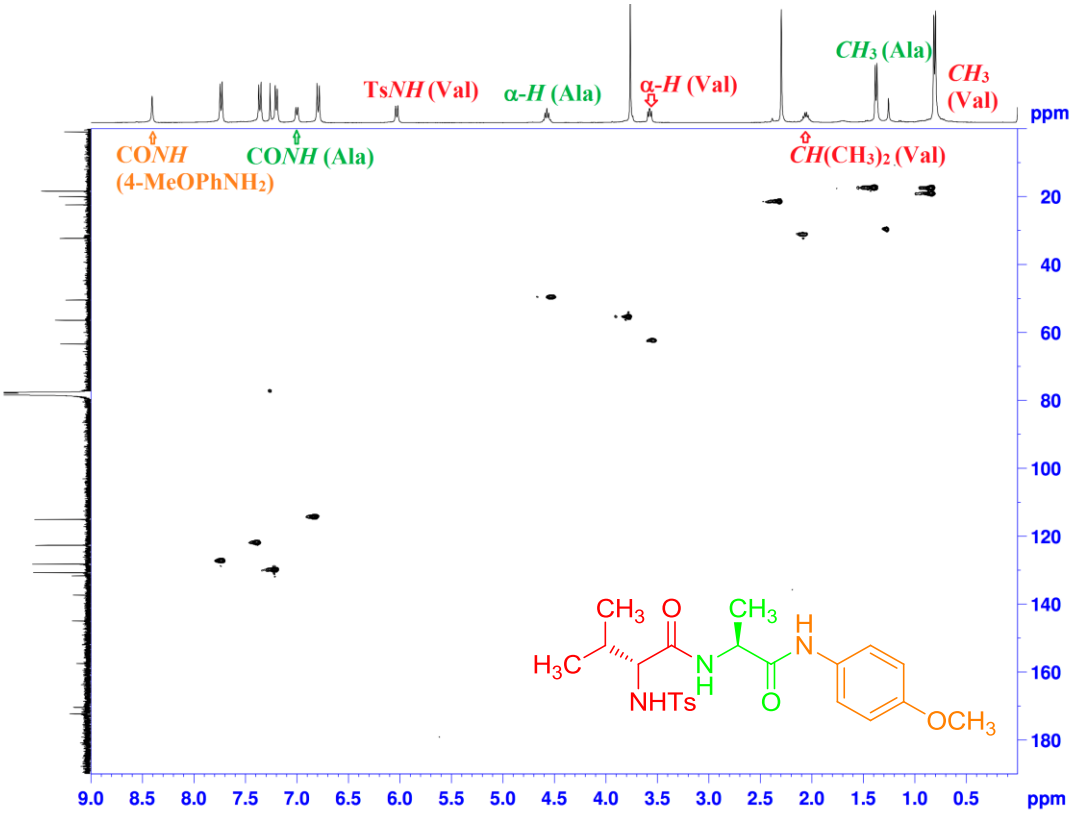


Figure S53. HSQC spectrum of (*R*,*S*)-G**17** in $CDCl_3$.

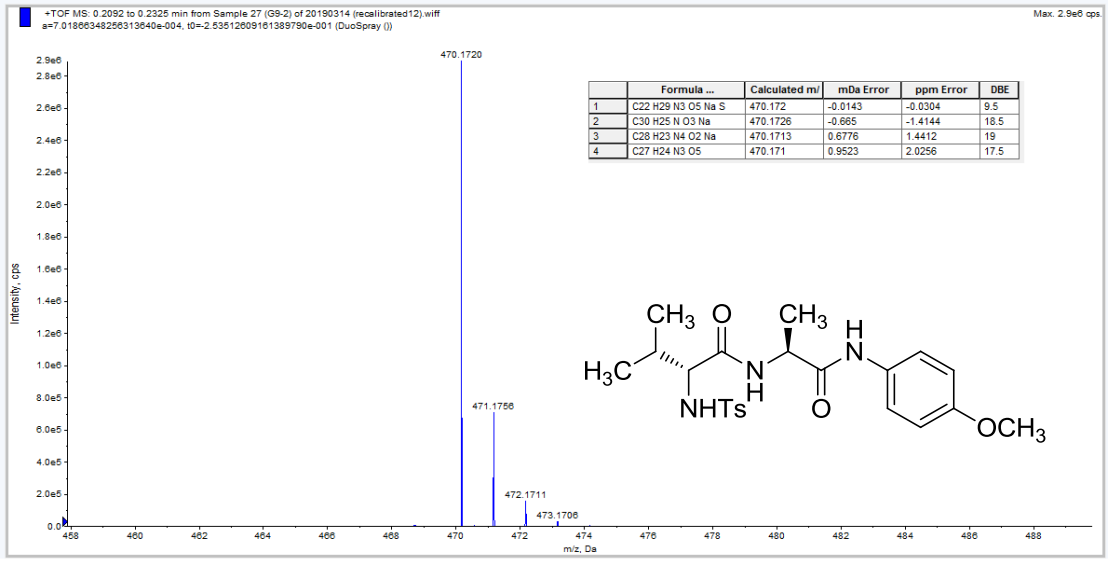


Figure S54. HRMS spectrum of (*R*,*S*)-G**17**.

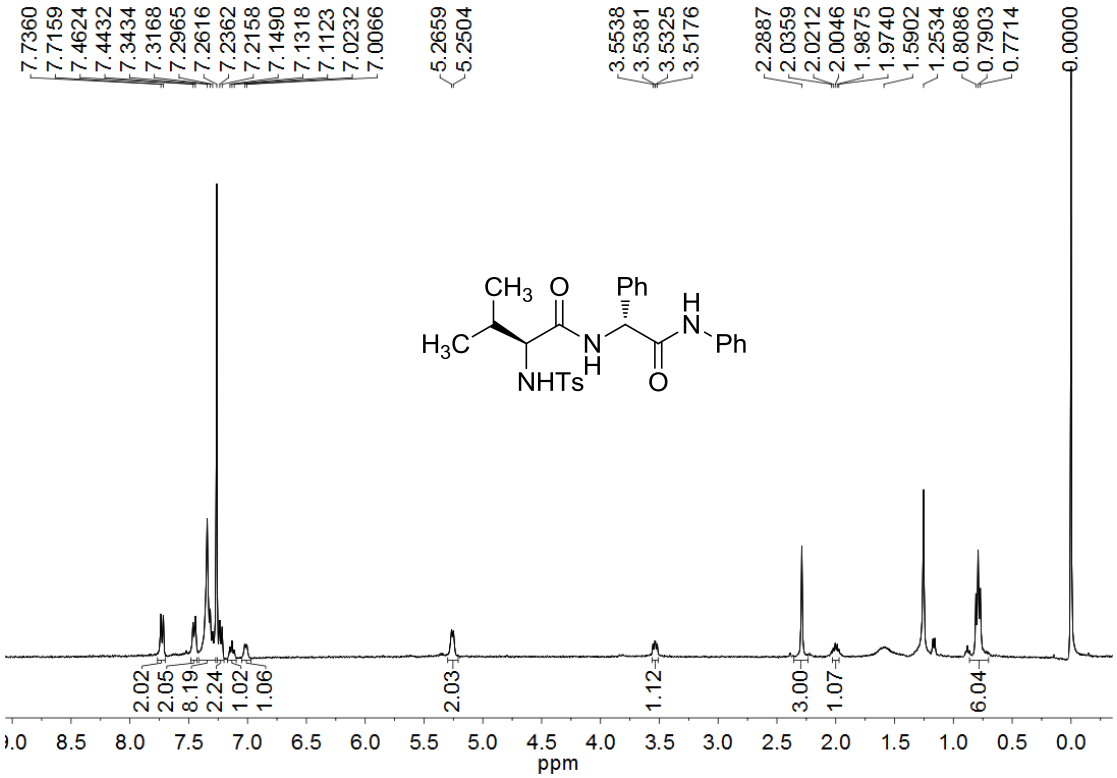


Figure S55. ^{1}H NMR spectrum of (*S*,*R*)-G**18** in $CDCl_3$ (400 MHz).

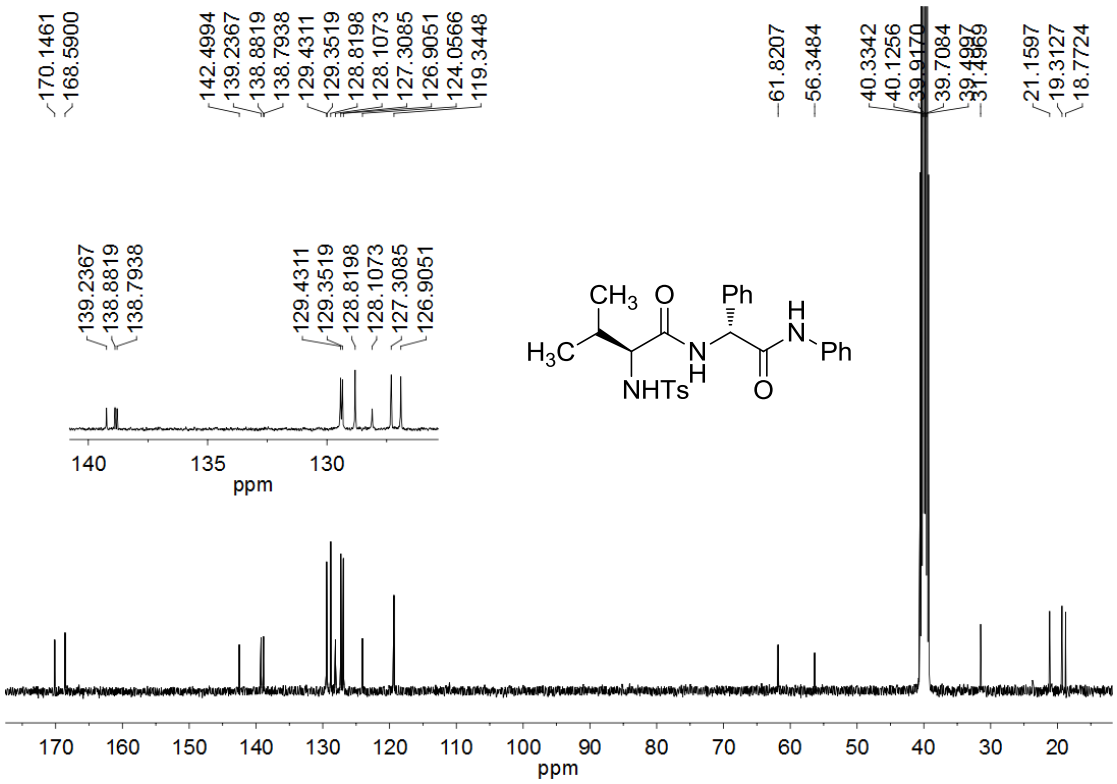


Figure S56. ^{13}C NMR spectrum of (*S*,*R*)-G**18** in DMSO-d_6 (100 MHz).

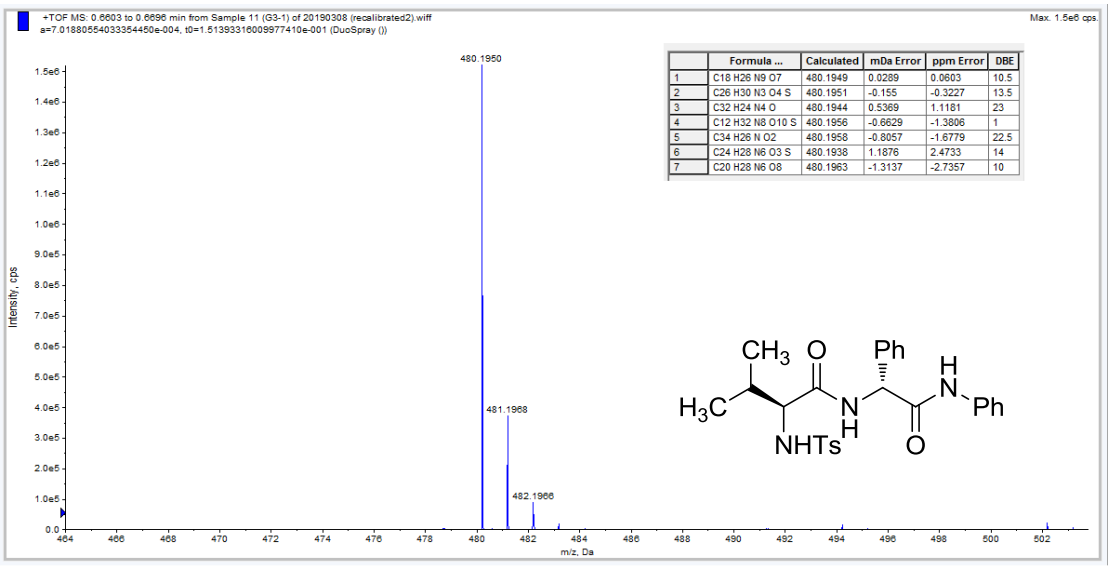


Figure S57. HRMS spectrum of (*S*,*R*)-G**18**.

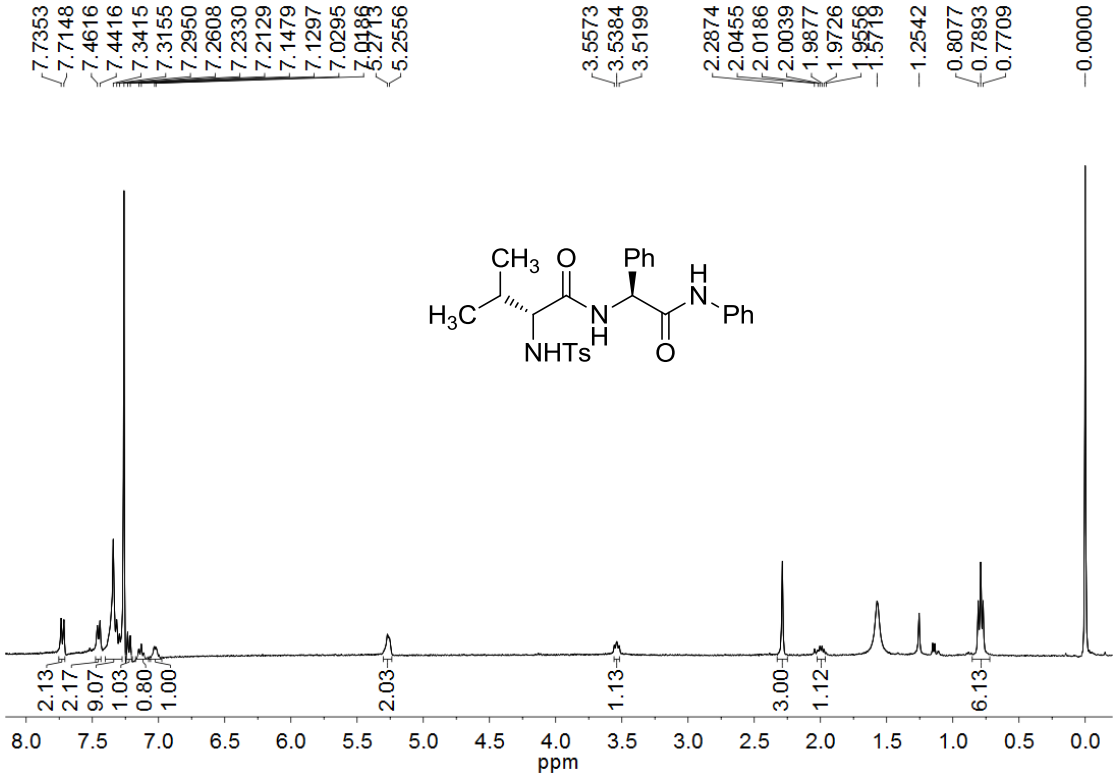


Figure S58. ^{1}H NMR spectrum of (*R*,*S*)-G**18** in $CDCl_3$ (400 MHz).

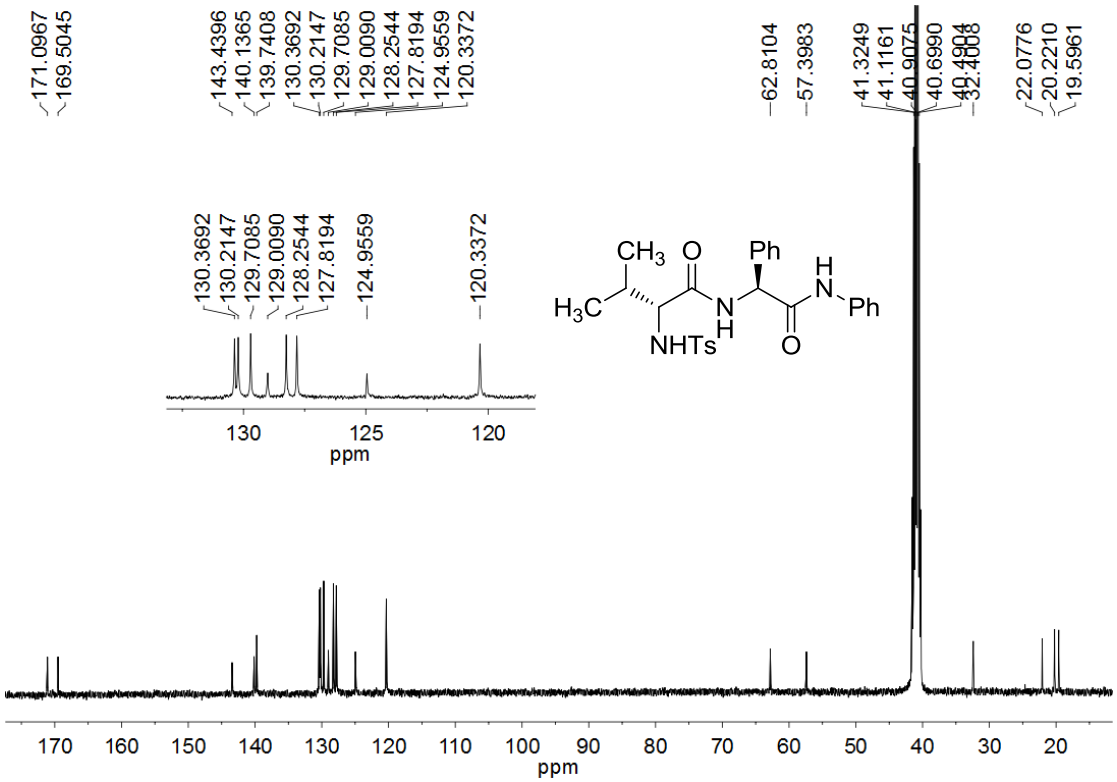


Figure S59. ^{13}C NMR spectrum of (*R*,*S*)-G**18** in DMSO-d_6 (100 MHz).

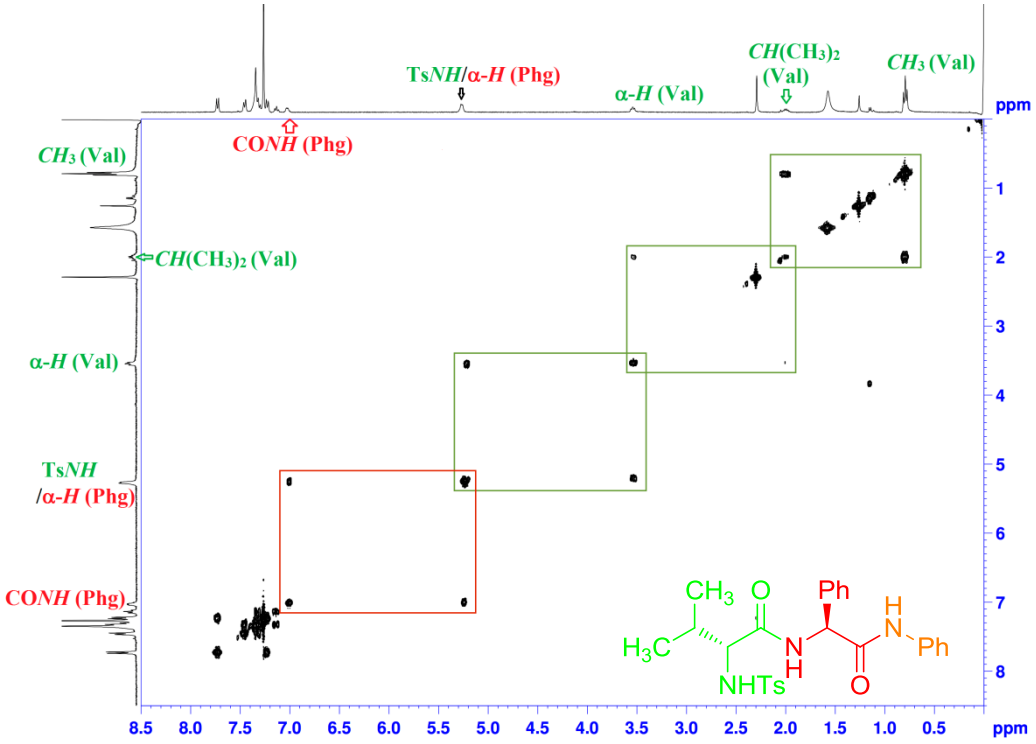


Figure S60. ^{1}H–^{1}H COSY spectrum of (*R*,*S*)-G**18** in $CDCl_3$ (400 MHz).

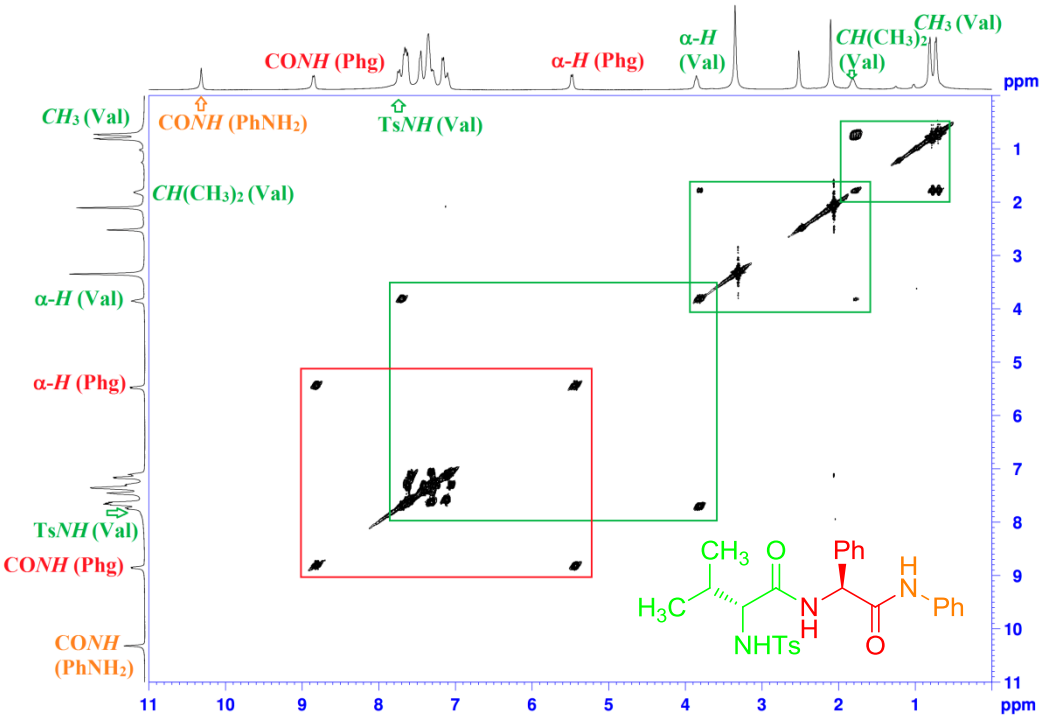


Figure S61. ^{1}H–^{1}H COSY spectrum of (*R*,*S*)-G**18** in DMSO-d_6 (400 MHz).

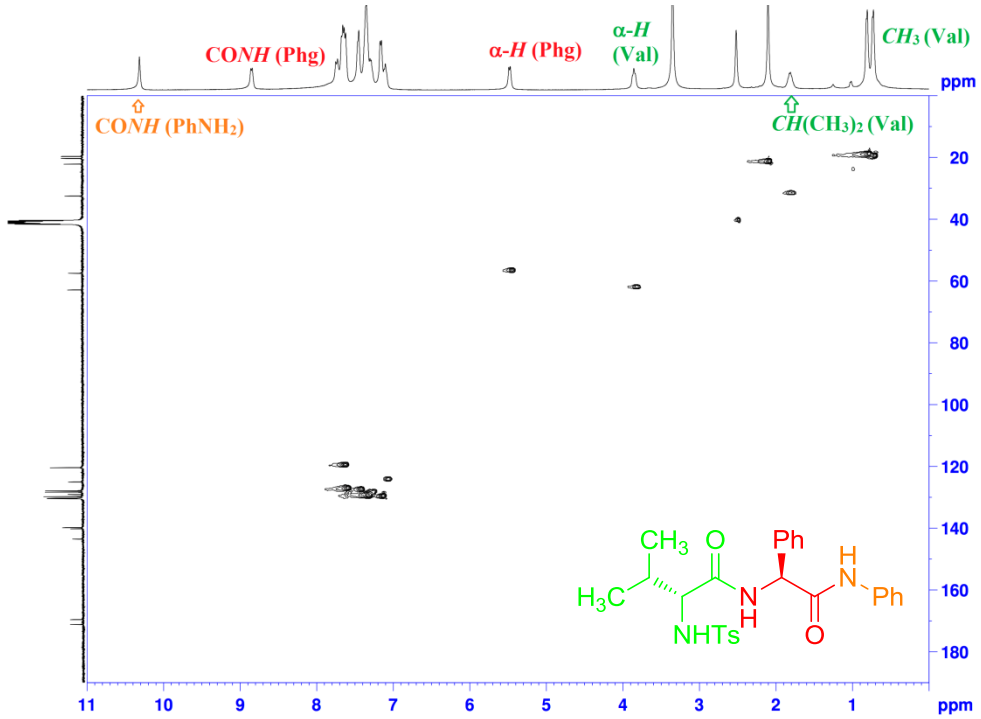


Figure S62. HSQC spectrum of (*R*,*S*)-G**18** in DMSO-d_6.

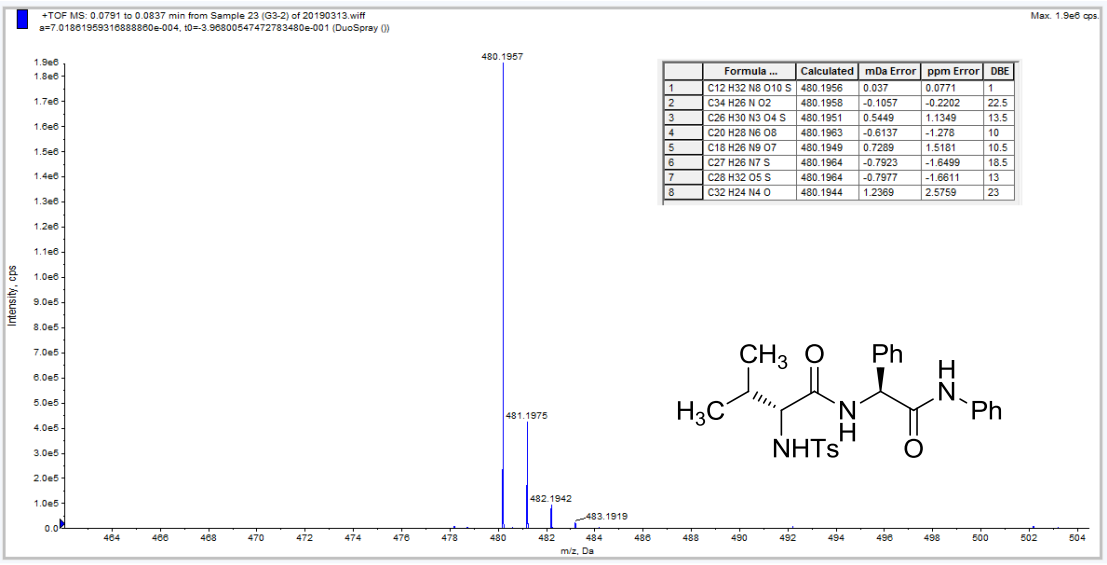


Figure S63. HRMS spectrum of (*R*,*S*)-G**18**.

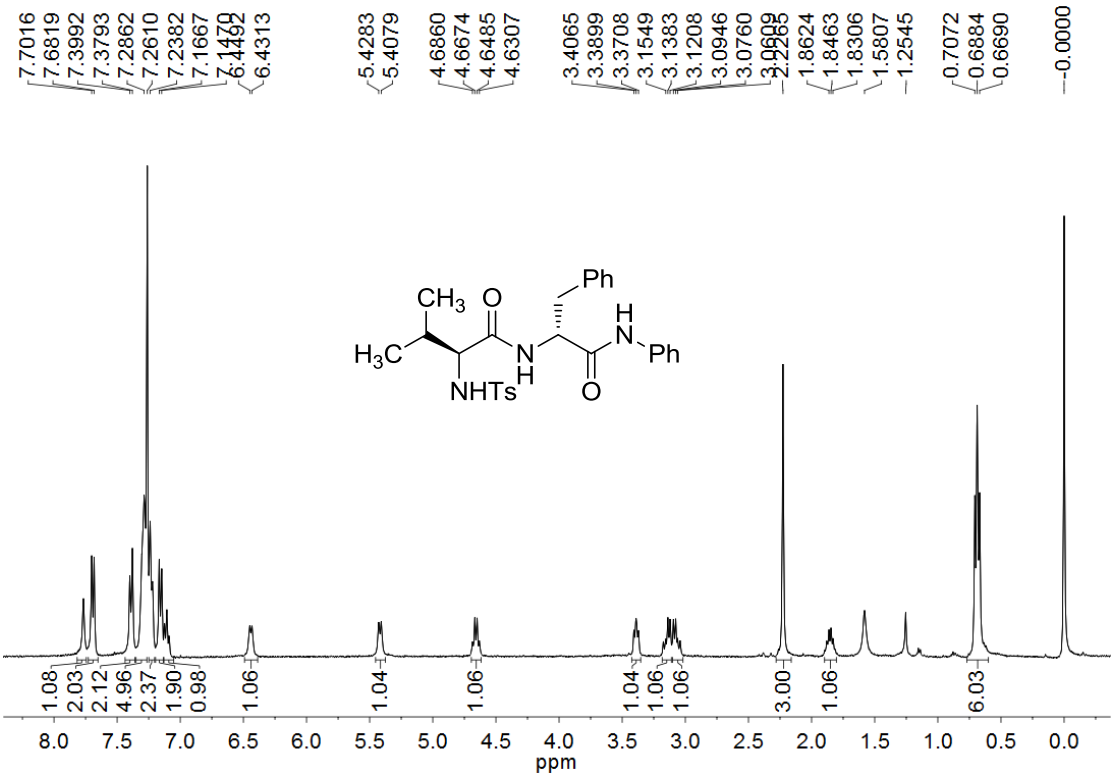


Figure S64. ^{1}H NMR spectrum of (*S*,*R*)-G**19** in $CDCl_3$ (400 MHz).

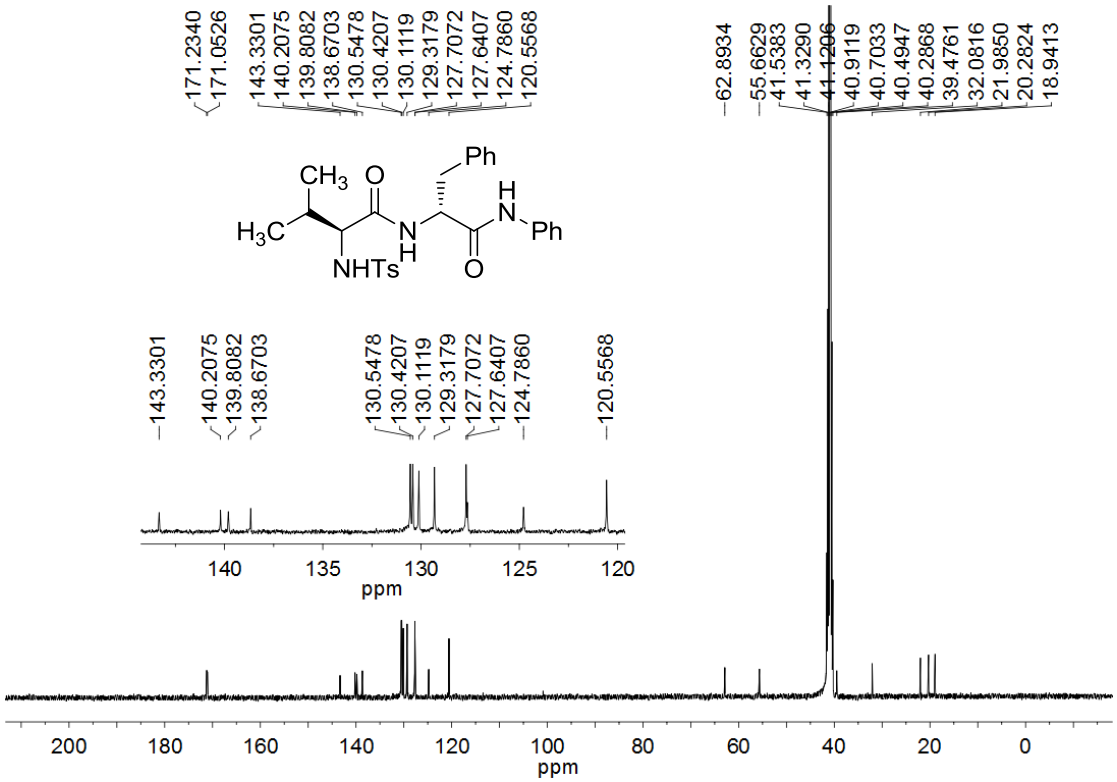


Figure S65. ^{13}C NMR spectrum of (*S*,*R*)-G**19** in DMSO-d_6 (100 MHz).

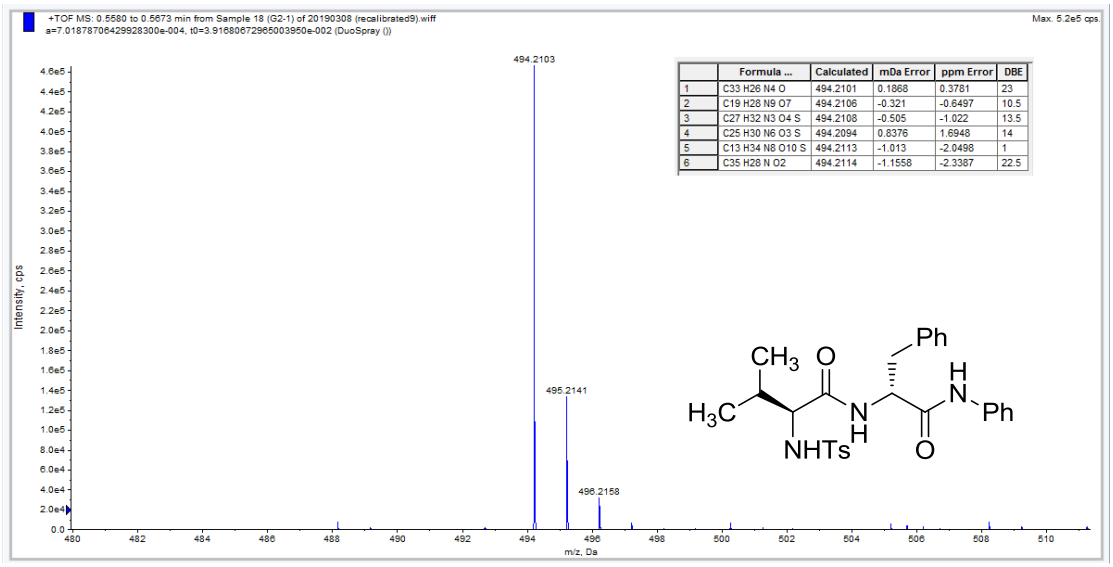


Figure S66. HRMS spectrum of (*S*,*R*)-G**19**.

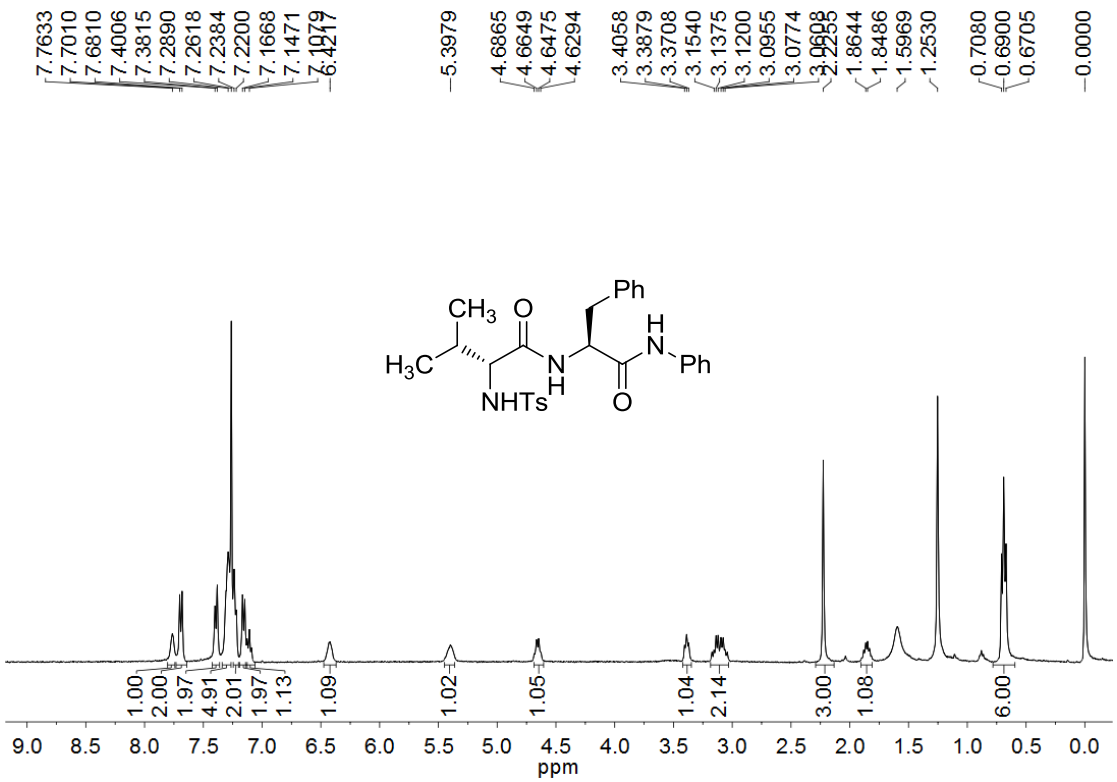


Figure S67. ^{1}H NMR spectrum of (*R*,*S*)-G**19** in $CDCl_3$ (400 MHz).

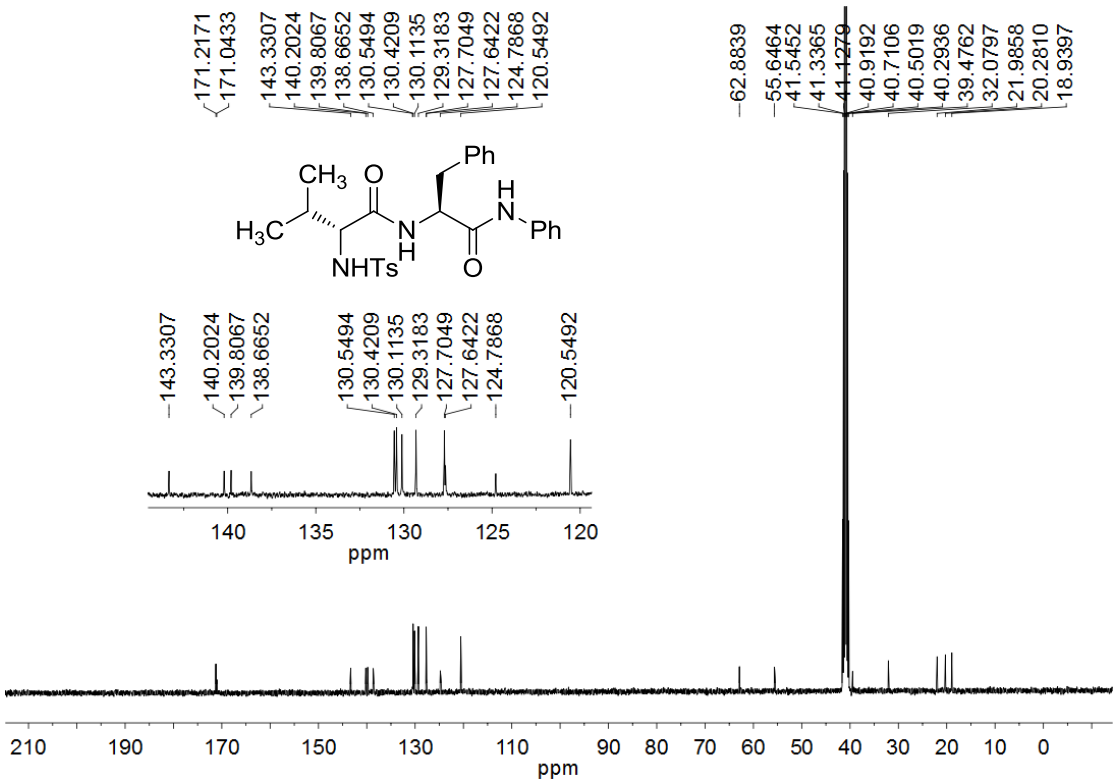


Figure S68. ^{13}C NMR spectrum of (*R*,*S*)-G**19** in DMSO-d_6 (100 MHz).

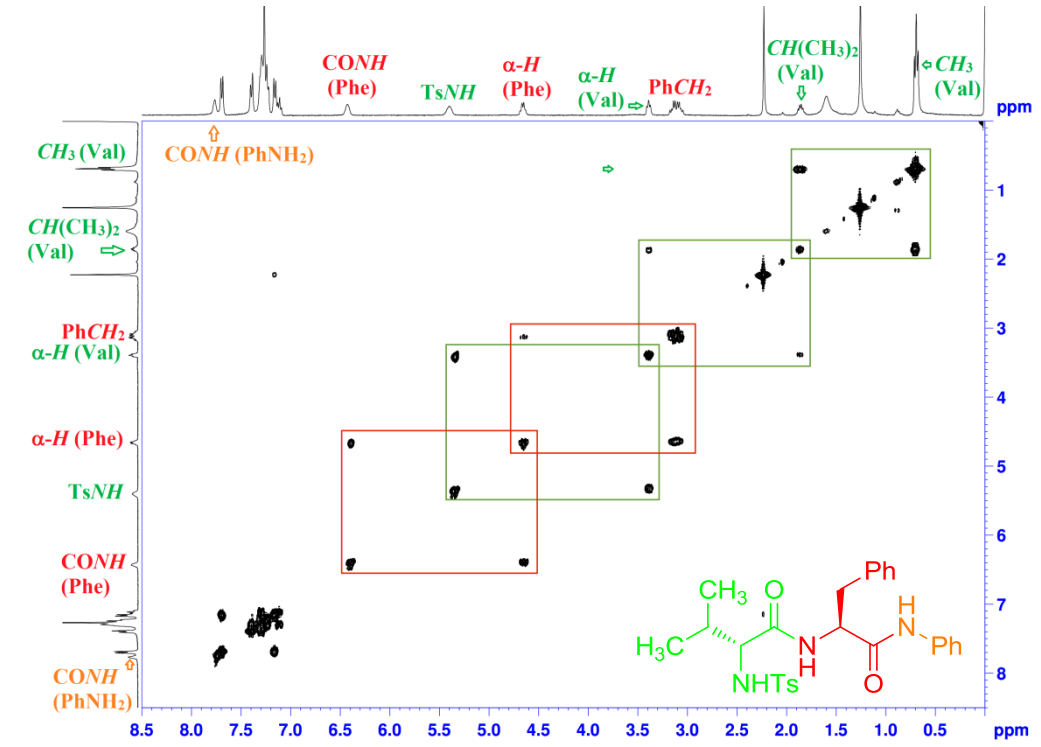


Figure S69. ^{1}H–^{1}H COSY spectrum of (*R*,*S*)-G**19** in $CDCl_3$ (400 MHz).

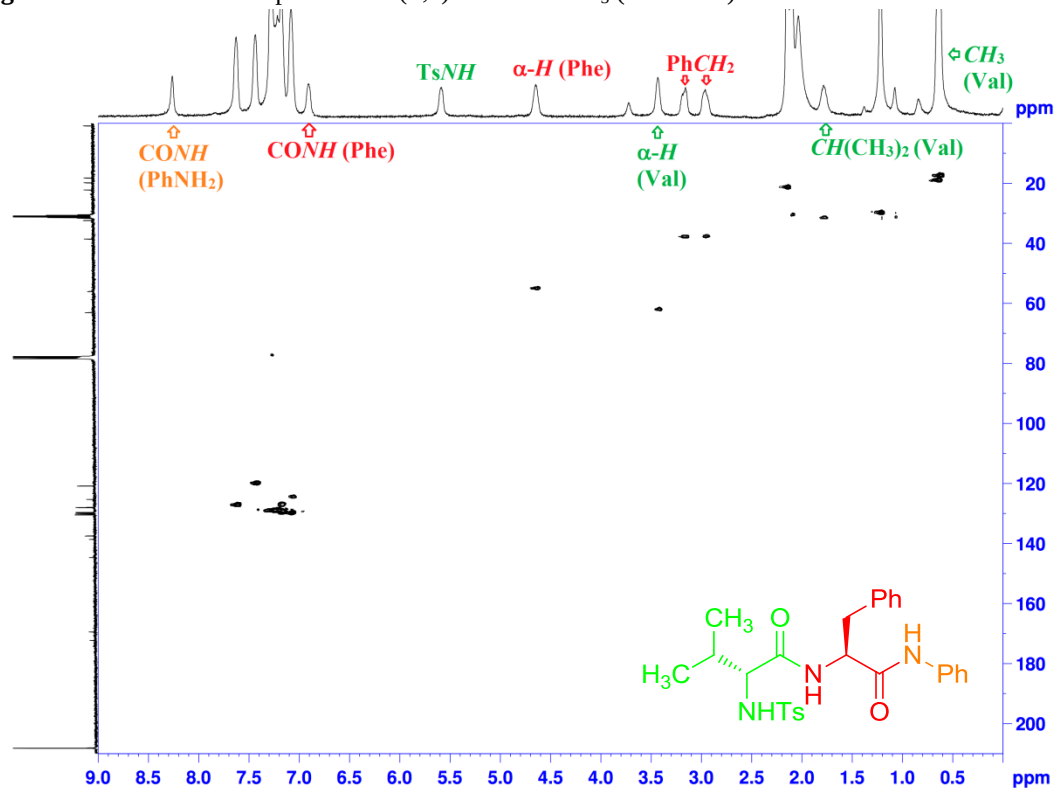


Figure S70. HSQC spectrum of (*R*,*S*)-G**19** in $CDCl_3$ containing 10% acetone-d_6.

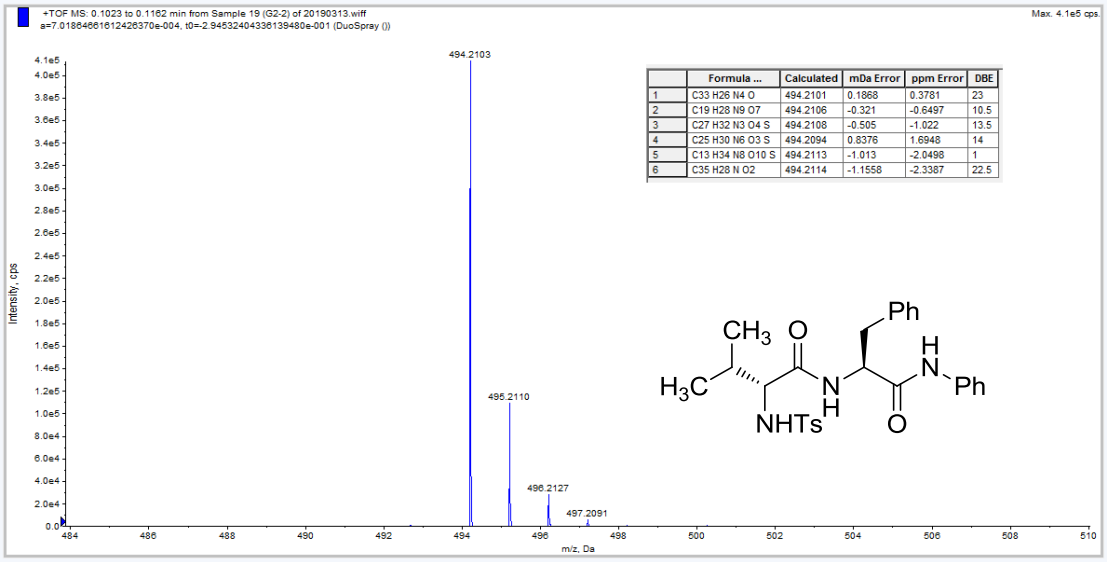


Figure S71. HRMS spectrum of (*R*,*S*)-G**19**.

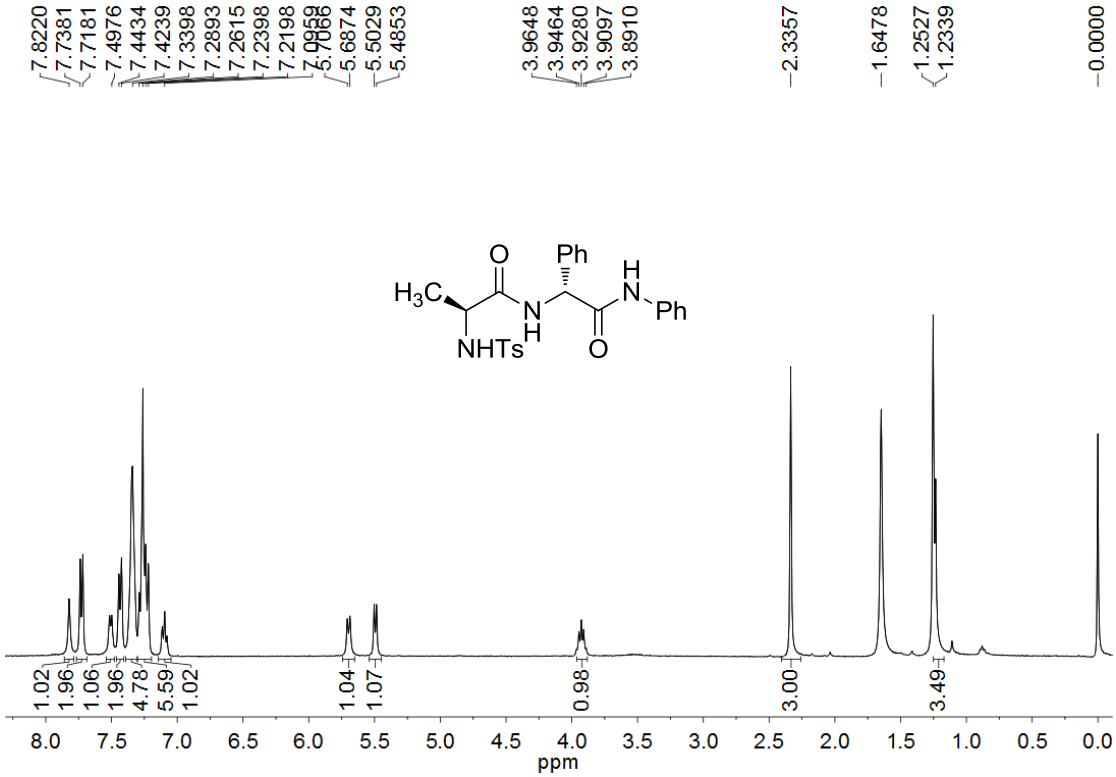


Figure S72. ^{1}H NMR spectrum of (*S*,*R*)-G**20** in $CDCl_3$ (400 MHz).

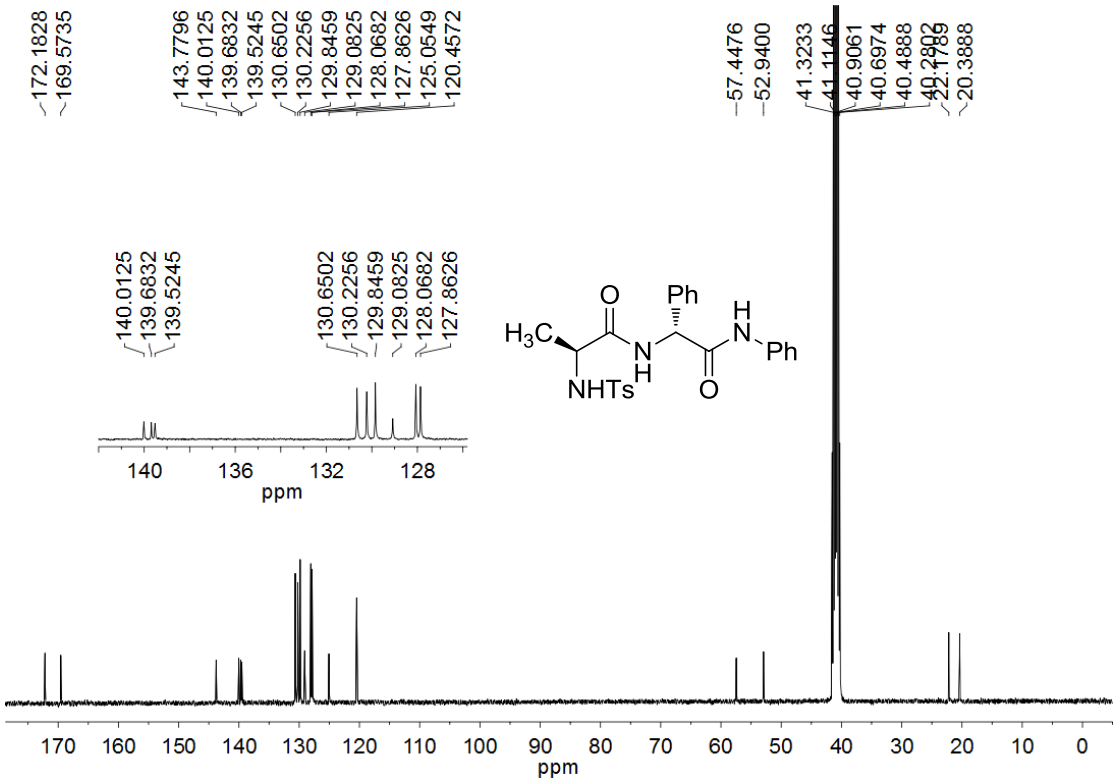


Figure S73. ^{13}C NMR spectrum of (*S*,*R*)-G**20** in DMSO-d_6 (100 MHz).

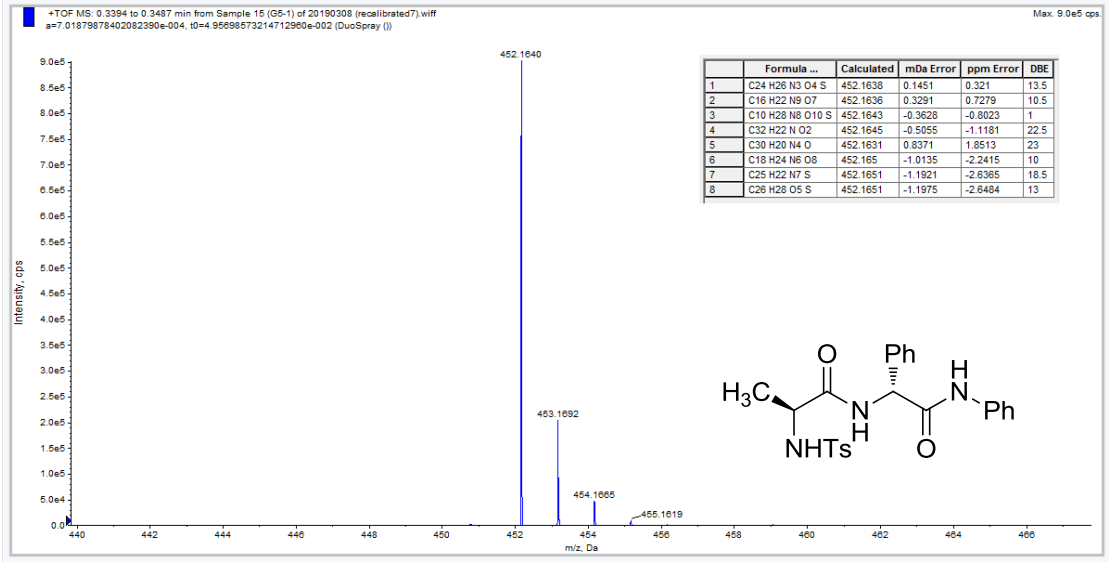


Figure S74. HRMS spectrum of (*S*,*R*)-G**20**.

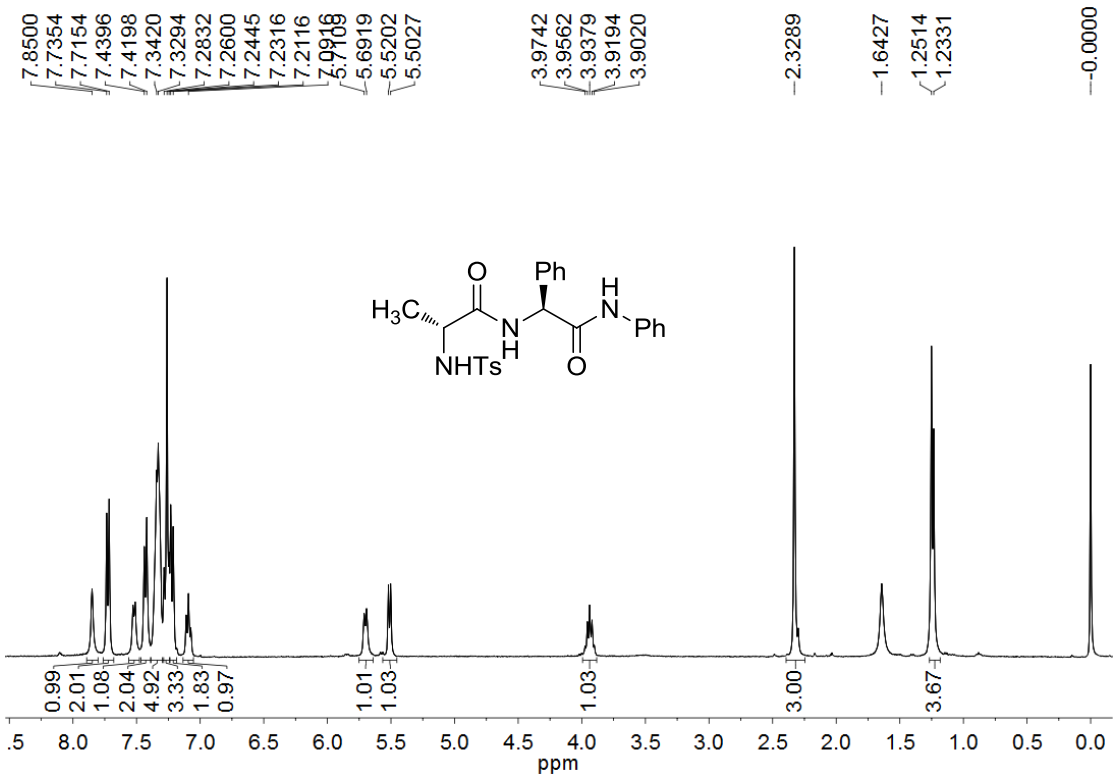


Figure S75. ^{1}H NMR spectrum of (*R*,*S*)-G**20** in $CDCl_3$ (400 MHz).

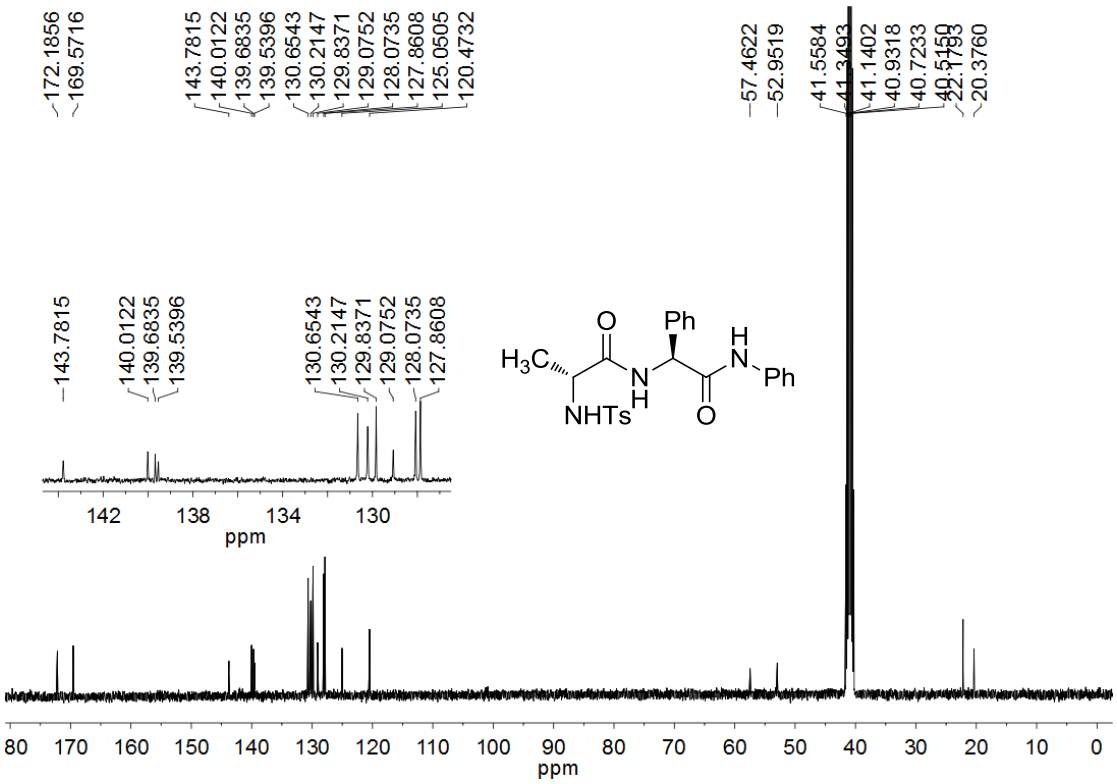


Figure S76. ^{13}C NMR spectrum of (*R*,*S*)-G**20** in DMSO-d_6.

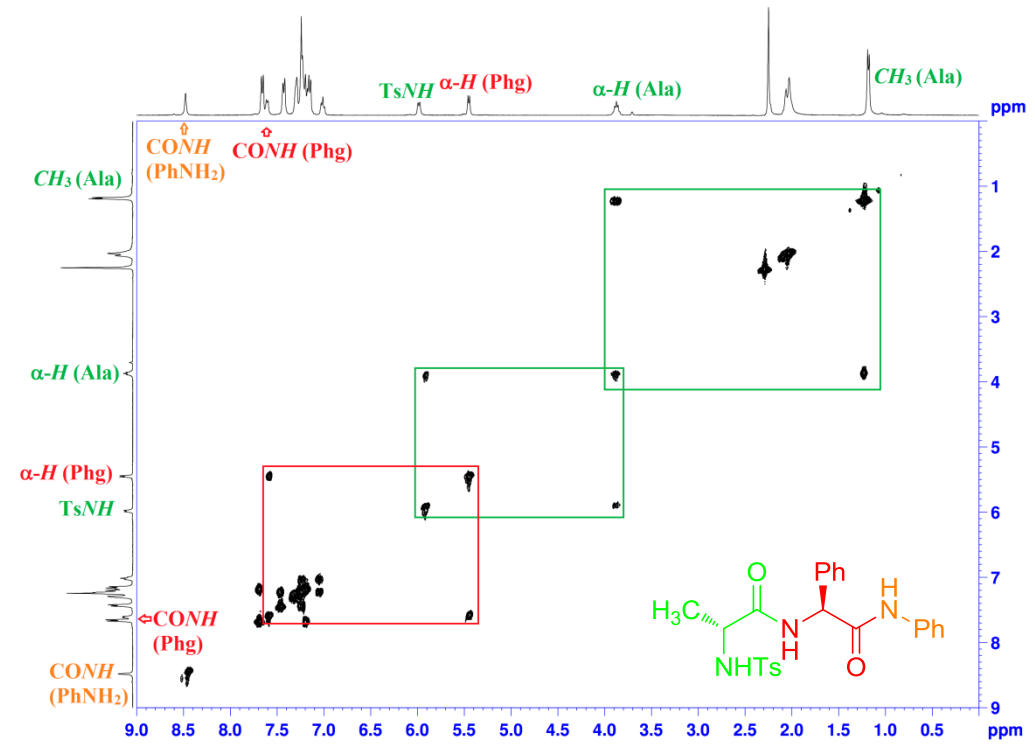


Figure S77. ^{1}H–^{1}H COSY spectrum of (*R*,*S*)-G**20** in $CDCl_3$ containing 10% acetone-d_6 (400 MHz).

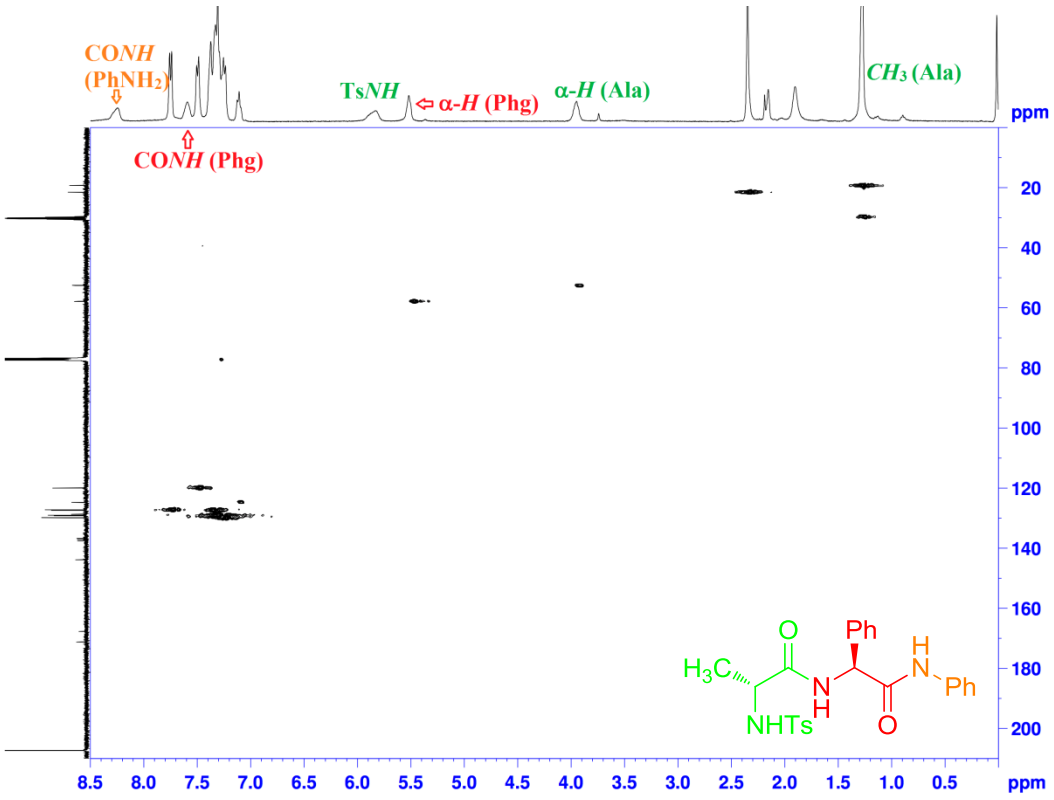


Figure S78. HSQC spectrum of (*R*,*S*)-G**20** in $CDCl_3$ containing 10 % acetone-d_6.

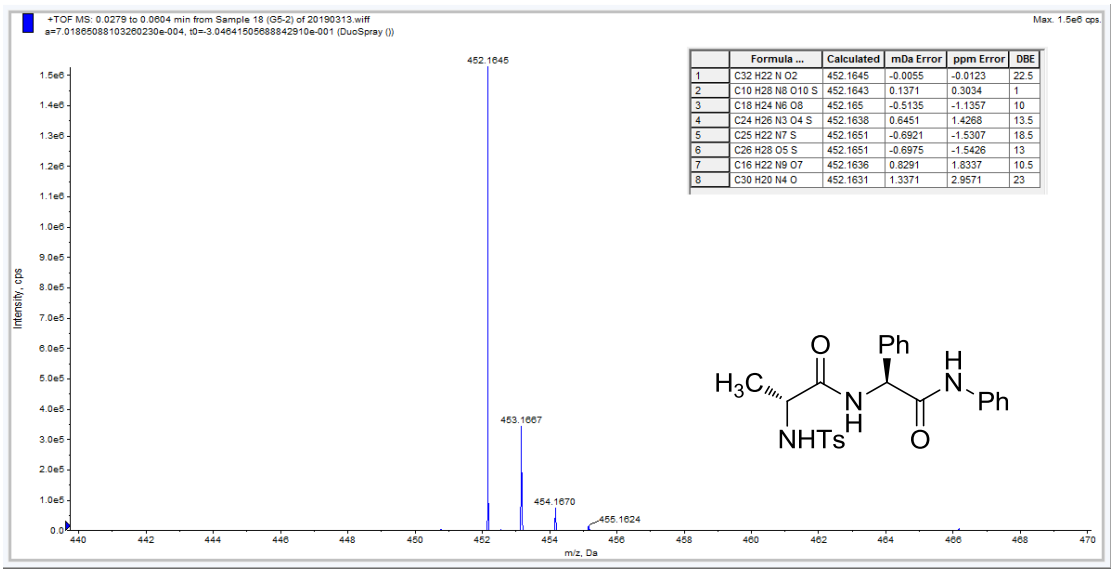


Figure S79. HRMS spectrum of (*R*,*S*)-G**20**.

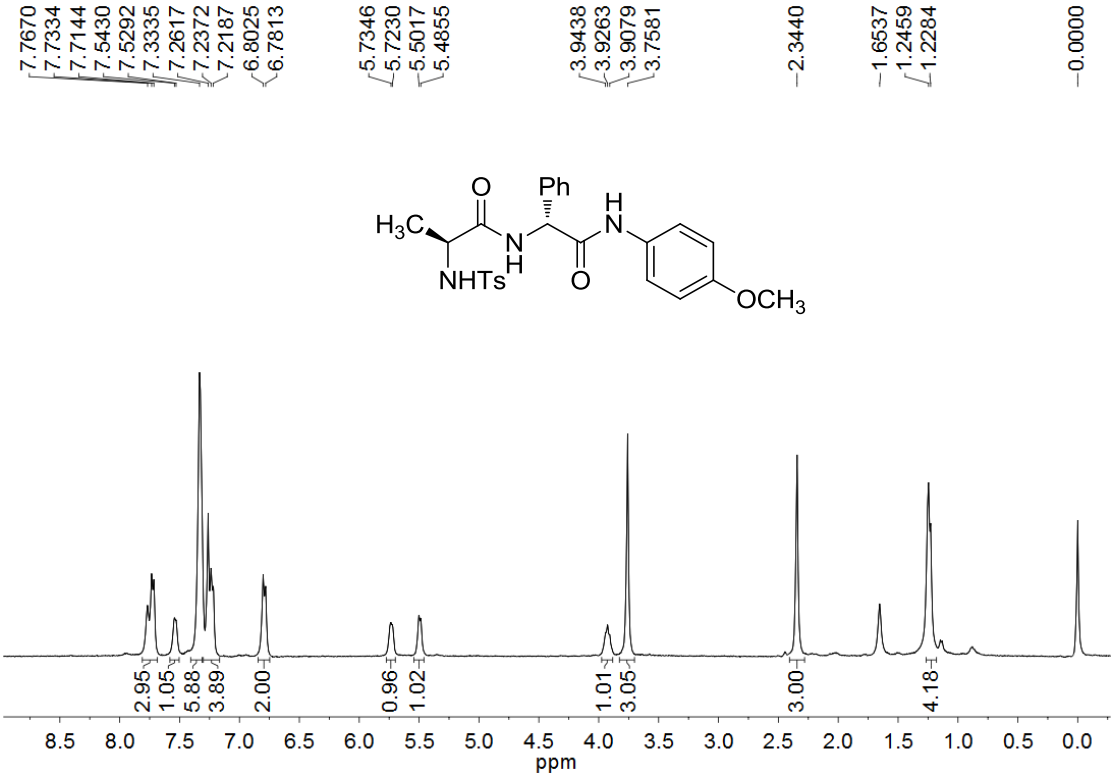


Figure S80. ^{1}H NMR spectrum of (*S*,*R*)-G**21** in $CDCl_3$ (400 MHz).

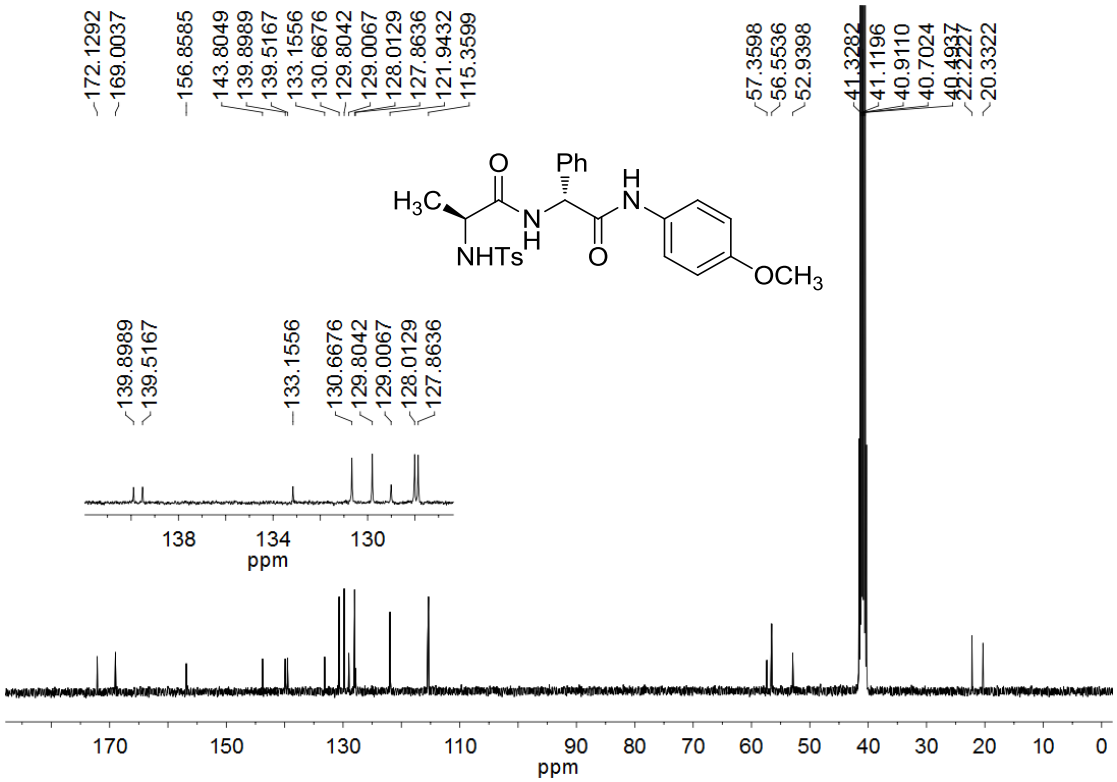


Figure S81. ^{13}C NMR spectrum of (*S*,*R*)-G**21** in DMSO-d_6 (100 MHz).

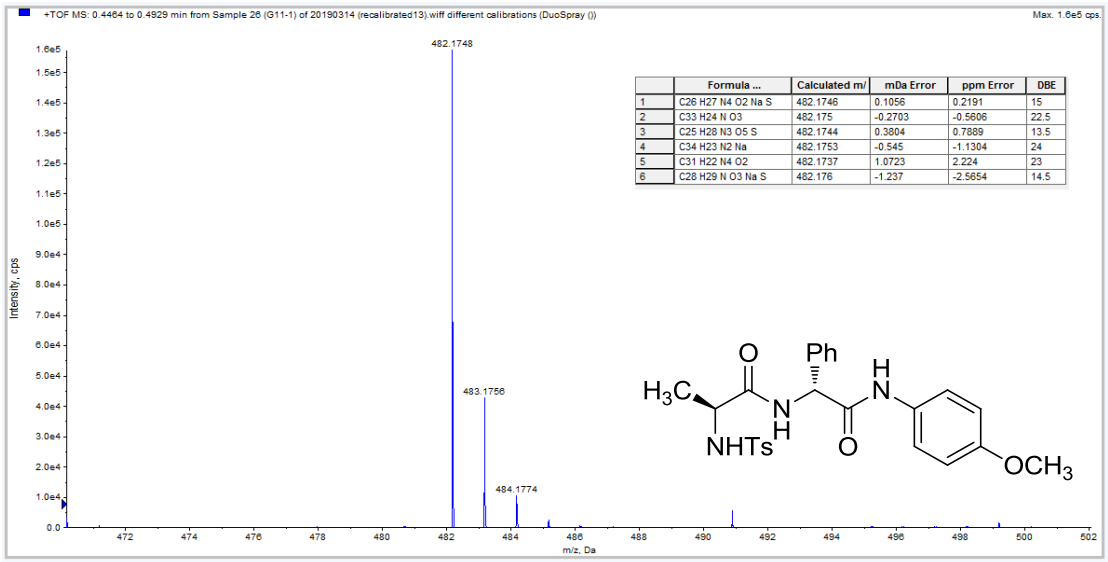


Figure S82. HRMS spectrum of (*S*,*R*)-G**21**.

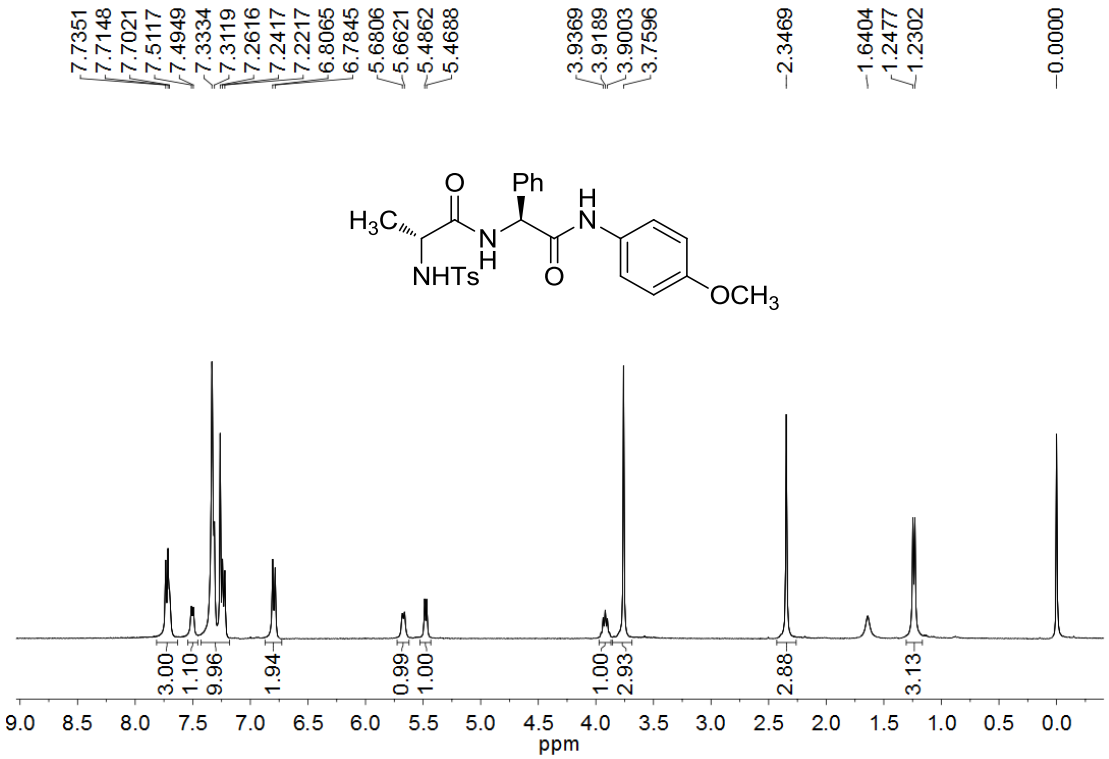


Figure S83. ^{1}H NMR spectrum of (*R*,*S*)-G**21** in $CDCl_3$ (400 MHz).

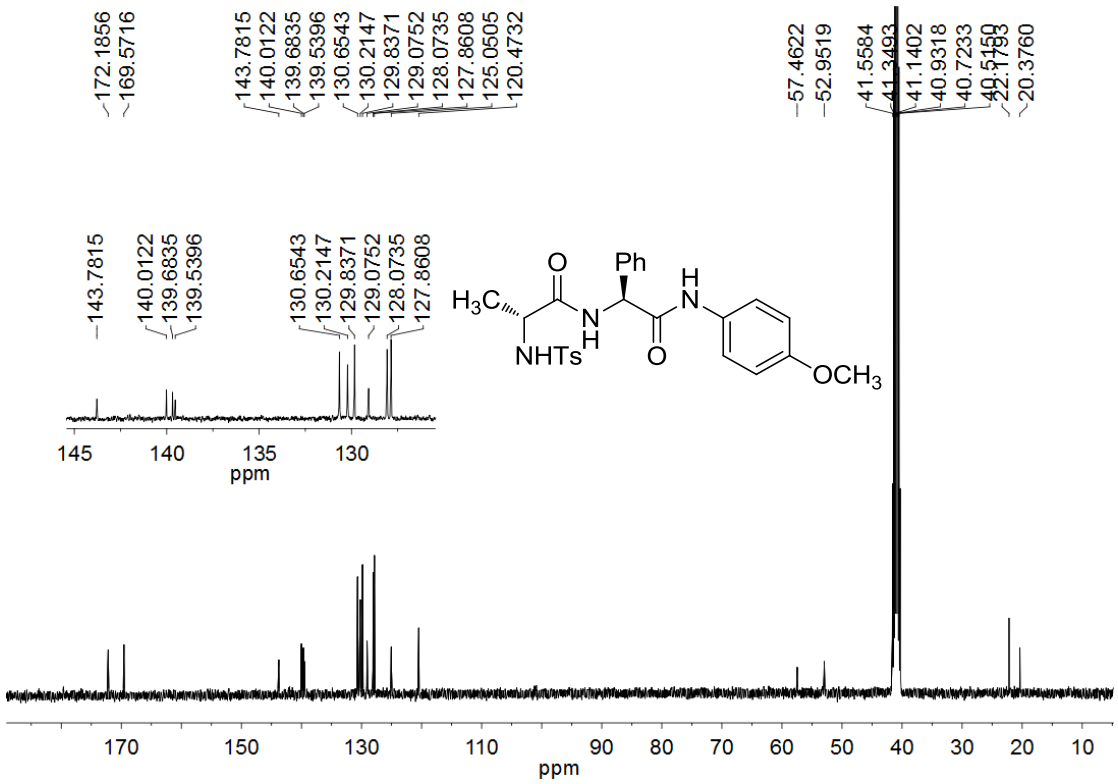


Figure S84. ^{13}C NMR spectrum of (*R*,*S*)-G**21** in DMSO-d_6 (100 MHz).

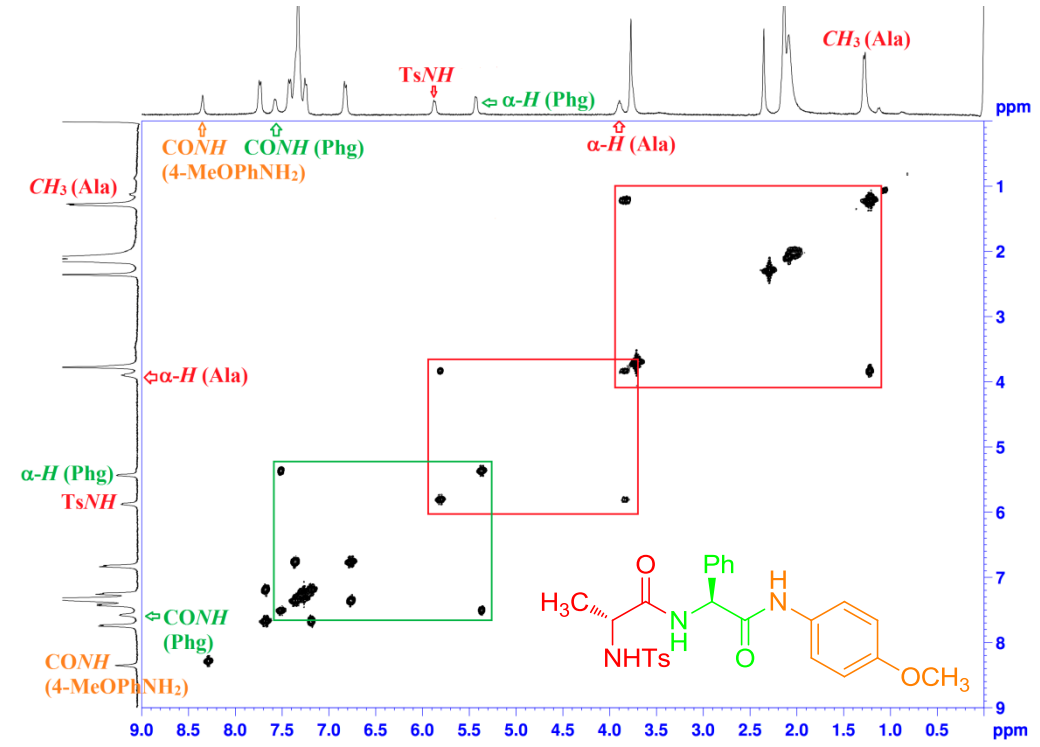


Figure S85. ^{1}H–^{1}H COSY spectrum of (*R*,*S*)-G**21** in $CDCl_3$ (5% acetone-d_6), (400 MHz).

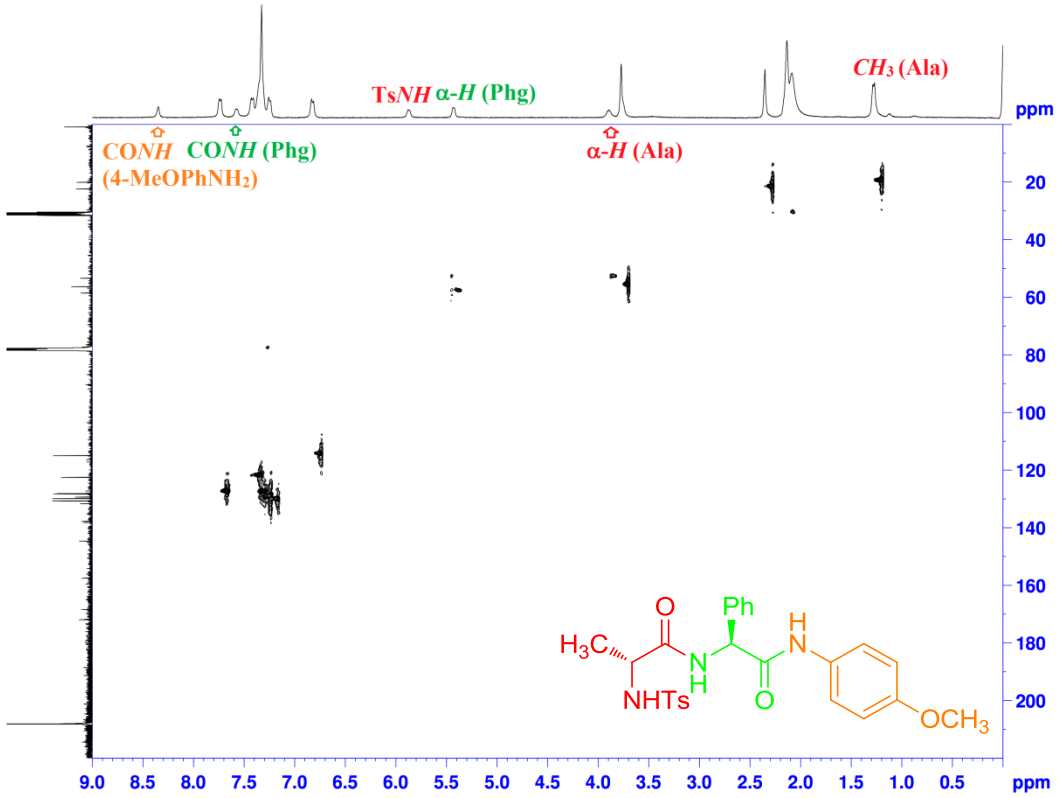


Figure S86. HSQC spectrum of (*R*,*S*)-G**21** in $CDCl_3$ (5% acetone-d_6).

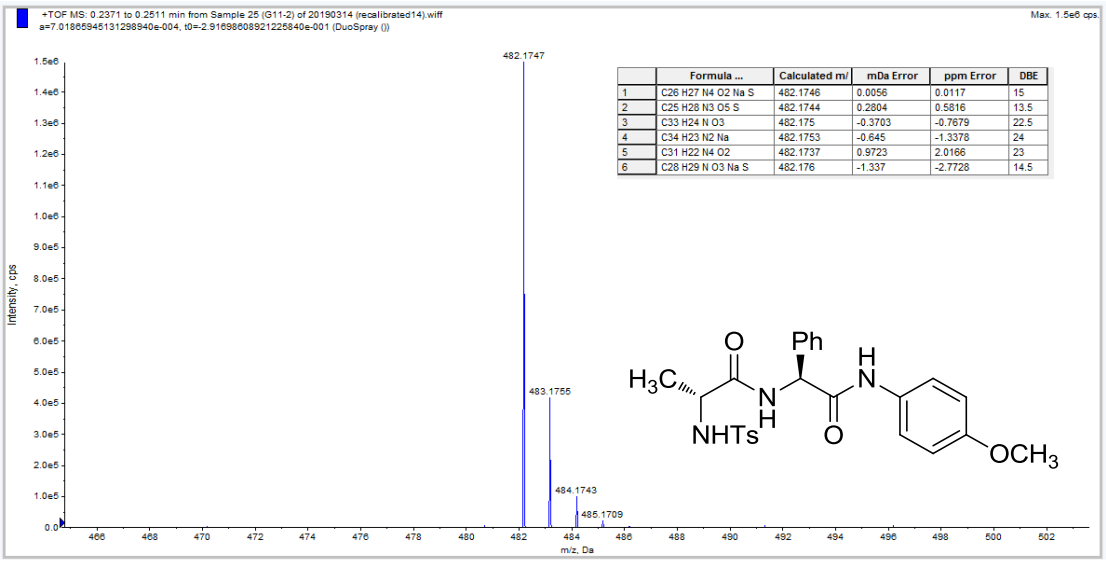


Figure S87. HRMS spectrum of (*R*,*S*)-G**21**.

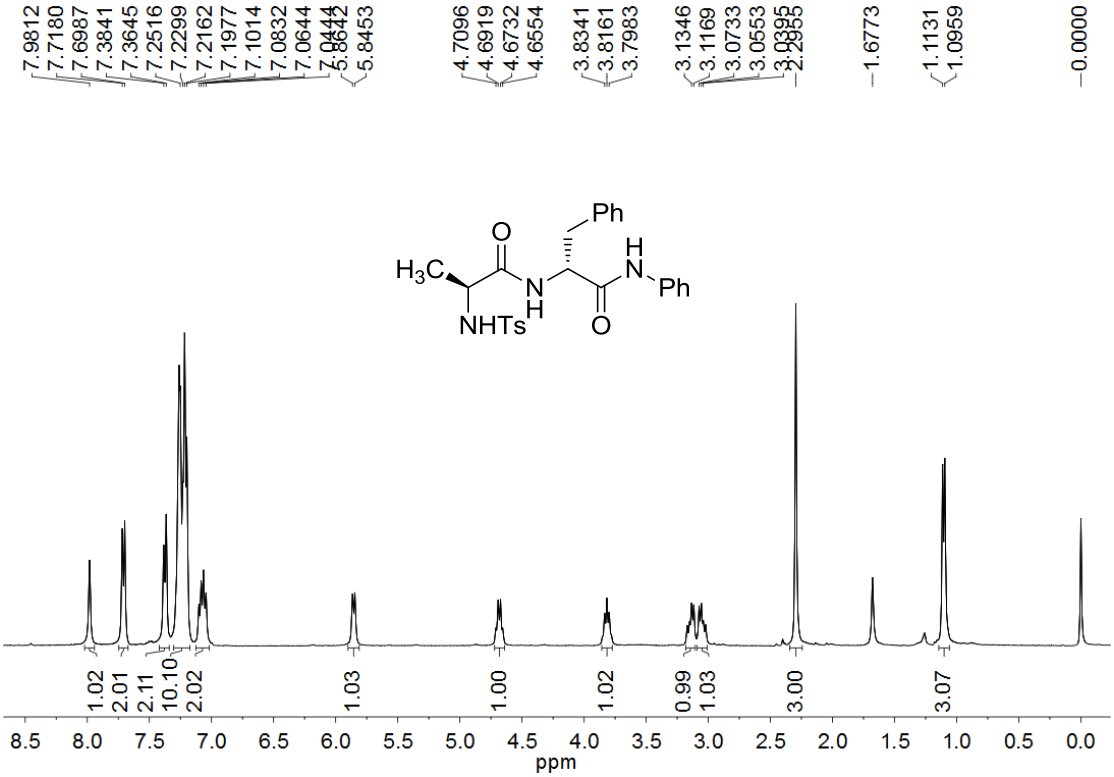


Figure S88. ^{1}H NMR spectrum of (*S*,*R*)-G**22** in $CDCl_3$ (400 MHz).

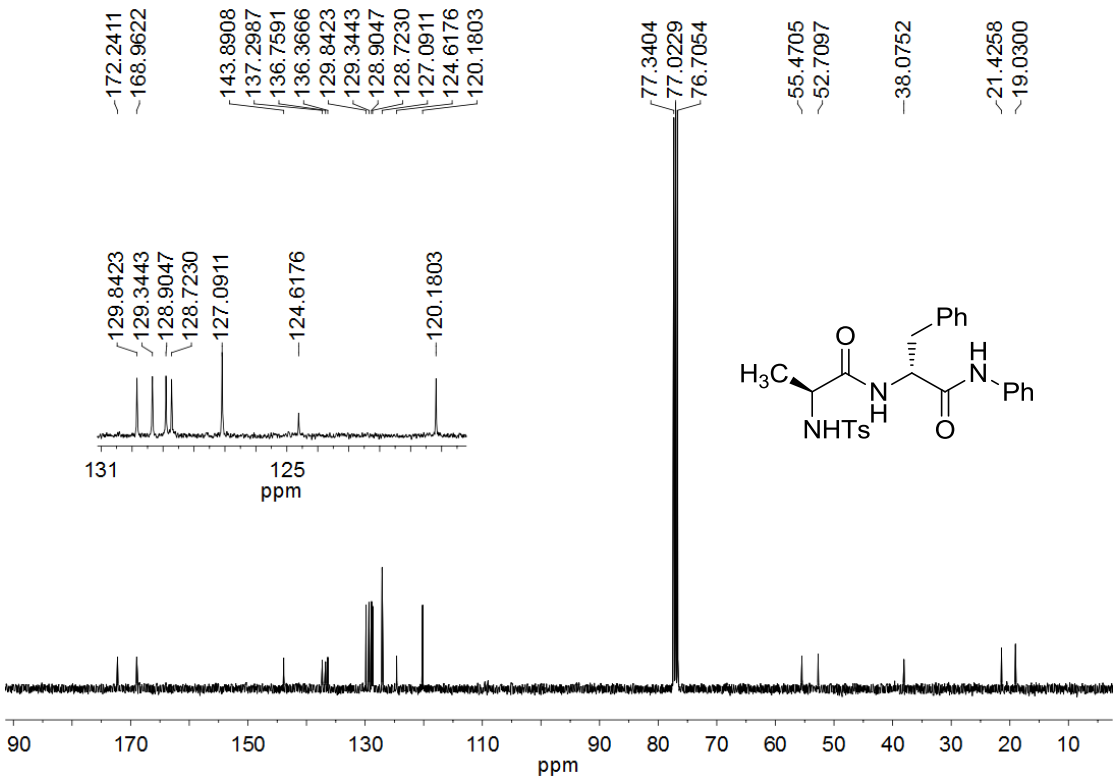


Figure S89. ^{13}C NMR spectrum of (*S*,*R*)-G**22** in $CDCl_3$ (100 MHz).

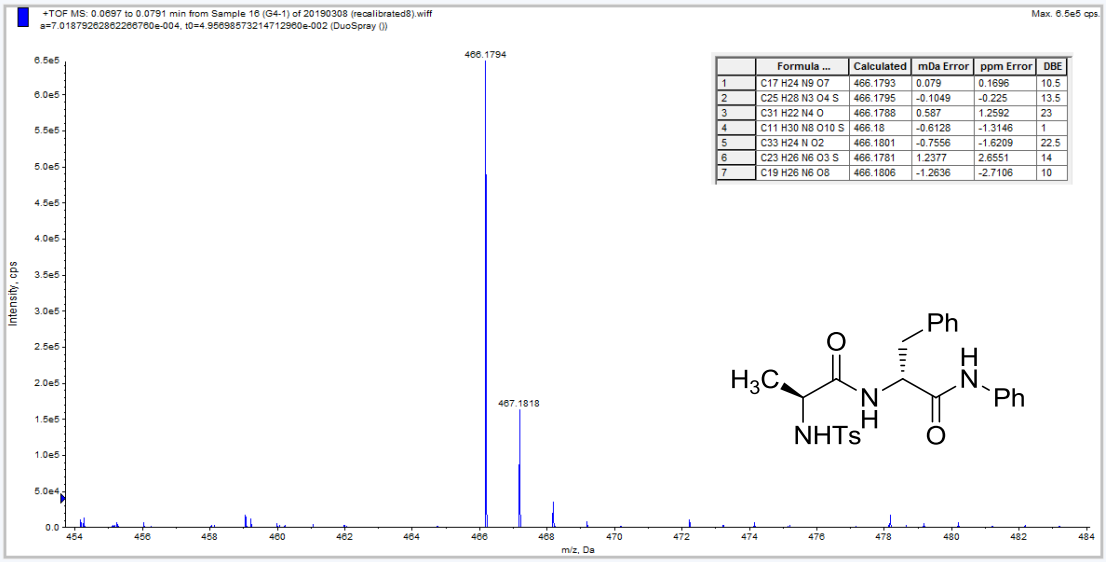


Figure S90. HRMS spectrum of (*S*,*R*)-G**22**.

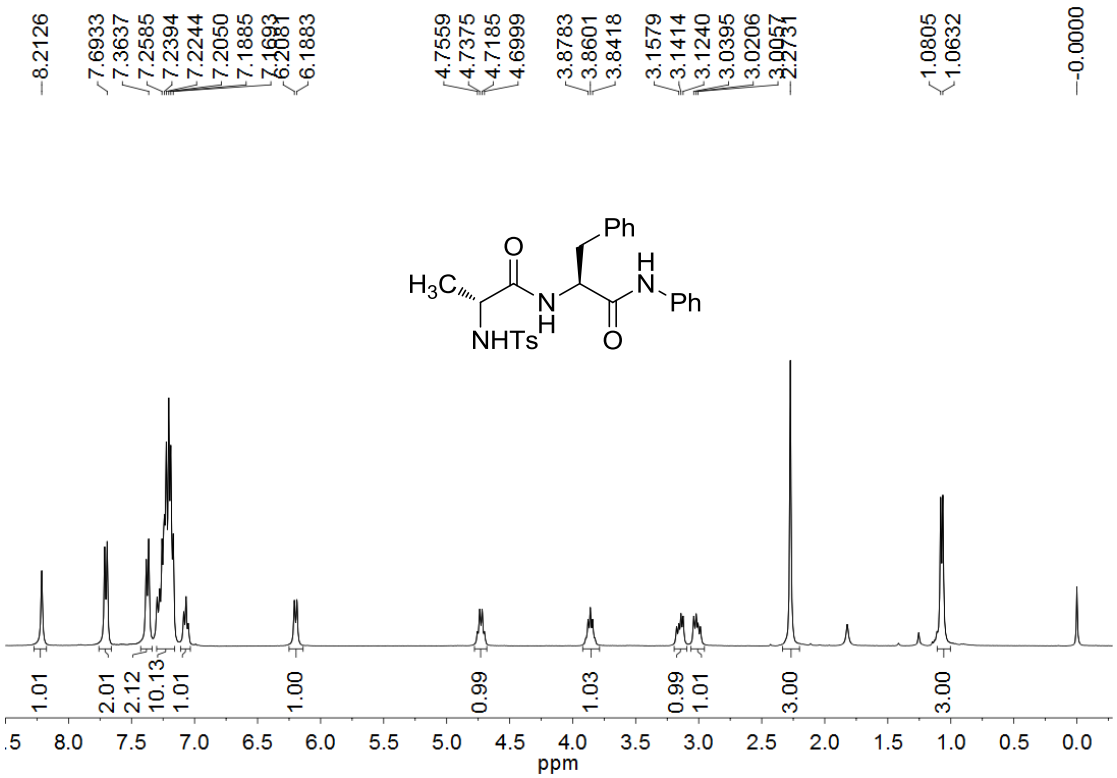


Figure S91. ^{1}H NMR spectrum of (*R*,*S*)-G**22** in $CDCl_3$ (400 MHz).

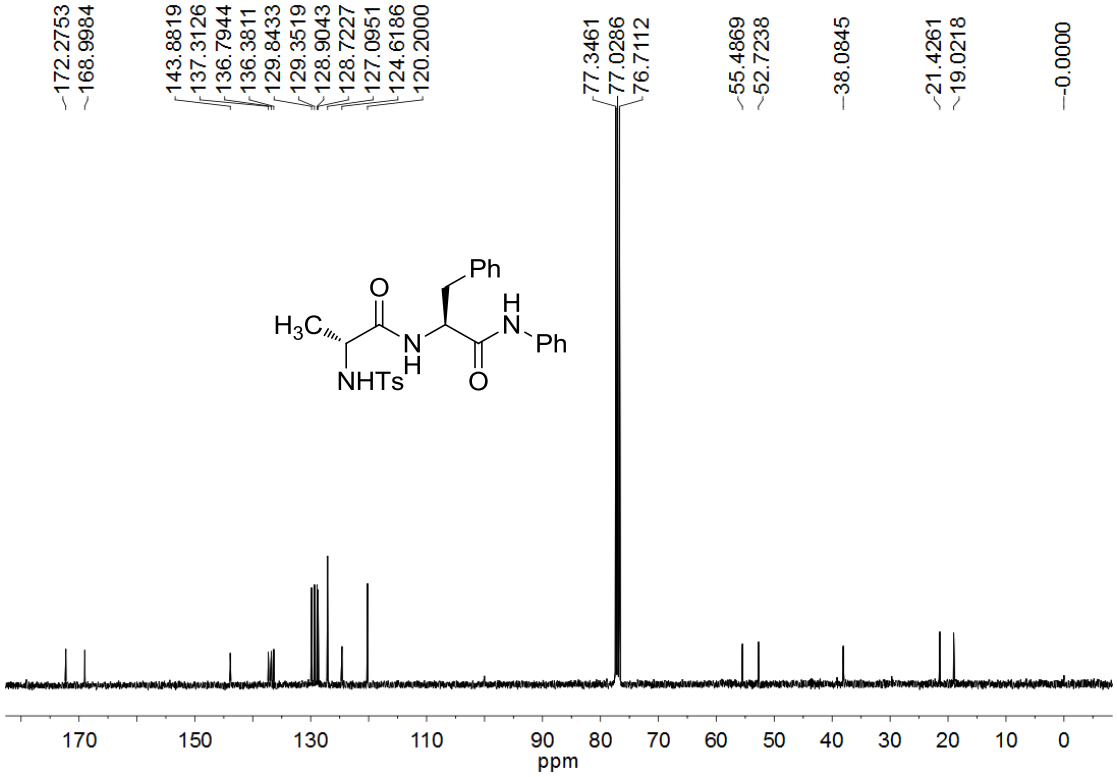


Figure S92. ^{13}C NMR spectrum of (*R*,*S*)-G**22** in $CDCl_3$ (100 MHz).

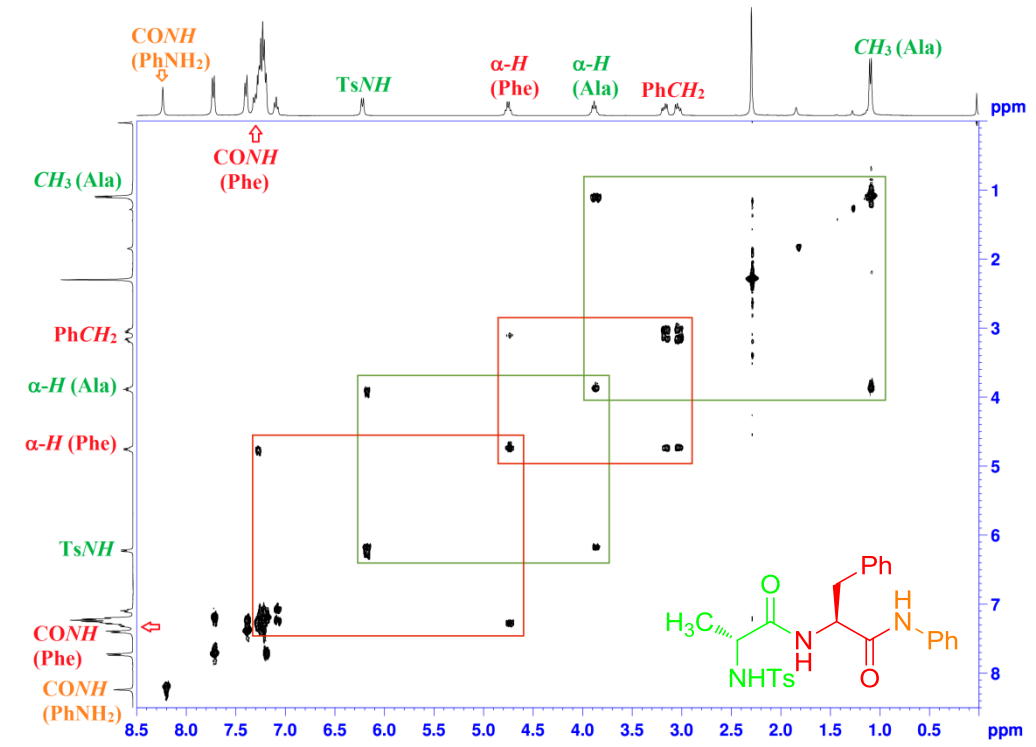


Figure S93. ^{1}H–^{1}H COSY spectrum of (*R*,*S*)-G**22** in $CDCl_3$ (400 MHz).

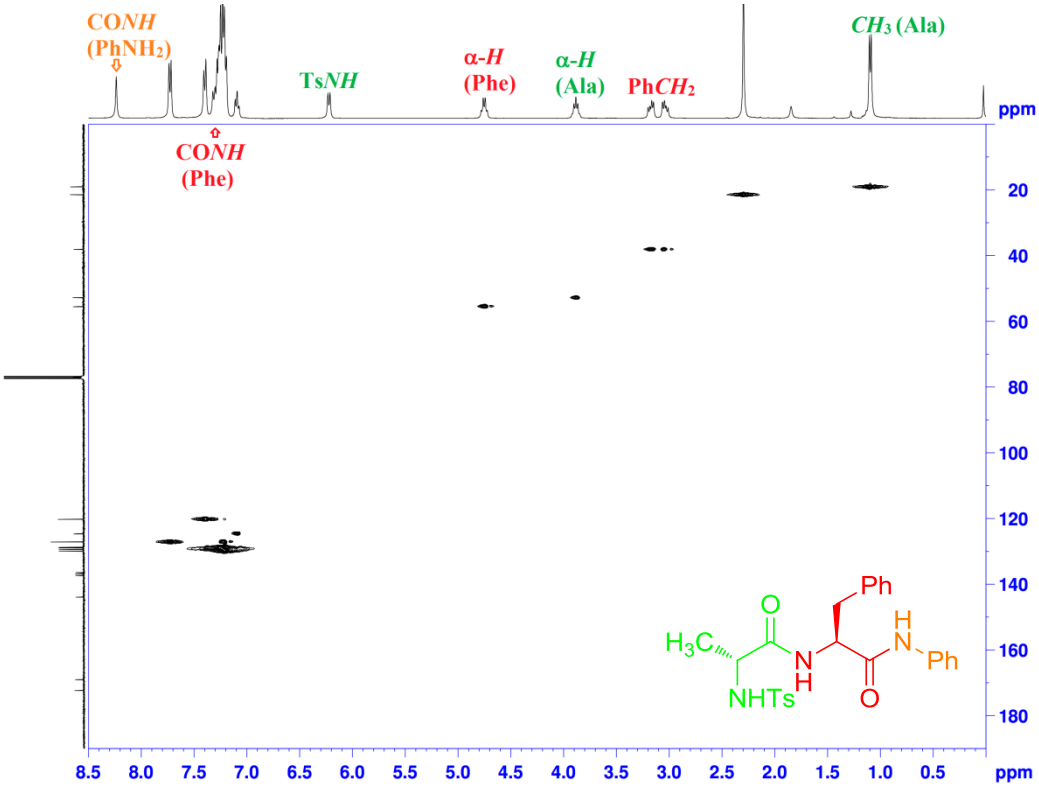


Figure S94. HSQC spectrum of (*R*,*S*)-G**22** in $CDCl_3$.

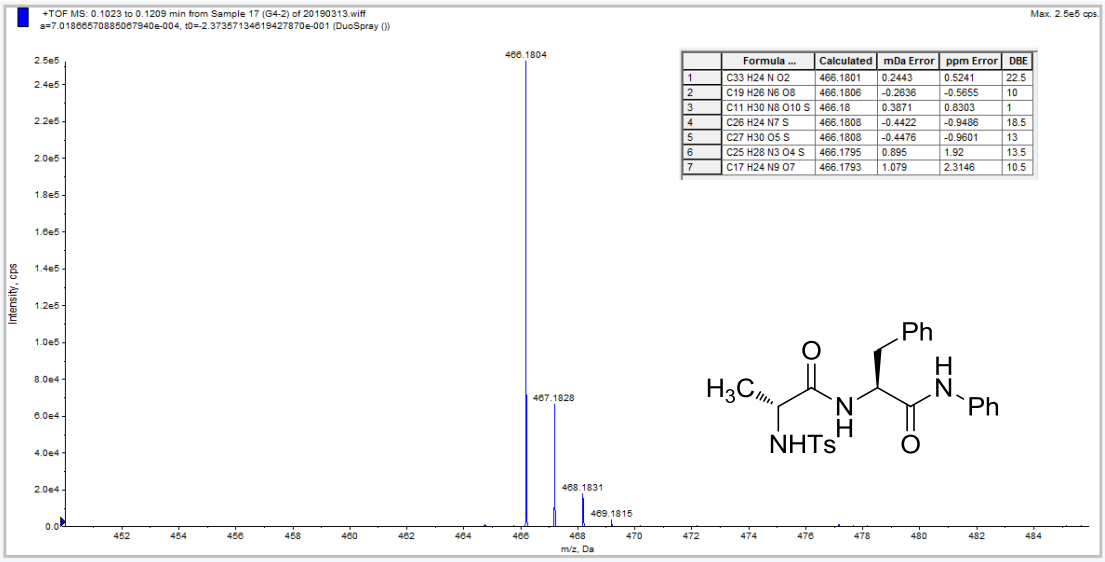


Figure S95. HRMS spectrum of (*R*,*S*)-G**22**.

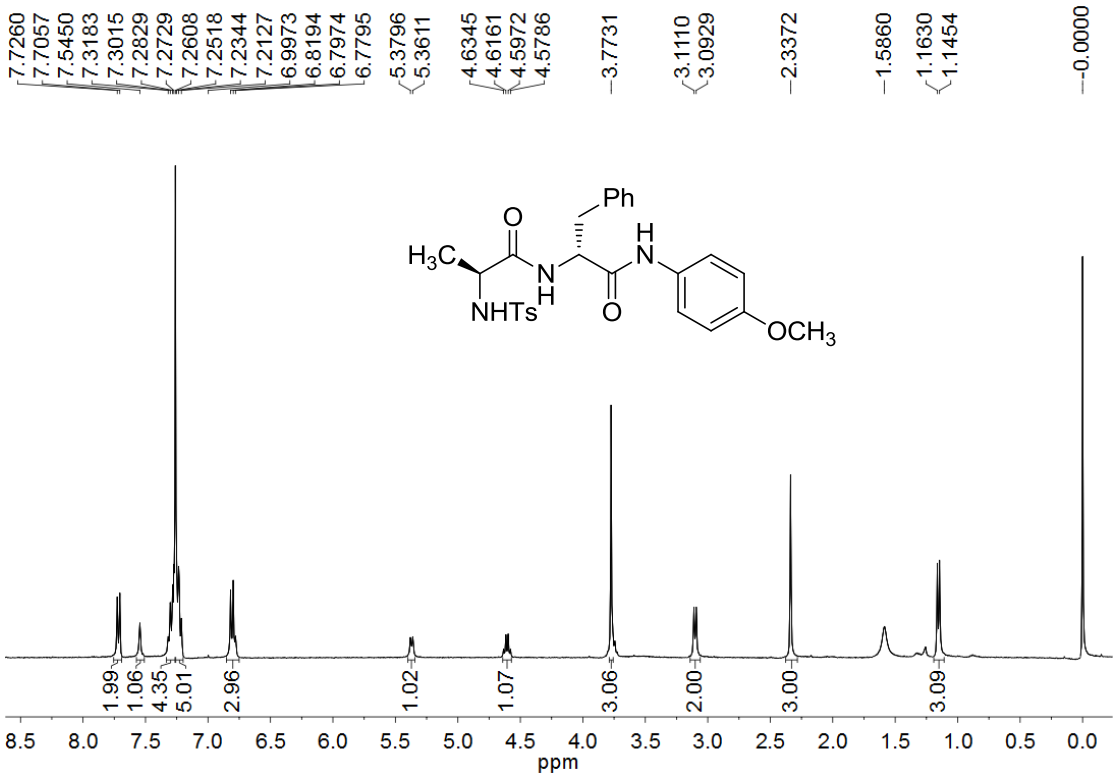


Figure S96. ^{1}H NMR spectrum of (*S*,*R*)-G**23** in $CDCl_3$ (400 MHz).

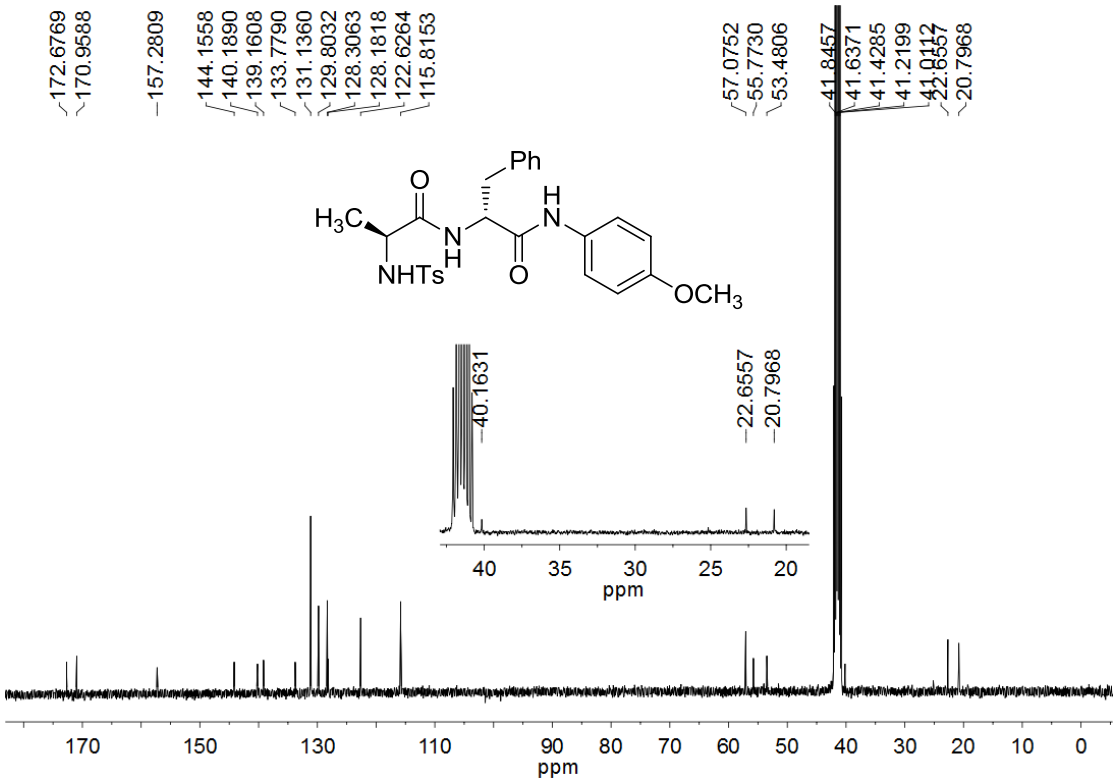


Figure S97. ^{13}C NMR spectrum of (*S*,*R*)-G**23** in DMSO-d_6 (100 MHz).

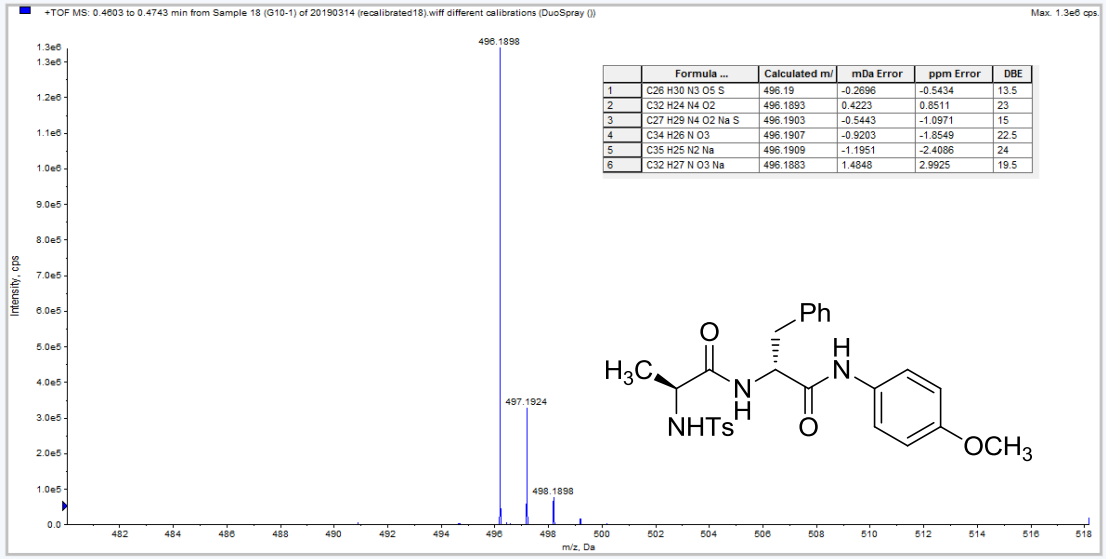


Figure S98. HRMS spectrum of (*S*,*R*)-G**23**.

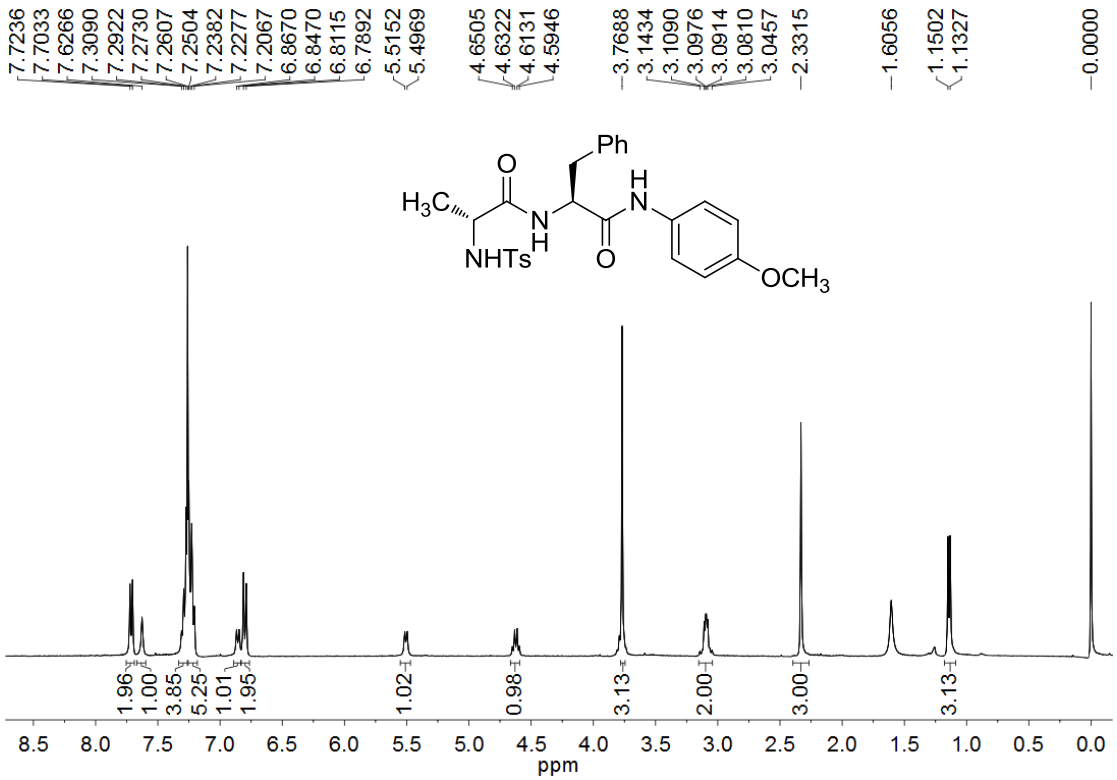


Figure S99. ^{1}H NMR spectrum of (*R*,*S*)-G**23** in $CDCl_3$ (400 MHz).

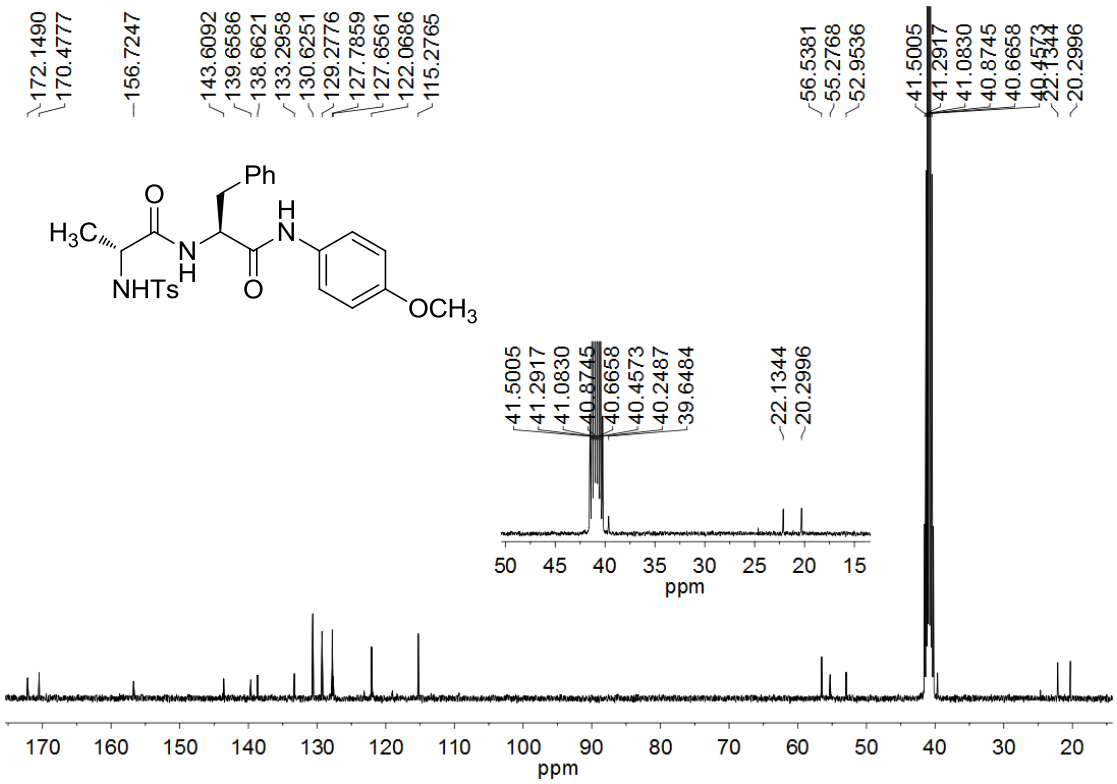


Figure S100. ^{13}C NMR spectrum of (*R*,*S*)-G**23** in DMSO-d_6 (100 MHz).

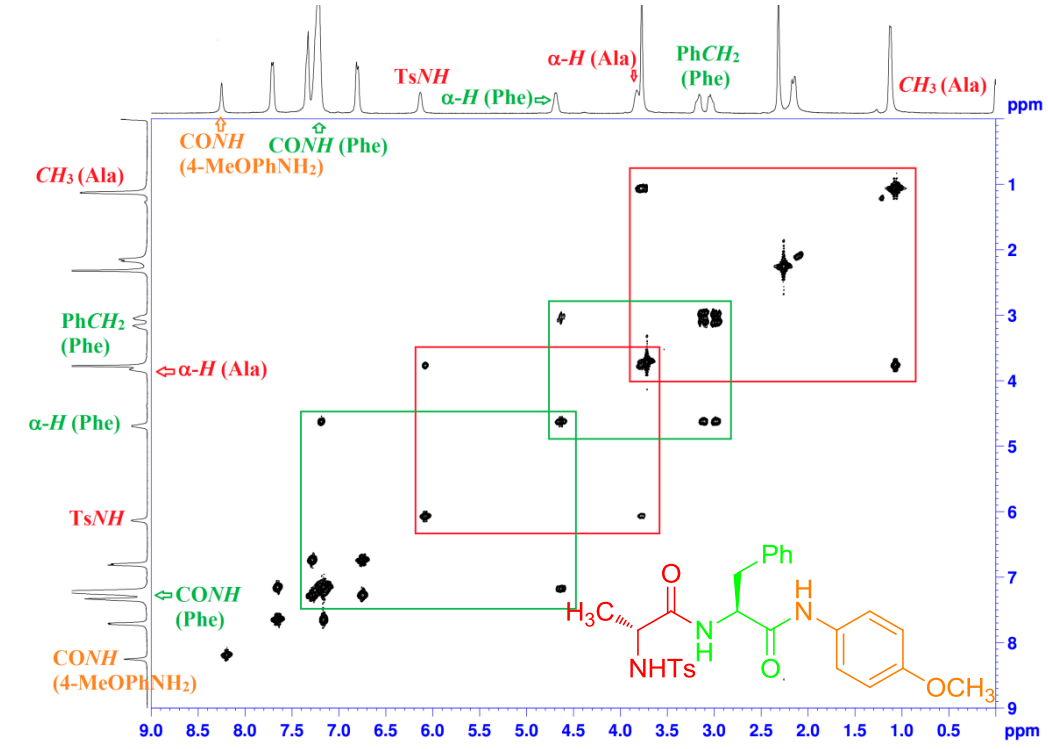


Figure S101. ^{1}H–^{1}H COSY spectrum of (*R*,*S*)-G**23** in $CDCl_3$ (400 MHz).

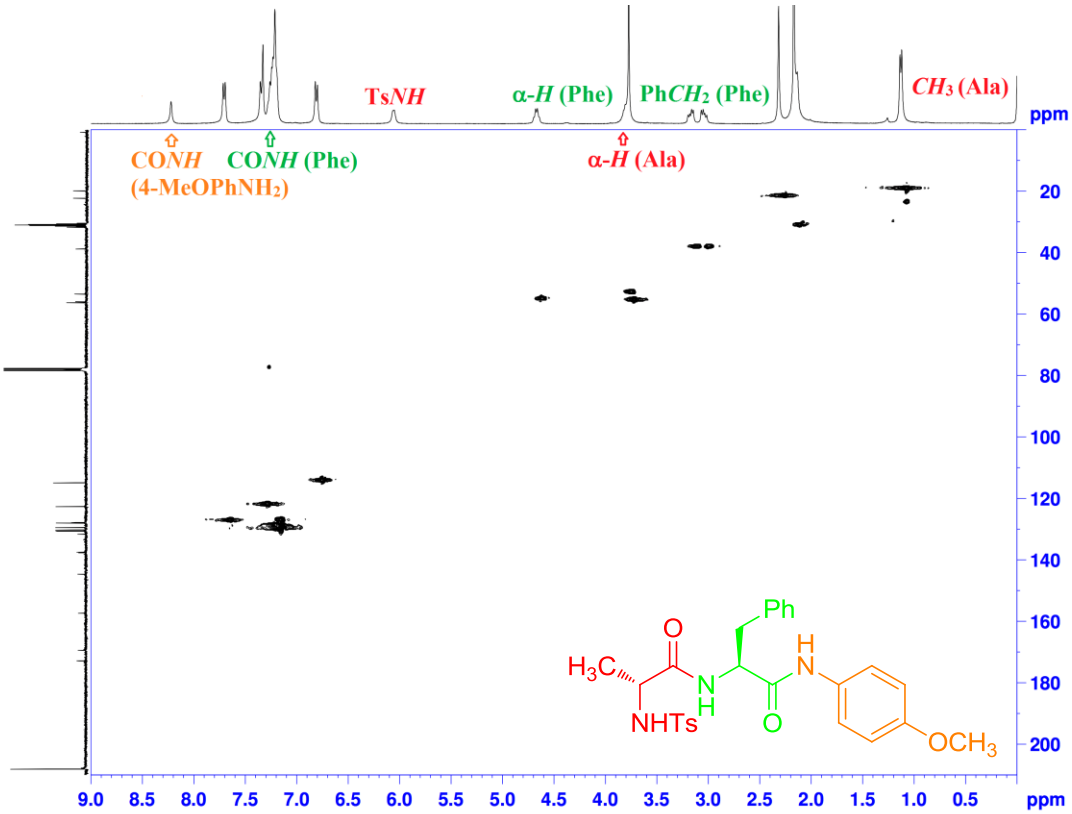


Figure S102. HSQC spectrum of (*R*,*S*)-G**23** in $CDCl_3$ (5% acetone-d_6).

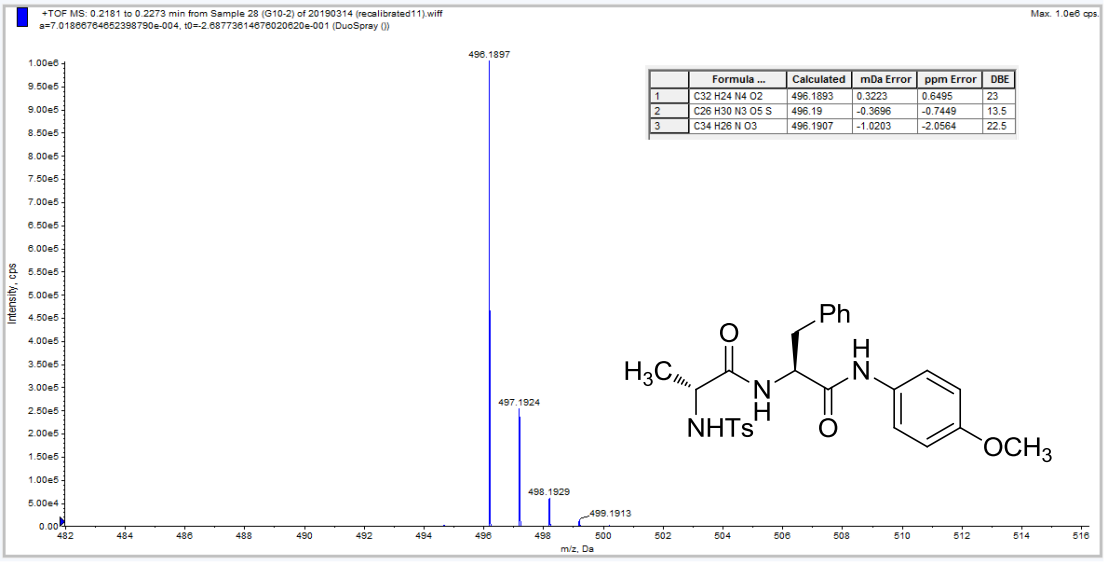


Figure S103. HRMS spectrum of (*R*,*S*)-G**23**.

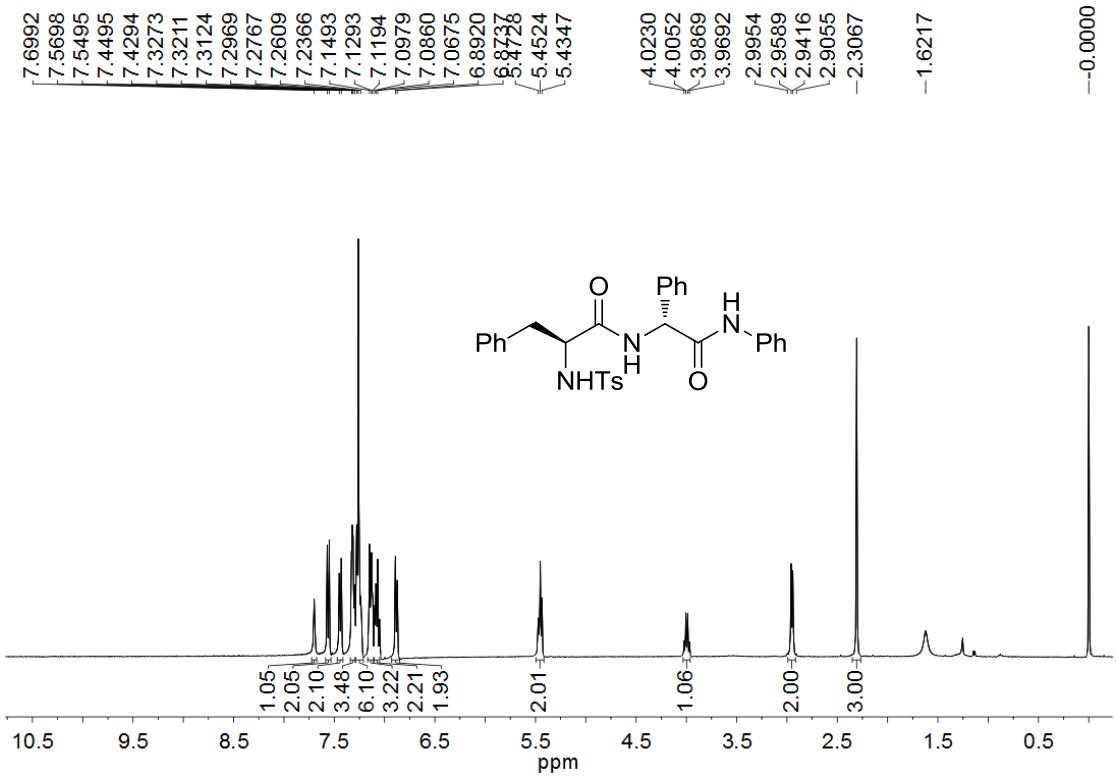


Figure S104. ^{1}H NMR spectrum of (*S*,*R*)-G**24** in $CDCl_3$ (400 MHz).

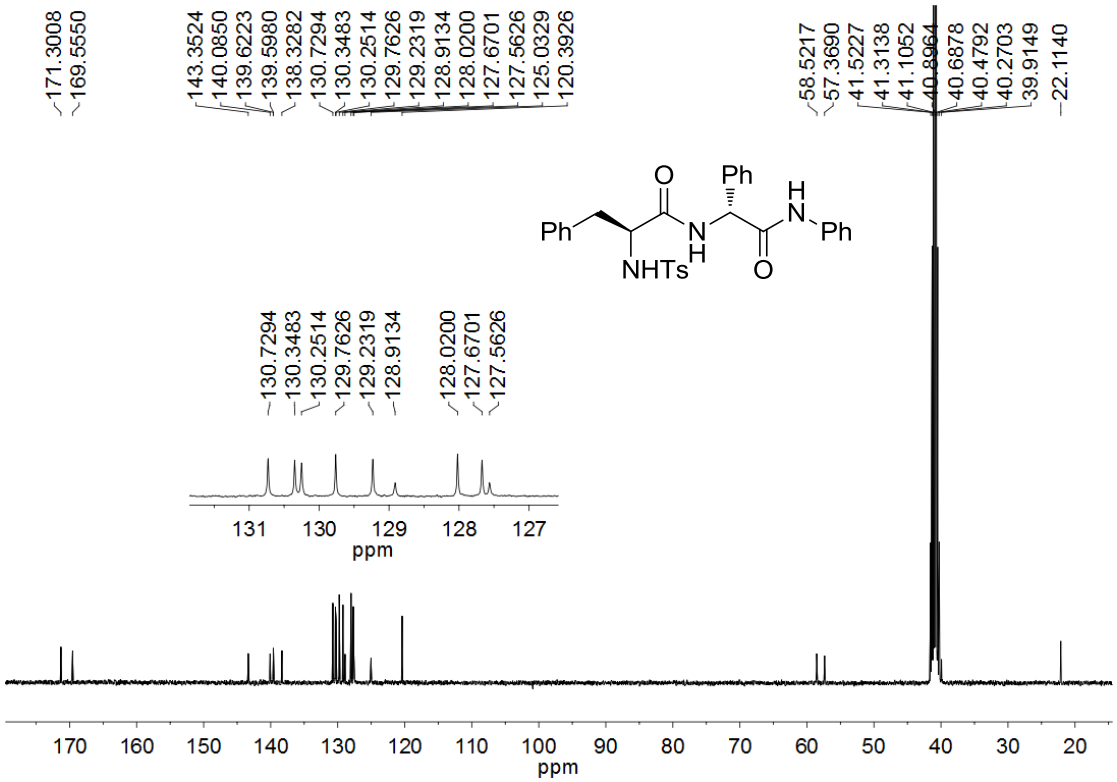


Figure S105. ^{13}C NMR spectrum of (*S*,*R*)-G**24** in DMSO-d_6 (100 MHz).

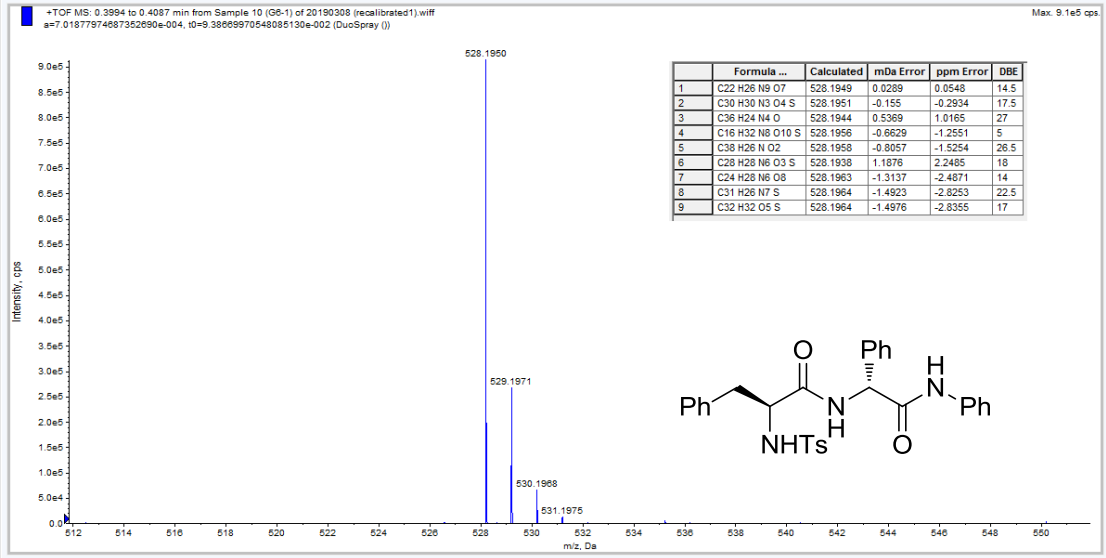


Figure S106. HRMS spectrum of (*S*,*R*)-G**24**.

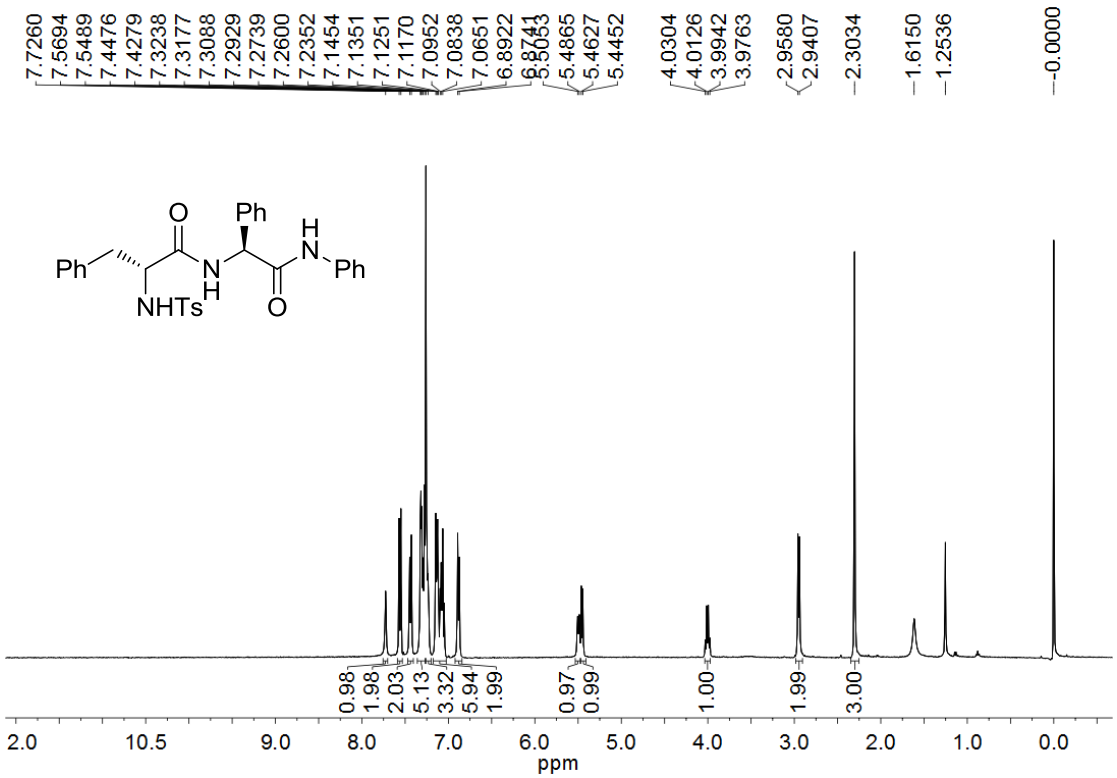


Figure S107. ^{1}H NMR spectrum of (*R*,*S*)-G**24** in $CDCl_3$ (400 MHz).

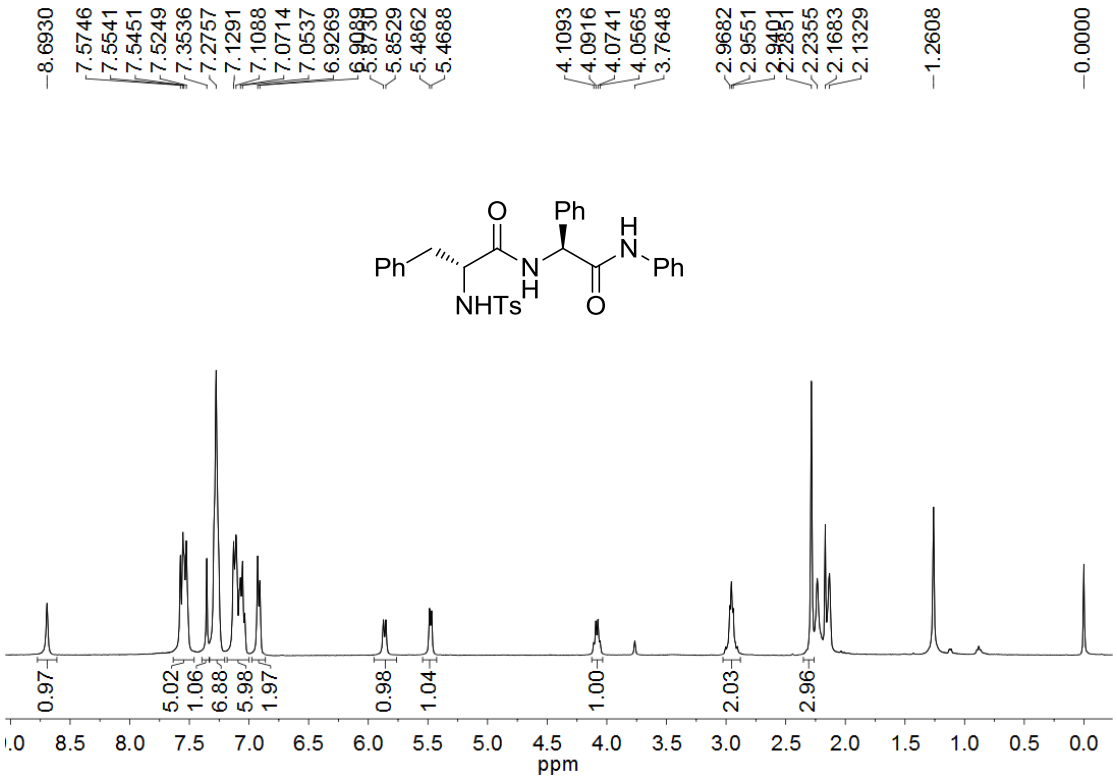


Figure S108. ^{13}C NMR spectrum of (*R*,*S*)-G**24** in $CDCl_3$ (5%acetone-d_6), (400 MHz).

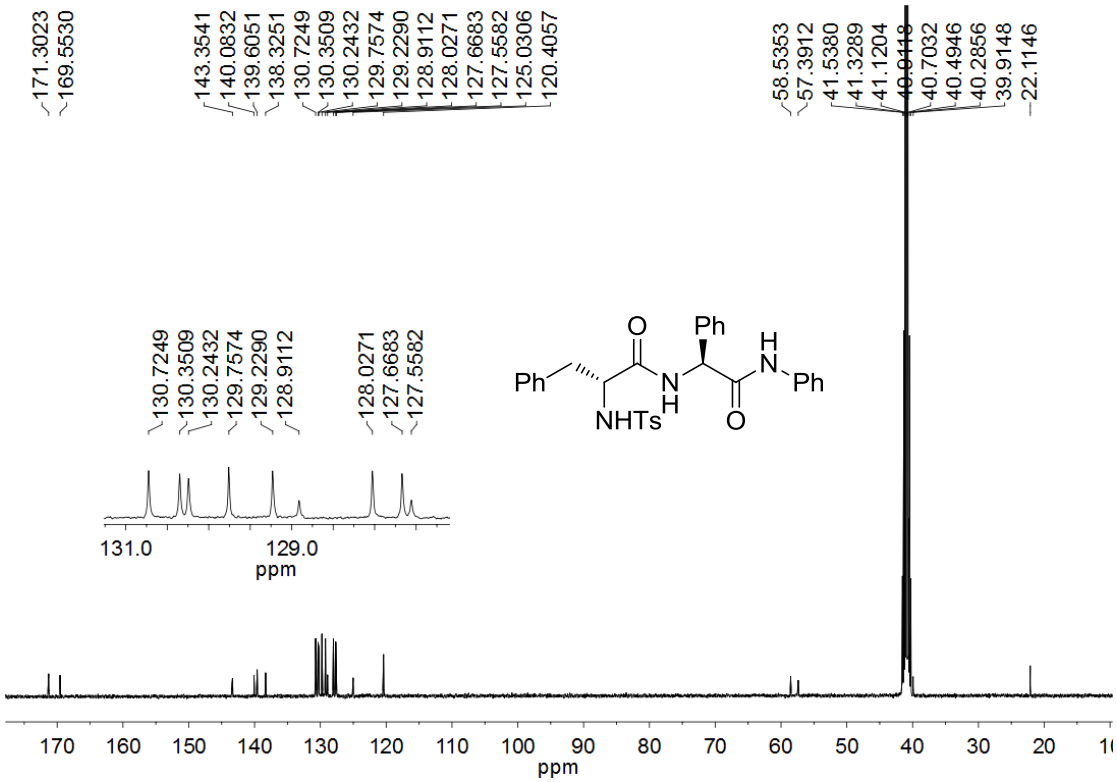


Figure S109. ^{13}C NMR spectrum of (*R*,*S*)-G**24** in DMSO-d_6 (100 MHz).

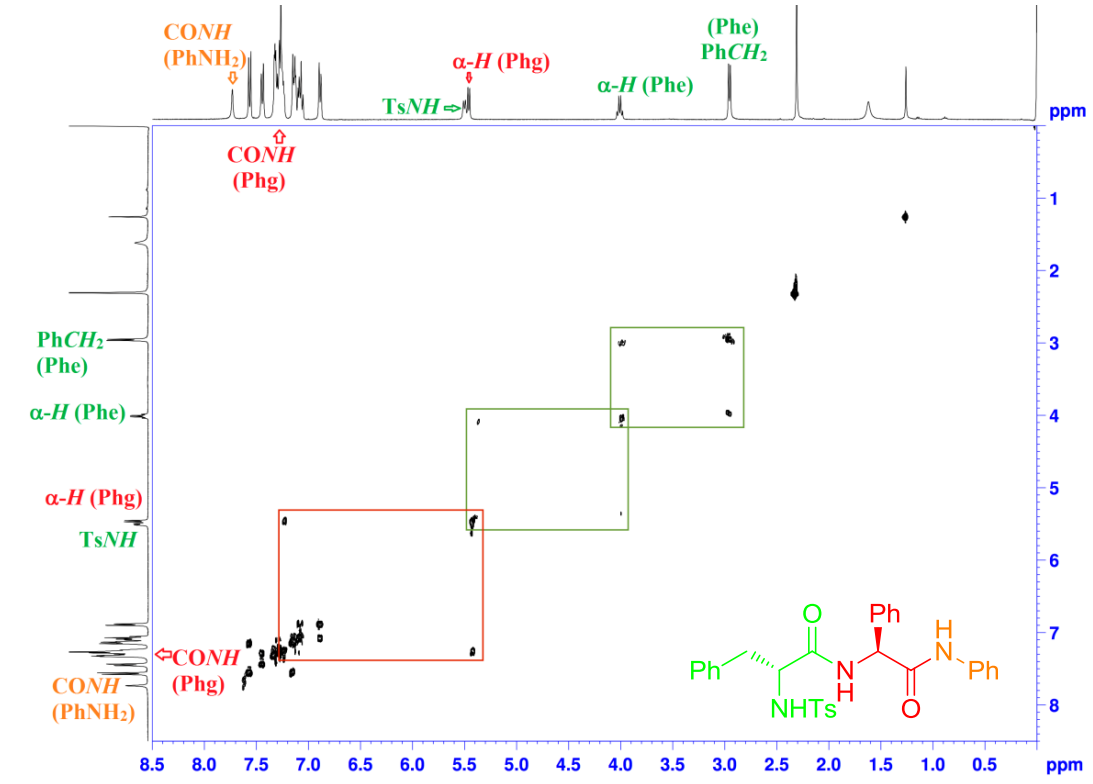


Figure S110. ^{1}H–^{1}H COSY spectrum of (*R*,*S*)-G**24** in $CDCl_3$ (400 MHz).

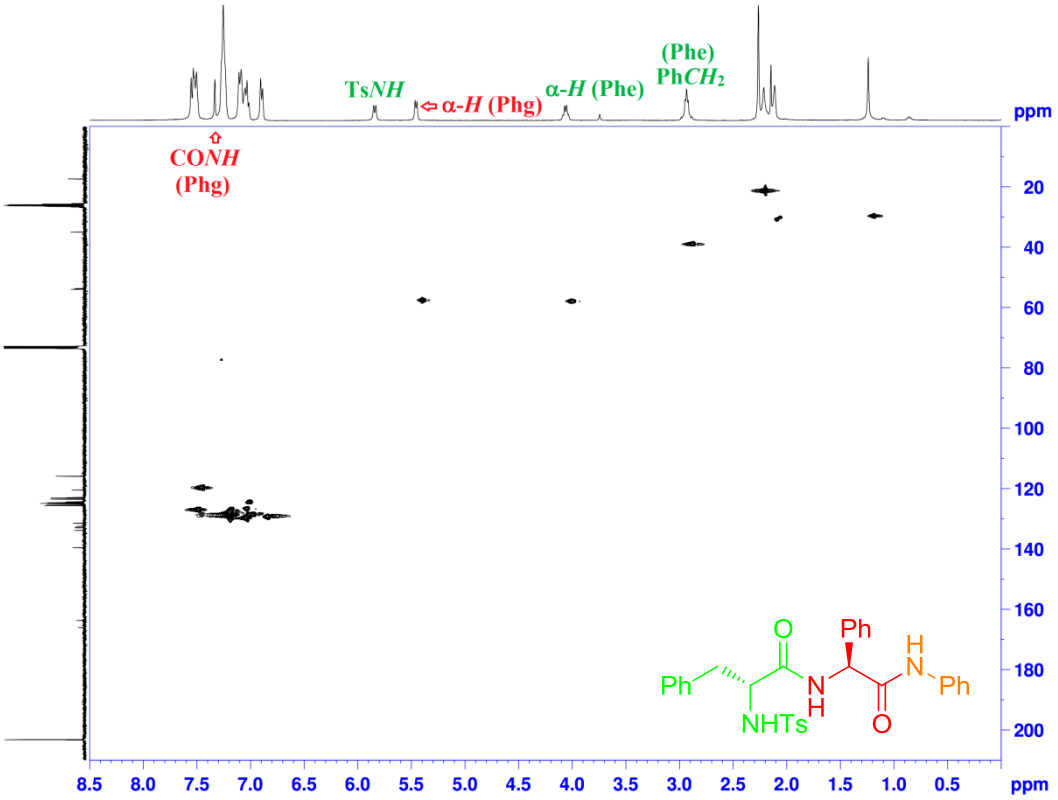


Figure S111. HSQC spectrum of (*R*,*S*)-G**24** in $CDCl_3$ (5% acetone-d_6).

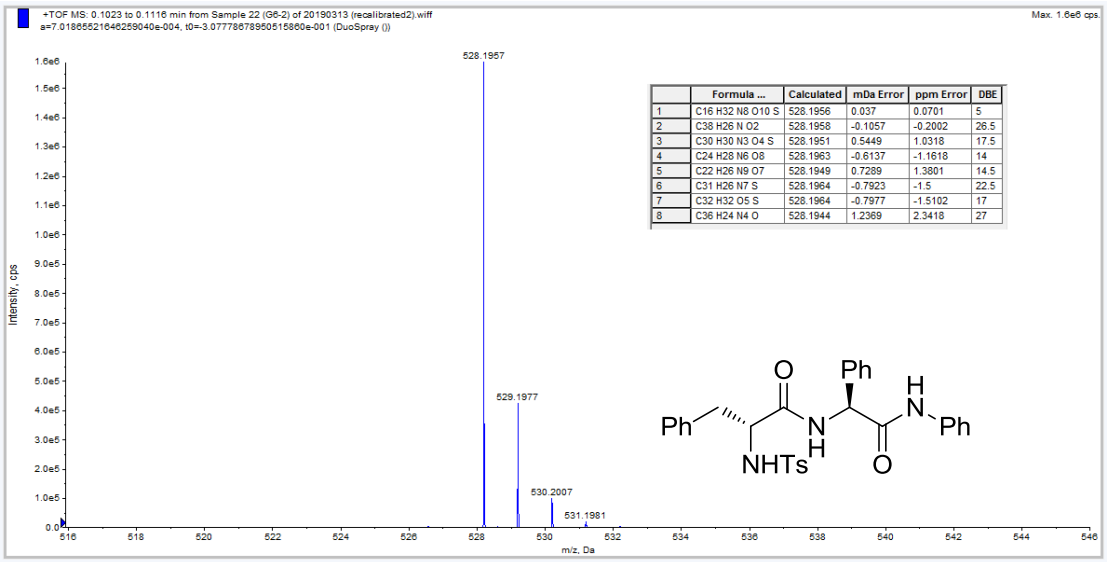


Figure S112. HRMS spectrum of (*R*,*S*)-G**24**.

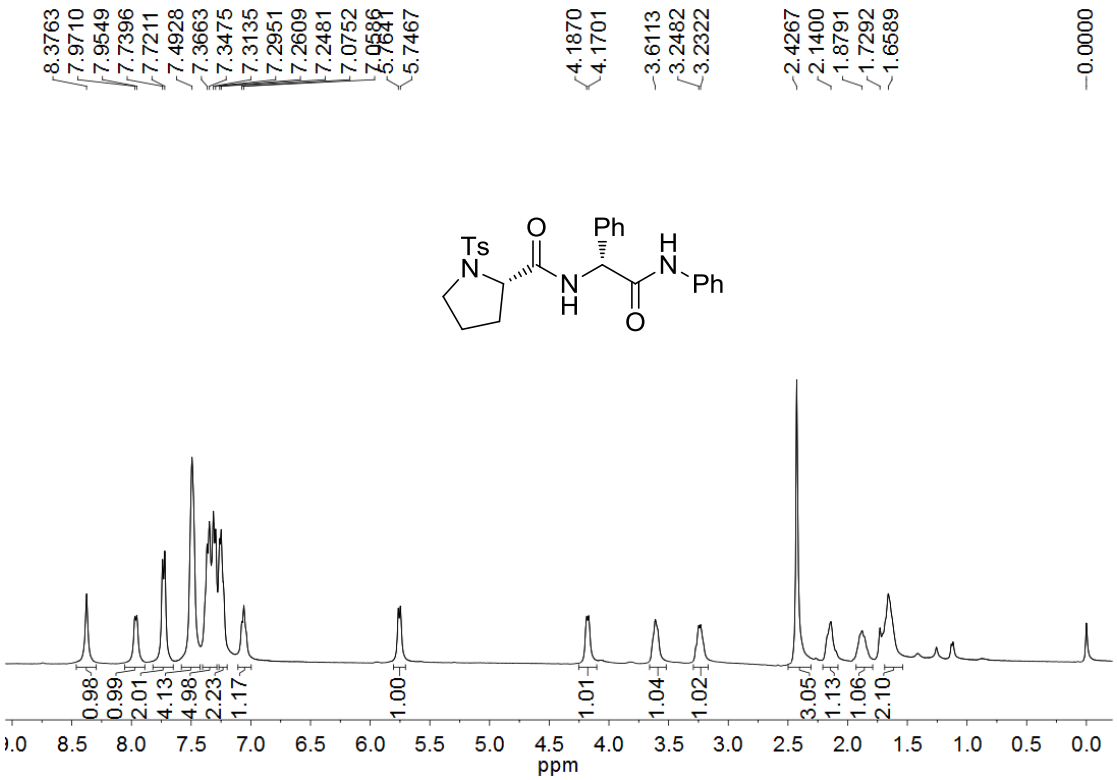


Figure S113. ^{13}C NMR spectrum of (*S*,*R*)-G**25** in $CDCl_3$ (400 MHz).

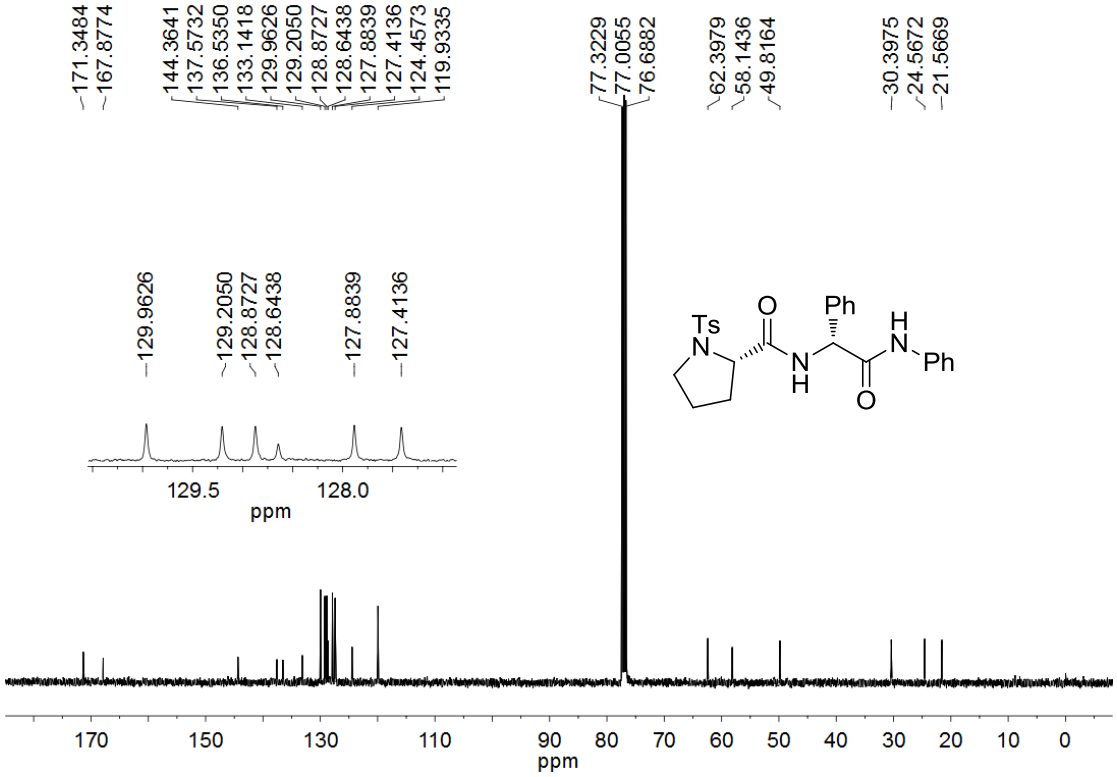


Figure S114. ^{13}C NMR spectrum of (*S*,*R*)-G**25** in $CDCl_3$ (100 MHz).

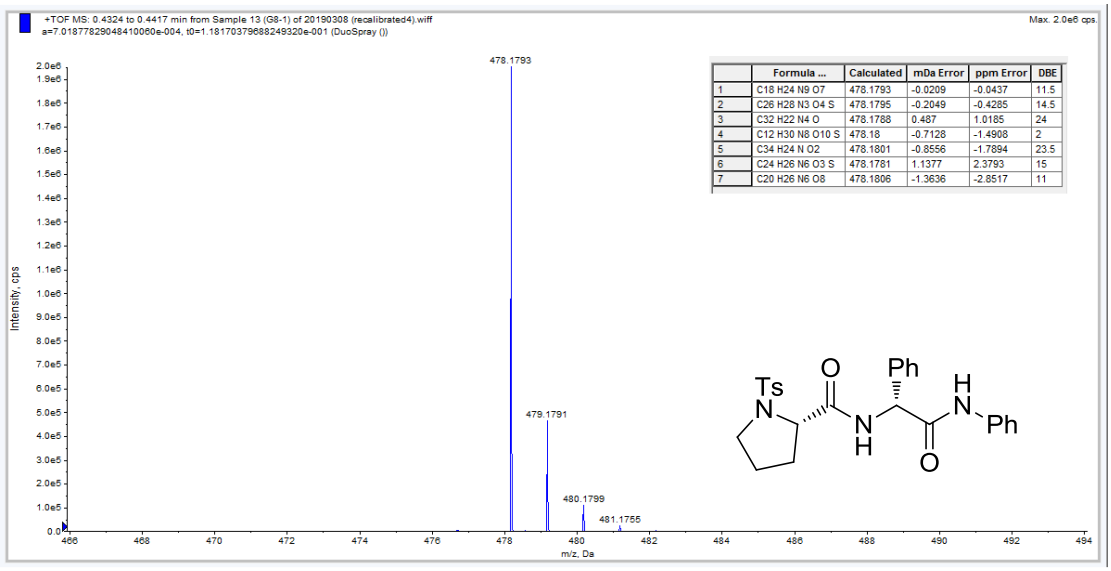


Figure S115. HRMS spectrum of (*S*,*R*)-G**25**.

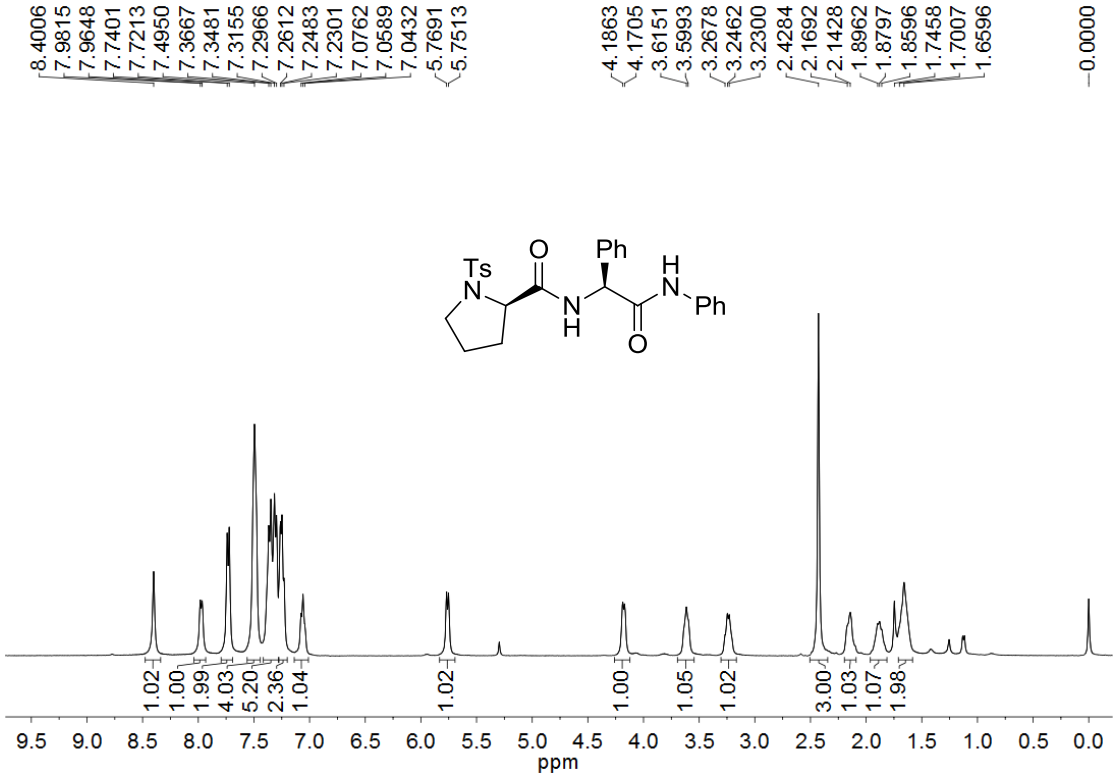


Figure S116. ^{1}H NMR spectrum of (*R*,*S*)-G**25** in $CDCl_3$ (400 MHz).

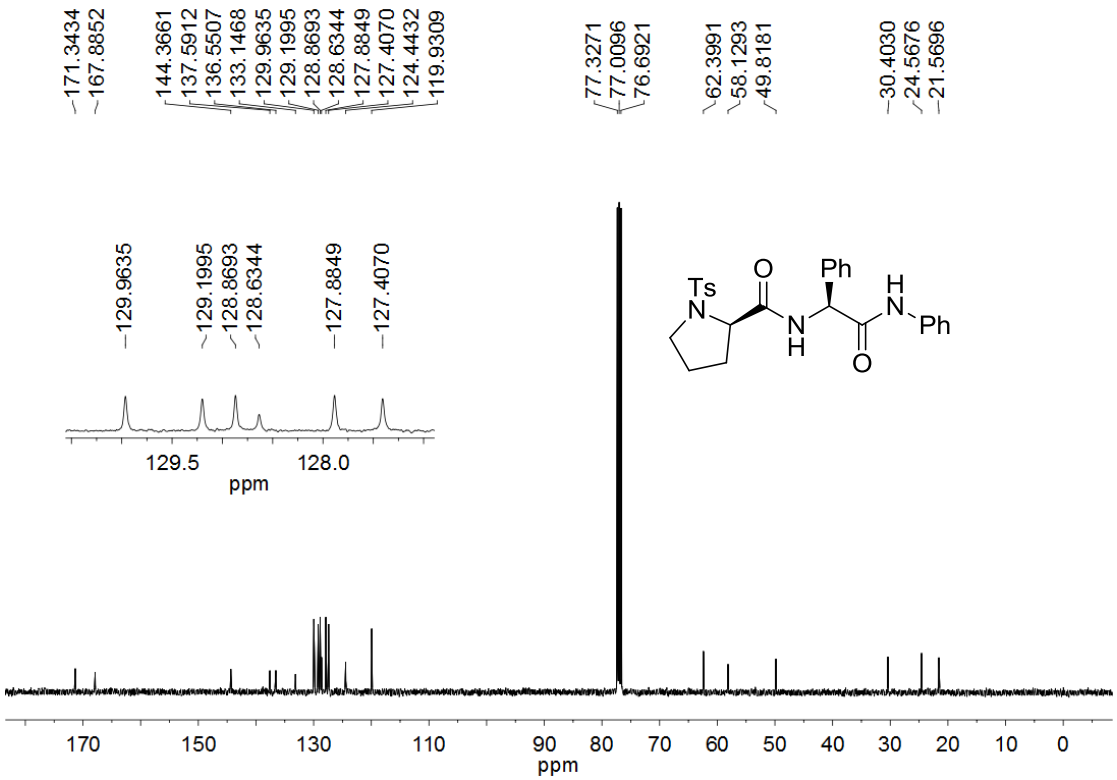


Figure S117. ^{13}C NMR spectrum of (*R*,*S*)-G**25** in $CDCl_3$ (100 MHz).

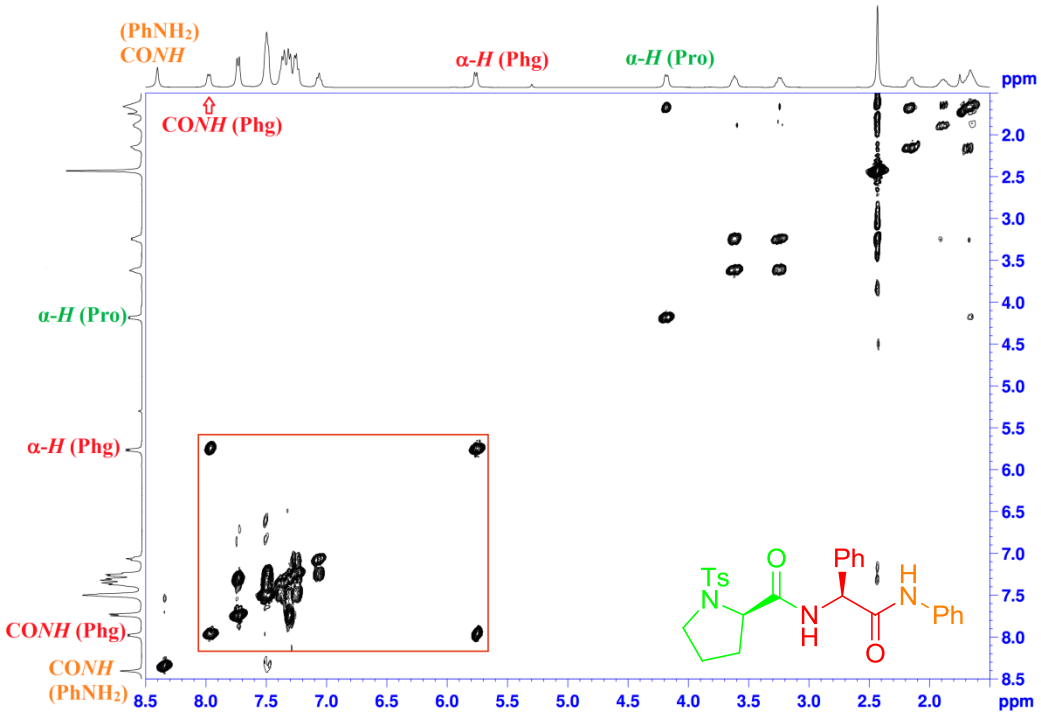


Figure S118. ^{1}H–^{1}H COSY spectrum of (*R*,*S*)-G**25** in $CDCl_3$ (400 MHz).

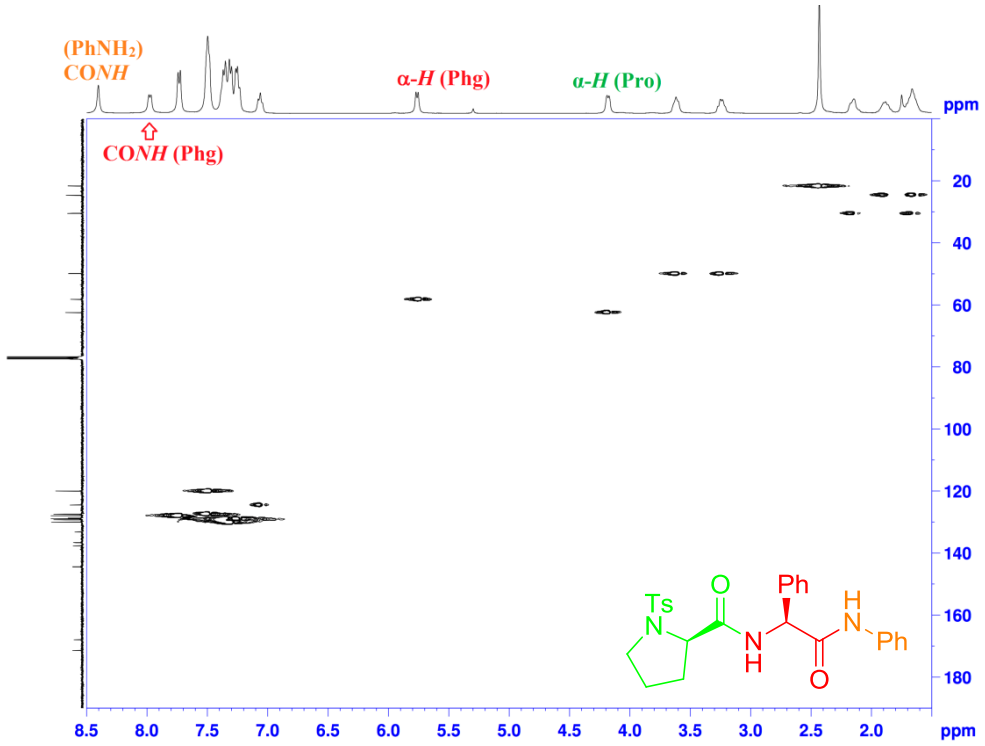


Figure S119. HSQC spectrum of (*R*,*S*)-G**25** in $CDCl_3$.

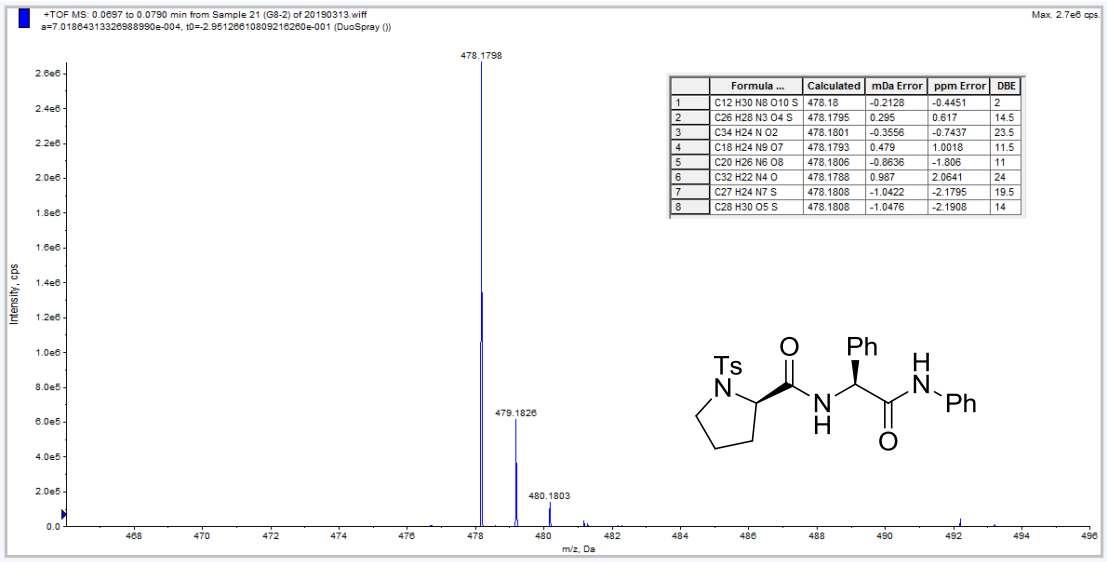


Figure S120. HRMS spectrum of (*R*,*S*)-G**25**.

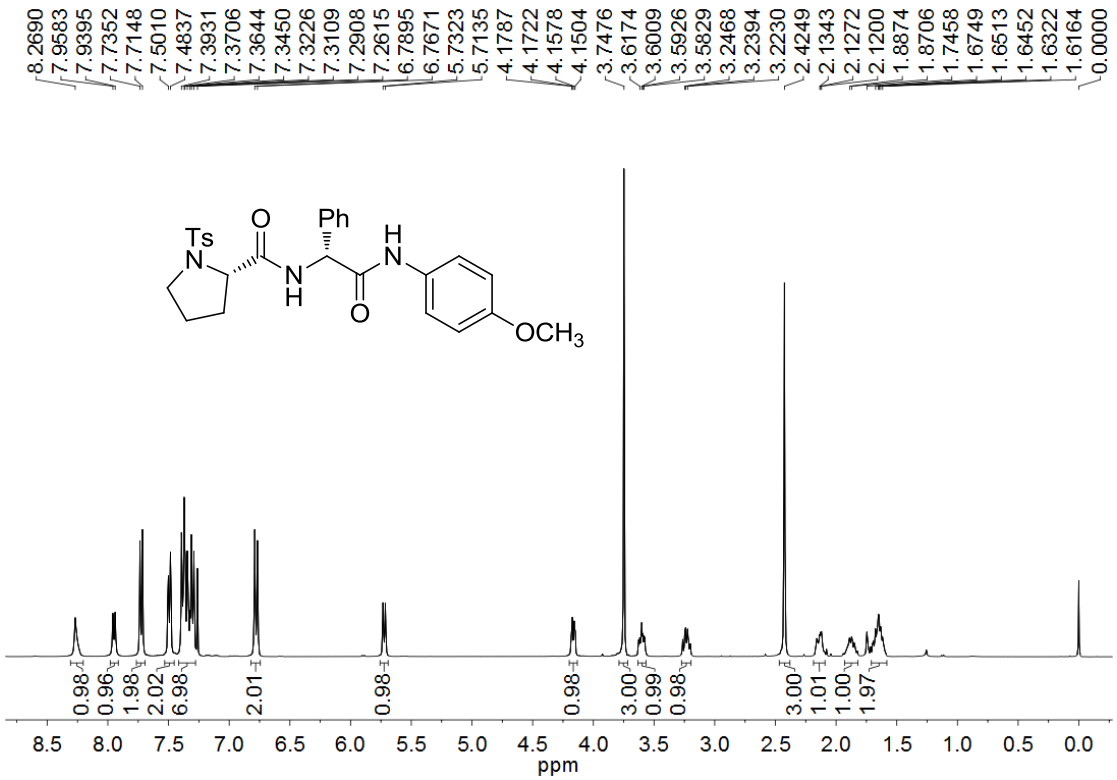


Figure S121. ^{1}H NMR spectrum of (*S*,*R*)-G**26** in $CDCl_3$ (400 MHz).

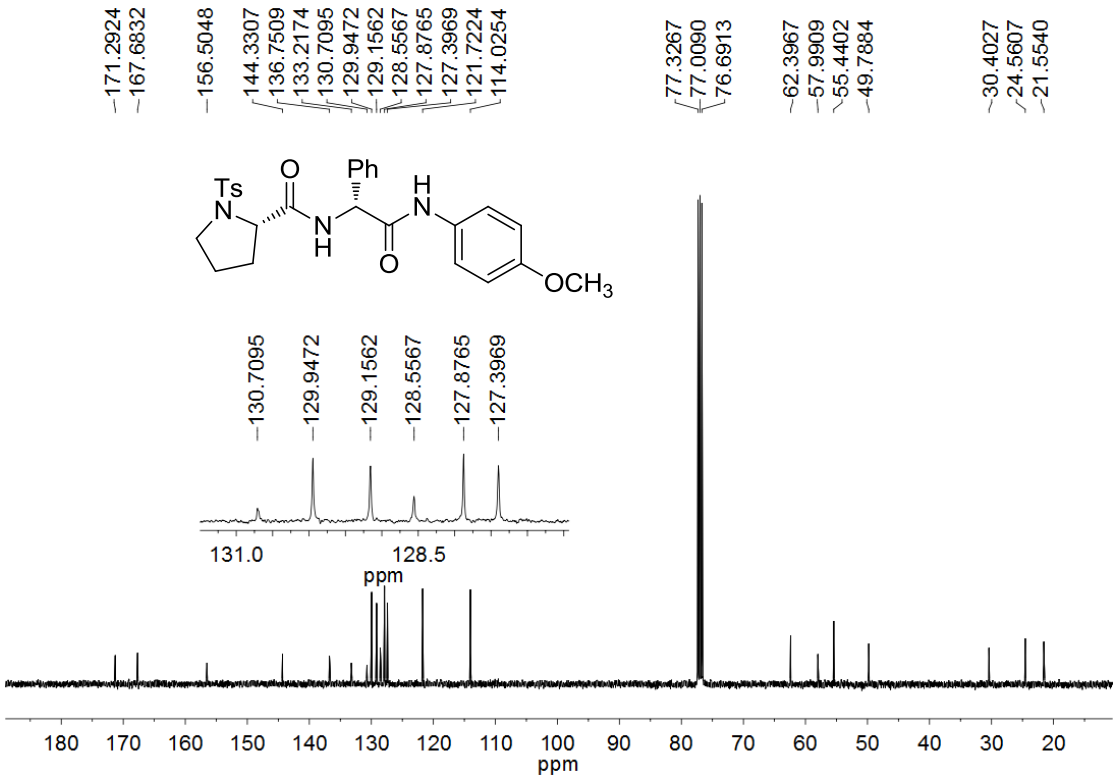
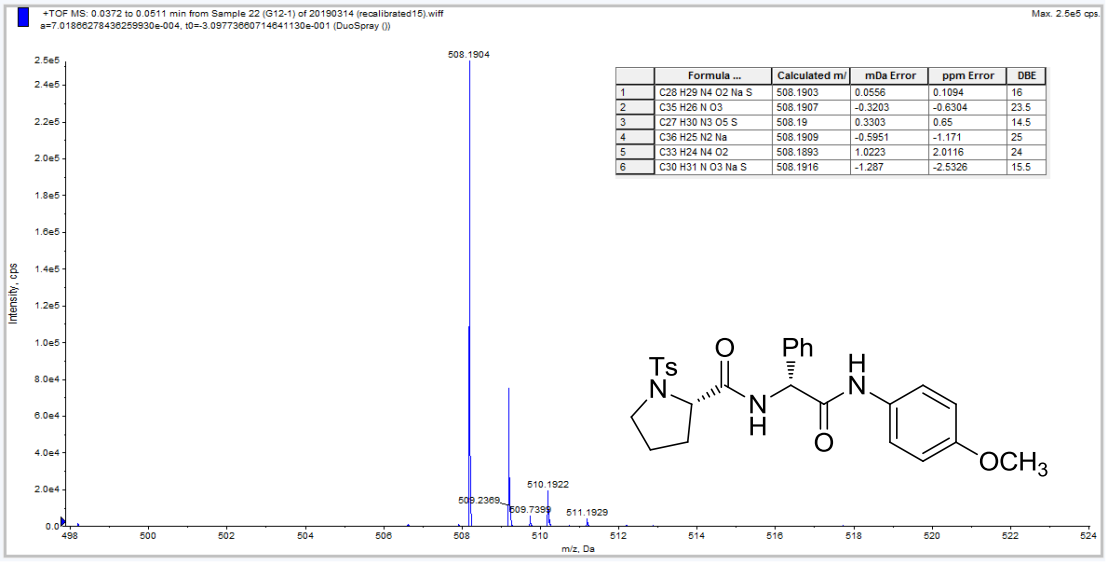


Figure S122. ^{13}C NMR spectrum of (*S*,*R*)-G**26** in $CDCl_3$ (100 MHz).



	Formula ...	Calculated m/	mDa Error	ppm Error	DBE
1	C28 H29 N4 O2 Na S	508.1903	0.0556	0.1094	16
2	C35 H26 N O3	508.1907	-0.3203	-0.6304	23.5
3	C27 H30 N3 O5 S	508.19	0.3303	0.65	14.5
4	C36 H25 N2 Na	508.1909	-0.5951	-1.171	25
5	C33 H24 N4 O2	508.1893	1.0223	2.0116	24
6	C30 H31 N O3 Na S	508.1916	-1.287	-2.5326	15.5

Figure S123. HRMS spectrum of (*S*,*R*)-G**26**.

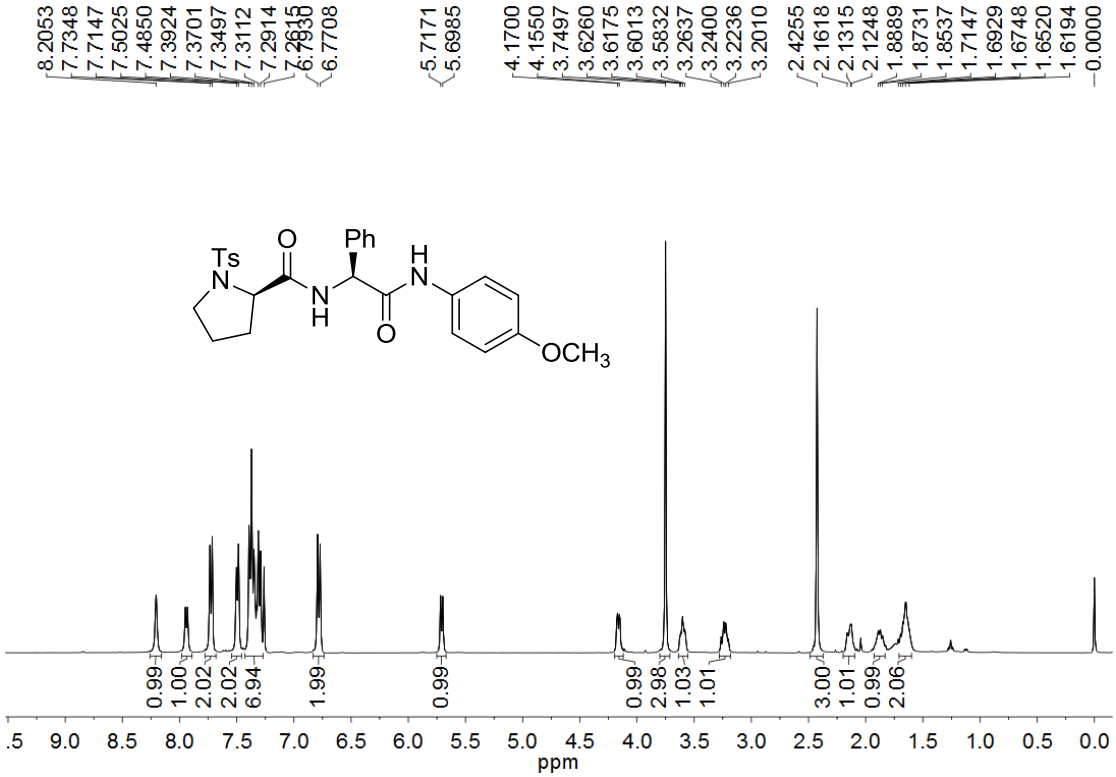


Figure S124. ^{1}H NMR spectrum of (*R*,*S*)-G**26** in $CDCl_3$ (400 MHz).

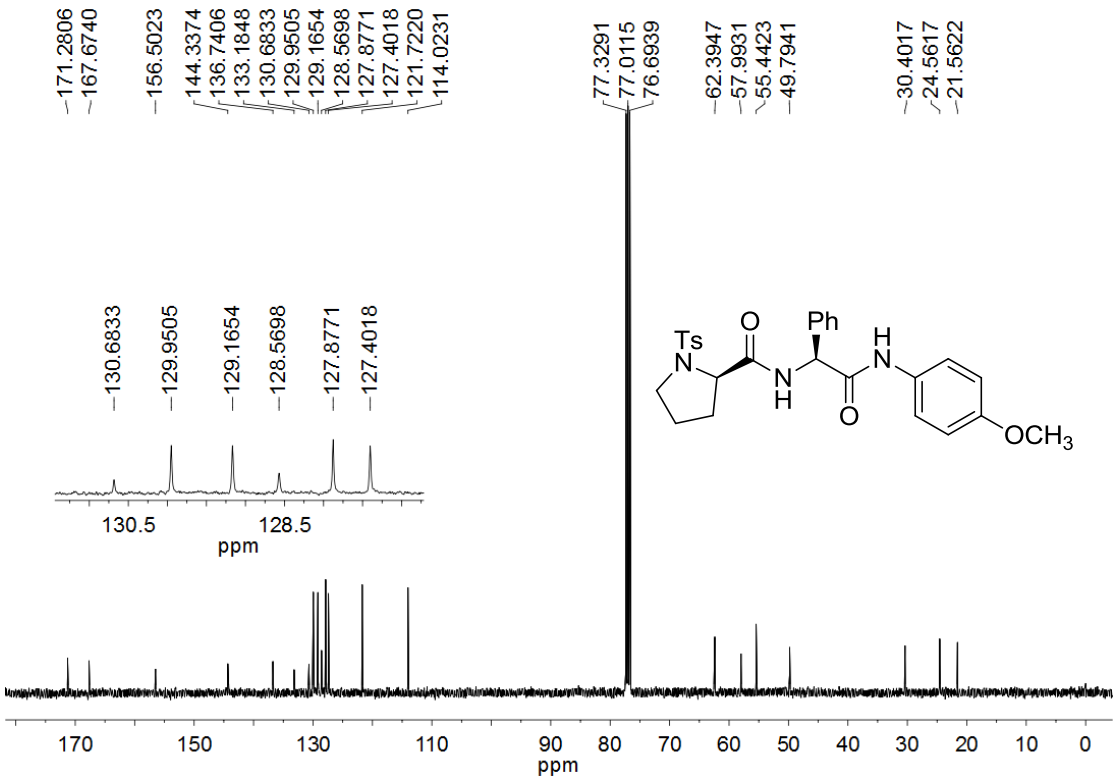


Figure S125. ^{13}C NMR spectrum of (*R*,*S*)-G**26** in $CDCl_3$ (100 MHz).

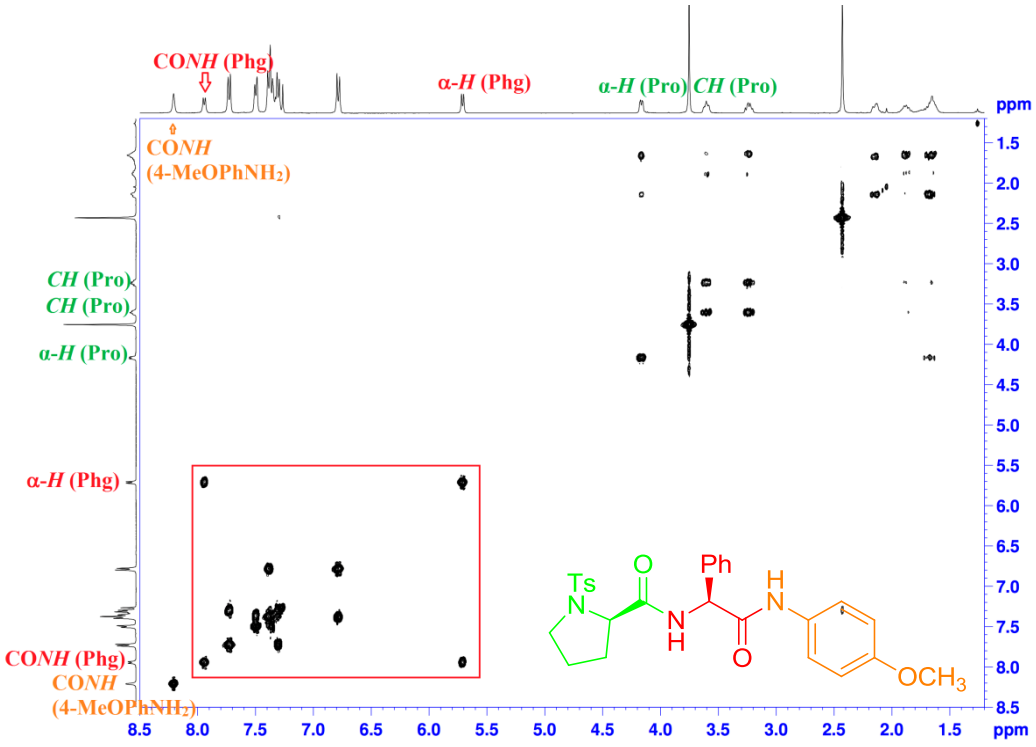


Figure S126. ^{1}H–^{1}H COSY spectrum of (*R*,*S*)-G**26** in $CDCl_3$ (400 MHz).

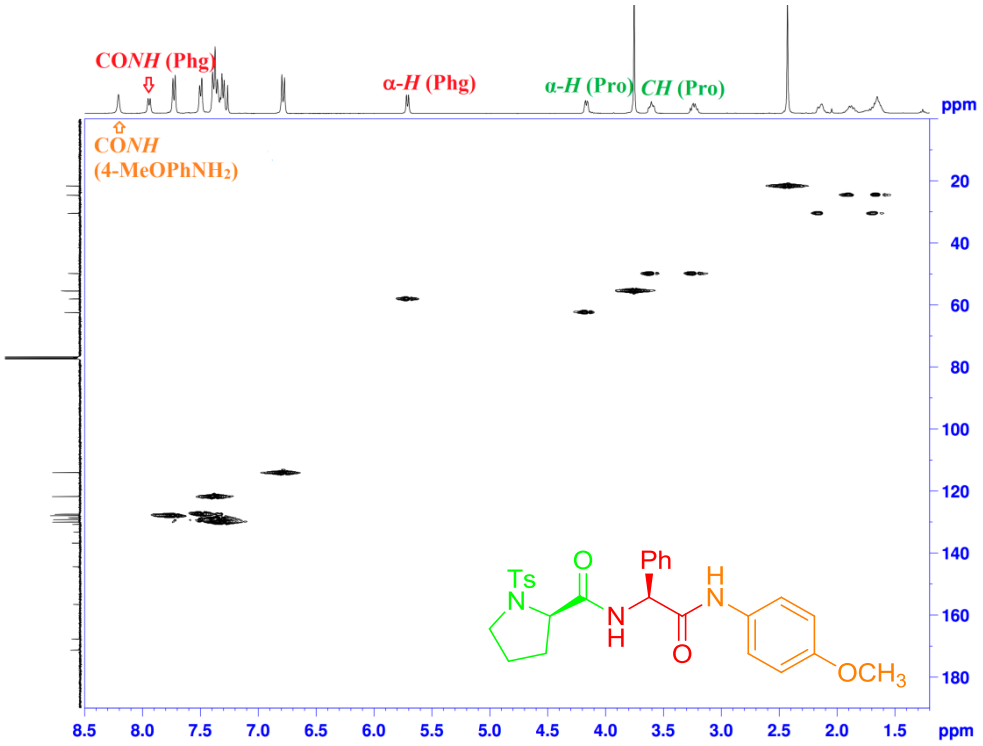


Figure S127. HSQC spectrum of (*R*,*S*)-G**26** in $CDCl_3$.

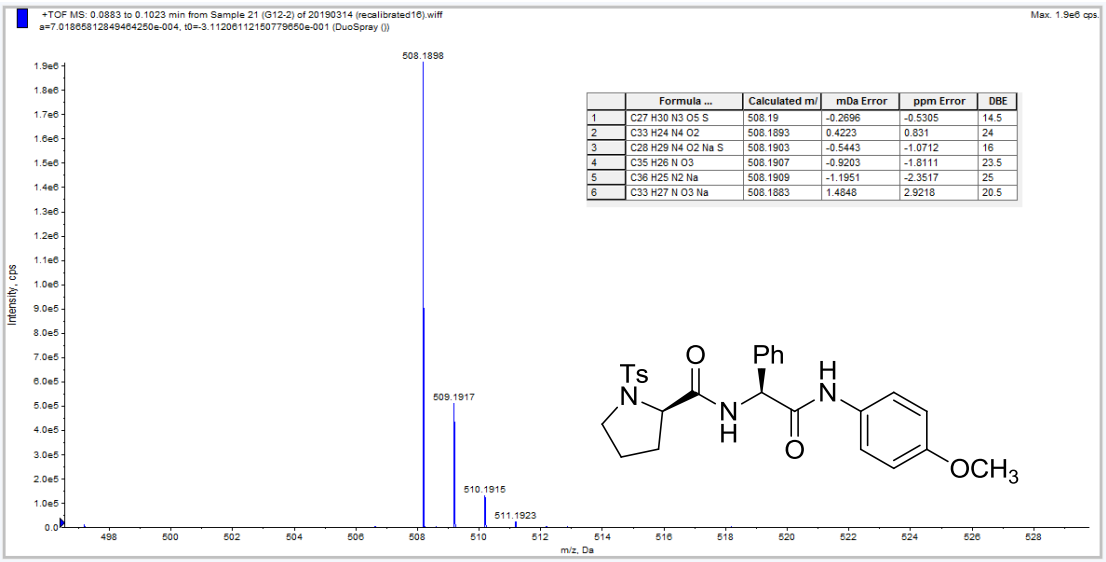


Figure S128. HRMS spectrum of (*R*,*S*)-G**26**.

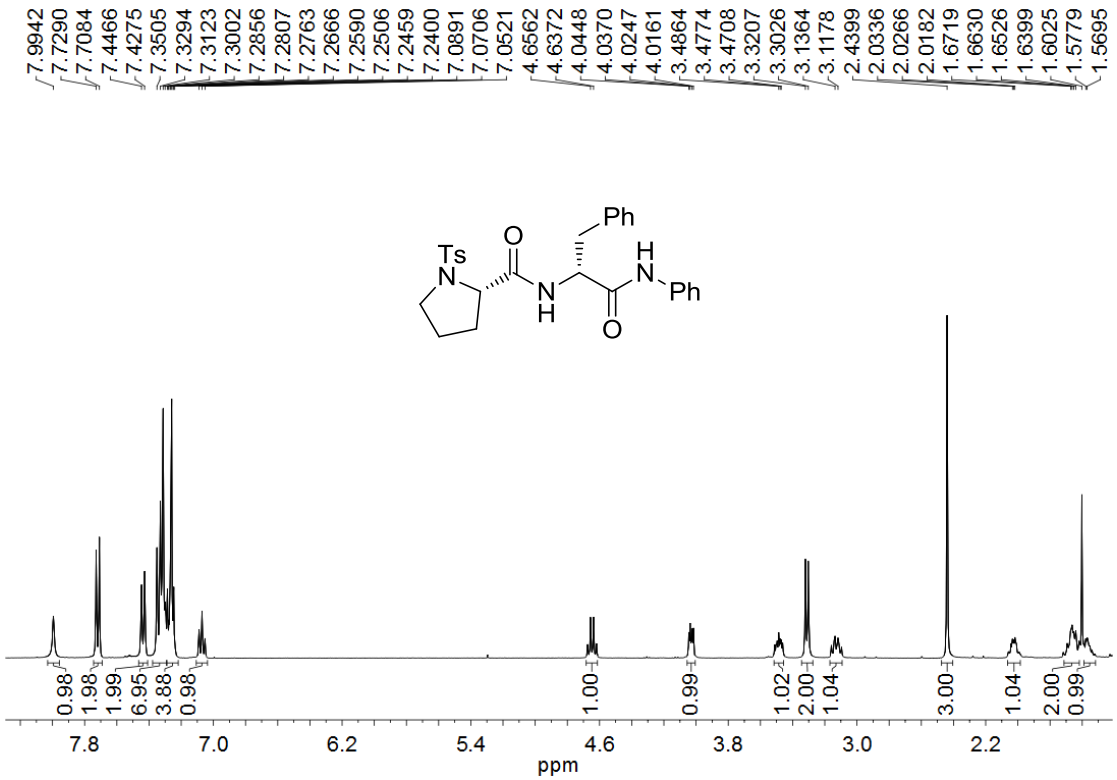


Figure S129. ^{1}H NMR spectrum of (*S*,*R*)-G**27** in $CDCl_3$ (400 MHz).

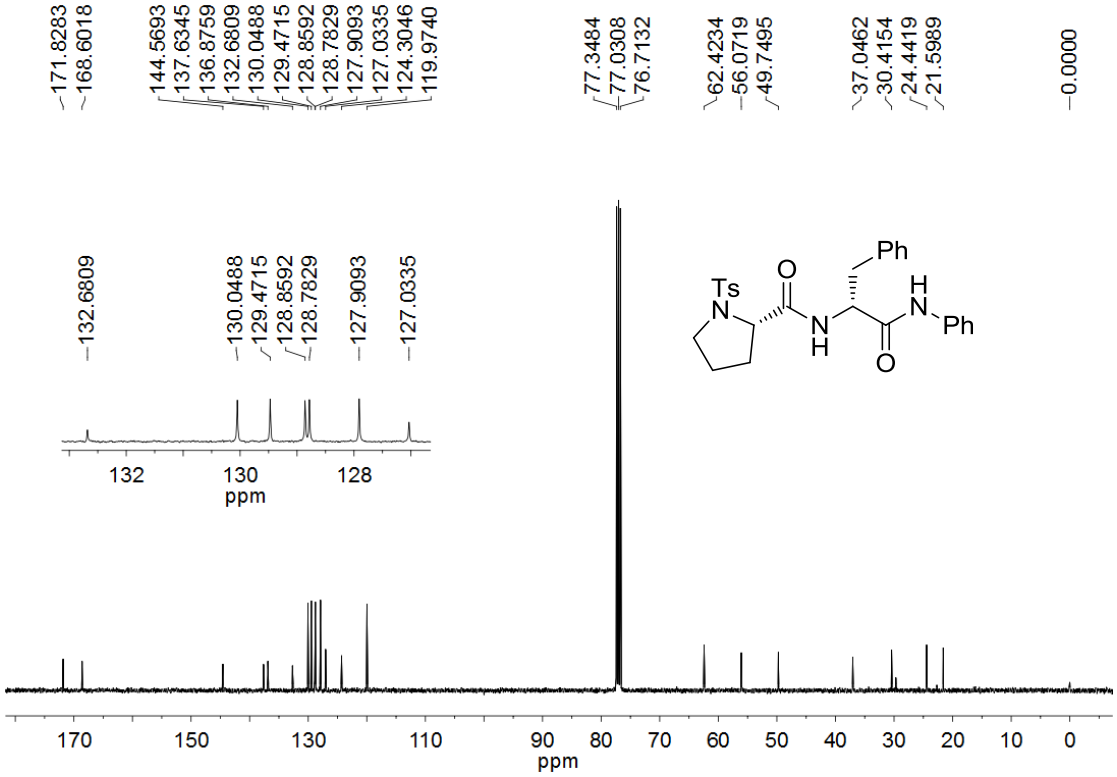


Figure S130. ^{13}C NMR spectrum of (*S*,*R*)-G**27** in $CDCl_3$ (100 MHz).

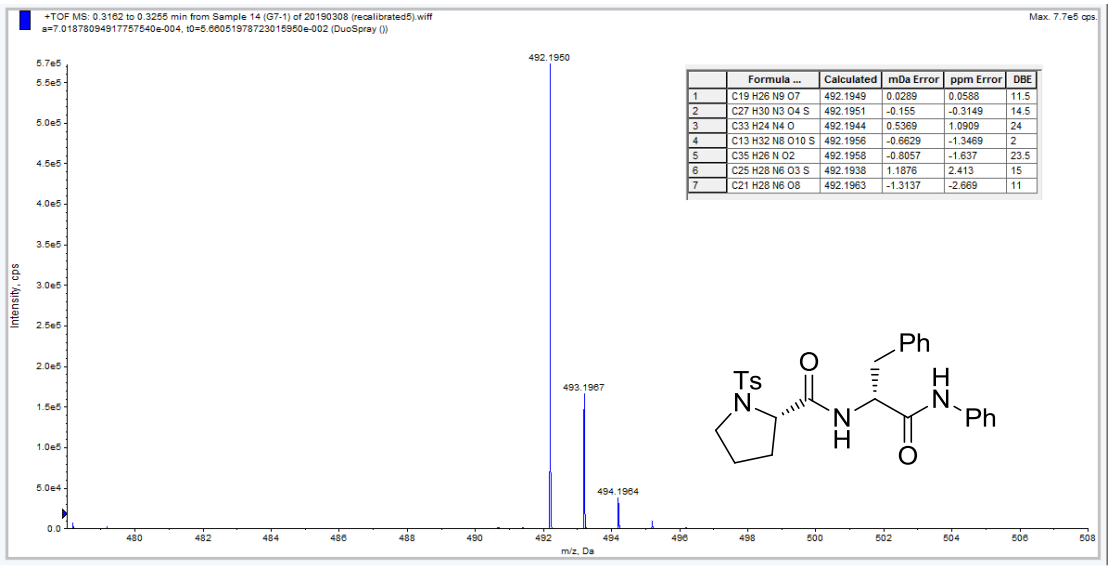


Figure S131. HRMS spectrum of (*S*,*R*)-G**27**.

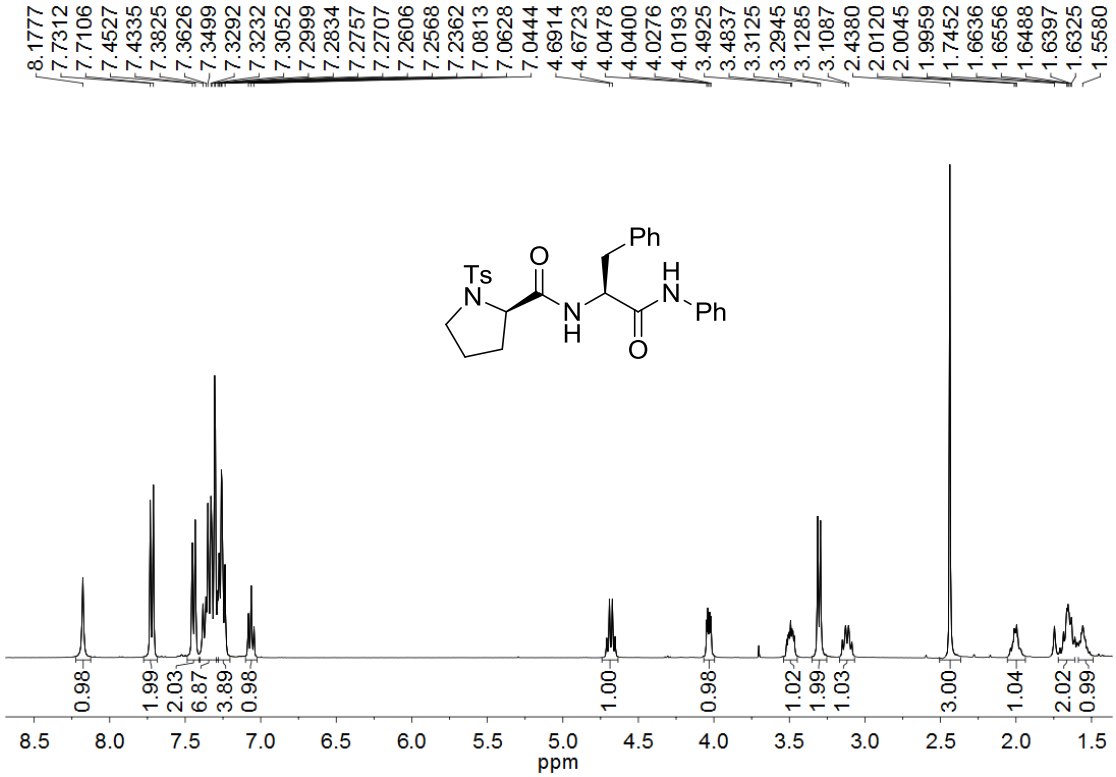


Figure S132. ^{1}H NMR spectrum of (*R*,*S*)-G**27** in $CDCl_3$ (400 MHz).

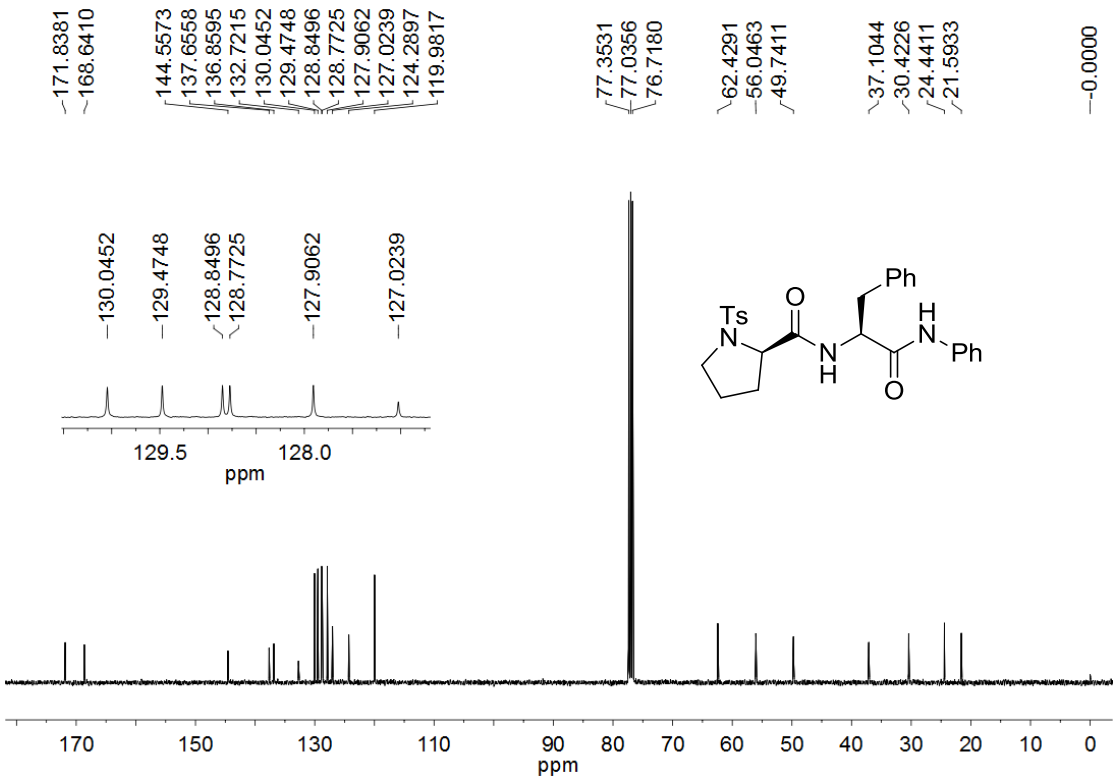


Figure S133. ^{13}C NMR spectrum of (*R*,*S*)-G**27** in $CDCl_3$ (100 MHz).

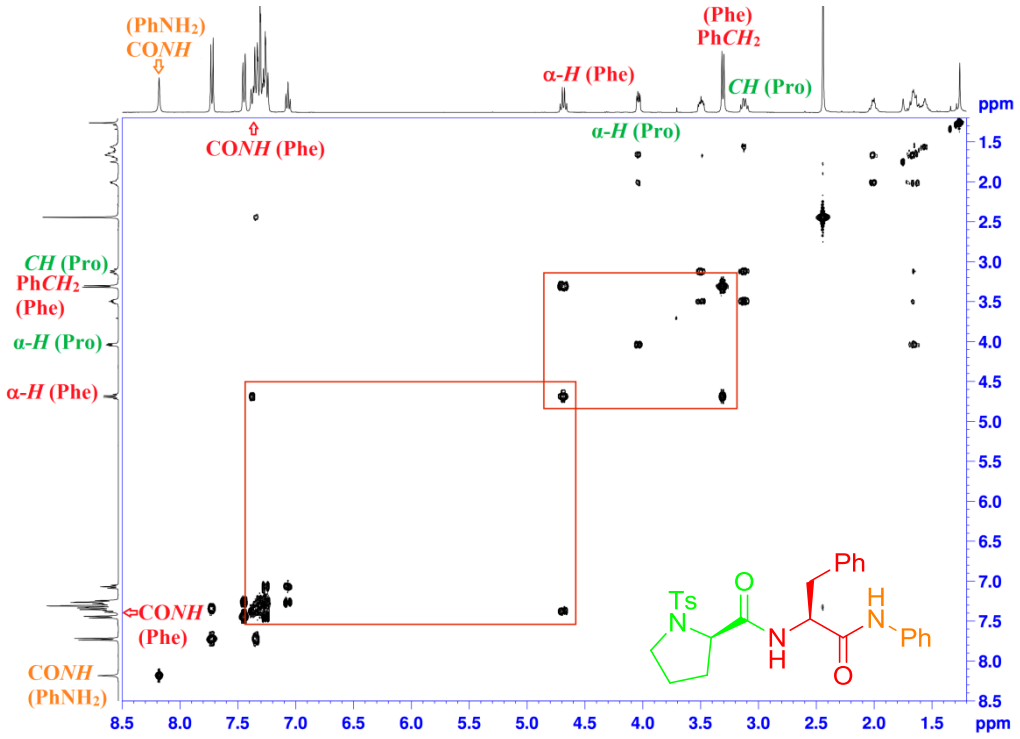


Figure S134. ^{1}H–^{1}H COSY spectrum of (*R*,*S*)-G**27** in $CDCl_3$ (400 MHz).

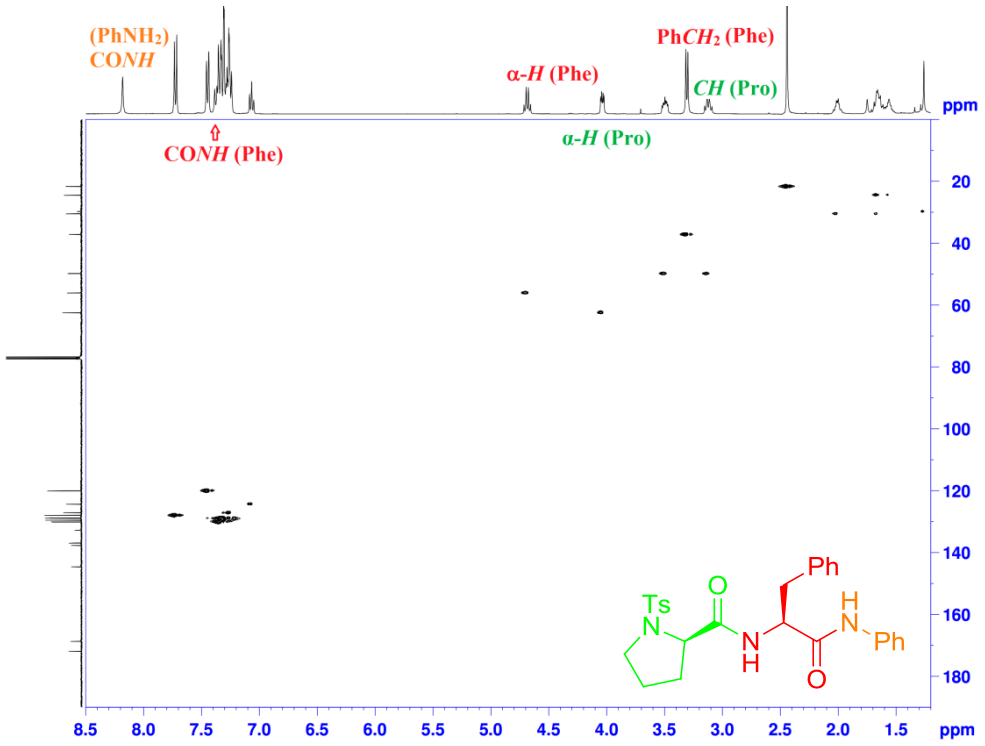


Figure S135. HSQC spectrum of (*R*,*S*)-G**27** in $CDCl_3$.

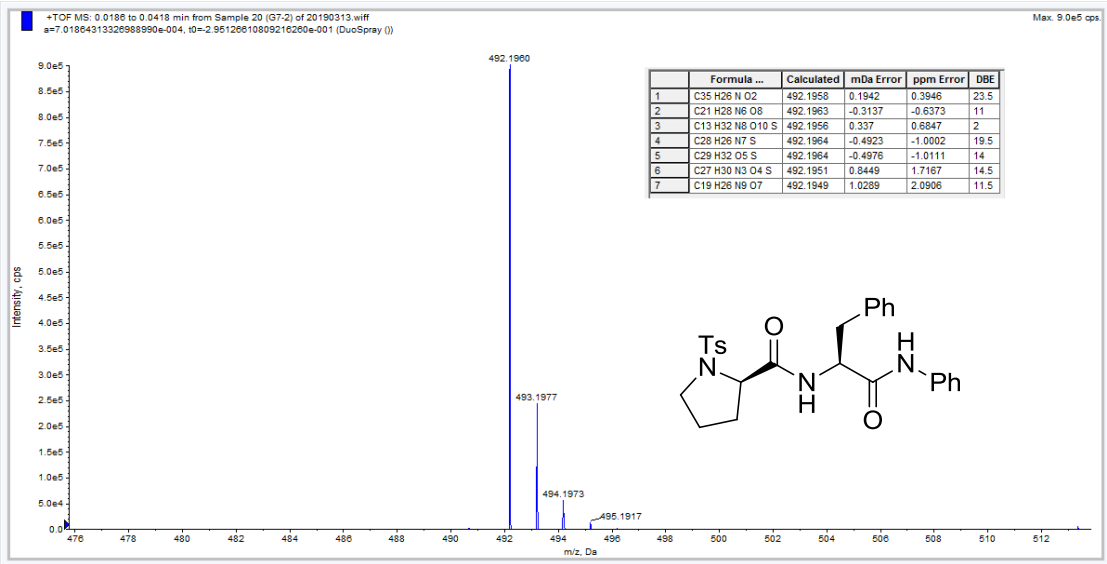


Figure S136. HRMS spectrum of (*R*,*S*)-G**27**.

^{1}H NMR spectra of enantiomeric discrimination of (±)-G16–27 in the presence of CSAs 1–9.

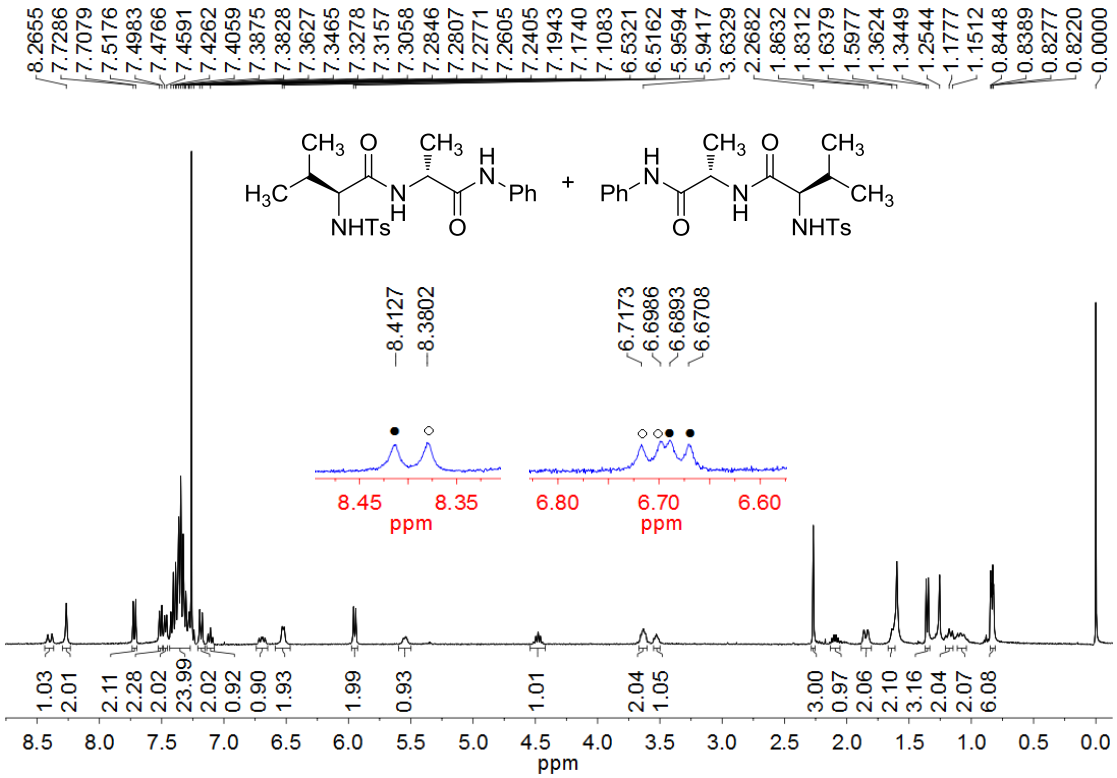


Figure S137. ^{1}H NMR spectrum of (±)-G**16** in the presence of CSA **1** in $CDCl_3$ (400 MHz), [(±)-G**16**] = 5 mM.

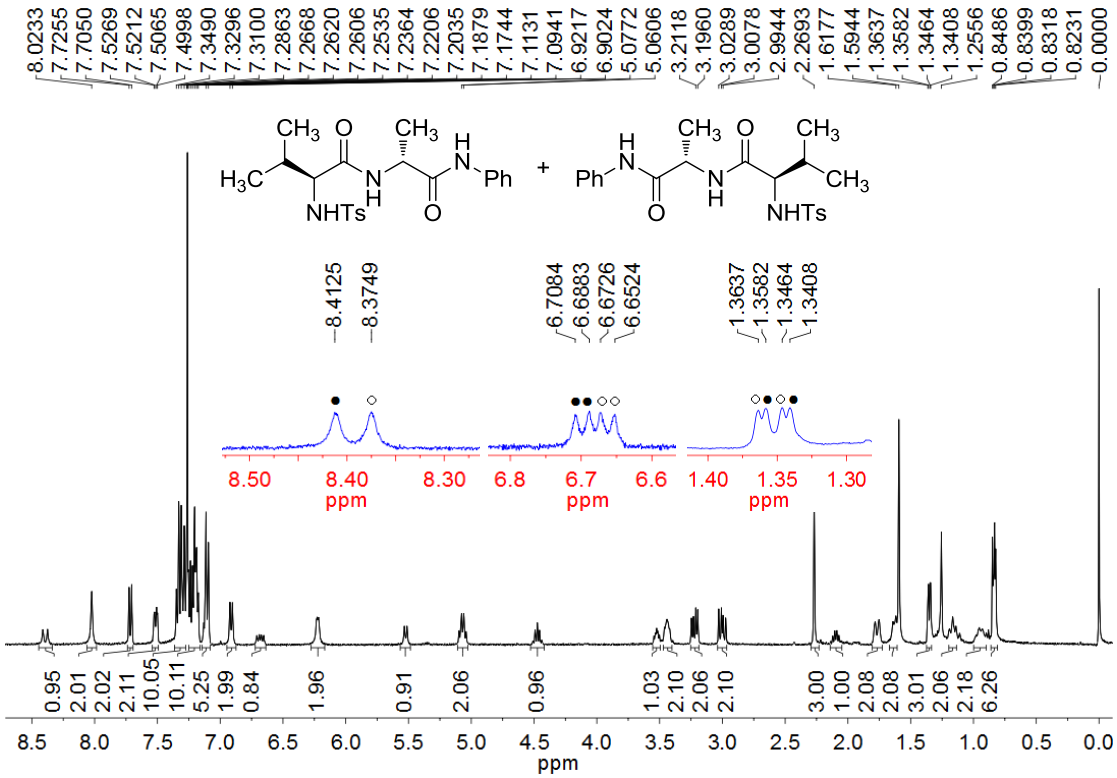


Figure S138. ^{1}H NMR spectrum of (±)-G**16** in the presence of CSA **2** in $CDCl_3$ (400 MHz), [(±)-G**16**] = 5 mM.

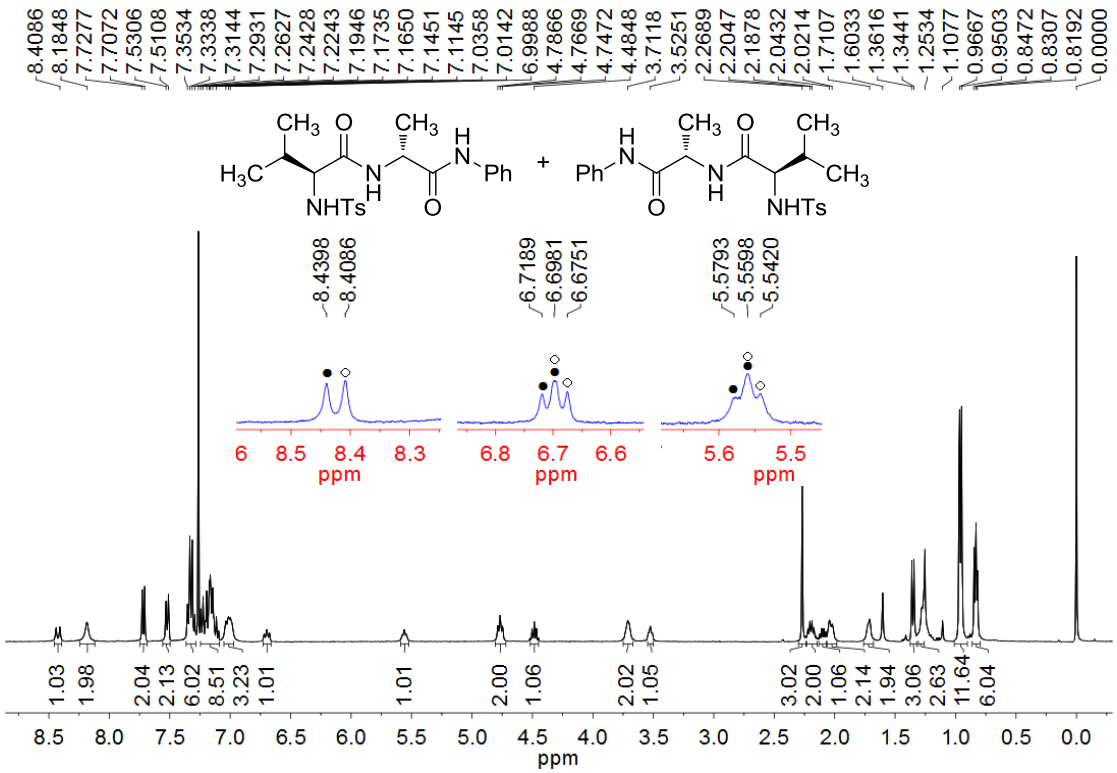


Figure S139. ^{1}H NMR spectrum of (±)-G**16** in the presence of CSA **3** in $CDCl_3$ (400 MHz), [(±)-G**16**] = 5 mM.

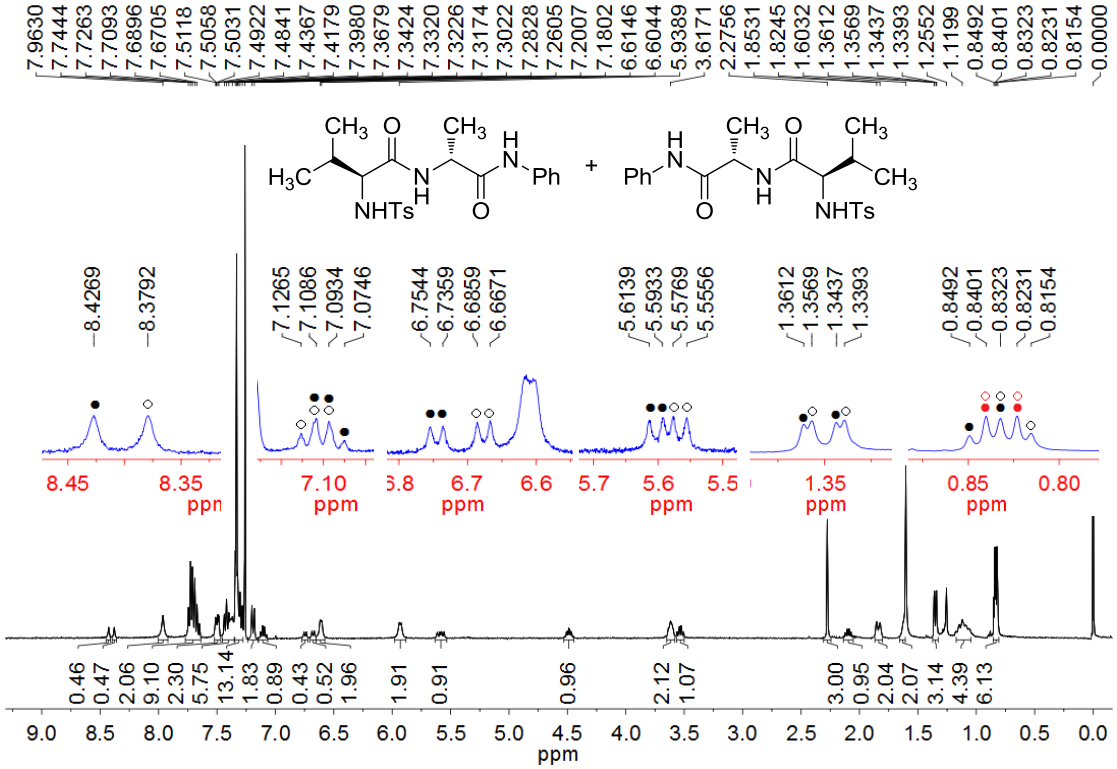


Figure S140. ^{1}H NMR spectrum of (±)-G**16** in the presence of CSA **4** in $CDCl_3$ (400 MHz), [(±)-G**16**] = 5 mM.

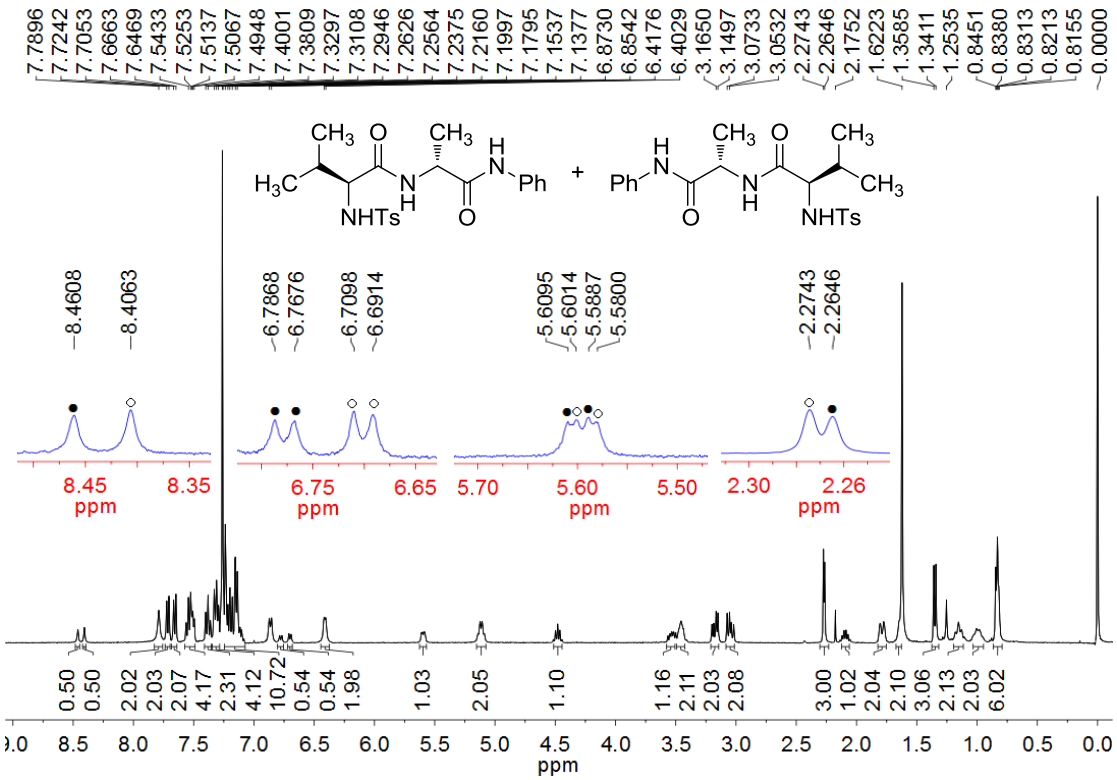


Figure S141. ^{1}H NMR spectrum of (±)-G**16** in the presence of CSA **5** in $CDCl_3$ (400 MHz), [(±)-G**16**] = 5 mM.

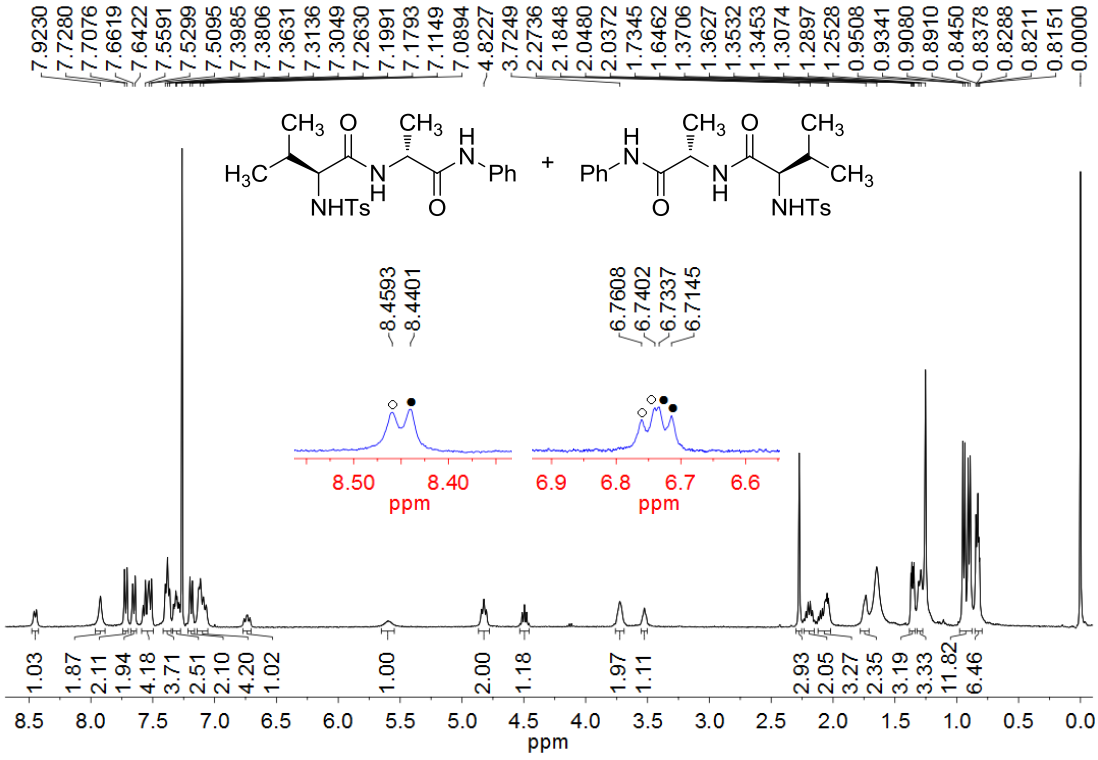


Figure S142. ^{1}H NMR spectrum of (±)-G**16** in the presence of CSA **6** in $CDCl_3$ (400 MHz), [(±)-G**16**] = 5 mM.

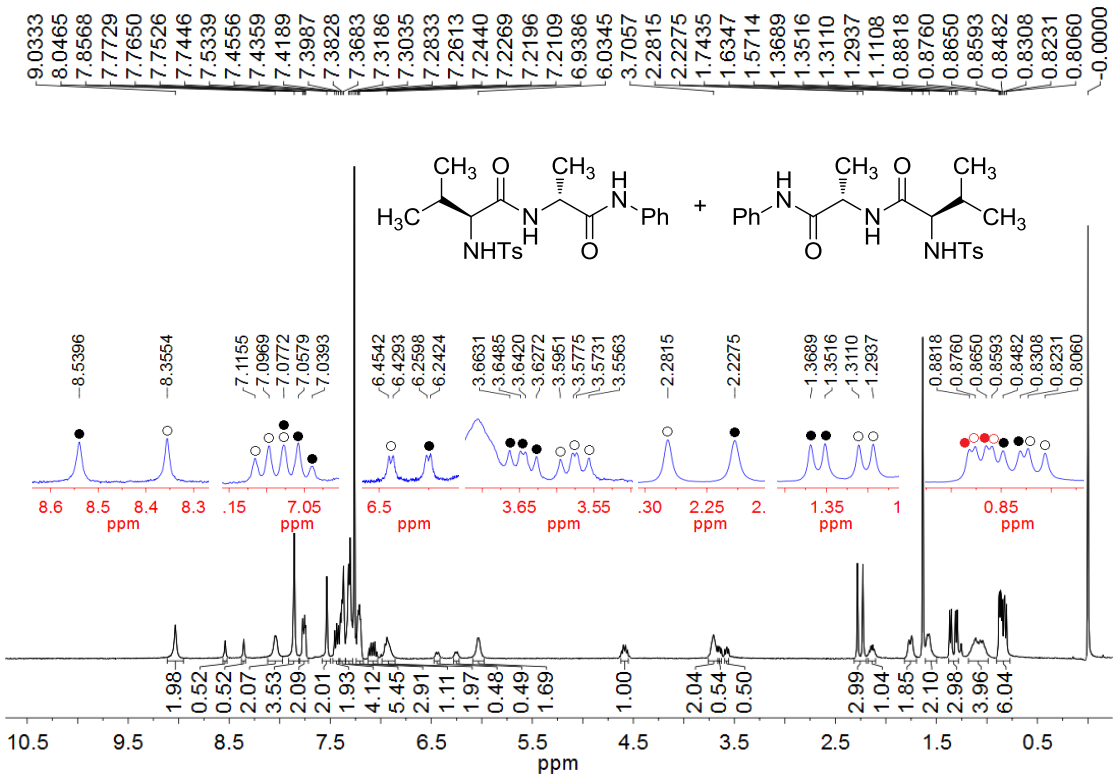


Figure S143. ^{1}H NMR spectrum of (±)-G**16** in the presence of CSA **7** in $CDCl_3$ (400 MHz), [(±)-G**16**] = 5 mM.

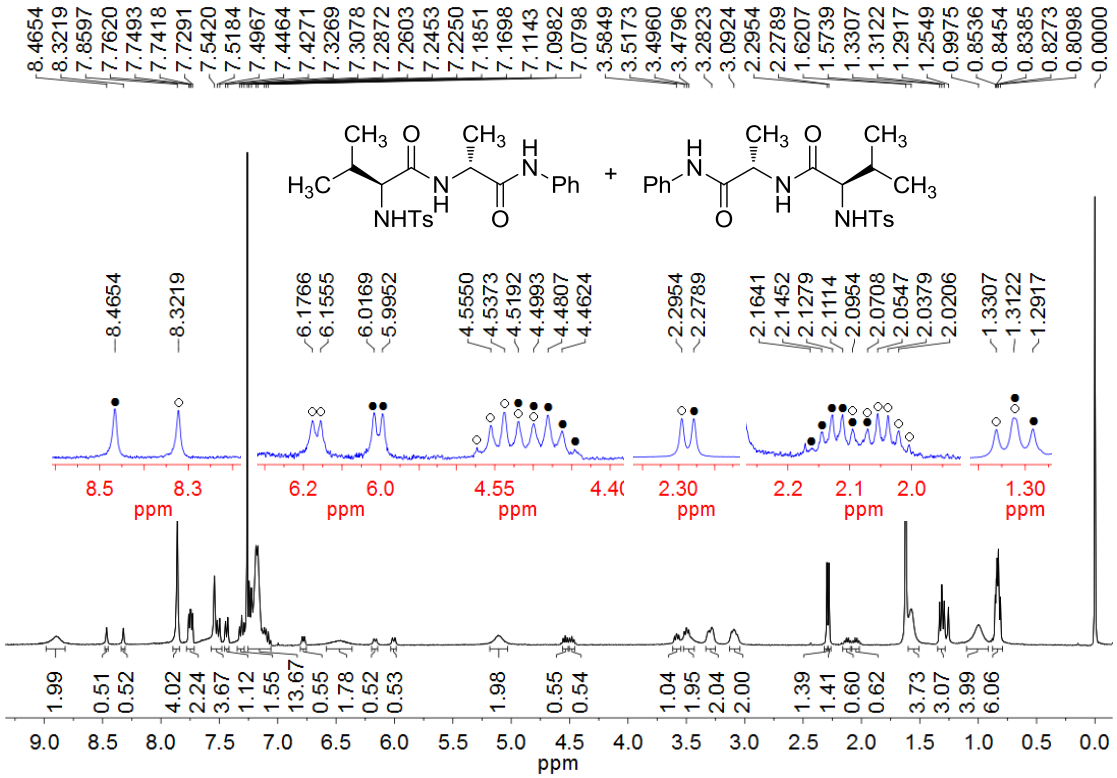


Figure S144. ^{1}H NMR spectrum of (±)-G**16** in the presence of CSA **8** in $CDCl_3$ (400 MHz), [(±)-G**16**] = 5 mM.

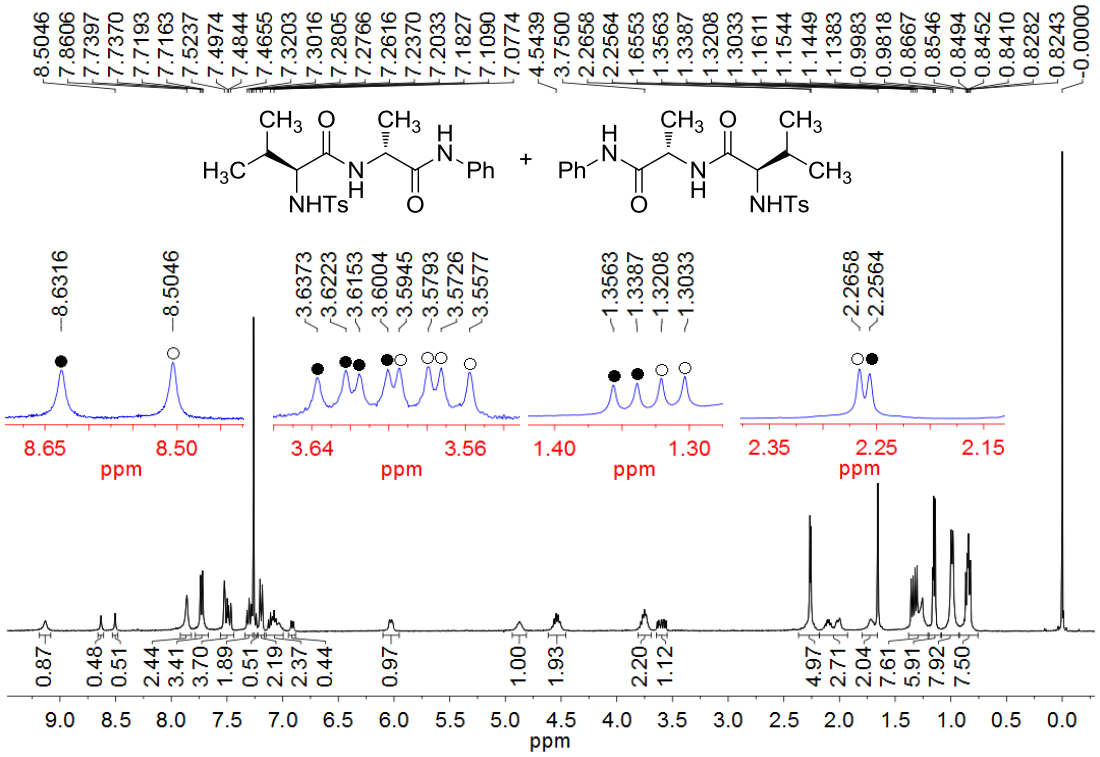


Figure S145. ^{1}H NMR spectrum of (±)-G**16** in the presence of CSA **9** in $CDCl_3$ (400 MHz), [(±)-G**16**] = 5 mM.

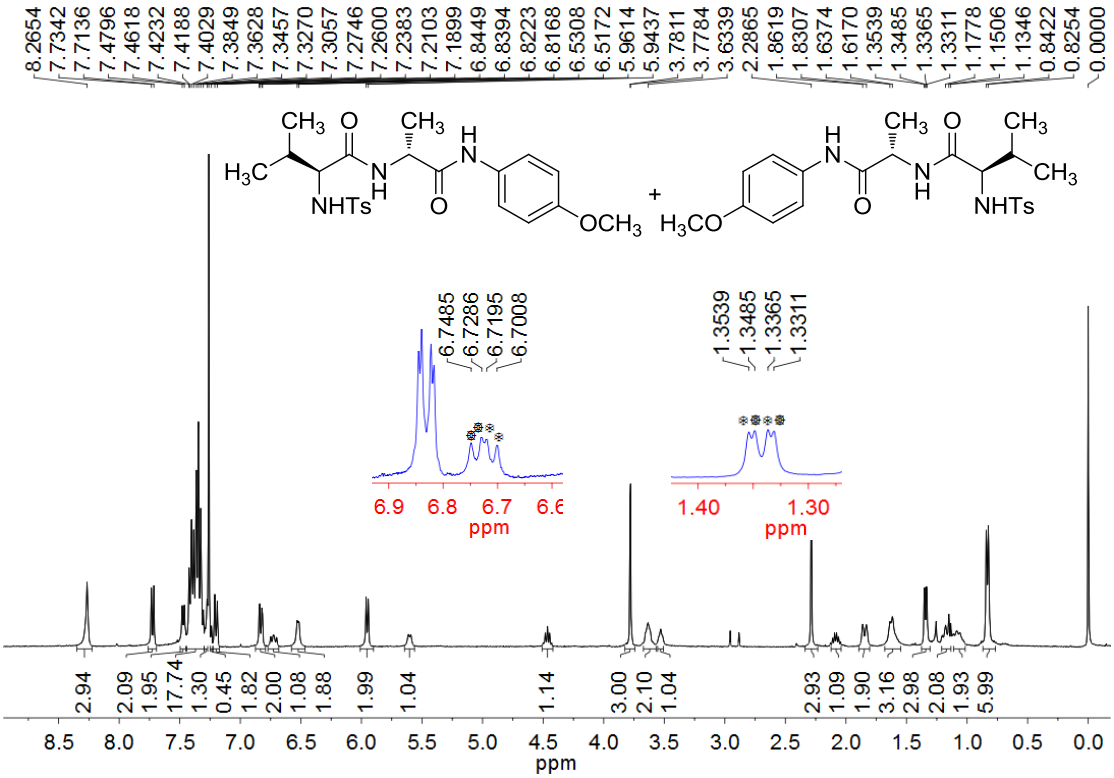


Figure S146. ^{1}H NMR spectrum of (±)-G**17** in the presence of CSA **1** in $CDCl_3$ (400 MHz), [(±)-G**17**] = 5 mM.

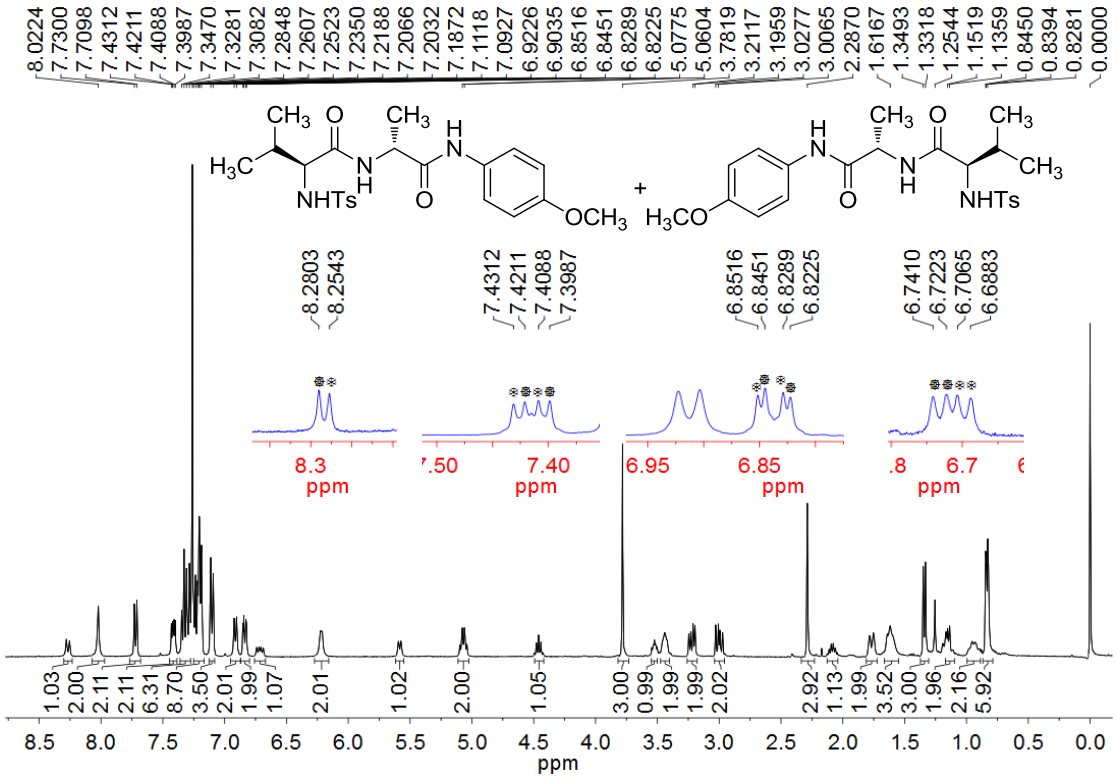


Figure S147. ^{1}H NMR spectrum of (±)-G**17** in the presence of CSA **2** in $CDCl_3$ (400 MHz), [(±)-G**17**] = 5 mM.

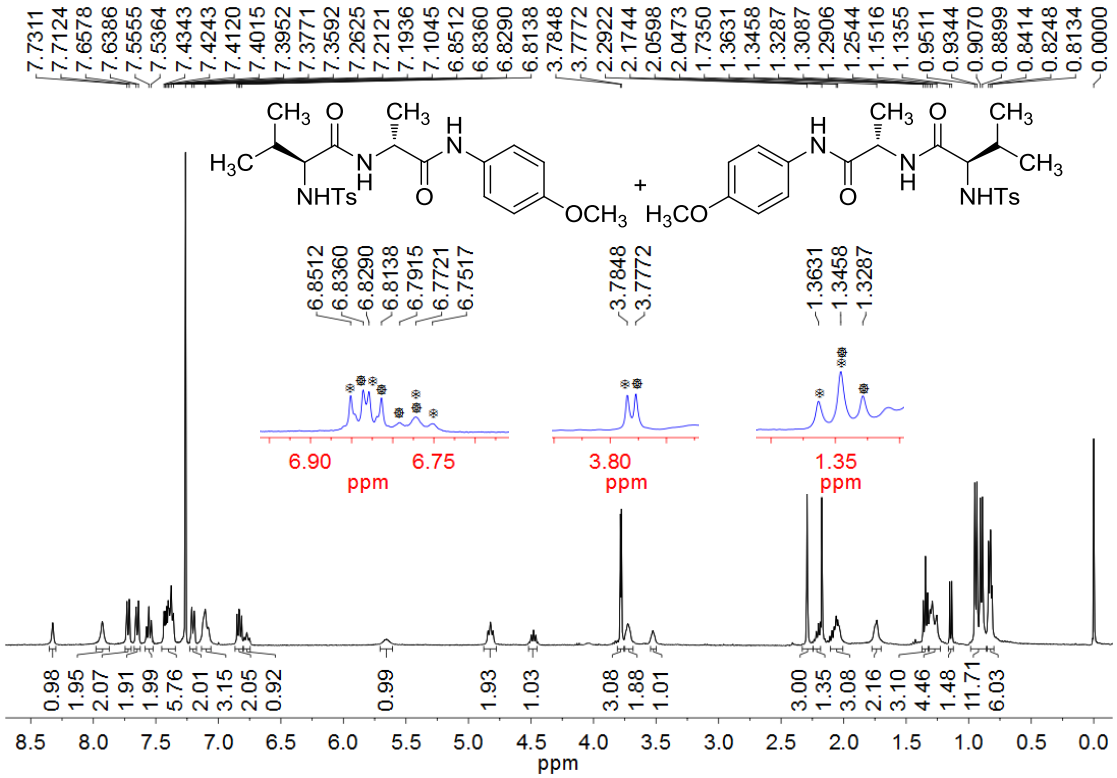


Figure S148. ^{1}H NMR spectrum of (±)-G**17** in the presence of CSA **3** in $CDCl_3$ (400 MHz), [(±)-G**17**] = 5 mM.

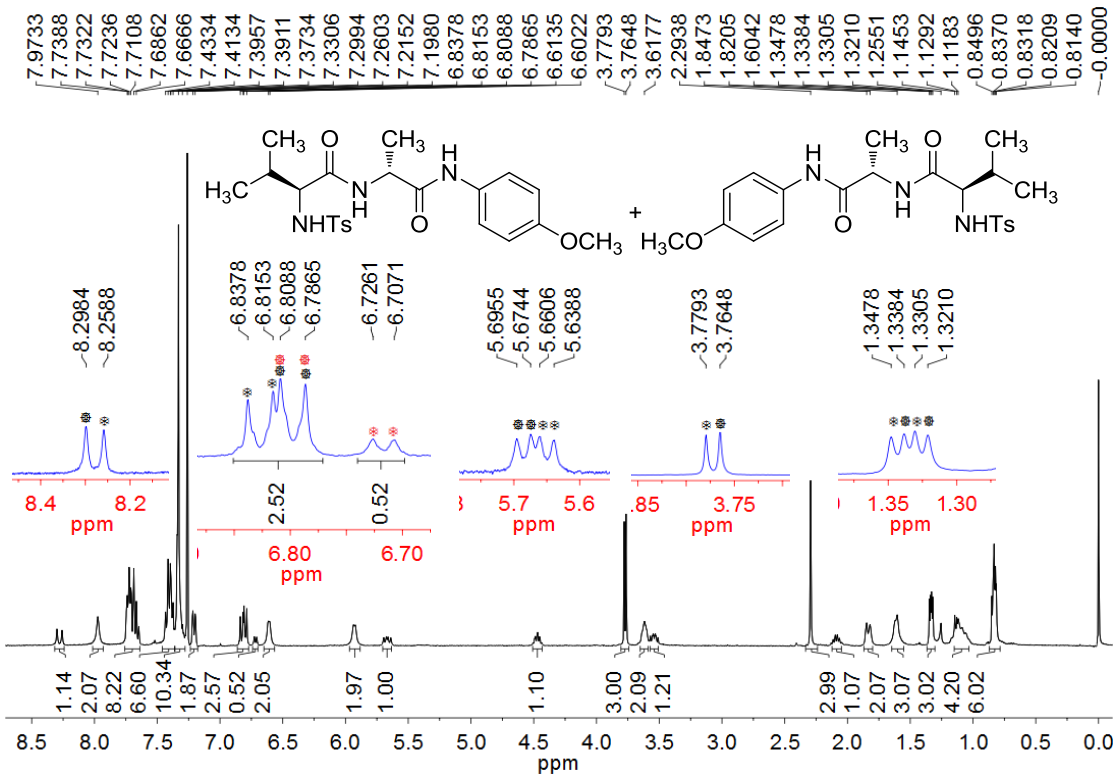


Figure S149. ^{1}H NMR spectrum of (±)-G**17** in the presence of CSA **4** in $CDCl_3$ (400 MHz), [(±)-G**17**] = 5 mM.

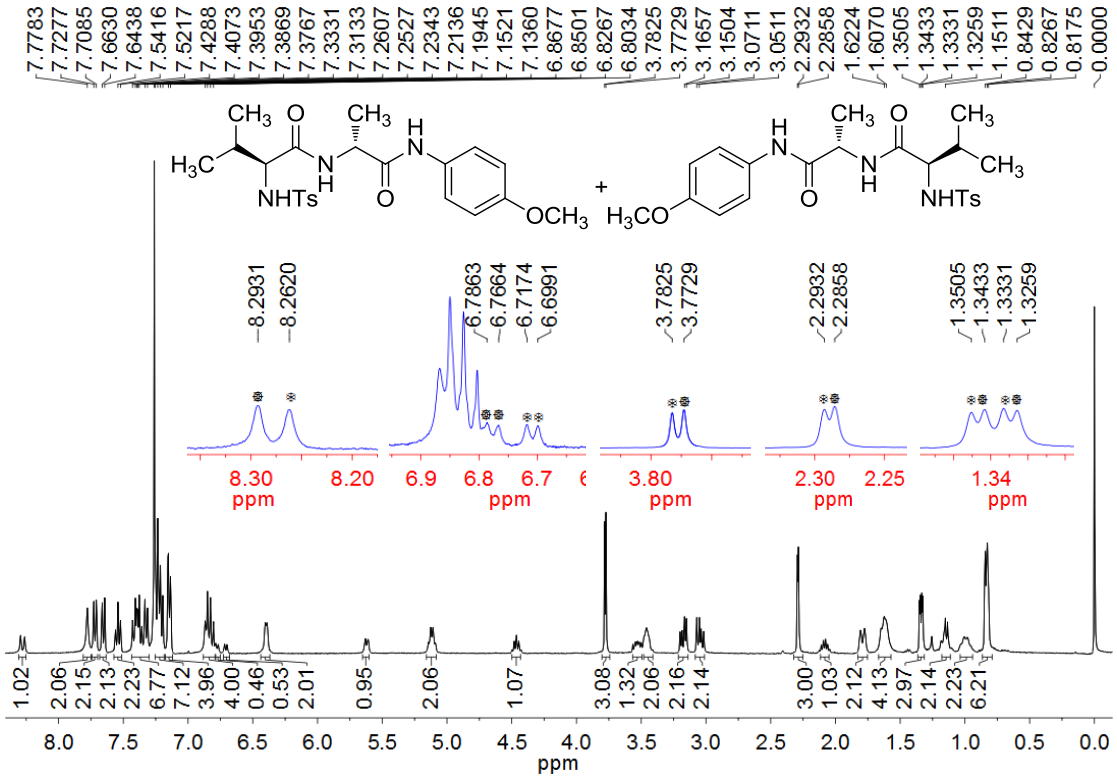


Figure S150. ^{1}H NMR spectrum of (±)-G**17** in the presence of CSA **5** in $CDCl_3$ (400 MHz), [(±)-G**17**] = 5 mM.

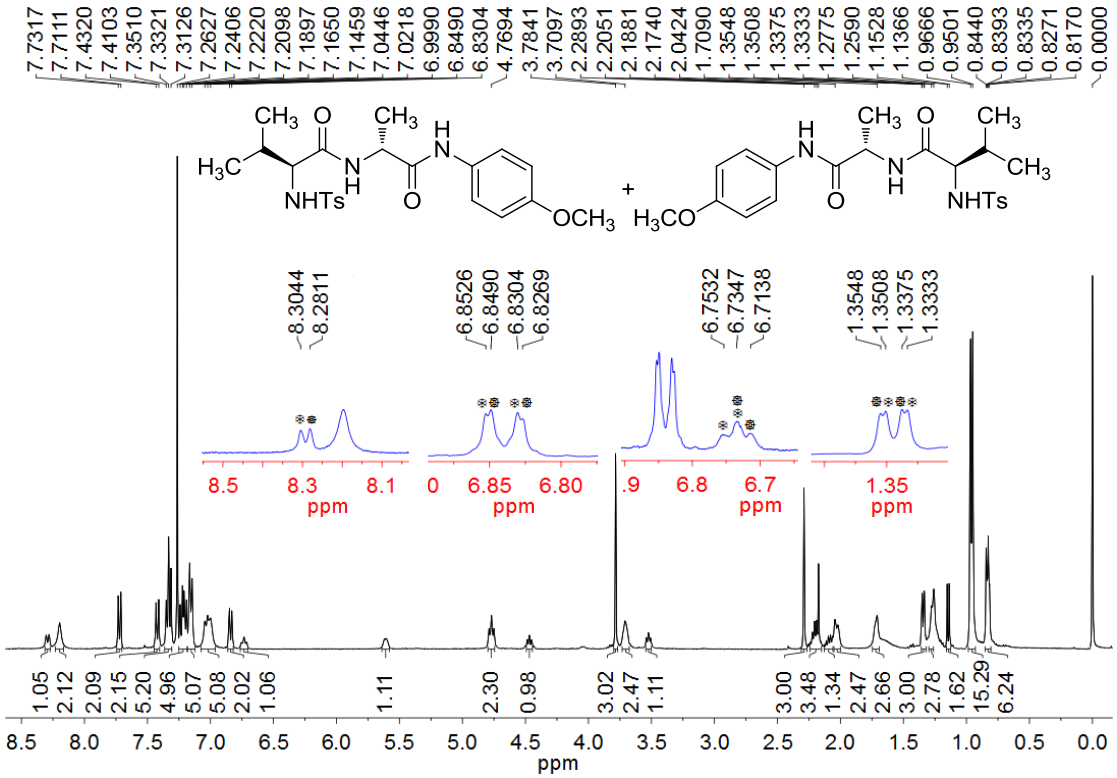


Figure S151. ^{1}H NMR spectrum of (±)-G**17** in the presence of CSA **6** in $CDCl_3$ (400 MHz), [(±)-G**17**] = 5 mM.

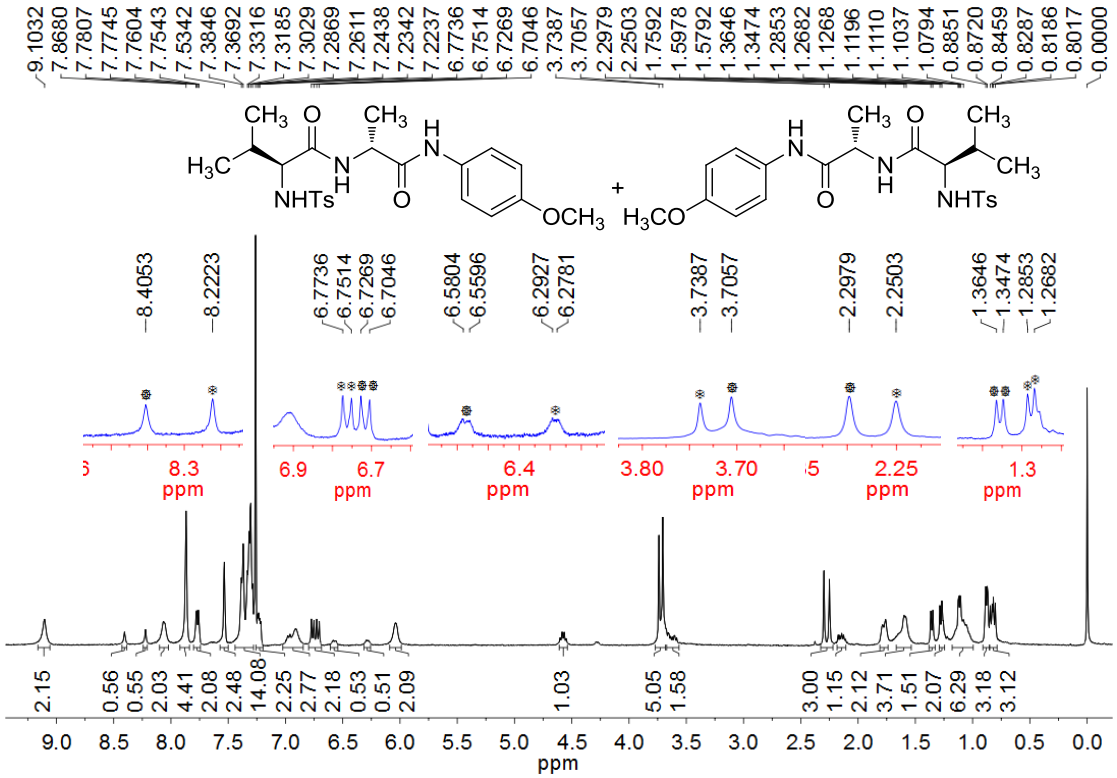


Figure S152. ^{1}H NMR spectrum of (±)-G**17** in the presence of CSA **7** in $CDCl_3$ (400 MHz), [(±)-G**17**] = 5 mM.

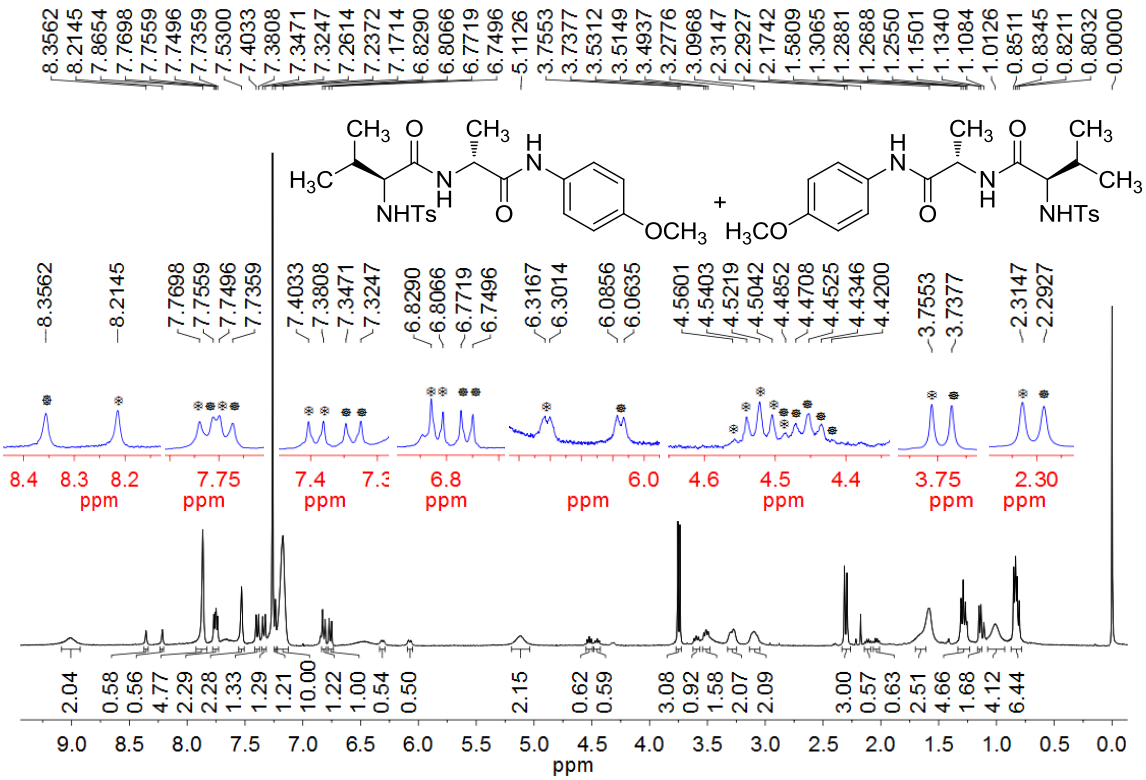


Figure S153. ^{1}H NMR spectrum of (±)-G**17** in the presence of CSA **8** in $CDCl_3$ (400 MHz), [(±)-G**17**] = 5 mM.

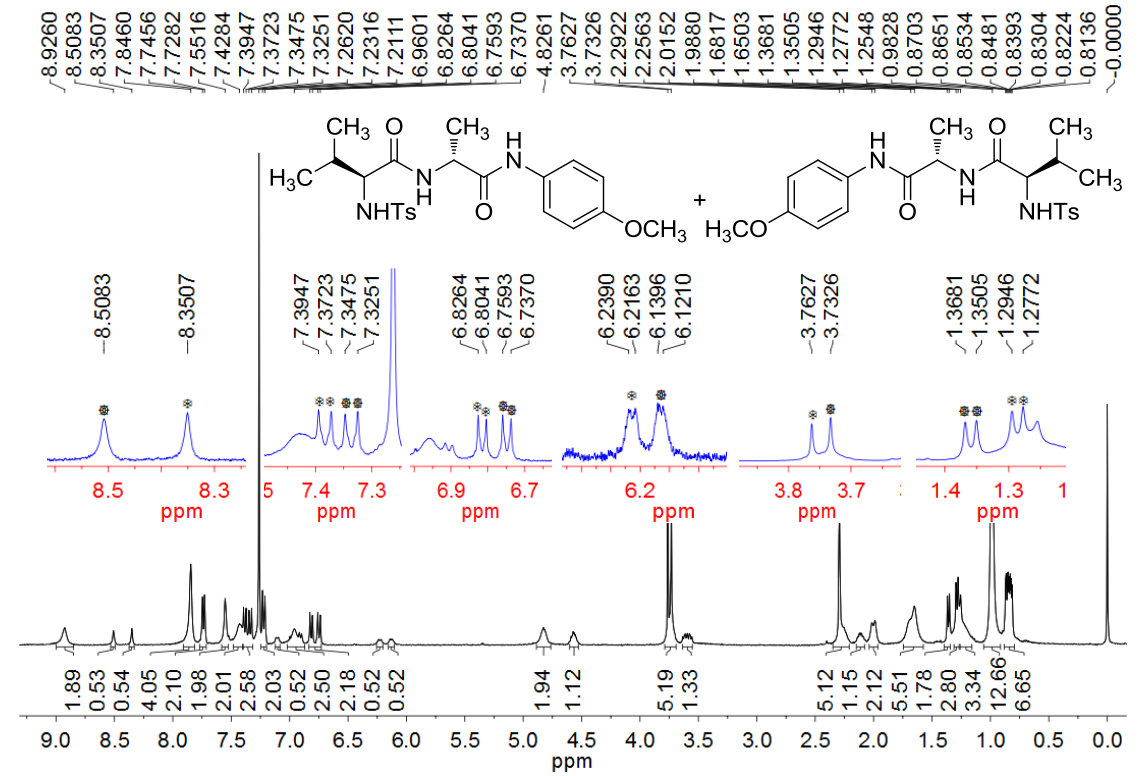


Figure S154. ^{1}H NMR spectrum of (±)-G**17** in the presence of CSA **9** in $CDCl_3$ (400 MHz), [(±)-G**17**] = 5 mM.

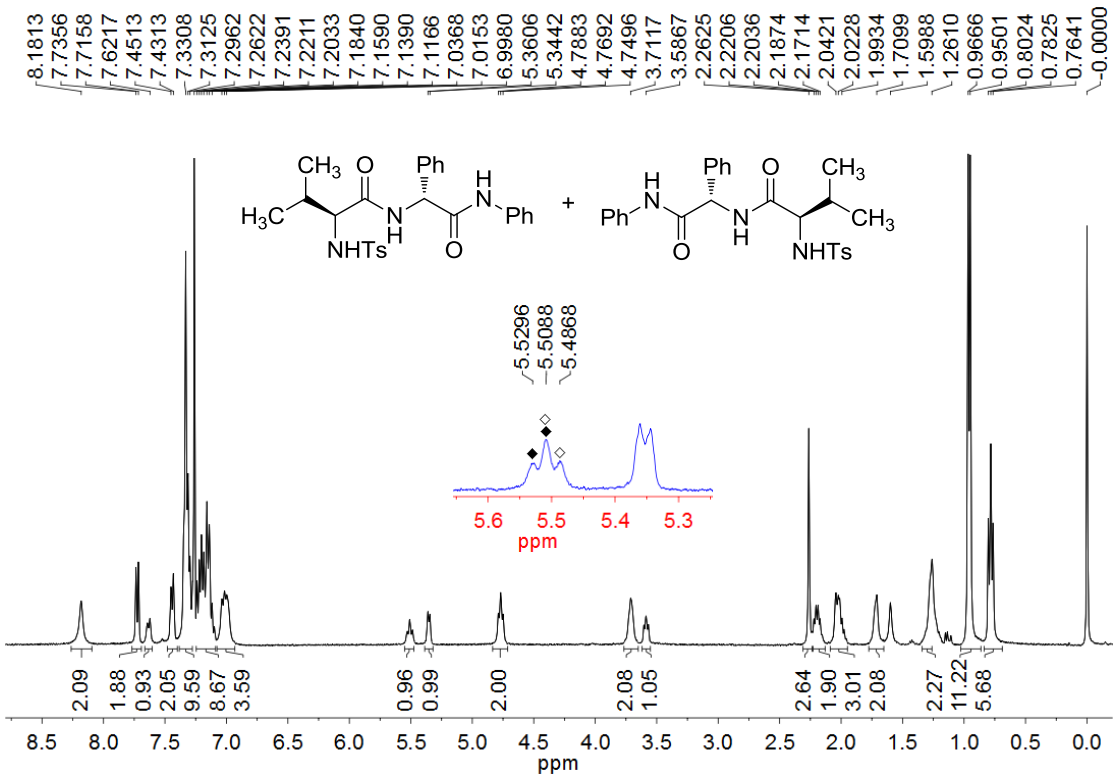


Figure S155. ^{1}H NMR spectrum of (±)-G**18** in the presence of CSA **3** in $CDCl_3$ (400 MHz), [(±)-G**18**] = 5 mM.

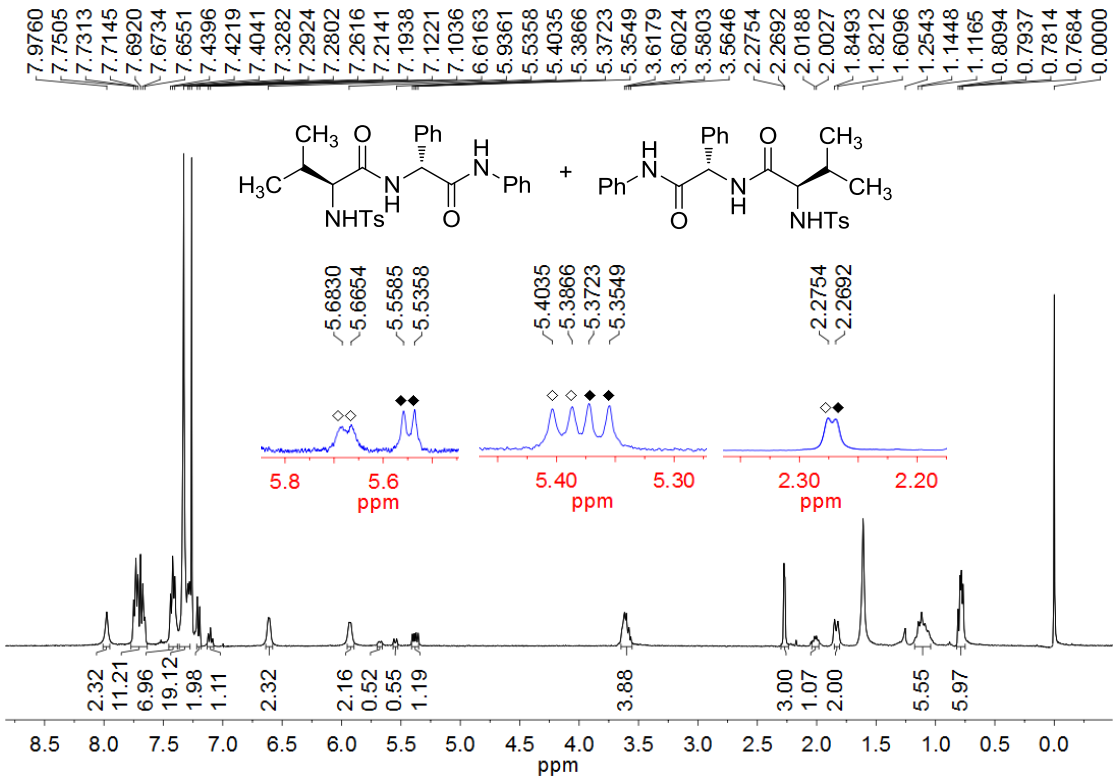


Figure S156. ^{1}H NMR spectrum of (±)-G**18** in the presence of CSA **4** in $CDCl_3$ (400 MHz), [(±)-G**18** = 5 mM.

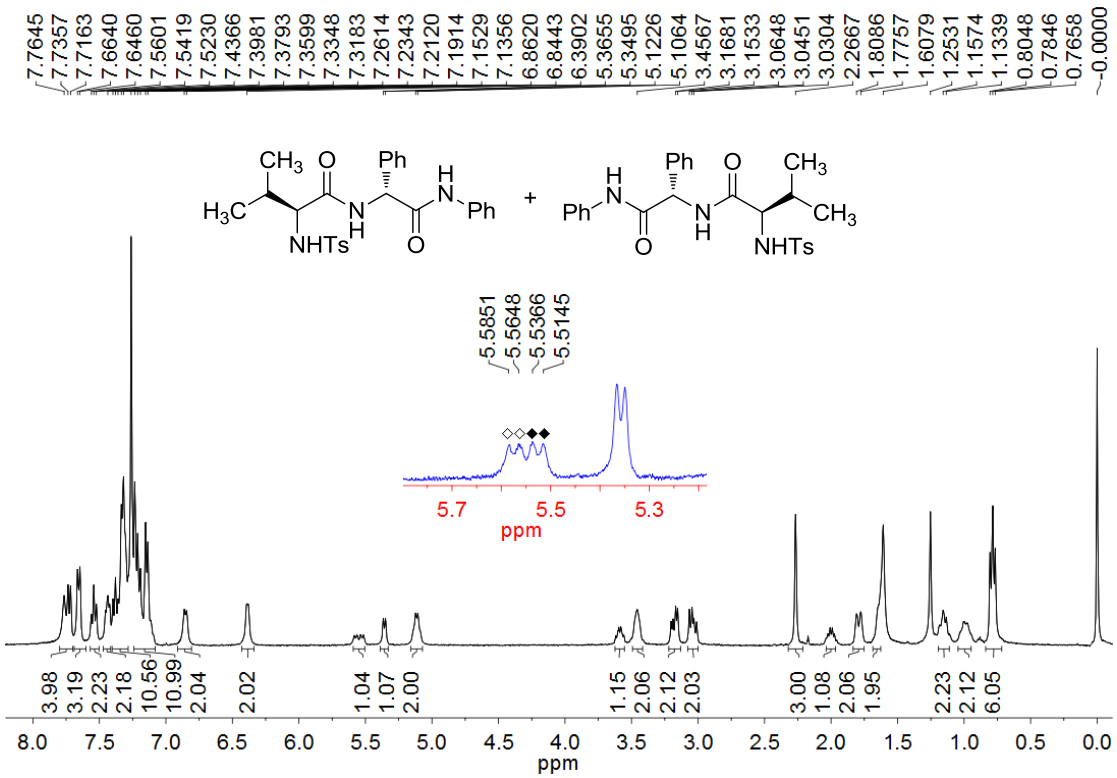


Figure S157. ^{1}H NMR spectrum of (±)-G**18** in the presence of CSA **5** in $CDCl_3$ (400 MHz), [(±)-G**18**] = 5 mM.

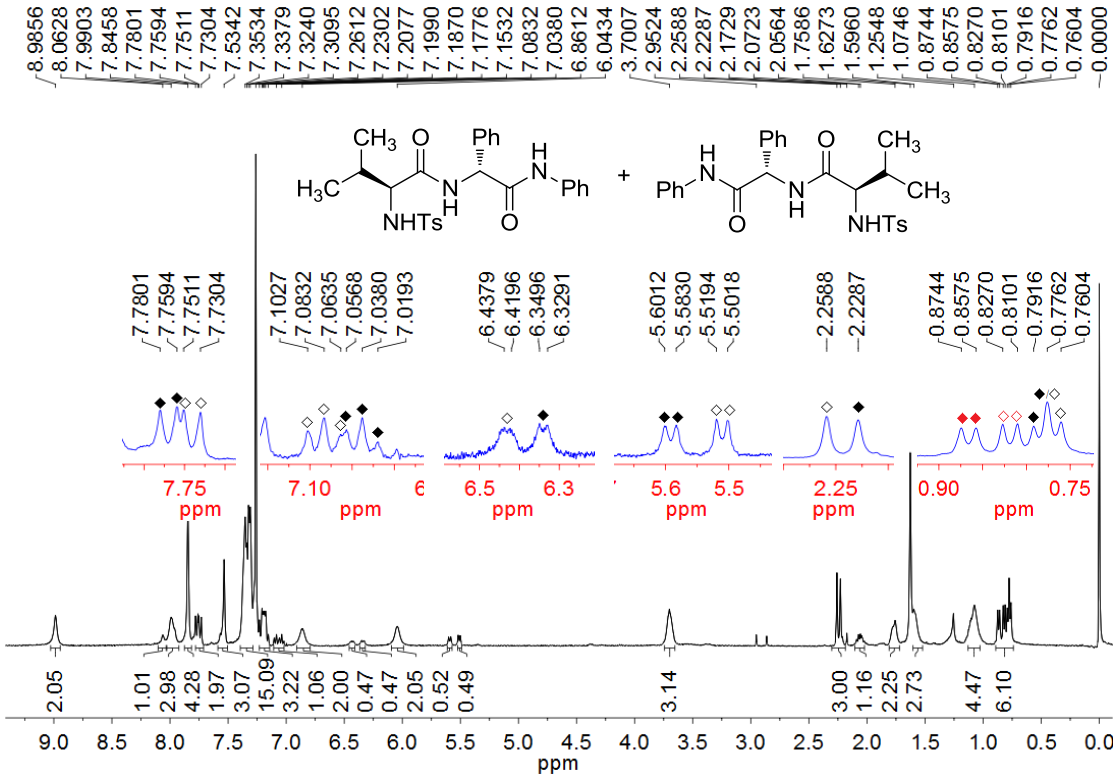


Figure S158. ^{1}H NMR spectrum of (±)-G**18** in the presence of CSA **7** in $CDCl_3$ (400 MHz), [(±)-G**18**] = 5 mM.

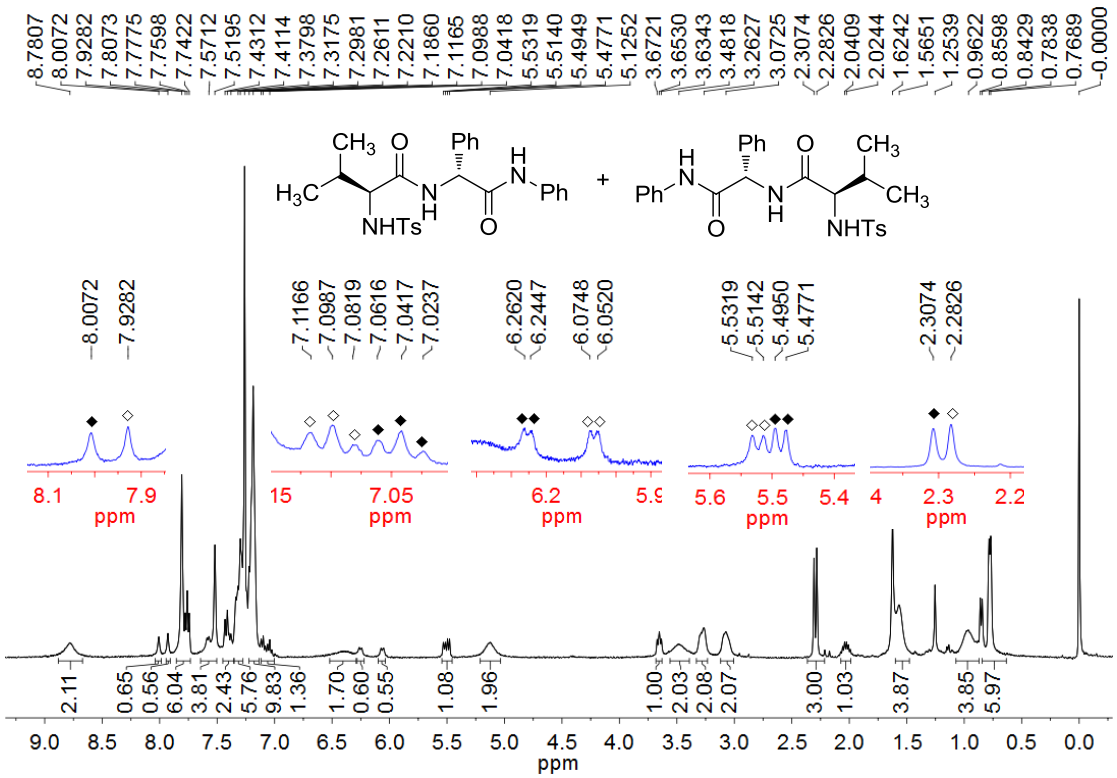


Figure S159. ^{1}H NMR spectrum of (±)-G**18** in the presence of CSA **8** in $CDCl_3$ (400 MHz), [(±)-G**18**] = 5 mM.

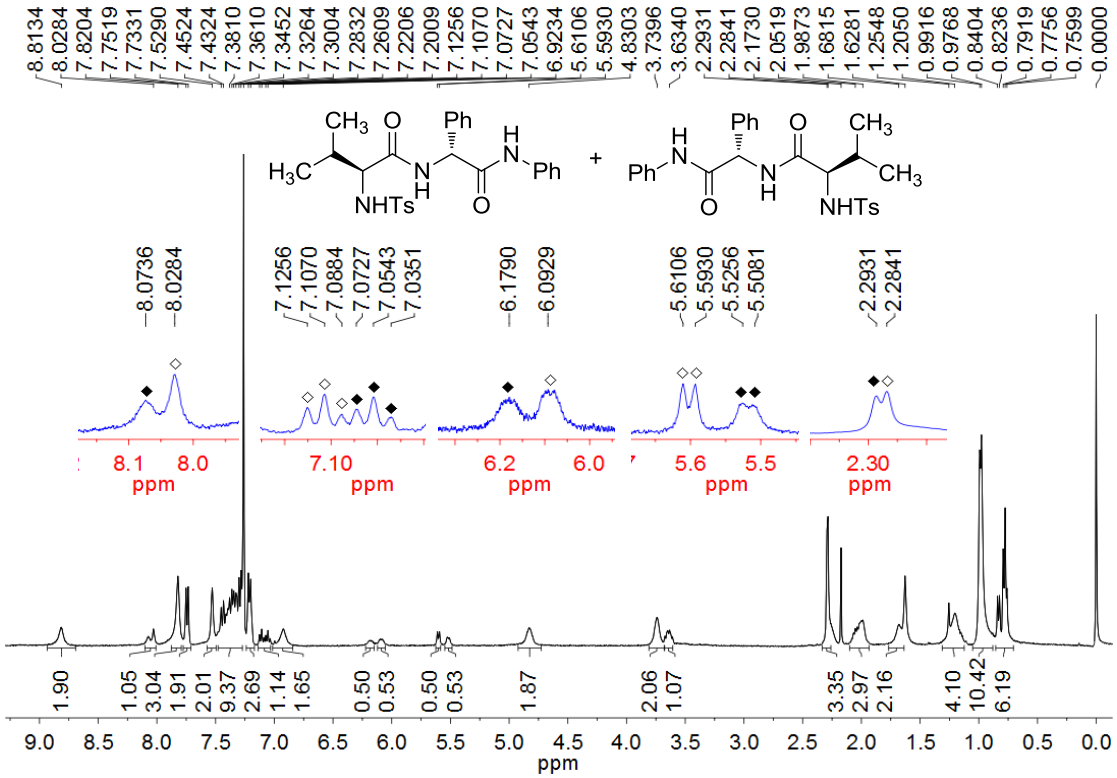


Figure S160. ^{1}H NMR spectrum of (±)-G**18** in the presence of CSA **9** in $CDCl_3$ (400 MHz), [(±)-G**18**] = 5 mM.

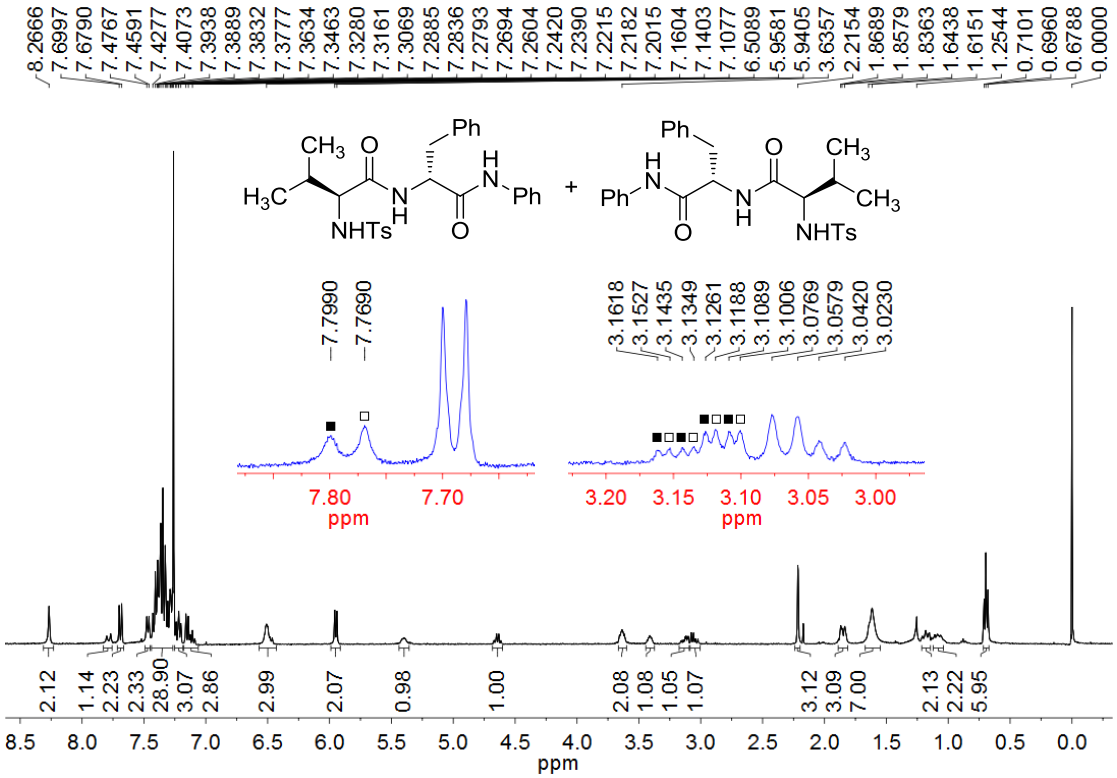


Figure S161. ^{1}H NMR spectrum of (±)-G**19** in the presence of CSA **1** in $CDCl_3$ (400 MHz), [(±)-G**19**] = 5 mM.

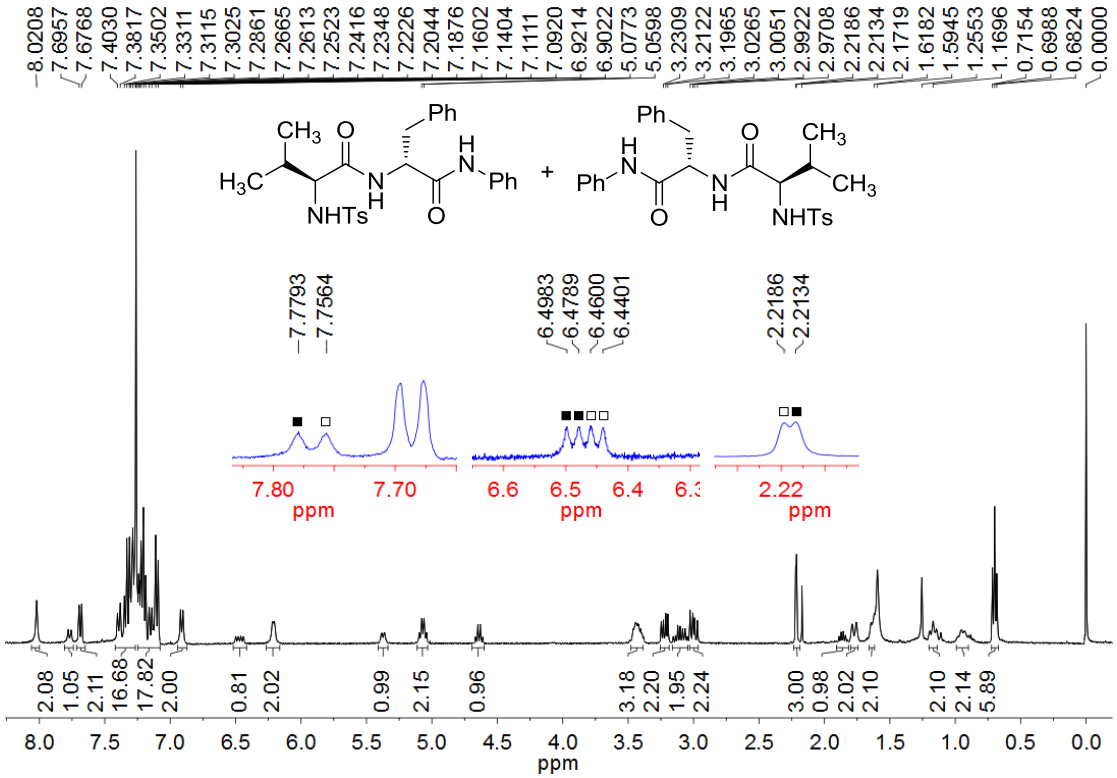


Figure S162. ^{1}H NMR spectrum of (±)-G**19** in the presence of CSA **2** in $CDCl_3$ (400 MHz), [(±)-G**19**] = 5 mM.

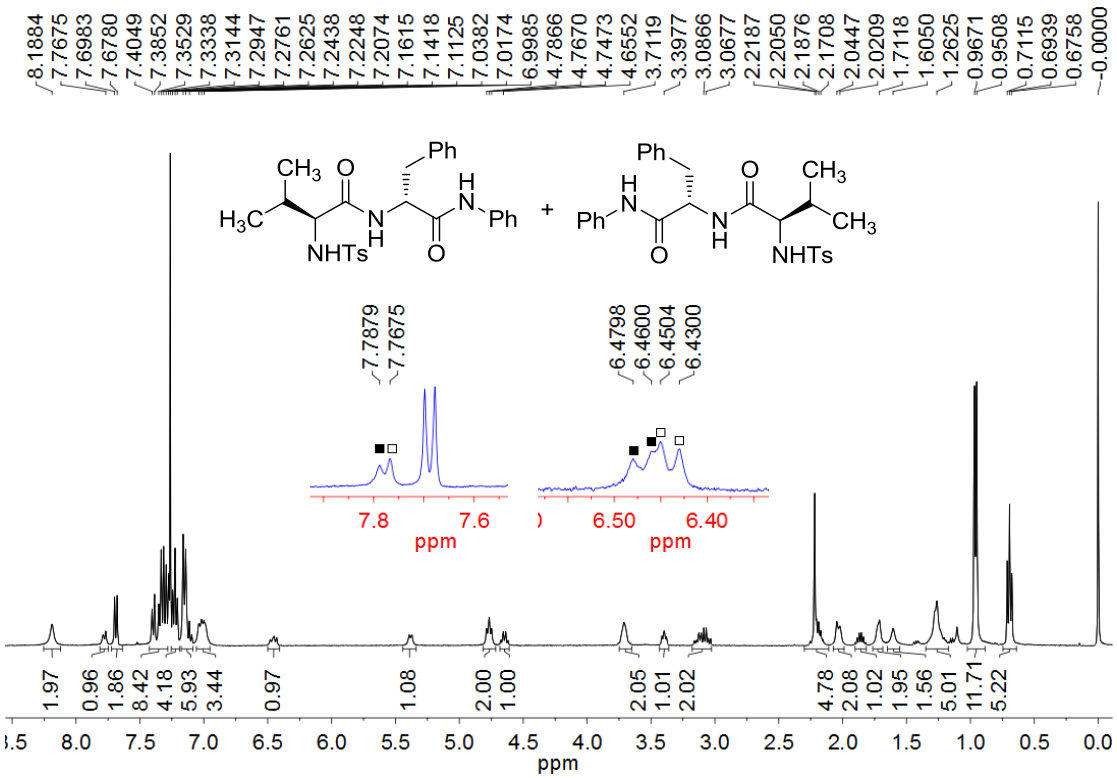


Figure S163. ^{1}H NMR spectrum of (±)-G**19** in the presence of CSA **3** in $CDCl_3$ (400 MHz), [(±)-G**19**] = 5 mM.

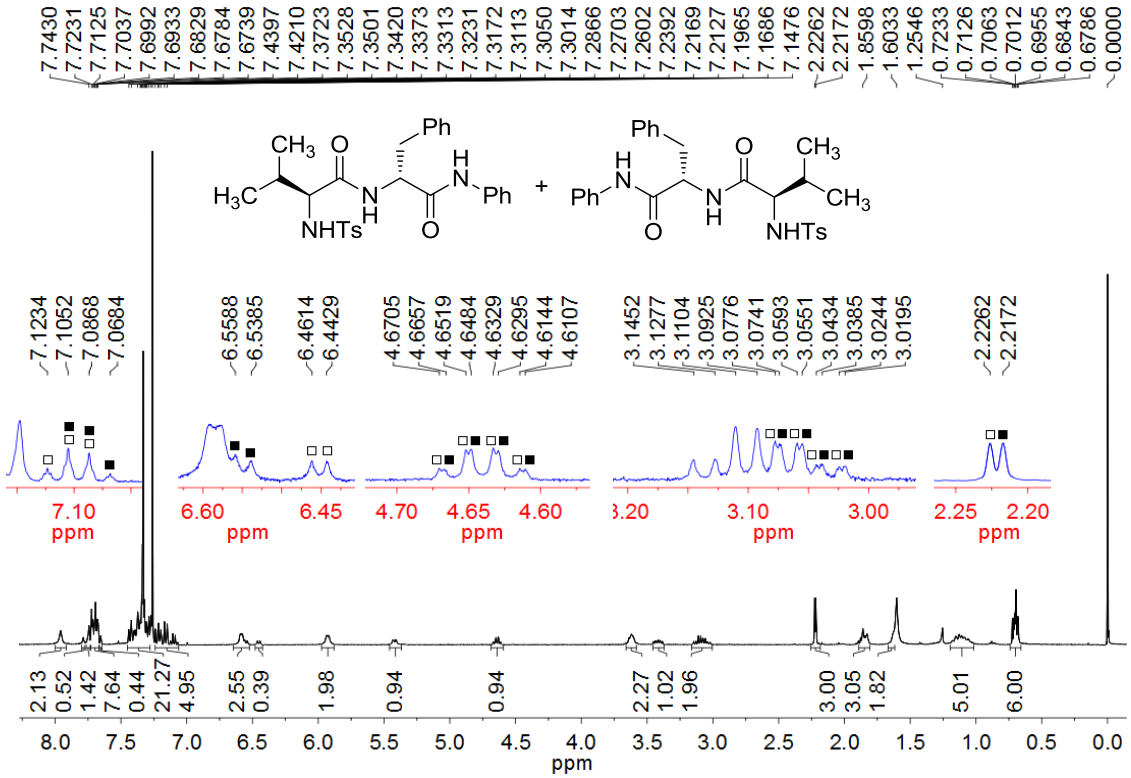


Figure S164. ^{1}H NMR spectrum of (±)-G**19** in the presence of CSA **4** in $CDCl_3$ (400 MHz), [(±)-G**19**] = 5 mM.

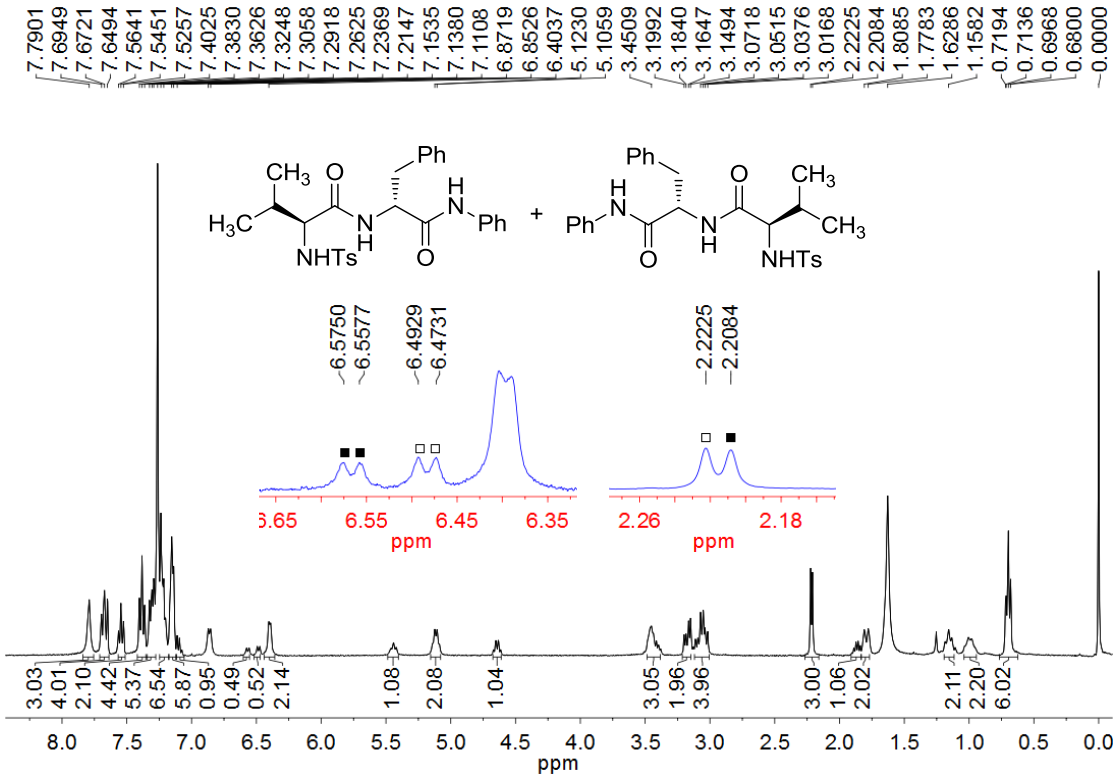


Figure S165. ^{1}H NMR spectrum of (±)-G**19** in the presence of CSA **5** in $CDCl_3$ (400 MHz), [(±)-G**19**] = 5 mM.

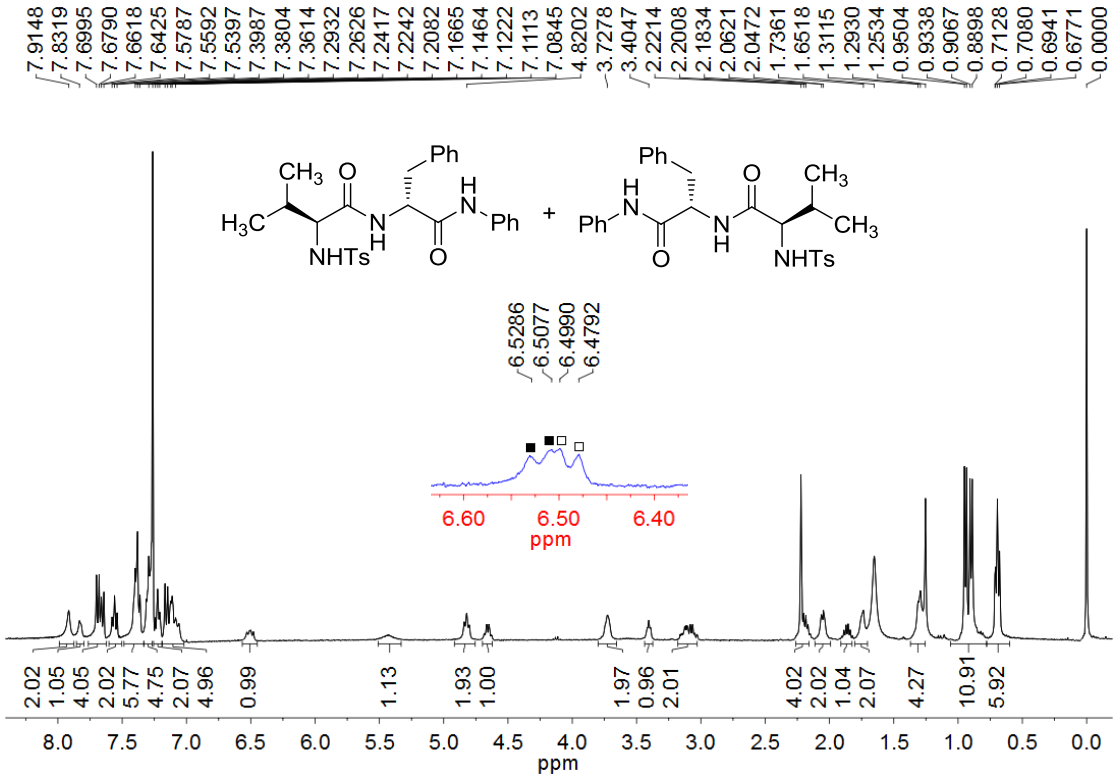


Figure S166. ^{1}H NMR spectrum of (±)-G**19** in the presence of CSA **6** in $CDCl_3$ (400 MHz), [(±)-G**19**] = 5 mM.

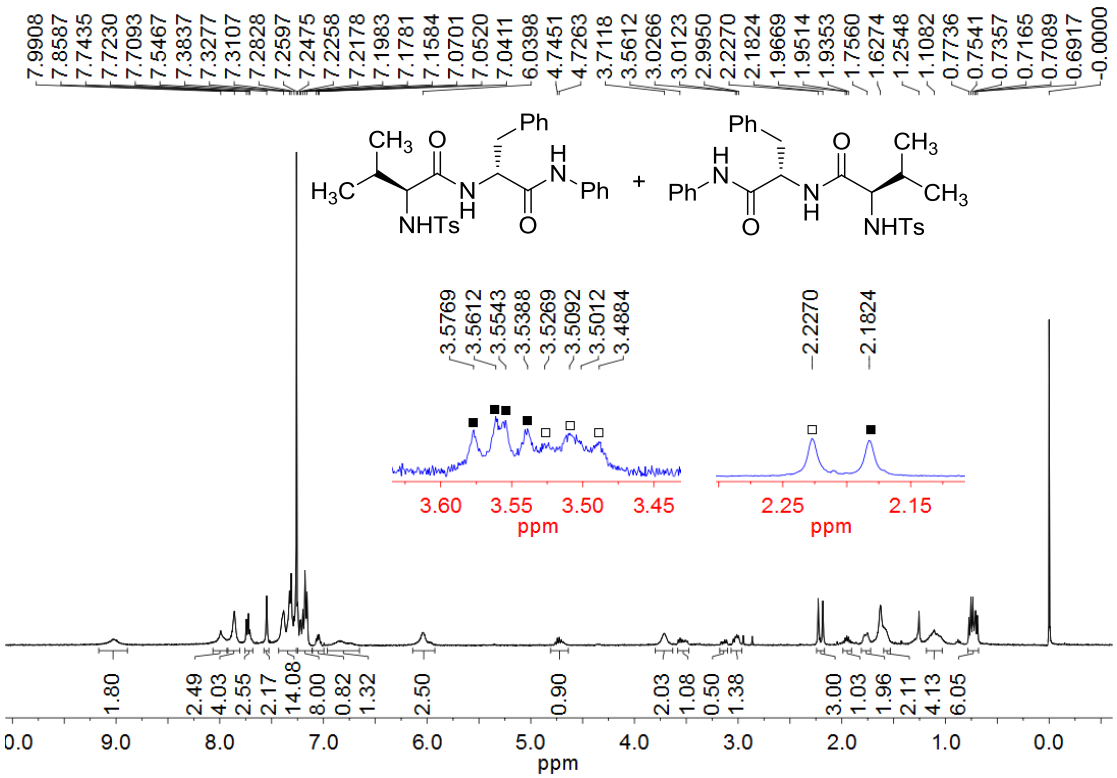


Figure S167. ^{1}H NMR spectrum of (±)-G**19** in the presence of CSA **7** in $CDCl_3$ (400 MHz), [(±)-G**19**] = 5 mM.

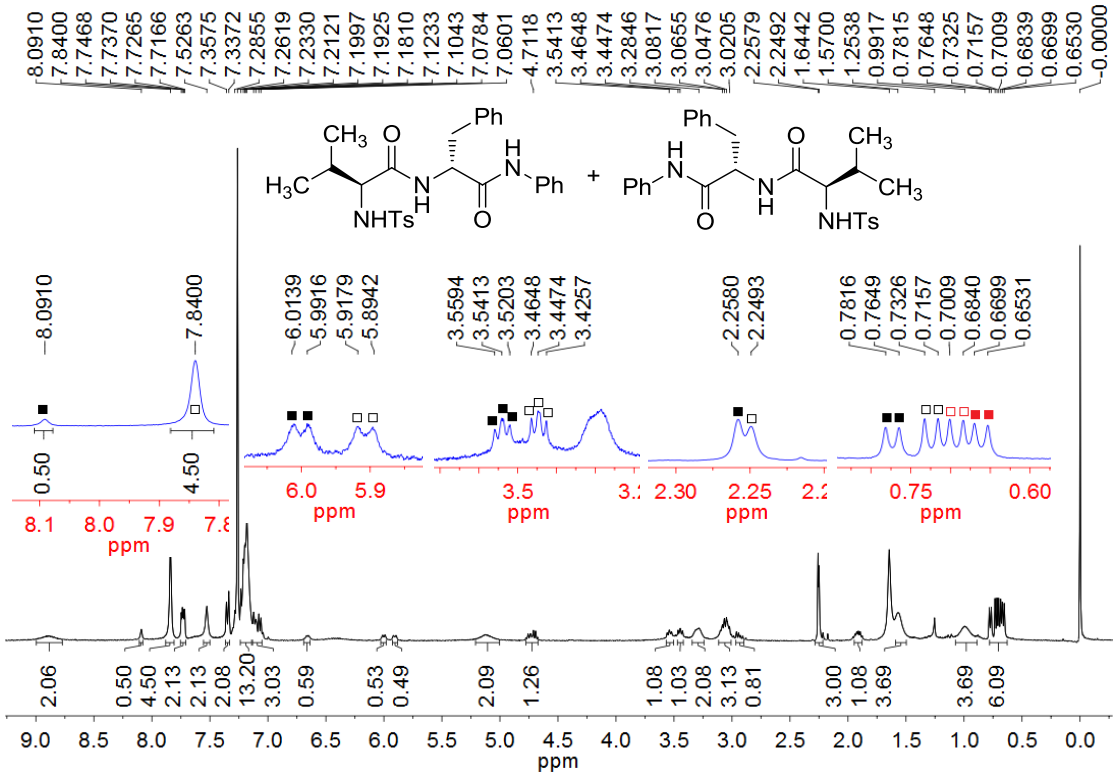


Figure S168. ^{1}H NMR spectrum of (±)-G**19** in the presence of CSA **8** in $CDCl_3$ (400 MHz), [(±)-G**13**9 = 5 mM.

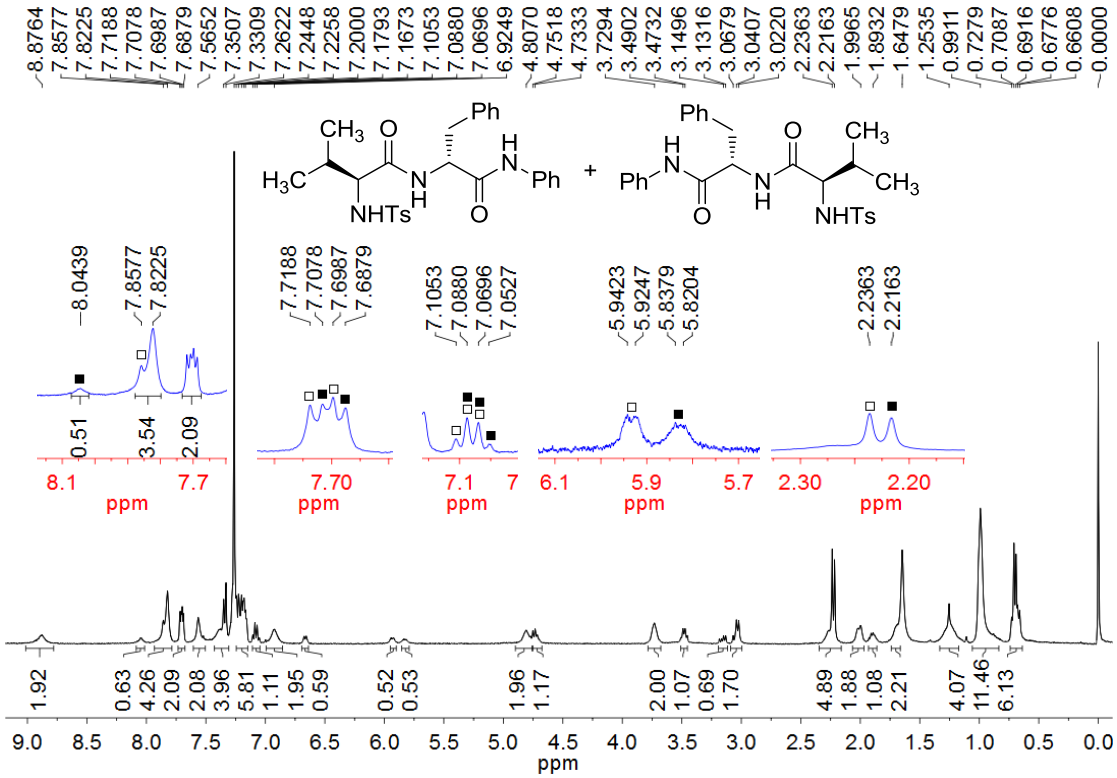


Figure S169. ^{1}H NMR spectrum of (±)-G**19** in the presence of CSA **9** in $CDCl_3$ (400 MHz), [(±)-G**19**] = 5 mM.

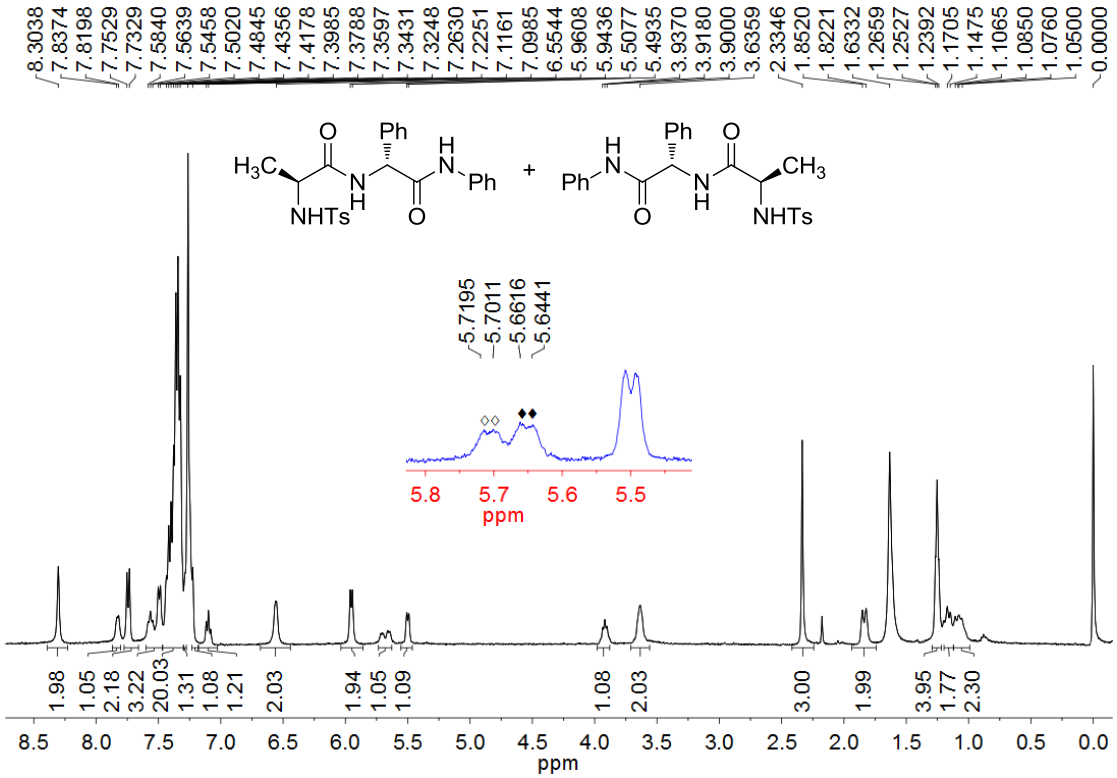


Figure S170. ^{1}H NMR spectrum of (±)-G**20** in the presence of CSA **1** in $CDCl_3$ (400 MHz), [(±)-G**20**] = 5 mM.

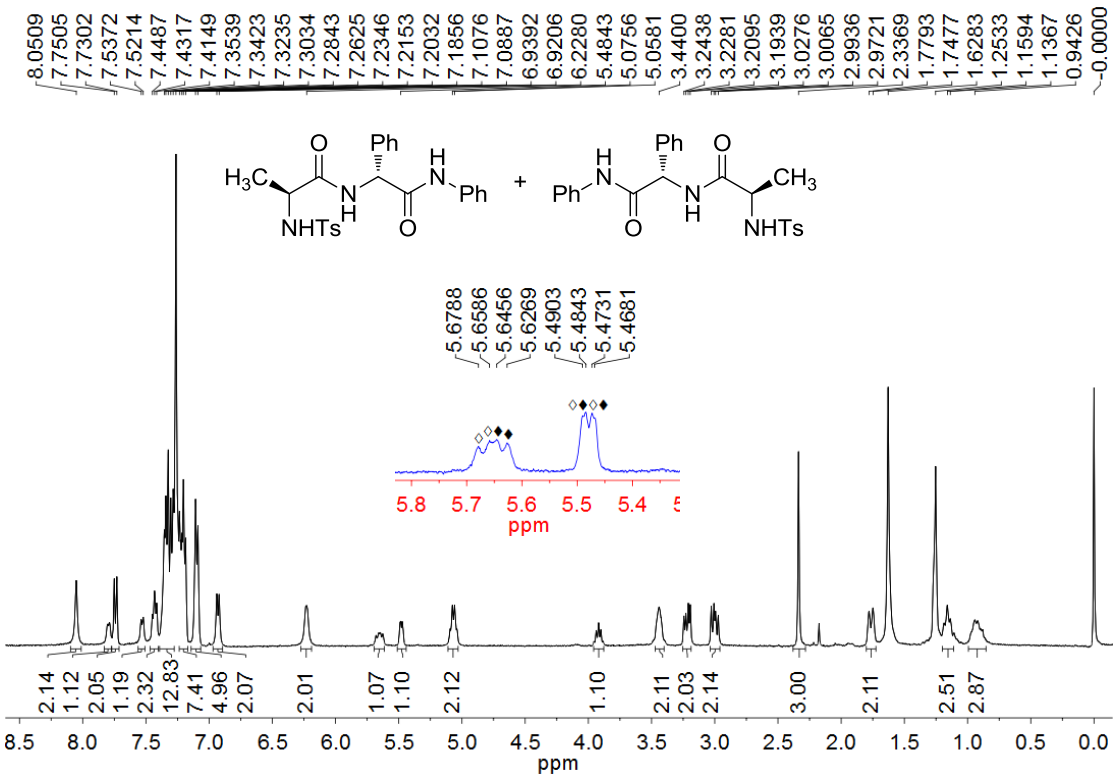


Figure S171. ^{1}H NMR spectrum of (±)-G**20** in the presence of CSA **2** in $CDCl_3$ (400 MHz), [(±)-G**20**] = 5 mM.

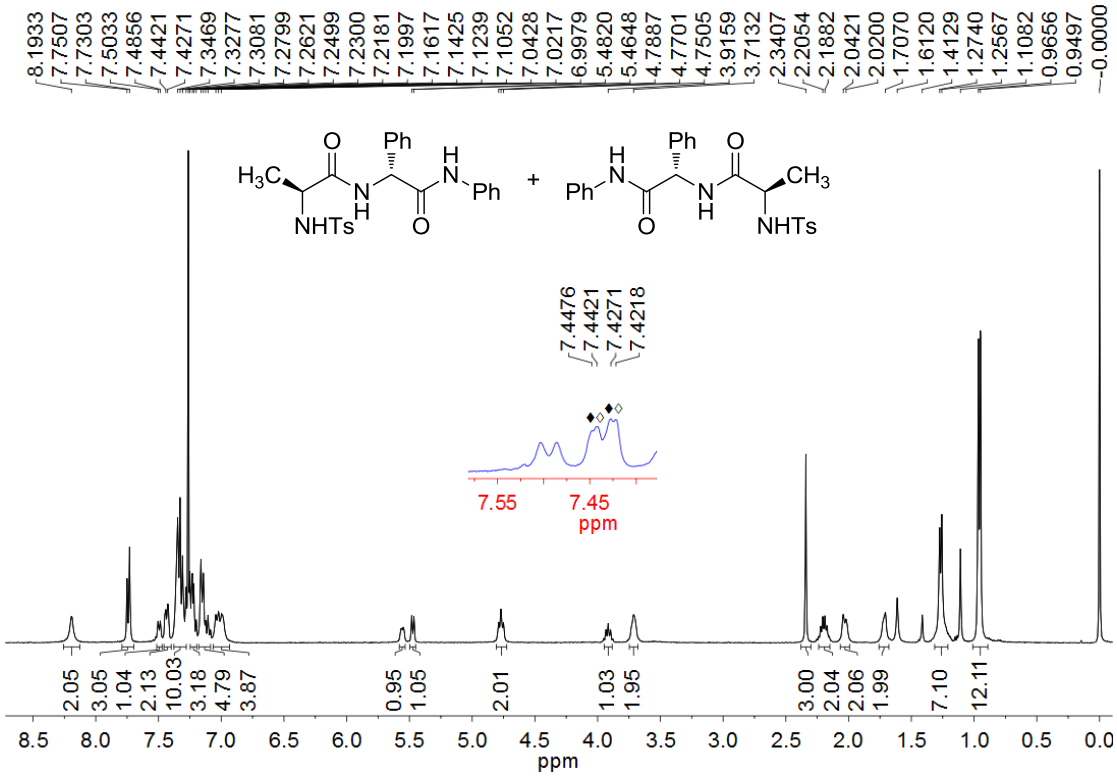


Figure S172. ^{1}H NMR spectrum of (±)-G**20** in the presence of CSA **3** in $CDCl_3$ (400 MHz), [(±)-G**20**] = 5 mM.

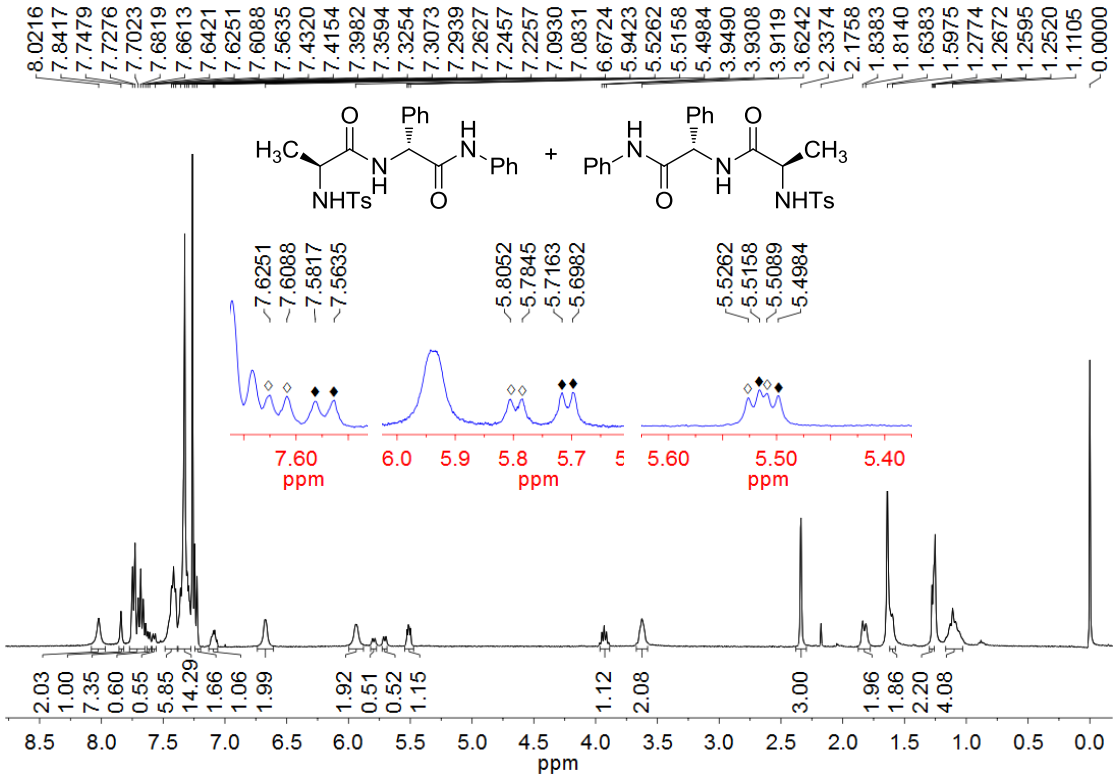


Figure S173. ^{1}H NMR spectrum of (±)-G**20** in the presence of CSA **4** in $CDCl_3$ (400 MHz), [(±)-G**20**] = 5 mM.

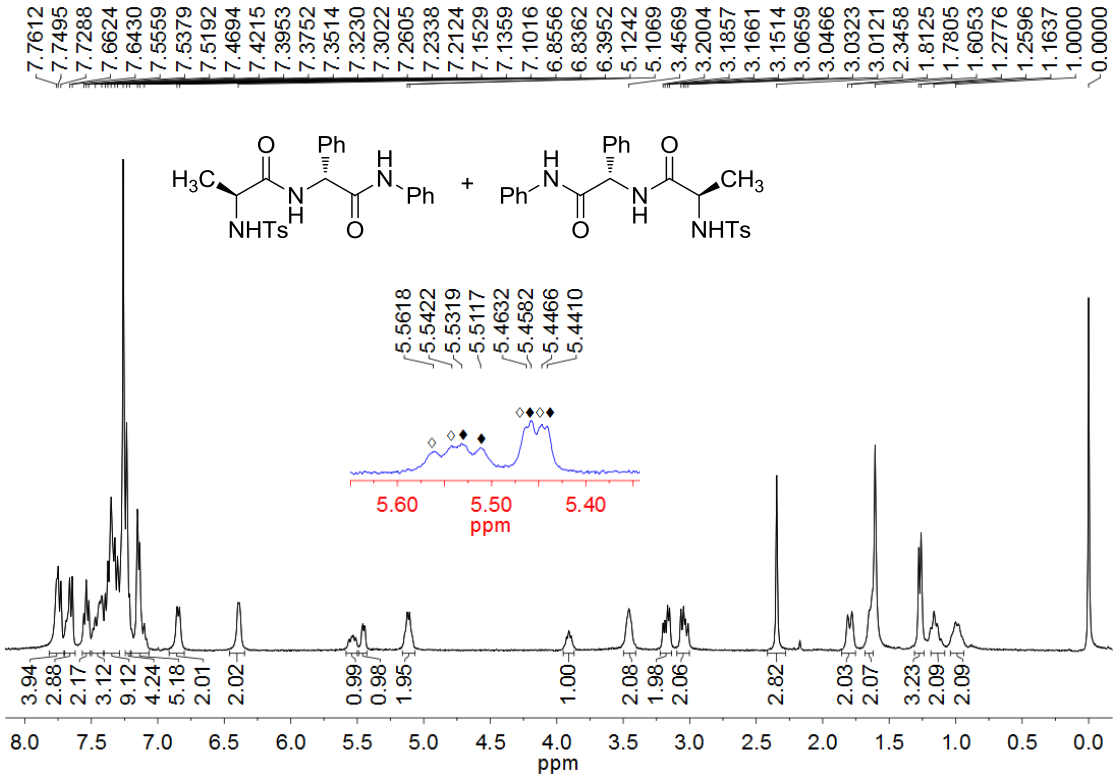


Figure S174. ^{1}H NMR spectrum of (±)-G**20** in the presence of CSA **5** in $CDCl_3$ (400 MHz), [(±)-G**20**] = 5 mM.

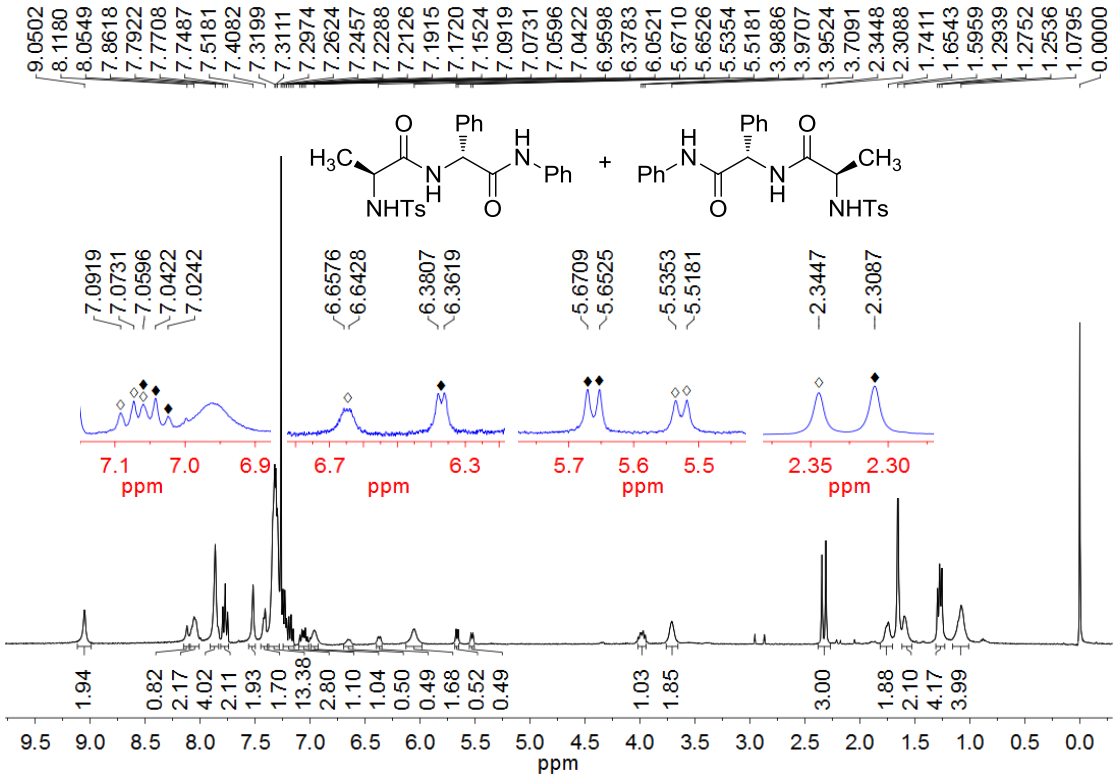


Figure S175. ^{1}H NMR spectrum of (±)-G**20** in the presence of CSA **7** in $CDCl_3$ (400 MHz), [(±)-G**20**] = 5 mM.

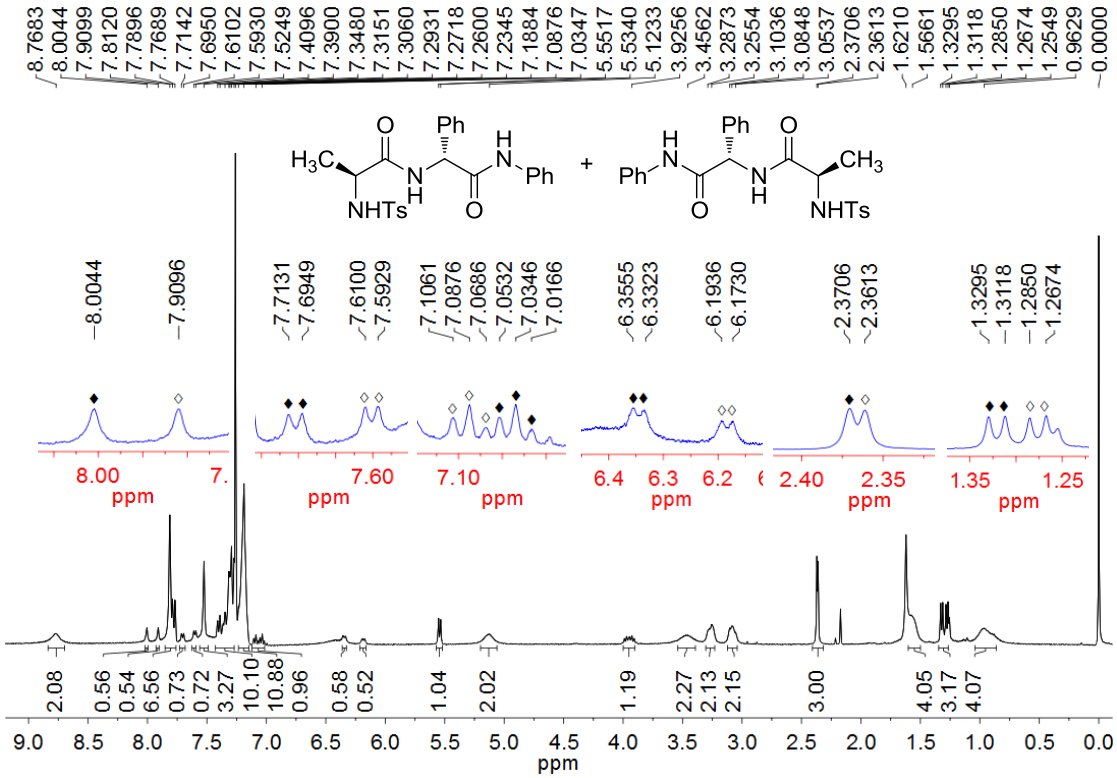


Figure S176. ^{1}H NMR spectrum of (±)-G**20** in the presence of CSA **8** in $CDCl_3$ (400 MHz), [(±)-G**20**] = 5 mM.

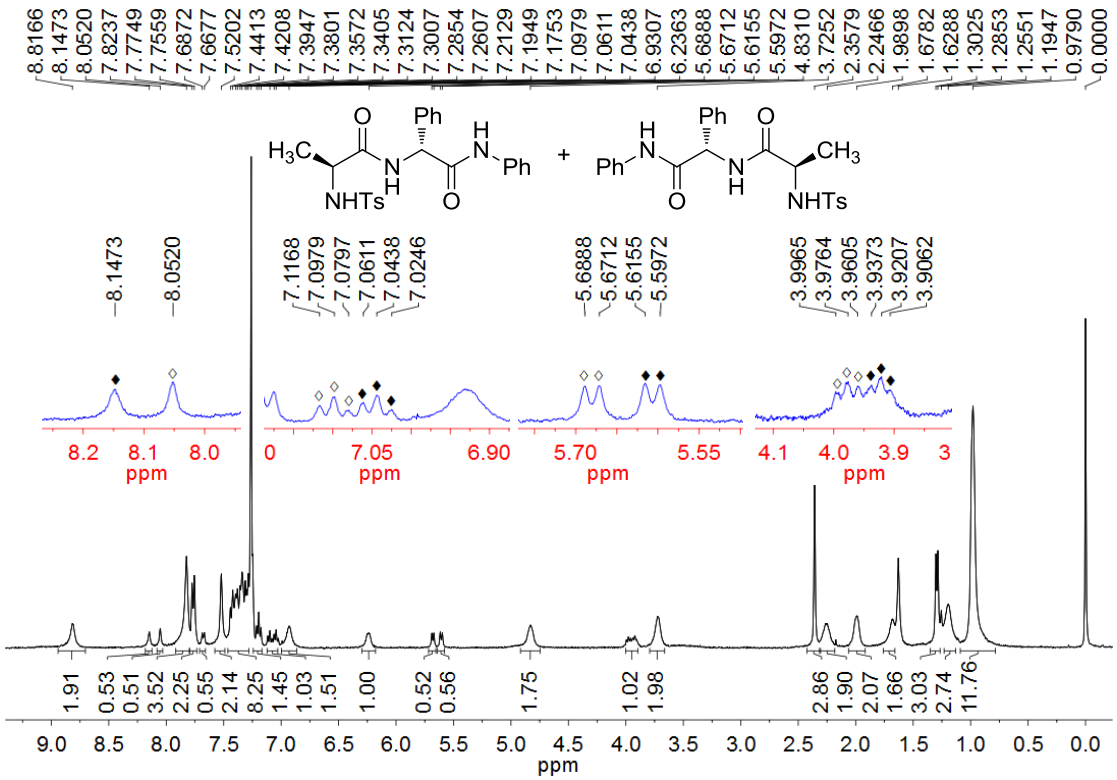


Figure S177. ^{1}H NMR spectrum of (±)-G**20** in the presence of CSA **9** in $CDCl_3$ (400 MHz), [(±)-G**20**] = 5 mM.

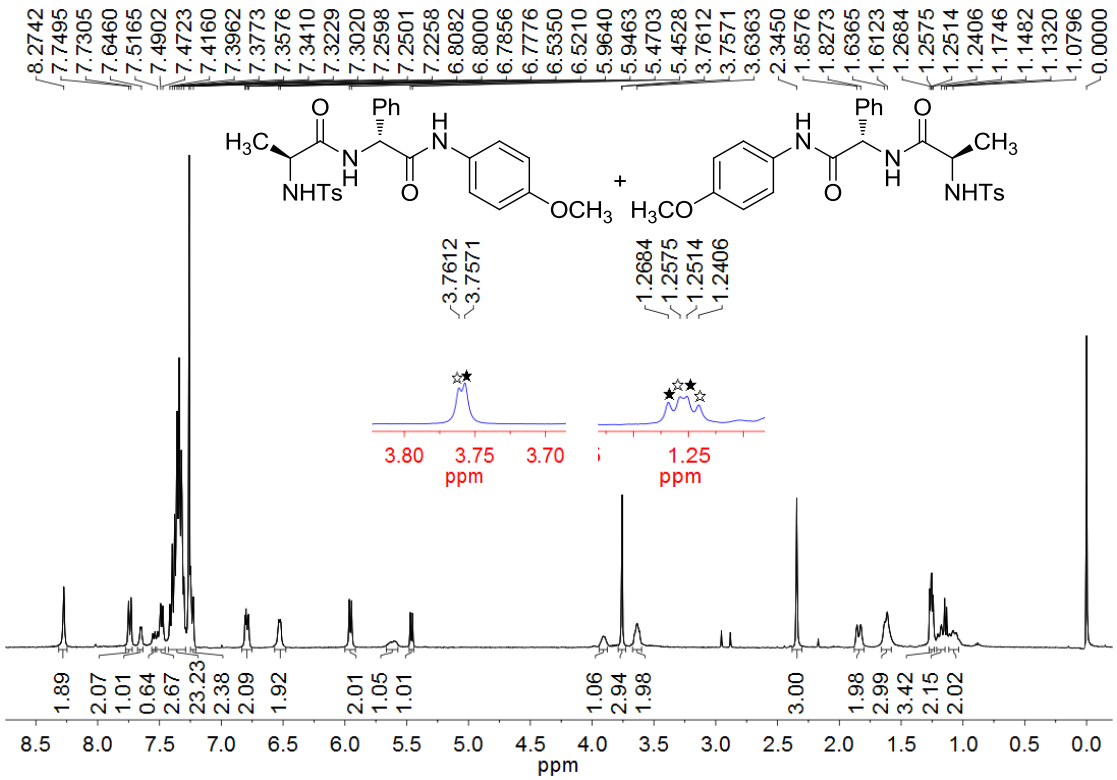


Figure S178. ^{1}H NMR spectrum of (±)-G**21** in the presence of CSA **1** in $CDCl_3$ (400 MHz), [(±)-G**21**] = 5 mM.

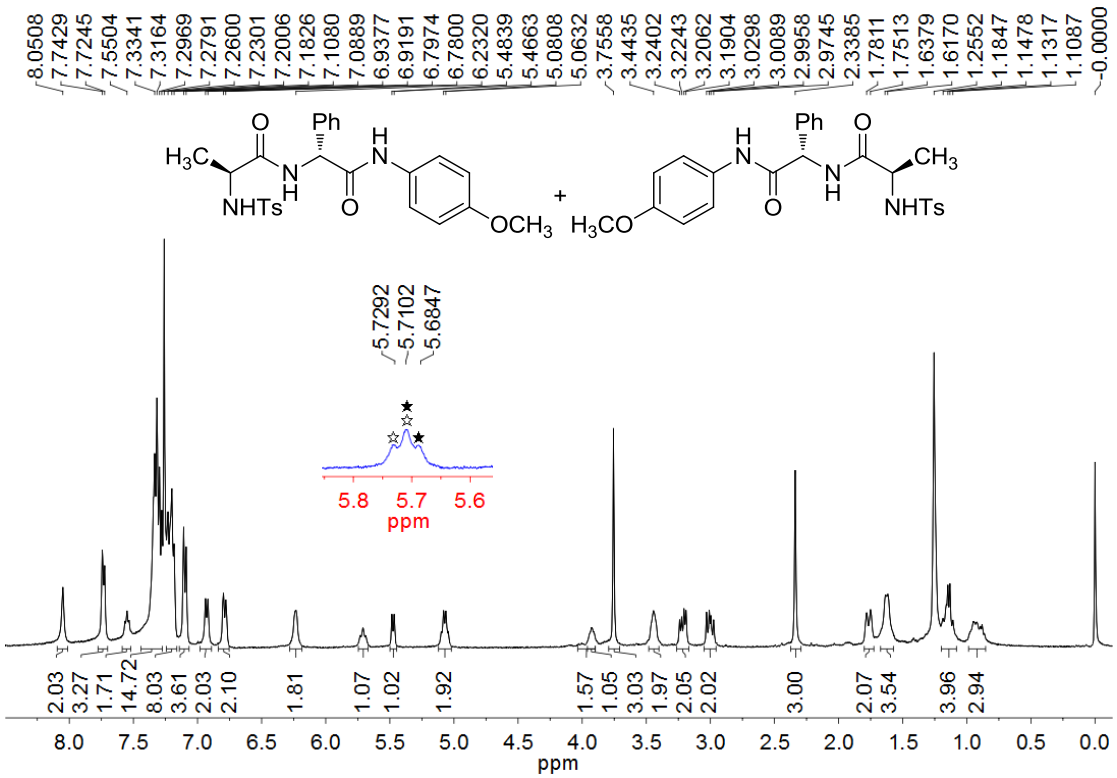


Figure S179. ^{1}H NMR spectrum of (±)-G**21** in the presence of CSA **2** in $CDCl_3$ (400 MHz), [(±)-G**21**] = 5 mM.

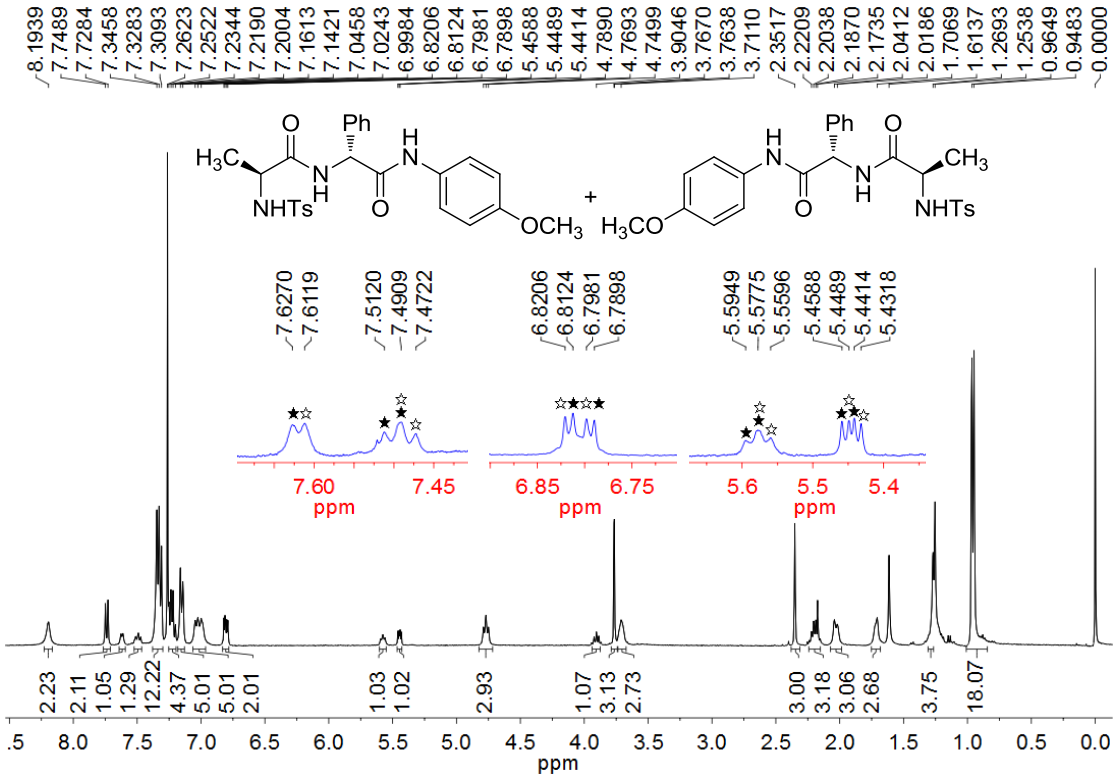


Figure S180. ^{1}H NMR spectrum of (±)-G**21** in the presence of CSA **3** in $CDCl_3$ (400 MHz), [(±)-G**21**] = 5 mM.

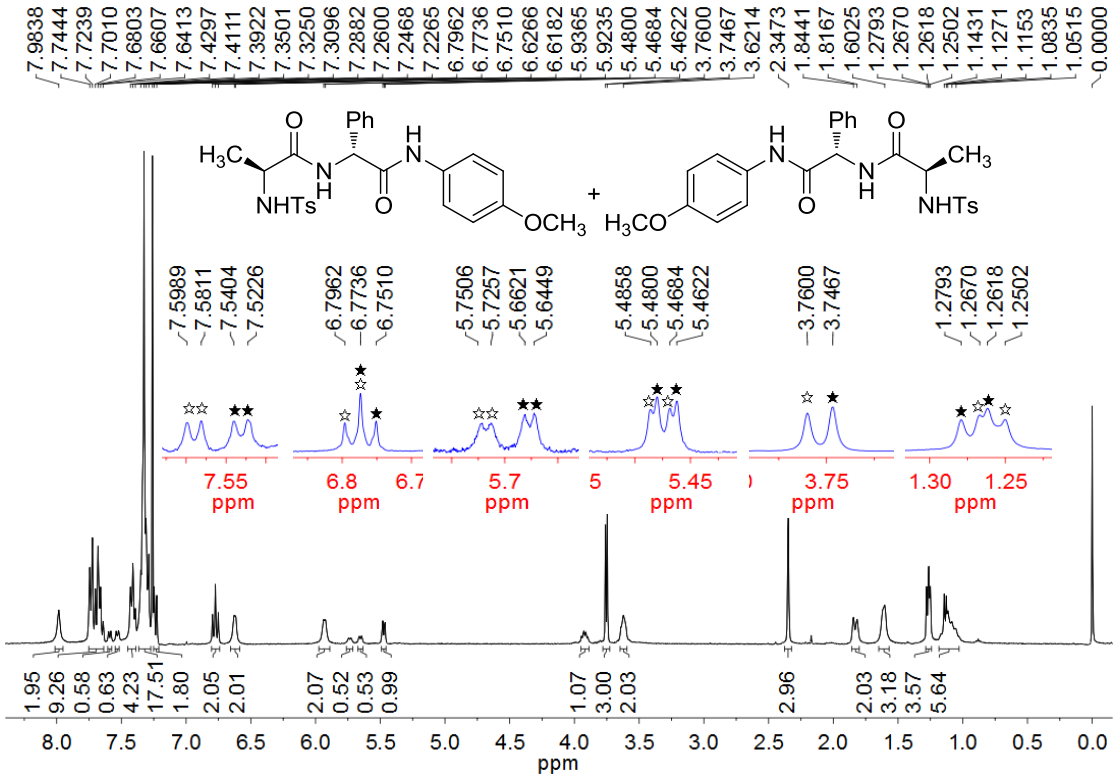


Figure S181. ^{1}H NMR spectrum of (±)-G**21** in the presence of CSA **4** in $CDCl_3$ (400 MHz), [(±)-G**21**] = 5 mM.

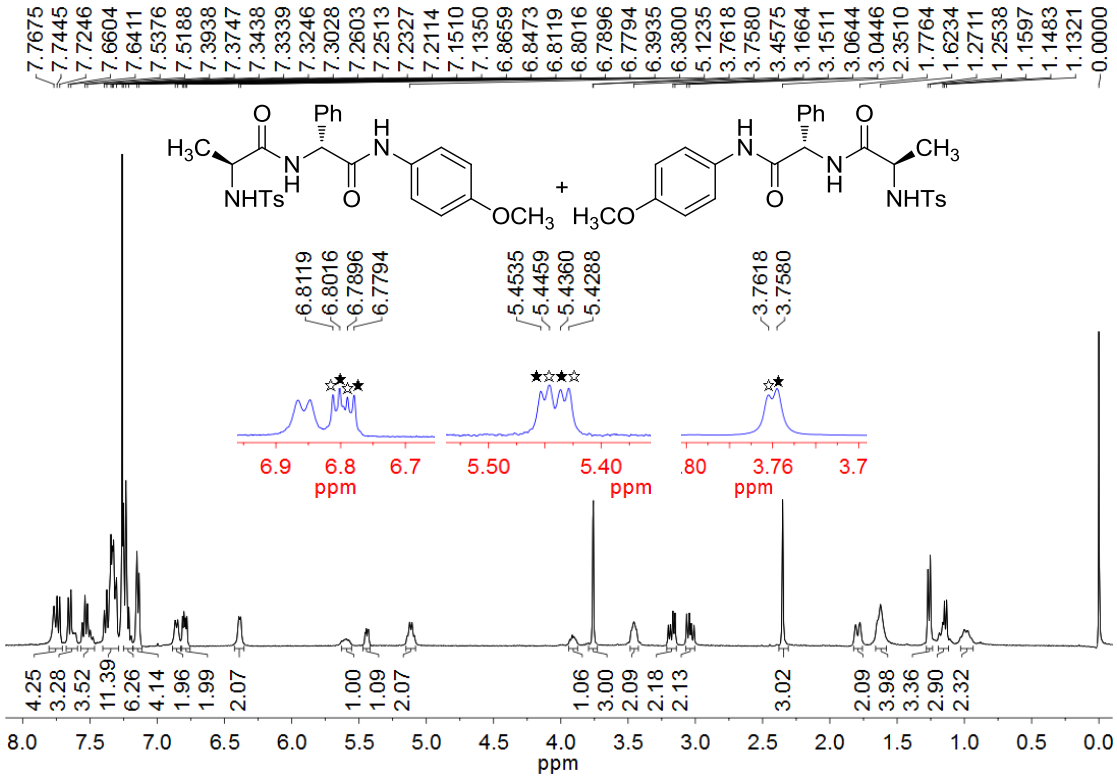


Figure S182. ^{1}H NMR spectrum of (±)-G**21** in the presence of CSA **5** in $CDCl_3$ (400 MHz), [(±)-G**21**] = 5 mM.

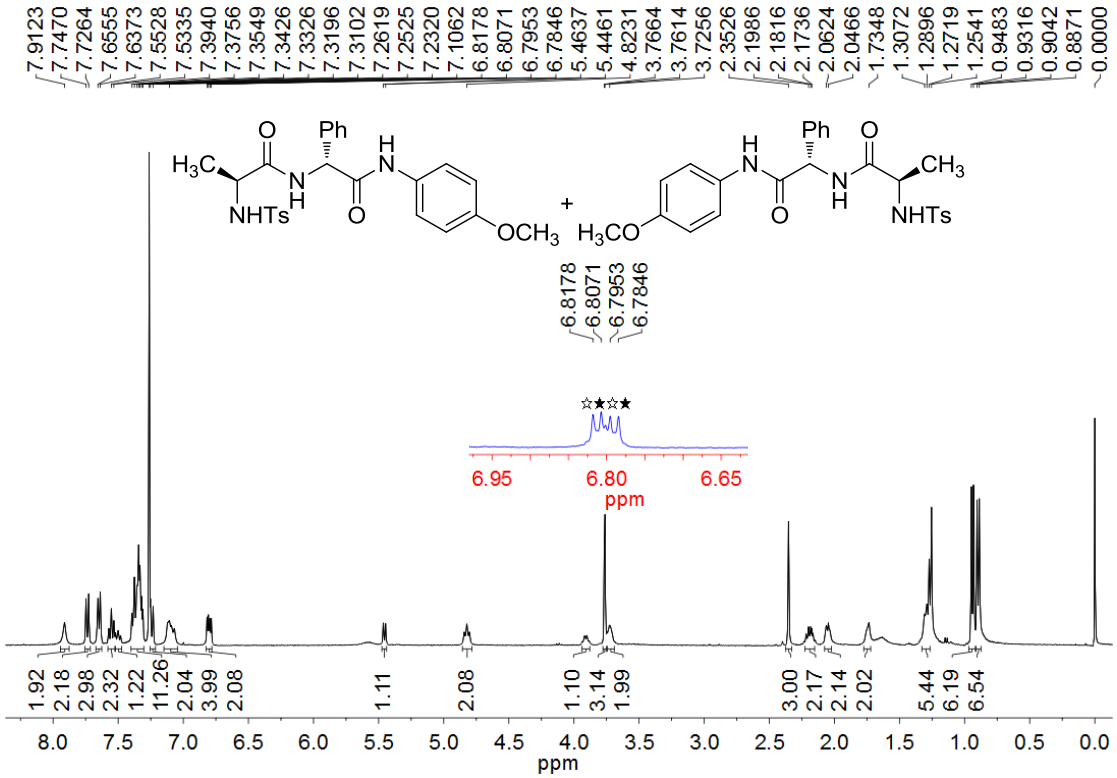


Figure S183. ^{1}H NMR spectrum of (±)-G**21** in the presence of CSA **6** in $CDCl_3$ (400 MHz), [(±)-G**21**] = 5 mM.

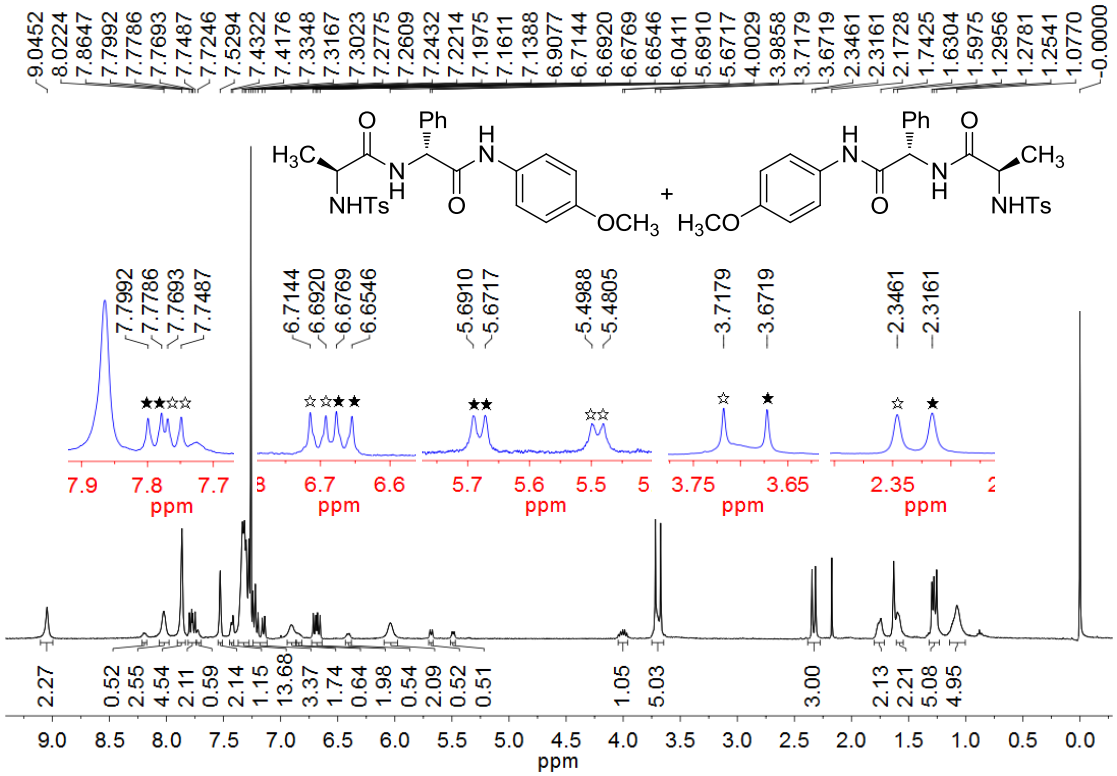


Figure S184. ^{1}H NMR spectrum of (±)-G**21** in the presence of CSA **7** in $CDCl_3$ (400 MHz), [(±)-G**21**] = 5 mM.

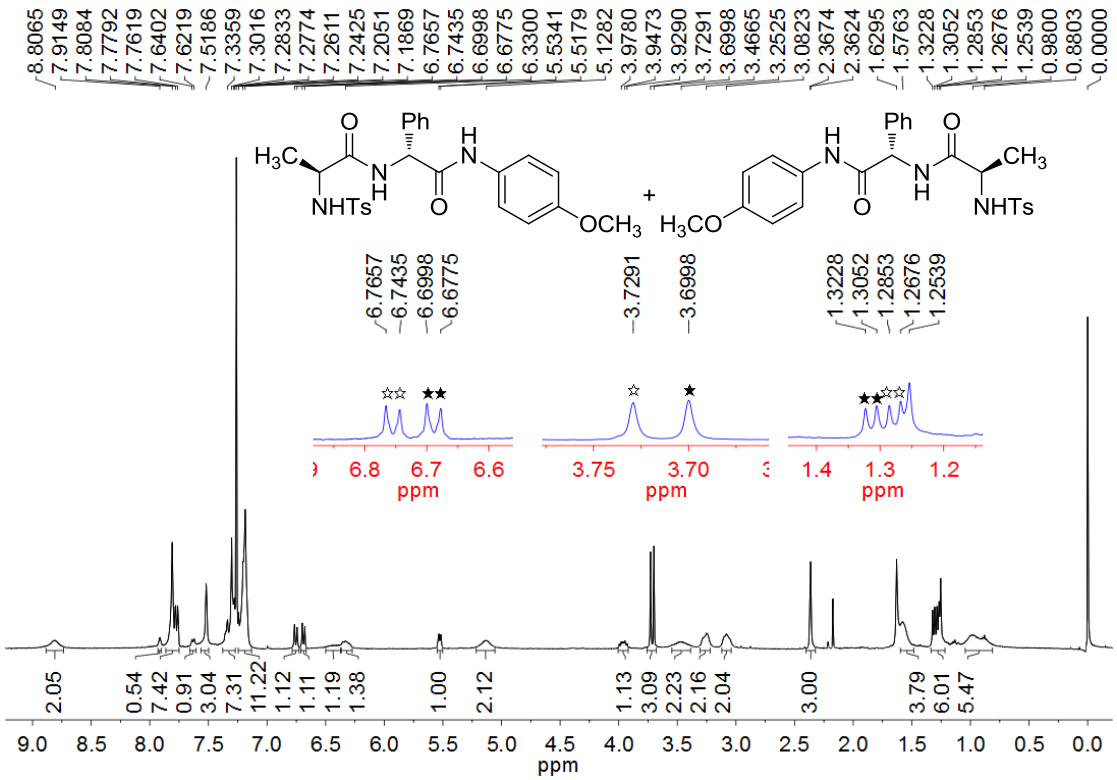


Figure S185. ^{1}H NMR spectrum of (±)-G**21** in the presence of CSA **8** in $CDCl_3$ (400 MHz), [(±)-G**21**] = 5 mM.

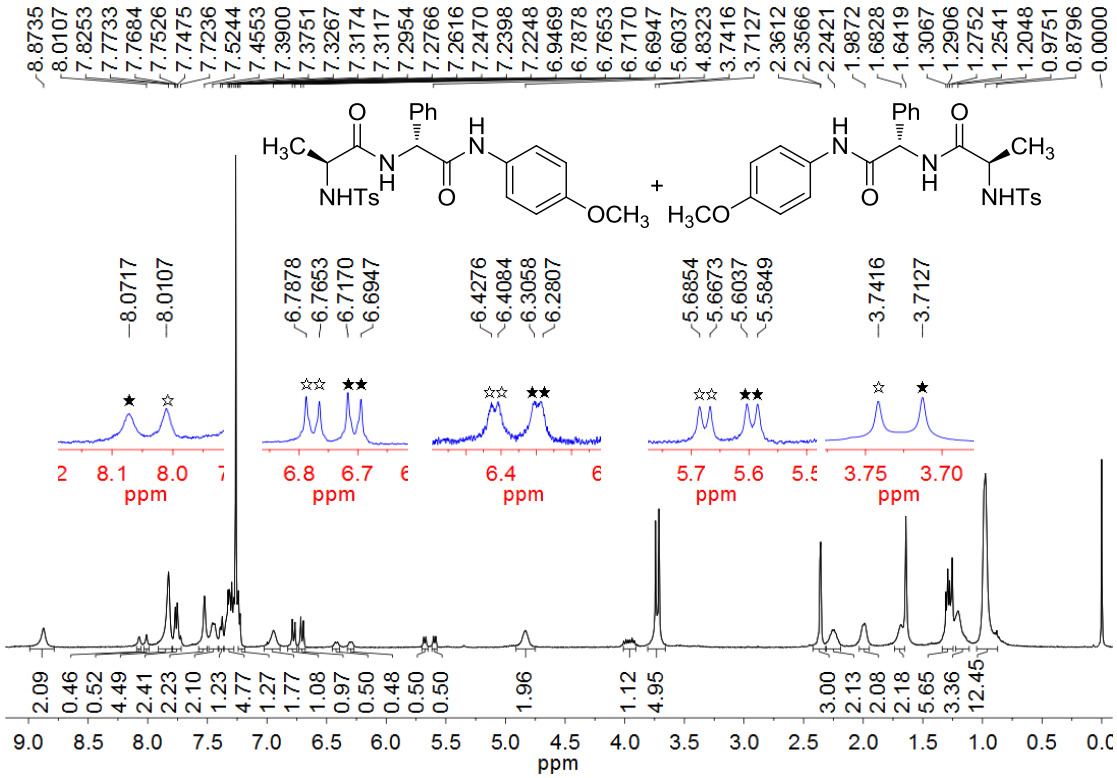


Figure S186. ^{1}H NMR spectrum of (±)-G**21** in the presence of CSA **9** in $CDCl_3$ (400 MHz), [(±)-G**21**] = 5 mM.

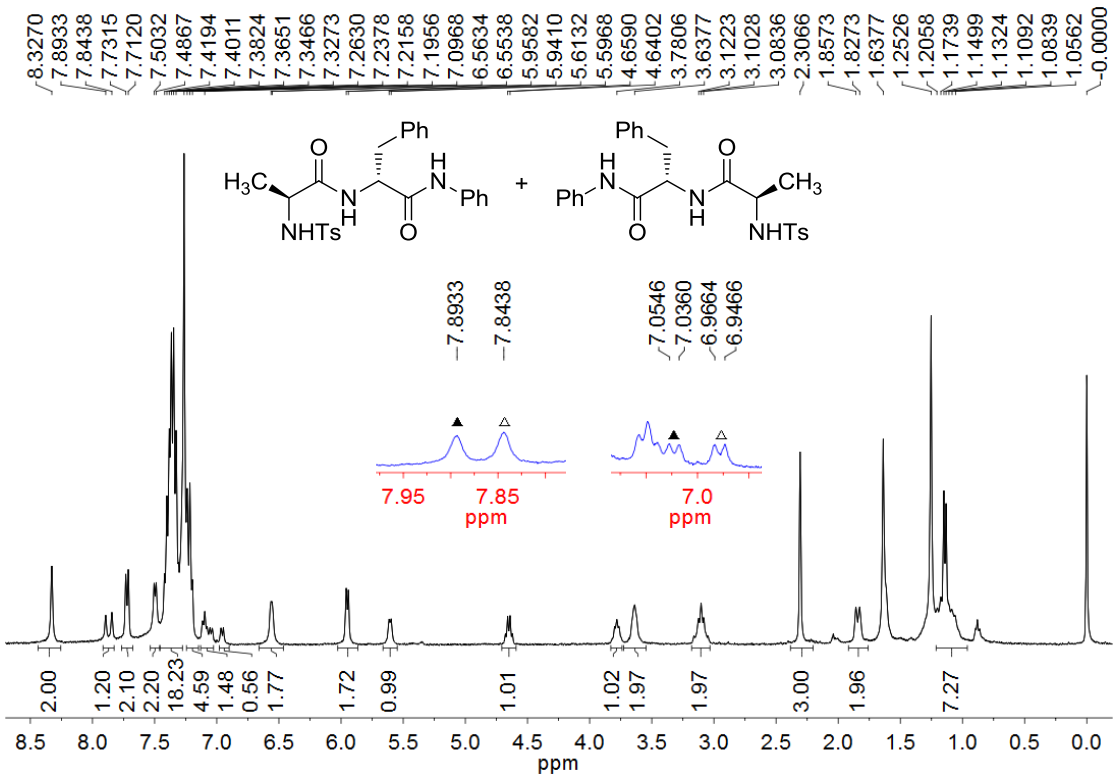


Figure S187. ^{1}H NMR spectrum of (±)-G**22** in the presence of CSA **1** in $CDCl_3$ (400 MHz), [(±)-G**22**] = 5 mM.

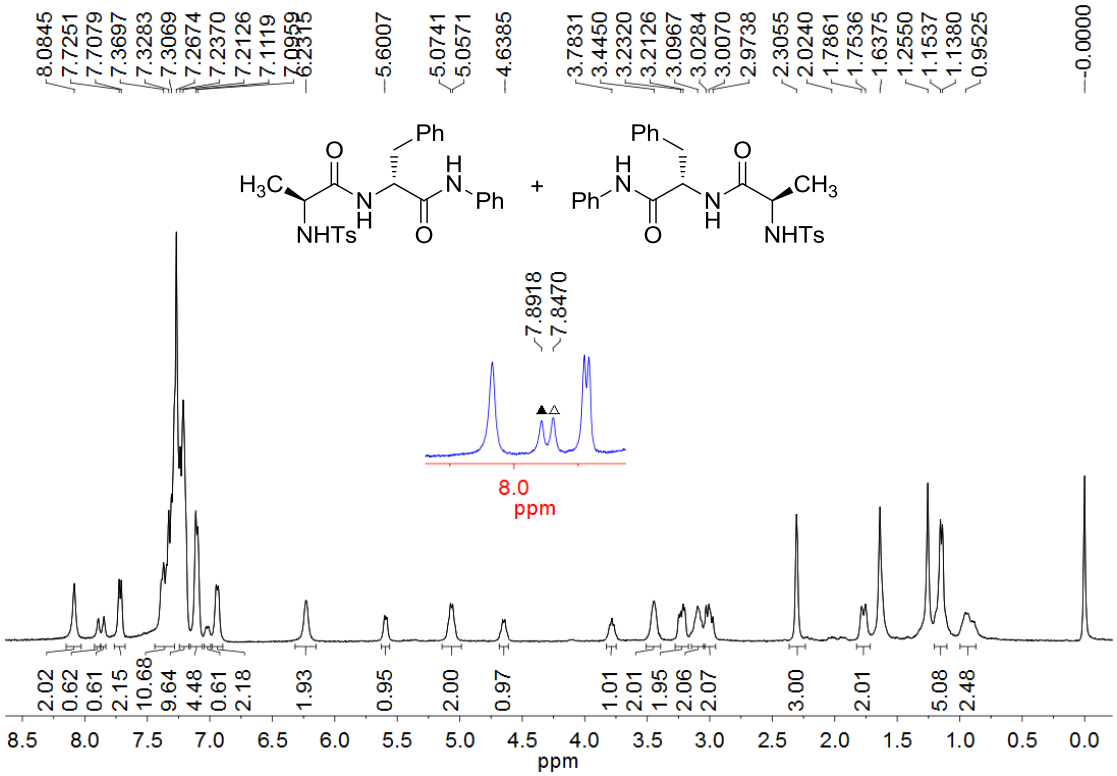


Figure S188. ^{1}H NMR spectrum of (±)-G**22** in the presence of CSA **2** in $CDCl_3$ (400 MHz), [(±)-G**22**] = 5 mM.

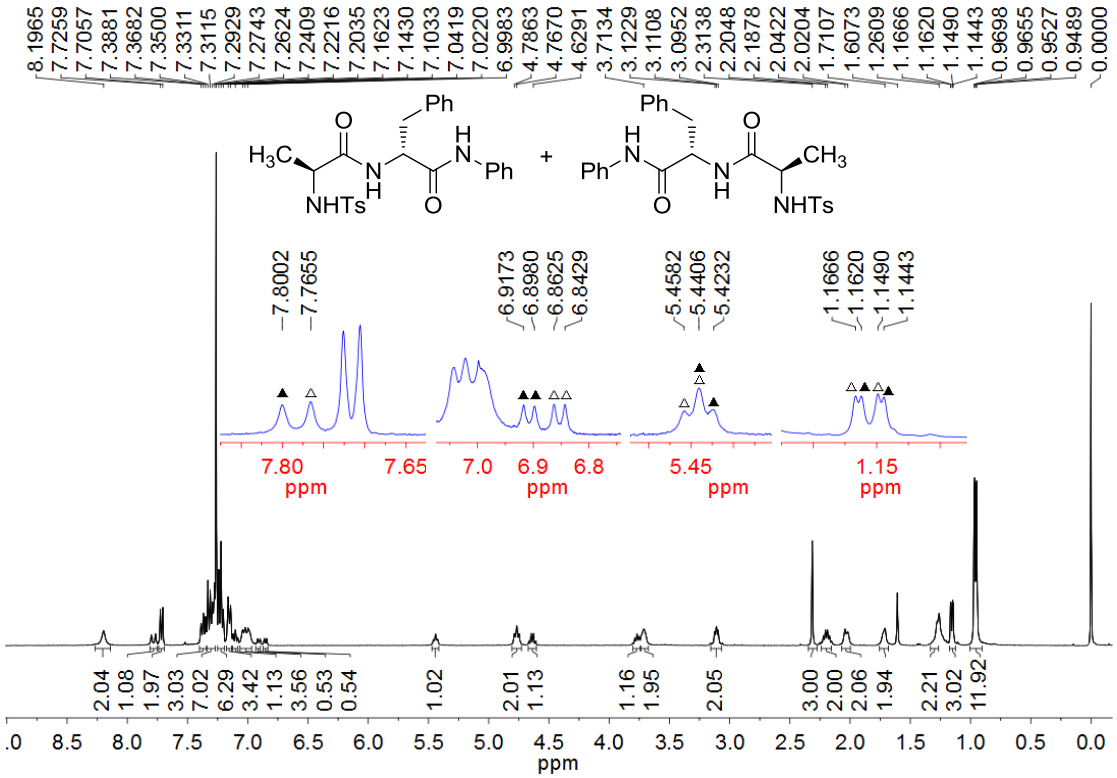


Figure S189. ^{1}H NMR spectrum of (±)-G**22** in the presence of CSA **3** in $CDCl_3$ (400 MHz), [(±)-G**22**] = 5 mM.

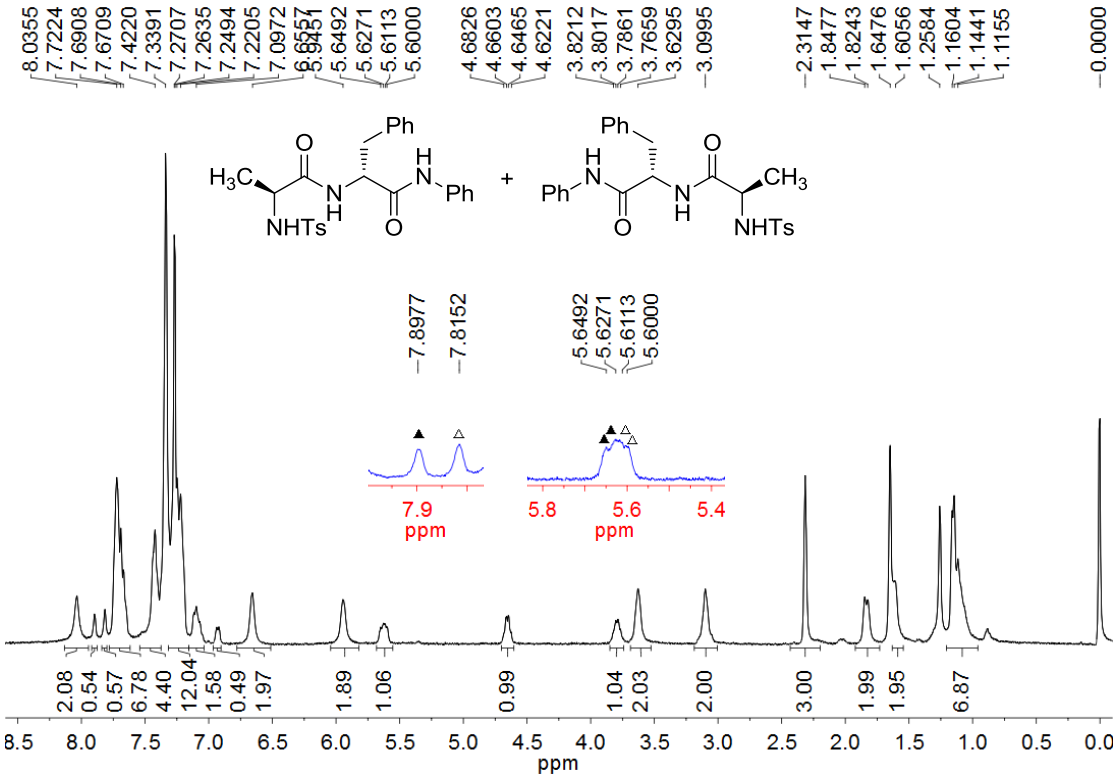


Figure S190. ^{1}H NMR spectrum of (±)-G**22** in the presence of CSA **4** in $CDCl_3$ (400 MHz), [(±)-G**22**] = 5 mM.

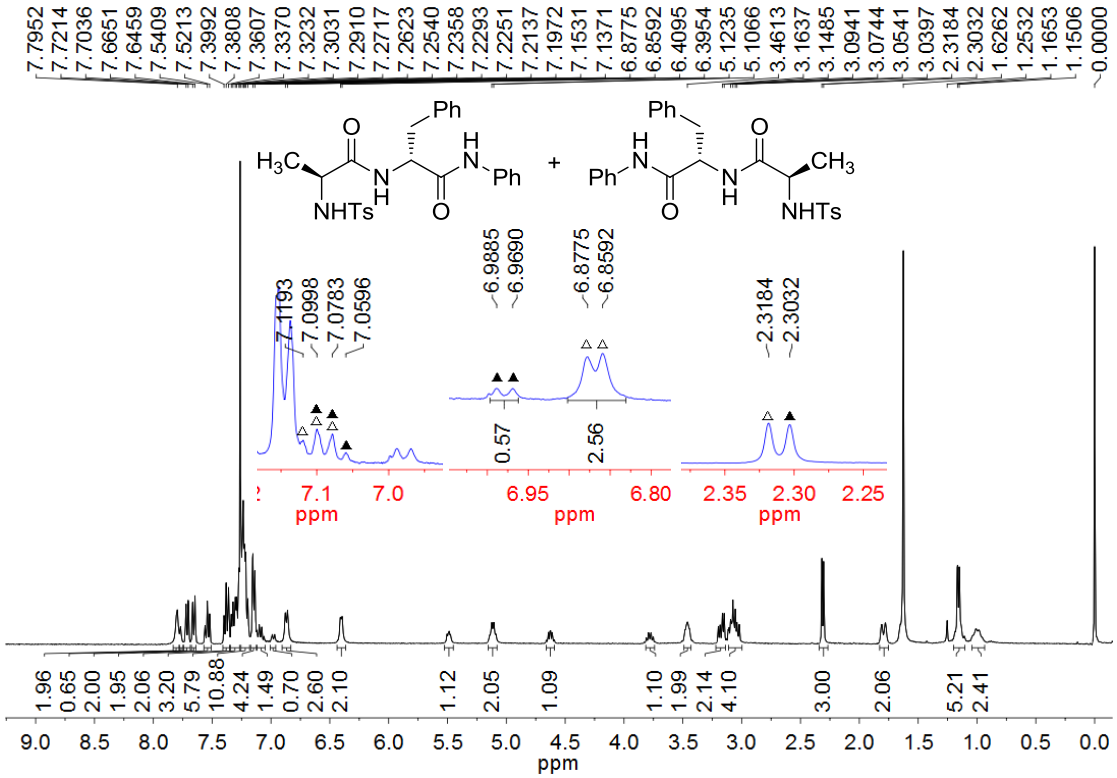


Figure S191. ^{1}H NMR spectrum of (±)-G**22** in the presence of CSA **5** in $CDCl_3$ (400 MHz), [(±)-G**22**] = 5 mM.

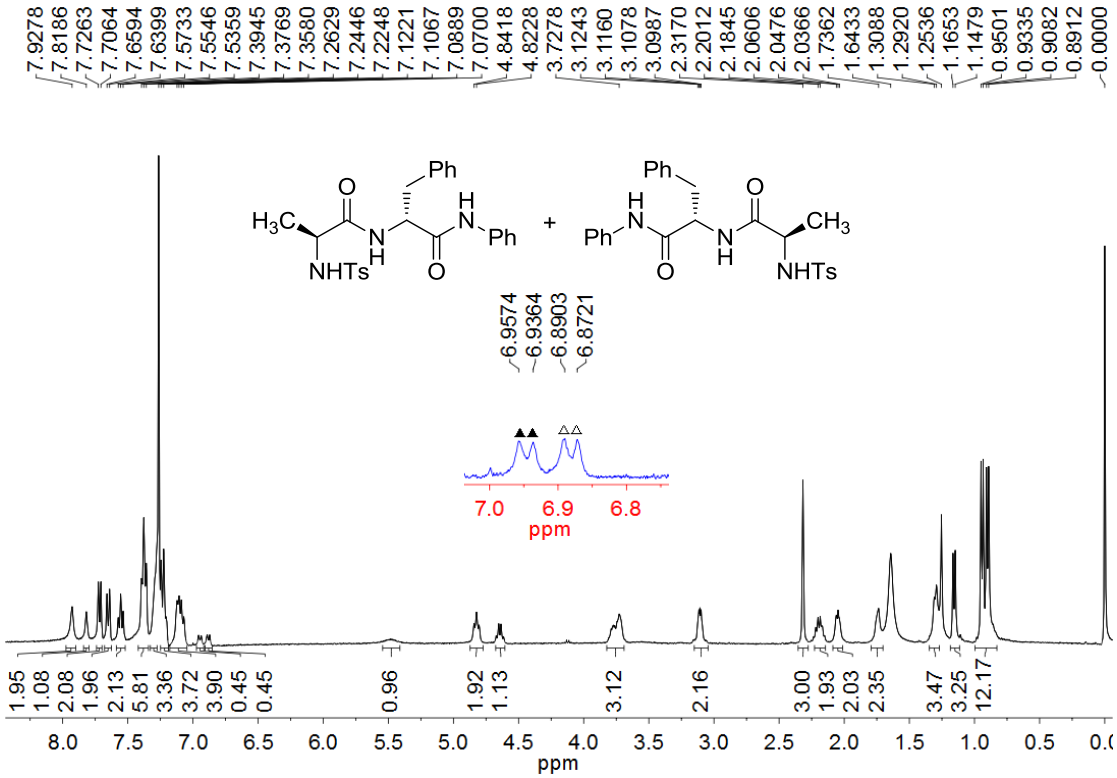


Figure S192. ^{1}H NMR spectrum of (±)-G**22** in the presence of CSA **6** in $CDCl_3$ (400 MHz), [(±)-G**22** = 5 mM.

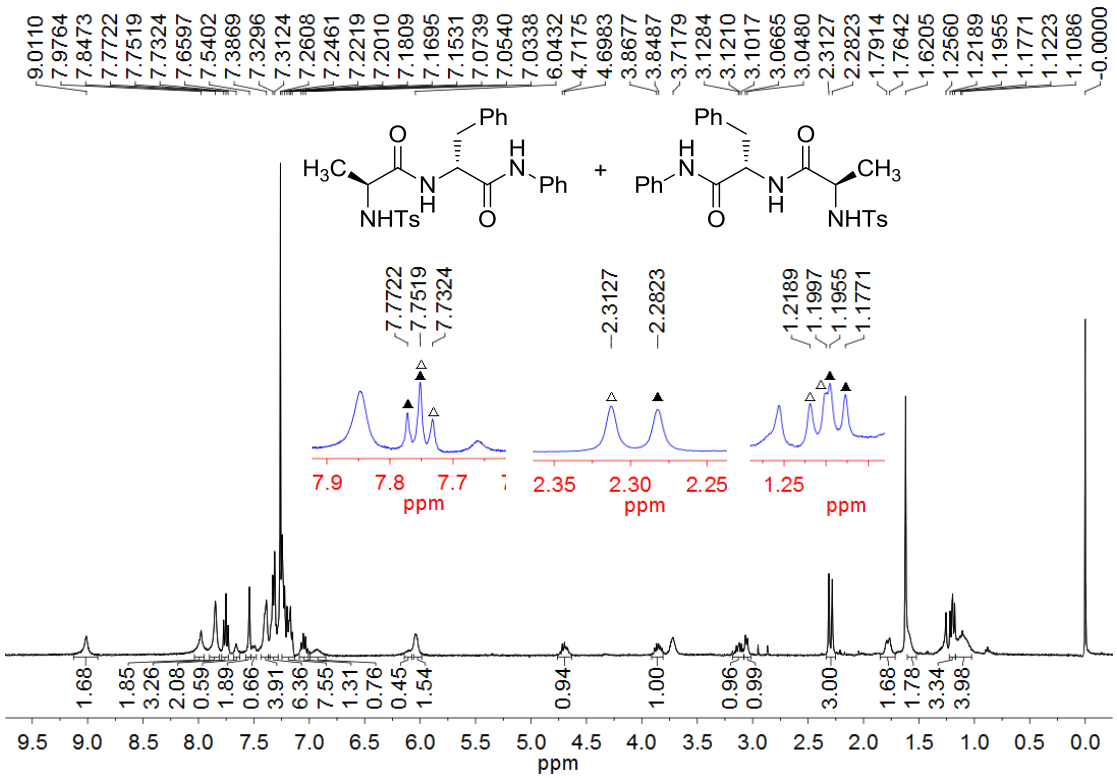


Figure S193. ^{1}H NMR spectrum of (±)-G**22** in the presence of CSA **7** in $CDCl_3$ (400 MHz), [(±)-G**22**] = 5 mM.

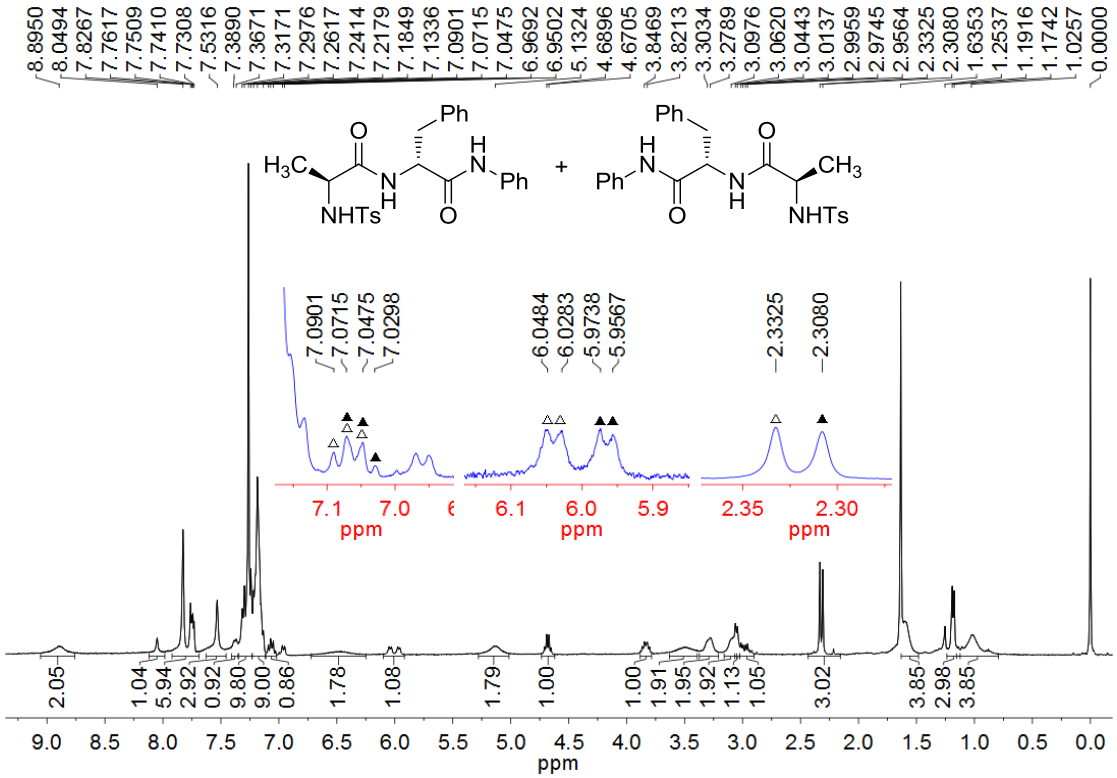


Figure S194. ^{1}H NMR spectrum of (±)-G**22** in the presence of CSA **8** in $CDCl_3$ (400 MHz), [(±)-G**22**] = 5 mM.

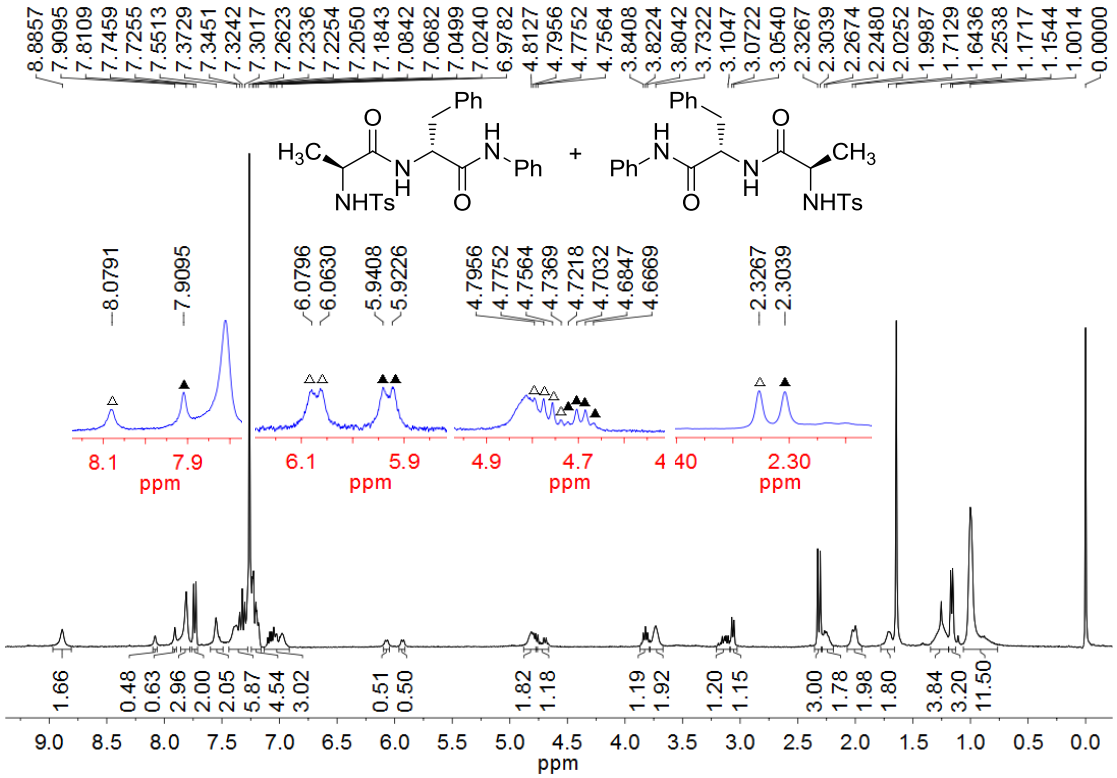


Figure S195. ^{1}H NMR spectrum of (±)-G**22** in the presence of CSA **9** in $CDCl_3$ (400 MHz), [(±)-G**22**] = 5 mM.

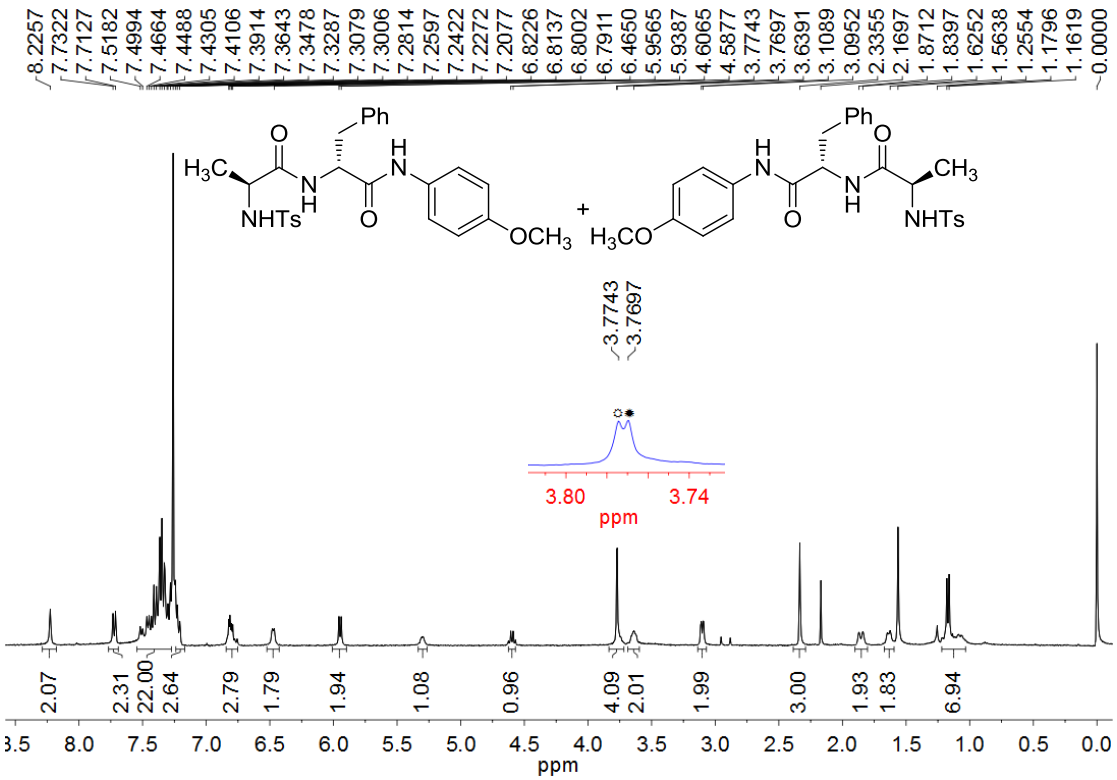


Figure S196. ^{1}H NMR spectrum of (±)-G**23** in the presence of CSA **1** in $CDCl_3$ (400 MHz), [(±)-G**23**] = 4 mM.

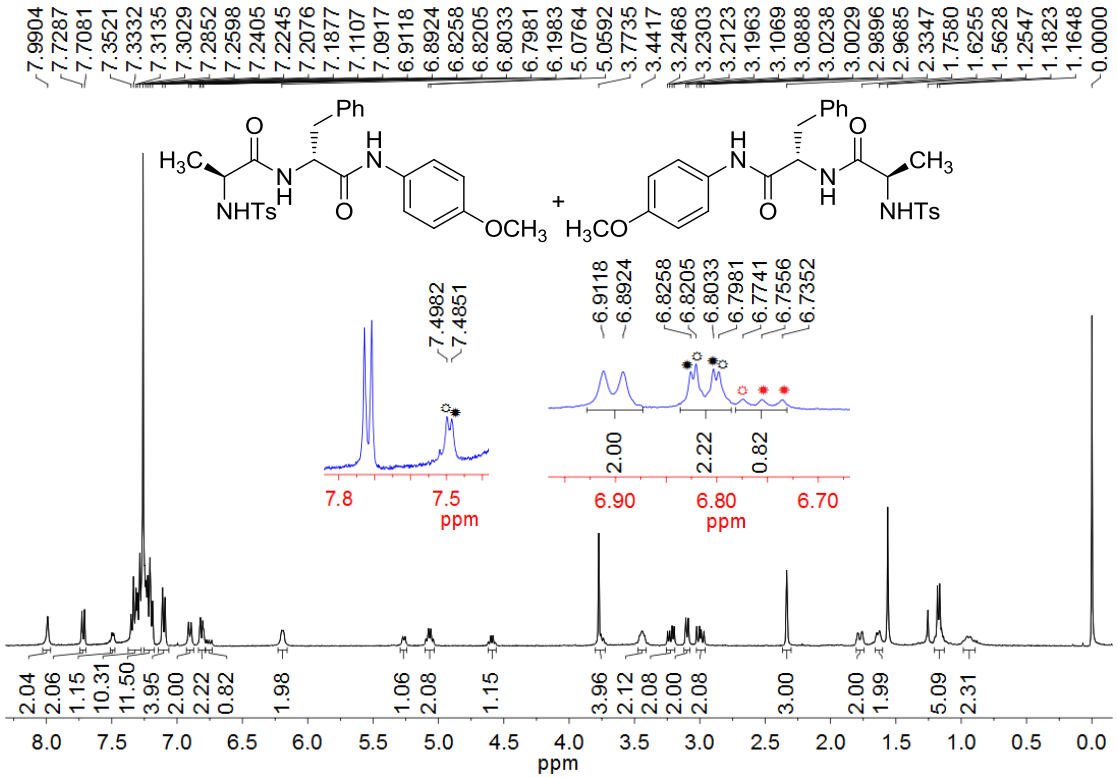


Figure S197. ^{1}H NMR spectrum of (±)-G**23** in the presence of CSA **2** in $CDCl_3$ (400 MHz), [(±)-G**23**] = 4 mM.

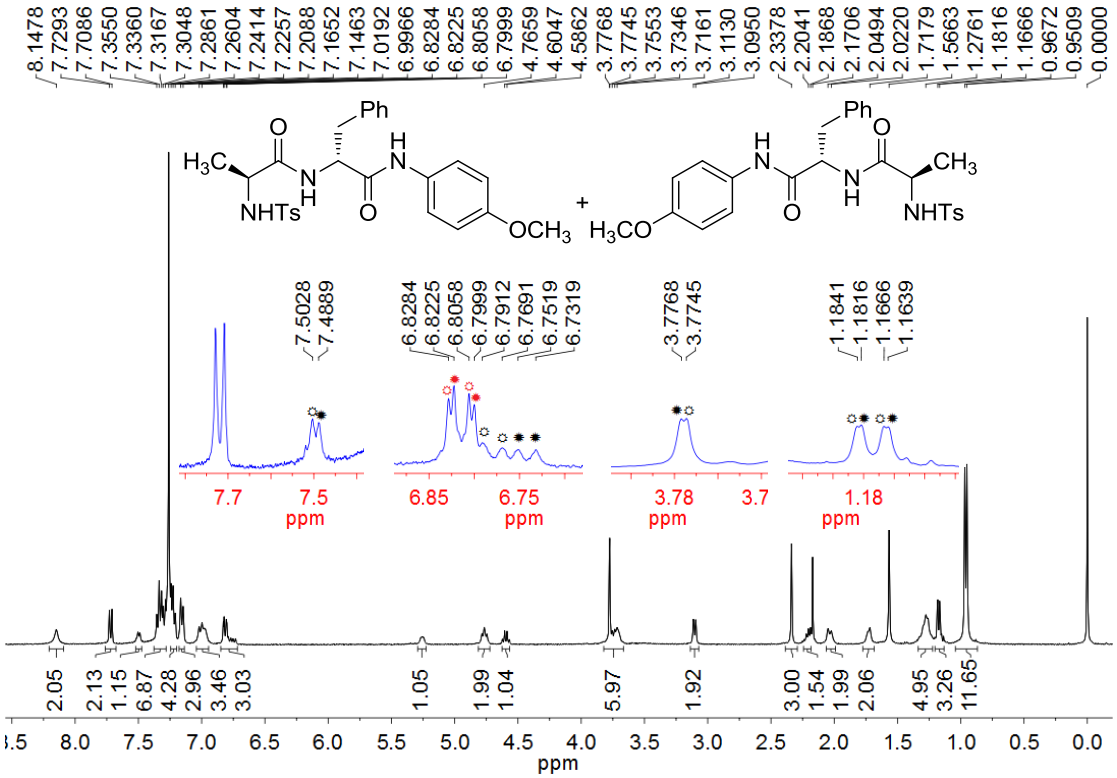


Figure S198. ^{1}H NMR spectrum of (±)-G**23** in the presence of CSA **3** in $CDCl_3$ (400 MHz), [(±)-G**23**] = 4 mM.

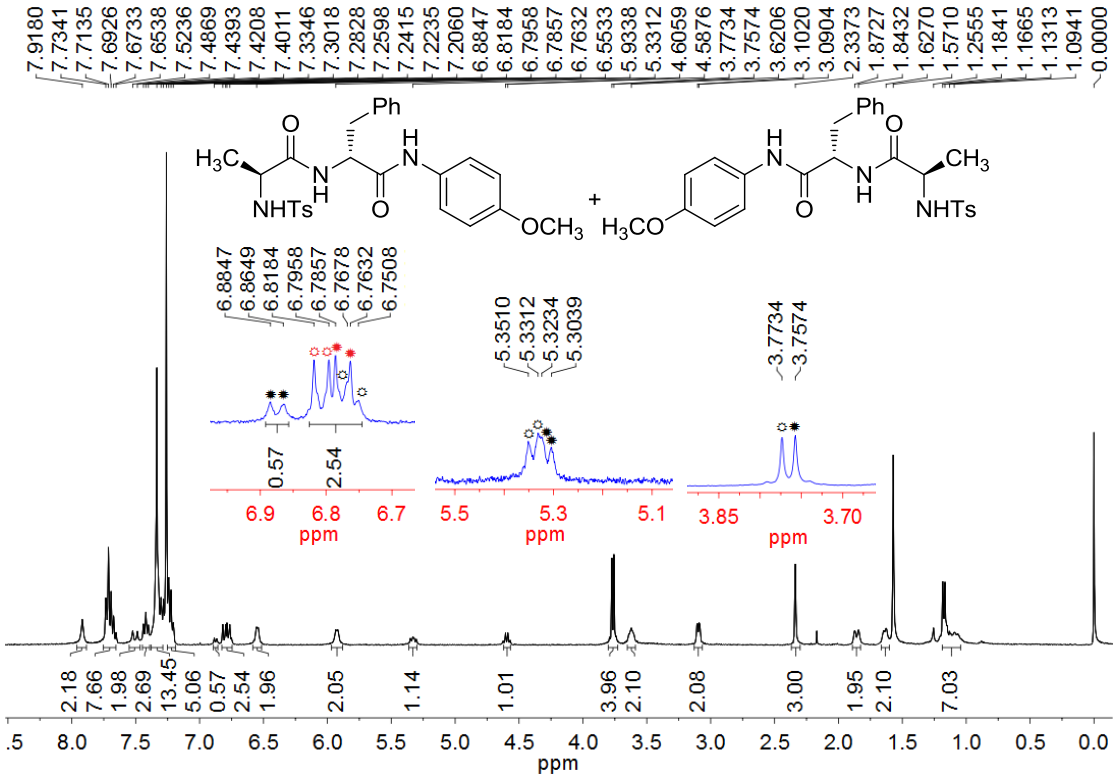


Figure S199. ^{1}H NMR spectrum of (±)-G**23** in the presence of CSA **4** in $CDCl_3$ (400 MHz), [(±)-G**23**] = 4 mM.

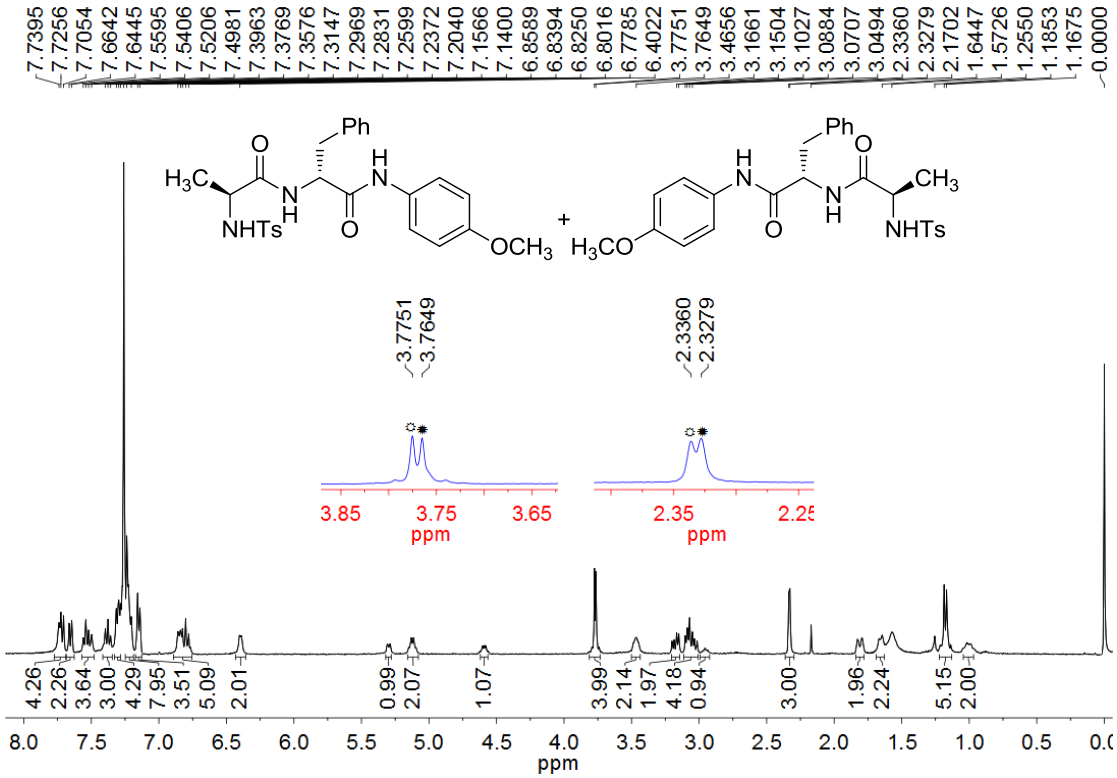


Figure S200. ^{1}H NMR spectrum of (±)-G**23** in the presence of CSA **5** in $CDCl_3$ (400 MHz), [(±)-G**23**] = 4 mM.

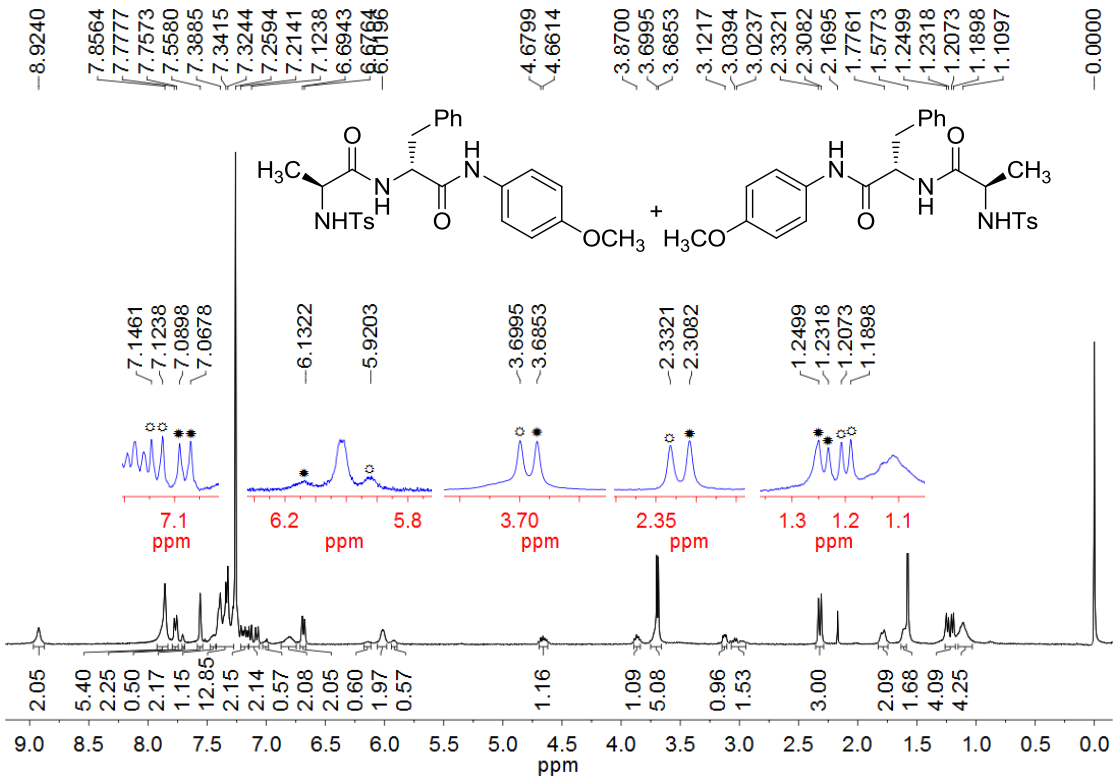


Figure S201. ^{1}H NMR spectrum of (±)-G**23** in the presence of CSA **7** in $CDCl_3$ (400 MHz), [(±)-G**23**] = 4 mM.

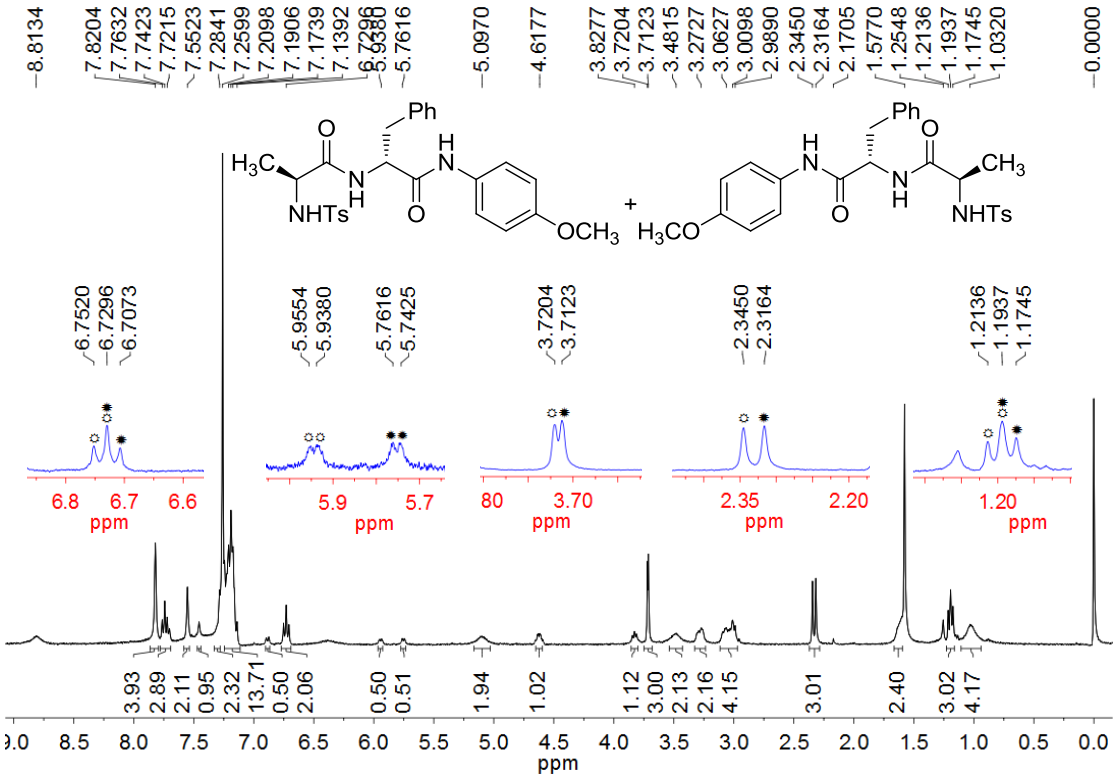


Figure S202. ^{1}H NMR spectrum of (±)-G**23** in the presence of CSA **8** in $CDCl_3$ (400 MHz), [(±)-G**23**] = 4 mM.

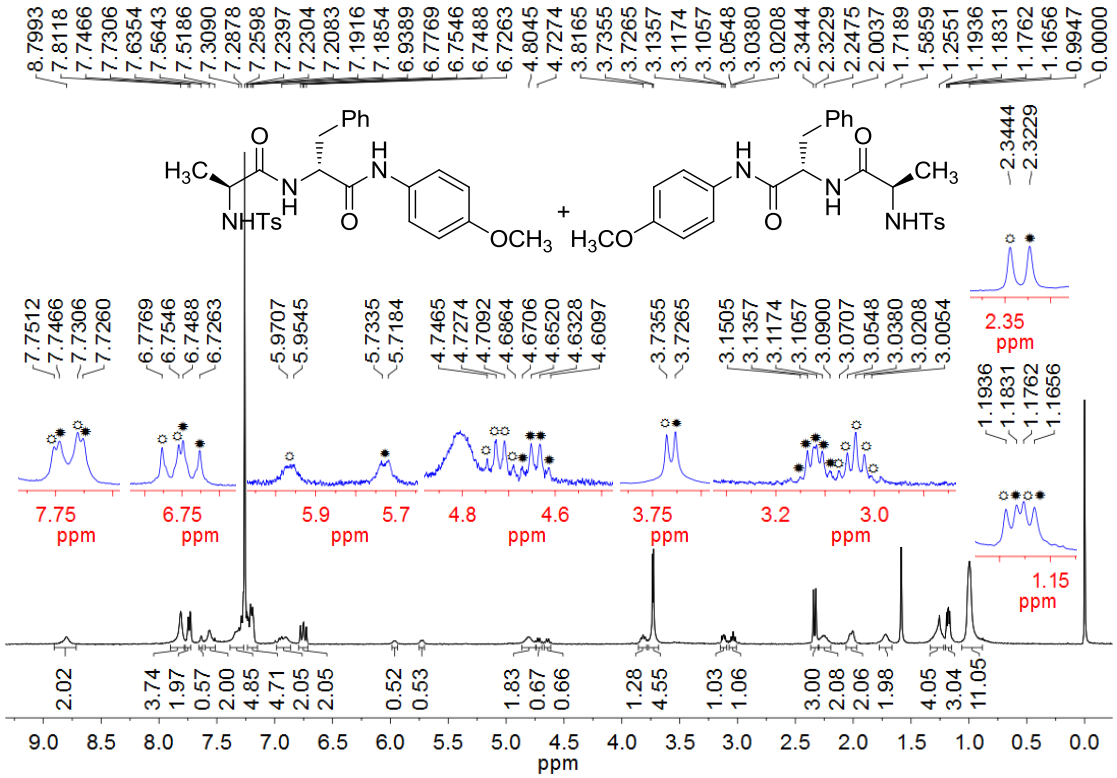


Figure S203. ^{1}H NMR spectrum of (±)-G**23** in the presence of CSA **9** in $CDCl_3$ (400 MHz), [(±)-G**23**] = 4 mM.

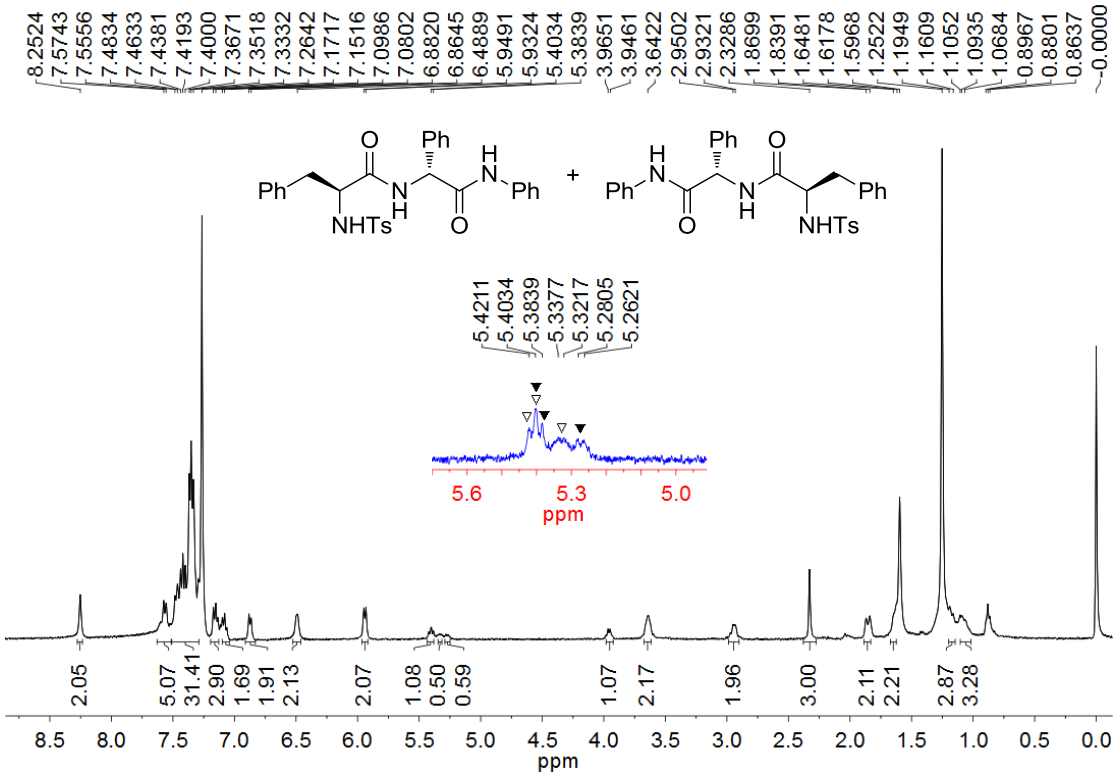


Figure S204. ^{1}H NMR spectrum of (±)-G**24** in the presence of CSA **1** in $CDCl_3$ (400 MHz), [(±)-G**24**] = 2.5 mM.

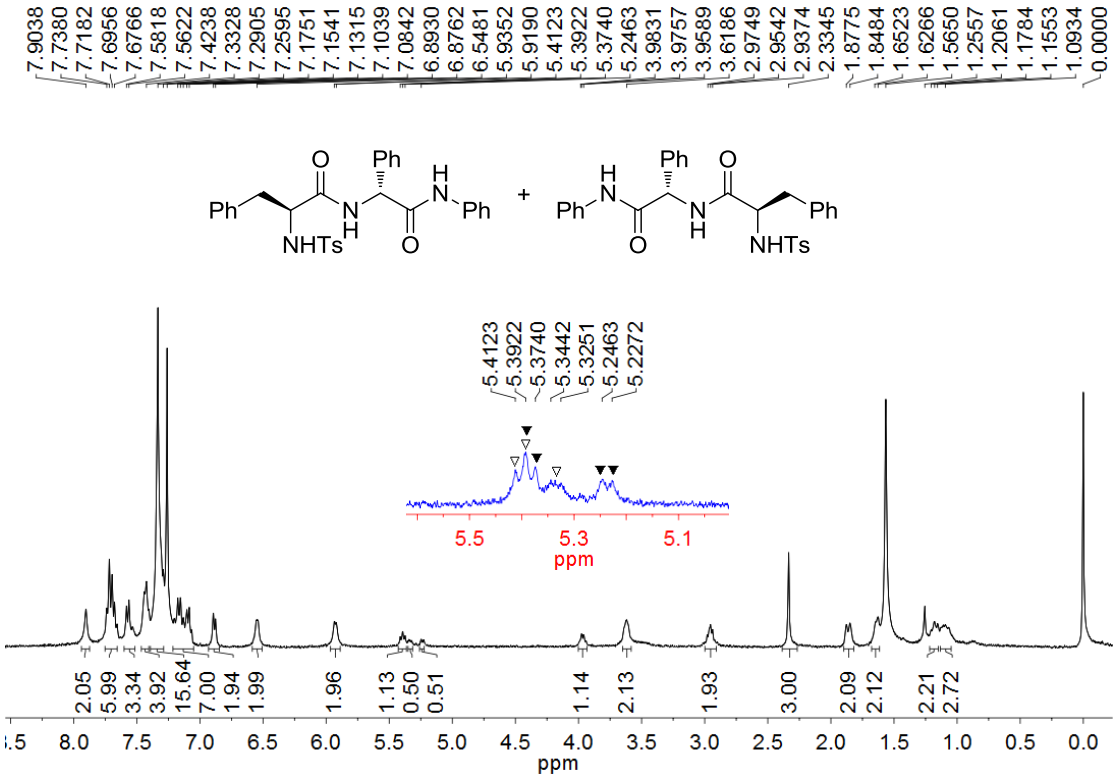


Figure S205. ^{1}H NMR spectrum of (±)-G**24** in the presence of CSA **4** in $CDCl_3$ (400 MHz), [(±)-G**24**] = 2.5 mM.

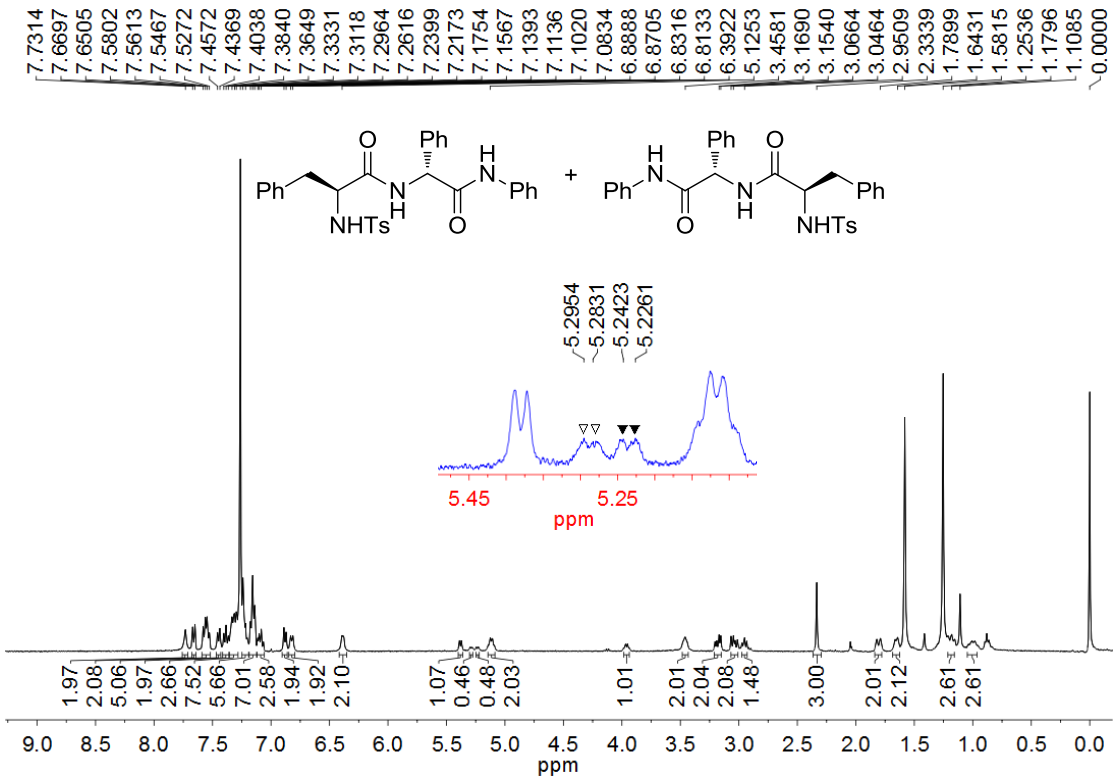


Figure S206. ^{1}H NMR spectrum of (±)-G**24** in the presence of CSA **5** in $CDCl_3$ (400 MHz), [(±)-G**24**] = 2.5 mM.

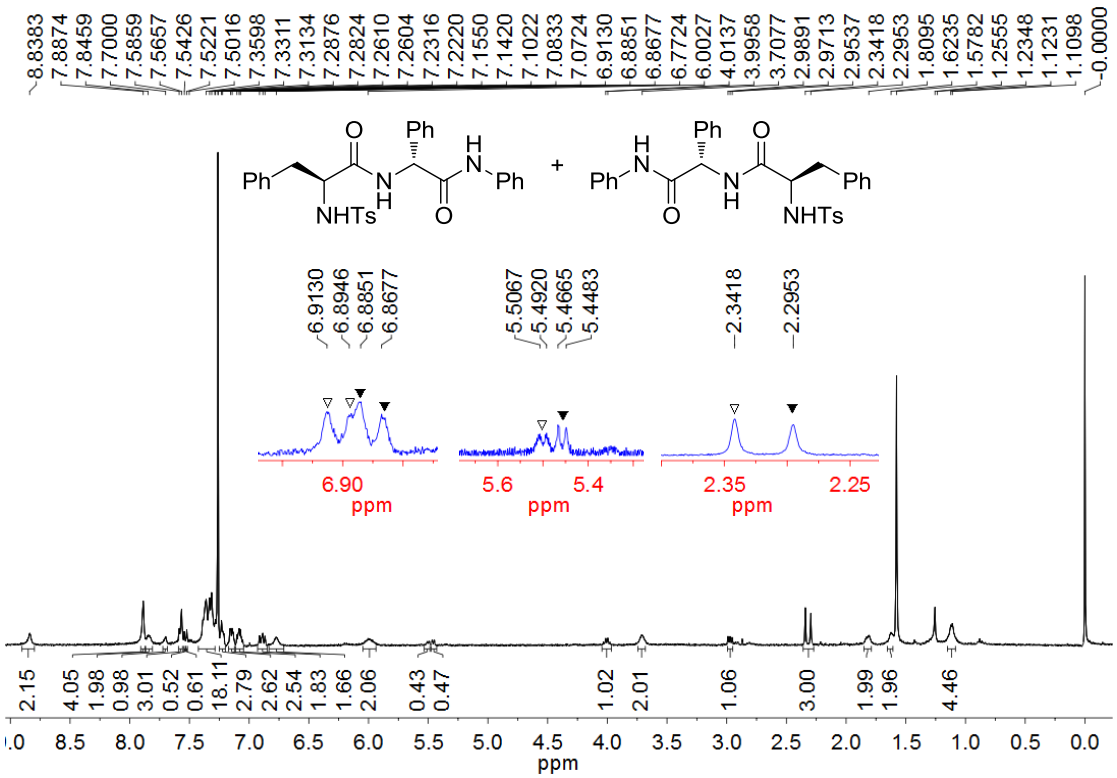


Figure S207. ^{1}H NMR spectrum of (±)-G**24** in the presence of CSA **7** in $CDCl_3$ (400 MHz), [(±)-G**24**] = 2.5 mM.

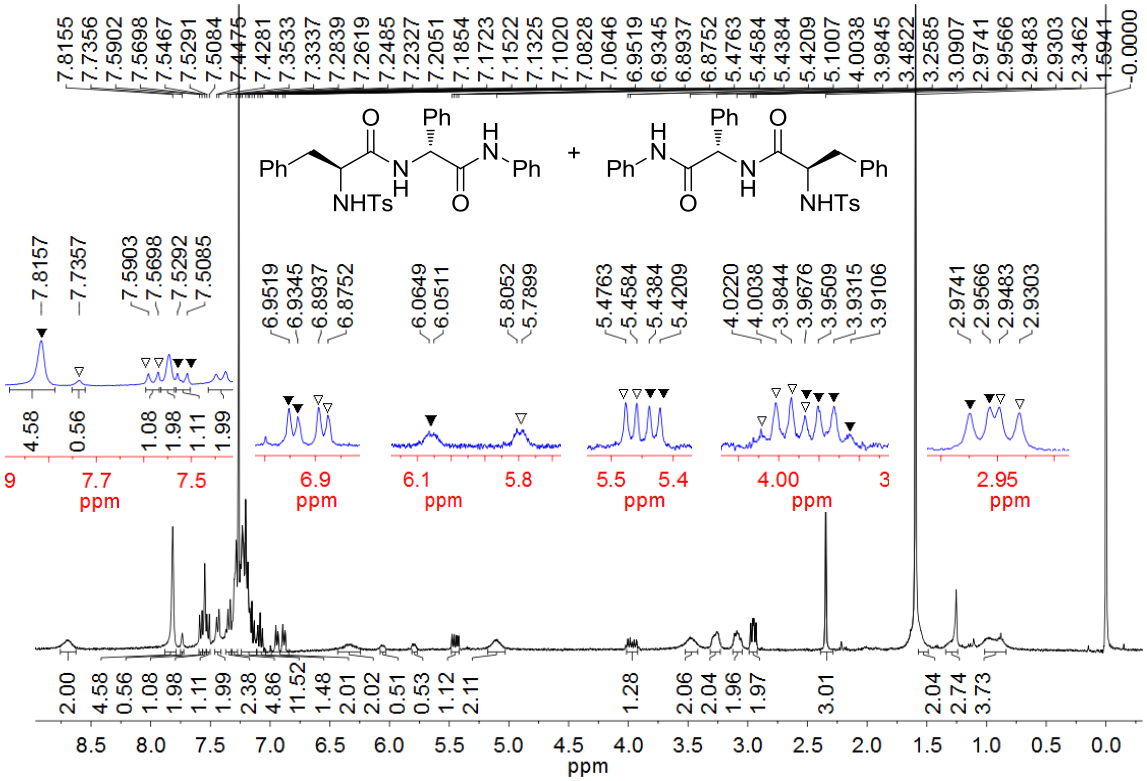


Figure S208. ^{1}H NMR spectrum of (±)-G**24** in the presence of CSA **8** in $CDCl_3$ (400 MHz), [(±)-G**24**] = 2.5 mM.

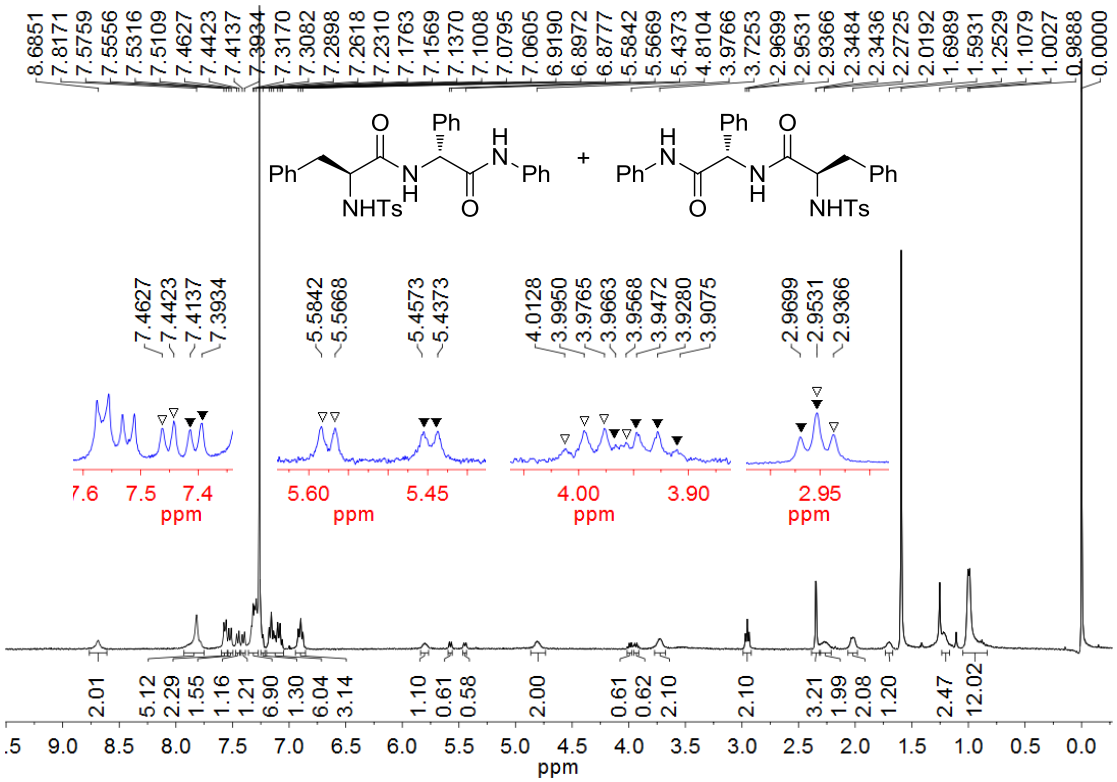


Figure S209. ^{1}H NMR spectrum of (±)-G**24** in the presence of CSA **9** in $CDCl_3$ (400 MHz), [(±)-G**24**] = 2.5 mM.

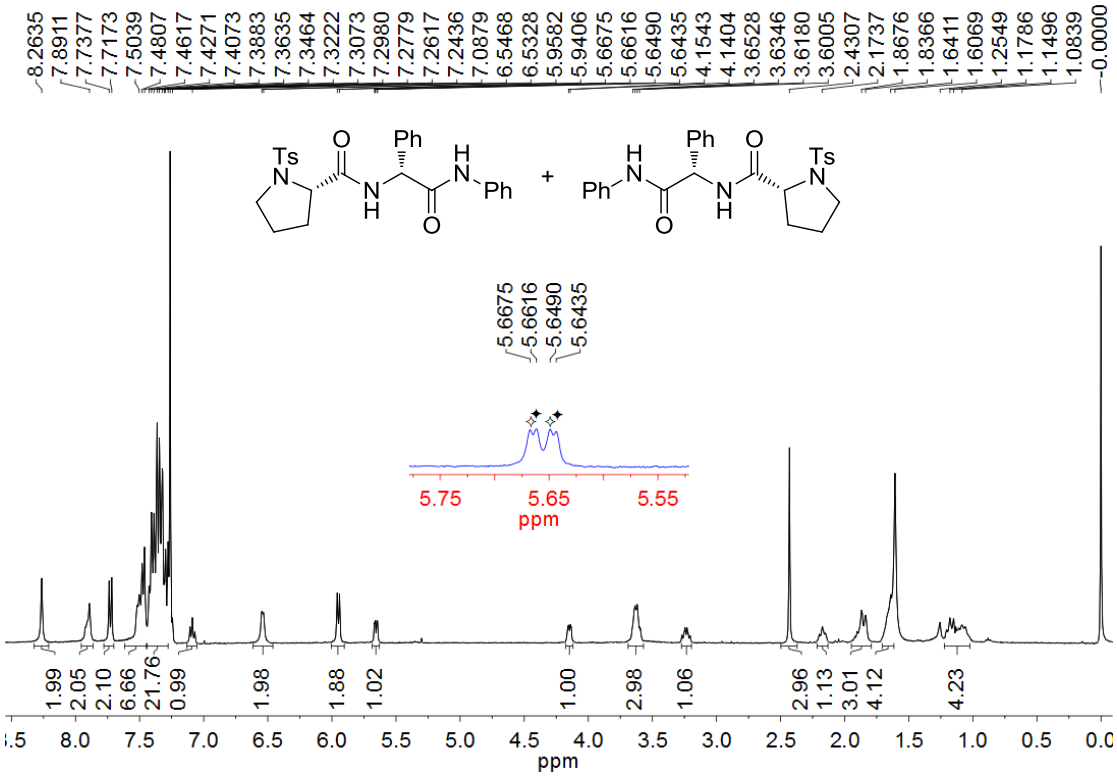


Figure S210. ^{1}H NMR spectrum of (±)-G**25** in the presence of CSA **1** in $CDCl_3$ (400 MHz), [(±)-G**25**] = 5 mM.

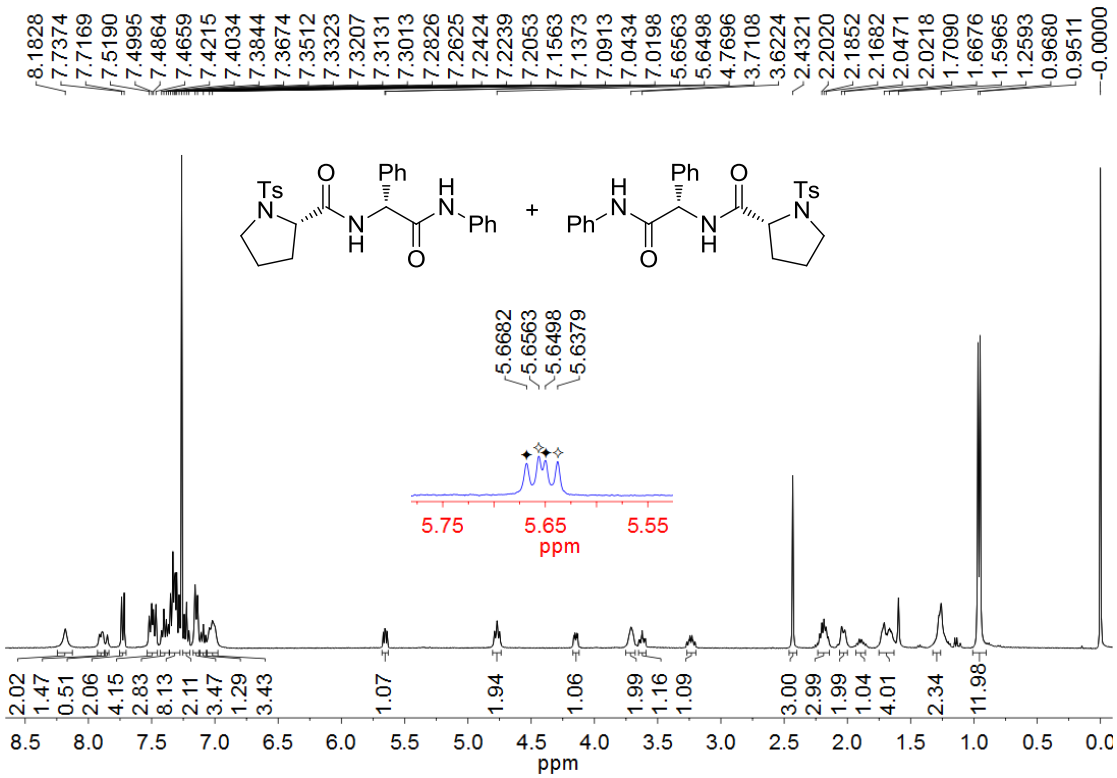


Figure S211. ^{1}H NMR spectrum of (±)-G**25** in the presence of CSA **3** in $CDCl_3$ (400 MHz), [(±)-G**25**] = 5 mM.

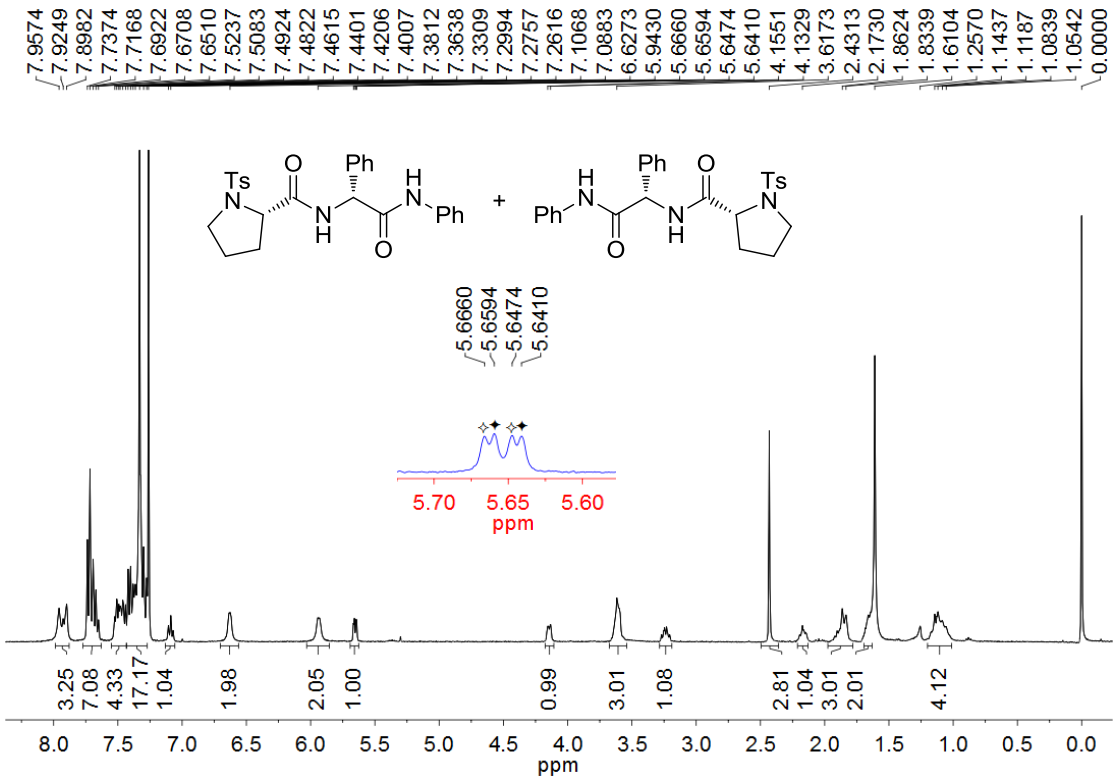


Figure S212. ^{1}H NMR spectrum of (±)-G**25** in the presence of CSA **4** in $CDCl_3$ (400 MHz), [(±)-G**25**] = 5 mM.

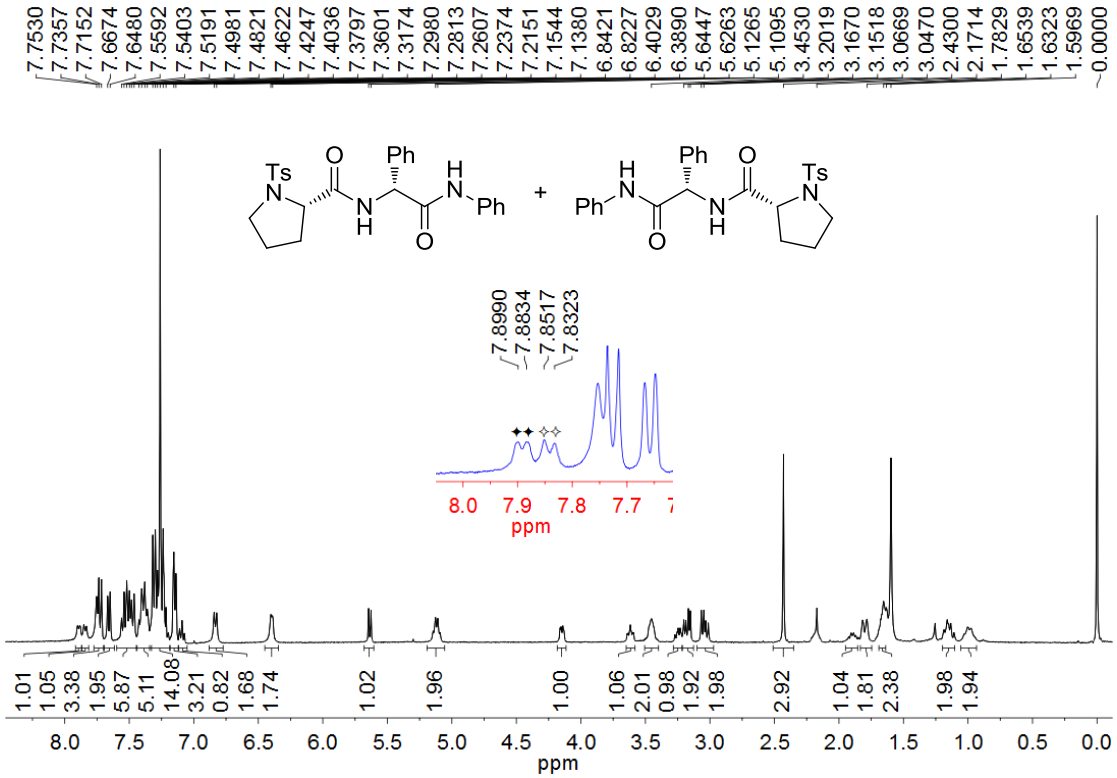


Figure S213. ^{1}H NMR spectrum of (±)-G**25** in the presence of CSA **5** in $CDCl_3$ (400 MHz), [(±)-G**25**] = 5 mM.

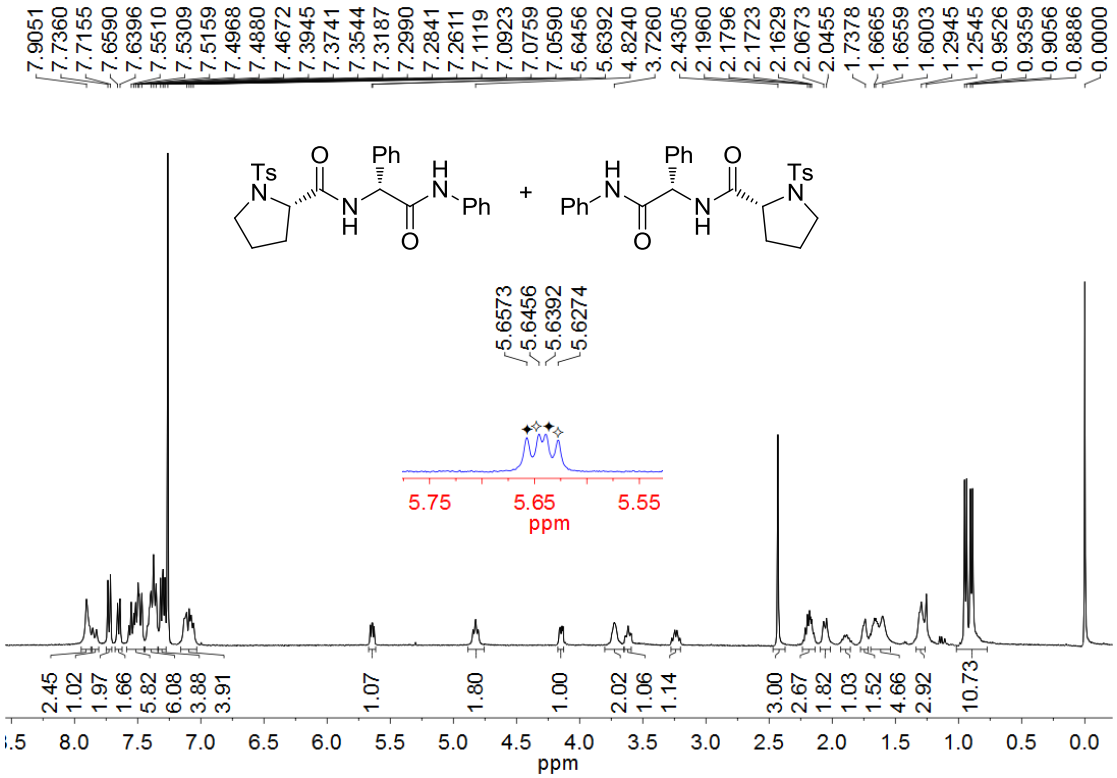


Figure S214. ^{1}H NMR spectrum of (±)-G**25** in the presence of CSA **6** in $CDCl_3$ (400 MHz), [(±)-G**25**] = 5 mM.

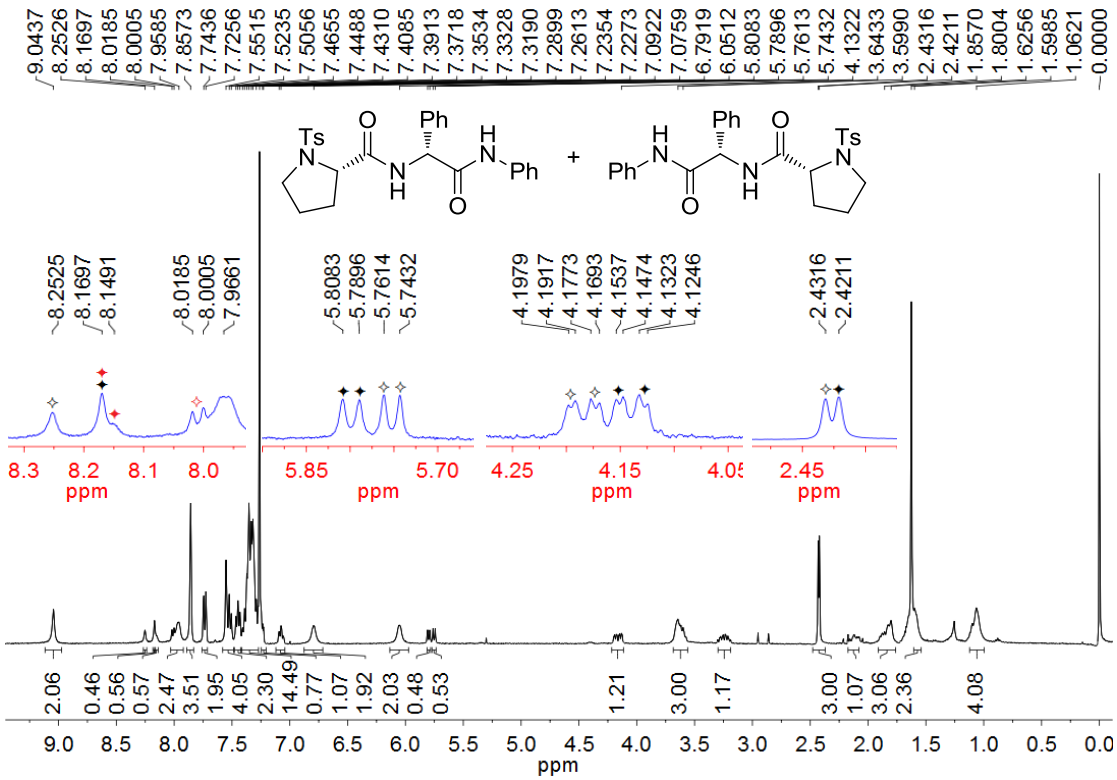


Figure S215. ^{1}H NMR spectrum of (±)-G**25** in the presence of CSA **7** in $CDCl_3$ (400 MHz), [(±)-G**25**] = 5 mM.

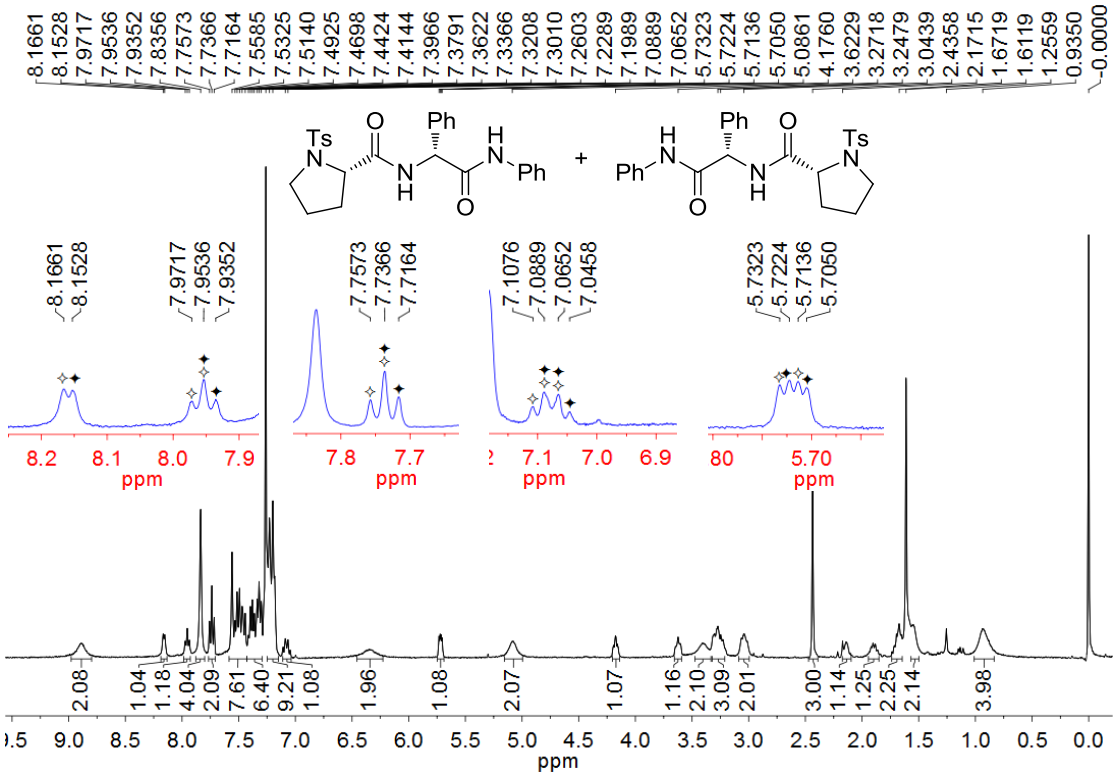


Figure S216. ^{1}H NMR spectrum of (±)-G**25** in the presence of CSA **8** in $CDCl_3$ (400 MHz), [(±)-G**25**] = 5 mM.

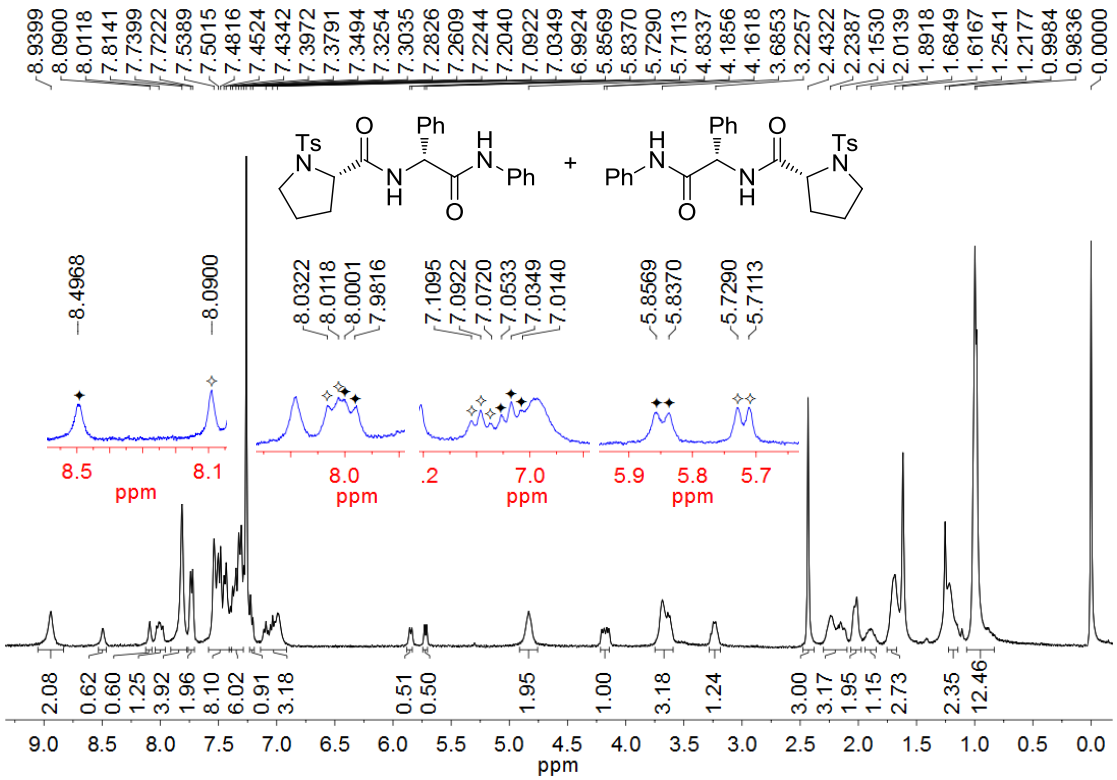


Figure S217. ^{1}H NMR spectrum of (±)-G**25** in the presence of CSA **9** in $CDCl_3$ (400 MHz), [(±)-G**25**] = 5 mM.

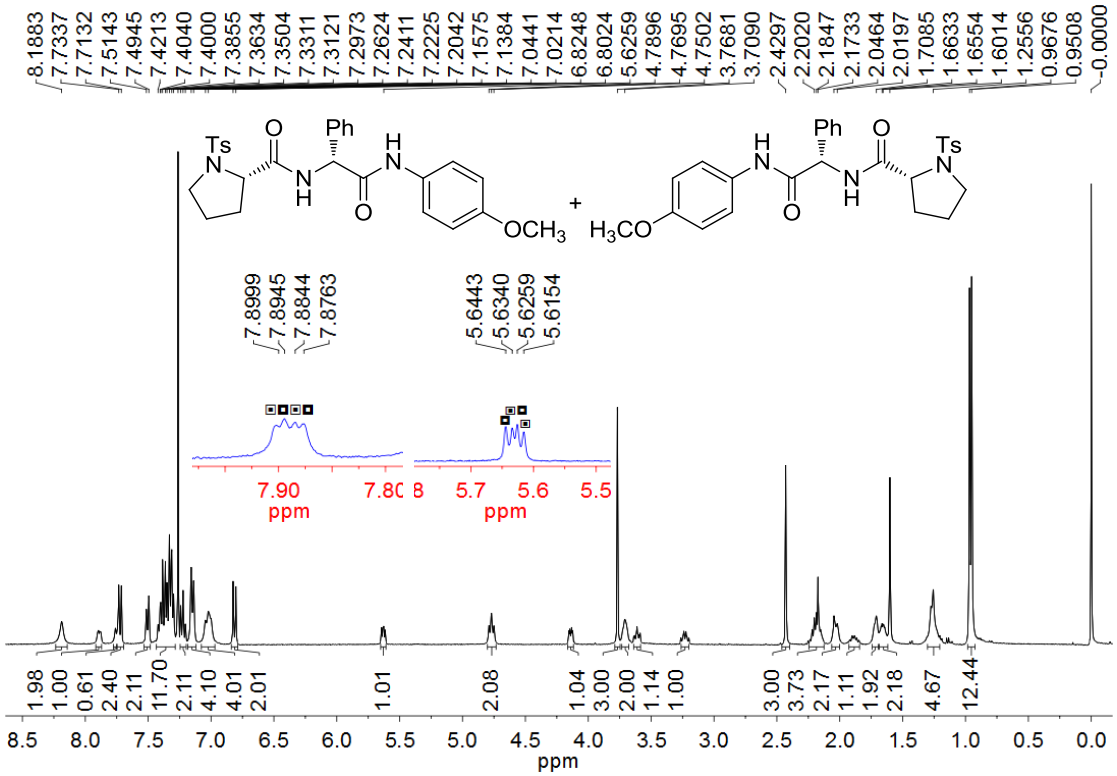


Figure S218. ^{1}H NMR spectrum of (±)-G**26** in the presence of CSA **3** in $CDCl_3$ (400 MHz), [(±)-G**26**] = 5 mM.

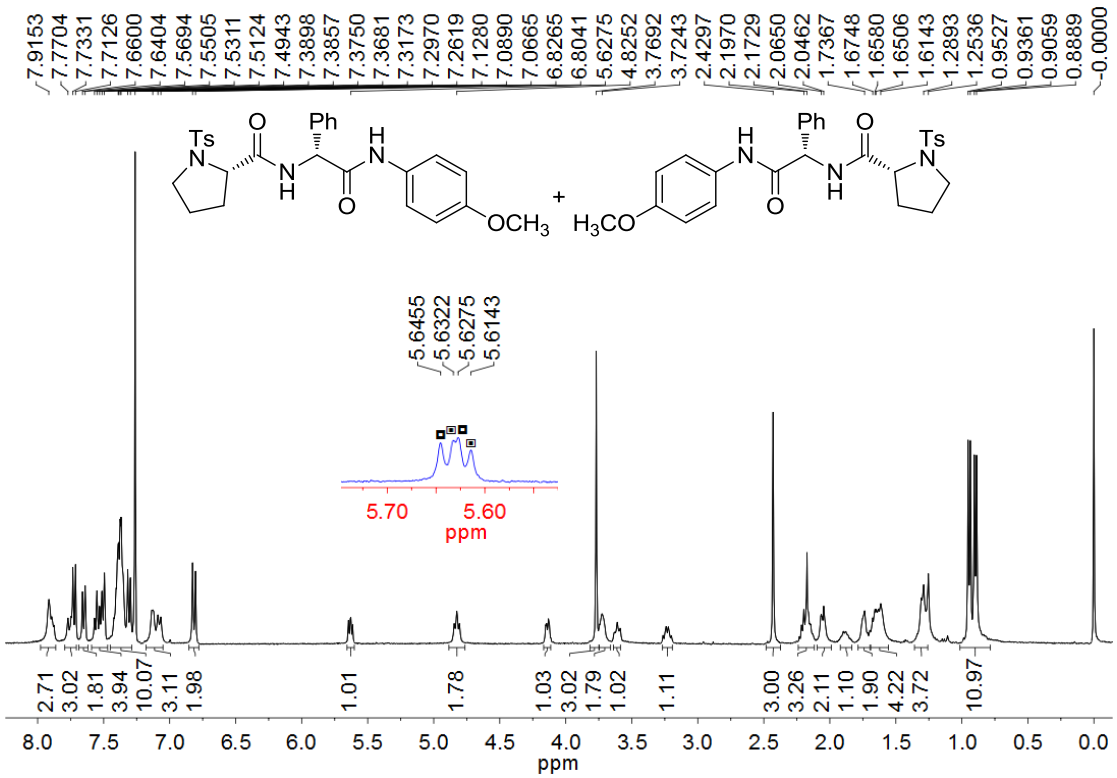


Figure S219. ^{1}H NMR spectrum of (±)-G**26** in the presence of CSA **6** in $CDCl_3$ (400 MHz), [(±)-G**26**] = 5 mM.

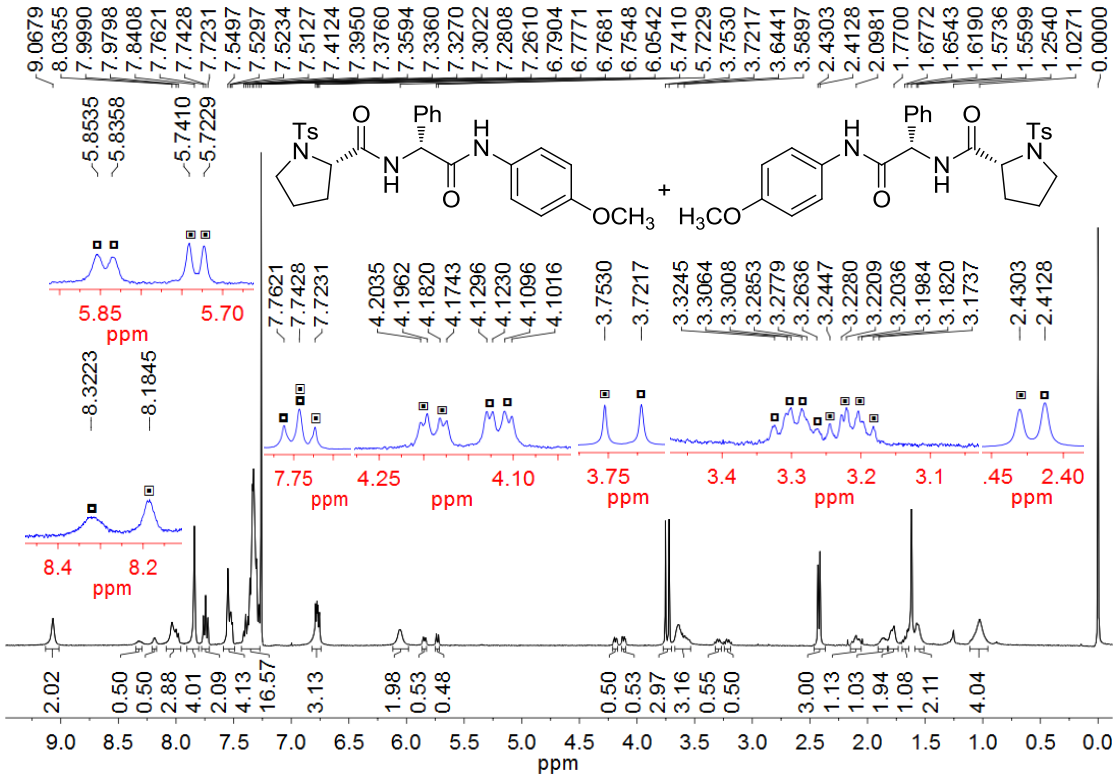


Figure S220. ^{1}H NMR spectrum of (±)-G**26** in the presence of CSA **7** in $CDCl_3$ (400 MHz), [(±)-G**26**] = 5 mM.

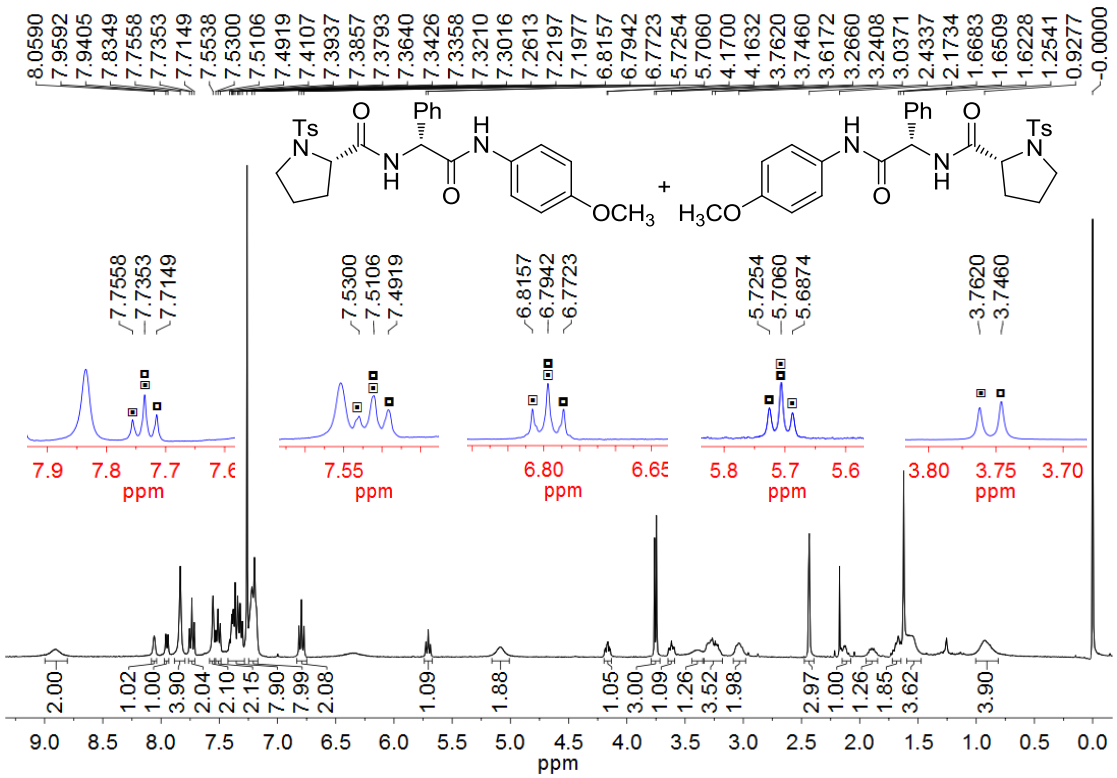


Figure S221. ^{1}H NMR spectrum of (±)-G**26** in the presence of CSA **8** in $CDCl_3$ (400 MHz), [(±)-G**26**] = 5 mM.

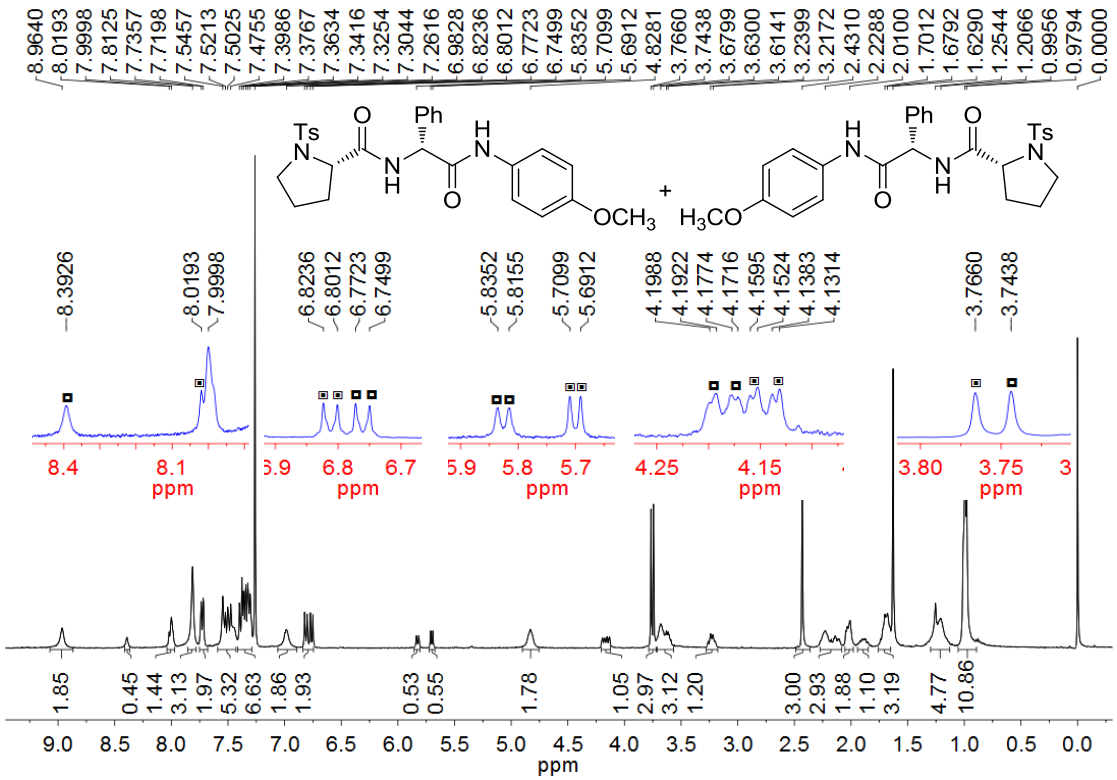


Figure S222. ^{1}H NMR spectrum of (±)-G**26** in the presence of CSA **9** in $CDCl_3$ (400 MHz), [(±)-G**26**] = 5 mM.

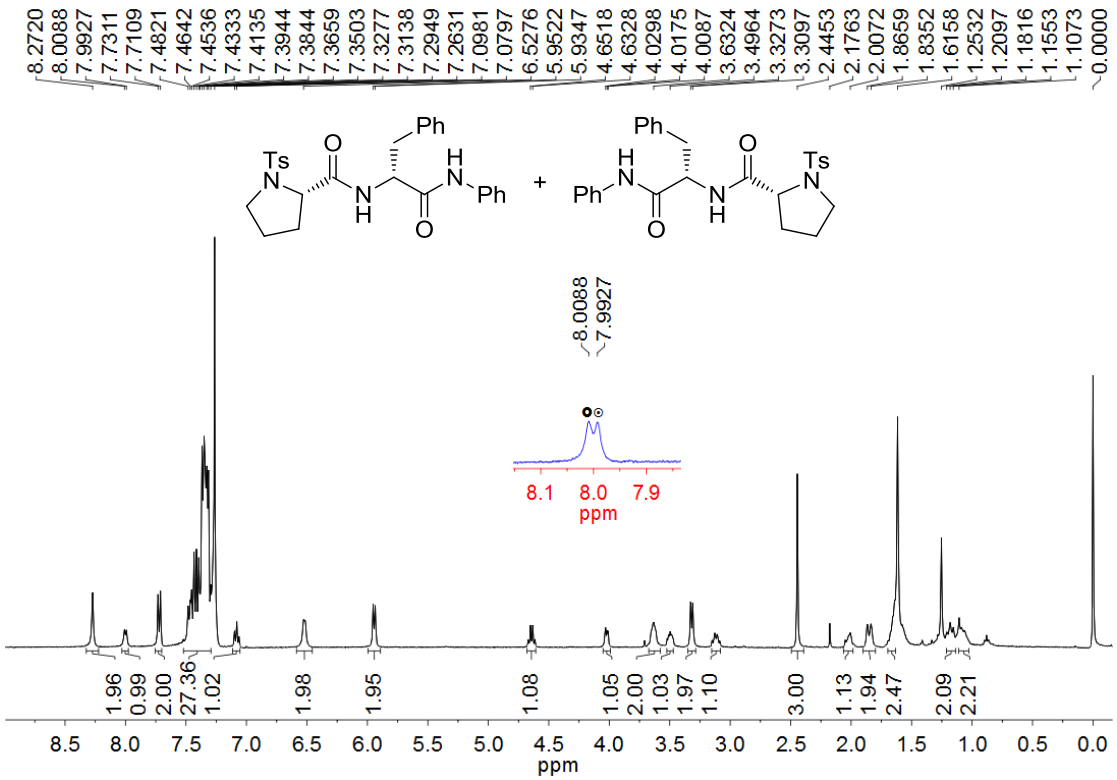


Figure S223. ^{1}H NMR spectrum of (±)-G**27** in the presence of CSA **1** in $CDCl_3$ (400 MHz), [(±)-G**27**] = 5 mM.

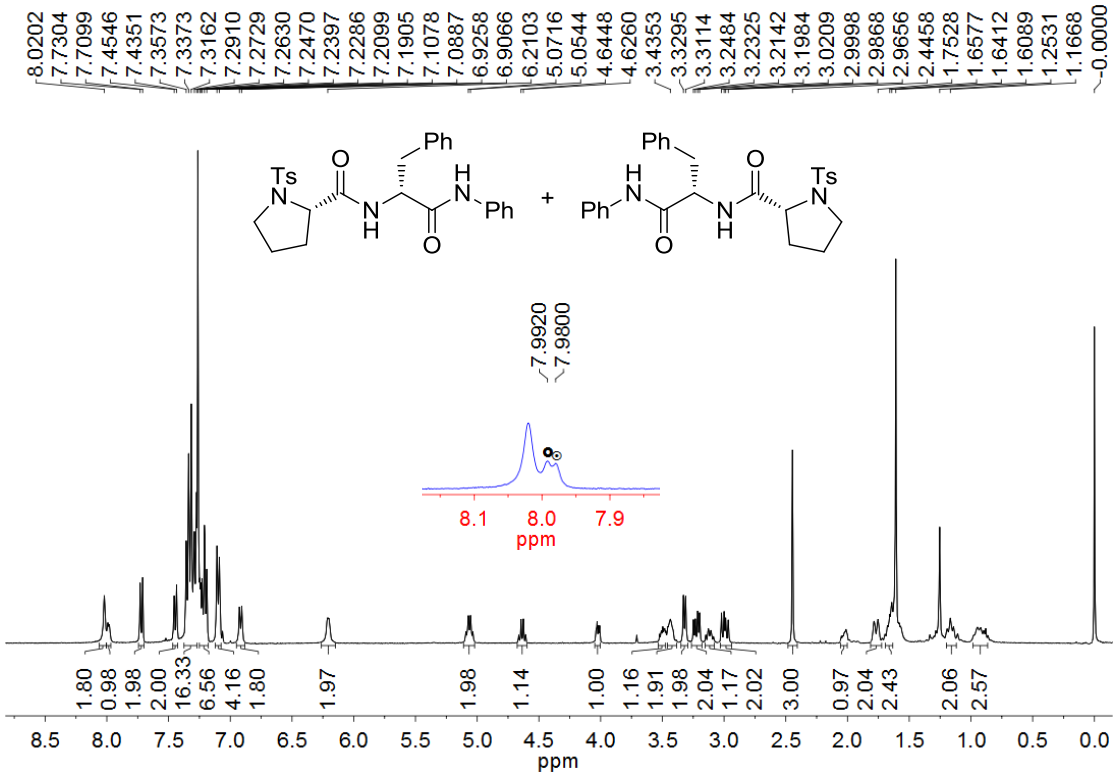


Figure S224. ^{1}H NMR spectrum of (±)-G**27** in the presence of CSA **2** in $CDCl_3$ (400 MHz), [(±)-G**27**] = 5 mM.

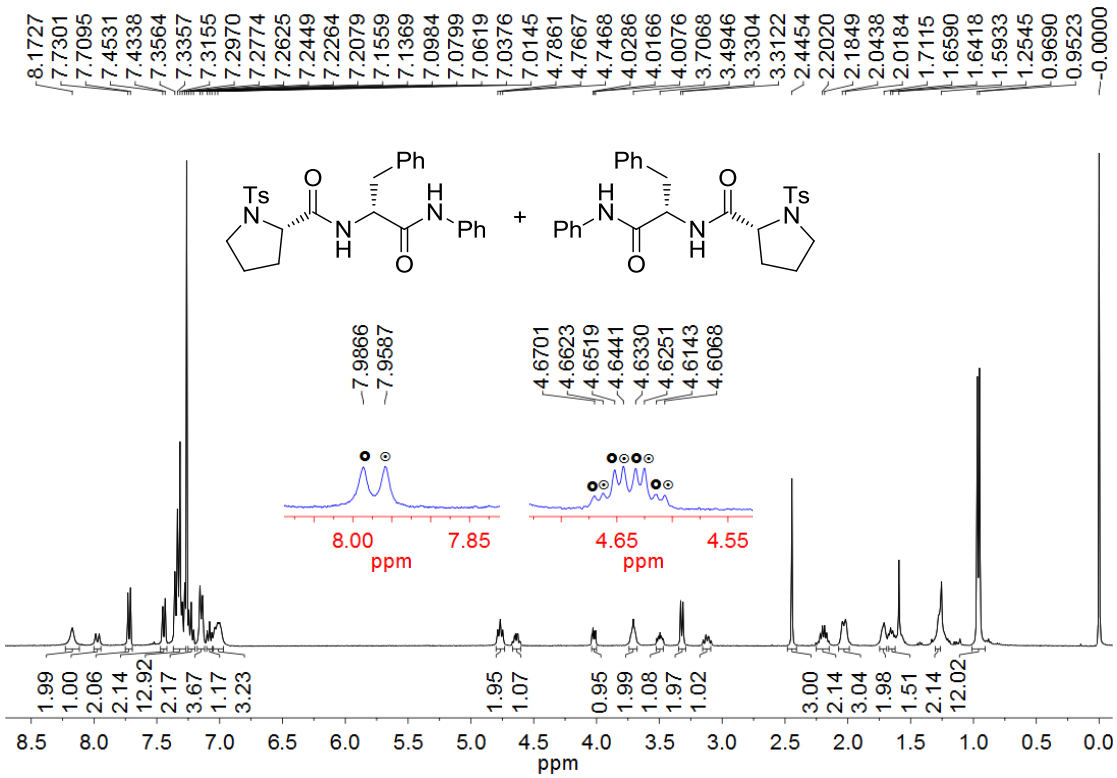


Figure S225. ^{1}H NMR spectrum of (±)-G**27** in the presence of CSA **3** in $CDCl_3$ (400 MHz), [(±)-G**27**] = 5 mM.

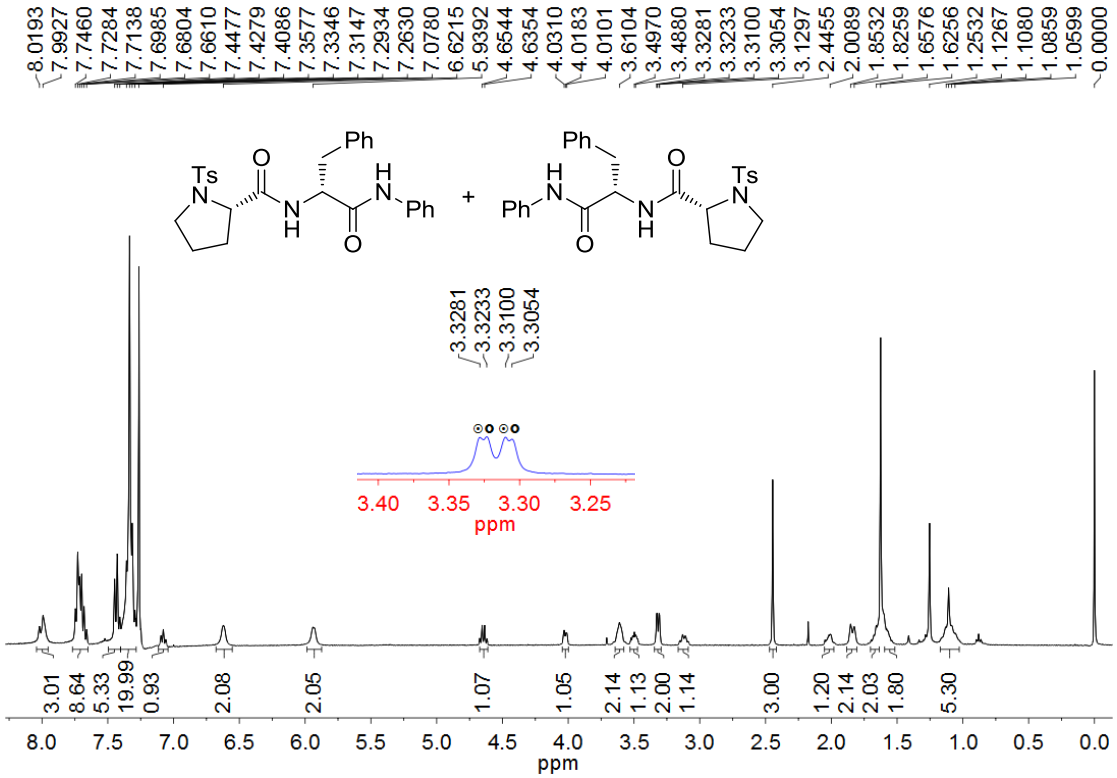


Figure S226. ^{1}H NMR spectrum of (±)-G**27** in the presence of CSA **4** in $CDCl_3$ (400 MHz), [(±)-G**27**] = 5 mM.

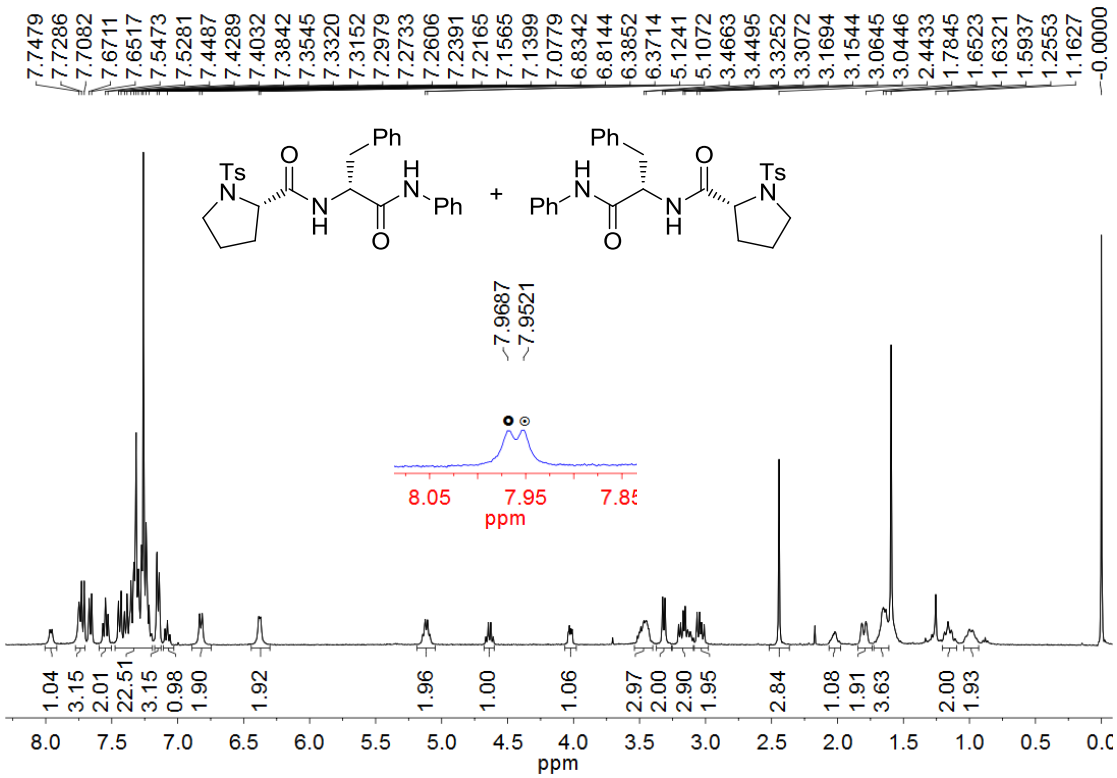


Figure S227. ^{1}H NMR spectrum of (±)-G**27** in the presence of CSA **5** in $CDCl_3$ (400 MHz), [(±)-G**27**] = 5 mM.

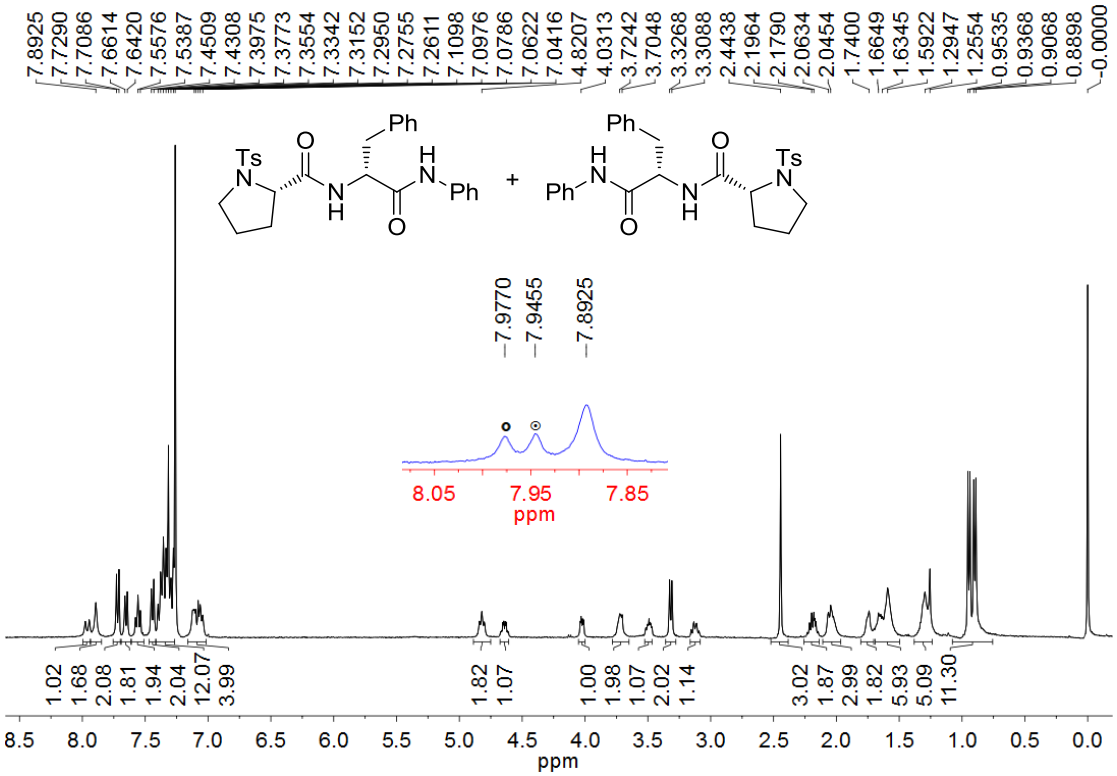


Figure S228. ^{1}H NMR spectrum of (±)-G**27** in the presence of CSA **6** in $CDCl_3$ (400 MHz), [(±)-G**27**] = 5 mM.

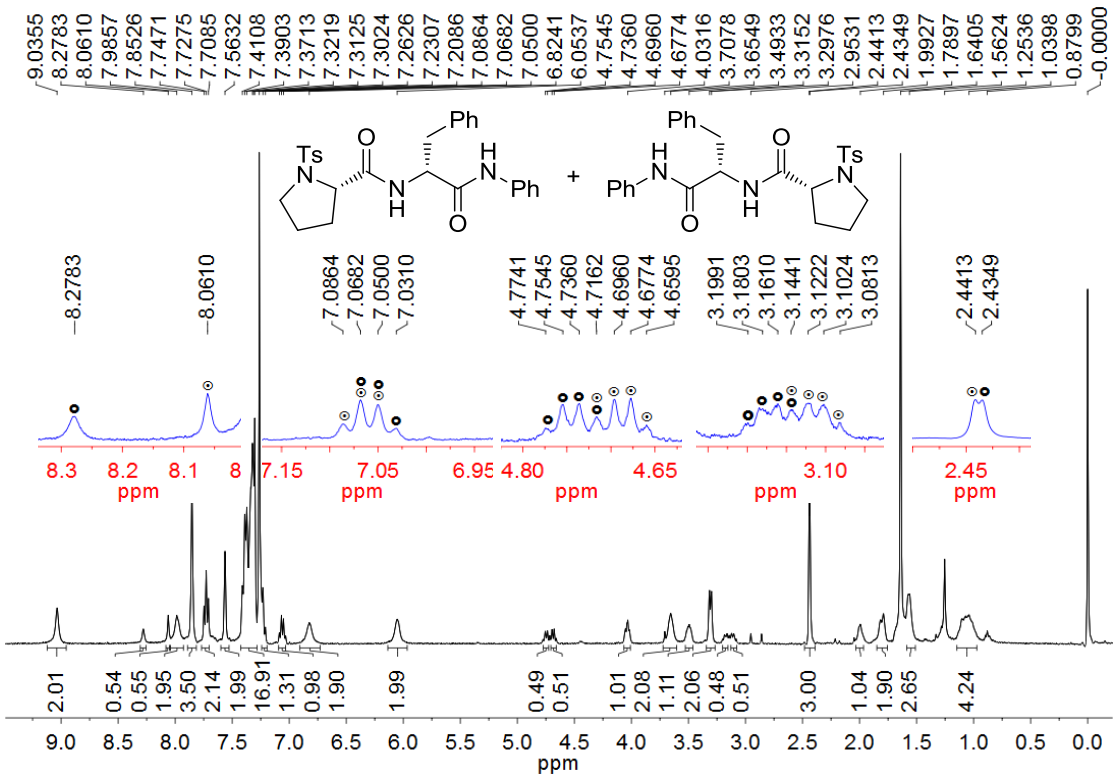


Figure S229. ^{1}H NMR spectrum of (±)-G**27** in the presence of CSA **7** in $CDCl_3$ (400 MHz), [(±)-G**27**] = 5 mM.

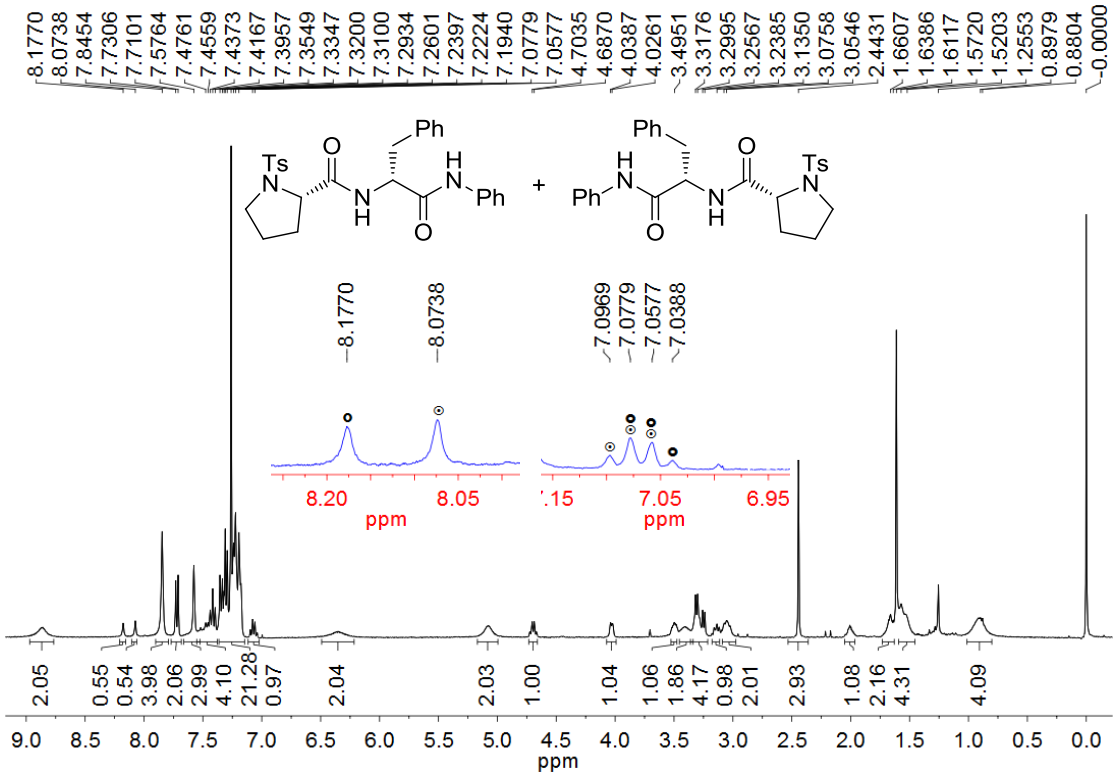


Figure S230. ^{1}H NMR spectrum of (±)-G**27** in the presence of CSA **8** in $CDCl_3$ (400 MHz), [(±)-G**27**] = 5 mM.

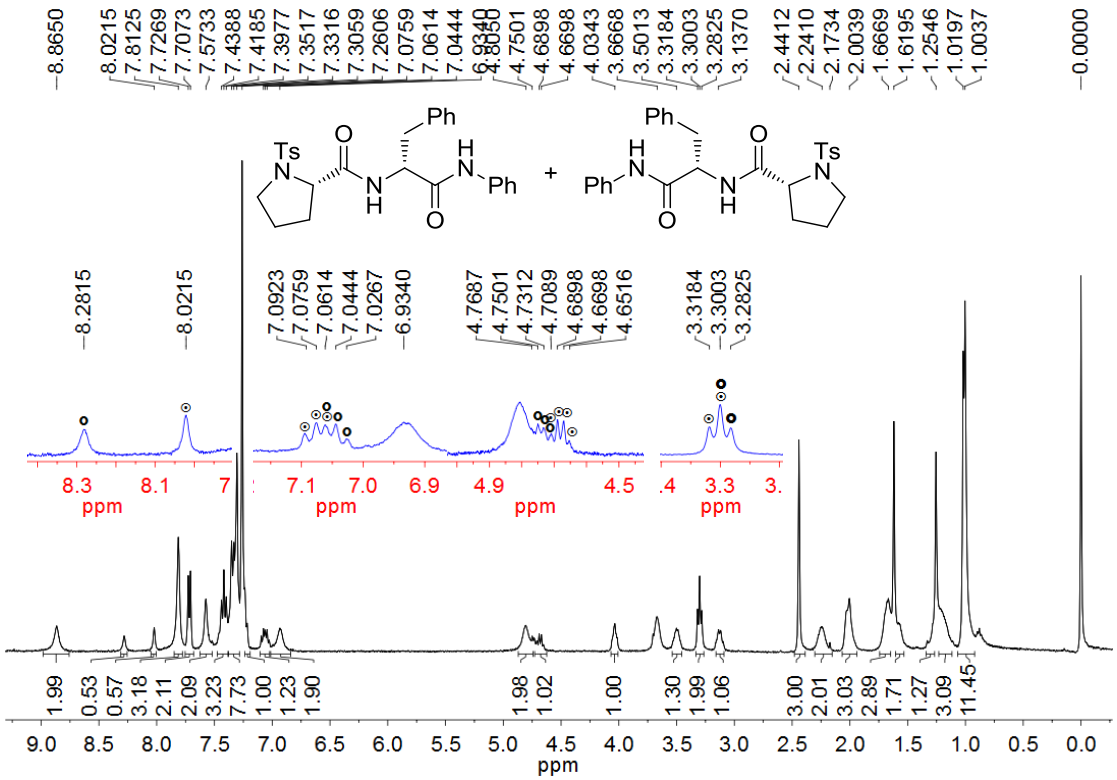


Figure S231. ^{1}H NMR spectrum of (±)-G**27** in the presence of CSA **9** in $CDCl_3$ (400 MHz), [(±)-G**27**] = 5 mM.