Highly enantioselective synthesis of spirocyclopropyloxindoles via Rh(II)-catalyzed asymmetric cyclopropanation reaction

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Supporting Information

General Information

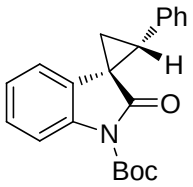
General experimental: All reactions were carried out under an atmosphere of argon using oven-dried glassware. Solvents were dried and degassed by standard methods. And alkenes used in the reaction were obtained from commercial sources and used without further purification. Flash column chromatography was performed using silica gel (300-400 mesh). Analytical thin-layer chromatography was performed using glass plates pre-coated with 200-300 mesh silica gel impregnated with a fluorescent indicator (254 nm). NMR spectra were recorded in $CDCl_3$ on a Varian Inova-400 NMR spectrometer (400 MHz); chemical shifts were reported in ppm with the solvent or internal TMS signals as reference, and coupling constants (J) were given in Hertz. The peak information was described as: br = broad, s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet, comp = composite. High-resolution mass spectra (HRMS) were recorded on a commercial apparatus (ESI Source). The characteristic data of **3b-3d** are consistent with the reported reference.[1] The dirhodium carboxylates catalysts are prepared according to the literature.[2]

General Procedure for the Catalytic Asymmetric Cyclopropanation

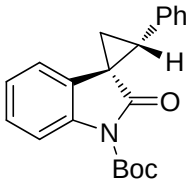
Method A: Rh_2(*S*-NTTL)$_4$ (0.001 mmol, 1.4 mg), alkenes (0.6 mmol or 1.0 mmol for alkyl alkenes) and DCE (1.0 mL) were added to a oven-dried flask containing a magnetic stirring bar under Ar. The reaction mixture was heated to 40 °C, and diazooxindole (0.2 mmol) in DCE (1.0 mL) was added slowly in 1 h *via* a syringe pump. After being stirred for another 2 h, the solvent was removed under reduced pressure (the diastereoselectivity was determined by ^{1}H NMR of the crude reaction mixture), and the resulting residue was purified by column chromatography on silica gel to give the pure products. The enantiomeric excesses of the products were determined by chiral HPLC analysis.

Method B: Rh_2(*S*-TBPTTL)$_4$ (0.001 mmol, 2.5 mg), alkenes (0.6 mmol) and DCE (1.0 mL) were added to a oven-dried flask containing a magnetic stirring bar under Ar. The reaction mixture was heated to 40 °C, and diazooxindole (0.2 mmol) in DCE (1.0 mL) was added slowly in 1 h *via* a syringe pump. After being stirred for another 2 h, the solvent was removed under reduced pressure (the diastereoselectivity was determined by ^{1}H NMR of the crude reaction mixture), and the resulting residue was purified by column chromatography on silica gel to afford the pure products. The enantiomeric excesses of the products were determined by chiral HPLC analysis.

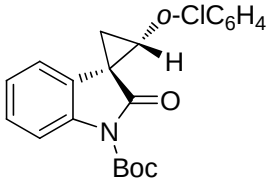
Characterization Data of Products 3 and 4



(*1S,2R*)-*tert*-Butyl 2'-Oxo-2-phenylspiro[cyclopropane-1,3'-indoline]-1'-carboxylate (*trans*-3a). Red oil (Method A: 60.9 mg, 91% yield, 93% *ee*). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.93 (d, *J* = 8.1 Hz, 1H), 7.36-7.27 (comp, 3H), 7.24-7.14 (comp, 3H), 6.78 (t, *J* = 7.6, 1H), 5.95 (d, *J* = 7.6, 1H), 3.42 (t, *J* = 8.7 Hz, 1H), 2.31-2.28 (m, 1H), 2.04-2.01 (m, 1H), 1.72 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 175.0, 149.3, 139.6, 134.2, 130.0, 128.4, 127.6, 126. 8, 126.3, 123.4, 120.4, 114.6, 84.1, 38.1, 33.6, 28.1, 24.1. HRMS (ESI) calculated for $C_{21}H_{21}NNaO_3$ $[M+Na]^+$: 358.1419, found 358.1431. HPLC analysis: Chiralpak IC, 254 nm, *i*PrOH/Hexanes = 3:97, 1.0 mL/min, R_t (major) = 17.2 min, R_t (minor) = 19.0 min.

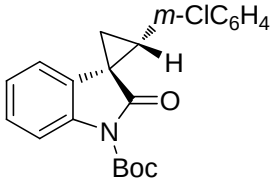


(*1R,2R*)-*tert*-Butyl 2'-Oxo-2-phenylspiro[cyclopropane-1,3'-indoline]-1'-carboxylate (*cis*-3a). Red oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.86 (d, *J* = 8.1 Hz, 1H), 7.41-7.23 (comp, 6H), 7.18 (t, *J* = 7.9 Hz, 1H), 6.95 (d, *J* = 7.4 Hz, 1H), 3.14 (t, *J* = 8.9 Hz, 1H), 2.46 (m, 1H), 2.09 (m, 1H), 1.57 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 171.7, 139.4, 133.7, 132.9, 129.5, 128.2, 127.5, 127.2, 124.2, 124.1, 118.0, 115.0, 84.1, 40.8, 34.6, 28.2, 24.3. HRMS (ESI) calculated for $C_{21}H_{21}NNaO_3$ $[M+Na]^+$: 358.1419, found 358.1431. HPLC analysis: Chiralpak IC, 254 nm, *i*PrOH/Hexanes = 3:97, 1.0 mL/min, R_t (minor) = 26.1 min, R_t (major) = 29.8 min.

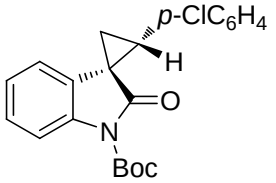


(*1S,2R*)-*tert*-Butyl 2-(2-Chlorophenyl)-2'-oxospiro[cyclopropane-1,3'-indoline]-1'-carboxylate (3e). Red oil (Method A: 57.7 mg, 78% yield, 89% *ee*). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.80 (d, *J* = 8.1 Hz, 1H),

7.34 (d, *J* = 7.6 Hz, 1H), 7.26-7.21 (m, 1H), 7.18-7.13 (m, 2H), 7.11-7.05 (m, 1H), 6.65 (t, *J* = 7.6 Hz, 1H), 5.73 (d, *J* = 7.4 Hz, 1H), 3.17 (t, *J* = 8.6 Hz, 1H), 2.27-2.24 (m, 1H), 1.93-1.90 (m, 1H), 1.60 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 175.0, 149.5, 139.9, 137.1, 133.1, 130.5, 129.5, 129.2, 127.1, 126.8, 126.2, 123.5, 119.2, 114.8, 84.3, 36.8, 33.6, 28.2, 23.7. HRMS (ESI) calculated for $C_{21}H_{20}ClNNaO_3$ $[M+Na]^+$: 392.1029, found 392.1041. HPLC analysis: Chiralpak IC, 254 nm, *i*PrOH/Hexanes = 3:97, 1.0 mL/min, R_t (major) = 12.2 min, R_t (minor) = 13.5 min.

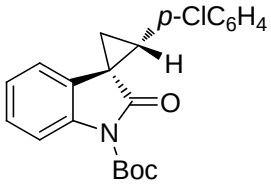


(*1S,2R*)-*tert*-Butyl 2-(3-Chlorophenyl)-2'-oxospiro[cyclopropane-1,3'-indoline]-1'-carboxylate (3f). Red oil (Method A: 60.6 mg, 82% yield, 88% *ee*). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.89 (d, *J* = 8.1 Hz, 1H), 7.27-7.17 (comp, 4H), 7.03 (d, *J* = 7.3 Hz, 1H), 6.81 (t, *J* = 7.6 Hz, 1H), 5.96 (d, *J* = 7.6 Hz, 1H), 3.33 (t, *J* = 8.7 Hz, 1H), 2.28-2.25 (m, 1H), 1.98-1.94 (m, 1H), 1.68 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 174.9, 149.4, 139.9, 136.6, 134. 4, 130.1, 129.8, 128.5, 128.0, 127.2, 126.0, 123.7, 120.5, 115.0, 84.5, 37.3, 33.7, 28.2, 24.0. HRMS (ESI) calculated for $C_{21}H_{20}ClNNaO_3$ $[M+Na]^+$: 392.1029, found 392.1037. HPLC analysis: Chiralpak IC, 254 nm, *i*PrOH/Hexanes = 3:97, 1.0 mL/min, R_t (major) = 14.6 min, R_t (minor) = 15.7 min.

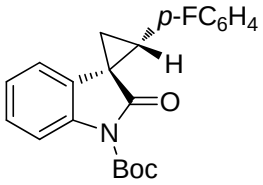


(*1S,2R*)-*tert*-Butyl 2-(4-Chlorophenyl)-2'-oxospiro[cyclopropane-1,3'-indoline]-1'-carboxylate (*trans*-3g). Red oil (Method A: 52.5 mg, 71% yield, 89% *ee*). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.89 (d, *J* = 8.1 Hz, 1H), 7.28-7.25 (m, 2H), 7.20-7.16 (m,1H), 7.10 (d, *J* = 8.2 Hz, 2H), 6.81 (t, *J* = 7.6 Hz, 1H), 5.93 (d, *J* = 7.6 Hz, 1H), 3.32 (t, *J* = 8.7 Hz, 1H), 2.29-2.26 (m, 1H), 1.96-1.93 (m, 1H), 1.68 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 175.0, 149.4, 139.9, 133.7, 133.1, 131.5, 128.8, 127.2, 126.1, 123.7, 120.5, 114.9, 84.5, 37.3, 33.7, 28.2, 24.2. HRMS (ESI) calculated for $C_{21}H_{20}ClNNaO_3$ $[M+Na]^+$: 392.1029, found 392.1034. HPLC analysis: Chiralpak IC, 254 nm, 3:97 *i*PrOH/Hexanes, 1.0

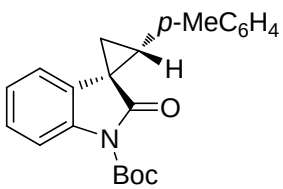
mL/min, R_t (major) = 11.8 min, R_t (minor) = 14.5 min.



(*1R,2R*)-*tert*-Butyl 2-(4-Chlorophenyl)-2'-oxospiro[cyclopropane-1,3'-indoline]-1'-carboxylate (*cis*-3g). Red oil (Method A: 94% *ee*). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.86 (d, *J* = 8.1 Hz, 1H), 7.25-7.15 (comp, 6H), 6.94 (d, *J* = 8.4 Hz, 1H), 3.08 (t, *J* = 8.9 Hz, 1H), 2.42-2.38 (m, 1H), 2.11-2.08 (m, 1H), 1.59 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 171.6, 149.3, 139.4, 133.3, 132.2, 130.8, 129.6, 128.3, 127.4, 124.3, 118.0, 115.0, 84.3, 39.8, 34.5, 28.2, 27.0. HRMS (ESI) calculated for $C_{21}H_{20}ClNNaO_3$ $[M+Na]^+$: 392.1029, found 392.1034. HPLC analysis: Chiralpak IC, 254 nm, *i*PrOH/Hexanes = 3:97, 1.0 mL/min, R_t (major) = 15.5 min, R_t (minor) = 16.6 min.

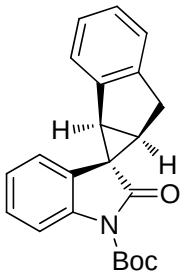


(*1S,2R*)-*tert*-Butyl 2-(4-Fluorophenyl)-2'-oxospiro[cyclopropane-1,3'-indoline]-1'-carboxylate (3h). Red oil (Method A: 51.5 mg, 73% yield, 88% *ee*). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.81 (d, *J* = 8.2 Hz, 1H), 7.13-7.02 (comp, 3H), 6.90 (t, *J* = 8.6 Hz, 2H), 6.74-6.70 (m, 1H), 5.85-5.58 (m, 1H), 3.25 (t, *J* = 8.6 Hz, 1H), 2.22-2.18 (m, 1H), 1.88-1.85 (m, 1H), 1.60 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 175.1, 162.2 (d, *J* = 245.2 Hz), 149.4, 139.8, 131.8 (d, *J* = 8.2 Hz), 130.3 (d, *J* = 3.2 Hz), 127.1, 126.2, 123.7, 120.5, 115.6 (d, *J* = 21.4 Hz), 114.9, 84.5, 37.3, 33.7, 28.2, 24.5. HRMS (ESI) calculated for $C_{21}H_{20}FNNaO_3$ $[M+Na]^+$: 376.1325, found 376.1321. HPLC analysis: Chiralpak IC, 254 nm, *i*PrOH/Hexanes = 3:97, 1.0 mL/min, R_t (major) = 4.8 min, R_t (minor) = 5.4 min.

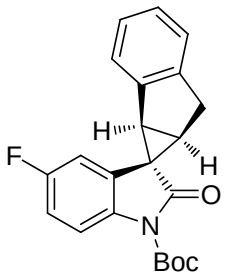


(*1S,2R*)-*tert*-Butyl 2'-Oxo-2-(p-tolyl)spiro[cyclopropane-1,3'-indoline]-1'-carboxylate (3i). Red oil (Method A:

51.7 mg, 74% yield, 89% *ee*). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.80 (d, *J* = 8.1 Hz, 1H), 7.10-7.05 (t, *J* = 7.6, 1H), 6.99 (comp, 4H), 6.70 (t, *J* = 7.6, 1H), 5.88 (d, *J* = 7.6 Hz, 1H), 3.28 (t, *J* = 8.7 Hz, 1H), 2.24 (s, 3H), 2.19-2.16 (m, 1H), 1.92-1.89 (m, 1H), 1.60 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 175.3, 149.4, 139.7, 137.4, 131.2, 130.0, 129.2, 126.7, 123.5, 120.5, 114.7, 84.3, 60.5, 38.1, 33.7, 28.2, 24.4, 21.2. HRMS (ESI) calculated for $C_{22}H_{23}NNaO_3$ $[M+Na]^+$: 372.1576, found 372.1573. HPLC analysis: Chiralpak IC, 254 nm, *i*PrOH/Hexanes = 3:97, 1.0 mL/min, R_t (major) = 10.7 min, R_t (minor) = 11.4 min.

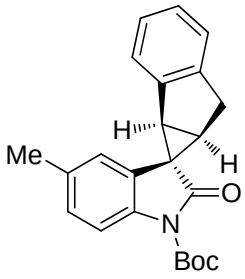


(*1S,1aR,6aR*)-*tert*-Butyl 2'-Oxo-6,6a-dihydro-1aH-spiro[cyclopropa[a]indene-1,3'-indoline]-1'-carboxylate (4a). Red solid (Method A: 54.1 mg, 78% yield, 94% *ee*; Method B: 51.3 mg, 74% yield, 98% *ee*). Mp 140-142 ^{o}C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.90 (d, *J* = 8.2 Hz, 1H), 7.34-7.26 (comp, 3H), 7.24-7.13 (m, 2H), 6.72 (t, *J* = 7.7 Hz, 1H), 5.65 (d, *J* = 7.7 Hz, 1H), 3.63-3.48 (m, 2H), 3.24 (d, *J* = 18.6 Hz, 1H), 2.99 (t, *J* = 6.5 Hz, 1H), 1.66 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 174.4, 149.4, 144.8, 140.6, 138.9, 128.0, 127.3, 127.0, 126.4, 125.0, 124.4, 123.8, 120.6, 114.9, 84.3, 44.8, 37.5, 36.0, 32.5, 28.3. HRMS (ESI) calculated for $C_{22}H_{21}NNaO_3$ $[M+Na]^+$: 370.1419, found 370.1432. HPLC analysis: Chiralpak IC, 2:98 *i*PrOH/Hexanes, 1.0 mL/min, R_t (major) = 21.5 min, R_t (minor) = 22.8 min; Chiralpak IC, 254 nm, *i*PrOH/Hexanes = 2:98, 1.0 mL/min, R_t (major) = 20.7 min, R_t (minor) = 22.1 min.



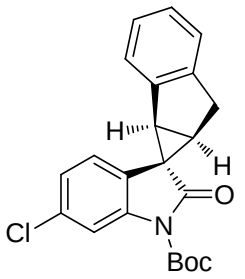
(*1S,1aR,6aR*)-*tert*-Butyl 5'-Fluoro-2'-oxo-6,6a-dihydro-1aH-spiro[cyclopropa[a]indene-1,3'-indoline]-1'-carboxylate

(4b). White solid (Method B: 45.3 mg, 62% yield, 93% *ee*). Mp 175-176 ^{o}C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.89-7.85 (m, 1H), 7.39-7.17 (comp, 4H), 6.87-6.82 (m, 1H), 5.36-5.33 (m, 1H), 3.67-3.51 (m, 2H), 3.22 (d, *J* = 18.7 Hz, 1H), 3.02 (t, *J* = 6.3 Hz, 1H), 1.65 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 174. 1, 159.4 (d, *J* = 239.5 Hz), 149.3, 144.4, 138.4, 136.6 (d, *J* = 2.2 Hz), 128.3, 127.6, 126.9 (d, *J* = 9.5 Hz), 126.4, 124.5, 115.8 (d, *J* = 8.3 Hz), 113.2, 108.4, 84.5, 45.0, 37.8, 36.1 (d, *J* = 2.1 Hz), 32.5, 28.2. HRMS (ESI) calculated for $C_{22}H_{20}FNNaO_3$ $[M+Na]^+$: 388.1325, found 388.1327. HPLC analysis: Chiralpak IC, 254 nm, *i*PrOH/Hexanes = 2:98, 1.0 mL/min, R_t (major) = 25.6 min, R_t (minor) = 27.8 min.



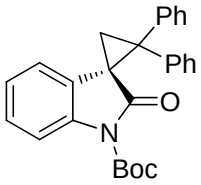
(*1S,1aR,6aR*)-*tert*-Butyl 5'-Methyl-2'-oxo-6,6a-dihydro-1aH-spiro[cyclopropa[a]indene-1,3'-indoline]-1'-carboxylate

(4c). Red solid (Method B: 62.1 mg, 86% yield, 99% *ee*). Mp 180-182 ^{o}C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.77 (d, *J* = 8.3 Hz, 1H), 7.37-7.27 (comp, 3H), 7.25-7.17 (m, 1H), 6.97 (d, *J* = 8.3 Hz, 1H), 5.43 (s, 1H), 3.65-3.49 (m, 2H), 3.25 (d, *J* = 18.5 Hz, 1H), 2.98 (t, *J* = 6.4 Hz, 1H), 1.99 (s, 3H), 1.66 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 174.6, 149.4, 144.9, 139.1, 138.2, 133.2, 127.9, 127.4, 127.3, 126.5, 125.0, 124.3, 121.5, 114.6, 84.2, 44.6, 37.3, 36.0, 32.5, 28.3, 21.2. HRMS (ESI) calculated for $C_{23}H_{23}NNaO_3$ $[M+Na]^+$: 384.1576, found 384.1582. HPLC analysis: Chiralpak IC, 254 nm, *i*PrOH/Hexanes = 2:98, 1.0 mL/min, R_t (major) = 37.7 min, R_t (minor) = 39.7 min.

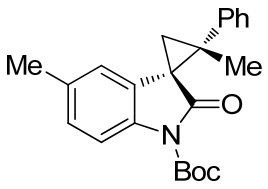


(*1S,1aR,6aR*)-*tert*-Butyl 6'-Chloro-2'-oxo-6,6a-dihydro-1aH-spiro[cyclopropa[a]indene-1,3'-indoline]-1'-carboxylate

(4d). White solid (Method B: 70.1 mg, 92% yield, 95% *ee*). Mp 178-180 ^{o}C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.97 (d, *J* = 2.0 Hz, 1H), 7.34-7.26 (comp, 3H), 7.25-7.17 (m, 1H), 6.71-6.69 (m, 1H), 5.52 (d, *J* = 8.3 Hz, 1H), 3.66-3.52 (m, 2H), 3.20 (d, *J* = 18.6 Hz, 1H), 3.00 (t, *J* = 6.4 Hz, 1H), 1.65 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 173.9, 149.1, 144.5, 141.4, 138.6, 134.0, 128.1, 127.5, 126.4, 124.4, 123.8, 123.4, 121.3, 115.6, 84.8, 44.9, 37.5, 35.8, 32.5, 28.2. HRMS (ESI) calculated for $C_{22}H_{20}ClNNaO_3$ $[M+Na]^+$: 404.1029, found 404.1028. HPLC analysis: Chiralpak IC, 254 nm, *i*PrOH/Hexanes = 2:98, 1.0 mL/min, R_t (major) = 25.0 min, R_t (minor) = 26.6 min.

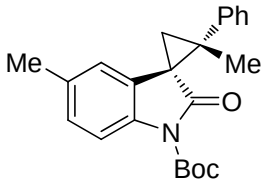


(*S*)-*tert*-Butyl 2'-Oxo-2,2-diphenylspiro[cyclopropane-1,3'-indoline]-1'-carboxylate (4e). White solid (Method A: 75.6 mg, 92% yield, 91% *ee*; Method B: 72.3 mg, 88% yield, 92% *ee*). Mp 179-181 ^{o}C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.87 (d, *J* = 8.1 Hz, 1H), 7.38-7.36 (m, 2H), 7.27-7.15 (comp, 9H), 6.76-6.72 (m, 1H), 5.67-5.64 (m, 1H), 2.70 (d, *J* = 4.7 Hz, 1H), 2.37 (d, *J* = 4.7 Hz, 1H), 1.60 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 172.2, 149.6, 140.2, 140.0, 139.8, 130.1, 129.2, 128.7, 128.6, 127.5, 127.2, 127.1, 127.0, 123.0, 122.3, 114.2, 84.0, 52.5, 37.9, 29.9, 28.3. HRMS (ESI) calculated for $C_{27}H_{25}NNaO_3$ $[M+Na]^+$: 434.1732, found 434.1747. HPLC analysis: Chiralpak IC, 254 nm, *i*PrOH/Hexanes = 2:98, 1.0 mL/min, R_t (major) = 24.3 min, R_t (minor) = 28.2 min.

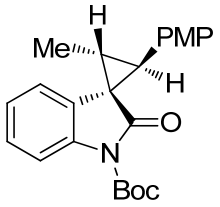


(*1S,2R*)-*tert*-Butyl 2,5'-Dimethyl-2'-oxo-2-phenylspiro[cyclopropane-1,3'-indoline]-1'-carboxylate (*trans*-4f). red oil (Method B: 35.7 mg, 49% yield, 95% *ee*) ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.66 (d, *J* = 8.3 Hz, 1H), 7.47-7.11 (comp, 5H), 6.89 (d, *J* = 8.3 Hz, 1H), 5.25 (s, 1H), 2.16 (d, *J* = 4.7 Hz, 1H), 2.08 (d, *J* = 4.7 Hz, 1H), 1.91 (s, 3H), 1.83 (s, 3H), 1.67 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 174.2, 149.6, 141.4, 136.8, 132.4, 128.5, 127.8, 127.3, 126.8, 122.3, 113.9, 84.0, 44.3, 36.8, 31.5, 28.3, 27.0, 21.2, 20.9. HRMS (ESI) calculated for $C_{23}H_{25}NNaO_3$ $[M+Na]^+$: 386.1732, found 386.1735.

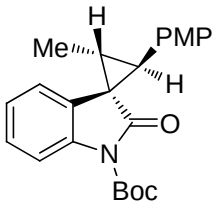
HPLC analysis: Chiralpak IC, 254 nm, *i*PrOH/Hexanes = 2:98, 1.0 mL/min, R_t (major) = 14.5 min, R_t (minor) = 16.3 min.



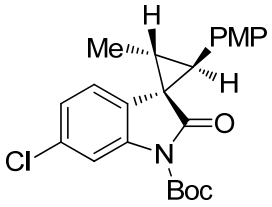
(*1R,2R*)-*tert*-Butyl 5'-methyl-2'-oxo-2-phenylspiro[cyclopropane-1,3'-indoline]-1'-carboxylate (*cis*-4f). red oil (Method B: 23.8 mg, 33% yield, 99% *ee*) ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.79 (d, *J* = 8.3 Hz, 1H), 7.29 (d, *J* = 7.3 Hz, 2H), 7.21 (comp, 3H), 7.11 (d, *J* = 8.3 Hz, 1H), 6.90 (s, 1H), 2.47 (d, *J* = 5.0 Hz, 1H), 2.39 (s, 3H), 1.80 (d, *J* = 5.0 Hz, 1H), 1.64 (s, 3H), 1.53 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) δ, 171.3, 149.6, 141.8, 138.2, 132.9, 128.7, 128.4, 127.6, 127.1, 127.0, 122.4, 114.5, 43.4, 38.7, 31.1, 28.2, 21.4, 21.2, 14.3. HRMS (ESI) calculated for $C_{23}H_{25}NNaO_3$ $[M+Na]^+$: 386.1732, found 386.1729. HPLC analysis: Chiralpak IC, 254 nm, *i*PrOH/Hexanes = 5:95, 1.0 mL/min, R_t (major) = 5.3 min, R_t (minor) = 6.7 min.



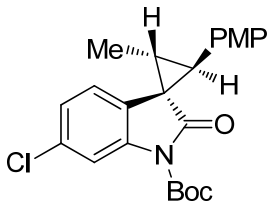
(*1S,2S,3R*)-*tert*-Butyl 2-(4-Methoxyphenyl)-3-methyl-2'-oxospiro[cyclopropane-1,3'-indoline]-1'-carboxylate (4g). Red oil (Method B: 31 mg, 46% yield, 98% *ee*) ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.89 (d, *J* = 8.1 Hz, 1H), 7.30 (m, 1H), 7.24-7.15 (comp, 3H), 7.06 (d, *J* = 7.4 Hz, 1H), 6.82 (d, *J* = 8.7 Hz, 2H), 3.78 (s, 3H), 2.94 (d, *J* = 8.9 Hz, 1H), 2.68-2.64 (m, 1H), 1.58 (s, 9H), 1.50 (d, *J* = 6.3 Hz, 3H); ^{13}C NMR (100 MHz, CDCl3) δ 172.0, 158.9, 149.5, 140.0, 130.4, 127.6, 126.8, 126.4, 123.7, 120.4, 115.0, 113.6, 84.1, 55.3, 46.7, 33.9, 31.1, 28.3, 12.7. HRMS (ESI) calculated for $C_{23}H_{25}NNaO_4$ $[M+Na]^+$: 402.1681, found 402.1674. HPLC analysis: Chiralpak IA, 254 nm, *i*PrOH/Hexanes = 3:97, 1.0 mL/min, R_t (major) = 9.8 min, R_t (minor) = 10.8 min.



(*1R,2S,3R*)-*tert*-Butyl 2-(4-Methoxyphenyl)-3-methyl-2'-oxospiro[cyclopropane-1,3'-indoline]-1'-carboxylate (4g'). Red oil (Method B: 31 mg, 46% yield, 87% *ee*) ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.83 (d, *J* = 8.1 Hz, 1H), 7.13 (d, *J* = 7.5 Hz, 1H), 7.06 (d, *J* = 8.4 Hz, 2H), 6.81-6.76 (comp, 3H), 5.95 (d, *J* = 7.6 Hz, 1H), 3.78 (s, 3H), 3.27 (d, *J* = 8.4 Hz, 1H), 2.36-2.28 (m, 1H), 1.68 (s, 9H), 1.63 (d, *J* = 6.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 173.6, 159.0, 149.6, 139.3, 131.1, 127.6, 127.0, 126.4, 123.5, 120.3, 114.6, 113.9, 84.3, 55.4, 45.1, 37.7, 35.3, 28.3, 11.5. HRMS (ESI) calculated for $C_{23}H_{25}NNaO_4$ $[M+Na]^+$: 402.1681, found 402.1670. HPLC analysis: Chiralpak IE, 254 nm, *i*PrOH/Hexanes = 5:95, 1.0 mL/min, R_t (major) = 9.7 min, R_t (minor) = 12.7 min.

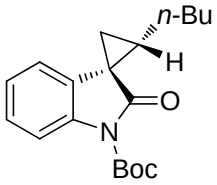


(*1S,2S,3R*)-*tert*-Butyl 6'-Chloro-2-(4-methoxyphenyl)-3-methyl-2'-oxospiro[cyclopropane-1,3'-indoline]-1'-carboxylate (4h). (Method B: 29.2 mg, 40% yield, 95% *ee*) ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.97 (d, *J* = 1.9 Hz, 1H), 7.22-7.14 (comp, 3H), 6.96 (d, *J* = 8.1 Hz, 1H), 6.84-6.80 (m, 2H), 3.78 (s, 3H), 2.93 (d, *J* = 8.9 Hz, 1H), 2.67 (m, 1H), 1.58 (s, 9H), 1.48 (d, *J* = 6.3 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 171.5, 159.0, 149.2, 140.8, 132.6, 130.3, 126.0, 125.9, 123.7, 121.1, 115.8, 113.7, 84.6, 55.3, 47.0, 39.1, 34.1, 28.2, 12.8. HRMS (ESI) calculated for $C_{23}H_{24}ClNNaO_4$ $[M+Na]^+$: 436.1292, found 436.1278. HPLC analysis: Chiralpak IA, 254 nm, *i*PrOH/Hexanes = 3:97, 1.0 mL/min, R_t (major) = 9.4 min, R_t (minor) = 10.4 min.

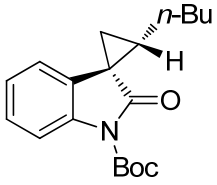


(*1R,2S,3R*)-*tert*-Butyl

6'-Chloro-2-(4-methoxyphenyl)-3-methyl-2'-oxospiro[cyclopropane-1,3'-indoline]-1'-carboxylate (4h'). Red oil (Method B: 43.9 mg, 59% yield, 78% *ee*) ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.91 (d, *J* = 1.9 Hz, 1H), 7.04 (d, *J* = 8.3 Hz, 2H), 6.84-6.74 (m, 3H), 5.83 (d, *J* = 8.2 Hz, 1H), 3.78 (s, 3H), 3.27 (d, *J* = 8.5 Hz, 1H), 2.35-2.27 (m, 1H), 1.68 (s, 9H), 1.63 (d, *J* = 6.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 173.1, 159.1, 149.4, 140.1, 132.3, 131.1, 126.6, 126.0, 123.5, 121.0, 115.3, 114.0, 84.7, 55.4, 45.3, 37.5, 35.4, 28.3, 11.5. HRMS (ESI) calculated for $C_{23}H_{24}ClNNaO_4$ $[M+Na]^+$: 436.1292, found 436.1282. HPLC analysis: Chiralpak IA, 254 nm, *i*PrOH/Hexanes = 1:99, 1.0 mL/min, R_t (major) = 28.8 min, R_t (minor) = 32.6 min.

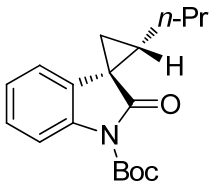


(*1S,2S*)-*tert*-butyl 2-Butyl-2'-oxospiro[cyclopropane-1,3'-indoline]-1'-carboxylate（*trans*-4i）. White oil (Method A: 48.2 mg, 77% yield, 93% *ee*). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.87 (d, *J* = 8.1 Hz, 1H), 7.28-7.19 (m, 1H), 7.05 (d, *J* = 7.6, 1H), 6.89-6.85 (m, 1H), 1.97-1.87 (m, 2H), 1.58 (s, 9H), 1.32-1.15 (comp, 7H), 0.76 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 176.0, 149.6, 140.0, 127.7, 126.9, 123.8, 120.4, 115.1, 84.3, 36.1, 32.4, 31.3, 28.3, 27.6, 26.4, 22.4, 14.0. HRMS (ESI) calculated for $C_{19}H_{25}NO_3$ $[M+H]^+$: 316.1913, found 316.1903. HPLC analysis: Chiralpak IC, 272 nm, *i*PrOH/Hexanes = 2:98, 1.0 mL/min, R_t (major) = 11.0 min, R_t (minor) = 11.8 min.

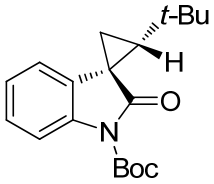


(*1R,2S*)-*tert*-Butyl 2-Butyl-2'-oxospiro[cyclopropane-1,3'-indoline]-1'-carboxylate (*cis*-4i). White oil (Method A: 75% *ee*) ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.84 (d, *J* = 8.1 Hz, 1H), 7.23 (m, 1H), 7.13-7.09 (m, 1H), 6.77 (d, *J* = 7.4 Hz, 1H), 1.89-1.79 (m, 3H), 1.65 (s, 9H), 1.42-1.24 (comp, 6H), 0.88 (m, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 174.2, 149.6, 138.9, 131.0, 126.7, 124.1, 117.6, 114.8, 84.1, 37.5, 31.8, 31.1, 28.3, 27.7, 25.7, 22.3, 14.2. HRMS (ESI) calculated for $C_{19}H_{25}NO_3$ $[M+H]^+$: 316.1913, found 316.1906. HPLC analysis: Chiralpak IC, 272 nm, *i*PrOH/Hexanes = 5:95, 1.0 mL/min, R_t

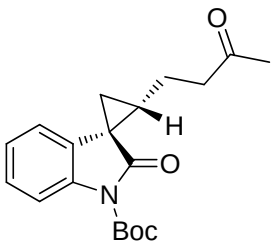
(major) = 6.6 min, R_t (minor) = 7.1 min.



(*1S,2S*)-*tert*-Butyl 2'-Oxo-2-propylspiro[cyclopropane-1,3'-indoline]-1'-carboxylate (4j). White oil (Method A: 47.0 mg, 78% yield, 94%) ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.94 (d, *J* = 8.2 Hz, 1H), 7.31-7.25 (m, 1H), 7.14-7.12 (m, 1H), 6.94 (d, *J* = 7.5 Hz, 1H), 2.06-1.93 (m, 2H), 1.65 (s, 9H), 1.46-1.22 (m, 5H), 0.86 (t, *J* = 7.4 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 175.9, 149.5, 140.0, 127.7, 126.8, 123.7, 120.4, 115.1, 84.2, 35.8, 32.2, 28.2, 27.5, 26.3, 22.4, 13.8. HRMS (ESI) calculated for $C_{18}H_{23}NNaO_3$ $[M+Na]^+$: 324.1576, found 324.1563. HPLC analysis: Chiralpak IC, 272 nm, *i*PrOH/Hexanes = 3:97, 1.0 mL/min, R_t (major/cis) = 7.1 min, R_t (minor/cis) = 8.1 min; R_t (major/trans) = 11.1 min, R_t (minor/trans) = 12.4 min.



(*1S,2R*)-*tert*-Butyl 2-(*tert*-Butyl)-2'-oxospiro[cyclopropane-1,3'-indoline]-1'-carboxylate (4k). White oil (Method A: 27.7 mg, 44% yield, 96% *ee*) ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.93 (d, *J* = 8.2 Hz, 1H), 7.26-7.18 (m, 2H), 7.10-7.06 (m, 1H), 2.02-1.90 (m, 2H), 1.77-1.73 (m 1H), 1.65 (s, 9H), 1.04 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 176.5, 149.5, 140.3, 127.7, 126.7, 123.3, 122.7, 115.1, 84.3, 50.7, 33.4, 32.0, 30.0, 28.3, 22.6. HRMS (ESI) calculated for $C_{19}H_{25}NNaO_3$ $[M+Na]^+$: 338.1732, found 338.1717. HPLC analysis: Chiralpak IB, 272 nm, *i*PrOH/Hexanes = 2:98, 1.0 mL/min, R_t (major) = 4.2 min, R_t (minor) = 4.8 min.



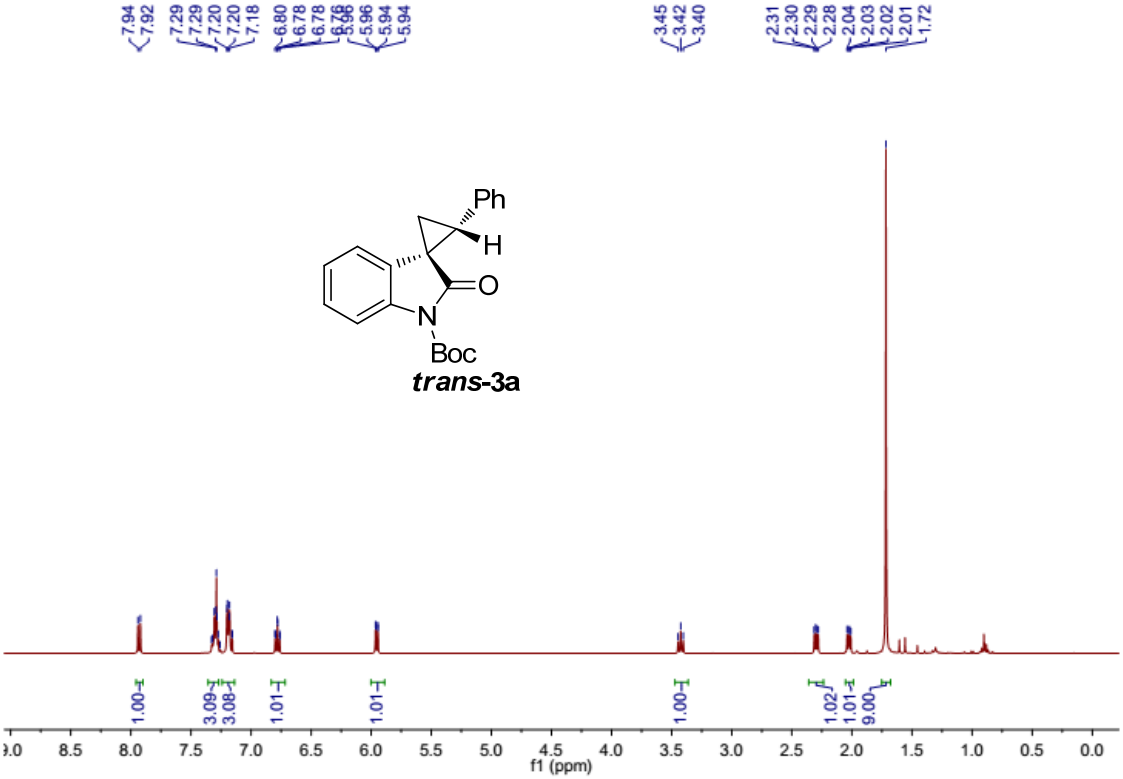
(*1S,2S*)-*tert*-Butyl

2'-Oxo-2-(3-oxobutyl)spiro[cyclopropane-1,3'-indoline]-1'-carboxylate (4l). White oil (Method A: 48.7 mg, 74% yield, 90% *ee*). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.94 (d, *J* = 8.1 Hz, 1H), 7.32-7.27 (m, 1H), 7.15-7.11 (m, 1H), 6.98 (d, *J* = 7.5 Hz, 1H), 2.54-2.44 (m, 1H), 2.36-2.28 (m, 1H), 2.16-1.85 (comp, 3H), 2.12 (s, 3H), 1.81-1.74 (m, 1H), 1.66 (s, 9H), 1.44-1.37 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 207.6, 175.6, 149.5, 140.0, 127.1, 127.0, 124.0, 120.4, 115.2, 84.4, 42.7, 34.7, 32.2, 30.1, 28.2, 26.0, 21.9. HRMS (ESI) calculated for $C_{19}H_{23}NNaO_4$ $[M+Na]^+$: 352.1525, found 352.1530. HPLC analysis: Chiralpak IB, 272 nm, *i*PrOH/Hexanes = 5:95, 1.0 mL/min, R_t (major) = 12.8 min, R_t (minor) = 21.4min.

Reference:

1 Z. Cao, F. Zhou, Y. Yu and J. Zhou, *Org. Lett.*, 2013, **15**, 42-45.

2 (a) S. Hashimoto, N. Watanabe, T. Sato, M. Shiro, S. Ikegami, *Tetrahedron lett.*, 1993, **34**, 5109-5112. (b) H. Tsutsui, T. Abe, S. Nakamura, M. Anada, S. Hashimoto, *Chem. Pharm. Bull.*, 2005, **53**, 1366-1368. (c) C. Liang, F. Collet, F. Robert-Peillard, P. Müller, R. H. Dodd, P. Dauban, *J. Am. Chem. Soc.*, 2008, **130**, 343-350. (d) P. Müller, Y. Allenbach, E. Robert, *Tetrahedron: Asymmetry*, 2003, **14**, 779-785.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7.20
7.18
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6.78
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5.94
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3.45
3.42
3.40
2.31
2.30
2.29
2.28
2.04
2.03
2.02
2.01
1.72
Ph
H
O
N
Boc
trans-3a
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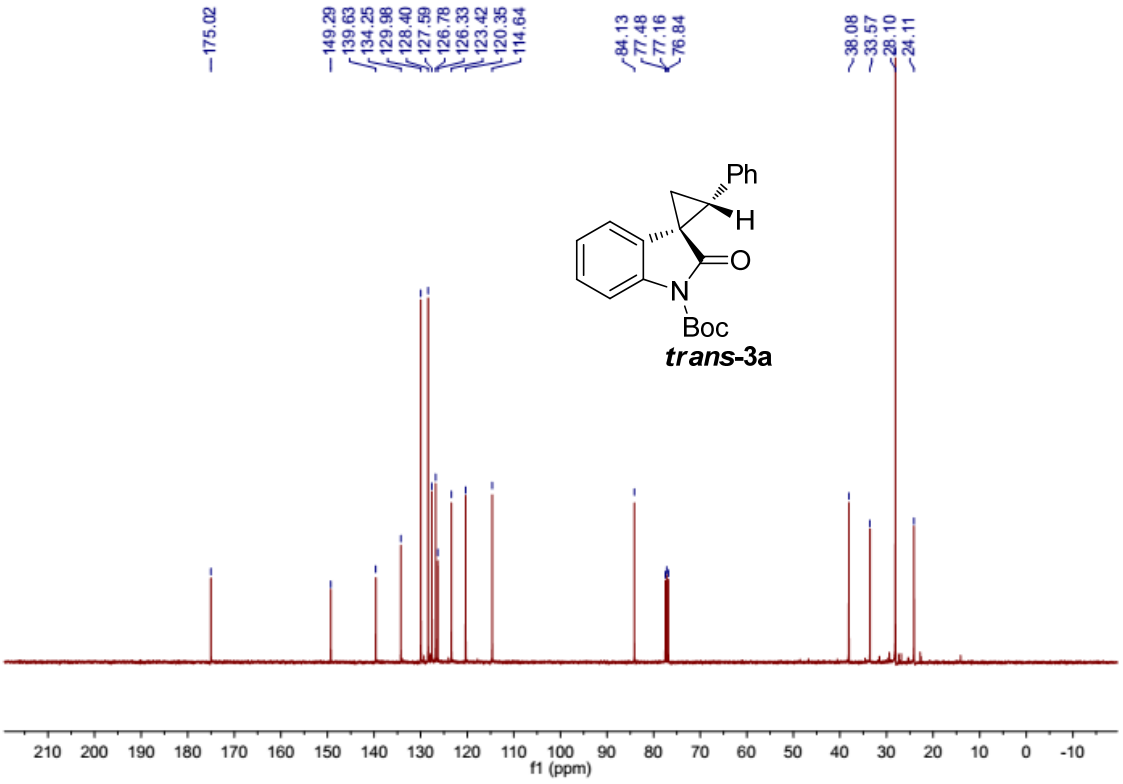
3.09
3.08
1.01
1.01
1.00
1.02
1.01
9.00
f1 (ppm)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128.40
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126.33
123.42
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114.64
84.13
77.48
77.16
76.84
38.08
33.57
28.10
24.11
Ph
H
O
N
Boc
trans-3a
f1 (ppm)


7.87
7.84
7.32
7.31
7.30
7.29
7.29
7.27
7.26
7.20
7.18
7.16
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2.44
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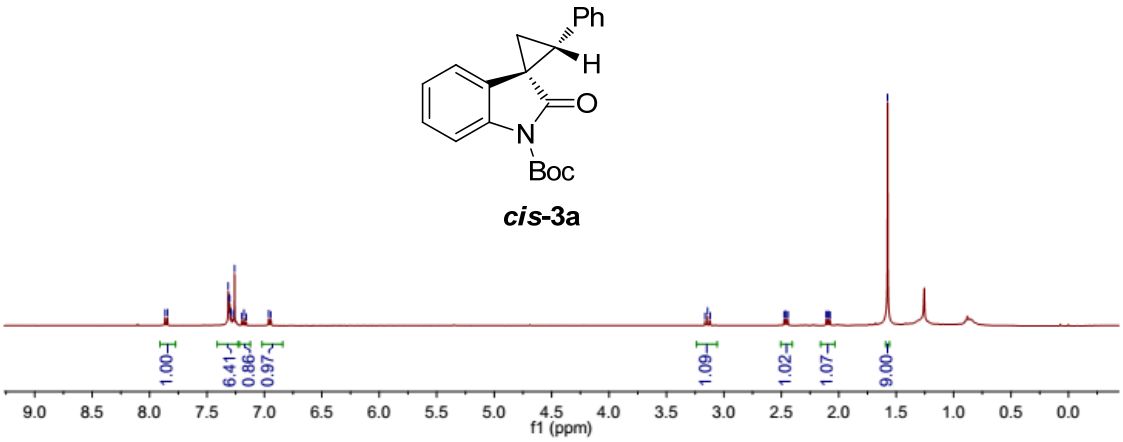
2.10
2.09
1.57


Ph
H
O
N
Boc
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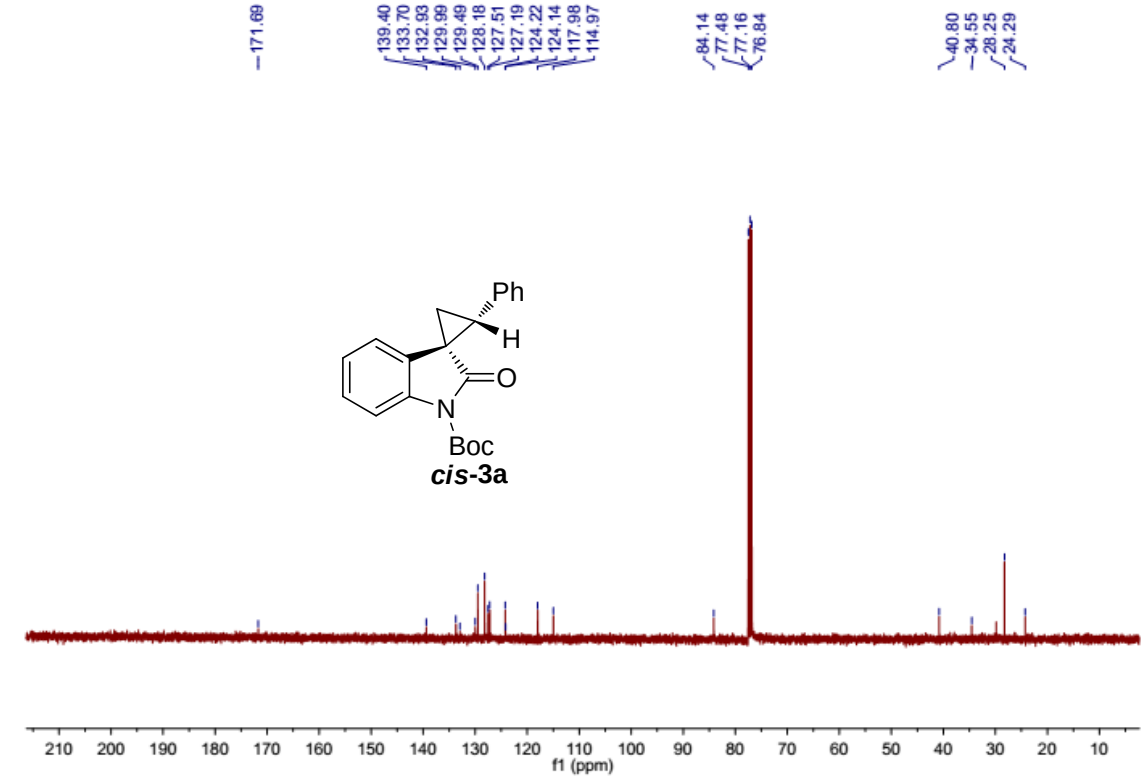
0.97
1.09
1.02
1.07
9.00
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

***cis*-3a**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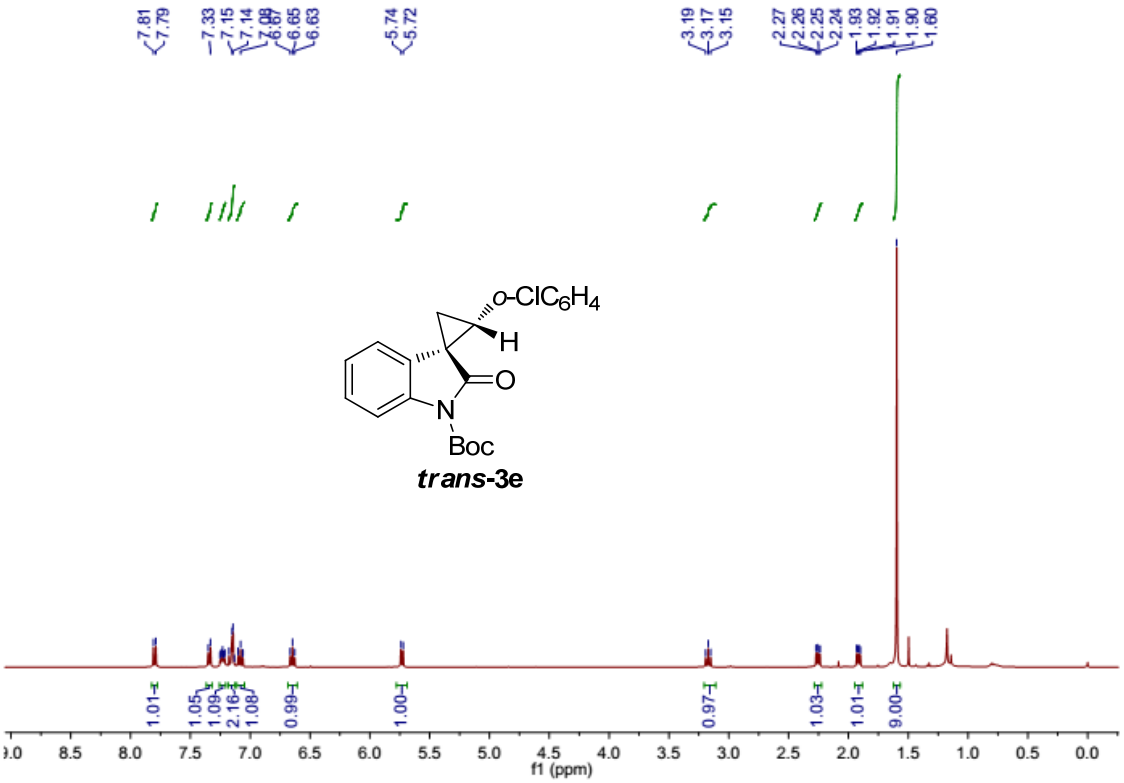
114.97
84.14
77.48
77.16
76.84
40.80
34.55
28.25
24.29
Ph
H
O
N
Boc
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10
f1 (ppm)

***cis*-3a**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O
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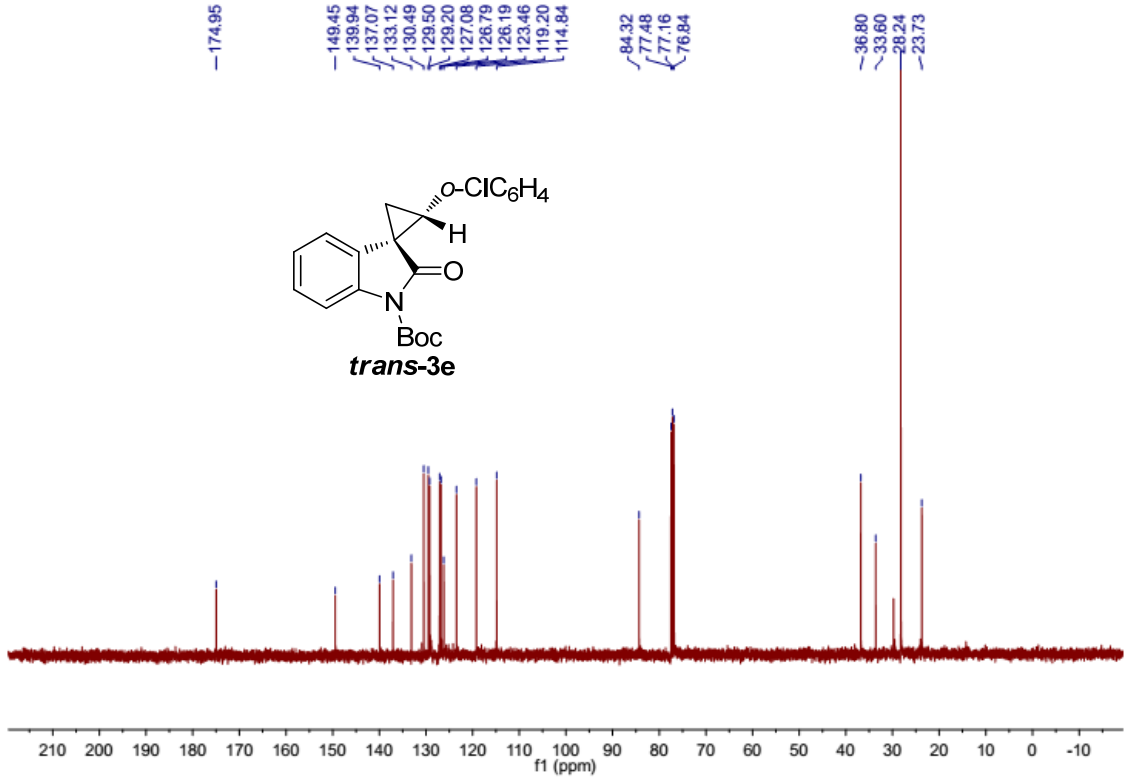
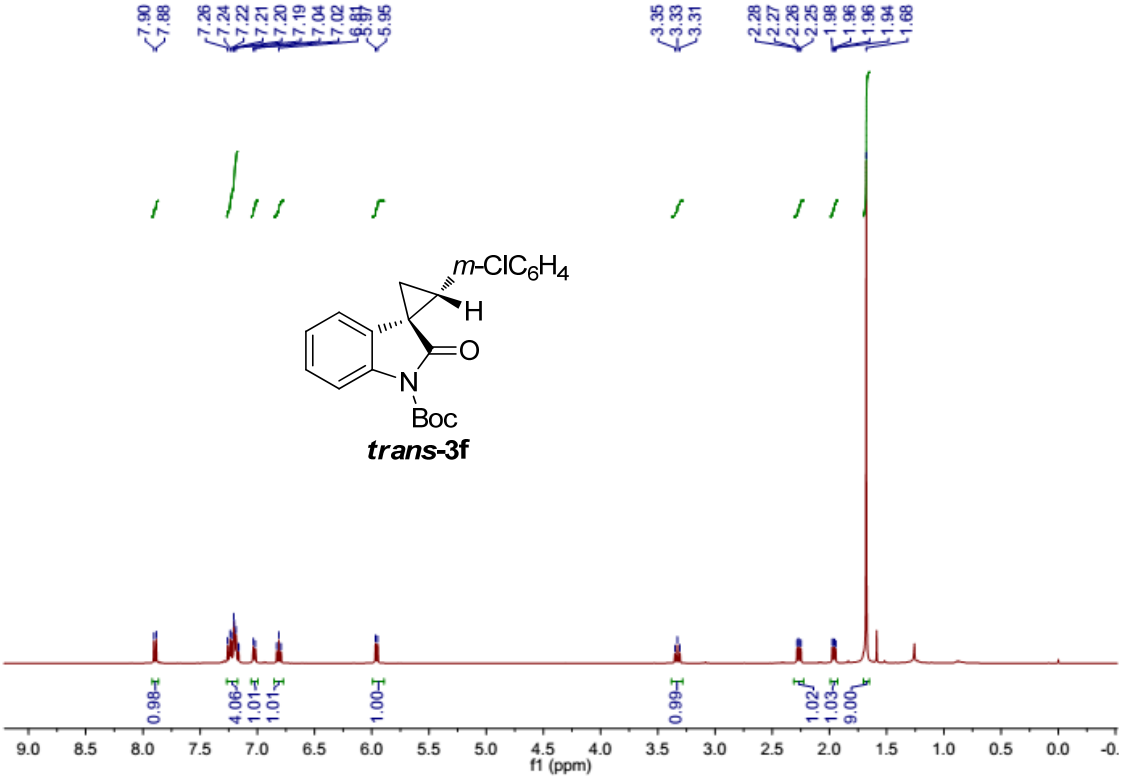
f1 (ppm)


o-ClC6H4
H
O
N
Boc
trans-3e
f1 (ppm)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7.24
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7.20
7.19
7.04
7.02
6.81
5.97
5.95
3.35
3.33
3.31
2.28
2.27
2.26
2.25
1.98
1.96
1.96
1.94
1.68
m-ClC6H4
H
O
N
Boc
trans-3f
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4.06
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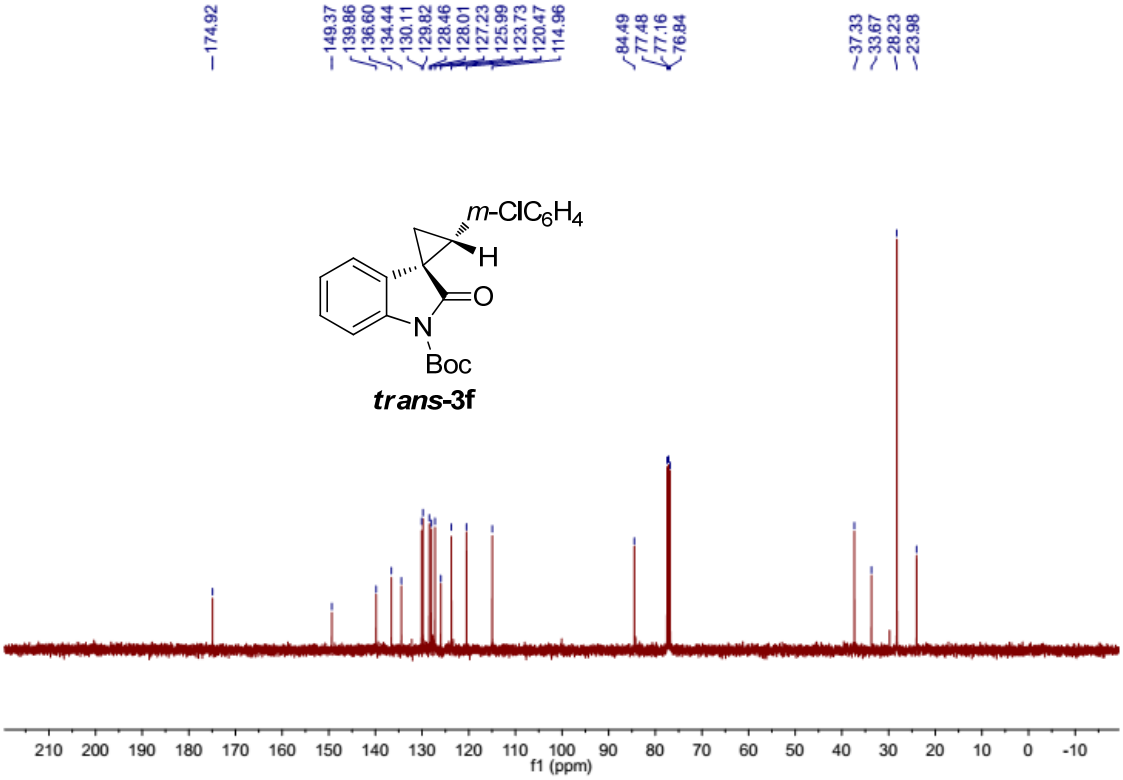
1.01
1.00
0.99
1.02
1.03
9.00
f1 (ppm)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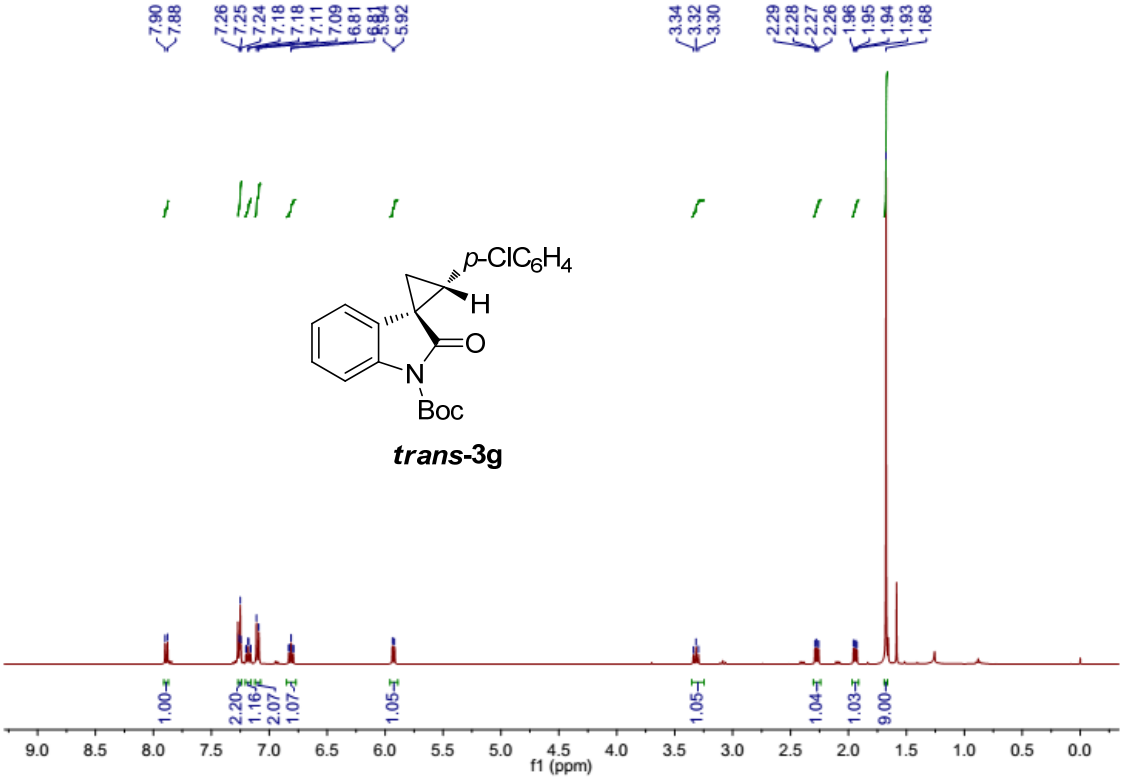
128.01
127.23
125.99
123.73
120.47
114.96
84.49
77.48
77.16
76.84
37.33
33.67
28.23
23.98
m-ClC6H4
H
O
N
Boc
trans-3f
f1 (ppm)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7.18
7.11
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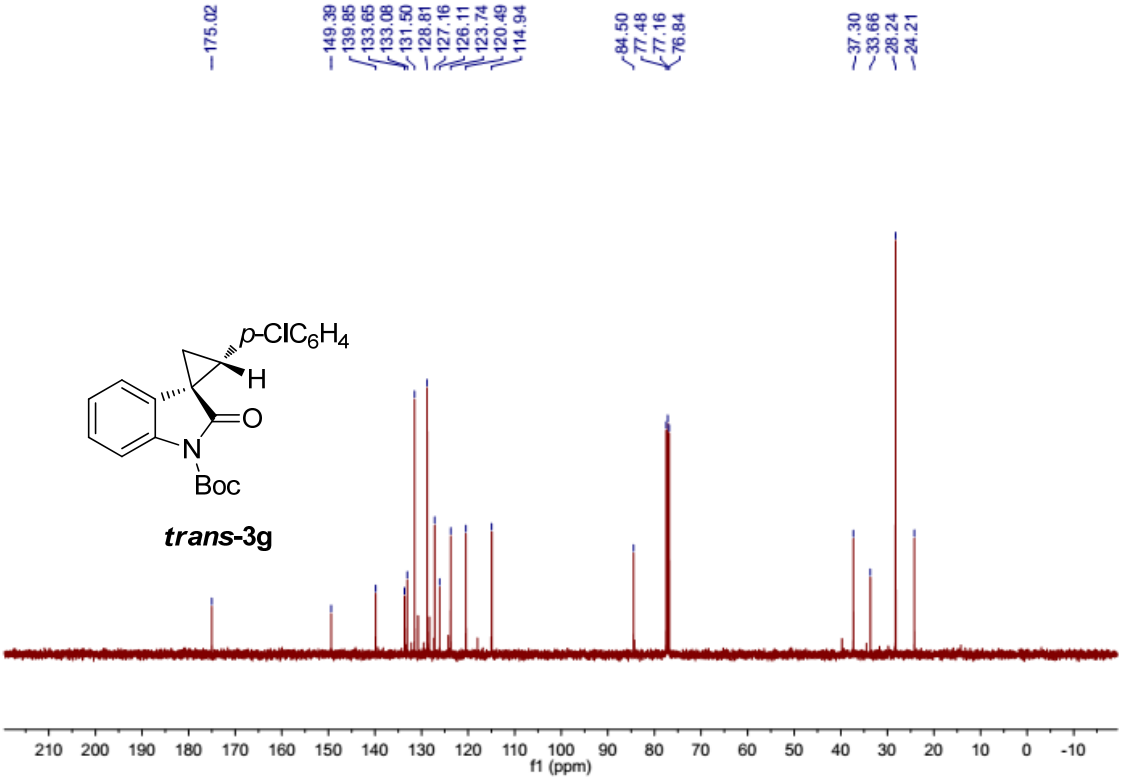
6.81
6.81
5.94
5.92
3.34
3.32
3.30
2.29
2.28
2.27
2.26
1.96
1.95
1.94
1.93
1.68
p-ClC6H4
H
O
N
Boc
trans-3g
1.00
2.20
1.16
2.07
1.07
1.05
1.05
1.04
1.03
9.00
f1 (ppm)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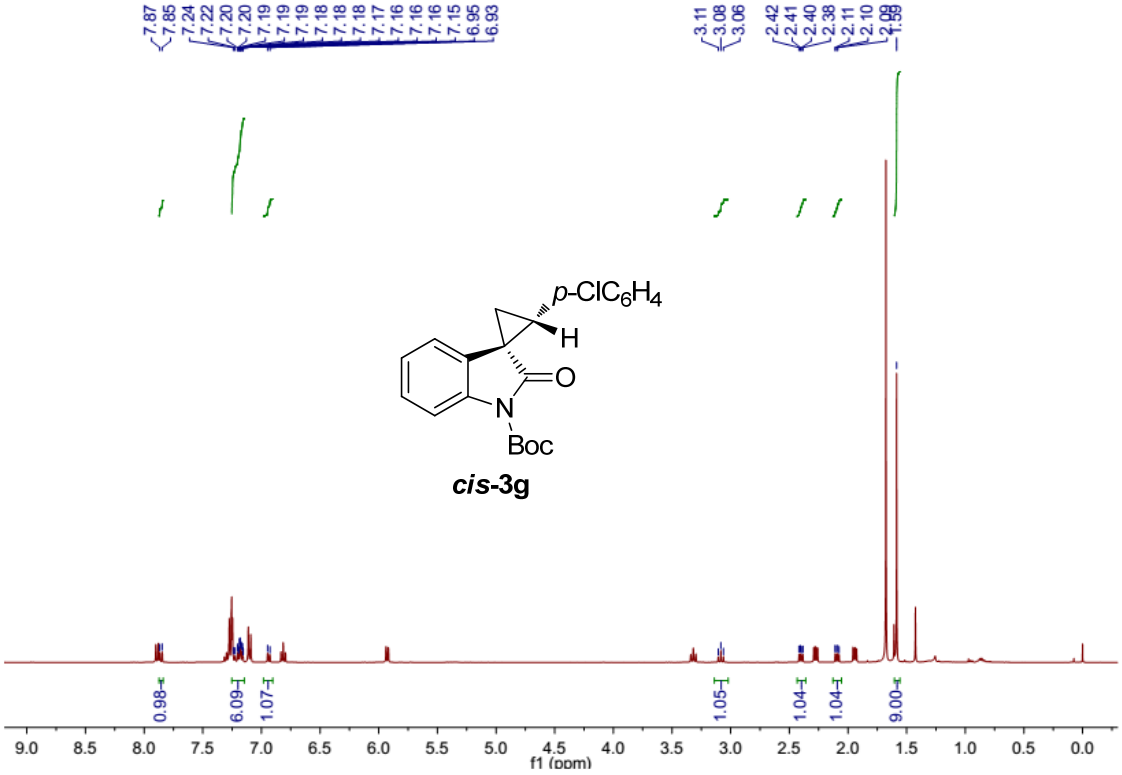
128.81
127.16
126.11
123.74
120.49
114.94
84.50
77.48
77.16
76.84
37.30
33.66
28.24
24.21
p-ClC6H4
H
O
N
Boc
trans-3g
f1 (ppm)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7.19
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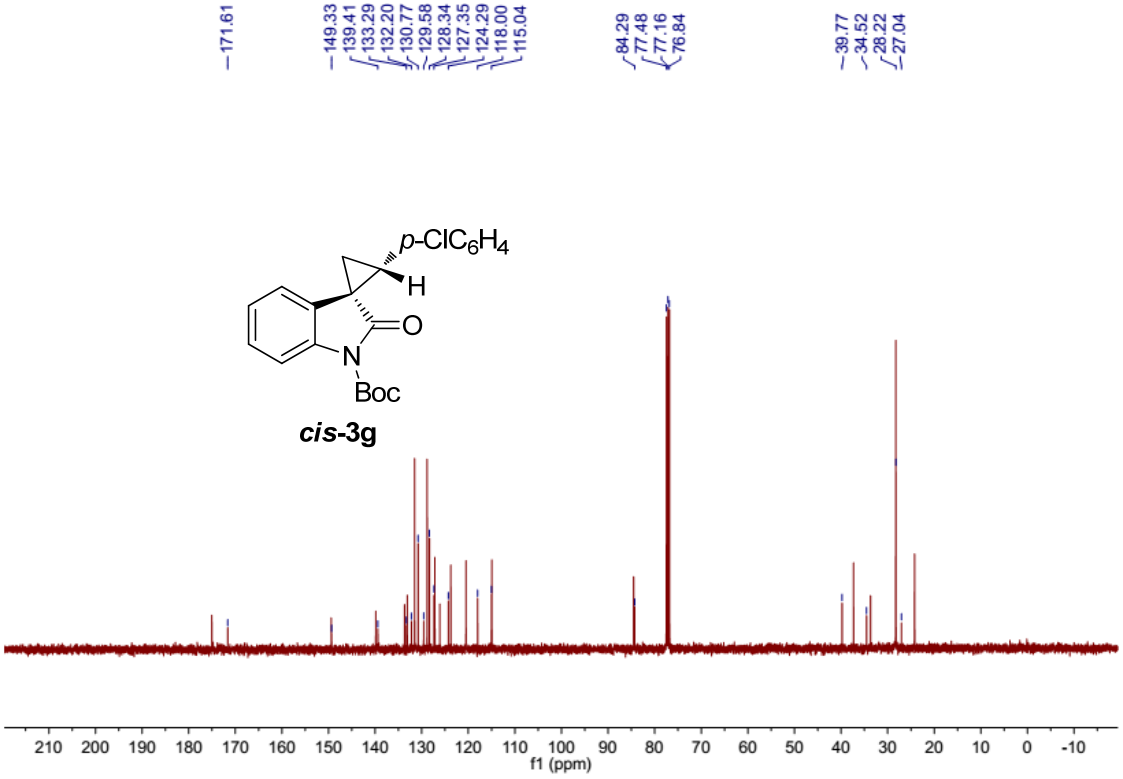
7.18
7.18
7.18
7.17
7.16
7.16
7.16
7.15
6.95
6.93
3.11
3.08
3.06
2.42
2.41
2.40
2.38
2.11
2.10
2.09
1.99
p-ClC6H4
H
O
N
Boc
0.98
6.09
1.07
1.05
1.04
1.04
9.00
f1 (ppm)

***cis*-3g**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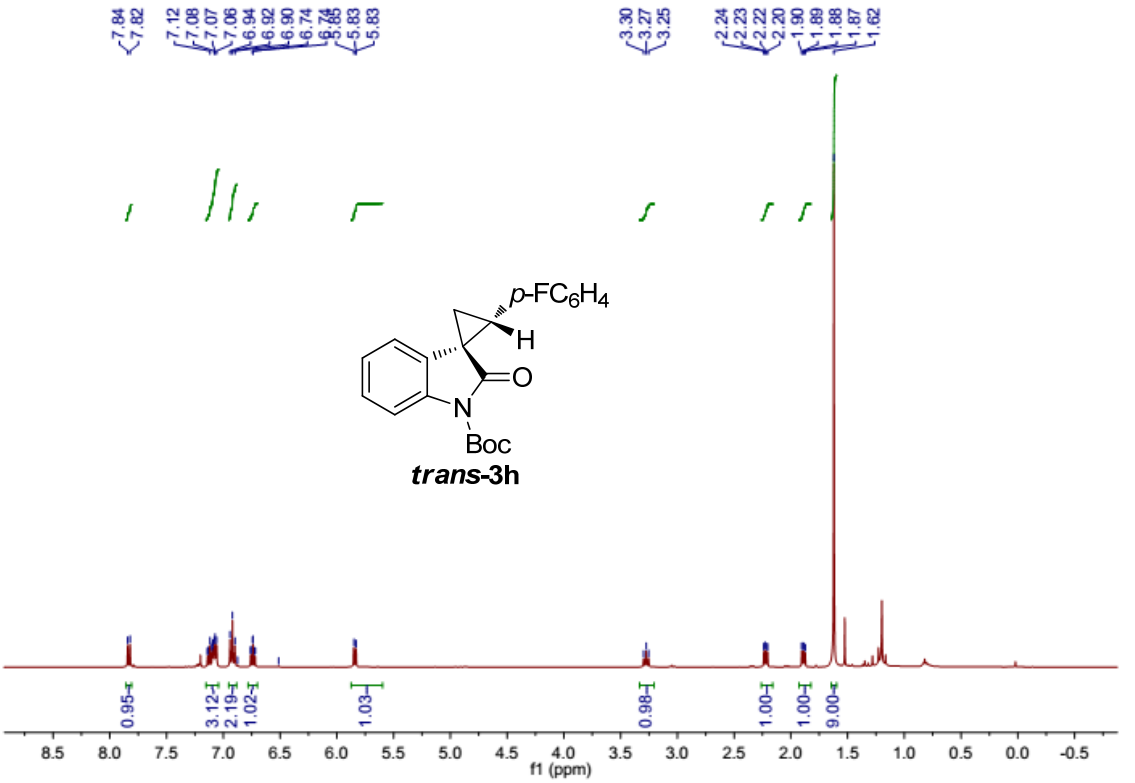
130.77
129.58
128.34
127.35
124.29
118.00
115.04
84.29
77.48
77.16
76.84
39.77
34.52
28.22
27.04
p-ClC6H4
H
O
N
Boc
f1 (ppm)

***cis*-3g**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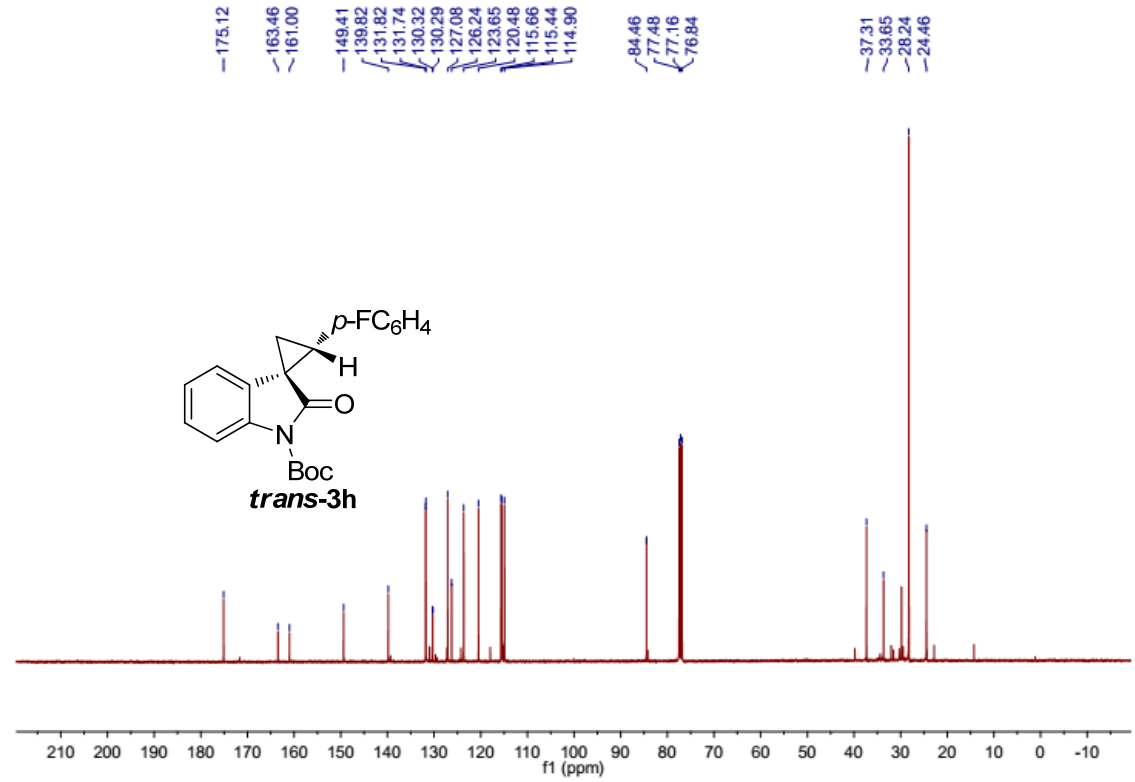
7.82
7.12
7.08
7.07
7.06
6.94
6.92
6.90
6.74
5.83
5.83
3.30
3.27
3.25
2.24
2.23
2.22
2.20
1.90
1.89
1.88
1.87
1.62
p-FC6H4
H
O
N
Boc
trans-3h
0.95
3.12
2.19
1.02
1.03
0.98
1.00
1.00
9.00
f1 (ppm)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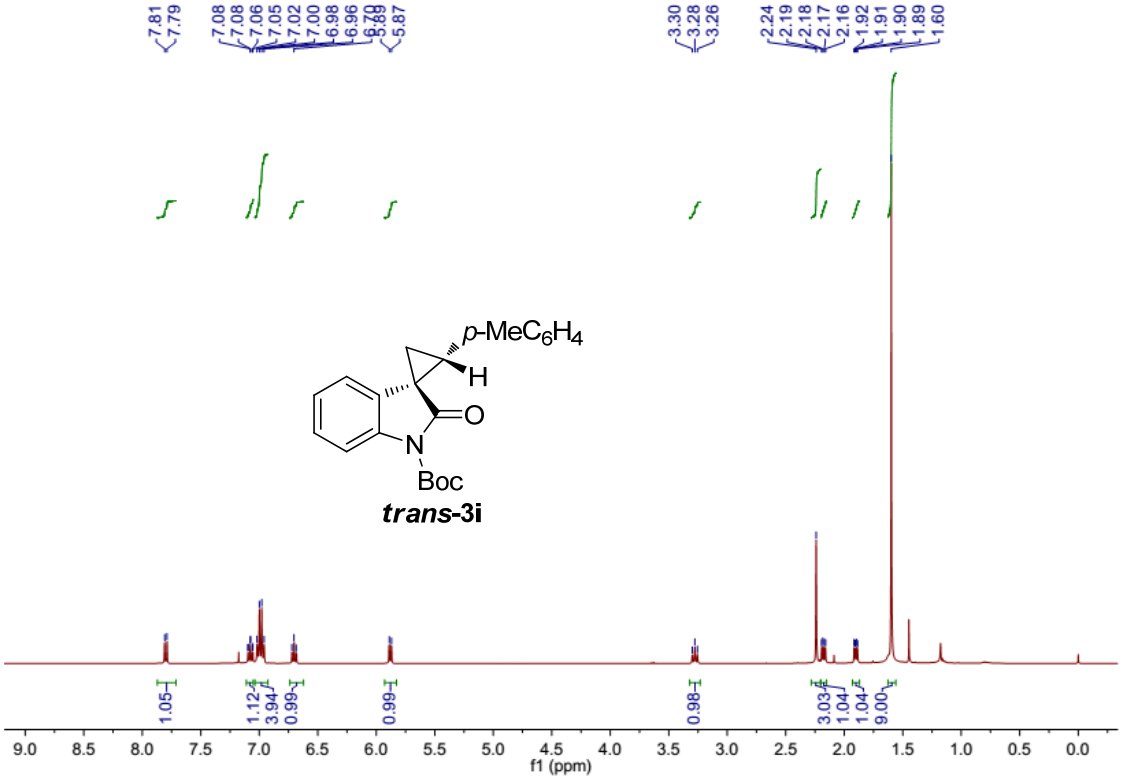
131.82
131.74
130.32
130.29
127.08
126.24
123.65
120.48
115.66
115.44
114.90
84.46
77.48
77.16
76.84
37.31
33.65
28.24
24.46
p-FC6H4
H
O
N
Boc
trans-3h
f1 (ppm)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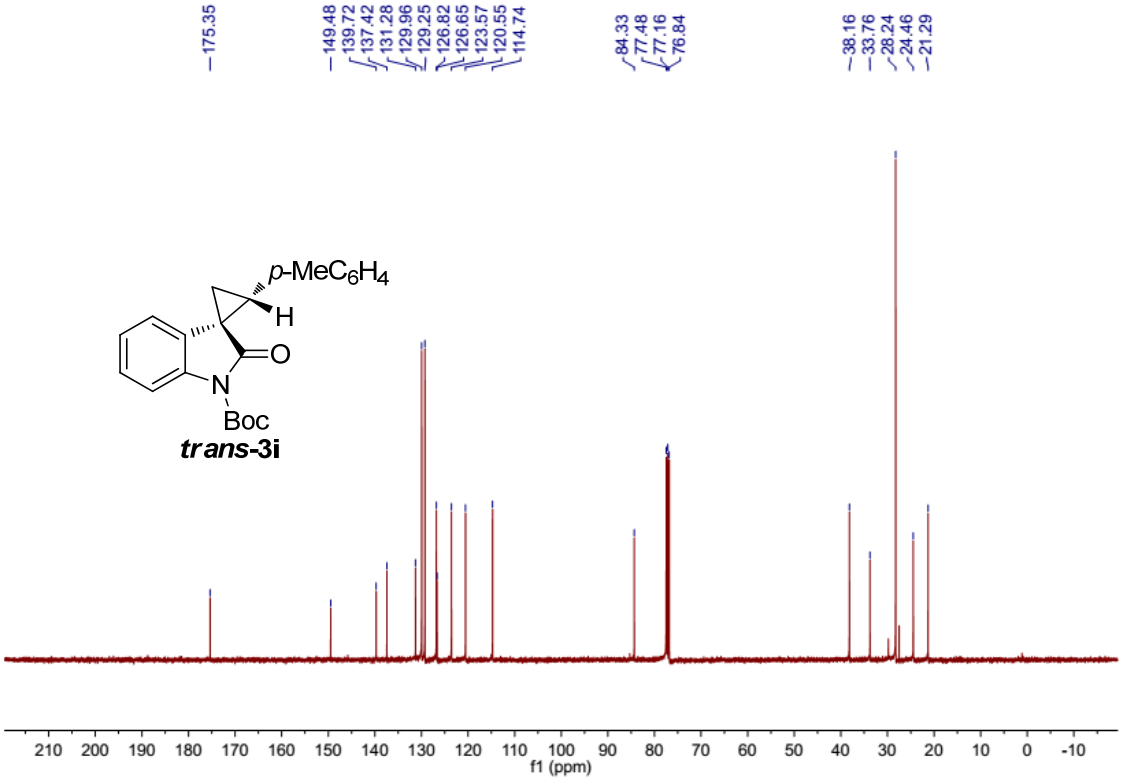
7.79
7.08
7.08
7.06
7.05
7.02
7.00
6.98
6.96
6.70
5.89
5.87
3.30
3.28
3.26
2.24
2.19
2.18
2.17
2.16
1.92
1.91
1.90
1.89
1.60
p-MeC6H4
H
O
N
Boc
trans-3i
1.05
1.12
3.94
0.99
0.99
0.98
3.03
1.04
1.04
9.00
f1 (ppm)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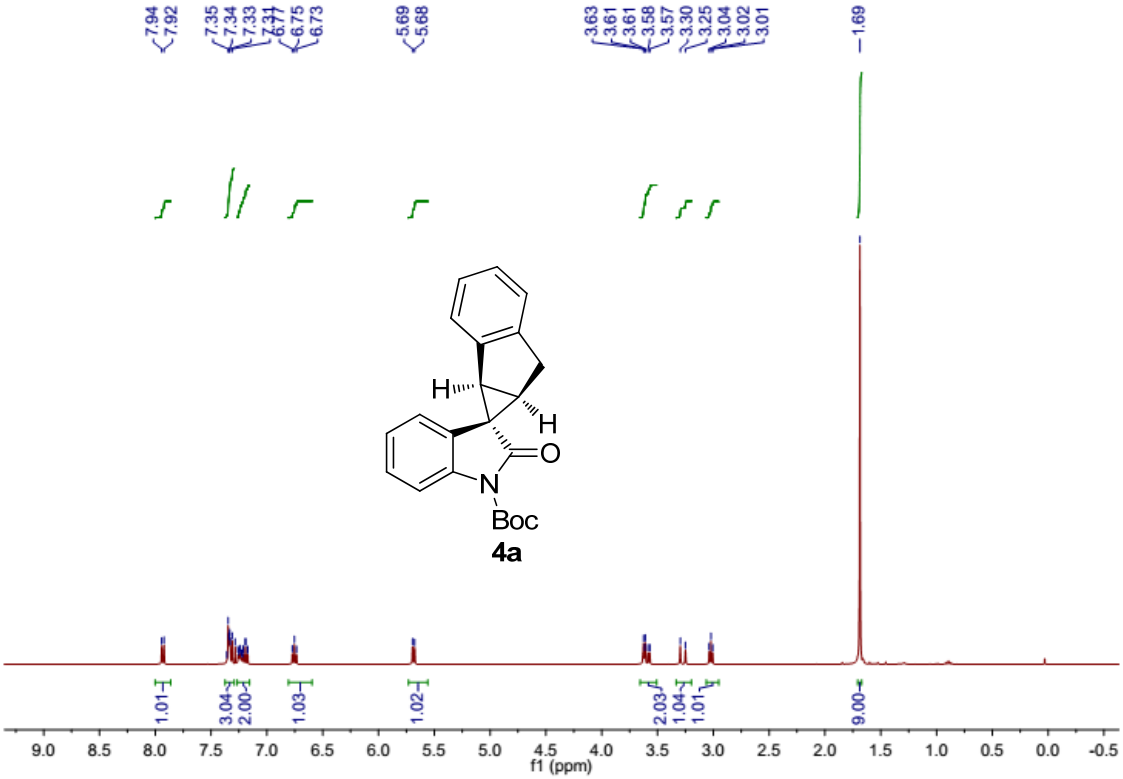
129.25
126.82
126.65
123.57
120.55
114.74
84.33
77.48
77.16
76.84
38.16
33.76
28.24
24.46
21.29
p-MeC6H4
H
O
N
Boc
trans-3i
f1 (ppm)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7.31
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6.75
6.73
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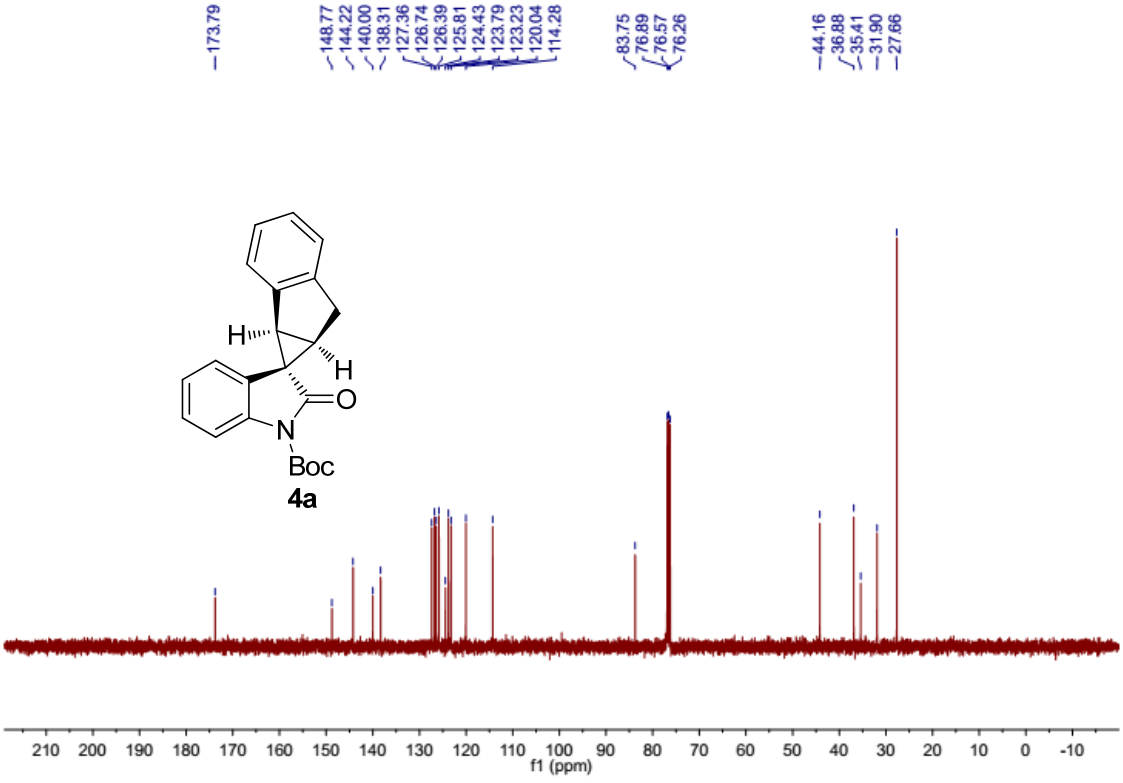
5.68
3.63
3.61
3.61
3.58
3.57
3.30
3.25
3.04
3.02
3.01
1.69
H
H
O
N
Boc
4a
1.01
3.04
2.00
1.03
1.02
2.03
1.04
1.01
9.00
f1 (ppm)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124.43
123.79
123.23
120.04
114.28
83.75
76.89
76.57
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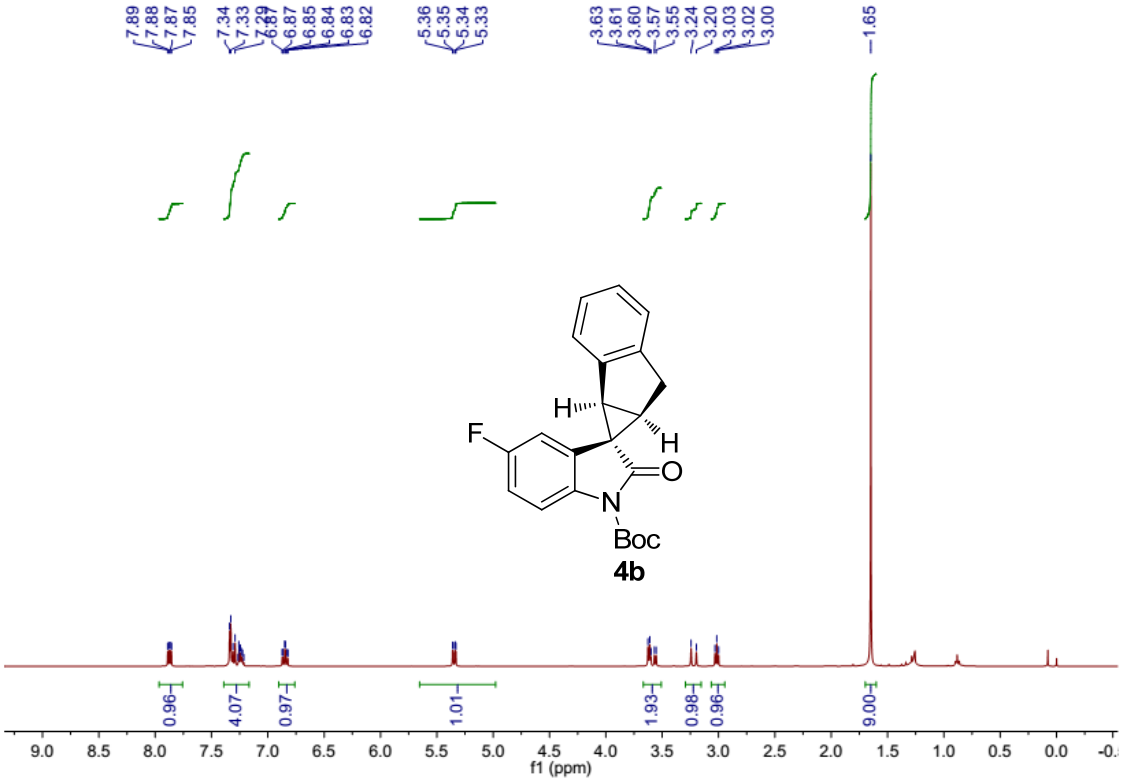
36.88
35.41
31.90
27.66
H
H
O
N
Boc
4a
f1 (ppm)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7.29
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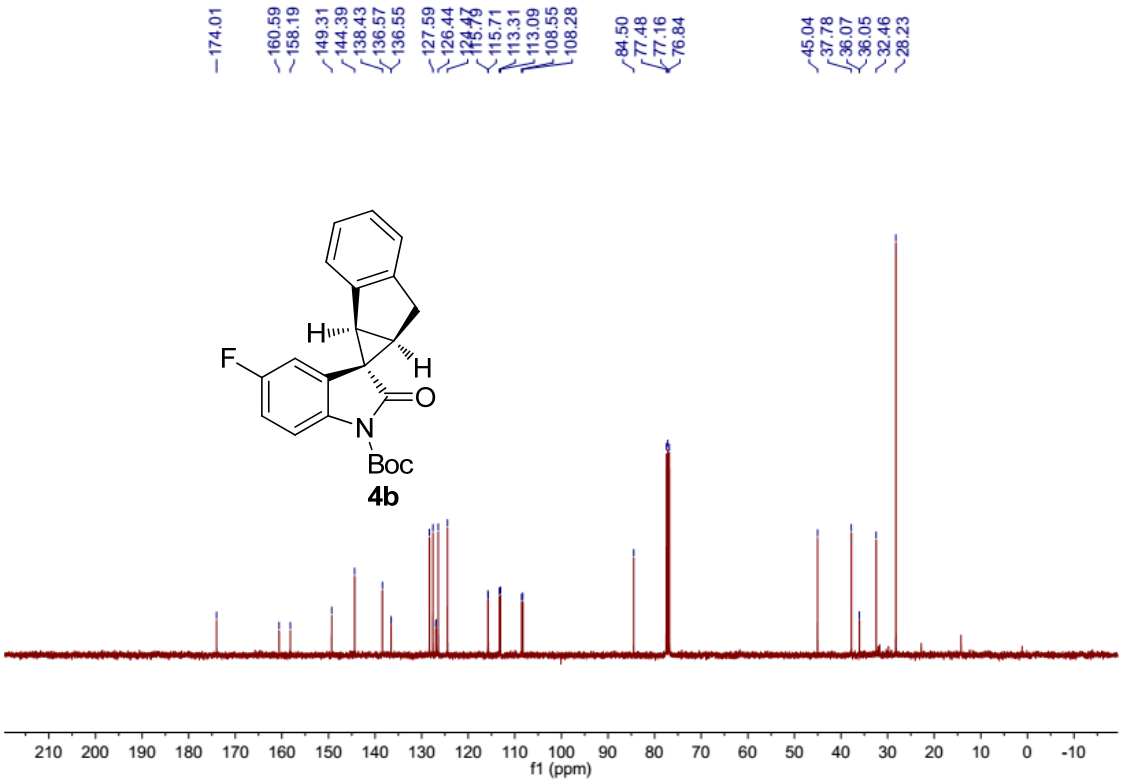
6.85
6.84
6.83
6.82
5.36
5.35
5.34
5.33
3.63
3.61
3.60
3.57
3.55
3.24
3.20
3.03
3.02
3.00
1.65
H
F
H
O
N
Boc
4b
0.96
4.07
0.97
1.01
1.93
0.98
0.96
9.00
f1 (ppm)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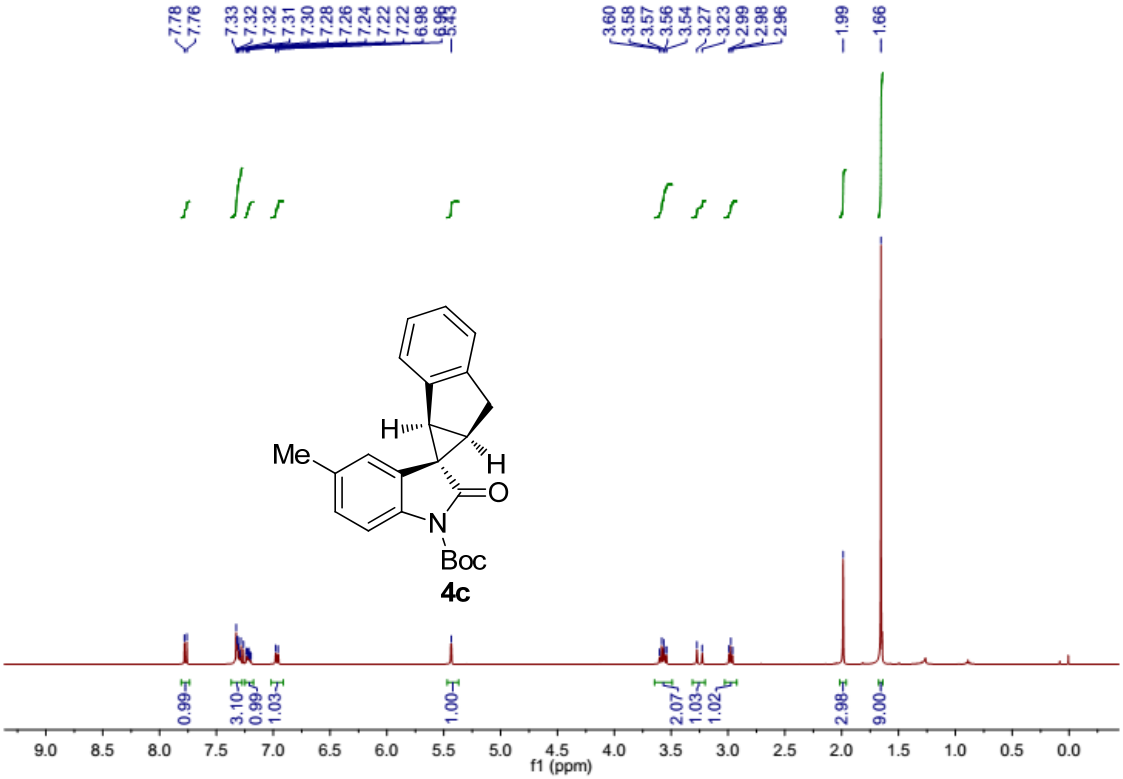
136.57
136.55
127.59
126.44
115.71
113.31
113.09
108.55
108.28
84.50
77.48
77.16
76.84
45.04
37.78
36.07
36.05
32.46
28.23
H
F
H
O
N
Boc
4b
f1 (ppm)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7.22
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6.96
5.43
3.60
3.58
3.57
3.56
3.54
3.27
3.23
2.99
2.98
2.96
1.99
1.66
Me
H
H
O
N
Boc
4c
0.99
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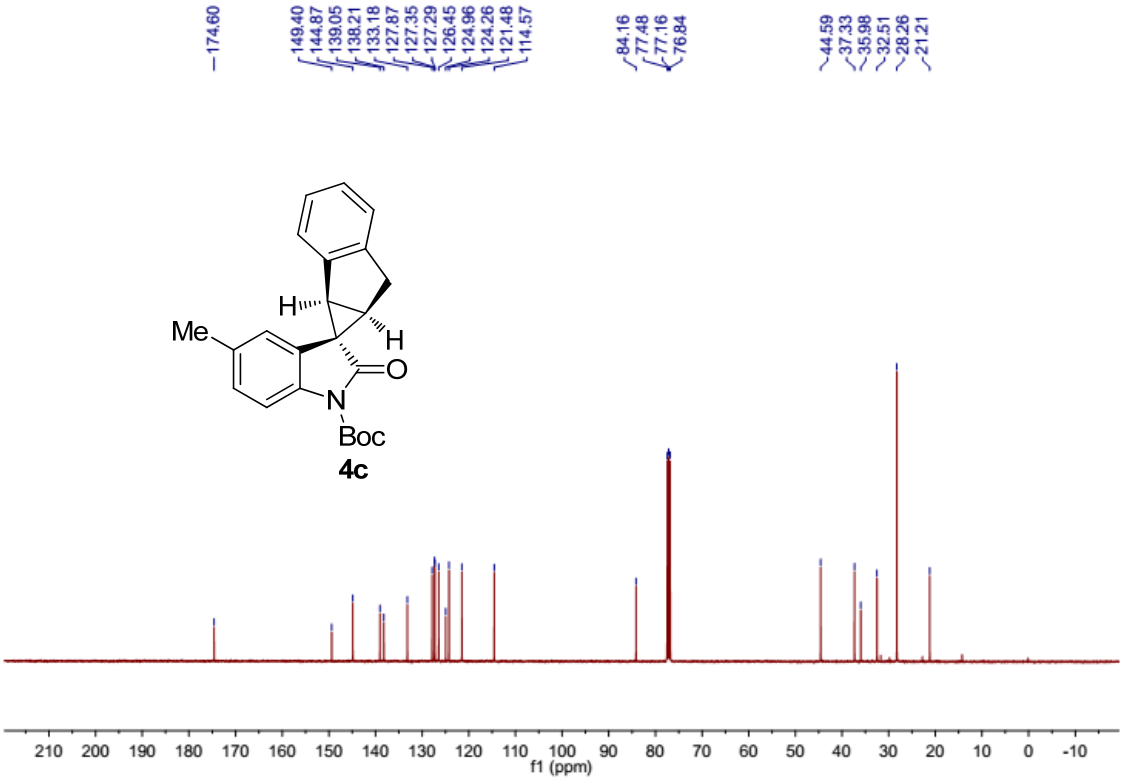
0.99
1.03
1.00
2.07
1.03
1.02
2.98
9.00
f1 (ppm)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127.35
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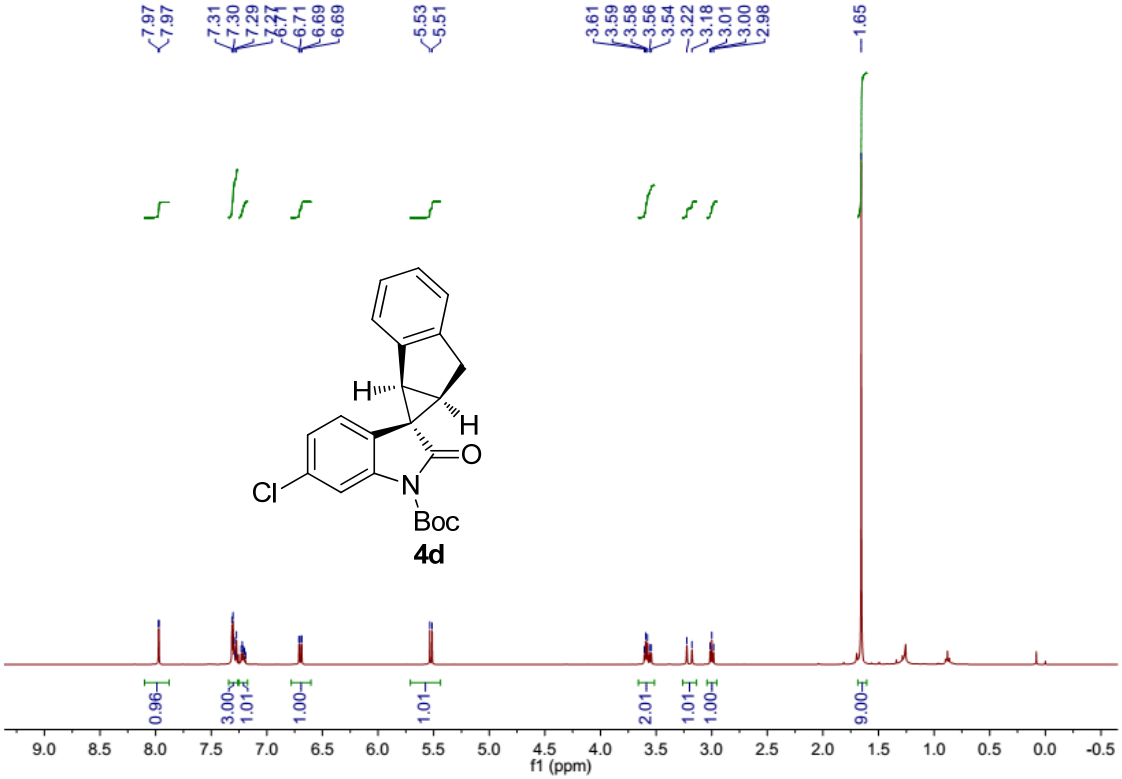
126.45
124.96
124.26
121.48
114.57
84.16
77.48
77.16
76.84
44.59
37.33
35.98
32.51
28.26
21.21
Me
H
H
O
N
Boc
4c
f1 (ppm)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6.71
6.69
6.69
5.53
5.51
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3.59
3.58
3.56
3.54
3.22
3.18
3.01
3.00
2.98
1.65
H
H
O
N
Cl
Boc
4d
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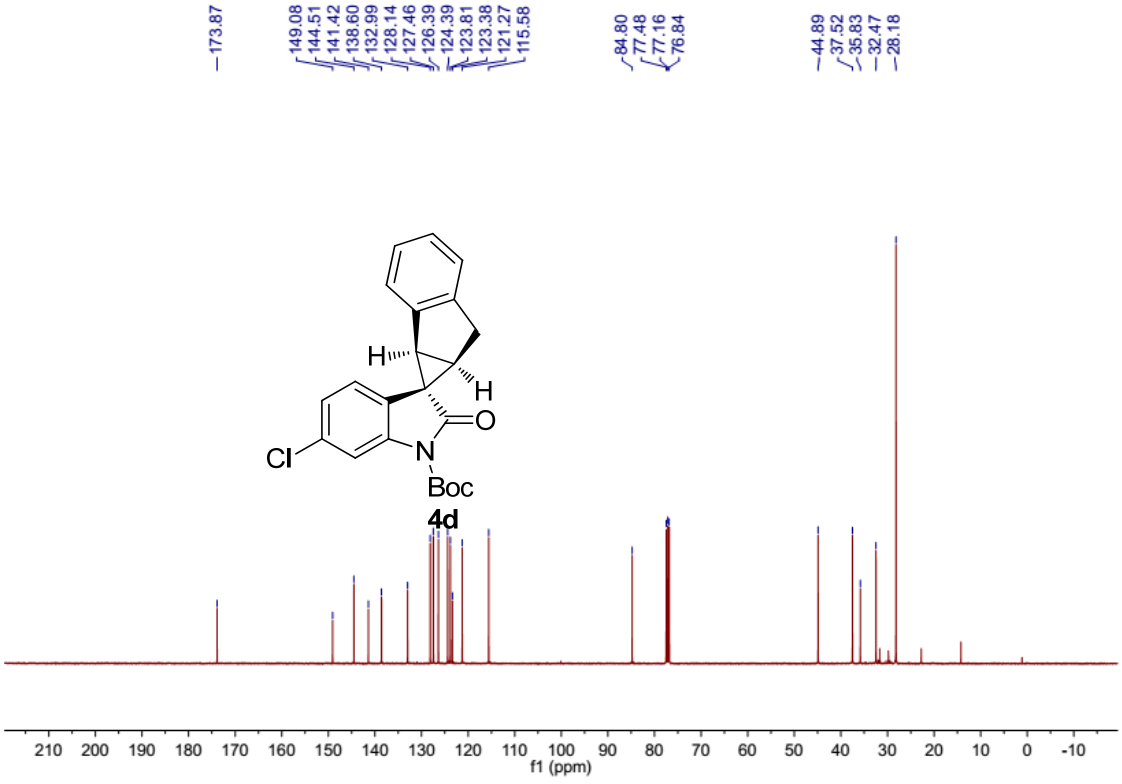
3.00
1.01
1.00
1.01
2.01
1.01
1.00
9.00
f1 (ppm)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123.81
123.38
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115.58
84.80
77.48
77.16
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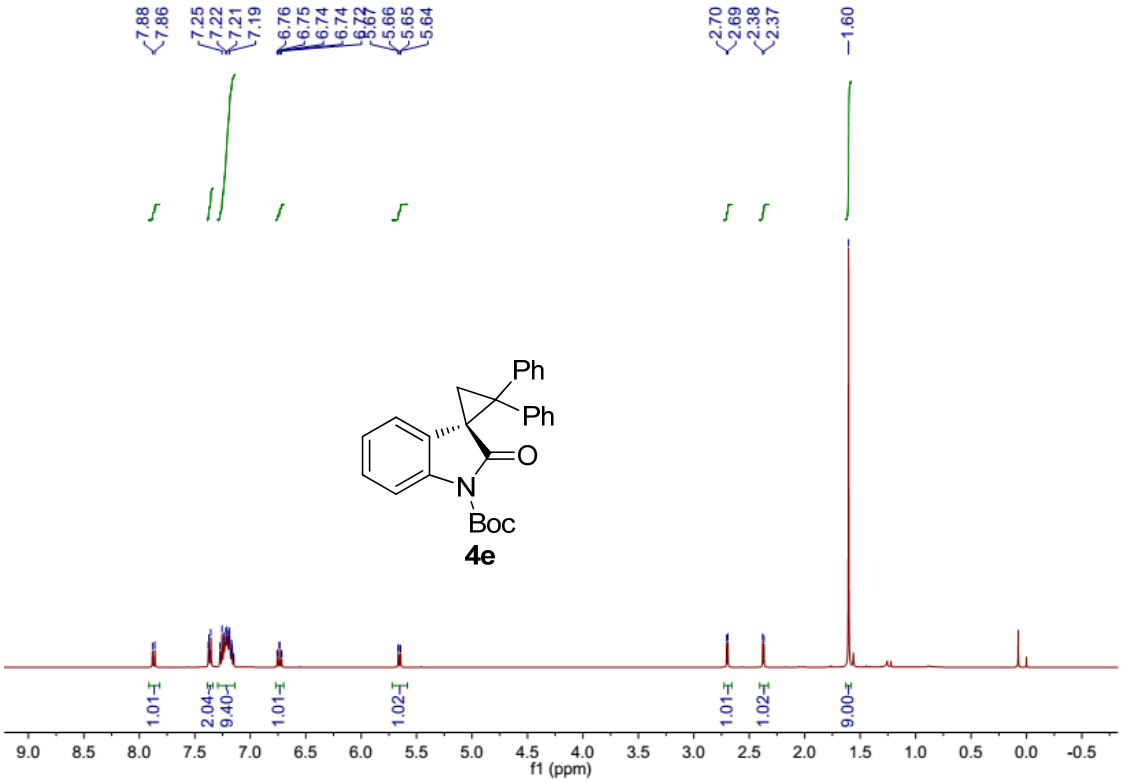
37.52
35.83
32.47
28.18
H
H
O
N
Cl
Boc
4d
f1 (ppm)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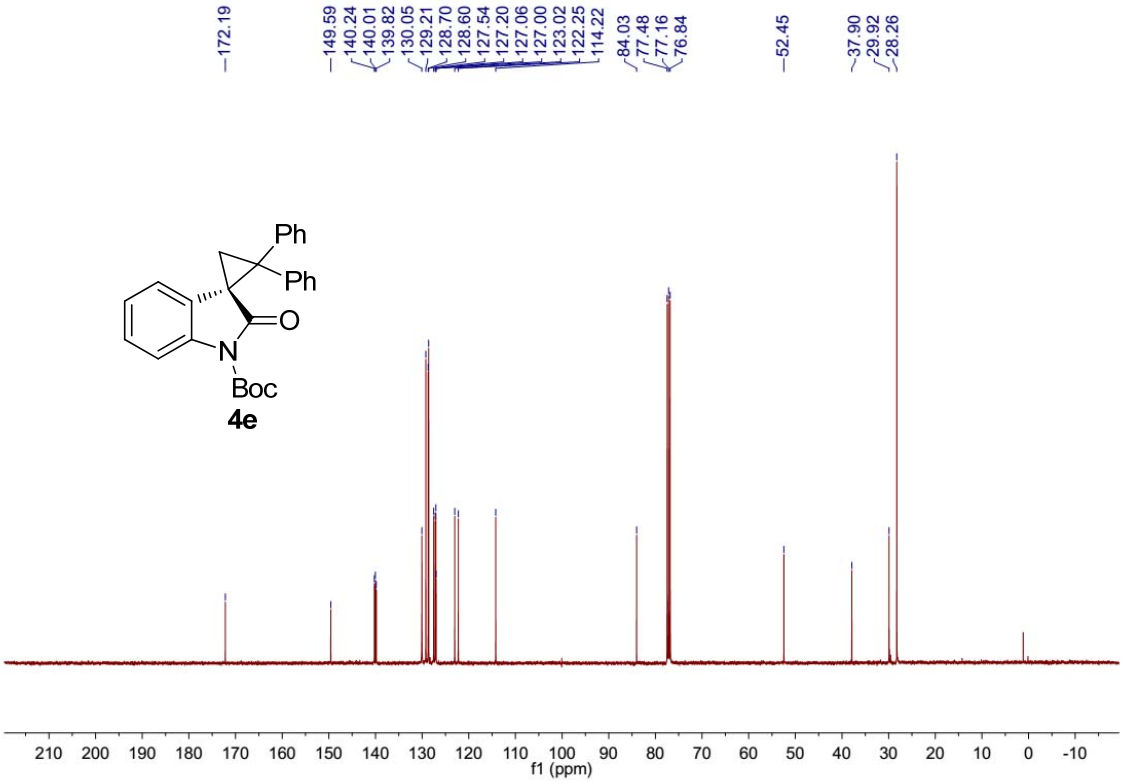
6.72
6.67
5.66
5.65
5.64
2.70
2.69
2.38
2.37
1.60
Ph
Ph
O
N
Boc
4e
1.01
2.04
9.40
1.01
1.02
1.01
1.02
9.00
f1 (ppm)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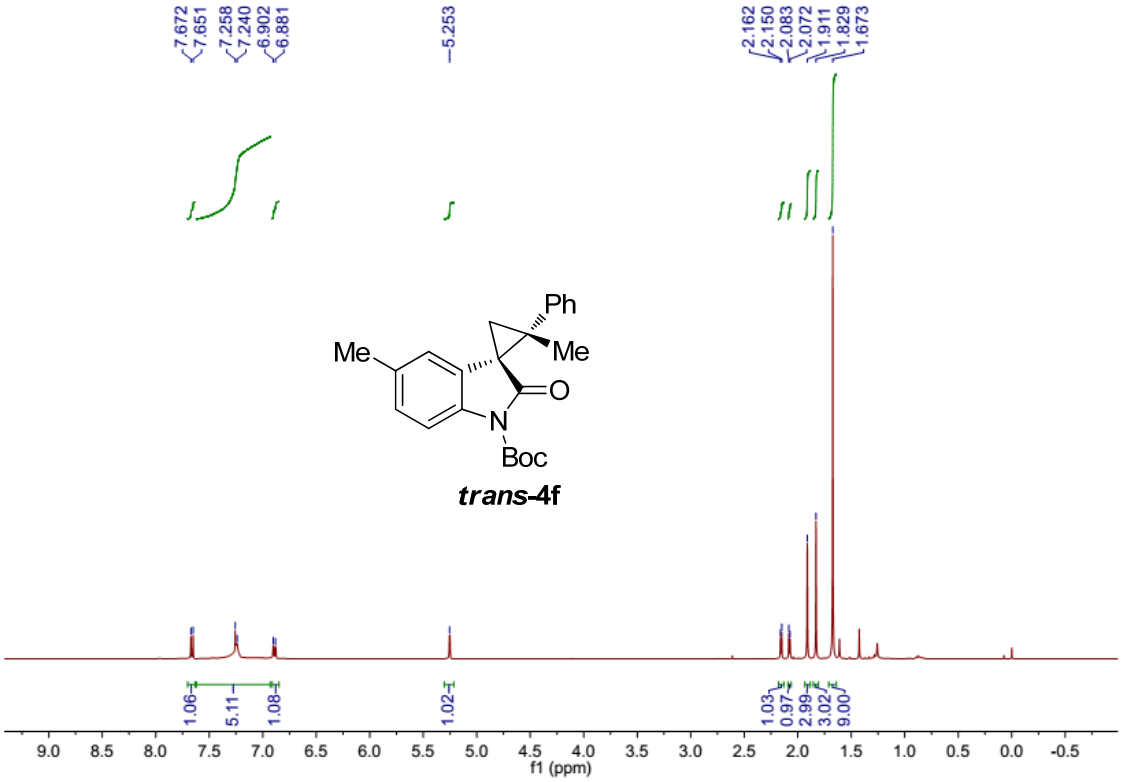
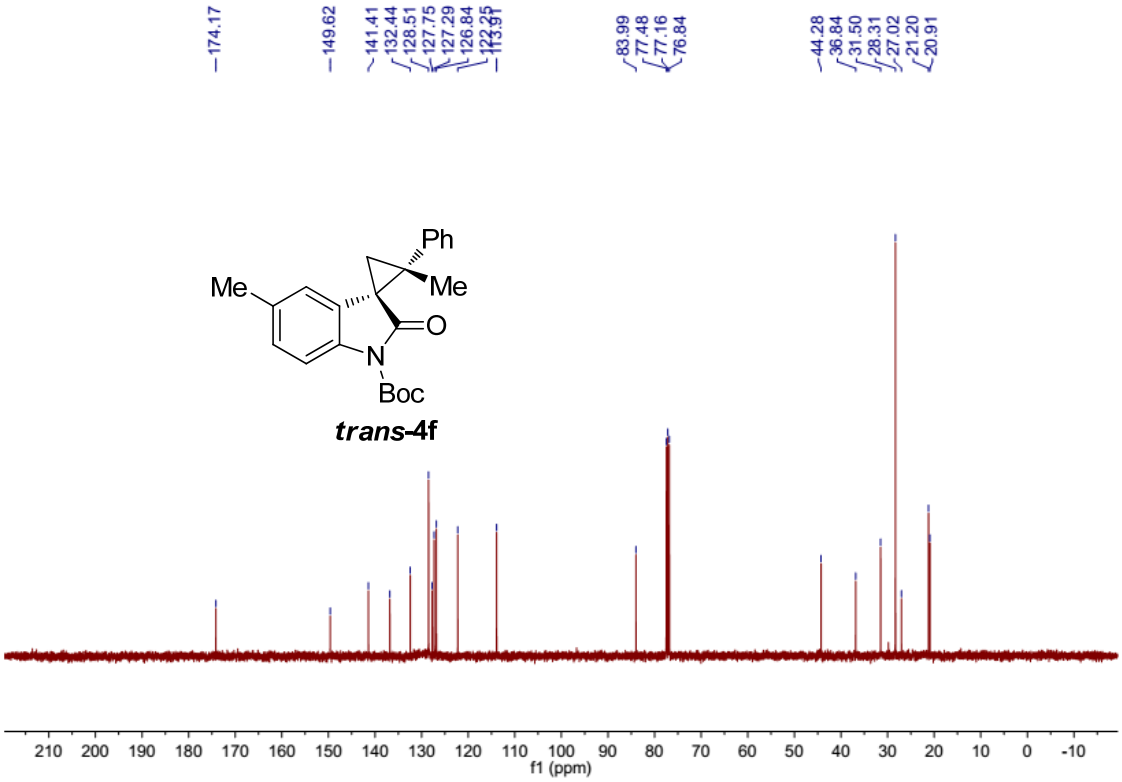
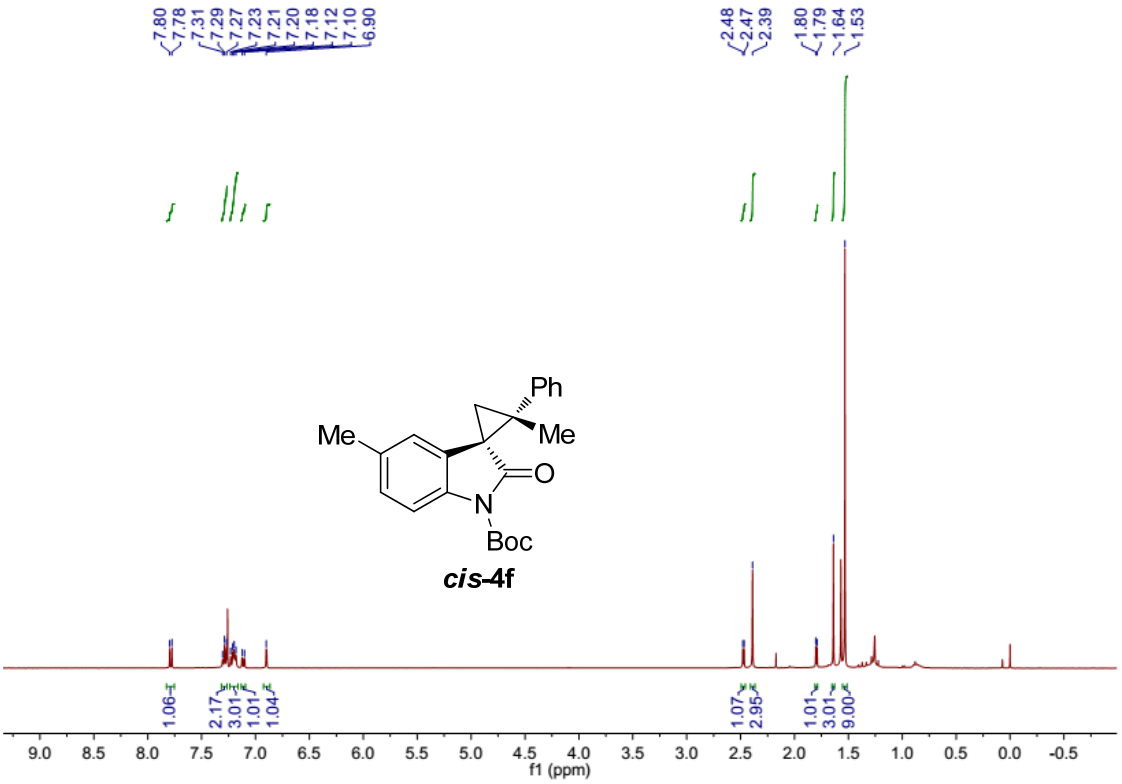
128.60
127.54
127.20
127.06
127.00
123.02
122.25
114.22
84.03
77.48
77.16
76.84
52.45
37.90
29.92
28.26
Ph
Ph
O
N
Boc
4e
f1 (ppm)



***trans*-4f**



***trans*-4f**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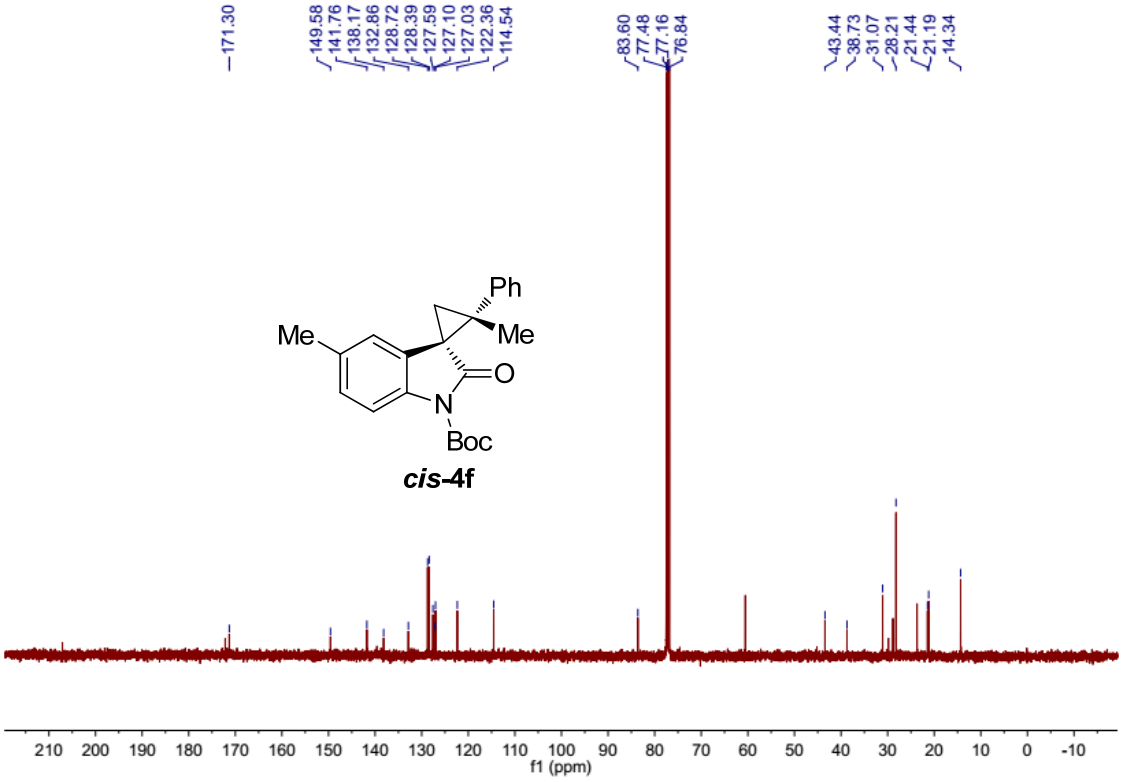
7.20
7.18
7.12
7.10
6.90
2.48
2.47
2.39
1.80
1.79
1.64
1.53
Ph
Me
Me
O
N
Boc
cis-4f
1.06
2.17
3.01
1.01
1.04
1.07
2.95
1.01
3.01
9.00
f1 (ppm)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128.72
128.39
127.59
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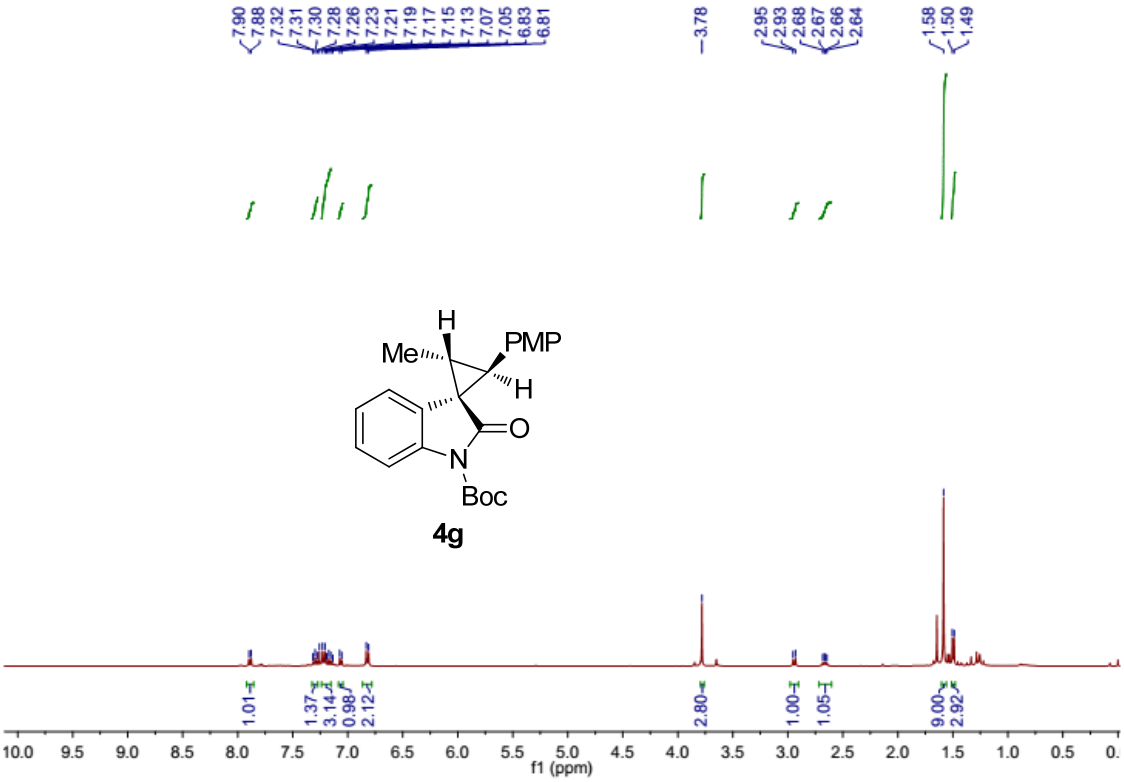
127.03
122.36
114.54
83.60
77.48
77.16
76.84
43.44
38.73
31.07
28.21
21.44
21.19
14.34
Ph
Me
Me
O
N
Boc
cis-4f
f1 (ppm)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7.88
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7.31
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7.28
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7.23
7.21
7.19
7.17
7.15
7.13
7.07
7.05
6.83
6.81
3.78
2.95
2.93
2.68
2.67
2.66
2.64
1.58
1.50
1.49
H
Me
PMP
H
O
N
Boc
4g
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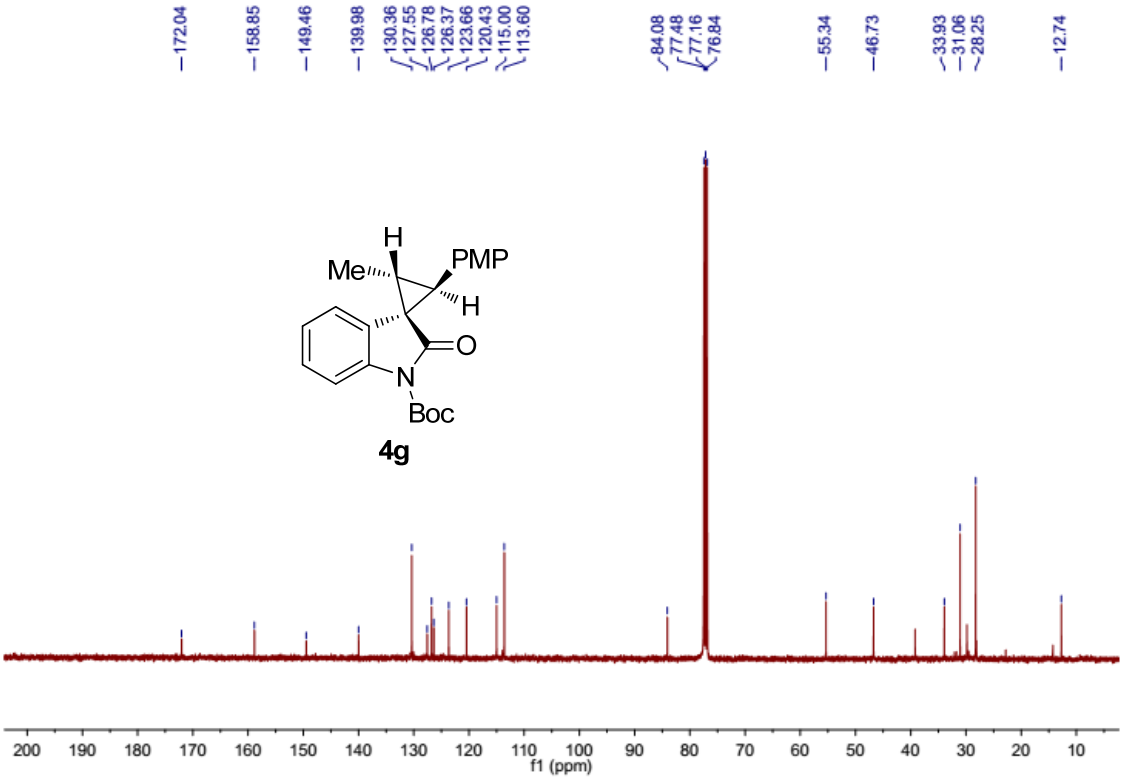
3.14
0.98
2.12
2.80
1.00
1.05
9.00
2.92
f1 (ppm)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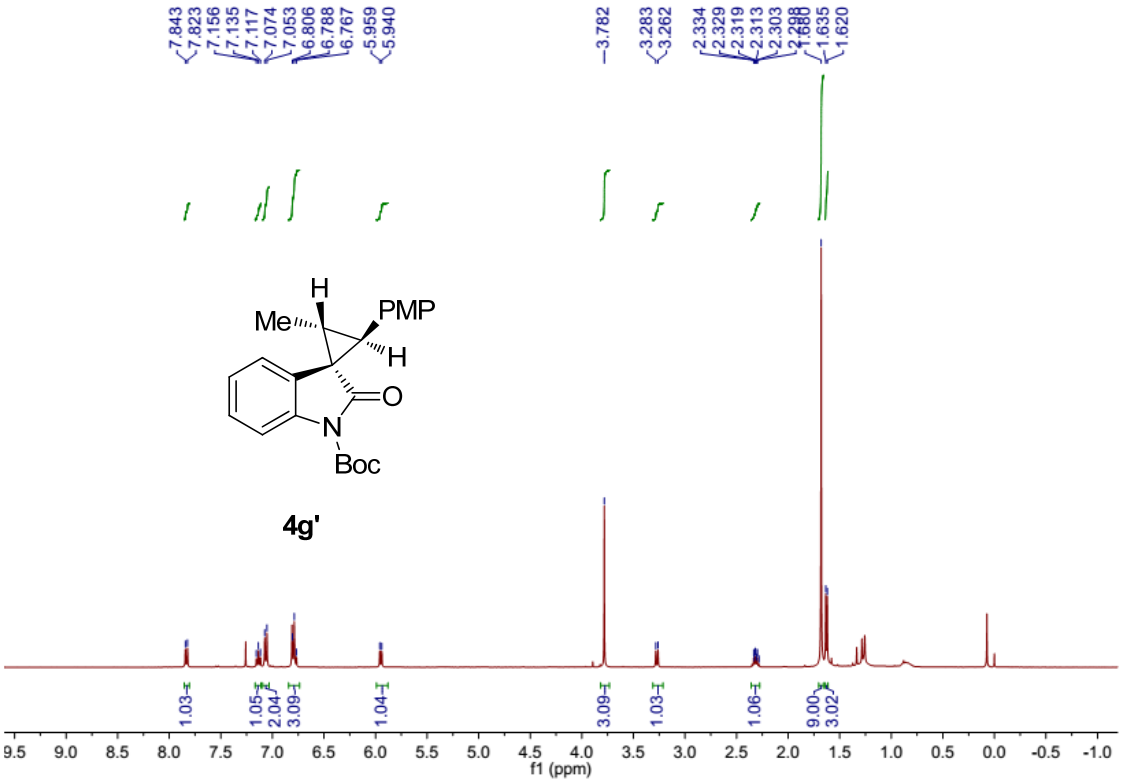
127.55
126.78
126.37
123.66
120.43
115.00
113.60
84.08
77.48
77.16
76.84
55.34
46.73
33.93
31.06
28.25
12.74
H
Me
PMP
H
O
N
Boc
4g
f1 (ppm)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6.767
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3.782
3.283
3.262
2.334
2.329
2.319
2.313
2.303
2.298
1.635
1.620
H
Me
PMP
H
O
N
Boc
1.03
1.05
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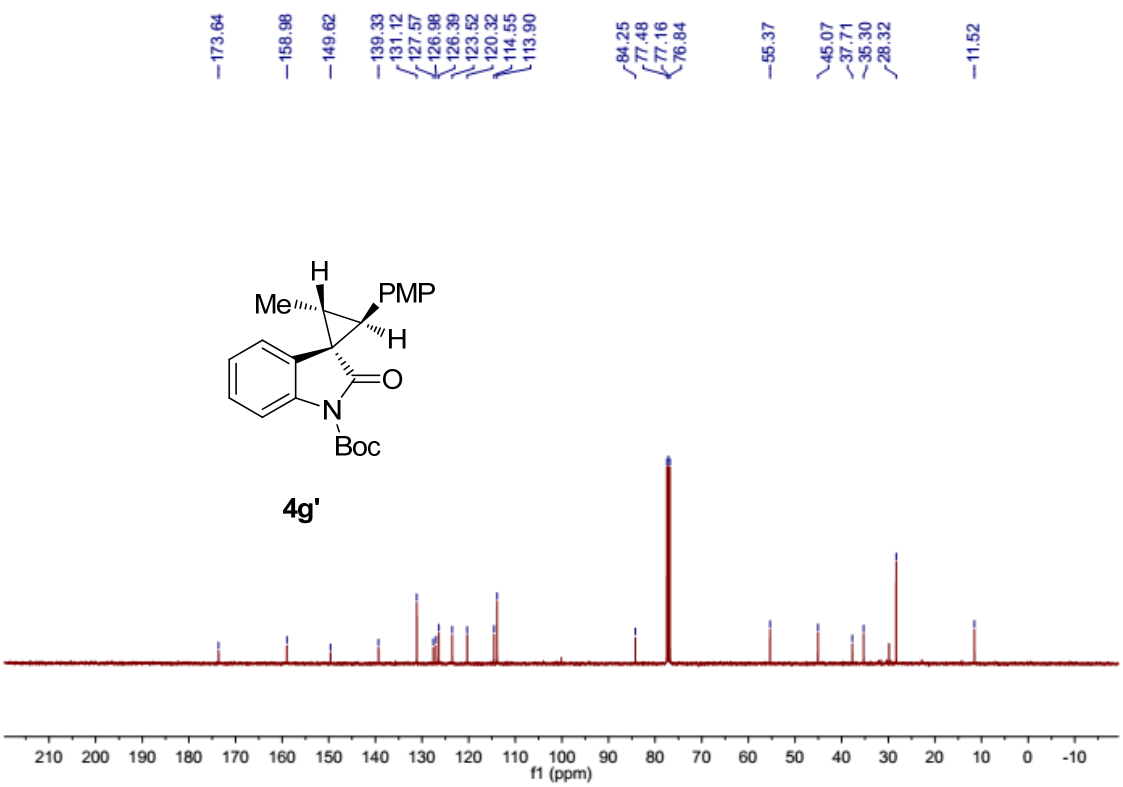
3.09
1.04
3.09
1.03
1.06
9.00
3.02
f1 (ppm)

4g'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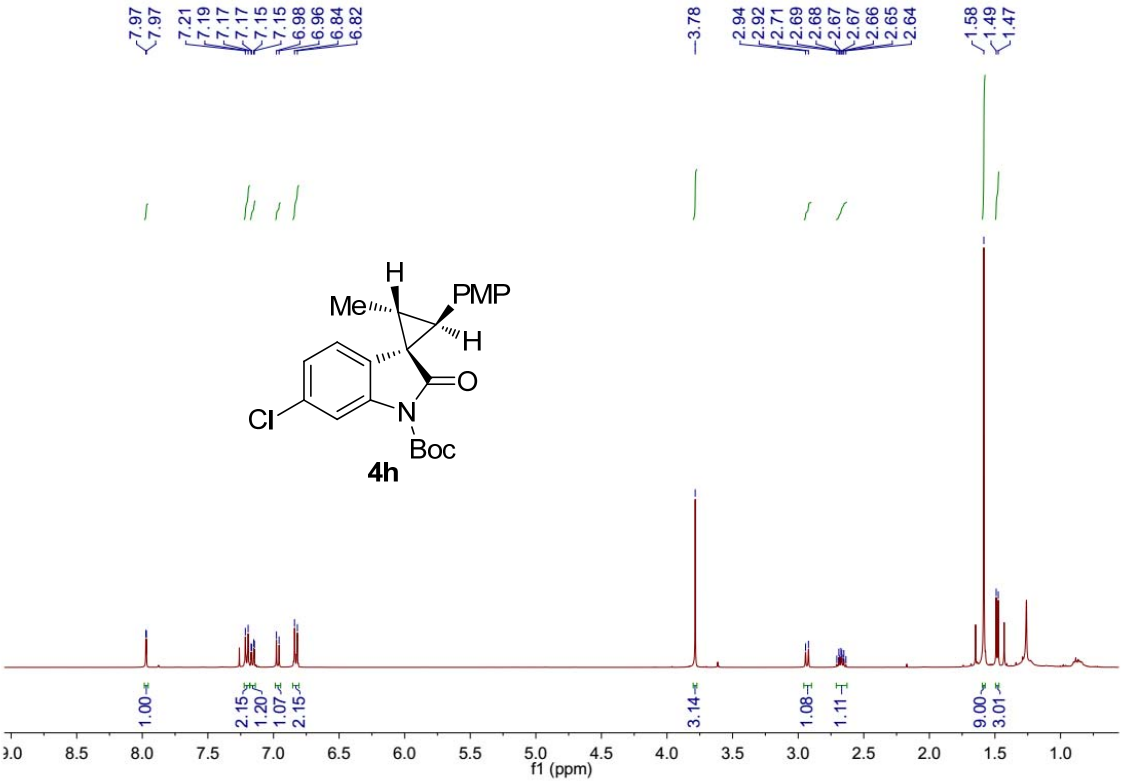
77.48
77.16
76.84
55.37
45.07
37.71
35.30
28.32
11.52
H
Me
PMP
H
O
N
Boc
f1 (ppm)

4g'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6.82
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2.69
2.68
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2.67
2.66
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1.47
H
Me
PMP
H
O
Cl
N
Boc
4h
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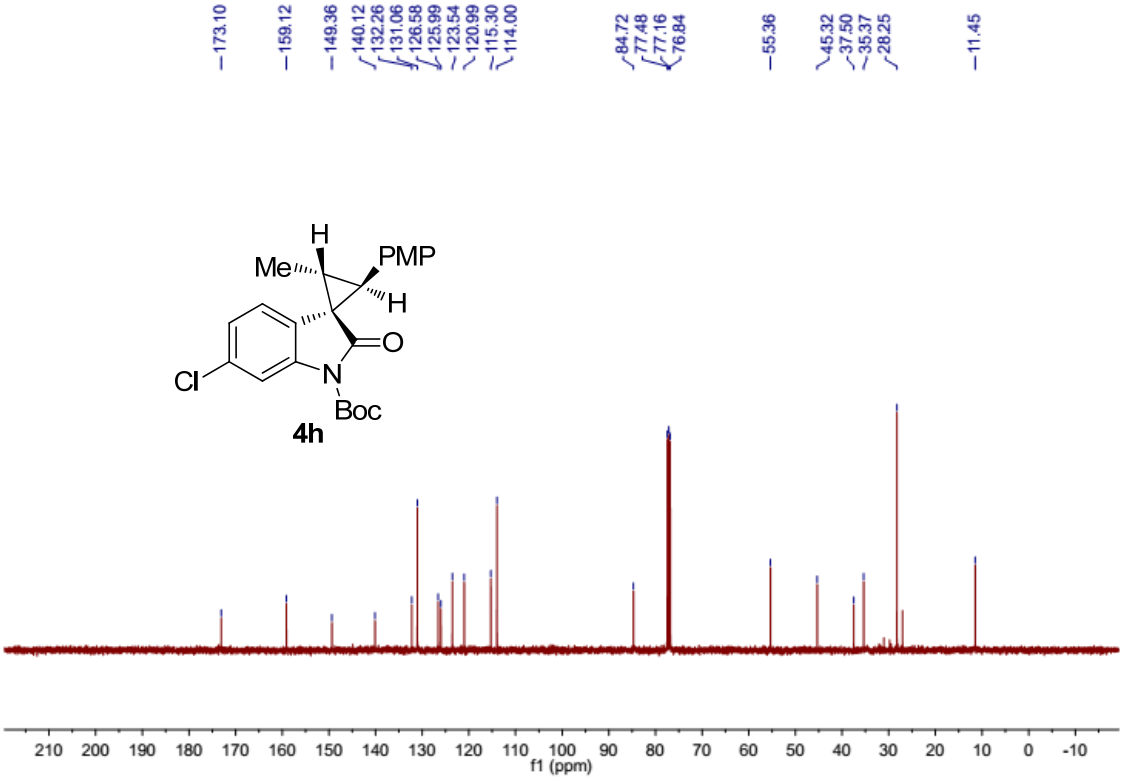
3.01
f1 (ppm)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77.16
76.84
55.36
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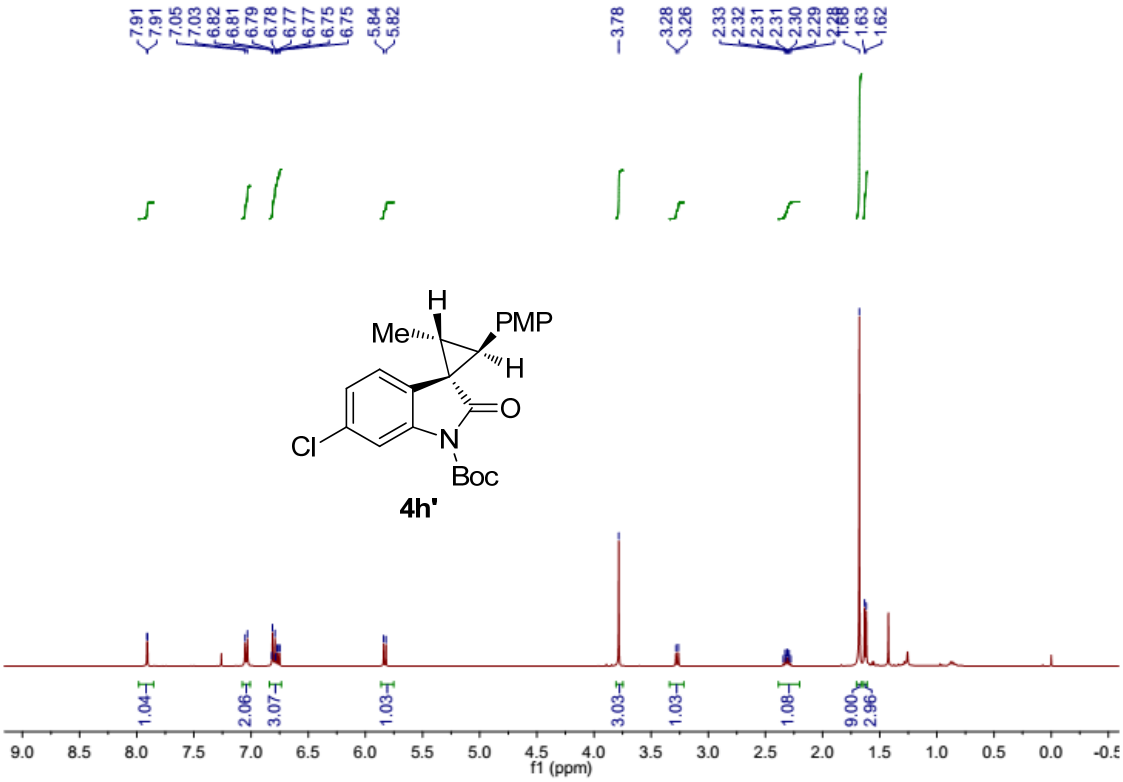
H
Me
PMP
H
O
Cl
N
Boc
4h
f1 (ppm)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H
Me
PMP
H
O
N
Cl
Boc
4h'
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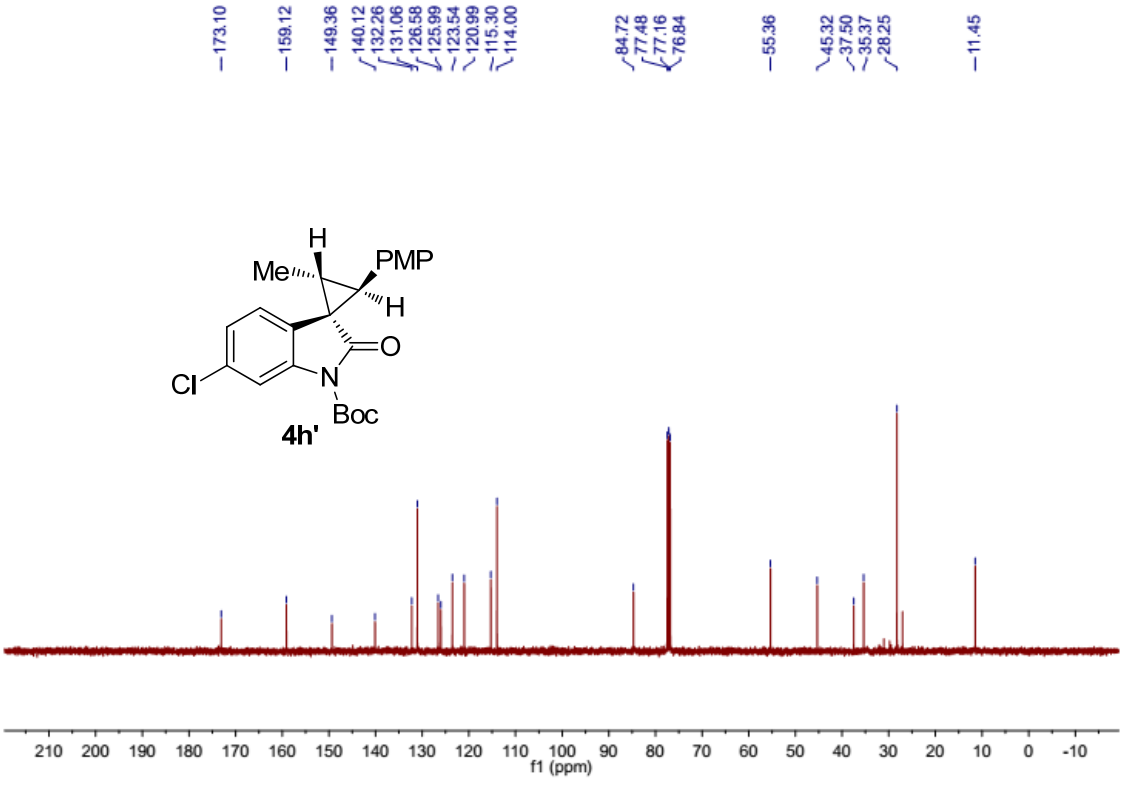
1.03
1.08
9.00
2.96
f1 (ppm)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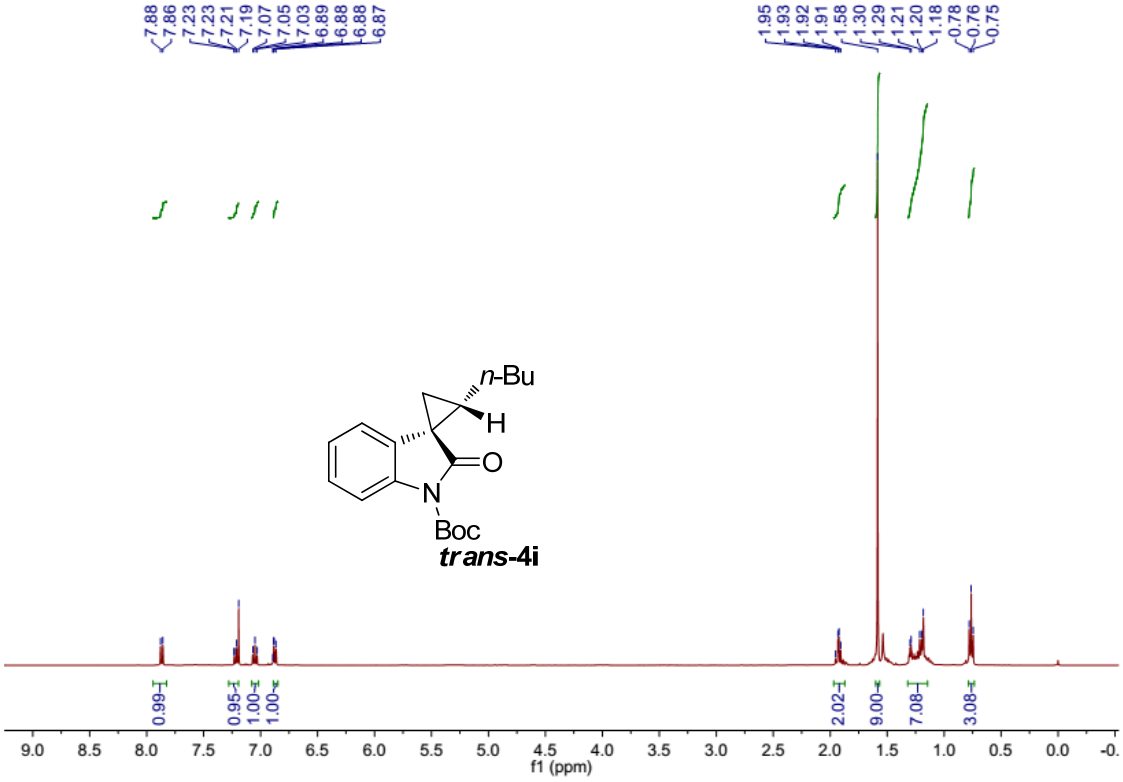
H
O
N
Cl
Boc
4h'
f1 (ppm)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1.93
1.92
1.91
1.58
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1.29
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1.20
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0.76
0.75
n-Bu
H
O
N
Boc
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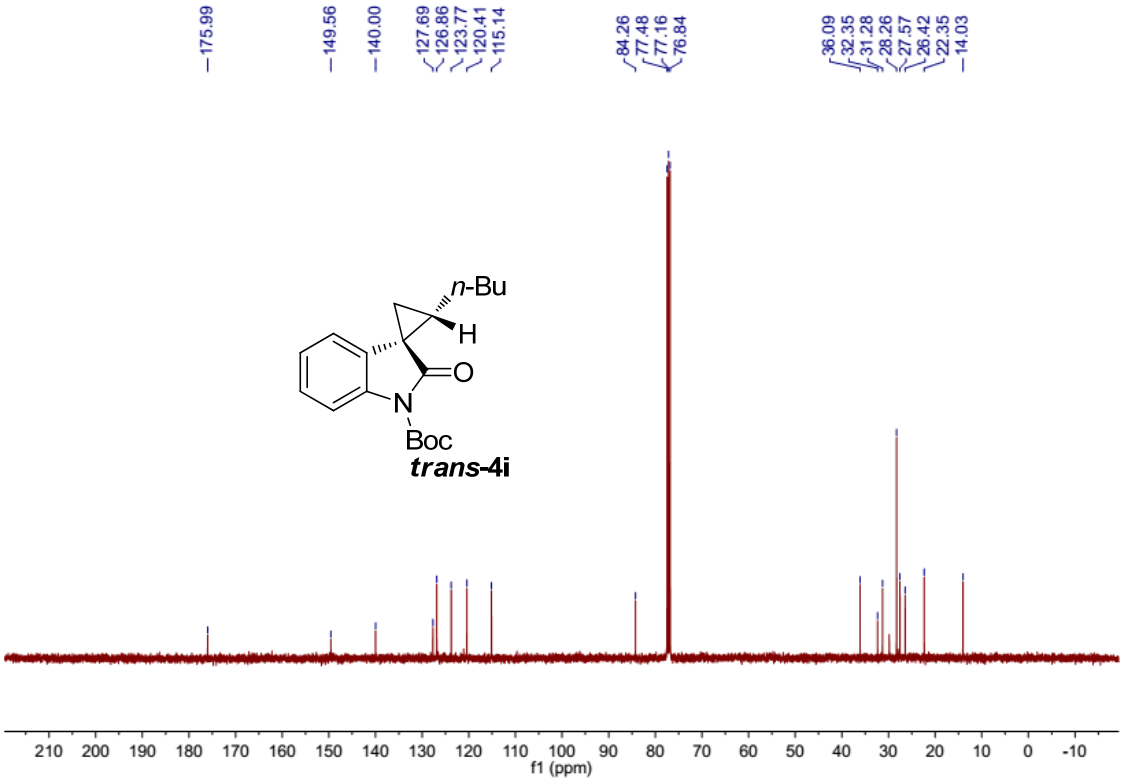
3.08
f1 (ppm)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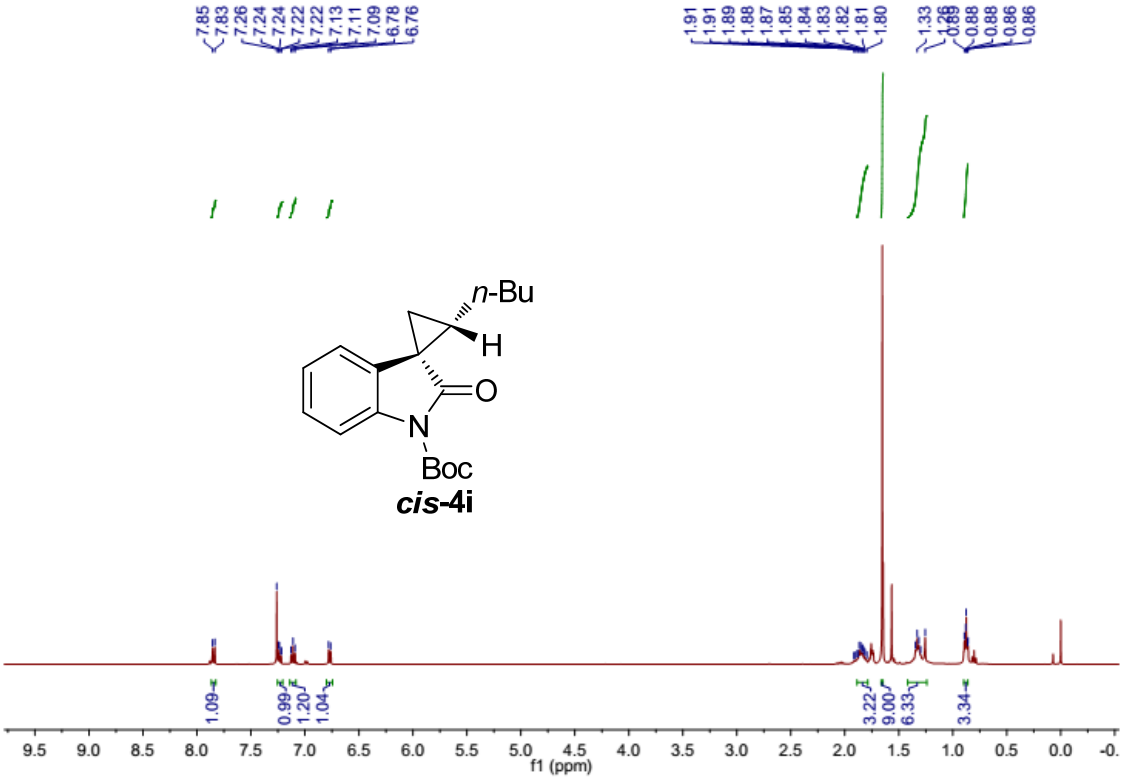
14.03
n-Bu
H
O
N
Boc
trans-4i
f1 (ppm)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0.88
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Boc
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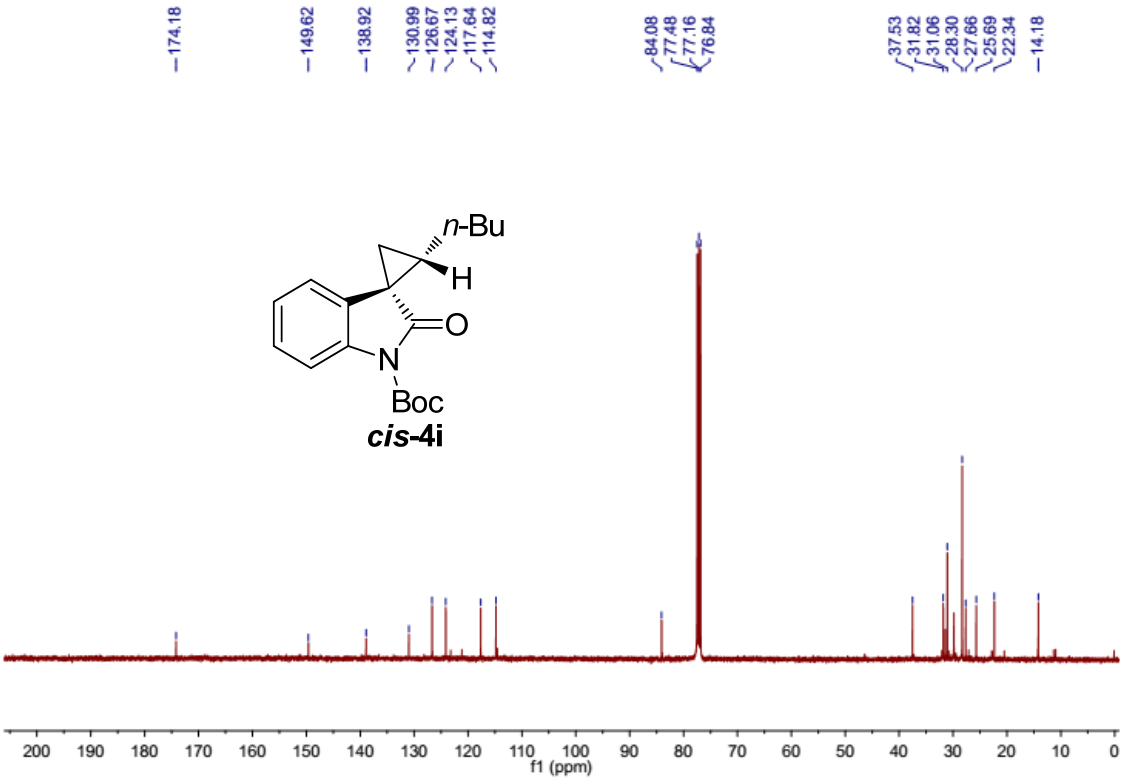
f1 (ppm)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31.82
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22.34
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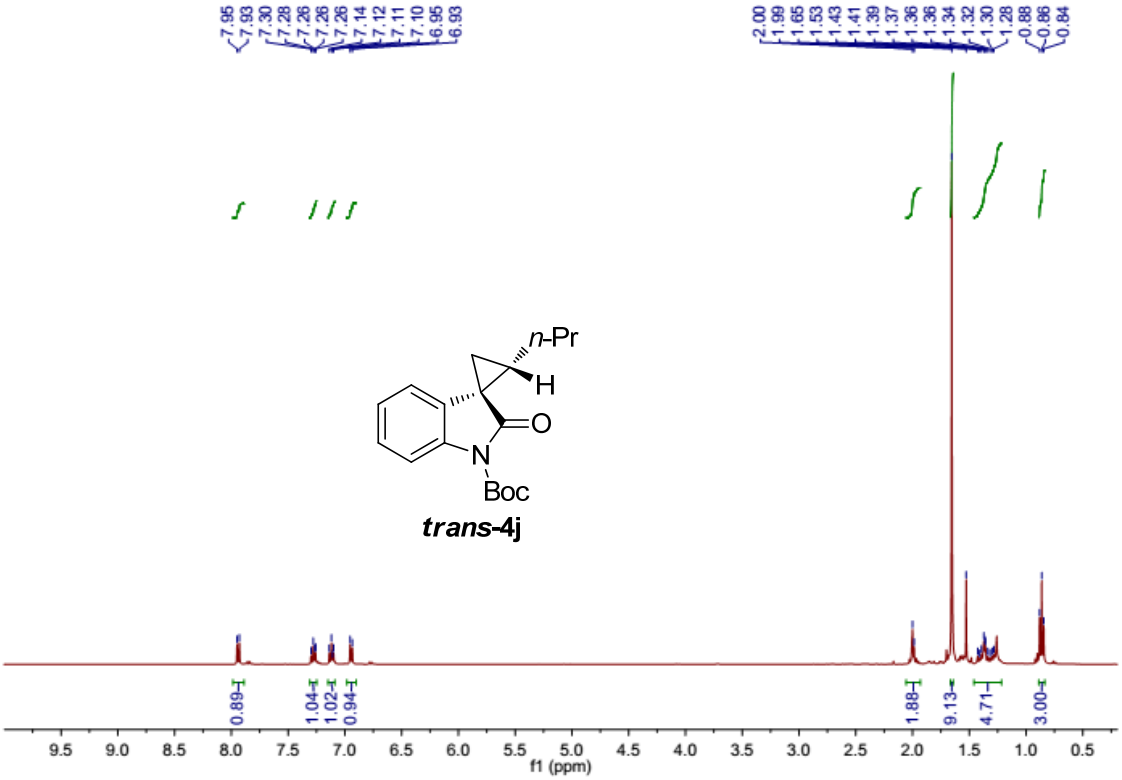
n-Bu
H
O
N
Boc
cis-4i
f1 (ppm)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O
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Boc
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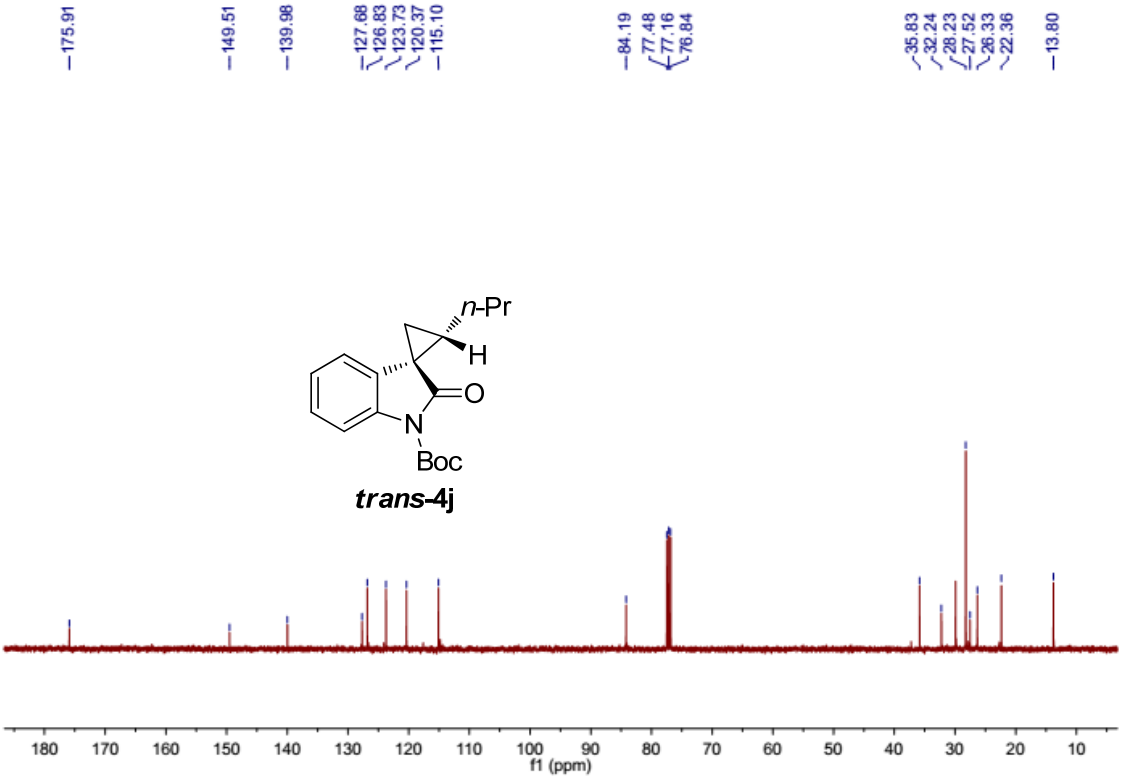
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H
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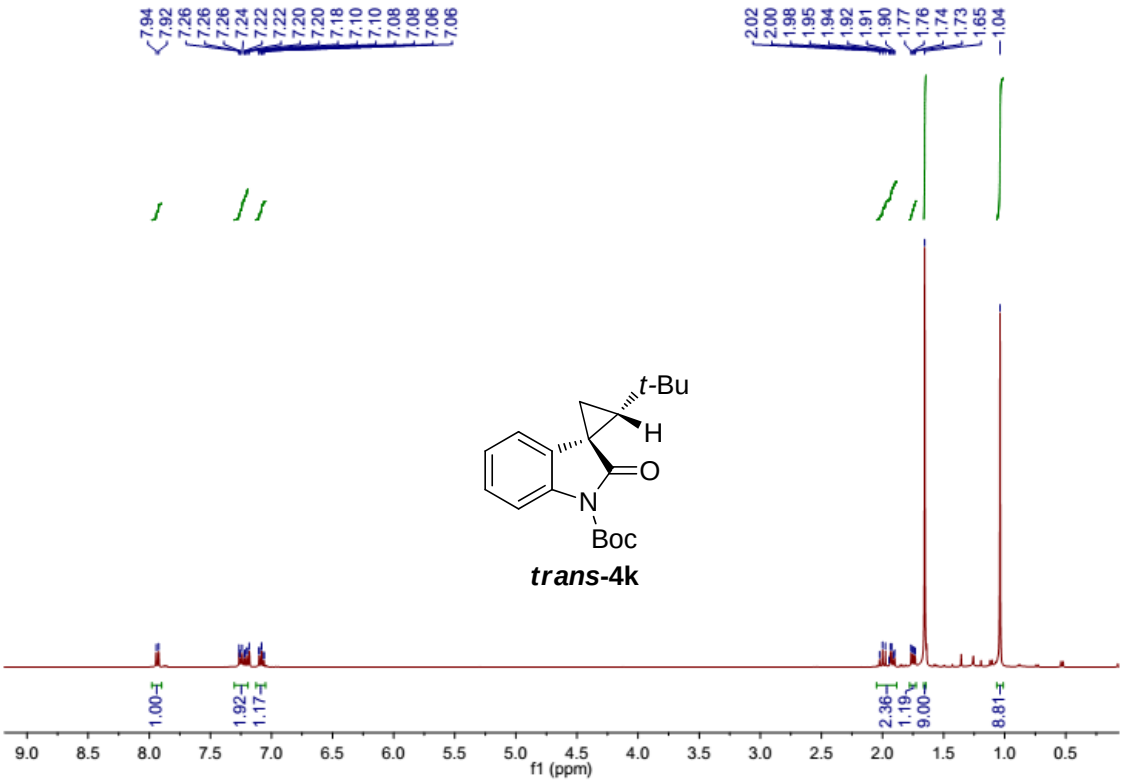
N
Boc
trans-4j
f1 (ppm)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1.76
1.74
1.73
1.65
1.04
t-Bu
H
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Boc
trans-4k
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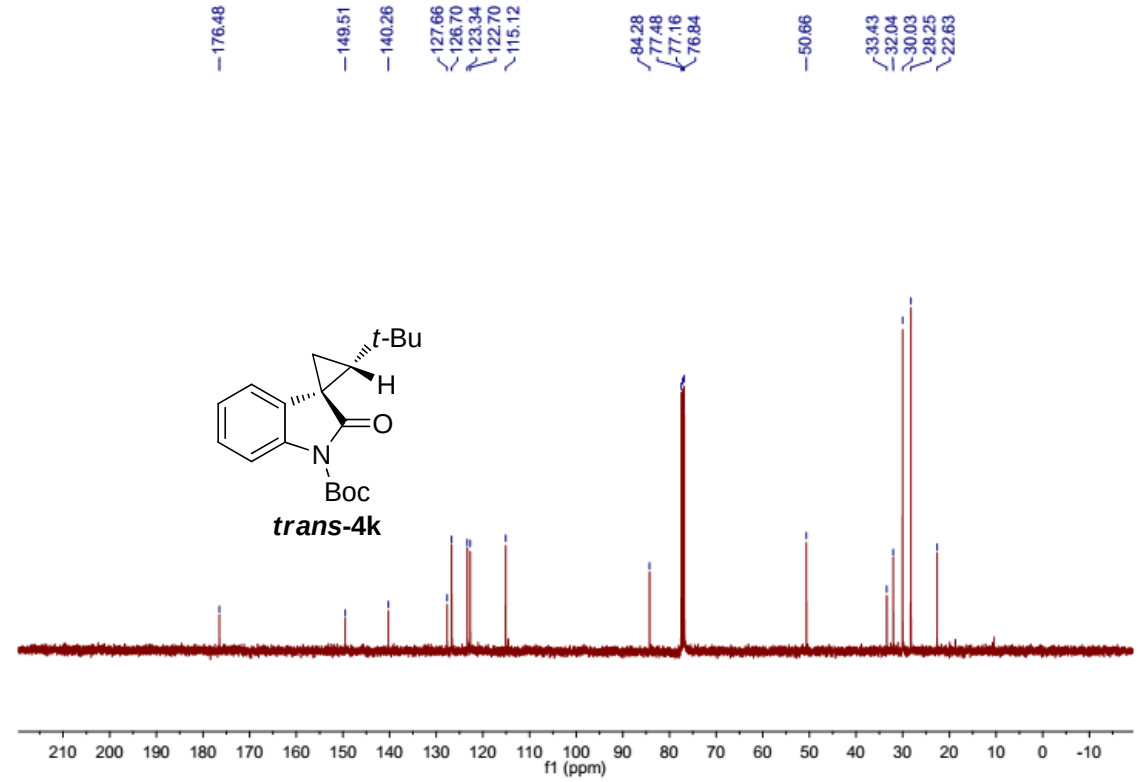
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77.16
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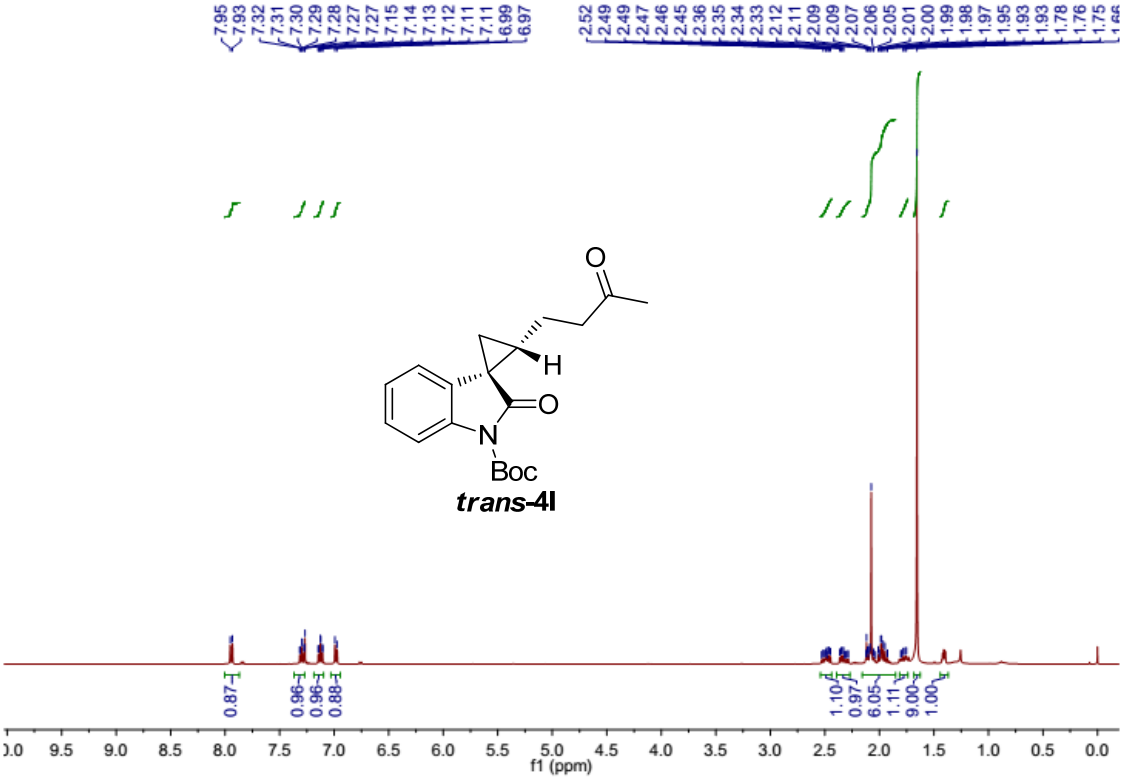
30.03
28.25
22.63
t-Bu
H
O
N
Boc
trans-4k
f1 (ppm)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2.00
1.99
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1.93
1.93
1.78
1.76
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O
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O
N
Boc
trans-4l
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1.11
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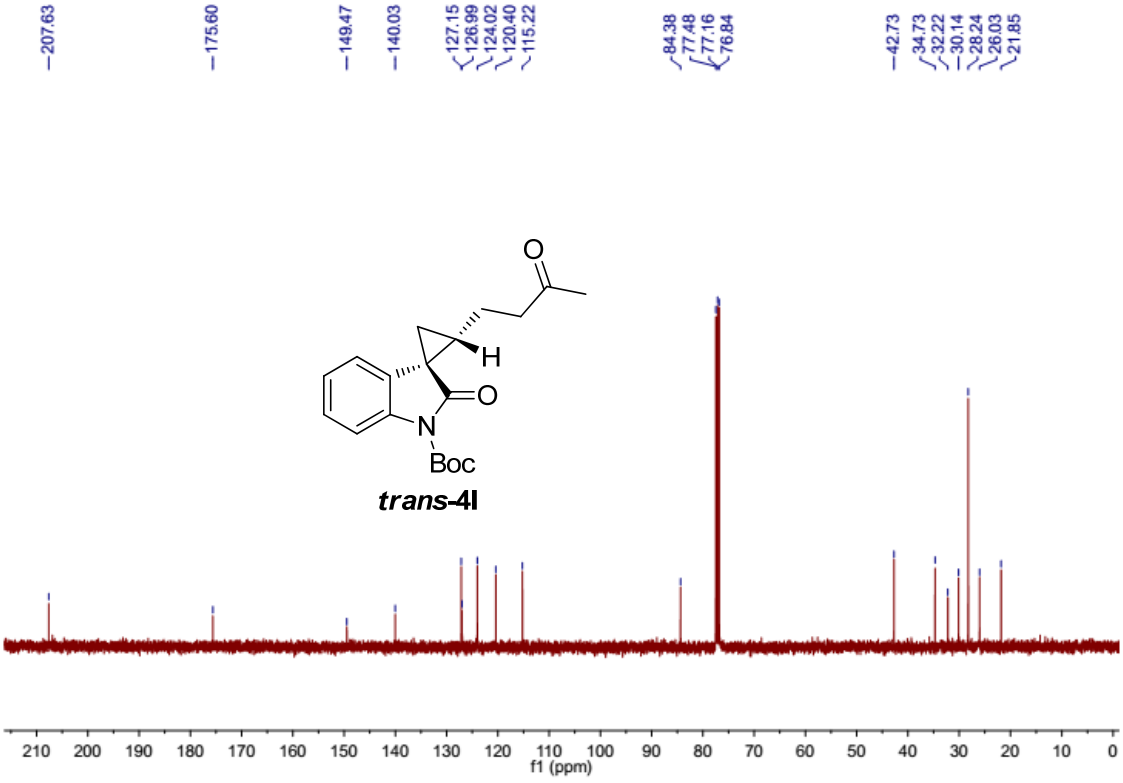
1.00
f1 (ppm)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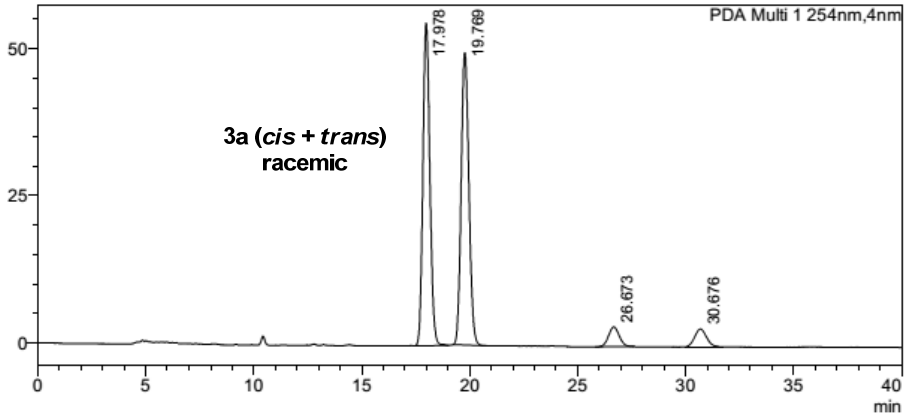
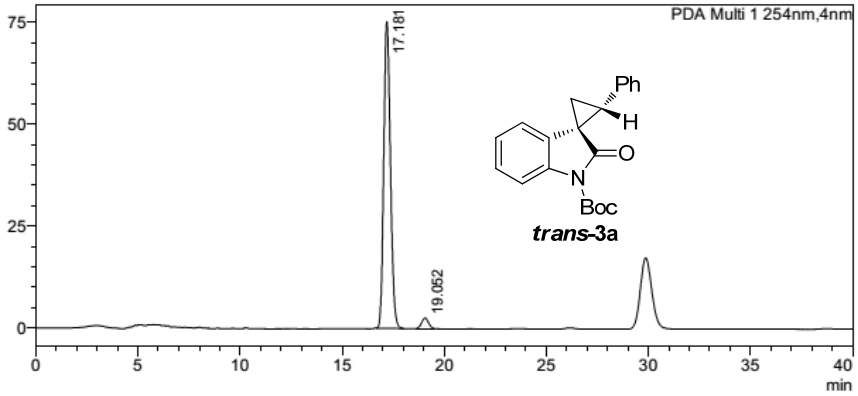
H
O
N
Boc
trans-4l
f1 (ppm)



<Peak Table>

PDA Ch1 254nm

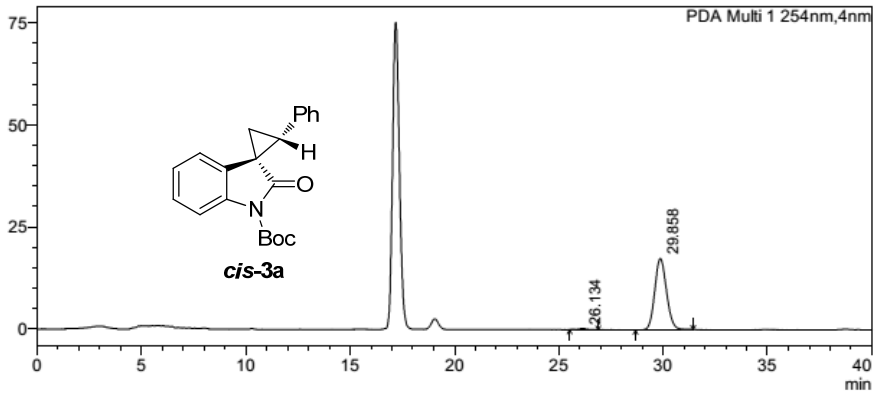
Peak#	Ret. Time	Area	Height	Area%
1	17.978	1226716	54860	45.540
2	19.769	1233447	49763	45.790
3	26.673	116956	3361	4.342
4	30.676	116603	3042	4.329
Total		2693722	111026	100.000



<Peak Table>

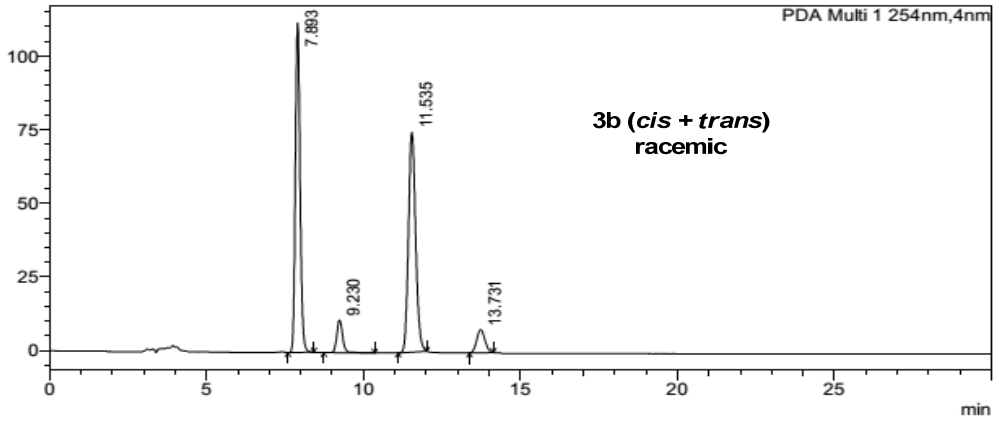
PDA Ch1 254nm

Peak#	Ret. Time	Area	Height	Area%
1	17.181	1705162	75200	96.558
2	19.052	60791	2563	3.442
Total		1765953	77763	100.000



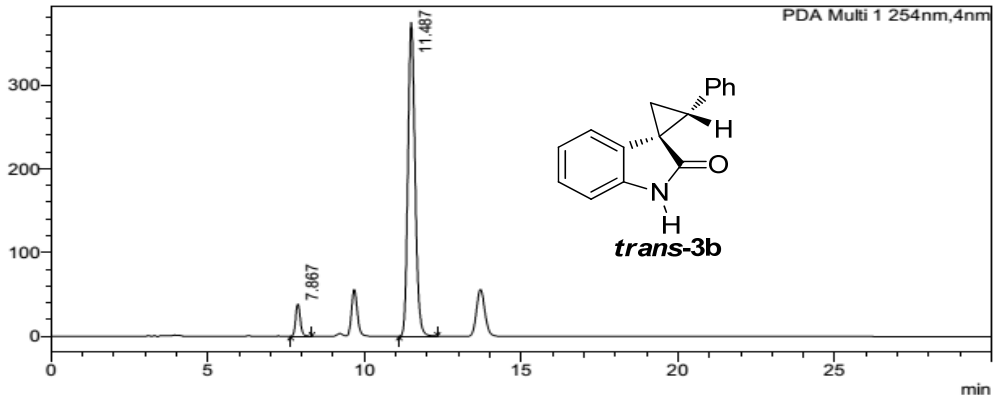
PDA Ch1 254nm

Peak#	Ret. Time	Area	Height	Area%
1	26.134	9014	278	1.301
2	29.858	683654	17490	98.699
Total		692668	17769	100.000



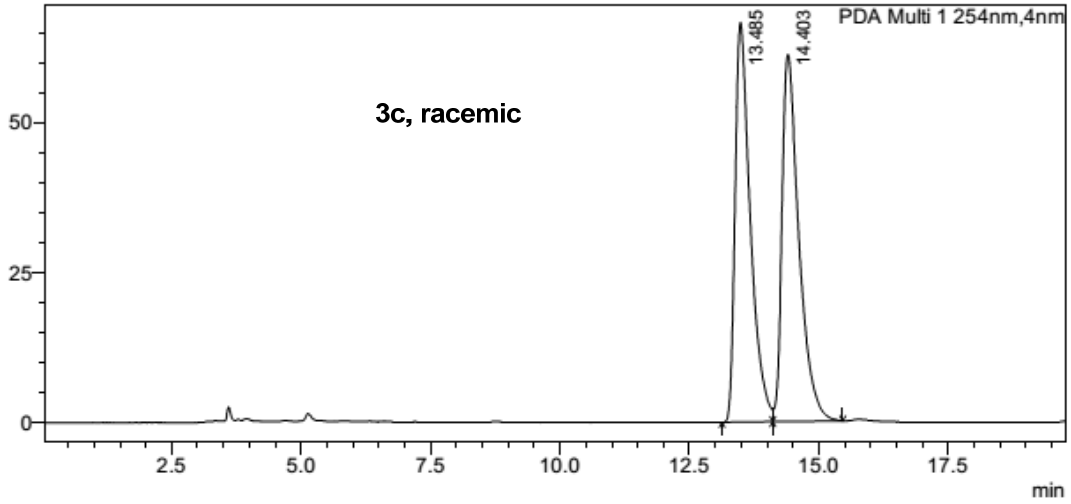
PDA Ch1 254nm

Peak#	Ret. Time	Area	Height	Area%
1	7.893	1168235	112065	44.255
2	9.230	143534	11100	5.437
3	11.535	1185789	74618	44.920
4	13.731	142200	7855	5.387
Total		2639757	205638	100.000



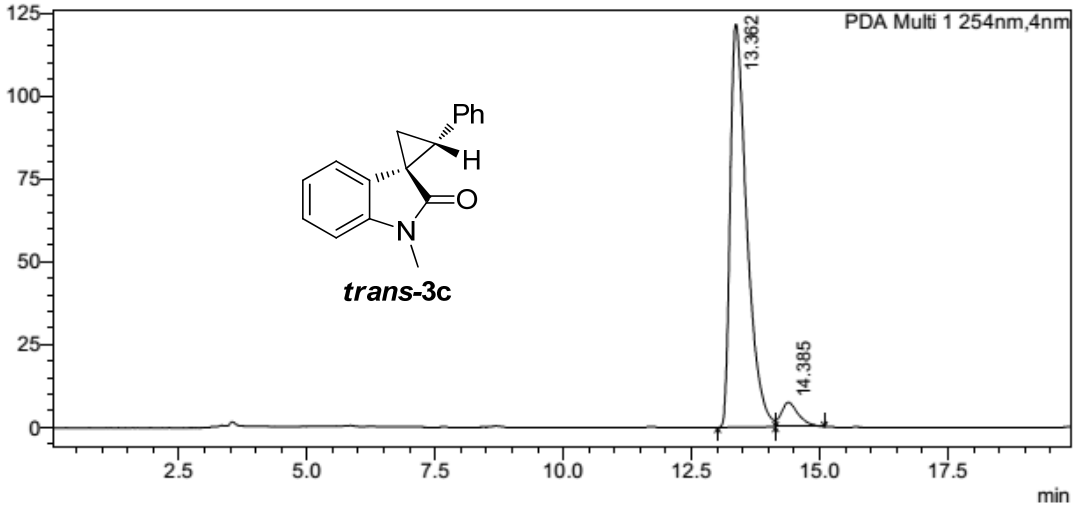
PDA Ch1 254nm

Peak#	Ret. Time	Area	Height	Area%
1	7.867	401450	38796	6.339
2	11.487	5931109	375005	93.661
Total		6332559	413801	100.000



PDA Ch1 254nm

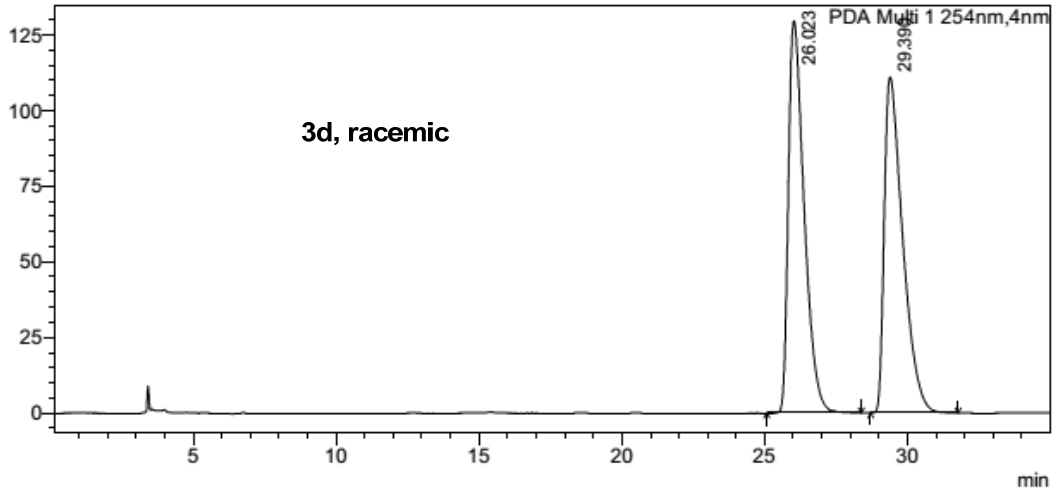
Peak#	Ret. Time	Area	Height	Area%
1	13.485	1368258	66732	49.558
2	14.403	1392691	61264	50.442
Total		2760949	127996	100.000



***trans*-3c**

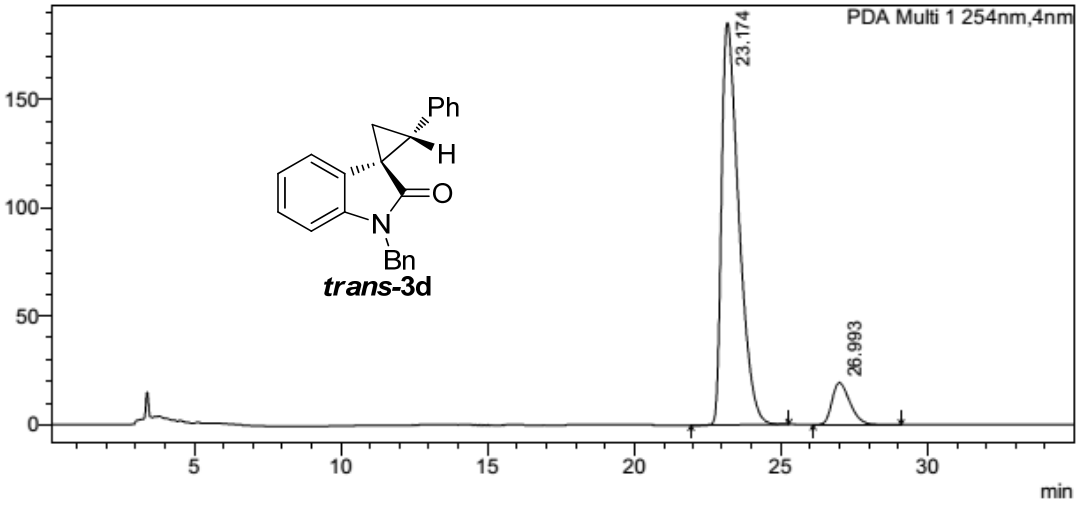
PDA Ch1 254nm

Peak#	Ret. Time	Area	Height	Area%
1	13.362	2749399	121549	94.001
2	14.385	175454	7380	5.999
Total		2924854	128929	100.000



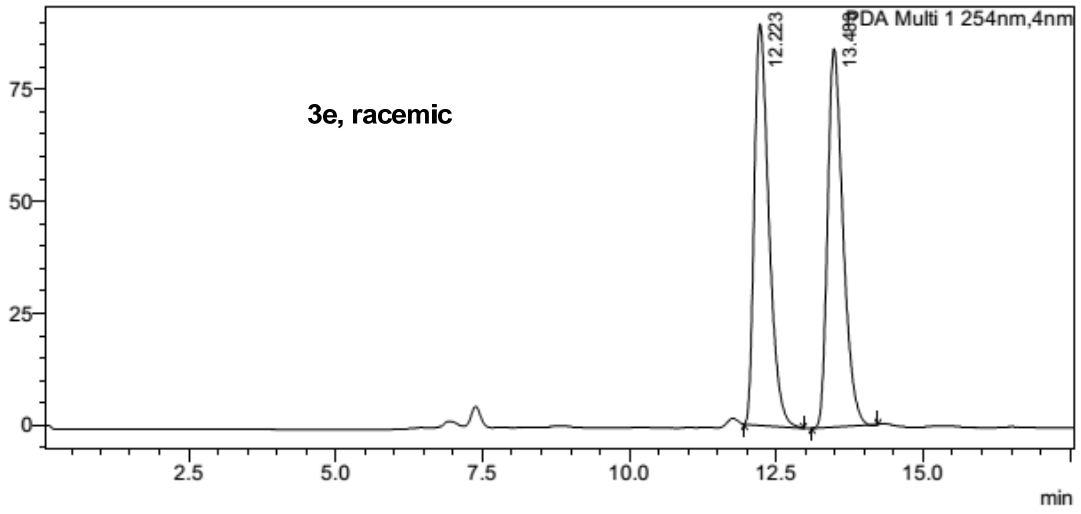
PDA Ch1 254nm

Peak#	Ret. Time	Area	Height	Area%
1	26.023	4990590	129680	49.970
2	29.390	4996671	111089	50.030
Total		9987262	240769	100.000



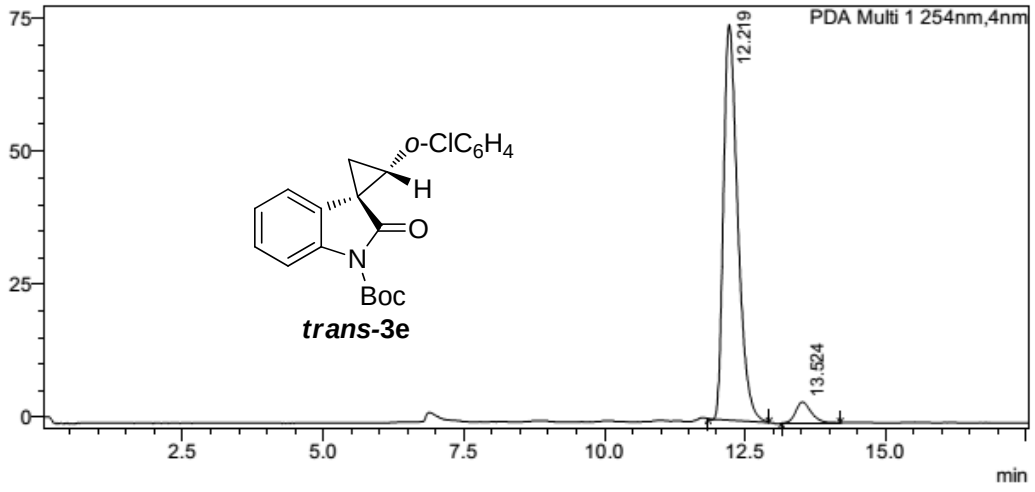
PDA Ch1 254nm

Peak#	Ret. Time	Area	Height	Area%
1	23.174	7584229	185501	90.241
2	26.993	820201	19344	9.759
Total		8404430	204845	100.000



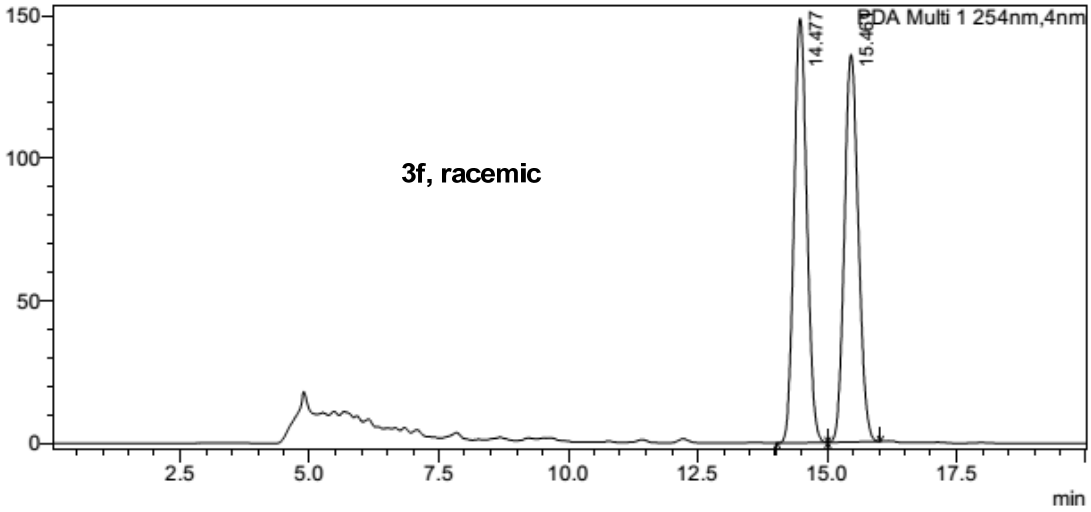
PDA Ch1 254nm

Peak#	Ret. Time	Area	Height	Area%
1	12.223	1544380	89613	49.944
2	13.483	1547828	84520	50.056
Total		3092207	174133	100.000



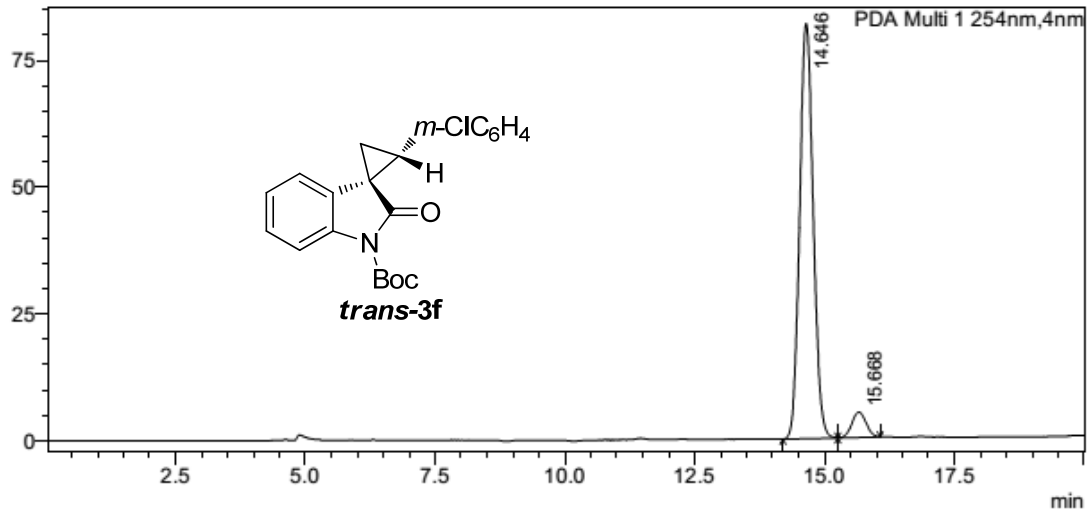
PDA Ch1 254nm

Peak#	Ret. Time	Area	Height	Area%
1	12.219	1310963	74406	94.418
2	13.524	77504	3984	5.582
Total		1388467	78390	100.000



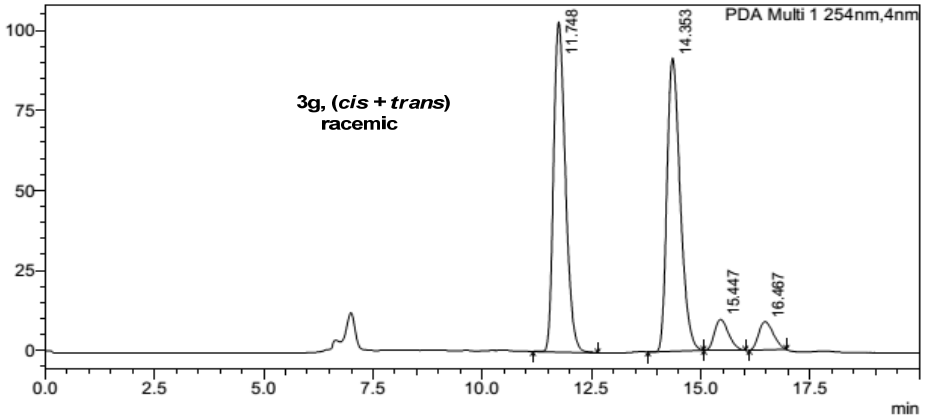
PDA Ch1 254nm

Peak#	Ret. Time	Area	Height	Area%
1	14.477	2579150	148794	50.316
2	15.461	2546712	135884	49.684
Total		5125862	284678	100.000



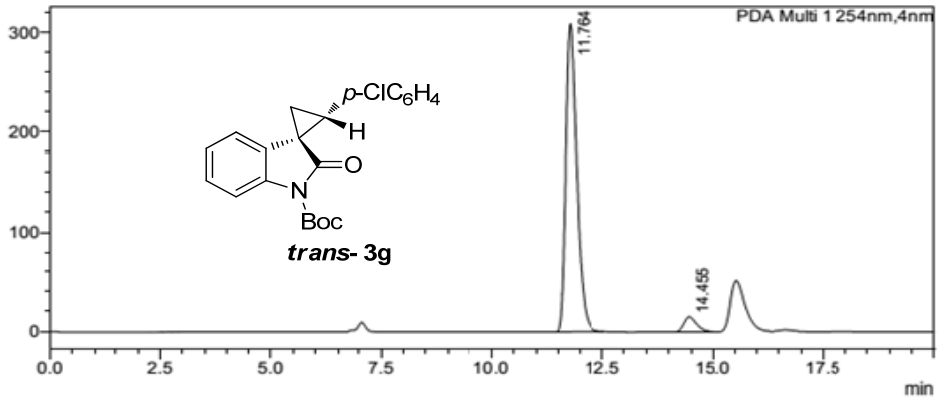
PDA Ch1 254nm

Peak#	Ret. Time	Area	Height	Area%
1	14.646	1486169	81771	93.935
2	15.668	95961	5017	6.065
Total		1582131	86788	100.000



PDA Ch1 254nm

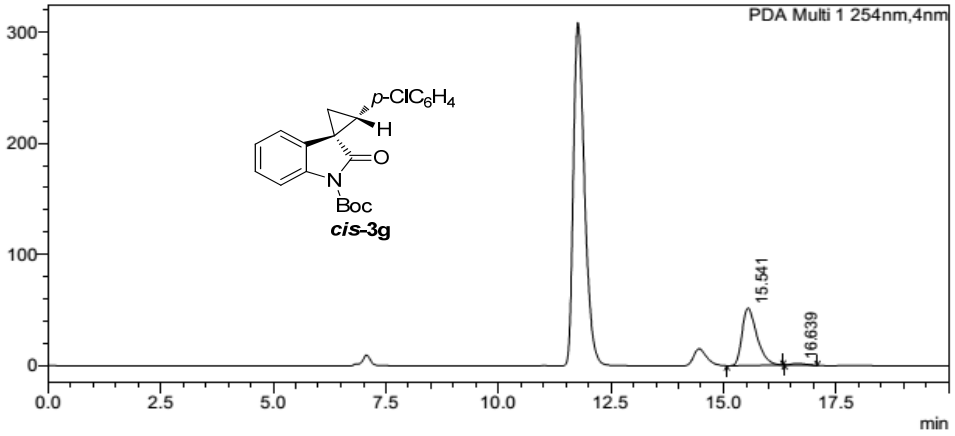
Peak#	Ret. Time	Area	Height	Area%
1	11.748	1945234	103173	45.207
2	14.353	1931682	91634	44.892
3	15.447	217768	9699	5.061
4	16.467	208277	8826	4.840
Total		4302962	213332	100.000



<Peak Table>

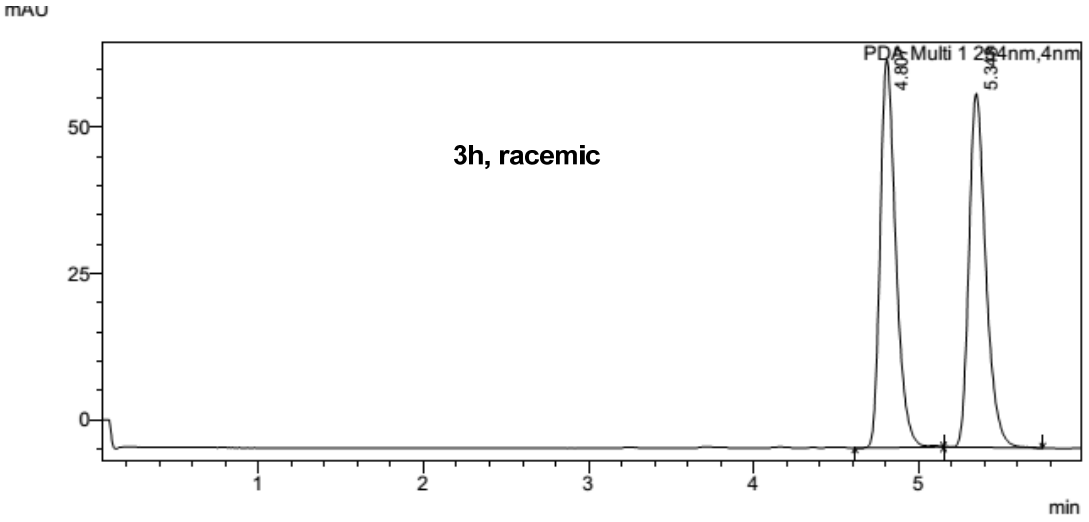
PDA Ch1 254nm

Peak#	Ret. Time	Area	Height	Area%
1	11.764	5332906	309436	94.593
2	14.455	304861	15301	5.407
Total		5637767	324737	100.000



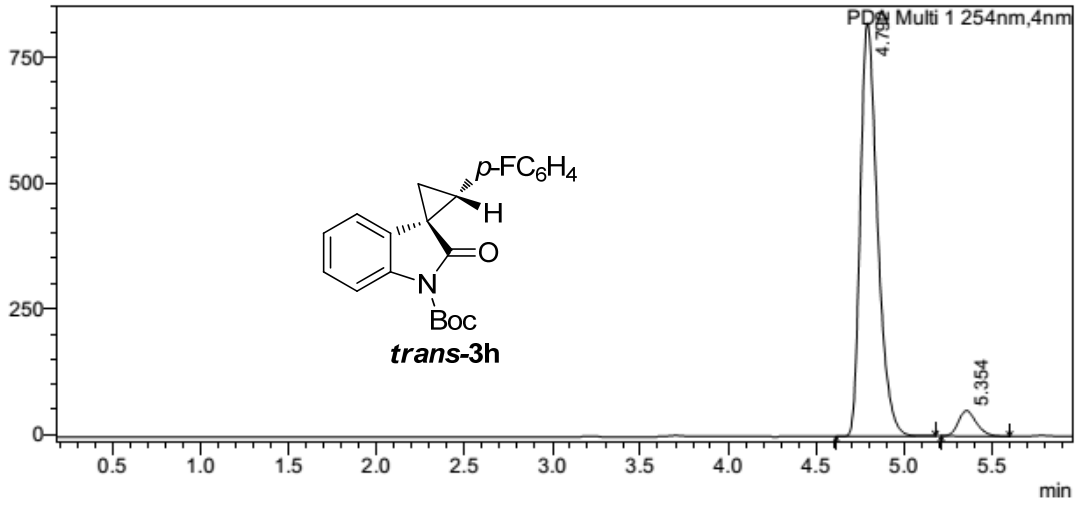
PDA Ch1 254nm

Peak#	Ret. Time	Area	Height	Area%
1	15.541	1186032	51634	97.148
2	16.639	34825	1691	2.852
Total		1220857	53325	100.000



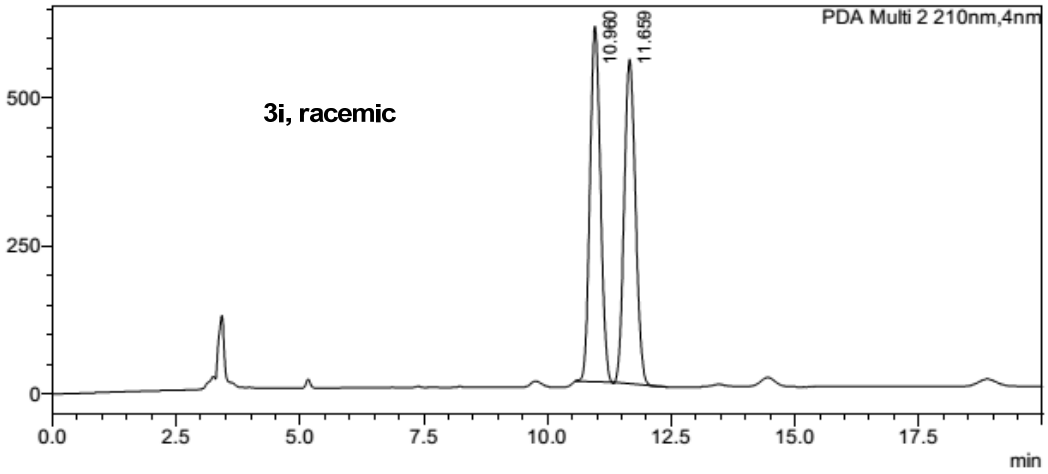
PDA Ch1 254nm

Peak#	Ret. Time	Area	Height	Area%
1	4.807	434936	66418	50.418
2	5.348	427730	60468	49.582
Total		862666	126887	100.000



PDA Ch1 254nm

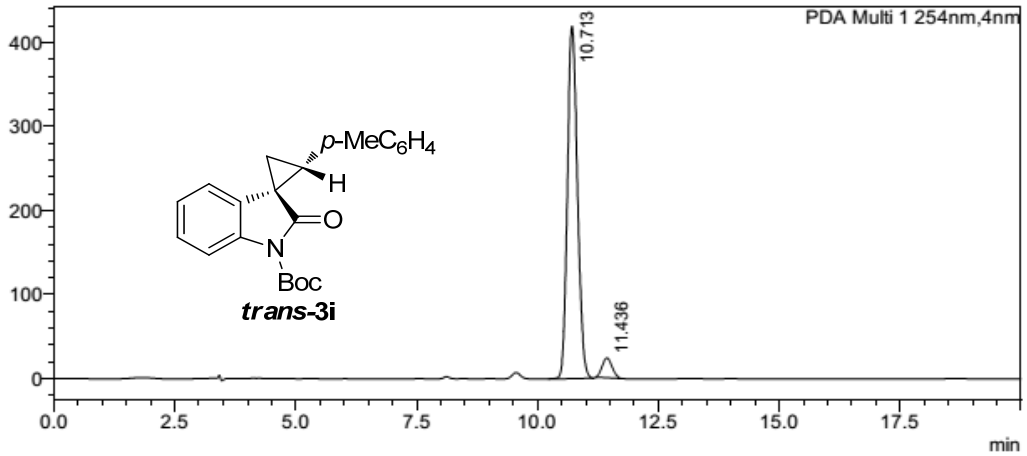
Peak#	Ret. Time	Area	Height	Area%
1	4.792	5604404	820555	93.823
2	5.354	368957	51034	6.177
Total		5973361	871589	100.000



<Peak Table>

PDA Ch2 210nm

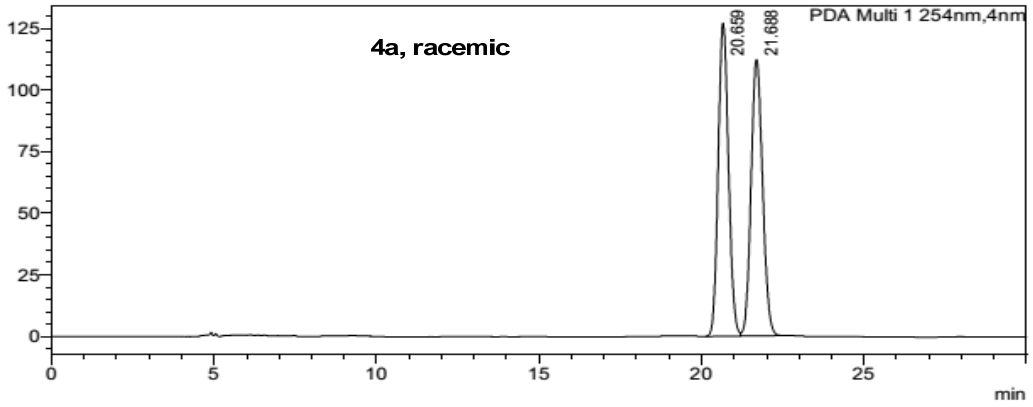
Peak#	Ret. Time	Area	Height	Area%
1	10.960	8809110	600488	50.372
2	11.659	8678898	548128	49.628
Total		17488009	1148615	100.000



<Peak Table>

PDA Ch1 254nm

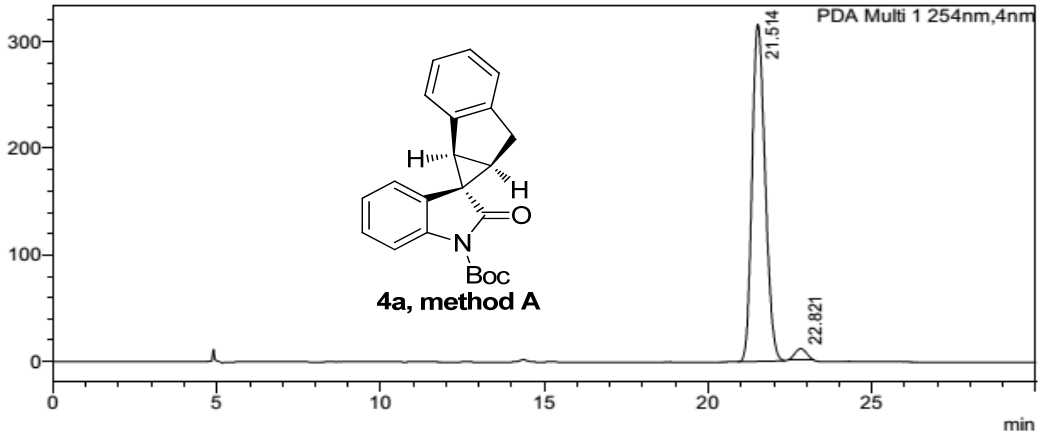
Peak#	Ret. Time	Area	Height	Area%
1	10.713	6044936	419373	94.671
2	11.436	340266	23459	5.329
Total		6385202	442832	100.000



<Peak Table>

PDA Ch1 254nm

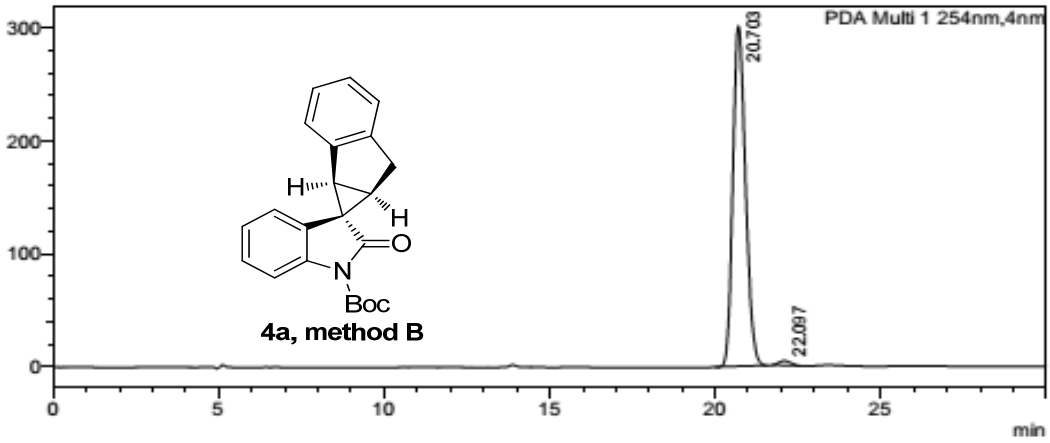
Peak#	Ret. Time	Area	Height	Area%
1	20.659	2798244	126990	50.321
2	21.688	2762528	112002	49.679
Total		5560773	238993	100.000



<Peak Table>

PDA Ch1 254nm

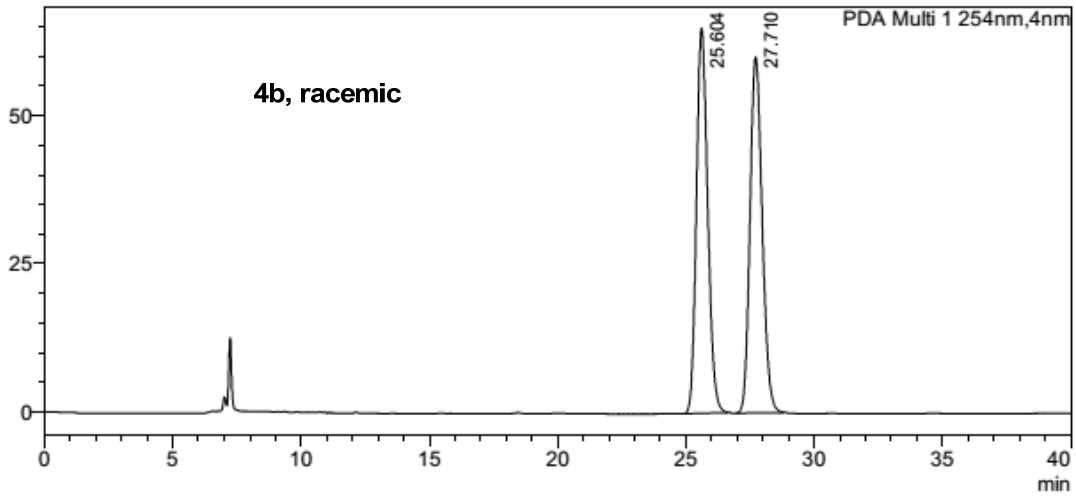
Peak#	Ret. Time	Area	Height	Area%
1	21.514	8757601	316022	96.955
2	22.821	275056	10896	3.045
Total		9032657	326918	100.000



<Peak Table>

PDA Ch1 254nm

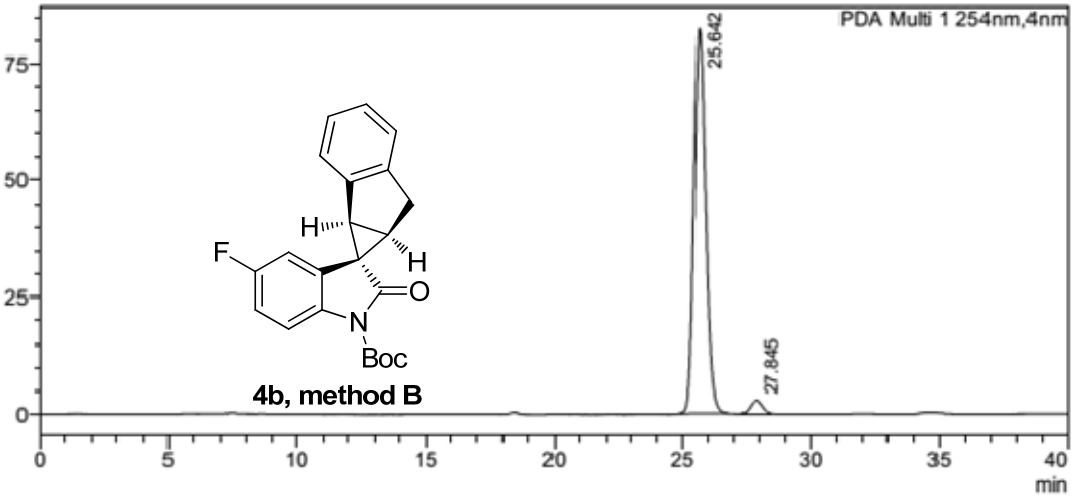
Peak#	Ret. Time	Area	Height	Area%
1	20.703	8013813	300763	98.790
2	22.097	98149	3909	1.210
Total		8111961	304672	100.000



<Peak Table>

PDA Ch1 254nm

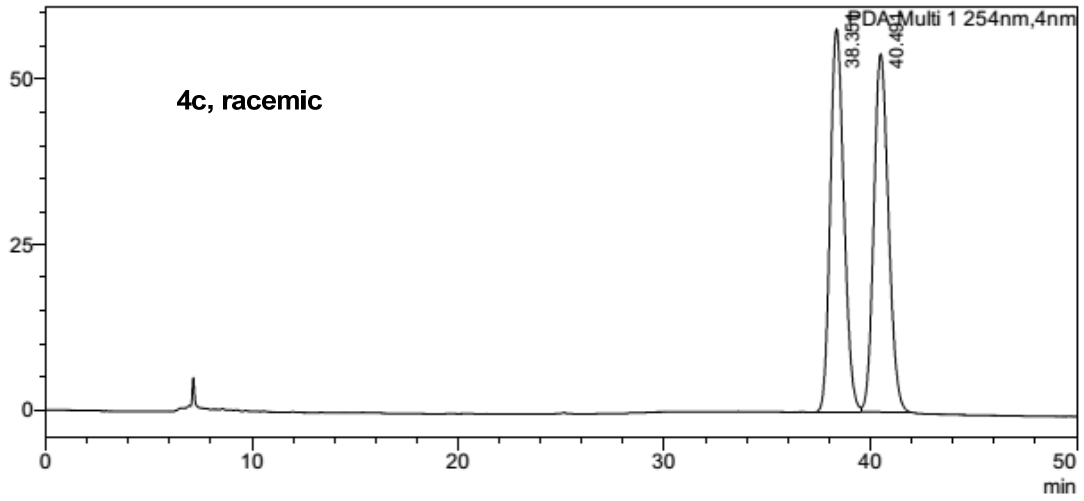
Peak#	Ret. Time	Area	Height	Area%
1	25.604	1981179	64882	49.992
2	27.710	1981800	60014	50.008
Total		3962979	124895	100.000



<Peak Table>

PDA Ch1 254nm

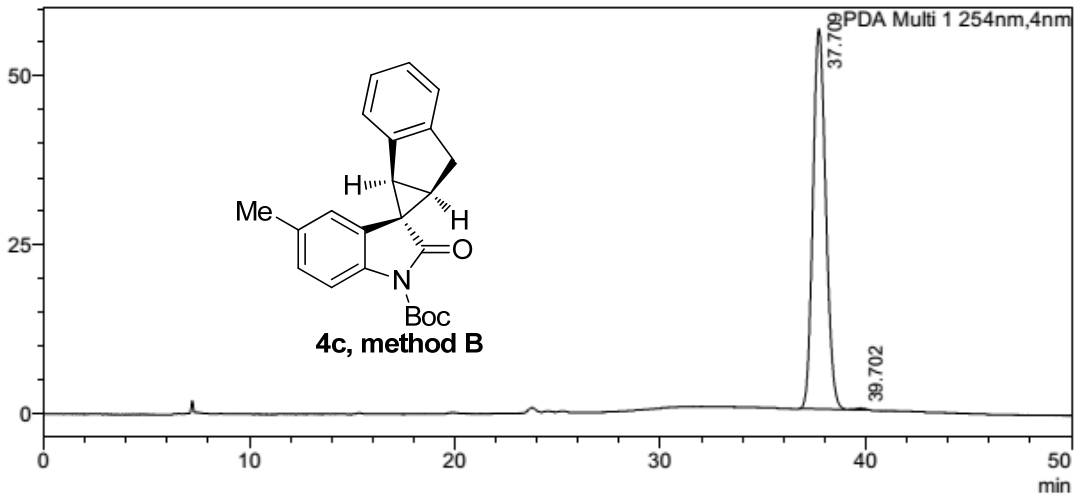
Peak#	Ret. Time	Area	Height	Area%
1	25.642	2543193	82525	96.374
2	27.845	95699	2960	3.626
Total		2638892	85485	100.000



<Peak Table>

PDA Ch1 254nm

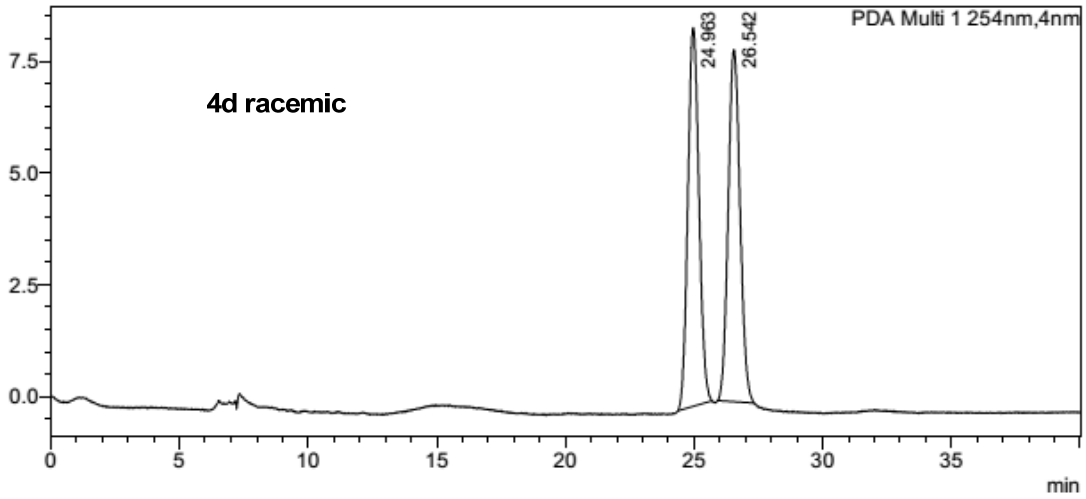
Peak#	Ret. Time	Area	Height	Area%
1	38.351	2674872	57951	49.926
2	40.491	2682760	54212	50.074
Total		5357632	112163	100.000



<Peak Table>

PDA Ch1 254nm

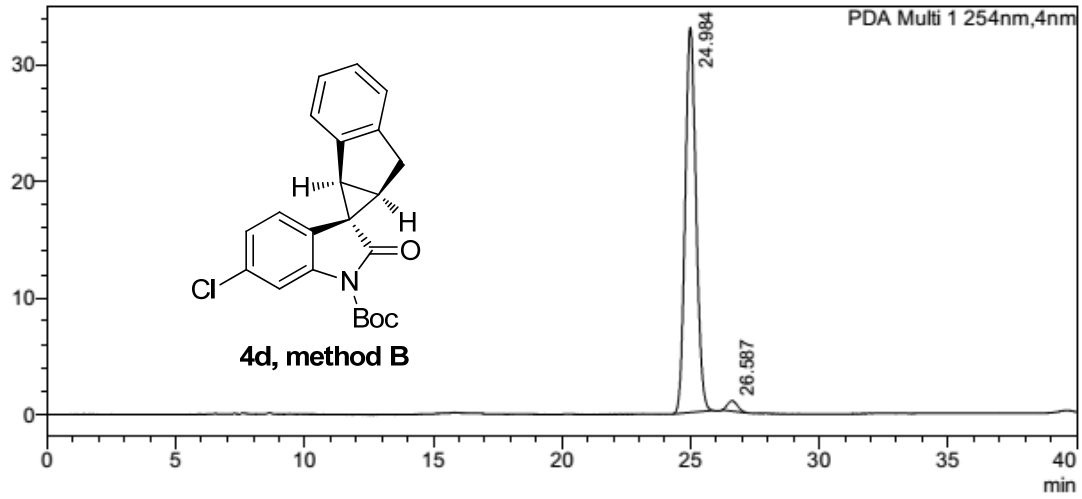
Peak#	Ret. Time	Area	Height	Area%
1	37.709	2437129	56214	99.692
2	39.702	7526	261	0.308
Total		2444655	56475	100.000



<Peak Table>

PDA Ch1 254nm

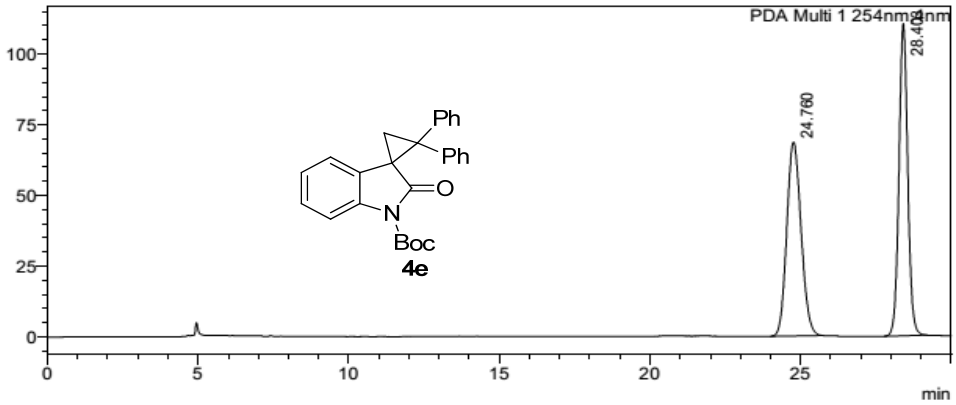
Peak#	Ret. Time	Area	Height	Area%
1	24.963	253824	8462	50.292
2	26.542	250877	7867	49.708
Total		504701	16328	100.000



<Peak Table>

PDA Ch1 254nm

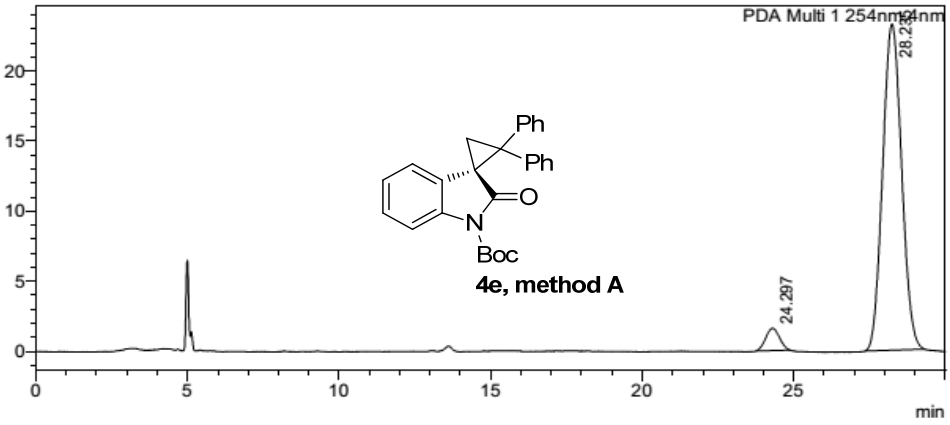
Peak#	Ret. Time	Area	Height	Area%
1	24.984	966882	33037	97.671
2	26.587	23052	871	2.329
Total		989934	33907	100.000



<Peak Table>

PDA Ch1 254nm

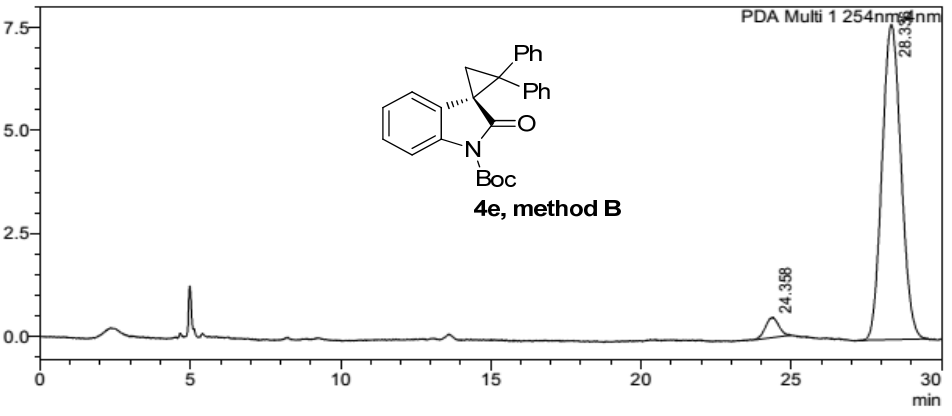
Peak#	Ret. Time	Area	Height	Area%
1	24.760	2260105	68459	50.280
2	28.403	2234919	110338	49.720
Total		4495024	178797	100.000



<Peak Table>

PDA Ch1 254nm

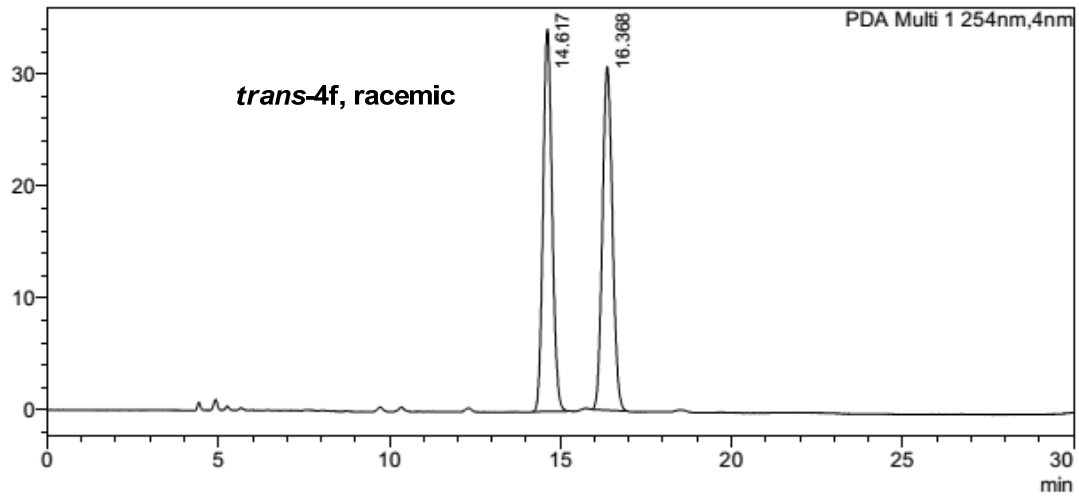
Peak#	Ret. Time	Area	Height	Area%
1	24.297	49884	1575	4.708
2	28.235	1009679	23250	95.292
Total		1059563	24825	100.000



<Peak Table>

PDA Ch1 254nm

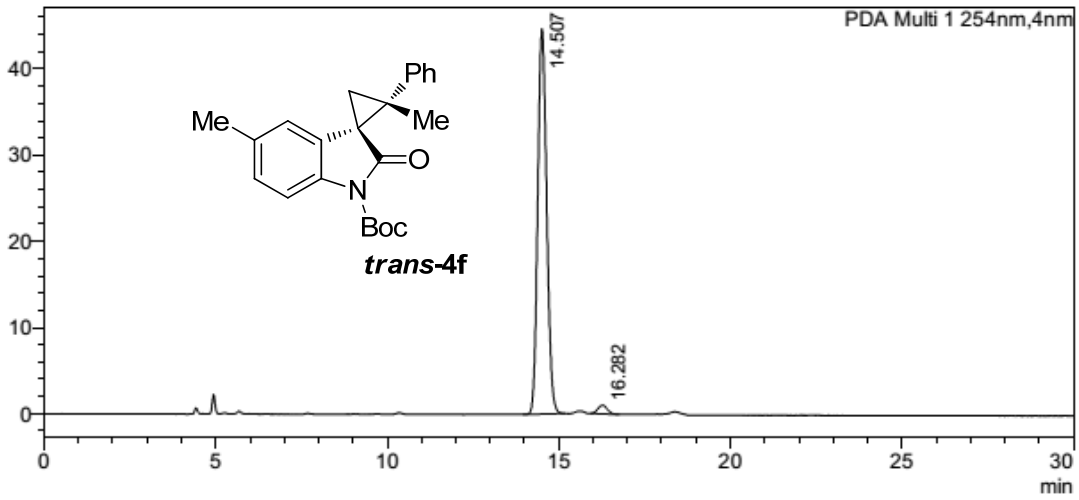
Peak#	Ret. Time	Area	Height	Area%
1	24.358	14889	492	4.241
2	28.336	336151	7652	95.759
Total		351040	8143	100.000



<Peak Table>

PDA Ch1 254nm

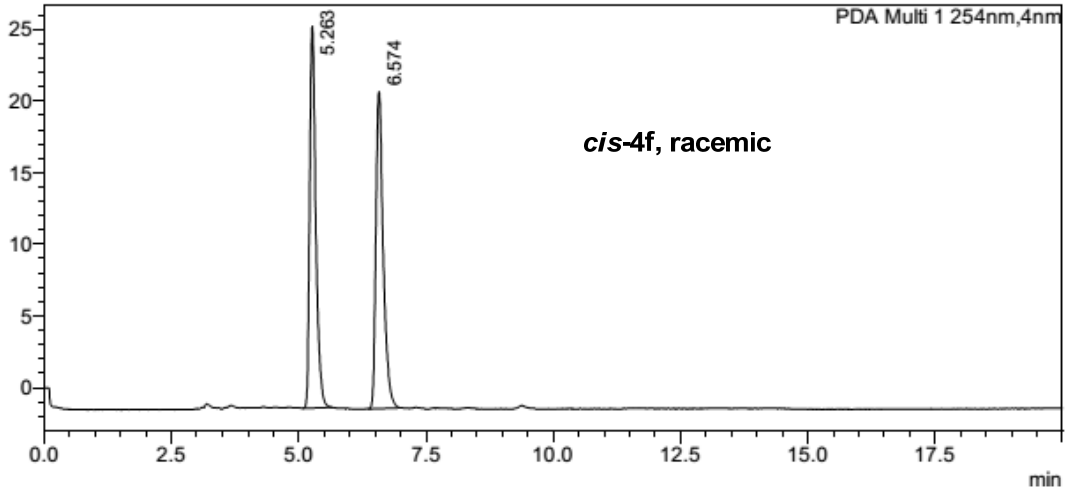
Peak#	Ret. Time	Area	Height	Area%
1	14.617	628043	34159	50.212
2	16.368	622752	30717	49.788
Total		1250795	64877	100.000



<Peak Table>

PDA Ch1 254nm

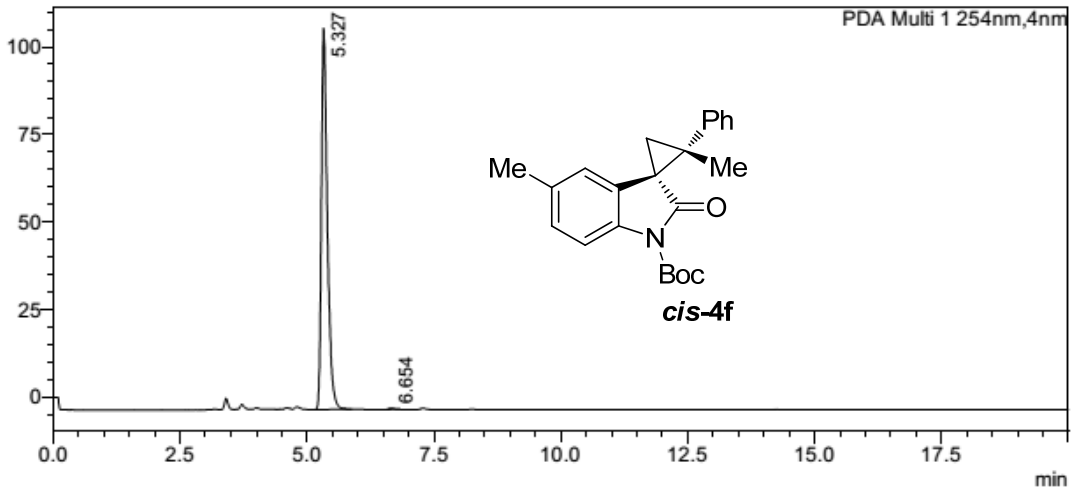
Peak#	Ret. Time	Area	Height	Area%
1	14.507	810512	44699	97.608
2	16.282	19867	1029	2.392
Total		830378	45728	100.000



<Peak Table>

PDA Ch1 254nm

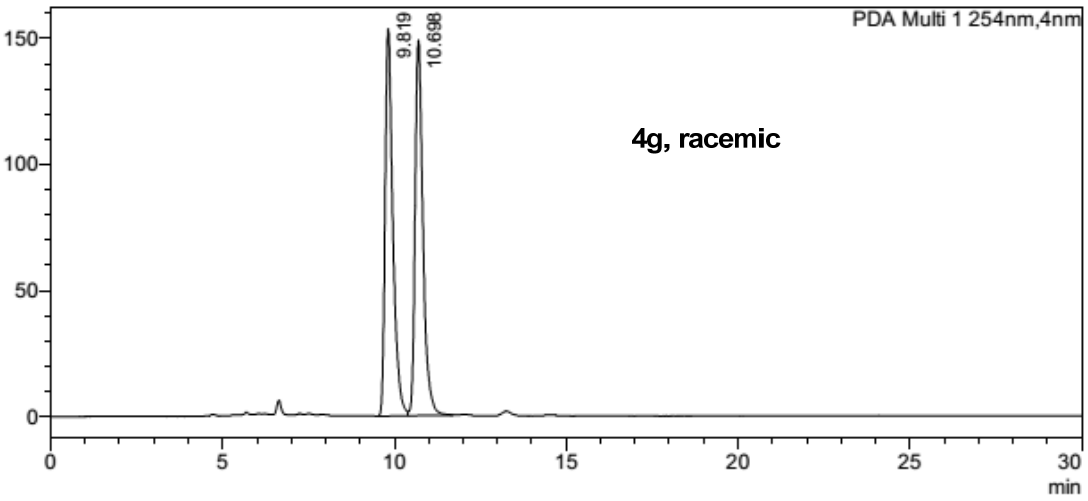
Peak#	Ret. Time	Area	Height	Area%
1	5.263	221006	26631	49.951
2	6.574	221435	22114	50.049
Total		442441	48745	100.000



<Peak Table>

PDA Ch1 254nm

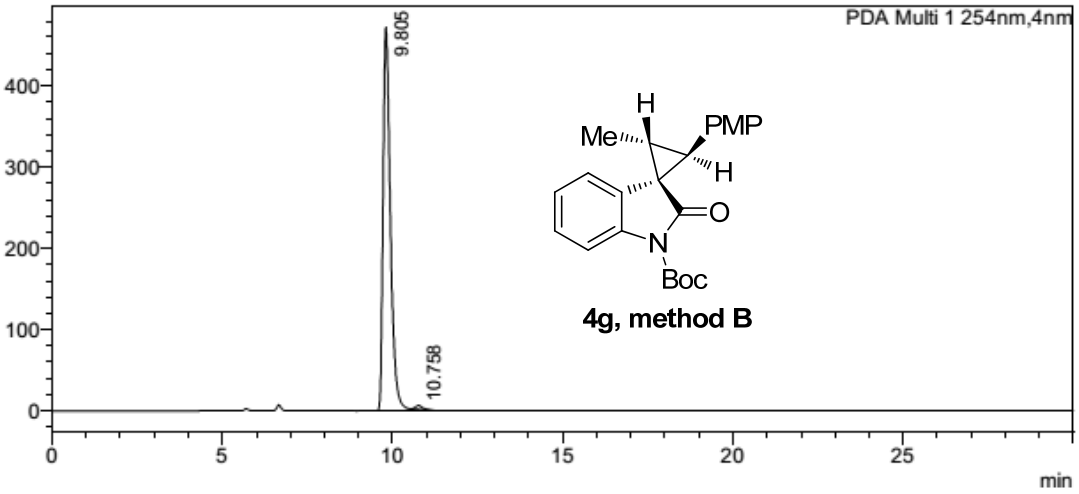
Peak#	Ret. Time	Area	Height	Area%
1	5.327	897445	108837	99.679
2	6.654	2890	355	0.321
Total		900335	109193	100.000



<Peak Table>

PDA Ch1 254nm

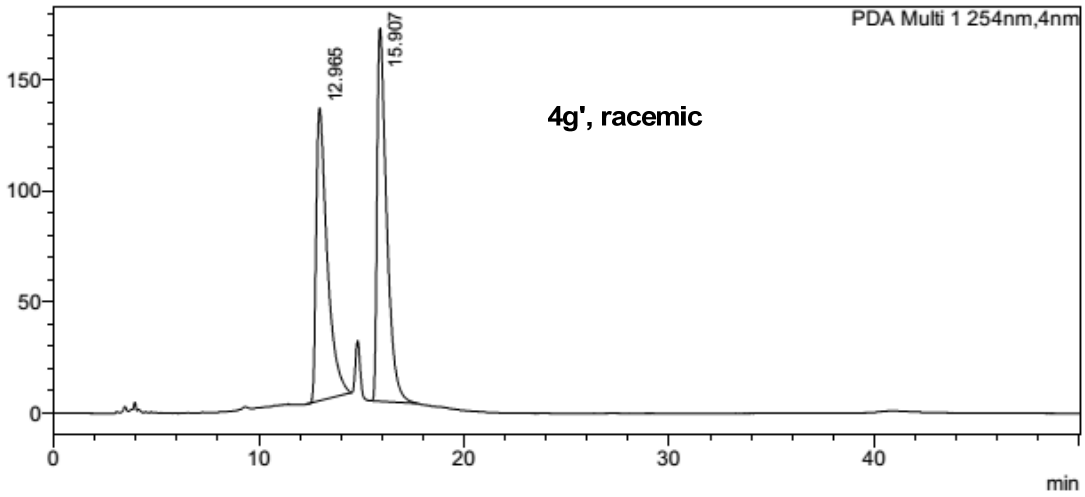
Peak#	Ret. Time	Area	Height	Area%
1	9.819	2416522	153605	50.361
2	10.698	2381911	148959	49.639
Total		4798433	302565	100.000



<Peak Table>

PDA Ch1 254nm

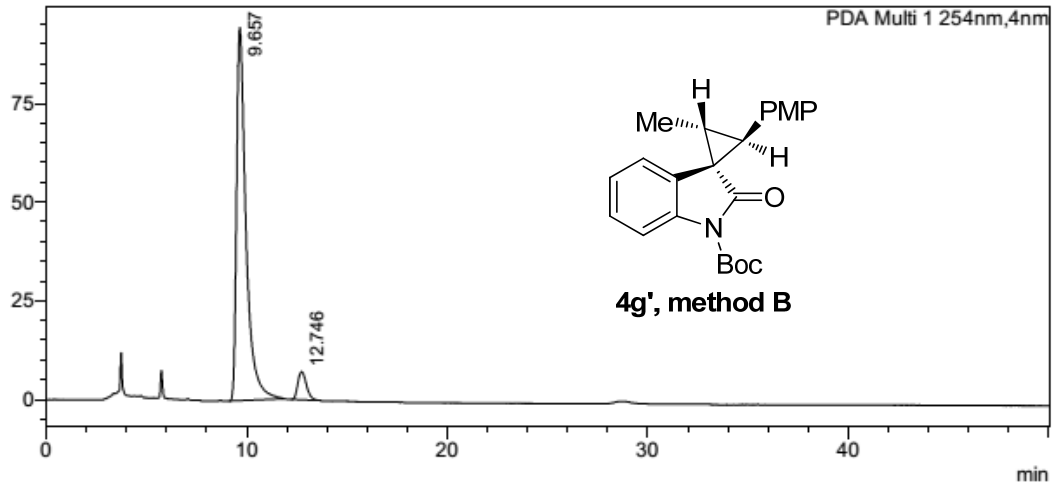
Peak#	Ret. Time	Area	Height	Area%
1	9.805	7091466	472342	99.064
2	10.758	66989	4599	0.936
Total		7158455	476941	100.000



<Peak Table>

PDA Ch1 254nm

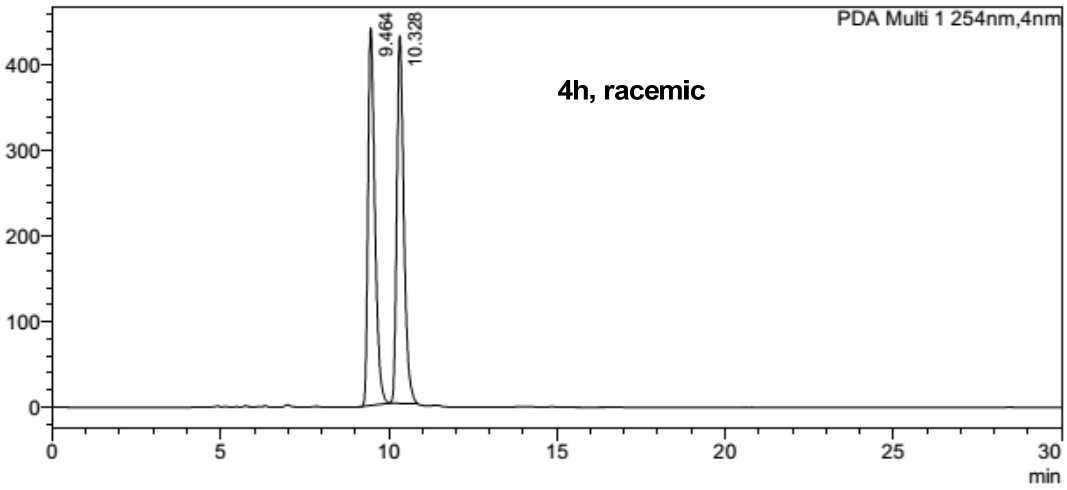
Peak#	Ret. Time	Area	Height	Area%
1	12.965	4963056	132124	47.642
2	15.907	5454353	168288	52.358
Total		10417410	300412	100.000



<Peak Table>

PDA Ch1 254nm

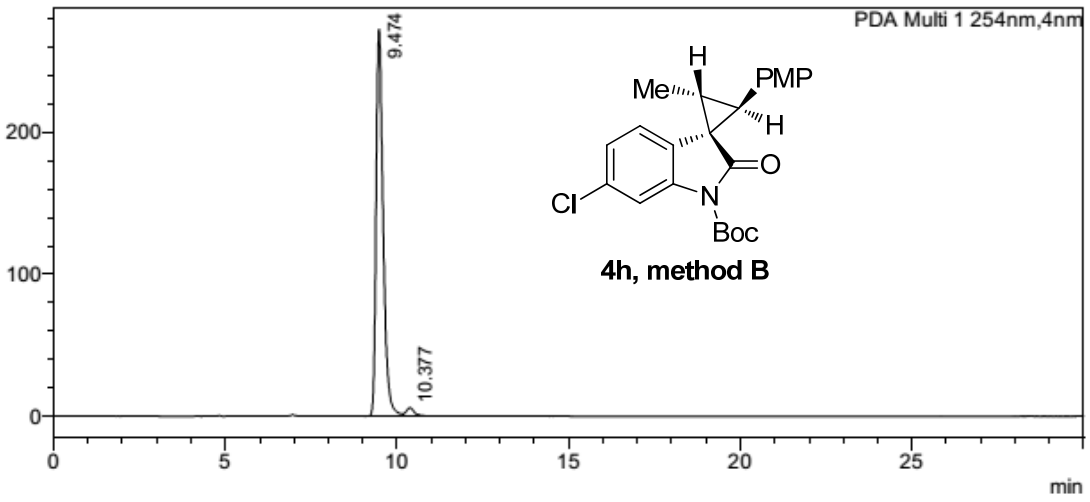
Peak#	Ret. Time	Area	Height	Area%
1	9.657	3098420	94480	93.605
2	12.746	211683	7146	6.395
Total		3310104	101625	100.000



<Peak Table>

PDA Ch1 254nm

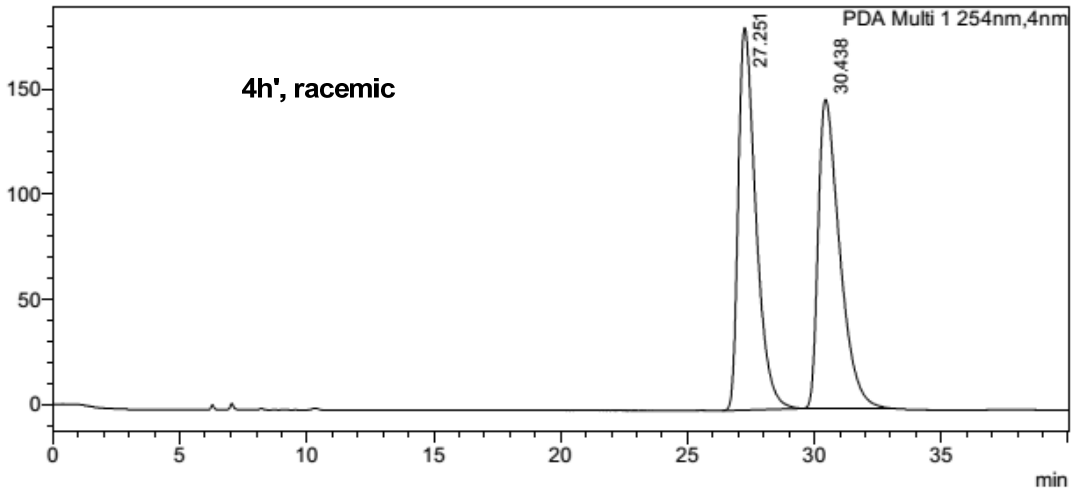
Peak#	Ret. Time	Area	Height	Area%
1	9.464	6288070	442208	50.136
2	10.328	6254065	430692	49.864
Total		12542135	872900	100.000



<Peak Table>

PDA Ch1 254nm

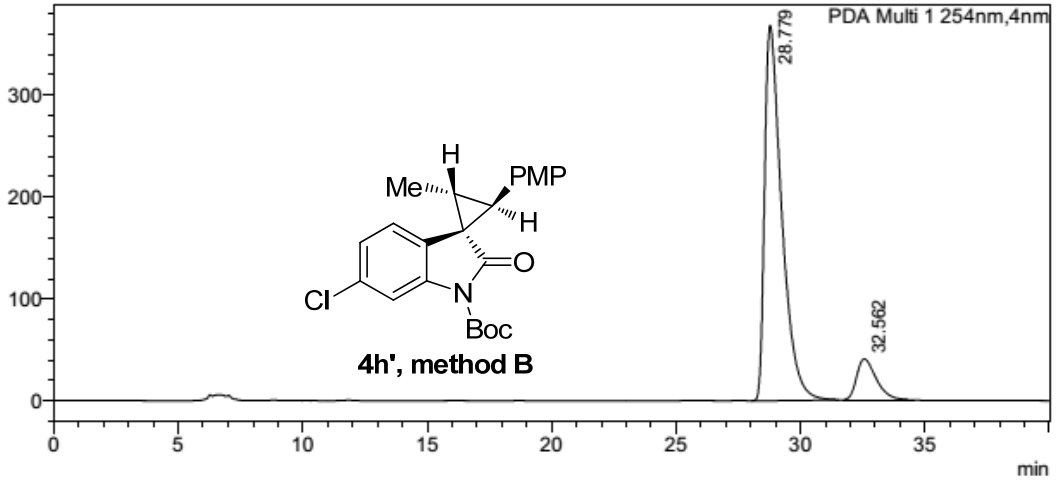
Peak#	Ret. Time	Area	Height	Area%
1	9.474	3875211	272731	97.619
2	10.377	94511	5817	2.381
Total		3969722	278548	100.000



<Peak Table>

PDA Ch1 254nm

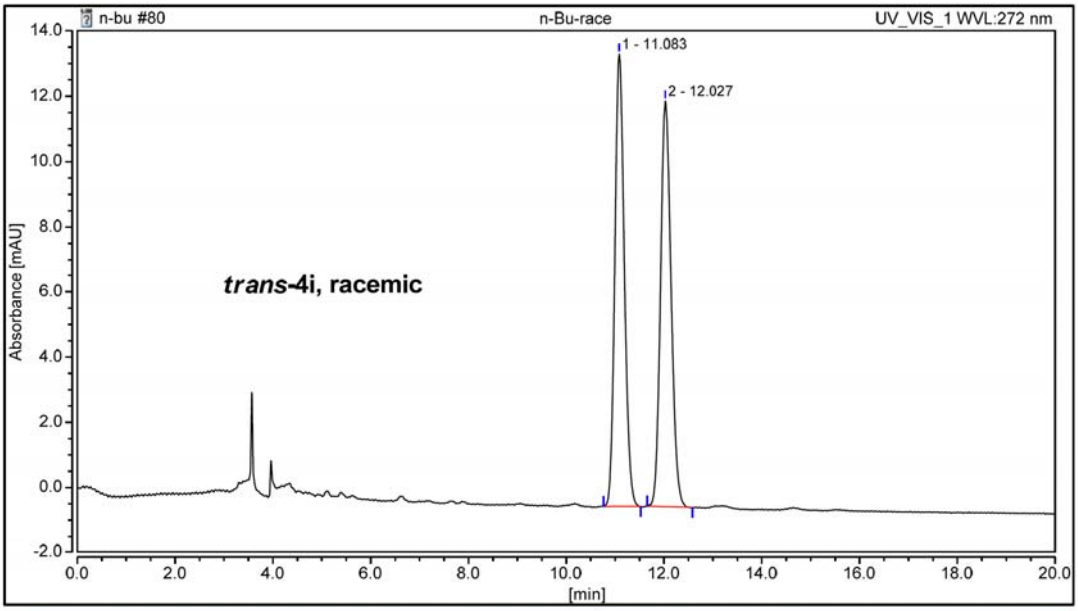
Peak#	Ret. Time	Area	Height	Area%
1	27.251	8785233	181599	50.317
2	30.438	8674646	146941	49.683
Total		17459879	328540	100.000



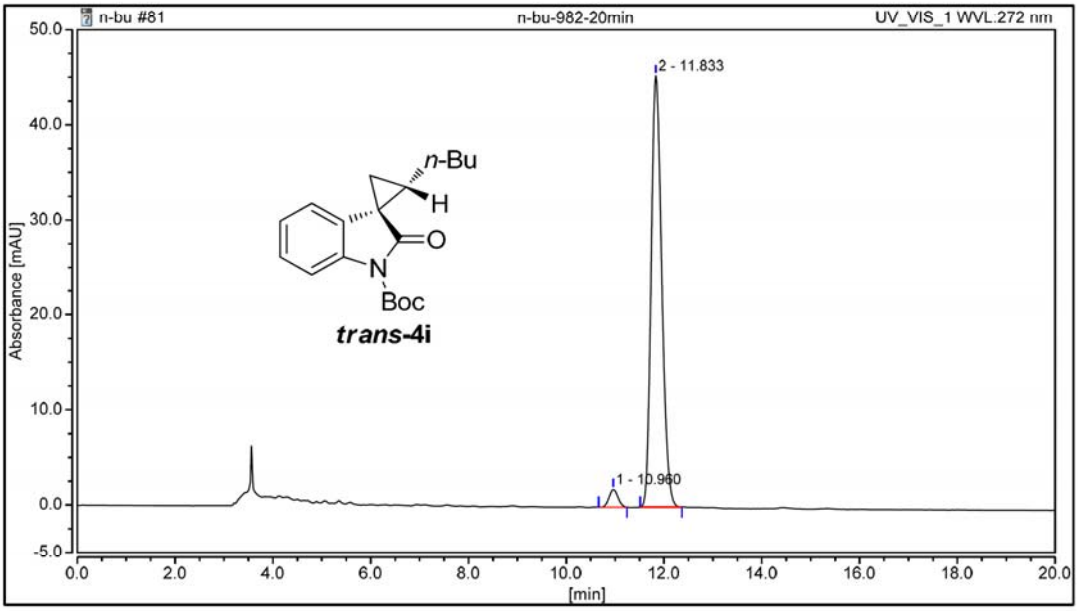
<Peak Table>

PDA Ch1 254nm

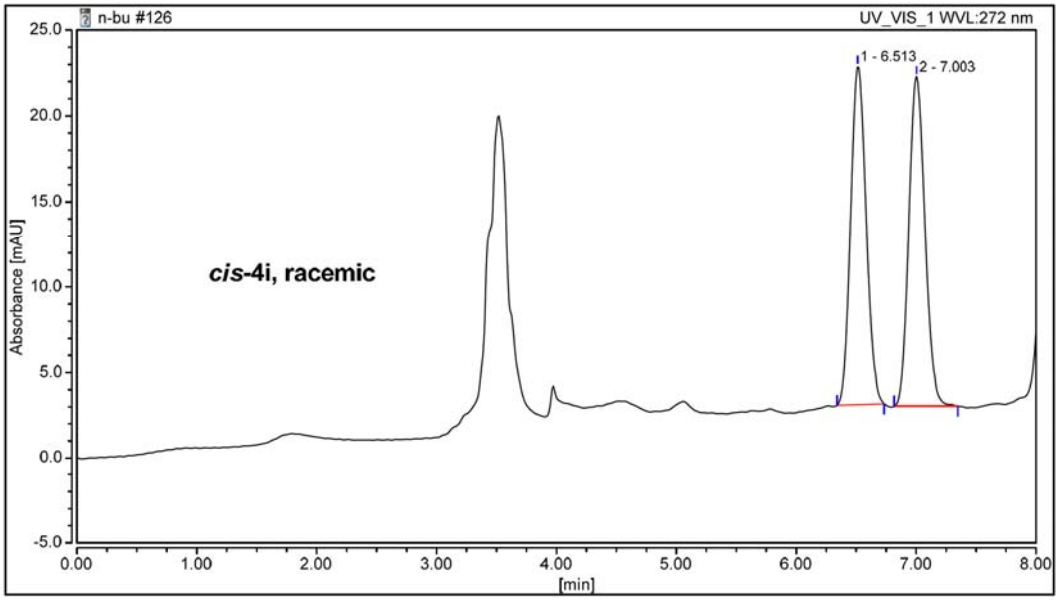
Peak#	Ret. Time	Area	Height	Area%
1	28.779	17677619	368871	88.838
2	32.562	2221111	40298	11.162
Total		19898729	409170	100.000



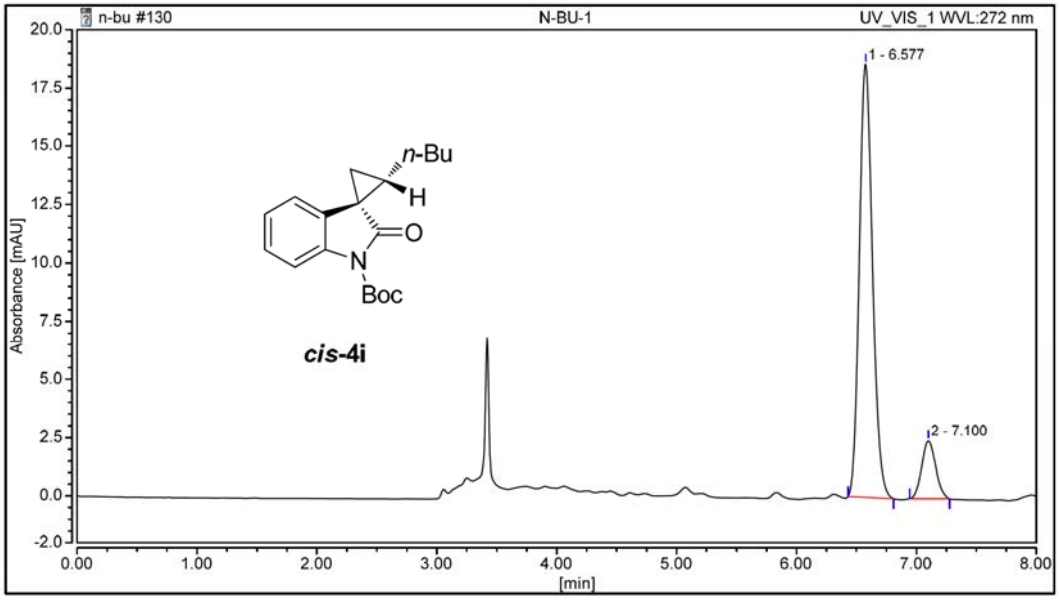
No.	Peak Name	Ret.Time min	Area mAU*min	Height mAU	Rel.Area %	Rel.Height %
1		11.083	3.112	13.869	50.00	52.71
2		12.027	3.113	12.442	50.00	47.29
Total			**6.225**	**26.311**	**100.00**	**100.00**



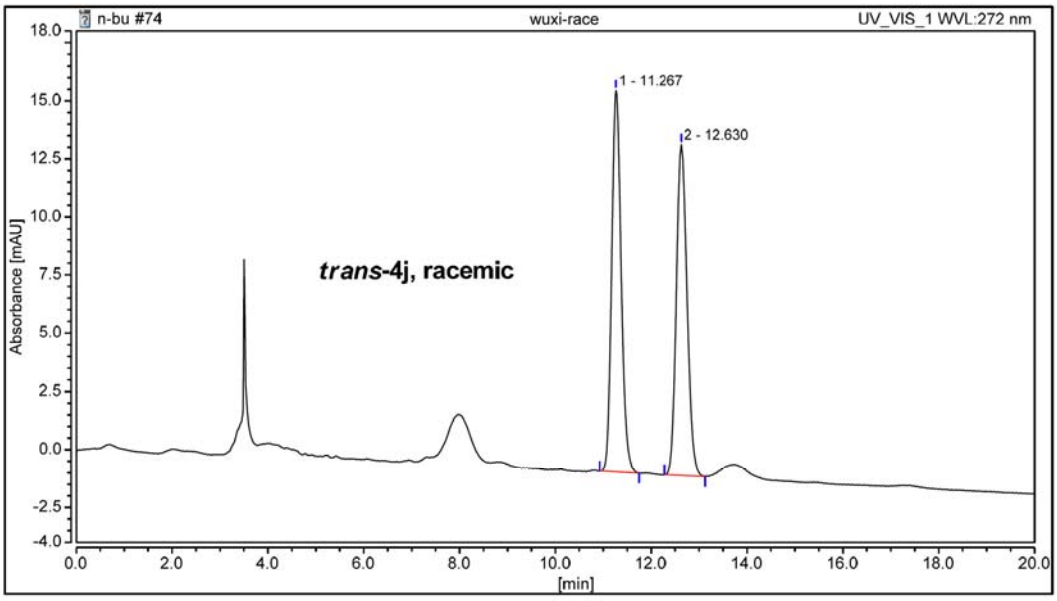
No.	Peak Name	Ret.Time min	Area mAU*min	Height mAU	Rel.Area %	Rel.Height %
1		10.960	0.397	1.882	3.40	3.98
2		11.833	11.307	45.390	96.60	96.02
Total			**11.705**	**47.272**	**100.00**	**100.00**



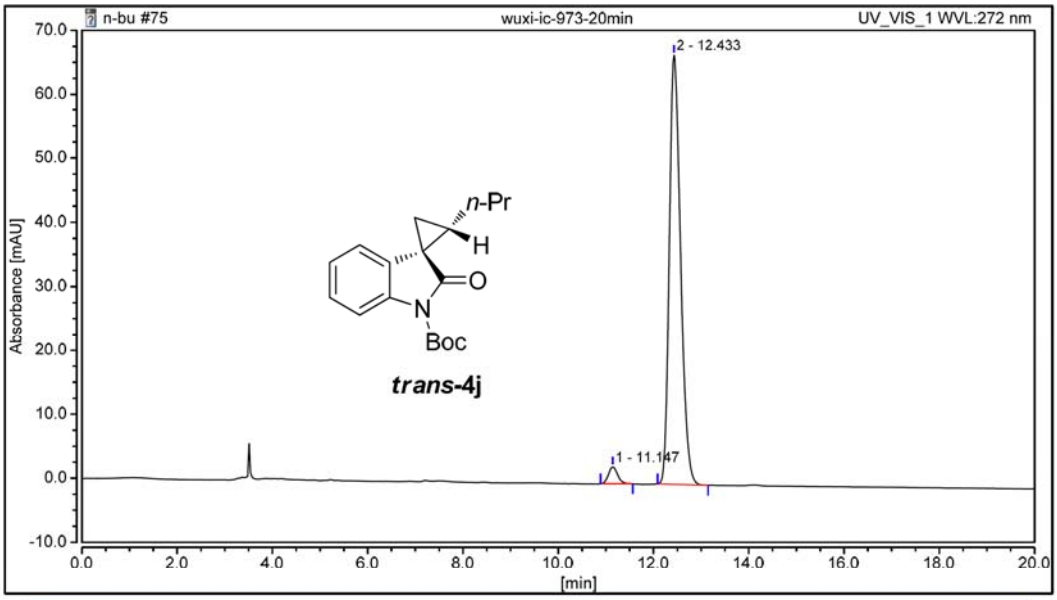
No.	Peak Name	Ret.Time min	Area mAU*min	Height mAU	Rel.Area %	Rel.Height %
1		6.513	2.956	19.782	49.58	50.65
2		7.003	3.005	19.272	50.42	49.35
Total			**5.961**	**39.054**	**100.00**	**100.00**



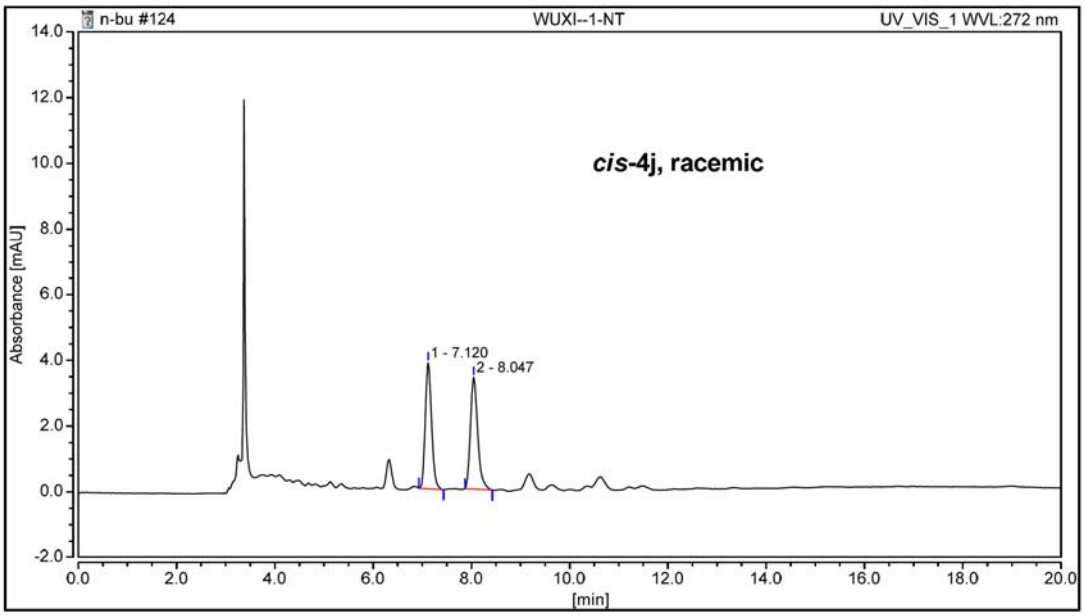
No.	Peak Name	Ret.Time min	Area mAU*min	Height mAU	Rel.Area %	Rel.Height %
1		6.577	2.364	18.585	87.63	88.18
2		7.100	0.334	2.491	12.37	11.82
Total			**2.697**	**21.075**	**100.00**	**100.00**



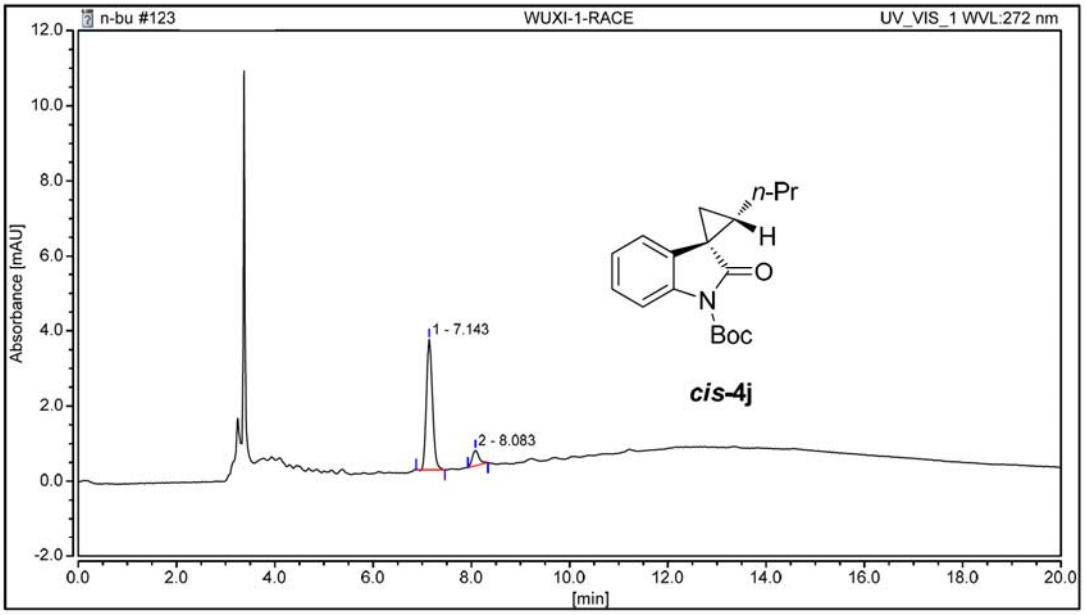
No.	Peak Name	Ret.Time min	Area mAU*min	Height mAU	Rel.Area %	Rel.Height %
1		11.267	3.732	16.407	50.40	53.53
2		12.630	3.673	14.244	49.60	46.47
Total			**7.405**	**30.651**	**100.00**	**100.00**



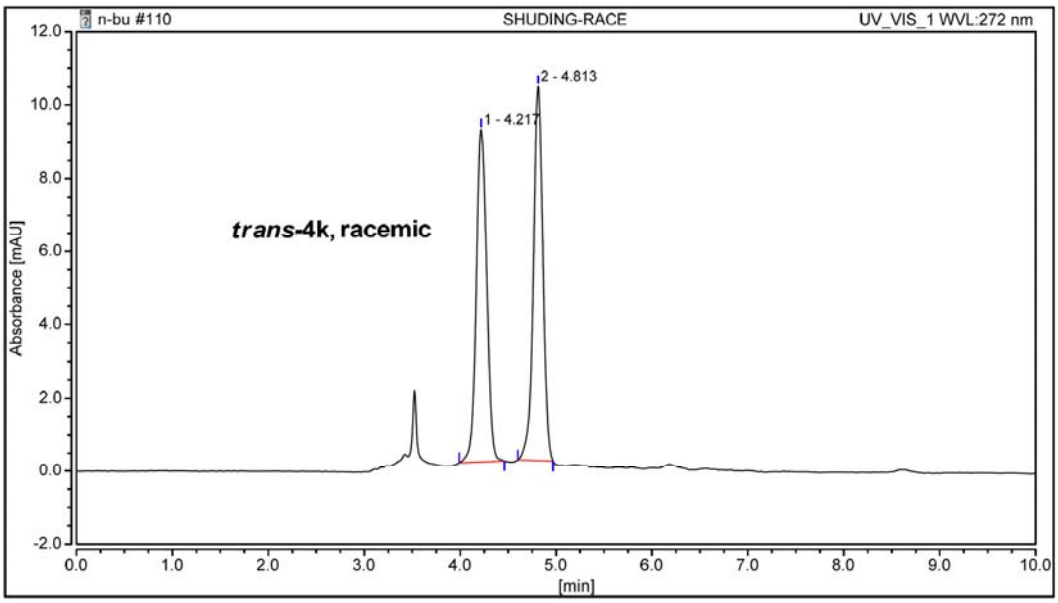
No.	Peak Name	Ret.Time min	Area mAU*min	Height mAU	Rel.Area %	Rel.Height %
1		11.147	0.596	2.635	3.15	3.78
2		12.433	18.331	67.097	96.85	96.22
Total			**18.927**	**69.732**	**100.00**	**100.00**



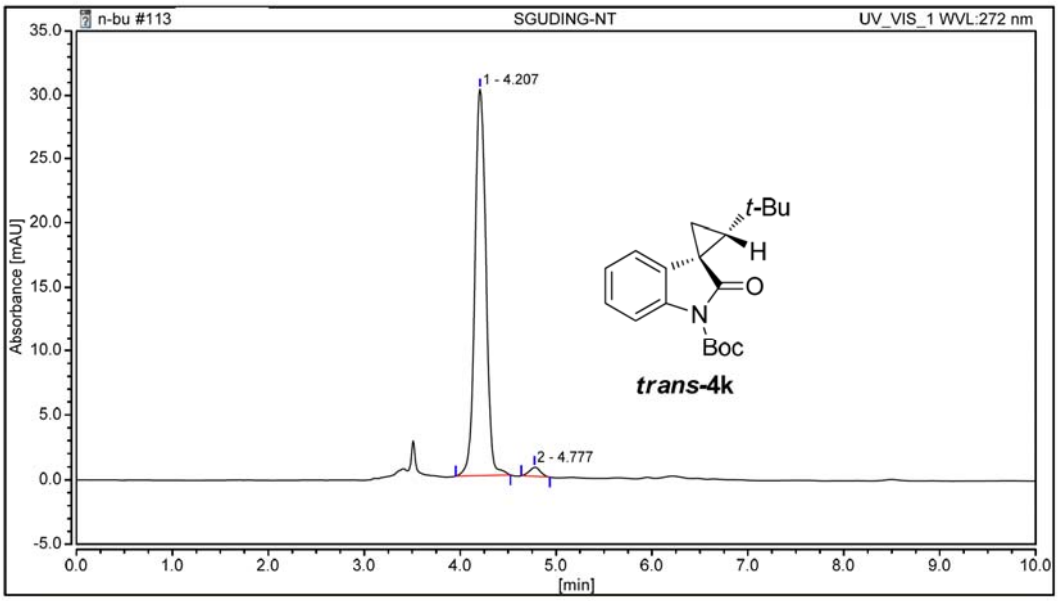
No.	Peak Name	Ret.Time min	Area mAU*min	Height mAU	Rel.Area %	Rel.Height %
1		7.120	0.564	3.831	49.27	53.01
2		8.047	0.581	3.396	50.73	46.99
Total			**1.144**	**7.227**	**100.00**	**100.00**



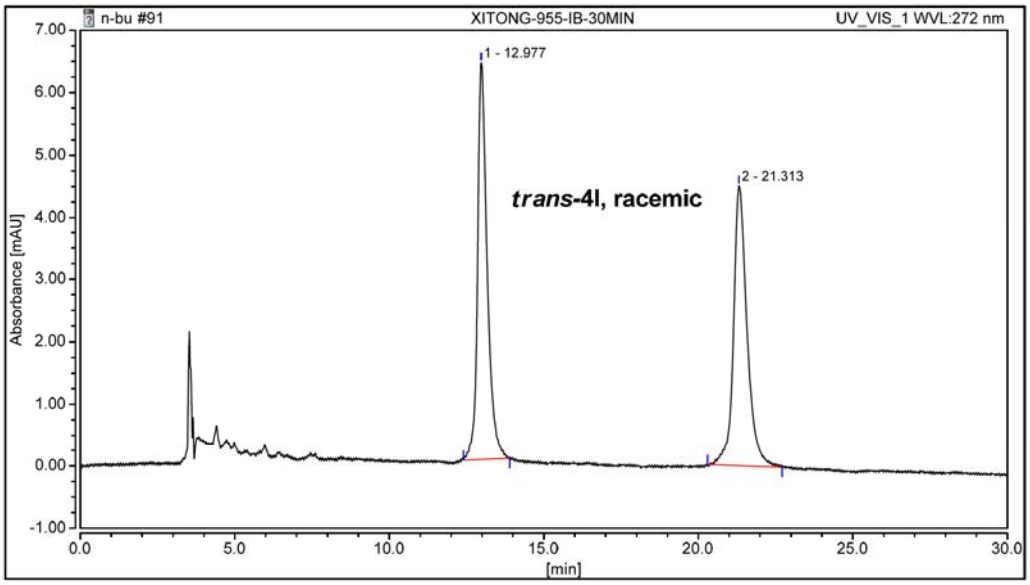
No.	Peak Name	Ret.Time min	Area mAU*min	Height mAU	Rel.Area %	Rel.Height %
1		7.143	0.491	3.468	88.38	89.48
2		8.083	0.064	0.408	11.62	10.52
Total			**0.555**	**3.876**	**100.00**	**100.00**



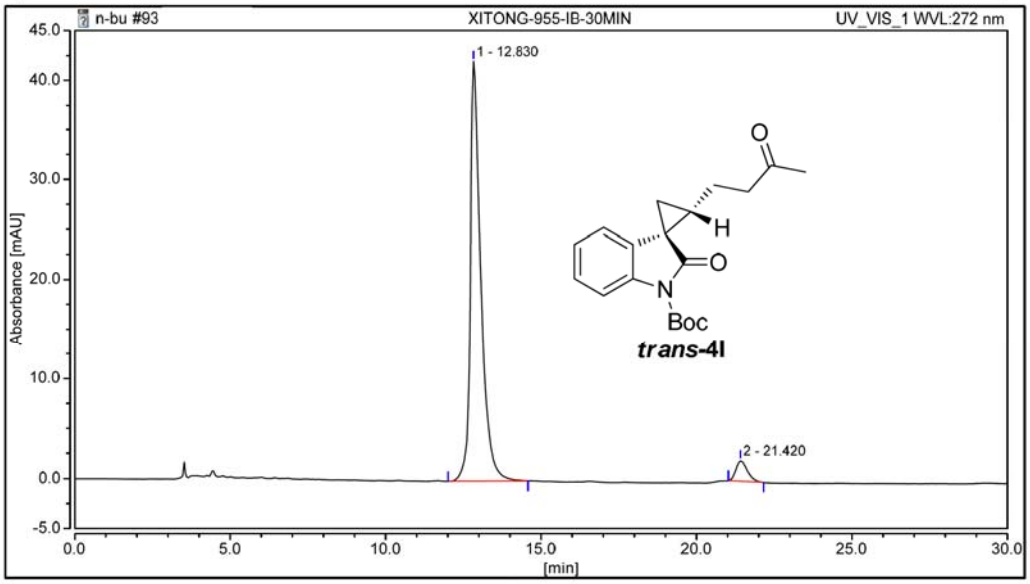
No.	Peak Name	Ret.Time min	Area mAU*min	Height mAU	Rel.Area %	Rel.Height %
1		4.217	1.146	9.090	49.42	47.05
2		4.813	1.173	10.229	50.58	52.95
Total			**2.319**	**19.319**	**100.00**	**100.00**



No.	Peak Name	Ret.Time min	Area mAU*min	Height mAU	Rel.Area %	Rel.Height %
1		4.207	3.999	30.154	97.74	97.72
2		4.777	0.093	0.705	2.26	2.28
Total			**4.092**	**30.859**	**100.00**	**100.00**

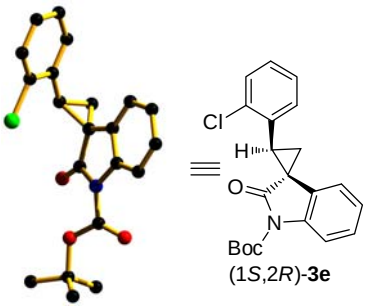


No.	Peak Name	Ret.Time min	Area mAU*min	Height mAU	Rel.Area %	Rel.Height %
1		12.977	2.300	6.378	50.08	58.57
2		21.313	2.293	4.511	49.92	41.43
Total			**4.593**	**10.889**	**100.00**	**100.00**



No.	Peak Name	Ret.Time min	Area mAU*min	Height mAU	Rel.Area %	Rel.Height %
1		12.830	16.547	42.198	95.03	95.37
2		21.420	0.866	2.048	4.97	4.63
Total			**17.413**	**44.245**	**100.00**	**100.00**

Single-crystal X-ray diffraction of 3e

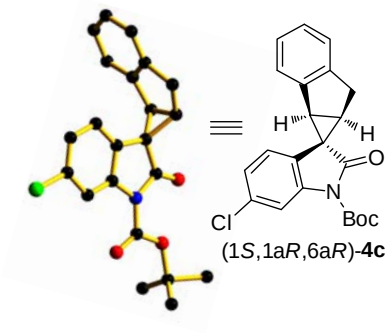


(1*S*,2*R*)-**3e**

Datablock: g151231a

```
Bond precision:  C-C = 0.0041 A          Wavelength=1.54184

Cell:            a=11.3609(3)     b=11.3606(3)     c=14.5605(4)
                 alpha=90         beta=90          gamma=90
Temperature:     293 K

                 Calculated                Reported
Volume           1879.28(9)                1879.28(9)
Space group      P 21 21 21                P 21 21 21
Hall group       P 2ac 2ab                 P 2ac 2ab
Moiety formula   C21 H20 Cl N O3           C21 H20 Cl N O3
Sum formula      C21 H20 Cl N O3           C21 H20 Cl N O3
Mr               369.83                    369.83
Dx,g cm-3        1.307                     1.307
Z                4                         4
Mu (mm-1)        1.964                     1.964
F000             776.0                     776.0
F000'            779.60
h,k,lmax         14,14,18                  14,13,18
Nref             3776[ 2154]               3725
Tmin,Tmax        0.475,0.745               0.156,1.000
Tmin'            0.359

Correction method= # Reported T Limits: Tmin=0.156 Tmax=1.000
AbsCorr = MULTI-SCAN

Data completeness= 1.73/0.99      Theta(max)= 73.610

R(reflections)= 0.0478( 3458)     wR2(reflections)= 0.1361( 3725)

S = 1.042                 Npar= 239
```

Single-crystal X-ray diffraction of 4c

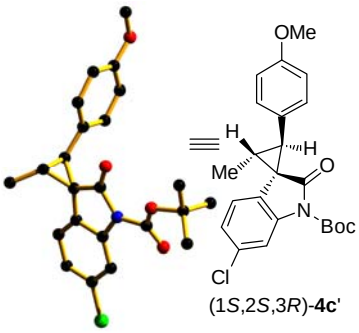


(1*S*,1a*R*,6a*R*)-**4c**

Datablock: g151224a

```
Bond precision:  C-C = 0.0041 A          Wavelength=1.54184

Cell:             a=7.3670(3)     b=12.1881(3)     c=21.7372(5)
                  alpha=90        beta=90          gamma=90
Temperature:      293 K

                  Calculated                Reported
Volume            1951.78(10)               1951.79(10)
Space group       P 21 21 21                P 21 21 21
Hall group        P 2ac 2ab                 P 2ac 2ab
Moiety formula    C22 H19 Cl N O3           C22 H19 Cl N O3
Sum formula       C22 H19 Cl N O3           C22 H19 Cl N O3
Mr                380.83                    380.83
Dx,g cm-3         1.296                     1.296
Z                 4                         4
Mu (mm-1)         1.909                     1.909
F000              796.0                     796.0
F000'             799.67
h,k,lmax          9,15,27                   9,15,27
Nref              3941[ 2271]               3871
Tmin,Tmax         0.592,0.751               0.425,1.000
Tmin'             0.488

Correction method= # Reported T Limits: Tmin=0.425 Tmax=1.000
AbsCorr = MULTI-SCAN

Data completeness= 1.70/0.98      Theta(max)= 73.670

R(reflections)= 0.0474( 3257)     wR2(reflections)= 0.1482( 3871)

S = 1.066                 Npar= 277
```

Single-crystal X-ray diffraction of 4h



(1*S*,2*S*,3*R*)-**4c'**

Datablock: g160416c

```
Bond precision:  C-C = 0.0046 A          Wavelength=0.71073

Cell:            a=9.0330(5)     b=9.8048(7)         c=12.0153(7)
                 alpha=90        beta=90.156(5)      gamma=90
Temperature:     223 K

                 Calculated                Reported
Volume           1064.15(11)               1064.15(12)
Space group      P 21                      P 1 21 1
Hall group       P 2yb                     P 2yb
Moiety formula   C23 H24 Cl N O4           C23 H24 Cl N O4
Sum formula      C23 H24 Cl N O4           C23 H24 Cl N O4
Mr               413.88                    413.88
Dx,g cm-3        1.292                     1.292
Z                2                         2
Mu (mm-1)        0.208                     0.208
F000             436.0                     436.0
F000'            436.49
h,k,lmax         10,11,14                  10,11,14
Nref             3764[ 2002]               3006
Tmin,Tmax        0.928,0.940               0.786,1.000
Tmin'            0.901

Correction method= # Reported T Limits: Tmin=0.786 Tmax=1.000
AbsCorr = MULTI-SCAN

Data completeness= 1.50/0.80      Theta(max)= 24.992

R(reflections)= 0.0362( 2690)     wR2(reflections)= 0.0857( 3006)

S = 1.048                 Npar= 267
```