Supporting Information for

Pd(II)-Catalyzed Enantioselective C(sp^3)−H Arylation of Cyclobutyl Ketones Using a Chiral Transient Directing Group

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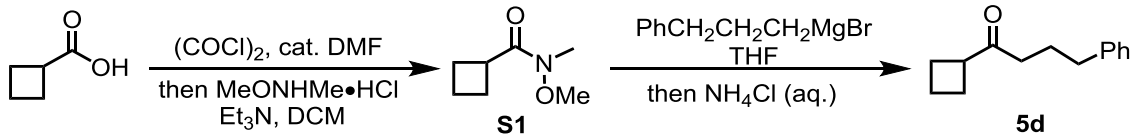
1. General Information

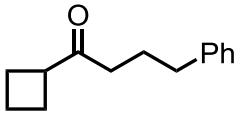
Ketone substrates were obtained from the commercial sources or synthesized following literature procedures. Aryl iodides were obtained from the commercial sources. Solvents were obtained from Sigma-Aldrich, Oakwood and Acros and used directly without further purification. Analytical thin layer chromatography was performed on 0.25 mm silica gel 60-F254. Visualization was carried out with UV light and Bromocresol Green Stain. ^{1}H NMR was recorded on Bruker DRX-600 instrument (500 MHz). Chemical shifts were quoted in parts per million (ppm) referenced to the literature values of tetramethylsilane. The following abbreviations (or combinations thereof) were used to explain multiplicities: s = singlet, d = doublet, t = triplet, q = quartet, p = pentet, m = multiplet, br = broad. coupling constants, J, were reported in Hertz unit (Hz). ^{13}C NMR spectra were recorded on Bruker DRX-600 instrument (126 MHz), and were fully decoupled by broad band proton decoupling. Chemical shifts were reported in ppm referenced to either the center line of a triplet at 77.16 ppm of chloroform-*d*. High-resolution mass spectra (HRMS) were recorded on an Agilent Mass spectrometer using ESI-TOF (electrospray ionization-time of flight). Enantiomeric excesses values were determined on a Hitachi LaChrom Elite HPLC system or Agilent Technologies supercritical fluid chromatography (SFC) system using commercially available chiral columns. Melting points were recorded on a Fisher-Johns 12-144 melting pointapparatus and are uncorrected. Optical rotation data was recorded on an Anton Paar 100 Modular Circular Polarimeter. X-ray crystallographic analysis was done at the X-ray crystallography facility, Department of Chemistry and Biochemistry, University of California, San Diego (UCSD).

2. General Procedure for the Preparation of Ketone Substrates[1]

Scheme S1



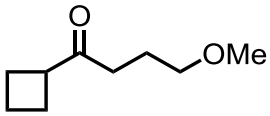
To a solution of cyclobutanecarboxylic acid (10.0 mmol) in DCM (12 mL) was added $(COCl)_2$ (11.0 mmol, 1.1 equiv.) and one drop of DMF at 0 °C. The reaction was allowed to warm to rt and stirred for 3 h, then concentrated in vacuo. The reaction mixture was dissolved in 24 mL of DCM and cooled to 0 °C. MeONHMe·HCl (12.0 mmol, 1.2 equiv.) was added in one portion, followed by triethylamine (30.0 mmol, 3.0 equiv.). The reaction was allowed to warm to rt and stirred overnight. Upon completion, 30 mL of water was added. The reaction mixture was extracted with diethyl ether (50 mL×3), and the combined organic layers were dried over anhydrous Na_2SO_4, filtered, concentrated. The amide **S1** used in the next step without further purification. To a solution of amide **S1** in THF was added phenylpropylmagnesium bromide (1.0 M in THF, 1.5 equiv.) at 0°C. The reaction was slowly warmed to rt and stirred for 4 h, then quenched with 1 M HCl solution. The reaction mixture was extracted with EtOAc (50 mL×3), and the combined organic layers were dried over anhydrous Na_2SO_4, filtered and concentrated. The crude product was then purified on silica gel to afford the desired ketone product **5d**.



5d

1-cyclobutyl-4-phenylbutan-1-one (5d)

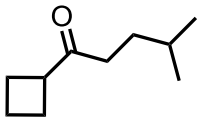
^{1}H NMR (500 MHz, $CDCl_3$) δ 7.31–7.23 (m, 2H), 7.17 (dd, *J* = 11.7, 7.0 Hz, 3H), 3.22 (pd, *J* = 8.6, 1.1 Hz, 1H), 2.65–2.53 (m, 2H), 2.35 (t, *J* = 7.3 Hz, 2H), 2.25–2.15 (m, 2H), 2.14–2.06 (m, 2H), 2.03–1.85 (m, 3H), 1.79 (m, 1H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 211.8, 141.8, 128.6, 128.4, 126.0, 45.5, 39.3, 35.3, 25.2, 24.5, 17.8.



5e

1-cyclobutyl-4-methoxybutan-1-one (5e)

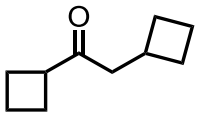
^{1}H NMR (500 MHz, $CDCl_3$) δ 3.37 (t, *J* = 6.2 Hz, 2H), 3.31 (s, 3H), 3.25 (qd, *J* = 8.5, 1.1 Hz, 1H), 2.44 (t, *J* = 7.2 Hz, 2H), 2.27–2.17 (m, 2H), 2.17–2.08 (m, 2H), 2.03–1.90 (m, 1H), 1.90–1.74 (m, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 211.7, 71.9, 58.6, 45.5, 36.4, 24.5, 23.7, 17.8.



5f

1-cyclobutyl-4-methylpentan-1-one (5f)

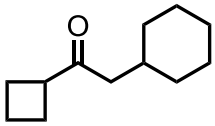
^{1}H NMR (500 MHz, $CDCl_3$) δ 3.24 (pd, *J* = 8.5, 2.9 Hz, 1H), 2.32 (td, *J* = 7.8, 3.1 Hz, 2H), 2.25–2.15 (m, 2H), 2.15–2.06 (m, 2H), 1.98–1.88 (m, 1H), 1.83–1.72 (m, 1H), 1.54–1.46 (m, 1H), 1.46–1.39 (m, 2H), 0.85 (d, *J* = 4.0 Hz, 6H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 212.5, 45.5, 38.2, 32.7, 27.9, 24.5, 22.5, 17.8.



5g

1,2-dicyclobutylethan-1-one (5g)

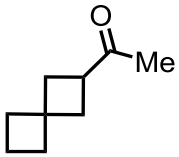
^{1}H NMR (500 MHz, $CDCl_3$) δ 3.17 (pd, *J* = 8.6, 3.0 Hz, 1H), 2.64 (pd, *J* = 7.9, 3.2 Hz, 1H), 2.43 (dd, *J* = 7.4, 3.1 Hz, 2H), 2.27–2.02 (m, 6H), 2.00–1.69 (m, 4H), 1.68–1.48 (m, 2H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 211.5, 47.4, 45.4, 31.6, 28.7, 24.3, 18.8, 17.7.



5h

1-cyclobutyl-2-cyclohexylethan-1-one (5h)

^{1}H NMR (500 MHz, $CDCl_3$) δ 3.22 (p, *J* = 8.6 Hz, 1H), 2.33–2.17 (m, 4H), 2.15–2.06 (m, 2H), 2.01–1.90 (m, 1H), 1.87–1.74 (m, 1H), 1.78–1.59 (m, 6H), 1.34–1.21 (m, 2H), 1.21–1.07 (m, 1H), 0.98–0.79 (m, 2H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 211.9, 45.9, 38.1, 33.6, 33.4, 26.4, 26.2, 24.3, 17.8.



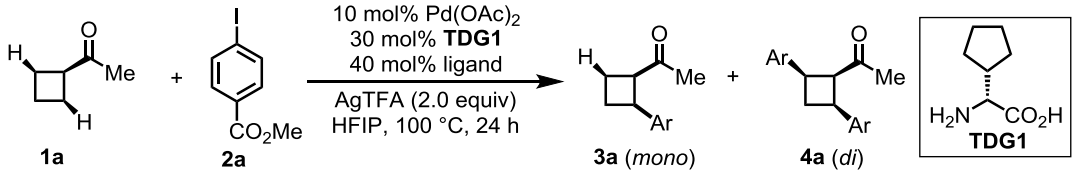
5i

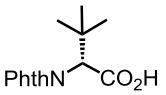
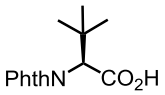
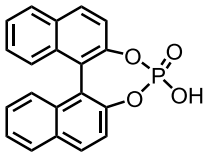
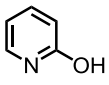
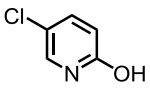
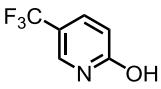
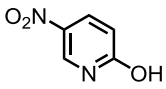
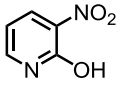
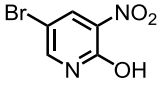
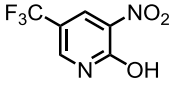
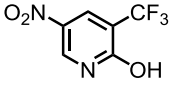
1-(spiro[3.3]heptan-2-yl)ethan-1-one (5i)

^{1}H NMR (500 MHz, $CDCl_3$) δ 3.13–3.03 (m, 1H), 2.20–2.12 (m, 4H), 2.08–1.97 (m, 5H), 1.90–1.84 (m, 2H), 1.83–1.74 (m, 2H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 210.1, 41.0, 39.5, 37.2, 35.5, 34.6, 27.3, 16.3.

3. Optimization of the Reaction Conditions

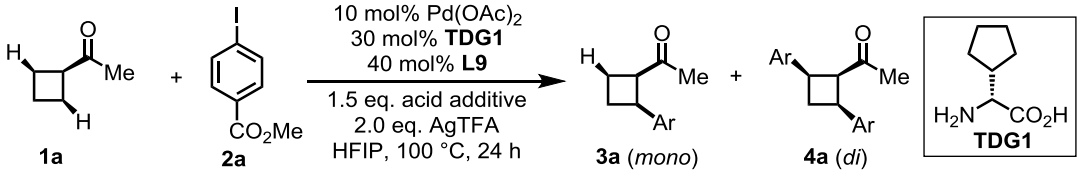
Table S1. Ligand Evaluation[a,b]

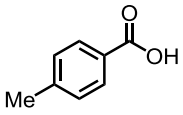
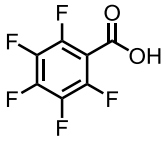


None	HOAc	^{t}BuCO$_2$H		
<5%, er N.D.	**L1** 30%, 33:67 er	**L2** 22%, 35:65 er	**L3** 11%, 34:66 er	**L4** 14%, 35:65 er
				
(±)-**L5** <5%, er N.D.	**L6** 14%, 25:75 er	**L7** 29%, 17:83 er 8% *di*	**L8** 16%, 18:82 er	**L9** 24%, 15:85 er 45% *di*
				
L10 32%, 25:75 er 48% *di*	**L11** 31%, 20:80 er 30% *di*	**L12** 25%, 17:83 er 52% *di*	**L13** 22%,19:81 er 43% *di*	

[a] Conditions: **1a** (0.2 mmol, 2.0 equiv), methyl 4-iodobenzoate (1.0 equiv), $Pd(OAc)_2$ (10 mol %), **TDG1** (30 mol %), ligand (40 mol %), AgTFA (2.0 equiv), HFIP (0.6 mL), 100 °C, under air, 24 h. [b] Yield determined by ^{1}H NMR analysis of the crude product using CH_2Br_2 as internal standard.

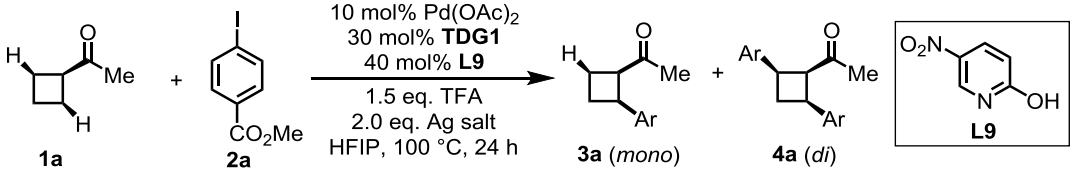
Table S2. Acid Additive Evaluation[a,b]



No additive	HOAc		
24%, 15:85 er 45% *di*	28%, 17:83 er 40% *di*	<5%, er N.D.	<5%, er N.D.
$TsOH•H_2O$	TFA (1.5 eq.)	TFA (3.75 eq.)	TFA (7.5 eq.)
<5%, er N.D.	25%, 14:86 er 45% *di*	29%, 22.5:77.5 er 48% *di*	29%, 27:73 er 52% *di*

[a] Conditions: **1a** (0.2 mmol, 2.0 equiv), methyl 4-iodobenzoate (1.0 equiv), $Pd(OAc)_2$ (10 mol %), **TDG1** (30 mol %), **L9** (40 mol %), acid additive (1.5 equiv), AgTFA (2.0 equiv), HFIP (0.6 mL), 100 °C, under air, 24 h. [b] Yield determined by ^1H NMR analysis of the crude product using CH_2Br_2 as internal standard.

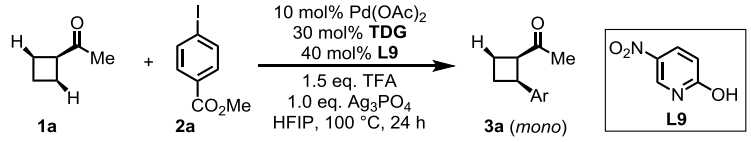
Table S3. Silver Salt Evaluation[a,b]



No silver salt **L9**	AgTFA	AgOAc	Ag_2CO_3	Ag_2O
N.R.	34%, 14:86 er 50% *di*	25%, 14:86 er 10% *di*	20%, 10:80 er 5% *di*	19%, 15:85 er 10% *di*
$AgNO_3$	AgF	Ag_3PO_4[c]	AgTFA K_3PO_4[d]	No pyridone ligand Ag_3PO_4[c]
N.R.	<5%, er N.D.	81%, 96:4 er <5% *di*	42%, 75:25 er 29% *di*	42%, 36:64 er <5% *di*
Na_3PO_4[e]	K_3PO_4[d]			
N.R.	N.R.			

[a] Conditions: **1a** (0.2 mmol, 2.0 equiv), methyl 4-iodobenzoate (1.0 equiv), $Pd(OAc)_2$ (10 mol %), **TDG1** (30 mol %), **L9** (40 mol %), TFA (1.5 equiv), silver salt (2.0 equiv), HFIP (0.6 mL), 100 °C, under air, 24 h. [b] Yield determined by ^1H NMR analysis of the crude product using CH_2Br_2 as internal standard. [c] Ag_3PO_4 (1.0 equiv). [d] K_3PO_4 (1.0 equiv). [e] Na_3PO_4 (1.0 equiv)

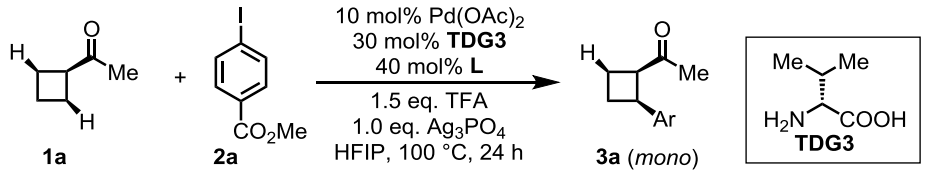
Table S4. Transient Directing Group Evaluation[a,b]

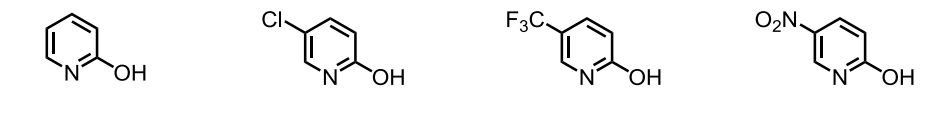


TDG1 82%, 96:4 er	**TDG2** 75%, 93:7 er	**TDG3** (*D*-Valine) 82%, 97:3 er	**TDG4** (*L*-Valine) 82%, 3:97 er	**TDG5** 78%, 96:4 er	**TDG6** 56%,99:1 er
TDG7 63%, 90:10 er	**TDG8** 55%, 83:17 er	**TDG9** 70%, 82:18 er	**TDG10** 79%, 91:9 er	**TDG11** 60%, 87:13 er	No **TDG** **L6** N.R.

[a] Conditions: **1a** (0.2 mmol, 2.0 equiv), methyl 4-iodobenzoate (1.0 equiv), $Pd(OAc)_2$ (10 mol %), **TDG** (30 mol %), **L9** (40 mol %), TFA (1.5 equiv), Ag_3PO_4 (1.0 equiv), HFIP (0.6 mL), 100 °C, under air, 24 h. [b] Yield determined by ^1H NMR analysis of the crude product using CH_2Br_2 as internal standard.

Table S5. Pyridone Ligand Evaluation in the Presence of Ag_3PO_4[a,b]



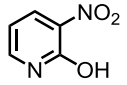


L6
52%, 62:38 er

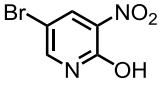
L7
48%, 87:13 er

L8
55%, 88:12 er

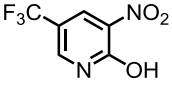
L9
82%, 97:3 er



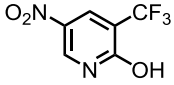
L10
72%, 97:3 er



L11
78%, 98:2 er



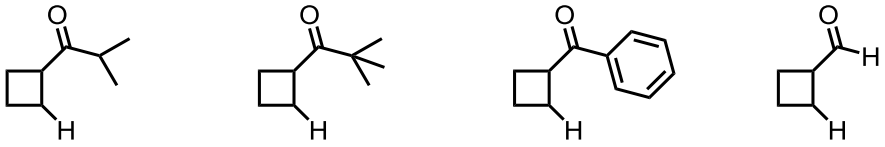
L12
82%, 98:2 er



L13
81%, 98:2 er

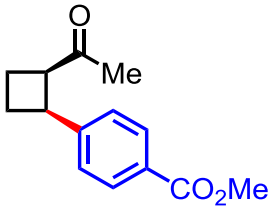
[a] Conditions: **1a** (0.2 mmol, 2.0 equiv), methyl 4-iodobenzoate (1.0 equiv), $Pd(OAc)_2$ (10 mol %), **TDG3** (30 mol %), **L** (40 mol %), TFA (1.5 equiv), Ag_3PO_4 (1.0 equiv), HFIP (0.6 mL), 100 °C, under air, 24 h. [b] Yield determined by ^1H NMR analysis of the crude product using CH_2Br_2 as internal standard.

Table S6: Selected Unsuccessful Substrates



4. General Procedure for the Enantioselective C(sp^3)–H Arylation and Characterization of the Products

To an oven-dried microwave tube (5 mL) equipped with a magnetic stir bar was added $Pd(OAc)_2$ (0.01 mmol, 10 mol %), transient directing groups (**TDG3**, 0.03 mmol, 30 mol %), ligand (**L12**, 0.04 mmol, 40 mol %), ArI (0.1 mmol), Ag_3PO_4 (0.1 mmol, 1.0 equiv), and solvent (HFIP, 0.6 mL and 0.15 mmol of TFA), followed by the ketone substrate (0.2 mmol, 2.0 equiv). The tube was sealed and stirred at room temperature for 10 min before heating to 100 °C for 24 h under vigorous stirring. Upon completion, the reaction mixture was cooled to room temperature and the dark brown suspension was passed through a pad of Celite and washed with acetone (1.0 mL × 3). The resulting solution was concentrated and purified by preparative thin-layer chromatography to afford the desired product. Unless otherwise specified, the racemic product was prepared according to previously reported procedure.[1,2]

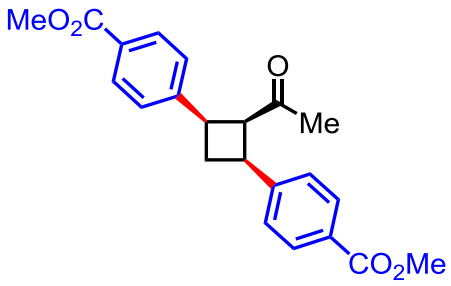


3a

methyl 4-((1*S*,2*R*)-2-acetylcyclobutyl)benzoate (3a)

Colorless oil (18.3 mg, 79% yield, 98:2 er). $[\alpha]_D^{20}$ = +18.6 (*c* = 1.0, $CHCl_3$). The er ratio was determined by a Hitachi LaChrom Elite HPLC analysis on a Chiralpak IC column (20% isopropanol in hexane, 2.0 mL/min) with retention time 8.4 min (major) and 21.9 min (minor). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.98 (d, *J* = 8.4 Hz, 2H), 7.31 (d, *J* = 8.0 Hz, 2H), 3.91 (s, 3H), 3.77 (q, *J* = 9.1 Hz, 1H), 3.36–3.27 (m, 1H), 2.27–2.14 (m, 4H), 2.06 (s, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 208.5, 167.1, 149.1, 130.0, 128.5, 126.8, 53.7, 52.2, 42.2, 27.9, 24.7, 21.6. HRMS (ESI-TOF) Calcd for $C_{14}H_{17}O_3^+$ $[M+H]^+$: 233.1178; found: 233.1173.

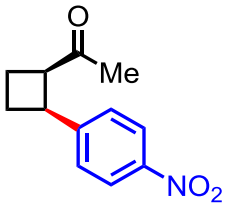
The absolute stereochemistry was assigned by analogy to compound **7a** and **7b**.



4a

dimethyl 4,4'-2-acetylcyclobutane-1,3-diyl)dibenzoate (4a)

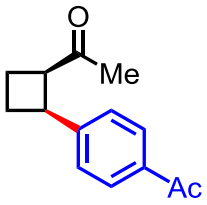
Colorless oil (50% yield under this condition: **TDG1** (30 mol %), **L9** (40 mol %), TFA (1.5 equiv), AgTFA (2.0 equiv), HFIP (0.6 mL), 100 °C, under air, 24 h.) ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.02 (d, *J* = 8.3 Hz, 4H), 7.36 (d, *J* = 8.3 Hz, 4H), 3.92 (s, 6H), 3.71 (td, *J* = 9.8, 8.0 Hz, 2H), 3.40 (t, *J* = 9.5 Hz, 1H), 2.74 (ddd, *J* = 10.7, 8.6, 7.7 Hz, 1H), 2.34 (q, *J* = 10.4 Hz, 1H), 1.98 (s, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 207.0, 167.0, 148.0, 130.2, 128.9, 127.0, 61.1, 52.3, 39.3, 32.5, 28.9. HRMS (ESI-TOF) Calcd for $C_{22}H_{23}O_5^+$ $[M+H]^+$: 367.1545; found: 367.1539.



3b

1-((1*R*,2*S*)-2-(4-nitrophenyl)cyclobutyl)ethan-1-one (3b)

Colorless oil (15.4 mg, 70% yield, 97:3 er). $[\alpha]_D^{20}$ = +17.2 (*c* = 1.0, $CHCl_3$). The er ratio was determined a Hitachi LaChrom Elite HPLC analysis on a Chiralpak IC column (20% isopropanol in hexane, 1.5 mL/min) with retention time 10.66 min (minor) and 11.74 min (major). ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.17 (d, *J* = 8.8 Hz, 2H), 7.38 (d, *J* = 8.2 Hz, 2H), 3.87 (q, *J* = 9.4 Hz, 1H), 3.36–3.25 (m, 1H), 2.33–2.25 (m, 2H), 2.22–2.16 (m, 2H), 2.10 (s, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 208.2, 151.5, 146.7, 127.6, 123.9, 53.6, 41.3, 27.7, 24.4, 22.1. HRMS (ESI-TOF) Calcd for $C_{13}H_{14}NO_3^+$ $[M+H]^+$: 220.0974; found: 220.0975. The absolute stereochemistry was assigned by analogy to compound **7a** and **7b**.

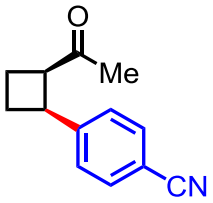


3c

1-(4-((1*S*,2*R*)-2-acetylcyclobutyl)phenyl)ethan-1-one (3c)

Colorless oil (13.9 mg, 64% yield, 98:2 er). $[\alpha]_D^{20}$ = +19.4 (*c* = 1.0, $CHCl_3$). The er ratio was determined by a Hitachi LaChrom Elite HPLC analysis on a Chiralpak IC column (20% isopropanol in hexane, 2.0 mL/min) with retention time 13.51 min (major) and 29.86 min (minor). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.91 (d, *J* = 8.4 Hz, 2H), 7.33 (d, *J* = 8.0 Hz, 2H), 3.82–3.75 (m, 1H), 3.36–3.27 (m, 1H), 2.59 (s, 3H), 2.27–2.13 (m, 4H), 2.06 (s, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 208.5, 197.9, 149.4, 135.6, 128.8, 128.8, 127.0, 126.9, 53.6, 42.1, 27.9, 26.7, 24.7, 21.6. HRMS (ESI-TOF) Calcd for $C_{14}H_{17}O_2^+$ $[M+H]^+$: 217.1229; found: 217.1225.

The absolute stereochemistry was assigned by analogy to compound **7a** and **7b**.



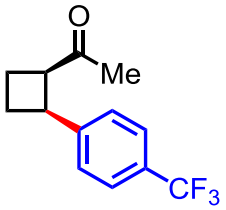
3d

4-((1*S*,2*R*)-2-acetylcyclobutyl)benzonitrile (3d)

Colorless oil (16 mg, 80% yield, 95:5 er). $[\alpha]_D^{20}$ = +38.9 (*c* = 1.0, $CHCl_3$). The er ratio was determined by a Hitachi LaChrom Elite HPLC analysis on a Chiralpak IC column (20% isopropanol in hexane, 1.5 mL/min) with retention time 12.49 min (minor) and 14.22 min (major). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.60 (d, *J* = 8.3 Hz, 2H), 7.34 (d, *J* = 8.2 Hz, 2H), 3.81 (q, *J* = 9.1 Hz, 1H), 3.3 –3.23 (m, 1H), 2.30–2.22 (m, 2H), 2.19–2.13 (m, 2H), 2.08 (s, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 208.3, 149.3, 132.4, 127.5, 119.1, 110.3, 53.6, 41.6,

27.7, 24.3, 22.0. HRMS (ESI-TOF) Calcd for $C_{13}H_{14}NO^+$ $[M+H]^+$: 200.1075; found: 200.1069.

The absolute stereochemistry was assigned by analogy to compound **7a** and **7b**.

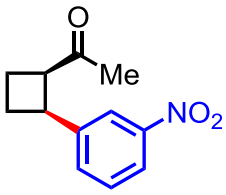


3e

1-((1*R*,2*S*)-2-(4-(trifluoromethyl)phenyl)cyclobutyl)ethan-1-one (3e)

Colorless oil (17.2 mg, 71% yield, 94:6 er). $[\alpha]_D^{20}$ = –124.5 (*c* = 1.0, $CHCl_3$). The er ratio was determined by a Hitachi LaChrom Elite HPLC analysis on a Chiralpak OD-H column (2% isopropanol in CO_2, 0.5 mL/min) with retention time 11.87 min (major) and 17.77 min (minor). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.56 (d, *J* = 7.7 Hz, 2H), 7.35 (d, *J* = 7.9 Hz, 2H), 3.78 (q, *J* = 8.4, 7.8 Hz, 1H), 3.33–3.25 (m, 1H), 2.28–2.14 (m, 4H), 2.07 (s, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 208.4, 147.9, 129.0, 128.7, 127.3, 127.1, 125.6, 125.5, 123.3, 53.7, 41.8, 27.9, 24.6, 21.7. HRMS (ESI-TOF) Calcd for $C_{13}H_{14}F_3O^+$ $[M+H]^+$: 243.0997; found: 243.0990.

The absolute stereochemistry was assigned by analogy to compound **7a** and **7b**.

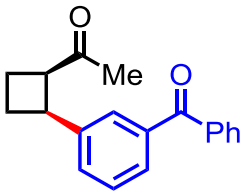


3f

1-((1*R*,2*S*)-2-(3-nitrophenyl)cyclobutyl)ethan-1-one (3f)

Colorless oil (16.4 mg, 75% yield, 97:3 er). $[\alpha]_D^{20}$ = +39.6 (*c* = 1.0, $CHCl_3$). The er ratio was determined by a Hitachi LaChrom Elite HPLC analysis on a Chiralpak IC column (20% isopropanol in hexane, 1.0 mL/min) with retention time 17.37 min (major) and 22.86 min (minor). ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.12–8.03 (m, 2H), 7.61–7.55 (m, 1H), 7.47 (t, *J* =

7.9 Hz, 1H), 3.86 (q, J = 9.4 Hz, 1H), 3.39–3.27 (m, 1H), 2.34–2.27 (m, 2H), 2.22–2.14 (m, 2H), 2.10 (s, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 208.2, 148.6, 146.0, 133.3, 129.5, 121.6, 121.5, 53.6, 41.1, 27.7, 24.5, 22.0. HRMS (ESI-TOF) Calcd for $C_{12}H_{14}NO_3^+$ $[M+H]^+$: 220.0974; found: 220.0969.

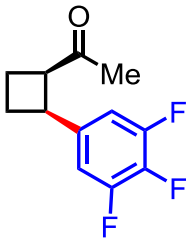


3g

1-((1*R*,2*S*)-2-(3-benzoylphenyl)cyclobutyl)ethan-1-one (3g)

Colorless oil (16.9 mg, 61% yield, 97:3 er). $[\alpha]_D^{20}$ = +9.1 (c = 1.0, $CHCl_3$). The er ratio was determined by a Hitachi LaChrom Elite HPLC analysis on a Chiralpak IC column (20% isopropanol in hexane, 1.5 mL/min) with retention time 14.87 min (major) and 16.89 min (minor). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.82–7.79 (m, 2H), 7.72 (t, J = 1.8 Hz, 1H), 7.60 (ddt, J = 8.8, 6.9, 1.4 Hz, 2H), 7.51–7.46 (m, 3H), 7.41 (t, J = 7.6 Hz, 1H), 3.77 (q, J = 9.2 Hz, 1H), 2.31–2.23 (m, 1H), 3.38–3.30 (m, 1H), 2.21–2.14 (m, 3H), 2.06 (s, 3H). HRMS (ESI-TOF) Calcd for $C_{19}H_{19}O_2^+$ $[M+H]^+$: 279.1385; found: 279.1379.

The absolute stereochemistry was assigned by analogy to compound **7a** and **7b**.



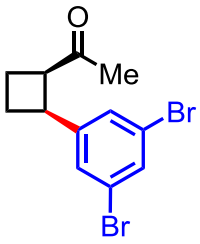
3h

1-((1*R*,2*S*)-2-(3,4,5-trifluorophenyl)cyclobutyl)ethan-1-one (3h)

Colorless oil (16.8 mg, 74% yield, 97:3 er). $[\alpha]_D^{20}$ = +11.9 (c = 1.0, $CHCl_3$). The er ratio was determined by a Hitachi LaChrom Elite HPLC analysis on a Chiralpak IC column (10% isopropanol in hexane, 0.5 mL/min) with retention time 11.75 min (major) and 12.24 min (minor). ^{1}H NMR (500 MHz, $CDCl_3$) δ 6.84 (ddd, J = 8.7, 6.5, 0.8 Hz, 2H), 3.71–3.65 (m,

1H), 3.22–3.15 (m, 1H), 2.26–2.18 (m, 2H), 2.13–2.03 (m, 5H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 208.2, 152.3 (dd, *J* = 10.4, 4.4 Hz), 150.3 (dd, *J* = 10.1, 4.4 Hz), 140.3, 139.4, 137.4, 110.7 (dd, *J* = 16.0, 4.8 Hz), 53.8, 40.9, 27.7, 24.4, 21.8. HRMS (ESI-TOF) Calcd for $C_{12}H_{12}F_3O^+$ $[M+H]^+$: 229.0840; found: 229.0833.

The absolute stereochemistry was assigned by analogy to compound **7a** and **7b**.

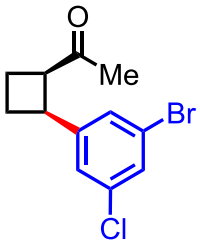


3i

1-((1*R*,2*S*)-2-(3,5-dibromophenyl)cyclobutyl)ethan-1-one (3i)

Colorless oil (26.9 mg, 82% yield, 99:1 er). $[\alpha]_D^{20}$ = +17.0 (*c* = 1.0, $CHCl_3$). The er ratio was determined by a Hitachi LaChrom Elite HPLC analysis on a Chiralpak IC column (20% isopropanol in hexane, 0.5 mL/min) with retention time 10.87 min (major) and 11.43 min (minor). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.50 (t, *J* = 1.8 Hz, 1H), 7.30 (dd, *J* = 1.8, 0.7 Hz, 2H), 3.74–3.66 (m, 1H), 3.28–3.21 (m, 1H), 2.26–2.17 (m, 2H), 2.14–2.06 (m, 5H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 208.1, 147.9, 132.1, 128.8, 123.1, 53.5, 40.9, 27.8, 24.5, 21.9. HRMS (ESI-TOF) Calcd for $C_{12}H_{13}Br_2O^+$ $[M+H]^+$: 330.9333; found: 330.9323.

The absolute stereochemistry was assigned by analogy to compound **7a** and **7b**.



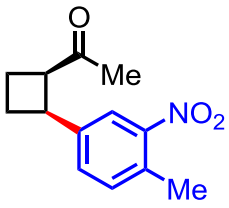
3j

1-((1*R*,2*S*)-2-(3-bromo-5-chlorophenyl)cyclobutyl)ethan-1-one (3j)

Colorless oil (17.7 mg, 62% yield, 97:3 er). $[\alpha]_D^{20}$ = +12.5 (*c* = 1.0, $CHCl_3$). The er ratio was determined by SFC analysis on a Chiralpak IC column (5% isopropanol in CO_2, 2.0

mL/min) with retention time 5.98 min (major) and 6.50 min (minor). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.35 (t, *J* = 1.8 Hz, 1H), 7.26–7.25 (m, 1H), 7.15–7.14 (m, 1H), 3.73–3.66 (m, 1H), 3.28–3.22 (m, 1H), 2.26–2.19 (m, 2H), 2.13–2.06 (m, 5H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 208.2, 147.6, 135.2, 129.5, 128.3, 125.9, 122.9, 53.5, 41.0, 27.8, 24.5, 21.9. HRMS (ESI-TOF) Calcd for $C_{12}H_{13}BrClO^+$ $[M+H]^+$: 286.9838; found: 286.9830.

The absolute stereochemistry was assigned by analogy to compound **7a** and **7b**.

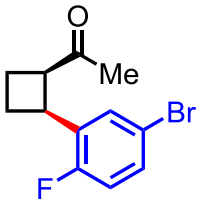


3k

1-((1*R*,2*S*)-2-(4-methyl-3-nitrophenyl)cyclobutyl)ethan-1-one (3k)

Colorless oil (9.3 mg, 40% yield, 93:7 er). $[\alpha]_D^{20}$ = +25.7 (*c* = 1.0, $CHCl_3$). The er ratio was determined by a Hitachi LaChrom Elite HPLC analysis on a Chiralpak IC column (20% isopropanol in hexane, 1.5 mL/min) with retention time 10.50 min (major) and 11.74 min (minor). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.64 (dd, *J* = 1.8, 0.9 Hz, 1H), 7.40–7.36 (m, 1H), 7.33 (d, *J* = 8.0 Hz, 1H), 4.14 (q, *J* = 9.3 Hz, 1H), 3.51–3.38 (m, 1H), 2.42–2.36 (m, 4H), 2.21–2.15 (m, 2H), 2.12 (s, 3H), 1.90 (dtd, *J* = 10.8, 9.9, 9.0 Hz, 1H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 208.4, 137.8, 135.1, 133.8, 128.2, 124.9, 51.6, 38.5, 27.7, 25.8, 21.6, 20.8. HRMS (ESI-TOF) Calcd for $C_{13}H_{16}NO_3^+$ $[M+H]^+$: 234.1130; found: 234.1127.

The absolute stereochemistry was assigned by analogy to compound **7a** and **7b**.



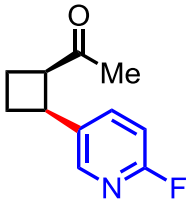
3l

1-((1*R*,2*S*)-2-(5-bromo-2-fluorophenyl)cyclobutyl)ethan-1-one (3l)

Colorless oil (20.0 mg, 74% yield, 99:1 er). $[\alpha]_D^{20}$ = +20.7 (*c* = 1.0, $CHCl_3$). The er ratio was determined by SFC analysis on a Chiralpak AS column (10% isopropanol in CO_2, 2.0

mL/min) with retention time 4.58 min (major) and 5.63 min (minor). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.36 (ddd, *J* = 6.6, 2.5, 0.8 Hz, 1H), 7.30 (dddd, *J* = 8.7, 4.5, 2.5, 0.4 Hz, 1H), 6.89 (dd, *J* = 9.9, 8.7 Hz, 1H), 3.90–3.81 (m, 1H), 3.39 (q, *J* = 8.8, 8.4 Hz, 1H), 2.30–2.12 (m, 4H), 2.09 (s, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 208.2, 161.0, 159.0, 132.9, 132.7, 131.3, 131.2, 131.02, 130.95, 117.5, 117.3, 116.82, 116.79, 52.1, 36.3, 28.0, 24.4, 21.8. HRMS (ESI-TOF) Calcd for $C_{12}H_{13}BrFO^{+}$ $[M+H]^{+}$: 271.0134; found: 271.0127.

The absolute stereochemistry was assigned by analogy to compound **7a** and **7b**.

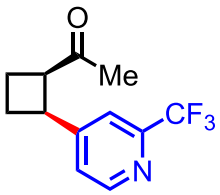


3m

1-((1*R*,2*S*)-2-(6-fluoropyridin-3-yl)cyclobutyl)ethan-1-one (3m)

Colorless oil (7.1 mg, 37% yield, 94:6 er). $[\alpha]_D^{20}$ = +9.4 (*c* = 1.0, $CHCl_3$). The er ratio was determined by SFC analysis on a Chiralpak IC column (10% isopropanol in CO_2, 2.0 mL/min) with retention time 6.95 min (major) and 7.56 min (minor). ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.10–8.06 (m, 1H), 7.72–7.65 (m, 1H), 6.88 (dd, *J* = 8.4, 2.9 Hz, 1H), 3.75 (q, *J* = 8.8, 8.3 Hz, 1H), 3.29–3.21 (m, 1H), 2.29–2.23 (m, 2H), 2.19–2.12 (m, 2H), 2.08 (s, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 208.2, 163.6, 161.7, 145.9, 145.8, 139.8, 139.7, 136.8, 136.7, 109.5, 109.2, 53.8, 38.6, 27.7, 24.5, 22.1. HRMS (ESI-TOF) Calcd for $C_{11}H_{13}FNO^{+}$ $[M+H]^{+}$: 194.0981; found: 194.0977.

The absolute stereochemistry was assigned by analogy to compound **7a** and **7b**.

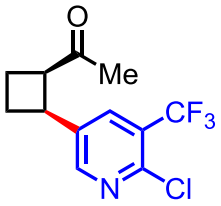


3n

1-((1*R*,2*S*)-2-(2-(trifluoromethyl)pyridin-4-yl)cyclobutyl)ethan-1-one (3n)

Colorless oil (10.2 mg, 42% yield, 92:8 er). $[\alpha]_D^{20}$ = –25.3 (*c* = 1.0, $CHCl_3$). The er ratio was determined by SFC analysis on a Chiralpak IC column (10% isopropanol in CO_2, 2.0 mL/min) with retention time 6.42 min (minor) and 7.62 min (major). ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.63 (d, *J* = 5.0 Hz, 1H), 7.56–7.50 (m, 1H), 7.34 (dt, *J* = 5.0, 1.1 Hz, 1H), 3.88 (q, *J* = 9.6 Hz, 1H), 3.35–3.25 (m, 1H), 2.37–2.29 (m, 2H), 2.23–2.16 (m, 2H), 2.12 (s, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 207.9, 154.8, 150.2, 148.7, 148.5, 124.8, 122.8, 120.6, 118.77, 118.75, 53.0, 40.2, 27.5, 23.6, 22.5. HRMS (ESI-TOF) Calcd for $C_{12}H_{13}F_3NO^+$ $[M+H]^+$: 244.0949; found: 244.0944.

The absolute stereochemistry was assigned by analogy to compound **7a** and **7b**.

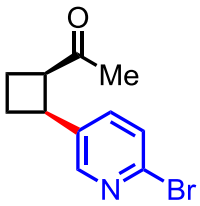


3o

1-((1*R*,2*S*)-2-(6-chloro-5-(trifluoromethyl)pyridin-3-yl)cyclobutyl)ethan-1-one (3o)

Colorless oil (14.0 mg, 50% yield, 88:12 er). $[\alpha]_D^{20}$ = –11.3 (*c* = 1.0, $CHCl_3$). The er ratio was determined by SFC analysis on a Chiralpak IC column (5% isopropanol in CO_2, 2.0 mL/min) with retention time 5.36 min (major) and 5.75 min (minor). ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.43 (dd, *J* = 1.8, 0.7 Hz, 1H), 7.91–7.84 (m, 1H), 3.90–3.82 (m, 1H), 3.32–3.23 (m, 1H), 2.38–2.29 (m, 2H), 2.21–2.15 (m, 2H), 2.11 (s, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 207.8, 150.9, 146.8, 138.3, 135.22, 135.18, 125.2, 125.0, 123.4, 121.2, 53.5, 37.8, 27.5, 23.9, 22.7. HRMS (ESI-TOF) Calcd for $C_{12}H_{12}ClF_3NO^+$ $[M+H]^+$: 278.0560; found: 278.0554.

The absolute stereochemistry was assigned by analogy to compound **7a** and **7b**.

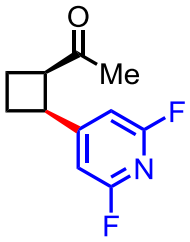


3p

1-((1*R*,2*S*)-2-(6-bromopyridin-3-yl)cyclobutyl)ethan-1-one (3p)

Colorless oil (8.8 mg, 35% yield, 95:5 er). $[\alpha]_D^{20}$ = +11.3 (*c* = 1.0, $CHCl_3$). The er ratio was determined by SFC analysis on a Chiralpak IC column (10% isopropanol in CO_2, 2.0 mL/min) with retention time 16.33 min (major) and 17.16 min (minor). ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.26–8.21 (m, 1H), 7.46–7.40 (m, 2H), 3.72 (q, *J* = 8.8, 8.1 Hz, 1H), 3.24 (q, *J* = 9.2, 8.8 Hz, 1H), 2.31–2.23 (m, 2H), 2.20–2.12 (m, 2H), 2.07 (s, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 208.1, 148.8, 140.1, 138.4, 137.3, 128.0, 53.6, 38.7, 27.7, 24.2, 22.2. HRMS (ESI-TOF) Calcd for $C_{11}H_{13}BrNO^+$ $[M+H]^+$: 254.0181 found: 254.0177.

The absolute stereochemistry was assigned by analogy to compound **7a** and **7b**.

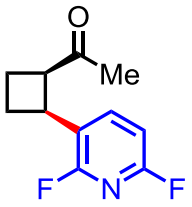


3q

1-((1*R*,2*S*)-2-(2,6-difluoropyridin-4-yl)cyclobutyl)ethan-1-one (3q)

Colorless oil (13.0 mg, 62% yield, 95:5 er). $[\alpha]_D^{20}$ = +9.8 (*c* = 1.0, $CHCl_3$). The er ratio was determined by SFC analysis on a Chiralpak IC column (3% isopropanol in CO_2, 2.0 mL/min) with retention time 7.97 min (minor) and 8.43 min (major). ^{1}H NMR (500 MHz, $CDCl_3$) δ 6.66 (s, 2H), 3.89–3.81 (m, 1H), 3.31–3.23 (m, 1H), 2.37–2.24 (m, 2H), 2.20–2.09 (m, 5H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 207.7, 163.6, 163.54, 163.48, 163.2, 163.0, 161.2, 161.1, 104.3, 104.2, 104.0, 103.9, 53.0, 40.1, 27.5, 23.6, 22.5. HRMS (ESI-TOF) Calcd for $C_{11}H_{12}F_2NO^+$ $[M+H]^+$: 212.0887; found: 212.0883.

The absolute stereochemistry was assigned by analogy to compound **7a** and **7b**.

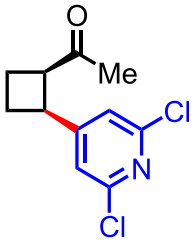


3r

1-((1*R*,2*S*)-2-(2,6-difluoropyridin-3-yl)cyclobutyl)ethan-1-one (3r)

Colorless oil (11.6 mg, 55% yield, 92:8 er). $[\alpha]_D^{20}$ = –103.0 (*c* = 1.0, $CHCl_3$). The er ratio was determined by SFC analysis on a Chiralpak IC column (5% isopropanol in CO_2, 2.0 mL/min) with retention time 6.0 min (major) and 6.70 min (minor). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.74 (dddd, *J* = 9.5, 8.2, 7.6, 0.8 Hz, 1H), 6.79 (dd, *J* = 8.1, 2.9 Hz, 1H), 3.82 (q, *J* = 9.1 Hz, 1H), 3.40 (q, *J* = 9.4 Hz, 1H), 2.34–2.24 (m, 2H), 2.21–2.13 (m, 2H), 2.11 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 208.1, 143.4, 143.31, 143.29, 143.2, 122.0, 121.9, 121.7, 121.6, 106.3, 106.2, 105.94, 105.88, 51.8, 35.5, 27.9, 24.2, 22.3. HRMS (ESI-TOF) Calcd for $C_{11}H_{12}F_2NO^+$ $[M+H]^+$: 212.0887; found: 212.0883.

The absolute stereochemistry was assigned by analogy to compound **7a** and **7b**.



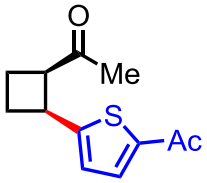
3s

1-((1*R*,2*S*)-2-(2,6-dichloropyridin-4-yl)cyclobutyl)ethan-1-one (3s)

Colorless oil (11.9 mg, 49% yield, 96:4 er). $[\alpha]_D^{20}$ = +13.8 (*c* = 1.0, $CHCl_3$). The er ratio was determined by SFC analysis on a Chiralpak IC column (5% isopropanol in CO_2, 2.0 mL/min) with retention time 13.08 min (minor) and 15.49 min (major). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.11 (s, 2H), 3.83–3.74 (m, 1H), 3.30–3.21 (m, 1H), 2.37–2.29 (m, 1H), 2.28–2.22 (m, 1H), 2.18–2.08 (m, 5H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 207.6, 158.8, 150.8, 121.2,

52.9, 39.5, 27.4, 23.4, 22.6. HRMS (ESI-TOF) Calcd for $C_{11}H_{12}Cl_2NO^+$ $[M+H]^+$: 244.0296; found: 244.0291.

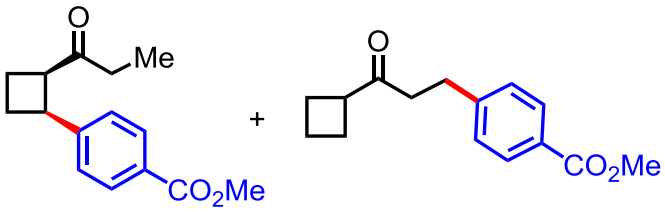
The absolute stereochemistry was assigned by analogy to compound **7a** and **7b**.



3t

1-(5-((1*S*,2*R*)-2-acetylcyclobutyl)thiophen-2-yl)ethan-1-one (3t)

Colorless oil (11.1 mg, 50% yield, 90:10 er). $[\alpha]_D^{20}$ = +20.3 (*c* = 1.0, $CHCl_3$). The er ratio was determined by SFC analysis on a Chiralpak IC column (20% isopropanol in CO_2, 2.0 mL/min) with retention time 8.85 min (major) and 15.15 min (minor). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.54 (d, *J* = 3.8 Hz, 1H), 6.88 (d, *J* = 3.8 Hz, 1H), 3.89 (q, *J* = 9.1 Hz, 1H), 3.38–3.26 (m, 1H), 2.52 (s, 3H), 2.37–2.31 (m, 1H), 2.22–2.13 (m, 3H), 2.08 (s, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 207.6, 190.6, 157.8, 142.5, 133.0, 124.9, 55.2, 38.2, 28.0, 26.8, 26.6, 21.3. HRMS (ESI-TOF) Calcd for $C_{12}H_{15}O_2S^+$ $[M+H]^+$: 223.0793; found: 223.0791.



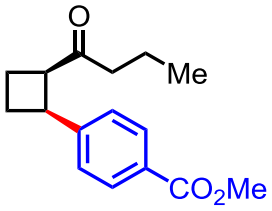
6a **6a'**

methyl 4-((1*S*,2*R*)-2-propionylcyclobutyl)benzoate (6a)

methyl 4-(3-cyclobutyl-3-oxopropyl)benzoate (6a')

Colorless oil as a 4:1 mixture of **6a** and **6a'** isomers (12.5 mg, 51% yield) **6a**: 96:4 er, $[\alpha]_D^{20}$ = +13.9 (*c* = 1.0, $CHCl_3$). The er ratio of **6a** was determined by SFC analysis on a Chiralpak IC column (5% isopropanol in CO_2, 2.0 mL/min) with retention time 15.04 min (major) and 18.71 min (minor). **6a**: ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.98 (d, *J* = 8.5 Hz, 2H), 7.30 (d, *J* = 8.0 Hz, 2H), 3.90 (s, 3H), 3.81–3.76 (m, 1H), 3.35–3.28 (m, 1H), 2.40–2.26 (m,

3H), 2.21–2.15 (m, 3H), 1.03 (t, J = 7.3 Hz, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 211.1, 167.1, 149.3, 129.9, 128.5, 126.8, 52.7, 52.2, 42.2, 34.0, 24.8, 21.7, 7.7. HRMS (ESI-TOF) Calcd for $C_{15}H_{19}O_3^+$ $[M+H]^+$: 247.1334; found: 247.1331. **6a'**: ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.96–7.93 (m, 0.50 H), 7.26–7.24 (m, 0.56H), 3.22 (pd, J = 8.6, 1.1 Hz, 0.25H), 2.95 (t, J = 7.5 Hz, 0.50H), 2.68 (t, J = 7.9, 7.1 Hz, 0.50H), 2.14–2.06 (m, 1H), 1.99–1.91 (m, 0.27H), 1.83–1.76 (m, 0.26H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 210.4, 171.5, 147.0, 128.4, 128.2, 127.7, 45.6, 39.5, 29.7, 23.9, 18.6, 7.7.

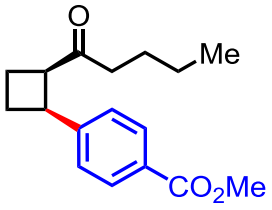


6b

methyl 4-((1*S*,2*R*)-2-butyrylcyclobutyl)benzoate (6b)

Colorless oil (16.1 mg, 62% yield, 98:2 er). $[\alpha]_D^{20}$ = –68.4 (*c* = 1.0, $CHCl_3$). The er ratio was determined by a Hitachi LaChrom Elite HPLC analysis on a Chiralpak IC column (20% isopropanol in hexane, 1.5 mL/min) with retention time 6.56 min (major) and 10.0 min (minor). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.98 (d, J = 8.3 Hz, 2H), 7.30 (d, J = 7.9 Hz, 2H), 3.91 (s, 3H), 3.81–3.76 (m, 1H), 3.34–3.28 (m, 1H), 2.36–2.24 (m, 3H), 2.20–2.13 (m, 3H), 1.60–1.49 (m, 2H), 0.87 (t, J = 7.4 Hz, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 210.7, 167.1, 149.3, 129.9, 128.4, 126.8, 53.0, 52.2, 42.8, 42.1, 24.8, 21.7, 17.1, 13.9. HRMS (ESI-TOF) Calcd for $C_{16}H_{21}O_3^+$ $[M+H]^+$: 261.1491; found: 261.1488.

The absolute stereochemistry was assigned by analogy to compound **7a** and **7b**.

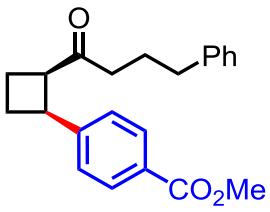


6c

methyl 4-((1*S*,2*R*)-2-pentanoylcyclobutyl)benzoate (6c)

Colorless oil (15.0 mg, 55% yield, 97:3 er). $[\alpha]_D^{20}$ = –133.0 (*c* = 1.0, $CHCl_3$). The er ratio was determined by a Hitachi LaChrom Elite HPLC analysis on a Chiralpak IC column (20% isopropanol in hexane, 1.5 mL/min) with retention time 6.12 min (major) and 9.42 min (minor). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.98 (d, *J* = 8.4 Hz, 2H), 7.30 (d, *J* = 8.0 Hz, 2H), 3.91 (s, 3H), 3.78 (q, *J* = 9.1 Hz, 1H), 3.34–3.27 (m, 1H), 2.33–2.26 (m, 3H), 2.19–2.13 (m, 3H), 1.55–1.48 (m, 2H), 1.32–1.20 (m, 2H), 0.86 (t, *J* = 7.3 Hz, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 210.8, 167.1, 149.3, 130.0, 128.4, 126.8, 53.0, 52.2, 42.1, 40.6, 25.8, 24.8, 22.5, 21.7, 14.0. HRMS (ESI-TOF) Calcd for $C_{17}H_{23}O_3^+$ $[M+H]^+$: 275.1647; found: 275.1646.

The absolute stereochemistry was assigned by analogy to compound **7a** and **7b**.

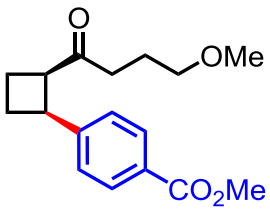


6d

methyl 4-((1*S*,2*R*)-2-(4-phenylbutanoyl)cyclobutyl)benzoate (6d)

Colorless oil (15.8 mg, 47% yield, 98:2 er). $[\alpha]_D^{20}$ = +9.7 (*c* = 1.0, $CHCl_3$). The er ratio was determined by a Hitachi LaChrom Elite HPLC analysis on a Chiralpak IC column (20% isopropanol in hexane, 1.5 mL/min) with retention time 9.05 min (major) and 12.90 min (minor). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.96 (d, *J* = 8.4 Hz, 2H), 7.28–7.25 (m, 4H), 7.20–7.17 (m, 1H), 7.11 (dd, *J* = 7.9, 1.1 Hz, 2H), 3.91 (s, 3H), 3.78–3.71 (m, 1H), 3.30–3.24 (m, 1H), 2.58 (dd, *J* = 8.3, 6.8 Hz, 2H), 2.35–2.22 (m, 3H), 2.19–2.10 (m, 3H), 1.91–1.83 (m, 2H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 149.2, 141.6, 130.0, 128.6, 128.5, 126.8, 126.1, 53.0, 52.2, 42.2, 40.0, 35.2, 25.0, 24.8, 21.6. HRMS (ESI-TOF) Calcd for $C_{22}H_{25}O_3^+$ $[M+H]^+$: 337.1804; found: 337.1799.

The absolute stereochemistry was assigned by analogy to compound **7a** and **7b**.

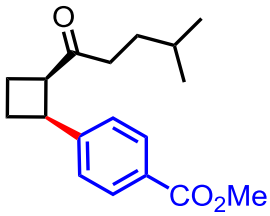


6e

methyl 4-((1*S*,2*R*)-2-(4-methoxybutanoyl)cyclobutyl)benzoate (6e)

Colorless oil (10.1 mg, 35% yield, 98:2 er). $[\alpha]_D^{20}$ = –140.0 (*c* = 1.0, $CHCl_3$). The er ratio was determined by SFC analysis on a Chiralpak AS column (2% isopropanol in CO_2, 2.0 mL/min) with retention time 4.92 min (major) and 10.11 min (minor). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.98 (d, *J* = 8.3 Hz, 2H), 7.30 (d, *J* = 8.6 Hz, 2H), 3.91 (s, 3H), 3.82–3.75 (m, 1H), 3.36–3.29 (m, 3H), 3.28 (s, 3H), 2.40 (qt, *J* = 17.5, 7.1 Hz, 2H), 2.28–2.22 (m, 1H), 2.22–2.12 (m, 3H), 1.83 (p, *J* = 6.5 Hz, 2H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 210.3, 167.2, 149.3, 129.9, 128.4, 126.8, 71.8, 58.6, 53.0, 52.2, 42.0, 37.3, 24.8, 23.6, 21.8. HRMS (ESI-TOF) Calcd for $C_{17}H_{23}O_4^+$ $[M+H]^+$: 291.1596; found: 291.1589.

The absolute stereochemistry was assigned by analogy to compound **7a** and **7b**.



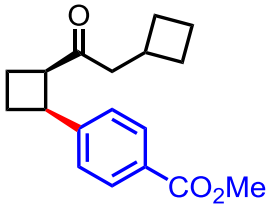
6f

methyl 4-((1*S*,2*R*)-2-(4-methylpentanoyl)cyclobutyl)benzoate (6g)

Colorless oil (13.8 mg, 51% yield, 97:3 er). $[\alpha]_D^{20}$ = –93.3 (*c* = 1.0, $CHCl_3$). The er ratio was determined by SFC analysis on a Chiralpak IC column (10% isopropanol in CO_2, 2.0 mL/min) with retention time 5.99 min (major) and 7.38 min (minor). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.98 (d, *J* = 8.3 Hz, 2H), 7.30 (d, *J* = 8.2 Hz, 2H), 3.91 (s, 3H), 3.78 (q, *J* = 8.2, 7.3 Hz, 1H), 3.36–3.28 (m, 1H), 2.36–2.22 (m, 3H), 2.21–2.12 (m, 3H), 1.46–1.41 (m, 3H), 0.84 (dd, *J* = 6.4, 2.7 Hz, 6H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 211.0, 167.2, 149.3, 130.0,

128.4, 126.8, 53.0, 52.2, 42.2, 38.9, 32.5, 27.8, 24.8, 22.5, 22.4, 21.8. HRMS (ESI-TOF) Calcd for $C_{18}H_{25}O_3^+$ $[M+H]^+$: 273.1491; found: 273.1488.

The absolute stereochemistry was assigned by analogy to compound **7a** and **7b**.

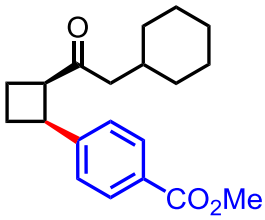


6g

methyl 4-((1*S*,2*R*)-2-(2-cyclobutylacetyl)cyclobutyl)benzoate (6g)

Colorless oil (11.4 mg, 40% yield, 97:3 er). $[\alpha]_D^{20}$ = –70.0 (*c* = 1.0, $CHCl_3$). The er ratio was determined by SFC analysis on a Chiralpak IC column (10% isopropanol in CO_2, 2.0 mL/min) with retention time 8.58 min (major) and 9.82 min (minor). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.98 (d, *J* = 8.3 Hz, 2H), 7.29 (d, *J* = 8.0 Hz, 2H), 3.91 (s, 3H), 3.76 (q, *J* = 8.6 Hz, 1H), 3.29–3.23 (m, 1H), 2.65 (p, *J* = 7.8 Hz, 1H), 2.42 (dd, *J* = 7.4, 4.9 Hz, 2H), 2.27–2.21 (m, 1H), 2.18–2.07 (m, 5H), 1.89–1.77 (m, 2H), 1.64–1.53 (m, 2H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 210.1, 167.2, 149.3, 130.0, 128.4, 126.8, 53.0, 52.2, 48.1, 42.0, 31.4, 28.7, 24.7, 21.7, 18.9. HRMS (ESI-TOF) Calcd for $C_{18}H_{23}O_3^+$ $[M+H]^+$: 287.1647; found: 287.1642.

The absolute stereochemistry was assigned by analogy to compound **7a** and **7b**.



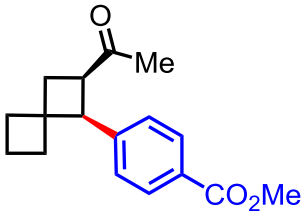
6h

methyl 4-((1*S*,2*R*)-2-(2-cyclohexylacetyl)cyclobutyl)benzoate (6h)

Colorless oil (13.8 mg, 44% yield, 96:4 er). $[\alpha]_D^{20}$ = +11.1 (*c* = 1.0, $CHCl_3$). The er ratio was determined by SFC analysis on a Chiralpak IC column (10% isopropanol in CO_2, 2.0 mL/min) with retention time 9.04 min (major) and 10.30 min (minor). ^{1}H NMR (500 MHz,

$CDCl_3$) δ 7.98 (d, *J* = 8.3 Hz, 2H), 7.29 (d, *J* = 8.3 Hz, 2H), 3.91 (s, 3H), 3.79–3.73 (m, 1H), 3.31–3.25 (m, 1H), 2.27–2.21 (m, 1H), 2.18–2.13 (m, 4H), 1.83–1.75 (m, 1H), 1.68–1.56 (m, 6H), 1.29–1.18 (m, 2H), 1.14–1.05 (m, 1H), 0.90–0.81 (m, 2H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 210.5, 167.2, 149.3, 129.9, 128.4, 126.8, 53.4, 52.2, 48.7, 42.1, 33.7, 33.5, 33.4, 26.3, 26.2, 26.2, 24.7, 21.6. HRMS (ESI-TOF) Calcd for $C_{20}H_{27}O_3^+$ $[M+H]^+$: 315.1960; found: 315.1958.

The absolute stereochemistry was assigned by analogy to compound **7a** and **7b**.

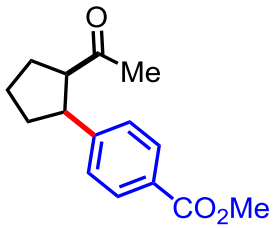


6i

methyl 4-((1*R*,2*R*)-2-acetylspiro[3.3]heptan-1-yl)benzoate (6i)

Colorless oil (17.9 mg, 66% yield, 95:5 er). $[\alpha]_D^{20}$ = +17.7 (*c* = 1.0, $CHCl_3$). The er ratio was determined by SFC analysis on a Chiralpak OD column (2% isopropanol in CO_2, 2.0 mL/min) with retention time 8.56 min (major) and 9.67 min (minor). ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.03 (d, *J* = 8.3 Hz, 2H), 7.28 (d, *J* = 8.3 Hz, 2H), 3.92 (s, 3H), 3.44–3.33 (m, 2H), 2.24 (d, *J* = 8.5 Hz, 2H), 2.03 (s, 3H), 2.01–1.93 (m, 2H), 1.83–1.76 (m, 1H), 1.72–1.68 (m, 2H), 1.54–1.46 (m, 1H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 208.5, 167.1, 145.2, 129.9, 128.7, 127.8, 52.2, 51.9, 45.5, 45.0, 35.1, 33.7, 29.7, 28.2, 16.1. HRMS (ESI-TOF) Calcd for $C_{17}H_{20}O_3^+$[M+H]$^+$: 273.1491; found: 273.1488.

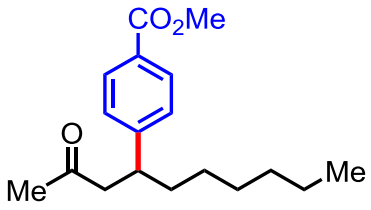
The absolute stereochemistry was assigned by analogy to compound **7a** and **7b**.



6j

methyl 4-(-2-acetylcyclopentyl)benzoate (6j)

Colorless oil, Ag_3PO_4: 10.3 mg, 42% yield, 70:30 er; $[\alpha]_D^{20} = -2.4$ (c = 1.0, $CHCl_3$). AgTFA: 9.1 mg, 37%, 61:39 er. The er ratios were determined by SFC analysis on a Chiralpak IC column (10% isopropanol in CO_2, 2.0 mL/min) with retention time 7.74 min (major), 10.99 min (minor) and 7.73 min (major), 10.97 min (minor), respectively. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.96 (d, J = 8.4 Hz, 2H), 7.30 (d, J = 8.0 Hz, 2H), 3.90 (s, 3H), 3.41–3.34 (m, 1H), 3.03 (td, J = 9.2, 7.7 Hz, 1H), 2.21–2.10 (m, 2H), 2.02 (s, 3H), 1.95–1.87 (m, 2H), 1.83–1.75 (m, 2H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 210.1, 167.1, 150.3, 130.1, 128.5, 127.4, 60.4, 52.2, 48.5, 35.7, 30.2, 30.1, 25.5. HRMS (ESI-TOF) Calcd for $C_{15}H_{19}O_3^+$ $[M+H]^+$: 247.1334; found: 247.1332.



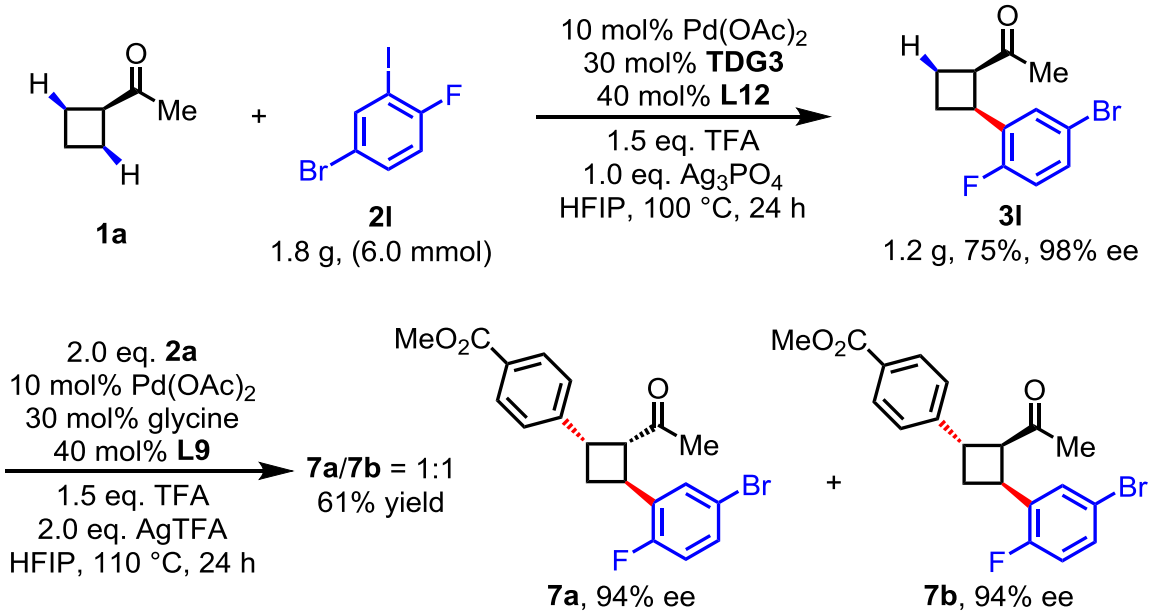
6k

methyl 4-(2-oxodecan-4-yl)benzoate (6k)

Colorless oil (9.3 mg, 32% yield, 60:40 er). The er ratio was determined by SFC analysis on a Chiralpak IC column (10% isopropanol in CO_2, 2.0 mL/min) with retention time 6.15 min (major) and 7.20 min (minor). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.96 (d, J = 8.3 Hz, 2H), 7.25 (d, J = 8.2 Hz, 2H), 3.90 (s, 3H), 3.22–3.15 (m, 1H), 2.73 (d, J = 7.1 Hz, 2H), 2.02 (s, 3H), 1.66–1.59 (m, 2H), 1.29–1.12 (m, 8H), 0.83 (t, J = 7.0 Hz, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 207.5, 167.2, 150.4, 130.0, 128.5, 127.7, 52.2, 50.6, 41.3, 36.4, 31.8, 30.8, 29.3, 27.4, 22.7, 14.2. HRMS (ESI-TOF) Calcd for $C_{18}H_{27}O_3^+$$[M+H]^+$: 291.1955; found: 291.1952.

5. Diverse Chiral Cyclobutanes via Sequential C–H Arylation

Scheme S2



The experiment was performed according to the above-described general procedure.

methyl 4-((1*S*,2*S*,3*S*)-2-acetyl-3-(5-bromo-2-fluorophenyl)cyclobutyl)benzoate (7a)

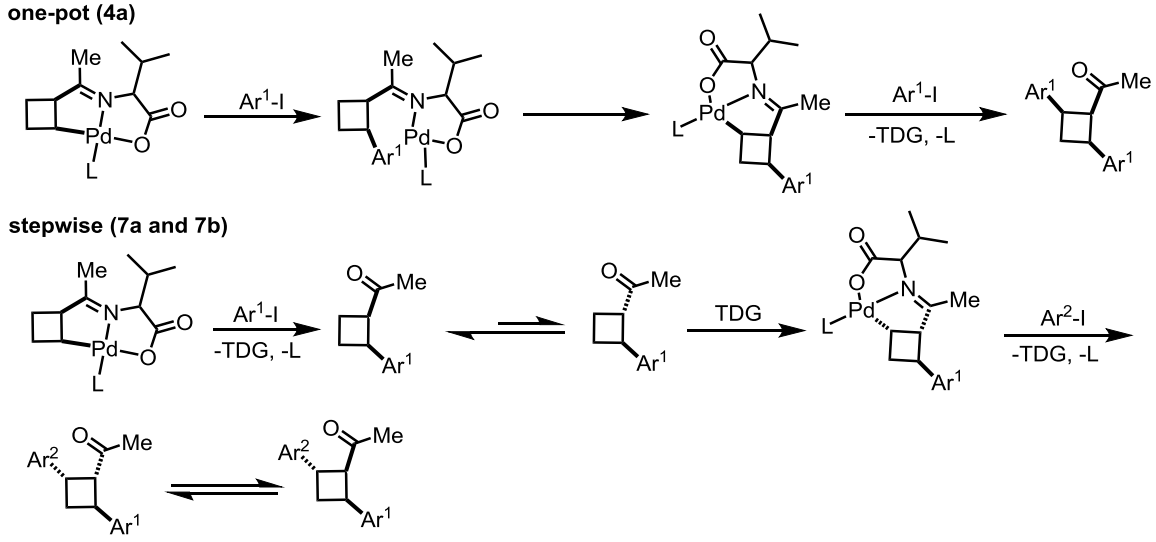
White solid (547 mg, 31.5% yield, 97:3 er), $[\alpha]_D^{20}$ = –3.8 (*c* = 1.0, $CHCl_3$), m.p.: 112−114 °C. The er ratio was determined by SFC analysis on a Chiralpak IC column (10% isopropanol in CO_2, 2.0 mL/min) with retention time 15.93 min (major) and 19.89 min (minor). ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.01 (d, *J* = 8.3 Hz, 2H), 7.40–7.35 (m, 3H), 7.32 (ddd, *J* = 8.7, 4.5, 2.5 Hz, 1H), 6.92 (dd, *J* = 10.0, 8.7 Hz, 1H), 4.41 (q, *J* = 9.1 Hz, 1H), 4.11 (td, *J* = 9.5, 5.4 Hz, 1H), 3.94–3.87 (m, 4H), 2.75–2.63 (m, 2H), 1.68 (s, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 205.6, 166.9, 161.3, 159.3, 146.0, 132.9, 132.8, 131.2, 131.1, 131.1, 130.1, 129.2, 127.9, 117.6, 117.4, 116.8, 56.7, 52.2, 40.6, 32.1, 32.1, 30.8, 30.8, 29.1. HRMS (ESI-TOF) Calcd for $C_{20}H_{19}BrFO_3^+$ $[M+H]^+$: 405.0502; found: 405.0489.

methyl 4-((1*S*,2*R*,3*S*)-2-acetyl-3-(5-bromo-2-fluorophenyl)cyclobutyl)benzoate (7b)

White solid (547mg, 31.5% yield, 97:3 er), $[\alpha]_D^{20}$ = +3.5 (*c* = 1.0, $CHCl_3$), m.p.: 124−126 °C. The er ratio was determined by SFC analysis on a Chiralpak IC column (10% isopropanol in CO_2, 2.0 mL/min) with retention time 12.79 min (minor) and 14.90 min (major). ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.04–7.98 (m, 2H), 7.51 (dd, *J* = 6.7, 2.5 Hz, 0.74H),

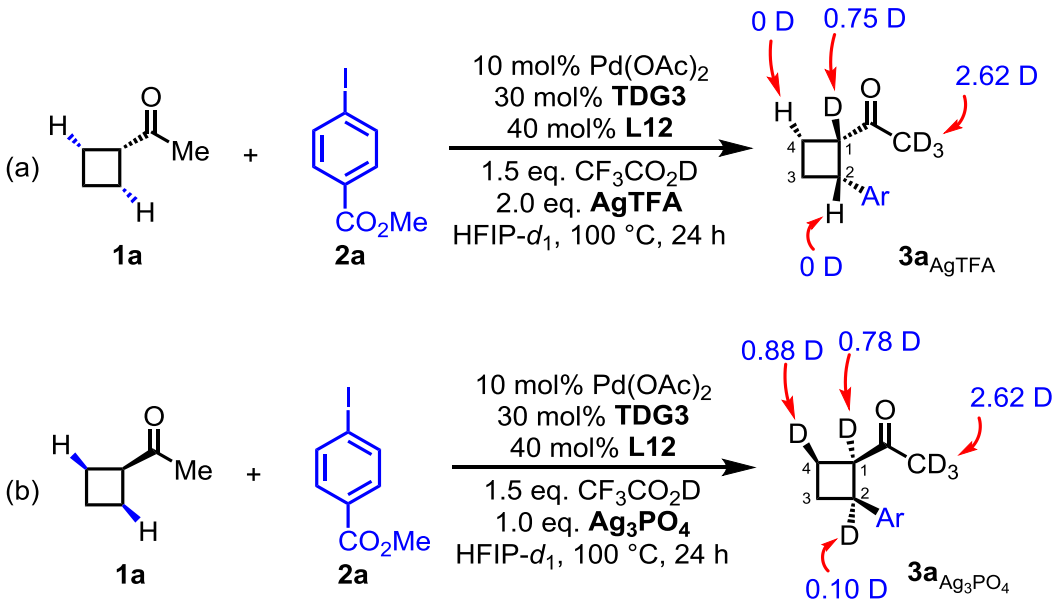
7.39 (dd, *J* = 6.6, 2.5 Hz, 0.28H), 7.38–7.31 (m, 3H), 6.93 (dt, *J* = 9.6, 8.3 Hz, 1H), 4.36–4.23 (m, 1.48H), 3.92 (s, 3H), 3.82–3.76 (m, 1H), 3.70 (q, *J* = 9.3 Hz, 0.30H), 3.47 (t, *J* = 9.6 Hz, 0.29H), 2.77–2.61 (m, 1.81H), 2.34 (q, *J* = 10.5 Hz, 0.29H), 2.03 (s, 0.80H), 1.85 (s, 2.15H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 206.8, 206.1, 167.1, 160.7, 158.8, 148.9, 147.8, 131.7, 131.7, 131.5, 131.5, 130.2, 130.1, 128.5, 127.0, 126.7, 117.2, 117.0, 59.5, 57.7, 52.2, 39.9, 37.4, 32.5, 32.5, 29.1, 28.9. HRMS (ESI-TOF) Calcd for $C_{20}H_{19}BrFO_3^+$ $[M+H]^+$: 405.0502; found: 405.0489.

Scheme S3 Proposed Mechanism of Two Different Di-Arylation Precedure



6. Deuterium-Labeling Experiments

Scheme S4



The experiment was performed according to the above-described general procedure using CF_3CO_2D and HFIP-d_1.

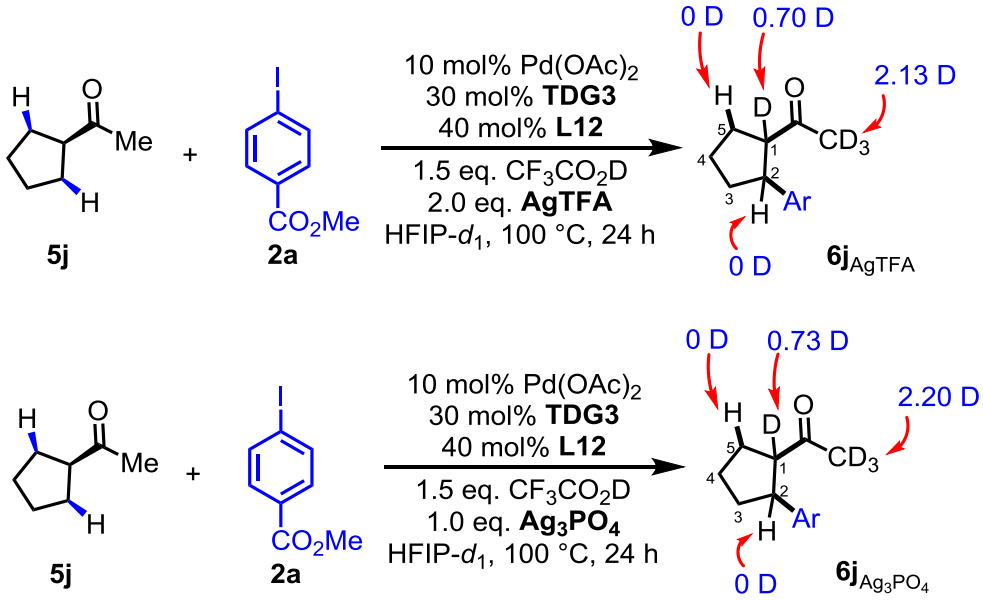
***d*-methyl 4-(2-acetylcyclobutyl)benzoate ($3a_{AgTFA}$)**

^{1}H NMR (500 MHz, $CDCl_3$) δ 8.03–7.99 (m, 2H), 7.35–7.30 (m, 1.81H), 3.93 (s, 3H), 3.79 (q, J = 7.6 Hz, 1H), 3.38–3.29 (m, 0.25H), 2.31–2.14 (m, 4H), 2.07–2.03 (m, 0.38H).

***d*-methyl 4-(2-acetylcyclobutyl)benzoate ($3a_{Ag_3PO_4}$)**

^{1}H NMR (500 MHz, $CDCl_3$) δ 8.03–7.96 (m, 2H), 7.35–7.30 (m, 1.08H), 3.93 (s, 3H), 3.78 (t, J = 8.8 Hz, 0,90H), 3.32 (t, J = 8.8 Hz, 0.22H), 2.32–2.13 (m, 3.12H), 2.07–2.03 (m, 0.38H).

Scheme S5



The experiment was performed according to the above-described general procedure using CF_3CO_2D and HFIP-d_1.

***d*-methyl 4-(2-acetylcyclopentyl)benzoate (6j$_{AgTFA}$)**

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.96 (t, *J* = 3.4 Hz, 2H), 7.30 (d, *J* = 8.4 Hz, 0.94H), 3.90 (s, 3H), 3.36 (t, *J* = 8.5 Hz, 1H), 3.03 (q, J = 8.8 Hz, 0.30H), 2.23–2.16 (m,1H), 2.14 – 2.08 (m, 1H), 2.03–1.97 (m, 0.87H), 1.94–1.86 (m, 2H), 1.83–1.74 (m, 2H).

***d*-methyl 4-(2-acetylcyclopentyl)benzoate (6j$_{Ag3PO4}$)**

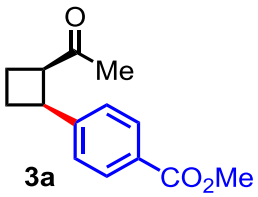
^{1}H NMR (500 MHz, $CDCl_3$) δ 7.99–7.93 (m, 2H), 7.30 (d, *J* = 8.4 Hz, 0.65H), 3.90 (s, 3H), 3.36 (t, *J* = 8.5 Hz, 1H), 3.03 (q, *J* = 8.5 Hz, 0.27H), 2.23–2.16 (m, 1H), 2.15–2.05 (m, 1H), 2.04–1.98 (m, 0.90H), 1.95–1.84 (m, 2H), 1.84–1.72 (m, 2H).

7. References

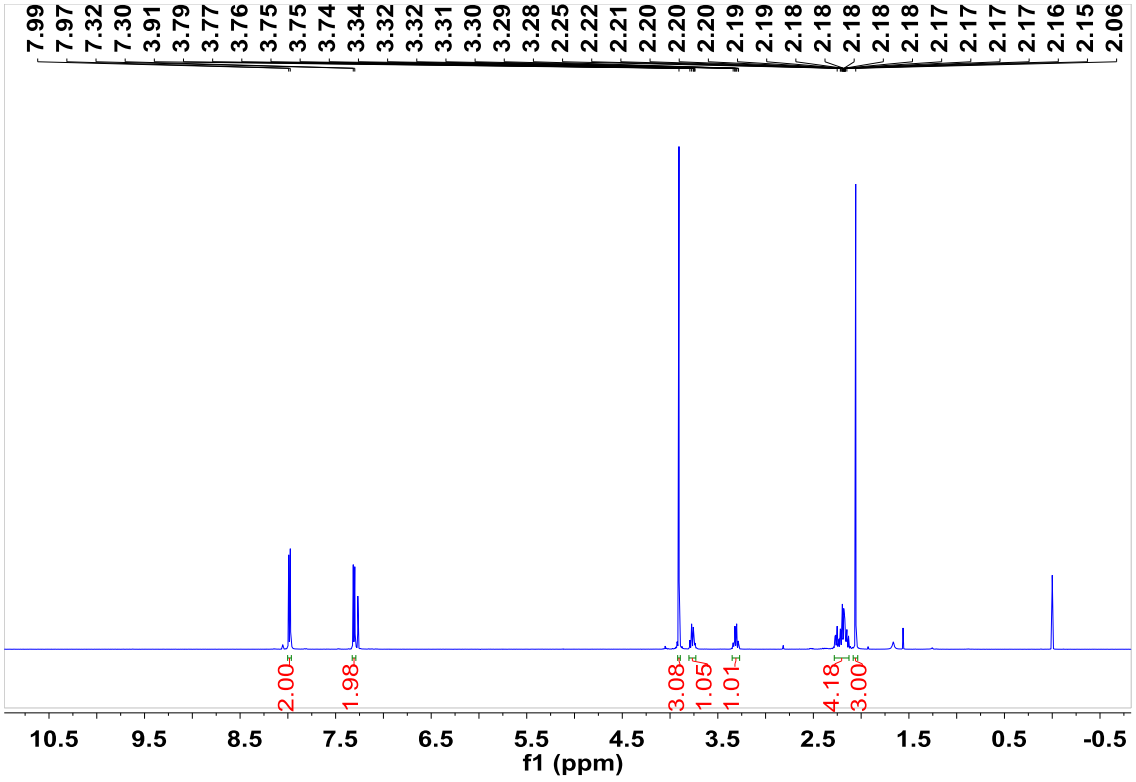
(1) Hong, K.; Park, H.; Yu, J.-Q. Methylene C(sp^3)−H Arylation of Aliphatic Ketones Using a Transient Directing Group. *ACS Catal.* **2017**, *7*, 6938.

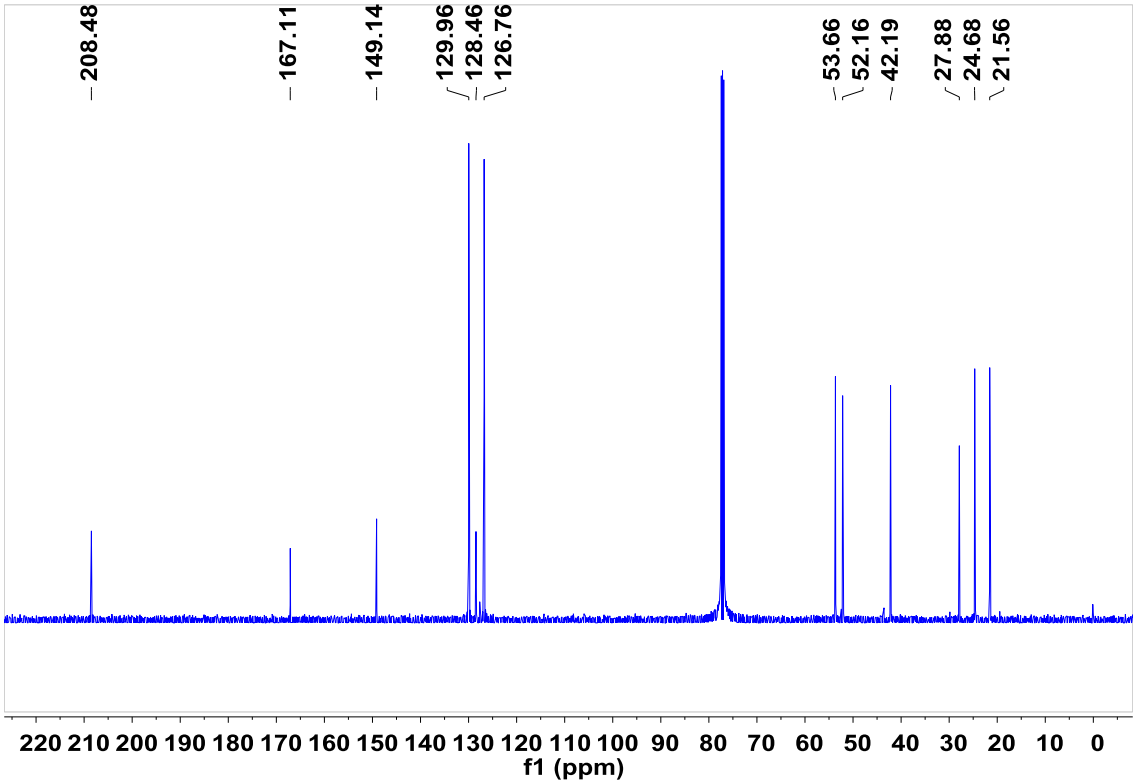
(2) Zhang, F.-L.; Hong, K.; Li, T.-J.; Park, H.; Yu, J.-Q. Functionalization of C(sp^3)−H Bonds Using a Transient Directing Group. *Science* **2016**, *351*, 252.

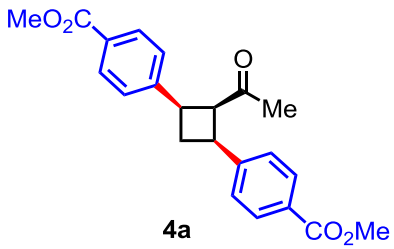
8. NMR spectra



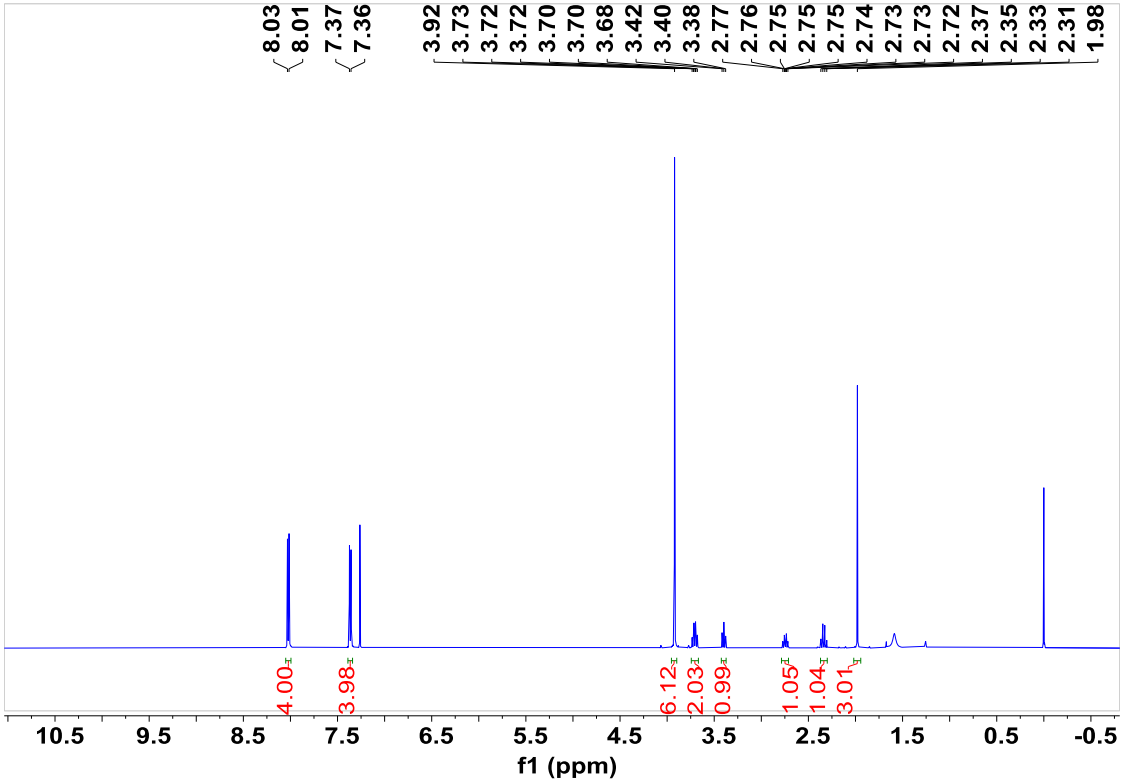
3a

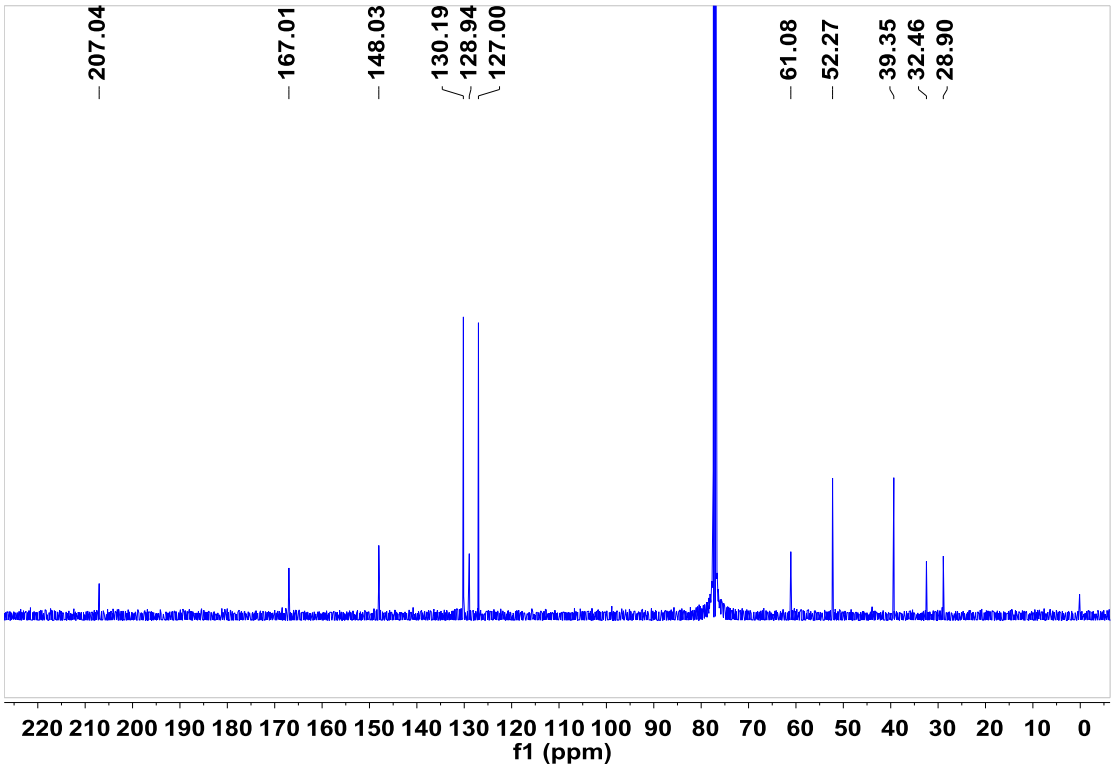


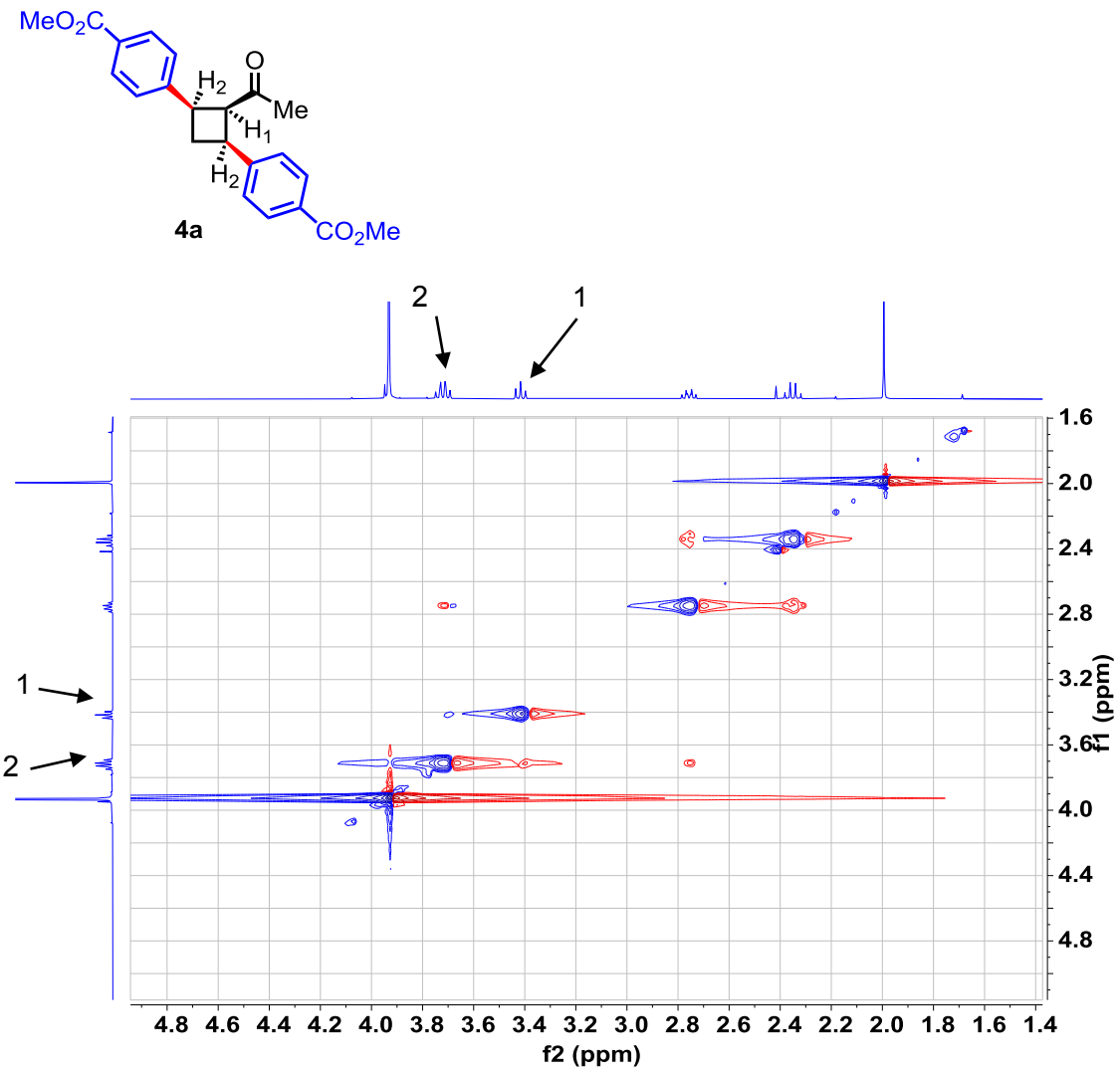



MeO2C
O
Me
CO2Me

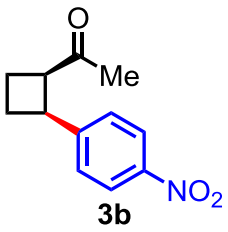
4a


8.03
8.01
7.37
7.36
3.92
3.73
3.72
3.72
3.70
3.70
3.68
3.42
3.40
3.38
2.77
2.76
2.75
2.75
2.75
2.74
2.73
2.73
2.72
2.37
2.35
2.33
2.31
1.98
4.00
3.98
6.12
2.03
0.99
1.05
1.04
3.01
10.5
9.5
8.5
7.5
6.5
5.5
4.5
3.5
2.5
1.5
0.5
-0.5
f1 (ppm)

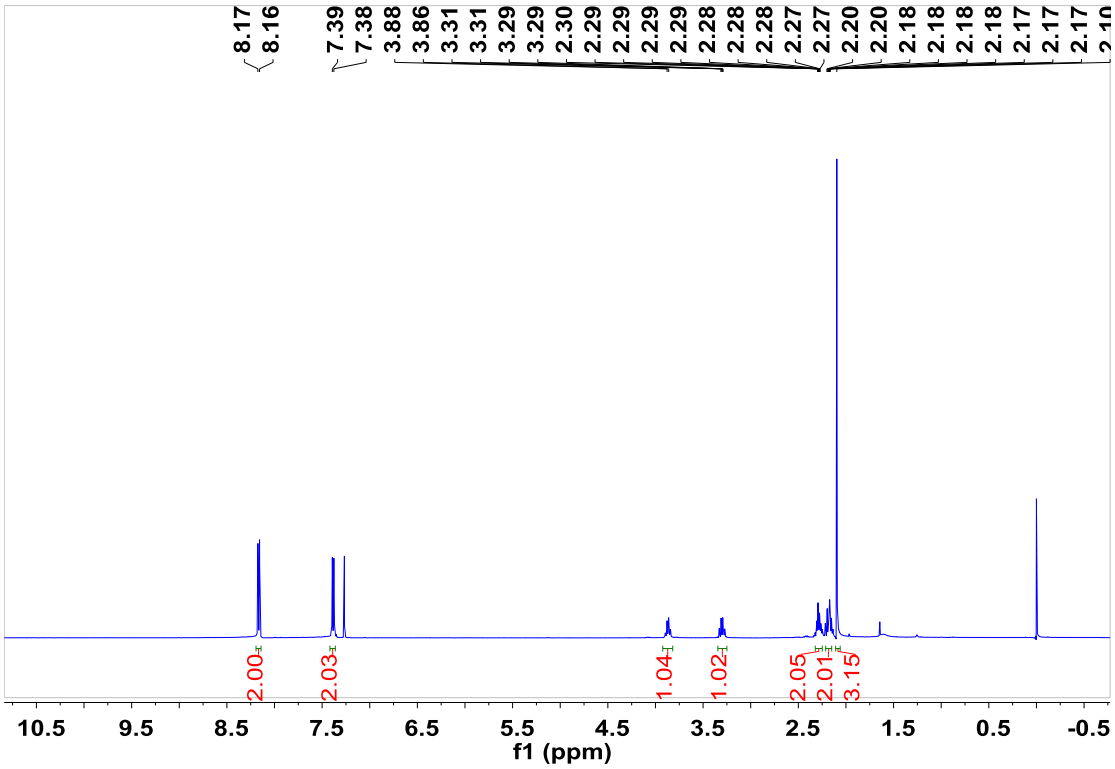

207.04
167.01
148.03
130.19
128.94
127.00
61.08
52.27
39.35
32.46
28.90
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

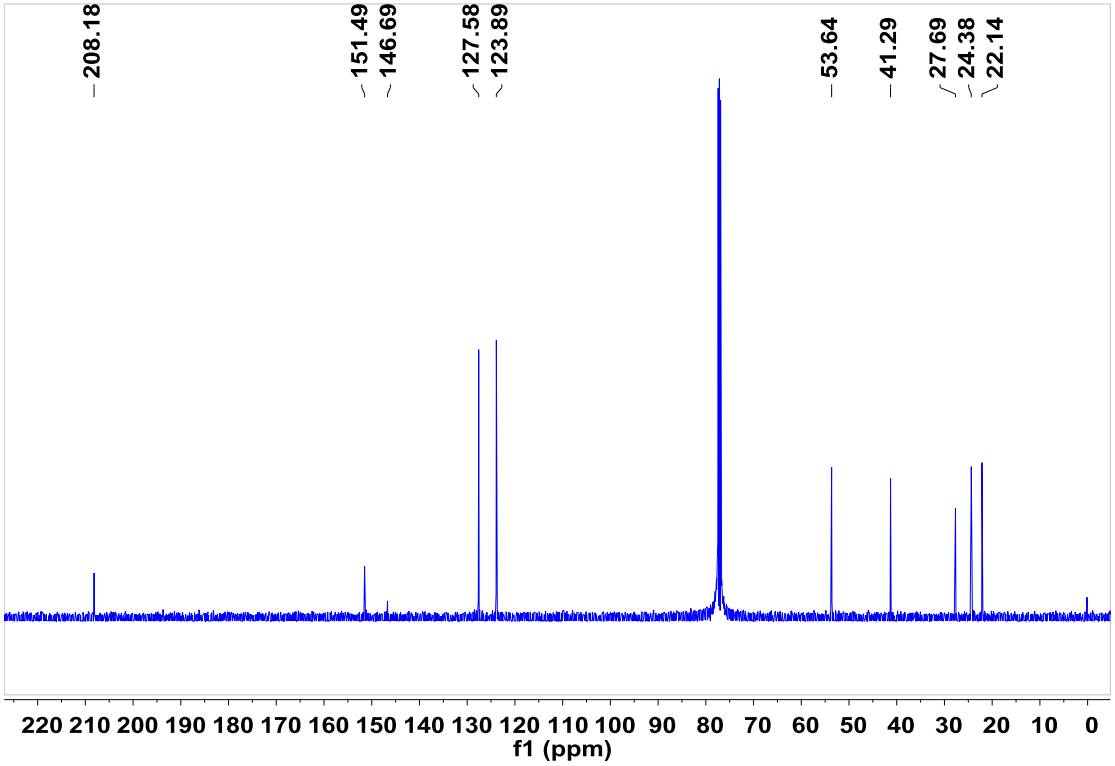


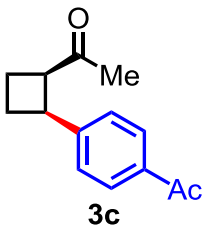
NOE 2D spectrum for **4a**


O
Me
NO2

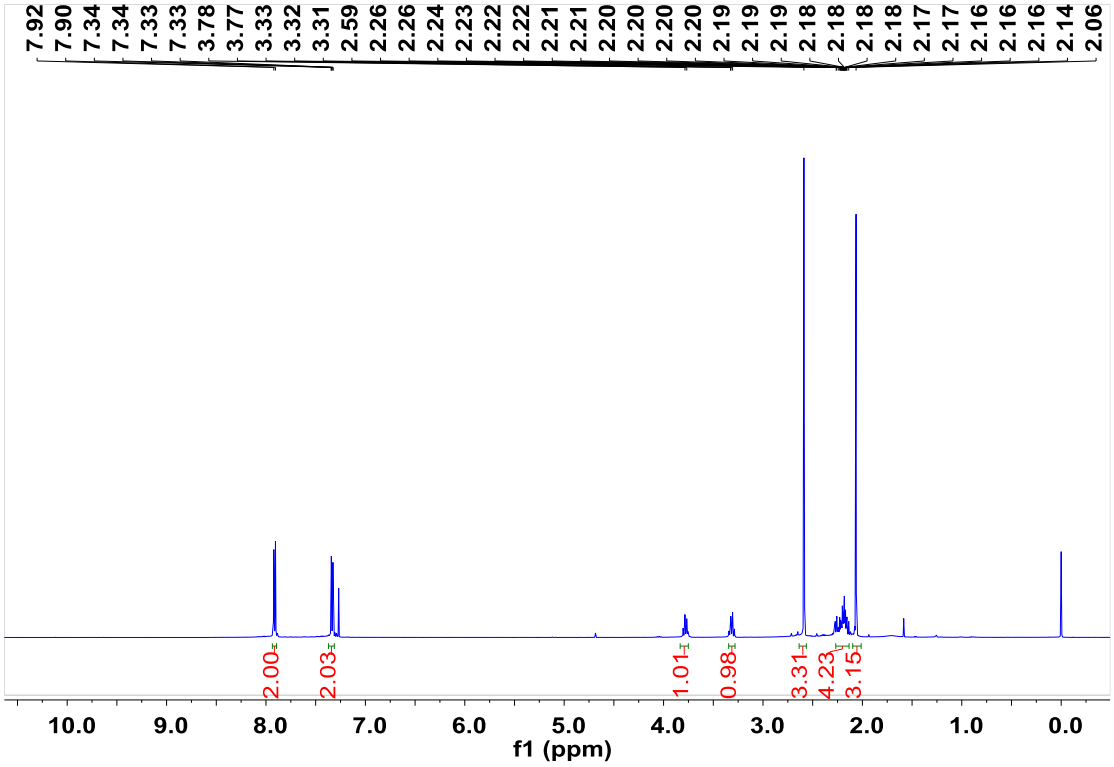
3b

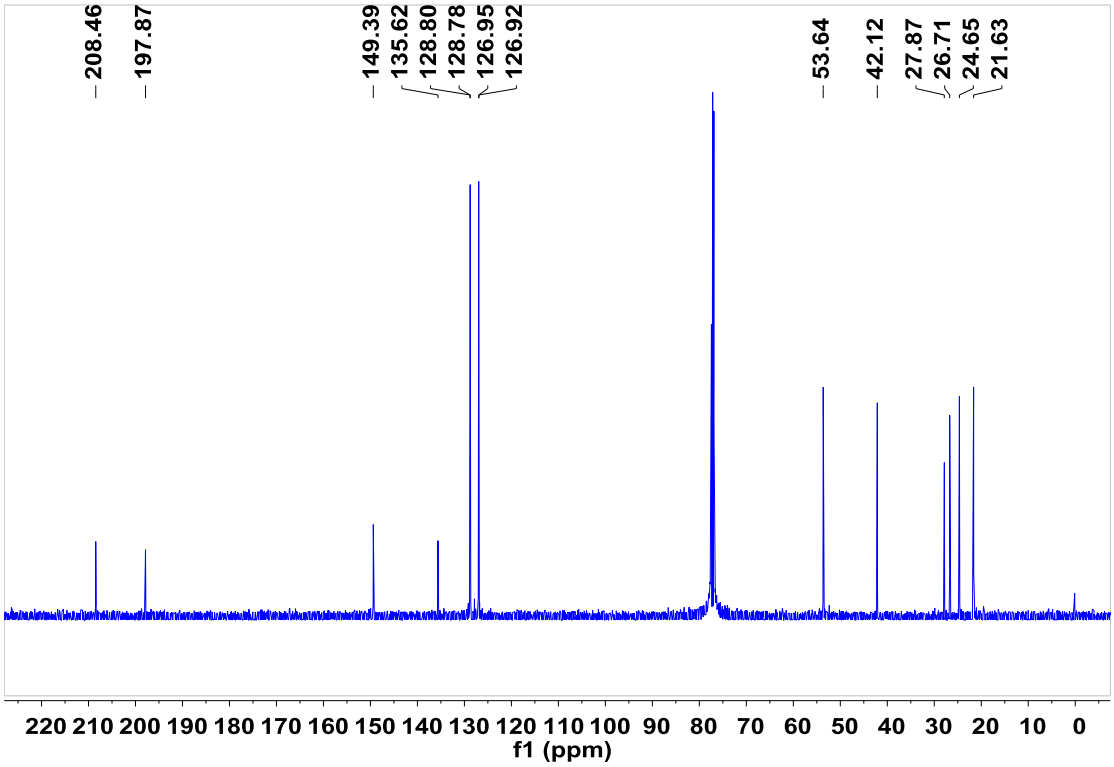

8.17
8.16
7.39
7.38
3.88
3.86
3.31
3.31
3.29
3.29
2.30
2.29
2.29
2.29
2.29
2.28
2.28
2.28
2.27
2.27
2.20
2.20
2.18
2.18
2.18
2.18
2.17
2.17
2.17
2.00
2.03
1.04
1.02
2.05
2.01
3.15
10.5
9.5
8.5
7.5
6.5
5.5
4.5
3.5
2.5
1.5
0.5
-0.5
f1 (ppm)

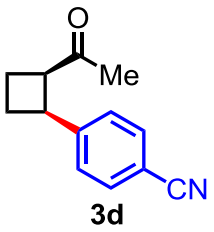

208.18
151.49
146.69
127.58
123.89
53.64
41.29
27.69
24.38
22.14
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


O
Me
Ac

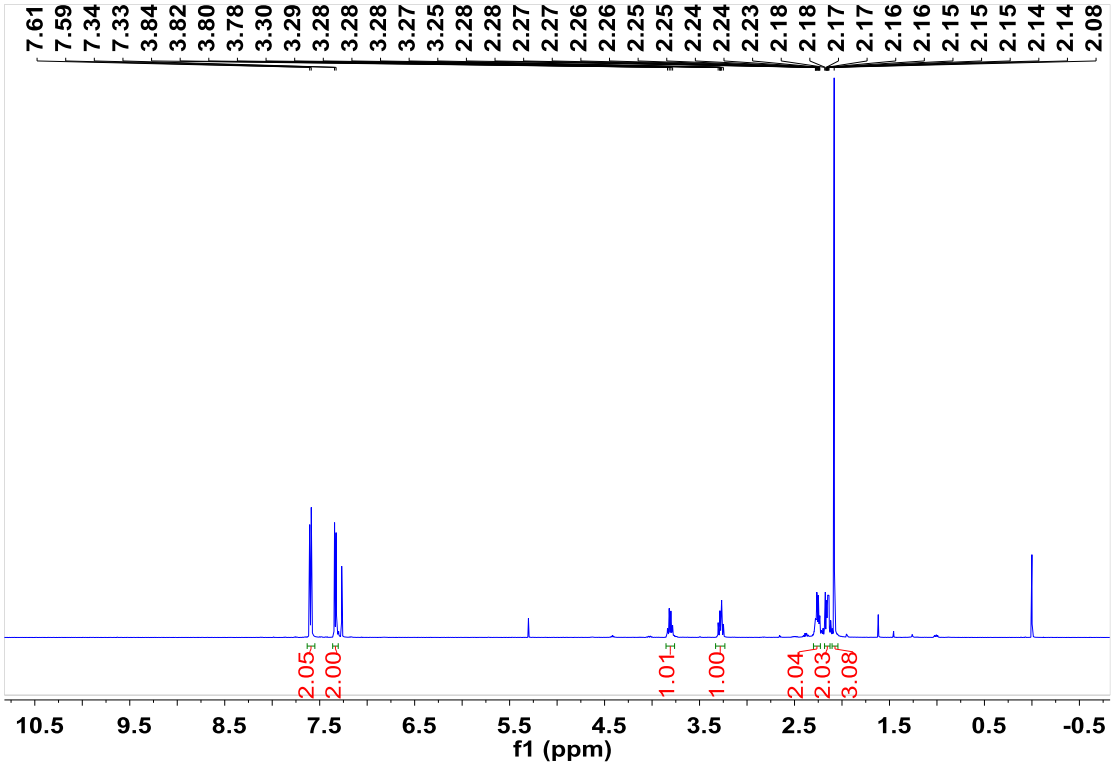
3c

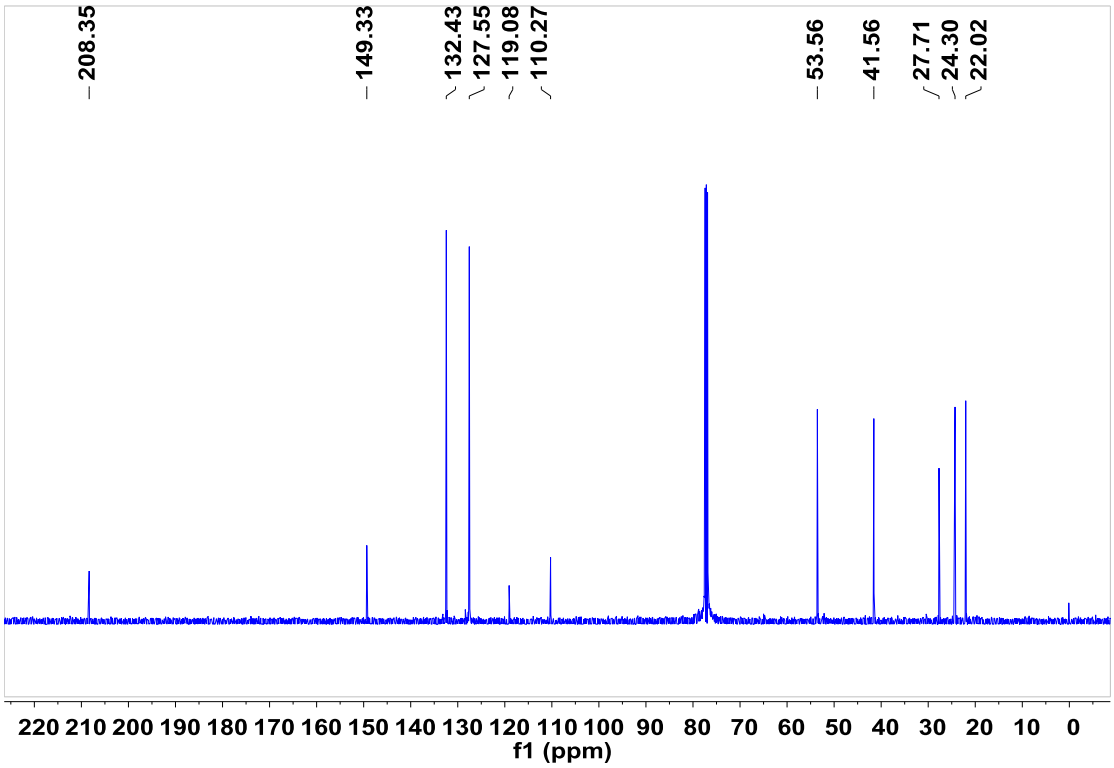

7.92 7.90 7.34 7.34 7.33 7.33 3.78 3.77 3.33 3.32 3.31 2.59 2.26 2.26 2.24 2.23 2.22 2.22 2.21 2.21 2.20 2.20 2.20 2.20 2.19 2.19 2.19 2.18 2.18 2.18 2.17 2.17 2.16 2.16 2.16 2.14 2.06
2.00 2.03 1.01 0.98 3.31 4.23 3.15
10.0 9.0 8.0 7.0 6.0 5.0 4.0 3.0 2.0 1.0 0.0
f1 (ppm)


208.46 197.87 149.39 135.62 128.80 128.78 126.95 126.92 53.64 42.12 27.87 26.71 24.65 21.63
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


O
Me
CN

3d


7.61 7.59 7.34 7.33 3.84 3.82 3.80 3.78 3.30 3.29 3.28 3.28 3.28 3.27 3.25 2.28 2.28 2.27 2.27 2.26 2.26 2.25 2.25 2.24 2.24 2.23 2.18 2.18 2.17 2.17 2.16 2.16 2.15 2.15 2.15 2.14 2.14 2.08
2.05 2.00 1.01 1.00 2.04 2.03 3.08
10.5 9.5 8.5 7.5 6.5 5.5 4.5 3.5 2.5 1.5 0.5 -0.5
f1 (ppm)


208.35 149.33 132.43 127.55 119.08 110.27 53.56 41.56 27.71 24.30 22.02
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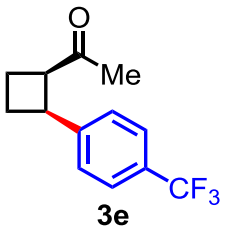
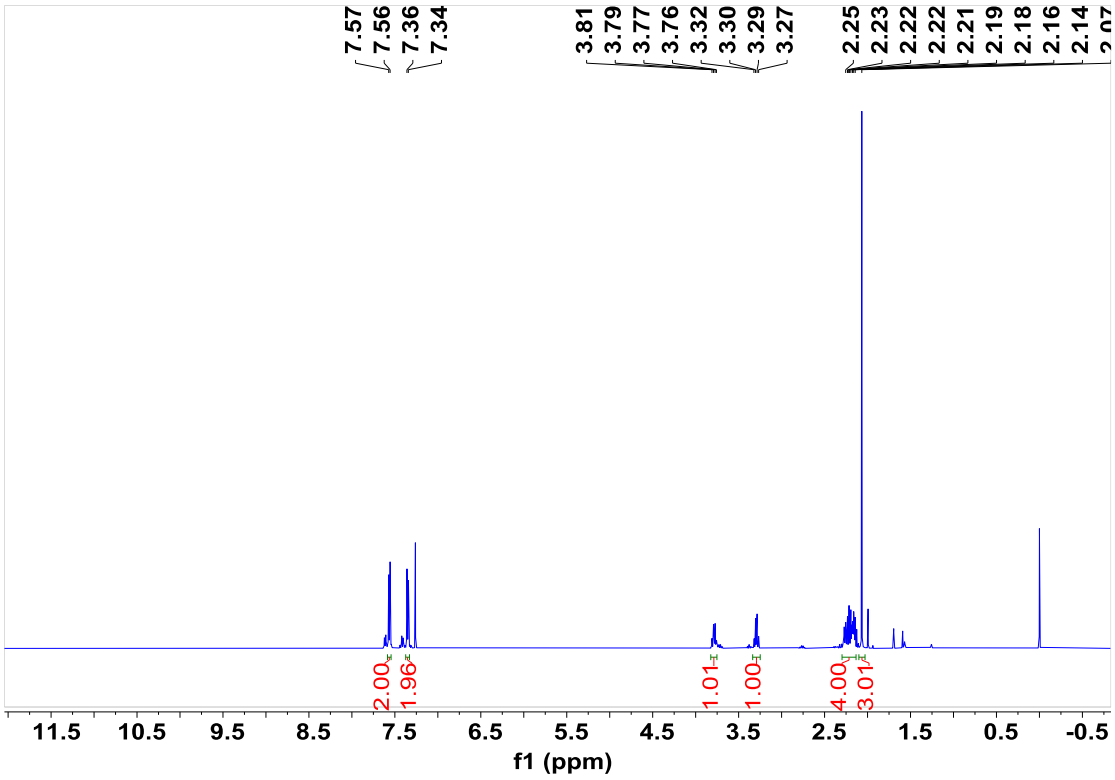
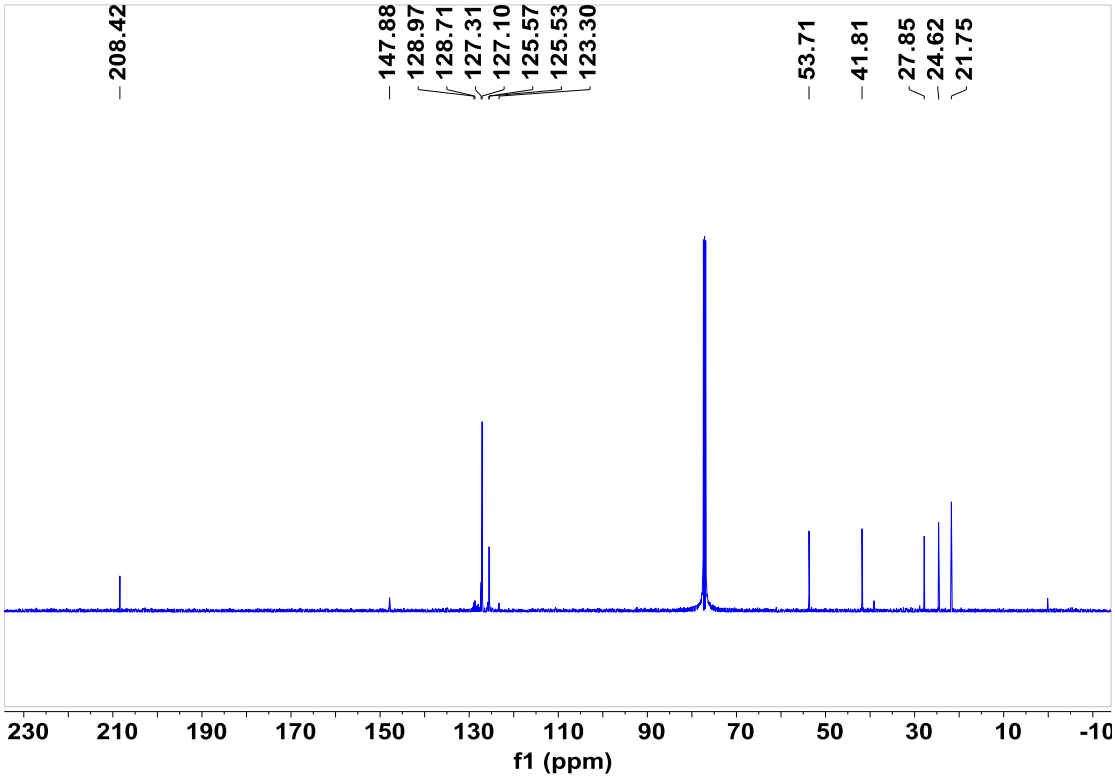
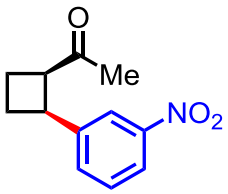
f1 (ppm)


O
Me
CF3

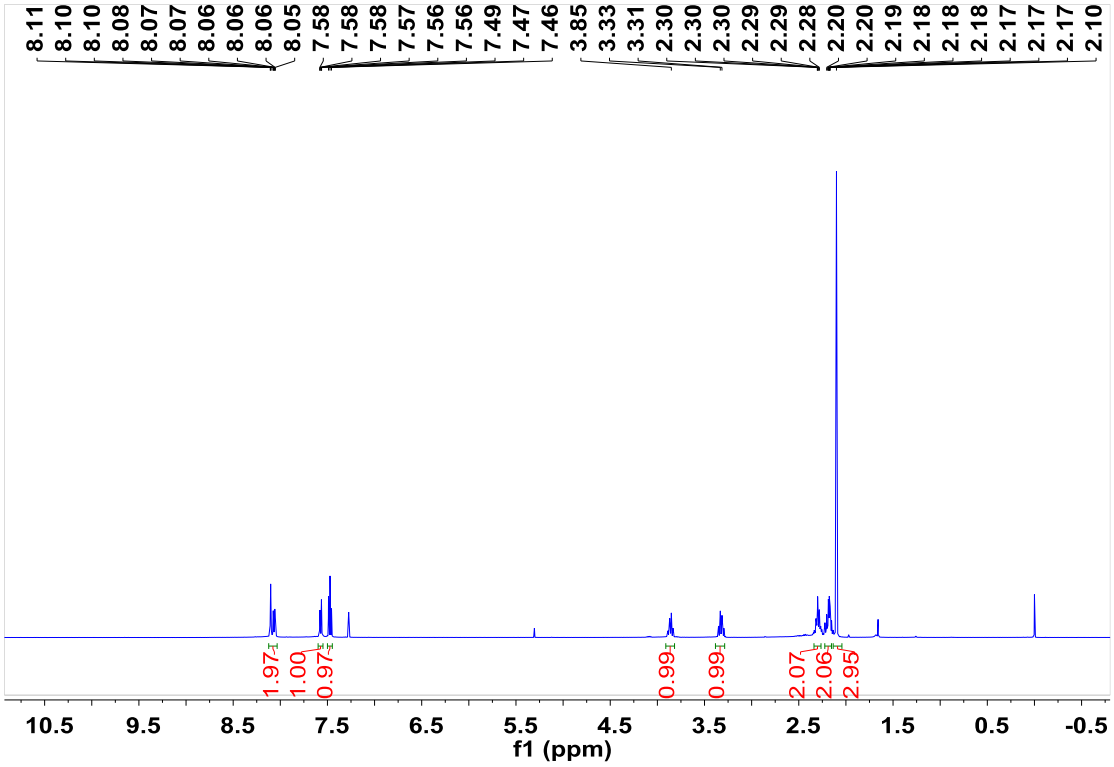
3e

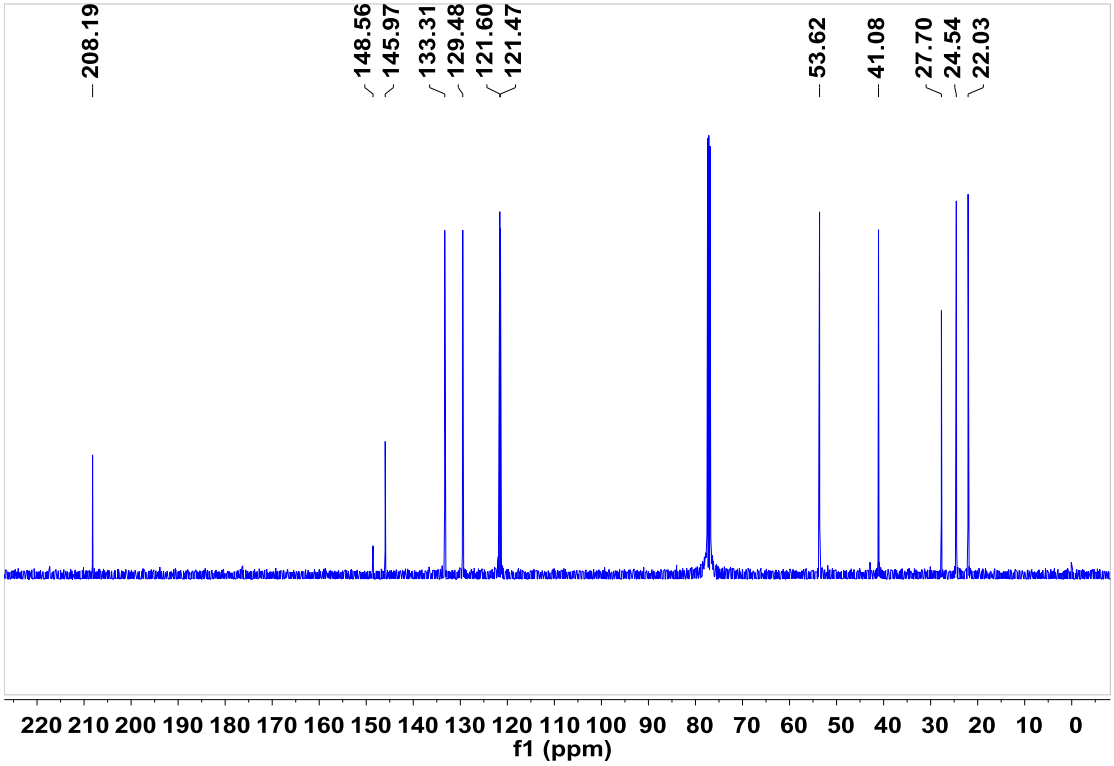

7.57
7.56
7.36
7.34
3.81
3.79
3.77
3.76
3.32
3.30
3.29
3.27
2.25
2.23
2.22
2.22
2.21
2.19
2.18
2.16
2.14
2.00
1.96
1.01
1.00
4.00
3.01
11.5
10.5
9.5
8.5
7.5
6.5
5.5
4.5
3.5
2.5
1.5
0.5
-0.5
f1 (ppm)

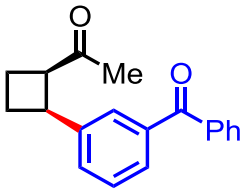

208.42
147.88
128.97
128.71
127.31
127.10
125.57
125.53
123.30
53.71
41.81
27.85
24.62
21.75
230
210
190
170
150
130
110
90
70
50
30
10
-10
f1 (ppm)


O
Me
NO2

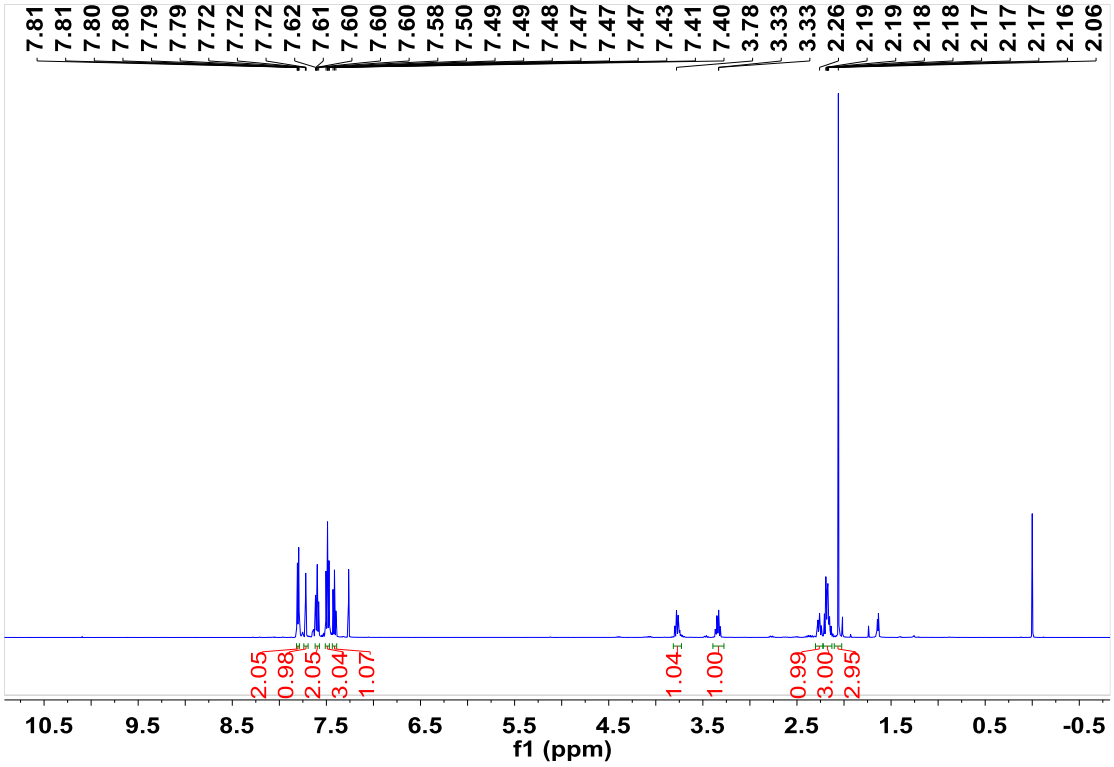
3f

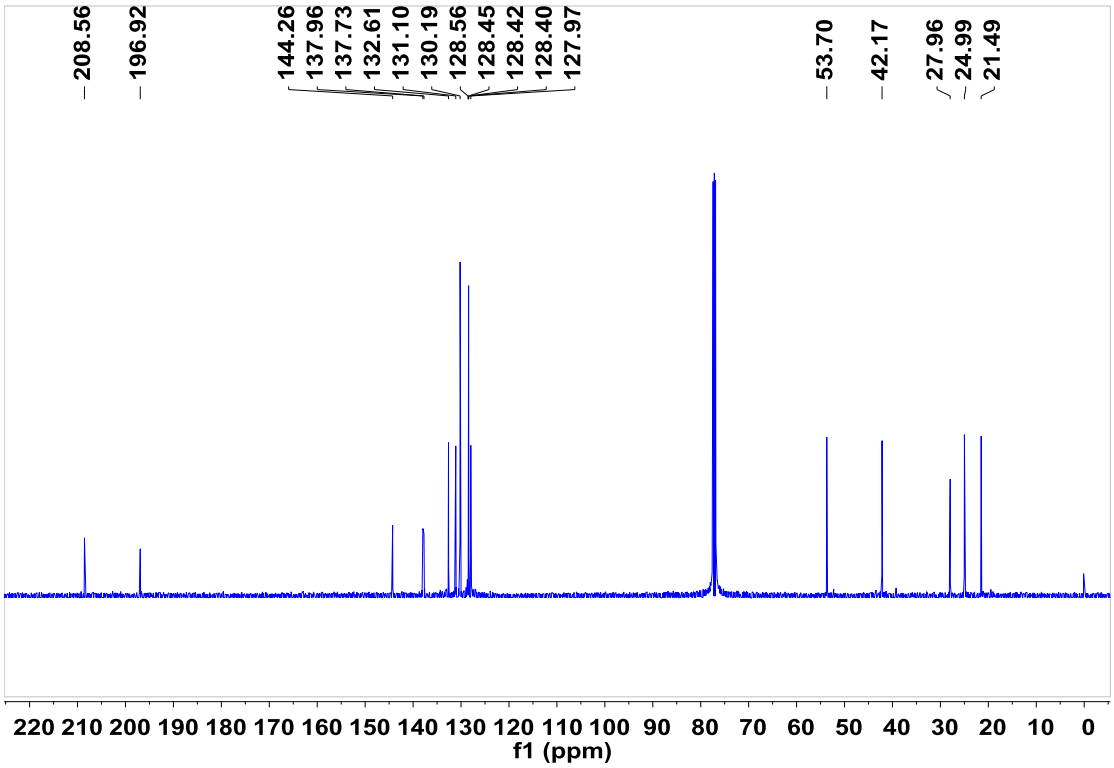

8.11 8.10 8.10 8.08 8.07 8.07 8.06 8.06 8.06 8.05
7.58 7.58 7.58 7.57 7.56 7.56 7.49 7.47 7.46
3.85 3.33 3.31 2.30 2.30 2.30 2.29 2.29 2.28
2.20 2.20 2.19 2.18 2.18 2.18 2.17 2.17 2.17 2.10
1.97 1.00 0.97 0.99 0.99 2.07 2.06 2.95
10.5 9.5 8.5 7.5 6.5 5.5 4.5 3.5 2.5 1.5 0.5 -0.5
f1 (ppm)

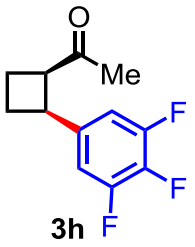

208.19
148.56 145.97 133.31 129.48 121.60 121.47
53.62 41.08 27.70 24.54 22.03
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


O
Me
O
Ph

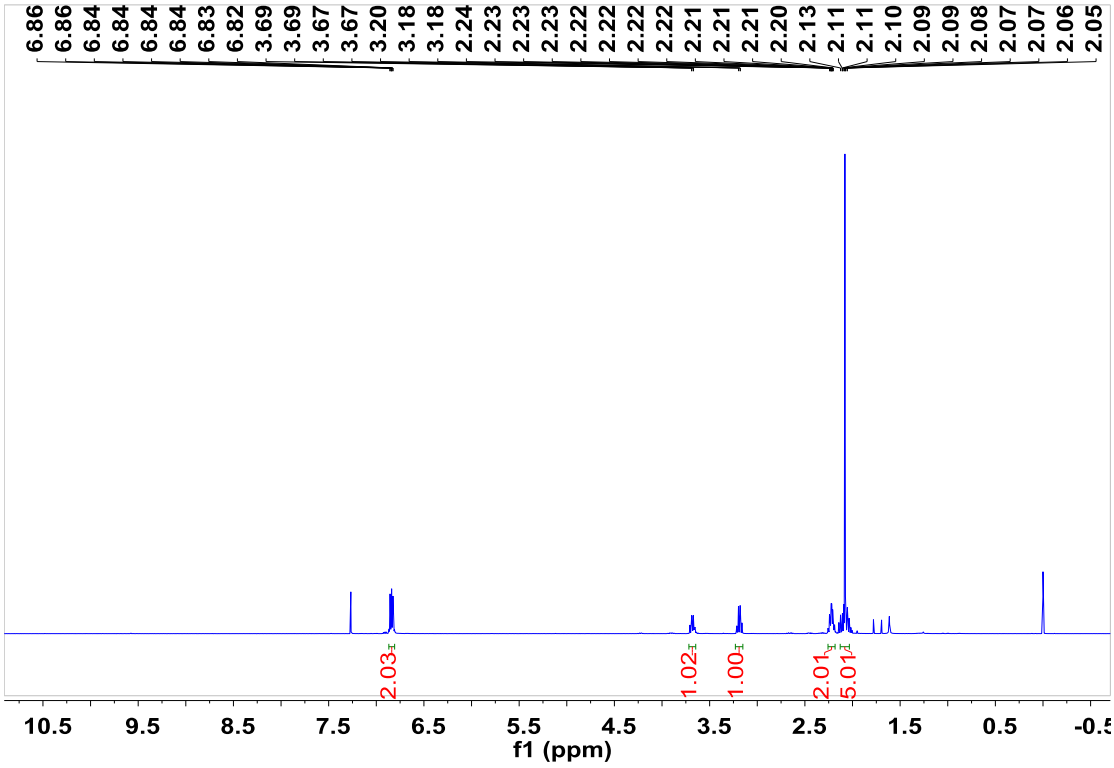
3g

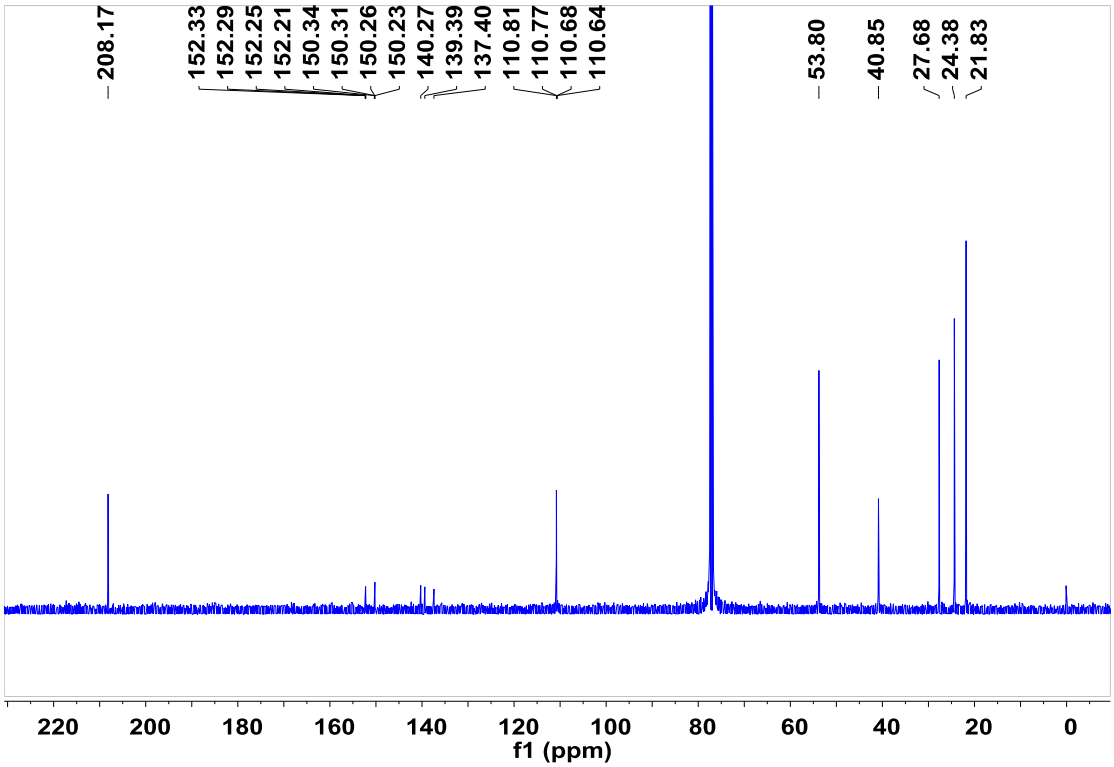

7.81 7.81 7.80 7.80 7.79 7.79 7.72 7.72 7.72 7.62 7.61 7.60 7.60 7.60 7.58 7.50 7.49 7.49 7.48 7.47 7.47 7.47 7.43 7.41 7.40 3.78 3.33 3.33 2.26 2.19 2.19 2.18 2.18 2.17 2.17 2.17 2.16 2.06
2.05 0.98 2.05 3.04 1.07 1.04 1.00 0.99 3.00 2.95
10.5 9.5 8.5 7.5 6.5 5.5 4.5 3.5 2.5 1.5 0.5 -0.5
f1 (ppm)

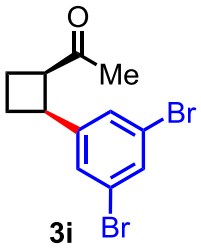

208.56 196.92 144.26 137.96 137.73 132.61 131.10 130.19 128.56 128.45 128.42 128.40 127.97 53.70 42.17 27.96 24.99 21.49
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


O
Me
F
F
F

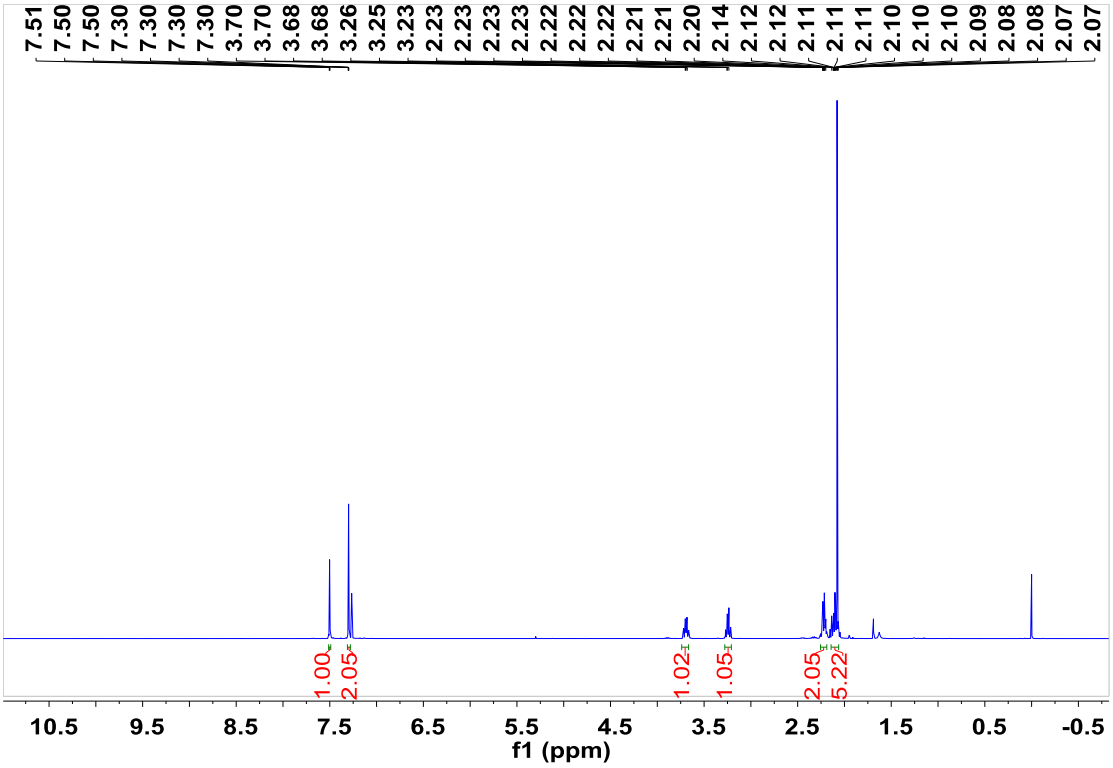
3h

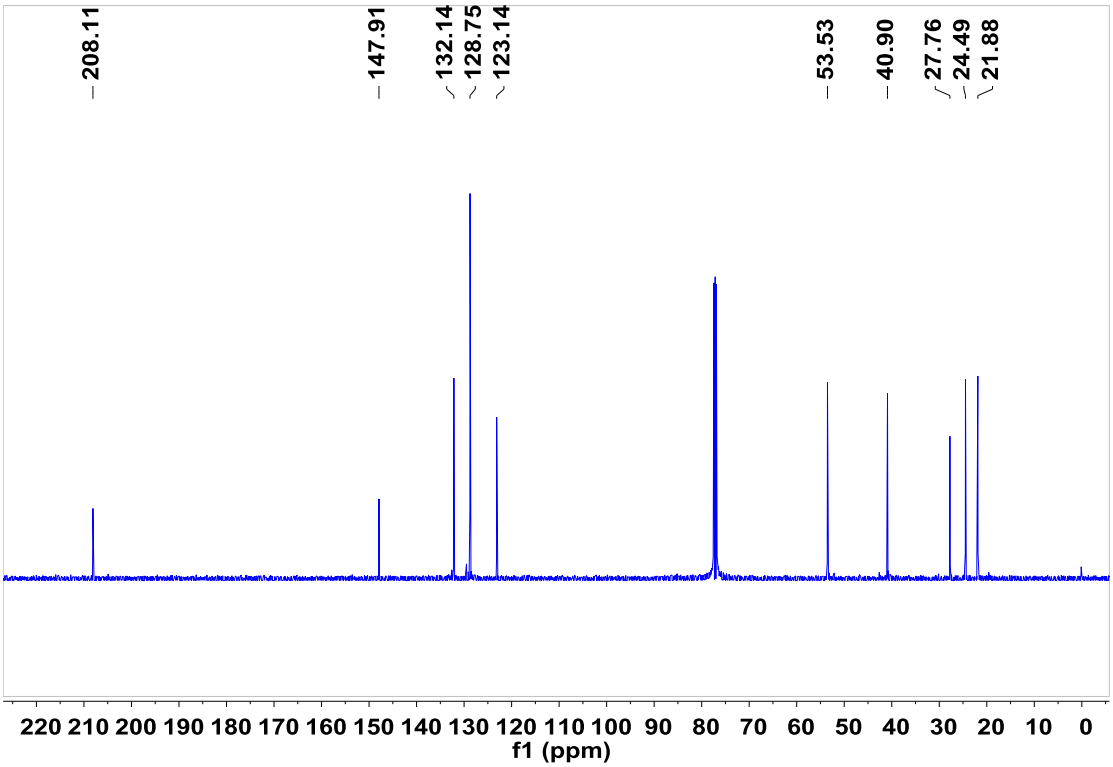

6.86 6.86 6.84 6.84 6.84 6.84 6.83 6.82 3.69 3.69 3.67 3.67 3.20 3.18 3.18 2.24 2.23 2.23 2.23 2.22 2.22 2.22 2.22 2.21 2.21 2.21 2.20 2.13 2.11 2.11 2.10 2.09 2.09 2.08 2.07 2.07 2.06 2.05
2.03
1.02
1.00
2.01
5.01
10.5 9.5 8.5 7.5 6.5 5.5 4.5 3.5 2.5 1.5 0.5
f1 (ppm)


208.17
152.33 152.29 152.25 152.21 150.34 150.31 150.26 150.23 140.27 139.39 137.40 110.81 110.77 110.68 110.64
53.80
40.85
27.68
24.38
21.83
220 200 180 160 140 120 100 80 60 40 20 0
f1 (ppm)


O
Me
Br
Br

3i


7.51 7.50 7.50 7.30 7.30 7.30 7.30 3.70 3.70 3.68 3.68 3.26 3.25 3.23 2.23 2.23 2.23 2.23 2.22 2.22 2.22 2.21 2.21 2.20 2.14 2.12 2.12 2.11 2.11 2.11 2.10 2.10 2.10 2.09 2.08 2.08 2.07 2.07
1.00
2.05
1.02
1.05
2.05
5.22
10.5 9.5 8.5 7.5 6.5 5.5 4.5 3.5 2.5 1.5 0.5 -0.5
f1 (ppm)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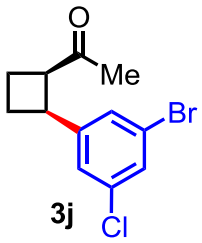
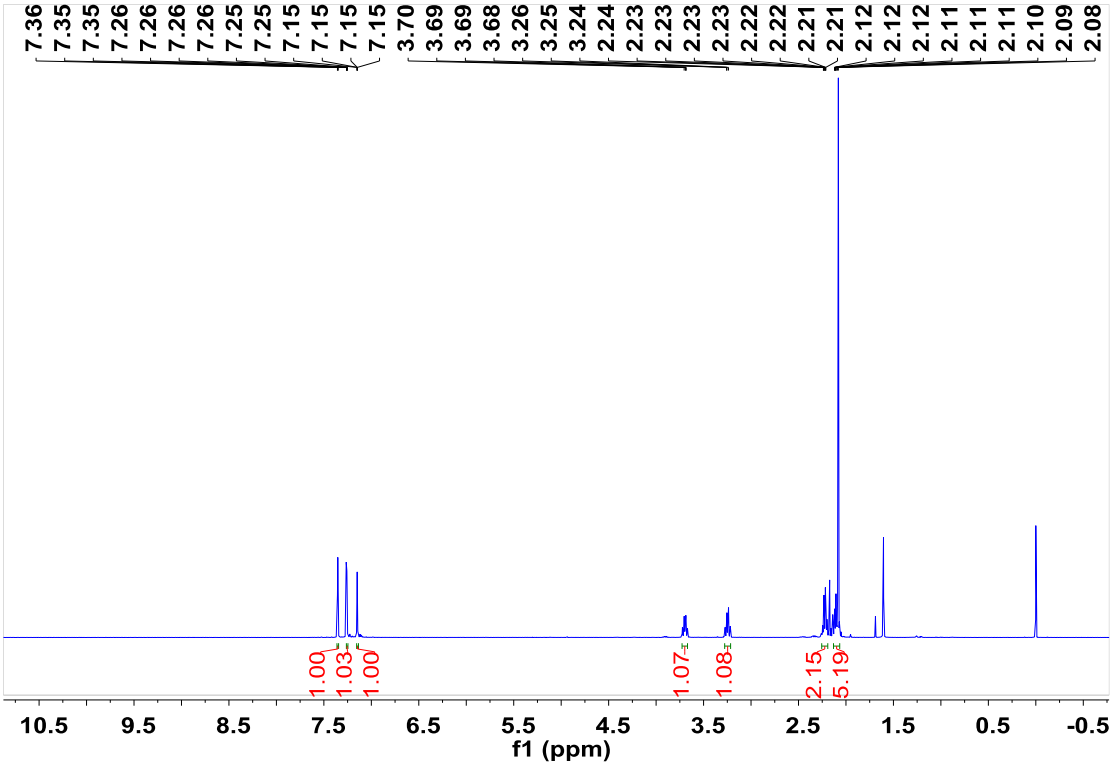
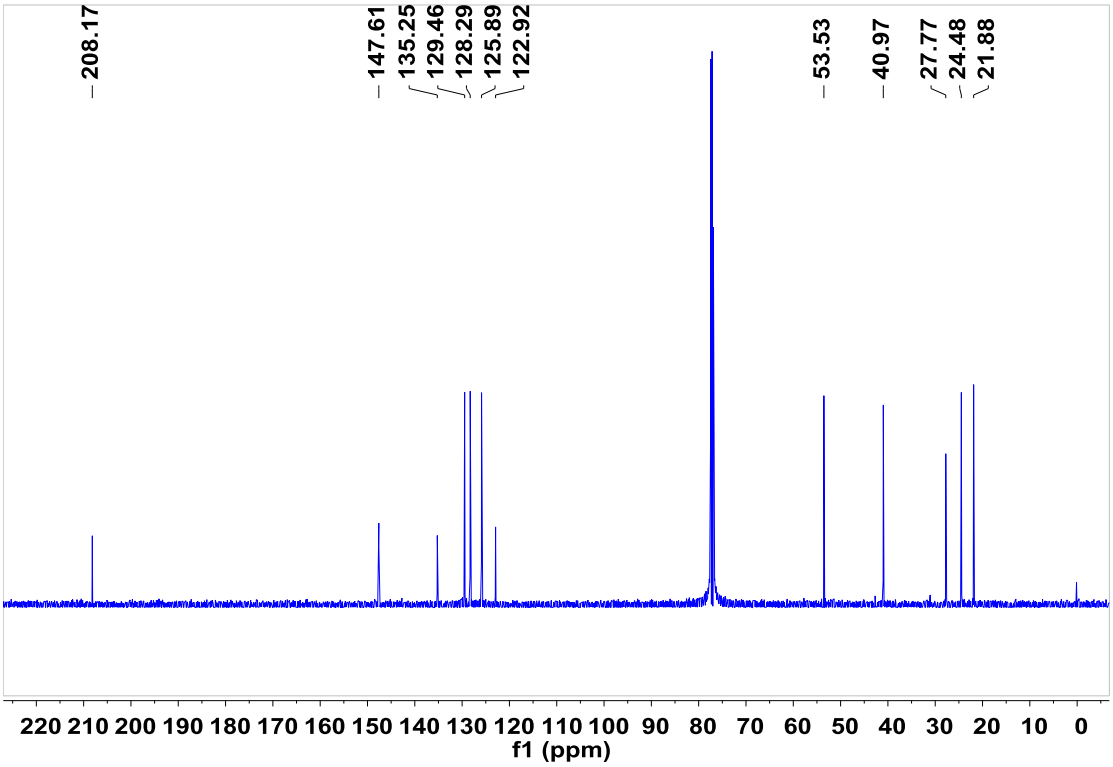
147.91
132.14
128.75
123.14
53.53
40.90
27.76
24.49
21.88
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


O
Me
Br
Cl

3j


7.36 7.35 7.35 7.26 7.26 7.26 7.26 7.25 7.25 7.15 7.15 7.15 7.15
3.70 3.69 3.69 3.68 3.26 3.25 3.24 2.24 2.23 2.23 2.23 2.23 2.22 2.22 2.21 2.21 2.12 2.12 2.12 2.11 2.11 2.11 2.10 2.09 2.08
1.00 1.03 1.00 1.07 1.08 2.15 5.19
10.5 9.5 8.5 7.5 6.5 5.5 4.5 3.5 2.5 1.5 0.5 -0.5
f1 (ppm)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147.61 135.25 129.46 128.29 125.89 122.92
53.53 40.97 27.77 24.48 21.88
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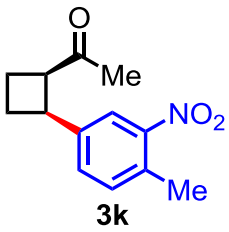
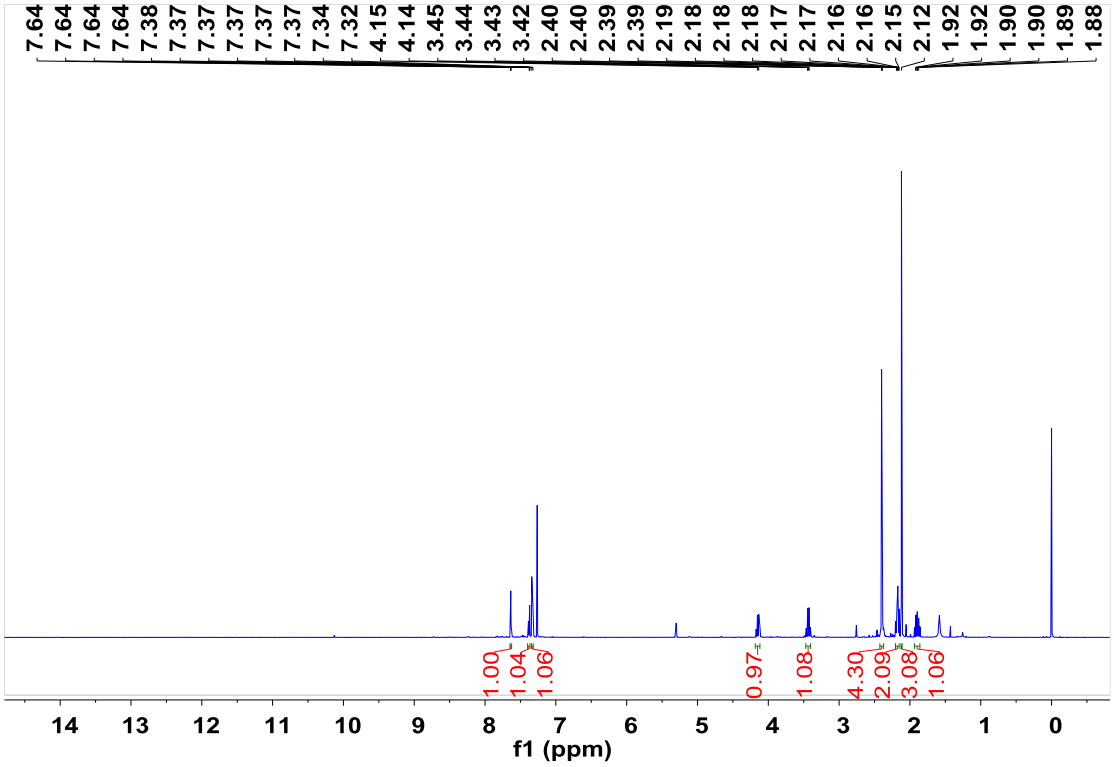
f1 (ppm)


O
Me
NO2
Me

3k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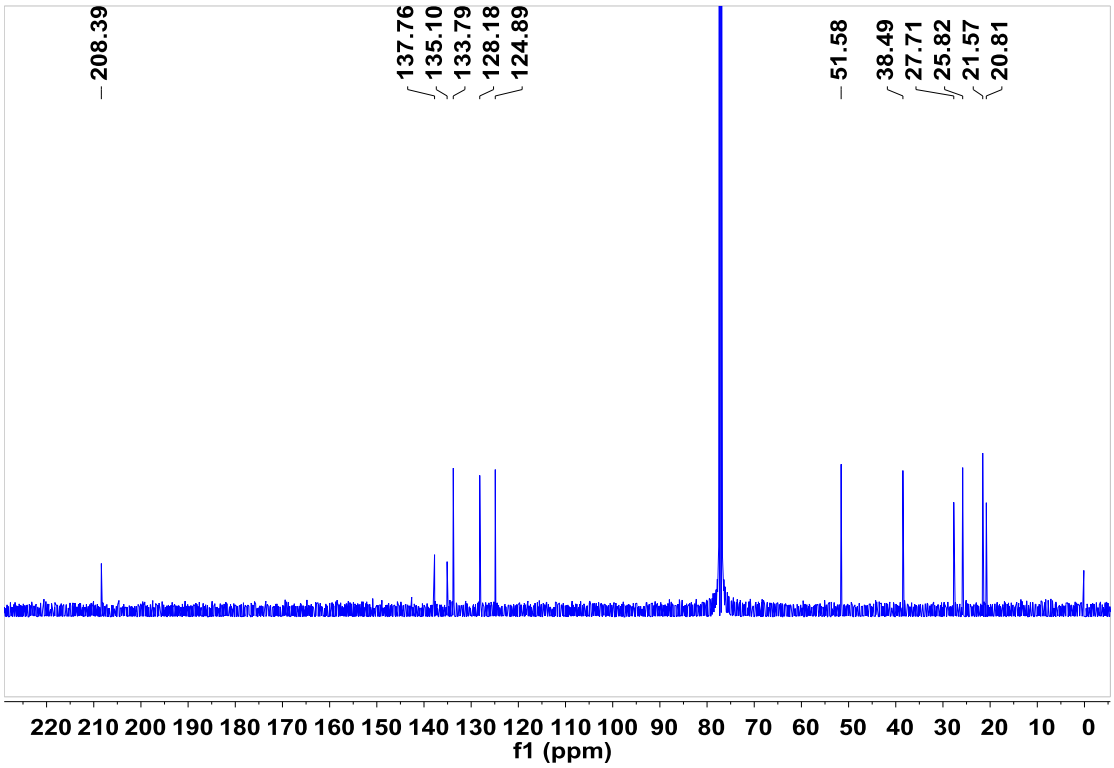
1.00 1.04 1.06 0.97 1.08 4.30 2.09 3.08 1.06
14 13 12 11 10 9 8 7 6 5 4 3 2 1 0
f1 (ppm)


208.39
137.76 135.10 133.79 128.18 124.89
51.58 38.49 27.71 25.82 21.57 20.81
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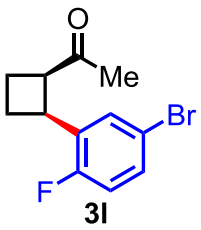
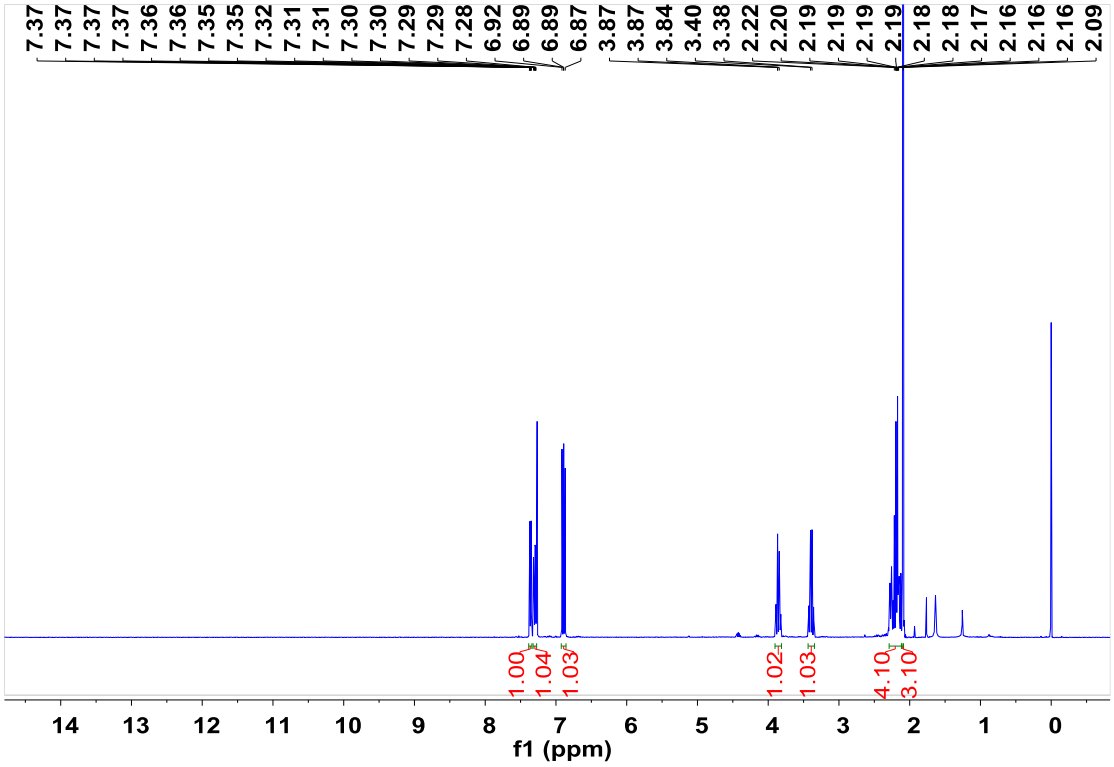
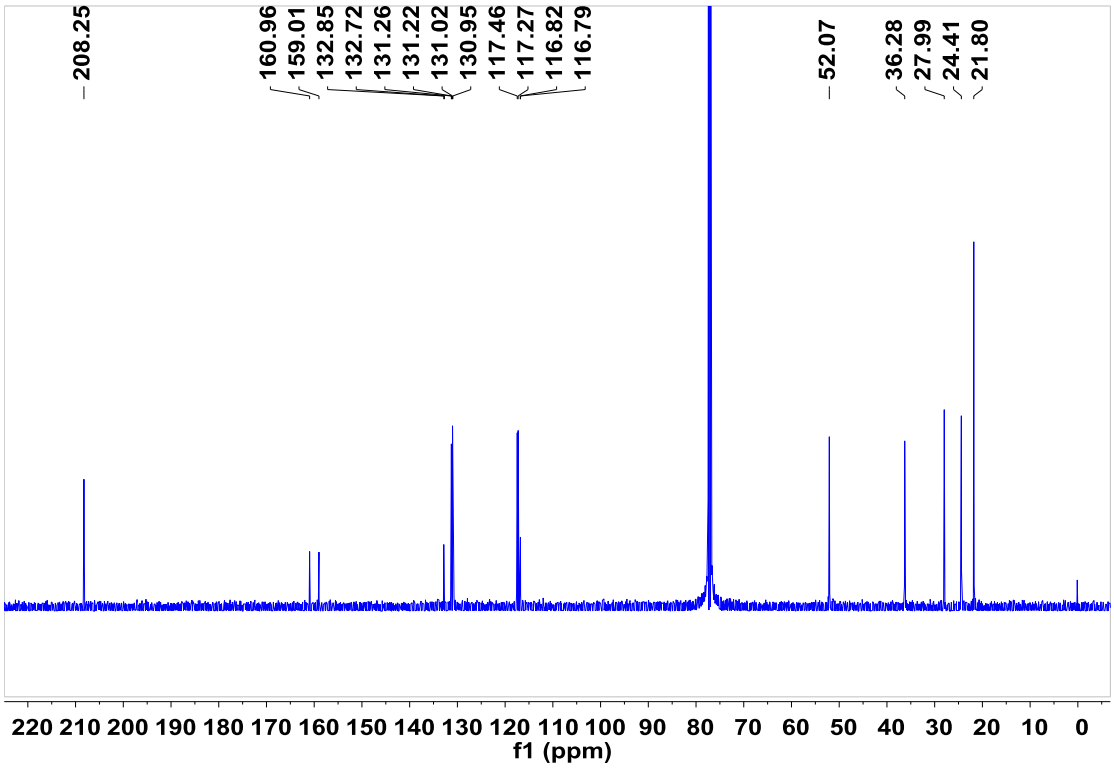
f1 (ppm)


O
Me
Br
F

3l


7.37 7.37 7.37 7.37 7.36 7.36 7.35 7.35 7.32 7.31 7.31 7.30 7.30 7.29 7.29 7.28 6.92 6.89 6.89 6.87
3.87 3.87 3.84 3.40 3.38 2.22 2.20 2.19 2.19 2.19 2.19 2.18 2.18 2.17 2.16 2.16 2.16 2.09
1.00 1.04 1.03 1.02 1.03 4.10 3.10
14 13 12 11 10 9 8 7 6 5 4 3 2 1 0
f1 (ppm)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160.96 159.01 132.85 132.72 131.26 131.22 131.02 130.95 117.46 117.27 116.82 116.79
52.07
36.28 27.99 24.41 21.80
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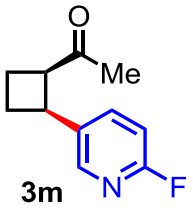
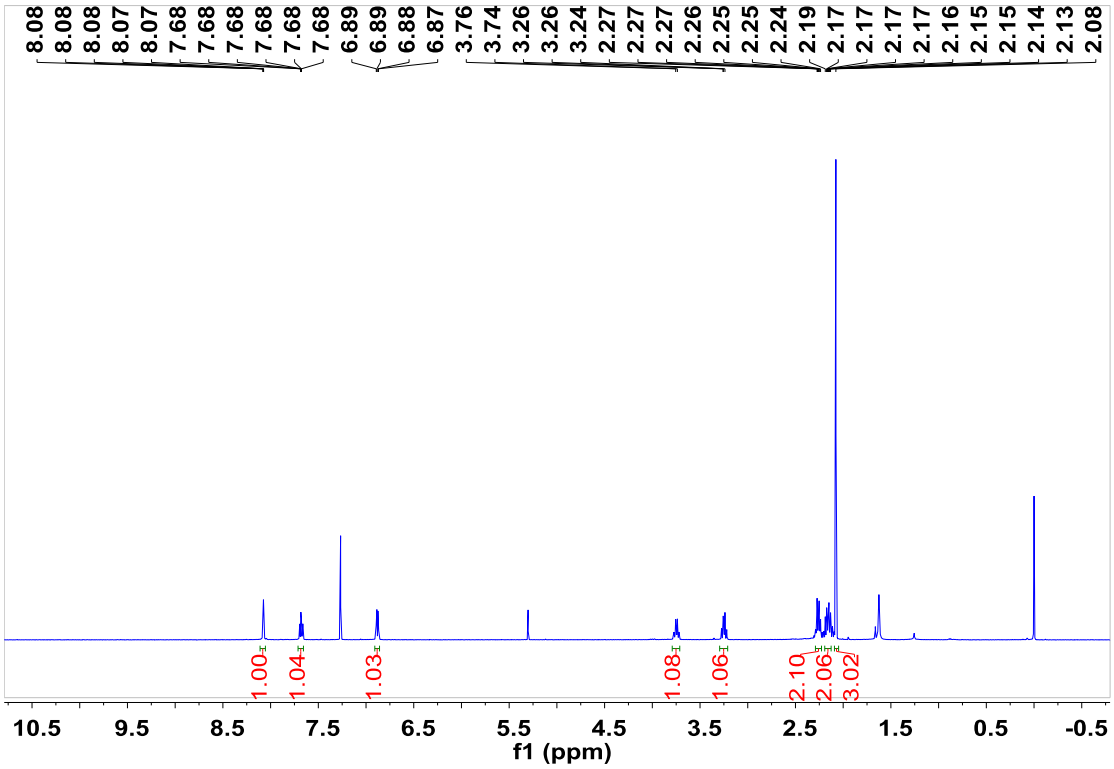
f1 (ppm)


O
Me
N
F

3m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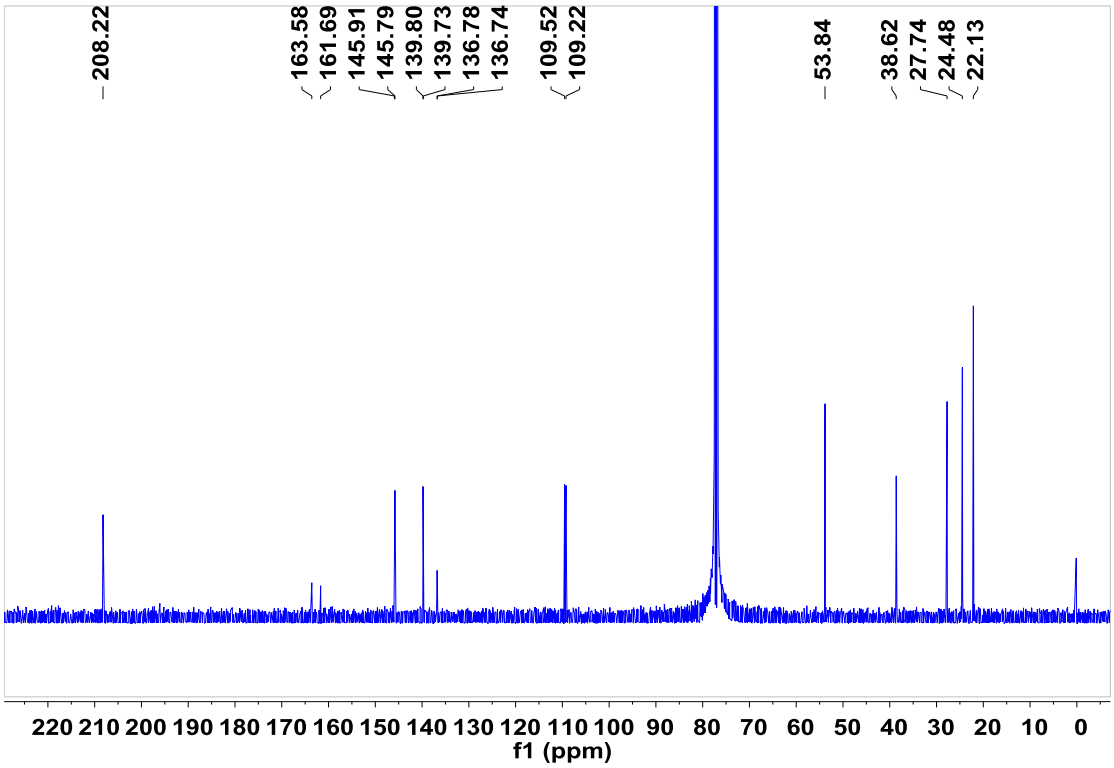
1.00 1.04 1.03 1.08 1.06 2.10 2.06 3.02
10.5 9.5 8.5 7.5 6.5 5.5 4.5 3.5 2.5 1.5 0.5 -0.5
f1 (ppm)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163.58 161.69 145.91 145.79 139.80 139.73 136.78 136.74 109.52 109.22
53.84
38.62 27.74 24.48 22.13
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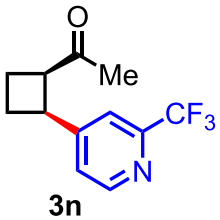
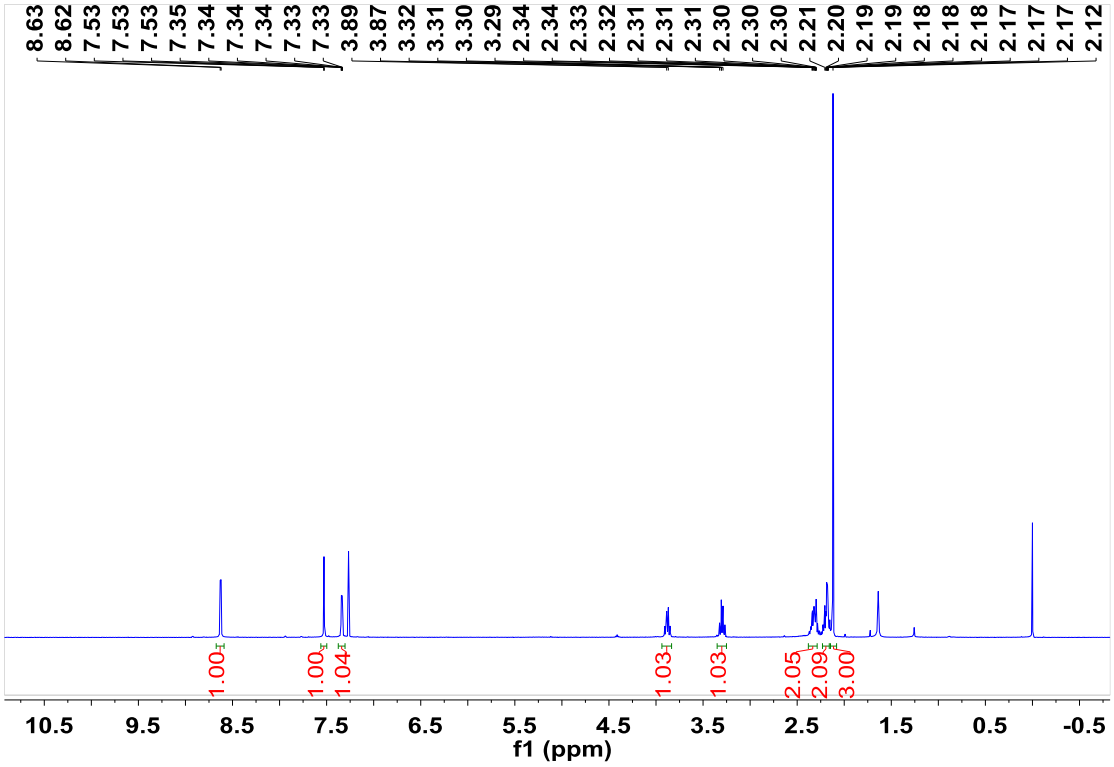
f1 (ppm)


O
Me
CF3
N

3n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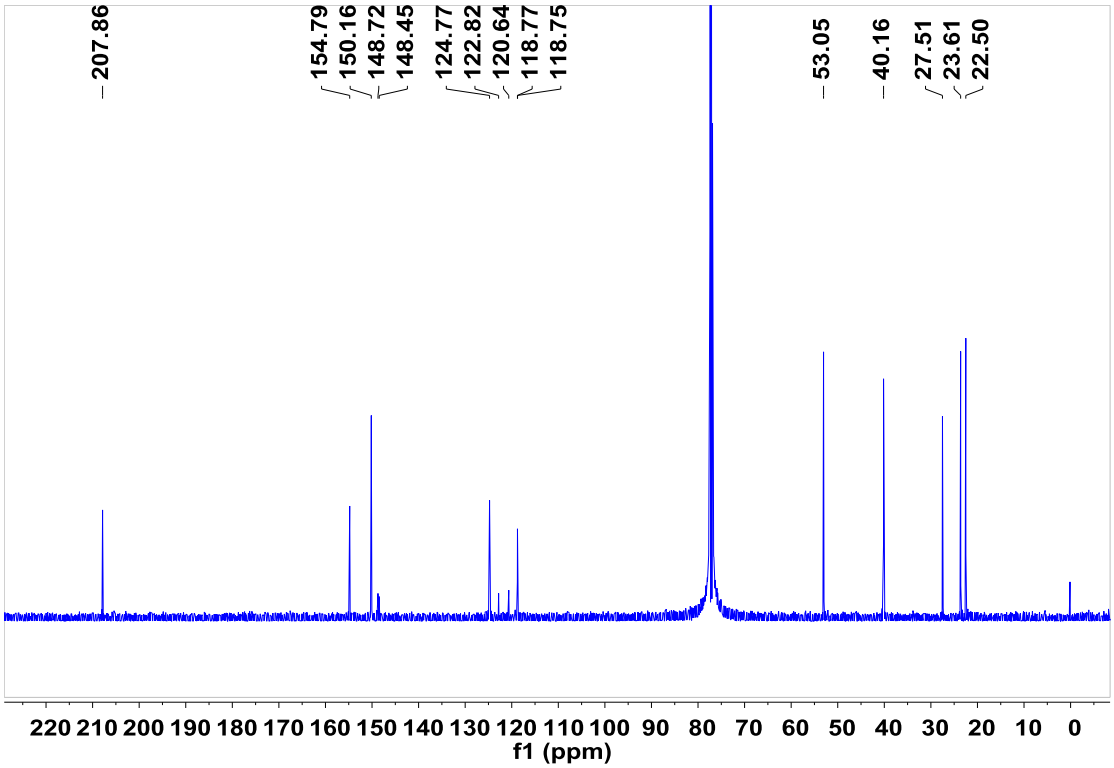
1.00 1.00 1.04 1.03 1.03 2.05 2.09 3.00
10.5 9.5 8.5 7.5 6.5 5.5 4.5 3.5 2.5 1.5 0.5 -0.5
f1 (ppm)


207.86
154.79 150.16 148.72 148.45 124.77 122.82 120.64 118.77 118.75
53.05 40.16 27.51 23.61 22.50
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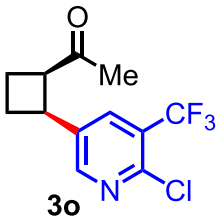
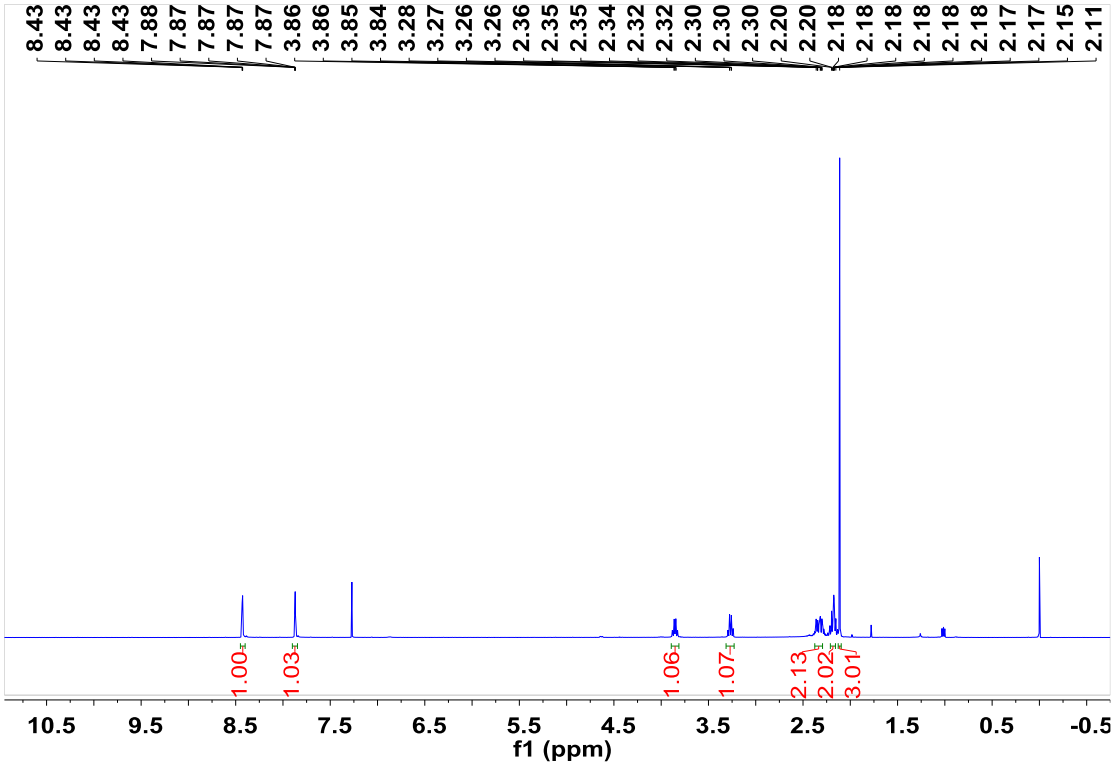
f1 (ppm)


O
Me
CF3
N
Cl

3o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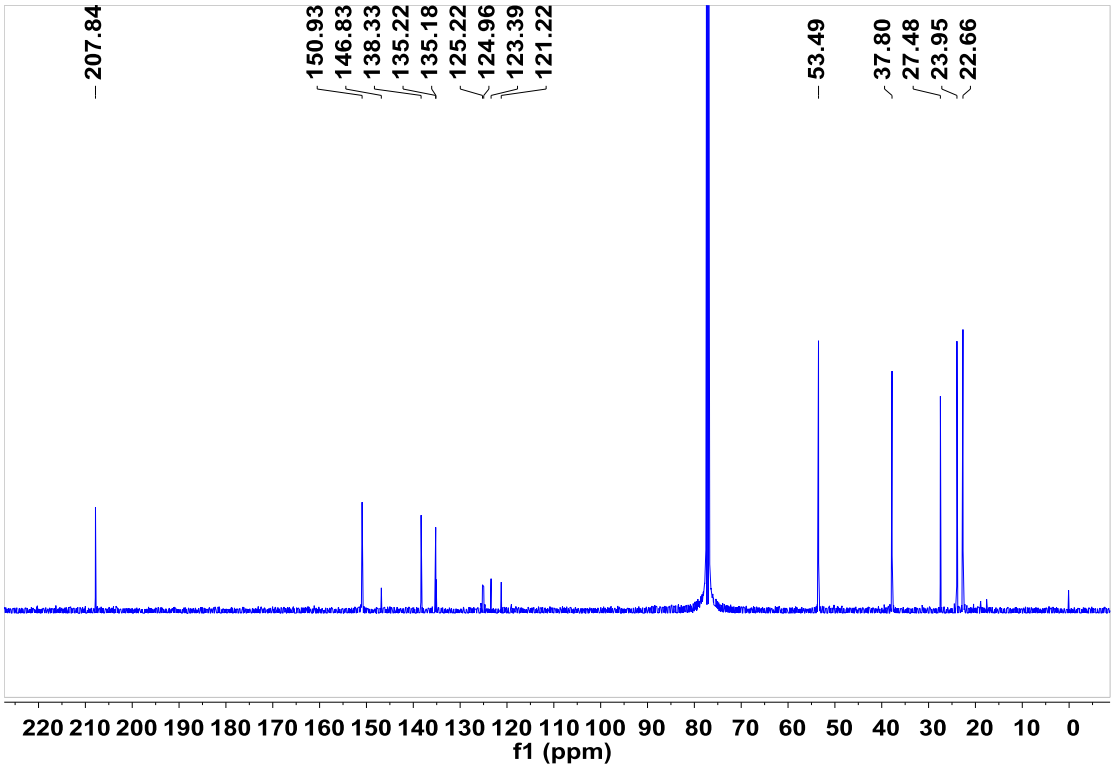
1.00
1.03
1.06
1.07
2.13
2.02
3.01
10.5 9.5 8.5 7.5 6.5 5.5 4.5 3.5 2.5 1.5 0.5 -0.5
f1 (ppm)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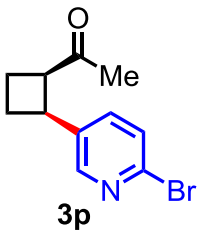
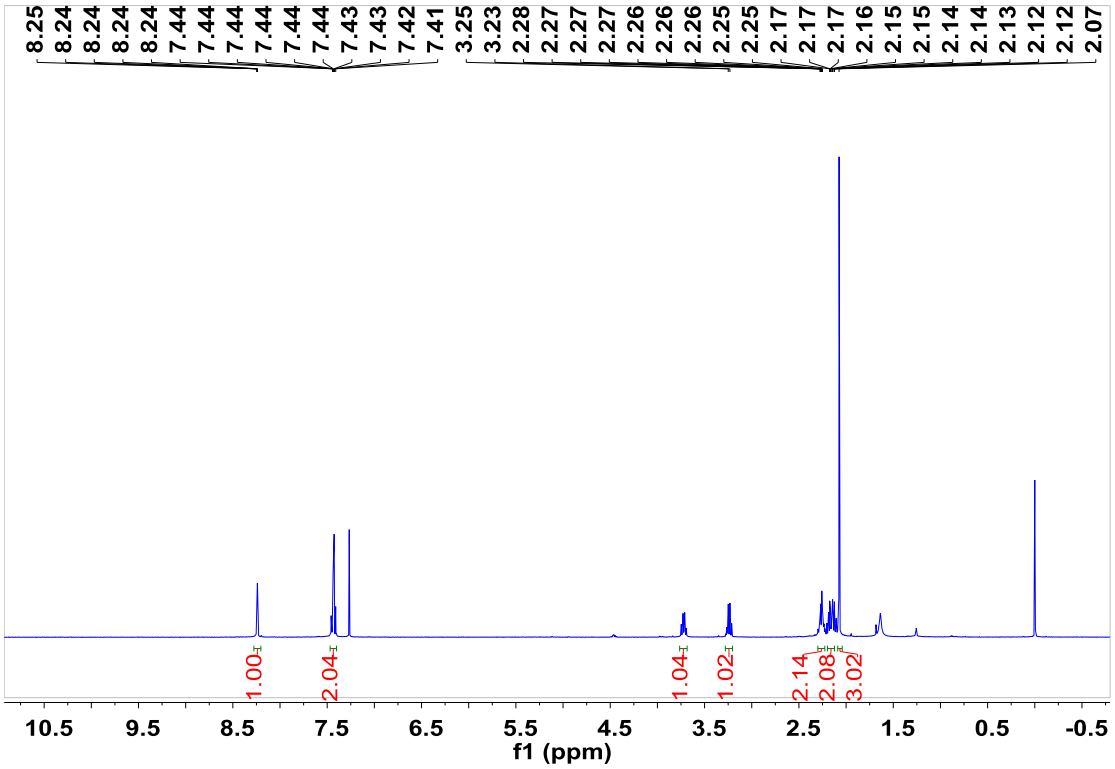
125.22
124.96
123.39
121.22
53.49
37.80
27.48
23.95
22.66
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


O
Me
N
Br

3p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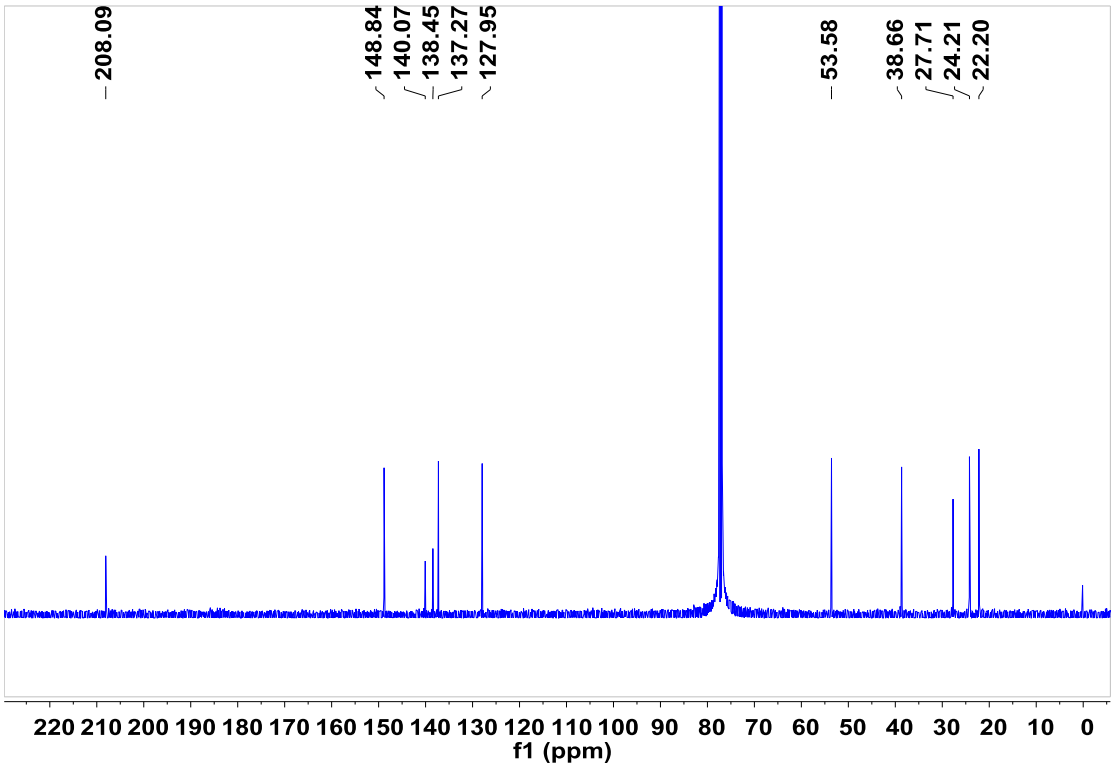
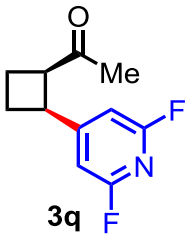
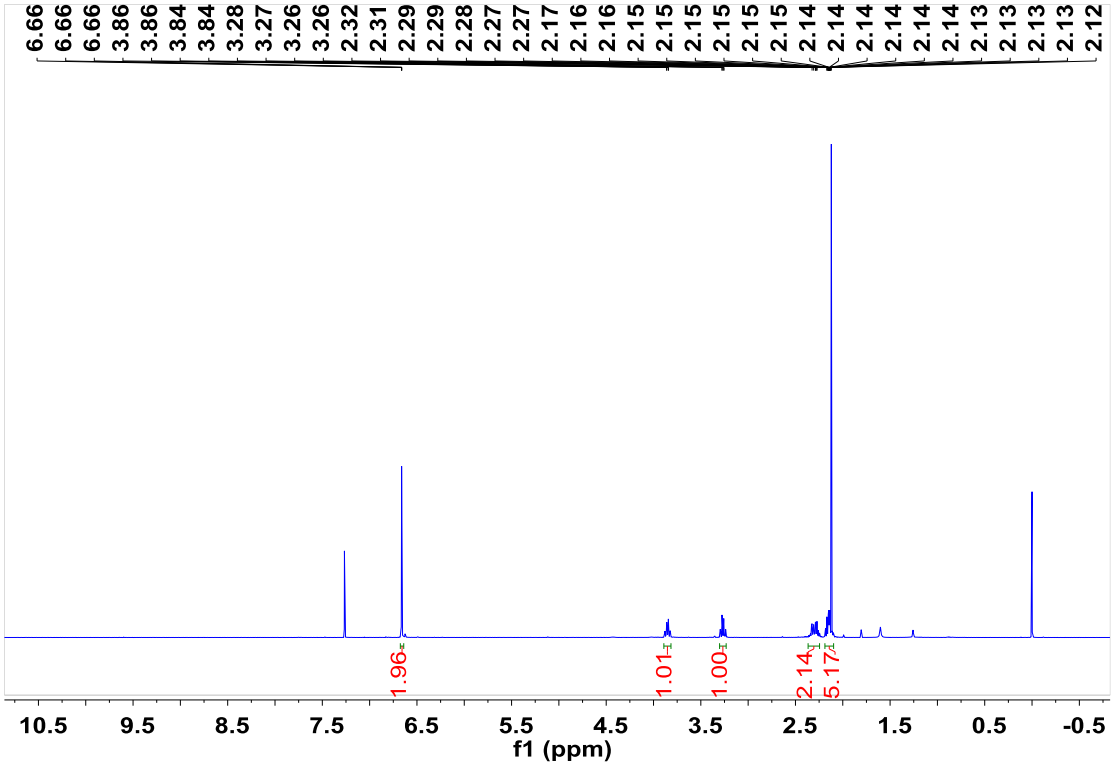
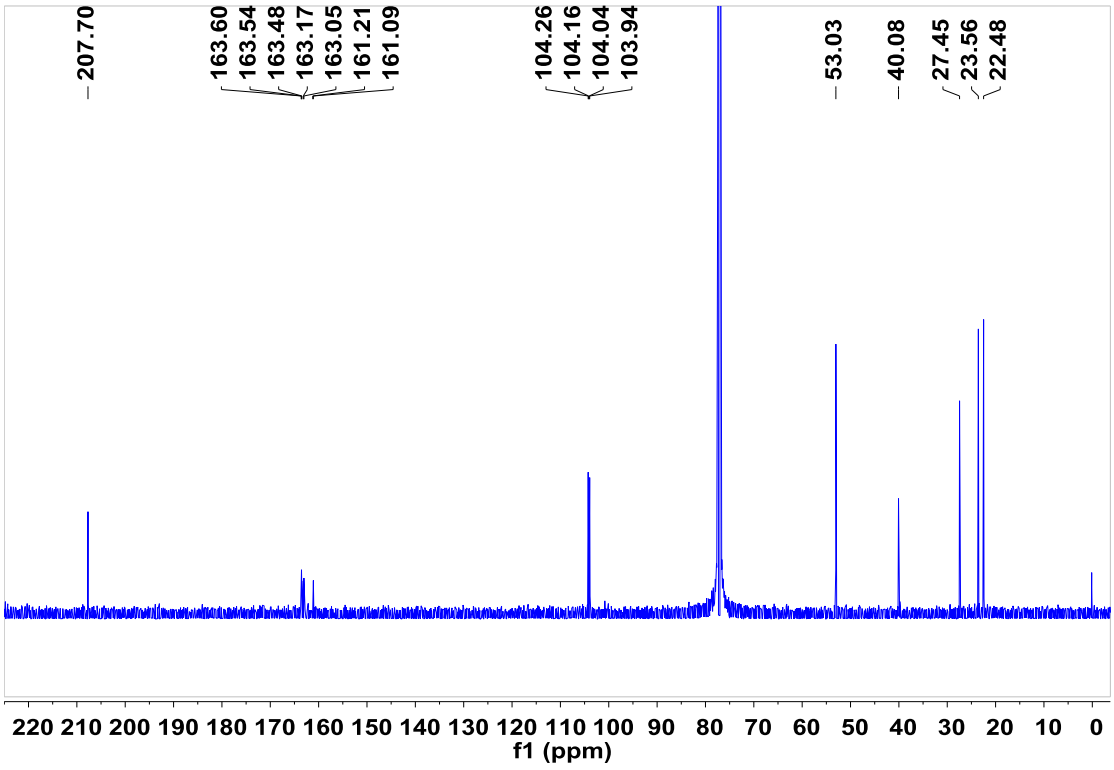
3.25 3.23 2.28 2.27 2.27 2.27 2.26 2.26 2.26 2.25 2.25 2.17 2.17 2.17 2.16 2.15 2.15 2.14 2.14 2.13 2.12 2.12 2.07
1.00 2.04 1.04 1.02 2.14 2.08 3.02
10.5 9.5 8.5 7.5 6.5 5.5 4.5 3.5 2.5 1.5 0.5 -0.5
f1 (ppm)

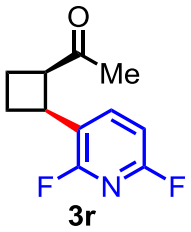

208.09
148.84 140.07 138.45 137.27 127.95
53.58
38.66 27.71 24.21 22.20
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


O
Me
F
N
F

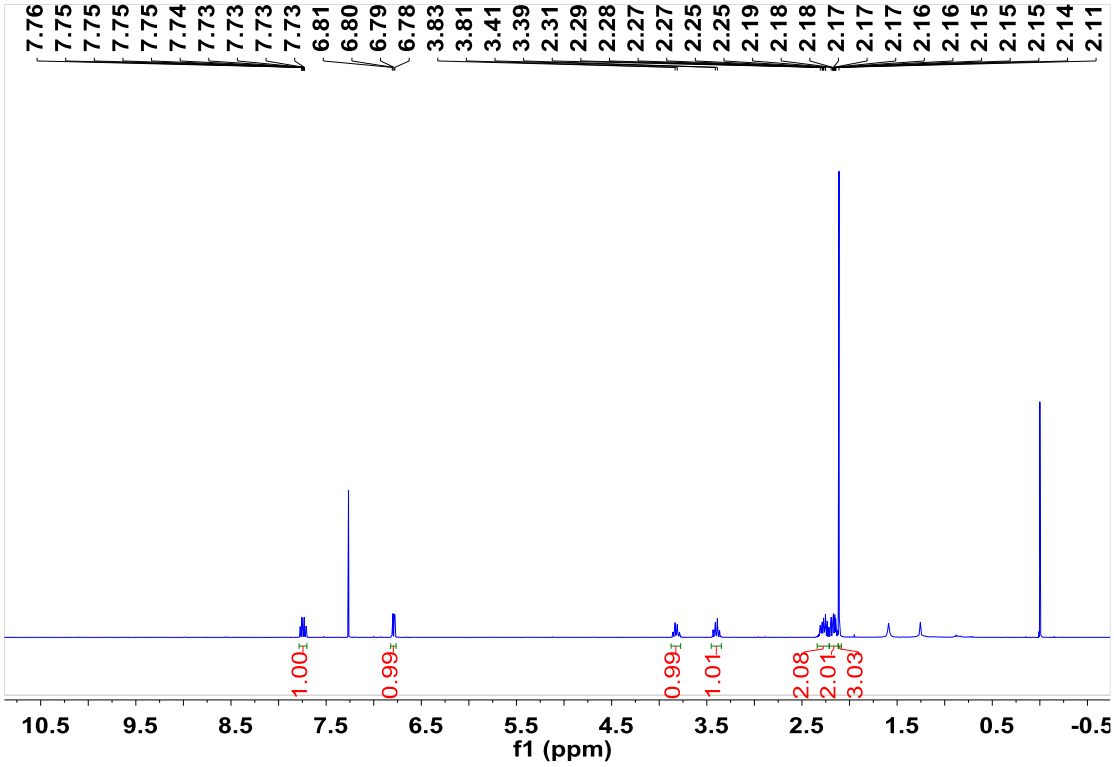
3q

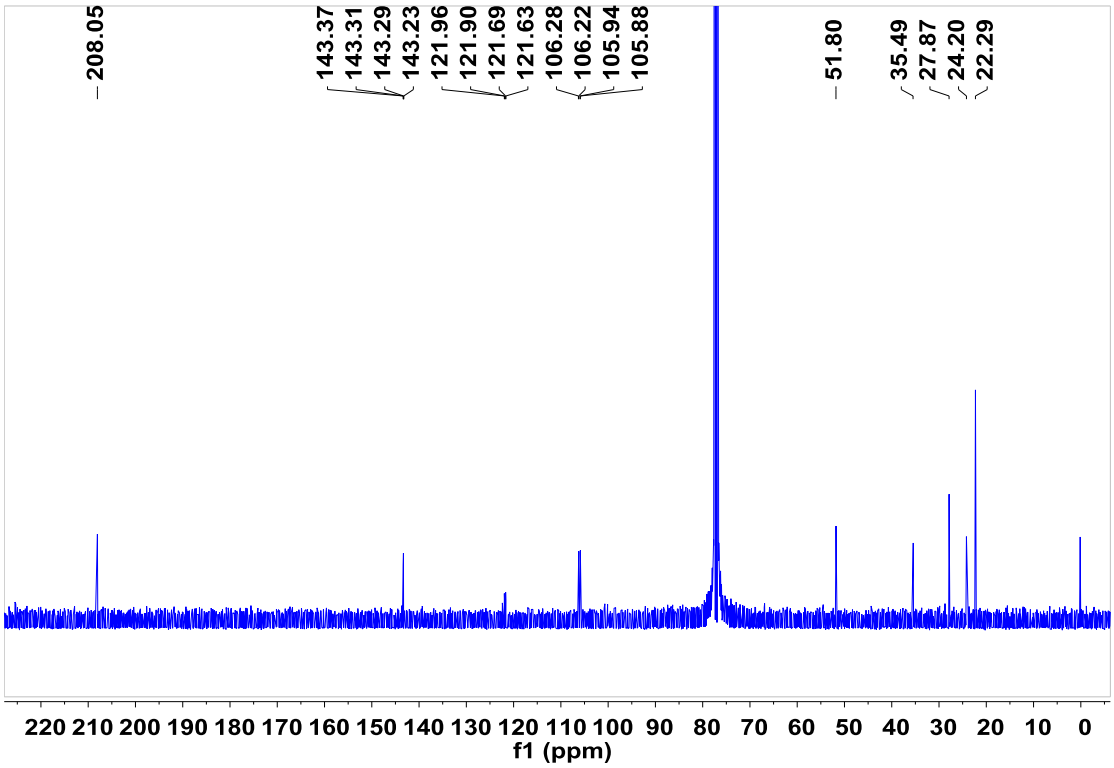

6.66 6.66 6.66 3.86 3.86 3.84 3.84 3.28 3.27 3.26 3.26 2.32 2.31 2.29 2.29 2.28 2.27 2.27 2.17 2.16 2.16 2.15 2.15 2.15 2.15 2.15 2.14 2.14 2.14 2.14 2.14 2.13 2.13 2.13 2.12
1.96
1.01
1.00
2.14
5.17
10.5 9.5 8.5 7.5 6.5 5.5 4.5 3.5 2.5 1.5 0.5 -0.5
f1 (ppm)

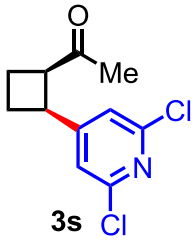

207.70
163.60 163.54 163.48 163.17 163.05 161.21 161.09
104.26 104.16 104.04 103.94
53.03
40.08
27.45
23.56
22.48
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


O
Me
F
N
F

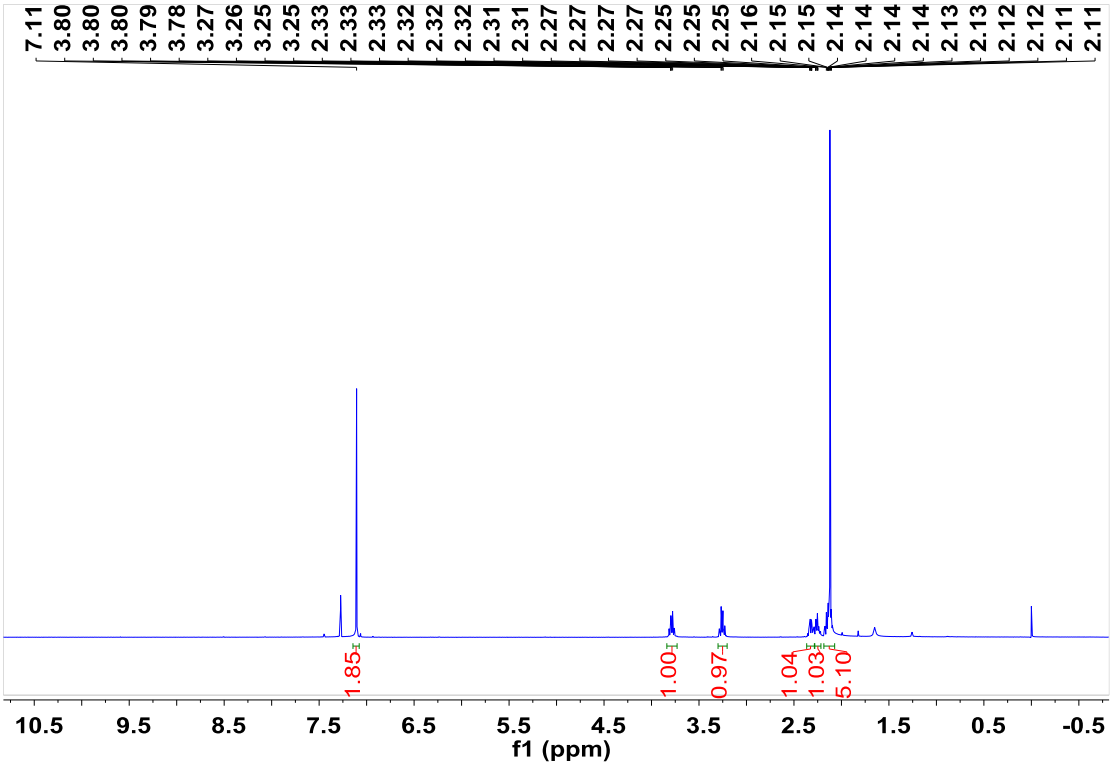
3r

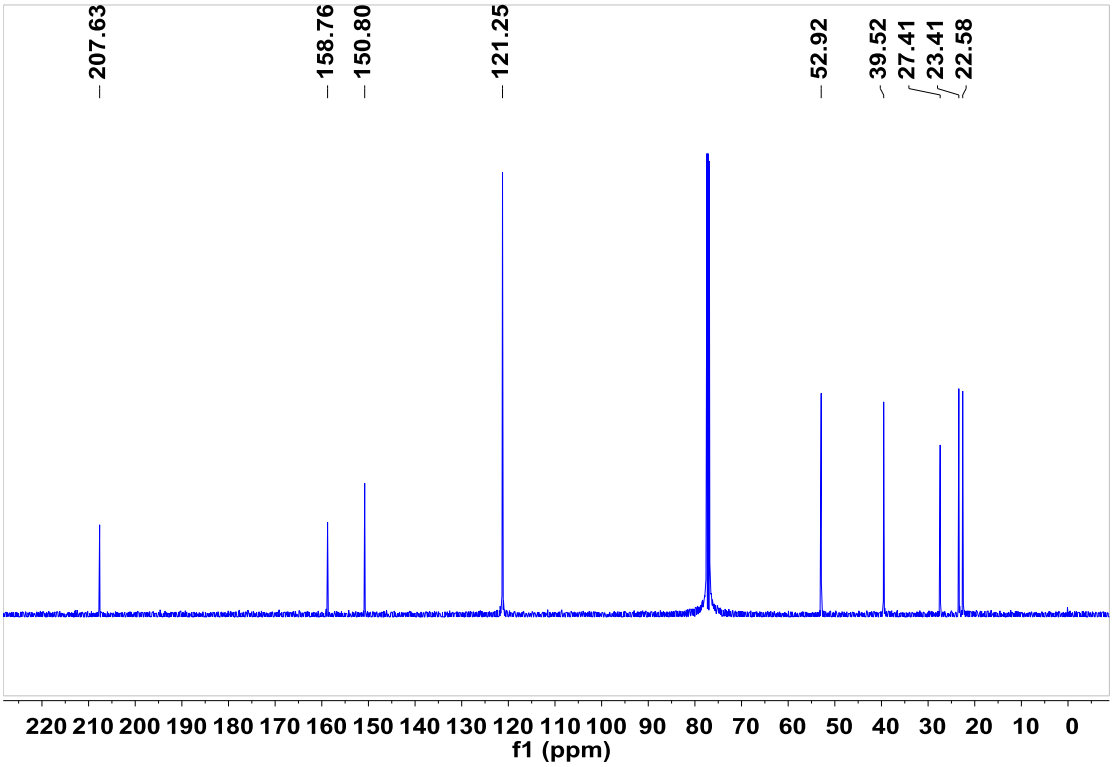

7.76 7.75 7.75 7.75 7.75 7.74 7.73 7.73 7.73 7.73 6.81 6.80 6.79 6.78 3.83 3.81 3.41 3.39 2.31 2.29 2.28 2.27 2.27 2.25 2.25 2.19 2.18 2.18 2.17 2.17 2.17 2.16 2.16 2.15 2.15 2.15 2.14 2.11
1.00 0.99 0.99 1.01 2.08 2.01 3.03
10.5 9.5 8.5 7.5 6.5 5.5 4.5 3.5 2.5 1.5 0.5 -0.5
f1 (ppm)

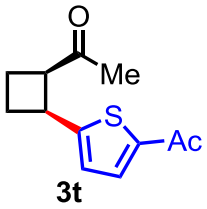

208.05
143.37 143.31 143.29 143.23 121.96 121.90 121.69 121.63 106.28 106.22 105.94 105.88
51.80
35.49 27.87 24.20 22.29
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


O
Me
Cl
N
Cl

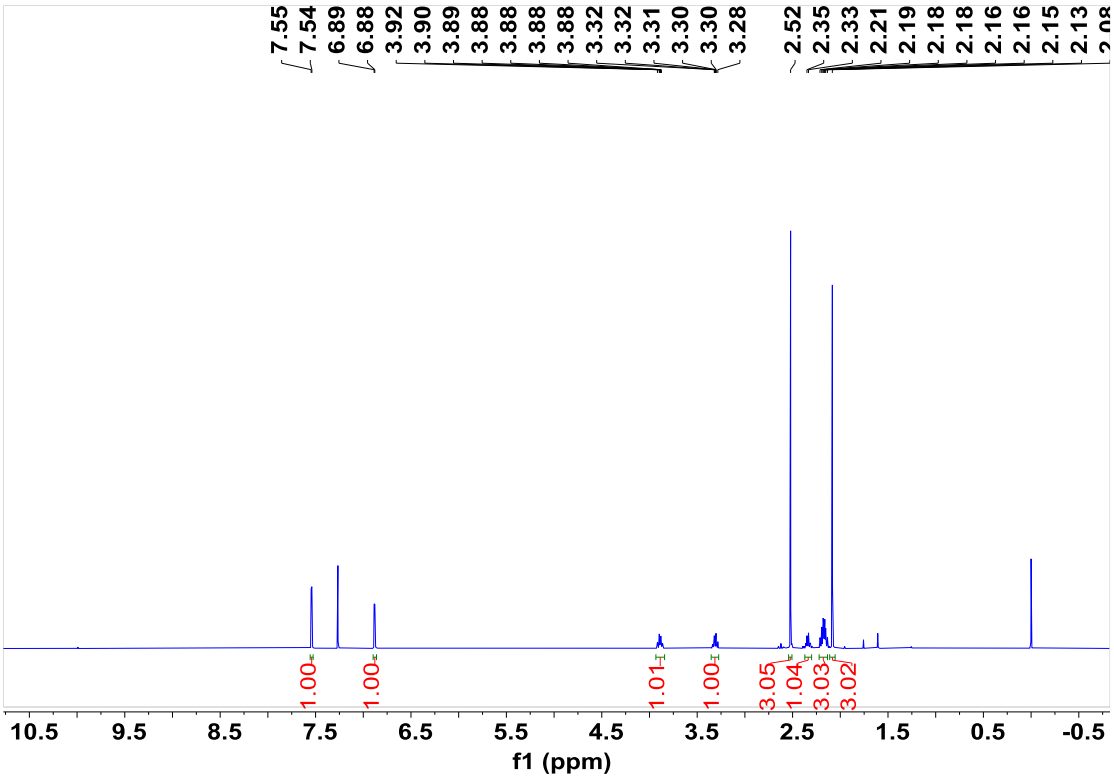
3s

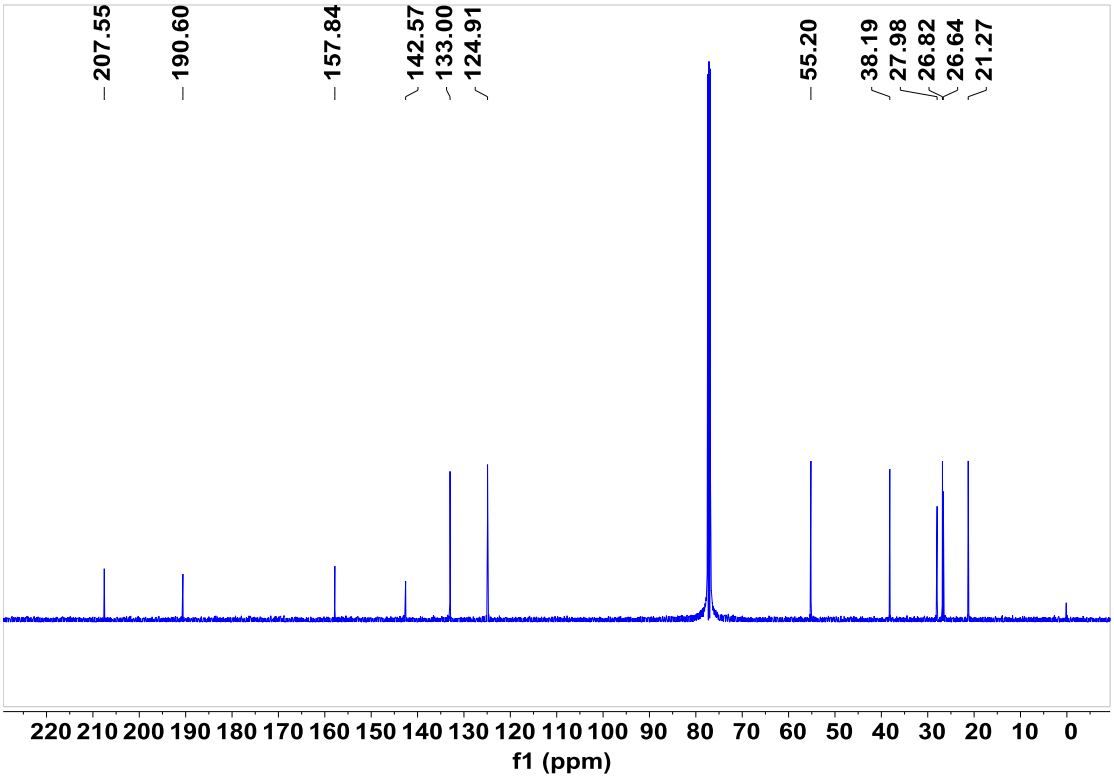

7.11 3.80 3.80 3.80 3.79 3.78 3.27 3.26 3.25 3.25 2.33 2.33 2.33 2.32 2.32 2.32 2.31 2.31 2.27 2.27 2.27 2.27 2.25 2.25 2.25 2.16 2.15 2.15 2.14 2.14 2.14 2.14 2.13 2.13 2.12 2.12 2.11 2.11
1.85
1.00
0.97
1.04
1.03
5.10
10.5 9.5 8.5 7.5 6.5 5.5 4.5 3.5 2.5 1.5 0.5 -0.5
f1 (ppm)

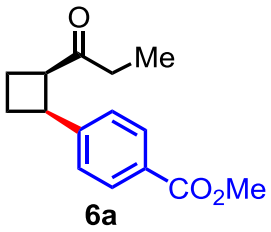

207.63
158.76
150.80
121.25
52.92
39.52
27.41
23.41
22.58
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


O
Me
S
Ac

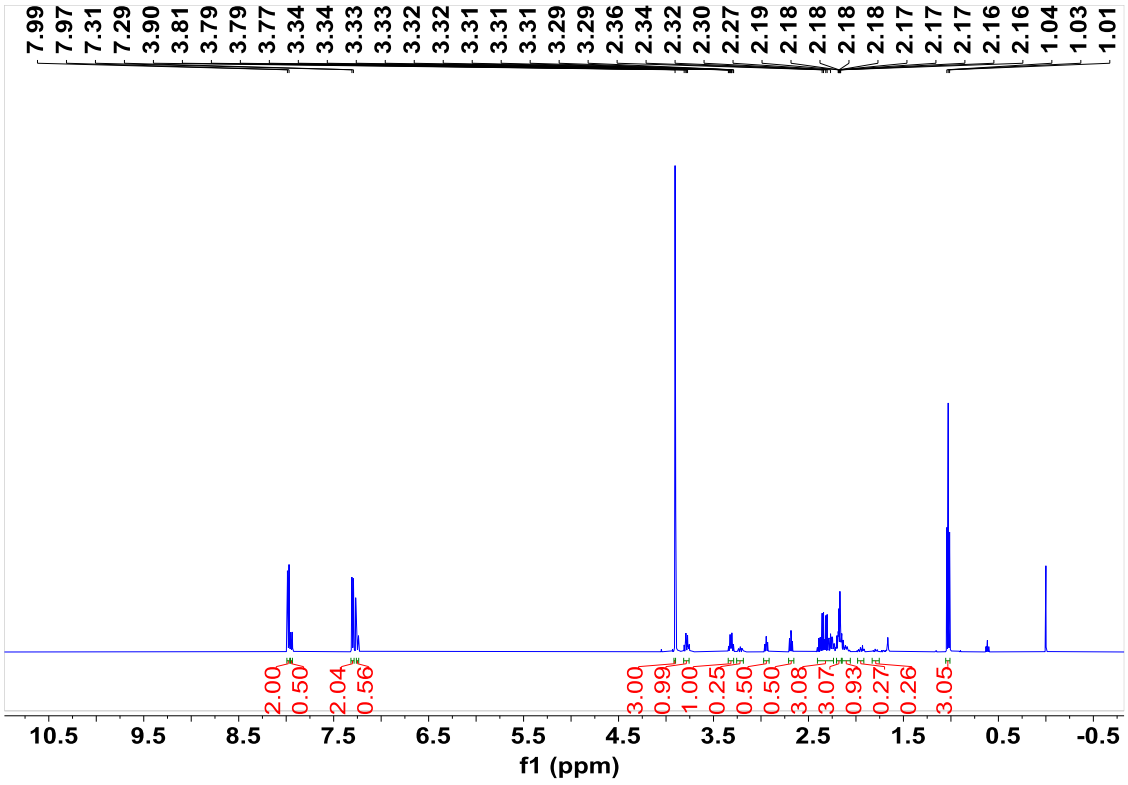
3t

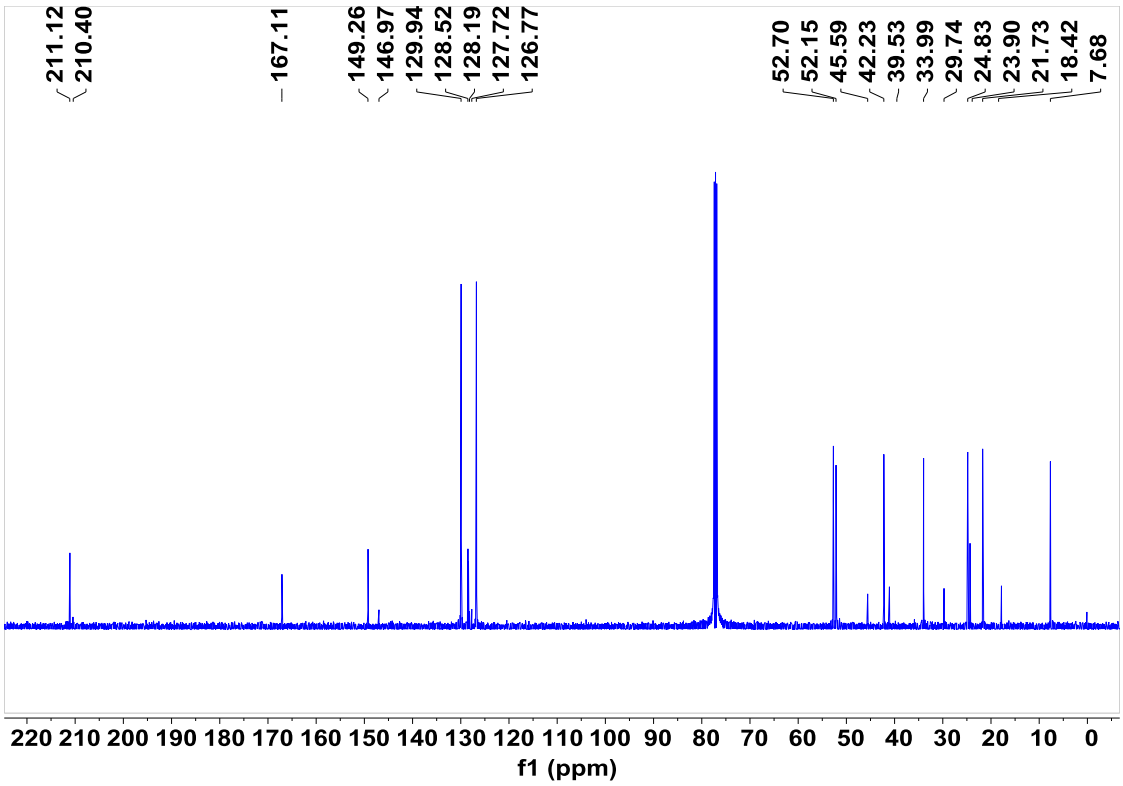

7.55
7.54
6.89
6.88
3.92
3.90
3.89
3.88
3.88
3.88
3.88
3.32
3.32
3.31
3.30
3.30
3.28
2.52
2.35
2.33
2.21
2.19
2.18
2.18
2.16
2.16
2.15
2.13
1.00
1.00
1.01
1.00
3.05
1.04
3.03
3.02
10.5
9.5
8.5
7.5
6.5
5.5
4.5
3.5
2.5
1.5
0.5
-0.5
f1 (ppm)

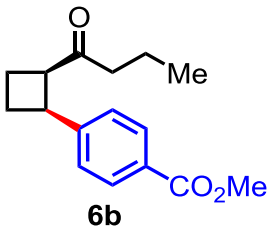

207.55
190.60
157.84
142.57
133.00
124.91
55.20
38.19
27.98
26.82
26.64
21.27
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


O
Me
CO2Me

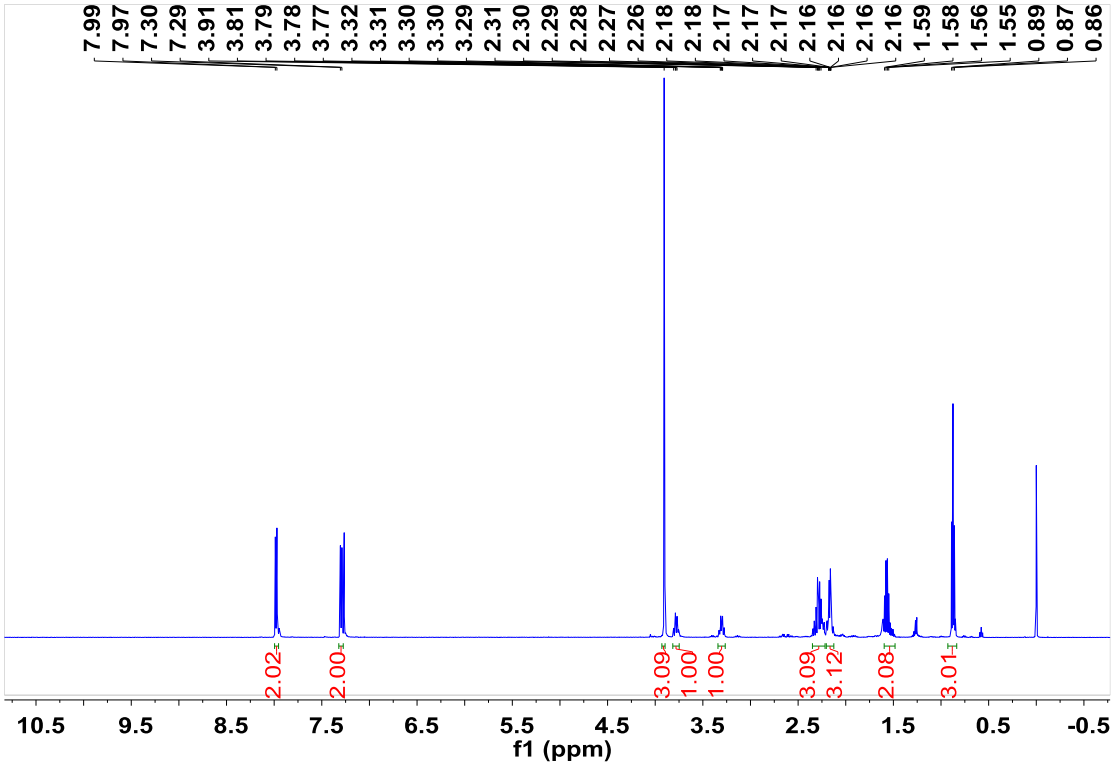
6a

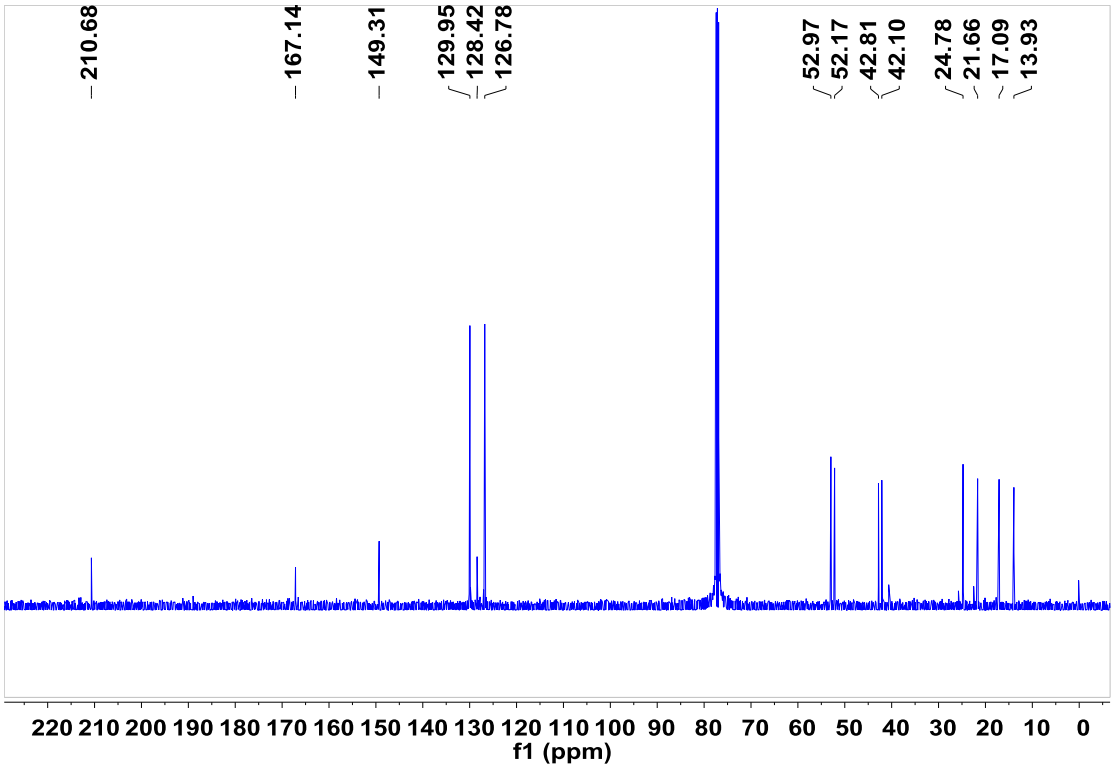

7.99 7.97 7.31 7.29 3.90 3.81 3.79 3.79 3.77 3.34 3.34 3.33 3.33 3.32 3.32 3.31 3.31 3.31 3.29 3.29 2.36 2.34 2.32 2.30 2.27 2.19 2.18 2.18 2.18 2.18 2.17 2.17 2.17 2.16 2.16 1.04 1.03 1.01
2.00 0.50 2.04 0.56 3.00 0.99 1.00 0.25 0.50 0.50 3.08 3.07 0.93 0.27 0.26 3.05
10.5 9.5 8.5 7.5 6.5 5.5 4.5 3.5 2.5 1.5 0.5 -0.5
f1 (ppm)

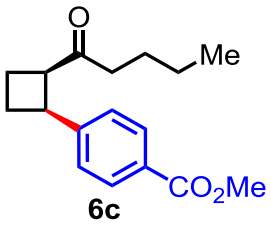

211.12 210.40 167.11 149.26 146.97 129.94 128.52 128.19 127.72 126.77 52.70 52.15 45.59 42.23 39.53 33.99 29.74 24.83 23.90 21.73 18.42 7.68
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


O
Me
CO2Me

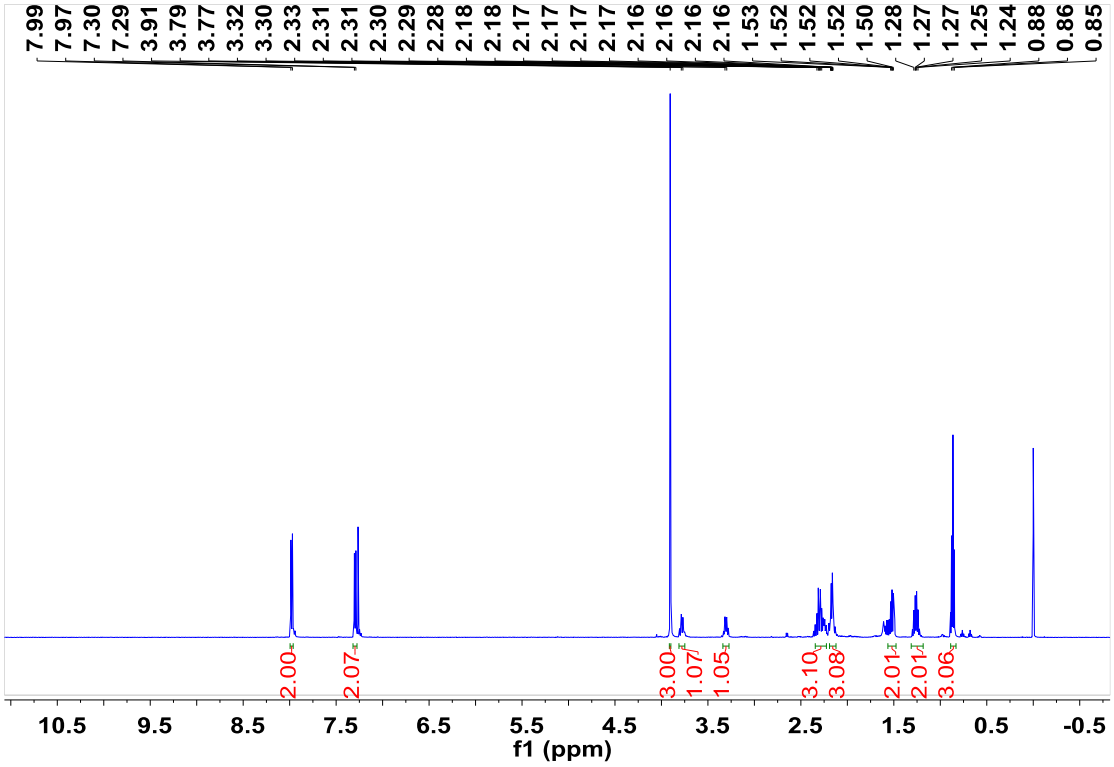
6b

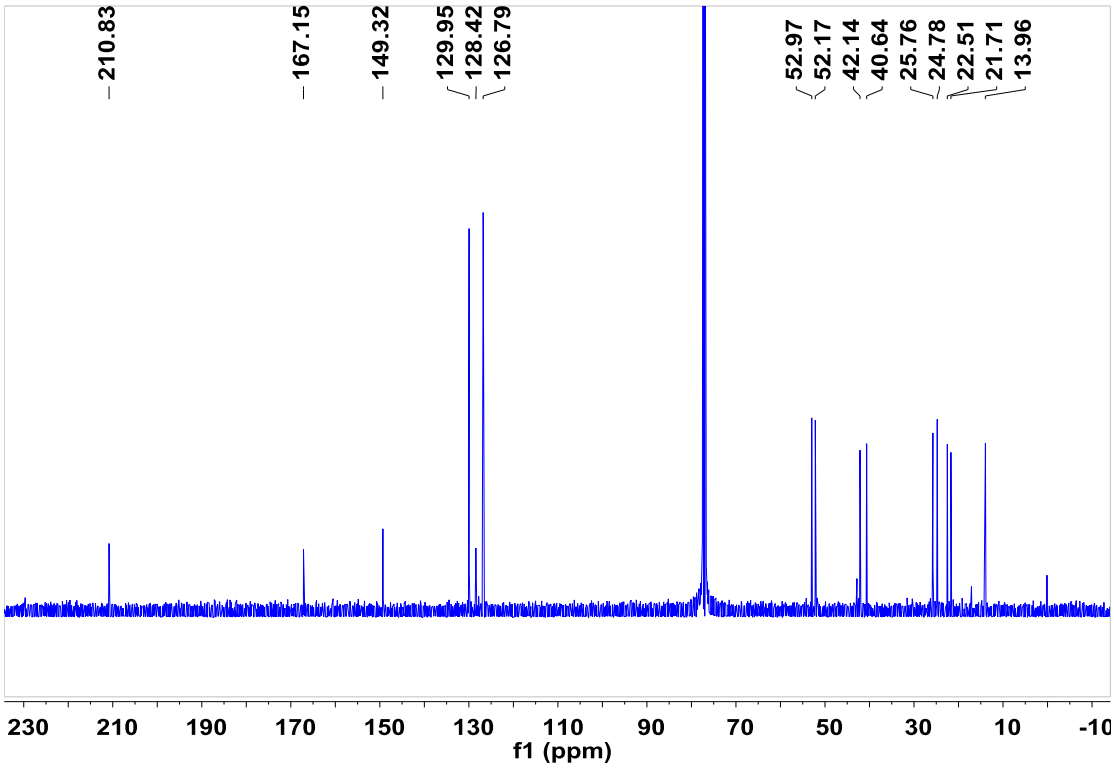

7.99
7.97
7.30
7.29
3.91
3.81
3.79
3.78
3.77
3.32
3.31
3.30
3.30
3.29
2.31
2.30
2.29
2.28
2.27
2.26
2.18
2.18
2.17
2.17
2.17
2.16
2.16
2.16
2.16
1.59
1.58
1.56
1.55
0.89
0.87
0.86
2.02
2.00
3.09
1.00
1.00
3.09
3.12
2.08
3.01
10.5
9.5
8.5
7.5
6.5
5.5
4.5
3.5
2.5
1.5
0.5
-0.5
f1 (ppm)

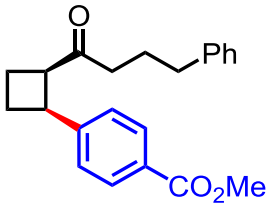

210.68
167.14
149.31
129.95
128.42
126.78
52.97
52.17
42.81
42.10
24.78
21.66
17.09
13.93
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


O
Me
CO2Me

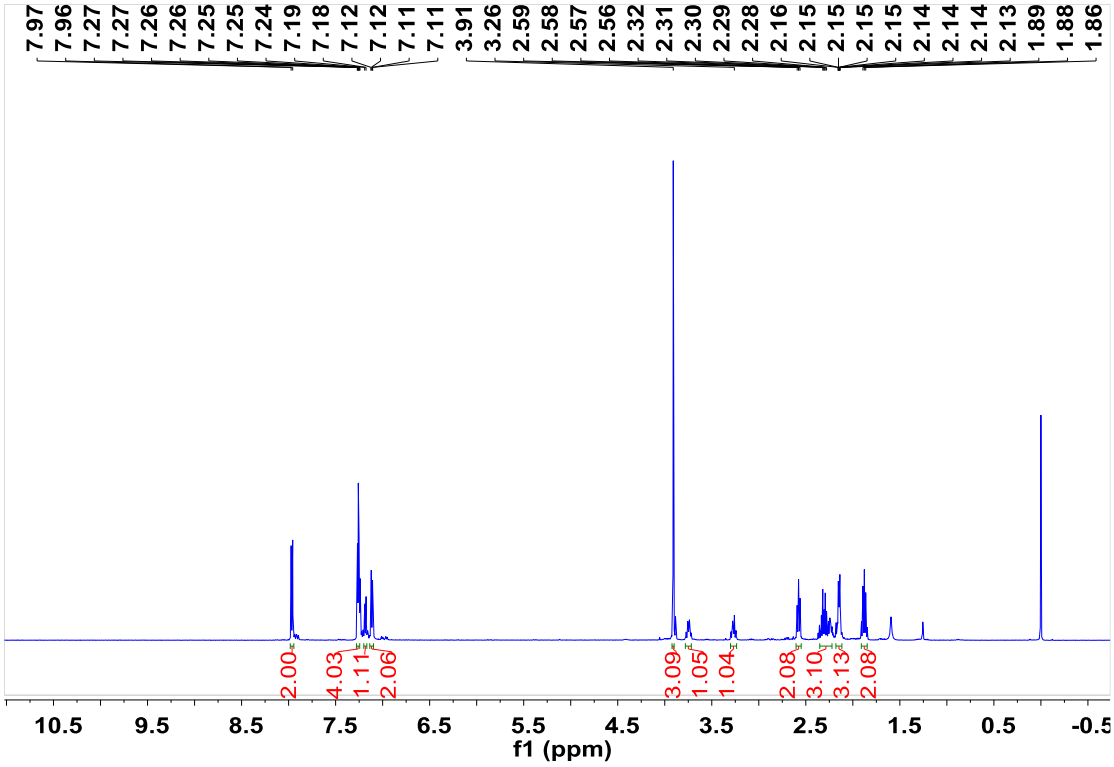
6c

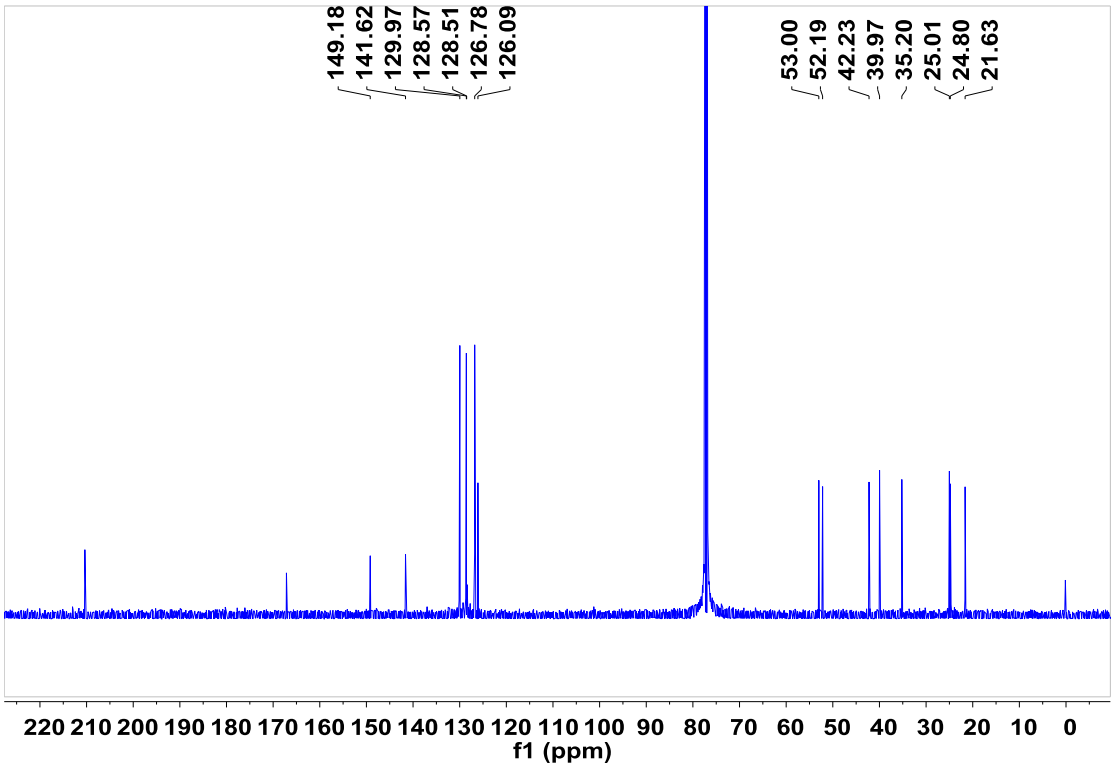

7.99 7.97 7.30 7.29 3.91 3.79 3.77 3.32 3.30 2.33 2.31 2.31 2.30 2.29 2.28 2.18 2.18 2.17 2.17 2.17 2.17 2.16 2.16 2.16 2.16 1.53 1.52 1.52 1.52 1.50 1.28 1.27 1.27 1.25 1.24 0.88 0.86 0.85
2.00 2.07 3.00 1.07 1.05 3.10 3.08 2.01 2.01 3.06
10.5 9.5 8.5 7.5 6.5 5.5 4.5 3.5 2.5 1.5 0.5 -0.5
f1 (ppm)


210.83 167.15 149.32 129.95 128.42 126.79 52.97 52.17 42.14 40.64 25.76 24.78 22.51 21.71 13.96
230 210 190 170 150 130 110 90 70 50 30 10 -10
f1 (ppm)


O
Ph
CO2Me

6d


7.97 7.96 7.27 7.27 7.26 7.26 7.25 7.25 7.24 7.19 7.18 7.12 7.12 7.11 7.11
3.91 3.26 2.59 2.58 2.57 2.56 2.32 2.31 2.30 2.29 2.28 2.16 2.15 2.15 2.15 2.14 2.14 2.14 2.13 1.89 1.88 1.86
2.00 4.03 1.11 2.06 3.09 1.05 1.04 2.08 3.10 3.13 2.08
10.5 9.5 8.5 7.5 6.5 5.5 4.5 3.5 2.5 1.5 0.5 -0.5
f1 (ppm)


149.18 141.62 129.97 128.57 128.51 126.78 126.09
53.00 52.19 42.23 39.97 35.20 25.01 24.80 21.63
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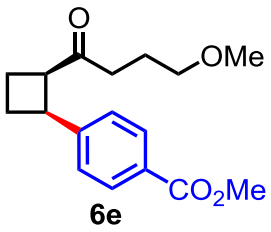
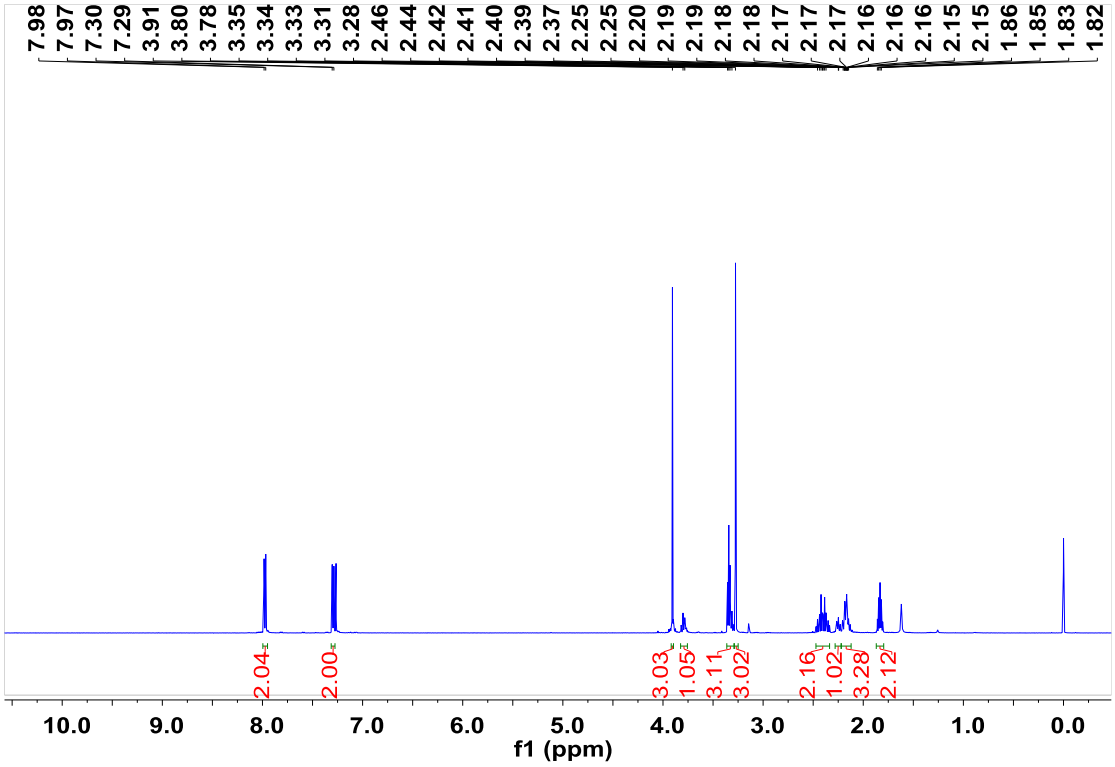
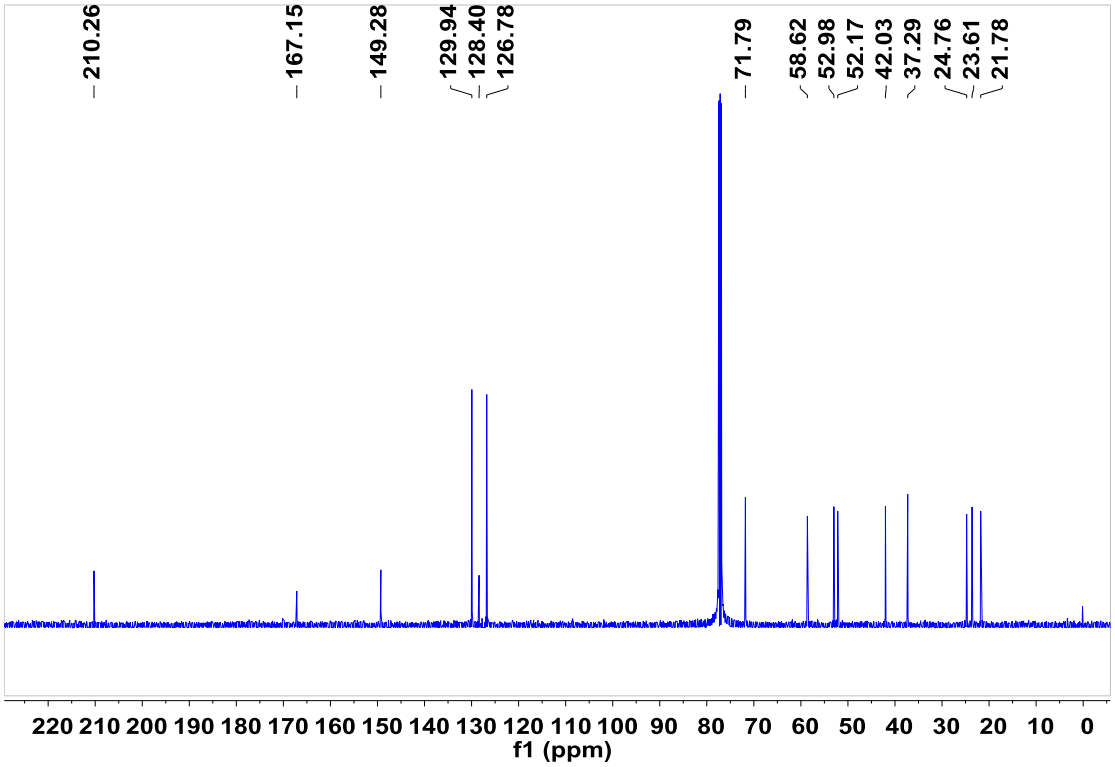
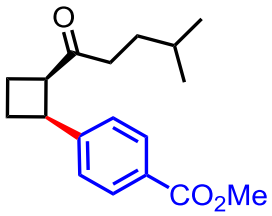
f1 (ppm)


O
OMe
CO2Me

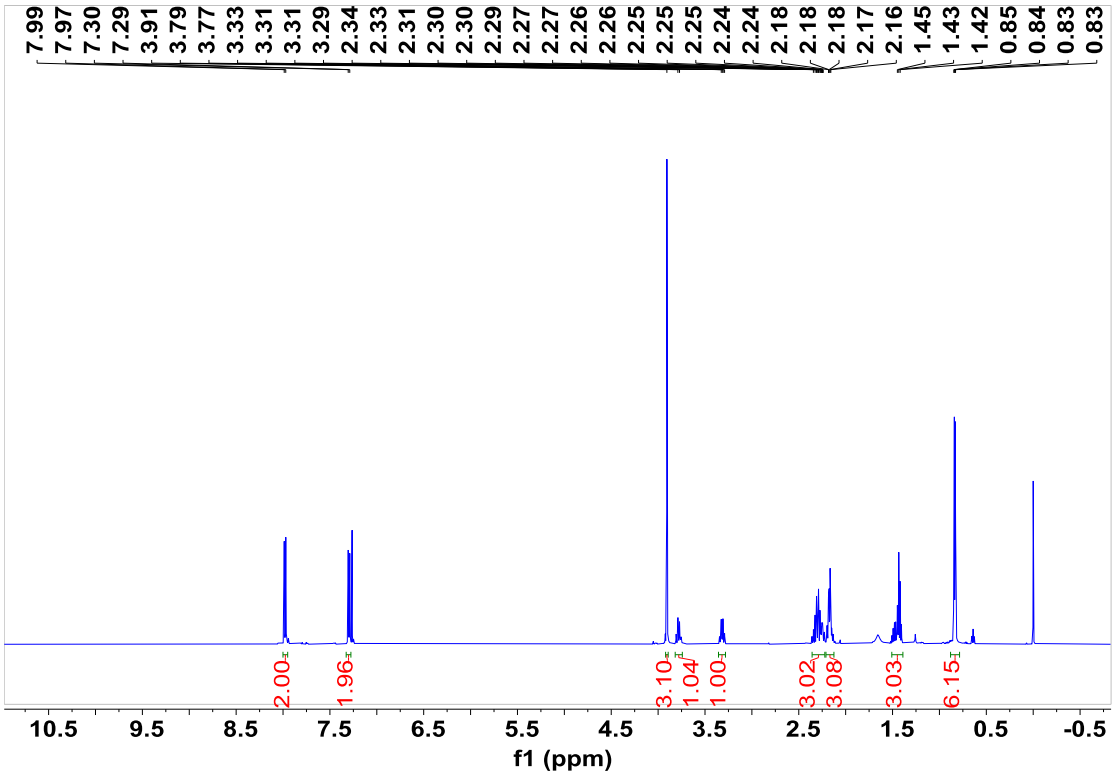
6e

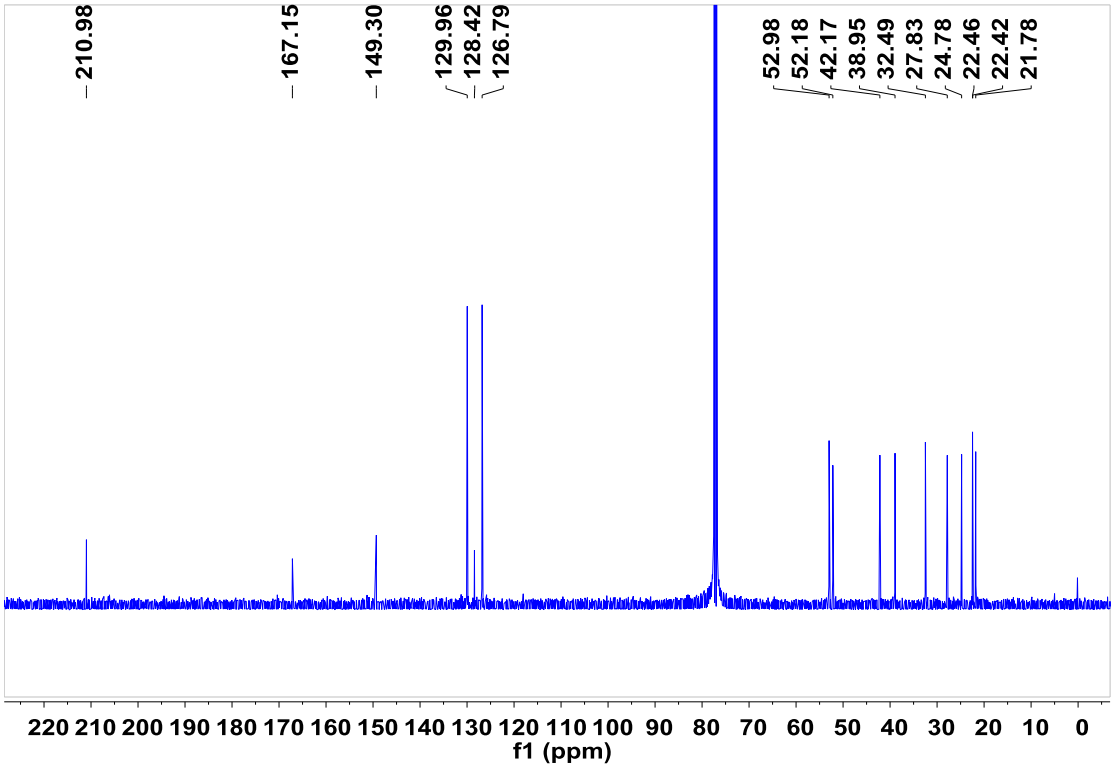

7.98 7.97 7.30 7.29 3.91 3.80 3.78 3.35 3.34 3.33 3.31 3.28 2.46 2.44 2.42 2.41 2.40 2.39 2.37 2.25 2.25 2.20 2.19 2.19 2.18 2.18 2.17 2.17 2.17 2.16 2.16 2.16 2.15 2.15 1.86 1.85 1.83 1.82
2.04 2.00 3.03 1.05 3.11 3.02 2.16 1.02 3.28 2.12
10.0 9.0 8.0 7.0 6.0 5.0 4.0 3.0 2.0 1.0 0.0
f1 (ppm)


210.26 167.15 149.28 129.94 128.40 126.78 71.79 58.62 52.98 52.17 42.03 37.29 24.76 23.61 21.78
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


O
CO2Me

6f


7.99 7.97 7.30 7.29 3.91 3.79 3.77 3.33 3.31 3.31 3.29 2.34 2.33 2.31 2.30 2.30 2.29 2.27 2.27 2.26 2.26 2.25 2.25 2.25 2.24 2.24 2.18 2.18 2.18 2.17 2.16 1.45 1.43 1.42 0.85 0.84 0.83 0.83
2.00 1.96 3.10 1.04 1.00 3.02 3.08 3.03 6.15
10.5 9.5 8.5 7.5 6.5 5.5 4.5 3.5 2.5 1.5 0.5 -0.5
f1 (ppm)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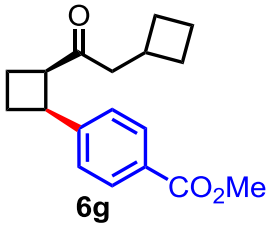
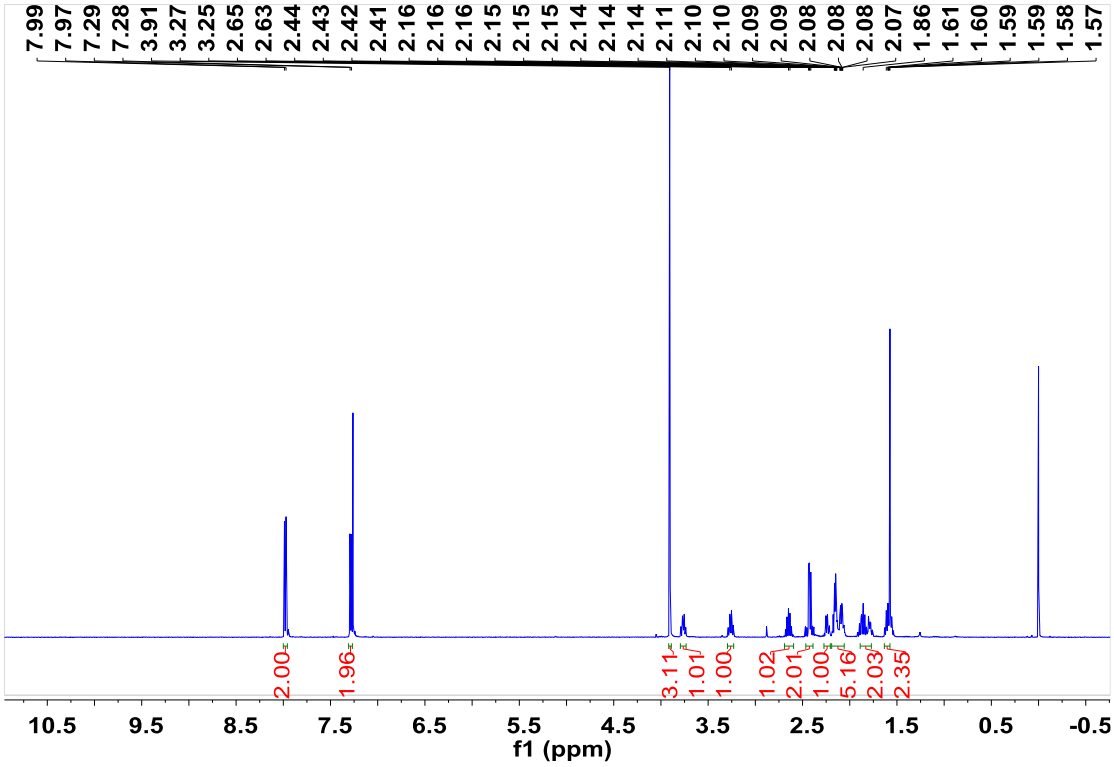
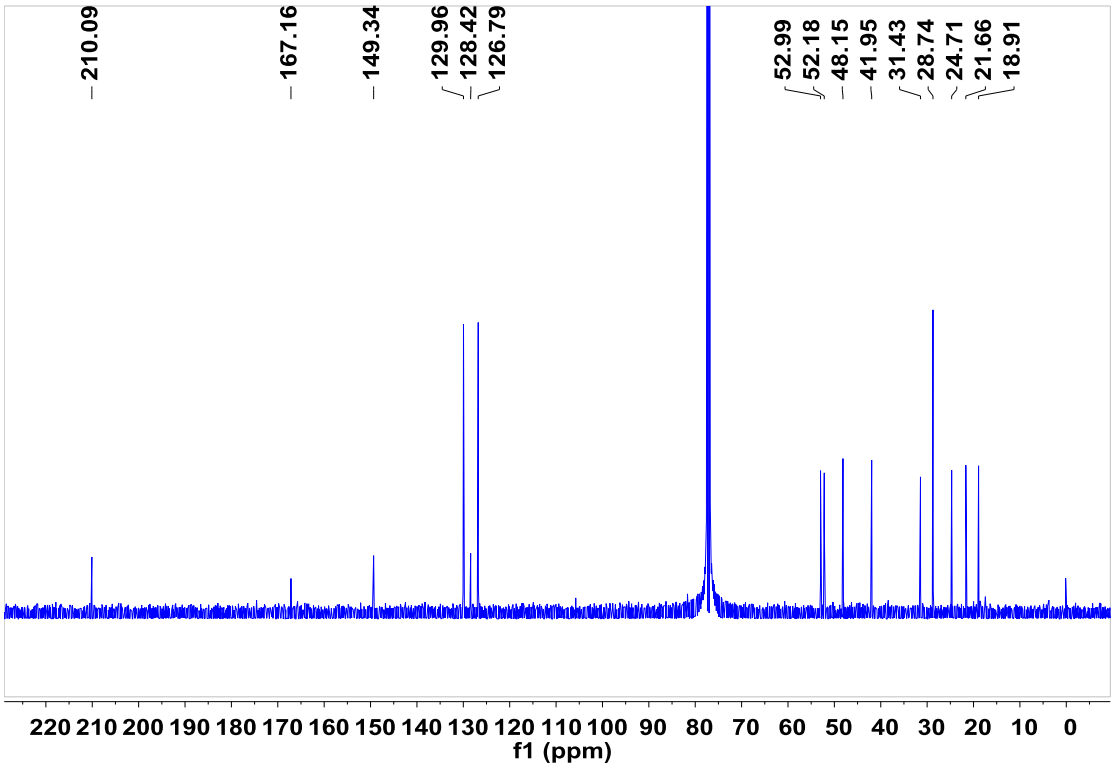
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


O
CO2Me

6g


7.99 7.97 7.29 7.28 3.91 3.27 3.25 2.65 2.63 2.44 2.43 2.42 2.41 2.16 2.16 2.16 2.15 2.15 2.15 2.14 2.14 2.14 2.11 2.10 2.10 2.09 2.09 2.08 2.08 2.08 2.07 1.86 1.61 1.60 1.59 1.59 1.58 1.57
2.00 1.96 3.11 1.01 1.00 1.02 2.01 1.00 5.16 2.03 2.35
10.5 9.5 8.5 7.5 6.5 5.5 4.5 3.5 2.5 1.5 0.5 -0.5
f1 (ppm)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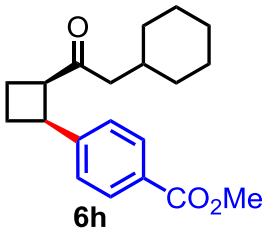
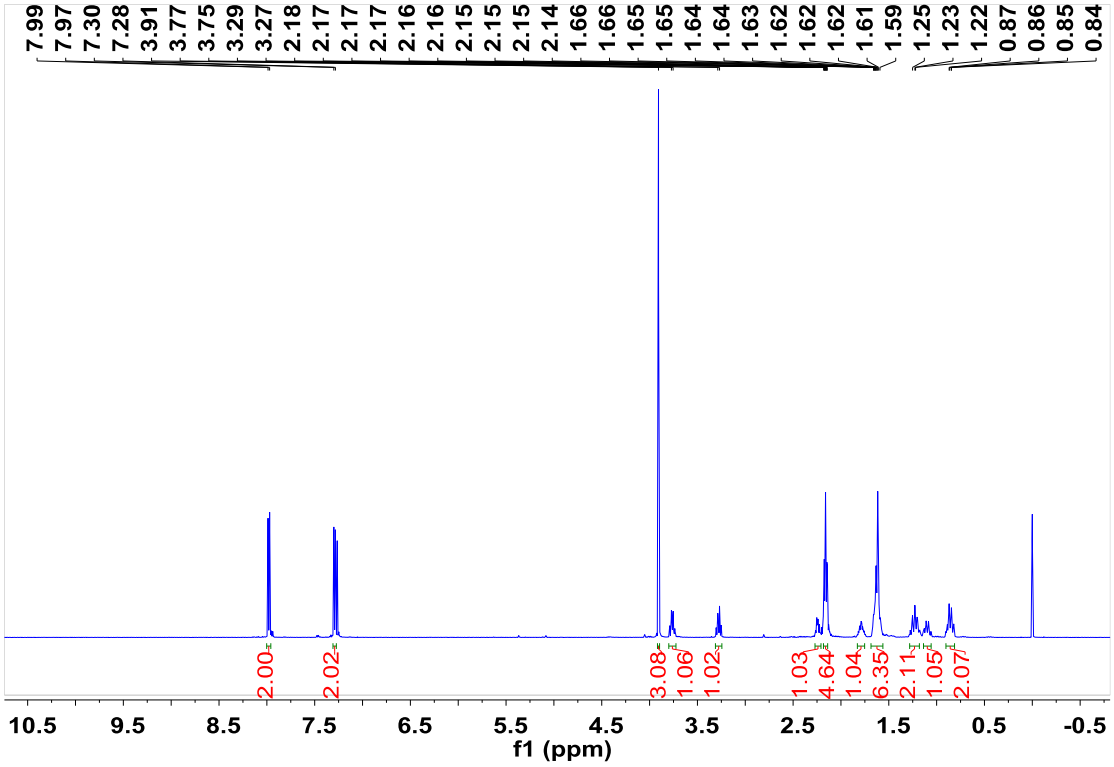
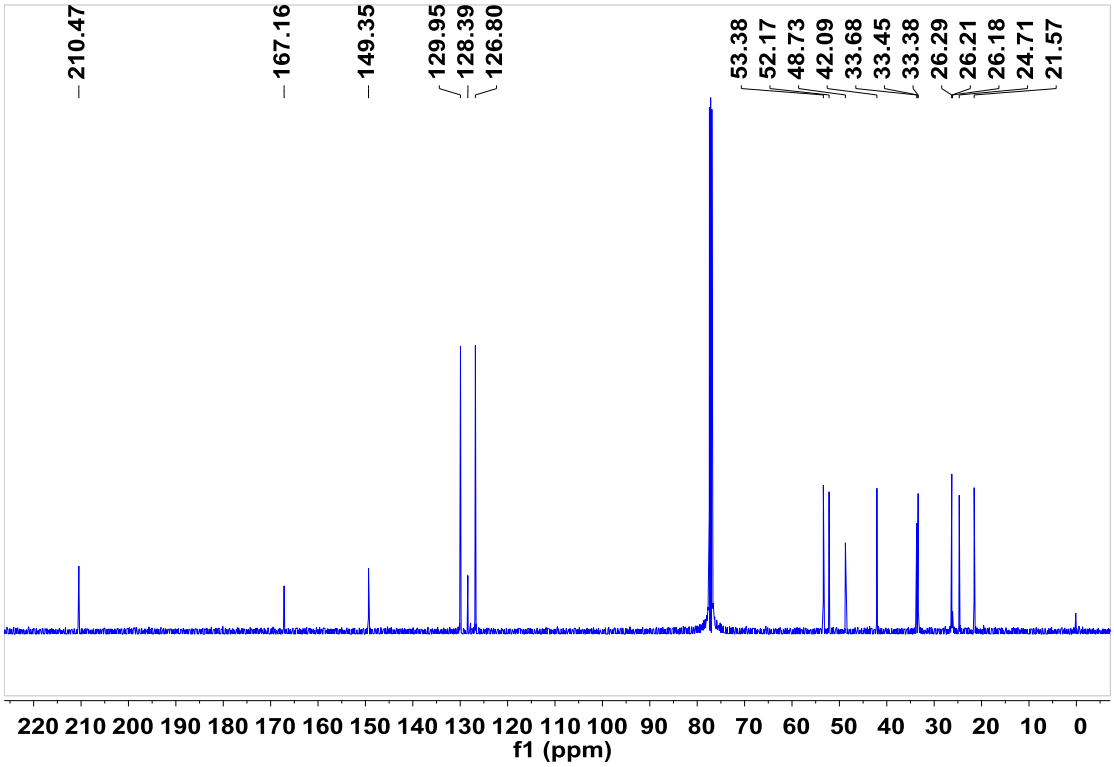
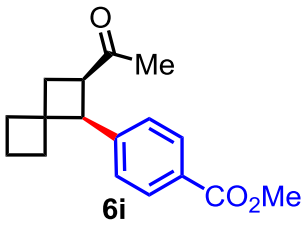
f1 (ppm)


O
CO2Me

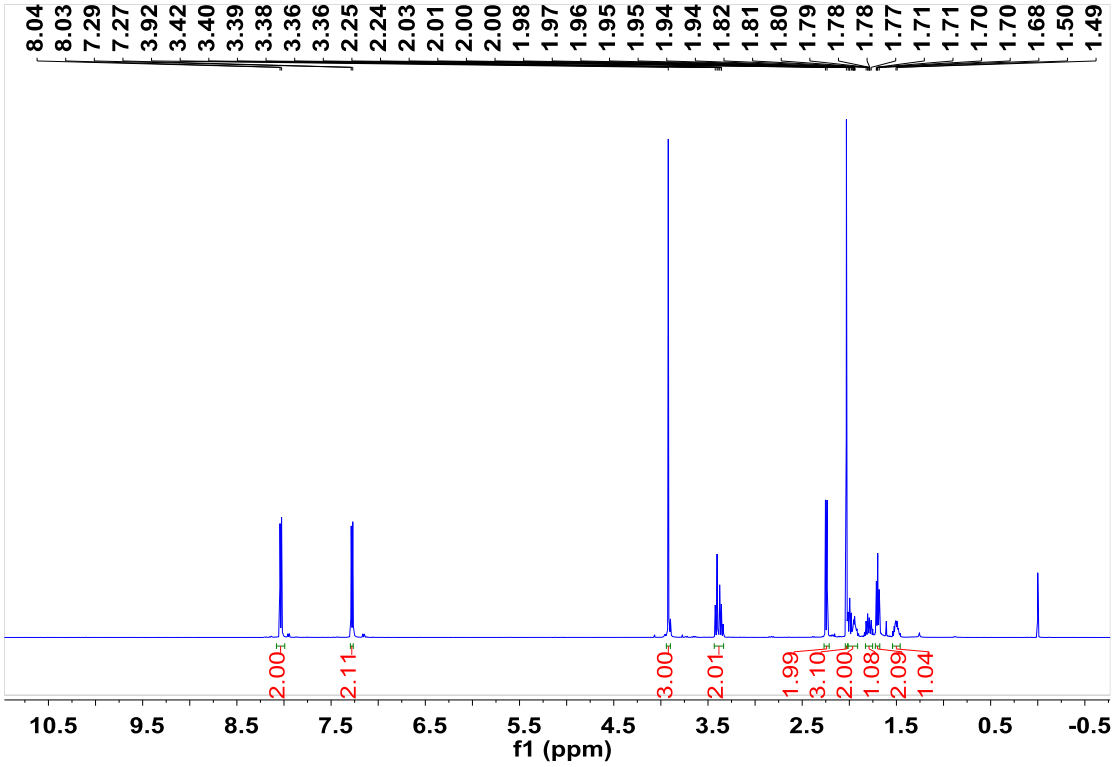
6h

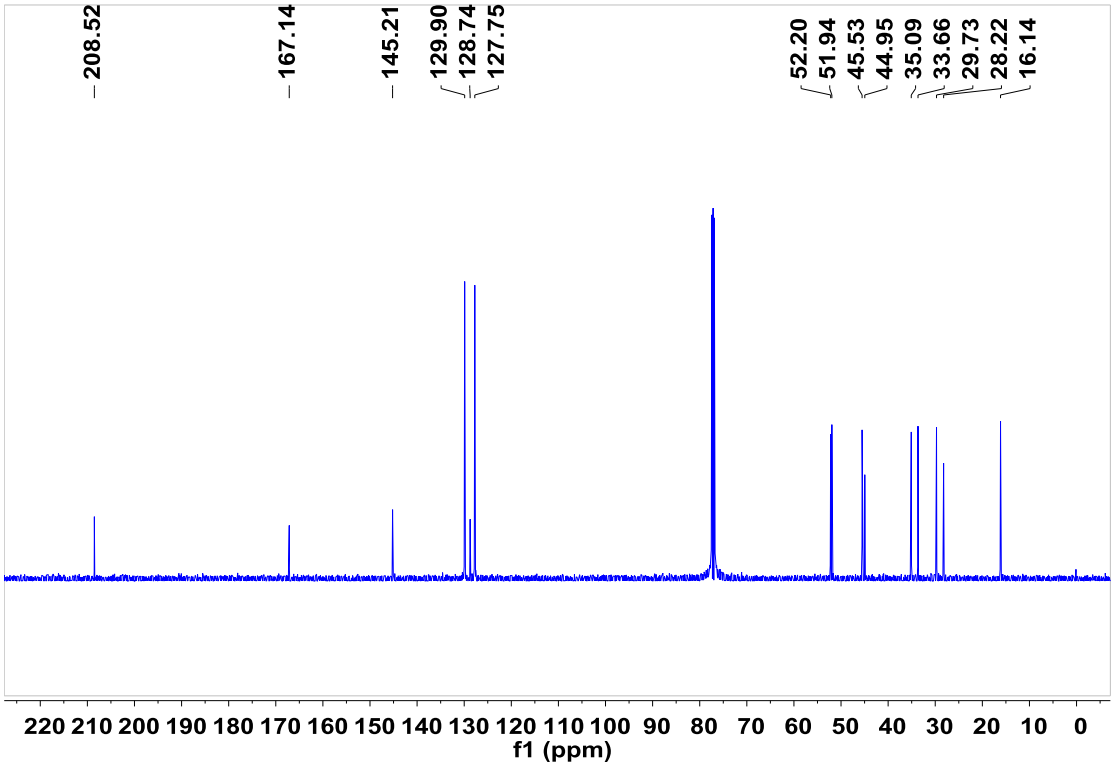

7.99 7.97 7.30 7.28 3.91 3.77 3.75 3.29 3.27 2.18 2.17 2.17 2.17 2.16 2.16 2.15 2.15 2.15 2.14 1.66 1.66 1.65 1.65 1.64 1.64 1.63 1.62 1.62 1.62 1.61 1.59 1.25 1.23 1.22 0.87 0.86 0.85 0.84
2.00 2.02 3.08 1.06 1.02 1.03 4.64 1.04 6.35 2.11 1.05 2.07
10.5 9.5 8.5 7.5 6.5 5.5 4.5 3.5 2.5 1.5 0.5 -0.5
f1 (ppm)

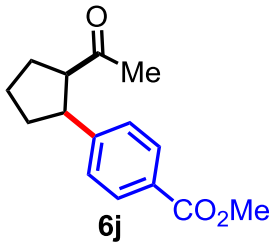

210.47 167.16 149.35 129.95 128.39 126.80 53.38 52.17 48.73 42.09 33.68 33.45 33.38 26.29 26.21 26.18 24.71 21.57
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


O
Me
CO2Me

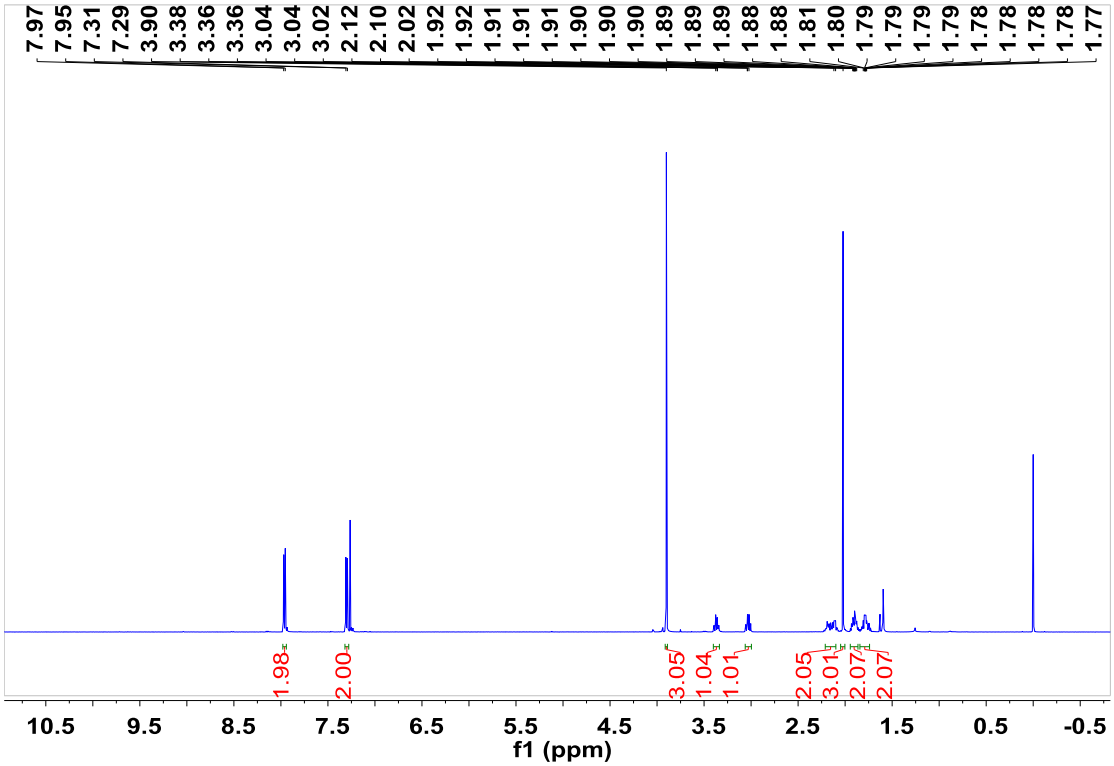
6i

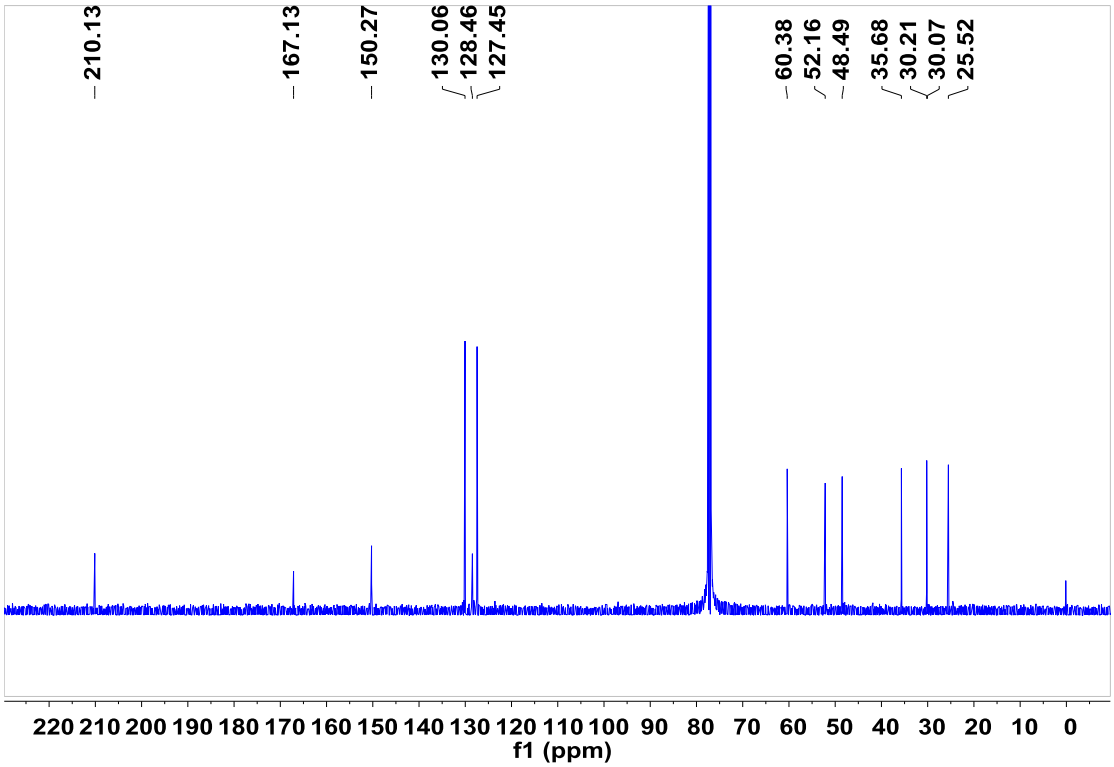

8.04 8.03 7.29 7.27 3.92 3.42 3.40 3.39 3.38 3.36 3.36 2.25 2.24 2.03 2.01 2.00 2.00 1.98 1.97 1.96 1.95 1.95 1.94 1.94 1.82 1.81 1.80 1.79 1.78 1.78 1.77 1.71 1.71 1.70 1.70 1.68 1.50 1.49
2.00 2.11 3.00 2.01 1.99 3.10 2.00 1.08 2.09 1.04
10.5 9.5 8.5 7.5 6.5 5.5 4.5 3.5 2.5 1.5 0.5 -0.5
f1 (ppm)

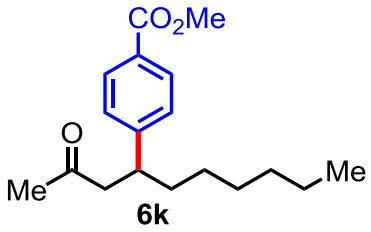

208.52 167.14 145.21 129.90 128.74 127.75 52.20 51.94 45.53 44.95 35.09 33.66 29.73 28.22 16.14
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


O
Me
CO2Me

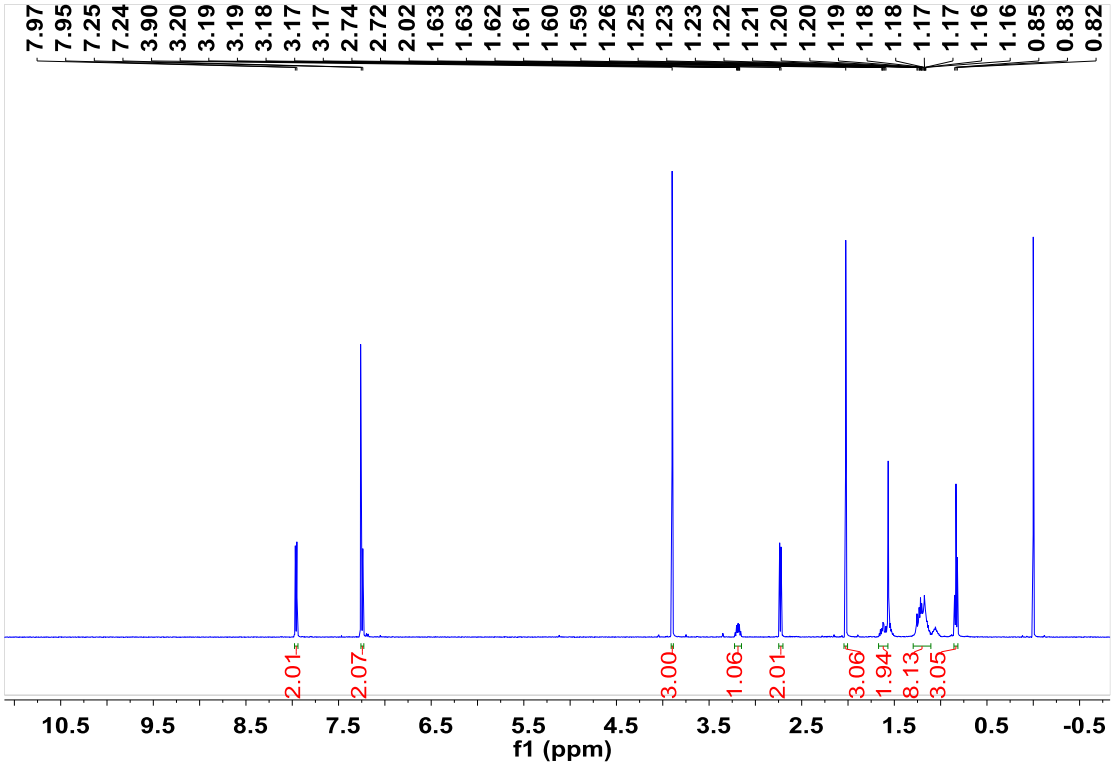
6j

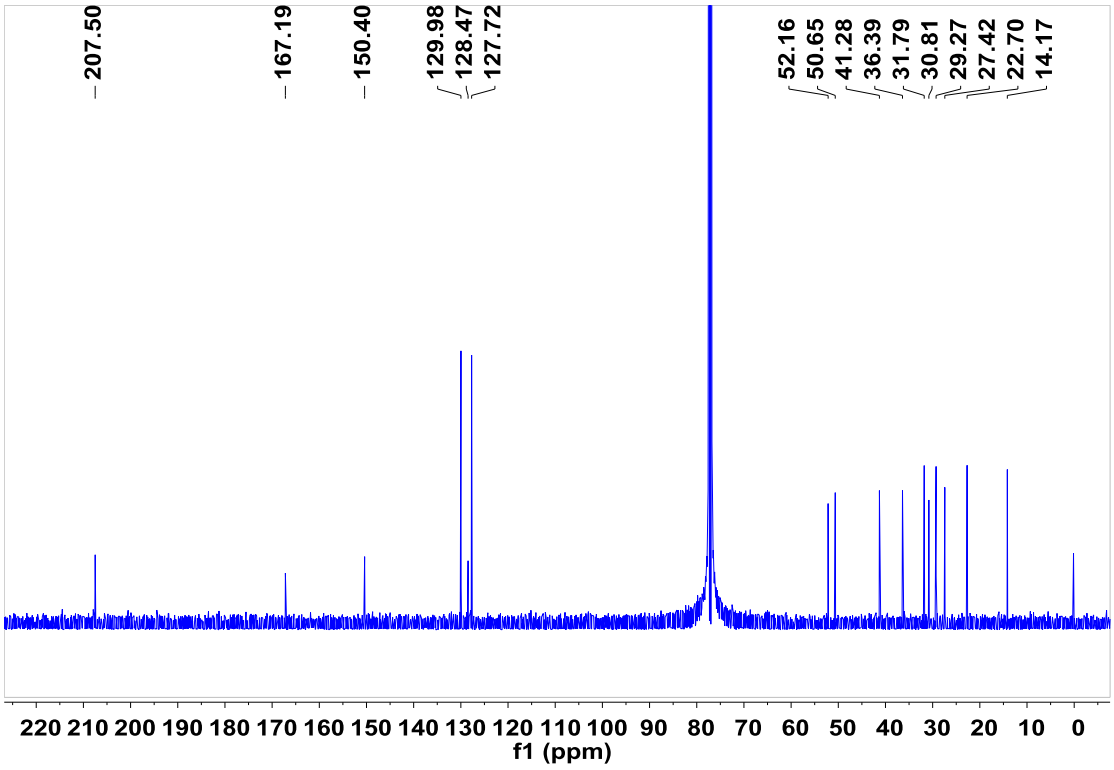

7.97 7.95 7.31 7.29 3.90 3.38 3.36 3.36 3.04 3.04 3.02 2.12 2.10 2.02 1.92 1.92 1.91 1.91 1.91 1.90 1.90 1.90 1.89 1.89 1.89 1.88 1.88 1.81 1.80 1.79 1.79 1.79 1.79 1.78 1.78 1.78 1.78 1.77
1.98
2.00
3.05
1.04
1.01
2.05
3.01
2.07
2.07
10.5 9.5 8.5 7.5 6.5 5.5 4.5 3.5 2.5 1.5 0.5 -0.5
f1 (ppm)

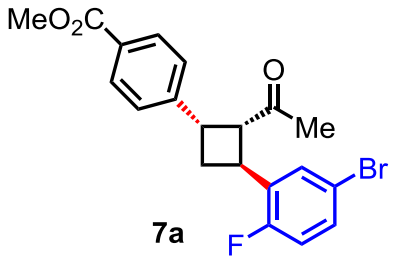

210.13
167.13
150.27
130.06
128.46
127.45
60.38
52.16
48.49
35.68
30.21
30.07
25.52
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


CO2Me
O
Me
Me

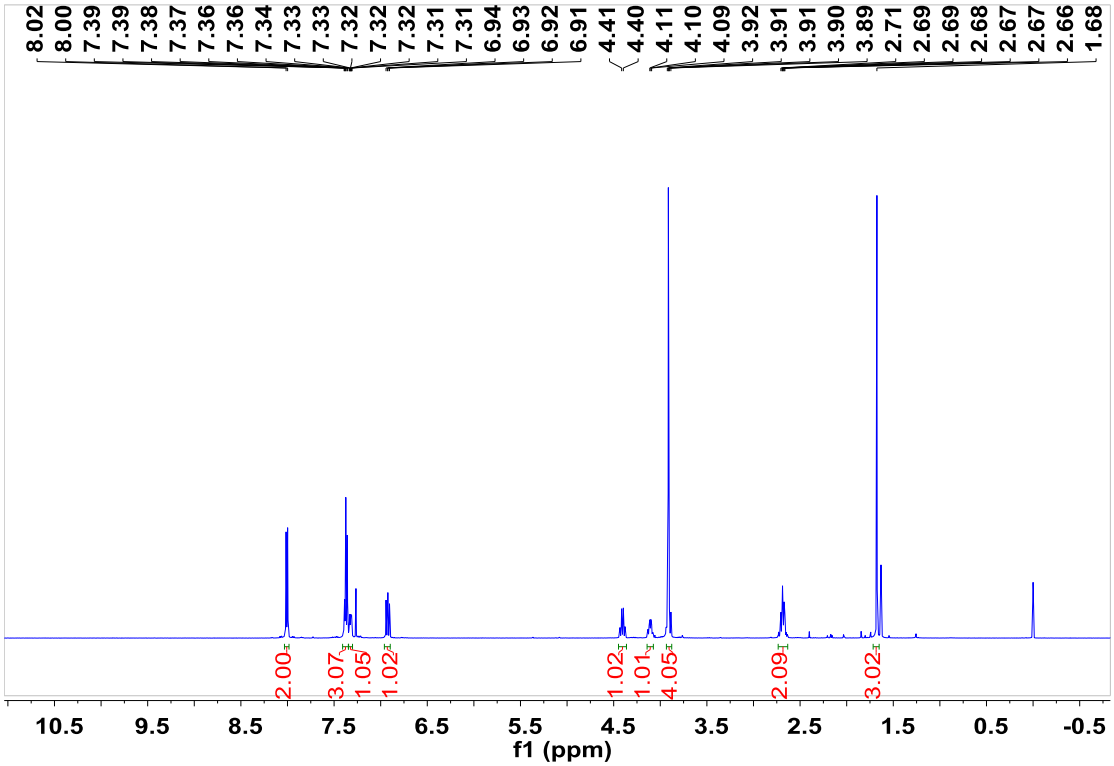
6k

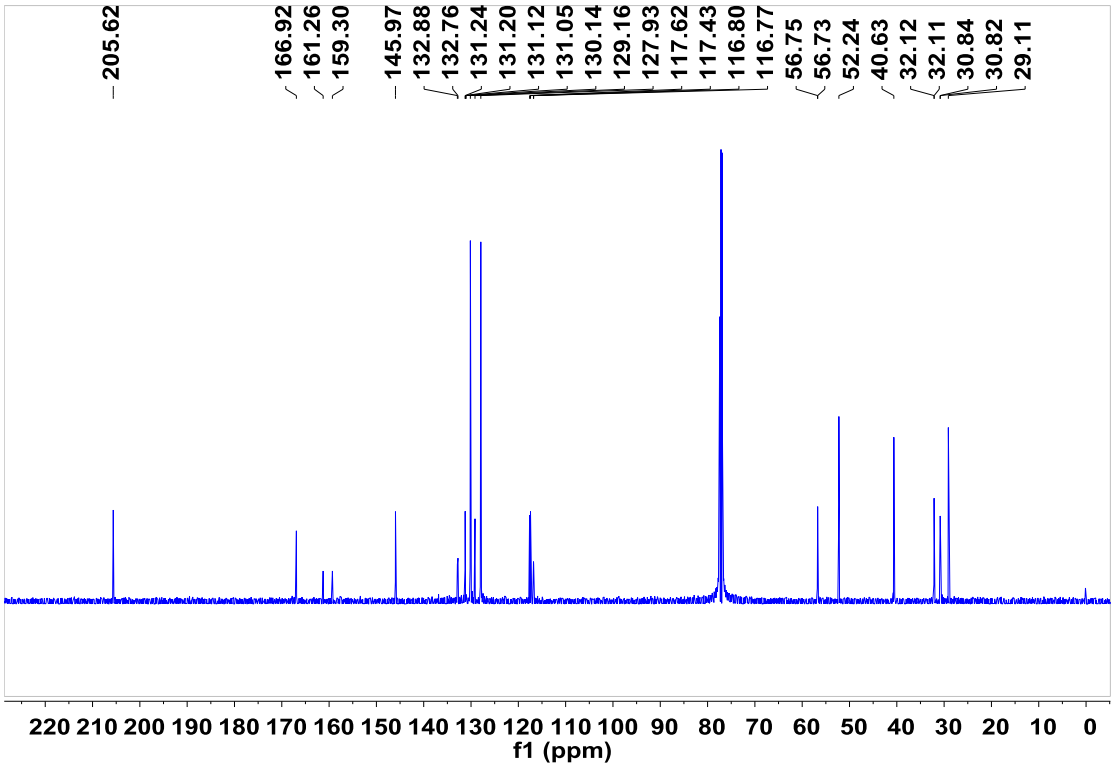

7.97 7.95 7.25 7.24 3.90 3.20 3.19 3.19 3.18 3.17 3.17 2.74 2.72 2.02 1.63 1.63 1.62 1.61 1.60 1.59 1.26 1.25 1.23 1.23 1.22 1.21 1.20 1.20 1.19 1.18 1.18 1.17 1.17 1.16 1.16 0.85 0.83 0.82
2.01 2.07 3.00 1.06 2.01 3.06 1.94 8.13 3.05
10.5 9.5 8.5 7.5 6.5 5.5 4.5 3.5 2.5 1.5 0.5 -0.5
f1 (ppm)

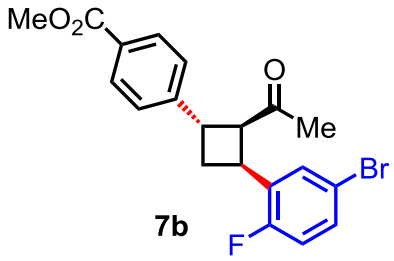

207.50 167.19 150.40 129.98 128.47 127.72 52.16 50.65 41.28 36.39 31.79 30.81 29.27 27.42 22.70 14.17
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


MeO2C
O
Me
Br
F

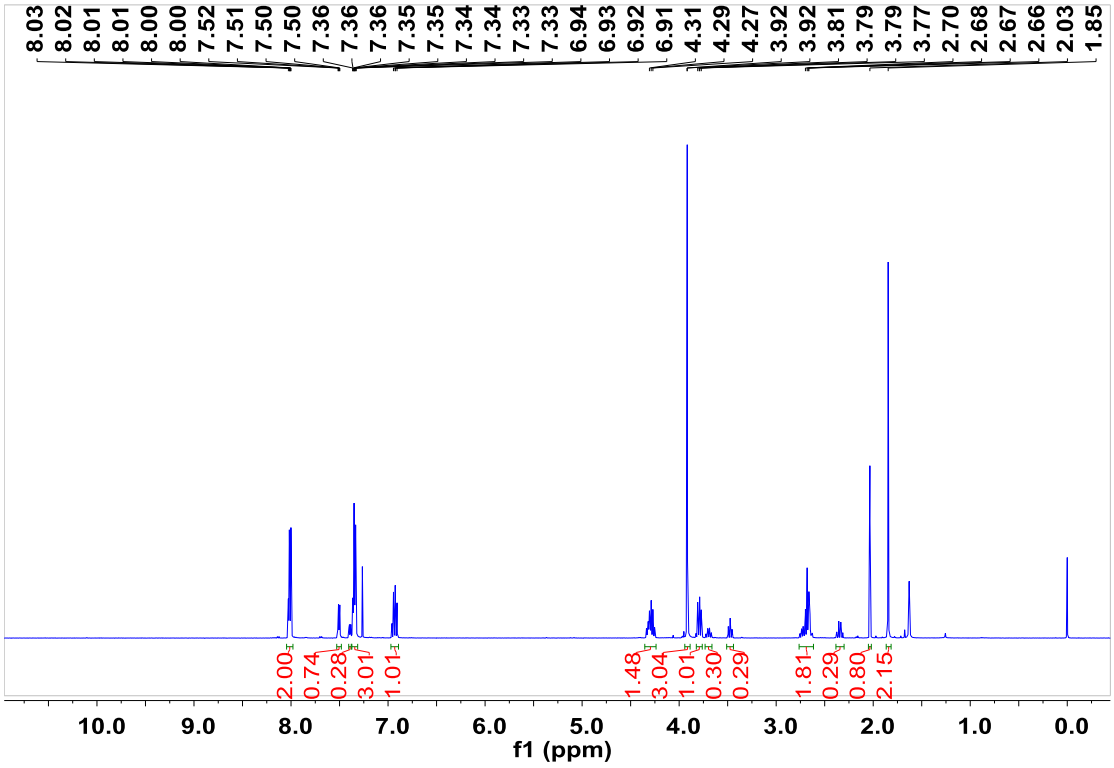
7a

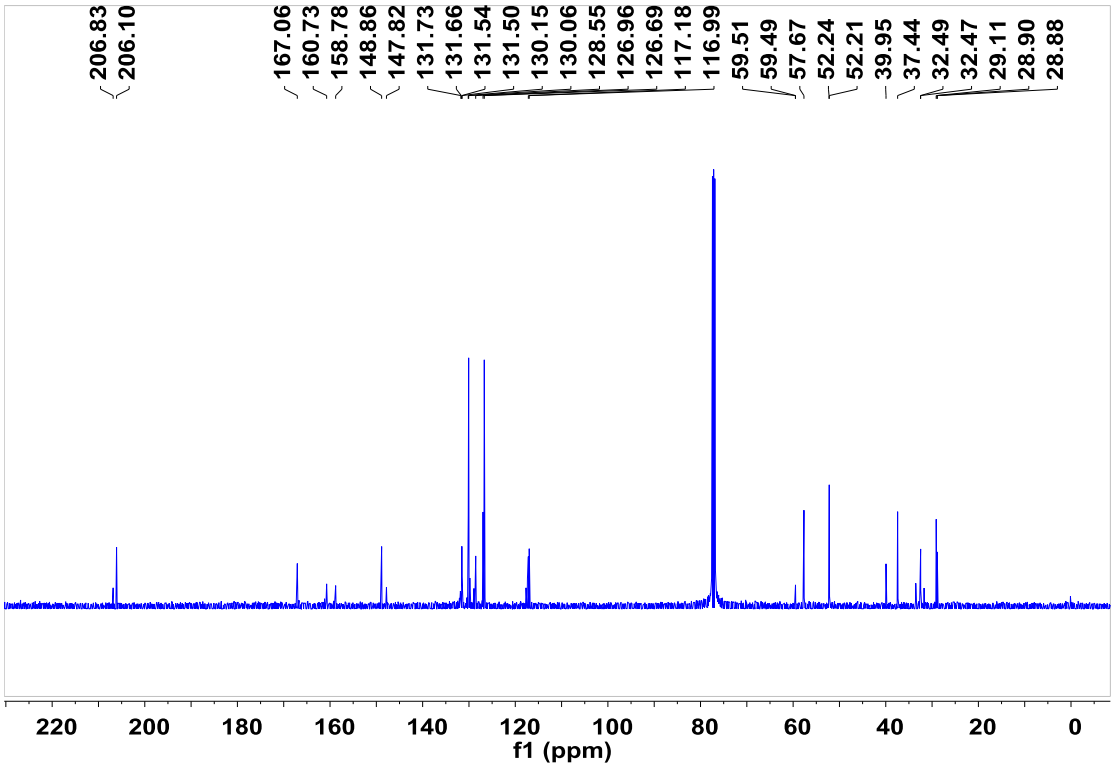

8.02 8.00 7.39 7.39 7.38 7.37 7.36 7.36 7.34 7.33 7.33 7.32 7.32 7.32 7.31 7.31 6.94 6.93 6.92 6.91 4.41 4.40 4.11 4.10 4.09 3.92 3.91 3.91 3.90 3.89 2.71 2.69 2.69 2.68 2.67 2.67 2.66 1.68
2.00 3.07 1.05 1.02 1.02 1.01 4.05 2.09 3.02
10.5 9.5 8.5 7.5 6.5 5.5 4.5 3.5 2.5 1.5 0.5 -0.5
f1 (ppm)

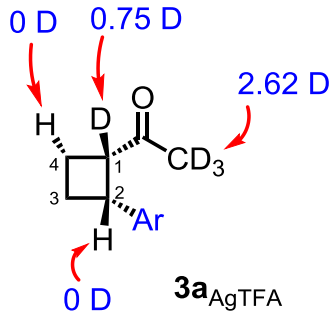

205.62
166.92 161.26 159.30 145.97 132.88 132.76 131.24 131.20 131.12 131.05 130.14 129.16 127.93 117.62 117.43 116.80 116.77 56.75 56.73 52.24 40.63 32.12 32.11 30.84 30.82 29.11
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


MeO2C
O
Me
Br
F

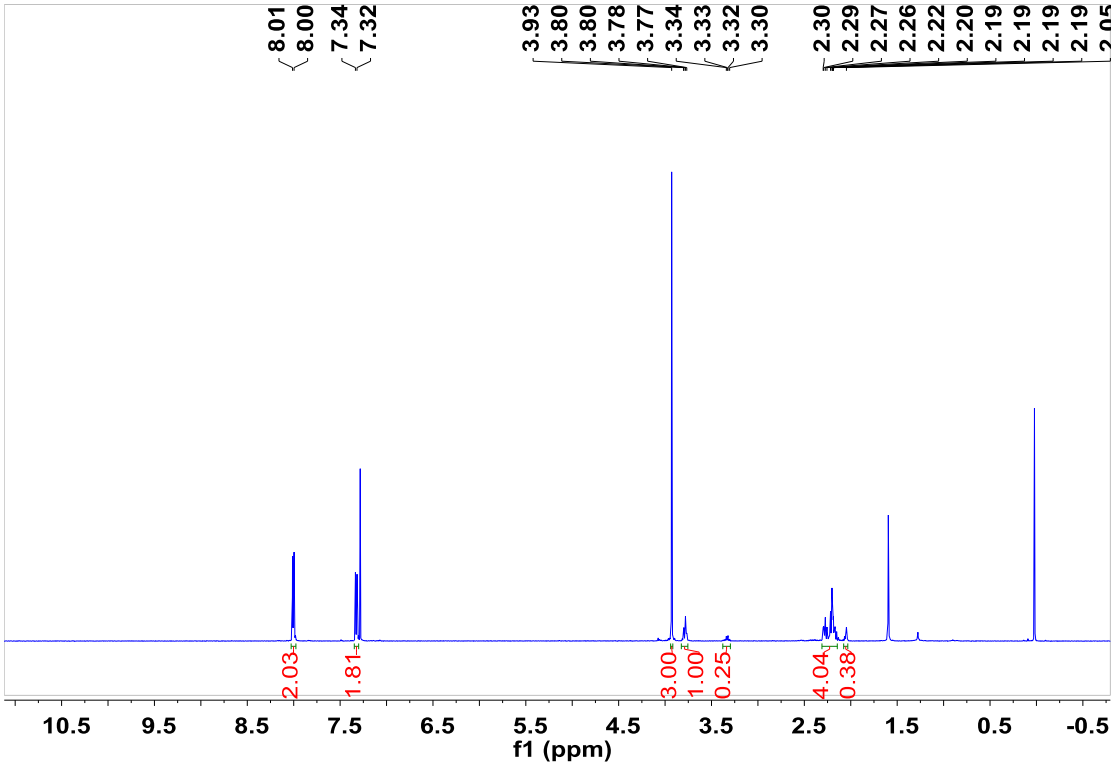
7b

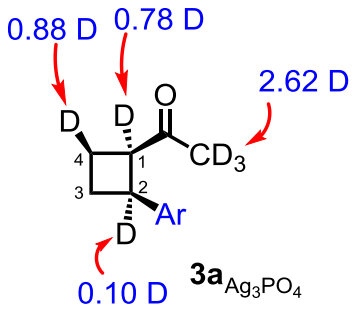

8.03 8.02 8.01 8.01 8.00 8.00 7.52 7.51 7.50 7.50 7.36 7.36 7.36 7.35 7.35 7.34 7.34 7.33 7.33 6.94 6.93 6.92 6.91 4.31 4.29 4.27 3.92 3.92 3.81 3.79 3.79 3.77 2.70 2.68 2.67 2.66 2.03 1.85
2.00 0.74 0.28 3.01 1.01 1.48 3.04 1.01 0.30 0.29 1.81 0.29 0.80 2.15
10.0 9.0 8.0 7.0 6.0 5.0 4.0 3.0 2.0 1.0 0.0
f1 (ppm)


206.83 206.10 167.06 160.73 158.78 148.86 147.82 131.73 131.66 131.54 131.50 130.15 130.06 128.55 126.96 126.69 117.18 116.99 59.51 59.49 57.67 52.24 52.21 39.95 37.44 32.49 32.47 29.11 28.90 28.88
220 200 180 160 140 120 100 80 60 40 20 0
f1 (ppm)

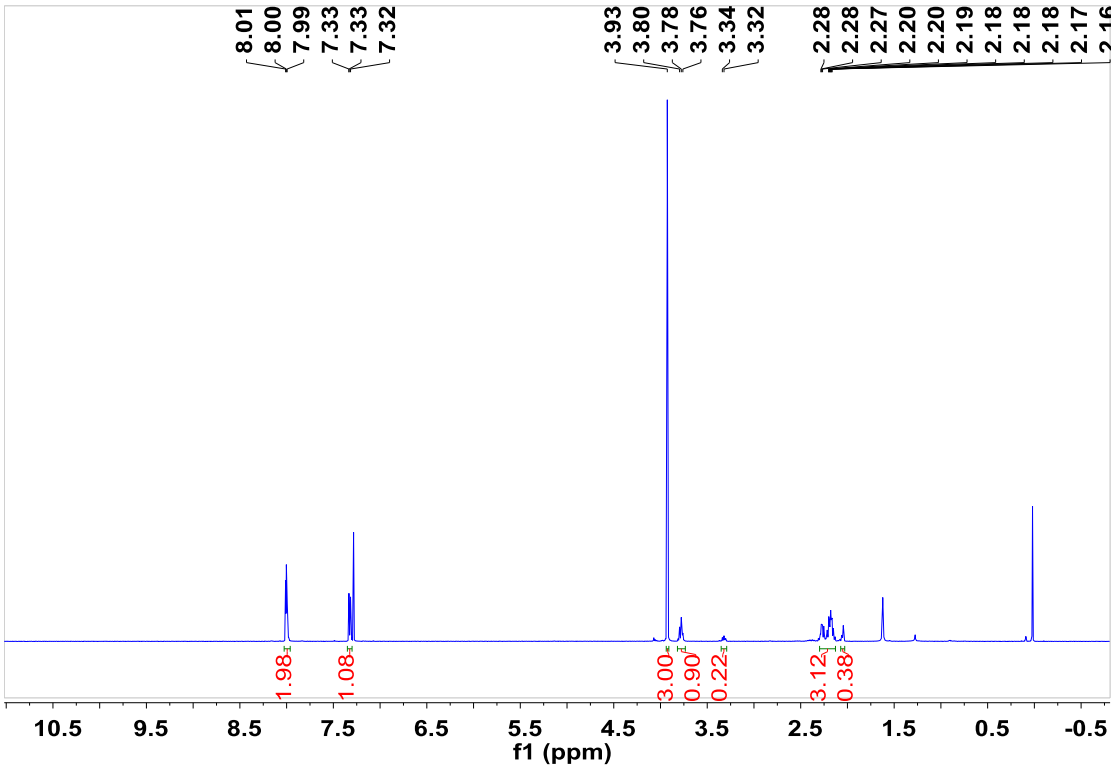

0 D
0.75 D
2.62 D
H
D
O
CD3
4
1
3
2
Ar
H
0 D

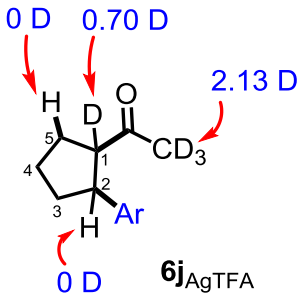
3a$_{AgTFA}$


8.01
8.00
7.34
7.32
3.93
3.80
3.80
3.78
3.77
3.34
3.33
3.32
3.30
2.30
2.29
2.27
2.26
2.22
2.20
2.19
2.19
2.19
2.19
2.03
1.81
3.00
1.00
0.25
4.04
0.38
10.5
9.5
8.5
7.5
6.5
5.5
4.5
3.5
2.5
1.5
0.5
-0.5
f1 (ppm)

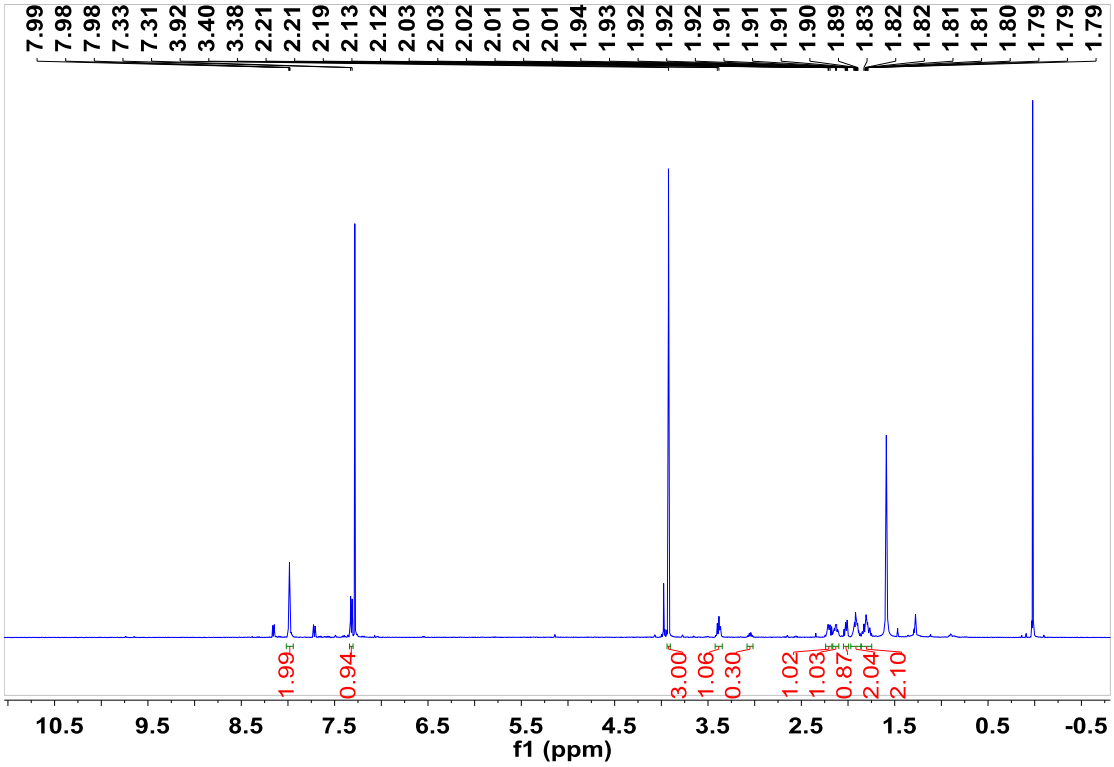

0.88 D
0.78 D
2.62 D
D
D
O
CD3
4
1
3
2
Ar
D
0.10 D

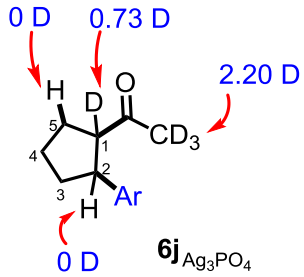
3a$_{Ag_3PO_4}$


8.01
8.00
7.99
7.33
7.33
7.32
3.93
3.80
3.78
3.76
3.34
3.32
2.28
2.28
2.27
2.20
2.20
2.19
2.18
2.18
2.18
2.17
1.98
1.08
3.00
0.90
0.22
3.12
0.38
10.5
9.5
8.5
7.5
6.5
5.5
4.5
3.5
2.5
1.5
0.5
-0.5
f1 (ppm)

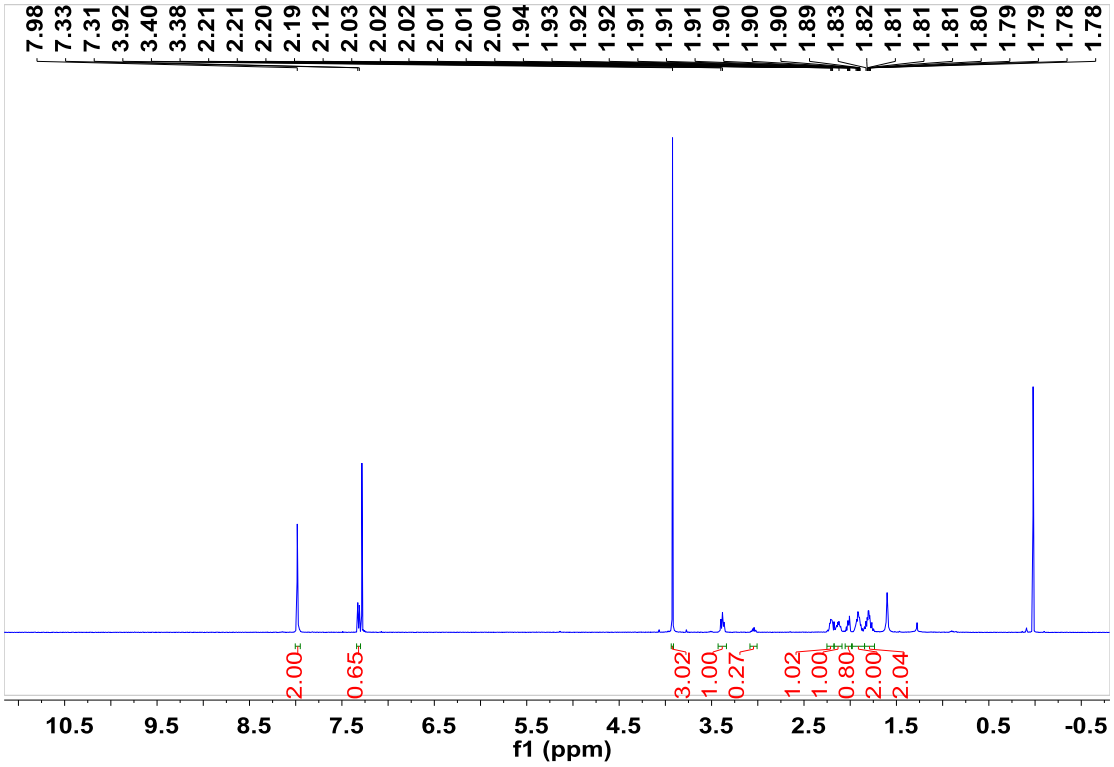

0 D
0.70 D
2.13 D
H
D
O
CD3
5
1
4
2
3
Ar
H
0 D

6j$_{AgTFA}$

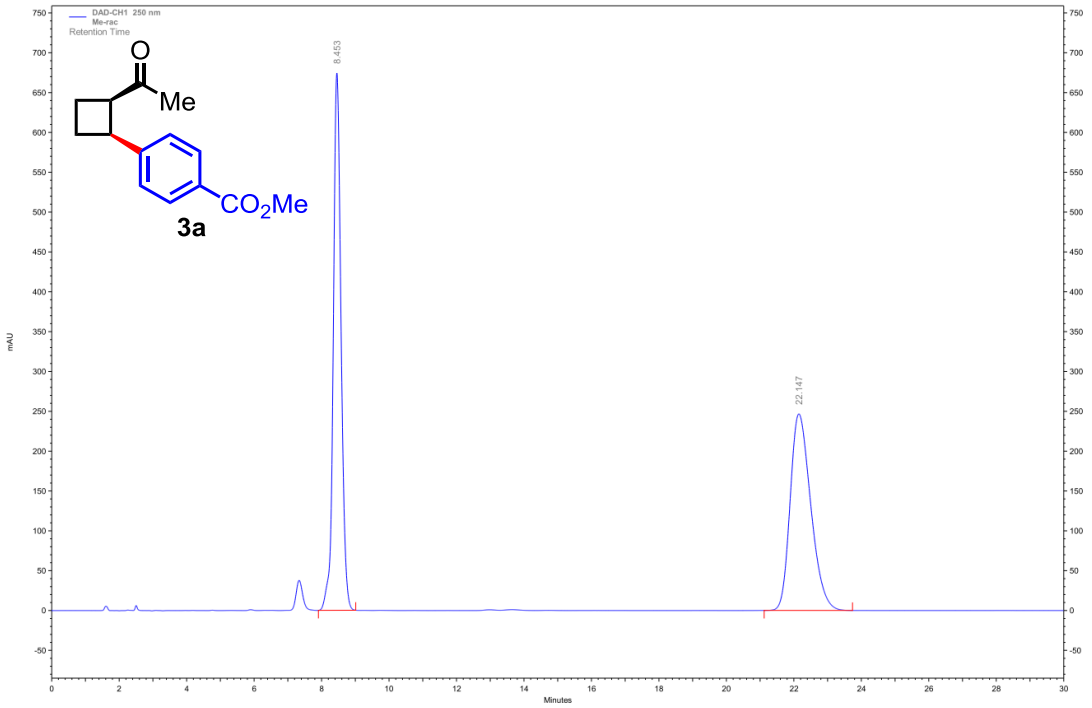

7.99 7.98 7.98 7.33 7.31 3.92 3.40 3.38 2.21 2.21 2.19 2.13 2.12 2.03 2.03 2.02 2.01 2.01 2.01 1.94 1.93 1.92 1.92 1.92 1.91 1.91 1.91 1.90 1.89 1.83 1.82 1.82 1.81 1.81 1.80 1.79 1.79 1.79
1.99
0.94
3.00
1.06
0.30
1.02
1.03
0.87
2.04
2.10
10.5 9.5 8.5 7.5 6.5 5.5 4.5 3.5 2.5 1.5 0.5 -0.5
f1 (ppm)


0 D
0.73 D
2.20 D
H
D
O
CD3
5
4
1
2
3
Ar
H
0 D

6j$_{Ag_3PO_4}$

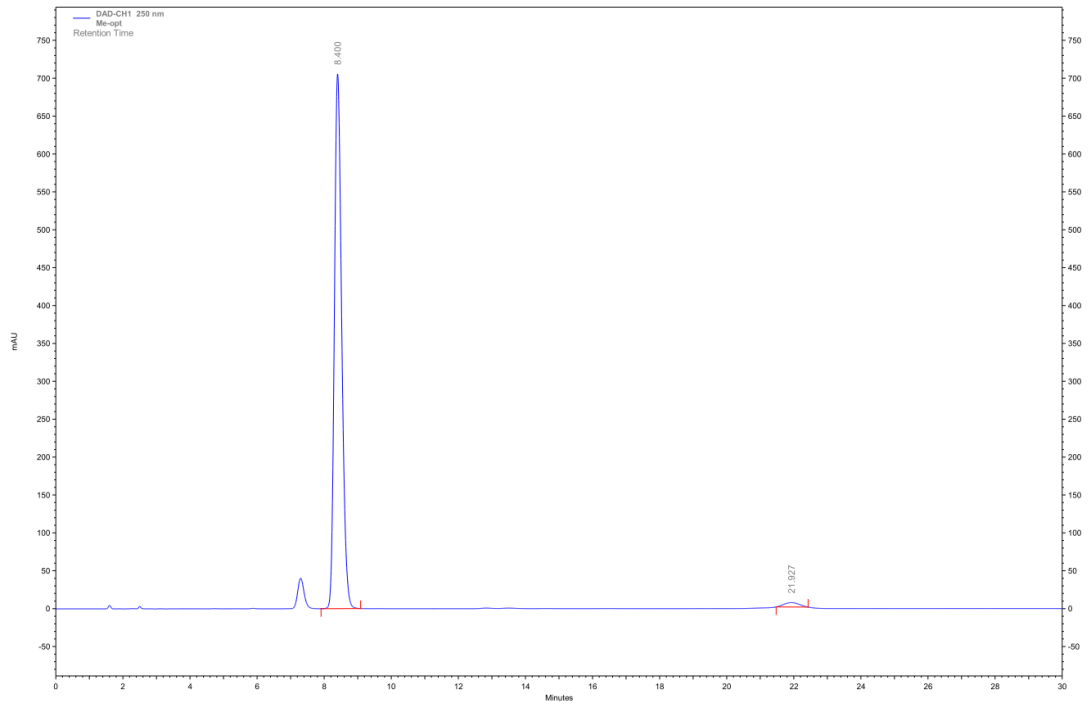

7.98 7.33 7.31 3.92 3.40 3.38 2.21 2.21 2.20 2.19 2.12 2.03 2.02 2.02 2.01 2.01 2.00 1.94 1.93 1.92 1.92 1.91 1.91 1.91 1.90 1.90 1.90 1.89 1.83 1.82 1.81 1.81 1.81 1.80 1.79 1.79 1.78 1.78
2.00 0.65 3.02 1.00 0.27 1.02 1.00 0.80 2.00 2.04
10.5 9.5 8.5 7.5 6.5 5.5 4.5 3.5 2.5 1.5 0.5 -0.5
f1 (ppm)

9. HPLC Data



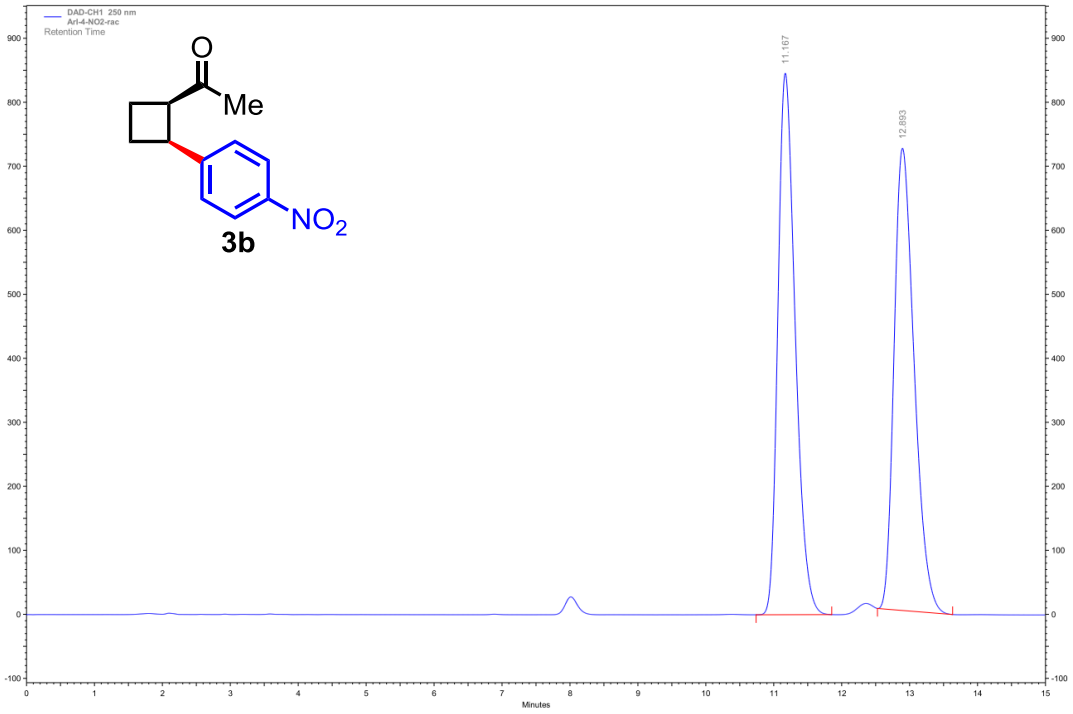
DAD-CH1 250 nm Results

Retention Time	Area	Area %
8.453	43106209	50.73
22.147	41870252	49.27



DAD-CH1 250 nm Results

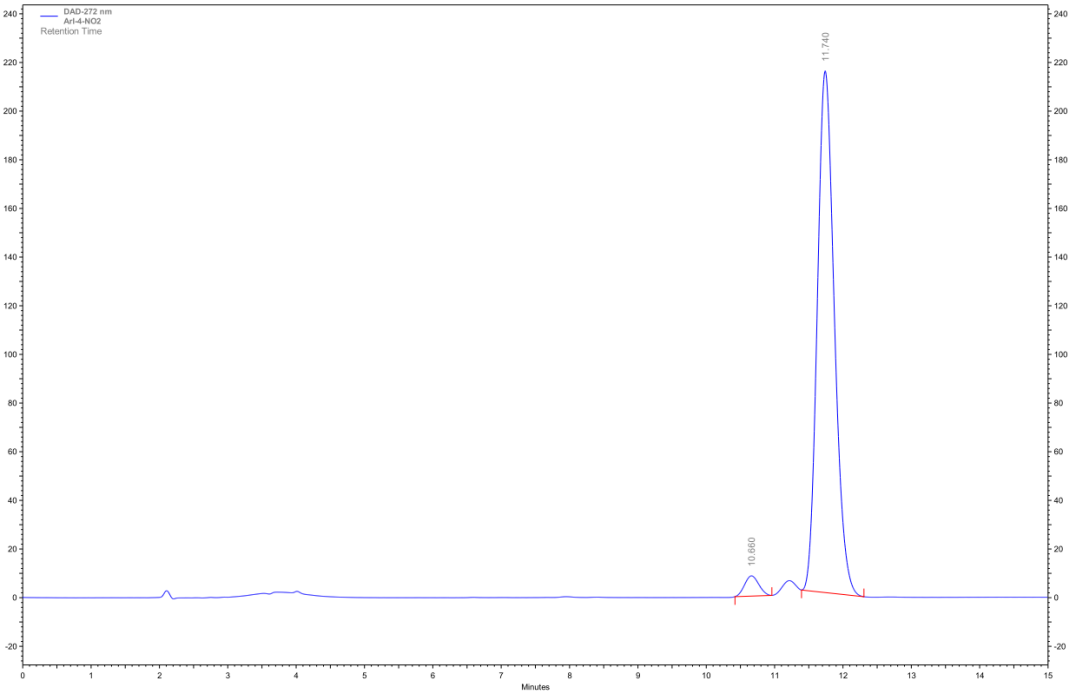
Retention Time	Area	Area %
8.400	42690583	98.31
21.927	733616	1.69



3b

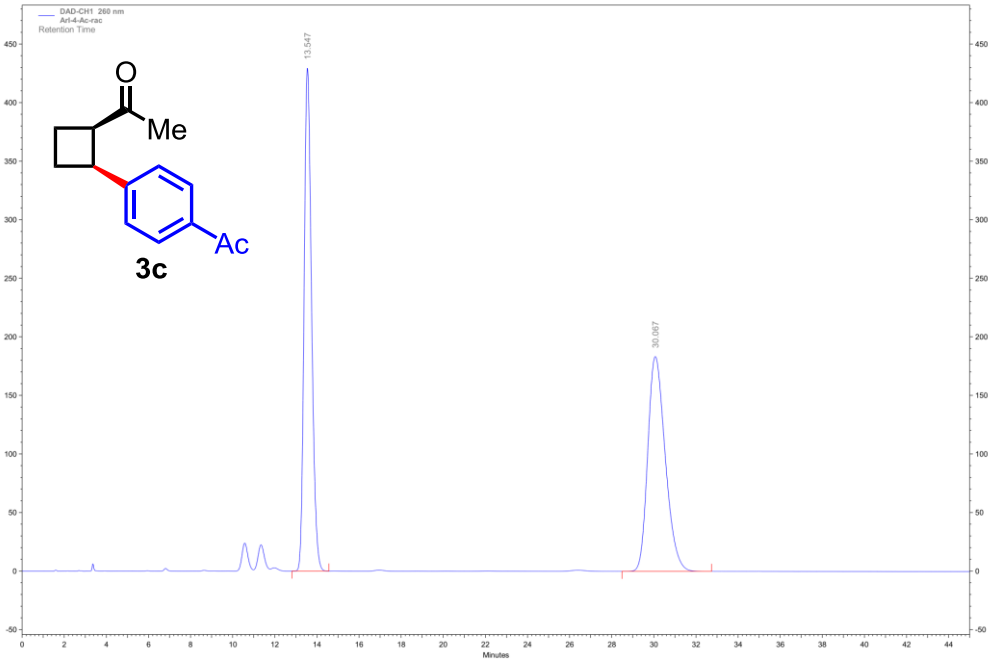
DAD-CH1 250 nm Results

Retention Time	Area	Area %
11.167	59951146	50.29
12.893	59263659	49.71



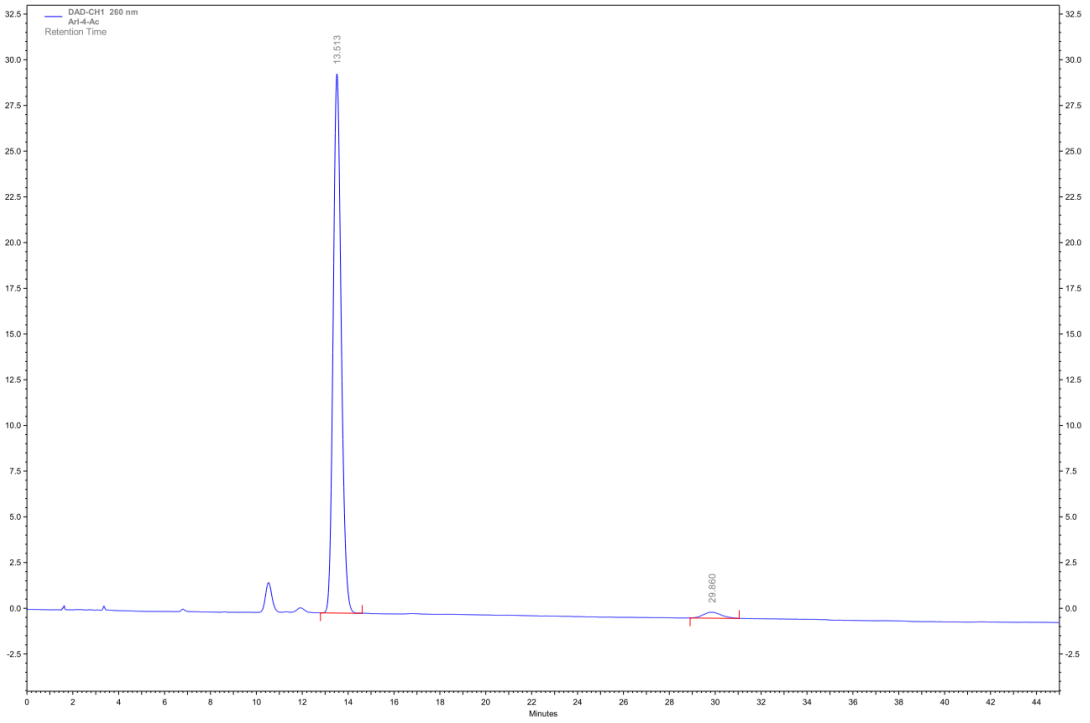
DAD-CH1 250 nm Results

Retention Time	Area	Area %
10.660	256371	3.15
11.740	7884071	96.85



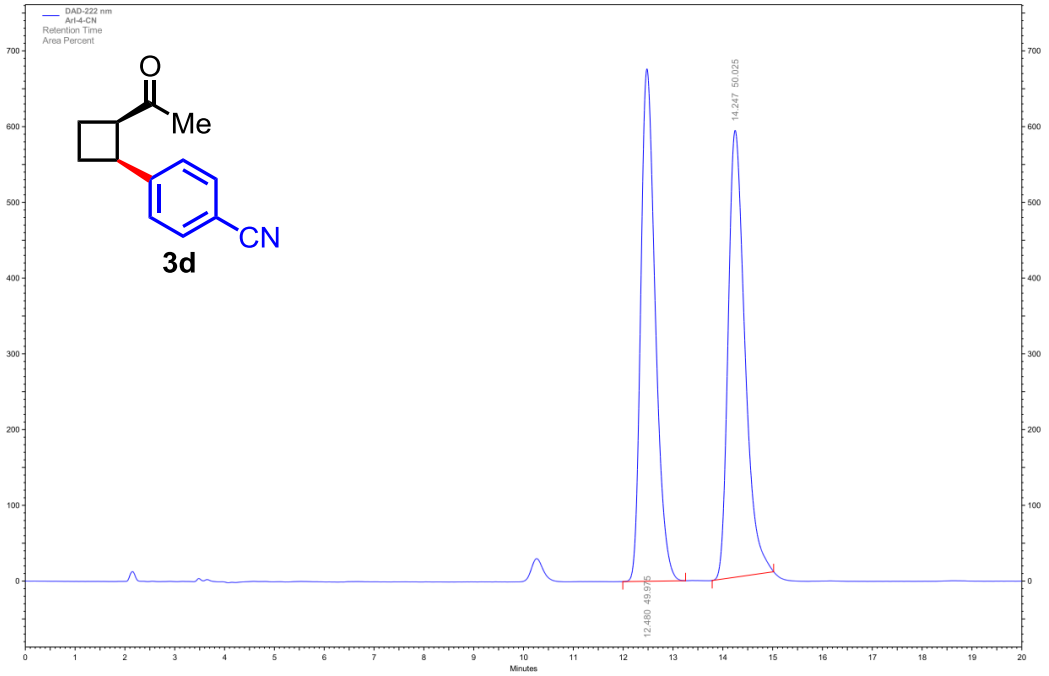
DAD-CH1 260 nm Results

Retention Time	Area	Area %
13.547	42257191	49.97
30.067	42313368	50.03



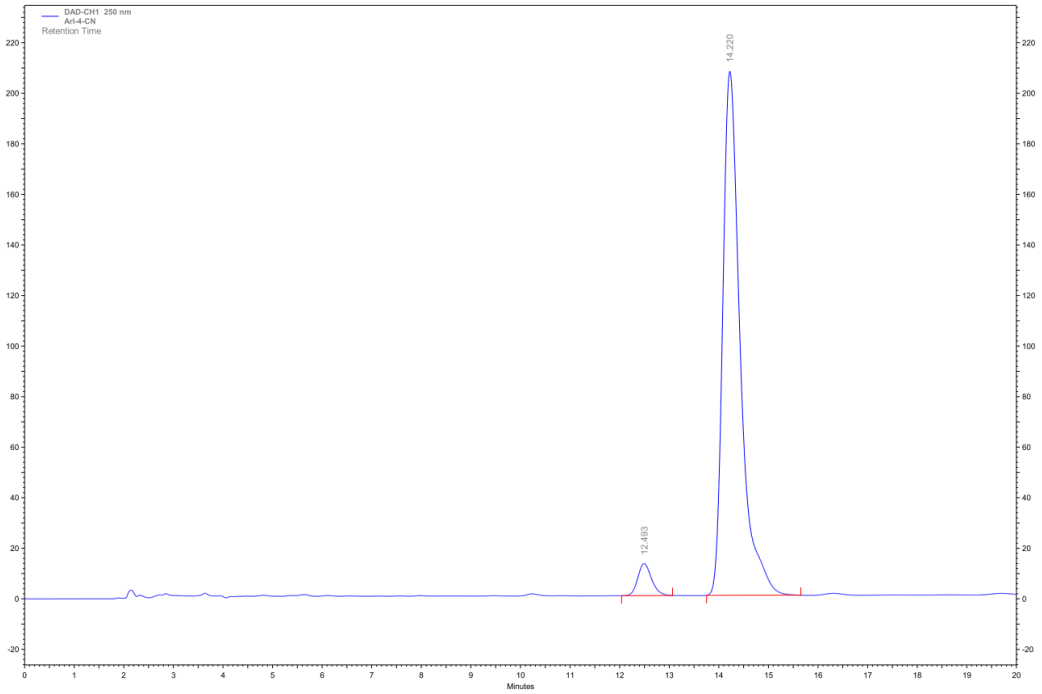
DAD-CH1 260 nm Results

Retention Time	Area	Area %
13.513	2869844	97.57
29.860	71579	2.43



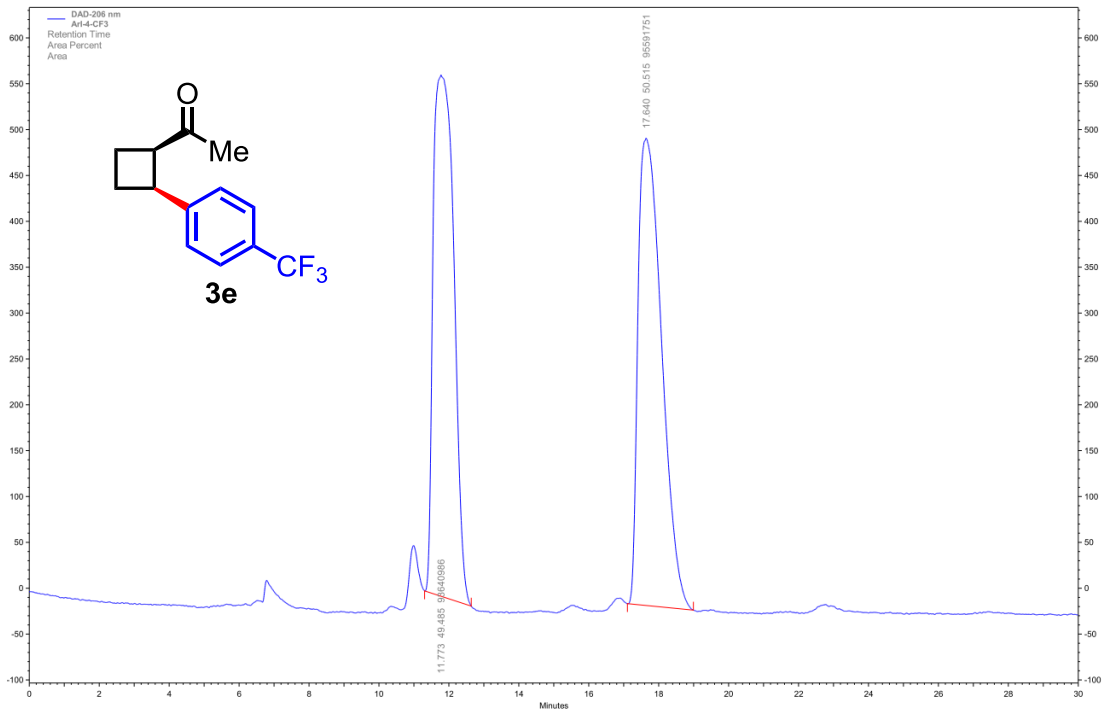
DAD-CH1 250 nm Results

Retention Time	Area	Area %
12.480	23932548	49.97
14.247	25407345	50.03



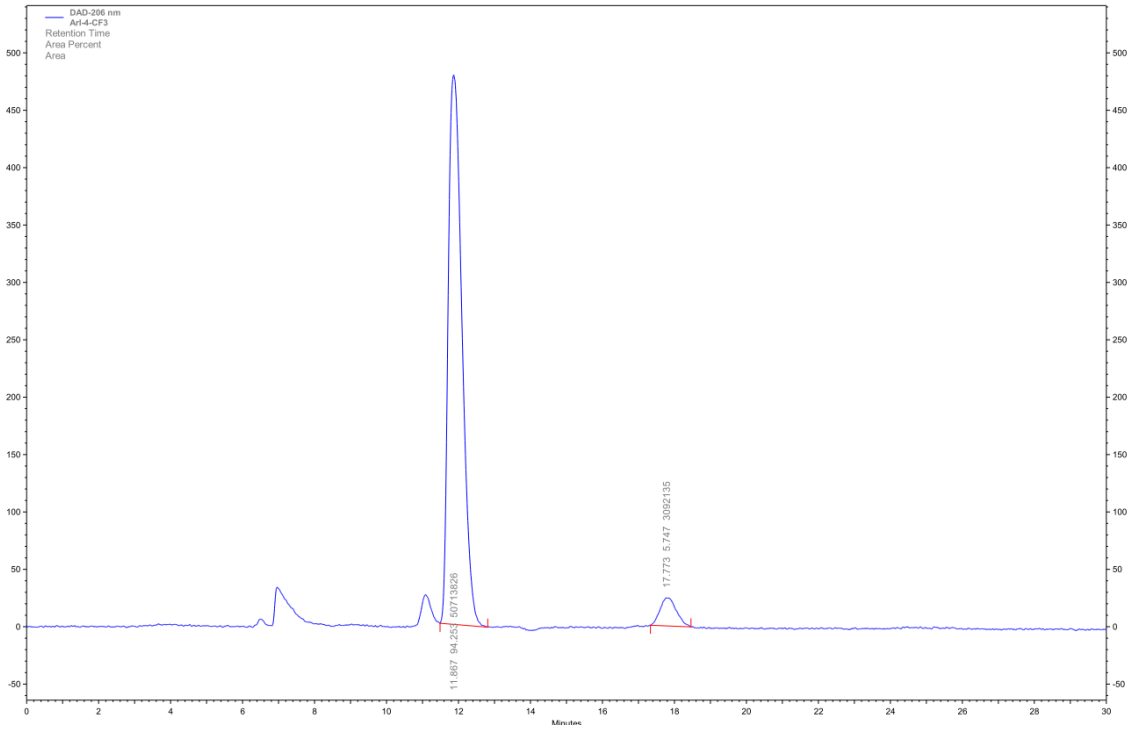
DAD-CH1 250 nm Results

Retention Time	Area	Area %
12.493	979580	4.61
14.220	20251991	95.39



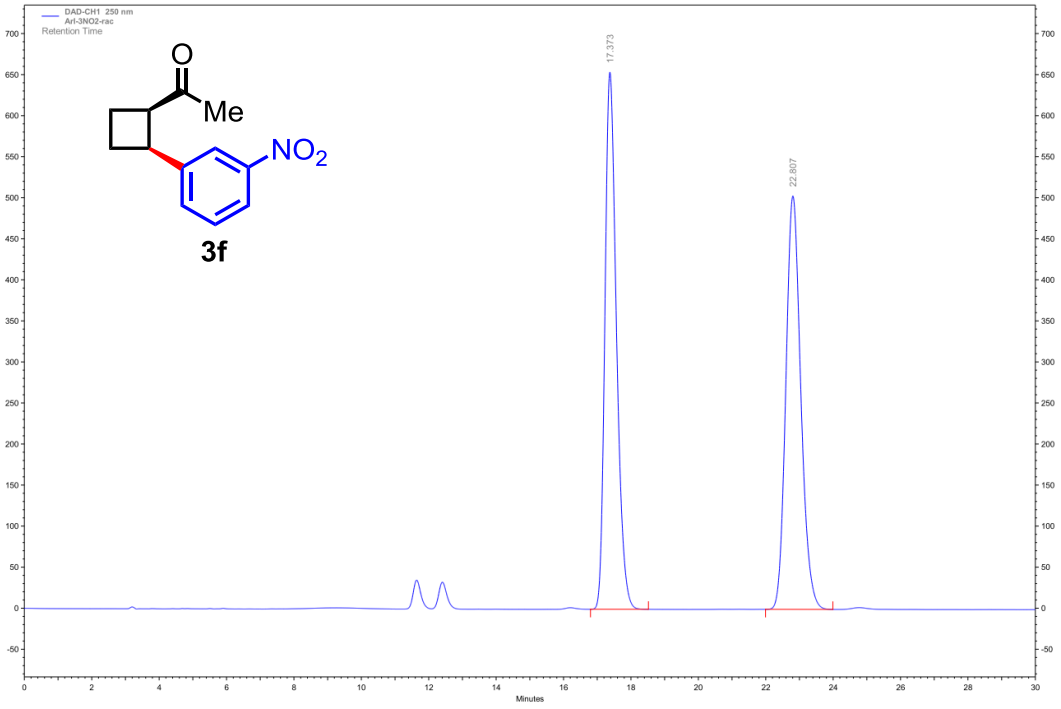
DAD-CH1 206 nm Results

Retention Time	Area	Area %
11.773	93640986	49.485
17.640	95591751	50.515



DAD-CH1 206 nm Results

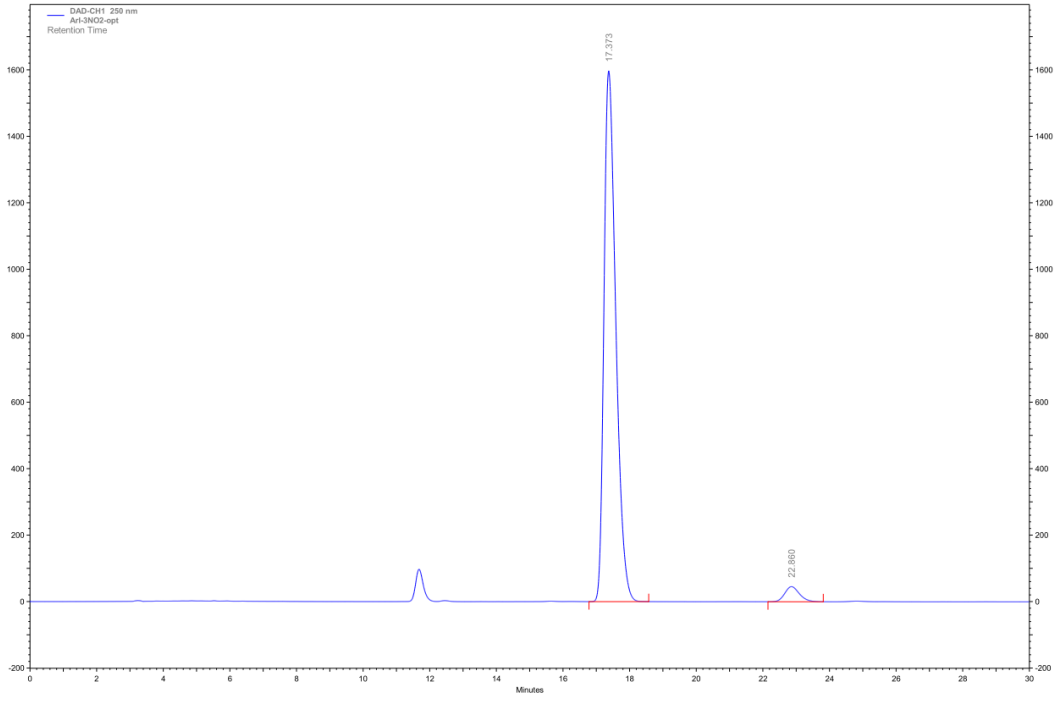
Retention Time	Area	Area %
11.867	50713826	94.253
17.773	3092135	5.747



3f

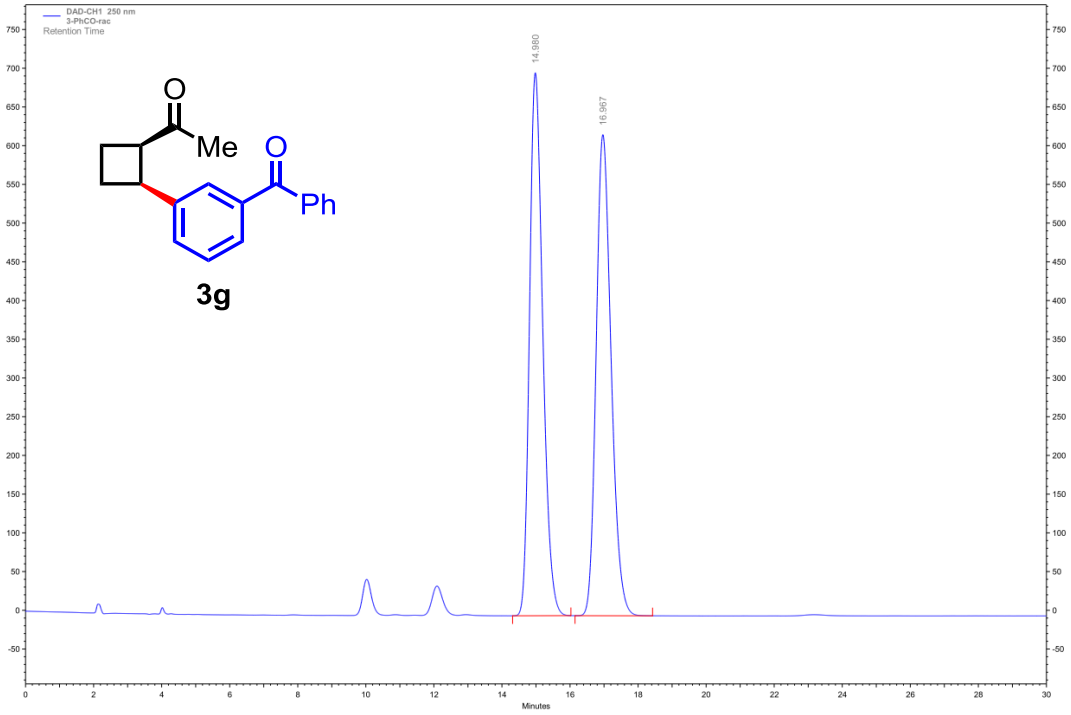
DAD-CH1 250 nm Results

Retention Time	Area	Area %
17.373	61998948	50.00
22.807	62002470	50.00



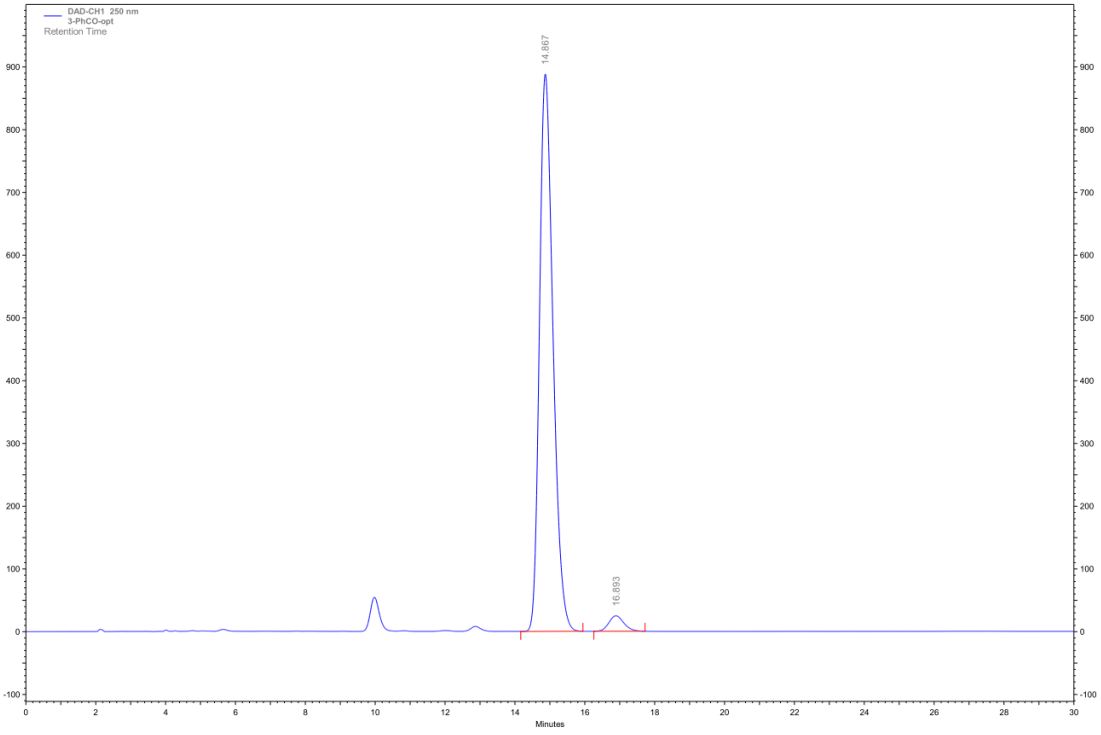
DAD-CH1 250 nm Results

Retention Time	Area	Area %
17.373	157702621	96.58
22.860	5582082	3.42



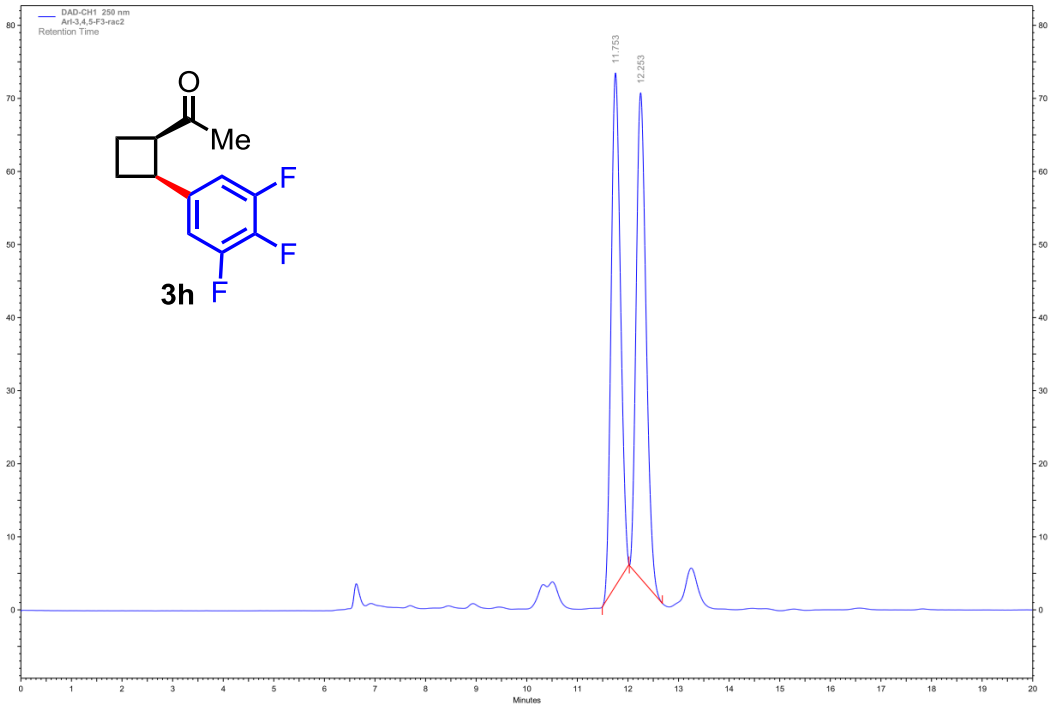
DAD-CH1 250 nm Results

Retention Time	Area	Area %
14.980	75679842	49.97
16.967	75783940	50.03



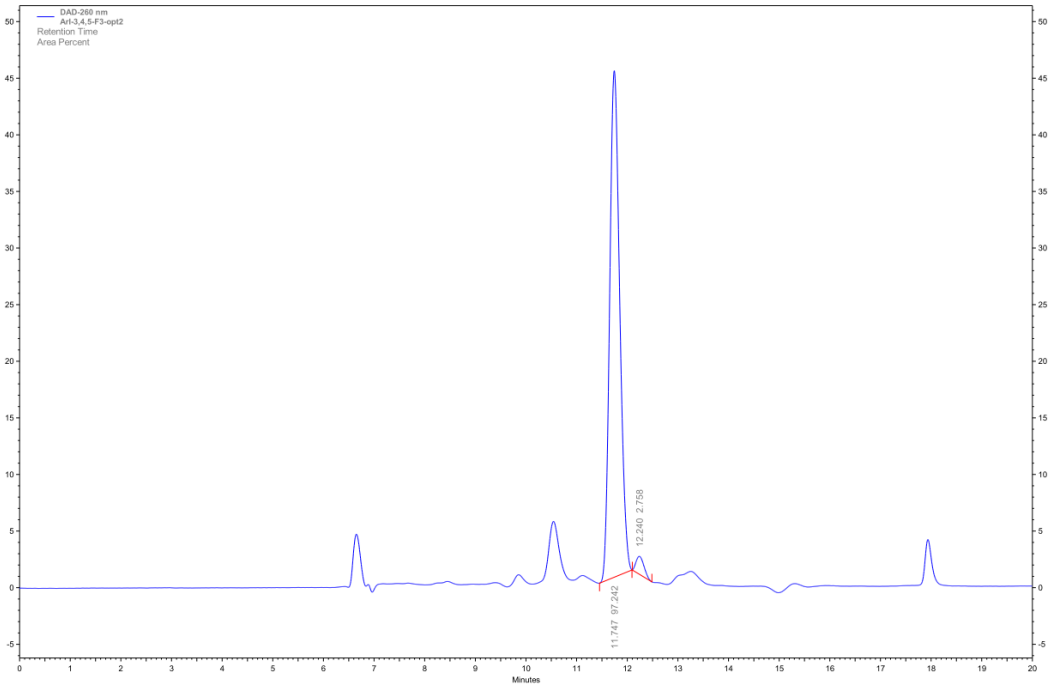
DAD-CH1 250 nm Results

Retention Time	Area	Area %
14.867	95884973	97.02
16.893	2942622	2.98



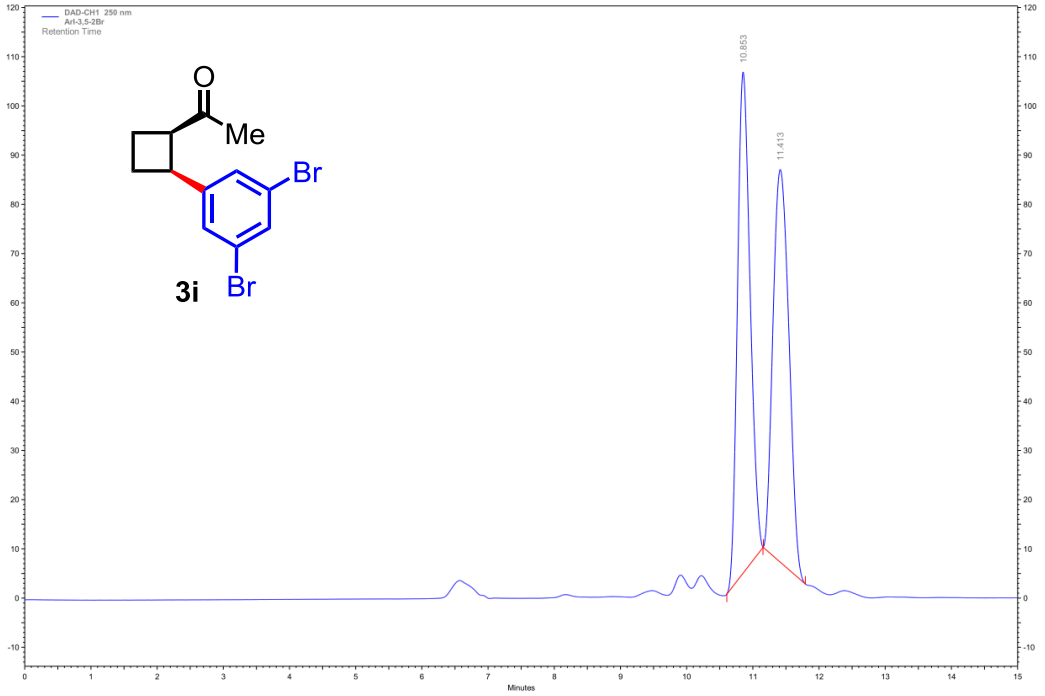
DAD-CH1 250 nm Results

Retention Time	Area	Area %
11.753	3620649	49.91
12.253	3633540	50.09



DAD-CH1 250 nm Results

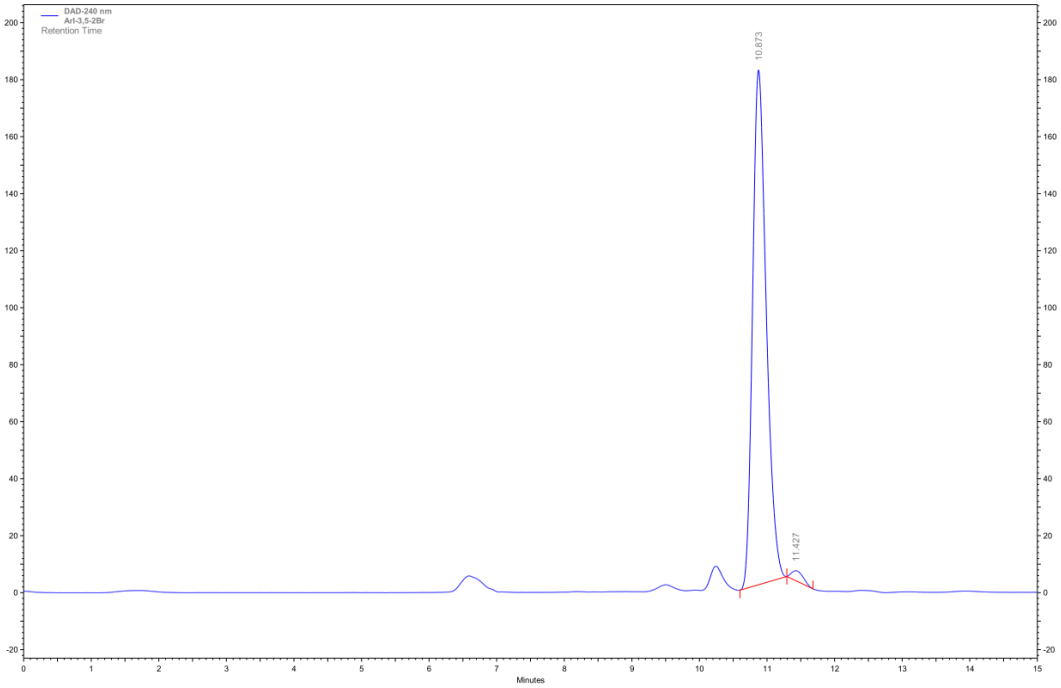
Retention Time	Area	Area %
11.747	1904981	97.25
12.240	53841	2.75



3i

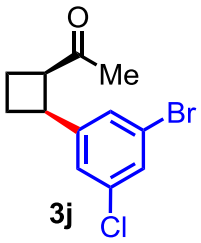
DAD-CH1 250 nm Results

Retention Time	Area	Area %
10.853	5399598	50.24
11.413	5348988	49.76

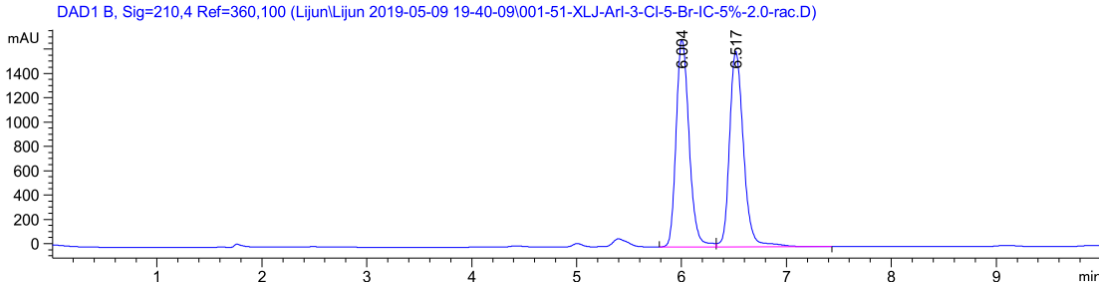


DAD-CH1 250 nm Results

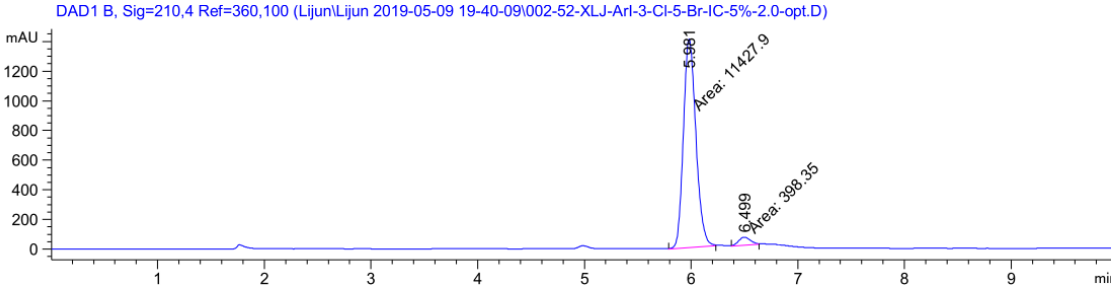
Retention Time	Area	Area %
10.873	2149725	98.66
11.407	29260	1.34



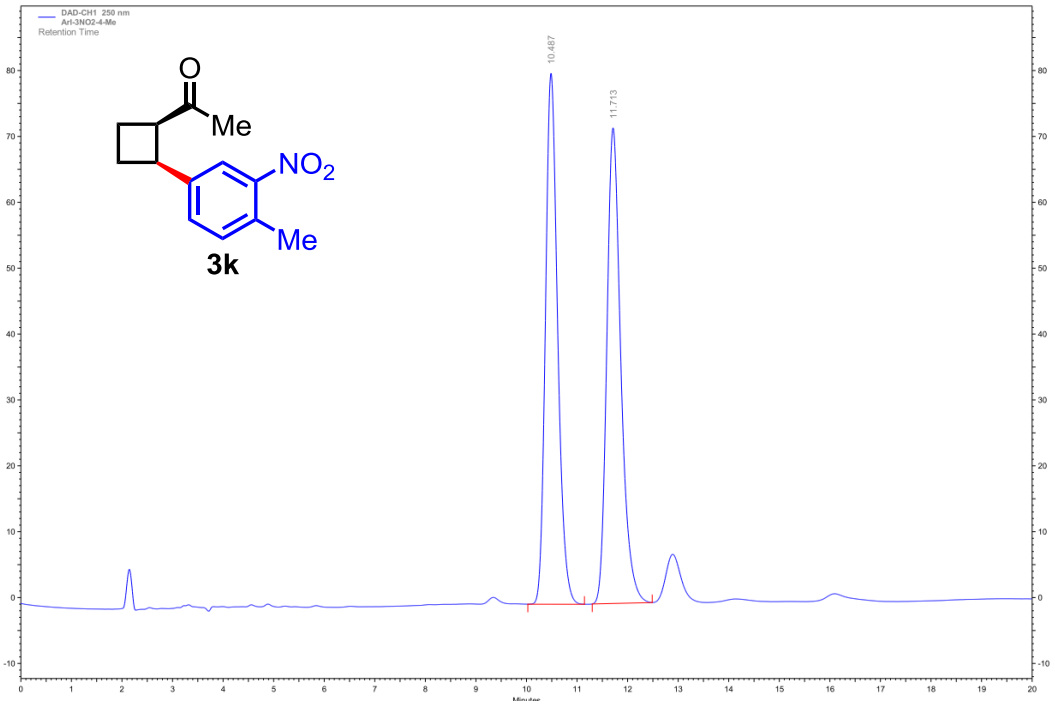
3j



Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	6.004	BV	0.1324	1.46543e4	1703.08911	49.6126
2	6.517	VB	0.1440	1.48831e4	1608.19824	50.3874

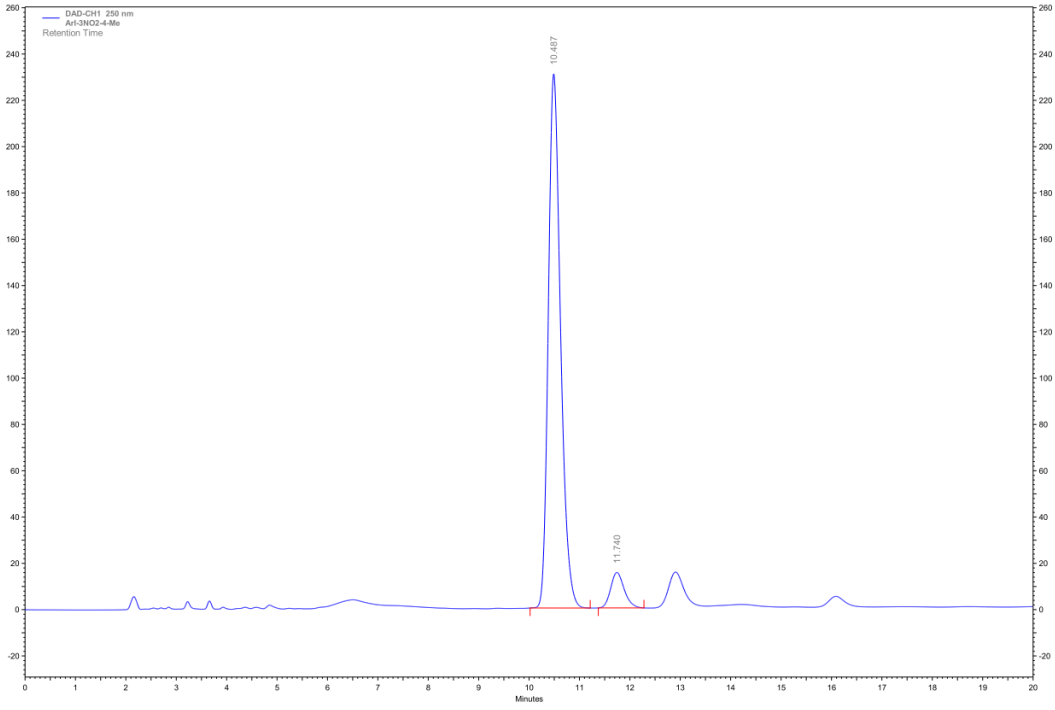


Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	5.981	MM	0.1354	1.14279e4	1406.34131	96.6316
2	6.499	MM	0.1244	398.35001	53.38059	3.3684



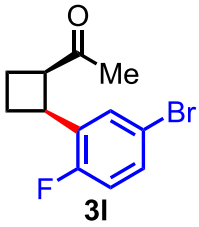
DAD-CH1 250 nm Results

Retention Time	Area	Area %
10.487	5376623	49.08
11.713	5578556	50.92

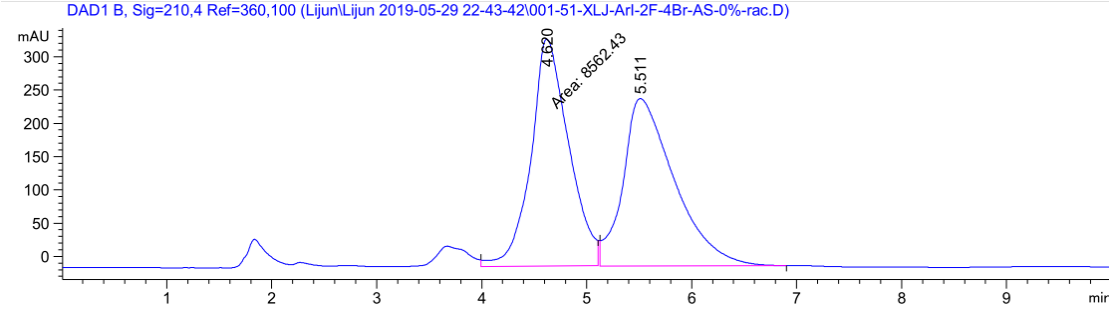


DAD-CH1 250 nm Results

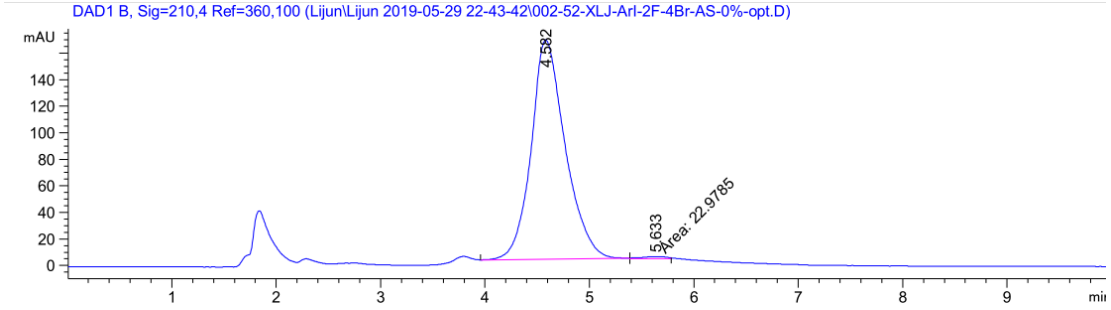
Retention Time	Area	Area %
10.487	15558558	93.14
11.740	1145510	6.86



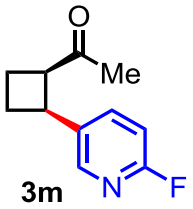
3l



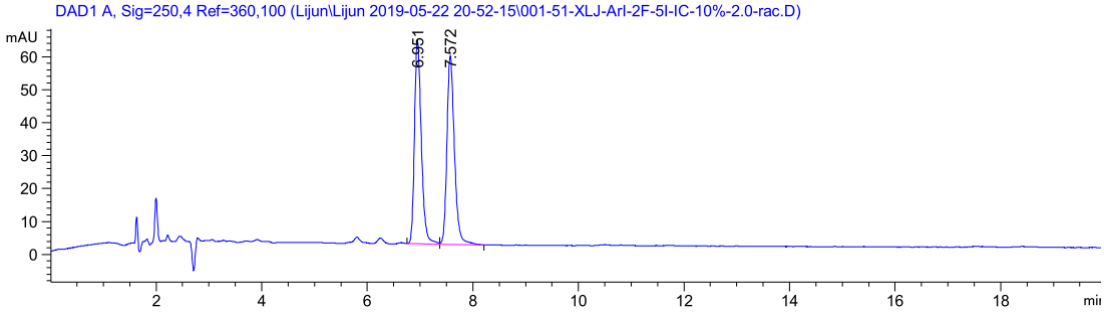
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	4.620	MM	0.4188	8562.43164	340.76370	49.8963
2	5.511	VB	0.5001	8598.02441	251.95415	50.1037



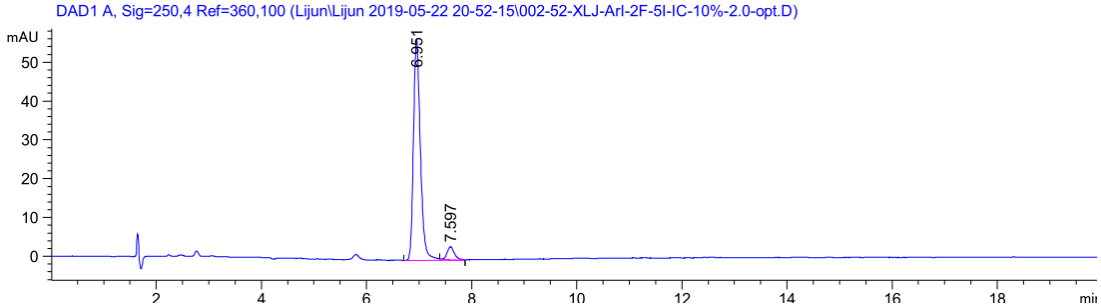
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	4.582	BB	0.3341	3737.61841	165.11324	99.3890
2	5.633	MM	0.2765	22.97846	1.38517	0.6110



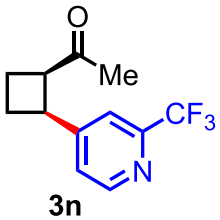
3m



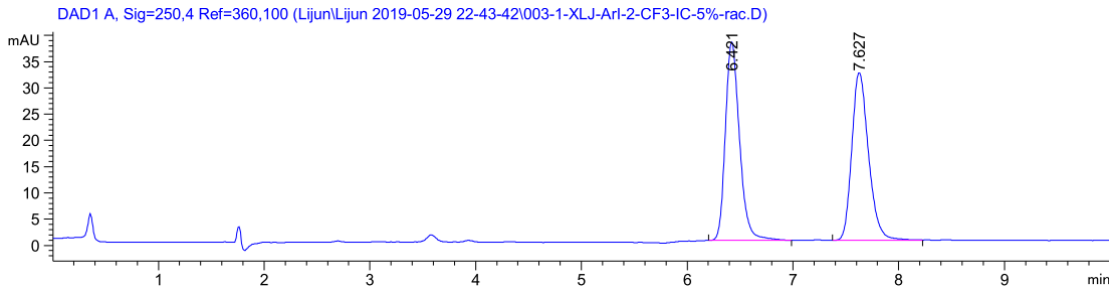
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	6.951	BV	0.1369	555.70782	61.81274	49.6309
2	7.572	VB	0.1509	563.97229	57.28272	50.3691



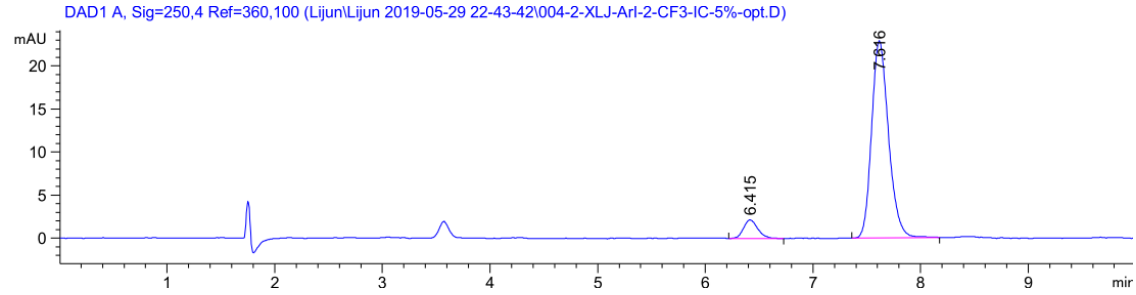
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	6.951	BV R	0.1387	519.40497	56.81502	94.1818
2	7.597	VB E	0.1449	32.08715	3.37446	5.8182



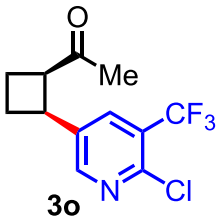
3n



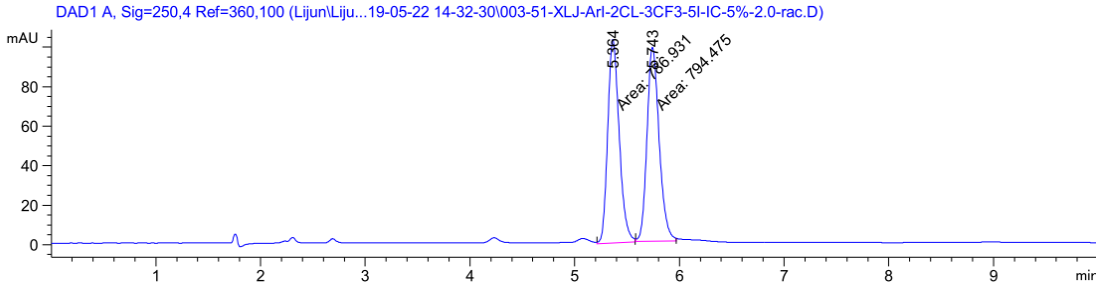
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	6.421	BB	0.1425	351.27402	37.78881	50.0834
2	7.627	BB	0.1699	350.10461	31.94782	49.9166



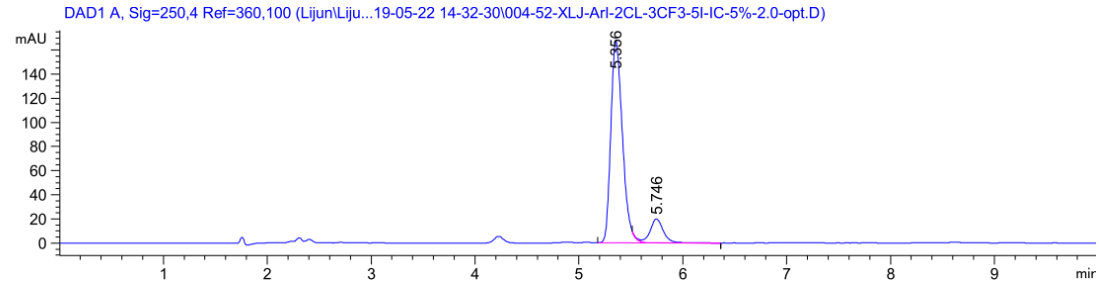
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	6.415	BB	0.1434	20.24956	2.15862	7.4900
2	7.616	BB	0.1693	250.10361	22.91296	92.5100



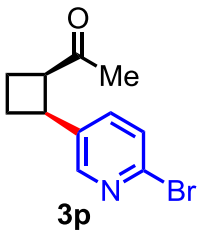
3o



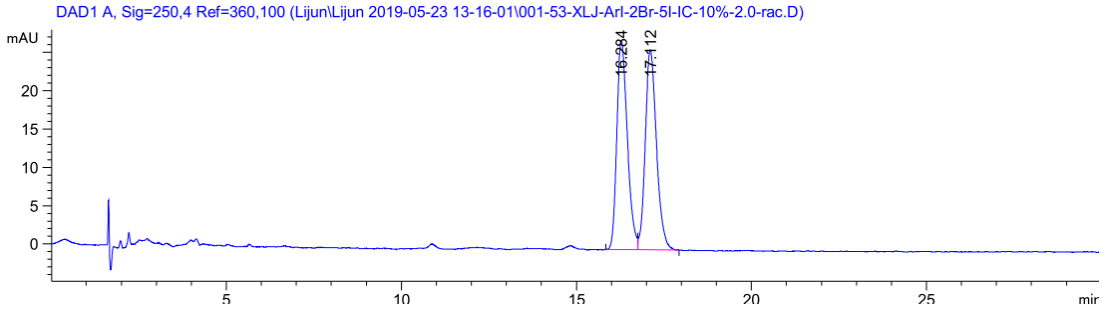
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	5.364	MM	0.1271	786.93103	103.21477	49.7615
2	5.743	MM	0.1347	794.47510	98.31642	50.2385



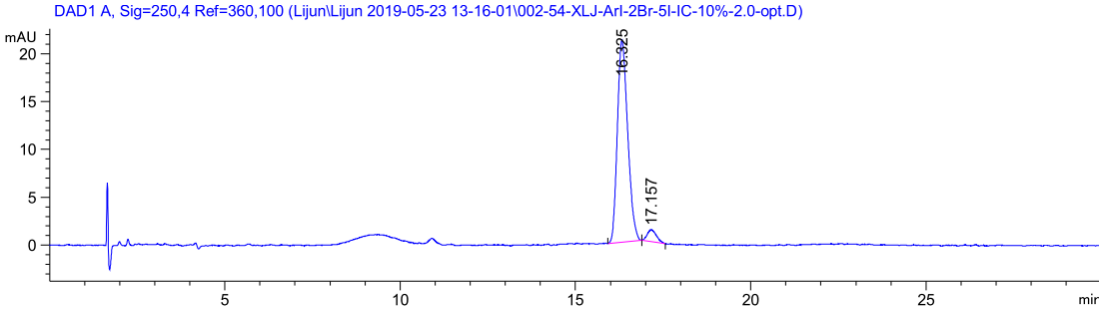
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	5.356	BV R	0.1188	1305.69495	167.75298	87.9672
2	5.746	VB E	0.1354	178.60246	19.77604	12.0328



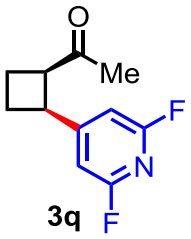
3p



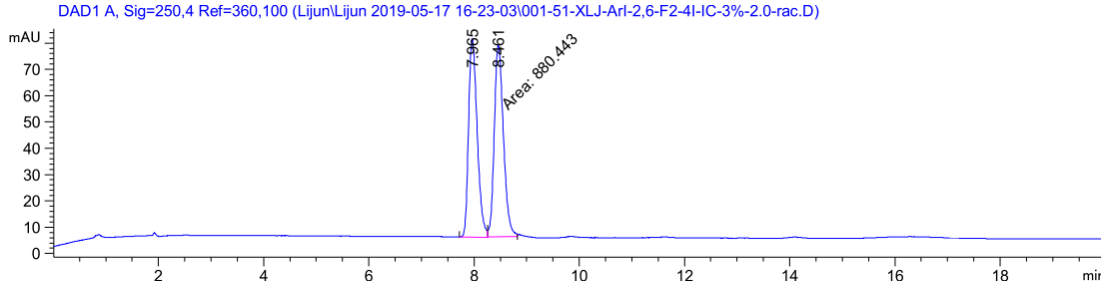
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	16.284	BV	0.3228	571.06885	27.21883	49.5780
2	17.112	VB	0.3420	580.79102	26.06443	50.4220



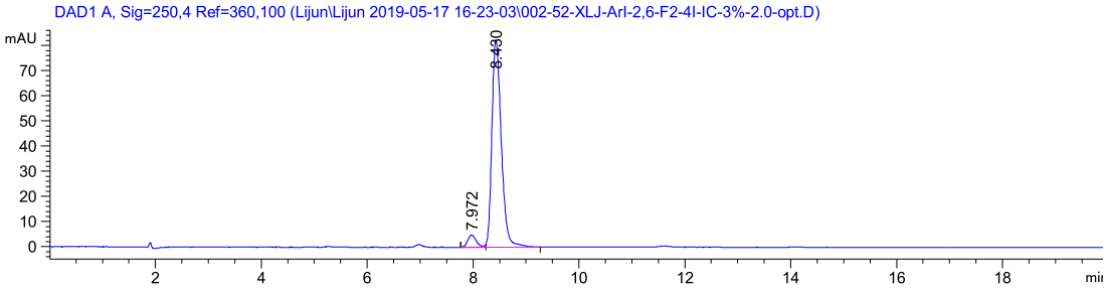
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	16.325	BB	0.3152	433.45526	21.13733	95.0080
2	17.157	BB	0.2307	22.77508	1.26098	4.9920



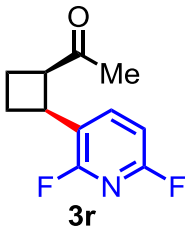
3q



Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	7.965	BV	0.1767	856.81567	75.34479	49.3200
2	8.461	MM	0.2017	880.44281	72.76616	50.6800

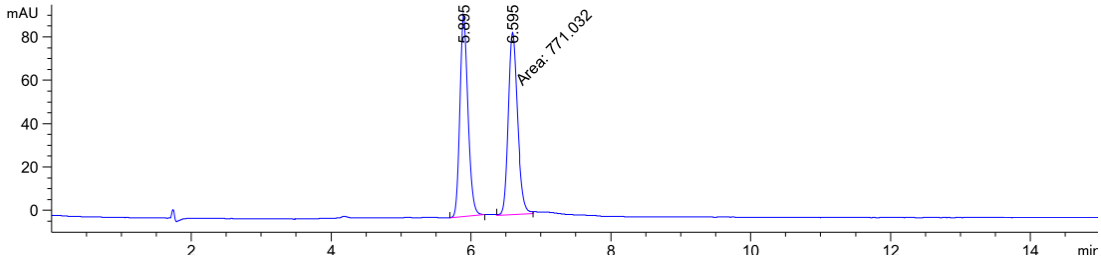


Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	7.972	BV E	0.1614	53.46638	4.89643	4.9942
2	8.430	VB R	0.1900	1017.10260	82.40462	95.0058



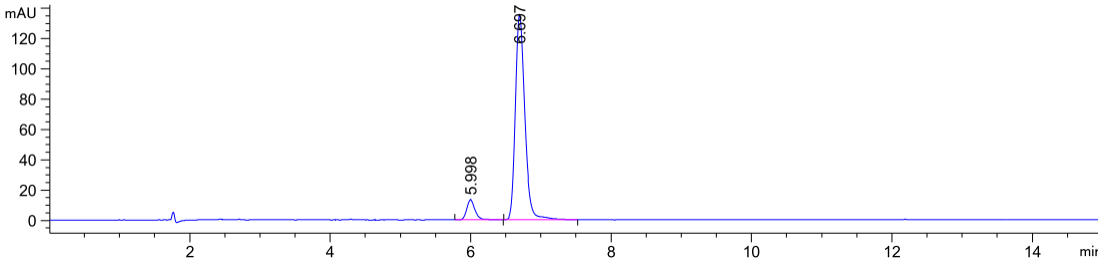
3r

DAD1 A, Sig=250,4 Ref=360,100 (Lijun\Lijun 2019-05-21 12-02-18\001-51-XLJ-ArI-26-2F-3I-IC-5%-2.0-rac.D)

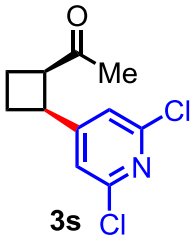


Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	5.895	BB	0.1255	761.34131	93.00169	49.6838
2	6.595	MM	0.1530	771.03217	83.96684	50.3162

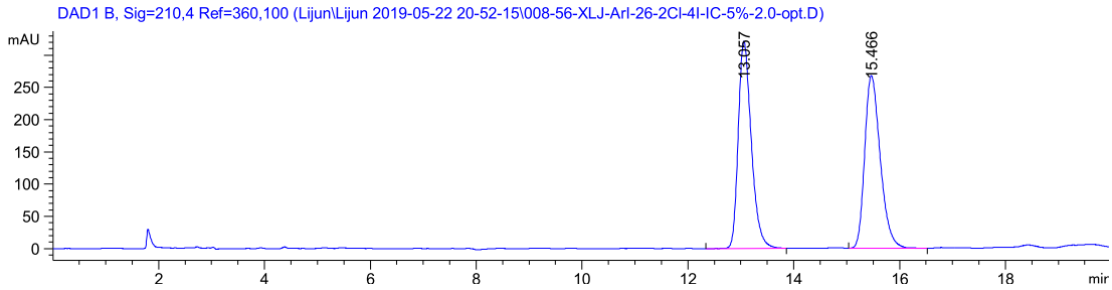
DAD1 A, Sig=250,4 Ref=360,100 (Lijun\Lijun 2019-05-23 13-16-01\004-57-XLJ-ArI-2F-3I-IC-5%-2.0-opt-2.D)



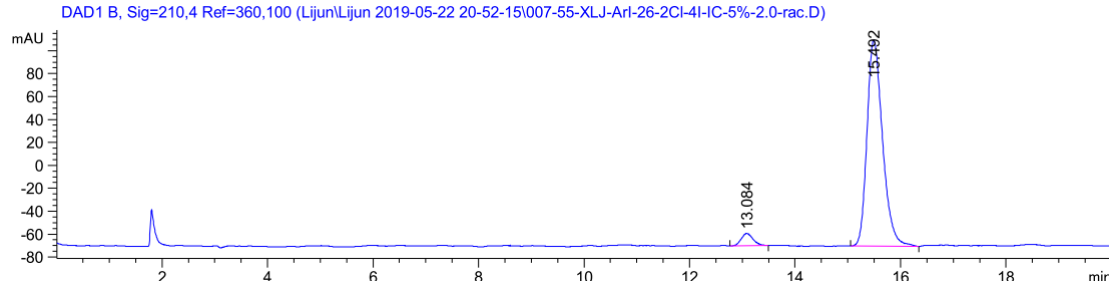
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	5.998	BB	0.1259	110.21342	13.40715	8.1106
2	6.697	BB	0.1418	1248.66992	135.10104	91.8894



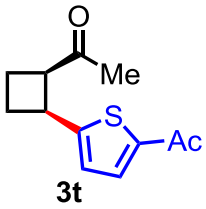
3s



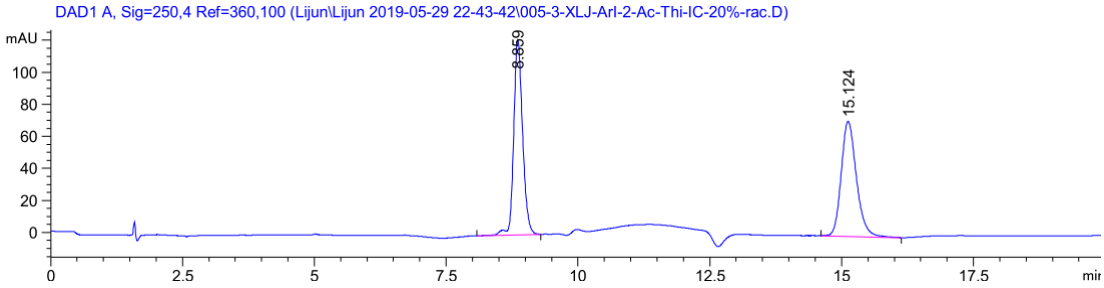
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	13.057	BB	0.2643	5522.30176	321.66049	50.2500
2	15.466	BB	0.3140	5467.35254	268.04251	49.7500



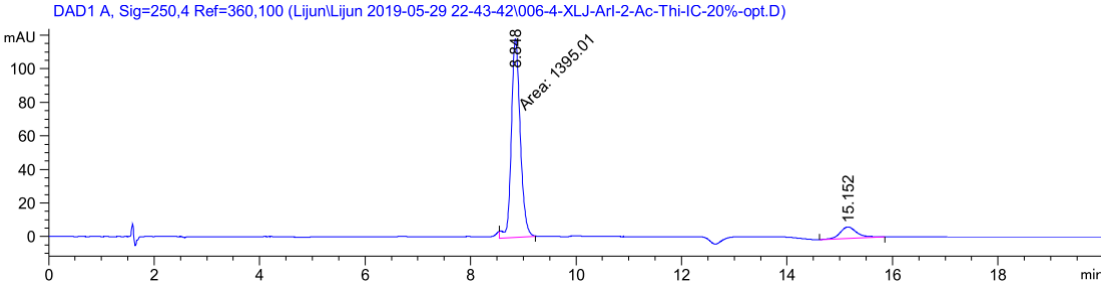
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	13.084	BB	0.2406	168.77231	10.78534	4.3950
2	15.492	BB	0.3124	3671.28857	179.63487	95.6050



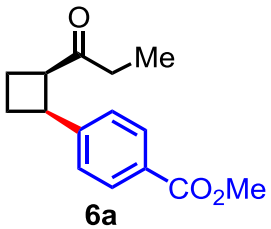
3t



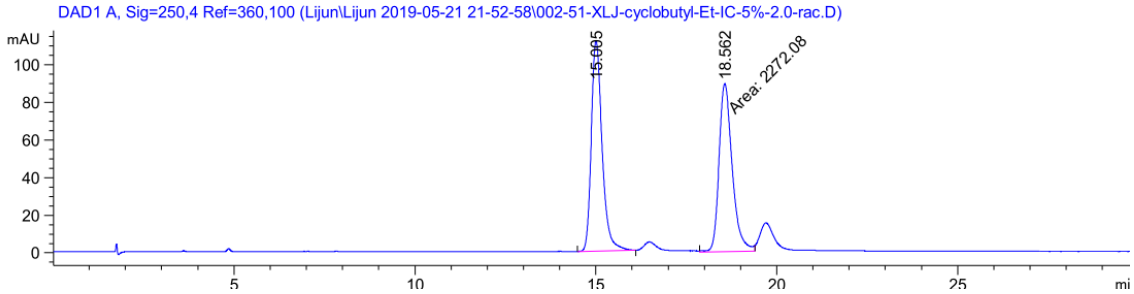
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	8.859	VB R	0.1762	1430.59680	121.73954	49.9282
2	15.124	BB	0.3074	1434.70874	71.71735	50.0718



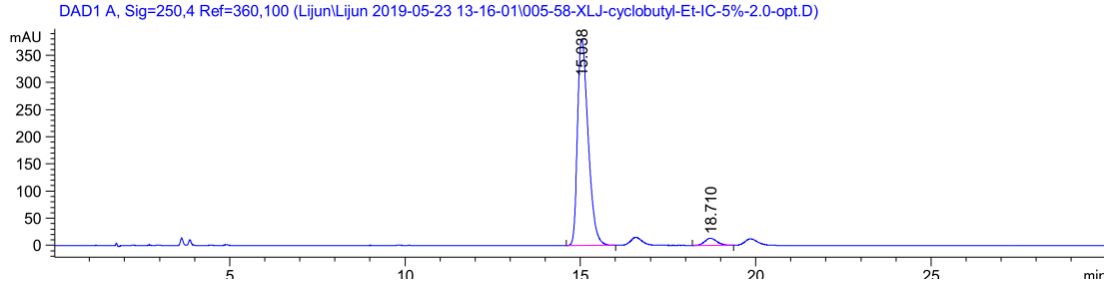
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	8.848	MM	0.1961	1395.00525	118.57172	90.3133
2	15.152	BB	0.3316	149.62393	6.83013	9.6867



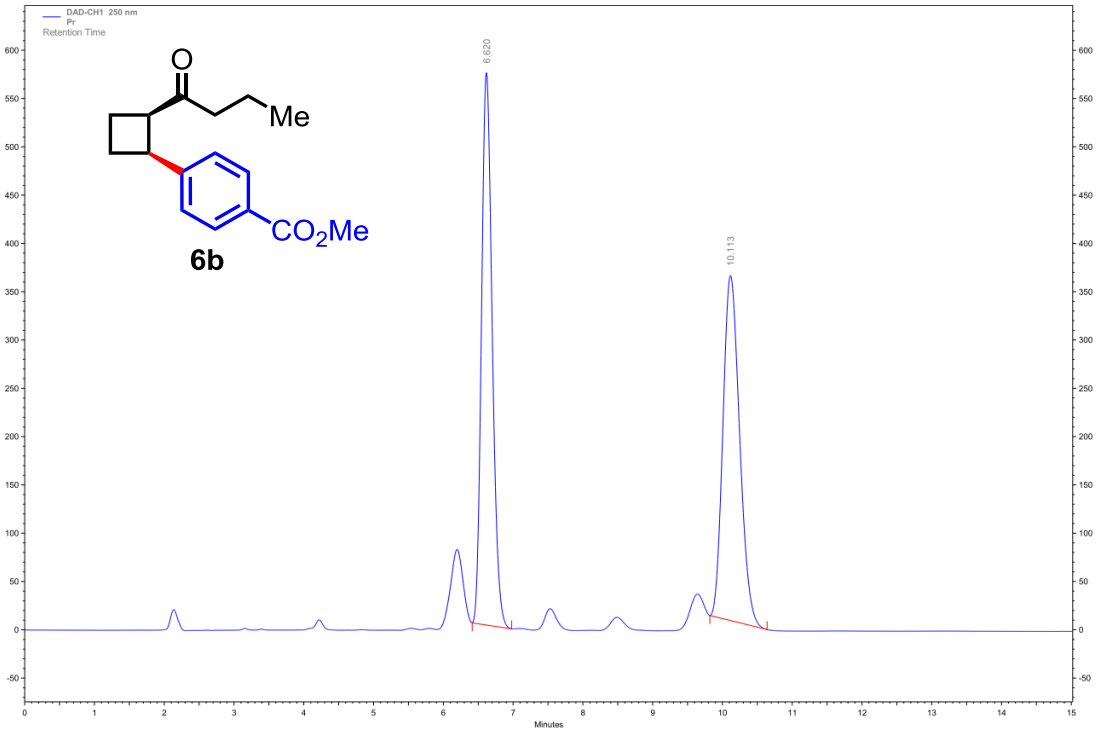
6a



Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	15.005	BB	0.3163	2320.00513	111.69574	50.5218
2	18.562	MM	0.4238	2272.08032	89.34408	49.4782

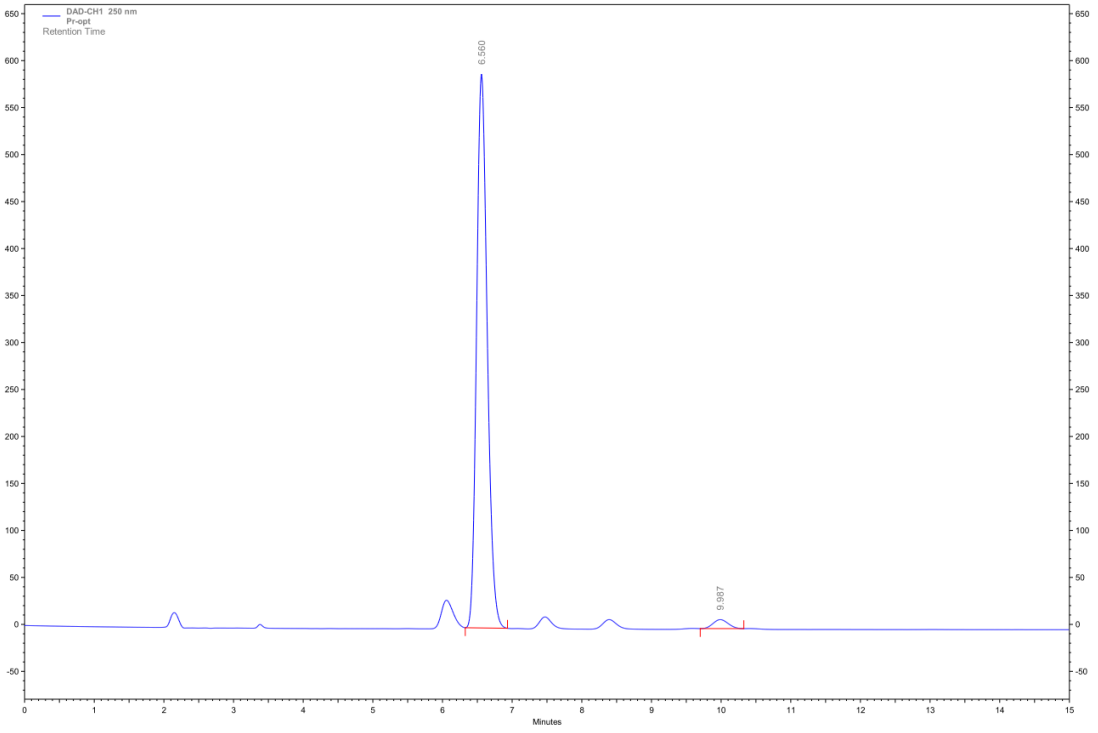


Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	15.038	BB	0.3276	8004.55029	380.26929	96.2042
2	18.710	BB	0.3647	315.82397	13.12700	3.7958



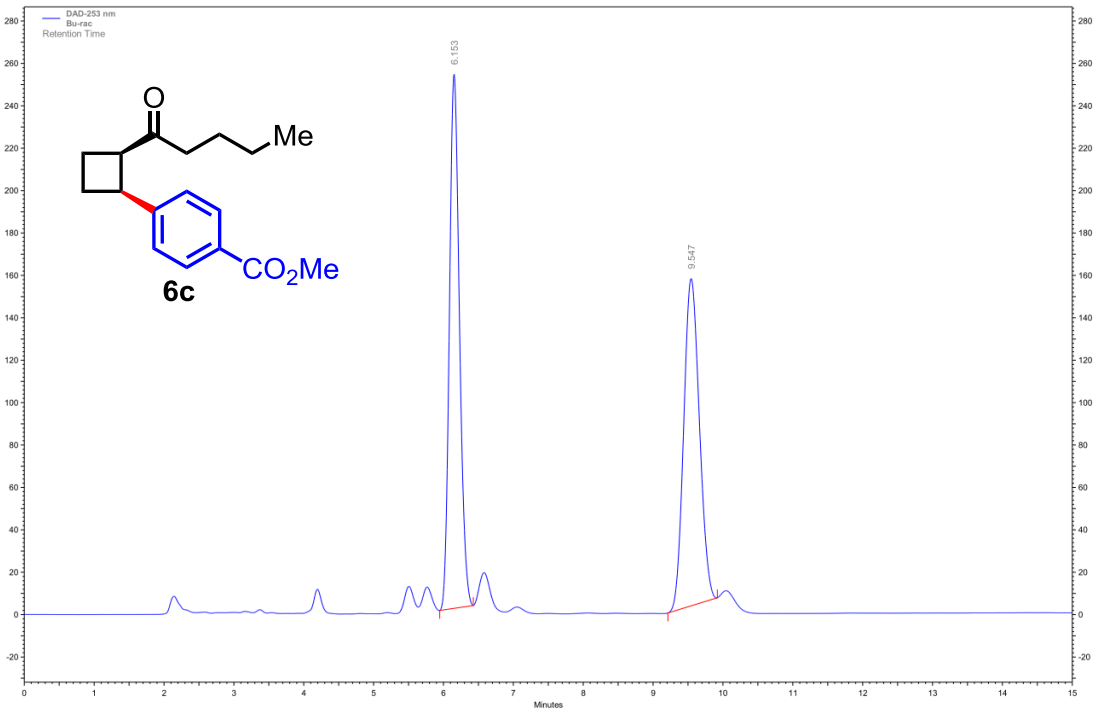
DAD-CH1 250 nm Results

Retention Time	Area	Area %
6.620	24299965	50.65
10.113	23675748	49.35



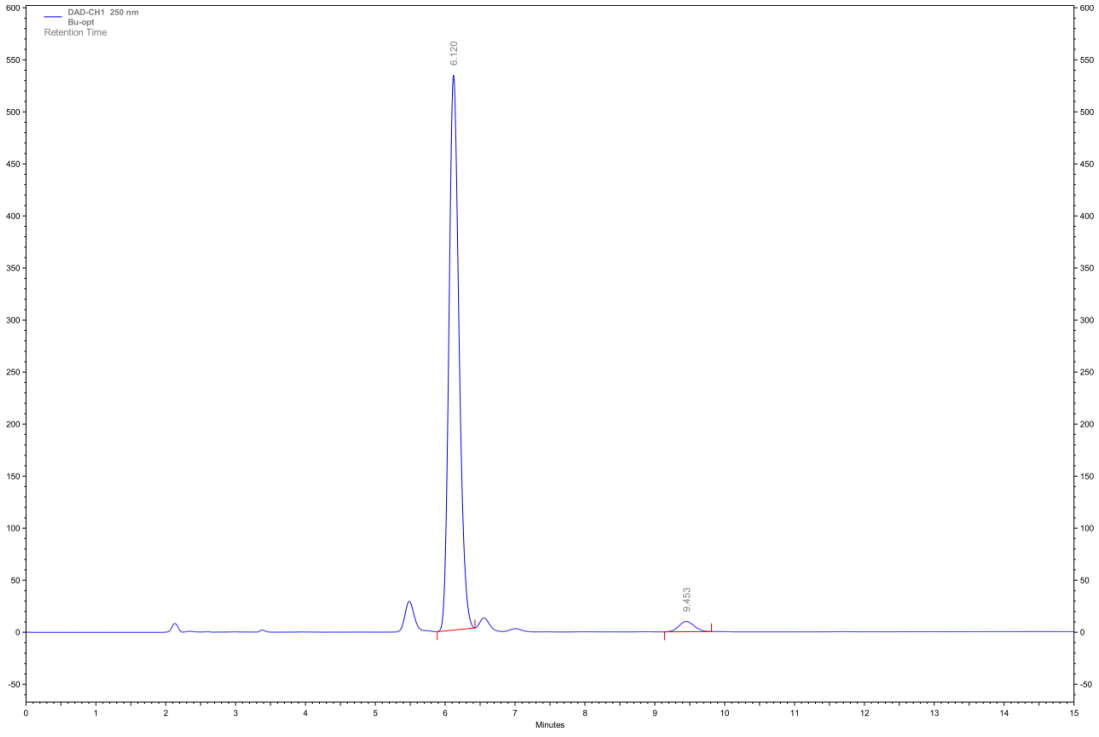
DAD-CH1 250 nm Results

Retention Time	Area	Area %
6.560	25009717	97.75
9.987	574833	2.25



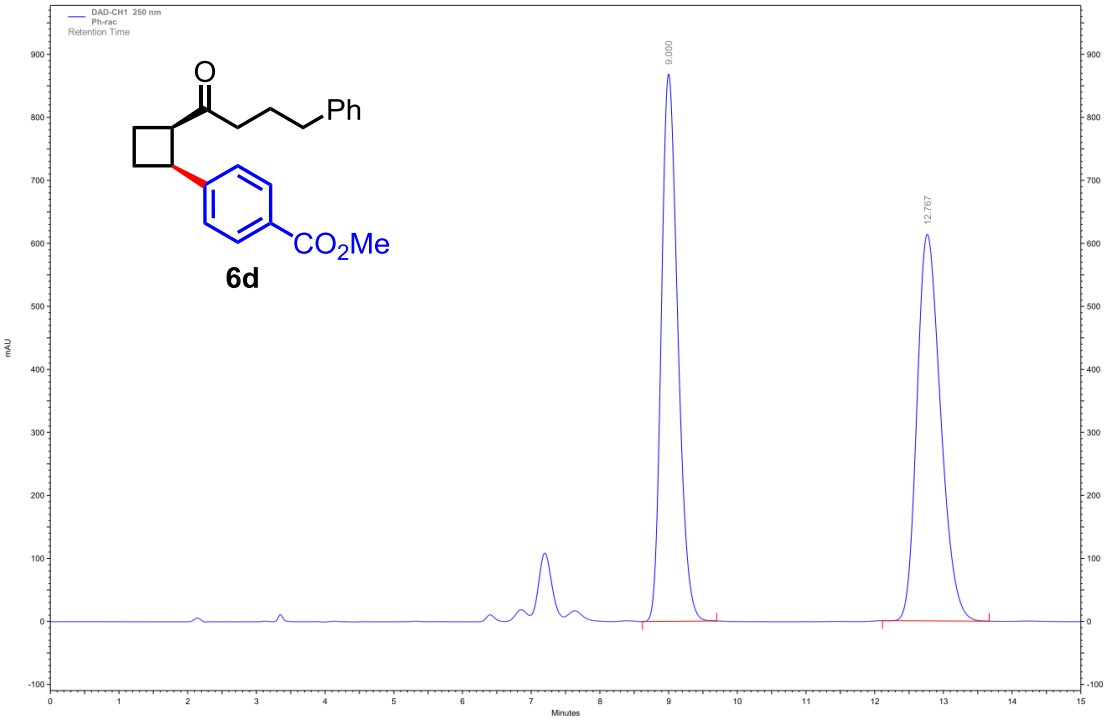
DAD-CH1 250 nm Results

Retention Time	Area	Area %
6.153	14130801	51.24
9.547	13449332	48.76



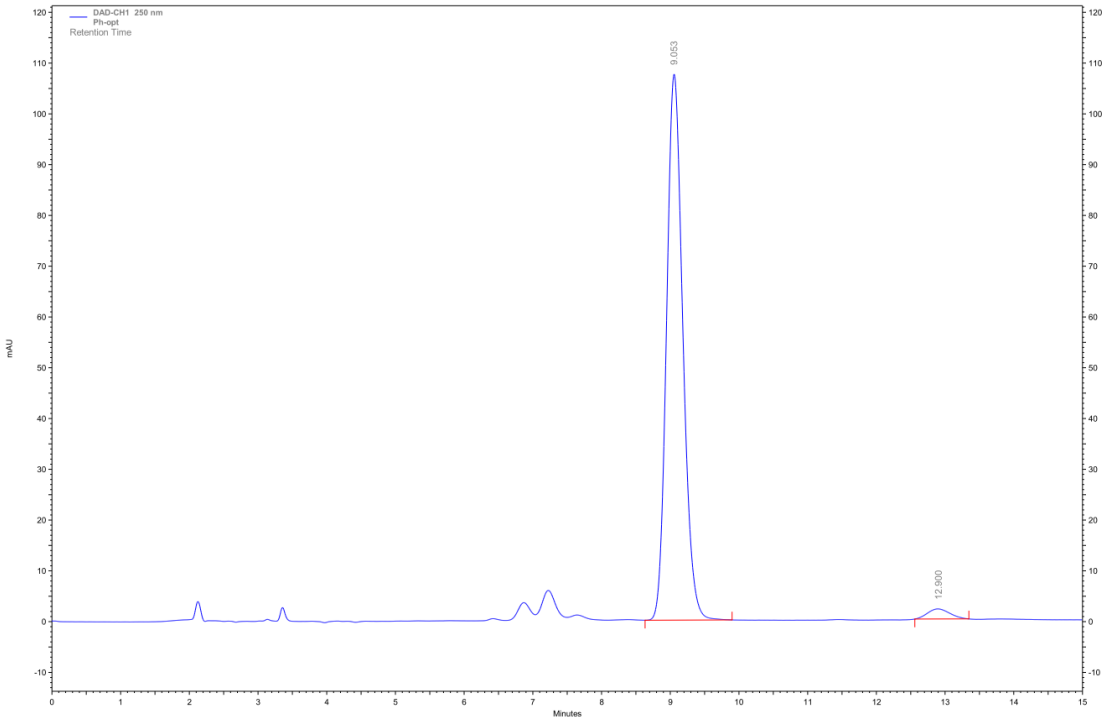
DAD-CH1 250 nm Results

Retention Time	Area	Area %
6.120	21319786	97.22
9.453	608622	2.78



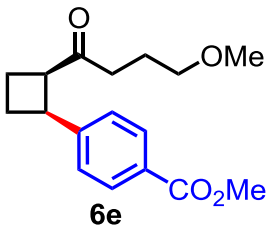
DAD-CH1 250 nm Results

Retention Time	Area	Area %
9.000	57906573	49.64
12.767	58756878	50.36

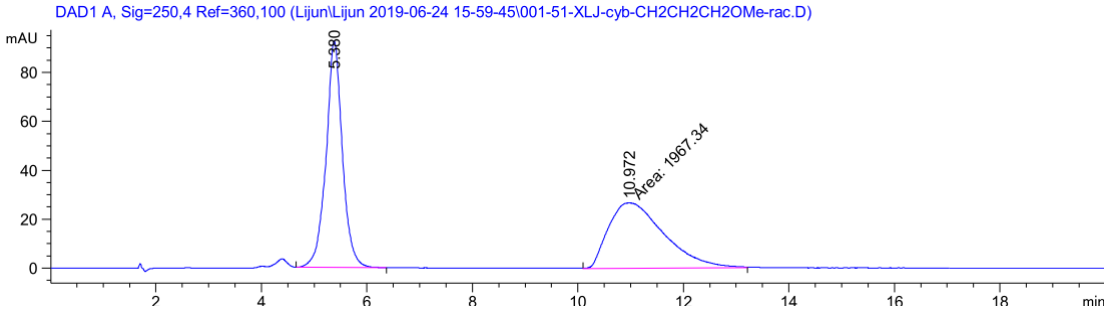


DAD-CH1 250 nm Results

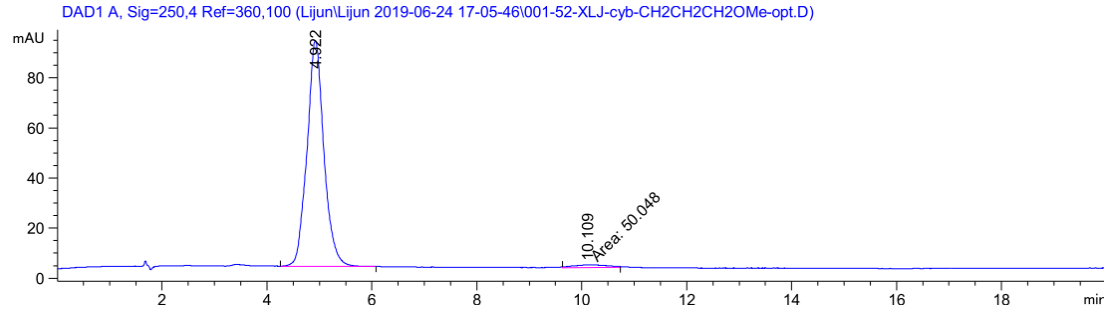
Retention Time	Area	Area %
9.053	7057445	97.54
12.900	177754	2.46



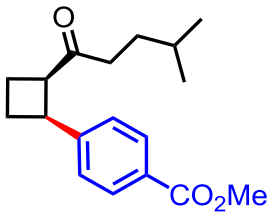
6e



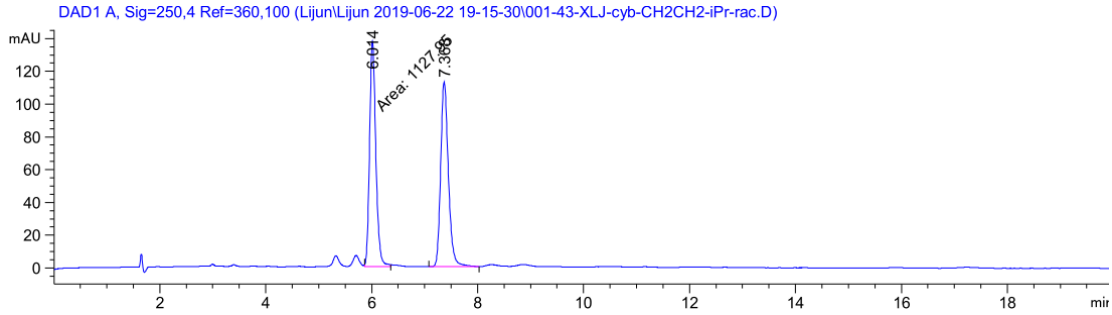
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	5.380	BB	0.3162	1999.42310	92.50969	50.4045
2	10.972	MM	1.2243	1967.33508	26.78123	49.5955



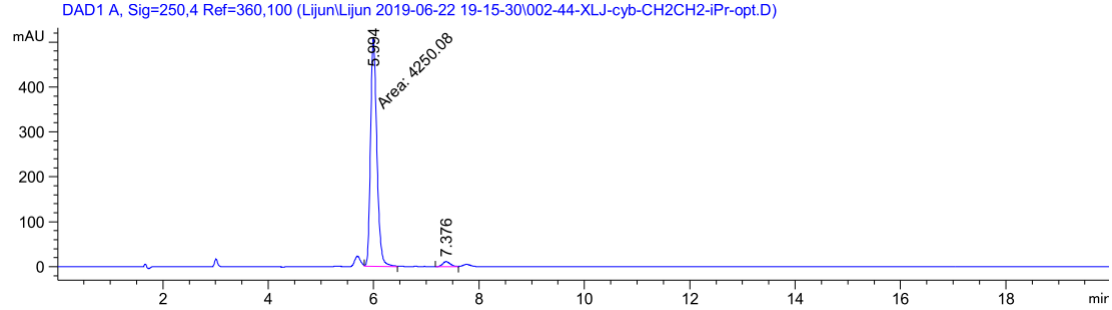
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	4.922	BB	0.3302	2022.87451	89.99692	97.5856
2	10.109	MM	0.7764	50.04798	1.07438	2.4144



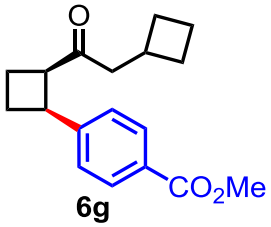
6f



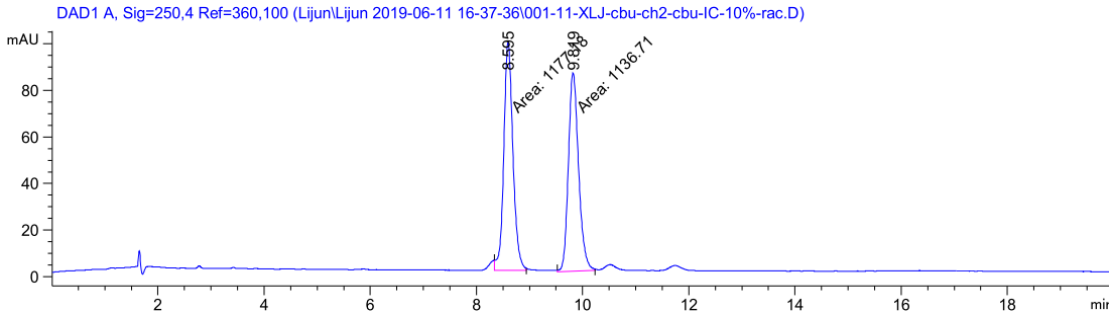
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	6.014	MM	0.1367	1127.95349	137.56294	49.7130
2	7.368	BB	0.1539	1140.97644	112.89320	50.2870



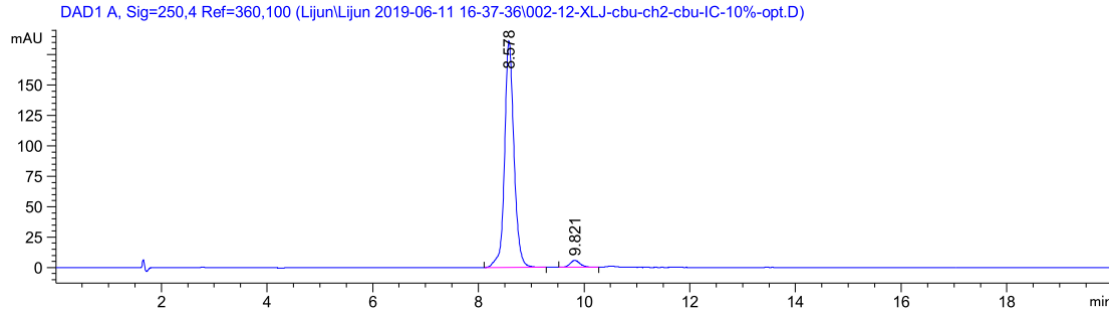
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	5.994	MM	0.1398	4250.07520	506.61185	97.3902
2	7.376	BV	0.1529	113.89037	11.36409	2.6098



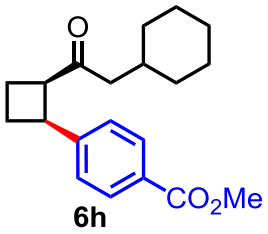
6g



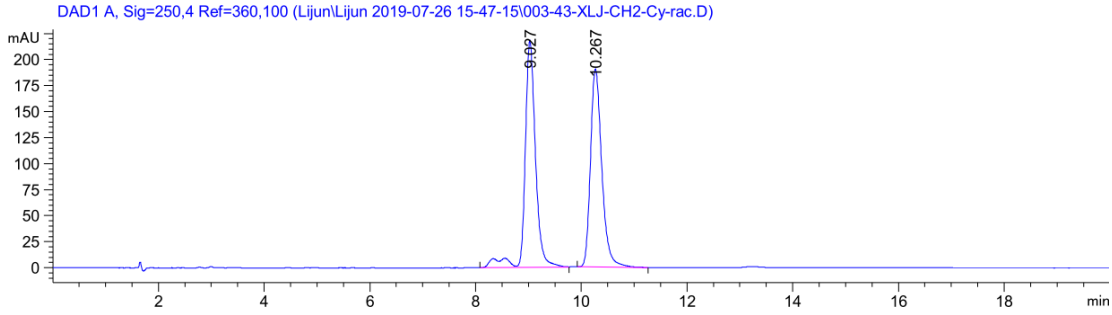
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	8.595	MM	0.2001	1177.17749	98.03753	50.8745
2	9.819	MM	0.2226	1136.70911	85.09312	49.1255



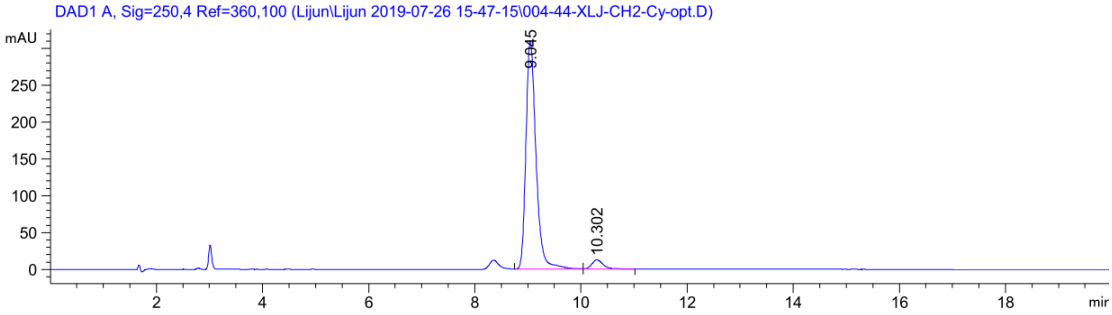
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	8.578	BB	0.1882	2301.31250	186.14893	96.8306
2	9.821	BB	0.2059	75.32481	5.71003	3.1694



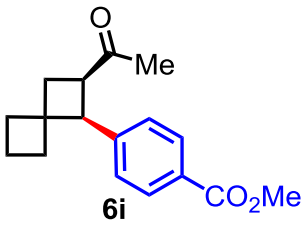
6h



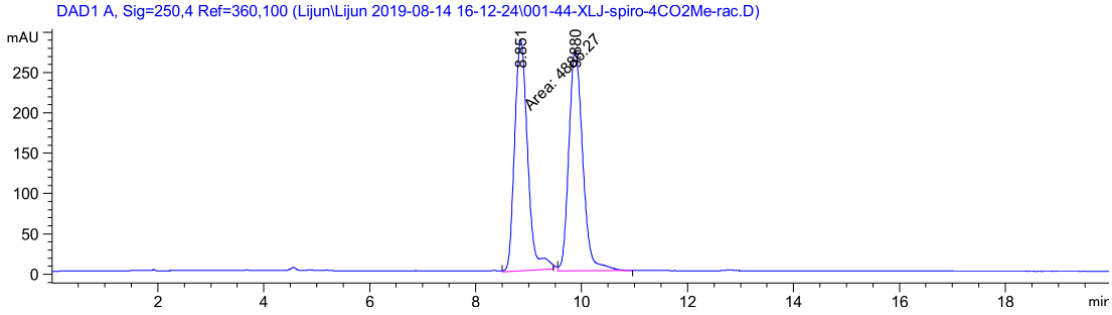
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	9.027	VB R	0.1975	3035.80908	217.80962	51.8482
2	10.267	BB	0.2269	2819.38086	190.23552	48.1518



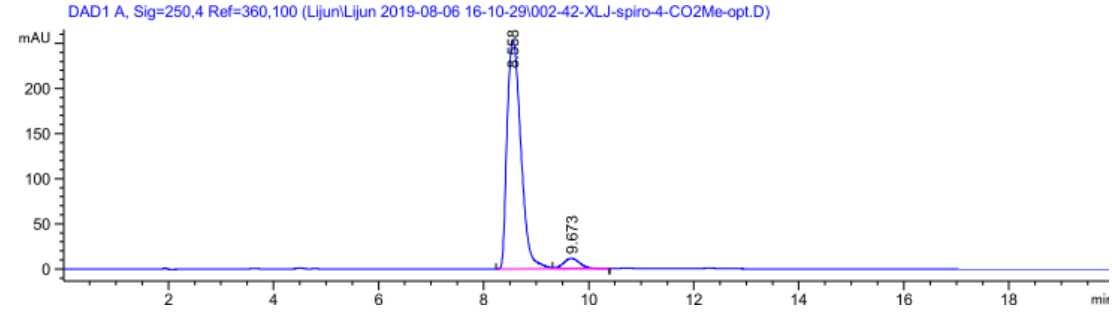
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	9.045	VV	0.2020	4097.41992	310.36560	95.6101
2	10.302	VB	0.2260	188.13153	12.61137	4.3899



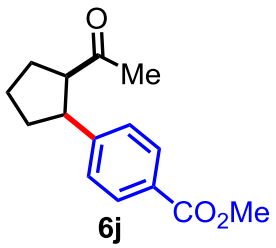
6i



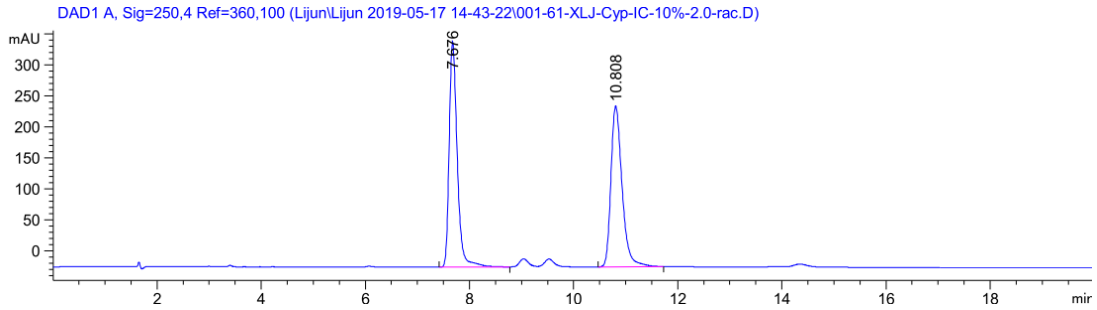
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	8.851	MM	0.2850	4886.26807	285.75439	49.5690
2	9.880	VB	0.2845	4971.24268	272.92963	50.4310



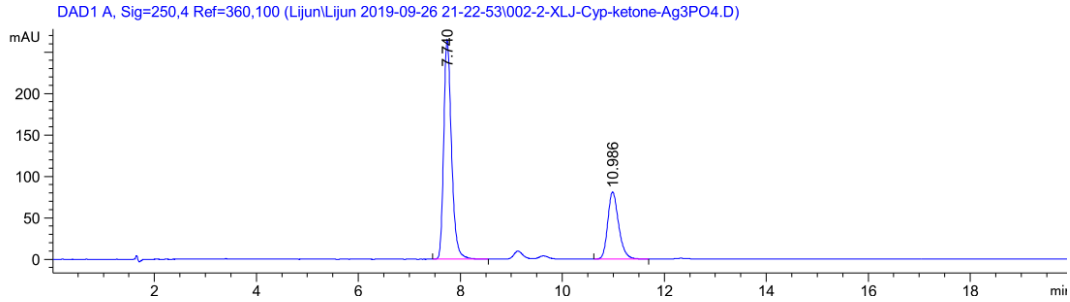
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	8.558	BV R	0.2930	4619.13281	253.03453	94.9575
2	9.673	VB E	0.3352	245.28772	11.48527	5.0425



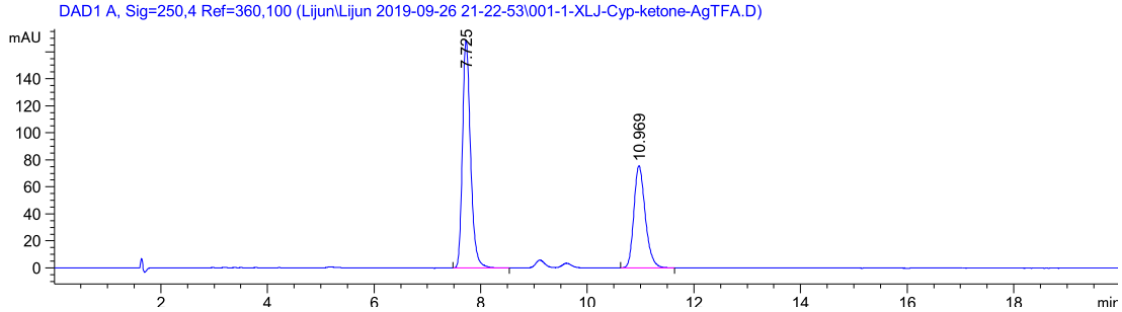
6j



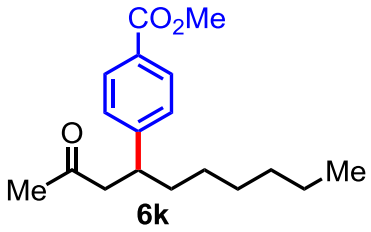
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	7.676	BB	0.1631	3898.86230	363.62344	50.2546
2	10.808	BB	0.2277	3859.35083	259.26367	49.7454



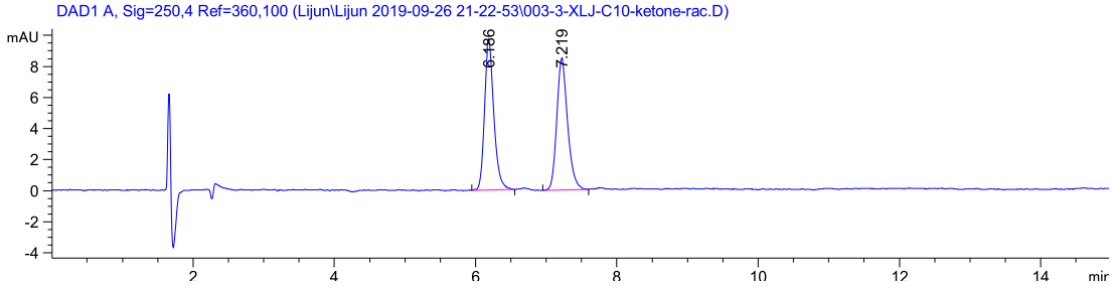
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	7.740	BB	0.1618	2809.53735	264.68832	70.0812
2	10.986	BB	0.2269	1199.43445	80.91444	29.9188



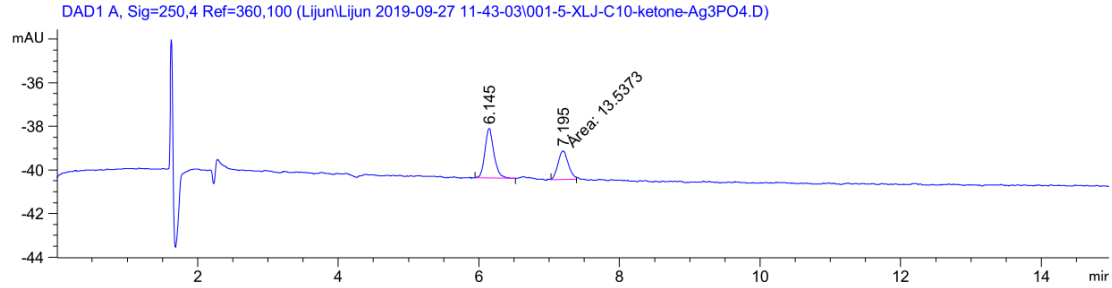
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	7.725	BB	0.1611	1779.71228	168.65359	61.3412
2	10.969	BB	0.2270	1121.62170	75.65052	38.6588



6k

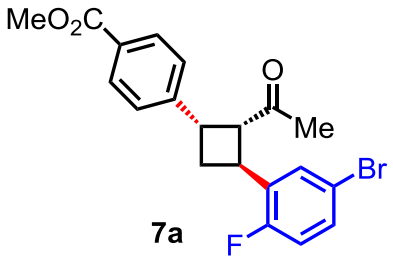


Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	6.186	BB	0.1417	89.70099	9.72083	49.6040
2	7.219	BB	0.1630	91.13331	8.50573	50.3960

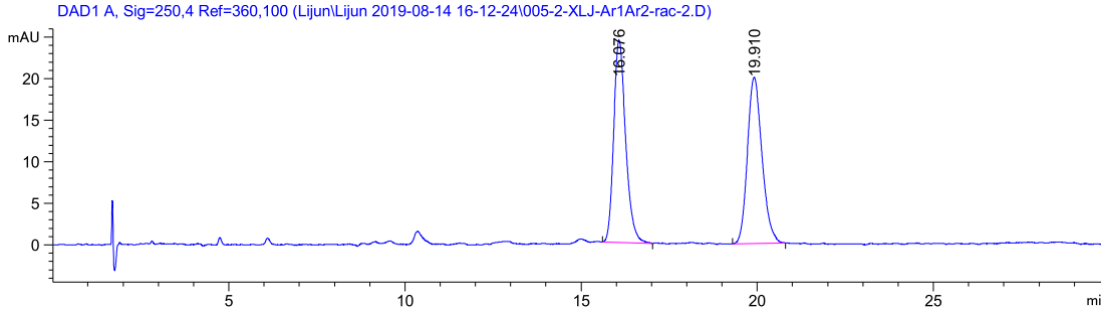


Signal 1: DAD1 A, Sig=250,4 Ref=360,100

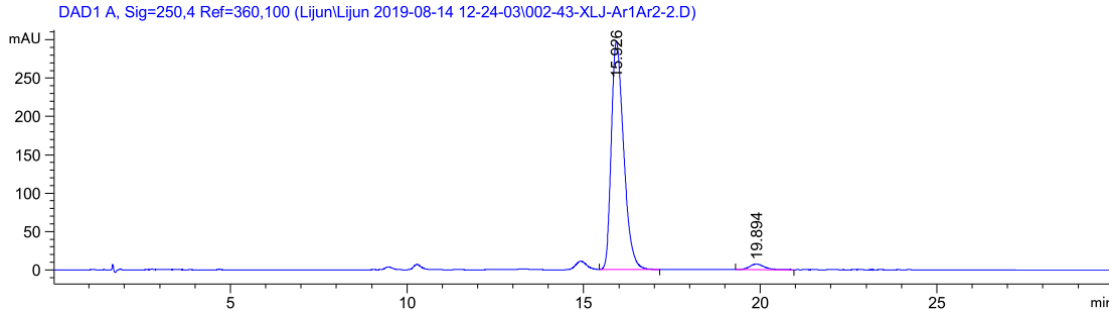
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	6.145	BB	0.1358	20.60308	2.27163	60.3481
2	7.195	MM	0.1719	13.53729	1.31283	39.6519



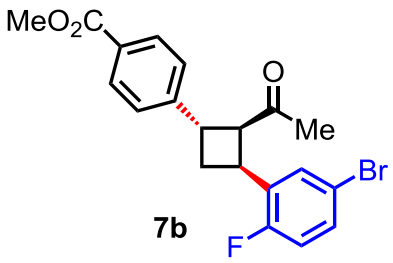
7a



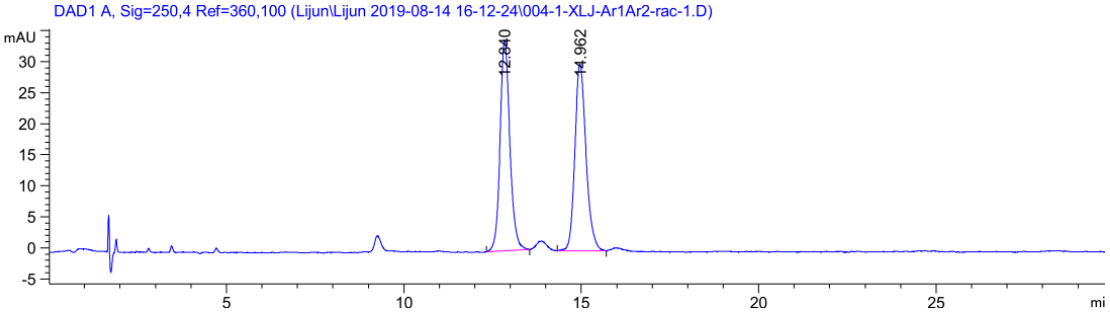
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	16.076	BB	0.3591	573.62939	24.32561	49.9344
2	19.910	BB	0.4295	575.13672	19.99709	50.0656



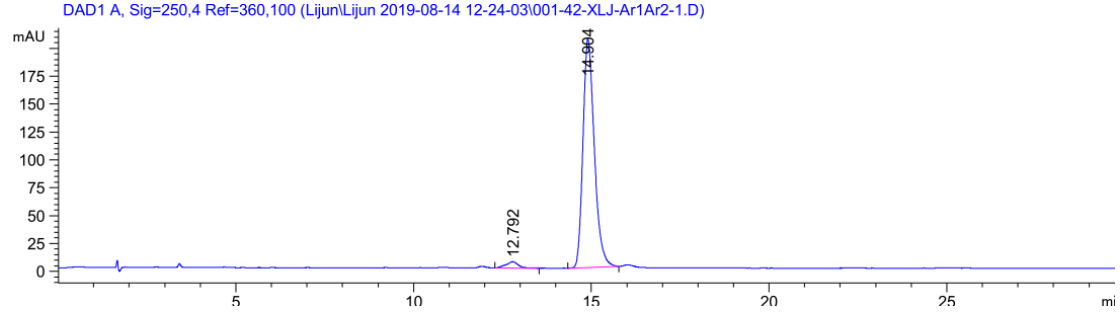
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	15.926	VB	0.3756	7237.36621	297.71201	97.1615
2	19.894	BB	0.4206	211.43762	7.24318	2.8385



7b



Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	12.840	BB	0.2930	648.24231	33.90818	49.6508
2	14.962	BB	0.3368	657.36176	30.09581	50.3492



Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	12.792	BB	0.3387	132.55385	5.58825	2.8320
2	14.904	BB	0.3415	4547.95215	204.47549	97.1680

10. X-Ray Crystallographic Data

Figure S1. X-Ray Structure of **7a**

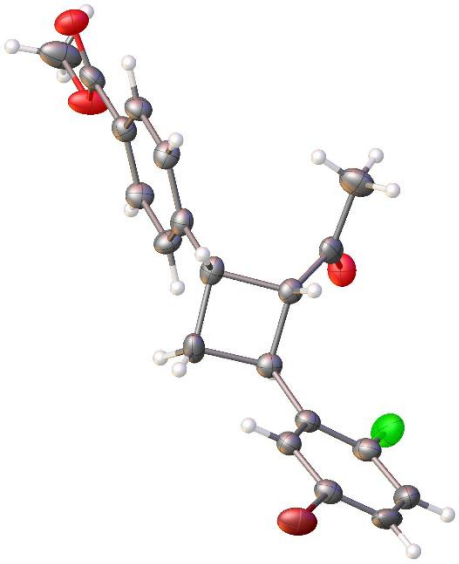


Table S6. Crystal data and structure refinement for **7a**

CCDC number	1965052	
Empirical formula	$C_{20}H_{18}BrFO_3$	
Formula weight	405.25	
Temperature	100.0 K	
Wavelength	1.54178 Å	
Crystal system	Orthorhombic	
Space group	$P2_12_12_1$	
Unit cell dimensions	a = 6.0286(2) Å	α= 90°.
	b = 15.4711(6) Å	β= 90°.
	c = 18.9210(5) Å	γ = 90°.
Volume	1764.74(10) $Å^3$	
Z	4	
Density (calculated)	1.525 Mg/m^3	
Absorption coefficient	3.404 mm^{-1}	
F(000)	824	
Crystal size	0.22 x 0.2 x 0.08 mm^3	
Crystal color, habit	colourless plate	
Theta range for data collection	3.690 to 66.738°.	
Index ranges	-6<=h<=7, -18<=k<=18, -19<=l<=22	
Reflections collected	12284	
Independent reflections	3121 [R(int) = 0.0310]	

Completeness to theta = 66.738°	99.8 %
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	0.5201 and 0.3722
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	3121 / 0 / 228
Goodness-of-fit on F^2	1.041
Final R indices [I>2sigma(I)]	R1 = 0.0255, wR2 = 0.0628
R indices (all data)	R1 = 0.0277, wR2 = 0.0639
Absolute structure parameter	-0.016(8)
Extinction coefficient	n/a
Largest diff. peak and hole	0.399 and -0.228 e.$Å^{-3}$

Figure S2. X-Ray Structure of **7b**

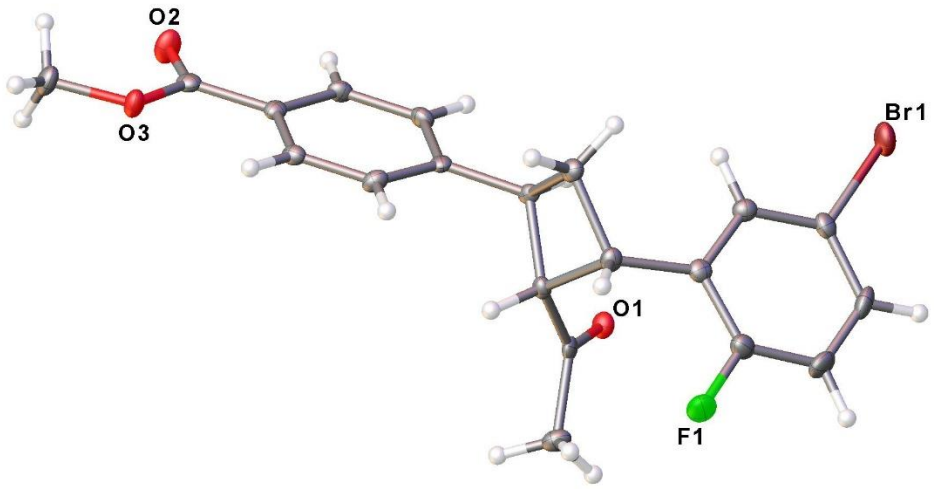


Table S7. Crystal data and structure refinement for **7b**

CCDC number	1965051	
Empirical formula	$C_{20}H_{18}BrFO_3$	
Formula weight	405.25	
Temperature	100.0 K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	P 21	
Unit cell dimensions	a = 5.5793(7) Å	α= 90°.
	b = 7.6061(7) Å	β= 95.929(5)°.
	c = 20.604(3) Å	γ = 90°.
Volume	869.69(18) $Å^3$	
Z	2	

Density (calculated)	1.548 Mg/m^3
Absorption coefficient	2.389 mm^{-1}
F(000)	412
Crystal size	0.3 x 0.2 x 0.1 mm^3
Theta range for data collection	1.987 to 27.115°.
Index ranges	-7<=h<=7, -9<=k<=9, -26<=l<=22
Reflections collected	6891
Independent reflections	3595 [R(int) = 0.0294]
Completeness to theta = 25.242°	99.5 %
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	0.7455 and 0.6028
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	3595 / 1 / 228
Goodness-of-fit on F^2	0.866
Final R indices [I>2sigma(I)]	R1 = 0.0284, wR2 = 0.0578
R indices (all data)	R1 = 0.0342, wR2 = 0.0595
Absolute structure parameter	0.029(6)
Largest diff. peak and hole	0.394 and -0.440 $e.Å^{-3}$