Supporting Information for

Natural Eutecto gels: Sustainable Catalytic Systems for C-C Bond Formation Reactions

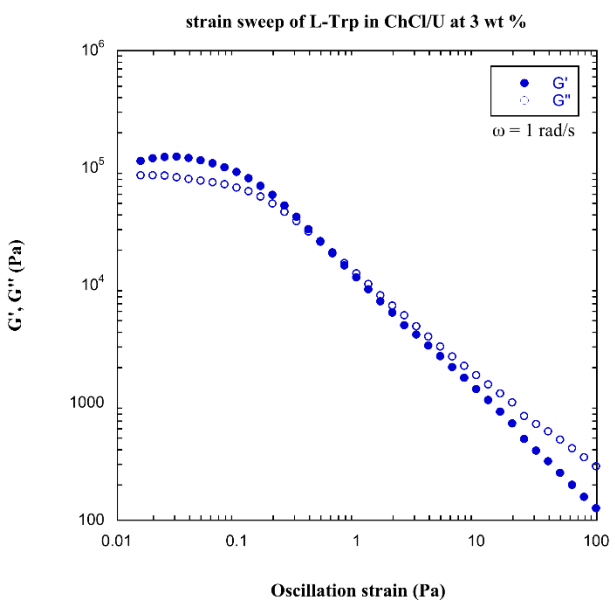
Table S1. Gelation tests performed in the presence of different gelators and solvents.

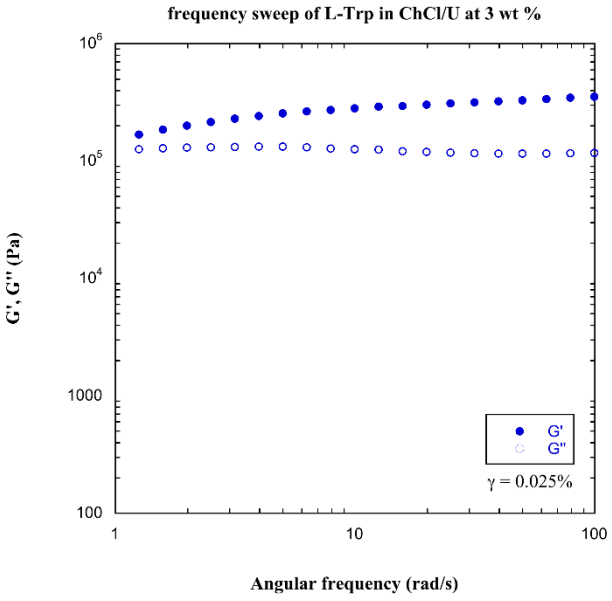
Gelator	DES	Conc. (w/w %)	Aspect[a]
L-Pro	ChCl/U (1:2)	3.0-5.0	G (3.0 %)
	DA/TBABr (1:2)	4.0-5.0	I
	ChCl/AcA (1:2)	2.0	S
	ChCl/Frut (1:2)	3.0-4.0	I
	$MePh_3PBr$/Gly (1:2)	2.0-3.0	S
L-Pro-NH_2	ChCl/U (1:2)	1.0-5.0	G (3.0%)
	DA/TBABr (1:2)	2.0-5.0	I
	ChCl/AcA (1:2)	2.0	S
	$MePh_3PBr$/Gly (1:2)	2.0-3.0	S
t-4-OH-L-Pro	ChCl/U (1:2)	1.0-5.0	G (3.0%)
	DA/TBABr (1:2)	2.0-5.0	I
	ChCl/AcA (1:2)	2.0	I
L-Ser	ChCl/U (1:2)	1.0-3.0	G (3.0%)
	DA/TBABr (1:2)	1.0-4.0	I
	ChCl/AcA (1:2)	2.0	I
	$MePh_3PBr$/Gly (1:2)	2.0-3.0	S
L-Iso	ChCl/U (1:2)	2.0	S
	DA/TBABr (1:2)	2.0	I
	ChCl/AcA (1:2)	2.0	I
L-Asp	ChCl/U (1:2)	3.0	I
L-Cys	ChCl/U (1:2)	1.0-3.0	I
L-Trp	ChCl/U (1:2)	1.0-2.0	G (2.0%)
	$MePh_3PBr$/Gly (1:2)	2.0	S

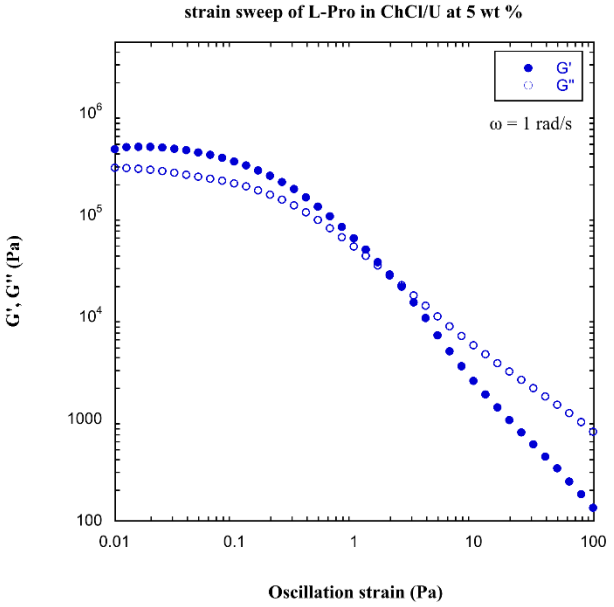
[a]G = gel; S = soluble; I = insoluble

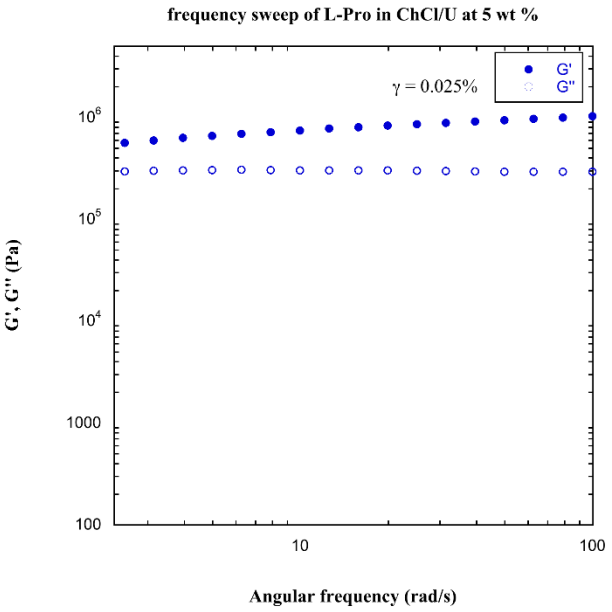
Table S2. Porosity of eutectogels determined in hexane.

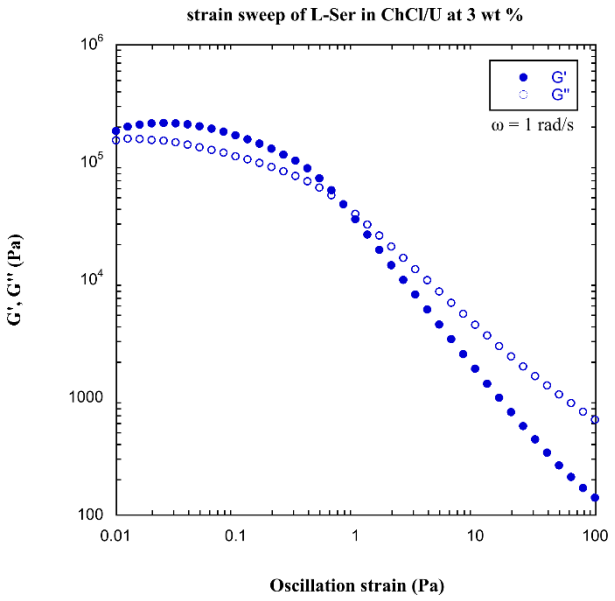
Eutectogel	P (%)
L-Pro/ChCl/U	86.9
t-4-OH-L-Pro/ChCl/U	86.3

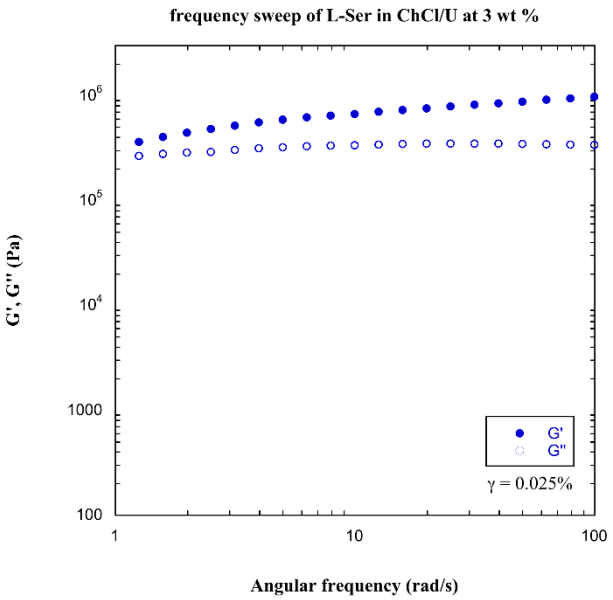

strain sweep of L-Trp in ChCl/U at 3 wt %
G'
G"
ω = 1 rad/s
G', G" (Pa)
Oscillation strain (Pa)

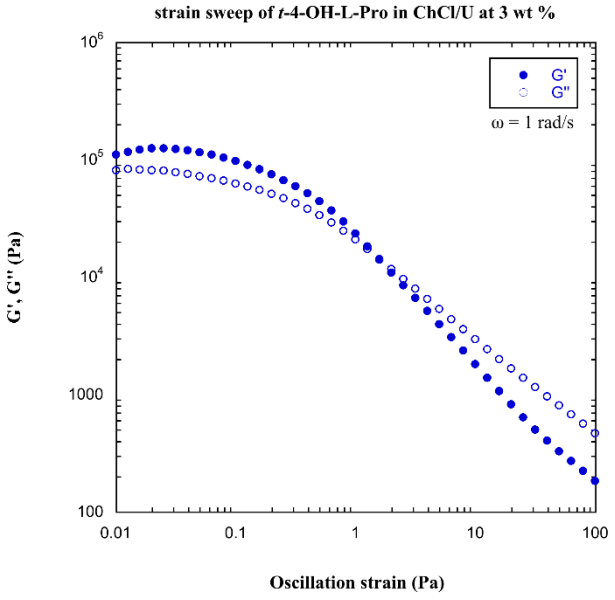

frequency sweep of L-Trp in ChCl/U at 3 wt %
G'
G"
γ = 0.025%
G', G" (Pa)
Angular frequency (rad/s)

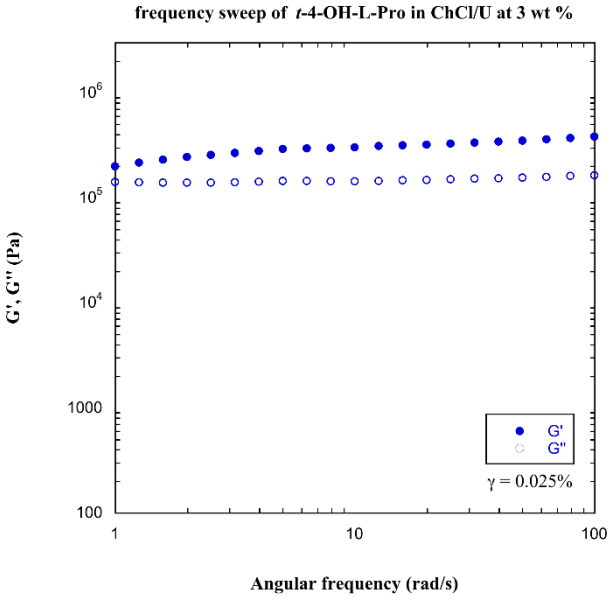

strain sweep of L-Pro in ChCl/U at 5 wt %
G'
G"
ω = 1 rad/s
G', G" (Pa)
Oscillation strain (Pa)


frequency sweep of L-Pro in ChCl/U at 5 wt %
γ = 0.025%
G'
G"
G', G" (Pa)
Angular frequency (rad/s)


strain sweep of L-Ser in ChCl/U at 3 wt %
G', G'' (Pa)
Oscillation strain (Pa)
G'
G''
ω = 1 rad/s


frequency sweep of L-Ser in ChCl/U at 3 wt %
G', G'' (Pa)
Angular frequency (rad/s)
G'
G''
γ = 0.025%


strain sweep of t-4-OH-L-Pro in ChCl/U at 3 wt %
G', G'' (Pa)
Oscillation strain (Pa)
G'
G''
ω = 1 rad/s


frequency sweep of t-4-OH-L-Pro in ChCl/U at 3 wt %
G', G'' (Pa)
Angular frequency (rad/s)
G'
G''
γ = 0.025%

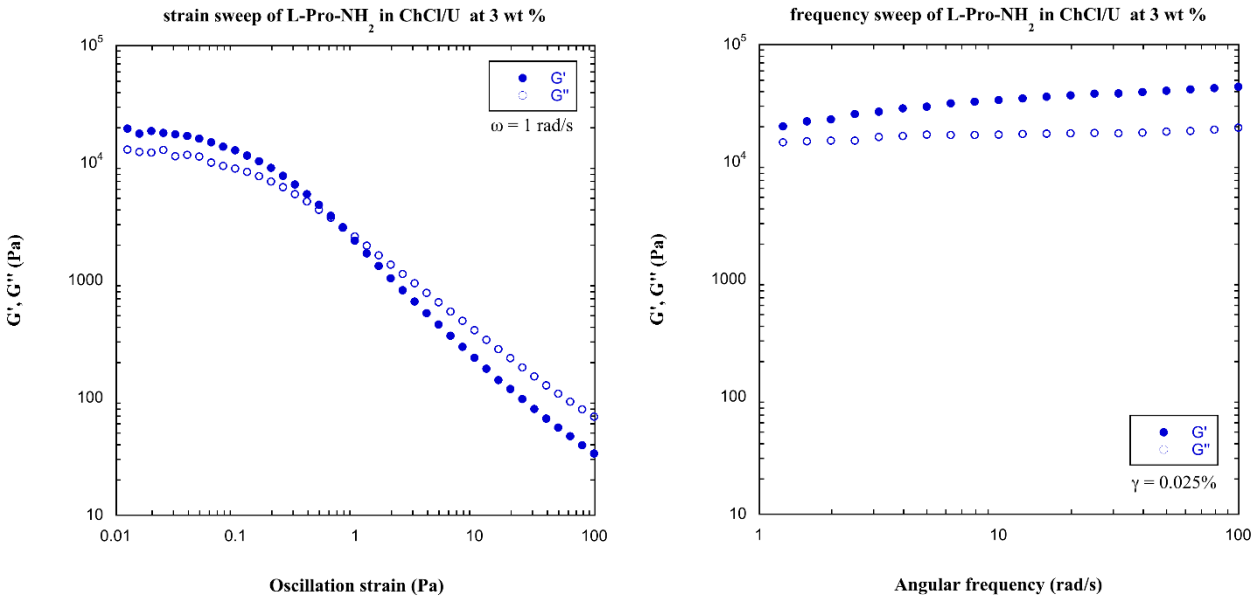


Figure S1. Strain and frequency sweeps of eutectogels.

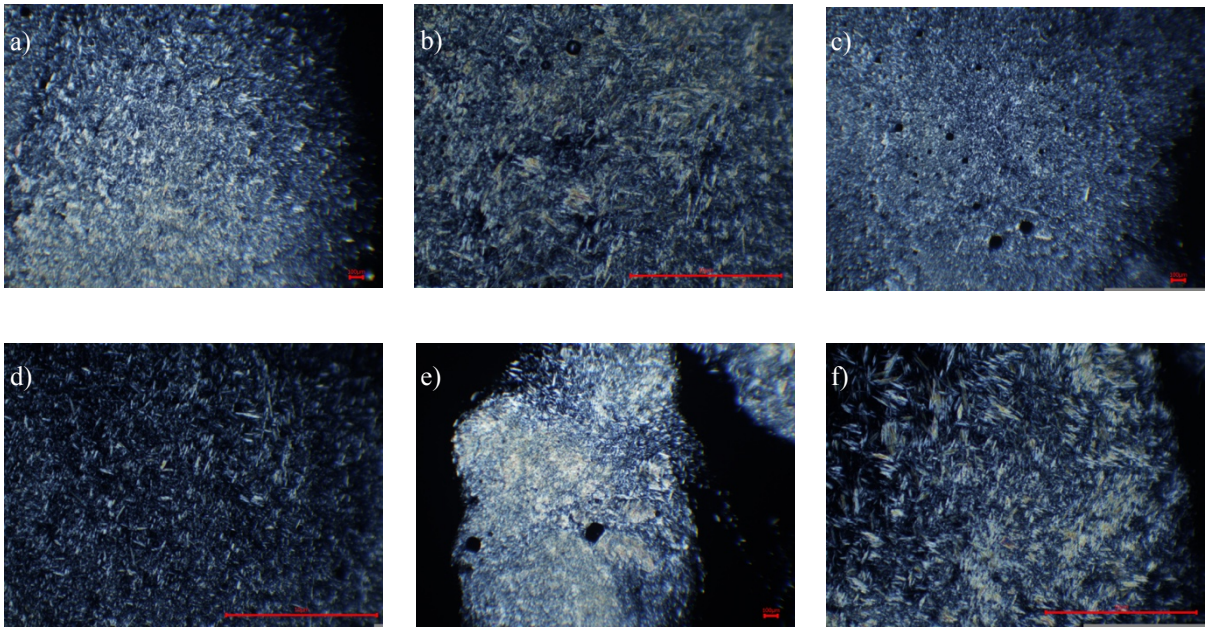
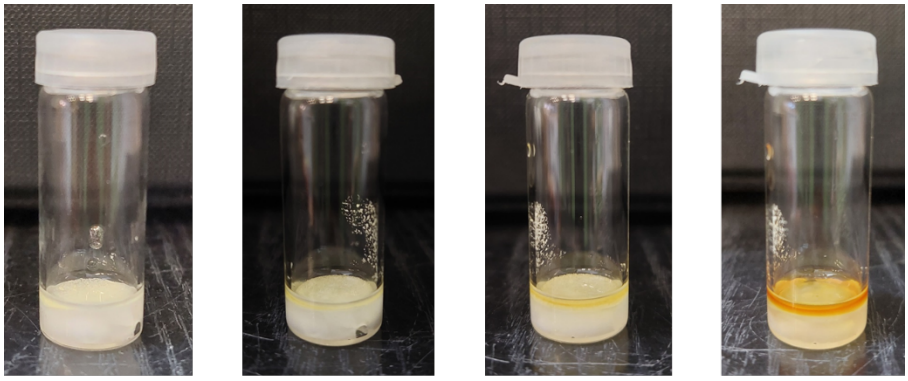


Figure S2. POM images for eutecto gels at 3.0 wt %: **a)- b**) L-Pro-NH_2/ChCl/U, **c) - d**) L-Ser/ChCl/U and **e) - f**) L-Trp/ChCl/U.

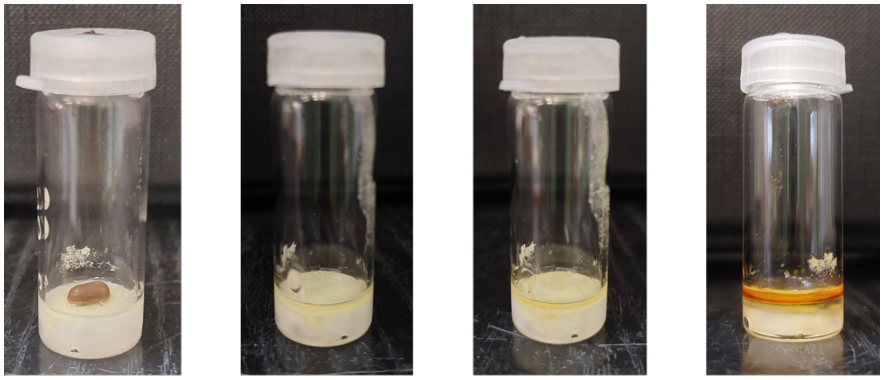
a) Evolution of the standard reaction



0 min 2 h 6 h 24 h

87% yield, 68% *ee*

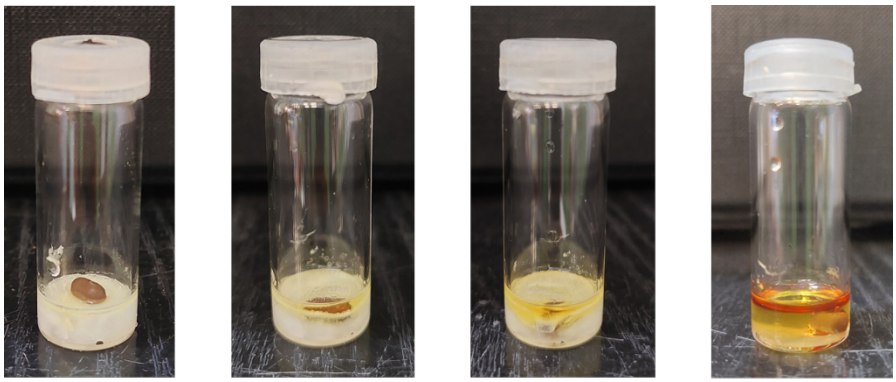
b) Evolution of the reaction stirring only the first 5 min



0 min 2 h 6 h 24 h

89% yield, 67% *ee*

c) Evolution of the reaction stirring during 24 h period



0 min 2 h 6 h 24 h

93% yield, 54% *ee*

Figure S3. Evolution of the reaction images.

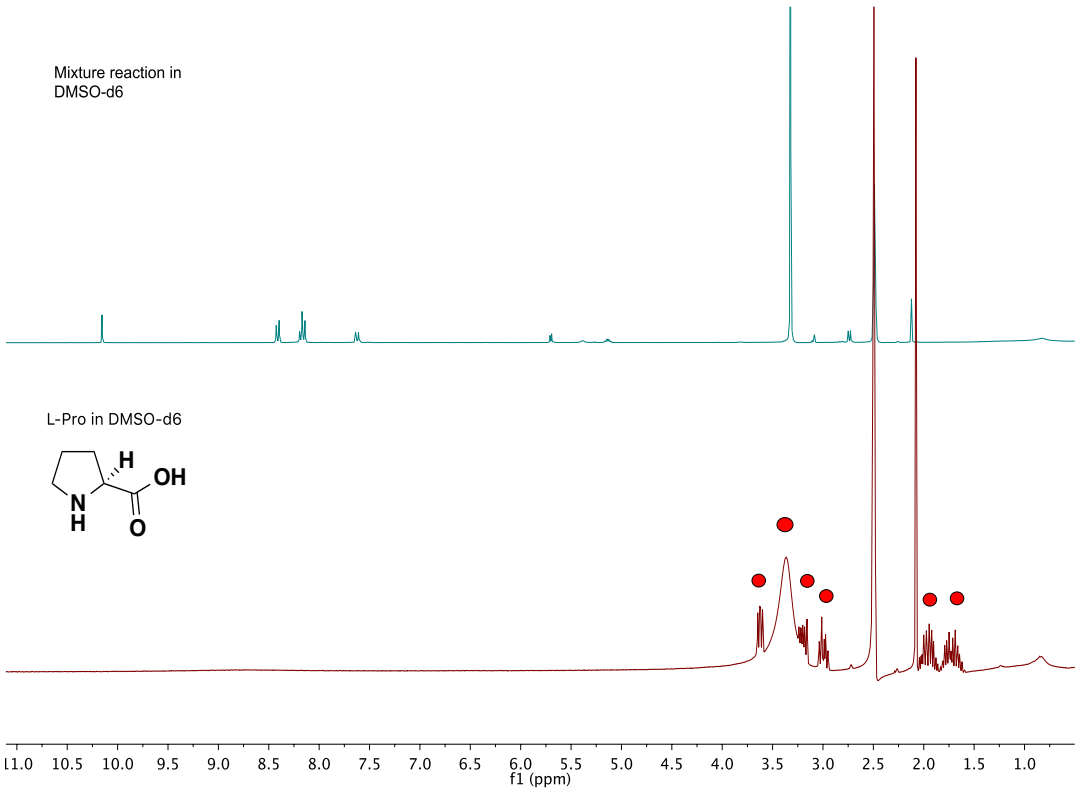


Figure S4. ^{1}H NMR spectra of L-Pro in DMSO-[d6] (down) and liquid phase (reaction mixture acetone/*p*-nitrobenzaldehyde) on the top of the gel after 24h (top).

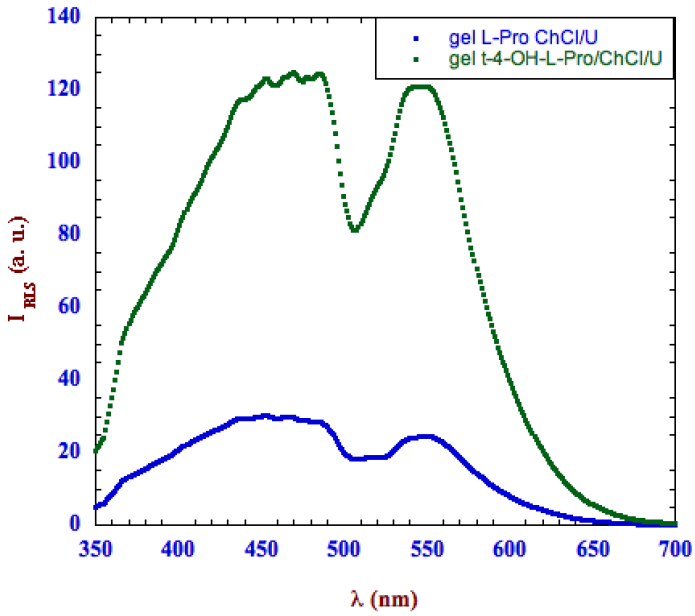
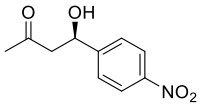
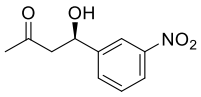


Figure S5. RLS spectra of L-Pro/ChCl/U and t-4-OH-L-Pro/ChCl/U eutectogels.

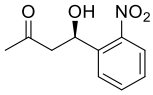
Spectra data of aldol products



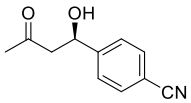
4-Hydroxy-4-(4-nitrophenyl)butan-2-one (3a):[1] ^{1}H NMR (300 MHz, $CDCl_3$): δ= 8.21 (d, J = 8.8 Hz, 2H, ArH), 7.54 (d, J = 8.8 Hz, 2H, ArH), 5.30-5.25 (m, 1H, CH_2CHOH), 3.62 (brs, 1H, OH), 2.90-2.85 (m, 2H, $COCH_2CH$), 2.23 (s, 3H, $COCH_3$) ppm; ^{13}C NMR (75 MHz, $CDCl_3$): δ = 208.6, 150.1, 147.5, 126.6, 123.9, 69.1, 51.6, 30.8 ppm.
The enantiomeric excess was determined by HPLC with a Chiracel AS column at 254 nm (*n*-hexane/*i*-PrOH: 85/15, 1.0 mL/min), t_R = 17.99 (major), t_R = 26.79 (minor).



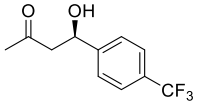
4-Hydroxy-4-(3-nitrophenyl)butan-2-one (3b):[1] ^{1}H NMR (300 MHz, $CDCl_3$): δ= 8.25-8.20 (m, 1H, ArH), 8.15-8.10 (m, 1H, ArH), 7.75-7.70 (m, 1H, ArH), 7.54 (t, J = 7.9 Hz, 1H), 5.30-5.25 (m, 1H, CH_2CHOH), 3.64 (d, J = 3.1 Hz, 1H, OH), 2.90-2.85 (m, 2H, $COCH_2CH$), 2.24 (s, 3H, $COCH_3$) ppm; ^{13}C NMR (75 MHz, $CDCl_3$): δ = 208.8, 148.5, 144.9, 131.9, 129.6, 122.7, 120.9, 68.9, 51.6, 30.9 ppm.
The enantiomeric excess was determined by HPLC with a Chiracel ADH column at 254 nm (*n*-hexane/*i*-PrOH: 95/5, 1.0 mL/min), t_R = 26.78 (major), t_R = 29.02 (minor).



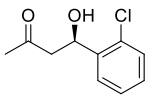
4-Hydroxy-4-(2-nitrophenyl)butan-2-one (3c):[1] ^{1}H NMR (300 MHz, $CDCl_3$): δ= 8.00-7.90 (m, 2H, ArH), 7.70-7.65 (m, 1H, ArH), 7.50-7.40 (m, 1H, ArH), 5.69 (dd, J = 9.4, 2.0 Hz, 1H, CH_2CHOH), 3.15 (dd, J = 17.8, 2.0 Hz, 1H, $COCH_2CH$), 2.80-2.70 (m, 1H, $COCH_2CH$), 2.24 (s, 3H, $COCH_3$) ppm; ^{13}C NMR (75 MHz, $CDCl_3$): δ = 208.8, 147.2, 138.3, 133.8, 128.3, 128.2, 124.5, 65.7, 51.0, 30.5 ppm.
The enantiomeric excess was determined by HPLC with a Chiracel ADH column at 254 nm (*n*-hexane/*i*-PrOH: 98/2, 1.0 mL/min), t_R = 42.34 (major), t_R = 45.77 (minor).



4-(1-Hydroxy-3-oxobutyl)benzonitrile (3d):[1] ^{1}H NMR (300 MHz, $CDCl_3$): δ= 7.70-7.60 (m, 2H, ArH), 7.50-7.45 (m, 2H, ArH), 5.25-5.20 (m, 1H, CH_2CHOH), 3.56 (d, J = 3.2 Hz, 1H, OH), 2.90-2.80 (m, 2H, $COCH_2CH$), 2.22 (s, 3H, $COCH_3$) ppm; ^{13}C NMR (75 MHz, $CDCl_3$): δ = 208.7, 148.1, 132.5, 126.4, 118.9, 111.5, 69.2, 51.6, 30.9 ppm.
The enantiomeric excess was determined by HPLC with a Chiracel ODH column at 230 nm (*n*-hexane/*i*-PrOH: 95/5, 1.0 mL/min), t_R = 34.30 (major), t_R = 40.06 (minor).

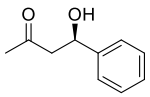


4-Hydroxy-4-(4-(trifluoromethyl)phenyl)butan-2-one (3e):[1] ^{1}H NMR (300 MHz, $CDCl_3$): δ= 7.61 (d, J = 8.2 Hz, 2H, ArH), 7.48 (d, J = 8.2 Hz, 2H, ArH), 5.22 (t, J = 6.1 Hz, 1H, CH_2CHOH), 2.85 (d, J = 6.1 Hz, 2H, $COCH_2CH$), 2.21 (s, 3H, $COCH_3$) ppm; ^{13}C NMR (75 MHz, $CDCl_3$): δ = 208.9, 146.7, 129.8, 128.5, 126.1, 125.6 (q, J = 3.8 Hz), 122.9, 69.4, 51.8, 30.9 ppm.
The enantiomeric excess was determined by HPLC with a Chiracel AS column at 210 nm (*n*-hexane/*i*-PrOH: 92/8, 1.0 mL/min), t_R = 8.97 (major), t_R = 11.35 (minor).

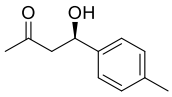


4-(2-Chlorophenyl)-4-hydroxybutan-2-one (3f):[1] ^{1}H NMR (300 MHz, $CDCl_3$): δ= 7.65-7.60 (m, 1H, ArH), 7.35-7.30 (m, 2H), 7.25-7.20 (m, 1H, ArH), 5.51 (dt, J = 9.7, 2.2 Hz, 1H, CH_2CHOH), 3.55 (d, J = 3.3 Hz, 1H, OH), 3.00 (dd, J = 17.7, 2.2 Hz, 1H, $COCH_2CH$), 2.68 (dd, J = 17.7, 9.7 Hz, 1H, $COCH_2CH$), 2.22 (s, 3H, $COCH_3$) ppm; ^{13}C NMR (75 MHz, $CDCl_3$): δ = 209.4, 140.1, 131.2, 129.4, 128.7, 127.4, 127.2, 66.7, 50.1, 30.8 ppm.

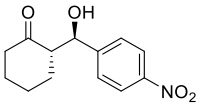
The enantiomeric excess was determined by HPLC with a Chiracel AS column at 210 nm (*n*-hexane/*i*-PrOH: 98/2, 1.0 mL/min), t_R = 16.96 (minor), t_R = 18.89 (major).



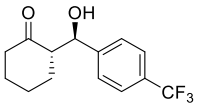
4-Hydroxy-4-phenylbutan-2-one (3g):[1] ^{1}H NMR (300 MHz, $CDCl_3$): δ= 7.40-7.35 (m, 4H, ArH), 7.35-7.30 (m, 1H, ArH), 5.20-5.15 (m, 1H, CH_2CHOH), 3.28 (d, J = 2.8 Hz, 1H, OH), 2.95-2.80 (m, 2H, $COCH_2CH$), 2.21 (s, 3H, $COCH_3$) ppm; ^{13}C NMR (75 MHz, $CDCl_3$): δ = 209.3, 142.8, 128.7, 127.9, 125.8, 70.0, 52.1, 30.9 ppm.
The enantiomeric excess was determined by HPLC with a Chiracel AS column at 210 nm (*n*-hexane/*i*-PrOH: 90/10, 1.0 mL/min), t_R = 9.95 (major), t_R = 12.34 (minor).



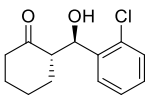
4-Hydroxy-4-(p-tolyl)butan-2-one (3h):[2] ^{1}H NMR (300 MHz, $CDCl_3$): δ= 7.25-7.20 (m, 2H, ArH), 7.20-7.15 (m, 2H, ArH), 5.13 (dd, J = 9.2, 3.2 Hz, 1H, CH_2CHOH), 2.95-2.80 (m, 2H, $COCH_2CH$), 2.34 (s, 3H, $ArCH^3$), 2.20 (s, 3H, $COCH_3$) ppm; ^{13}C NMR (75 MHz, $CDCl_3$): δ = 209.4, 139.8, 137.6, 129.4, 125.7, 69.9, 52.1, 31.0, 21.3 ppm.
The enantiomeric excess was determined by HPLC with a Chiracel ASH column at 210 nm (*n*-hexane/*i*-PrOH: 90/10, 1.0 mL/min), t_R = 9.09 (major), t_R = 11.28 (minor).



2-(Hydroxy(4-nitrophenyl)methyl)cyclohexan-1-one (3i) *syn:anti* (1:4):[1] ^{1}H NMR (300 MHz, $CDCl_3$): δ= 8.25-8.20 (m, 2H, ArH), 7.55-7.50 (m, 2H, ArH), 5.49 (d, J = 2.2 Hz, 1H *syn*, C*H*OH), 4.90 (d, J = 8.4 Hz, 1H *anti*, C*H*OH), 2.65-2.60 (m, 1H), 2.55-2.50 (m, 1H), 2.40-2.30 (m, 1H), 2.15-2.10 (m, 1H), 1.90-1.80 (m, 1H), 1.70-1.50 (m, 3H), 1.45-1.35 (m, 1H) ppm; ^{13}C NMR (75 MHz, $CDCl_3$): δ = 214.9 (*anti*), 214.2 (*syn*), 149.2, 148.5, 147.7, 147.2, 128.0 (*anti*), 126.7 (*syn*), 123.7 (*anti*), 123.6 (*syn*), 74.2 (*anti*), 70.3 (*syn*), 57.3 (*anti*), 56.9 (*syn*), 42.8 (2C, *anti, syn*), 30.9 (*anti*), 28.0 (*syn*), 27.8 (anti), 26.1 (*syn*), 24.9 (*syn*), 24.8 (*anti*) ppm.
The enantiomeric excess was determined by HPLC with a Chiracel ADH column at 254 nm (*n*-hexane/*i*-PrOH: 90/10, 1.0 mL/min), *syn*: t_R = 18.72 (major), t_R = 20.66 (minor), *anti*: t_R = 24.06 (minor), t_R = 27.25 (major).

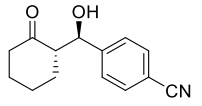


2-(Hydroxy(4-(trifluoromethyl)phenyl)methyl)cyclohexan-1-one *syn:anti* (1:2) (3j):[1] ^{1}H NMR (300 MHz, $CDCl_3$): δ= 7.65-7.60 (m, 2H, ArH), 7.50-7.40 (m, 2H, ArH), 5.45 (d, J = 1.0 Hz, 1H *syn*, C*H*OH), 4.85 (d, J = 8.6 Hz, 1H *anti*, C*H*OH), 2.65-2.60 (m, 1H), 2.50-2.45 (m, 1H), 2.40-2.30 (m, 1H), 2.15-2.00 (m, 1H), 1.90-1.75 (m, 1H), 1.70-1.65 (m, 1H), 1.60-1.50 (m, 2H), 1.40-1.30 (m, 1H) ppm; ^{13}C NMR (75 MHz, $CDCl_3$): δ = 215.3 (*anti*), 214.6 (*syn*), 145.6 (*syn*), 145.1 (*anti*), 130.4 (*anti*), 127.5 (*anti*), 126.2 (*anti*), 125.5 (q, J = 3.5 Hz, *anti*), 125.3 (q, J = 3.5 Hz, *syn*), 74.4 (*anti*), 70.4 (*syn*), 57.4 (*anti*), 57.1 (*syn*), 42.8 (2C, *anti, syn*), 30.9 (*anti*), 28.1 (*syn*), 27.9 (*anti*), 26.0 (*syn*), 25.0 (*syn*), 24.9 (*anti*) ppm.
The enantiomeric excess was determined by HPLC with a Chiracel AD column at 210 nm (*n*-hexane/*i*-PrOH: 90/10, 1.0 mL/min), *syn*: t_R = 7.21 (major), t_R = 8.38 (minor), *anti*: t_R = 10.70 (minor), t_R = 13.55 (major).

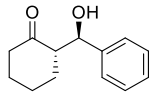


2-((2-Chlorophenyl)(hydroxy)methyl)cyclohexan-1-one *syn:anti* (1:3.3) (3k):[1] ^{1}H NMR (300 MHz, $CDCl_3$): δ= 7.60-7.55 (m, 1H, ArH), 7.35-7.30 (m, 2H, ArH), 7.25-7.20 (m, 1H, ArH), 5.71 (d, J = 1.8 Hz, 1H *syn*, C*H*OH), 5.35 (d, J = 8.1 Hz, 1H *anti*, C*H*OH), 2.85-2.80 (m, 1H *syn*), 2.70-2.65 (m 1H), 2.50-2.40 (m, 1H), 2.40-2.30 (m, 1H), 2.15-2.05 (m, 1H), 1.85-1.80 (m, 1H), 1.70-1.50 (m, 4H) ppm; ^{13}C NMR (75 MHz, $CDCl_3$): δ = 215.4 (*anti*), 215.0 (*syn*), 139.2

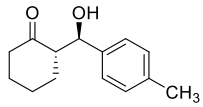
(*anti*), 138.7 (*syn*), 133.1 (*anti*), 129.4 (*anti*), 128.9 (*anti*), 128.7 (*syn*), 128.4 (*anti*), 128.3 (*syn*), 70.4 (*syn*), 127.4 (*anti*), 126.8 (*syn*), 70.6 (*anti*), 67.9 (*syn*), 57.7 (*anti*), 53.7 (*syn*), 42.9 (*anti*), 30.6 (*anti*), 28.1 (*syn*), 28.0 (*anti*), 26.1 (*syn*), 25.1 (*anti*), 25.0 (*syn*) ppm.
The enantiomeric excess was determined by HPLC with a Chiracel ODH column at 210 nm (*n*-hexane/*i*-PrOH: 95/5, 1.0 mL/min), *syn*: t_R = 7.22 (major), t_R = 8.38 (minor), *anti*: t_R = 9.35 (major), t_R = 11.87 (minor).



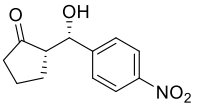
4-(Hydroxy(2-oxocyclohexyl)methyl)benzonitrile (3l) *syn:anti* (1:2.5):[3] ^{1}H NMR (300 MHz, $CDCl_3$): δ= 7.65-7.60 (m, 2H, ArH), 7.45-7.40 (m, 2H, ArH), 5.43 (d, J = 2.0 Hz, 1H *syn*, C*H*OH), 4.84 (d, J = 8.5 Hz, 1H *anti*, C*H*OH), 2.60-2.50 (m, 2H), 2.45-2.30 (m, 1H), 2.15-2.10 (m, 1H), 1.90-1.75 (m, 3H), 1.40-1.30 (m, 1H) ppm; ^{13}C NMR (75 MHz, $CDCl_3$): δ = 215.0 (*anti*), 214.3 (*syn*), 147.1 (*syn*), 146.5 (*anti*), 132.3 (*anti*), 133.2 (*syn*), 127.9 (*anti*), 126.7 (*syn*), 119.0 (*syn*), 118.9 (*anti*), 111.9 (*anti*), 111.0 (*syn*), 74.4 (*anti*), 70.3 (*syn*), 57.3 (*anti*), 56.9 (*syn*), 42.8 (2C, *anti*, *syn*), 30.9 (*anti*), 28.0 (*syn*), 27.8 (*anti*), 26.0 (*syn*), 24.9 (*syn*), 24.8 (*anti*) ppm.
The enantiomeric excess was determined by HPLC with a Chiracel ADH column at 240 nm (*n*-hexane/*i*-PrOH: 95/5, 1.0 mL/min), *syn*: t_R = 31.58 (major), t_R = 39.68 (minor), *anti*: t_R = 46.53 (minor), t_R = 59.58 (major).



2-(Hydroxy(phenyl)methyl)cyclohexan-1-one (3m) *syn:anti* (1:2.5):[4] ^{1}H NMR (300 MHz, $CDCl_3$): δ= 7.40-7.30 (m, 5H), 5.40 (d, J = 2.1 Hz, 1H *syn*, C*H*OH), 4.79 (d, J = 8.8 Hz, 1H *anti*, C*H*OH), 2.90-2.80 (m, 1H *syn*), 2.65-2.50 (m, 1H), 2.50-2.30 (m, 2H), 2.15-2.10 (m, 1H), 2.00-1.50 (m, 4H), 1.35-1.30 (m, 1H) ppm; ^{13}C NMR (75 MHz, $CDCl_3$): δ = 215.7 (*anti*), 202.0, 141.1, 135.7, 130.4, 128.5, 128.3, 128.1, 127.2, 125.9, 74.9 (*anti*), 70.8 (*syn*), 57.6 (*anti*), 57.3 (*syn*), 42.9 (*anti*), 40.5 (*syn*), 31.0 (*anti*), 28.1 (*syn*), 28.0 (*anti*), 26.2 (*syn*), 25.0 (*syn*), 24.9 (*anti*) ppm.
The enantiomeric excess was determined by HPLC with a Chiracel ODH column at 210 nm (*n*-hexane/*i*-PrOH: 95/5, 0.5 mL/min), *syn*: t_R = 18.79 (major), t_R = 21.48 (minor), *anti*: t_R = 25.58 (major), t_R = 37.91 (minor).

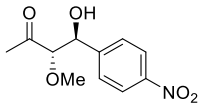


2-(Hydroxy(*p*-tolyl)methyl)cyclohexan-1-one (3n):[5] ^{1}H NMR (400 MHz, $CDCl_3$): δ= 7.25-7.10 (m, 4H, ArH), 4.75 (dd, J = 8.7, 2.7 Hz, 1H, C*H*OH), 3.89 (d, J = 2.7 Hz, 1H, OH), 2.70-2.55 (m, 1H), 2.55-2.45 (m, 1H), 2.40-2.35 (m, 1H), 2.34 (s, 3H, CH_3), 2.08 (m, 1H), 1.85-1.75 (m, 1H), 1.70-1.50 (m, 2H), 1.35-1.20 (m, 2H) ppm; ^{13}C NMR (101 MHz, $CDCl_3$): δ = 209.2, 137.7, 136.0, 129.2, 127.1, 74.7, 57.6, 42.8, 31.1, 28.0, 24.9, 21.3 ppm.
The enantiomeric excess was determined by HPLC with a Chiracel ODH column at 210 nm (*n*-hexane/*i*-PrOH: 98/2, 1.0 mL/min), *syn*: t_R = 8.25 (major), t_R = 8.76 (minor), *anti*: t_R = 10.79 (major), t_R = 14.26 (minor).

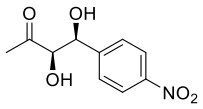


2-(Hydroxy(4-nitrophenyl)methyl)cyclopentan-1-one (3o) *syn:anti* (1.8:1):[1] ^{1}H NMR (300 MHz, $CDCl_3$): δ= 8.25-8.20 (m, 2H, ArH), 7.55-7.50 (m, 2H), 5.43 (d, J = 2.9 Hz, 1H *syn*, C*H*OH), 4.85 (d, J = 9.2 Hz, 1H *anti*, C*H*OH), 2.50-2.10 (m, 3H), 2.10-1.90 (m, 2H), 1.85-1.45 (m, 2H) ppm; ^{13}C NMR (75 MHz, $CDCl_3$): δ = 222.4 (*anti*), 219.5 (*syn*), 150.2, 148.8, 147.8, 147.3, 127.5 (*anti*), 126.5 (*syn*), 123.9 (*anti*), 123.8 (*syn*), 74.6 (*anti*), 70.7 (*syn*), 56.2 (*syn*), 55.2 (*anti*), 39.1 (*syn*), 38.7 (*anti*), 27.0 (*anti*), 22.6 (*syn*), 20.5 (2C, *anti, syn*) ppm.

The enantiomeric excess was determined by HPLC with a Chiracel AD column at 280 nm (*n*-hexane/*i*-PrOH: 96/4, 1.0 mL/min), *syn*: t_R = 32.64 (major), t_R = 48.06 (minor), *anti*: t_R = 56.63 (minor), t_R = 61.25 (major).



4-Hydroxy-3-methoxy-4-(4-nitrophenyl)butan-2-one (3p) *syn:anti* (1:3.3):[1] ^{1}H NMR (300 MHz, $CDCl_3$): δ= 8.25-8.20 (m, 2H, ArH), 7.60-7.55 (m, 2H, ArH), 5.05 (d, J = 4.0 Hz, 1H *syn*, C*H*OH), 5.03 (d, J = 6.3 Hz, 1H *anti*, C*H*OH), 3.77 (d, J = 4.0 Hz, 1H *syn*, C*H*OCH_3), 3.70 (d, J = 6.3 Hz, 1H *anti*, C*H*OCH_3), 3.38 (s, 3H *syn*, $COCH_3$), 3.32 (s, 3H *anti*, $COCH_3$) ppm; ^{13}C NMR (75 MHz, $CDCl_3$): δ = 210.0 (*anti*), 147.9, 147.5, 146.9, 127.8 (*anti*), 127.3 (*syn*), 123.7 (*syn*), 123.6 (*anti*), 90.1 (*syn*), 89.7 (*anti*), 73.5 (*anti*), 73.4 (*syn*), 59.8 (2C, *anti*, *syn*), 27.7 (*syn*), 27.6 (*anti*) ppm.
The enantiomeric excess was determined by HPLC with a Chiracel ODH column at 280 nm (*n*-hexane/*i*-PrOH: 90/10, 0.8 mL/min), *anti*: t_R = 15.34 (major), t_R = 18.14 (minor), *syn*: t_R = 19.05 (minor), t_R = 24.24 (major).



3,4-Dihydroxy-4-(4-nitrophenyl)butan-2-one (3q) *syn:anti* (1.6:1):[6] ^{1}H NMR (300 MHz, $CDCl_3$): δ= 8.30-8.25 (m, 2H, ArH), 7.65-7.60 (m, 2H, ArH), 5.23 (d, J = 2.5 Hz, 1H *syn*, CC*H*OH), 5.11 (d, J = 4.7 Hz, 1H *anti*, CC*H*OH), 4.49 (d, J = 4.7 Hz, 1H *anti*, COC*H*OH), 4.42 (d, J = 2.5 Hz, 1H *syn*, COC*H*OH), 2.38 (s, 3H *syn*), 2.02 (s, 3H *anti*) ppm; ^{13}C NMR (75 MHz, $CDCl_3$): δ = 206.7, 206.6, 147.4, 127.4, 127.3, 123.9, 80.7, 80.1, 74.6, 73.1, 26.1 ppm.
The enantiomeric excess was determined by HPLC with a Chiracel ADH column at 254 nm (*n*-hexane/*i*-PrOH: 80/20, 0.8 mL/min), *anti*: t_R = 10.62 (minor), t_R = 11.93 (major), *syn*: t_R = 13.77 (minor), t_R = 17.35 (major).

References

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[2] Yu, N.; Han, S.; Yu, H. *Tetrahedron* **2015**, *71*, 4665-4669.

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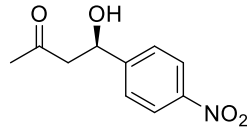
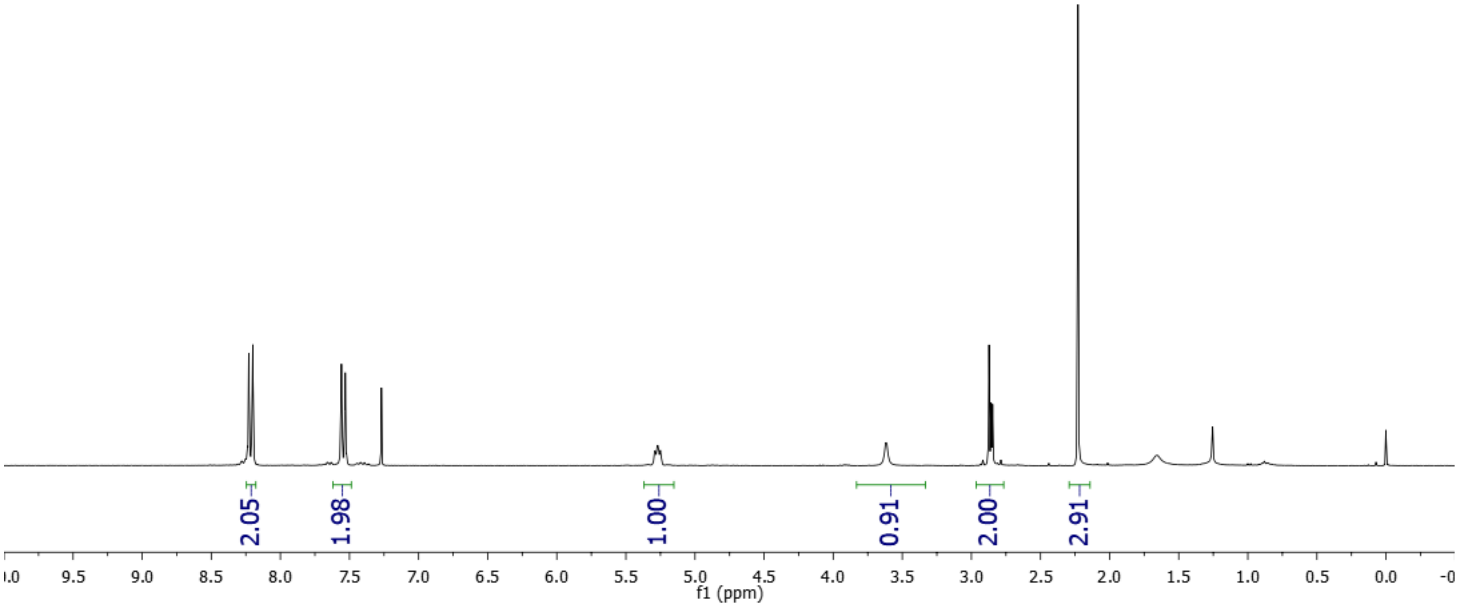
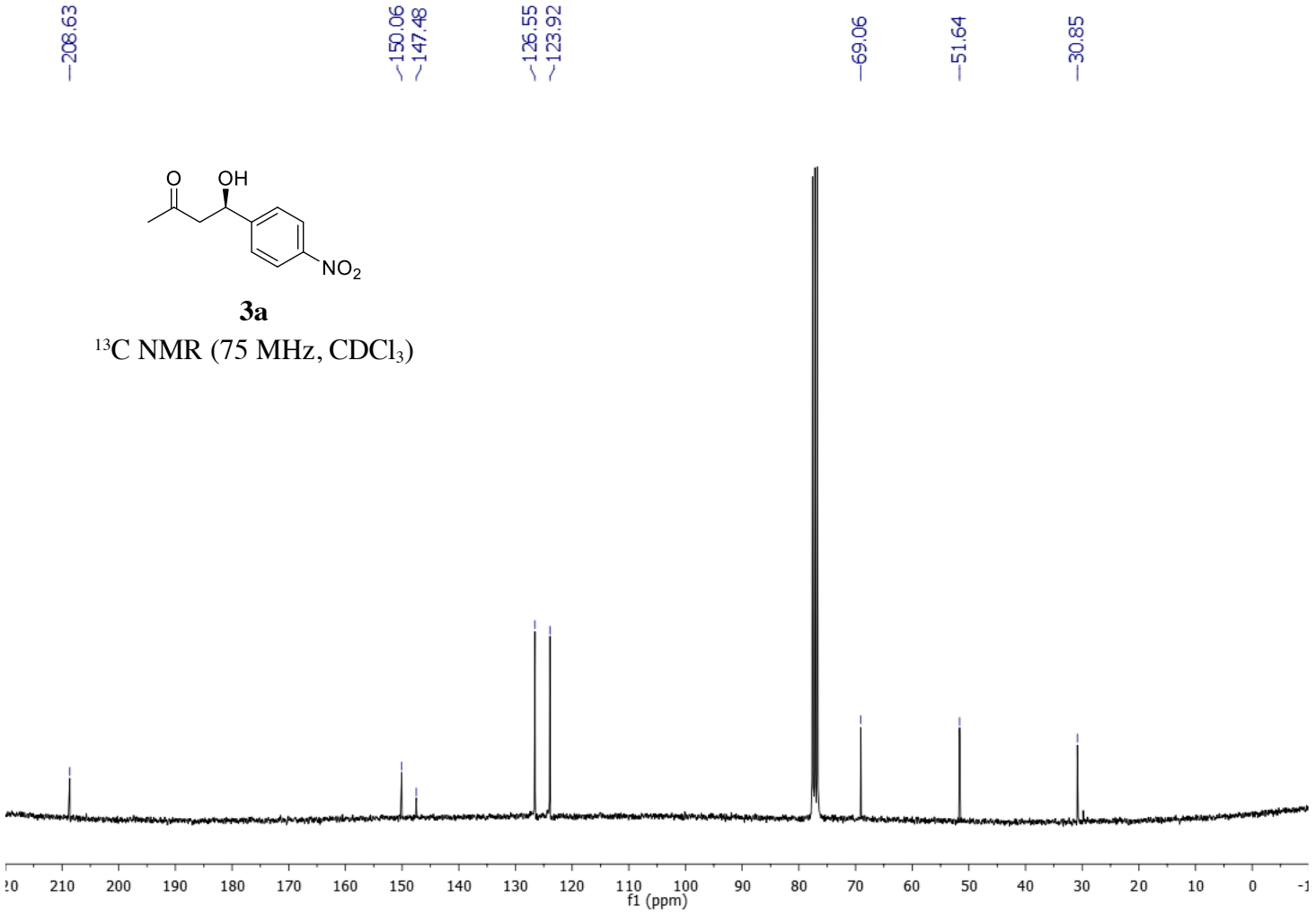
NMR spectra for aldol products



3a

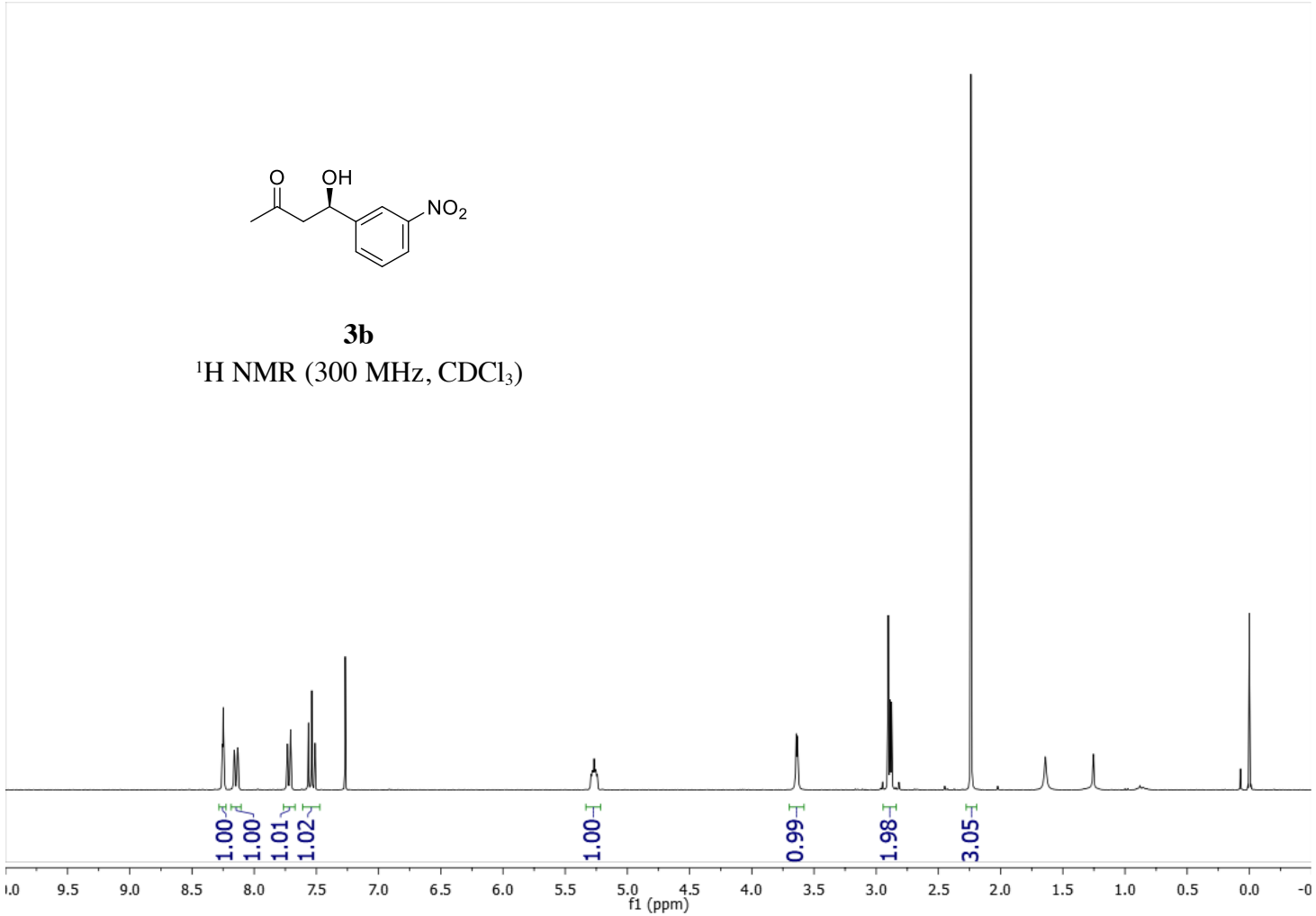
^{1}H NMR (300 MHz, $CDCl_3$)





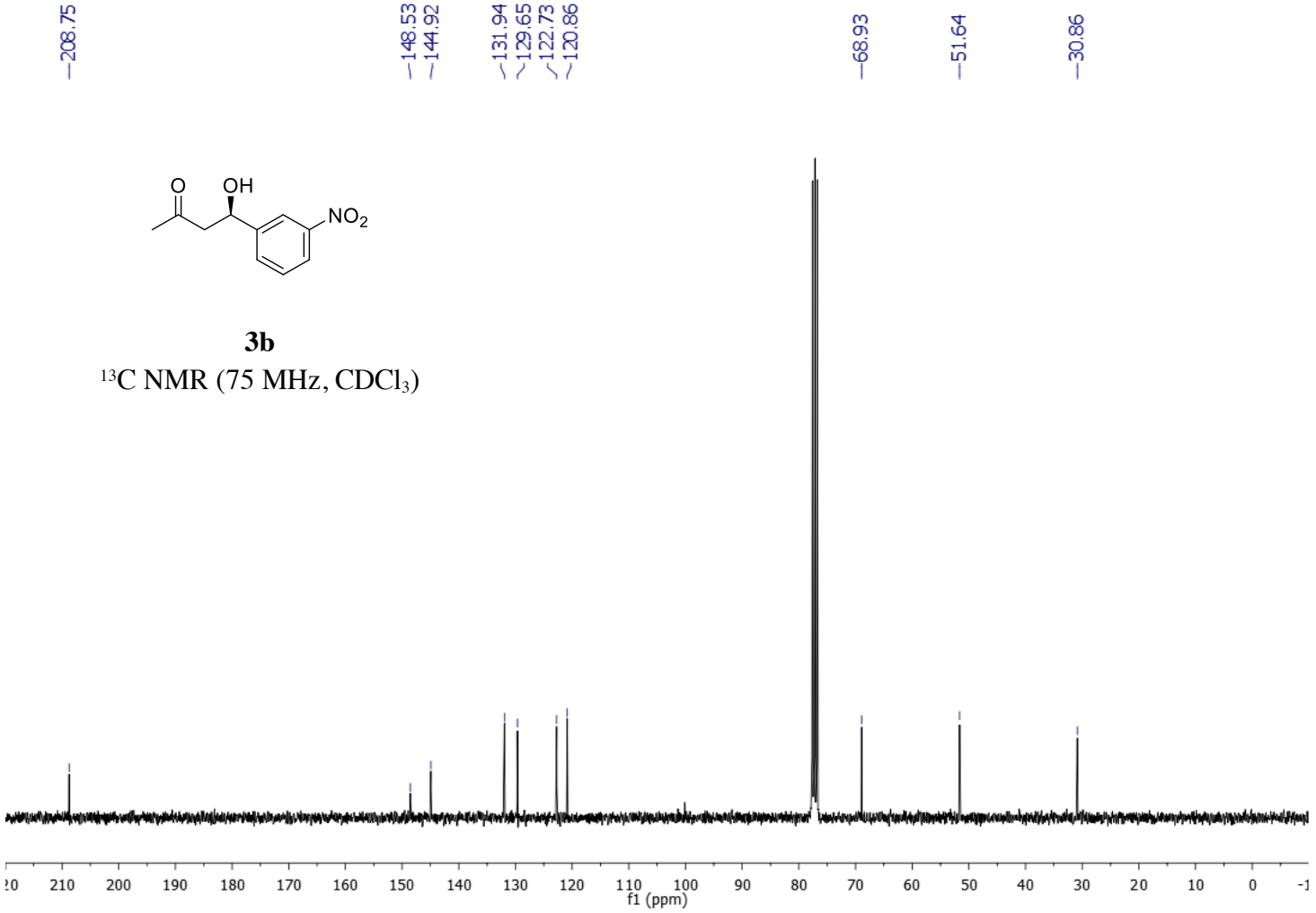
3a

^{13}C NMR (75 MHz, $CDCl_3$)



3b

^{1}H NMR (300 MHz, $CDCl_3$)

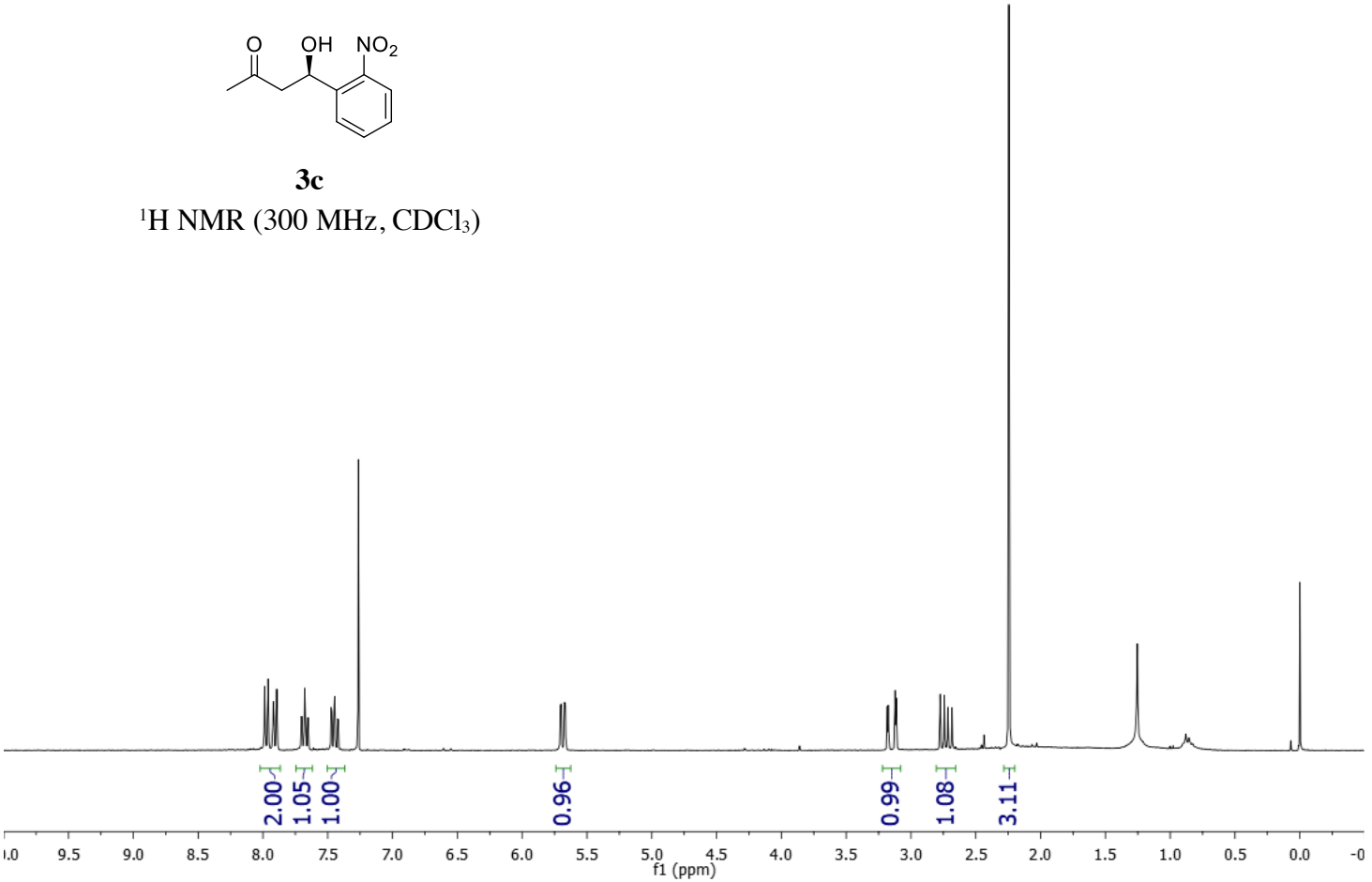


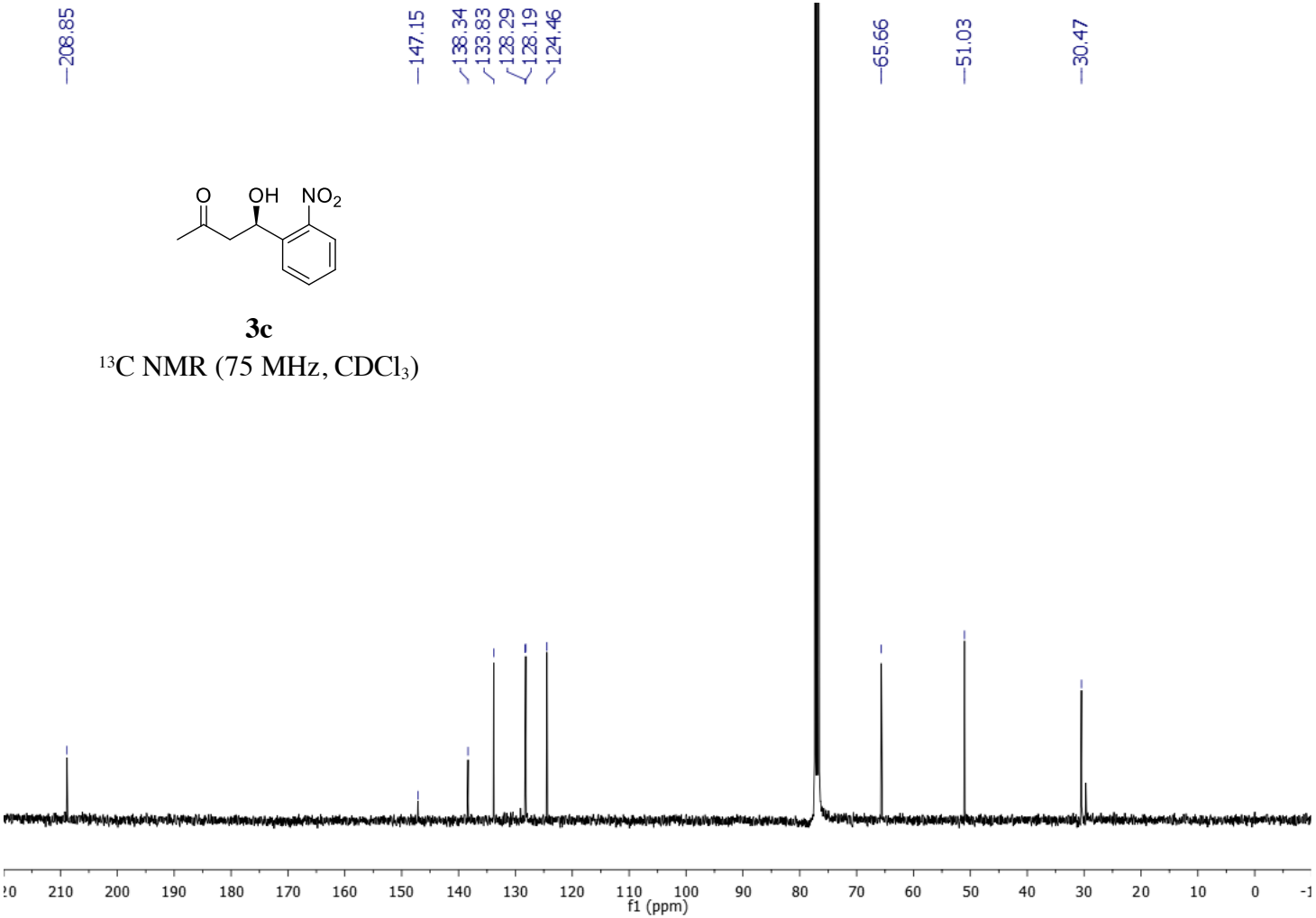
3b

^{13}C NMR (75 MHz, $CDCl_3$)

3c

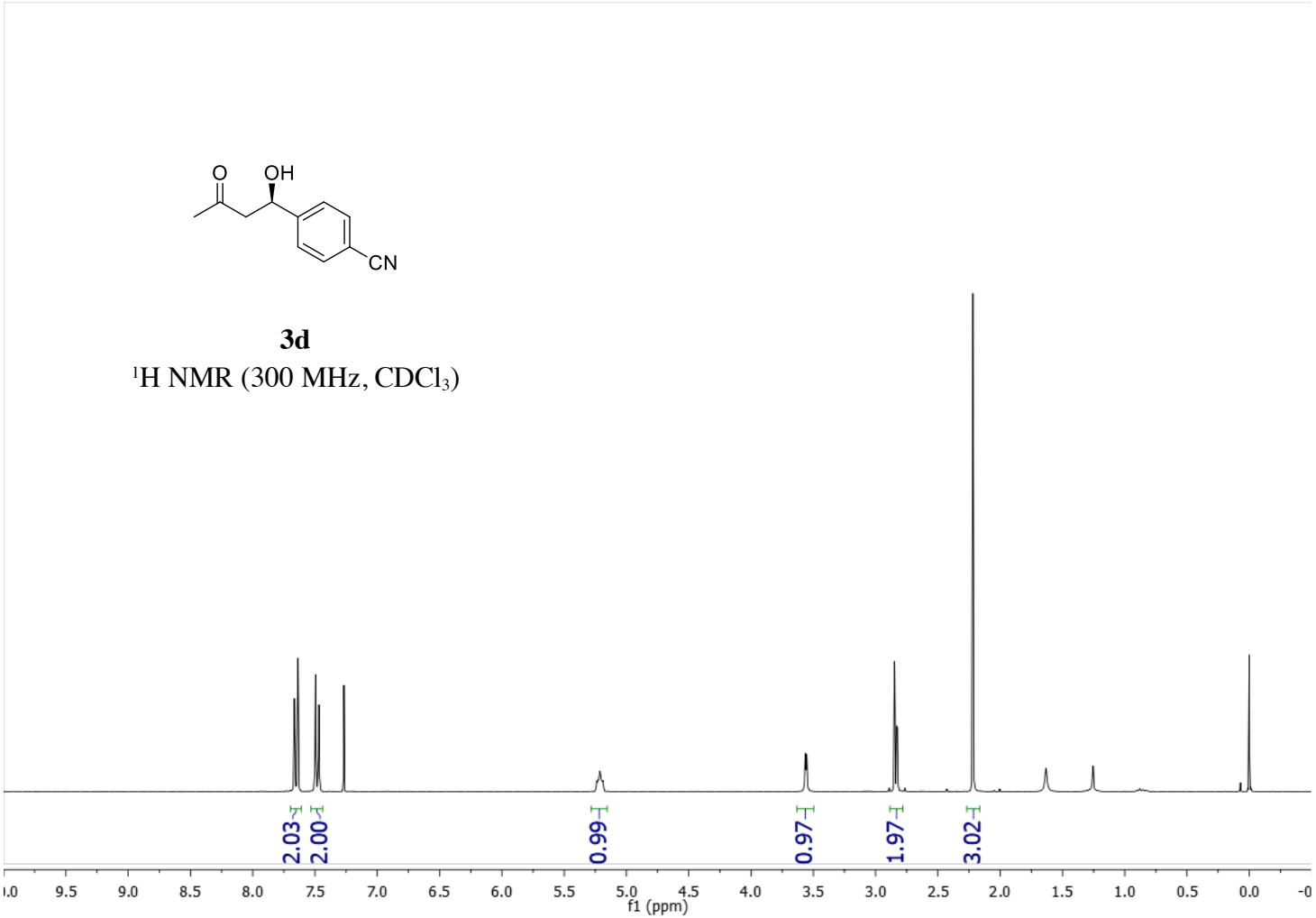
^{1}H NMR (300 MHz, $CDCl_3$)

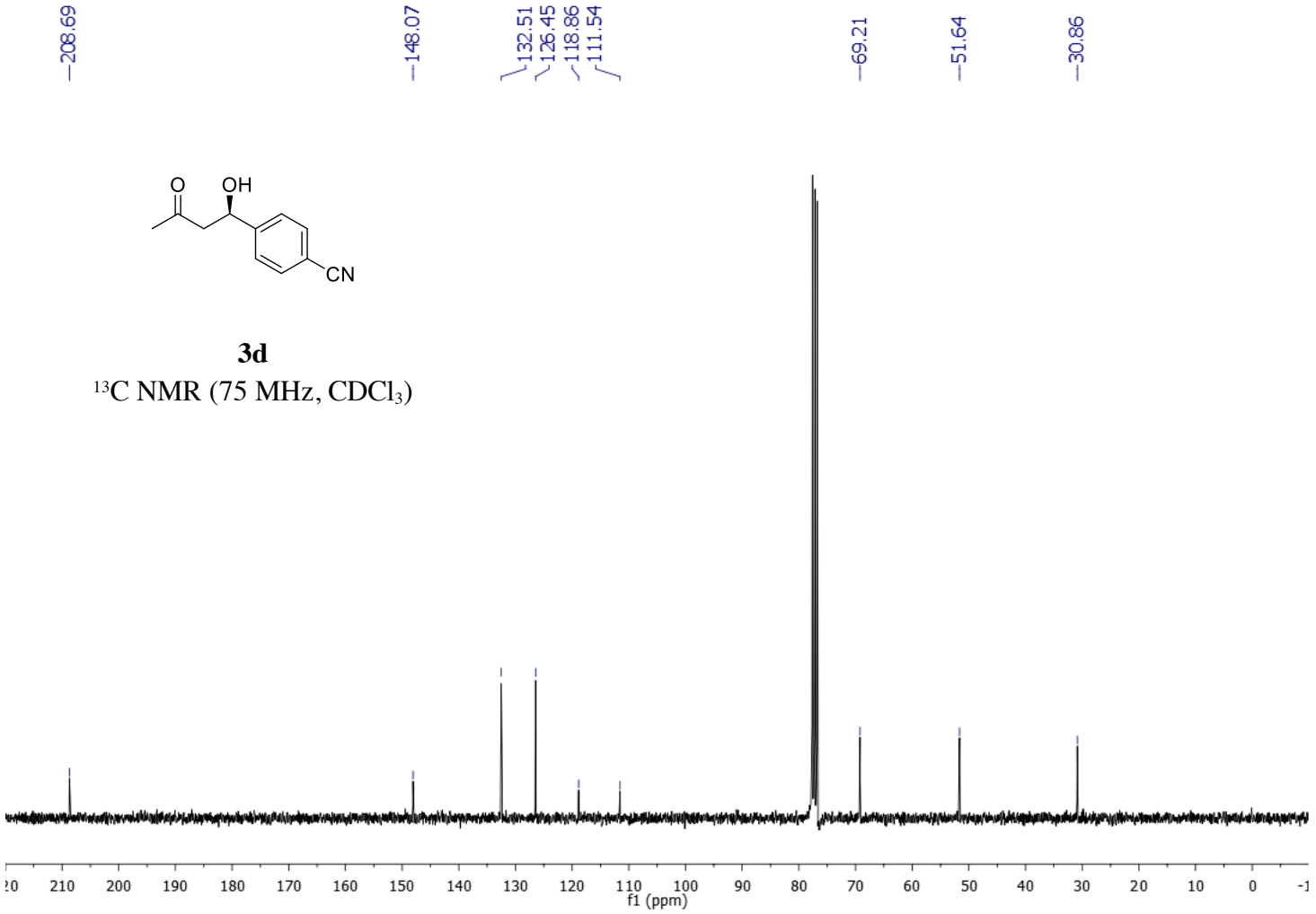




3c

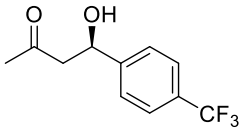
^{13}C NMR (75 MHz, $CDCl_3$)


O
OH
CN
3d
1H NMR (300 MHz, CDCl3)
2.03
2.00
0.99
0.97
1.97
3.02
f1 (ppm)



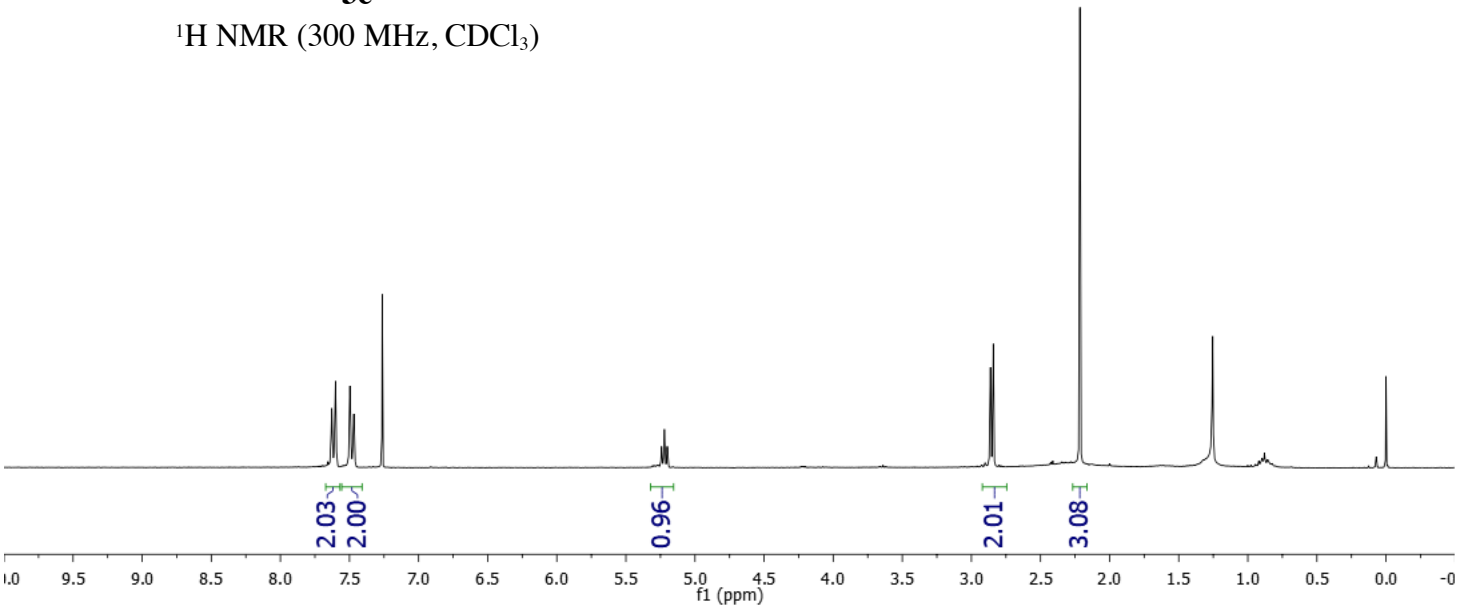
3d

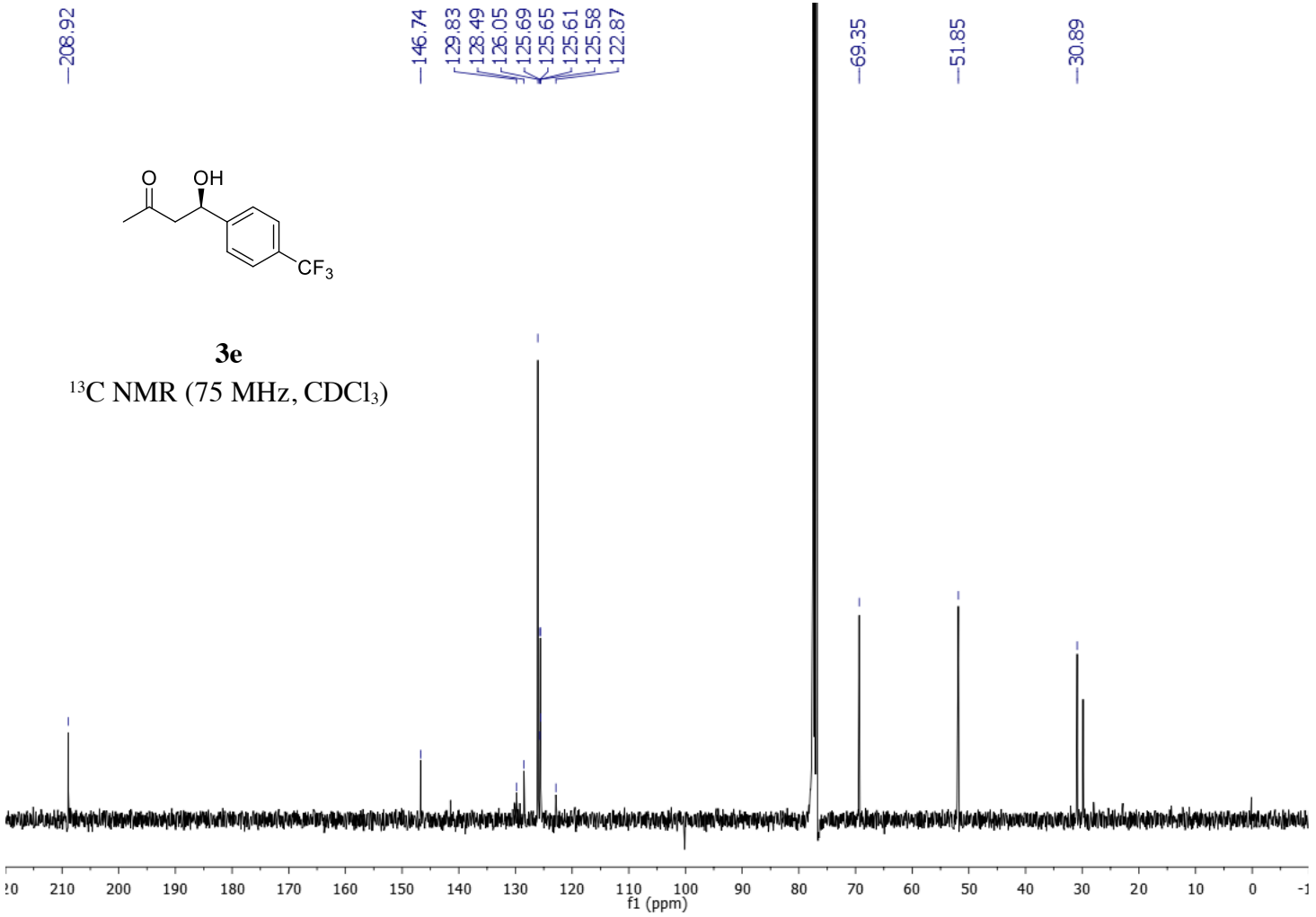
^{13}C NMR (75 MHz, $CDCl_3$)



3e

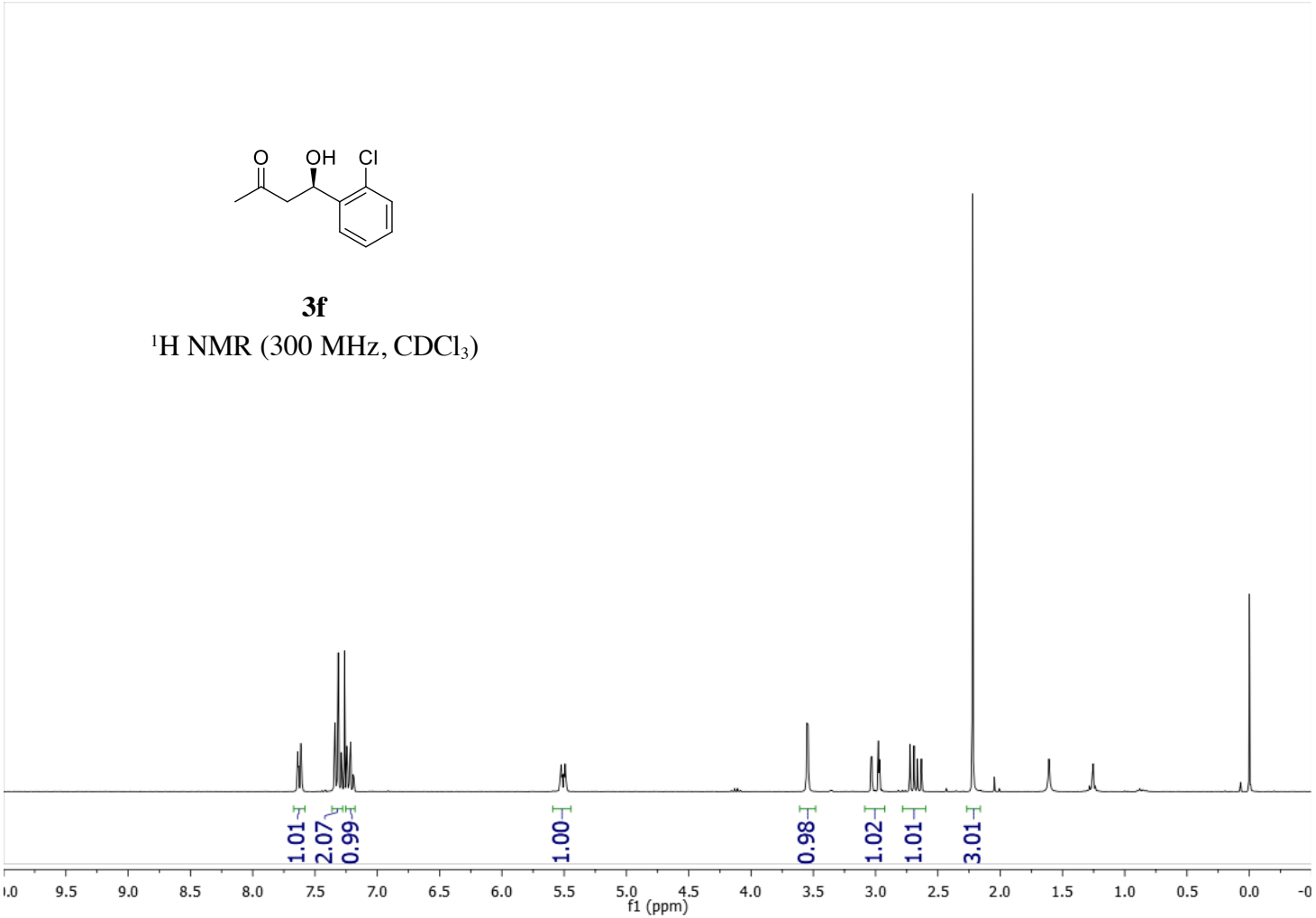
^{1}H NMR (300 MHz, $CDCl_3$)





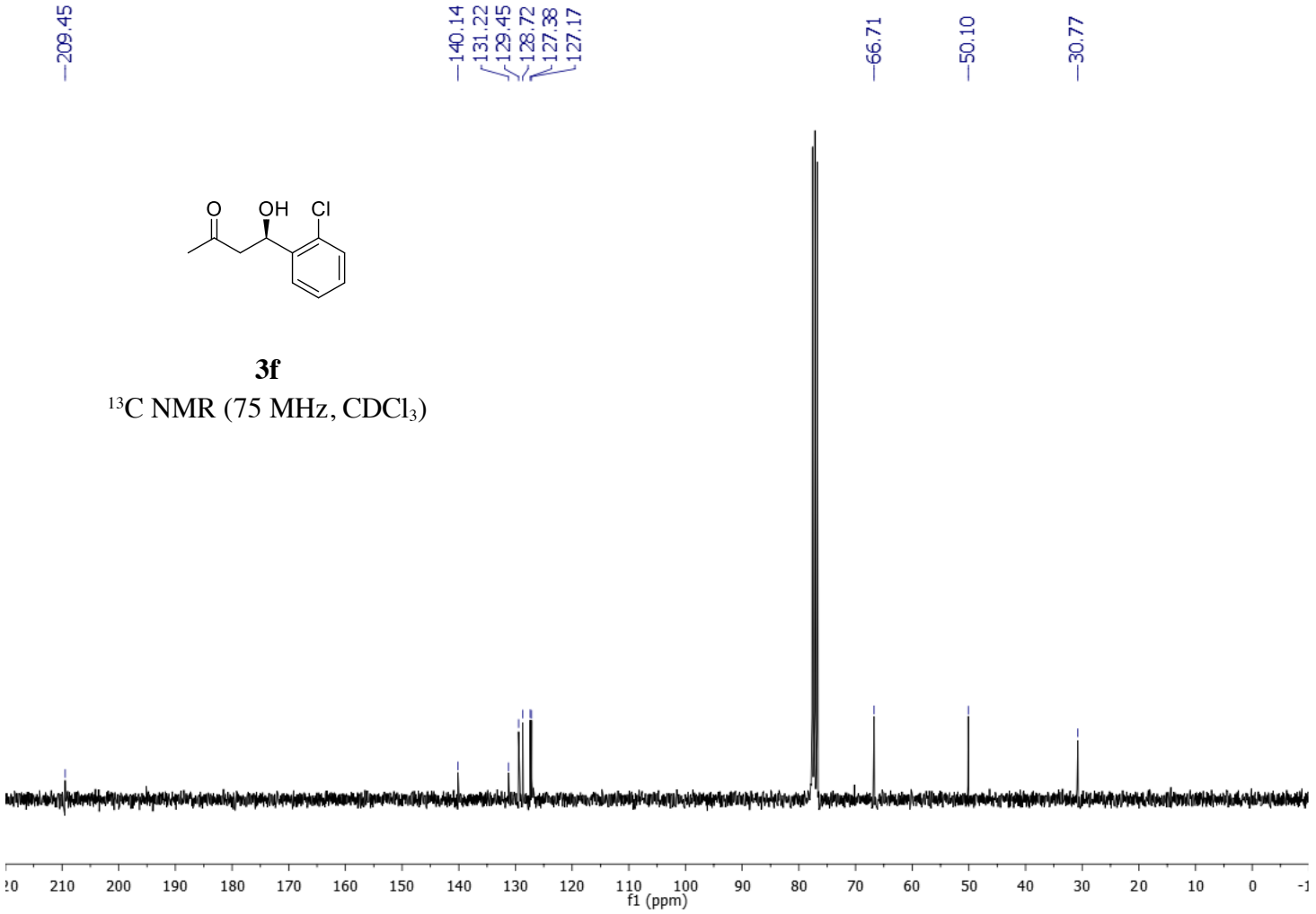
3e

^{13}C NMR (75 MHz, $CDCl_3$)



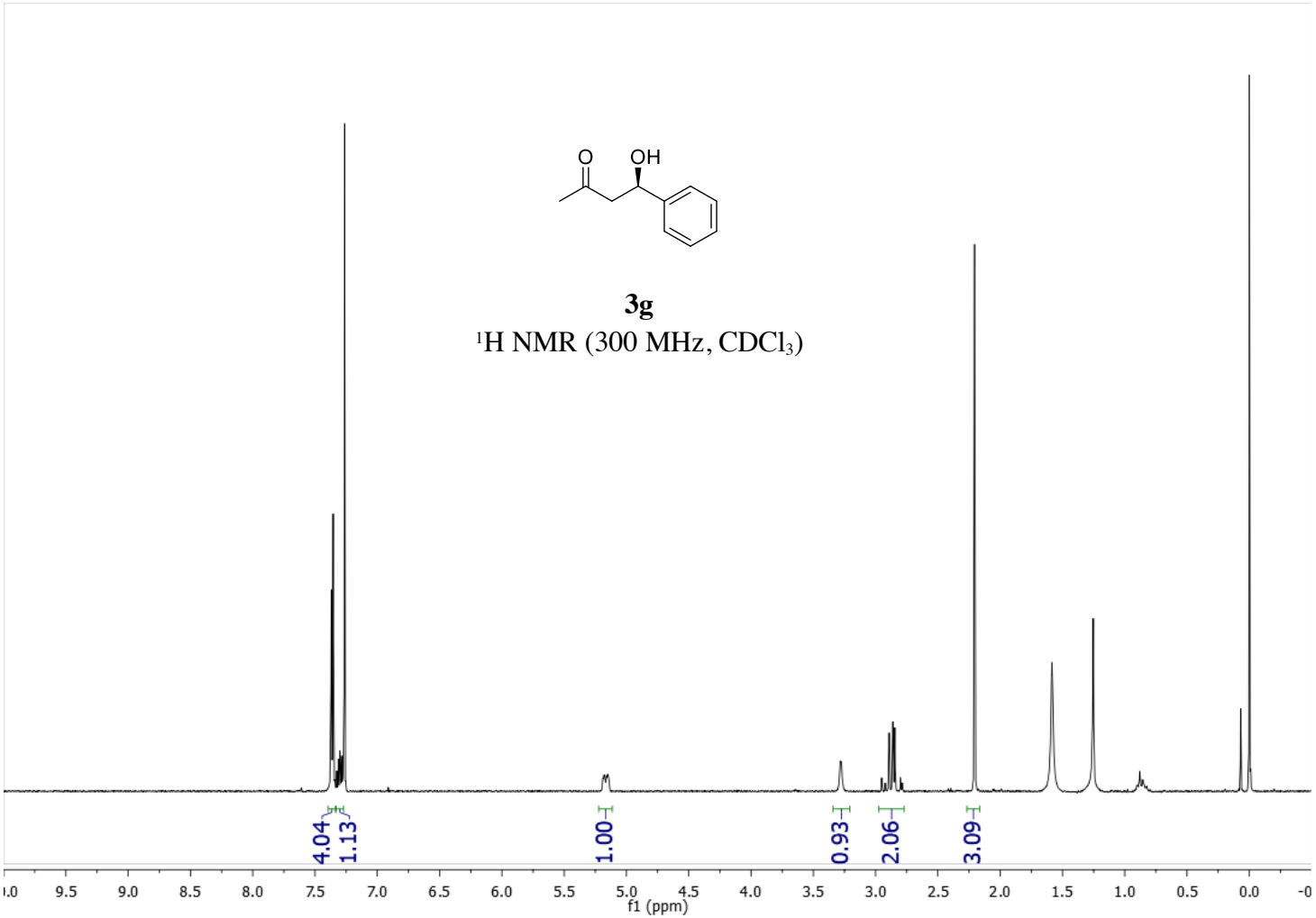
3f

^{1}H NMR (300 MHz, $CDCl_3$)



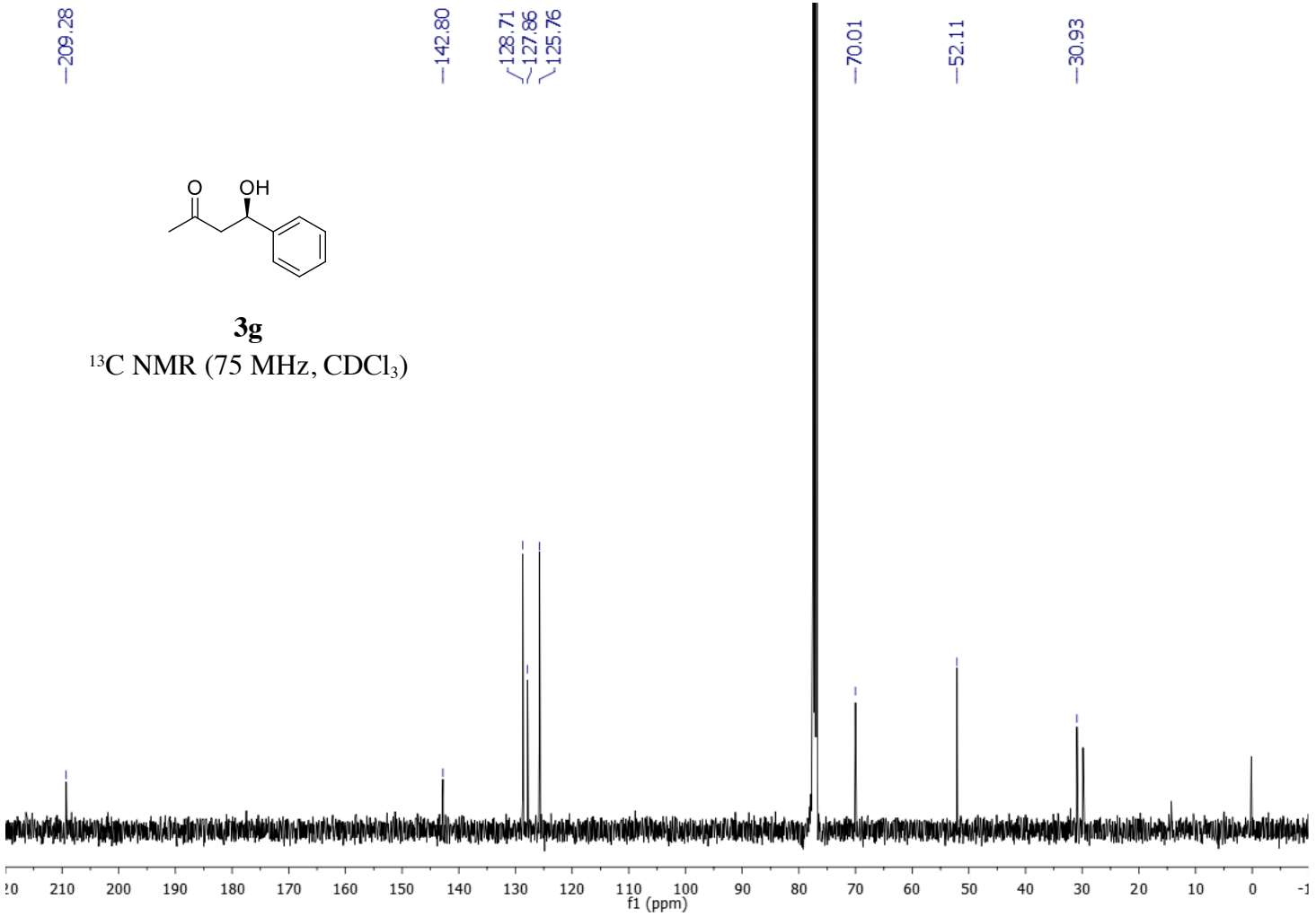
3f

^{13}C NMR (75 MHz, $CDCl_3$)



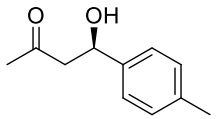
3g

^{1}H NMR (300 MHz, $CDCl_3$)



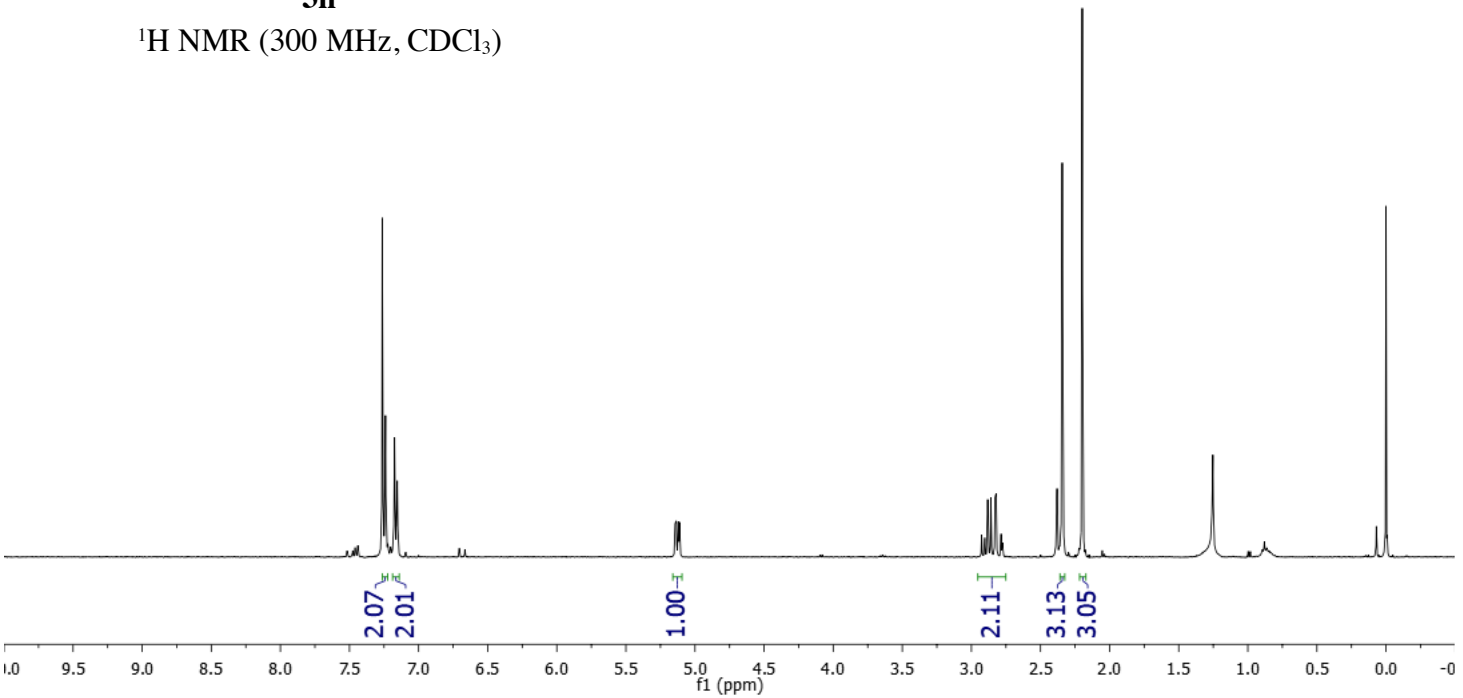
3g

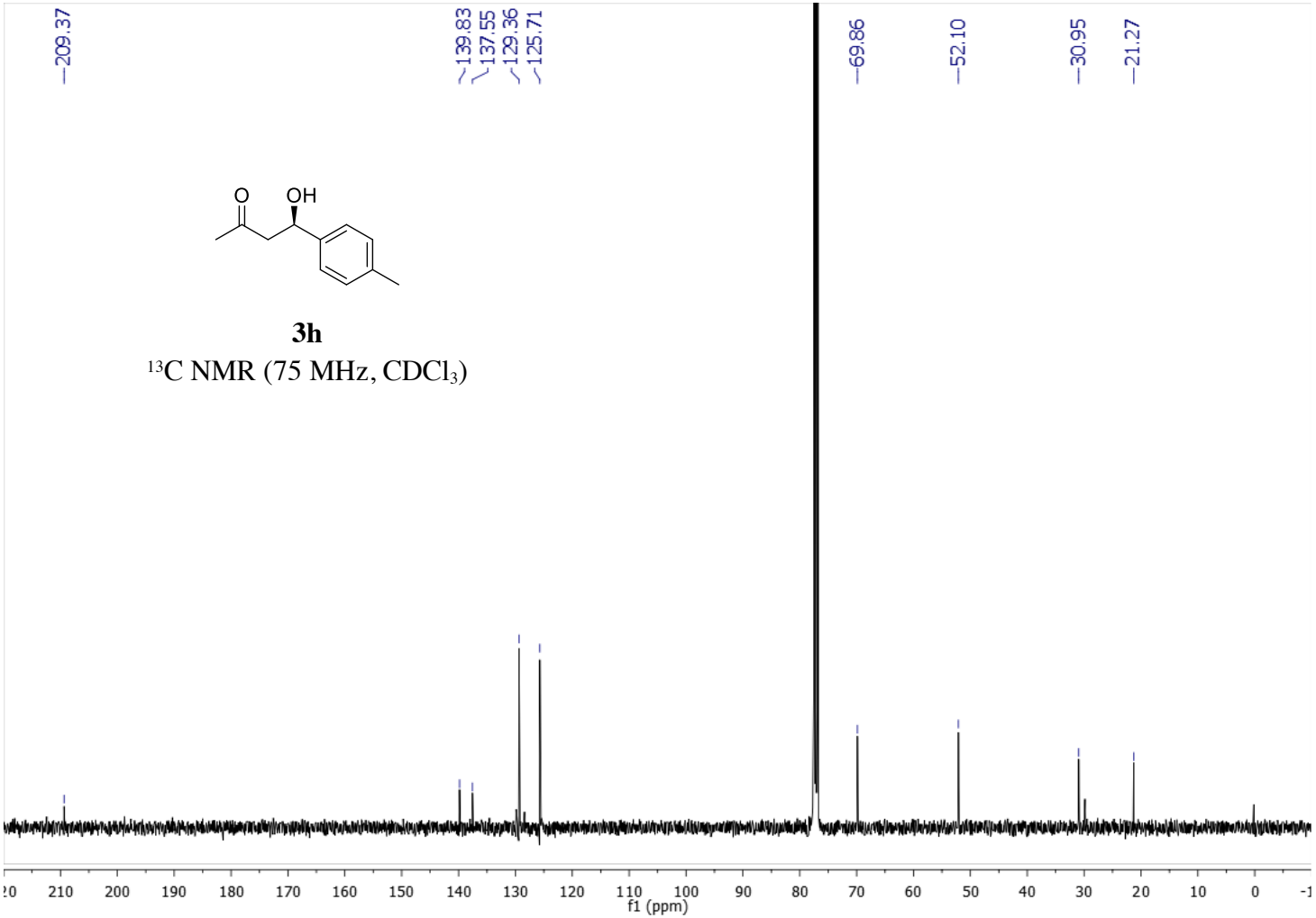
^{13}C NMR (75 MHz, $CDCl_3$)



3h

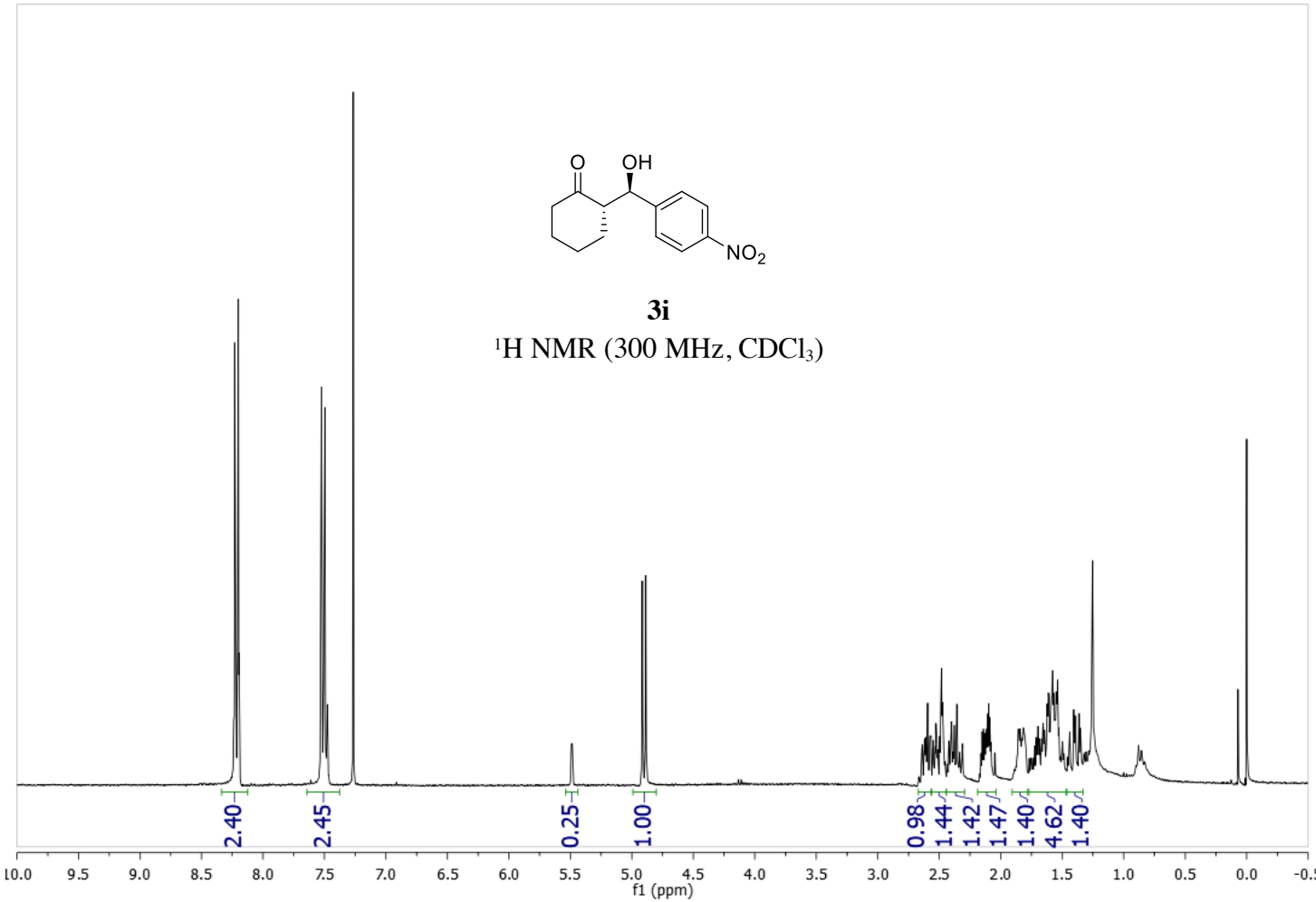
^{1}H NMR (300 MHz, $CDCl_3$)





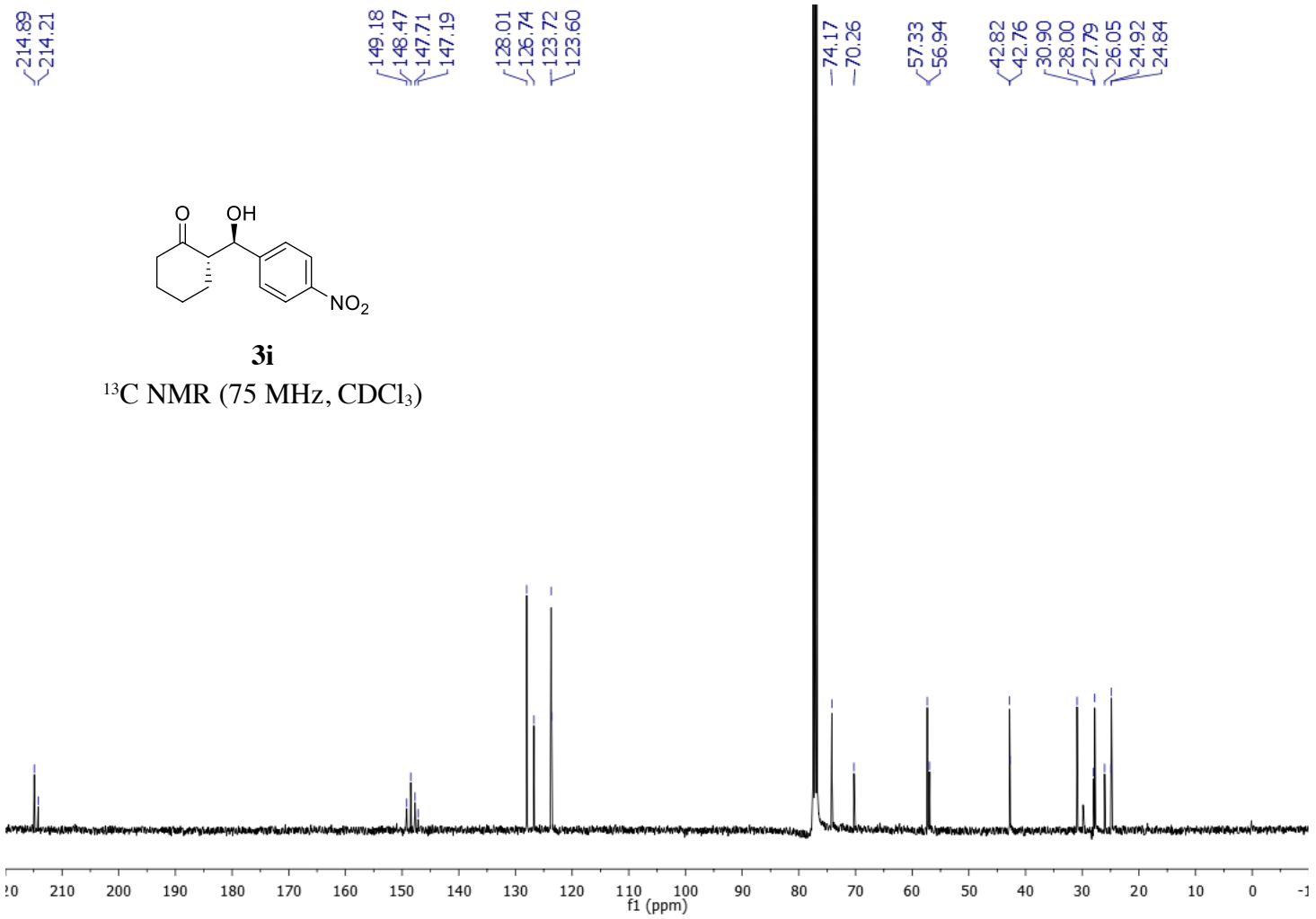
3h

^{13}C NMR (75 MHz, $CDCl_3$)



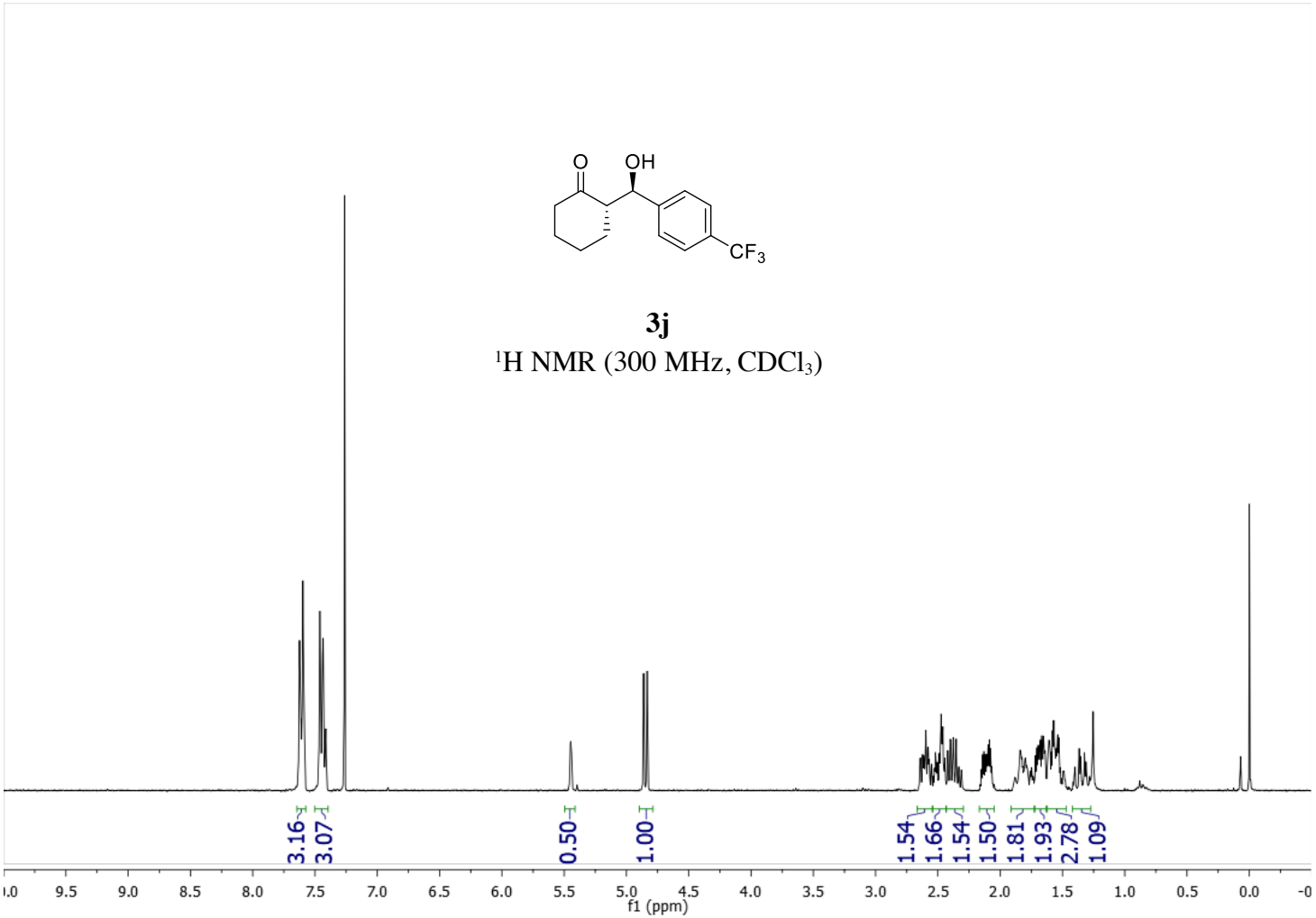
3i

^{1}H NMR (300 MHz, $CDCl_3$)



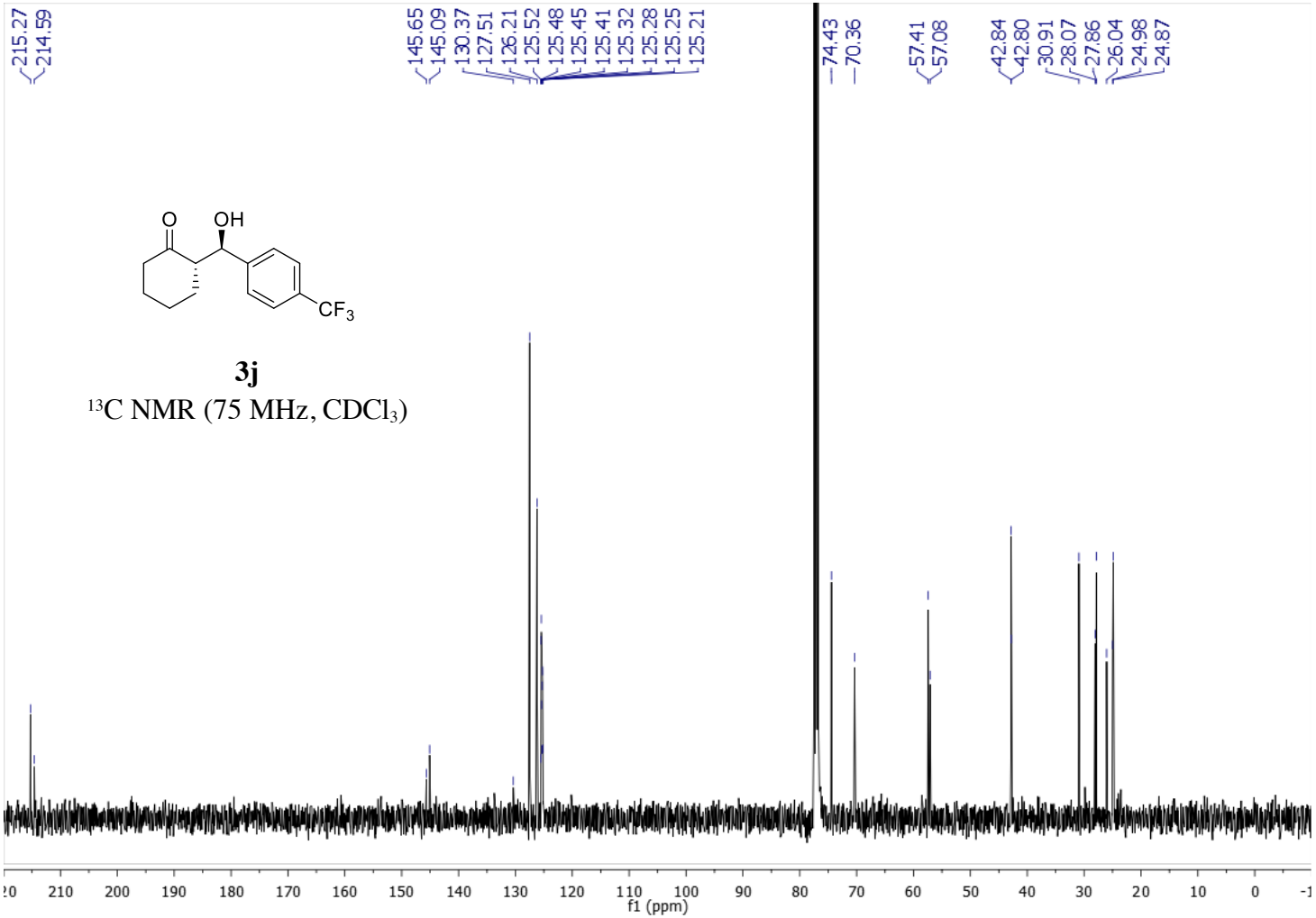
3i

^{13}C NMR (75 MHz, $CDCl_3$)



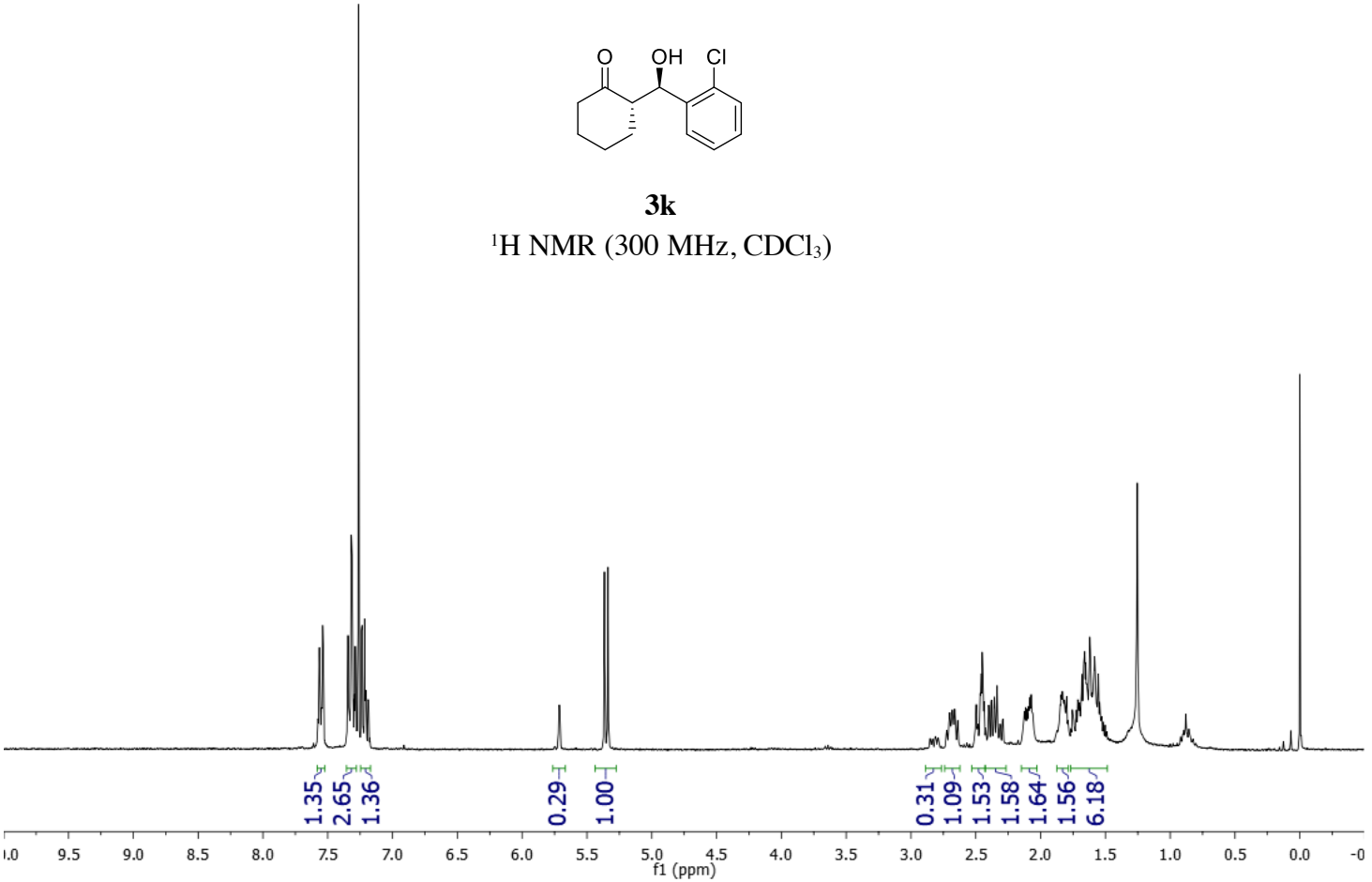
3j

^{1}H NMR (300 MHz, $CDCl_3$)



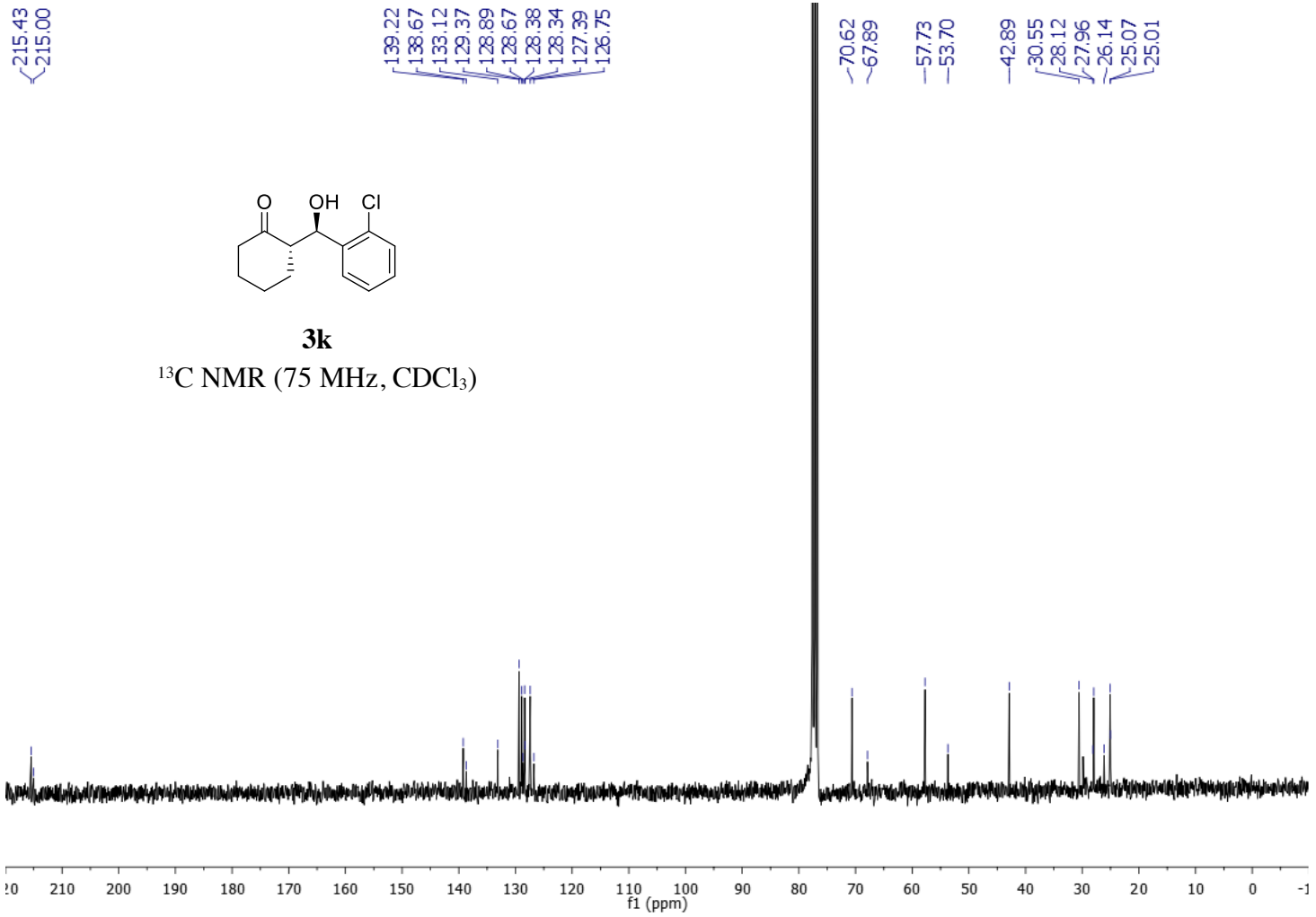
3j

^{13}C NMR (75 MHz, $CDCl_3$)



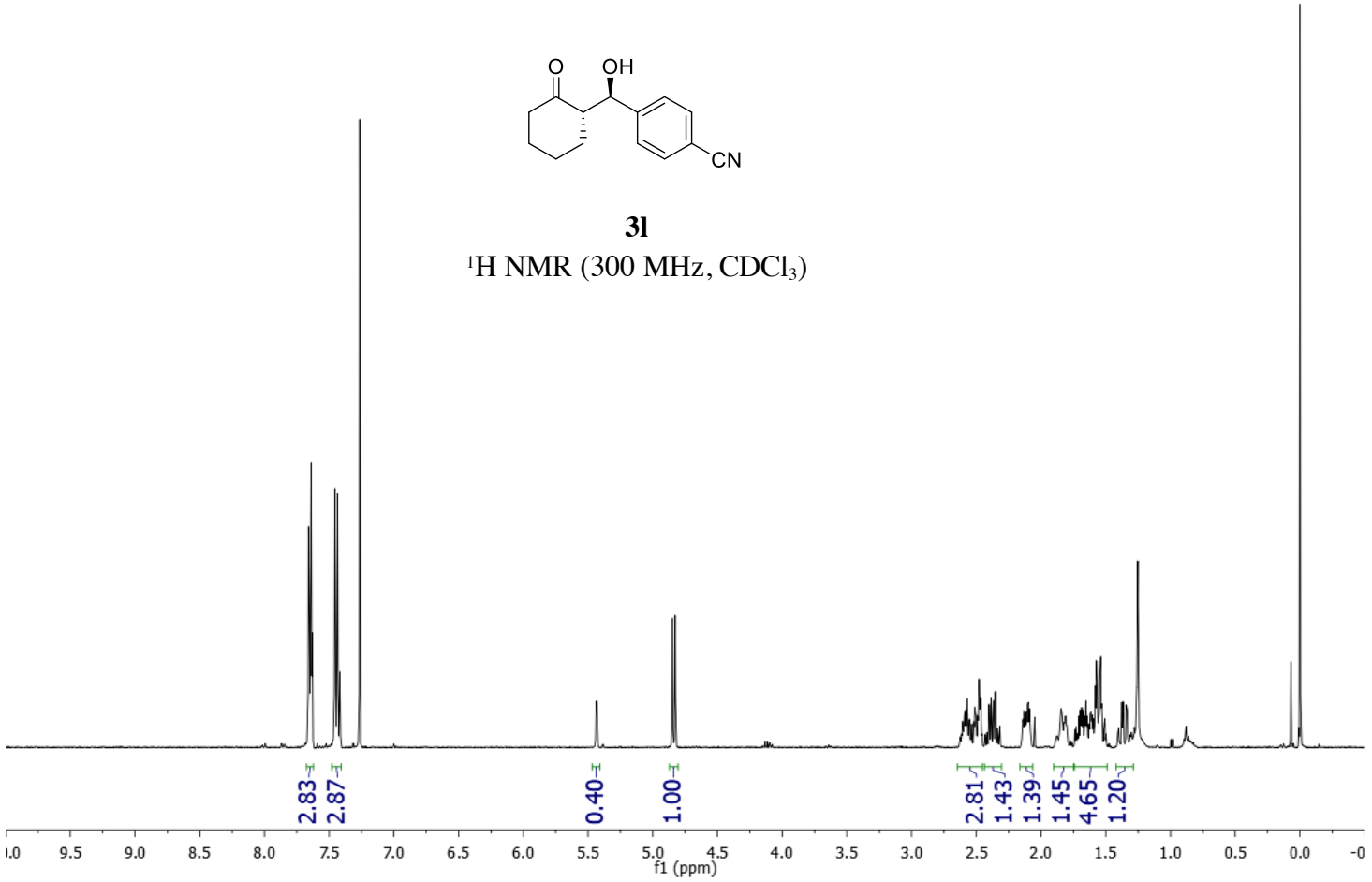
3k

^{1}H NMR (300 MHz, $CDCl_3$)



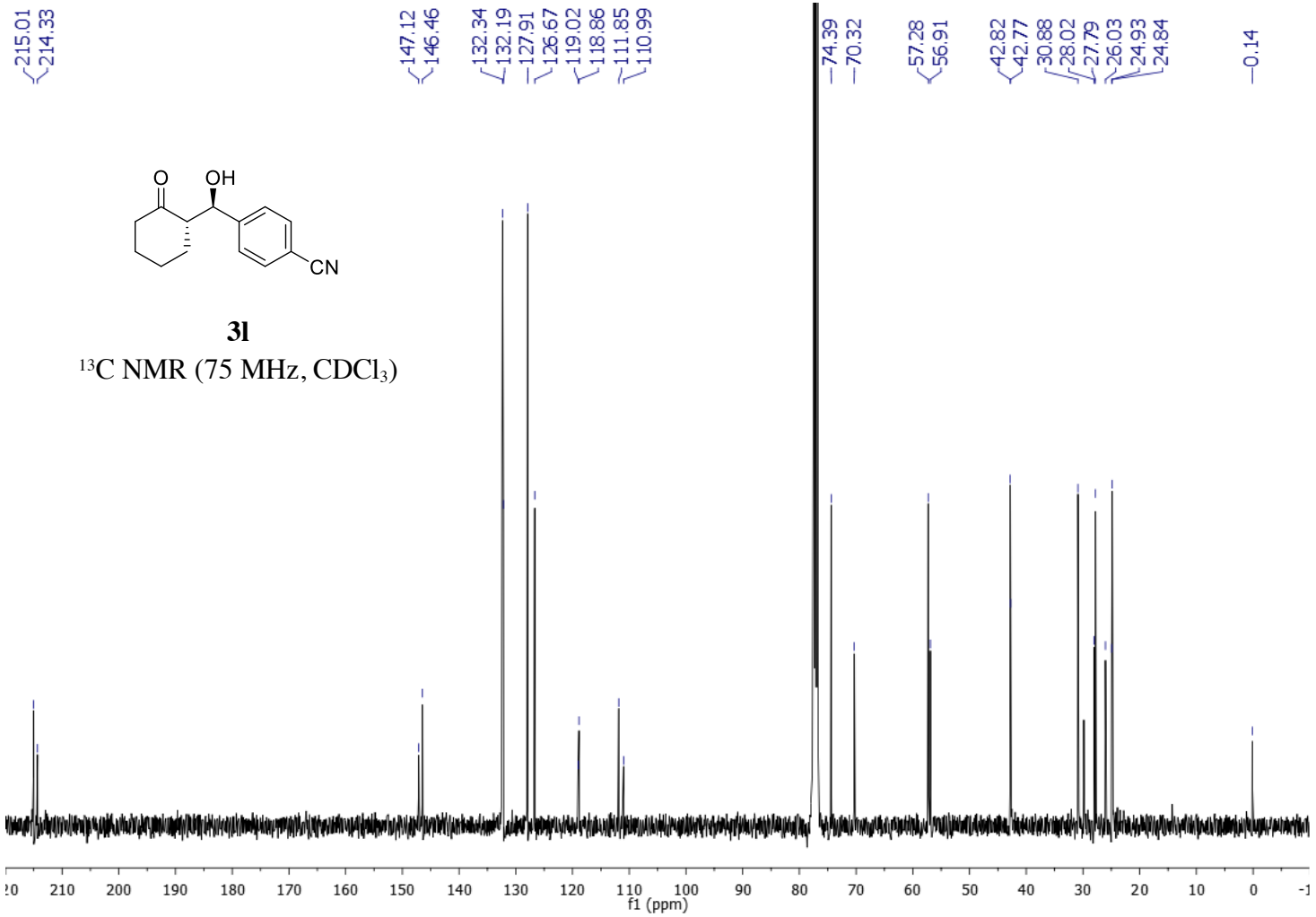
3k

^{13}C NMR (75 MHz, $CDCl_3$)



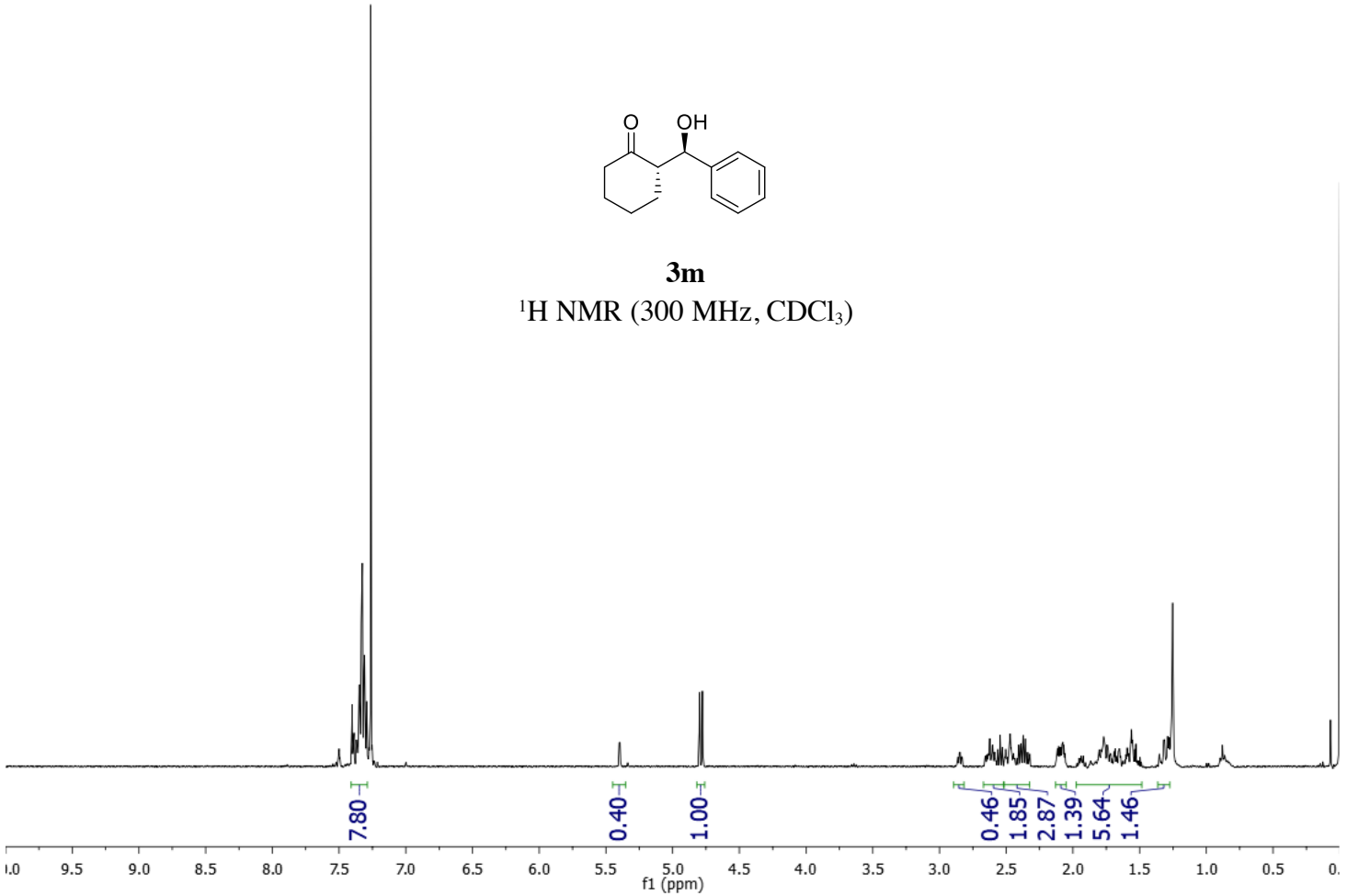
3l

^{1}H NMR (300 MHz, $CDCl_3$)



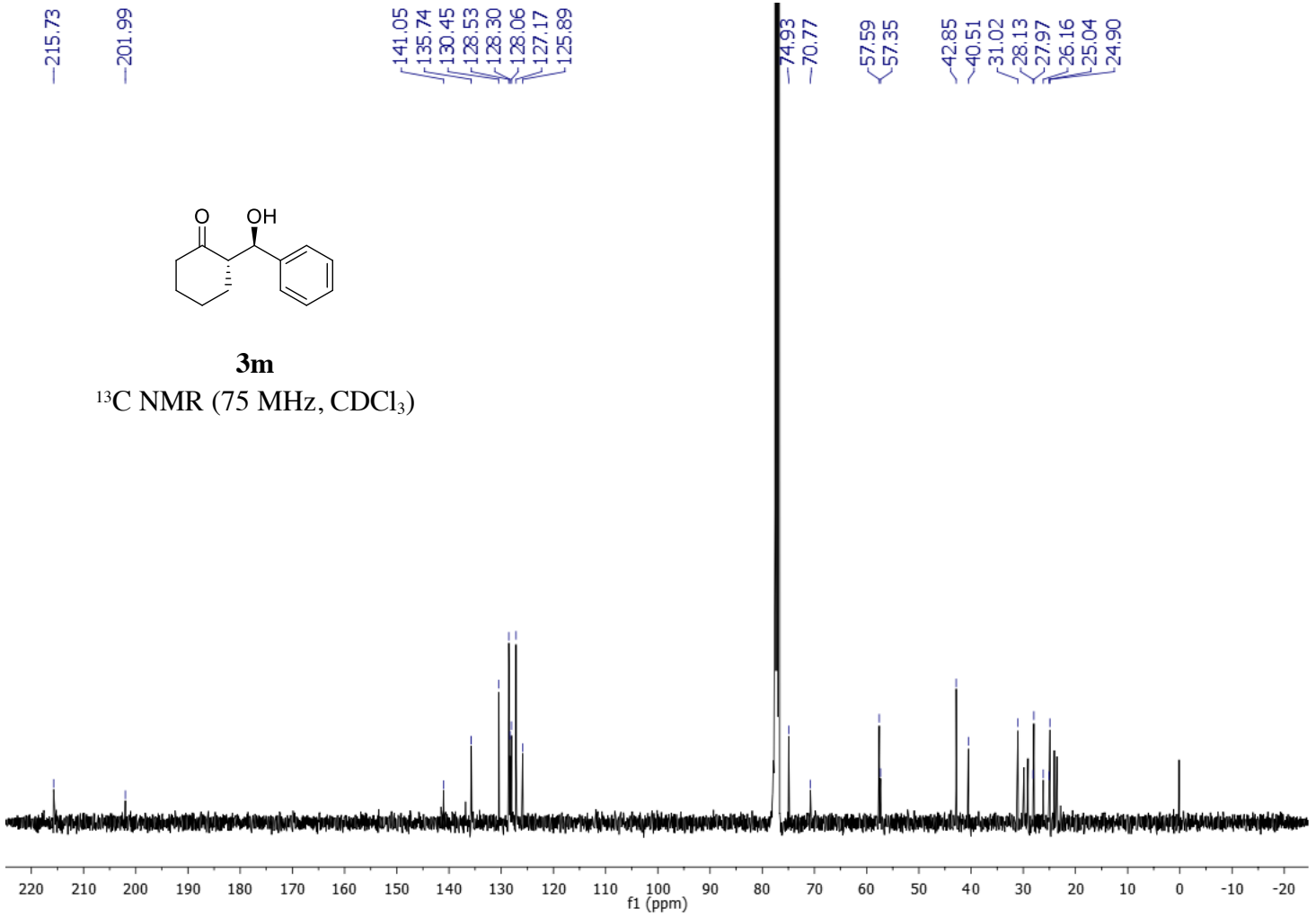
3l

^{13}C NMR (75 MHz, $CDCl_3$)



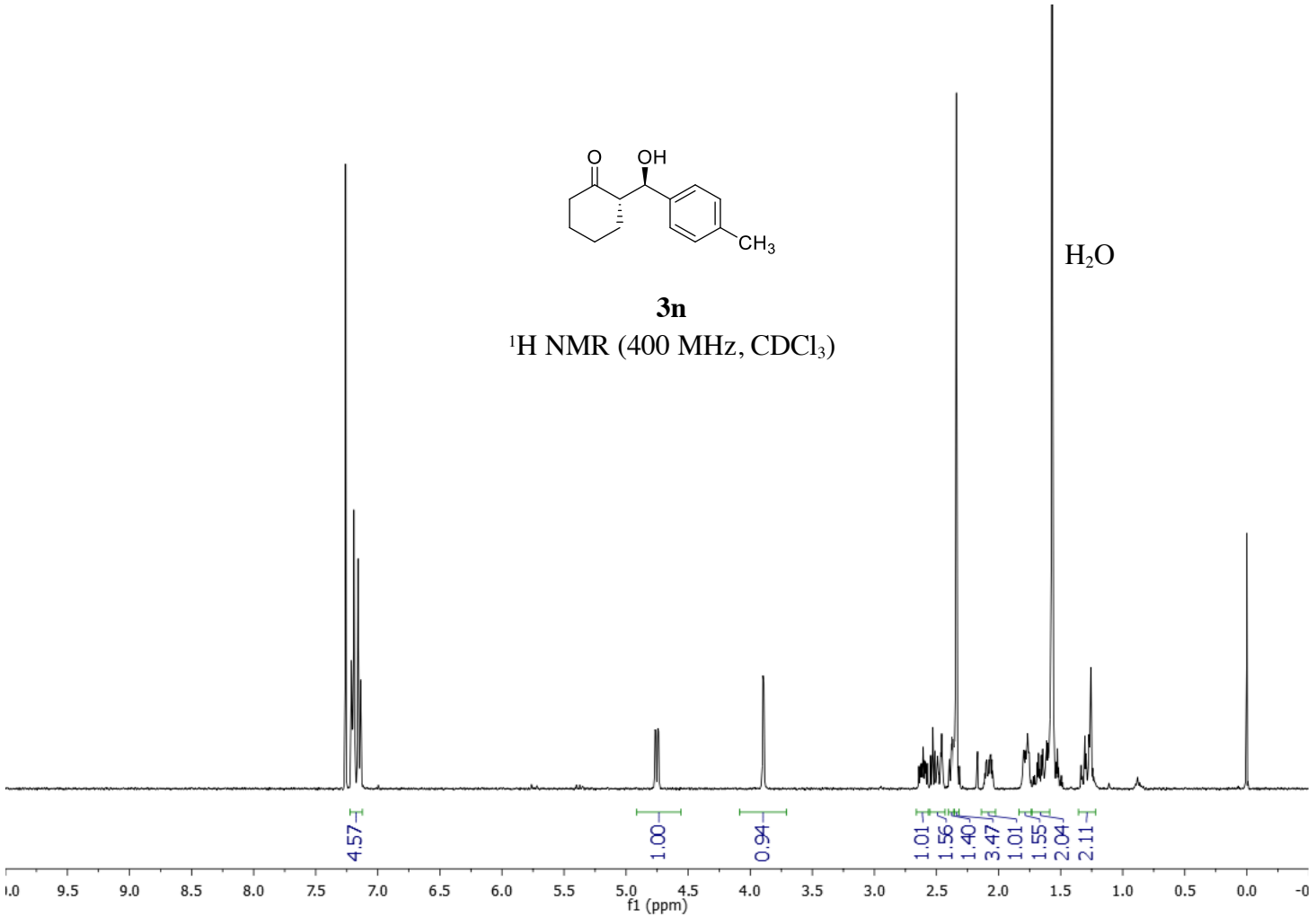
3m

^{1}H NMR (300 MHz, $CDCl_3$)



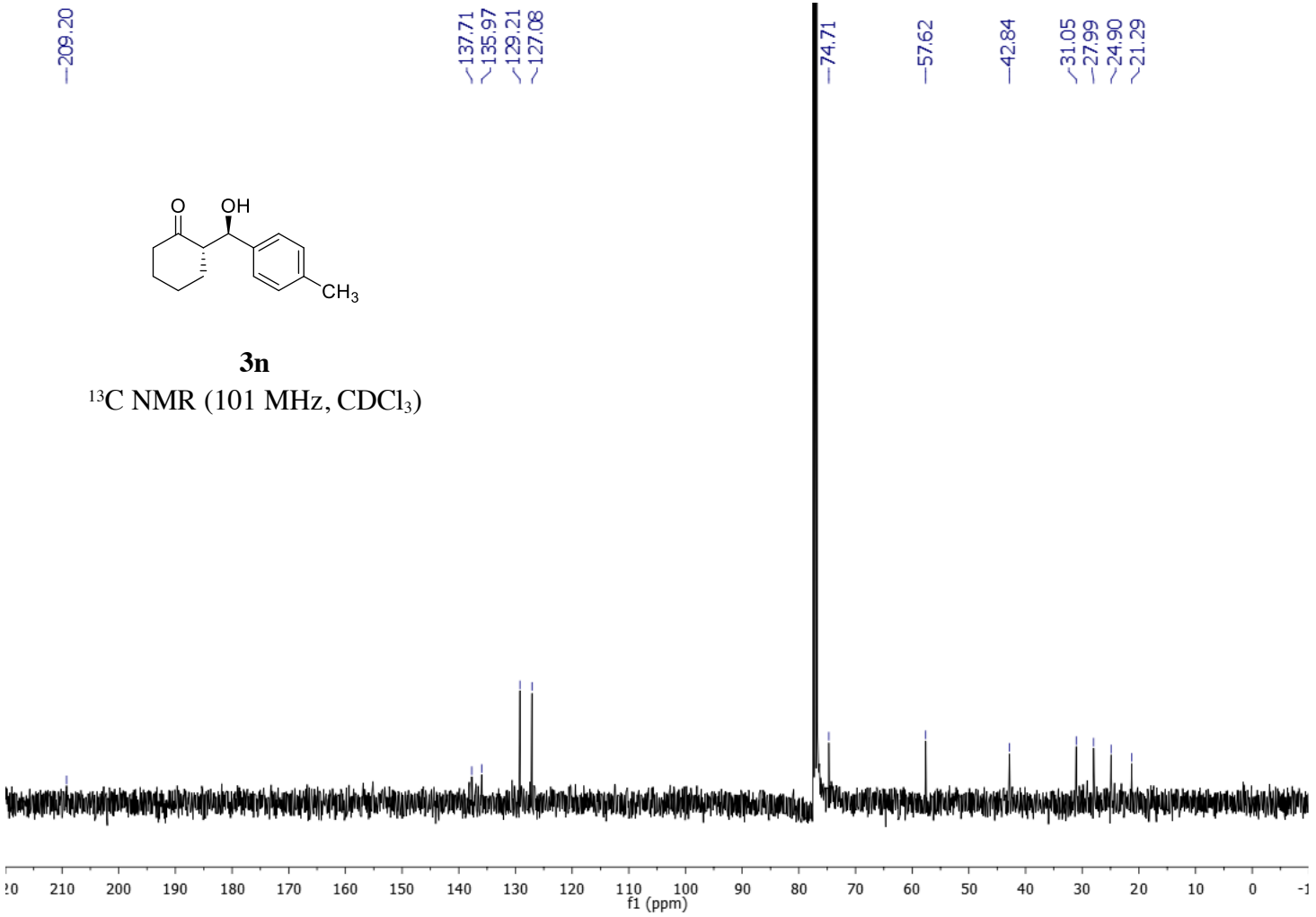
3m

^{13}C NMR (75 MHz, $CDCl_3$)



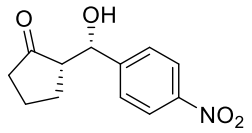
3n

^{1}H NMR (400 MHz, $CDCl_3$)



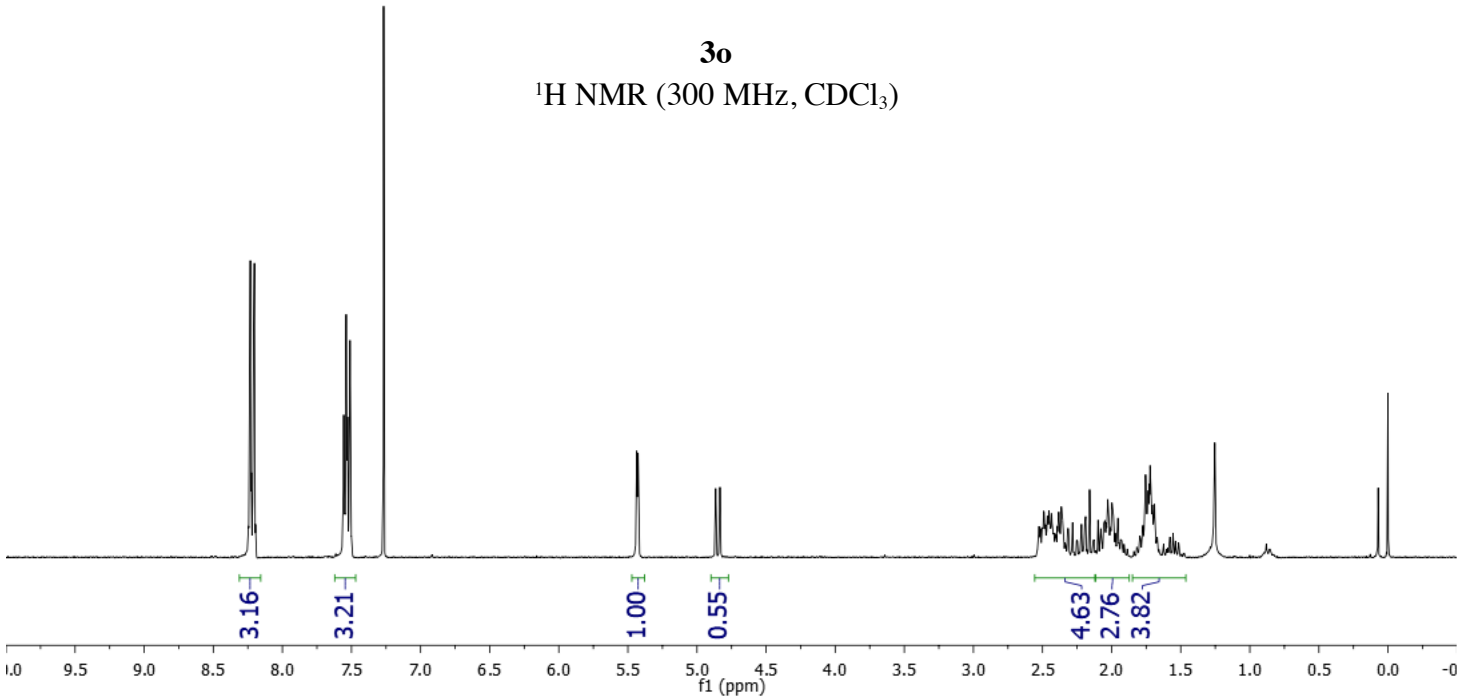
3n

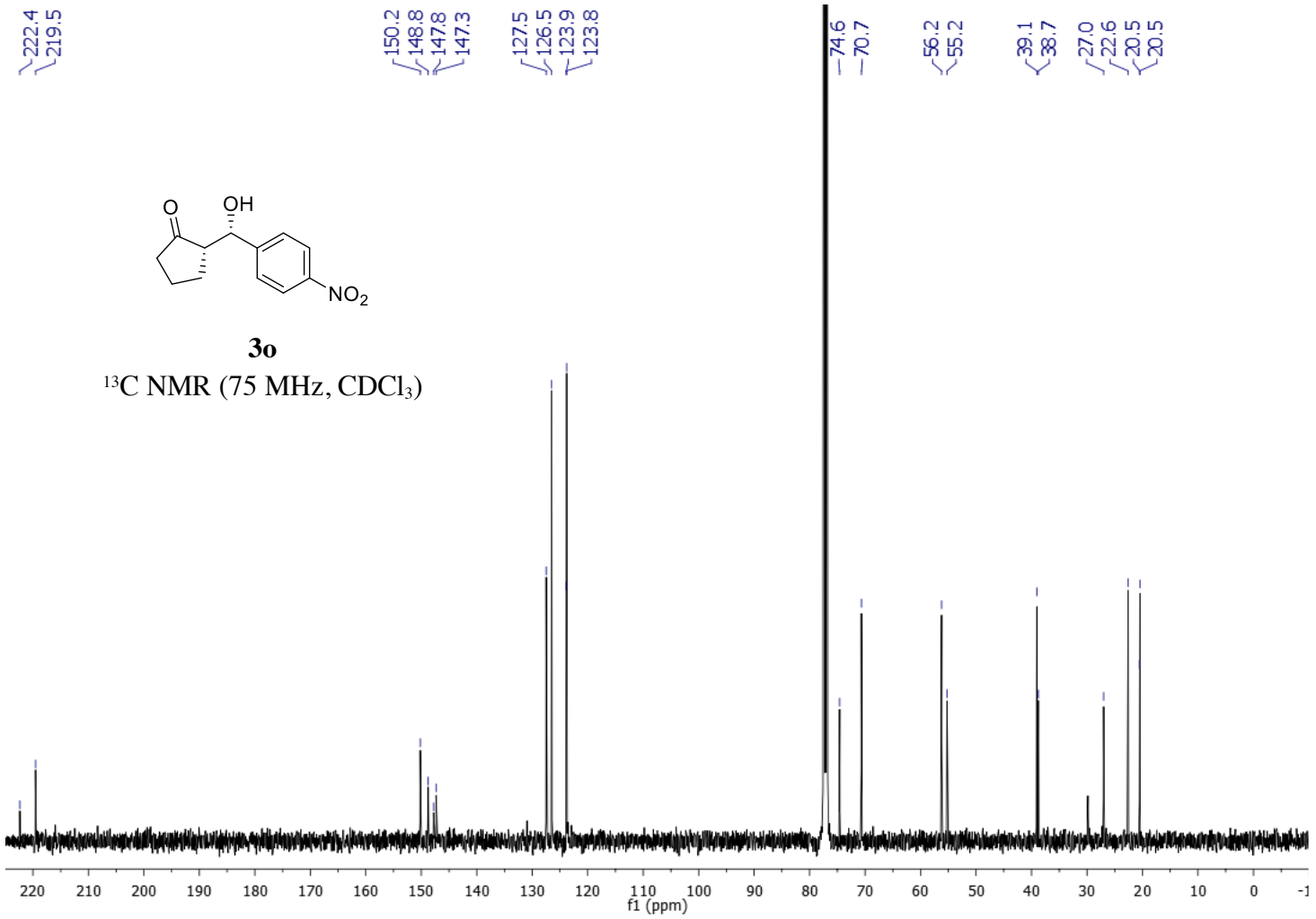
^{13}C NMR (101 MHz, $CDCl_3$)



3o

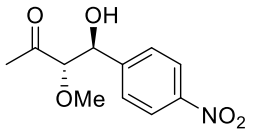
^{1}H NMR (300 MHz, $CDCl_3$)





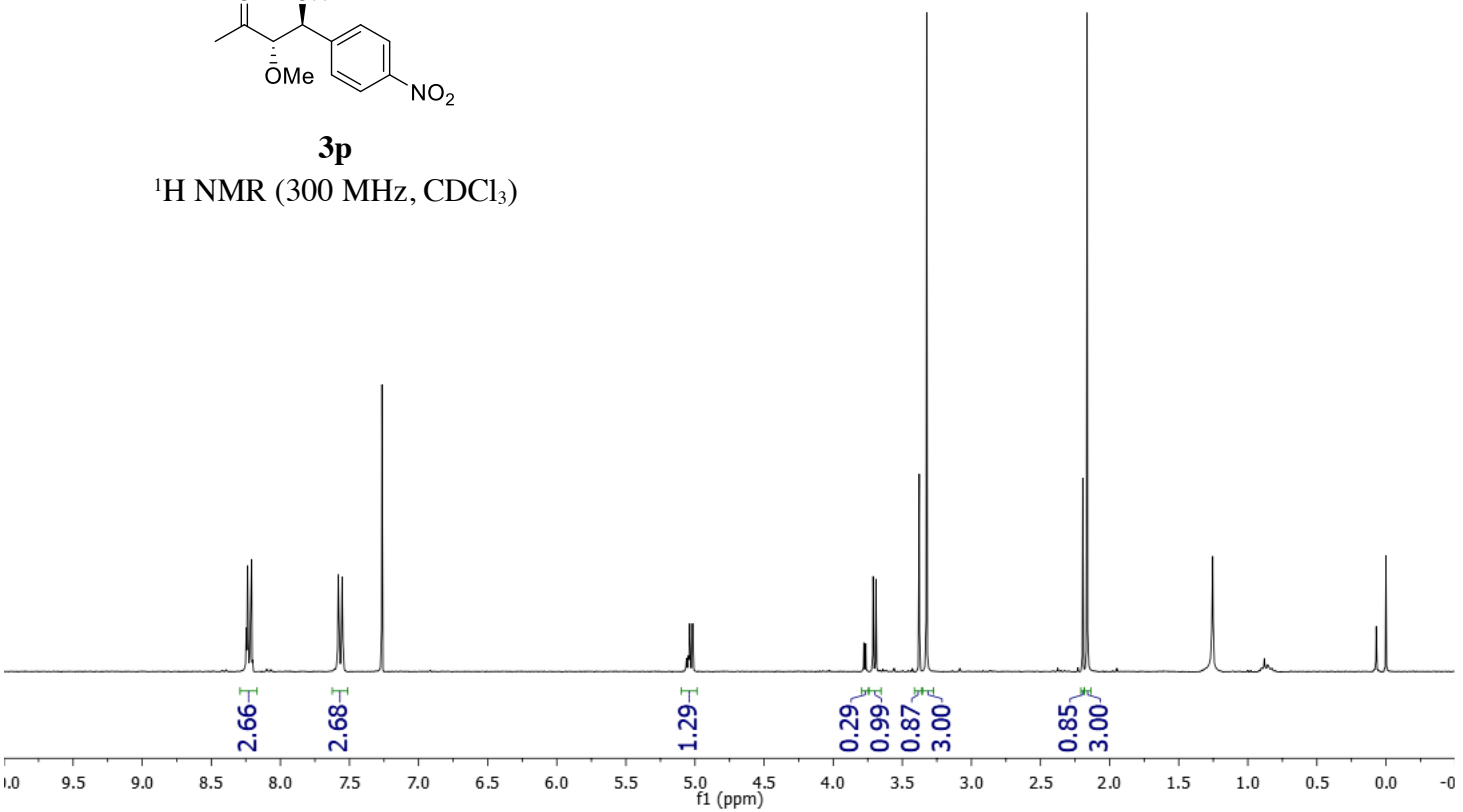
3o

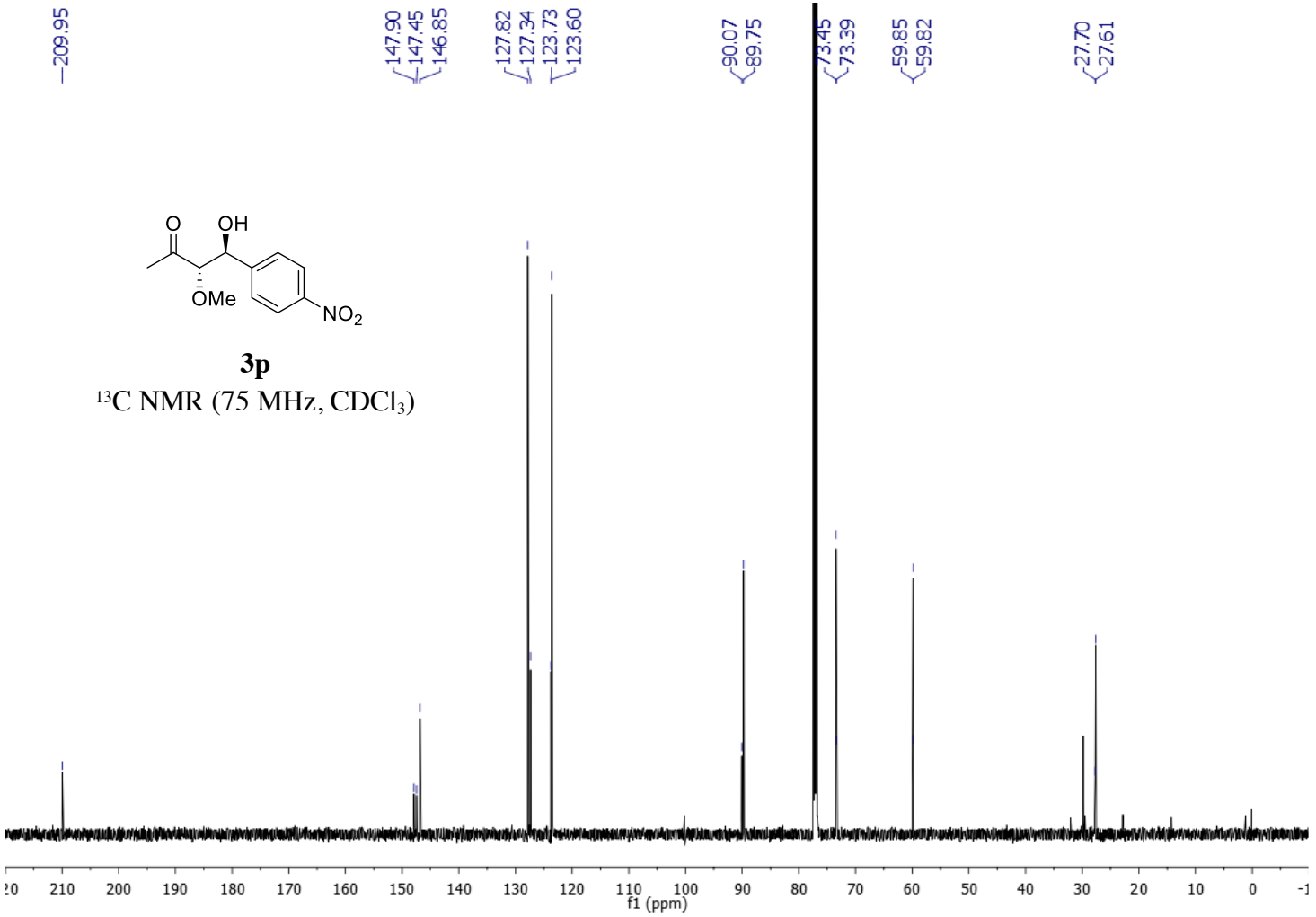
^{13}C NMR (75 MHz, $CDCl_3$)



3p

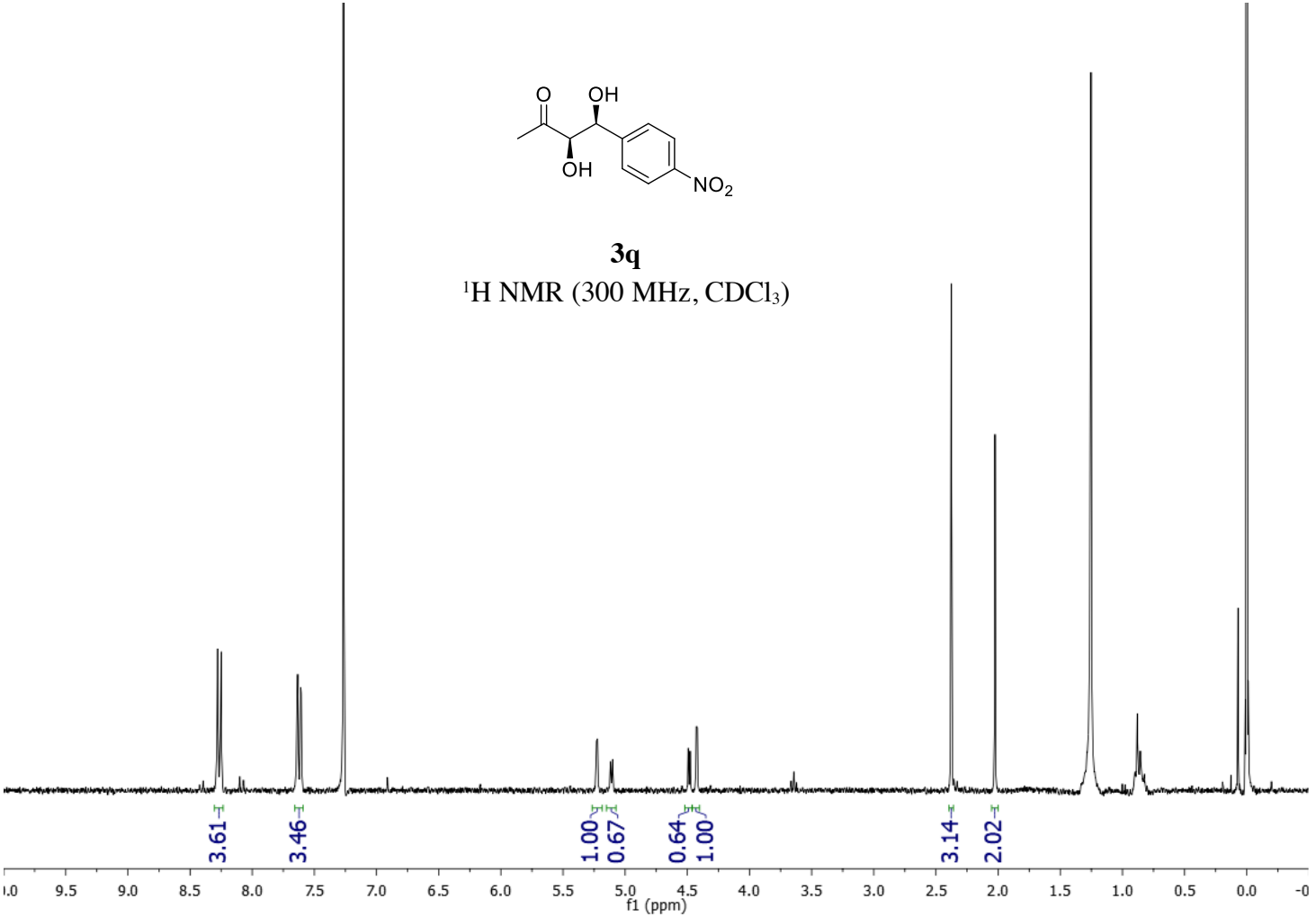
^{1}H NMR (300 MHz, $CDCl_3$)





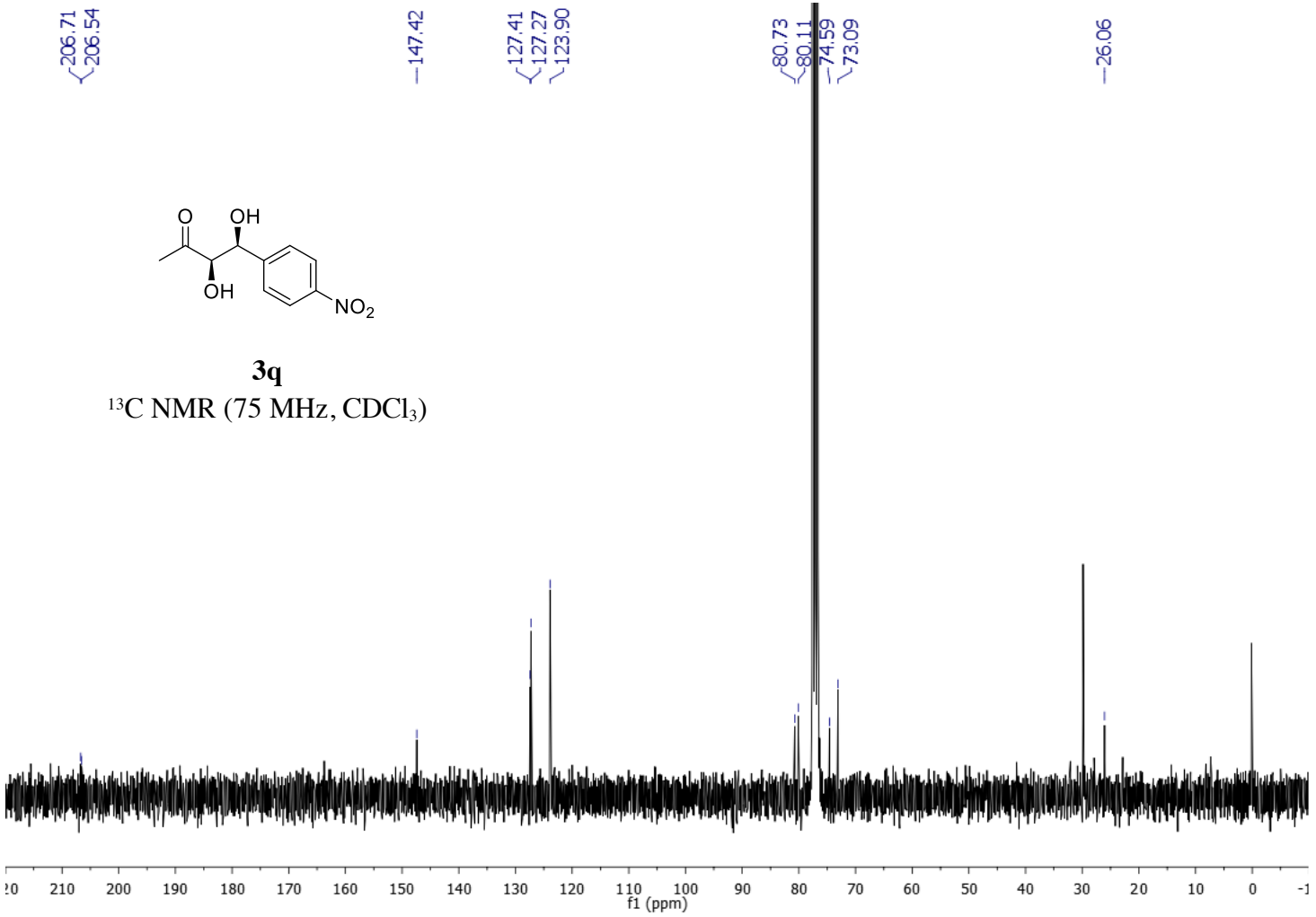
3p

^{13}C NMR (75 MHz, $CDCl_3$)



3q

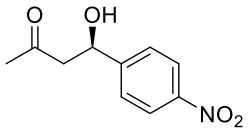
^{1}H NMR (300 MHz, $CDCl_3$)



3q

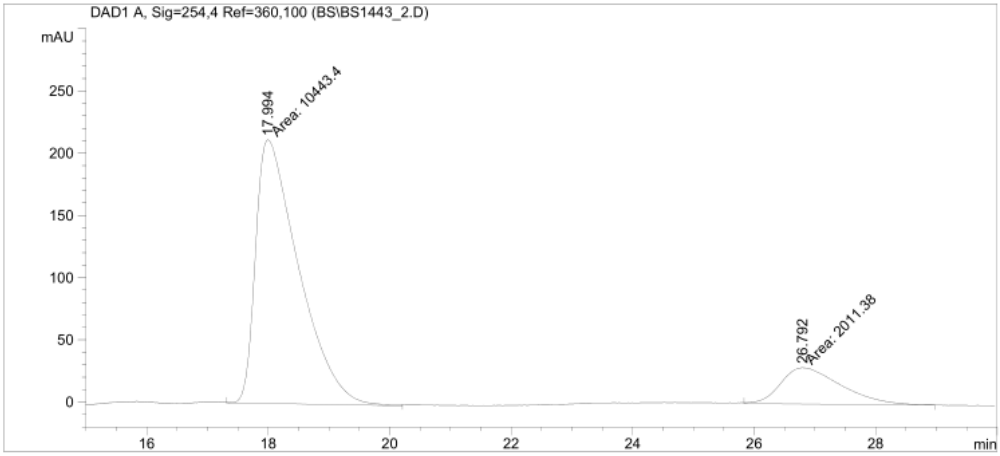
^{13}C NMR (75 MHz, $CDCl_3$)

HPLC for aldol products

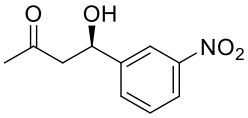


3a

```
Data File C:\HPCHEM\2\DATA\BS\BS1443_2.D                    Sample Name: BS1443_2
    AS 85:15 HEX/IPA, 1.0 mL/min, HPLC2

    ===================================================================
    Injection Date  : 02/12/2019 15:09:12
    Sample Name     : BS1443_2                    Location : Vial 2
    Acq. Operator   : BS
    Acq. Instrument : HPLC 2                    Inj Volume : 5 µl
    Acq. Method     : C:\HPCHEM\2\METHODS\C6_1_60.M
    Last changed    : 08/11/2019 10:28:57  by DA
    Analysis Method : C:\HPCHEM\2\METHODS\C2_1_40.M
    Last changed    : 02/12/2019 15:56:15  by BS
                      (modified after loading)
    cOLUMNA 2
    ===================================================================
```



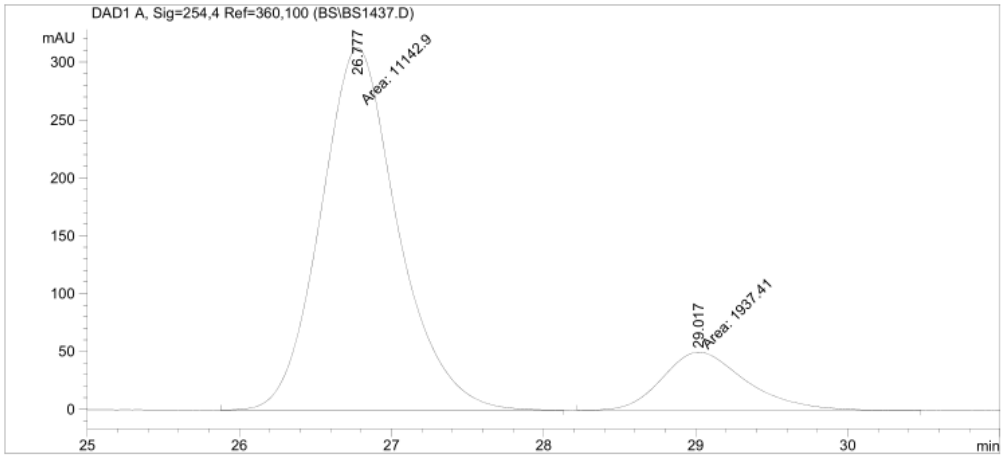

```
    ===================================================================
                              Area Percent Report
    ===================================================================

    Sorted By             :      Signal
    Multiplier            :      1.0000
    Dilution              :      1.0000
    Sample Amount         :      1.00000  [ng/ul]    (not used in calc.)
    Use Multiplier & Dilution Factor with ISTDs


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

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]   [mAU*s]     [mAU]       %
    ----|-------|----|-------|----------|----------|--------|
       1  17.994 MM    0.8215 1.04434e4   211.88654  83.8505
       2  26.792 MM    1.1442 2011.38062   29.29814  16.1495

    Totals :                  1.24547e4   241.18467
```



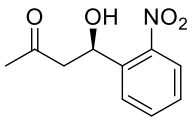
3b

Data File C:\HPCHEM\2\DATA\BS\BS1437.D Sample Name: BS1437
AD-H 95:5 HEX/IPA, 1.0 mL/min, HPLC2

```
==================================================================
Injection Date  : 20/11/2019 17:57:54
Sample Name     : BS1437                        Location : Vial 4
Acq. Operator   : BS
Acq. Instrument : HPLC 2                      Inj Volume : 6 µl
Acq. Method     : C:\HPCHEM\2\METHODS\C4.M
Last changed    : 20/11/2019 18:31:25  by BS
                  (modified after loading)
Analysis Method : C:\HPCHEM\2\METHODS\C6_1_60.M
Last changed    : 22/11/2019 12:37:21  by BS
                  (modified after loading)
==================================================================
```



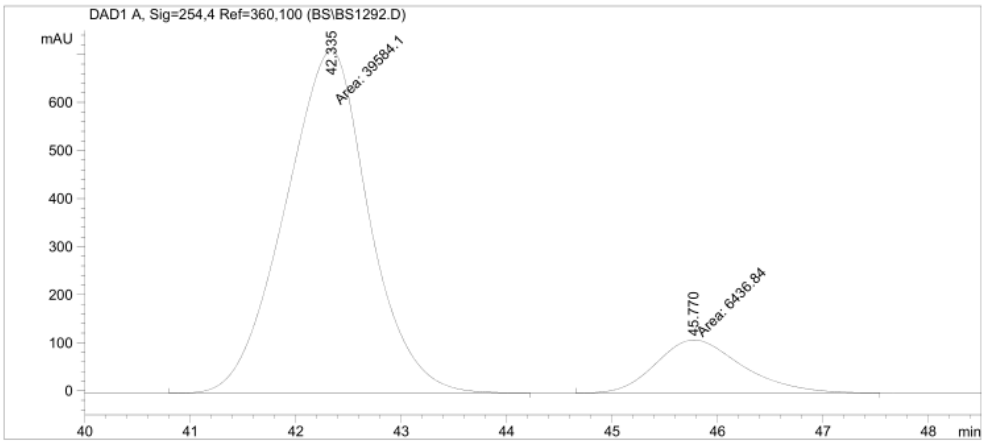

```
==================================================================
                        Area Percent Report
==================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Sample Amount         :      1.00000  [ng/ul]   (not used in calc.)
Use Multiplier & Dilution Factor with ISTDs


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

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1  26.777 MM    0.5916 1.11429e4   313.92798  85.1884
   2  29.017 MM    0.6432 1937.40869   50.19857  14.8116

Totals :                  1.30803e4   364.12655
```



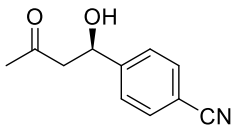
3c

Data File C:\HPCHEM\2\DATA\BS\BS1292.D Sample Name: BS1292
AD-H 98:2 HEX/IPA, 1.0 mL/min, HPLC2

```
==================================================================
Injection Date  : 11/07/2019 9:28:00
Sample Name     : BS1292                        Location : Vial 1
Acq. Operator   : BS
Acq. Instrument : HPLC 2                      Inj Volume : 5 µl
Acq. Method     : C:\HPCHEM\2\METHODS\C4.M
Last changed    : 11/07/2019 9:30:53  by BS
                  (modified after loading)
Analysis Method : C:\HPCHEM\2\METHODS\C3_1_60.M
Last changed    : 31/10/2019 17:44:05  by DA
                  (modified after loading)
cOLUMNA 2
==================================================================
```



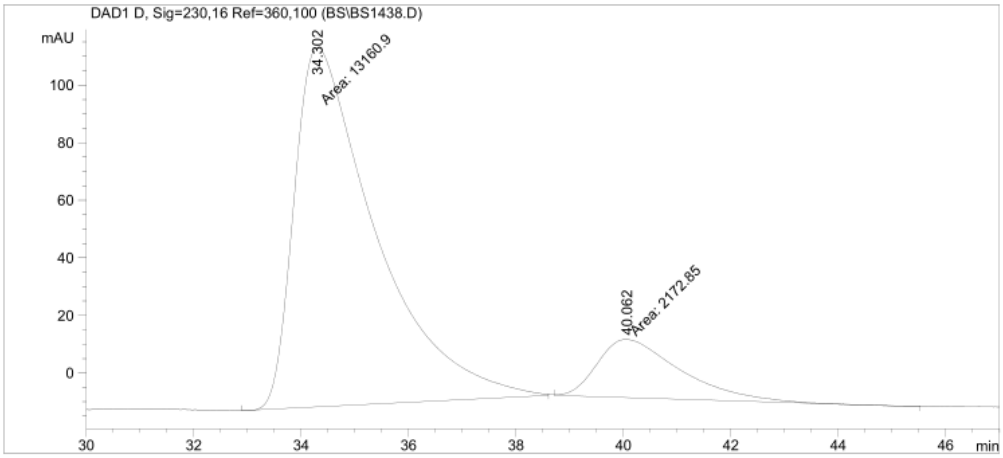

```
==================================================================
                        Area Percent Report
==================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Sample Amount         :      1.00000  [ng/ul]   (not used in calc.)
Use Multiplier & Dilution Factor with ISTDs


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

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1  42.335 MM    0.9239 3.95841e4   714.09467  86.0133
   2  45.770 MM    0.9622 6436.83545  111.49776  13.9867

Totals :                  4.60210e4   825.59242
```



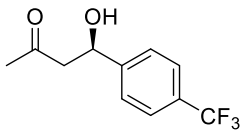
3d

Data File C:\HPCHEM\2\DATA\BS\BS1438.D Sample Name: BS1438
OD-H 95:5 HEX/IPA, 1.0 mL/min, HPLC2

```
==========================================================================
Injection Date  : 21/11/2019 9:18:44
Sample Name     : BS1438                          Location : Vial 3
Acq. Operator   : BS
Acq. Instrument : HPLC 2                        Inj Volume : 6 µl
Acq. Method     : C:\HPCHEM\2\METHODS\C2_1_40.M
Last changed    : 21/11/2019 10:07:12  by BS
                  (modified after loading)
Analysis Method : C:\HPCHEM\2\METHODS\C6_1_60.M
Last changed    : 22/11/2019 12:33:01  by BS
                  (modified after loading)
==========================================================================
```



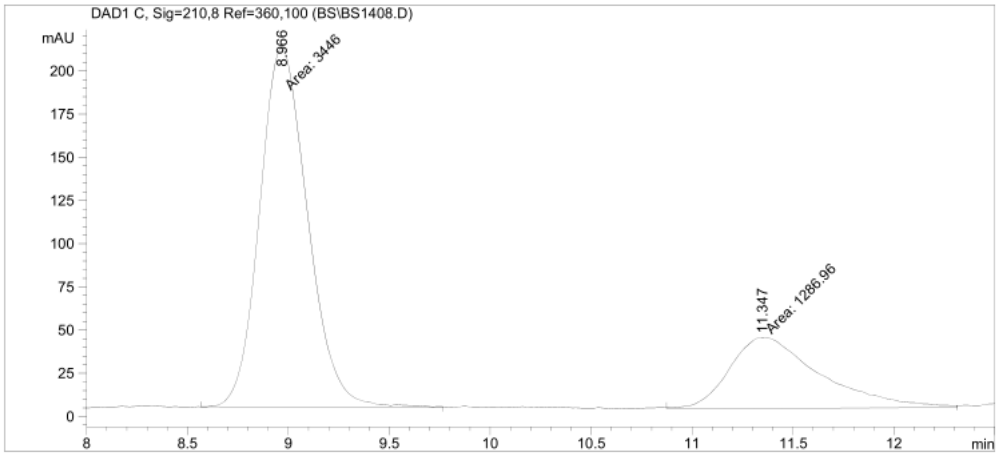

```
==========================================================================
                          Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Sample Amount         :      1.00000  [ng/ul]    (not used in calc.)
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 D, Sig=230,16 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1  34.302 MM    1.7575 1.31609e4   124.80685  85.8296
   2  40.062 MM    1.7831 2172.84546   20.30966  14.1704

Totals :                  1.53337e4   145.11652
```



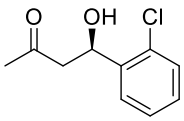
3e

Data File C:\HPCHEM\2\DATA\BS\BS1408.D Sample Name: BS1408
AS 92:8 HEX/IPA, 1.0 mL/min, HPLC2

```
==========================================================================
Injection Date  : 21/11/2019 10:45:45
Sample Name     : BS1408                          Location : Vial 5
Acq. Operator   : BS
Acq. Instrument : HPLC 2                        Inj Volume : 6 µl
Acq. Method     : C:\HPCHEM\2\METHODS\C6_1_60.M
Last changed    : 21/11/2019 11:03:10  by BS
                  (modified after loading)
Analysis Method : C:\HPCHEM\2\METHODS\C6_1_60.M
Last changed    : 22/11/2019 12:55:31  by BS
                  (modified after loading)
==========================================================================
```




```
==========================================================================
                          Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Sample Amount         :      1.00000  [ng/ul]    (not used in calc.)
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 C, Sig=210,8 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   8.966 MM    0.2761 3446.00464  208.04527  72.8086
   2  11.347 MM    0.5213 1286.96191   41.14829  27.1914

Totals :                  4732.96655  249.19356
```

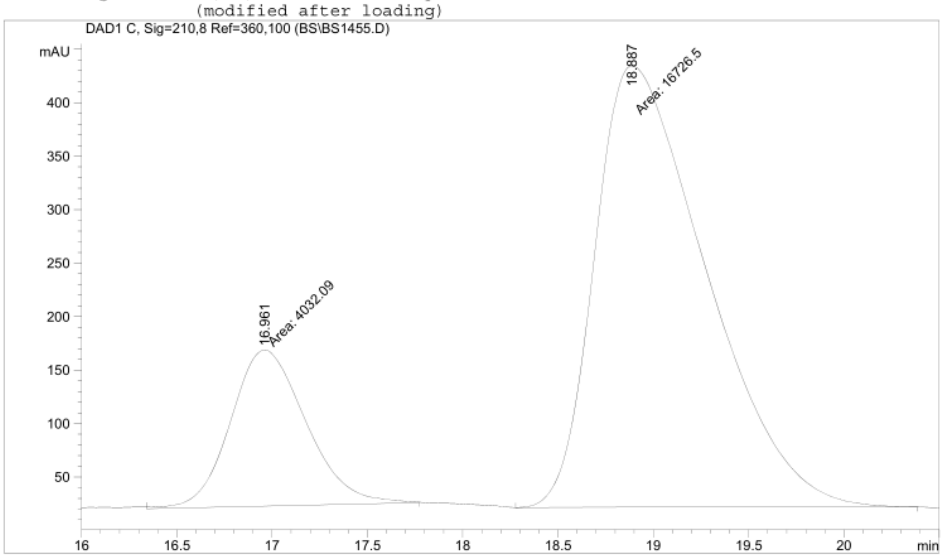


3f

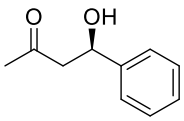
Data File C:\HPCHEM\2\DATA\BS\BS1455.D Sample Name: BS1455

AS 98/2 HEX:IPA 1.0 ML/MIN

```
==================================================================
Injection Date  : 16/12/2019 9:42:01
Sample Name     : BS1455                        Location : Vial 1
Acq. Operator   : BS
Acq. Instrument : HPLC 2                      Inj Volume : 5 µl
Acq. Method     : C:\HPCHEM\2\METHODS\C6_1_60.M
Last changed    : 16/12/2019 10:11:30  by BS
                  (modified after loading)
Analysis Method : C:\HPCHEM\2\METHODS\C4.M
Last changed    : 22/01/2020 13:19:25  by DF
                  (modified after loading)
```

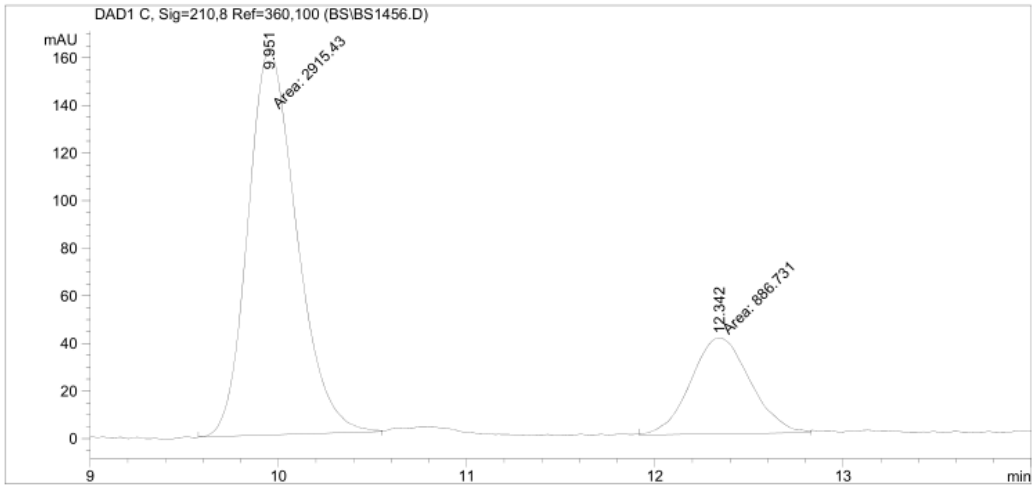



```
==================================================================
                        Area Percent Report
==================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Sample Amount         :      1.00000  [ng/ul]   (not used in calc.)
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 C, Sig=210,8 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]       [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1  16.961 MM    0.4594 4032.09131  146.29199  19.4237
   2  18.887 MM    0.6753 1.67265e4   412.80487  80.5763

Totals :                  2.07586e4   559.09686
```

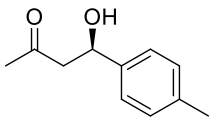


3g

```
Data File C:\HPCHEM\2\DATA\BS\BS1456.D                              Sample Name: BS1456
    AS 90/10 HX:IPA 1.0 ML/MIN

    ===================================================================
    Injection Date  : 16/12/2019 11:12:03          Seq. Line :   1
    Sample Name     : BS1456                        Location : Vial 2
    Acq. Operator   : BS                               Inj :   1
    Acq. Instrument : HPLC 2                      Inj Volume : 5 µl
    Acq. Method     : C:\HPCHEM\2\METHODS\C6_1_60.M
    Last changed    : 08/11/2019 10:28:57  by DA
    Analysis Method : C:\HPCHEM\2\METHODS\C6_1_60.M
    Last changed    : 16/12/2019 12:28:33  by BS
                      (modified after loading)
    ===================================================================
```

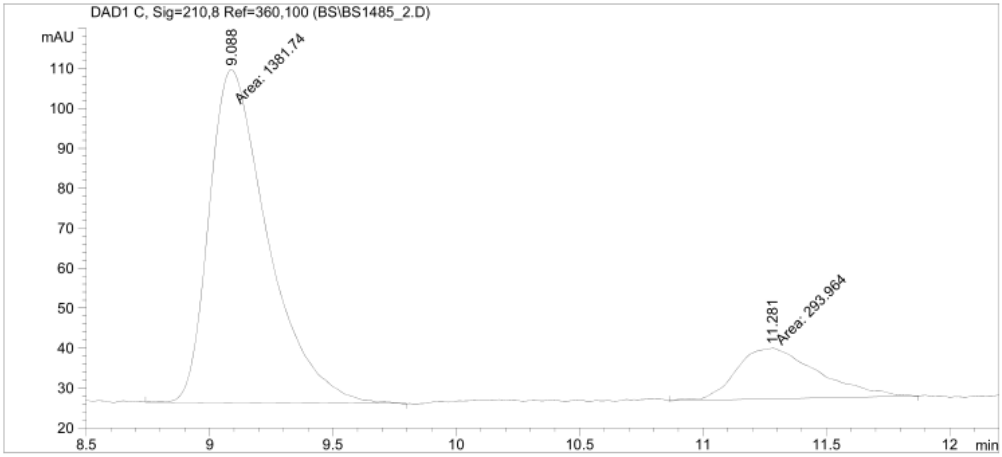



```
    ===================================================================
                            Area Percent Report
    ===================================================================

    Sorted By             :      Signal
    Multiplier            :      1.0000
    Dilution              :      1.0000
    Use Multiplier & Dilution Factor with ISTDs


    Signal 1: DAD1 C, Sig=210,8 Ref=360,100

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]   [mAU*s]     [mAU]       %
    ----|-------|----|-------|----------|----------|--------|
       1   9.951 MM    0.3008 2915.43115  161.53714  76.6783
       2  12.342 MM    0.3657  886.73053   40.40839  23.3217

    Totals :                  3802.16168  201.94553
```

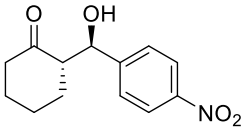


3h

```
Data File C:\HPCHEM\2\DATA\BS\BS1485_2.D                          Sample Name: BS1485_2
    AS-H 90/10 HEX:IPA 1.0 mL/min HPLC 2

    ===================================================================
    Injection Date  : 06/02/2020 9:27:08
    Sample Name     : BS1485_2                        Location : Vial 1
    Acq. Operator   : BS
    Acq. Instrument : HPLC 2                       Inj Volume : 6 µl
    Acq. Method     : C:\HPCHEM\2\METHODS\C3_1_60.M
    Last changed    : 06/02/2020 9:56:46  by BS
                      (modified after loading)
    Analysis Method : C:\HPCHEM\2\METHODS\C3_1_60.M
    Last changed    : 06/02/2020 11:29:27  by BS
                      (modified after loading)
    cOLUMNA 2
    ===================================================================
```



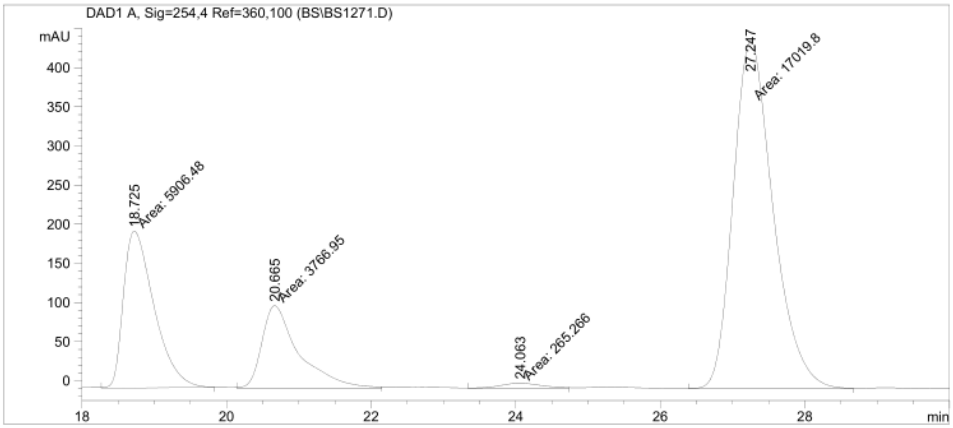

```
    ===================================================================
                              Area Percent Report
    ===================================================================

    Sorted By             :      Signal
    Multiplier            :      1.0000
    Dilution              :      1.0000
    Sample Amount         :      1.00000  [ng/ul]   (not used in calc.)
    Use Multiplier & Dilution Factor with ISTDs


    Signal 1: DAD1 C, Sig=210,8 Ref=360,100

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]   [mAU*s]     [mAU]       %
    ----|-------|----|-------|----------|----------|--------|
       1   9.088 MM    0.2759 1381.73975   83.47313  82.4573
       2  11.281 MM    0.3853  293.96359   12.71603  17.5427

    Totals :                  1675.70334   96.18916
```



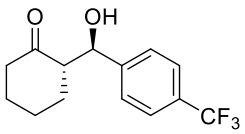
anti-**3i**

Data File C:\HPCHEM\2\DATA\BS\BS1271.D Sample Name: BS1271
ADH 90:10 HEX/IPA, 1.0 mL/min, HPLC2

```
==================================================================
Injection Date  : 01/07/2019 12:16:25
Sample Name     : BS1271                        Location : Vial 1
Acq. Operator   : BS
Acq. Instrument : HPLC 2                      Inj Volume : 5 µl
Acq. Method     : C:\HPCHEM\2\METHODS\C4.M
Last changed    : 01/07/2019 12:17:39  by BS
                  (modified after loading)
Analysis Method : C:\HPCHEM\2\METHODS\C3_1_60.M
Last changed    : 19/12/2019 16:09:26  by DA
                  (modified after loading)
cOLUMNA 2
==================================================================
```



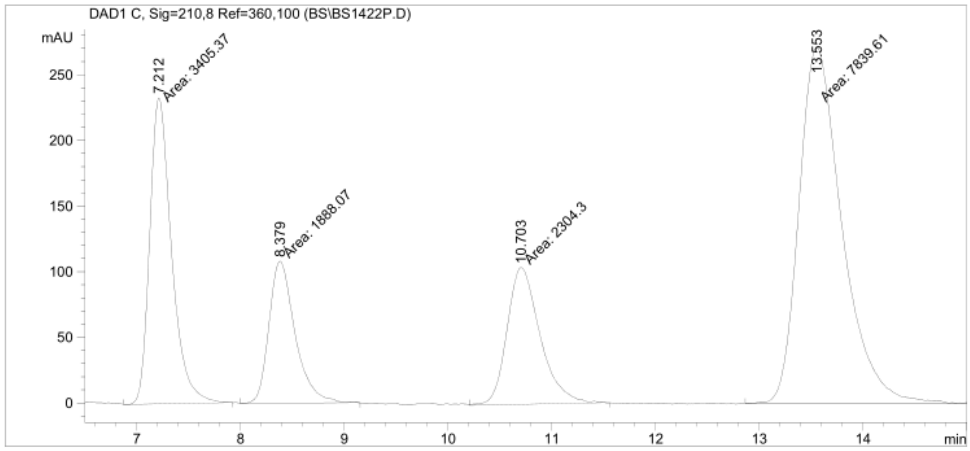

```
==================================================================
                        Area Percent Report
==================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Sample Amount         :      1.00000  [ng/ul]   (not used in calc.)
Use Multiplier & Dilution Factor with ISTDs


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

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1  18.725 MM    0.4911 5906.48193  200.44827  21.9095
   2  20.665 MM    0.5948 3766.94849  105.55300  13.9731
   3  24.063 MM    0.6746  265.26608    6.55345   0.9840
   4  27.247 MM    0.6389 1.70198e4   443.96204  63.1334

Totals :                  2.69585e4   756.51676
```



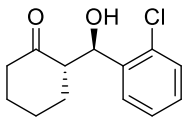
anti-**3j**

Data File C:\HPCHEM\2\DATA\BS\BS1422P.D Sample Name: BS1422P
AD 90/10 HEX:IPA 1 mL/min HPLC 2

```
==========================================================================
Injection Date  : 17/01/2020 11:51:52
Sample Name     : BS1422P                        Location : Vial 1
Acq. Operator   : BS
Acq. Instrument : HPLC 2                        Inj Volume : 6 µl
Acq. Method     : C:\HPCHEM\2\METHODS\C5.M
Last changed    : 17/01/2020 12:17:31  by BS
                  (modified after loading)
Analysis Method : C:\HPCHEM\2\METHODS\C1_1_60M.M
Last changed    : 29/01/2020 15:07:24  by BS
                  (modified after loading)
==========================================================================
```

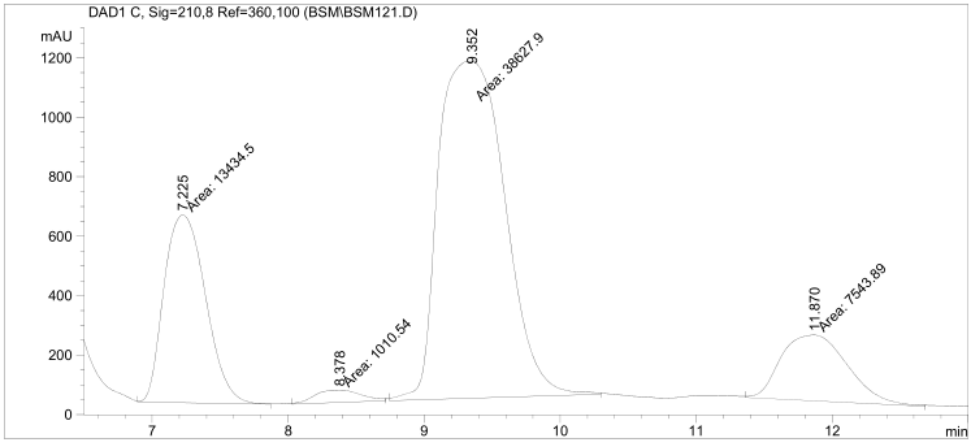



```
==========================================================================
                        Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Sample Amount         :      1.00000  [ng/ul]   (not used in calc.)
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 C, Sig=210,8 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   7.212 MM    0.2434 3405.36792  233.14369  22.0593
   2   8.379 MM    0.2908 1888.07043  108.20387  12.2305
   3  10.703 MM    0.3684 2304.30103  104.25667  14.9268
   4  13.553 MM    0.4815 7839.60840  271.37921  50.7834

Totals :                  1.54373e4   716.98344
```

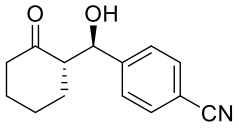


anti-**3k**

```
Data File C:\HPCHEM\2\DATA\BSM\BSM121.D                              Sample Name: BSM121
    OD-H 95:5 HEX/IPROH, 1.0 ML/min, HPLC2

    ===================================================================
    Injection Date  : 21/03/2019 12:49:54
    Sample Name     : BSM121                          Location : Vial 1
    Acq. Operator   : BSM
    Acq. Instrument : HPLC 2                        Inj Volume : 5 µl
    Acq. Method     : C:\HPCHEM\2\METHODS\C2_1_60.M
    Last changed    : 21/03/2019 12:31:10  by BSM
                      (modified after loading)
    Analysis Method : C:\HPCHEM\2\METHODS\C3_1_60.M
    Last changed    : 19/12/2019 9:45:22  by DA
                      (modified after loading)
    cOLUMNA 2
    ===================================================================
```




```
    ===================================================================
                              Area Percent Report
    ===================================================================

    Sorted By             :      Signal
    Multiplier            :      1.0000
    Dilution              :      1.0000
    Sample Amount         :      1.00000  [ng/ul]   (not used in calc.)
    Use Multiplier & Dilution Factor with ISTDs


    Signal 1: DAD1 C, Sig=210,8 Ref=360,100

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]       [min]   [mAU*s]     [mAU]       %
    ----|-------|----|-------|----------|----------|--------|
       1   7.225 MM    0.3544 1.34345e4   631.85455  22.1630
       2   8.378 MM    0.4035 1010.54034   41.74266   1.6671
       3   9.352 MM    0.5677 3.86279e4  1133.95313  63.7247
       4  11.870 MM    0.5665 7543.89014  221.93658  12.4452

    Totals :                  6.06169e4  2029.48693
```

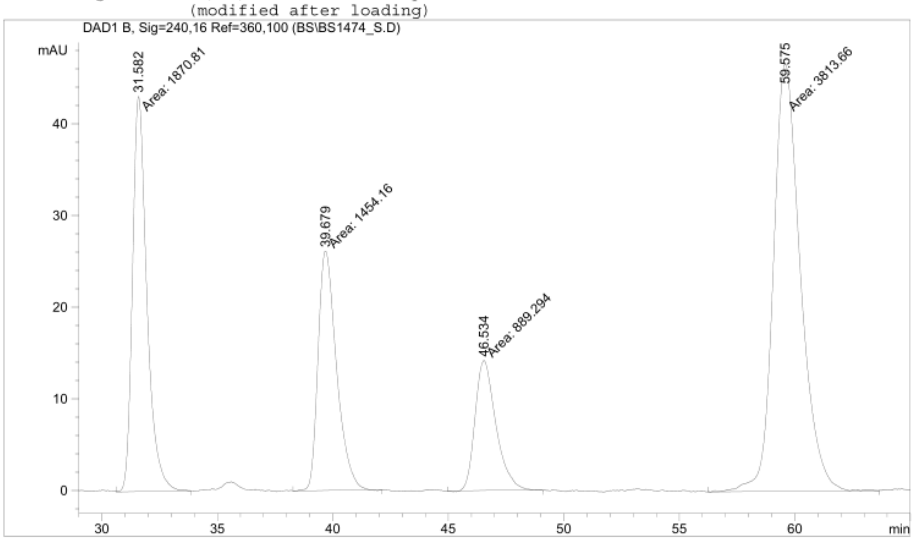


anti-**3l**

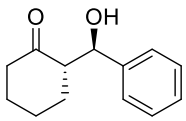
Data File C:\HPCHEM\2\DATA\BS\BS1474_S.D Sample Name: BS1474_S

AD-H 95/5 HEX:IPA 1.0 mL/min HPLC 2

```
==========================================================================
Injection Date  : 05/02/2020 14:36:21
Sample Name     : BS1474_S                        Location : Vial 1
Acq. Operator   : BS
Acq. Instrument : HPLC 2                        Inj Volume : 5 µl
Acq. Method     : C:\HPCHEM\2\METHODS\C4.M
Last changed    : 05/02/2020 15:45:24  by BS
                  (modified after loading)
Analysis Method : C:\HPCHEM\2\METHODS\C4.M
Last changed    : 05/02/2020 15:55:22  by BS
                  (modified after loading)
```

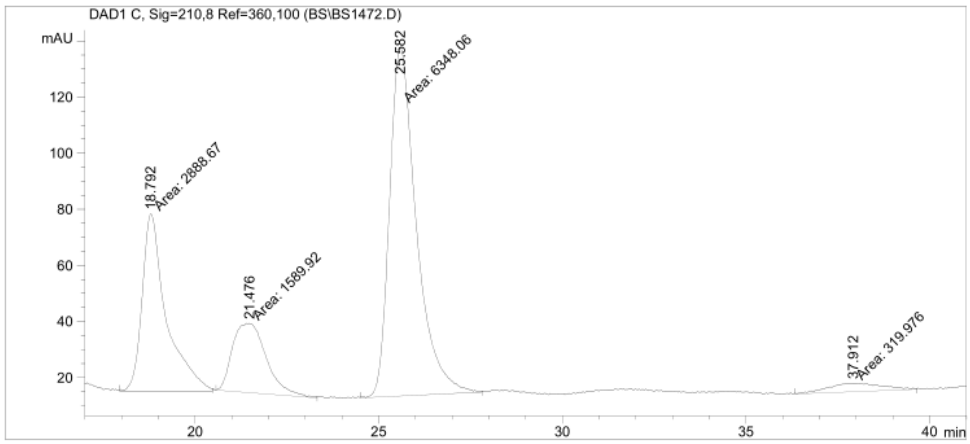



```
==========================================================================
                           Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Sample Amount         :      1.00000  [ng/ul]   (not used in calc.)
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 B, Sig=240,16 Ref=360,100

Peak RetTime Type  Width     Area      Height      Area
  #   [min]        [min]    [mAU*s]    [mAU]        %
----|-------|----|-------|----------|----------|--------|
   1  31.582 MM    0.7230 1870.80701   43.12420  23.3038
   2  39.679 MM    0.9283 1454.15674   26.10822  18.1138
   3  46.534 MM    1.0446  889.29352   14.18940  11.0775
   4  59.575 MM    1.3631 3813.65552   46.63028  47.5049

Totals :                  8027.91278  130.05210
```

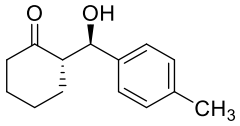


anti-**3m**

```
Data File C:\HPCHEM\2\DATA\BS\BS1472.D                          Sample Name: BS1472
    OD-H 95/5 HEX:IPA 0.5 ML/MIN

    ===================================================================
    Injection Date  : 13/01/2020 9:57:26
    Sample Name     : BS1472                        Location : Vial 1
    Acq. Operator   : BS
    Acq. Instrument : HPLC 2                      Inj Volume : 5 µl
    Acq. Method     : C:\HPCHEM\2\METHODS\C2_1_60.M
    Last changed    : 13/01/2020 10:55:44  by BS
                      (modified after loading)
    Analysis Method : C:\HPCHEM\2\METHODS\C2_1_60.M
    Last changed    : 13/01/2020 11:13:03  by BS
                      (modified after loading)
    cOLUMNA 2
    ===================================================================
```



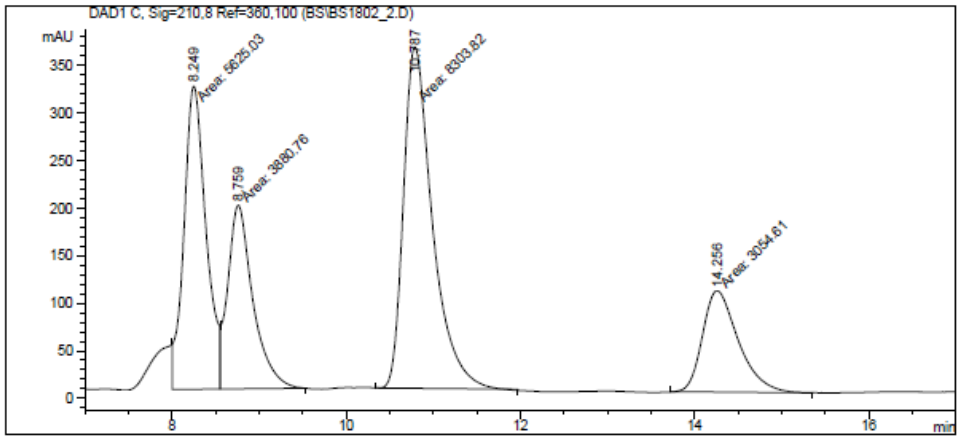

```
    ===================================================================
                              Area Percent Report
    ===================================================================

    Sorted By             :      Signal
    Multiplier            :      1.0000
    Dilution              :      1.0000
    Sample Amount         :      1.00000  [ng/ul]   (not used in calc.)
    Use Multiplier & Dilution Factor with ISTDs


    Signal 1: DAD1 C, Sig=210,8 Ref=360,100

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]   [mAU*s]     [mAU]       %
    ----|-------|----|-------|----------|----------|--------|
       1  18.792 MM    0.7607 2888.66821   63.29018  25.9152
       2  21.476 MM    1.0693 1589.92151   24.78108  14.2637
       3  25.582 MM    0.8516 6348.06348  124.24226  56.9505
       4  37.912 MM    1.7057  319.97641    3.12646   2.8706

    Totals :                  1.11466e4   215.43998
```



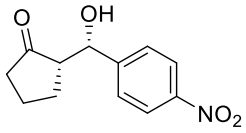
anti-**3n**

Data File C:\HPCHEM\2\DATA\BS\BS1802_2.D Sample Name: BS1802_2
OD-H, 1.0 mL/min, 98/2 Hx/IPA

```
==========================================================================
Injection Date  : 07/07/2021 10:35:43
Sample Name     : BS1802 2                      Location : Vial 2
Acq. Operator   : BS
Acq. Instrument : HPLC 2                      Inj Volume : 5 µl
Acq. Method     : C:\HPCHEM\2\METHODS\C2_1_60.M
Last changed    : 07/07/2021 11:14:01  by BS
                  (modified after loading)
Analysis Method : C:\HPCHEM\2\METHODS\C2_1_60.M
Last changed    : 07/07/2021 13:25:45  by BS
                  (modified after loading)
cOLUMNA 2
==========================================================================
```



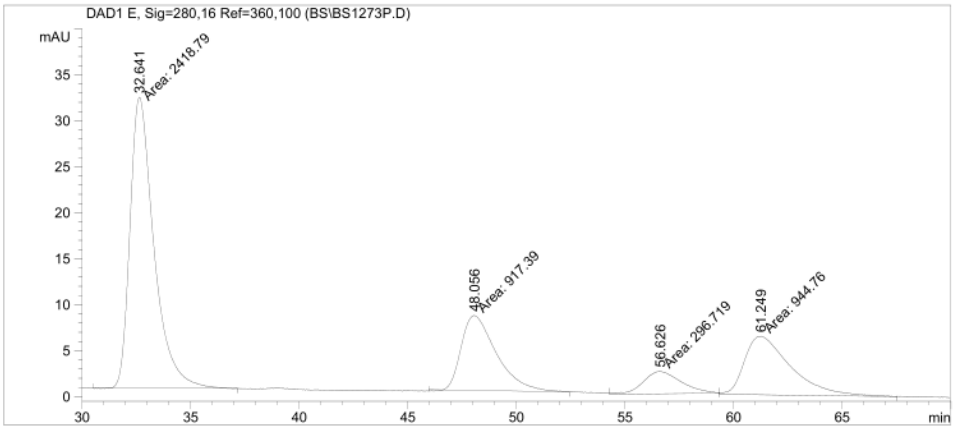

```
==========================================================================
                          Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Sample Amount         :      1.00000  [ng/ul]   (not used in calc.)
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 C, Sig=210,8 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]      [min]    [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   8.249 FM    0.2940 5625.02734  318.92517  26.9602
   2   8.759 FM    0.3347 3880.75928  193.22963  18.6001
   3  10.787 MM    0.3854 8303.82129  359.12814  39.7993
   4  14.256 MM    0.4773 3054.61060  106.65953  14.6404
```



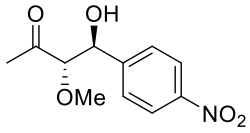
syn-**3o**

Data File C:\HPCHEM\2\DATA\BS\BS1273P.D Sample Name: BS1273P
AD 96:4 HEX/IPA, 1.0 mL/min, HPLC2

```
==================================================================
Injection Date  : 05/07/2019 10:36:43
Sample Name     : BS1273P                        Location : Vial 2
Acq. Operator   : BS
Acq. Instrument : HPLC 2                       Inj Volume : 6 µl
Acq. Method     : C:\HPCHEM\2\METHODS\C5.M
Last changed    : 05/07/2019 11:40:20  by BS
                  (modified after loading)
Analysis Method : C:\HPCHEM\2\METHODS\C3_1_60.M
Last changed    : 31/10/2019 18:14:57  by DA
                  (modified after loading)
cOLUMNA 2
==================================================================
```



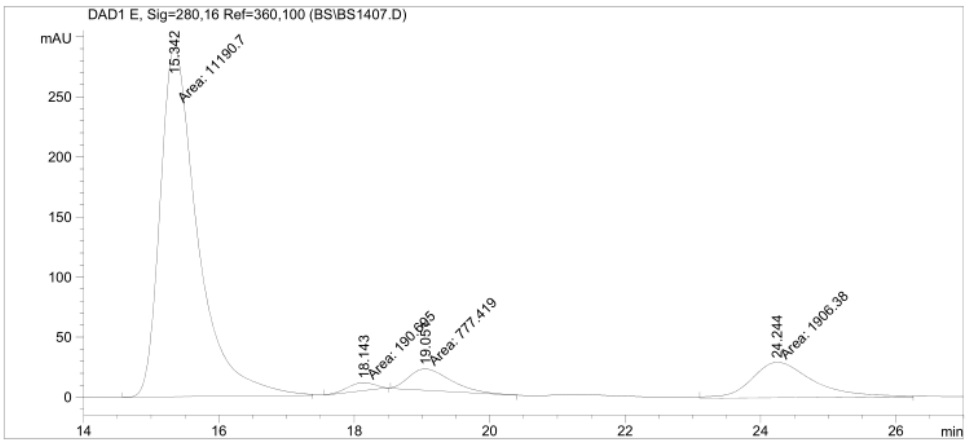

```
==================================================================
                        Area Percent Report
==================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Sample Amount         :      1.00000  [ng/ul]   (not used in calc.)
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 E, Sig=280,16 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1  32.641 MM    1.2760 2418.78662   31.59271  52.8390
   2  48.056 MM    1.8759  917.38977    8.15066  20.0406
   3  56.626 MM    2.0309  296.71857    2.43508   6.4819
   4  61.249 MM    2.4673  944.75983    6.38193  20.6385

Totals :                  4577.65479   48.56038
```



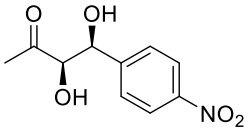
anti-**3p**

Data File C:\HPCHEM\2\DATA\BS\BS1407.D Sample Name: BS1407
OD-H 90:10 HEX/IPA, 0.8 mL/min, HPLC2

```
==========================================================================
Injection Date  : 20/11/2019 16:03:02
Sample Name     : BS1407                          Location : Vial 1
Acq. Operator   : BS
Acq. Instrument : HPLC 2                        Inj Volume : 6 µl
Acq. Method     : C:\HPCHEM\2\METHODS\C2_1_40.M
Last changed    : 20/11/2019 16:27:06  by BS
                  (modified after loading)
Analysis Method : C:\HPCHEM\2\METHODS\C6_1_60.M
Last changed    : 22/11/2019 12:40:56  by BS
                  (modified after loading)
==========================================================================
```




```
==========================================================================
                          Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Sample Amount         :      1.00000  [ng/ul]    (not used in calc.)
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 E, Sig=280,16 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1  15.342 MM    0.6425 1.11907e4   290.29651  79.5631
   2  18.143 MM    0.4769  190.69528    6.66449   1.3558
   3  19.051 MM    0.7255  777.41852   17.85838   5.5272
   4  24.244 MM    1.0714 1906.37500   29.65425  13.5538

Totals :                  1.40652e4   344.47363
```

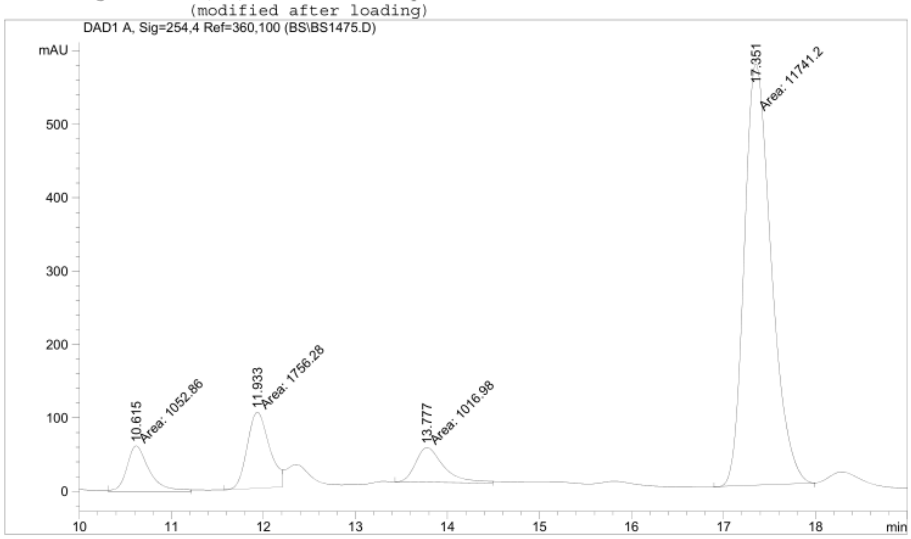


syn-**3q**

Data File C:\HPCHEM\2\DATA\BS\BS1475.D Sample Name: BS1475

AD-H 80/20 HEX:IPA 0.8 ML/MIN

```
==========================================================================
Injection Date  : 13/01/2020 15:15:36
Sample Name     : BS1475                          Location : Vial 1
Acq. Operator   : BS
Acq. Instrument : HPLC 2                        Inj Volume : 5 µl
Acq. Method     : C:\HPCHEM\2\METHODS\C4.M
Last changed    : 13/01/2020 15:39:12  by BS
                  (modified after loading)
Analysis Method : C:\HPCHEM\2\METHODS\C4.M
Last changed    : 13/01/2020 18:33:58  by BS
                  (modified after loading)
```




```
==========================================================================
                        Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Sample Amount         :      1.00000  [ng/ul]   (not used in calc.)
Use Multiplier & Dilution Factor with ISTDs


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

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1  10.615 MM    0.2827 1052.86279   62.07030   6.7633
   2  11.933 MF    0.2815 1756.28406  103.98099  11.2818
   3  13.777 MM    0.3577 1016.98077   47.39058   6.5328
   4  17.351 MM    0.3406 1.17412e4   574.50885  75.4221

Totals :                  1.55674e4   787.95072
```