Supporting Information for

Copper-Catalyzed Radical Coupling/Fragmentation Reaction: Efficient Access to β-Oxophosphine Oxides

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TABLE OF CONTENTS

1. General information

Solvents were purified and dried by standard methods prior to use. All commercially available reagents were used without further purification unless otherwise noted. Oxygen- and moisture-sensitive reactions were carried out under argon atmosphere. Column chromatography was generally performed on silica gel (200-300 mesh) and reactions were monitored by thin layer chromatography (TLC) using silica gel GF254 plates with UV light to visualize the course of reaction. Melting points were determined with a digital Koffer apparatus and were uncorrected. ^{1}H, ^{13}C and ^{31}P NMR data were recorded on a 400 MHz spectrometer using $CDCl_3$ as solvent at room temperature. The chemical shifts (δ) are reported in ppm and coupling constants (J) in Hz. High-resolution mass spectra (HRMS) were obtained on a FT-ICR spectrometer.

2. Synthesis of the starting materials

Substrates **1a**, **1c**, **1d-1g**, **1j**, **1k**, **1m**, **1o**, **1q**-**1s** were prepared accroding to the **Method A**. [1-3] **1h**, **1i**, **1l**, **1n**, **1p** were prepared accroding to the **Method B**. [1-3] **1b** was prepared accroding to the **Method C.** [4] **1t** was prepared accroding to the **Method d.** [5] Spectroscopy data of the known compounds matches with the data reported in the corresponding references.

Method A:

To a solution of alkenyl bromide (4.0 mmol) in dry THF was added a solution of *t*-BuLi (1.6 M in pentane, 8.0 mmol) at -78 °C under an argon atmosphere for 20 min. The solution was stirred at -78 °C for 1.5 h. A solution of ketone or aldehyde (5.2 mmol, 1.3 equiv) in dry THF was added to the reaction mixture and stirred at -78 °C for 1 h. Then the reaction was allowed to warm to room temperature. The reaction mixture was quenched with H_2O and extracted with ethyl acetate. The combined organic layer was washed with brine, dried over anhydrous Na_2SO_4, filtered, and concentrated under reduced pressure. The crude reaction mixture was purified by flash column chromatography on silica gel to afford the corresponding product.

Method B:

A three-necked flask equipped with an addition funnel, a condenser and a stir bar was charged with Mg turnings (12.0 mmol) under an argon atmosphere. Dry THF (8 mL) and bromoethane (0.26 mmol) were added via syringe. Then iodine (8.0 mg) was added. After stirring at rt for 10 min, alkenyl bromide (4.0 mmol) in dry THF (5 mL) was then added dropwise over 40 min via the addition funnel. The reaction was then heated to reflux for 3 h and was cooled to room temperature and ketone (5.6 mmol) was added. The resulting reaction mixture was stirred at

room temperature for 6 h, then quenched with saturated aqueous NH_4Cl solution and extracted twice with ethyl acetate. The combined organic layer was washed with brine, dried over anhydrous Na_2SO_4, filtered, and concentrated under reduced pressure. The crude reaction mixture was purified by flash column chromatography on silica gel to afford the corresponding product.

Method C:

To a solution of Mg (10.0 mmol) in Et_2O, bromobenzene (10.0 mmol) was added at 0 ^oC. The reaction mixture was heated to reflux for 2 h, CuI (10% mmol) was then added at rt. The reaction mixture was allowed to stir at same temperature for 0.5 h, propargyl alcohol (4.0 mmol) in Et_2O was added dropwise at rt. Then, the reaction was heated to reflux for 24 h, after cooling to rt, quenched with saturated aqueous NH_4Cl solution and extracted twice with ethyl acetate. The combined organic layer was washed with brine, dried over anhydrous Na_2SO_4, filtered, and concentrated under reduced pressure. The crude reaction mixture was purified by flash column chromatography on silica gel to afford the corresponding product.

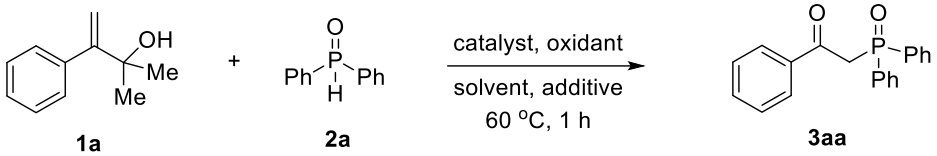
Method D:

To a solution of indene (1.0 equiv.) in anhydrous tetrahydrofuran at -78 ^oC was added n-butyllithium (1.0 equiv.) over 10 minutes. The mixture was allowed to warm to room temperature and stirred for a further 6 h, before it was cooled again to -78 ^oC. The appropriate ketone (1.0 equiv.) was then added dropwise into the solution over 15 minutes. The reaction was then allowed to warm slowly to room temperature and then stirred for a further 6 h before being quenched with water. The organic layer was then extracted three times with diethyl ether, combined, washed once with brine and dried ($MgSO_4$) before concentrating in vacuo. The product was purified by flash chromatography using silica gel and/or basic aluminium oxide, followed by recrystallization from hexane to afford the desired product in high purity.

3. General procedure for the copper-catalyzed radical reaction.

A mixture of allylic alcohols **1** (0.2 mmol), *H*-phosphine oxides **2** (0.4 mmol) and $CuSO_4·5H_2O$ (10 mmol %), TBHP (2.0 equiv), 4 Å MS (40.0 mg) in DCE (2.0 mL) was stirred under an atomersphere of argon at 60 ^oC for 1 h. The resulting mixture was concentrated under vacuum and the crude product was purified by silica gel chromatography using petroleum ether–AcOEt (3:1-2:1, v/v) as the eluent to give the corresponding products.

4. The detail optimization of the reaction conditions[a]

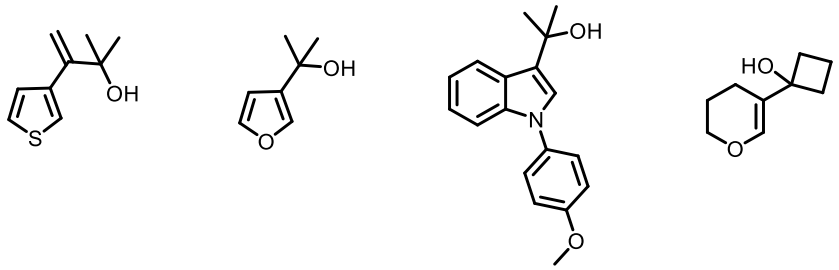


1a **2a** **3aa**

Entry	Catalyst	Oxidant	Solvent	Additive	Yield [%][b]
1	$Cu(NO_3)_2{\cdot}3H_2O$	TBHP	CH_3CN	4 Å MS	46
2	$AgNO_3$	TBHP	CH_3CN	4 Å MS	30
3	Cu	TBHP	CH_3CN	4 Å MS	65
4	CuCl	TBHP	CH_3CN	4 Å MS	42
5	Cu_2O	TBHP	CH_3CN	4 Å MS	39
6	$CuSO_4$	TBHP	CH_3CN	4 Å MS	45
7	$Cu(OAc)_2$	TBHP	CH_3CN	4 Å MS	51
8	$CuBr_2$	TBHP	CH_3CN	4 Å MS	54
9	CuBr	TBHP	CH_3CN	4 Å MS	46
10	CuO	TBHP	CH_3CN	4 Å MS	38
11	CuI	TBHP	CH_3CN	4 Å MS	60
12	$CuSO_4{\cdot}5H_2O$	TBHP	CH_3CN	4 Å MS	74
13	$CuSO_4{\cdot}5H_2O$	TBHP	THF	4 Å MS	29
14	$CuSO_4{\cdot}5H_2O$	TBHP	DMF	4 Å MS	10
15	**$CuSO_4{\cdot}5H_2O$**	**TBHP**	**DCE**	**4 Å MS**	**94**
16	$CuSO_4{\cdot}5H_2O$	$K_2S_2O_8$	DCE	4 Å MS	20
17	$CuSO_4{\cdot}5H_2O$	--	DCE	4 Å MS	30
18	--	TBHP	DCE	4 Å MS	trace
19	$CuSO_4{\cdot}5H_2O$	TBHP	DCE	--	26
20	$CuSO_4{\cdot}5H_2O$	TBHP	DCE	4 Å MS[c]	67
21	$CuSO_4{\cdot}5H_2O$	TBHP	DCE	4 Å MS[d]	85
22	$CuSO_4{\cdot}5H_2O$	TBHP	DCE	4 Å MS[e]	95
23[f]	$CuSO_4{\cdot}5H_2O$	TBHP	DCE	4 Å MS	70
24[g]	$CuSO_4{\cdot}5H_2O$	TBHP	DCE	4 Å MS	90

[a] Reaction conditions : **1a** (0.2 mmol, 1.0 equiv.), **2a** (0.4 mmol, 2.0 equiv.), catalyst (10 mol%), oxidant (0.4 mmol, 2.0 equiv.), 4 Å MS (40.0 mg), solvent (2.0 ml) at 60 °C under argon for 1 h. [b] Isolated yields. [c] 4 Å MS (10.0 mg). [d] 4 Å MS (30.0 mg). [e] 4 Å MS (100.0 mg). [f] $CuSO_4{\bullet}5H_2O$ (5 mol%). [g] In the air.

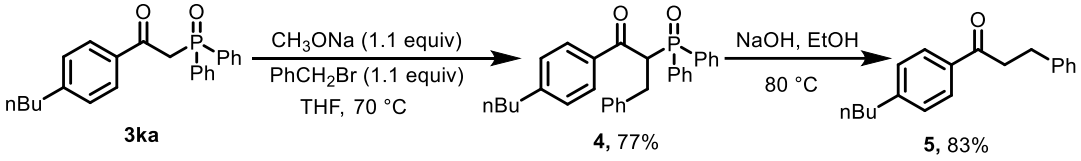
5. The failed substrates bearing heterocycle for this copper-catalyzed radical reaction.



6. Gram scale preparation of 3ka.

A mixture of **1k** (1.09 g, 5 mmol), **2a** (2.02 g, 10 mmol) and $CuSO_4·5H_2O$ (10 mmol %), TBHP (2.0 equiv), 4 Å MS (1.1 g) in DCE (25 mL) was stirred under an atomersphere of argon at 60 °C for 1 h. The resulting mixture was concentrated under vacuum and the crude product was purified by silica gel chromatography using petroleum ether–AcOEt (3:1-2:1, v/v) as the eluent to give the corresponding products.

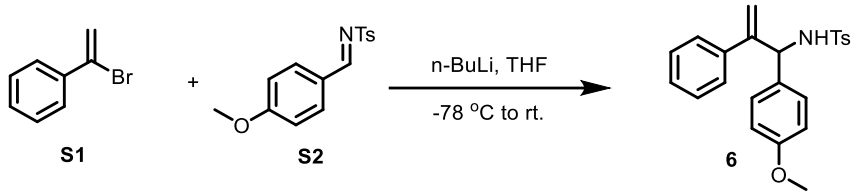
7. Synthesis of 1-(4-butylphenyl)-3-phenylpropan-1-one 5.



A tube was charged with a magnetic stir-bar, **3ka** (0.50 mmol), sodium methoxide (0.55 mmol), and anhydrous THF (10.0 mL). The tube was stirred vigorously under air at 70 °C. After 1 h benzyl bromide (0.55 mmol) was added and stirring was continued at 70 °C for 12 h. The reaction mixture was allowed to cool to ambient temperature, and then transferred to a round-bottom flask. Silica gel was added, and the solvent was removed under reduced pressure to afford a free-flowing powder. This powder was then dryloaded onto a silica gel column and purified by flash chromatography using petroleum ether: AcOEt (2:1, v/v) as the eluent to give the corresponding product **4** in 77% yield.

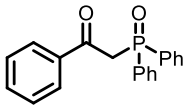
A tube was charged with a magnetic stir-bar, **4** (0.11 mmol), 4 M aqueous NaOH (2 mL), ethanol (2 mL). The reaction mixture was heated with stirring at 80 °C for 1 h. The reaction mixture was allowed to cool to ambient temperature, and then transferred to a round-bottom flask. Silica gel was added, and the solvent was removed under reduced pressure to afford a free-flowing powder. This powder was then dryloaded onto a silica gel column and purified by flash chromatography using petroleum ether as the eluent to give the corresponding product **5** in 83% yield.

8. Preparation of 6.



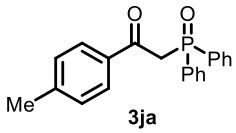
n-BuLi (2.5 M in THF, 1.5 eq.) was added dropwise to a solution of (1-bromovinyl)benzene **S1** (1.0 eq.) in dry THF at -78 °C under argon. The resulting mixture was then stirred at the same temperature for 1.5 h. Imine **S2** (2.0 eq.) was then condensed into the reaction mixture and it was stirred for an additional 0.5 h at -78 °C. The suspension was then slowly warmed to 22 °C and monitored by TLC analysis until start materials was disappeared completely. A saturated solution of NH_4Cl was then added and the mixture was extracted with EtOAc, and dried over $MgSO_4$. Concentration and purification by chromatography on silica gel (PE / EtOAc = 5 : 1) afforded the **6** as colorless oil.

9. Characterization data for all products.



3aa

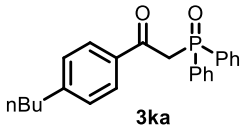
White solid; Mp: 137−139°C. **^{1}H NMR (400 MHz, $CDCl_3$)** δ 8.02 – 7.97 (m, 2H), 7.85 – 7.77 (m, 4H), 7.54 (ddd, *J* = 7.1, 5.2, 1.3 Hz, 3H), 7.50 – 7.40 (m, 6H), 4.16 (d, *J* = 15.4 Hz, 2H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ (192.78, 192.72, d, *J* = 6.1 Hz), 136.77, 133.57, (132.23, 131.20, d, *J* = 104.0 Hz), (132.12, 132.10, d, *J* = 2.0 Hz), (131.06, 130.96 d, *J* = 10.1 Hz), 129.16, (128.61, 128.49, d, *J* = 12.1 Hz), 128.46, (43.43, 42.85, d, *J* = 58.6 Hz). **^{31}P NMR (162 MHz, $CDCl_3$)** δ 27.15. **HRMS** (ESI): m/z $[M+H]^+$ calculated for $C_{20}H_{18}O_2P$: 321.1039, found: 321.1048.



3ja

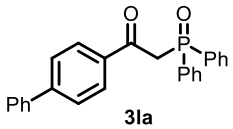
White solid; Mp: 123−125°C. **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.89 (d, *J* = 8.2 Hz, 2H), 7.85 – 7.76 (m, 4H), 7.56 – 7.50 (m, 2H), 7.50 – 7.43 (m, 4H), 7.22 (d, *J* = 8.0 Hz, 2H), 4.12 (d, *J* = 15.3 Hz, 2H), 2.38 (s, 3H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ (192.31, 192.26, d, *J* = 5.1 Hz), 144.61, 134.39, (132.36, 131.34, d, *J* = 103.0 Hz), (132.09, 132.06, d, *J* = 3.0 Hz), (131.10, 131.00, d, *J* = 10.1 Hz), 129.36, 129.19, (128.61, 128.49, d, *J* = 12.1 Hz), (43.39, 42.82, d, *J* = 57.6 Hz), 21.68. **^{31}P NMR (162 MHz,**

$CDCl_3$) δ 27.18. **HRMS** (ESI): m/z $[M+H]^+$ calculated for $C_{21}H_{20}O_2P$: 335.1195, found: 335.1196.



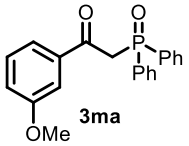
3ka

White solid; Mp: 98−99°C. **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.90 (d, *J* = 8.3 Hz, 2H), 7.85 – 7.77 (m, 4H), 7.56 – 7.50 (m, 2H), 7.49 – 7.42 (m, 4H), 7.22 (d, *J* = 8.3 Hz, 2H), 4.13 (d, *J* = 15.4 Hz, 2H), 2.70 – 2.55 (m, 2H), 1.65 – 1.51 (m, 2H), 1.32 (dt, *J* = 14.7, 7.4 Hz, 2H), 0.92 (t, *J* = 7.3 Hz, 3H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ (192.25, 192.20, d, *J* = 5.1 Hz), 149.34, 134.64, (132.45, 131.43, d, *J* = 103.0 Hz), (132.00, 131.98, d, *J* = 2.0 Hz), (131.10, 131.00, d, *J* = 10.1 Hz), 129.31, 128.54, (128.50, 128.42, d, *J* = 8.1 Hz), (43.36, 42.78, d, *J* = 58.6 Hz), 35.59, 33.01, 22.17, 13.79. **^{31}P NMR (162 MHz, $CDCl_3$)** δ 27.16. **HRMS** (ESI): m/z $[M+H]^+$ calculated for $C_{24}H_{26}O_2P$:377.1665, found: 377.1667.



3la

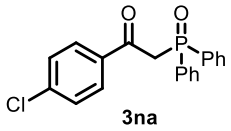
White solid; Mp: 148−150°C. **^{1}H NMR (400 MHz, $CDCl_3$)** δ 8.07 (d, *J* = 8.3 Hz, 2H), 7.82 (dd, *J* = 12.0, 7.2 Hz, 4H), 7.61 (dd, *J* = 15.7, 7.8 Hz, 4H), 7.54 – 7.35 (m, 9H), 4.17 (d, *J* = 15.2 Hz, 2H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ (192.17, 192.12, d, *J* = 5.1 Hz), 146.01, 139.52, 135.43, (132.19, 131.16, d, *J* = 104.0 Hz), (132.06, 132.04, d, *J* = 2.0 Hz), (130.98, 130.89, d, *J* = 9.1 Hz), 129.77, 128.80, (128.56, 128.44, d, *J* = 12.1 Hz), 128.17, 127.12, 127.00, (43.43, 42.85, d, *J* = 58.6 Hz). **^{31}P NMR (162 MHz, $CDCl_3$)** δ 27.17. **HRMS** (ESI): m/z $[M+H]^+$ calculated for $C_{26}H_{22}O_2P$:397.1352, found: 397.1358.



3ma

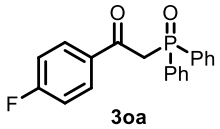
White solid; Mp: 105−107°C. **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.86 – 7.76 (m, 4H), 7.61 (d, *J* = 7.7 Hz, 1H), 7.57 – 7.51 (m, 2H), 7.50 – 7.43 (m, 5H), 7.33 (t, *J* = 8.0 Hz, 1H), 7.09 (dd, *J* = 8.2, 2.1 Hz, 1H),

4.14 (d, *J* = 15.3 Hz, 2H), 3.81 (s, 3H). **^{13}C NMR (101 MHz, $CDCl_3$)** *δ* (192.63, 192.58, d, *J* = 5.1 Hz), 159.57, 138.17, (132.31, 131.28, d, *J* = 104.0 Hz), (132.13, 132.10, d, *J* = 3.0 Hz), (131.10, 131.01, d, *J* = 9.1 Hz), 129.50, (128.63, 128.50, d, *J* = 13.1 Hz), 122.19, 120.57, 112.55, 55.37, (43.60, 43.02, d, *J* = 58.6 Hz). **^{31}P NMR (162 MHz, $CDCl_3$)** *δ* 27.12. **HRMS** (ESI): m/z $[M+H]^+$ calculated for $C_{21}H_{20}O_3P$: 351.1145, found: 351.1147.



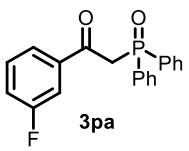
3na

White solid; Mp: 167−168°C. **^{1}H NMR (400 MHz, $CDCl_3$)** *δ* 7.96 (d, *J* = 8.6 Hz, 2H), 7.85 – 7.74 (m, 4H), 7.57 – 7.51 (m, 2H), 7.47 (ddd, *J* = 7.1, 5.4, 2.4 Hz, 4H), 7.39 (d, *J* = 8.6 Hz, 2H), 4.11 (d, *J* = 15.2 Hz, 2H). **^{13}C NMR (101 MHz, $CDCl_3$)** *δ* (191.56, 191.51, d, *J* = 5.1 Hz), 140.12, 135.20, (132.19, 131.16, d, *J* = 104.0 Hz), (132.19, 132.16, d, *J* = 3.0 Hz), (131.00, 130.91, d, *J* = 9.1 Hz), 130.68, 128.74, (128.66, 128.54, d, *J* = 12.1 Hz), (43.80, 43.24, d, *J* = 56.6 Hz). **^{31}P NMR (162 MHz, $CDCl_3$)** *δ* 26.60. **HRMS** (ESI): m/z $[M+H]^+$ calculated for $C_{20}H_{17}ClO_2P$:355.0649, found: 355.0648.



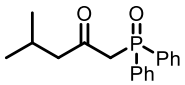
3oa

White solid; Mp: 164−165°C. **^{1}H NMR (400 MHz, $CDCl_3$)** *δ* 8.12 – 8.00 (m, 2H), 7.86 – 7.75 (m, 4H), 7.57 – 7.51 (m, 2H), 7.47 (ddd, *J* = 7.0, 5.4, 2.4 Hz, 4H), 7.09 (t, *J* = 8.6 Hz, 2H), 4.12 (d, *J* = 15.3 Hz, 2H). **^{13}C NMR (101 MHz, $CDCl_3$)** *δ* (191.15, 191.10, d, *J* = 5.1 Hz), (167.27, 164.72, d, *J* = 257.6 Hz), (133.36, 133.34, d, *J* = 2.0 Hz), (132.20, 132.17, d, *J* = 3.0 Hz), (132.16, 131.13, d, *J* = 104.0 Hz), (132.12, 132.03, d, *J* = 9.1 Hz), (131.04, 130.95, d, *J* = 9.1 Hz), (128.67, 128.54, d, *J* = 13.1 Hz), (115.69, 115.47, d, *J* = 22.2 Hz), (43.72, 43.15, d, *J* = 57.6 Hz). **^{31}P NMR (162 MHz, $CDCl_3$)** *δ* 26.95. **HRMS** (ESI): m/z $[M+H]^+$ calculated for $C_{20}H_{17}FO_2P$: 339.0945, found: 339.0944.



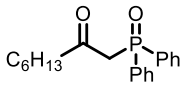
3pa

White solid; Mp: 95−96°C. **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.88 – 7.75 (m, 5H), 7.68 – 7.61 (m, 1H), 7.58 – 7.51 (m, 2H), 7.51 – 7.44 (m, 4H), 7.44 – 7.38 (m, 1H), 7.27 – 7.21 (m, 1H), 4.13 (d, *J* = 15.2 Hz, 2H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ (191.66, 191.61, d, *J* = 5.1 Hz), (163.82, 161.36, d, *J* = 248.5 Hz), (138.98, 138.92, d, *J* = 6.1 Hz), (132.25, 132.22, d, *J* = 3.0 Hz), (132.15, 131.12, d, *J* = 104.0 Hz), (131.07, 130.97, d, *J* = 10.1 Hz), (130.22, 130.14, d, *J* = 8.1 Hz), (128.70, 128.57, d, *J* = 13.1 Hz), (125.30, 125.27, d, *J* = 3.0 Hz), (120.69, 120.48, d, *J* = 21.2 Hz), (115.69, 115.47, d, *J* = 22.2 Hz), (43.81, 43.24, d, *J* = 57.6 Hz). **^{31}P NMR (162 MHz, $CDCl_3$)** δ 26.76. **HRMS** (ESI): m/z $[M+H]^+$ calculated for $C_{20}H_{17}FO_2P$: 339.0945, found: 339.0949.



3ra

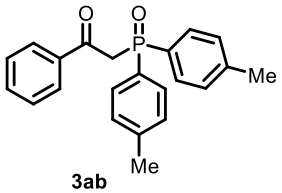
Yellow oil. **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.80 – 7.73 (m, 4H), 7.58 – 7.53 (m, 2H), 7.52 – 7.46 (m, 4H), 3.58 (d, *J* = 15.0 Hz, 2H), 2.53 (d, *J* = 6.8 Hz, 2H), 2.07 (dt, *J* = 17.2, 6.7 Hz, 1H), 0.82 (d, *J* = 6.7 Hz, 6H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ (202.79, 202.74, d, *J* = 5.1 Hz), (132.24, 131.22, d, *J* = 103.0 Hz), (132.24, 132.22, d, *J* = 2.0 Hz), (130.90, 130.80, d, *J* = 10.1 Hz), (128.77, 128.65, d, *J* = 12.1 Hz), 54.04, (47.52, 46.96, d, *J* = 56.6 Hz), 24.02, 22.26. **^{31}P NMR (162 MHz, $CDCl_3$)** δ 26.78. **HRMS** (ESI): m/z $[M+H]^+$ calculated for $C_{18}H_{22}O_2P$:301.1352, found: 301.1354.



3sa

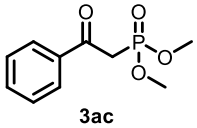
Yellow oil. **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.80 – 7.73 (m, 4H), 7.58 – 7.46 (m, 6H), 3.60 (d, *J* = 15.0 Hz, 2H), 2.64 (t, *J* = 7.3 Hz, 2H), 1.54 – 1.41 (m, 2H), 1.26 – 1.13 (m, 6H), 0.84 (t, *J* = 7.0 Hz, 3H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ (203.28, 203.22, d, *J* = 6.1 Hz), (132.30, 131.27, d, *J* = 104.0 Hz), (132.23, 132.20, d, *J* = 3.0 Hz), (130.90, 130.80, d, *J* = 10.1 Hz), (128.77, 128.65, d, *J* = 12.1 Hz),

(47.28, 46.72, d, *J* = 56.6 Hz), 45.33, 31.48, 28.45, 23.16, 22.42, 14.00. **^{31}P NMR (162 MHz, $CDCl_3$)** *δ* 26.69. **HRMS** (ESI): m/z $[M+H]^+$ calculated for $C_{20}H_{26}O_2P$:329.1665, found: 329.1666.



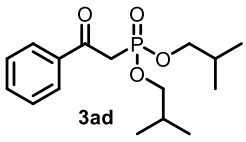
3ab

White solid; Mp: 91−93°C. **^{1}H NMR (400 MHz, $CDCl_3$)** *δ* 7.99 (d, *J* = 7.5 Hz, 2H), 7.67 (dd, *J* = 12.0, 8.0 Hz, 4H), 7.52 (t, *J* = 7.4 Hz, 1H), 7.40 (t, *J* = 7.7 Hz, 2H), 7.25 (dd, *J* = 8.2, 2.7 Hz, 4H), 4.11 (d, *J* = 15.4 Hz, 2H), 2.37 (s, 6H). **^{13}C NMR (101 MHz, $CDCl_3$)** *δ* (192.98, 192.93, d, *J* = 5.1 Hz), (142.57, 142.54, d, *J* = 3.0 Hz), 136.99, 133.40, (131.12, 131.01, d, *J* = 11.1 Hz), (129.33, 129.21, d, *J* = 12.1 Hz), 129.21, (129.21, 128.18, d, *J* = 104.0 Hz), 128.40, (43.76, 43.19, d, *J* = 57.6 Hz), 21.52. **^{31}P NMR (162 MHz, $CDCl_3$)** *δ* 27.63. **HRMS** (ESI): m/z $[M+H]^+$ calculated for $C_{26}H_{22}O_2P$:397.1352, found: 397.1358. **HRMS** (ESI): m/z $[M+H]^+$ calculated for $C_{22}H_{22}O_2P$:349.1352, found: 349.1354.



3ac

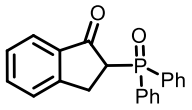
Yellow oil. **^{1}H NMR (400 MHz, $CDCl_3$)** *δ* 8.05 – 7.96 (m, 2H), 7.65 – 7.58 (m, 1H), 7.50 (dd, *J* = 10.6, 4.8 Hz, 2H), 3.81 (s, 3H), 3.78 (s, 3H), 3.67 (d, *J* = 22.6 Hz, 2H). **^{13}C NMR (101 MHz, $CDCl_3$)** *δ* (191.76, 191.69, d, *J* = 7.1 Hz), 136.26, 133.85, 128.96, 128.70, (53.29, 53.22, d, *J* = 7.1 Hz), (37.99, 36.68, d, *J* = 132.3 Hz). **^{31}P NMR (162 MHz, $CDCl_3$)** *δ* 23.06. **HRMS** (ESI): m/z $[M+H]^+$ calculated for $C_{10}H_{14}O_4P$:229.0624, found: 229.0628.



3ad

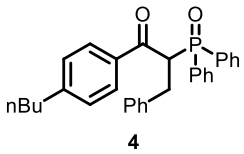
Yellow oil. **^{1}H NMR (400 MHz, $CDCl_3$)** *δ* 8.02 (d, *J* = 7.7 Hz, 2H), 7.59 (t, *J* = 7.3 Hz, 1H), 7.48 (t, *J* = 7.6 Hz, 2H), 3.89 – 3.79 (m, 4H), 3.66 (d, *J* = 22.9 Hz, 2H), 1.87 (td, *J* = 13.3, 6.6 Hz, 2H), 0.88 (d, *J*

= 6.7 Hz, 12H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ (191.89, 191.82, d, *J* = 7.1 Hz), 136.44, 133.63, 129.03, 128.58, (72.40, 72.33, d, *J* = 7.1 Hz), (38.74, 37.46, d, *J* = 129.3 Hz), (29.13, 29.06, d, *J* = 7.1 Hz), (18.57, 18.56, d, *J* = 1.0 Hz). **^{31}P NMR (162 MHz, $CDCl_3$)** δ 19.73. **HRMS** (ESI): m/z $[M+H]^+$ calculated for $C_{16}H_{26}O_4P$:313.1563, found: 313.1565.



3ta

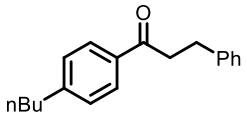
White solid; Mp: 86−88°C. **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.94 (dd, *J* = 12.0, 6.9 Hz, 2H), 7.71 (dd, *J* = 11.9, 7.2 Hz, 2H), 7.62 (d, *J* = 7.7 Hz, 1H), 7.59 – 7.49 (m, 4H), 7.48 – 7.42 (m, 2H), 7.38 (dd, *J* = 12.6, 5.1 Hz, 2H), 7.31 (d, *J* = 7.4 Hz, 1H), 3.96 (ddd, *J* = 15.7, 8.4, 3.5 Hz, 1H), 3.70 – 3.55 (m, 1H), 3.49 – 3.36 (m, 1H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ (199.87, 199.84, d, *J* = 3.0 Hz), (153.10, 153.05, d, *J* = 5.1 Hz), (136.95, 136.94, d, *J* = 1.0 Hz), 13 5.01, (132.40, 131.38, d, *J* = 103.0 Hz), (132.19, 132.16, d, *J* = 3.0 Hz), (132.09, 132.06, d, *J* = 3.0 Hz), (131.66, 131.57, d, *J* = 9.1 Hz), (131.41, 131.31, d, *J* = 10.1 Hz), (130.35, 129.33, d, *J* = 103.0 Hz), (128.74, 128.62, d, *J* = 12.1 Hz), (128.31, 128.18, d, *J* = 13.1 Hz), 127.61, 126.29, 124.11, (49.09, 48.46, d, *J* = 63.6 Hz), 28.27. **^{31}P NMR (162 MHz, $CDCl_3$)** δ 32.22. HRMS (ESI) m/z: $[M + H]^+$ calcd for $C_{21}H_{18}O_2P$ 333.1039, found 333.1045.



4

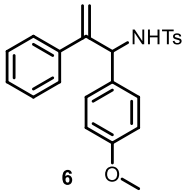
White solid; Mp: 133−135°C.**^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.99 – 7.88 (m, 2H), 7.80 – 7.73 (m, 2H), 7.45 – 7.38 (m, 6H), 7.34 (ddd, *J* = 7.1, 5.4, 2.4 Hz, 2H), 7.15 – 7.05 (m, 5H), 6.96 (d, *J* = 8.2 Hz, 2H), 4.83 (ddd, *J* = 15.8, 11.3, 2.4 Hz, 1H), 3.56 (ddd, *J* = 13.9, 11.4, 4.8 Hz, 1H), 3.30 – 3.16 (m, 1H), 2.51 (t, *J* = 7.6 Hz, 2H), 1.53 – 1.47 (m, 2H), 1.29 – 1.23 (m, 2H), 0.89 (t, *J* = 7.3 Hz, 3H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ (196.82, 196.79, d, *J* = 3.0 Hz), 148.39, (139.15, 139.01, d, *J* = 14.1 Hz), 135.94, (131.98, 131.96, d, *J* = 2.0 Hz), (131.90, 131.88, d, *J* = 2.0 Hz), (131.83, 131.74, d, *J* = 9.1

Hz), (131.38, 131.29, d, *J* = 9.1 Hz), (130.78, 129.79, d, *J* = 100.0 Hz), 130.32, (128.44, 128.41, d, *J* = 3.0 Hz), 128.30, 128.10, 126.39, (54.19, 53.65, d, *J* =54.5 Hz), 35.34, 33.79, 32.91, 22.00, 13.73. **^{31}P NMR (162 MHz, $CDCl_3$)** δ 29.34. **HRMS** (ESI): m/z $[M+H]^+$ calculated for $C_{31}H_{32}O_2P$:467.2134, found: 467.2135.



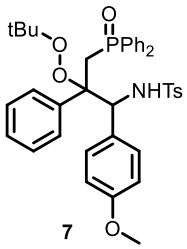
5

Colorless oil. **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.88 (d, *J* = 8.2 Hz, 2H), 7.33 – 7.18 (m, 7H), 3.33 – 3.22 (m, 2H), 3.06 (dd, *J* = 10.1, 5.3 Hz, 2H), 2.73 – 2.60 (m, 2H), 1.65 – 1.56 (m, 2H), 1.36 (dt, *J* = 14.9, 7.4 Hz, 2H), 0.93 (t, *J* = 7.3 Hz, 3H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 198.9, 148.8, 141.4, 134.6, 128.6, 128.5, 128.4, 128.2, 126.1, 40.4, 35.7, 33.2, 30.2, 22.3, 13.9. **HRMS** (ESI): m/z $[M+H]^+$ calculated for $C_{19}H_{23}O$:267.1743, found: 267.1746.



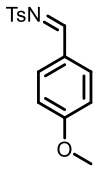
6

Viscous oil. **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.58 (d, *J* = 8.3 Hz, 2H), 7.21 – 7.14 (m, 5H), 7.14 – 7.03 (m, 4H), 6.76 – 6.69 (m, 2H), 5.41 (d, *J* = 7.6 Hz, 1H), 5.36 (s, 1H), 5.19 (s, 1H), 5.11 (d, *J* = 7.6 Hz, 1H), 3.73 (s, 3H), 2.39 (s, 3H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 159.0, 147.3, 143.1, 138.9, 137.5, 131.1, 129.3, 128.6, 128.3, 127.8, 127.2, 126.8, 116.0, 113.9, 60.3, 55.2, 21.4. **HRMS** (ESI): m/z $[M+H]^+$ calculated for $C_{23}H_{24}NO_3S$:394.1471, found: 394.1474.



7

White solid; Mp: 79−82°C. **^{1}H NMR (400 MHz, $CDCl_3$)** δ 8.21 (d, J = 7.7 Hz, 1H), 7.74 – 7.65 (m, 2H), 7.64 – 7.56 (m, 2H), 7.47 (dd, J = 7.3, 1.4 Hz, 1H), 7.44 – 7.38 (m, 3H), 7.38 – 7.30 (m, 4H), 7.23 – 7.16 (m, 2H), 7.05 (t, J = 7.3 Hz, 1H), 7.01 – 6.89 (m, 4H), 6.80 (d, J = 8.6 Hz, 2H), 6.38 (d, J = 8.8 Hz, 2H), 5.27 (d, J = 8.9 Hz, 1H), 3.67 (s, 3H), 3.37 (dd, J = 15.4, 11.4 Hz, 1H), 3.15 (dd, J = 15.4, 12.2 Hz, 1H), 2.28 (s, 3H), 1.01 (s, 9H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 158.41, 141.76, (138.64, 138.60, d, J = 4.0 Hz), 138.54, (134.47, 134.39, d, J = 8.1 Hz), (133.44, 133.38, d, J = 6.1 Hz), (131.42, 131.39, d, J = 3.0 Hz), (131.26, 131.23, d, J = 3.0 Hz), (130.90, 130.80, d, J = 10.1 Hz), 130.72, (130.62, 130.53, d, J = 9.1 Hz), 128.68, (128.52, 128.40, d, J = 12.1 Hz), (128.36, 128.24, d, J = 12.1 Hz), 127.92, 127.70, 127.29, 126.98, 126.82, 112.25, (86.56, 86.53, d, J = 3.0 Hz), 80.89, 63.41, 55.02, (35.35, 34.65， d, J = 70.7 Hz), 26.55, 21.25. **HRMS** (ESI): m/z $[M+H]^+$ calculated for $C_{39}H_{43}NO_6PS$:684.2543, found: 684.2545.



8

White solid; Mp: 124−126°C. **^{1}H NMR (400 MHz, $CDCl_3$)** δ 8.93 (s, 1H), 7.87 (d, J = 8.2 Hz, 4H), 7.32 (d, J = 8.1 Hz, 2H), 6.96 (d, J = 8.8 Hz, 2H), 3.87 (s, 3H), 2.41 (s, 3H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 169.1, 165.2, 144.2, 135.7, 133.6, 129.6, 127.8, 125.1, 114.6, 55.6, 21.5. **HRMS** (ESI): m/z $[M+H]^+$ calculated for $C_{15}H_{16}NO_3S$:290.0845, found: 290.0848.

References

[1] Z. Shen, X. Pan, Y. Lai, J. Hu, X. Wan, X. Li, H. Zhang, W. Xie, *Chem. Sci.* **2015**, *6*, 6986.

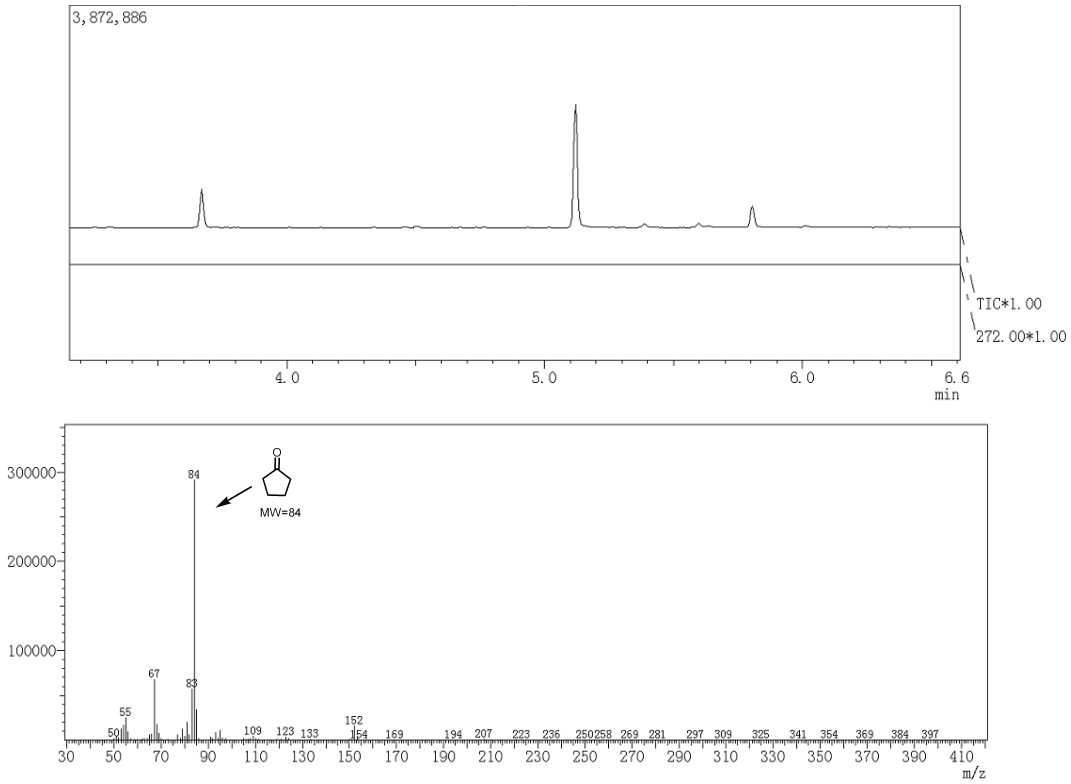
[2] X. Shu, M. Zhang, Y. He, H. Frei, F. D. Toste, *J. Am. Chem. Soc.* **2014**, *136*, 5844.

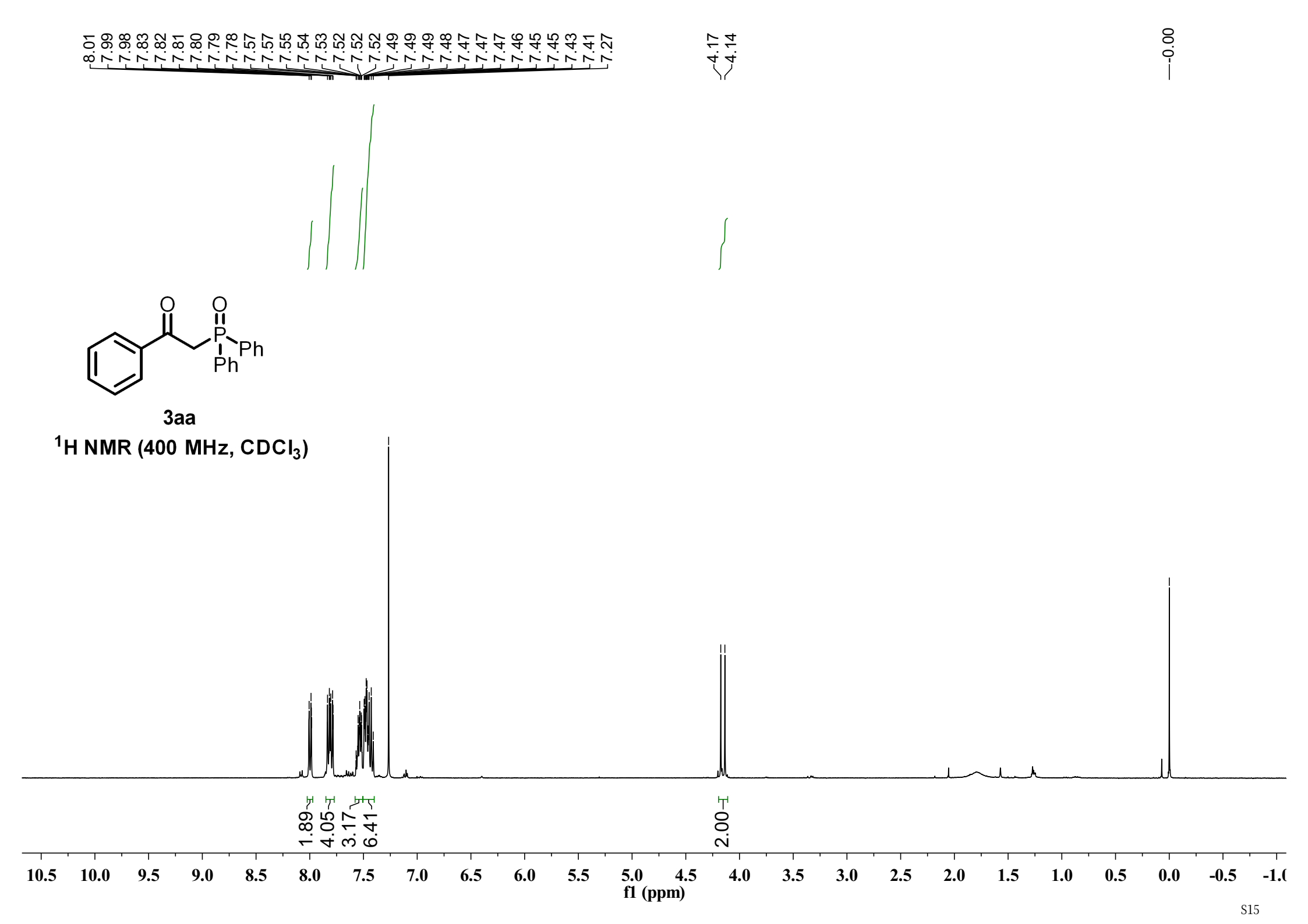
[3] W. Weng, J. Sun, P. Li, B. Zhang, *Chem. Eur. J.* **2017**, *23*, 9752.

[4] Z. Duan, X. Hu, C. Zhang, D. Wang, S. Yu, Z. Zheng, *J. Org. Chem.* **2009**, *74*, 9191.

[5] D. H. Lukamto, M. J. Gaunt, *J. Am. Chem. Soc.* **2017**, *139*, 9160.

GC-MS spectrum of cyclopentanone

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7.99
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7.57
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7.47
7.47
7.46
7.45
7.45
7.43
7.41
7.27
4.17
4.14
0.00
O
O
P
Ph
Ph
3aa
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4.05
3.17
6.41
2.00
10.5
10.0
9.5
9.0
8.5
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7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
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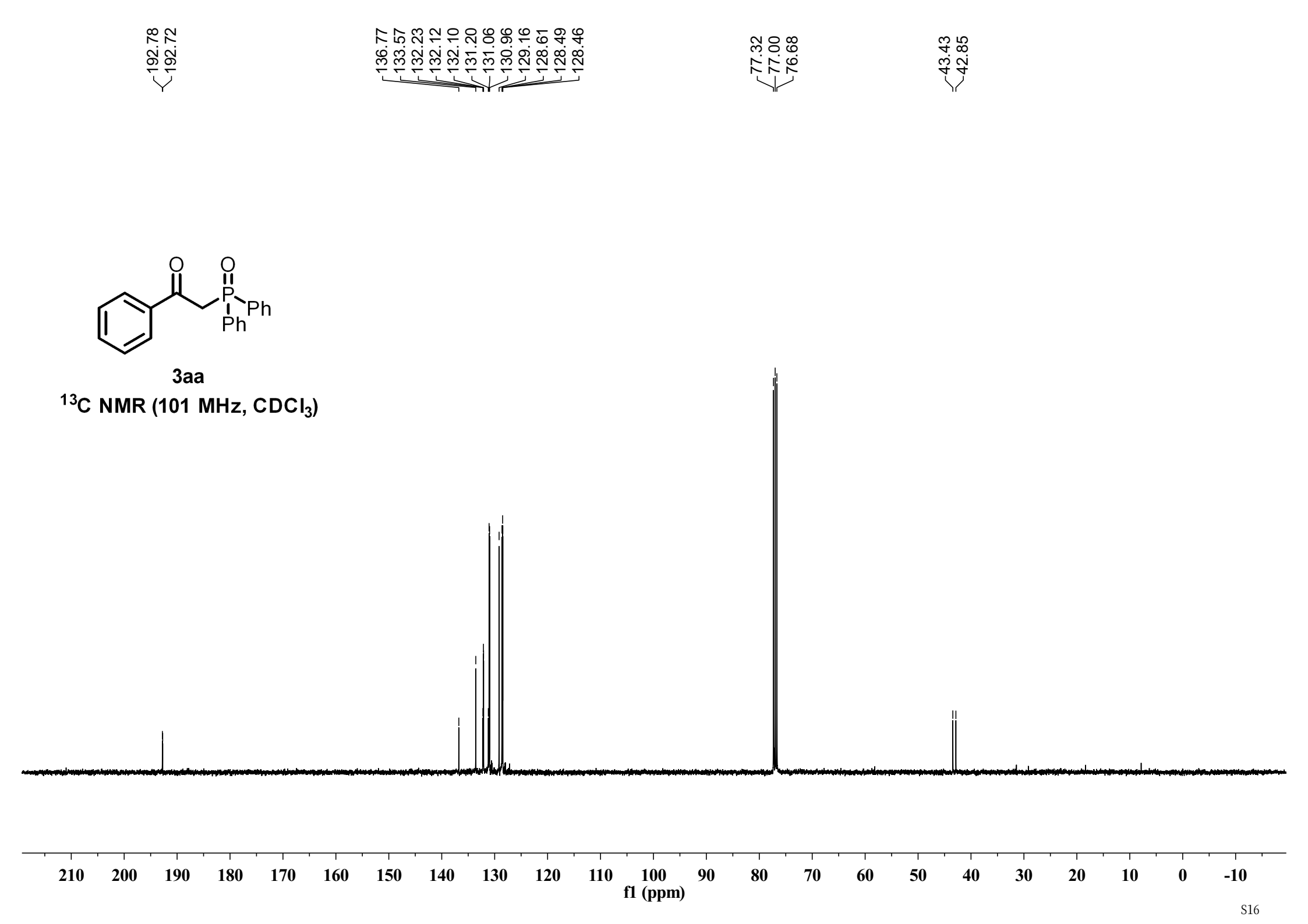
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)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192.72
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132.12
132.10
131.20
131.06
130.96
129.16
128.61
128.49
128.46
77.32
77.00
76.68
43.43
42.85
O
O
P
Ph
Ph
3aa
13C NMR (101 MHz, CDCl3)
210
200
190
180
170
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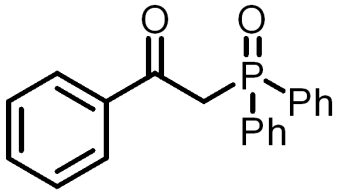
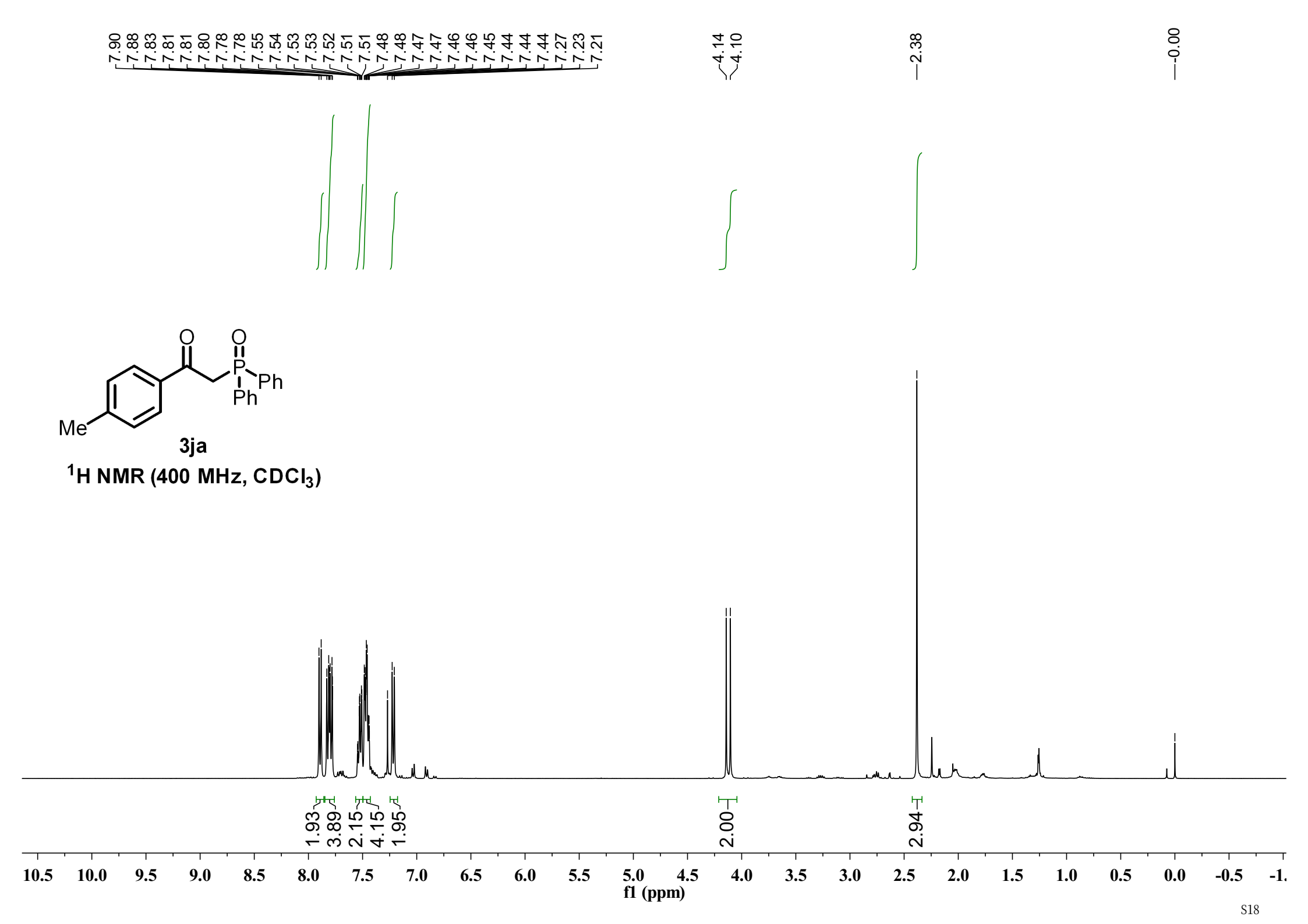
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)



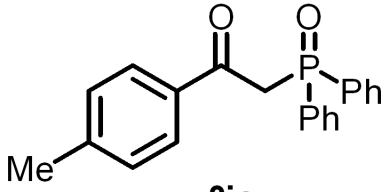
3aa

^{31}P NMR (162 MHz, $CDCl_3$)



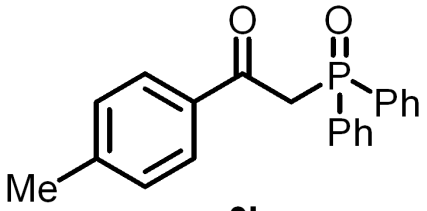
3ja

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



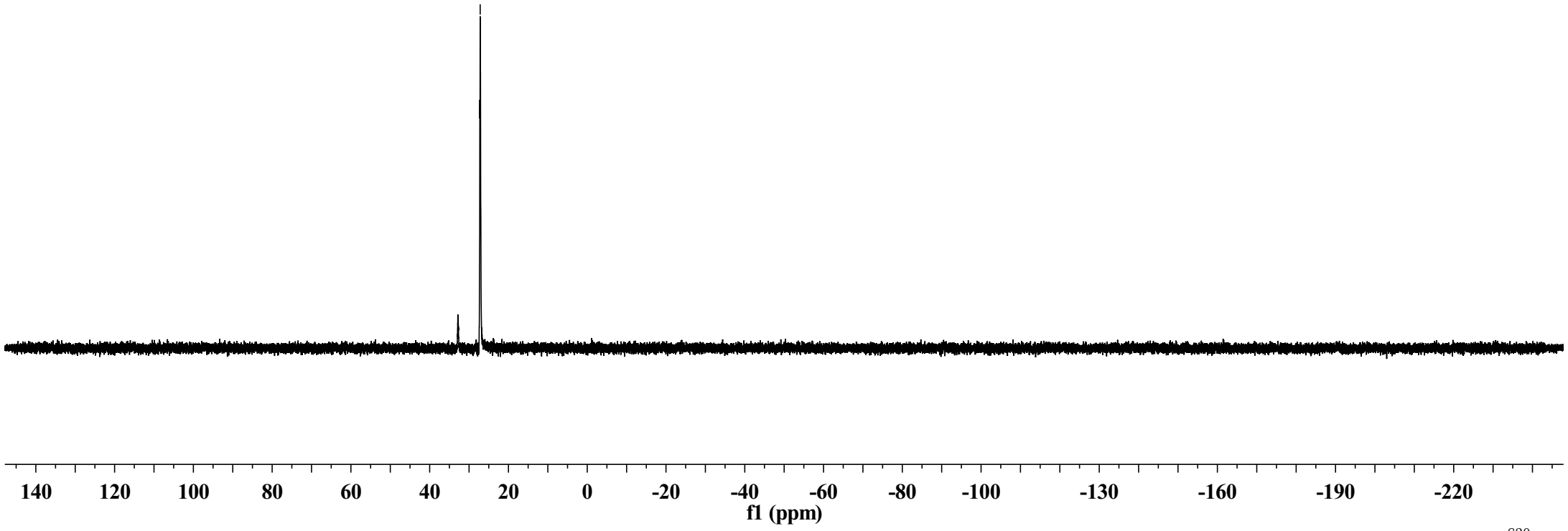
3ja

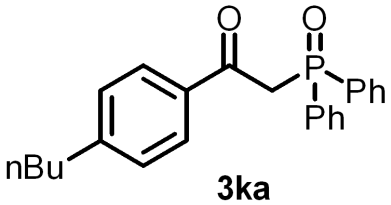
^{13}C NMR (101 MHz, $CDCl_3$)



3ja

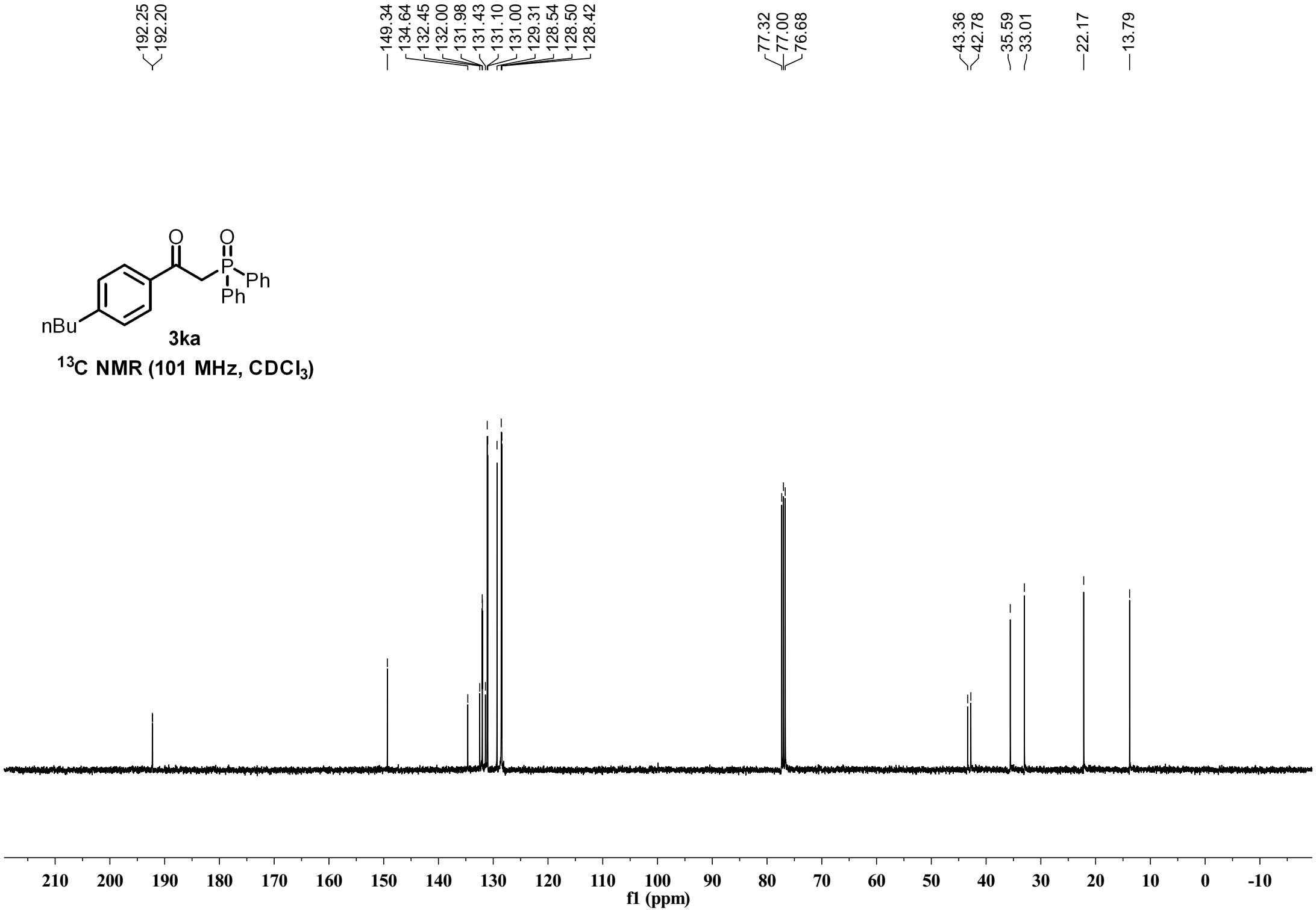
^{31}P NMR (162 MHz, $CDCl_3$)





3ka

^{1}H NMR (400 MHz, $CDCl_3$)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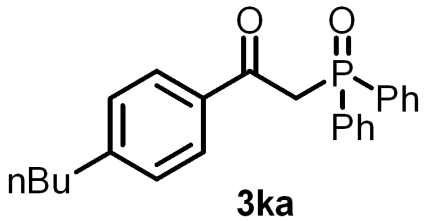
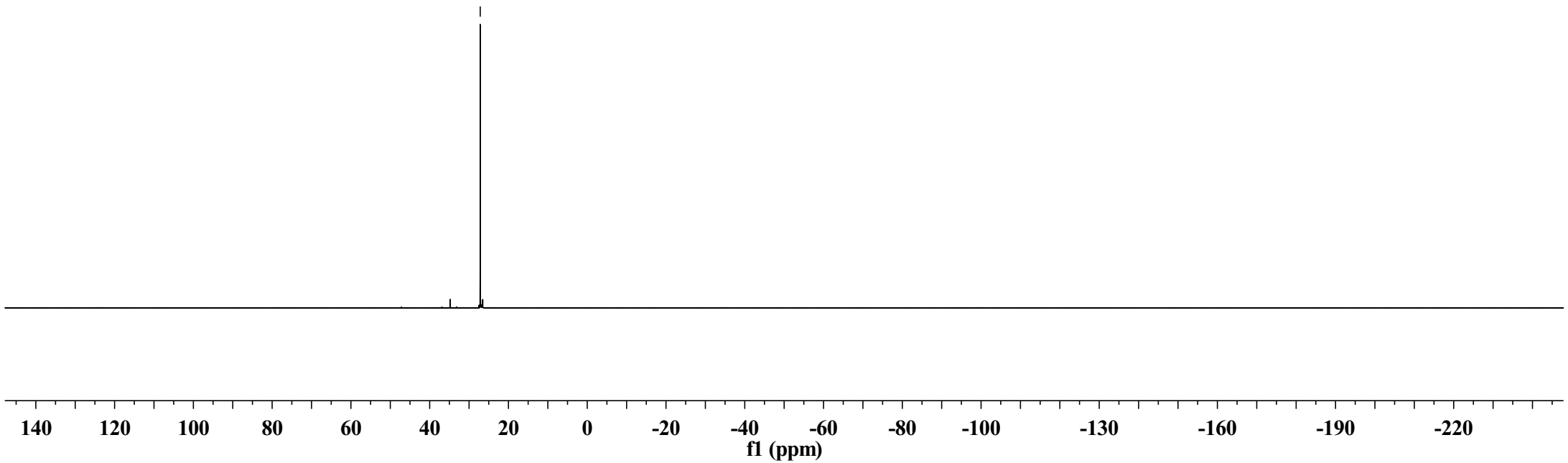
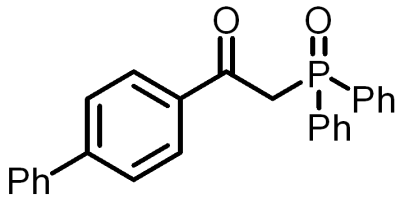
128.42
77.32
77.00
76.68
43.36
42.78
35.59
33.01
22.17
13.79
O
O
P
Ph
Ph
nBu
3ka
13C NMR (101 MHz, CDCl3)
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)



3ka

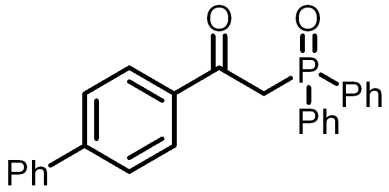
^{31}P NMR (162 MHz, $CDCl_3$)





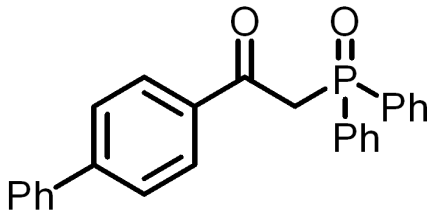
3la

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



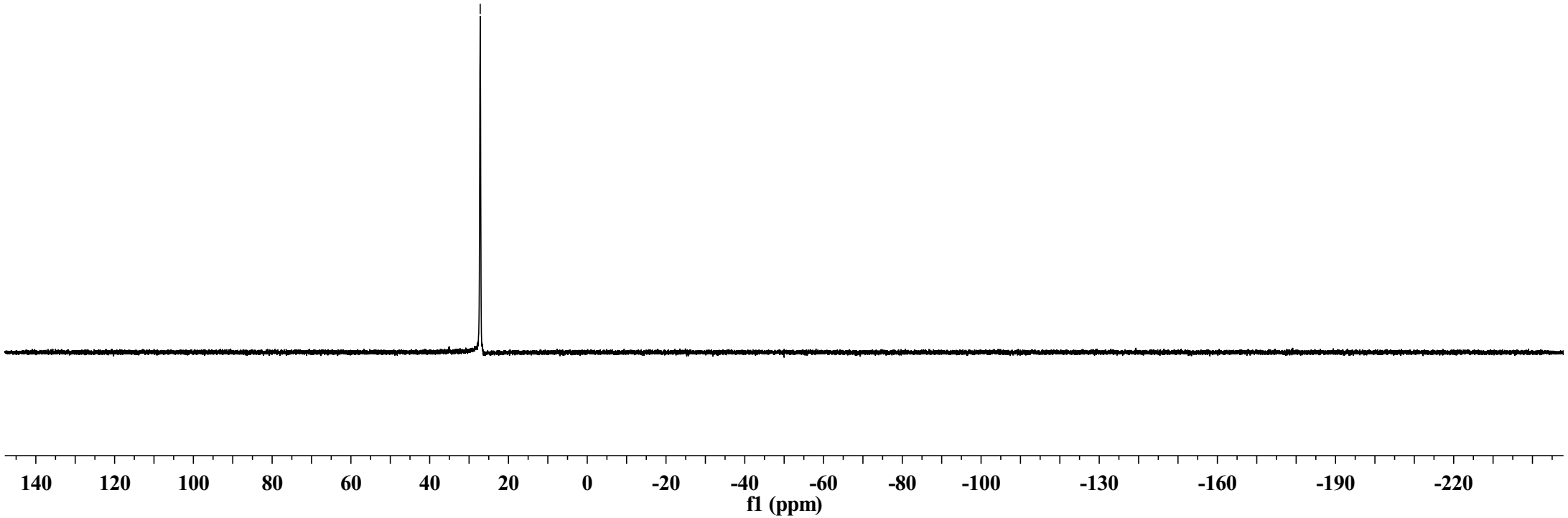
3la

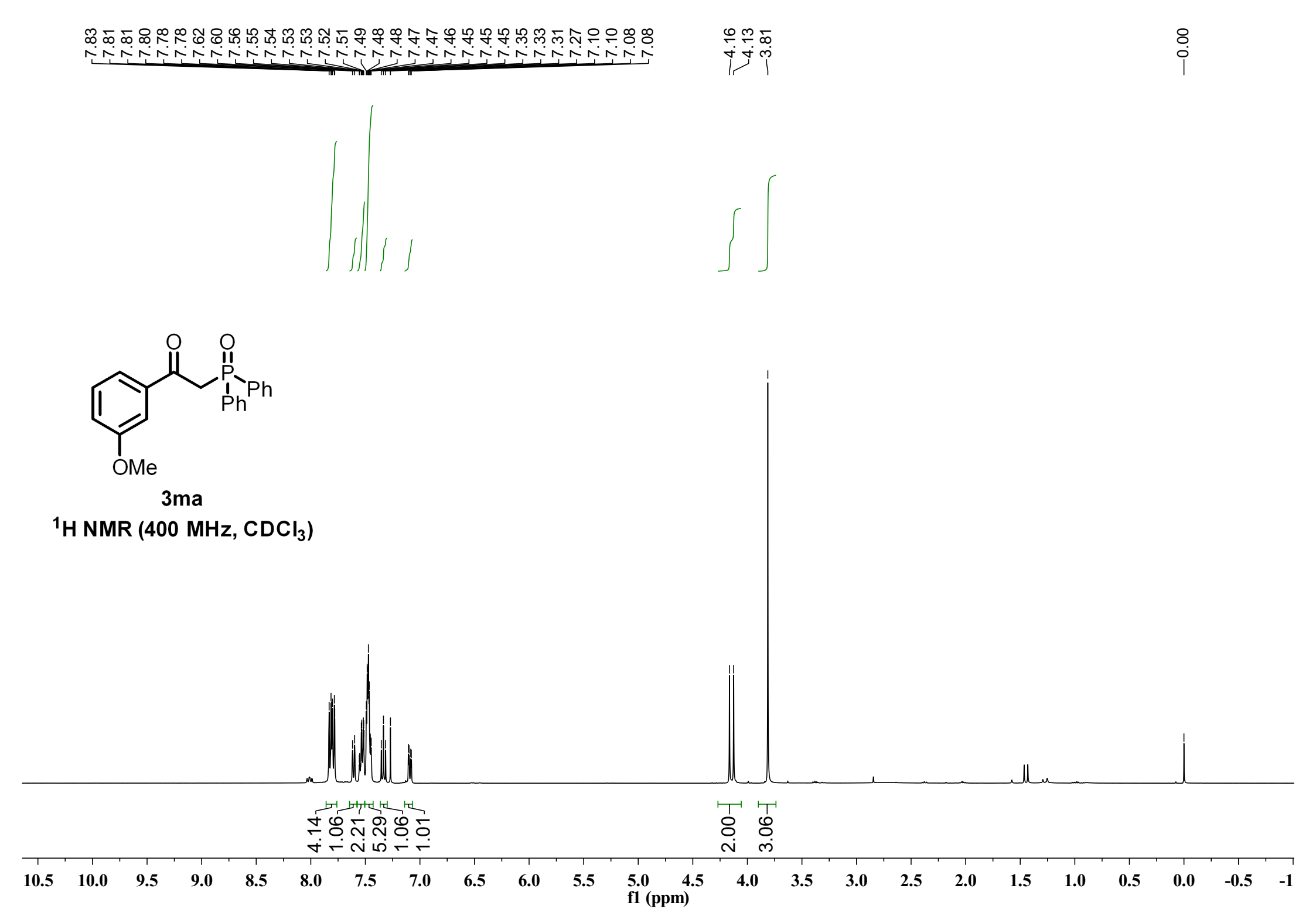
^{13}C NMR (101 MHz, $CDCl_3$)



3la

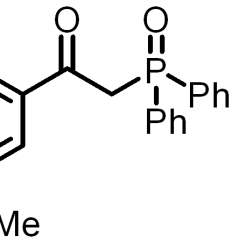
^{31}P NMR (162 MHz, $CDCl_3$)





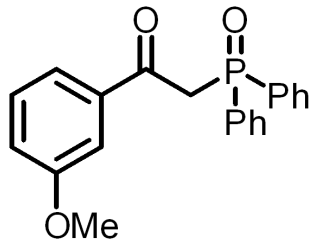
3ma

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



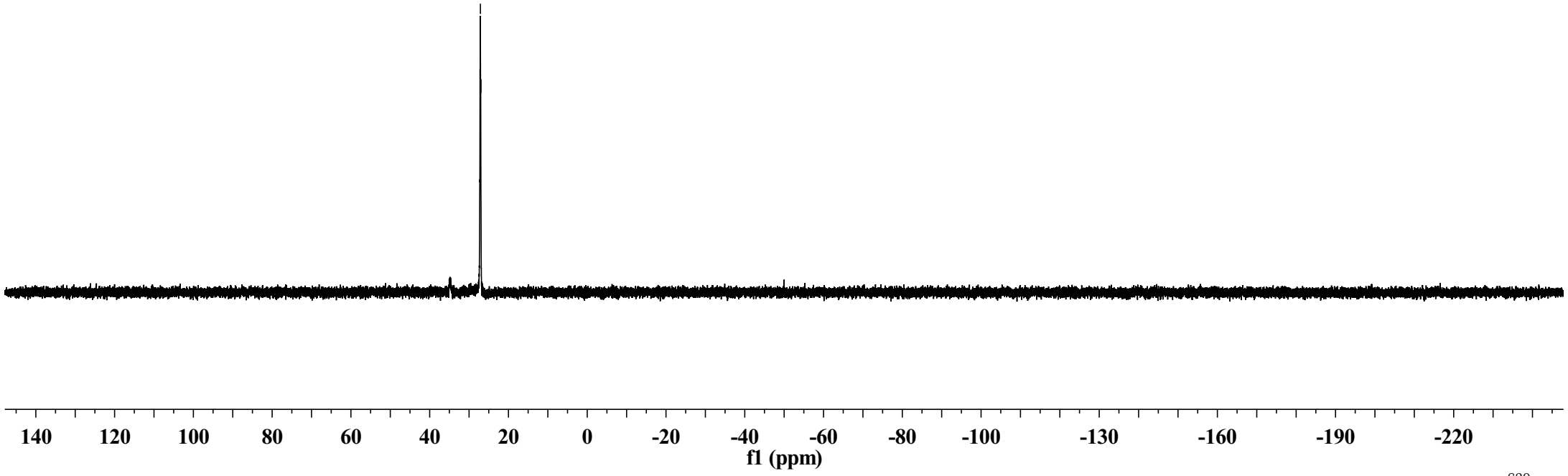
3ma

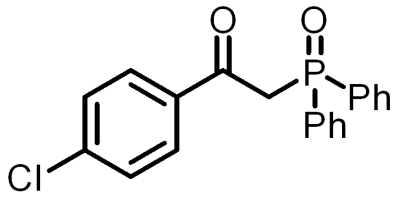
^{13}C NMR (101 MHz, $CDCl_3$)



3ma

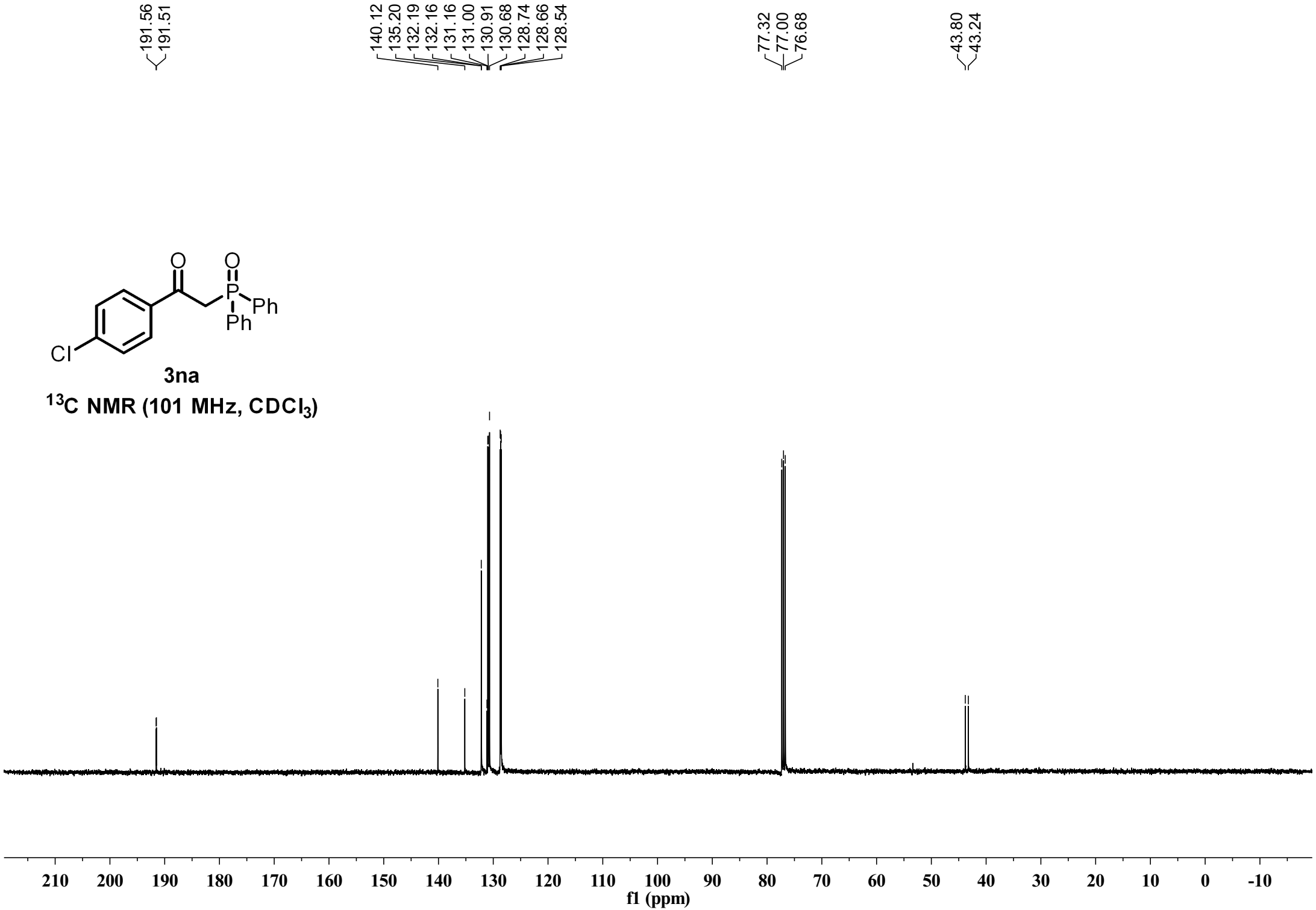
^{31}P NMR (162 MHz, $CDCl_3$)

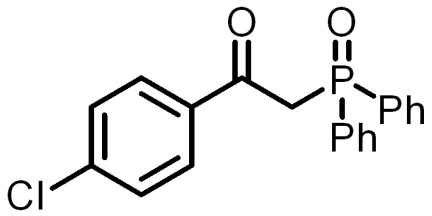




3na

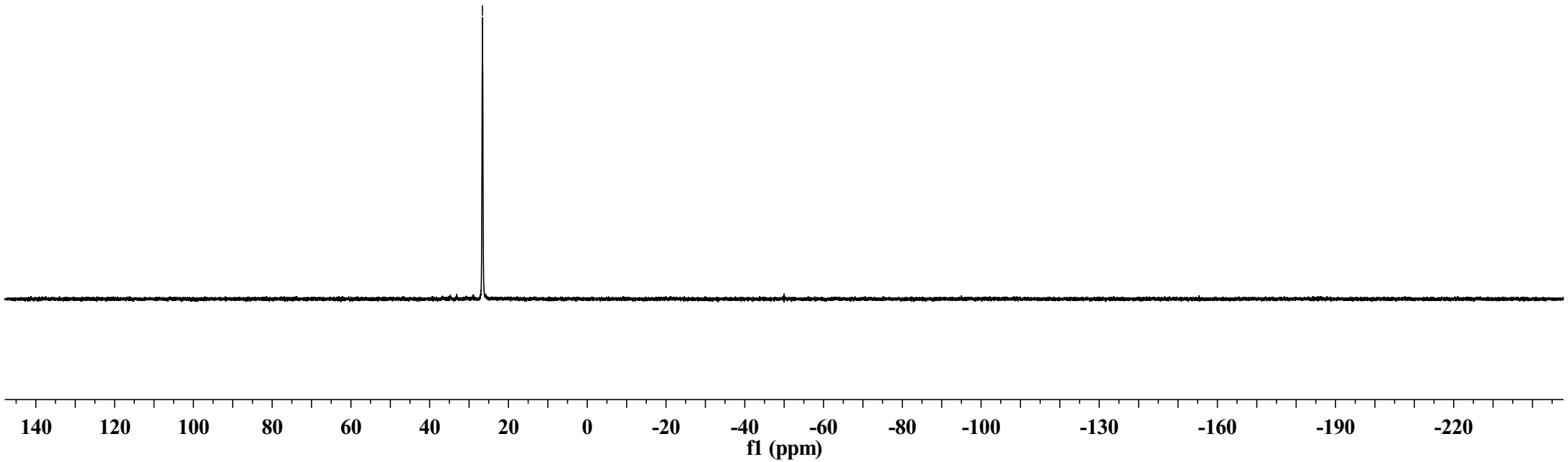
^{1}H NMR (400 MHz, $CDCl_3$)

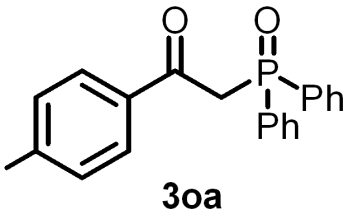

191.56
191.51
140.12
135.20
132.19
132.16
131.16
131.00
130.91
130.68
128.74
128.66
128.54
77.32
77.00
76.68
43.80
43.24
O
O
P
Ph
Ph
Cl
3na
13C NMR (101 MHz, CDCl3)
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)



3na

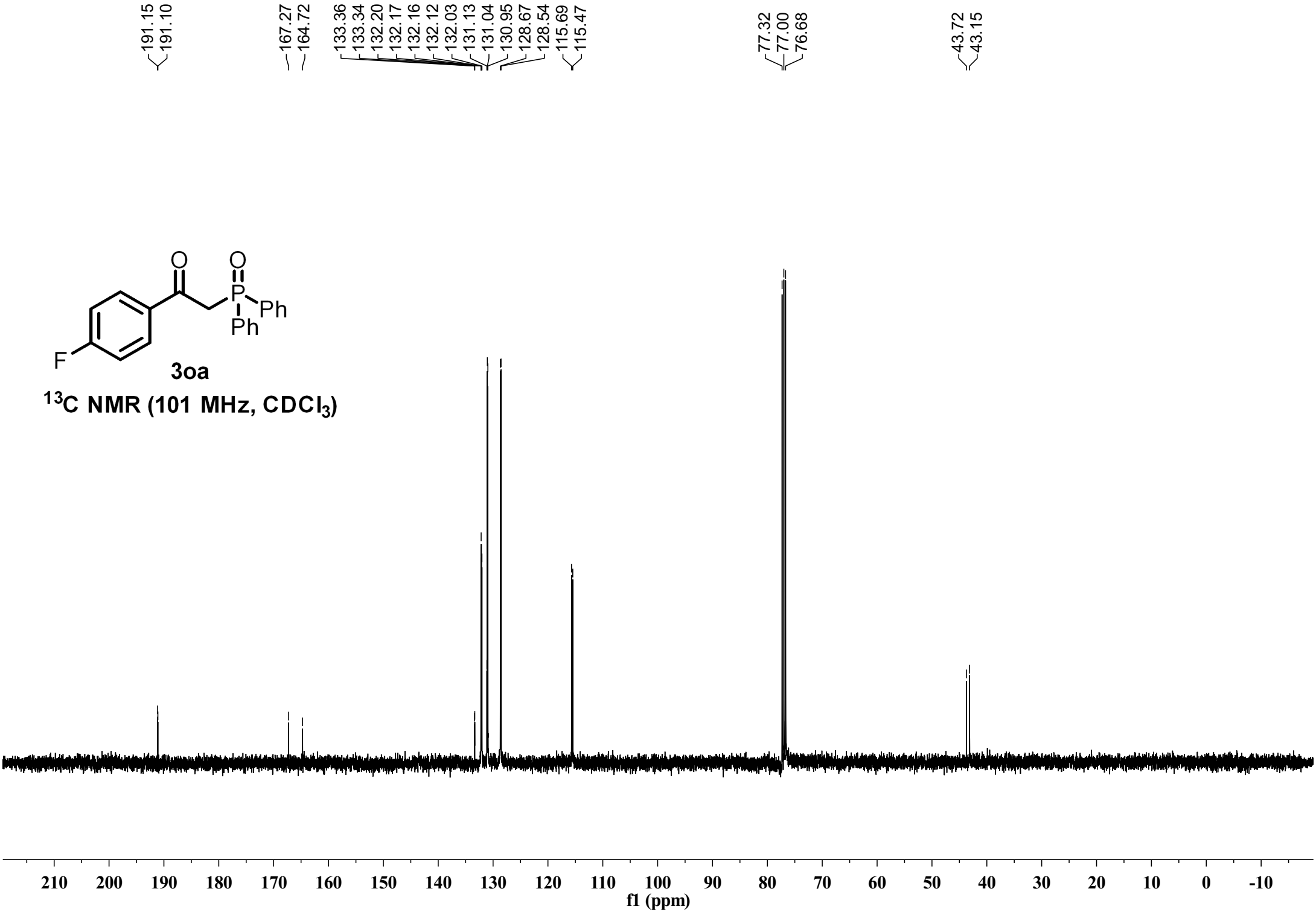
^{31}P NMR (162 MHz, $CDCl_3$)

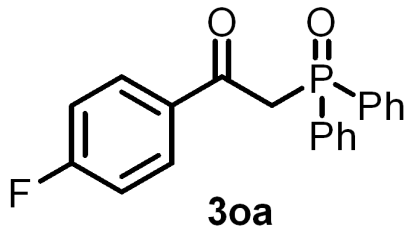




3oa

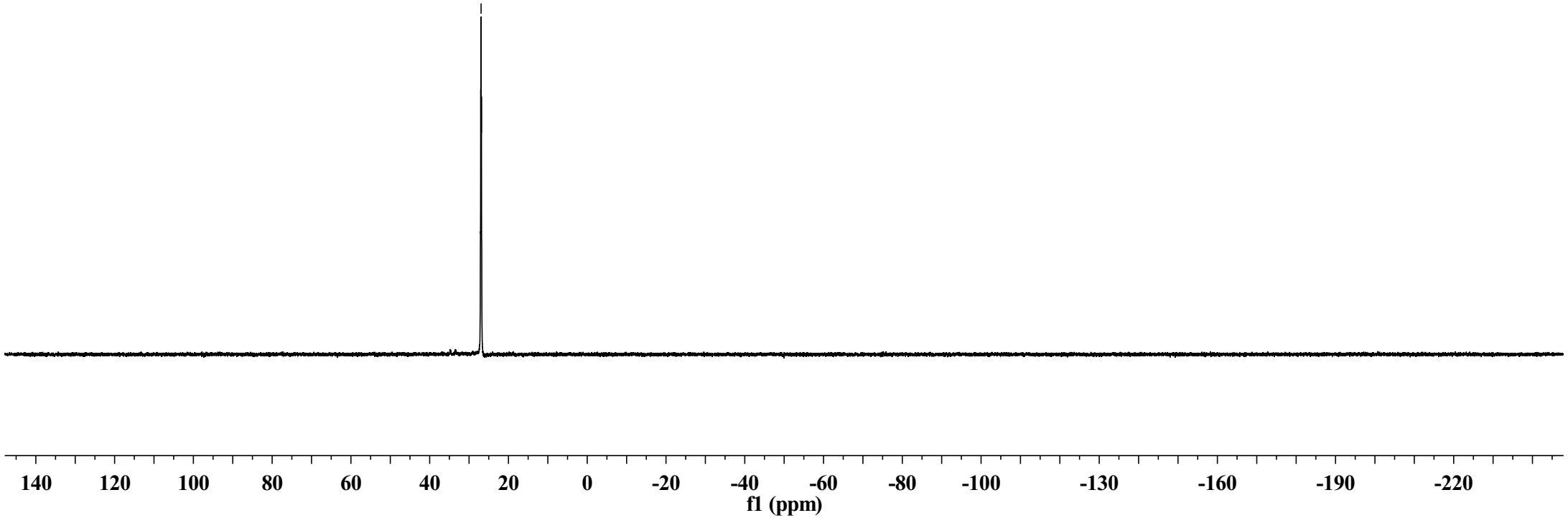
^{1}H NMR (400 MHz, $CDCl_3$)

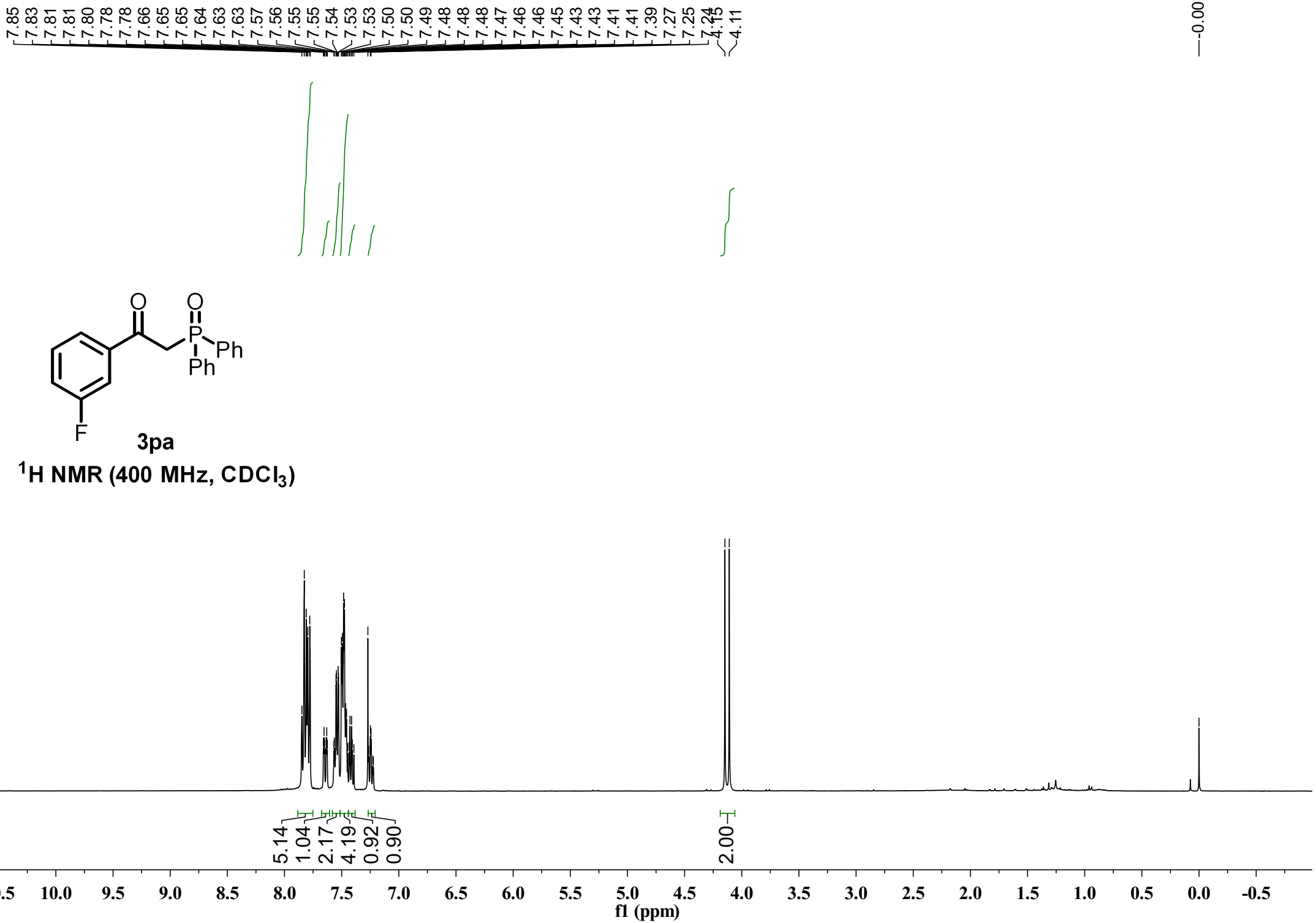

191.15
191.10
167.27
164.72
133.36
133.34
132.20
132.17
132.16
132.12
132.03
131.13
131.04
130.95
128.67
128.54
115.69
115.47
77.32
77.00
76.68
43.72
43.15
O
O
P
Ph
Ph
F
3oa
13C NMR (101 MHz, CDCl3)
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

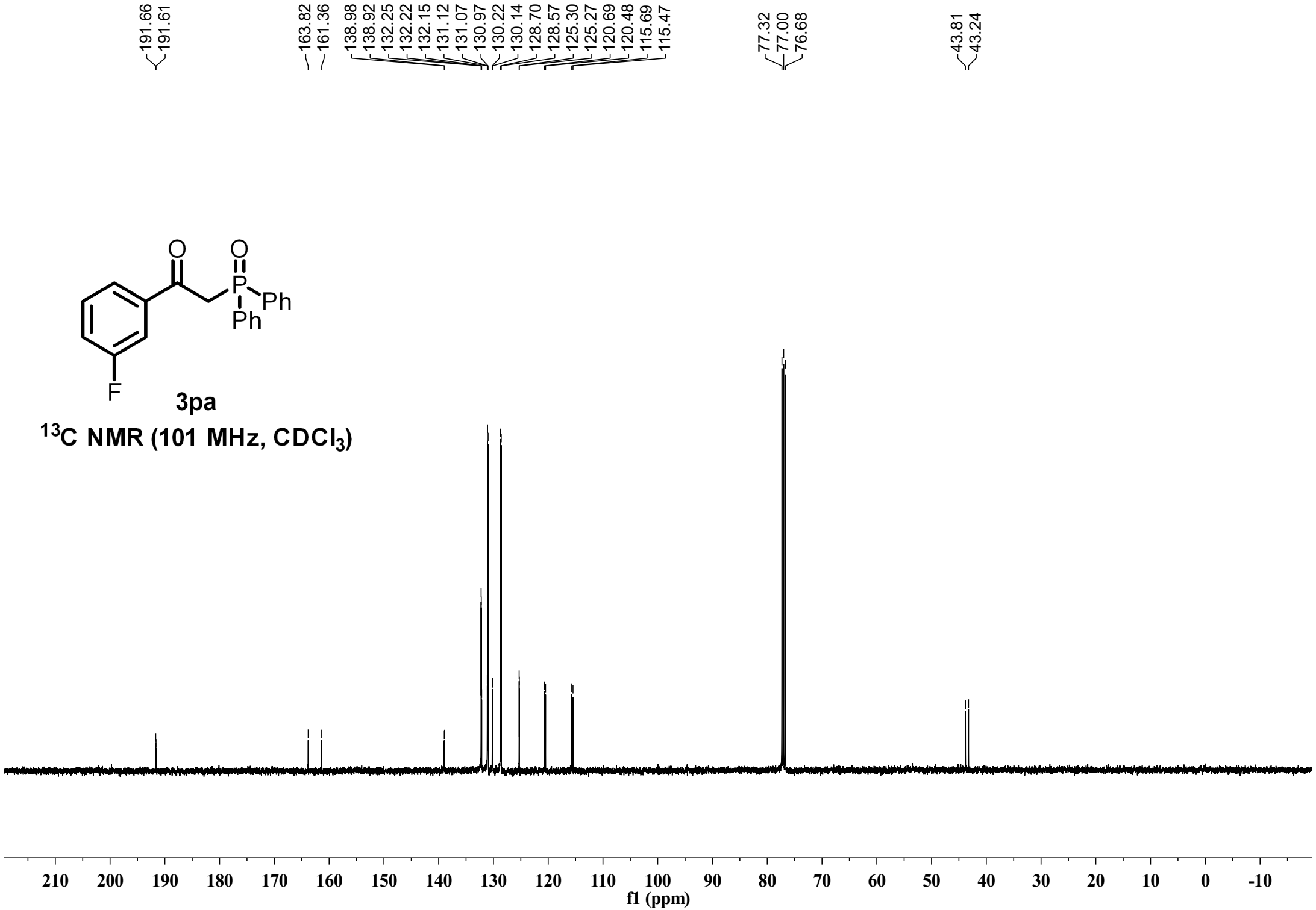


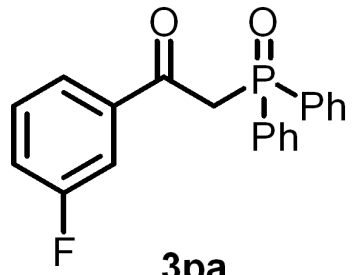
3oa

^{31}P NMR (162 MHz, $CDCl_3$)



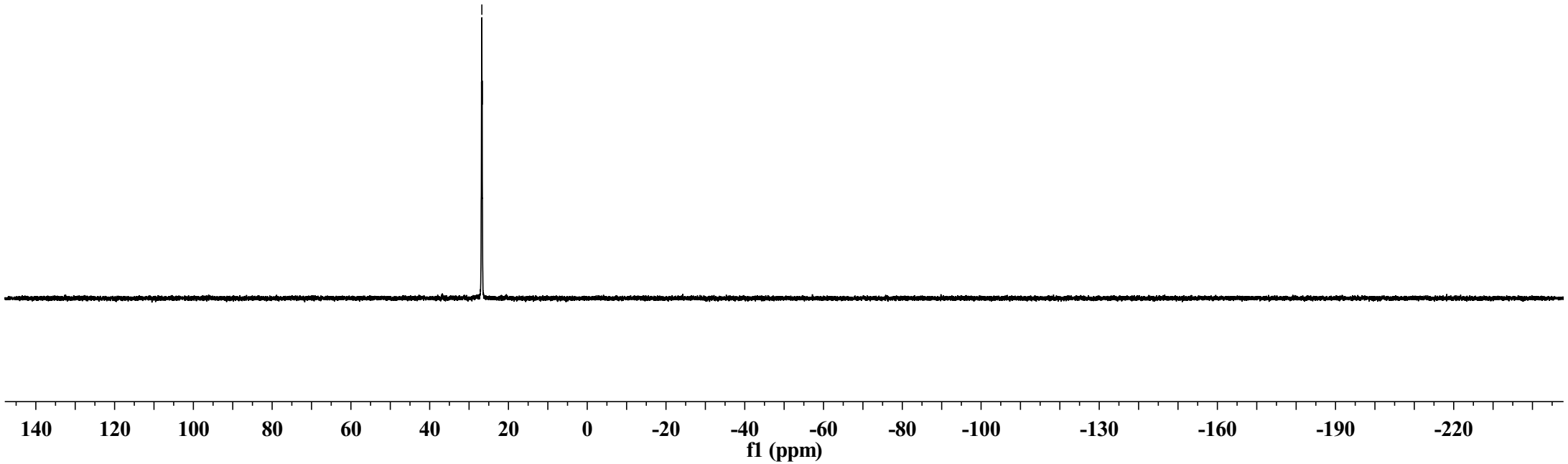

7.85
7.83
7.81
7.81
7.80
7.78
7.78
7.66
7.65
7.65
7.64
7.63
7.63
7.57
7.56
7.55
7.55
7.54
7.53
7.53
7.50
7.50
7.49
7.48
7.48
7.48
7.47
7.46
7.46
7.45
7.43
7.43
7.41
7.41
7.39
7.27
7.25
7.24
4.15
4.11
-0.00
O
O
P
Ph
Ph
F
3pa
1H NMR (400 MHz, CDCl3)
5.14
1.04
2.17
4.19
0.92
0.90
2.00
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)

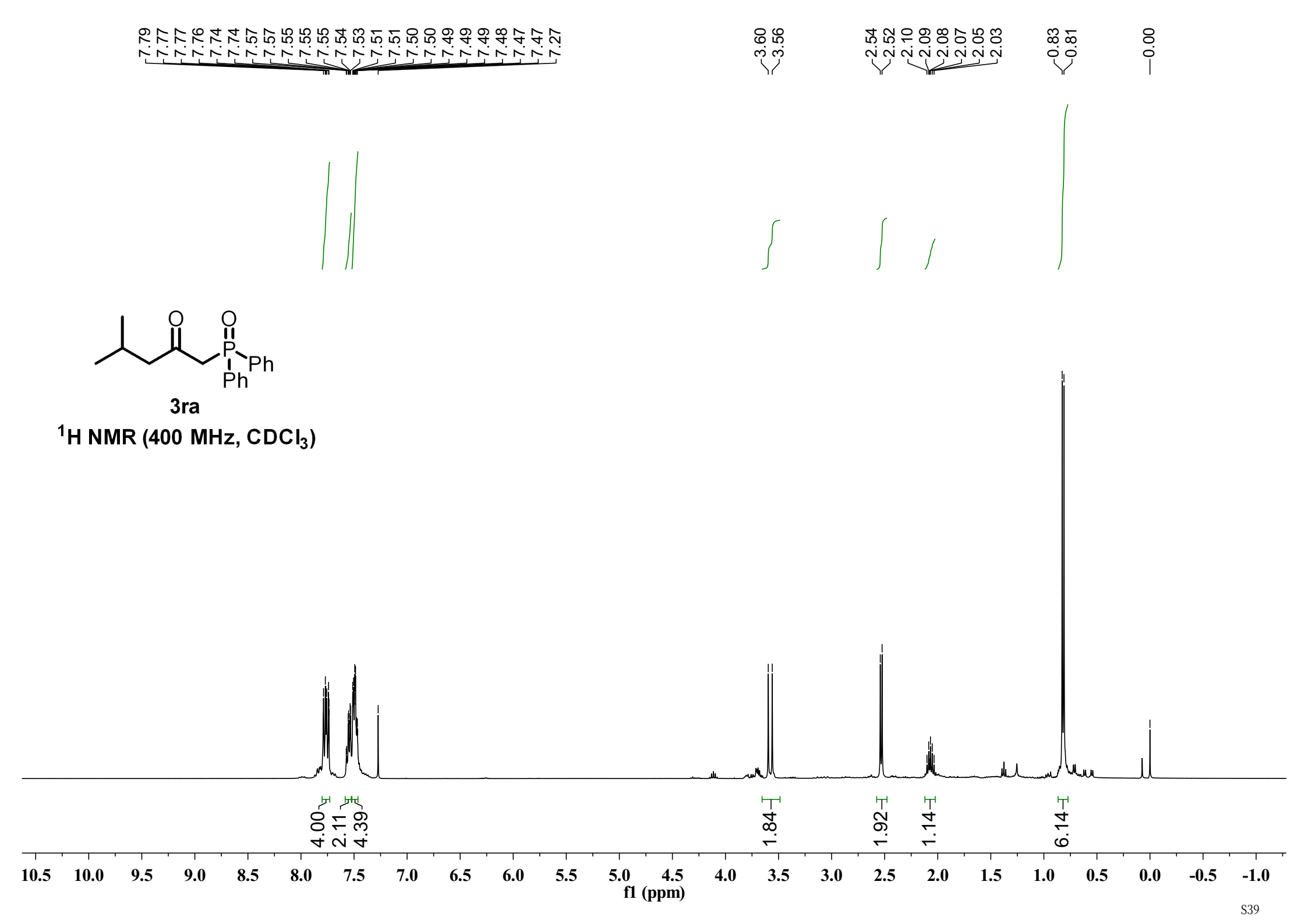

191.66
191.61
163.82
161.36
138.98
138.92
132.25
132.22
132.15
131.12
131.07
130.97
130.22
130.14
128.70
128.57
125.30
125.27
120.69
120.48
115.69
115.47
77.32
77.00
76.68
43.81
43.24
O
O
P
Ph
Ph
F
3pa
13C NMR (101 MHz, CDCl3)
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)



3pa

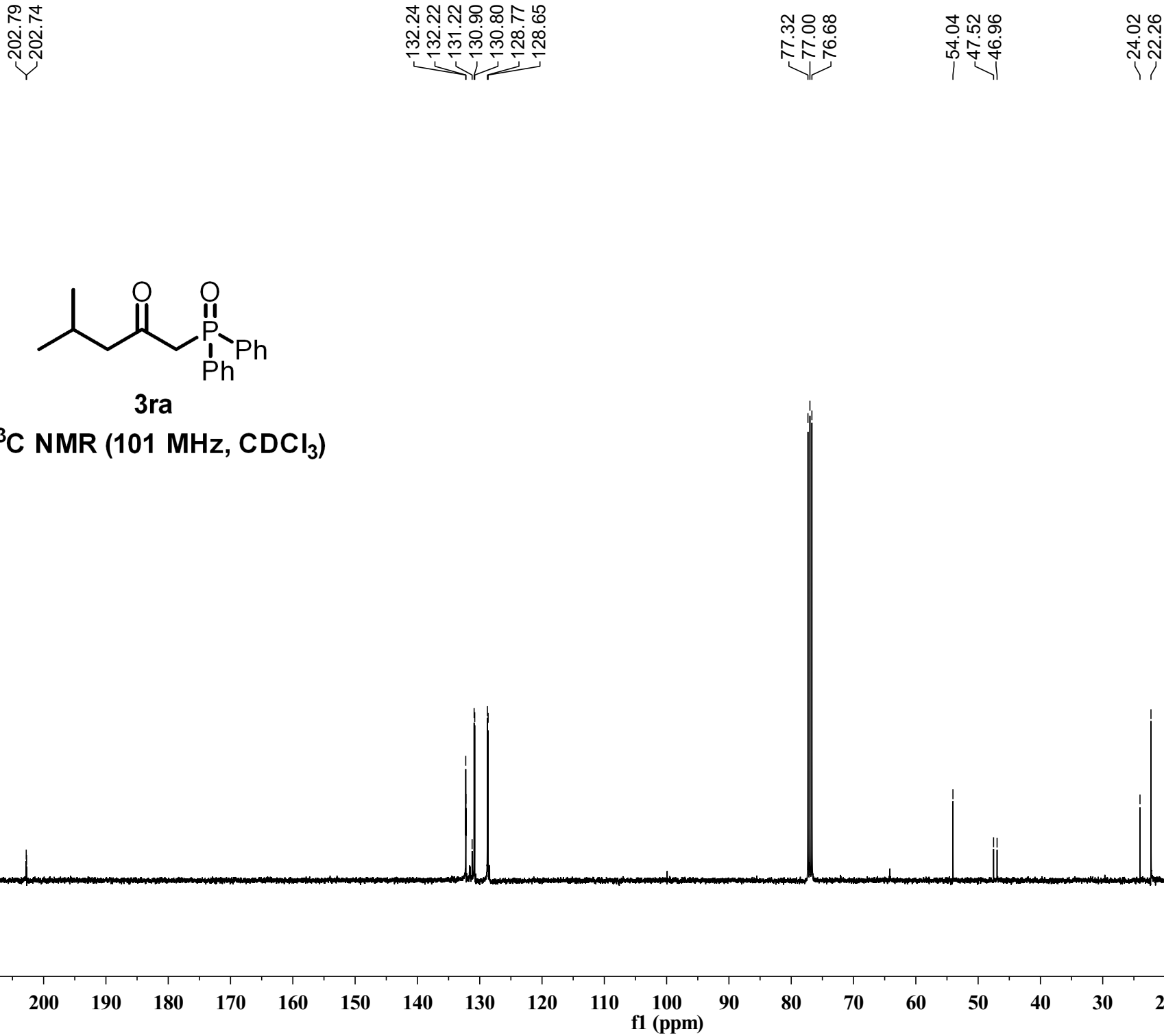
^{31}P NMR (162 MHz, $CDCl_3$)

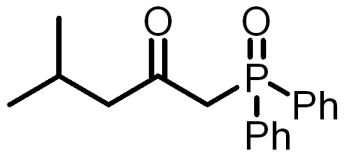




3ra

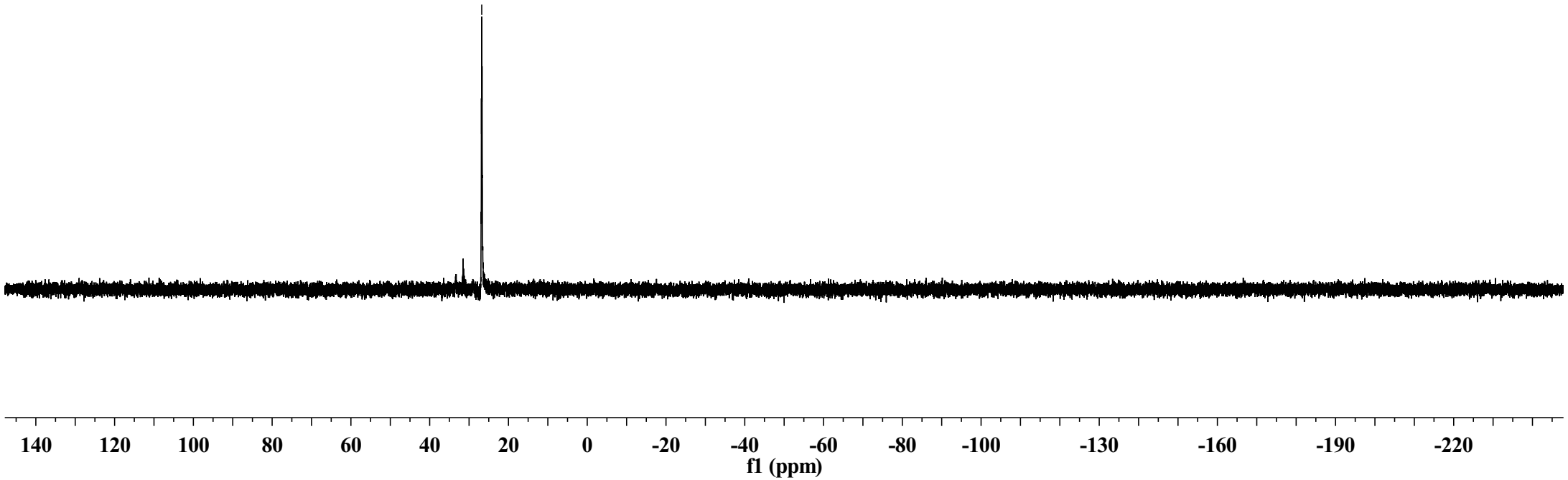
^{1}H NMR (400 MHz, $CDCl_3$)

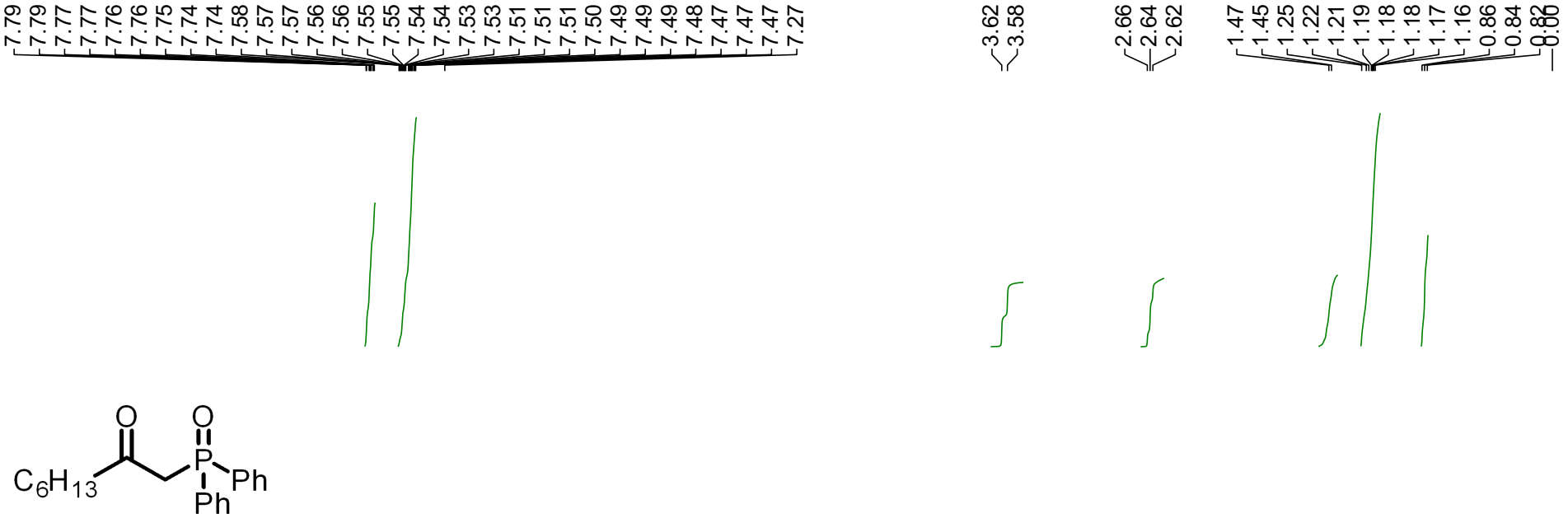

202.79
202.74
132.24
132.22
131.22
130.90
130.80
128.77
128.65
77.32
77.00
76.68
54.04
47.52
46.96
24.02
22.26
O
O
P
Ph
Ph
3ra
13C NMR (101 MHz, CDCl3)
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)



3ra

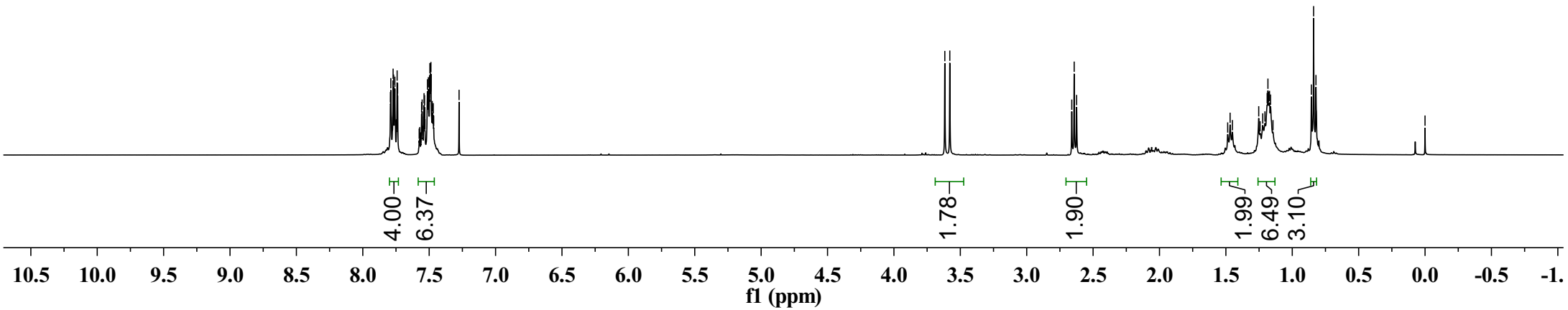
^{31}P NMR (162 MHz, $CDCl_3$)

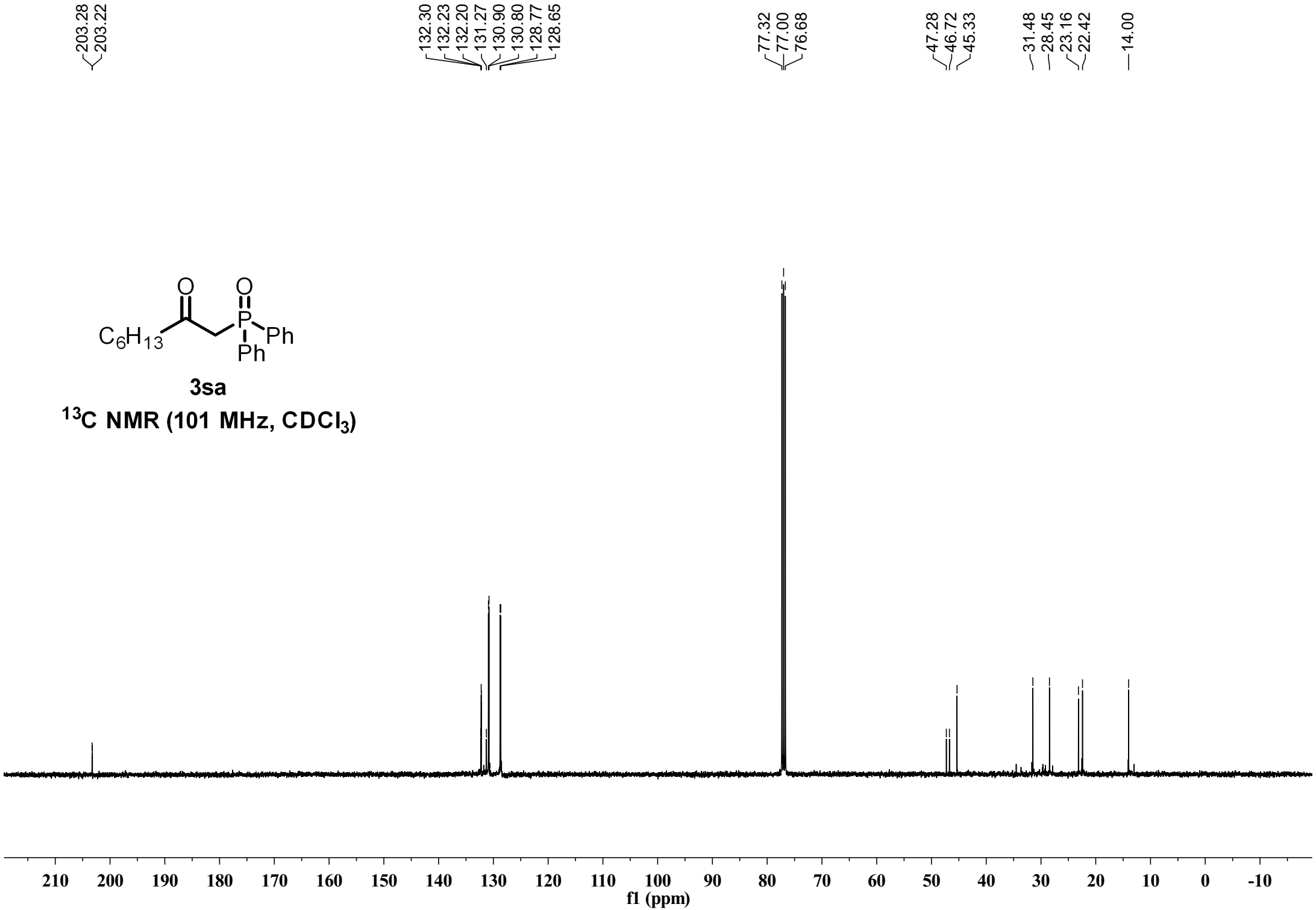


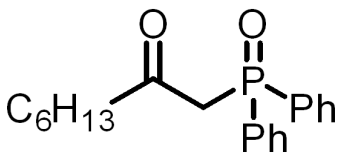


3sa

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

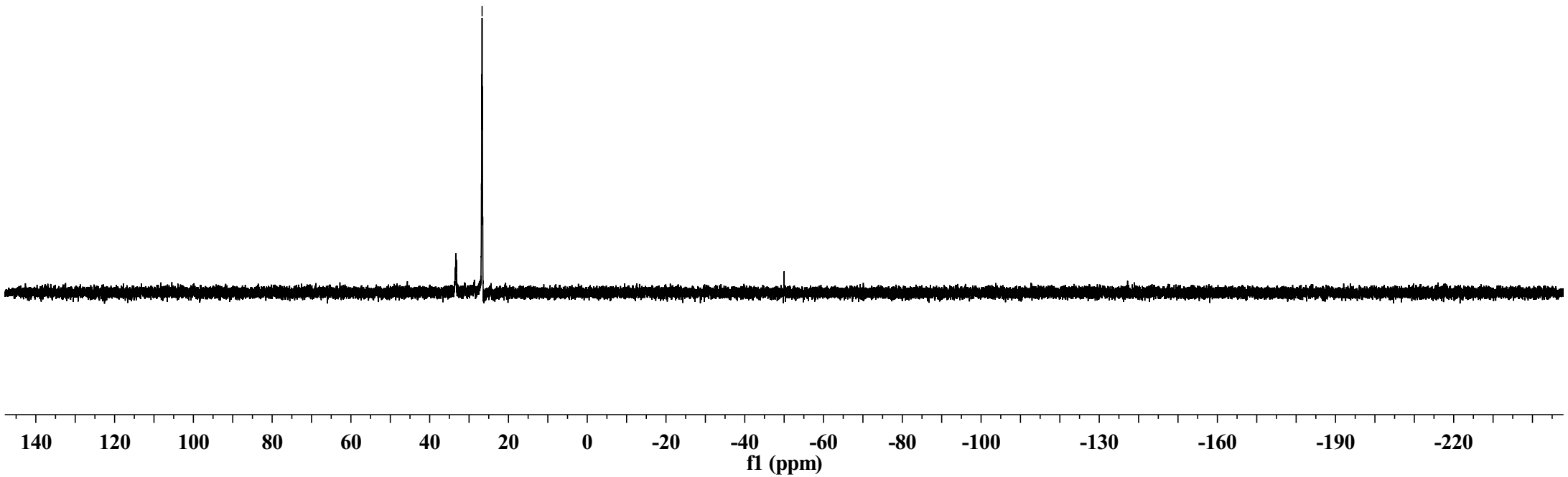


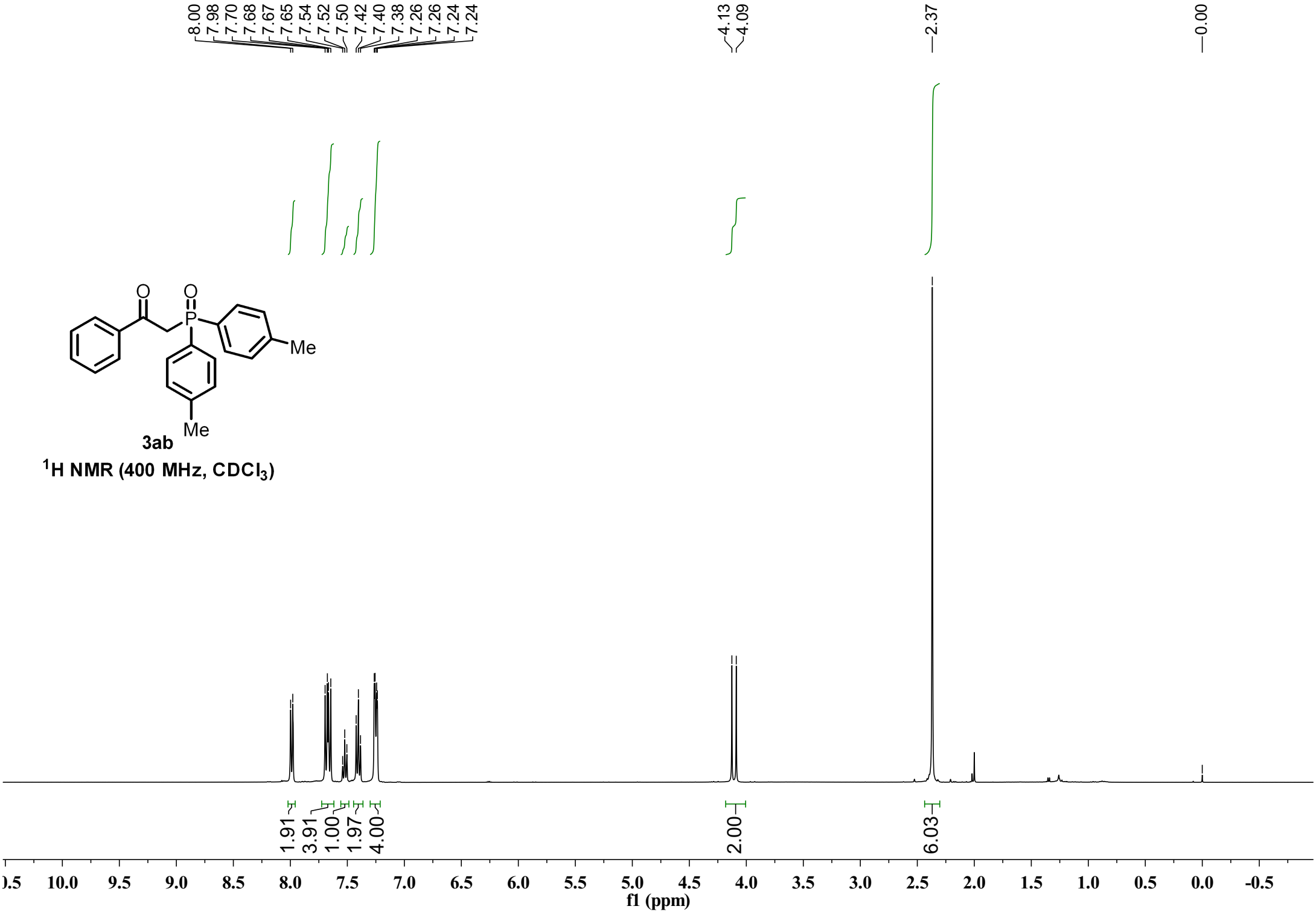

203.28
203.22
132.30
132.23
132.20
131.27
130.90
130.80
128.77
128.65
77.32
77.00
76.68
47.28
46.72
45.33
31.48
28.45
23.16
22.42
14.00
O
O
P
Ph
Ph
C6H13
3sa
13C NMR (101 MHz, CDCl3)
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)



3sa

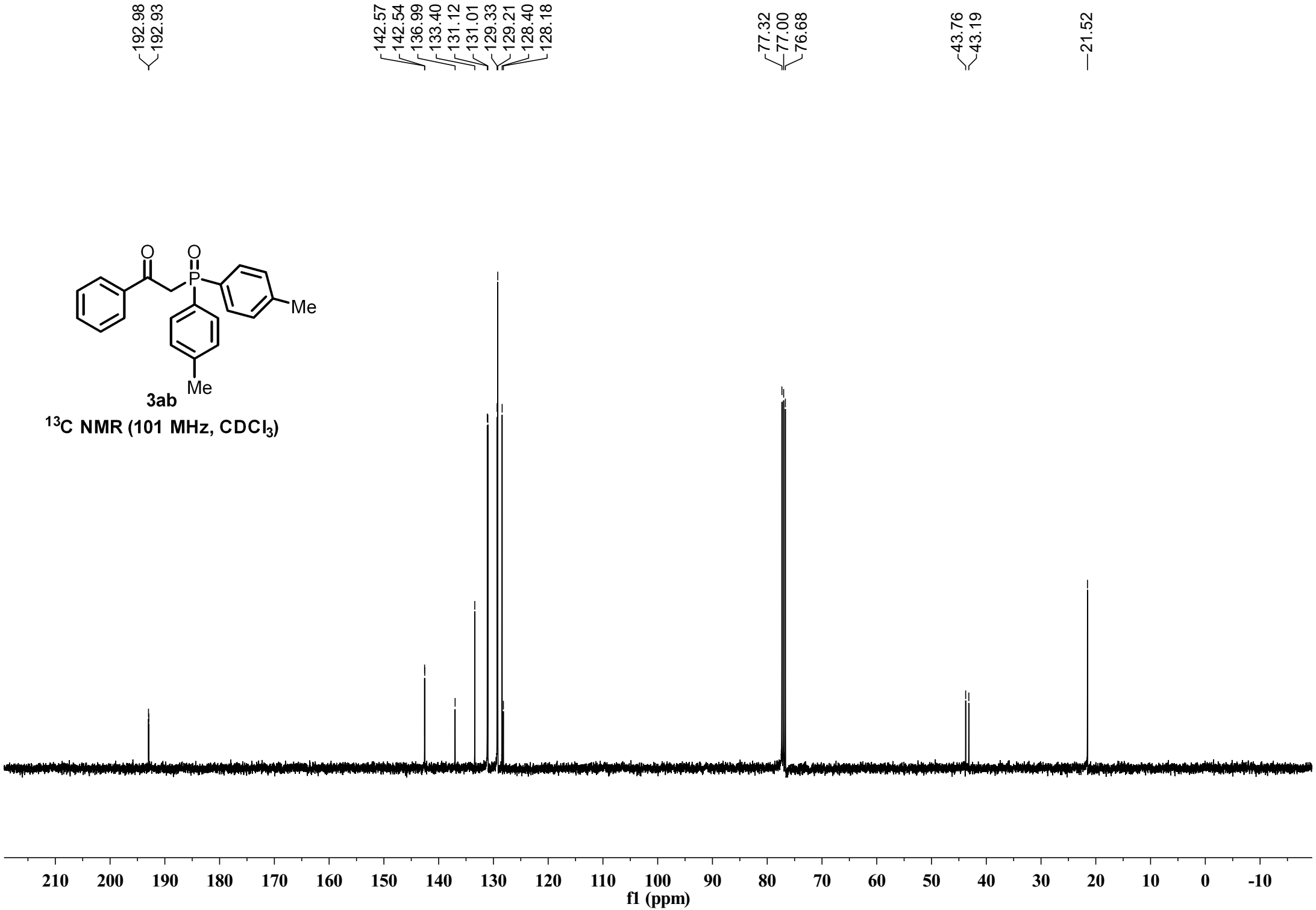
^{31}P NMR (162 MHz, $CDCl_3$)

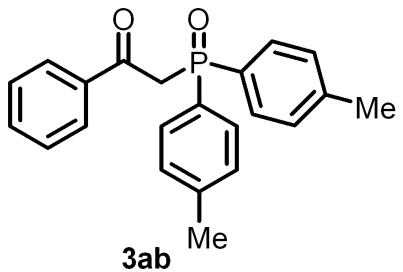




3ab

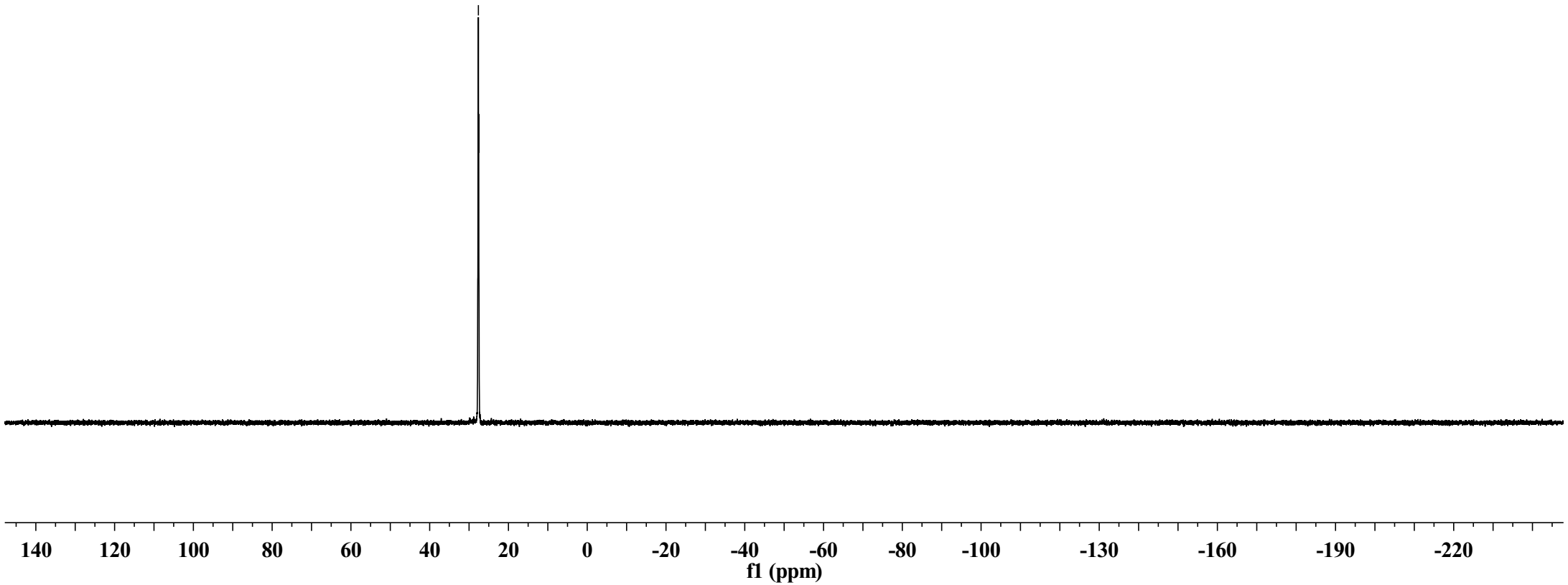
^{1}H NMR (400 MHz, $CDCl_3$)

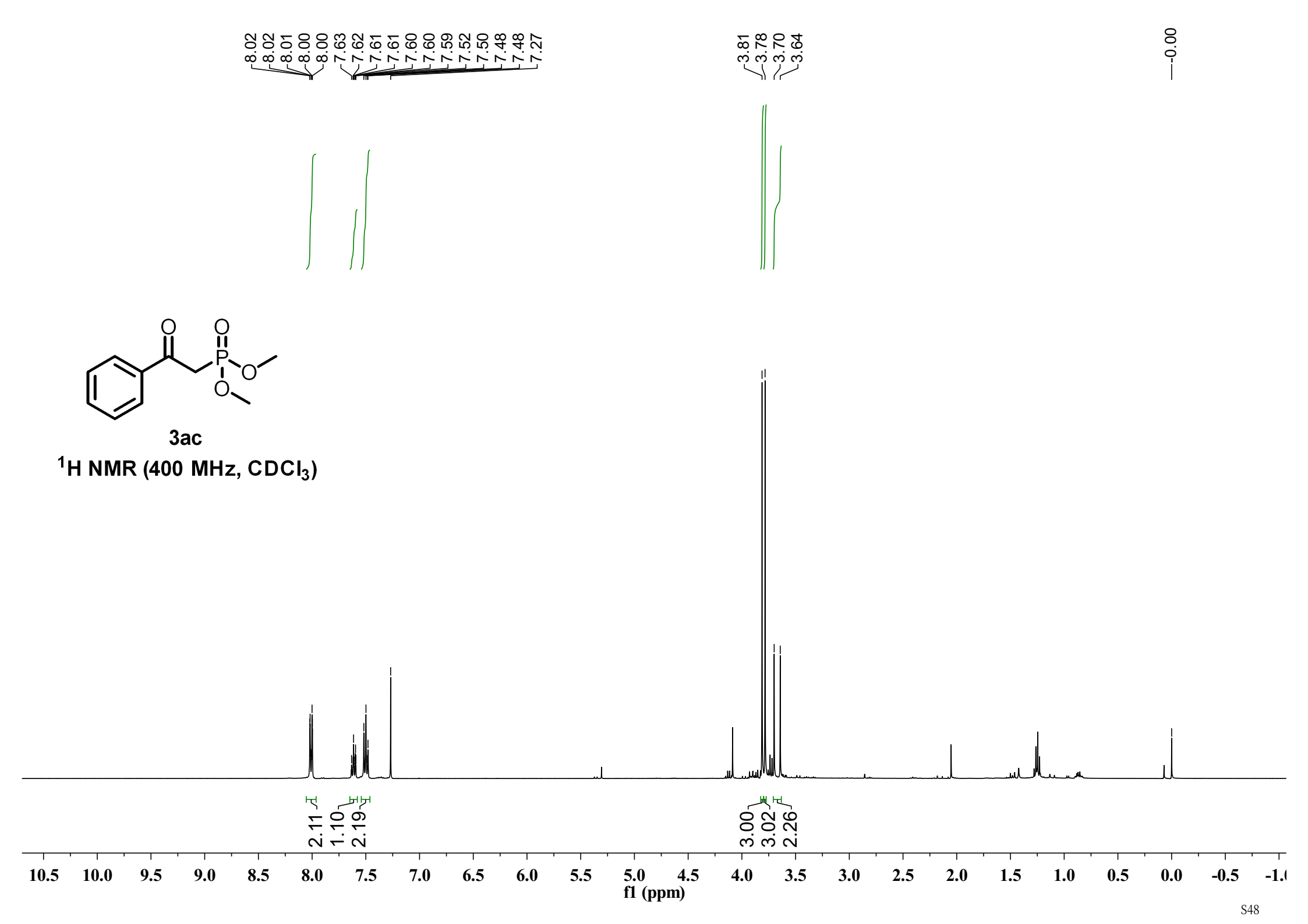

192.98
192.93
142.57
142.54
136.99
133.40
131.12
131.01
129.33
129.21
128.40
128.18
77.32
77.00
76.68
43.76
43.19
21.52
O
O
P
Me
Me
3ab
13C NMR (101 MHz, CDCl3)
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)



3ab

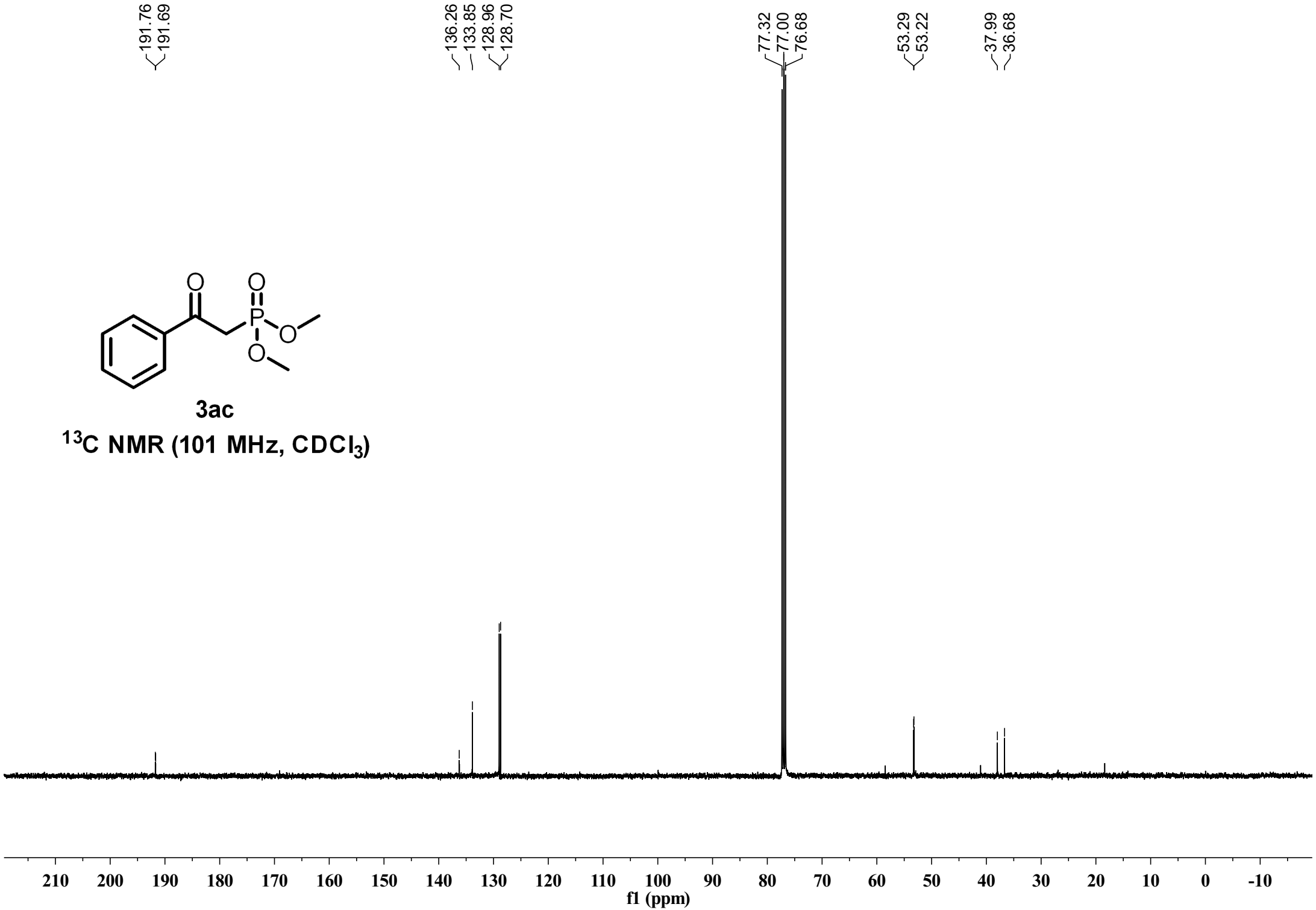
^{31}P NMR (162 MHz, $CDCl_3$)

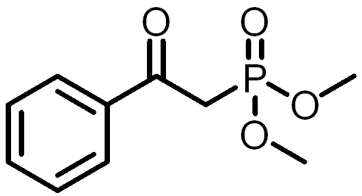




3ac

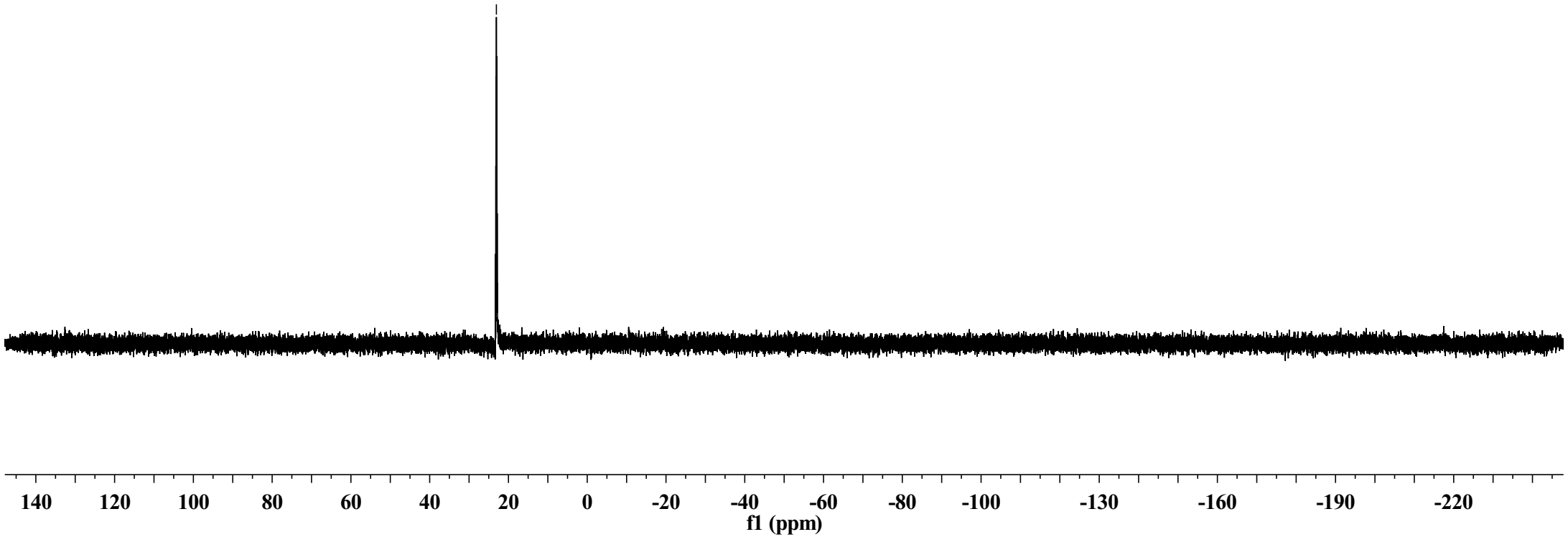
^{1}H NMR (400 MHz, $CDCl_3$)

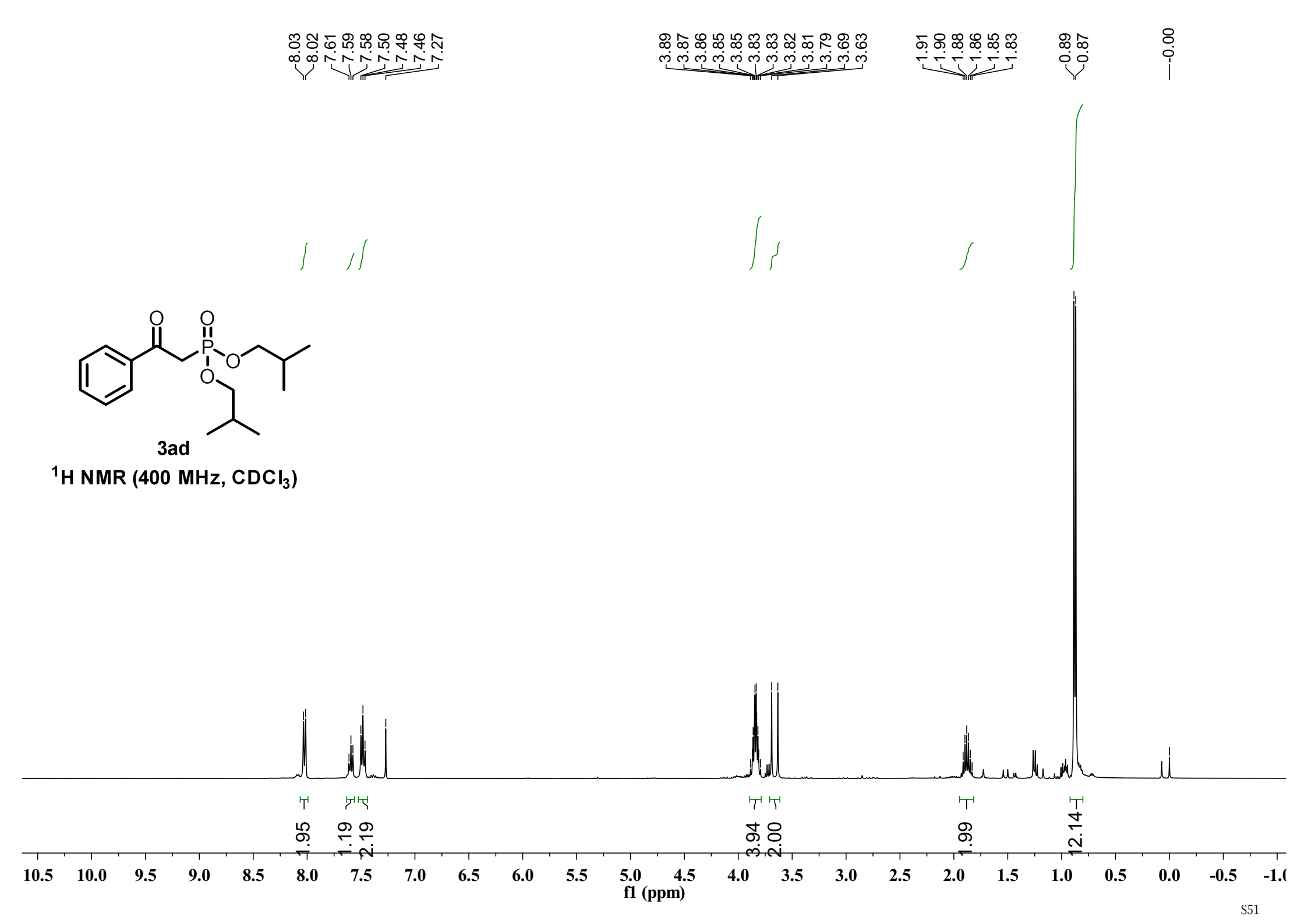

191.76
191.69
136.26
133.85
128.96
128.70
77.32
77.00
76.68
53.29
53.22
37.99
36.68
O
O
P
O
O
3ac
13C NMR (101 MHz, CDCl3)
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)



3ac

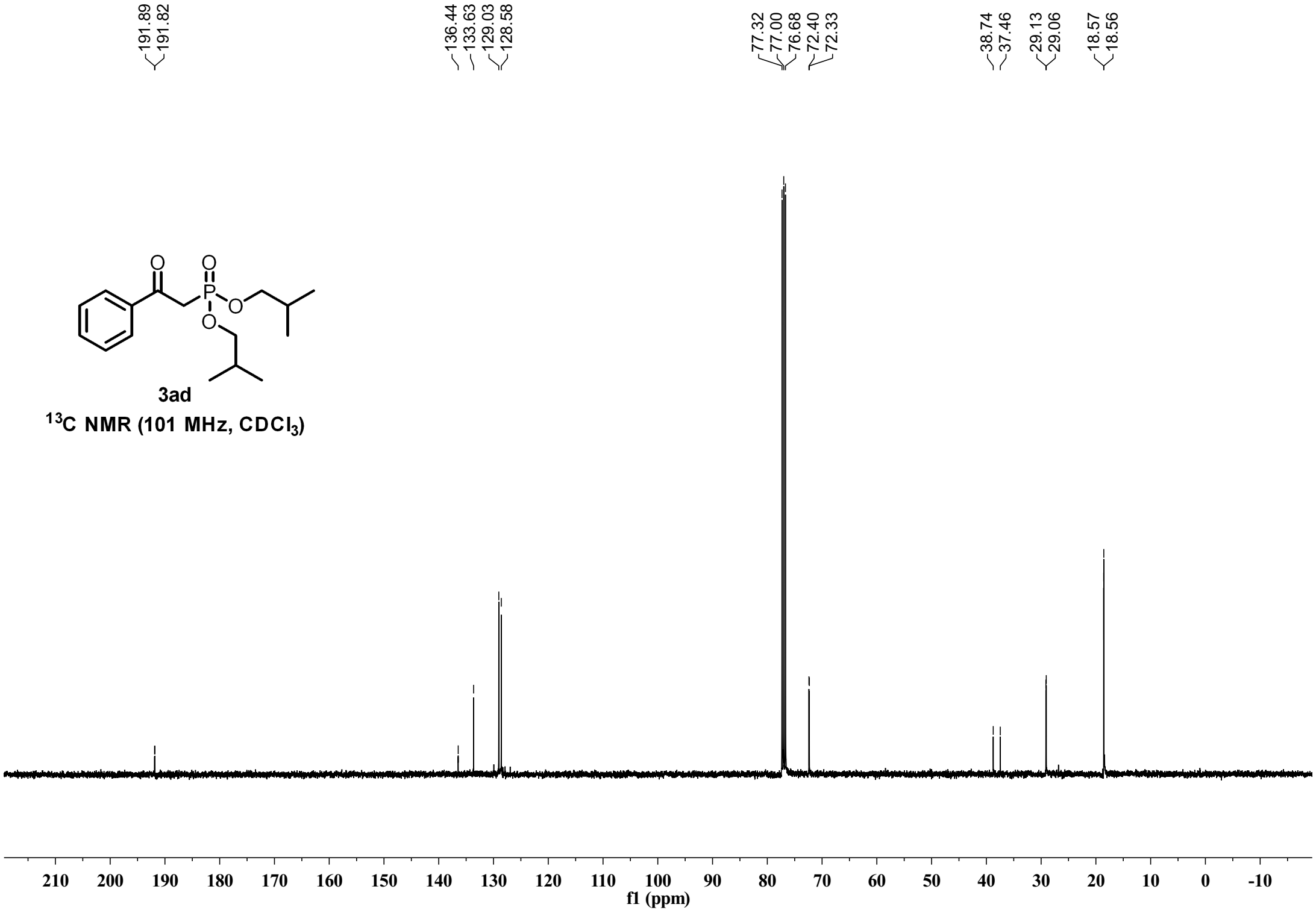
^{31}P NMR (162 MHz, $CDCl_3$)

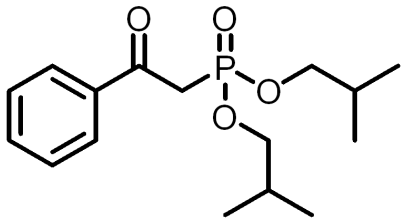




3ad

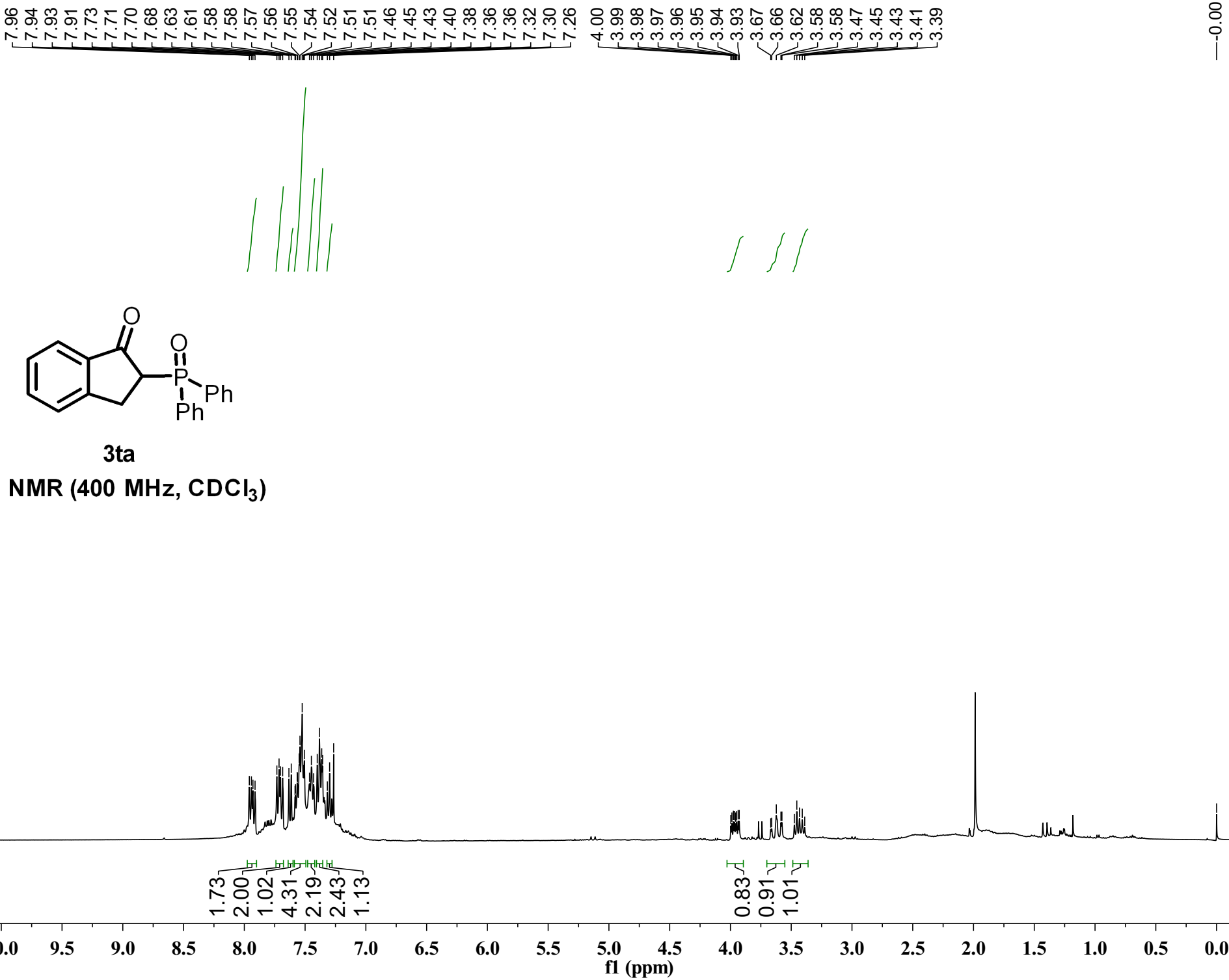
^{1}H NMR (400 MHz, $CDCl_3$)


191.89
191.82
136.44
133.63
129.03
128.58
77.32
77.00
76.68
72.40
72.33
38.74
37.46
29.13
29.06
18.57
18.56
O
O
P
O
O
3ad
^{13}C NMR (101 MHz, CDCl$_3$)
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)



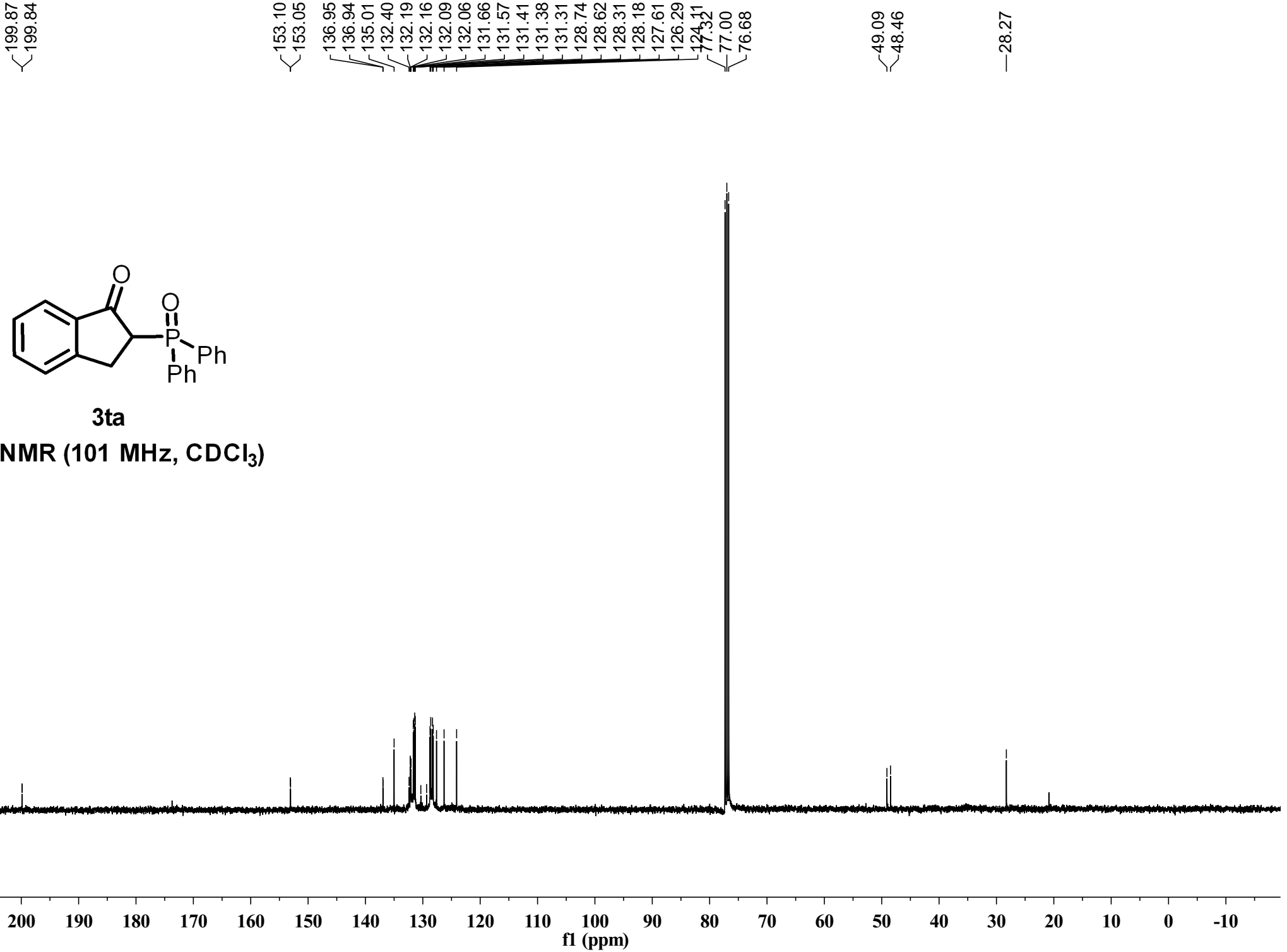
3ad

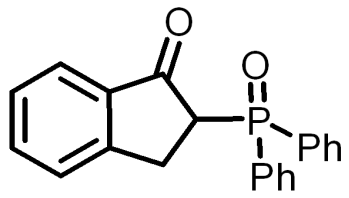
^{31}P NMR (162 MHz, $CDCl_3$)



3ta

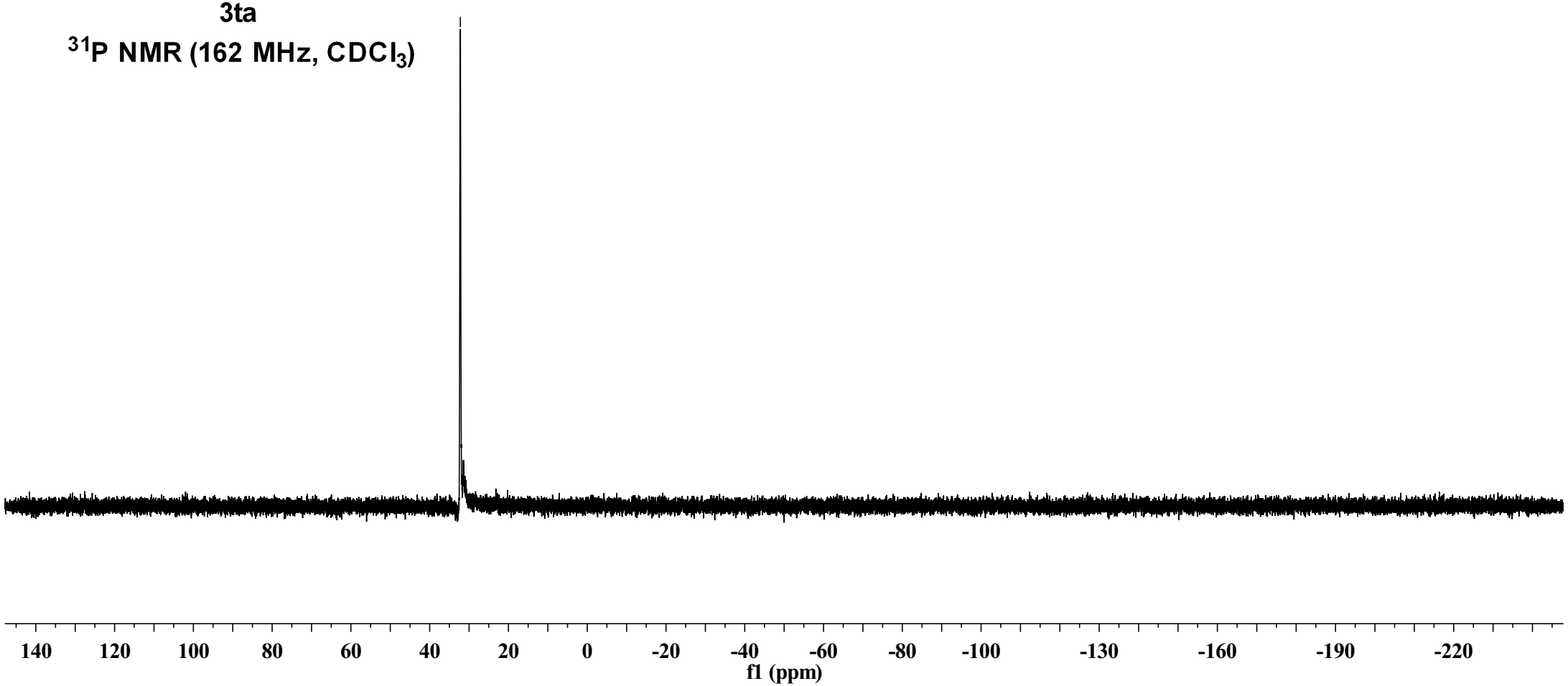
^{1}H NMR (400 MHz, $CDCl_3$)

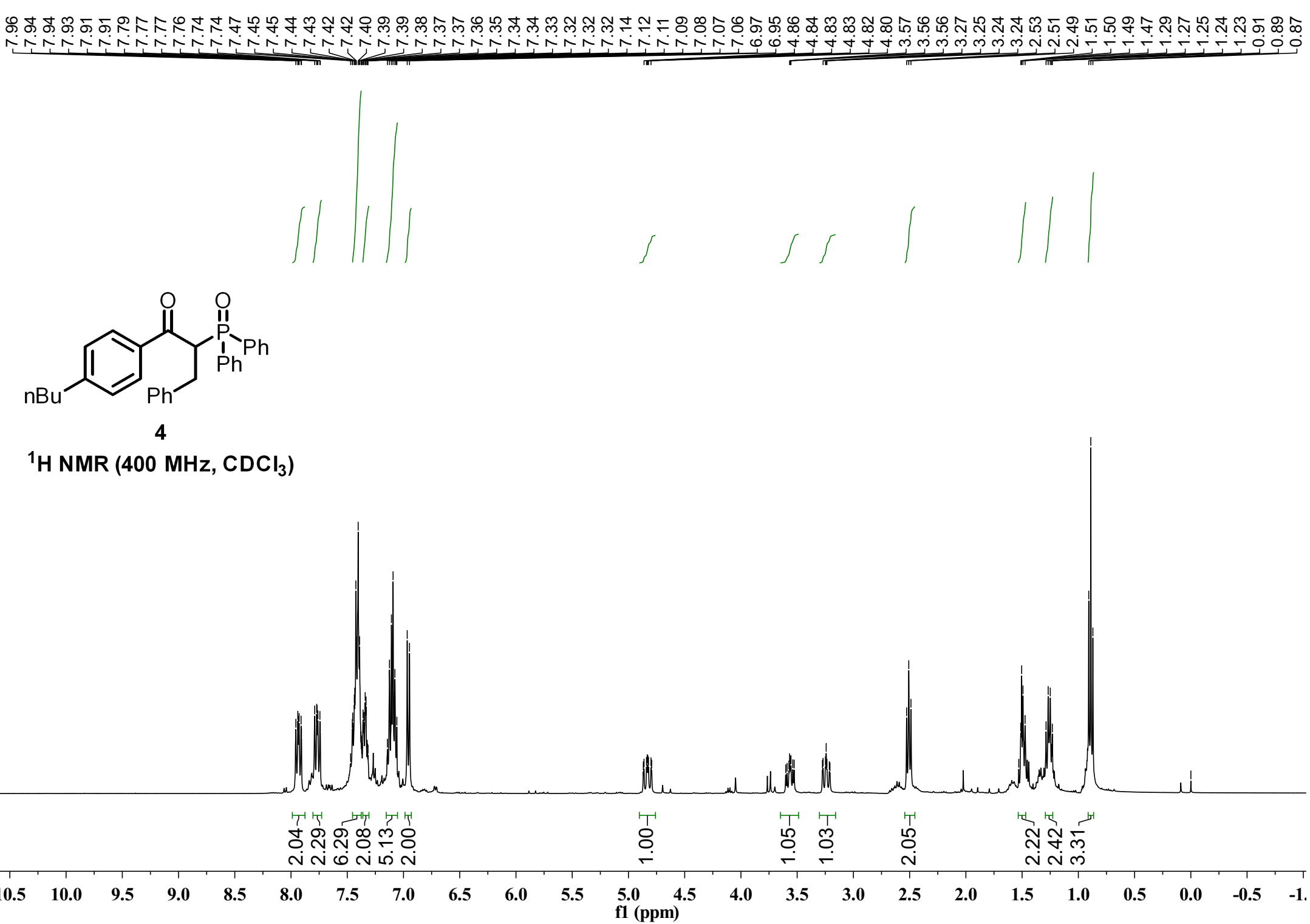

199.87
199.84
153.10
153.05
136.95
136.94
135.01
132.40
132.19
132.16
132.09
132.06
131.66
131.57
131.41
131.38
131.31
128.74
128.62
128.31
128.18
127.61
126.29
124.11
77.32
77.00
76.68
49.09
48.46
28.27
O
O
P
Ph
Ph
3ta
13C NMR (101 MHz, CDCl3)
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

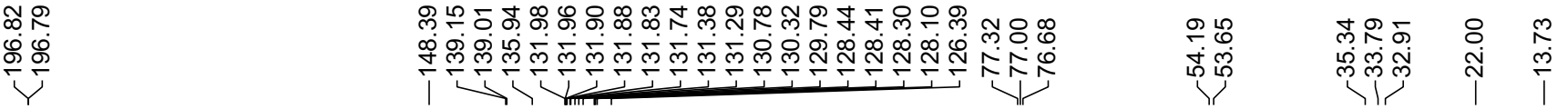


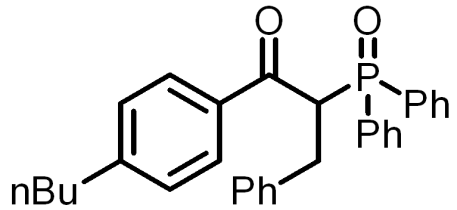
3ta

^{31}P NMR (162 MHz, $CDCl_3$)



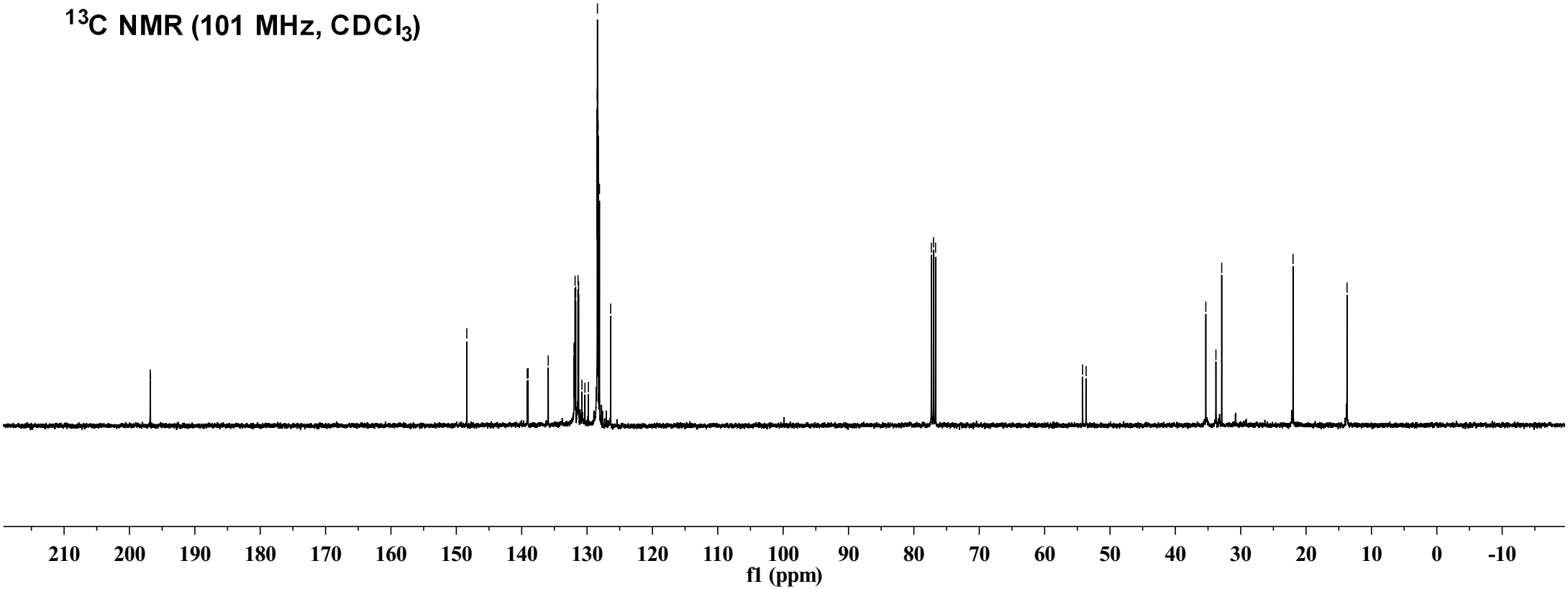

7.96 7.94 7.94 7.93 7.91 7.91 7.79 7.77 7.77 7.76 7.74 7.74 7.47 7.45 7.45 7.44 7.43 7.42 7.42 7.40 7.39 7.39 7.38 7.37 7.37 7.36 7.35 7.34 7.34 7.33 7.32 7.32 7.32 7.14 7.12 7.11 7.09 7.08 7.07 7.06 6.97 6.95 4.86 4.84 4.83 4.83 4.82 4.80 3.57 3.56 3.56 3.27 3.25 3.24 3.24 2.53 2.51 2.49 1.51 1.50 1.49 1.47 1.29 1.27 1.25 1.24 1.23 0.91 0.89 0.87
O
O
P
Ph
Ph
nBu
Ph
4
1H NMR (400 MHz, CDCl3)
2.04 2.29 6.29 2.08 5.13 2.00 1.00 1.05 1.03 2.05 2.22 2.42 3.31
10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)

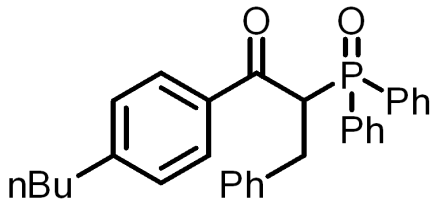




4

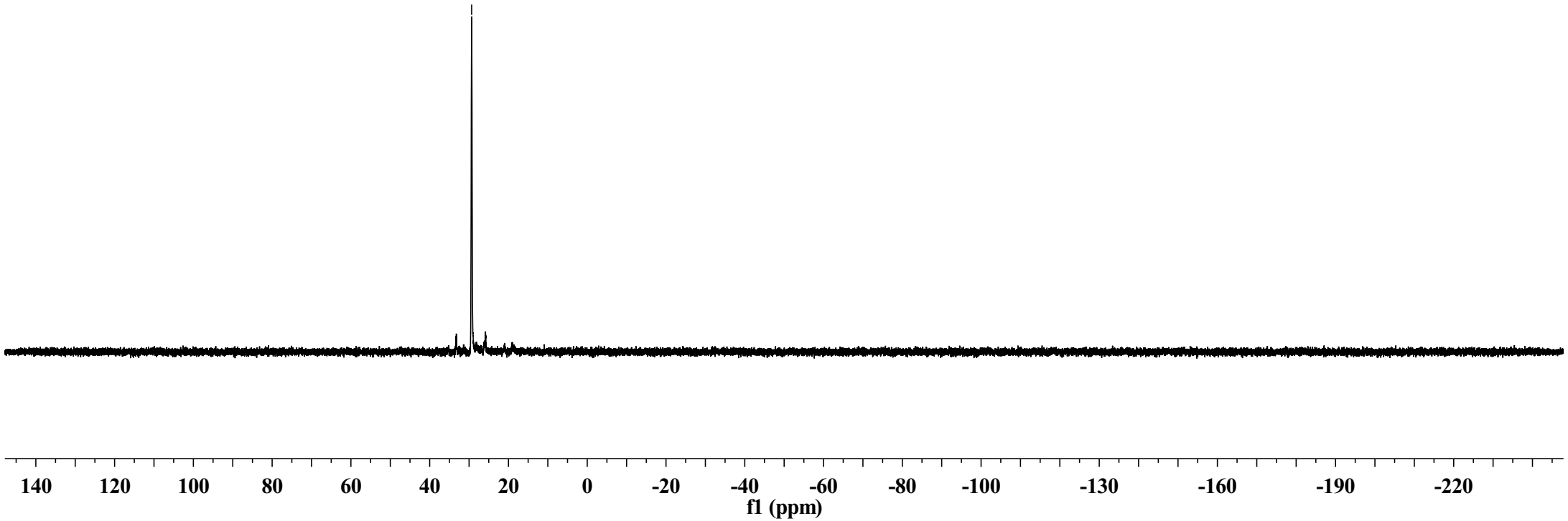
^{13}C NMR (101 MHz, $CDCl_3$)

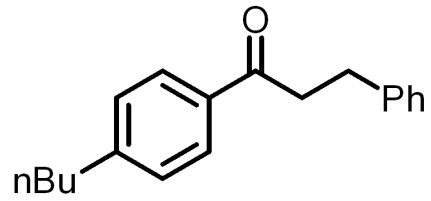




4

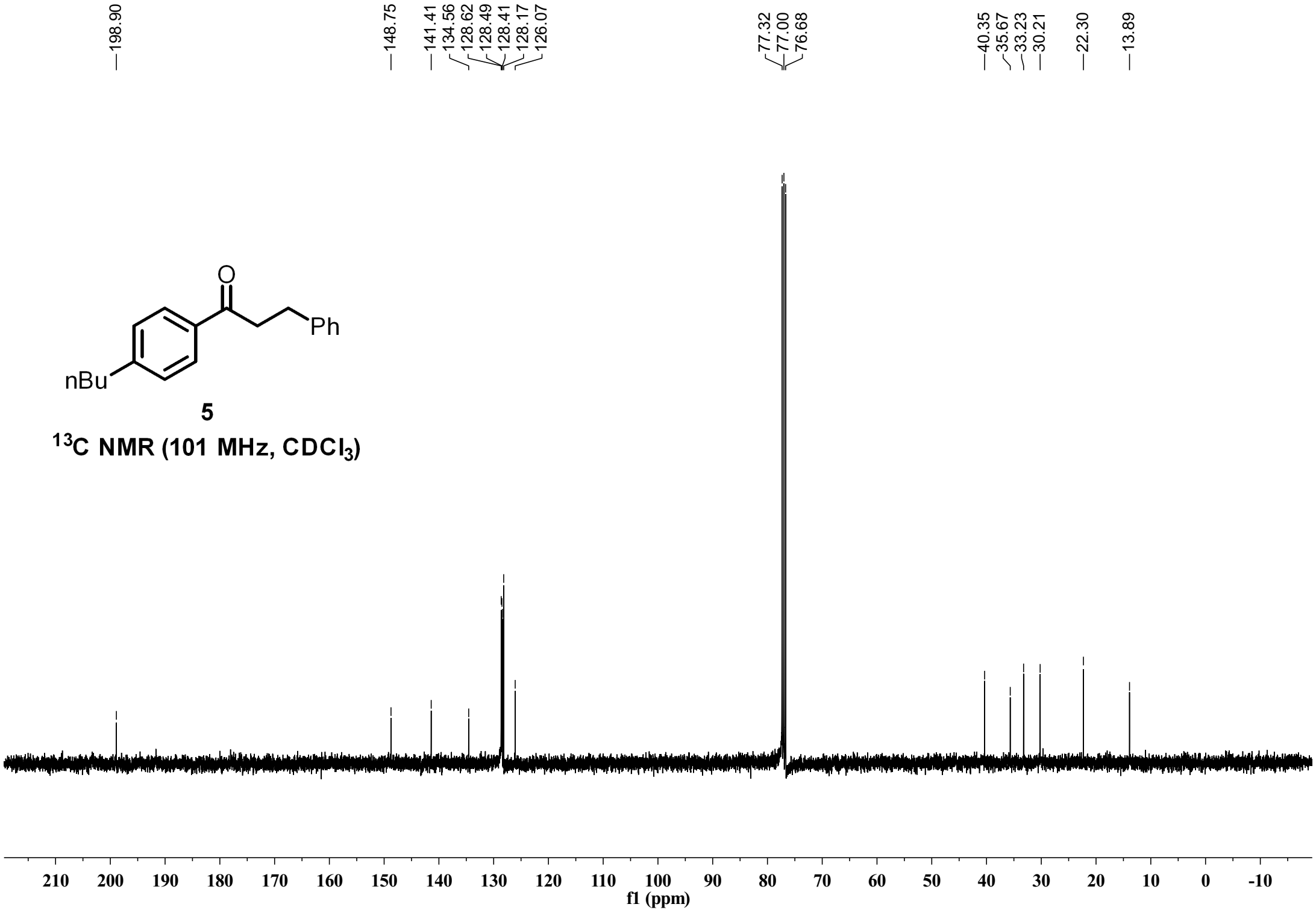
^{31}P NMR (162 MHz, $CDCl_3$)

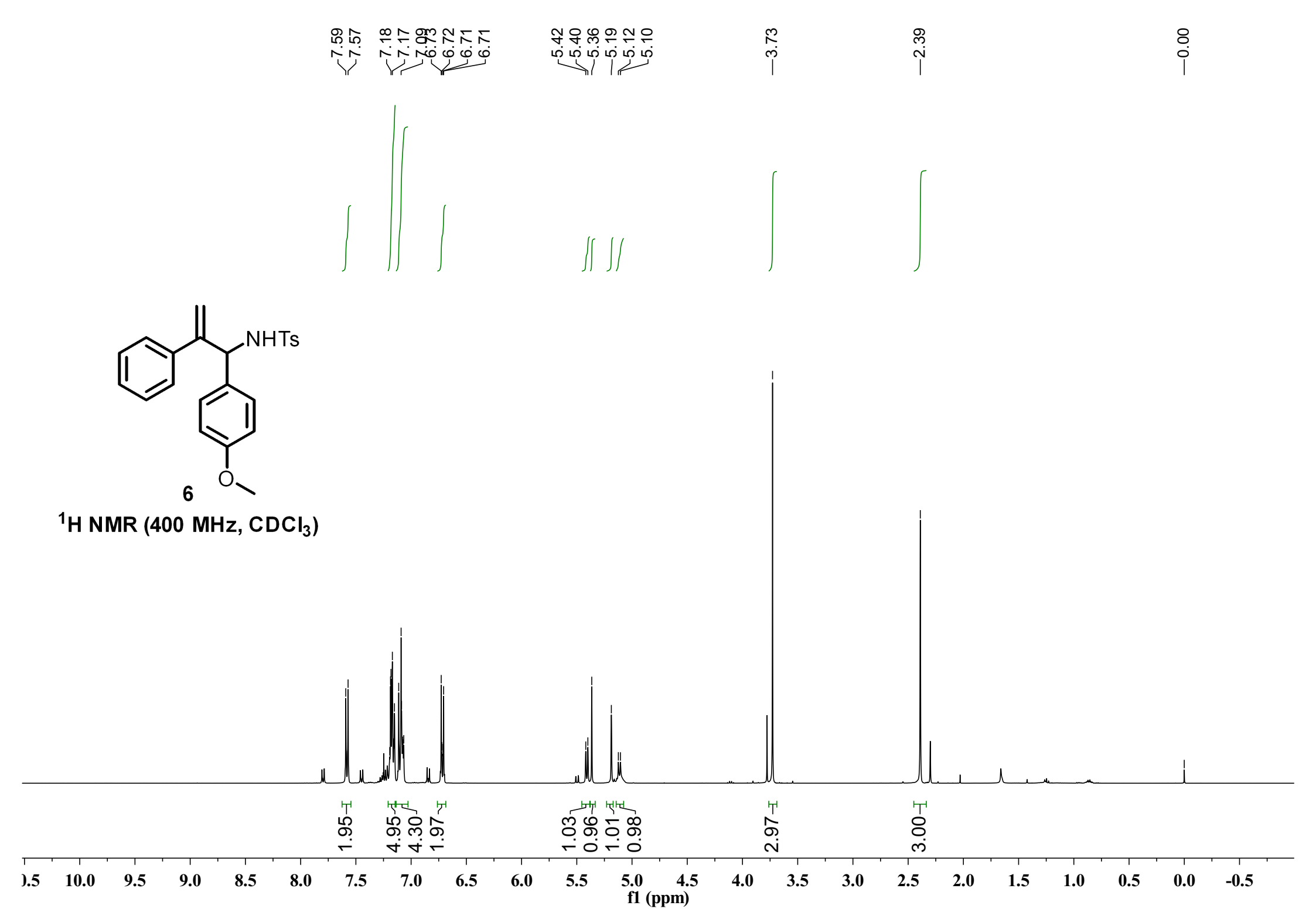




5

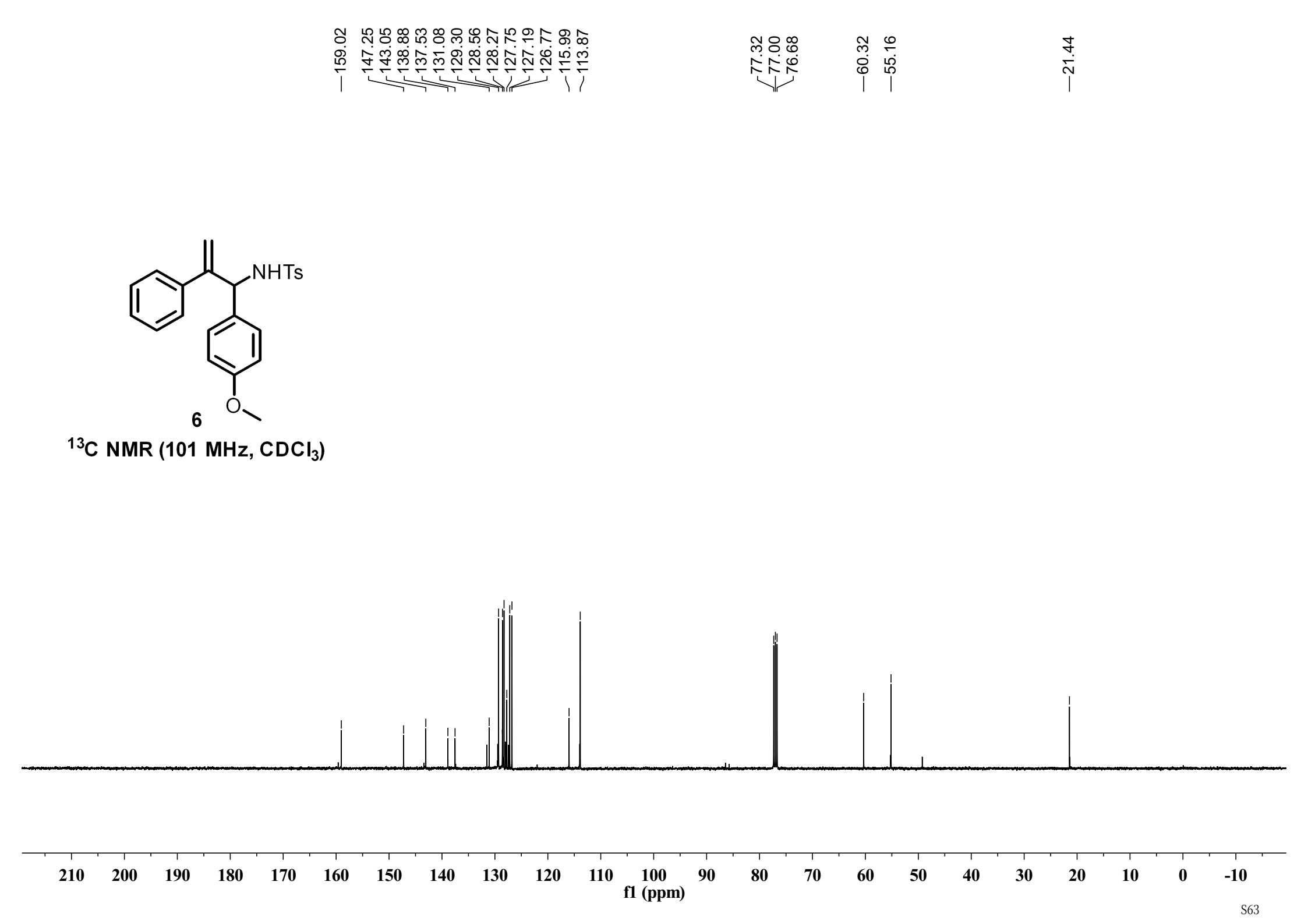
^{1}H NMR (400 MHz, $CDCl_3$)

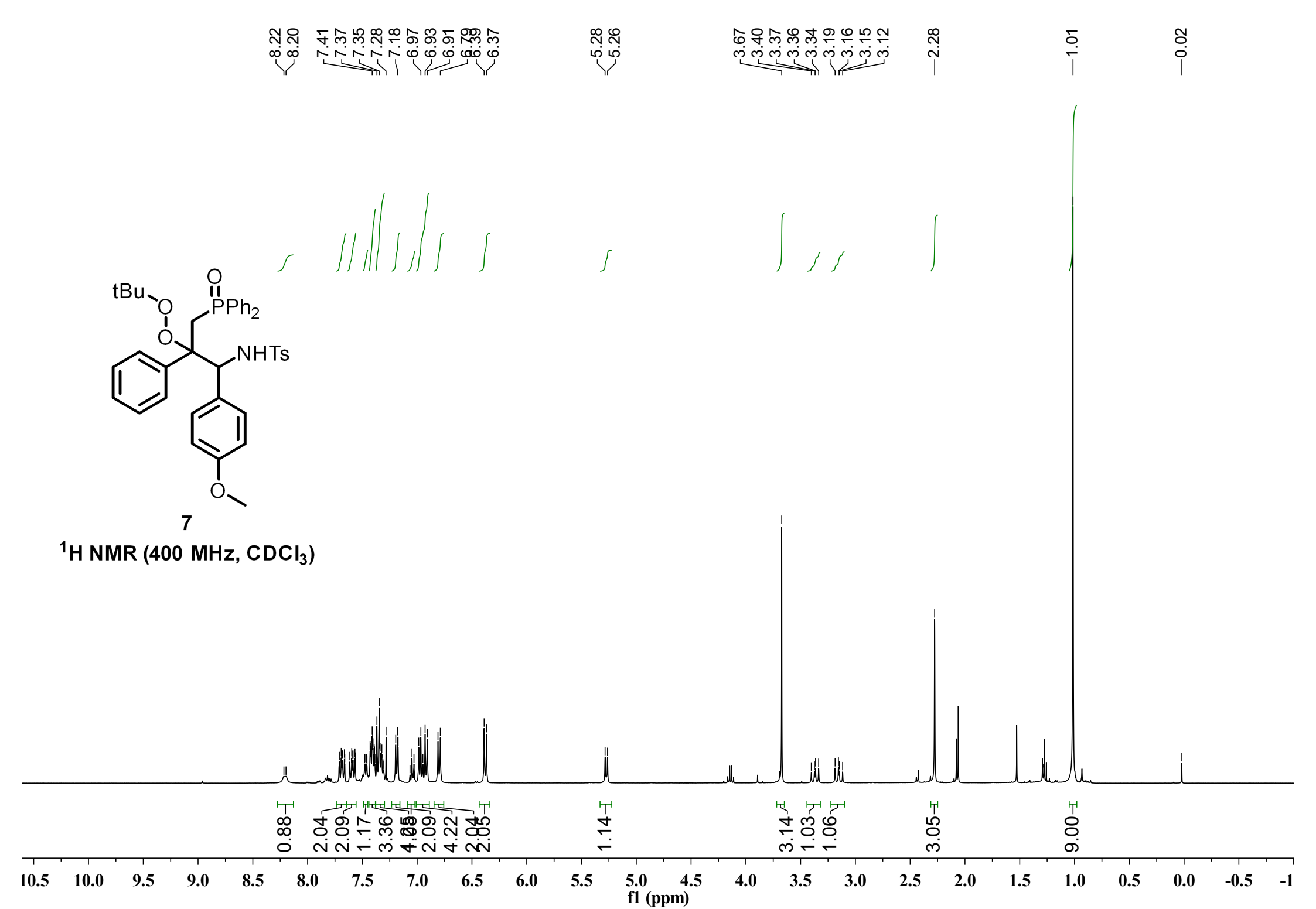

198.90
148.75
141.41
134.56
128.62
128.49
128.41
128.17
126.07
77.32
77.00
76.68
40.35
35.67
33.23
30.21
22.30
13.89
O
Ph
nBu
5
13C NMR (101 MHz, CDCl3)
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

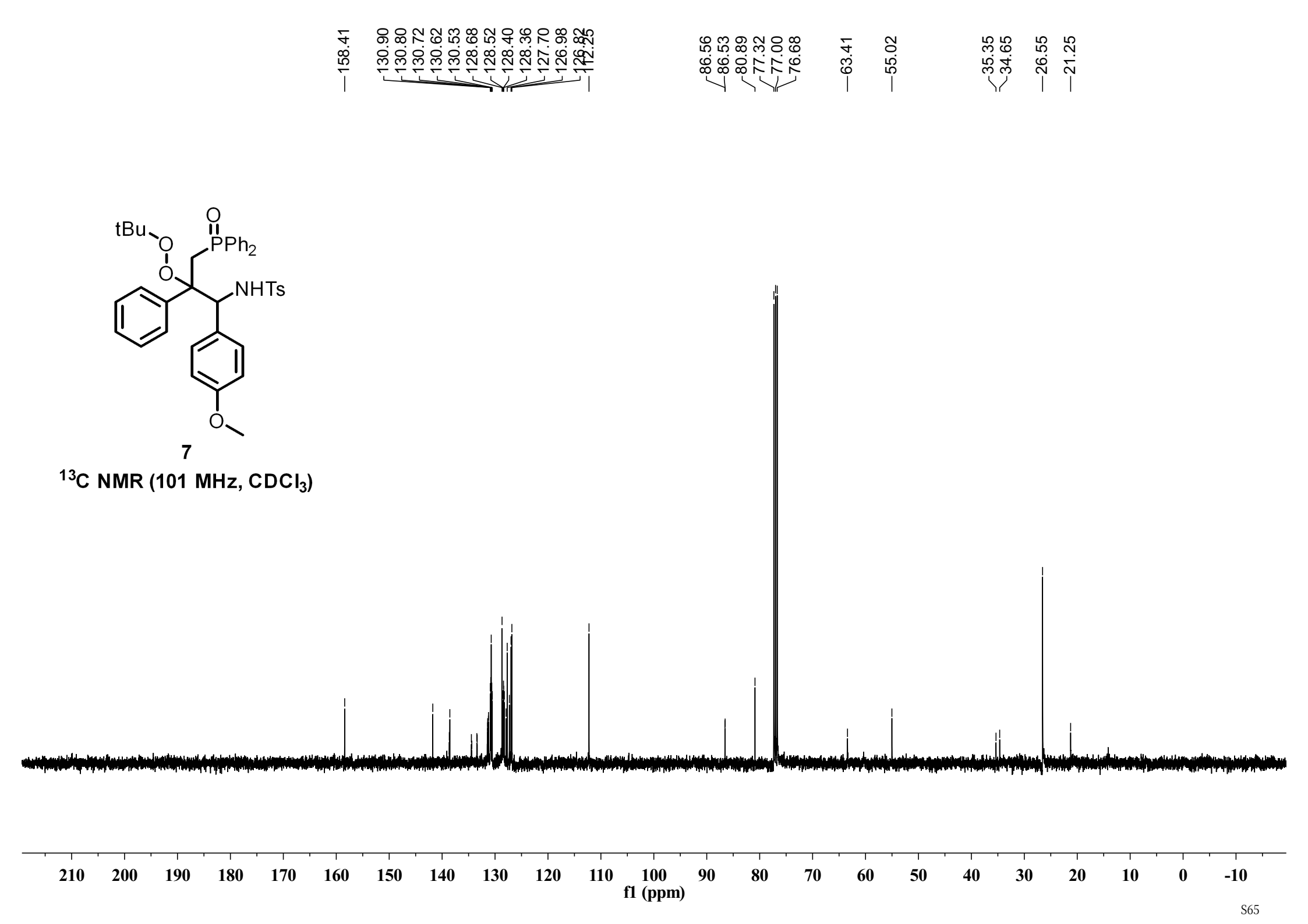


6

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

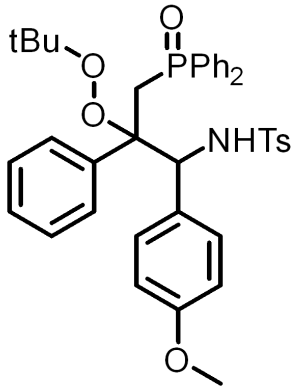

159.02
147.25
143.05
138.88
137.53
131.08
129.30
128.56
128.27
127.75
127.19
126.77
115.99
113.87
77.32
77.00
76.68
60.32
55.16
21.44
NHTs
O
6
13C NMR (101 MHz, CDCl3)
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)


8.22
8.20
7.41
7.37
7.35
7.28
7.18
6.97
6.93
6.91
6.79
6.39
6.37
5.28
5.26
3.67
3.40
3.37
3.36
3.34
3.19
3.16
3.15
3.12
2.28
1.01
0.02
tBu
O
O
PPh2
NHTs
O
O
7
1H NMR (400 MHz, CDCl3)
0.88
2.04
2.09
1.17
3.36
2.09
4.22
2.04
2.05
1.14
3.14
1.03
1.06
3.05
9.00
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
-1
f1 (ppm)



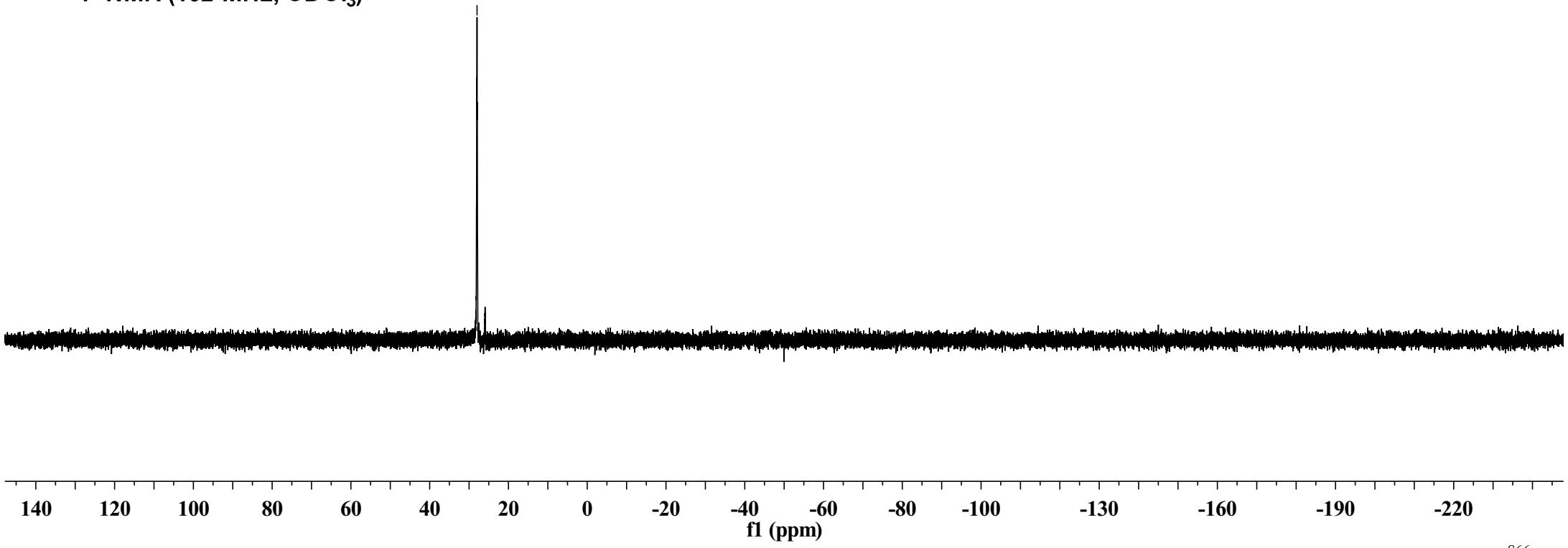
7

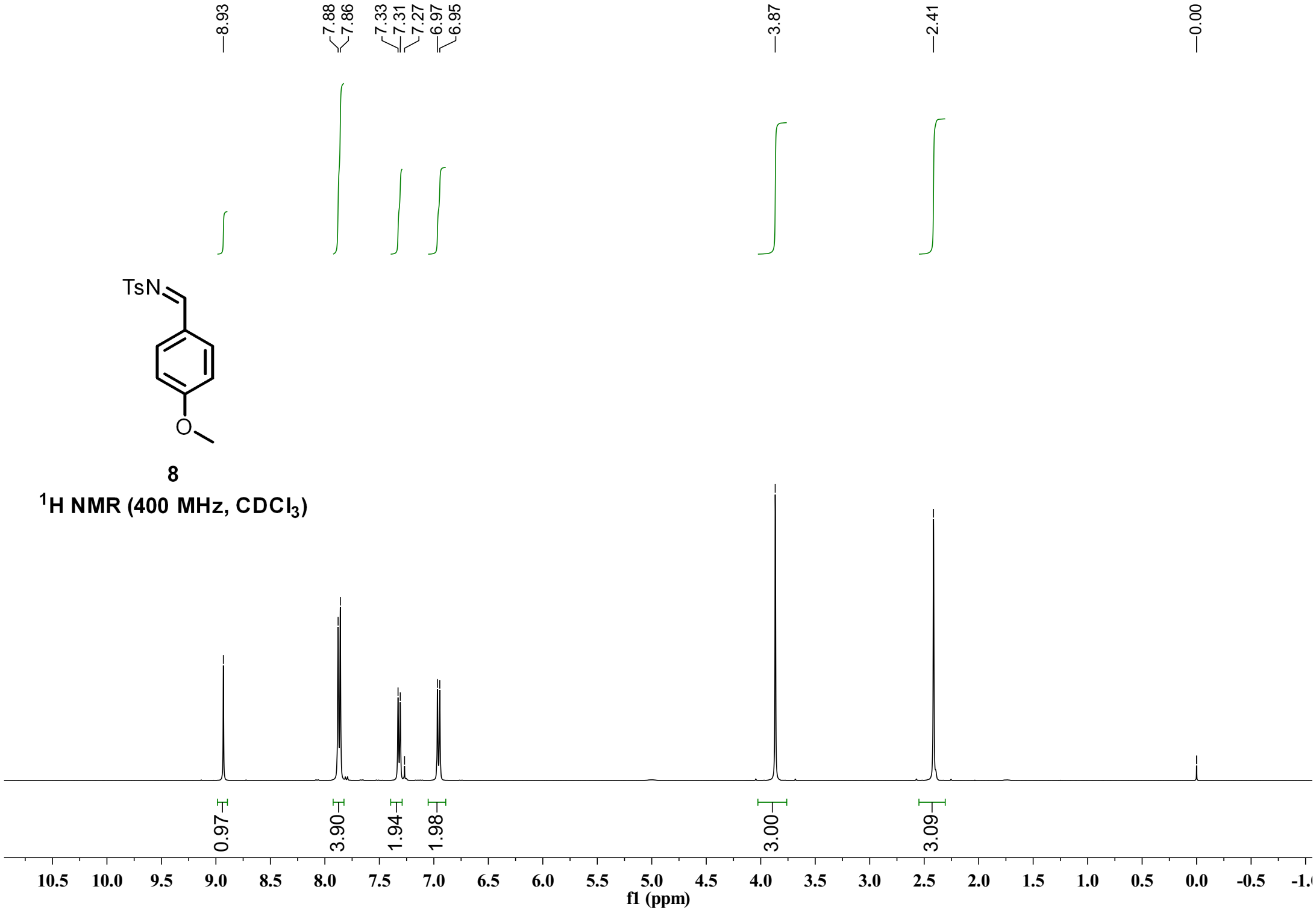
^{13}C NMR (101 MHz, $CDCl_3$)

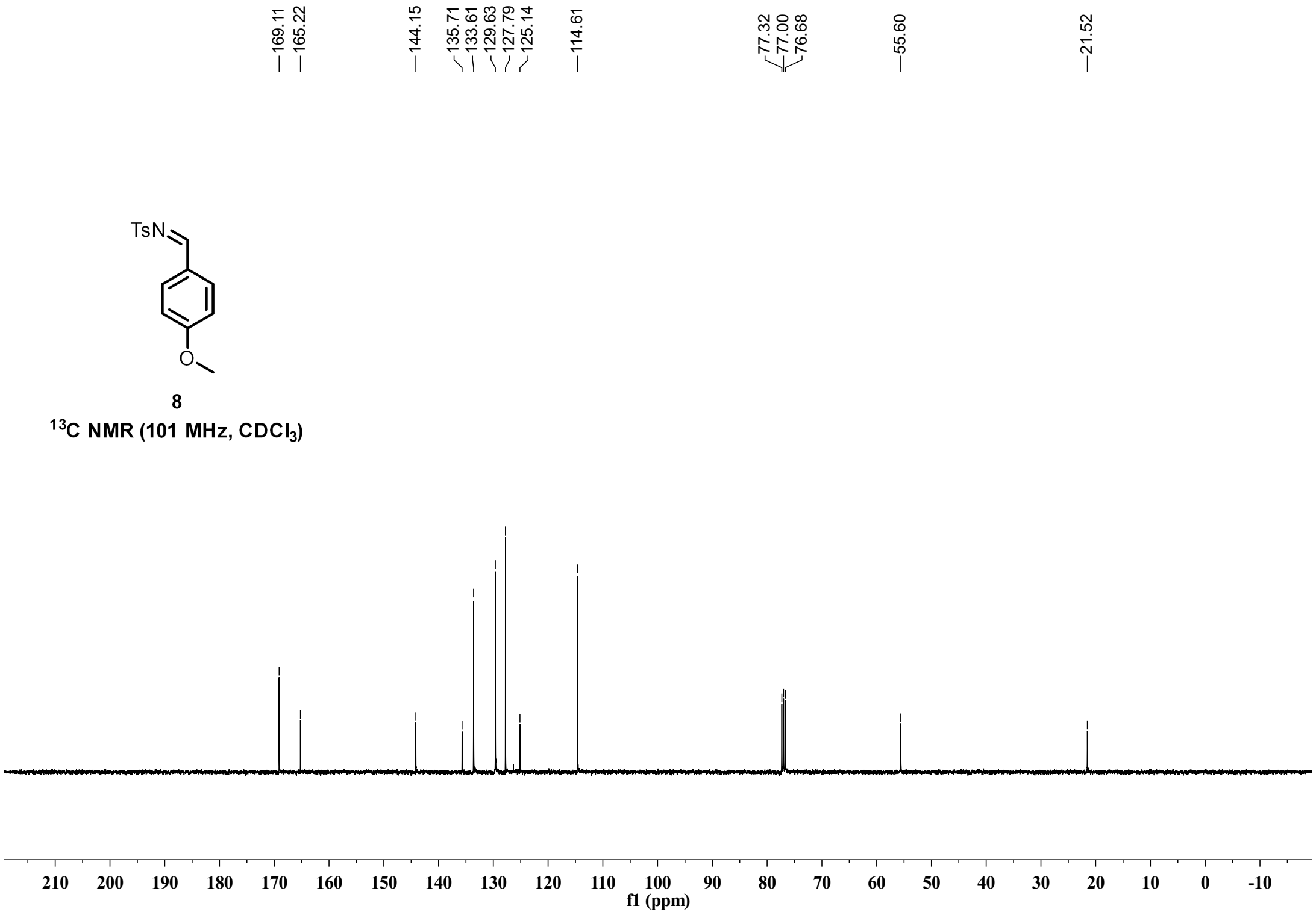


7

^{31}P NMR (162 MHz, $CDCl_3$)




8.93
7.88
7.86
7.33
7.31
7.27
6.97
6.95
3.87
2.41
0.00
TsN
O
8
^{1}H NMR (400 MHz, CDCl$_3$)
0.97
3.90
1.94
1.98
3.00
3.09
10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)



8

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