

Supporting Informations

Ir(III)-Catalyzed Carbenoid Functionalization of Benzamides: Synthesis of *N*-Methoxyisoquinolinediones and *N*-Methoxyisoquinolinones

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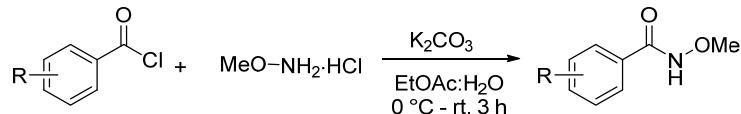
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1. General Methods

Unless otherwise stated, all commercial reagents and solvents were used without additional purification. Analytical thin layer chromatography (TLC) was performed on pre-coated silica gel 60 F₂₅₄ plates. Visualization on TLC was achieved by the use of UV light (254 nm). Column chromatography was carried out by using spectrochem silica gel (60–120, 100–200, 230–400 mesh). ¹H NMR spectra were recorded on Bruker AC 200 MHz, JEOL AL-400 (400 MHz), Bruker DRX 400/500 MHz spectrometers. TMS was used as an internal standard and the chemical shifts were reported in parts per million (δ) relative to internal standard TMS (0 ppm) or CDCl₃ (7.27 ppm). In case of the peak patterns are indicated as follows: s, singlet; d, doublet; dd, doublet of doublet; t, triplet; m, multiplet; q, quartet. The coupling constants, J are reported in Hertz (Hz). ¹³C NMR spectra were obtained by JEOL AL-400 (100 MHz), Bruker DRX (125 MHz), and Bruker DRX (100 MHz) spectrometers and referenced to the internal solvent signals (central peak is 77.0 ppm in CDCl₃). For carbon appearing as doublet, peak at higher value was mentioned along with coupling constant in Hz. High-resolution mass spectra (HRMS) were recorded on a Thermo Scientific Q-Exactive, Accela 1250 pump. Fourier transform infrared (FT-IR) spectra were taken on a UATR (Single Reflection Diamond) for the Spectrum Two in the 600–4000 cm⁻¹ region. Dichloro(η^5 -pentamethylcyclopentadienyl)iridium(III) dimer (99%) was purchased from Alfa Aesar, and all the silver salts were purchased from Aldrich Chemicals and Alfa Asear. All the amides¹ and diazo compounds^{2,3} were prepared according to reported procedure.

2. General Procedure for the Preparation of Starting Materials

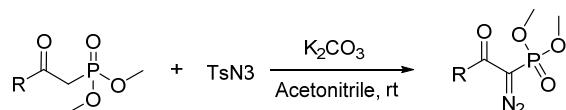
2.1. Preparation of amides (1)¹



To a solution of K₂CO₃ (6.0 mmol, 2.0 equiv) in a mixture of EtOAc/H₂O (30 mL, 2:1) was added *O*-methylhydroxylamine hydrochloride (3.6 mmol, 1.2 equiv). The resulting solution was cooled to 0°C, followed by dropwise addition of the benzoyl chloride (3.0 mmol, 1.0 equiv). The reaction mixture was warmed to room temperature and stirred for overnight. The organic

phase was separated and the aqueous phase was extracted with EtOAc (20 mL × 3). The combined organic layers were dried over Na₂SO₄, filtered, and evaporated under reduced pressure. The pure products were obtained by flash column chromatography.

2.2. Preparation of Diazo substrate (2)^{2,3}

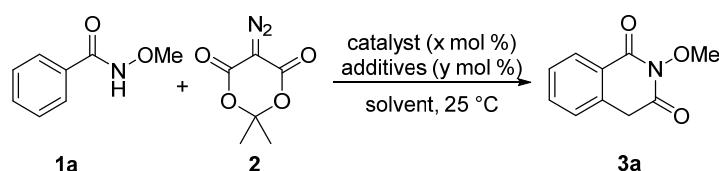


To a stirred solution of Meldrum's acid or phosphonate (3.0 mmol) and TsN₃ (712.0 mg, 3.6 mmol) in acetonitrile (10 mL) was added K₂CO₃ (624.0 mg, 4.52 mmol) and the resulting mixture was stirred at room temperature for over night. After completion of reaction, the reaction mixture was filtered through a pad of celite and washed with EtOAc (10 mL x 3). The solvent was evaporated under reduced pressure and the residue was purified by flash column chromatography using EtOAc/Pet. ether as eluent to furnish the pure diazo compound.

3. Optimization of Reaction Conditions

To a screw capped vial with a spinvane triangular-shaped Teflon stirbar were added N-methoxybenzamide (**1a**, 22.7 mg, 0.15 mmol), dimethyl (1-diazo-2-oxopropyl)phosphonate (**2a**, 28.1 mg, 0.11 mmol), catalyst, additive, and solvent (0.7 mL) under air. The reaction mixture was stirred at room temperature for mentioned time. After the reaction, the reaction mixture was filtered through a pad of celite and then washed with ethyl acetate (5 mL x 3). Solvents were removed under reduced pressure and the crude yield was measured by ¹H NMR spectrum using an internal standard (dibromomethane).

Table S1. Optimization of Reaction Conditions^a



Entry	Catalyst (mol %)	Additives (mol %)	Solvent	Time (h)	Yield ^b (%)
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1	[IrCp*Cl ₂] ₂ (2.0)	AgNTf ₂ (8.0)	MeOH	10	25
2	[IrCp*Cl ₂] ₂ (2.0)	AgNTf ₂ (8.0)	THF	10	58
3	[IrCp*Cl ₂] ₂ (2.0)	AgNTf ₂ (8.0)	Dioxane	10	72
4	[IrCp*Cl ₂] ₂ (2.0)	AgNTf ₂ (8.0)	DCE	10	93
5	[IrCp*Cl ₂] ₂ (2.0)	AgNTf ₂ (8.0)	DCE	5	95 (90)
6	[IrCp*Cl ₂] ₂ (2.0)	AgSbF ₆ (8.0)	DCE	5	80
7	[IrCp*Cl ₂] ₂ (2.0)	AgBF ₄ (8.0)	DCE	5	72
8	[IrCp*Cl ₂] ₂ (2.0)	AgOAc (8.0)	DCE	5	45
9	[IrCp*Cl ₂] ₂ (2.0)	AgOC(O)CF ₃ (8.0)	DCE	5	61
10	[IrCp*Cl ₂] ₂ (1.0)	AgNTf ₂ (4.0)	DCE	5	68
11	[IrCp*Cl ₂] ₂ (2.0)	AgNTf ₂ (8.0)	DCE	1	60
12	[IrCp*Cl ₂] ₂ (2.0)	LiNTf ₂ (8.0)	DCE	5	15
13	[IrCp*Cl ₂] ₂ (2.0)	None	DCE	5	12
14	None	AgNTf ₂ (8.0)	DCE	5	0
15	[RhCp*Cl ₂] ₂ (2.0)	AgSbF ₆ (8.0)	DCE	5	<5
16	[RhCp*Cl ₂] ₂ (2.0)	AgSbF ₆ (8.0)	THF	5	<5
17	[Ru(<i>p</i> -Cymene)Cl ₂] ₂ (2.0)	AgNTf ₂ (8.0)	DCE	5	<5

^aReaction conditions: **1a** (0.15 mmol), **2a** (1.1 equiv), with catalyst and additives in solvent (1 mL) at 25 °C. ^bCrude yield was mentioned based on ¹H NMR (CH₂Br₂ as an internal standard); isolated yield in parentheses.

4. Experimental Procedure for Substrate Scope & Spectroscopic Data of Products

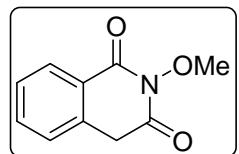
4.1. General procedure for Substrate Scope of *N*-Methoxyisoquinolinedione

To a screw capped vial with a spinvane triangular-shaped Teflon stirbar were added (hetero)aryl oxime (0.15 mmol), diazocompounds (0.17 mmol), [IrCp*Cl₂]₂ (2.0 mol%, 2.4 mg), AgNTf₂ (8.0 mol%, 4.7 mg), and 1,2-dichloroethane (1.0 mL) under air. The reaction mixture was stirred at room temperature for 5 h. After completion of the reaction, the reaction mixture was filtered through a pad of celite and then washed with EtOAc (5 mL x 3). Solvents were removed under reduced pressure and the residue was purified by column chromatography (EtOAc/Petroleum ether) to obtain the desired pure product.

4.2. Spectroscopic data of Synthesized Compounds

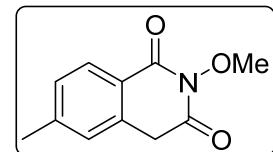
2-Methoxyisoquinoline-1,3(2H,4H)-dione (3a)⁴

Following the general procedure compound **3a** was obtained in 90% yield (26.0 mg) as yellow solid; R_f 0.4 (50% ethyl acetate/pet. ether); mp: 130–106 °C; FT-IR: $\bar{\nu}_{\text{max}}$ 3681, 2923, 1722, 1688, 1252, 682 cm⁻¹. ¹H NMR (500 MHz, CDCl₃): δ 3.95 (s, 3H), 4.13 (s, 2H), 7.25 (s, 1H), 7.43 (t, J = 7.6 Hz, 1H), 7.55–7.61 (m, 1H), 8.15 (d, J = 7.6 Hz, 1H); ¹³C NMR (125 MHz, CDCl₃): δ 37.4, 64.0, 125.0, 127.3, 127.8, 128.9, 133.1, 133.9, 161.1, 165.5; HRMS (ESI+): calcd. for C₁₀H₁₀O₃N [M+H]⁺: 192.0655, found: 192.0654.



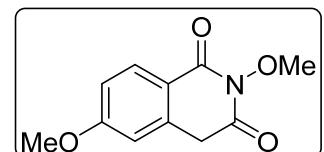
2-Methoxy-6-methylisoquinoline-1,3(2H,4H)-dione (3b)⁴

Following the general procedure compound **3b** was obtained in 92% yield (28.3 mg) as yellow solid; R_f 0.4 (50% ethyl acetate/pet. ether); mp: 150–153 °C; FT-IR: $\bar{\nu}_{\text{max}}$ 3681, 2981, 1719, 1689, 1257, 684 cm⁻¹. ¹H NMR (500 MHz, CDCl₃): δ 2.42 (s, 3H), 3.98 (s, 3H), 4.10 (s, 2H), 7.08 (s, 1H), 7.26 (s, 1H), 8.08 (d, J = 7.9 Hz, 1H); ¹³C NMR (125 MHz, CDCl₃): δ 21.7, 37.4, 64.1, 122.6, 127.8, 129.0, 129.1, 133.1, 145.2, 161.2, 165.7; HRMS (ESI+): calcd. for C₁₁H₁₂O₃N [M+H]⁺: 206.0812, found: 206.0813.



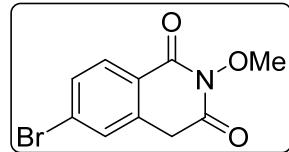
2,6-Dimethoxyisoquinoline-1,3(2H,4H)-dione (3c)⁴

Following the general procedure compound **3c** was obtained in 90% yield (30 mg) as yellow solid; R_f 0.3 (60% ethyl acetate/pet. ether); mp: 135–137 °C; FT-IR: $\bar{\nu}_{\text{max}}$ 3712, 2922, 1721, 1689, 1345, 692 cm⁻¹. ¹H NMR (500 MHz, CDCl₃): δ 3.87 (s, 3H), 3.97 (s, 3H), 4.10 (s, 2H), 6.70 (s, 1H), 6.96 (dd, J = 8.9, 2.4 Hz, 1H), 8.11 (d, J = 8.9 Hz, 1H); ¹³C NMR (125 MHz, CDCl₃): δ 37.6, 55.6, 64.1, 111.6, 114.5, 117.8, 131.2, 135.4, 160.8, 164.1, 165.5; HRMS (ESI+): calcd. for C₁₁H₁₂O₄N [M+H]⁺: 222.0761, found: 222.0761.



6-Bromo-2-methoxyisoquinoline-1,3(2H,4H)-dione (3d)⁴

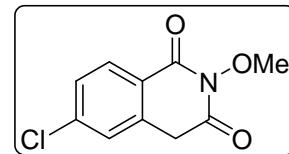
Following the general procedure compound **3d** was obtained in 83% yield (33.6 mg) as yellow solid; R_f 0.3 (50% ethyl acetate/pet. ether); mp: 205–208 °C; FT-IR: $\bar{\nu}_{\text{max}}$ 3684, 2920, 1718, 1678, 1304, 731, 682 cm⁻¹. ¹H NMR (400 MHz, CDCl₃): δ 3.99 (s, 3H), 4.14 (s, 2H), 7.48 (s, 1H), 7.61 (d, J = 8.6



Hz, 1H), 8.07 (d, J = 8.3 Hz, 1H); ^{13}C NMR (100 MHz, CDCl_3): δ 37.1, 64.2, 124.1, 129.3, 130.4, 130.6, 131.6, 134.8, 160.5, 164.7; HRMS (ESI+): calcd. for $\text{C}_{10}\text{H}_9\text{O}_3\text{NBr} [\text{M}+\text{H}]^+$: 269.9760, found: 269.9760.

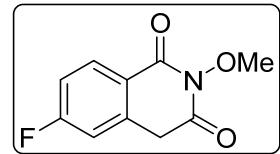
6-Chloro-2-methoxyisoquinoline-1,3(2H,4H)-dione (3e)⁴

Following the general procedure compound **3e** was obtained in 85% yield (28.8 mg) as yellow solid; R_f 0.3 (50% ethyl acetate/pet. ether); mp: 190-192 °C; FT-IR: $\bar{\nu}_{\text{max}}$ 3712, 2973, 1728, 1644, 1334, 888, 662 cm^{-1} . ^1H NMR (400 MHz, CDCl_3): δ 3.35 (br. s., 2H), 3.91 (s, 3H), 7.26 (d, J =1.7 Hz, 1H), 7.38 (dd, J = 8.6, 1.7 Hz, 1H), 8.06 (d, J = 8.6 Hz, 1H); ^{13}C NMR (100 MHz, CDCl_3): δ 36.7, 64.0, 123.4, 127.3, 128.5, 130.4, 134.6, 134.7, 140.6, 160.5, 165.1; HRMS (ESI+): calcd. for $\text{C}_{10}\text{H}_9\text{O}_3\text{NCl} [\text{M}+\text{H}]^+$: 226.0265, found: 226.0265.



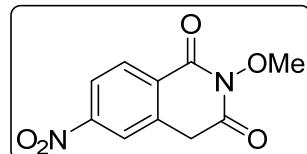
6-Fluoro-2-methoxyisoquinoline-1,3(2H,4H)-dione (3f)⁴

Following the general procedure compound **3f** was obtained in 82% yield (25.6 mg) as yellow solid; R_f 0.3 (50% ethyl acetate/pet. ether); mp: 160-162 °C; FT-IR: $\bar{\nu}_{\text{max}}$ 3681, 2863, 1718, 1678, 1033, 731, 765, 677 cm^{-1} . ^1H NMR (400 MHz, CDCl_3): δ 3.98 (s, 3H), 4.15 (s, 2H), 6.95–7.03 (m, 1H), 7.17 (td, J = 8.5, 2.3 Hz, 1H), 8.23 (dd, J = 8.9, 5.7 Hz, 1H); ^{13}C NMR (100 MHz, CDCl_3): δ 37.4, 64.2, 114.2 (d, J = 22.8 Hz), 115.9 (d, J = 22.8 Hz), 121.6 (d, J = 1.9 Hz), 132.0 (d, J = 9.6 Hz), 136.0 (d, J = 9.6 Hz), 160.2, 164.8 (d, J = 13.4 Hz), 167.3; HRMS (ESI+): calcd. for $\text{C}_{10}\text{H}_9\text{O}_3\text{NF} [\text{M}+\text{H}]^+$: 210.0561, found: 210.0561.



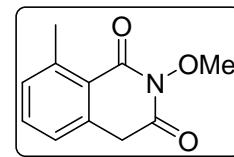
2-Methoxy-6-nitroisoquinoline-1,3(2H,4H)-dione (3g)

Following the general procedure compound **3f** was obtained in 62% yield (22.0 mg) as yellow solid; R_f 0.3 (60% ethyl acetate/pet. ether); mp: 210-213 °C; FT-IR: $\bar{\nu}_{\text{max}}$ 3681, 2973, 1729, 1644, 1334, 1033, 728, 666 cm^{-1} . ^1H NMR (400 MHz, CDCl_3): δ 4.04 (s, 3H), 4.30 (s, 2H), 8.20 (s, 1H), 8.31 (d, J = 8.3 Hz, 1H), 8.45 (d, J = 8.8 Hz, 1H); ^{13}C NMR (100 MHz, CDCl_3): δ 37.4, 64.4, 122.6, 122.8, 130.1, 130.9, 134.7, 150.9, 159.5, 164.1; HRMS (ESI+): calcd. for $\text{C}_{10}\text{H}_9\text{O}_5\text{N}_2 [\text{M}+\text{H}]^+$: 237.0506, found: 237.0507.



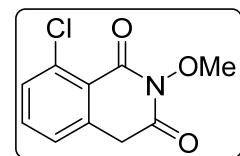
2-Methoxy-8-methylisoquinoline-1,3(2H,4H)-dione (3h)⁴

Following the general procedure compound **3h** was obtained in 72% yield (22.2 mg) as yellow solid; R_f 0.4 (50% ethyl acetate/pet. ether); mp: 142–146 °C; FT-IR: $\bar{\nu}_{\text{max}}$ 3681, 2864, 1730, 1691, 1340, 1065, 634 cm⁻¹. ¹H NMR (400 MHz, CDCl₃): δ 2.79 (s, 3H), 4.00 (s, 3H), 4.14 (s, 2H), 7.13 (d, J = 7.3 Hz, 1H), 7.22–7.30 (m, 1H), 7.41–7.50 (m, 1H); ¹³C NMR (100 MHz, CDCl₃): δ 23.1, 37.8, 64.0, 123.1, 125.6, 131.6, 132.9, 134.4, 143.3, 161.6, 165.2; HRMS (ESI+): calcd. for C₁₁H₁₂O₃N [M+H]⁺: 206.0812, found: 206.0813.



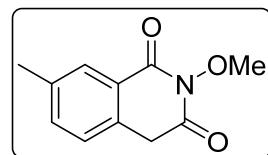
8-Chloro-2-methoxyisoquinoline-1,3(2H,4H)-dione (3i)⁴

Following the general procedure compound **3i** was obtained in 66% yield (22.4 mg) as yellow solid; R_f 0.3 (50% ethyl acetate/pet. ether); mp: 140–143 °C; FT-IR: $\bar{\nu}_{\text{max}}$ 3681, 2864, 1730, 1691, 1340, 1033, 679, 634 cm⁻¹. ¹H NMR (500 MHz, CDCl₃): δ 3.98 (s, 3H), 4.16 (s, 2H), 7.18–7.24 (m, 1H), 7.47–7.52 (m, 2H); ¹³C NMR (125 MHz, CDCl₃): δ 37.8, 64.1, 122.0, 126.4, 131.8, 133.6, 136.0, 136.7, 158.8, 164.2; HRMS (ESI+): calcd. for C₁₀H₉O₃NCl [M+H]⁺: 226.0265, found: 226.0265.



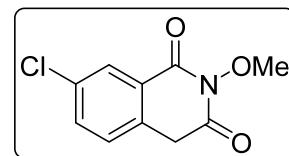
2-Methoxy-7-methylisoquinoline-1,3(2H,4H)-dione (3j)⁴

Following the general procedure compound **3j** was obtained in 75% yield (23.2 mg) as yellow solid; R_f 0.4 (50% ethyl acetate/pet. ether); mp: 141–143 °C; FT-IR: $\bar{\nu}_{\text{max}}$ 3712, 1981, 1731, 1691, 1263, 1033, 781, 726 cm⁻¹. ¹H NMR (400 MHz, CDCl₃): δ 2.43 (s, 3H), 3.99 (s, 3H), 4.11 (s, 2H), 7.17 (d, J = 7.8 Hz, 1H), 7.42 (d, J = 7.8 Hz, 1H), 8.02 (s, 1H); ¹³C NMR (100 MHz, CDCl₃): δ 21.0, 37.2, 64.1, 124.9, 127.2, 129.1, 130.2, 135.0, 138.0, 161.3, 165.7; HRMS (ESI+): calcd. for C₁₁H₁₂O₃N [M+H]⁺: 206.0812, found: 206.0813.



7-Chloro-2-methoxyisoquinoline-1,3(2H,4H)-dione (3k-1)

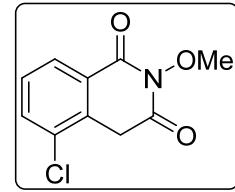
Following the general procedure compound **3k-1** was obtained in 33% yield (11.2 mg) as yellow solid; R_f 0.3 (50% ethyl acetate/pet. ether); mp: 142–145 °C; FT-IR: $\bar{\nu}_{\text{max}}$ 3681, 2869, 1719, 1693, 1263, 1033, 888, 662 cm⁻¹. ¹H NMR (400 MHz, CDCl₃): δ 4.00 (s, 3H), 4.14 (s, 2H), 7.25 (d, J = 8.2 Hz, 1H), 7.55–7.61 (m, 1H), 8.20 (d,



$J = 2.3$ Hz, 1H); ^{13}C NMR (100 MHz, CDCl_3): δ 37.1, 64.2, 126.8, 128.8, 128.8, 131.4, 134.2, 134.4, 160.1, 164.9; HRMS (ESI+): calcd. for $\text{C}_{10}\text{H}_9\text{O}_3\text{NCl} [\text{M}+\text{H}]^+$: 226.0265, found: 226.0267.

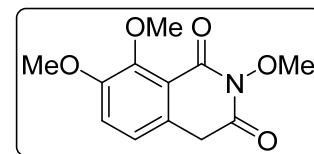
5-Chloro-2-methoxyisoquinoline-1,3(2H,4H)-dione (**3k-2**)

Following the general procedure compound **3k-2** was obtained in 28% yield (9.4 mg) as yellow solid; R_f 0.3 (50% ethyl acetate/pet. ether); mp: 140-144 °C; FT-IR: $\bar{\nu}_{\text{max}}$ 3681, 2866, 1734, 1688, 1033, 752, 642 cm^{-1} . ^1H NMR (400 MHz, CDCl_3): δ 4.01 (s, 3H), 4.15 (s, 2H), 7.45 – 7.49 (m, 1H), 7.68 (dd, $J = 8.0, 1.0$ Hz, 1H), 8.18 (dd, $J = 7.8, 1.0$ Hz, 1H); ^{13}C NMR (100 MHz, CDCl_3): δ 36.2, 64.2, 127.0, 127.8, 129.0, 131.3, 133.0, 134.4, 160.2, 164.3; HRMS (ESI+): calcd. for $\text{C}_{10}\text{H}_9\text{O}_3\text{NCl} [\text{M}+\text{H}]^+$: 226.0265, found: 226.0266.



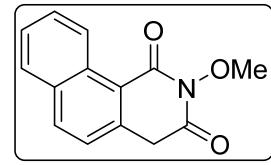
2,7,8-Trimethoxyisoquinoline-1,3(2H,4H)-dione (**3l**)

Following the general procedure compound **3l** was obtained in 62% yield (23.3 mg) as pale yellow solid; R_f 0.3 (70% ethyl acetate/pet. ether); mp: 157-159 °C; FT-IR: $\bar{\nu}_{\text{max}}$ 3681, 2973, 1718, 1678, 1492, 1033, 765, 677 cm^{-1} . ^1H NMR (500 MHz, CDCl_3): δ 3.89 (s, 3H), 3.93 (s, 3H), 3.97 (s, 3H), 4.05 (s, 2H), 6.97 (d, $J = 8.5$ Hz, 1H), 7.15 (d, $J = 8.5$ Hz, 1H); ^{13}C NMR (125 MHz, CDCl_3): δ 36.9, 56.3, 61.4, 64.0, 117.8, 119.1, 123.1, 125.5, 151.1, 153.1, 158.9, 165.3; HRMS (ESI+): calcd. for $\text{C}_{12}\text{H}_{14}\text{O}_5\text{N} [\text{M}+\text{H}]^+$: 252.0866, found: 252.0867.



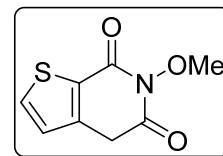
2-Methoxybenzo[h]isoquinoline-1,3(2H,4H)-dione (**3m**)

Following the general procedure compound **3m** was obtained in 65% yield (23.5 mg) as yellow solid; R_f 0.4 (50% ethyl acetate/pet. ether); mp: 185-188 °C; FT-IR: $\bar{\nu}_{\text{max}}$ 3681, 2844, 1722, 1687, 1251, 1070, 749, 647 cm^{-1} . ^1H NMR (400 MHz, CDCl_3): δ 4.07 (s, 3H), 4.28 (s, 2H), 7.30 (d, $J = 8.2$ Hz, 1H), 7.59–7.64 (m, 1H), 7.74 (ddd, $J = 8.6, 7.0, 1.4$ Hz, 1H), 7.87–7.92 (m, 1H), 8.07 (d, $J = 8.2$ Hz, 1H), 9.67 (d, $J = 8.7$ Hz, 1H); ^{13}C NMR (100 MHz, CDCl_3): δ 38.5, 64.1, 119.2, 124.5, 126.1, 126.9, 128.8, 129.6, 131.6, 133.0, 135.5, 135.6, 161.9, 164.9; HRMS (ESI+): calcd. for $\text{C}_{14}\text{H}_{12}\text{O}_3\text{N} [\text{M}+\text{H}]^+$: 242.0812, found: 242.0812.



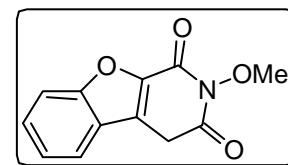
6-Methoxy-3a,7a-dihydrothieno[2,3-c]pyridine-5,7(4H,6H)-dione (3n)

Following the general procedure compound **3n** was obtained in 77% yield (22.8 mg) as yellow solid; R_f 0.3 (60% ethyl acetate/pet. ether); mp: 165–168 °C; FT-IR: $\bar{\nu}_{\text{max}}$ 3681, 3091, 2973, 1781, 1688, 1424, 1249, 864, 755 cm^{-1} . ^1H NMR (400 MHz, CDCl_3): δ 4.00 (s, 3H), 4.09 (s, 2H), 7.02 (d, $J = 5.0$ Hz, 2H), 7.76 (d, $J = 5.0$ Hz, 2H); ^{13}C NMR (100 MHz, CDCl_3): δ 35.6, 64.3, 126.5, 128.0, 135.1, 139.9, 156.9, 165.8; HRMS (ESI+): calcd. for $\text{C}_8\text{H}_8\text{O}_3\text{NS} [\text{M}+\text{H}]^+$: 198.0219, found: 198.0219.



2-Methoxybenzofuro[2,3-c]pyridine-1,3(2H,4H)-dione (3o)

Following the general procedure compound **3o** was obtained in 80% yield (27.8 mg) as brown solid; R_f 0.3 (60% ethyl acetate/pet. ether); mp: 200–202 °C; FT-IR: $\bar{\nu}_{\text{max}}$ 3712, 2866, 1730, 1696, 1379, 1179, 1060, 744 cm^{-1} . ^1H NMR (400 MHz, CDCl_3): δ 4.04 (s, 3H), 4.19 (s, 2H), 7.39–7.44 (m, 1H), 7.55–7.60 (m, 1H), 7.68 (d, $J = 8.3$ Hz, 1H), 7.65 (d, $J = 8.3$ Hz, 1); ^{13}C NMR (100 MHz, CDCl_3): δ 31, 65, 113, 121, 122, 124, 125, 127, 129, 140, 157, 165; HRMS (ESI+): calcd. for $\text{C}_{12}\text{H}_{10}\text{O}_4\text{N} [\text{M}+\text{H}]^+$: 232.0604, found: 232.0604.



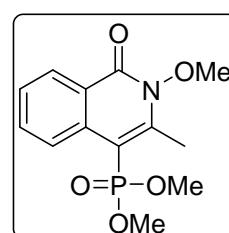
4.3. General procedure for Substrate Scope of *N*-Methoxyisoquinolinone

To a screw capped vial with a spinvane triangular-shaped Teflon stirbar were added (hetero)aryl oxime (0.15 mmol), diazocompounds (0.17 mmol), $[\text{IrCp}^*\text{Cl}_2]_2$ (2.0 mol%, 2.4 mg), AgNTf_2 (8.0 mol%, 4.7 mg), and 1,2-dichloroethane (1.0 mL) under air. The reaction mixture was stirred at 35 °C for 10 h. After completion of the reaction, the reaction mixture was filtered through a pad of celite and then washed with EtOAc (5 mL x 3). Solvents were removed under reduced pressure and the residue was purified by column chromatography ($\text{EtOAc}/\text{Petroleum ether}$) to obtain the desired pure product.

4.2. Spectroscopic data of Synthesized Compounds

Dimethyl (2-methoxy-3-methyl-1-oxo-1,2-dihydroisoquinolin-4-yl)phosphonate (5a)

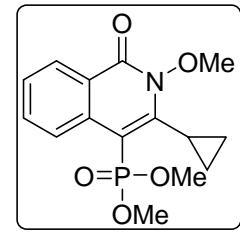
Following the general procedure compound **5a** was obtained in 90% yield (40.1 mg) as white solid; R_f 0.3 (70% ethyl acetate/pet. ether); mp: 108–110



°C; FT-IR: $\bar{\nu}_{\text{max}}$ 3681, 2855, 1678, 1606, 1395, 1050, 1012, 795, 691 cm⁻¹. ¹H NMR (500 MHz, CDCl₃): δ 2.98 (d, J = 2.1 Hz, 3H), 3.78 (s, 3H), 3.80 (s, 3H), 4.10 (s, 3H), 7.49 (t, J = 7.5 Hz, 1H), 7.66–7.73 (m, 1H), 8.32 (d, J = 8.5 Hz, 1H), 8.45 (d, J = 8.2 Hz, 1H); ¹³C NMR (125 MHz, CDCl₃): δ 15.4 (d, J = 1.9 Hz), 52.3 (d, J = 5.6 Hz), 63.7, 99.7 (d, J = 199.1 Hz), 125.4 (d, J = 14.4 Hz), 126.0, 126.5, 127.6, 132.8, 134.8 (d, J = 9.5 Hz), 151.1 (d, J = 23.2 Hz), 158.3; HRMS (ESI+): calcd. for C₁₃H₁₇O₅NP [M+H]⁺: 298.0839, found: 298.0838.

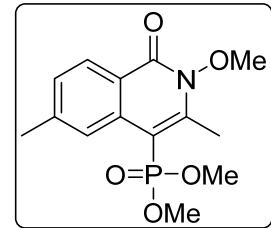
Dimethyl (3-cyclopropyl-2-methoxy-1-oxo-1,2-dihydroisoquinolin-4-yl)phosphonate (5b)

Following the general procedure compound **5b** was obtained in 88% yield (42.7 mg) as yellow oil; R_f 0.3 (70% ethyl acetate/pet. ether); FT-IR: $\bar{\nu}_{\text{max}}$ 3468, 2949, 1669, 1307, 1262, 1050, 1017, 824, 749, 696 cm⁻¹. ¹H NMR (500 MHz, CDCl₃): δ 0.96–1.01 (m, 2H), 1.23–1.28 (m, 2H), 2.19–2.29 (m, 1H), 3.76 (s, 3H), 3.79 (s, 3H), 4.12 (s, 3H), 7.44–7.50 (m, 1H), 7.65 (ddd, J = 8.5, 7.1, 1.8 Hz, 1H), 8.14 (d, J = 8.2 Hz, 1H), 8.40 (dt, J = 8.1, 1.7 Hz, 1H); ¹³C NMR (125 MHz, CDCl₃): δ 9.5, 12.8 (d, J = 3.7 Hz), 52.5 (d, J = 5.8 Hz), 64.2, 102.9 (d, J = 204.2 Hz), 125.7 (d, J = 14.3 Hz), 126.1 (d, J = 2.8 Hz), 126.7, 127.5, 132.5, 134.8 (d, J = 8.7 Hz), 154.5 (d, J = 20.2 Hz), 158.5; HRMS (ESI+): calcd. for C₁₅H₁₉O₅NP [M+H]⁺: 324.0995, found: 324.0995.



Dimethyl (2-methoxy-3,6-dimethyl-1-oxo-1,2-dihydroisoquinolin-4-yl)phosphonate (5c)

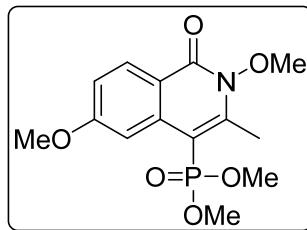
Following the general procedure compound **5c** was obtained in 84% yield (39.3 mg) as white solid; R_f 0.3 (70% ethyl acetate/pet. ether); mp: 113–116 °C; FT-IR: $\bar{\nu}_{\text{max}}$ 3681, 2844, 1666, 1225, 1190, 1050, 1017, 833, 773, 695 cm⁻¹. ¹H NMR (400 MHz, CDCl₃): δ 2.49 (s, 3H), 2.93 (d, J = 2.2 Hz, 3H), 3.75 (s, 3H), 3.78 (s, 3H), 4.06 (s, 3H), 7.25–7.33 (m, 1H), 8.11 (s, 1H), 8.31 (dd, J = 8.3, 1.7 Hz, 1H); ¹³C NMR (100 MHz, CDCl₃): δ 15.5 (d, J = 2.3 Hz), 22.4, 52.3 (d, J = 5.4 Hz), 63.7, 98.9 (d, J = 198.2 Hz), 123.2 (d, J = 14.5 Hz), 125.7 (d, J = 1.9 Hz), 127.6, 128.1, 134.8 (d, J = 9.7 Hz), 143.5, 151.0 (d, J = 23.2 Hz), 158.2; HRMS (ESI+): calcd. for C₁₄H₁₉O₅NP [M+H]⁺: 312.0995, found: 312.0995.



Dimethyl (2,6-dimethoxy-3-methyl-1-oxo-1,2-dihydroisoquinolin-4-yl)phosphonate (5d)

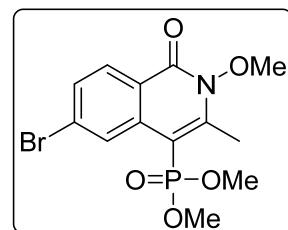
Following the general procedure compound **5d** was obtained in 85% yield (41.8 mg) as white

solid; R_f 0.3 (70% ethyl acetate/pet. ether); mp: 128-131 °C; FT-IR: $\bar{\nu}_{\text{max}}$ 3681, 2844, 1694, 1445, 1058, 1033, 683, 645 cm^{-1} . ^1H NMR (400 MHz, CDCl_3): δ 2.91 (d, $J = 1.5$ Hz, 3H), 3.75–3.77 (m, 3H), 3.77–3.81 (m, 3H), 3.91 (s, 3H), 4.07 (s, 3H), 7.05 (dd, $J = 8.8, 1.7$ Hz, 1H), 7.88 (d, $J = 2.0$ Hz, 1H), 8.34 (d, $J = 8.8$ Hz, 1H); ^{13}C NMR (100 MHz, CDCl_3): δ 15.6 (d, $J = 2.3$ Hz), 52.3 (d, $J = 5.5$ Hz), 55.4, 63.8, 98.4 (d, $J = 199.2$ Hz), 108.0, 115.6, 119.1 (d, $J = 14.4$ Hz), 137.0 (d, $J = 9.3$ Hz), 151.5 (d, $J = 22.3$ Hz), 158.0, 162.0; HRMS (ESI+): calcd. for $\text{C}_{14}\text{H}_{19}\text{O}_6\text{NP} [\text{M}+\text{H}]^+$: 328.0945, found: 328.0943.



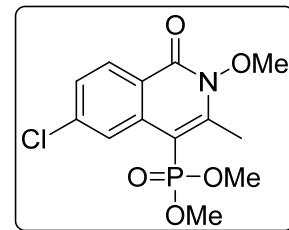
Dimethyl (6-bromo-2-methoxy-3-methyl-1-oxo-1,2-dihydroisoquinolin-4-yl)phosphonate (5e)

Following the general procedure compound **5e** was obtained in 77% yield (43.5 mg) as yellow solid; R_f 0.3 (60% ethyl acetate/pet. ether); mp: 137-139 °C; FT-IR: $\bar{\nu}_{\text{max}}$ 3858, 3745, 2951, 1666, 1226, 1190, 1001, 833, 773, 695 cm^{-1} . ^1H NMR (400 MHz, CDCl_3): δ 2.93 (s, 3H), 3.77 (s, 3H), 3.80 (s, 3H), 4.07 (s, 3H), 7.57 (d, $J = 8.6$ Hz, 1 H), 8.26 (d, $J = 8.3$ Hz, 1 H), 8.53 (s, 1 H); ^{13}C NMR (100 MHz, CDCl_3): δ 15.6, 52.5 (d, $J = 5.5$ Hz), 63.8, 98.5 (d, $J = 199.5$ Hz), 124.1 (d, $J = 13.9$ Hz), 128.4, 128.8, 129.2, 129.9, 1.36.3 (d, $J = 9.2$ Hz), 152.1 (d, $J = 22.5$ Hz), 157.8; HRMS (ESI+): calcd. for $\text{C}_{13}\text{H}_{16}\text{O}_5\text{NBrP} [\text{M}+\text{H}]^+$: 375.9944, found: 375.9947.



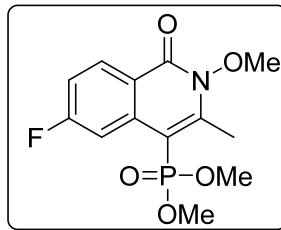
Dimethyl (6-chloro-2-methoxy-3-methyl-1-oxo-1,2-dihydroisoquinolin-4-yl)phosphonate (5f)

Following the general procedure compound **5f** was obtained in 72% yield (35.8 mg) as white solid; R_f 0.3 (60% ethyl acetate/pet. ether); mp: 142-145 °C; FT-IR: $\bar{\nu}_{\text{max}}$ 3680, 2845, 1680, 1257, 1174, 1101, 1016, 988, 808, 758, 647 cm^{-1} . ^1H NMR (400 MHz, CDCl_3): δ 2.93 (s, 3H), 3.77 (s, 3H), 3.80 (s, 3H), 4.07 (s, 3H), 7.57 (d, $J = 8.6$ Hz, 1H), 8.26 (d, $J = 8.3$ Hz, 1H), 8.53 (s, 1H); ^{13}C NMR (100 MHz, CDCl_3): δ 15.6, 52.5 (d, $J = 5.4$ Hz), 63.8, 98.5 (d, $J = 199.6$ Hz), 124.1 (d, $J = 14.1$ Hz), 128.4, 128.8, 129.2, 129.9, 1.36.3 (d, $J = 9.3$ Hz), 152.1 (d, $J = 22.5$ Hz), 157.8; HRMS (ESI+): calcd. for $\text{C}_{13}\text{H}_{16}\text{O}_5\text{NClP} [\text{M}+\text{H}]^+$: 332.0449, found: 332.0449.



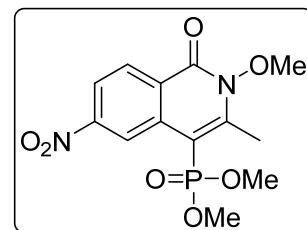
Dimethyl (6-fluoro-2-methoxy-3-methyl-1-oxo-1,2-dihydroisoquinolin-4-yl)phosphonate (5g)

Following the general procedure compound **5g** was obtained in 80% yield (38.0 mg) as white solid; R_f 0.3 (60% ethyl acetate/pet. ether); mp: 129-133 °C; FT-IR: $\bar{\nu}_{\text{max}}$ 3681, 2863, 1678, 1356, 1170, 1054, 1007, 819, 765, 654 cm⁻¹. ¹H NMR (400 MHz, CDCl₃): δ 2.93 (s, 3H), 3.80 (s, 3H), 3.77 (s, 3H), 4.07 (s, 3H), 7.57 (d, J = 8.6 Hz, 1H), 8.26 (d, J = 8.3 Hz, 1H), 8.53 (s, 1H); ¹³C NMR (100 MHz, CDCl₃): δ 15.6, 52.3 (d, J = 5.4 Hz), 63.8, 98.7 (dd, J = 3.1, 199.3 Hz), 111.9 (d, J = 25.0 Hz), 115.2 (d, J = 23.8 Hz), 122.0 (d, J = 14.3 Hz), 130.6 (d, J = 10.7 Hz), 137.2 (dd, J = 9.2, 11.7 Hz), 152.2 (d, J = 22.7 Hz), 157.6, 166.3 (d, J = 252.0 Hz); HRMS (ESI+): calcd. for C₁₃H₁₆O₅NFP [M+H]⁺: 316.0745, found: 316.0744.



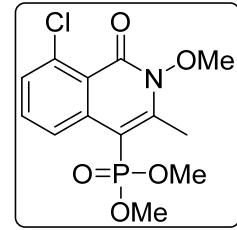
Dimethyl (2-methoxy-3-methyl-6-nitro-1-oxo-1,2-dihydroisoquinolin-4-yl)phosphonate (5h)

Following the general procedure compound **5h** was obtained in 66% yield (34.0 mg) as pale yellow solid; R_f 0.3 (80% ethyl acetate/pet. ether); mp: 157-159 °C; FT-IR: $\bar{\nu}_{\text{max}}$ 3092, 2854, 1667, 1525, 1334, 1174, 1014, 855, 739, 667 cm⁻¹. ¹H NMR (500 MHz, CDCl₃): δ 3.00 (d, J = 2.3 Hz, 3H), 3.84 (s, 3H), 3.86 (s, 3H), 4.13 (s, 3H), 8.23 (dd, J = 8.8, 2.3 Hz, 1H), 8.58–8.61 (m, 1H), 9.32 (d, J = 1.9 Hz, 1H); ¹³C NMR (100 MHz, CDCl₃): δ 15.7 (d, J = 1.7 Hz), 52.7 (d, J = 5.9 Hz), 64.0, 99.8 (d, J = 201.3 Hz), 120.2, 122.3, 129.1 (d, J = 13.5 Hz), 129.6, 135.8 (d, J = 8.7 Hz), 150.4, 153.2 (d, J = 22.1 Hz), 157.2; HRMS (ESI+): calcd. for C₁₃H₁₆O₇N₂P [M+H]⁺: 343.0690, found: 343.0688.



Dimethyl (8-chloro-2-methoxy-3-methyl-1-oxo-1,2-dihydroisoquinolin-4-yl)phosphonate (5i)

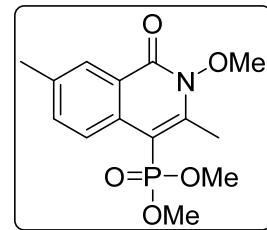
Following the general procedure compound **5i** was obtained in 70% yield (34.8 mg) as pale yellow solid; R_f 0.3 (60% ethyl acetate/pet. ether); mp: 102-105 °C; FT-IR: $\bar{\nu}_{\text{max}}$ 3681, 2865, 1687, 1275, 1184, 1048, 1017, 901, 804, 735, 661 cm⁻¹. ¹H NMR (200 MHz, CDCl₃): δ 2.91 (d, J = 2.1 Hz, 3H), 3.72 (s, 3H), 3.77 (s, 3H), 4.05 (s, 3H), 7.42–7.55 (m, 2H), 8.26 (dd, J = 6.9, 2.8 Hz, 1H); ¹³C NMR (100 MHz, CDCl₃): δ 15.7, 52.4 (d, J = 5.7 Hz), 63.7, 98.3 (d, J = 200.8 Hz), 121.8 (d, J = 14.5 Hz), 125.0, 129.8, 132.1, 135.1 (d, J = 3.0 Hz), 137.9 (d, J = 10.3 Hz).



Hz), 152.1 (d, J = 22.2 Hz), 156.3; HRMS (ESI+): calcd. for $C_{13}H_{16}O_5NClP$ [M+H]⁺: 332.0449, found: 332.0449.

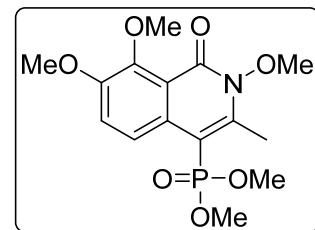
Dimethyl (2-methoxy-3,7-dimethyl-1-oxo-1,2-dihydroisoquinolin-4-yl)phosphonate (5j)

Following the general procedure compound **5j** was obtained in 62% yield (29.0 mg) as white solid; R_f 0.3 (70% ethyl acetate/pet. ether); mp: 120–122 °C; FT-IR: $\bar{\nu}_{\text{max}}$ 3681, 2844, 1692, 1235, 1190, 1064, 1017, 833, 783, 695 cm⁻¹. ¹H NMR (200 MHz, CDCl₃): δ 2.46 (s, 17 H), 2.94 (d, J = 2.3 Hz, 16 H), 3.73 (s, 17 H), 3.78 (s, 17 H), 4.07 (s, 17 H), 7.48 (dd, J = 8.7, 1.8 Hz, 5 H), 8.18 (d, J = 8.7 Hz, 6 H), 8.23 (br. s., 5 H); ¹³C NMR (100 MHz, CDCl₃): δ 15.3, 21.0, 52.2 (d, J = 5.1 Hz), 63.7, 99.0 (d, J = 199.5 Hz), 125.3 (d, J = 14.2 Hz), 125.9, 127.1, 132.4 (d, J = 9.3 Hz), 134.3, 136.7, 150.0 (d, J = 23.7 Hz), 158.3; HRMS (ESI+): calcd. for $C_{14}H_{19}O_5NP$ [M+H]⁺: 312.0995, found: 312.0992.



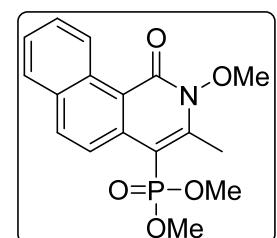
Dimethyl (2,7,8-trimethoxy-3-methyl-1-oxo-1,2-dihydroisoquinolin-4-yl)phosphonate (5k)

Following the general procedure compound **5k** was obtained in 75% yield (40.2 mg) as light yellow oil; R_f 0.3 (80% ethyl acetate/pet. ether); FT-IR: $\bar{\nu}_{\text{max}}$ 3681, 2845, 1659, 1290, 1050, 1016, 834, 780, 724 cm⁻¹. ¹H NMR (500 MHz, CDCl₃): δ 2.89 (d, J = 2.4 Hz, 3H), 3.76 (s, 3H), 3.74 (s, 3H), 3.95 (s, 3H), 3.93 (s, 3H), 4.05 (s, 3H), 7.31 (d, J = 9.2 Hz, 1H), 8.08 (d, J = 9.2 Hz, 1H); ¹³C NMR (125 MHz, CDCl₃): δ 15.9, 52.4 (d, J = 5.7 Hz), 63.7, 99.1 (d, J = 200.5 Hz), 119.2 (d, J = 14.0 Hz), 123.3 (d, J = 2.7 Hz), 126.9, 127.2, 127.9, 128.6, 131.4, 133.8, 137.4 (d, J = 9.5 Hz), 151.8 (d, J = 22.8 Hz), 158.5; HRMS (ESI+): calcd. for $C_{15}H_{21}O_7NP$ [M+H]⁺: 358.1050, found: 358.1049.



Dimethyl (2-methoxy-3-methyl-1-oxo-1,2-dihydrobenzo[h]isoquinolin-4-yl)phosphonate (5l)

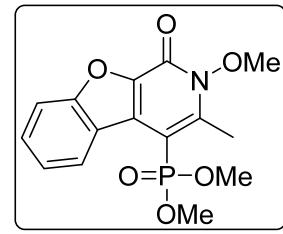
Following the general procedure compound **5l** was obtained in 77% yield (40.2 mg) as brown sticky solid; R_f 0.3 (60% ethyl acetate/pet. ether); mp: 107–109 °C; FT-IR: $\bar{\nu}_{\text{max}}$ 3531, 2950, 1659, 1243, 1032, 1018, 825, 750, 626 cm⁻¹. ¹H NMR (500 MHz, CDCl₃): δ 3.05 (d, J = 2.1 Hz, 3H), 3.78 (s, 3H), 3.80 (s, 3H), 4.15 (s, 3H), 7.63 (td, J = 7.4, 1.1 Hz, 1H), 7.75



(ddd, $J = 8.7, 7.0, 1.4$ Hz, 1H), 7.89 (d, $J = 7.9$ Hz, 1H), 8.04 (d, $J = 9.2$ Hz, 1H), 8.38 (d, $J = 9.2$ Hz, 1H), 10.10 (d, $J = 8.9$ Hz, 1H); ^{13}C NMR (125 MHz, CDCl_3): δ 15.4 (d, $J = 1.9$ Hz), 52.3 (d, $J = 5.6$ Hz), 63.7, 99.7 (d, $J = 199.1$ Hz), 125.4 (d, $J = 14.4$ Hz), 126.0, 126.5, 127.6, 132.8, 134.8 (d, $J = 9.5$ Hz), 151.1 (d, $J = 23.2$ Hz), 158.3; HRMS (ESI+): calcd. for $\text{C}_{17}\text{H}_{19}\text{O}_5\text{NP} [\text{M}+\text{H}]^+$: 348.0995, found: 348.0995.

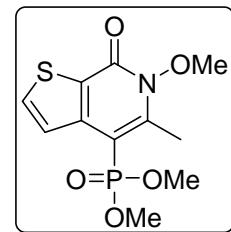
Dimethyl (2-methoxy-3-methyl-1-oxo-1,2-dihydrobenzofuro[2,3-c]pyridin-4-yl)phosphonate (**5m**)

Following the general procedure compound **5m** was obtained in 62% yield (31.4 mg) as pale yellow solid; R_f 0.3 (70% ethyl acetate/pet. ether); mp: 189–192 °C; FT-IR: $\bar{\nu}_{\text{max}}$ 3681, 2865, 1692, 1347, 1240, 1033, 1017, 754, 636 cm^{-1} . ^1H NMR (200 MHz, CDCl_3): δ 3.05 (d, $J = 2.0$ Hz, 3H), 3.78 (s, 3H), 3.84 (s, 3H), 4.18 (s, 3H), 7.35–7.47 (m, 1H), 7.57 (td, $J = 7.7, 1.3$ Hz, 1H), 7.65–7.74 (m, 1H), 8.39 (d, $J = 8.2$ Hz, 1H); ^{13}C NMR (100 MHz, CDCl_3): δ 15.1, 52.5 (d, $J = 5.7$ Hz), 64.3, 95.3 (d, $J = 206.3$ Hz), 112.5, 122.1, 123.7, 125.6, 127.2 (d, $J = 8.3$ Hz), 129.1, 142.8 (d, $J = 18.6$ Hz), 150.3 (d, $J = 24.8$ Hz), 151.1, 157.2; HRMS (ESI+): calcd. for $\text{C}_{15}\text{H}_{17}\text{O}_6\text{NP} [\text{M}+\text{H}]^+$: 338.0788, found: 338.0786.



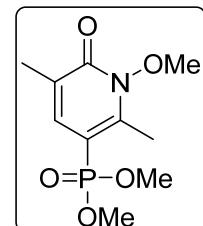
Dimethyl (6-methoxy-5-methyl-7-oxo-3a,6,7,7a-tetrahydrothieno[2,3-c]pyridin-4-yl)phosphonate (**5n**)

Following the general procedure compound **5n** was obtained in 77% yield (35.0 mg) as white solid; R_f 0.3 (70% ethyl acetate/pet. ether); mp: 108–111 °C; FT-IR: $\bar{\nu}_{\text{max}}$ 3681, 2923, 1664, 1506, 1181, 1050, 971, 805, 775, 683 cm^{-1} . ^1H NMR (500 MHz, CDCl_3): δ 2.87 (d, $J = 2.1$ Hz, 3H), 3.75 (s, 3H), 3.77 (s, 3H), 4.10 (s, 3H), 7.71 (d, $J = 5.5$ Hz, 1H), 7.75 (d, $J = 5.2$ Hz, 1H); ^{13}C NMR (125 MHz, CDCl_3): δ 15.1 (d, $J = 1.9$ Hz), 52.4 (d, $J = 5.4$ Hz), 64.0, 98.3 (d, $J = 201.8$ Hz), 126.2, 128.9 (d, $J = 16.6$ Hz), 133.6, 143.3 (d, $J = 9.7$ Hz), 150.5 (d, $J = 22.4$ Hz), 154.7; HRMS (ESI+): calcd. for $\text{C}_{11}\text{H}_{15}\text{O}_5\text{NPS} [\text{M}+\text{H}]^+$: 304.0403, found: 304.0402.



Dimethyl (1-methoxy-2,5-dimethyl-6-oxo-1,6-dihdropyridin-3-yl)phosphonate (**5o**)

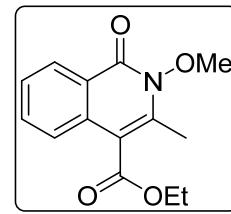
Following the general procedure compound **5o** was obtained in 80% yield (31.5 mg) as light yellow oil; R_f 0.3 (40% ethyl acetate/pet. ether); FT-IR: $\bar{\nu}_{\text{max}}$ 3468,



2923, 1660, 1393, 1182, 1049, 1016, 825, 756, 636 cm^{-1} . ^1H NMR (500 MHz, CDCl_3): δ 2.15 (s, 3H), 2.66 (s, 3H), 3.76 (s, 3H), 3.79 (s, 3H), 4.06 (s, 3H), 7.46 (d, $J = 11.1$ Hz, 1H); ^{13}C NMR (125 MHz, CDCl_3): δ 15.3 (d, $J = 2.9$ Hz), 16.7, 52.8 (d, $J = 5.5$ Hz), 63.6, 100.2 (d, $J = 207.4$ Hz), 128.1 (d, $J = 14.8$ Hz), 137.6 (d, $J = 8.2$ Hz), 150.8 (d, $J = 20.7$ Hz), 159.3; HRMS (ESI+): calcd. for $\text{C}_{10}\text{H}_{17}\text{O}_5\text{NP} [\text{M}+\text{H}]^+$: 262.0839, found: 262.0837.

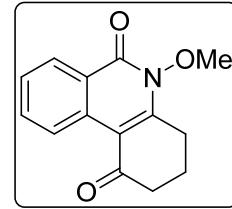
Ethyl 2-methoxy-3-methyl-1-oxo-1,2-dihydroisoquinoline-4-carboxylate (**5p**)⁵

Following the general procedure compound **5p** was obtained in 60% yield (23.6 mg) as light yellow oil; R_f 0.4 (40% ethyl acetate/pet. ether); FT-IR: $\bar{\nu}_{\text{max}}$ 2881, 2838, 1715, 1663, 1488, 1287, 1116, 1047, 988, 782, 693 cm^{-1} . ^1H NMR (400 MHz, CDCl_3): δ 1.44 (t, $J = 7.3$ Hz, 3H), 2.54–2.57 (m, 3H), 4.09 (s, 3H), 4.47 (q, $J = 6.9$ Hz, 2H), 7.48 (ddd, $J = 7.9, 5.2, 3.0$ Hz, 1H), 7.65–7.71 (m, 2H), 8.41–8.46 (m, 1H); ^{13}C NMR (100 MHz, CDCl_3): δ 14.2, 14.9, 61.7, 63.8, 109.6, 123.8, 125.3, 126.6, 127.8, 132.8, 133.1, 140.7, 158.2, 166.8; HRMS (ESI+): calcd. for $\text{C}_{14}\text{H}_{16}\text{O}_4\text{N} [\text{M}+\text{H}]^+$: 262.1074, found: 262.1071.



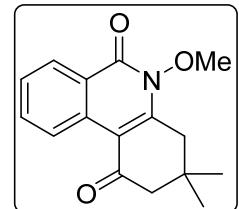
5-Methoxy-3,4-dihydrophenanthridine-1,6(2H,5H)-dione (**5q**)

Following the general procedure compound **5q** was obtained in 58% yield (21.2 mg) as white solid; R_f 0.3 (40% ethyl acetate/pet. ether); mp: 130–134 °C; FT-IR: $\bar{\nu}_{\text{max}}$ 3681, 2844, 1680, 1642, 1388, 1183, 1033, 977, 771, 693 cm^{-1} . ^1H NMR (500 MHz, CDCl_3): δ 2.18–2.23 (m, 2H), 2.63–2.67 (m, 2H), 3.17 (t, $J = 6.1$ Hz, 2H), 4.12 (s, 3H), 7.47–7.52 (m, 1H), 7.71–7.75 (m, 1H), 8.41 (dd, $J = 8.0, 1.1$ Hz, 1H), 9.24 (d, $J = 8.4$ Hz, 1H); ^{13}C NMR (125 MHz, CDCl_3): δ 20.6, 24.8, 38.8, 64.1, 109.8, 125.0, 126.3, 127.1, 127.3, 133.3, 133.7, 153.1, 158.5, 196.1; HRMS (ESI+): calcd. for $\text{C}_{14}\text{H}_{14}\text{O}_3\text{N} [\text{M}+\text{H}]^+$: 244.0968, found: 244.0968.



5-Methoxy-3,3-dimethyl-3,4-dihydrophenanthridine-1,6(2H,5H)-dione (5r)⁵

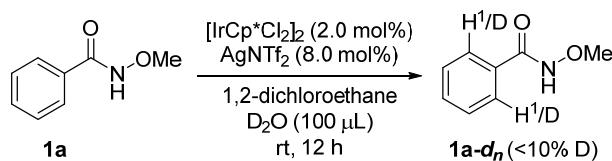
Following the general procedure compound **5r** was obtained in 64% yield (26.0 mg) as light yellow solid; R_f 0.4 (50% ethyl acetate/pet. ether); mp: 126-128 °C; FT-IR: $\bar{\nu}_{\text{max}}$ 3681, 2873, 1686, 1655, 1374, 1105, 1033, 923, 719, 695 cm⁻¹. ¹H NMR (500 MHz, CDCl₃): δ 1.20 (s, 6H), 2.53 (s, 2H), 3.03 (s, 2H), 4.11 (s, 3H), 7.51 (td, J = 7.6, 1.1 Hz, 1H), 7.75 (ddd, J = 8.6, 7.1, 1.6 Hz, 1H), 8.43 (dd, J = 8.1, 1.1 Hz, 1H), 9.28 (d, J = 8.5 Hz, 1H); ¹³C NMR (100 MHz, CDCl₃): δ 28.2, 31.8, 38.3, 52.5, 64.1, 108.8, 125.0, 126.2, 127.1, 127.4, 133.1, 133.7, 151.4, 158.7, 196.3; HRMS (ESI+): calcd. for C₁₆H₁₈O₃N [M+H]⁺: 272.1281, found: 272.1280.



5. Experimental Procedures of the Mechanistic Studies

6.1. Deuterium Exchange Study in absence of Diazo compound

To a screw capped vial with a spinvane triangular-shaped Teflon stirbar were added N-methoxybenzamide (**1a**, 22.7 mg, 0.15 mmol), $[\text{IrCp}^*\text{Cl}_2]_2$ (2.4 mg, 2.0 mol %), AgNTf_2 (4.7 mg, 8.0 mol %), 1,2-dichloroethane (1.0 mL) and D_2O (100 μL) under air. The reaction mixture was stirred at room temperature for 12 h. Next, the reaction mixture was filtered through a pad of celite and then washed with CH_2Cl_2 (5 mL x 3). Solvents were removed under reduced pressure and the compound was analyzed by ^1H NMR analysis.



Acquisition Time (sec)	3.9584	Comment	Ravindra	Date	19 Jan 2016 16:25:44
Date Stamp	19 Jan 2016 16:25:44				
File Name	\172.16.2.4\nmr_data\AV200\2016#\AV200JAN_16#\AV200\data\Administrator\nmr\tue4av2#041\1\PDATA\1\1r				
Frequency (MHz)	200.13	Nucleus	^1H	Number of Transients	4
Original Points Count	16384	Owner	Administrator	Points Count	32768
Receiver Gain	512.00	Solvent	CHLOROFORM-d	Pulse Sequence	zg30
Spectrum Offset (Hz)	1229.4108	Spectrum Type	STANDARD	Sweep Width (Hz)	4138.95
				Temperature (degree C)	27.000

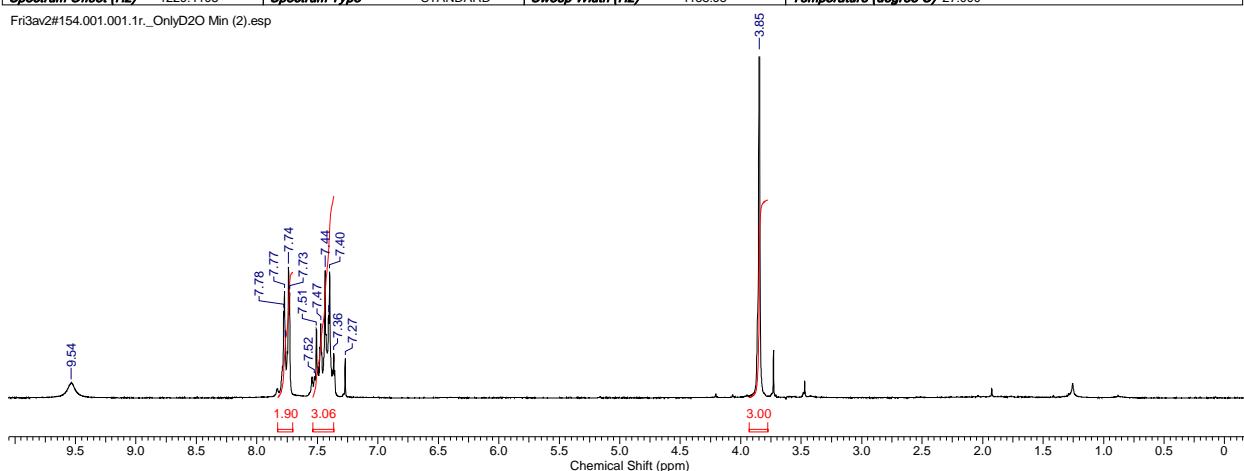
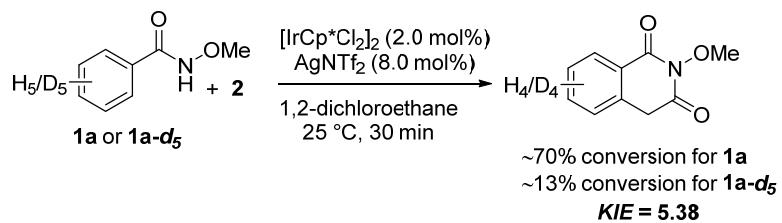


Figure S1. ^1H NMR spectra of **1a-d_n** in CDCl_3

6.2. Study of Kinetic Isotop Effect

6.2.1. Parallel experiments



N-methoxybenzamide (**1a**, 15.2 mg, 0.1 mmol) or N-methoxybenzamide-*d*₅ (**1a-d**₅, 15.7 mg, 0.1 mmol) were added to two separate screw capped vials with spinvane triangular-shaped Teflon stirbar, along with diazo compound **2** (18.8 mg, 0.11 mmol), [IrCp*Cl₂]₂ (1.6 mg, 2.0 mol %), AgNTf₂ (3.1 mg, 8.0 mol %) and 1,2-dichloroethane (0.75 mL) under atmospheric conditions. Each of the reaction mixture was stirred at 25 °C for 30 min. Then both the reaction mixture was separately filtered through a pad of celite and the celite pad was washed with EtOAc (5 mL x 3). The solvent was removed under reduced pressure to afford the crude product **3a** and **3a-d**₄. From the ¹H NMR analysis the ratio of **3a** and **3a-d**₄ found to be 1.43:1.

Note: Since the reaction is too fast and the conversion is high (70% for 3a), we have not included this results in main text.

6.2.2. Competitive experiments

N-methoxybenzamide (**1a**, 15.2 mg, 0.1 mmol) and *N*-methoxybenzamide-*d*₅ (**1a-d**₅, 15.7 mg, 0.1 mmol) were added to screw capped vials with spinvane triangular-shaped Teflon stirbar, along with diazo compound **2** (18.8 mg, 0.11 mmol), [IrCp*Cl₂]₂ (1.6 mg, 2.0 mol %), AgNTf₂ (3.1 mg, 8.0 mol %) and 1,2-dichloroethane (0.75 mL) under atmospheric conditions. The reaction mixture was stirred at 25 °C for 10 min and then filtered through a pad of celite and the celite pad was washed with EtOAc (5 mL x 3). The solvent was removed under reduced pressure and the residue was purify by column chromatography (methanol/dichloromethana) to afford mixture of **3a** and **3a-d**₄. From the ¹H NMR analysis the ratio of **3a** and **3a-d**₄ found to be 4:1.

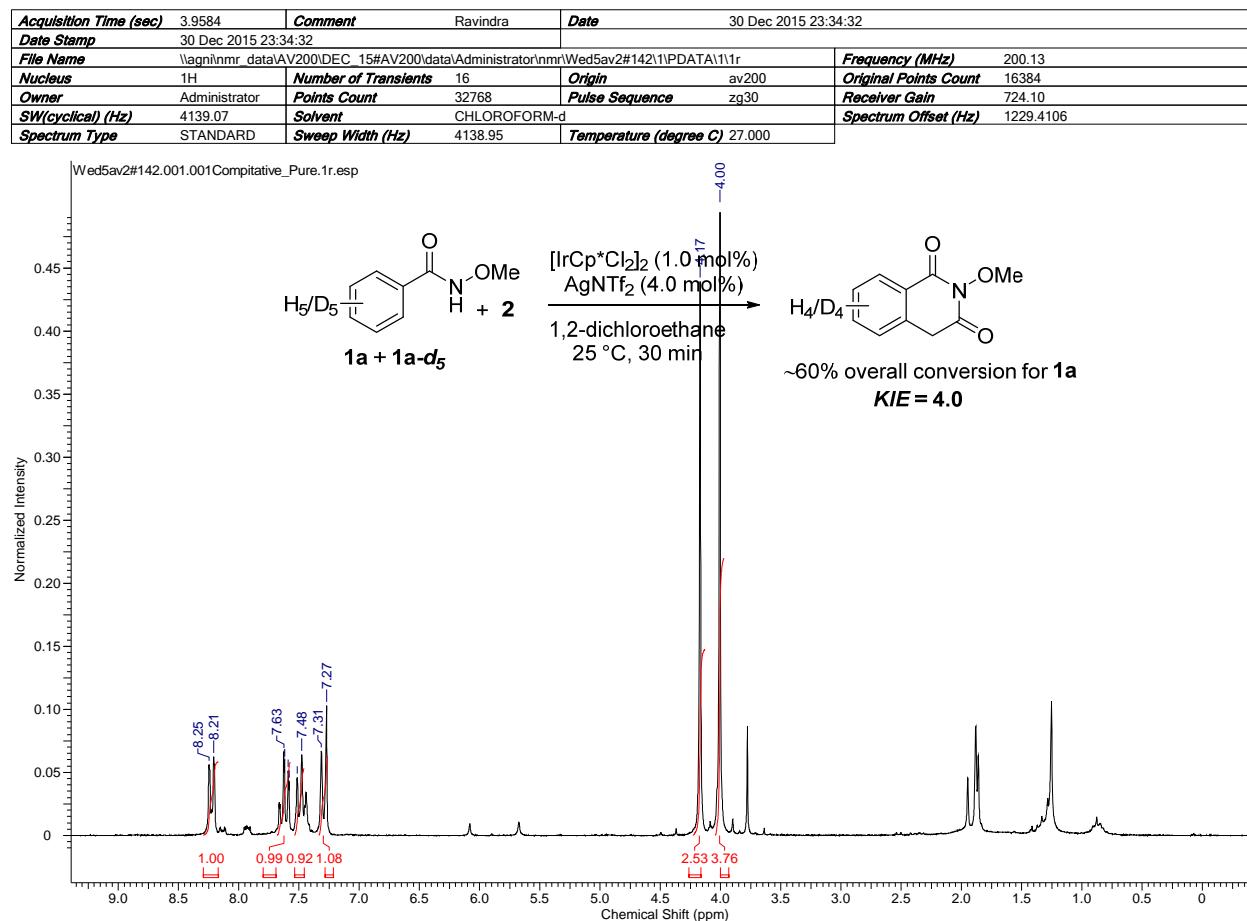


Figure S2. ¹H NMR spectra of **3a-d**₅ and **3a-d**₄ in CDCl₃.

References:

1. Fukui, Y.; Liu, P.; Liu, Q.; He, Z.-T.; Wu, N.-Y.; Tian, P.; Lin, G.-Q. *J. Am. Chem. Soc.* **2014**, *136*, 15607.
2. Meffre, P.; Hermann, S.; Durand, P.; Reginato, G.; Riu, A. *Tetrahedron* **2002**, *58*, 5159–5162.
3. Koskinen, A. M. P.; Muñoz, L. *J. Chem. Soc., Chem. Commun.* **1990**, 652–653
4. Shi, J.; Zhou, J.; Yan, Y.; Jia, J.; Liu, X.; Song, H.; Xu, H. E.; Yi, W. *Chem. Commun.* **2015**, *51*, 668.
5. Shi, L.; Yu, K.; Wang, B. *Chem. Commun.* **2015**, *51*, 17277.

Appendix 1

X-Ray Crystallographic Information

7.1. General information

Single-crystal X-ray data of **4c**, **6** and **18** were collected on a Bruker KAPPA APEX II CCD Duo diffractometer, using graphite-monochromated Mo K α radiation ($\lambda = 0.71073 \text{ \AA}$). The X-ray generator was operated at 50 kV and 30 mA. A preliminary set of cell constants and an orientation matrix were calculated from total 36 frames. The X-ray data acquisition was monitored by APEX 2 program suit. All the data were corrected for Lorentz-polarization and absorption effects using SAINT and SADABS programs integrated in APEX 2 package. The structures were solved by direct methods and refined by full matrix least squares, based on F^2 , using SHELX-97. Molecular diagrams were generated using Mercury programs. Geometrical calculations were performed using SHELXTL and PLATON. The H atoms for the **4c**, **6** and **18** were placed in geometrically idealized positions (C-H = 0.93 and C-H = 0.97 \AA for the phenyl and methylene H atoms) and constrained to ride on their parent atoms [Uiso(H) = 1.2 Ueq(C)].

7.2. X-Ray data for compound **3i**

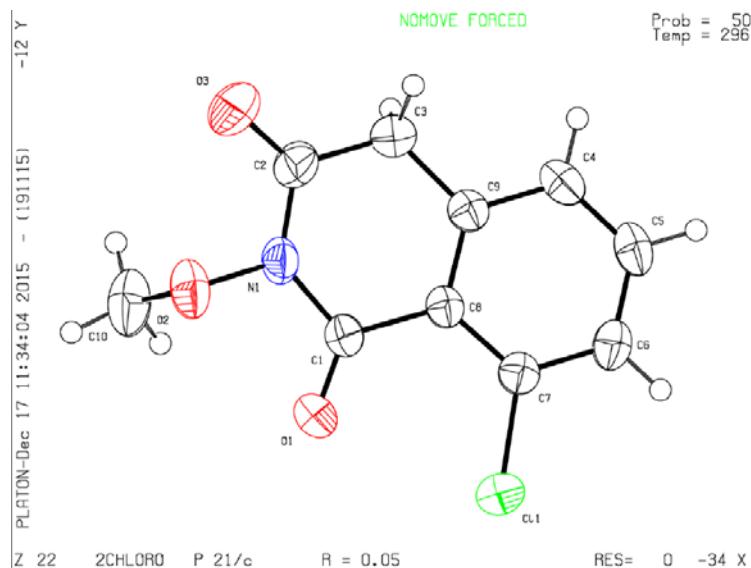


Figure S3. ORTEP structure of compound **5i**

Details data:

Table S2. Crystal data and structure refinement for **3i**.

Identification code	2CHLORO
Empirical formula	C10 H8 Cl N O3
Formula weight	225.62
Temperature	296(2) K
Wavelength	0.71073 Å
Crystal system, space group	Monoclinic, P 21/c
Unit cell dimensions	a = 9.2286(6) Å alpha = 90 deg. b = 12.6651(9) Å beta = 96.240(3) deg. c = 8.3177(6) Å gamma = 90 deg.
Volume	966.42(12) Å^3
Z, Calculated density	4, 1.551 Mg/m^3
Absorption coefficient	0.379 mm^-1
F(000)	464
Crystal size	0.498 x 0.325 x 0.124 mm
Theta range for data collection	2.742 to 28.345 deg.
Limiting indices	-12<=h<=12, -16<=k<=16, -11<=l<=11
Reflections collected / unique	30222 / 2406 [R(int) = 0.0507]
Completeness to theta = 25.242	99.9 %
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	2406 / 0 / 137
Goodness-of-fit on F^2	1.024
Final R indices [I>2sigma(I)]	R1 = 0.0511, wR2 = 0.1419
R indices (all data)	R1 = 0.0697, wR2 = 0.1560
Extinction coefficient	n/a
Largest diff. peak and hole	0.550 and -0.239 e.Å^-3

Table S3. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters (Å^2 x 10^3) for **3i**.

U(eq) is defined as one third of the trace of the orthogonalized Uij tensor.

	x	y	z	U(eq)
Cl(1)	6498(1)	2546(1)	10670(1)	54(1)
O(1)	3423(2)	2352(2)	10086(3)	69(1)
O(2)	791(2)	3058(1)	9511(2)	52(1)
O(3)	261(2)	4609(2)	7432(3)	92(1)

N(1)	1904(2)	3527(2)	8769(2)	40(1)
C(1)	3301(2)	3136(2)	9265(3)	38(1)
C(2)	1508(2)	4348(2)	7722(3)	48(1)
C(3)	2719(2)	4854(2)	6967(3)	46(1)
C(4)	5379(3)	5204(2)	7179(3)	43(1)
C(5)	6796(2)	4980(2)	7765(3)	46(1)
C(6)	7110(2)	4155(2)	8830(3)	41(1)
C(7)	5988(2)	3554(2)	9319(2)	35(1)
C(8)	4528(2)	3759(2)	8744(2)	32(1)
C(9)	4241(2)	4596(2)	7657(2)	35(1)
C(10)	179(3)	2172(2)	8594(4)	66(1)

Table S4. Bond lengths [Å] and angles [deg] for **5i**.

Cl(1)-C(7)	1.732(2)
O(1)-C(1)	1.204(3)
O(2)-N(1)	1.388(2)
O(2)-C(10)	1.437(3)
O(3)-C(2)	1.196(3)
N(1)-C(2)	1.380(3)
N(1)-C(1)	1.400(3)
C(1)-C(8)	1.483(3)
C(2)-C(3)	1.485(3)
C(3)-C(9)	1.495(3)
C(3)-H(3A)	0.9700
C(3)-H(3B)	0.9700
C(4)-C(5)	1.375(3)
C(4)-C(9)	1.395(3)
C(4)-H(4)	0.9300
C(5)-C(6)	1.380(3)
C(5)-H(5)	0.9300
C(6)-C(7)	1.381(3)
C(6)-H(6)	0.9300
C(7)-C(8)	1.405(3)
C(8)-C(9)	1.399(3)
C(10)-H(10A)	0.9600
C(10)-H(10B)	0.9600
C(10)-H(10C)	0.9600
N(1)-O(2)-C(10)	111.39(18)

C(2)-N(1)-O(2)	116.42(17)
C(2)-N(1)-C(1)	128.65(18)
O(2)-N(1)-C(1)	114.81(17)
O(1)-C(1)-N(1)	119.06(19)
O(1)-C(1)-C(8)	125.3(2)
N(1)-C(1)-C(8)	115.67(18)
O(3)-C(2)-N(1)	121.2(2)
O(3)-C(2)-C(3)	123.2(2)
N(1)-C(2)-C(3)	115.61(18)
C(2)-C(3)-C(9)	117.52(19)
C(2)-C(3)-H(3A)	107.9
C(9)-C(3)-H(3A)	107.9
C(2)-C(3)-H(3B)	107.9
C(9)-C(3)-H(3B)	107.9
H(3A)-C(3)-H(3B)	107.2
C(5)-C(4)-C(9)	120.1(2)
C(5)-C(4)-H(4)	120.0
C(9)-C(4)-H(4)	120.0
C(4)-C(5)-C(6)	120.6(2)
C(4)-C(5)-H(5)	119.7
C(6)-C(5)-H(5)	119.7
C(7)-C(6)-C(5)	119.7(2)
C(7)-C(6)-H(6)	120.2
C(5)-C(6)-H(6)	120.2
C(6)-C(7)-C(8)	121.31(19)
C(6)-C(7)-Cl(1)	115.95(16)
C(8)-C(7)-Cl(1)	122.74(16)
C(9)-C(8)-C(7)	117.86(18)
C(9)-C(8)-C(1)	119.69(18)
C(7)-C(8)-C(1)	122.45(18)
C(8)-C(9)-C(4)	120.5(2)
C(8)-C(9)-C(3)	121.15(18)
C(4)-C(9)-C(3)	118.34(19)
O(2)-C(10)-H(10A)	109.5
O(2)-C(10)-H(10B)	109.5
H(10A)-C(10)-H(10B)	109.5
O(2)-C(10)-H(10C)	109.5
H(10A)-C(10)-H(10C)	109.5
H(10B)-C(10)-H(10C)	109.5

Table S5. Anisotropic displacement parameters ($\text{A}^2 \times 10^3$) for **3i**.

The anisotropic displacement factor exponent takes the form:

$$-2 \pi^2 [h^2 a^*{}^2 U_{11} + \dots + 2 h k a^* b^* U_{12}]$$

	U11	U22	U33	U23	U13	U12
Cl(1)	45(1)	52(1)	63(1)	16(1)	-5(1)	3(1)
O(1)	47(1)	58(1)	98(2)	39(1)	-4(1)	-12(1)
O(2)	42(1)	53(1)	65(1)	-8(1)	21(1)	-11(1)
O(3)	37(1)	99(2)	141(2)	46(2)	18(1)	21(1)
N(1)	31(1)	41(1)	50(1)	1(1)	11(1)	-4(1)
C(1)	35(1)	34(1)	43(1)	2(1)	2(1)	-5(1)
C(2)	36(1)	45(1)	64(1)	2(1)	7(1)	7(1)
C(3)	42(1)	42(1)	53(1)	7(1)	3(1)	4(1)
C(4)	52(1)	35(1)	45(1)	4(1)	16(1)	-3(1)
C(5)	43(1)	45(1)	53(1)	-2(1)	19(1)	-11(1)
C(6)	31(1)	46(1)	47(1)	-7(1)	8(1)	-2(1)
C(7)	36(1)	32(1)	37(1)	-4(1)	5(1)	0(1)
C(8)	33(1)	28(1)	35(1)	-5(1)	8(1)	-2(1)
C(9)	38(1)	31(1)	37(1)	-3(1)	7(1)	0(1)
C(10)	46(1)	64(2)	87(2)	-18(2)	12(1)	-17(1)

Table S6. Torsion angles [deg] for **3i**.

C(10)-O(2)-N(1)-C(2)	92.1(3)
C(10)-O(2)-N(1)-C(1)	-91.6(2)
C(2)-N(1)-C(1)-O(1)	-173.8(2)
O(2)-N(1)-C(1)-O(1)	10.3(3)
C(2)-N(1)-C(1)-C(8)	7.5(3)
O(2)-N(1)-C(1)-C(8)	-168.32(17)
O(2)-N(1)-C(2)-O(3)	-2.8(4)
C(1)-N(1)-C(2)-O(3)	-178.5(3)
O(2)-N(1)-C(2)-C(3)	178.73(19)
C(1)-N(1)-C(2)-C(3)	3.0(4)
O(3)-C(2)-C(3)-C(9)	168.9(3)
N(1)-C(2)-C(3)-C(9)	-12.7(3)
C(9)-C(4)-C(5)-C(6)	-0.1(3)
C(4)-C(5)-C(6)-C(7)	-0.4(3)
C(5)-C(6)-C(7)-C(8)	0.4(3)
C(5)-C(6)-C(7)-Cl(1)	-179.45(17)

C(6)-C(7)-C(8)-C(9)	0.0(3)
Cl(1)-C(7)-C(8)-C(9)	179.86(14)
C(6)-C(7)-C(8)-C(1)	-179.14(19)
Cl(1)-C(7)-C(8)-C(1)	0.7(3)
O(1)-C(1)-C(8)-C(9)	173.6(2)
N(1)-C(1)-C(8)-C(9)	-7.8(3)
O(1)-C(1)-C(8)-C(7)	-7.2(3)
N(1)-C(1)-C(8)-C(7)	171.34(18)
C(7)-C(8)-C(9)-C(4)	-0.5(3)
C(1)-C(8)-C(9)-C(4)	178.69(19)
C(7)-C(8)-C(9)-C(3)	178.94(18)
C(1)-C(8)-C(9)-C(3)	-1.9(3)
C(5)-C(4)-C(9)-C(8)	0.5(3)
C(5)-C(4)-C(9)-C(3)	-178.9(2)
C(2)-C(3)-C(9)-C(8)	12.4(3)
C(2)-C(3)-C(9)-C(4)	-168.1(2)

7.3.1. X-ray data of compound 5c

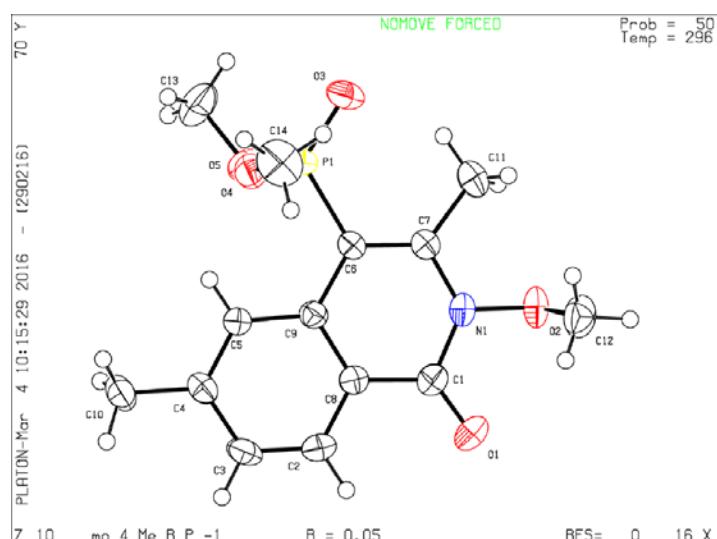


Figure S4. ORTEP diadram of compound 5c.

Table S7. Crystal data and structure refinement for 5c.

Identification code	mo_4_Me_R_0m
Empirical formula	C15 H18 O5 P
Formula weight	309.26

Temperature	296(2) K	
Wavelength	0.71073 Å	
Crystal system	Triclinic	
Space group	P-1	
Unit cell dimensions	a = 8.0151(6) Å b = 9.9592(8) Å c = 10.3467(8) Å	$\alpha = 69.516(4)^\circ$. $\beta = 72.945(4)^\circ$. $\gamma = 89.454(4)^\circ$.
Volume	735.66(10) Å ³	
Z	2	
Density (calculated)	1.396 Mg/m ³	
Absorption coefficient	0.206 mm ⁻¹	
F(000)	326	
Crystal size	? x ? x ? mm ³	
Theta range for data collection	2.893 to 28.459°.	
Index ranges	-10<=h<=10, -13<=k<=13, -13<=l<=13	
Reflections collected	18852	
Independent reflections	3680 [R(int) = 0.0519]	
Completeness to theta = 25.242°	99.8 %	
Refinement method	Full-matrix least-squares on F ²	
Data / restraints / parameters	3680 / 0 / 195	
Goodness-of-fit on F ²	1.220	
Final R indices [I>2sigma(I)]	R1 = 0.0624, wR2 = 0.1707	
R indices (all data)	R1 = 0.1036, wR2 = 0.1925	
Extinction coefficient	n/a	
Largest diff. peak and hole	0.672 and -0.377 e.Å ⁻³	

Table S8. (x 10⁴) and equivalent isotropic displacement parameters (Å² x 10³) for **5c**. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
P(1)	5988(1)	7498(1)	2424(1)	35(1)
O(1)	9151(3)	6234(2)	-2872(2)	58(1)
O(2)	9130(2)	8903(2)	-3012(2)	44(1)
O(3)	6343(3)	8984(2)	2287(2)	56(1)
O(4)	6614(2)	6382(2)	3657(2)	41(1)
O(5)	3996(2)	6952(2)	2907(2)	45(1)

C(1)	8459(3)	6411(3)	-1742(3)	39(1)
C(2)	8441(3)	7785(2)	-1682(2)	24(1)
C(3)	7694(3)	8154(2)	-486(3)	37(1)
C(4)	6893(3)	7085(2)	816(3)	32(1)
C(5)	5851(3)	4429(2)	2166(2)	32(1)
C(6)	5705(3)	3033(2)	2195(3)	34(1)
C(7)	6529(4)	2760(3)	930(3)	43(1)
C(8)	7418(4)	3866(3)	-322(3)	43(1)
C(9)	7552(3)	5276(2)	-351(3)	34(1)
C(10)	6770(3)	5587(2)	903(2)	30(1)
C(11)	11019(4)	9125(3)	-3400(3)	53(1)
C(12)	7799(5)	9756(3)	-814(3)	67(1)
C(13)	8364(4)	6587(4)	3710(4)	66(1)
C(14)	2790(4)	7171(4)	4136(3)	60(1)
C(15)	4662(4)	1831(3)	3552(3)	44(1)

Table S9. Bond lengths [Å] and angles [°] for **5c**.

P(1)-O(3)	1.4589(19)
P(1)-O(5)	1.5663(19)
P(1)-O(4)	1.5769(19)
P(1)-C(4)	1.797(2)
O(1)-C(1)	1.212(3)
O(2)-C(2)	1.390(2)
O(2)-C(11)	1.446(3)
O(4)-C(13)	1.439(3)
O(5)-C(14)	1.438(3)
C(1)-C(2)	1.391(3)
C(1)-C(9)	1.463(3)
C(2)-C(3)	1.380(3)
C(3)-C(4)	1.365(3)
C(3)-C(12)	1.508(3)
C(4)-C(10)	1.465(3)
C(5)-C(6)	1.386(3)
C(5)-C(10)	1.406(3)
C(5)-H(5)	0.9300

C(6)-C(7)	1.400(4)
C(6)-C(15)	1.503(3)
C(7)-C(8)	1.369(4)
C(7)-H(7)	0.9300
C(8)-C(9)	1.398(3)
C(8)-H(8)	0.9300
C(9)-C(10)	1.405(3)
C(11)-H(11A)	0.9600
C(11)-H(11B)	0.9600
C(11)-H(11C)	0.9600
C(12)-H(12A)	0.9600
C(12)-H(12B)	0.9600
C(12)-H(12C)	0.9600
C(13)-H(13A)	0.9600
C(13)-H(13B)	0.9600
C(13)-H(13C)	0.9600
C(14)-H(14A)	0.9600
C(14)-H(14B)	0.9600
C(14)-H(14C)	0.9600
C(15)-H(15A)	0.9600
C(15)-H(15B)	0.9600
C(15)-H(15C)	0.9600

O(3)-P(1)-O(5)	114.83(11)
O(3)-P(1)-O(4)	112.40(11)
O(5)-P(1)-O(4)	101.85(10)
O(3)-P(1)-C(4)	116.44(11)
O(5)-P(1)-C(4)	103.00(11)
O(4)-P(1)-C(4)	106.86(10)
C(2)-O(2)-C(11)	110.79(19)
C(13)-O(4)-P(1)	120.05(18)
C(14)-O(5)-P(1)	119.83(18)
O(1)-C(1)-C(2)	120.8(2)
O(1)-C(1)-C(9)	125.7(2)
C(2)-C(1)-C(9)	113.5(2)
C(3)-C(2)-O(2)	117.4(2)

C(3)-C(2)-C(1)	127.3(2)
O(2)-C(2)-C(1)	115.1(2)
C(4)-C(3)-C(2)	118.8(2)
C(4)-C(3)-C(12)	127.2(2)
C(2)-C(3)-C(12)	113.9(2)
C(3)-C(4)-C(10)	119.7(2)
C(3)-C(4)-P(1)	120.57(18)
C(10)-C(4)-P(1)	119.71(17)
C(6)-C(5)-C(10)	122.3(2)
C(6)-C(5)-H(5)	118.9
C(10)-C(5)-H(5)	118.9
C(5)-C(6)-C(7)	118.9(2)
C(5)-C(6)-C(15)	120.8(2)
C(7)-C(6)-C(15)	120.3(2)
C(8)-C(7)-C(6)	120.2(2)
C(8)-C(7)-H(7)	119.9
C(6)-C(7)-H(7)	119.9
C(7)-C(8)-C(9)	120.7(2)
C(7)-C(8)-H(8)	119.6
C(9)-C(8)-H(8)	119.6
C(8)-C(9)-C(10)	120.6(2)
C(8)-C(9)-C(1)	117.7(2)
C(10)-C(9)-C(1)	121.7(2)
C(9)-C(10)-C(5)	117.2(2)
C(9)-C(10)-C(4)	118.9(2)
C(5)-C(10)-C(4)	123.8(2)
O(2)-C(11)-H(11A)	109.5
O(2)-C(11)-H(11B)	109.5
H(11A)-C(11)-H(11B)	109.5
O(2)-C(11)-H(11C)	109.5
H(11A)-C(11)-H(11C)	109.5
H(11B)-C(11)-H(11C)	109.5
C(3)-C(12)-H(12A)	109.5
C(3)-C(12)-H(12B)	109.5
H(12A)-C(12)-H(12B)	109.5
C(3)-C(12)-H(12C)	109.5

H(12A)-C(12)-H(12C)	109.5
H(12B)-C(12)-H(12C)	109.5
O(4)-C(13)-H(13A)	109.5
O(4)-C(13)-H(13B)	109.5
H(13A)-C(13)-H(13B)	109.5
O(4)-C(13)-H(13C)	109.5
H(13A)-C(13)-H(13C)	109.5
H(13B)-C(13)-H(13C)	109.5
O(5)-C(14)-H(14A)	109.5
O(5)-C(14)-H(14B)	109.5
H(14A)-C(14)-H(14B)	109.5
O(5)-C(14)-H(14C)	109.5
H(14A)-C(14)-H(14C)	109.5
H(14B)-C(14)-H(14C)	109.5
C(6)-C(15)-H(15A)	109.5
C(6)-C(15)-H(15B)	109.5
H(15A)-C(15)-H(15B)	109.5
C(6)-C(15)-H(15C)	109.5
H(15A)-C(15)-H(15C)	109.5
H(15B)-C(15)-H(15C)	109.5

Symmetry transformations used to generate equivalent atoms:

Table S10. ($\text{\AA}^2 \times 10^3$) for **5c**. The anisotropic displacement factor exponent takes the form: $-2\pi^2 [h^2 a^{*2} U^{11} + \dots + 2 h k a^{*} b^{*} U^{12}]$

	U ¹¹	U ²²	U ³³	U ²³	U ¹³	U ¹²
P(1)	45(1)	28(1)	34(1)	-15(1)	-8(1)	2(1)
O(1)	76(2)	58(1)	31(1)	-21(1)	3(1)	-1(1)
O(2)	42(1)	42(1)	31(1)	2(1)	-6(1)	-6(1)
O(3)	88(2)	34(1)	50(1)	-23(1)	-15(1)	3(1)
O(4)	47(1)	40(1)	37(1)	-12(1)	-14(1)	-2(1)
O(5)	41(1)	55(1)	44(1)	-27(1)	-8(1)	7(1)
C(1)	40(1)	43(1)	31(1)	-14(1)	-6(1)	1(1)
C(2)	27(1)	21(1)	15(1)	-1(1)	-1(1)	-3(1)
C(3)	42(1)	28(1)	36(1)	-10(1)	-7(1)	1(1)
C(4)	40(1)	24(1)	30(1)	-9(1)	-8(1)	2(1)

C(5)	39(1)	26(1)	28(1)	-10(1)	-6(1)	1(1)
C(6)	38(1)	26(1)	37(1)	-9(1)	-14(1)	2(1)
C(7)	58(2)	26(1)	48(2)	-19(1)	-16(1)	3(1)
C(8)	57(2)	40(1)	36(1)	-22(1)	-9(1)	6(1)
C(9)	40(1)	31(1)	30(1)	-12(1)	-9(1)	4(1)
C(10)	37(1)	26(1)	28(1)	-11(1)	-10(1)	5(1)
C(11)	42(2)	60(2)	44(2)	-12(1)	-2(1)	-11(1)
C(12)	101(3)	28(1)	45(2)	-4(1)	3(2)	-2(2)
C(13)	54(2)	67(2)	83(2)	-26(2)	-31(2)	7(2)
C(14)	52(2)	76(2)	41(2)	-19(2)	-1(1)	13(2)
C(15)	50(2)	29(1)	45(2)	-7(1)	-14(1)	-4(1)

Table S11. displacement parameters ($\text{\AA}^2 \times 10^{-3}$) for **5c**.

	x	y	z	U(eq)
H(5)	5322	4606	3011	38
H(7)	6473	1824	941	51
H(8)	7939	3677	-1162	52
H(11A)	11330	9458	-2728	80
H(11B)	11479	9831	-4363	80
H(11C)	11503	8233	-3368	80
H(12A)	7128	9939	32	100
H(12B)	7335	10219	-1598	100
H(12C)	9001	10131	-1083	100
H(13A)	9146	6136	3126	99
H(13B)	8376	6161	4695	99
H(13C)	8738	7599	3344	99
H(14A)	3163	6727	4977	91
H(14B)	1638	6746	4309	91
H(14C)	2765	8185	3944	91
H(15A)	5011	1851	4358	66
H(15B)	4874	923	3435	66
H(15C)	3435	1949	3731	66

7.3. X-ray data of compound 5n

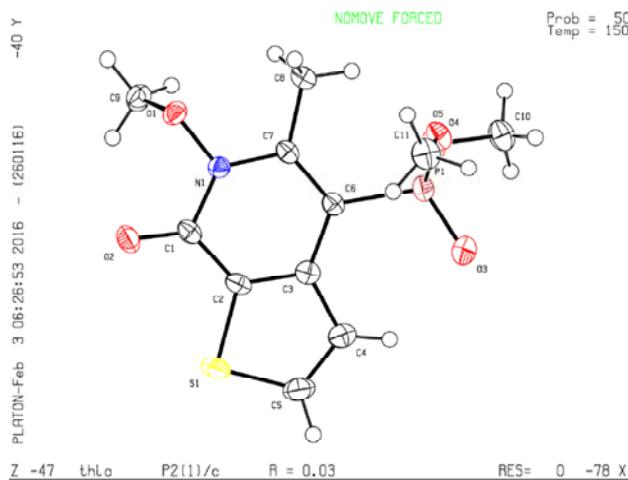


Figure S5. ORTEP diagram of compound 5n

Table S12. Crystal data and structure refinement for **5n**.

Identification code	5n	
Empirical formula	C11 H14 N O5 P S	
Formula weight	303.26	
Temperature	150(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	P2(1)/c	
Unit cell dimensions	a = 9.0499(5) Å	α= 90°.
	b = 9.3721(6) Å	β= 105.5360(10)°.
	c = 16.1145(10) Å	γ = 90°.
Volume	1316.84(14) Å ³	
Z	4	
Density (calculated)	1.530 Mg/m ³	
Absorption coefficient	0.383 mm ⁻¹	
F(000)	632	
Crystal size	0.35 x 0.25 x 0.15 mm ³	
Theta range for data collection	2.34 to 24.98°.	
Index ranges	-10<=h<=10, -11<=k<=11, -19<=l<=18	
Reflections collected	11778	
Independent reflections	2318 [R(int) = 0.0207]	
Completeness to theta = 24.98°	100.0 %	

Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	0.9449 and 0.8777
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	2318 / 0 / 176
Goodness-of-fit on F^2	0.975
Final R indices [$I > 2\sigma(I)$]	$R_1 = 0.0295$, $wR_2 = 0.0753$
R indices (all data)	$R_1 = 0.0312$, $wR_2 = 0.0765$
Largest diff. peak and hole	0.380 and -0.289 e. \AA^{-3}

Table S13. Atomic coordinates ($\times 10^4$) and equivalent isotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for **5n**. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
S(1)	5297(1)	4388(1)	7845(1)	34(1)
P(1)	10213(1)	7875(1)	8671(1)	23(1)
O(1)	6514(1)	7788(1)	5638(1)	23(1)
O(2)	4630(1)	5949(1)	6077(1)	29(1)
O(3)	10291(2)	7378(2)	9542(1)	33(1)
O(4)	11642(1)	7486(1)	8331(1)	30(1)
O(5)	10159(1)	9553(1)	8565(1)	28(1)
N(1)	6903(2)	7184(1)	6461(1)	20(1)
C(1)	5822(2)	6238(2)	6628(1)	21(1)
C(2)	6314(2)	5683(2)	7481(1)	23(1)
C(3)	7646(2)	6090(2)	8091(1)	21(1)
C(4)	7834(2)	5279(2)	8870(1)	28(1)
C(5)	6674(2)	4340(2)	8814(1)	36(1)
C(6)	8611(2)	7168(2)	7865(1)	20(1)
C(7)	8229(2)	7675(2)	7033(1)	20(1)
C(8)	9115(2)	8740(2)	6669(1)	27(1)
C(9)	6881(2)	6801(2)	5035(1)	28(1)
C(10)	13175(2)	7557(2)	8904(1)	36(1)
C(11)	8864(2)	10342(2)	8704(1)	33(1)

Table 14. Bond lengths [\AA] and angles [$^\circ$] for **5n**.

S(1)-C(5) 1.718(2)

S(1)-C(2)	1.7182(17)
P(1)-O(3)	1.4642(14)
P(1)-O(4)	1.5759(13)
P(1)-O(5)	1.5811(13)
P(1)-C(6)	1.7942(17)
O(1)-N(1)	1.3963(17)
O(1)-C(9)	1.444(2)
O(2)-C(1)	1.230(2)
O(4)-C(10)	1.447(2)
O(5)-C(11)	1.453(2)
N(1)-C(7)	1.382(2)
N(1)-C(1)	1.399(2)
C(1)-C(2)	1.425(2)
C(2)-C(3)	1.390(2)
C(3)-C(4)	1.438(2)
C(3)-C(6)	1.444(2)
C(4)-C(5)	1.354(3)
C(4)-H(4)	0.9500
C(5)-H(5)	0.9500
C(6)-C(7)	1.376(2)
C(7)-C(8)	1.496(2)
C(8)-H(8A)	0.9800
C(8)-H(8B)	0.9800
C(8)-H(8C)	0.9800
C(9)-H(9A)	0.9800
C(9)-H(9B)	0.9800
C(9)-H(9C)	0.9800
C(10)-H(10A)	0.9800
C(10)-H(10B)	0.9800
C(10)-H(10C)	0.9800
C(11)-H(11A)	0.9800
C(11)-H(11B)	0.9800
C(11)-H(11C)	0.9800
C(5)-S(1)-C(2)	90.22(9)
O(3)-P(1)-O(4)	115.25(8)

O(3)-P(1)-O(5)	114.32(8)
O(4)-P(1)-O(5)	101.38(7)
O(3)-P(1)-C(6)	113.49(8)
O(4)-P(1)-C(6)	104.07(7)
O(5)-P(1)-C(6)	107.07(7)
N(1)-O(1)-C(9)	109.51(12)
C(10)-O(4)-P(1)	120.25(12)
C(11)-O(5)-P(1)	119.55(12)
C(7)-N(1)-O(1)	116.87(13)
C(7)-N(1)-C(1)	127.51(14)
O(1)-N(1)-C(1)	115.35(13)
O(2)-C(1)-N(1)	121.57(16)
O(2)-C(1)-C(2)	127.11(16)
N(1)-C(1)-C(2)	111.30(14)
C(3)-C(2)-C(1)	124.78(15)
C(3)-C(2)-S(1)	113.26(13)
C(1)-C(2)-S(1)	121.94(13)
C(2)-C(3)-C(4)	110.50(15)
C(2)-C(3)-C(6)	118.77(15)
C(4)-C(3)-C(6)	130.70(16)
C(5)-C(4)-C(3)	112.12(17)
C(5)-C(4)-H(4)	123.9
C(3)-C(4)-H(4)	123.9
C(4)-C(5)-S(1)	113.87(15)
C(4)-C(5)-H(5)	123.1
S(1)-C(5)-H(5)	123.1
C(7)-C(6)-C(3)	118.48(15)
C(7)-C(6)-P(1)	121.36(13)
C(3)-C(6)-P(1)	120.09(13)
C(6)-C(7)-N(1)	118.82(15)
C(6)-C(7)-C(8)	126.32(15)
N(1)-C(7)-C(8)	114.87(15)
C(7)-C(8)-H(8A)	109.5
C(7)-C(8)-H(8B)	109.5
H(8A)-C(8)-H(8B)	109.5
C(7)-C(8)-H(8C)	109.5

H(8A)-C(8)-H(8C)	109.5
H(8B)-C(8)-H(8C)	109.5
O(1)-C(9)-H(9A)	109.5
O(1)-C(9)-H(9B)	109.5
H(9A)-C(9)-H(9B)	109.5
O(1)-C(9)-H(9C)	109.5
H(9A)-C(9)-H(9C)	109.5
H(9B)-C(9)-H(9C)	109.5
O(4)-C(10)-H(10A)	109.5
O(4)-C(10)-H(10B)	109.5
H(10A)-C(10)-H(10B)	109.5
O(4)-C(10)-H(10C)	109.5
H(10A)-C(10)-H(10C)	109.5
H(10B)-C(10)-H(10C)	109.5
O(5)-C(11)-H(11A)	109.5
O(5)-C(11)-H(11B)	109.5
H(11A)-C(11)-H(11B)	109.5
O(5)-C(11)-H(11C)	109.5
H(11A)-C(11)-H(11C)	109.5
H(11B)-C(11)-H(11C)	109.5

Symmetry transformations used to generate equivalent atoms:

Table S15. Anisotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for **5n**. The anisotropic displacement factor exponent takes the form: $-2\pi^2 [h^2 a^* a^2 U^{11} + \dots + 2 h k a^* b^* U^{12}]$

	U ¹¹	U ²²	U ³³	U ²³	U ¹³	U ¹²
S(1)	39(1)	33(1)	34(1)	0(1)	15(1)	-16(1)
P(1)	20(1)	24(1)	23(1)	-4(1)	4(1)	-2(1)
O(1)	27(1)	23(1)	19(1)	4(1)	4(1)	1(1)
O(2)	22(1)	33(1)	30(1)	-5(1)	5(1)	-7(1)
O(3)	33(1)	38(1)	24(1)	-2(1)	4(1)	-4(1)
O(4)	19(1)	39(1)	31(1)	-8(1)	3(1)	1(1)
O(5)	25(1)	25(1)	33(1)	-7(1)	7(1)	-5(1)
N(1)	21(1)	19(1)	19(1)	0(1)	6(1)	-1(1)
C(1)	20(1)	18(1)	28(1)	-5(1)	9(1)	-2(1)

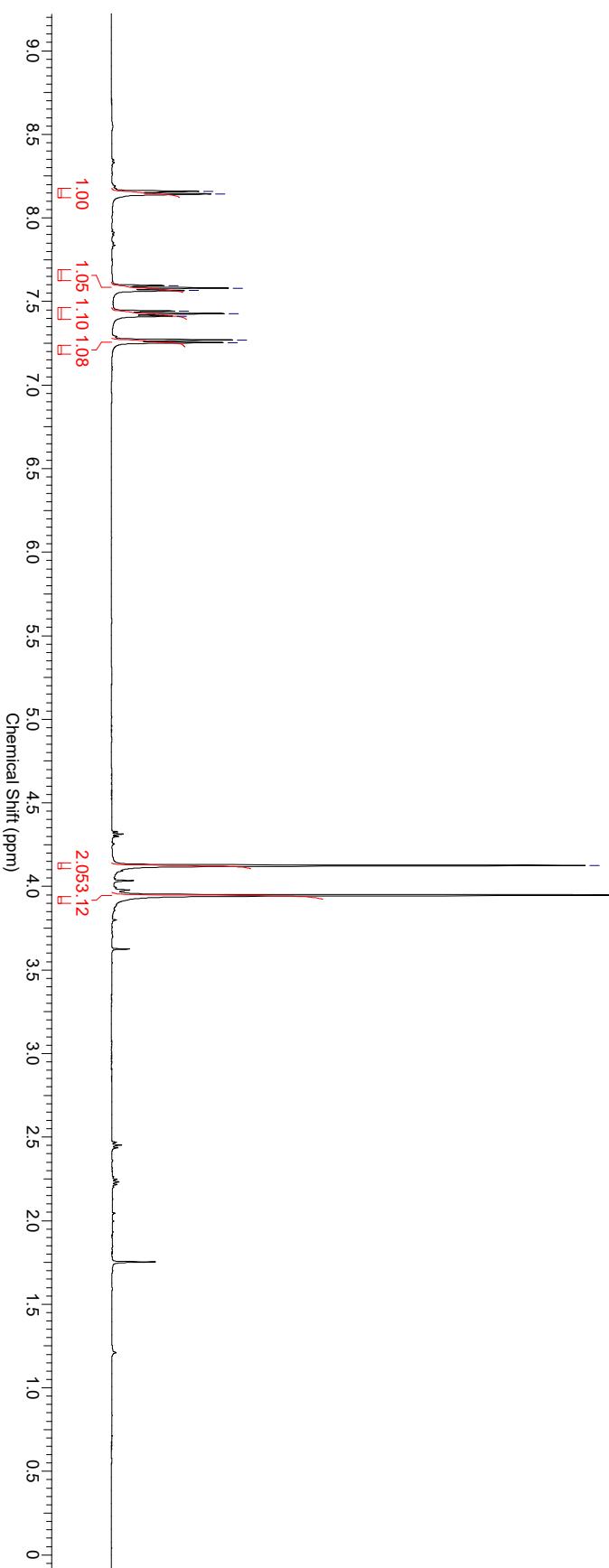
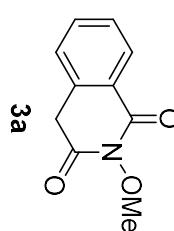
C(2)	24(1)	19(1)	28(1)	-2(1)	12(1)	-4(1)
C(3)	23(1)	18(1)	24(1)	-3(1)	10(1)	2(1)
C(4)	34(1)	25(1)	26(1)	1(1)	11(1)	1(1)
C(5)	49(1)	32(1)	30(1)	6(1)	17(1)	-6(1)
C(6)	19(1)	19(1)	22(1)	-3(1)	7(1)	-1(1)
C(7)	18(1)	17(1)	25(1)	-3(1)	7(1)	-1(1)
C(8)	27(1)	25(1)	28(1)	1(1)	7(1)	-7(1)
C(9)	28(1)	35(1)	22(1)	-2(1)	7(1)	-1(1)
C(10)	20(1)	46(1)	40(1)	2(1)	3(1)	-3(1)
C(11)	35(1)	26(1)	37(1)	-6(1)	10(1)	3(1)

Appendix 2

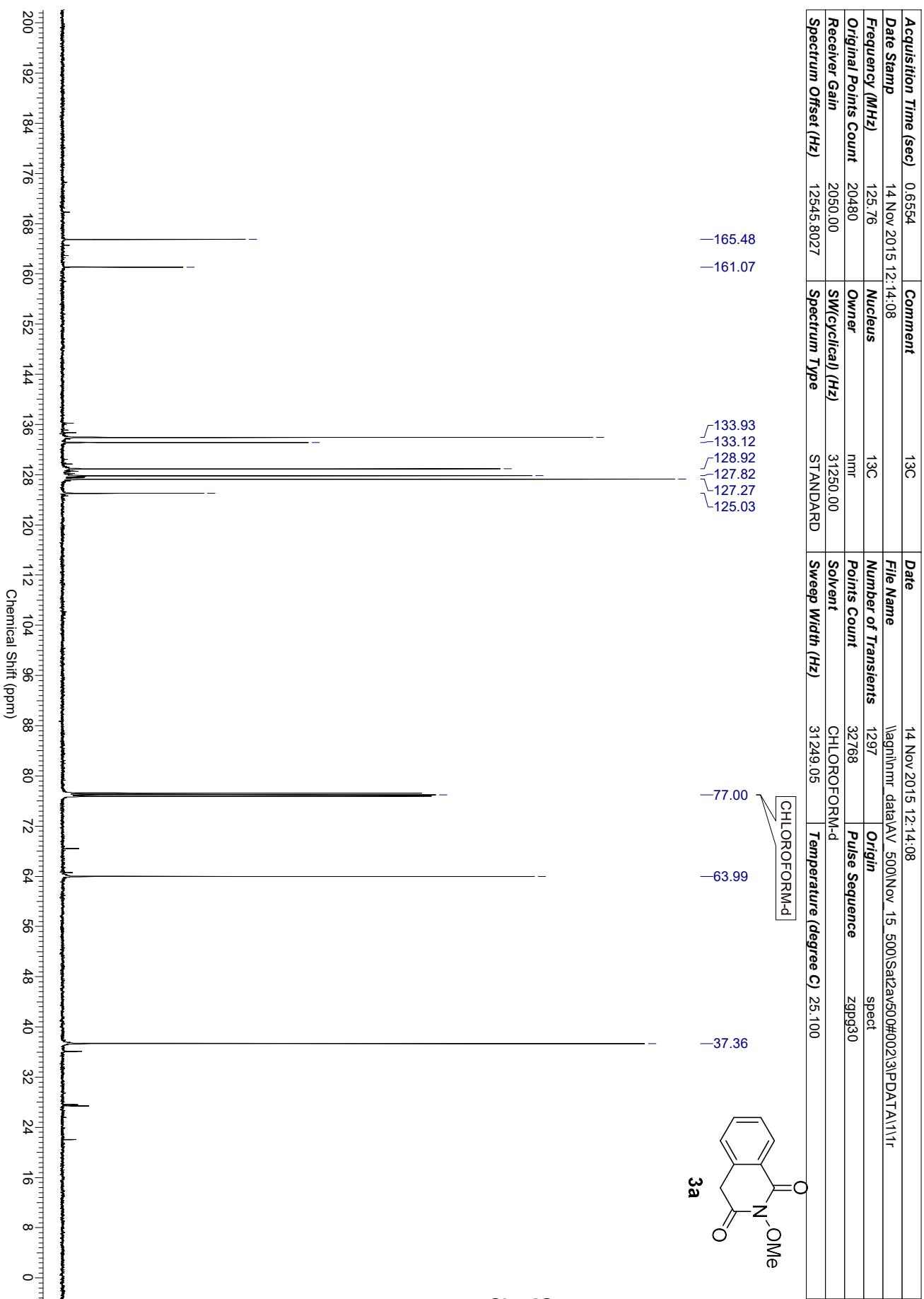
^1H & ^{13}C NMR and HRMS Spectra

¹H NMR Spectra for Compound 3a in CDCl₃

Acquisition Time (sec)	1.6000	Comment	Venkanna 1H	Date	14-Nov-2015 11:10:08
Date Stamp	14 Nov 2015 11:10:08			File Name	\lagnmr_dataAV_500Nov_15_500Sat2av500#002\1PDATA\11r
Frequency (MHz)	500.13	Nucleus	1H	Number of Transients	60
Original Points Count	16000	Owner	nmr	Points Count	32768
Receiver Gain	128.00	SW(cyclical) (Hz)	10000.00	Solvent	CHLOROFORM-d
Spectrum Offset (Hz)	2489.8574	Spectrum Type	STANDARD	Sweep Width (Hz)	9999.70

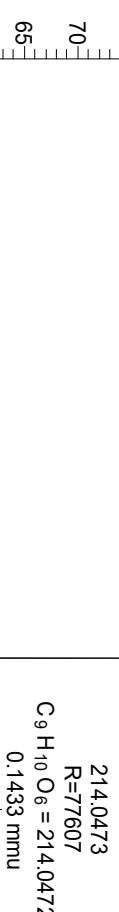
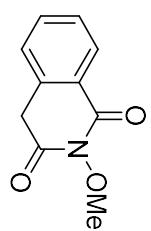
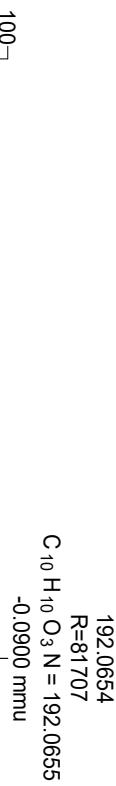


¹³C NMR Spectra for Compound 3a in CDCl₃

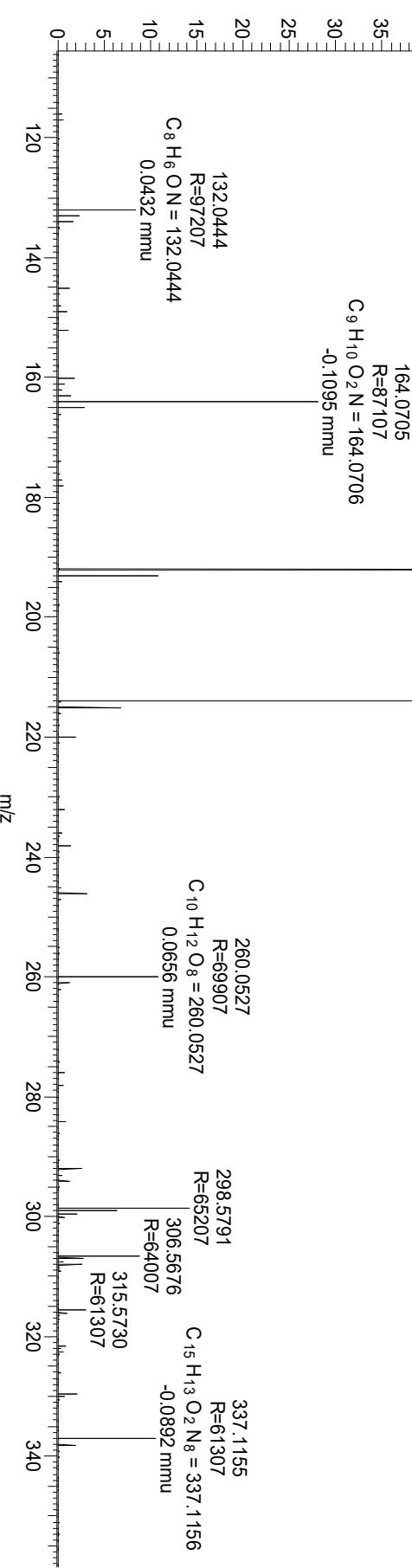


HRMS Spectra for Compound 3a in MeOH

RSP-1 #97 RT: 0.43 AV: 1 NL: 1.13E9
T: FTMS + p ESI Full ms [100.00-1500.00]

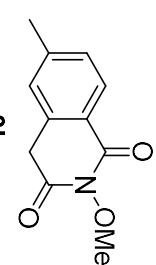
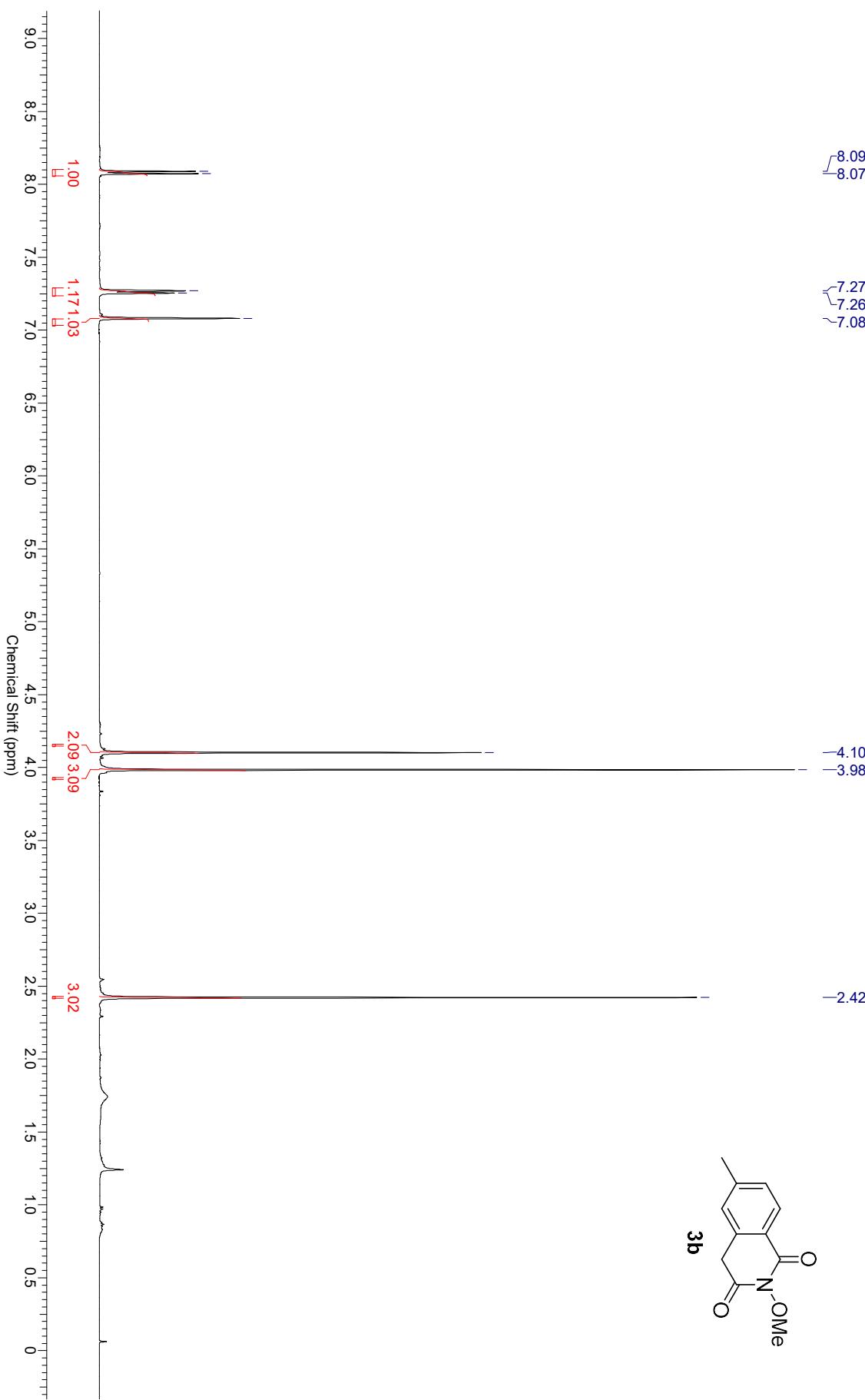


Relative Abundance

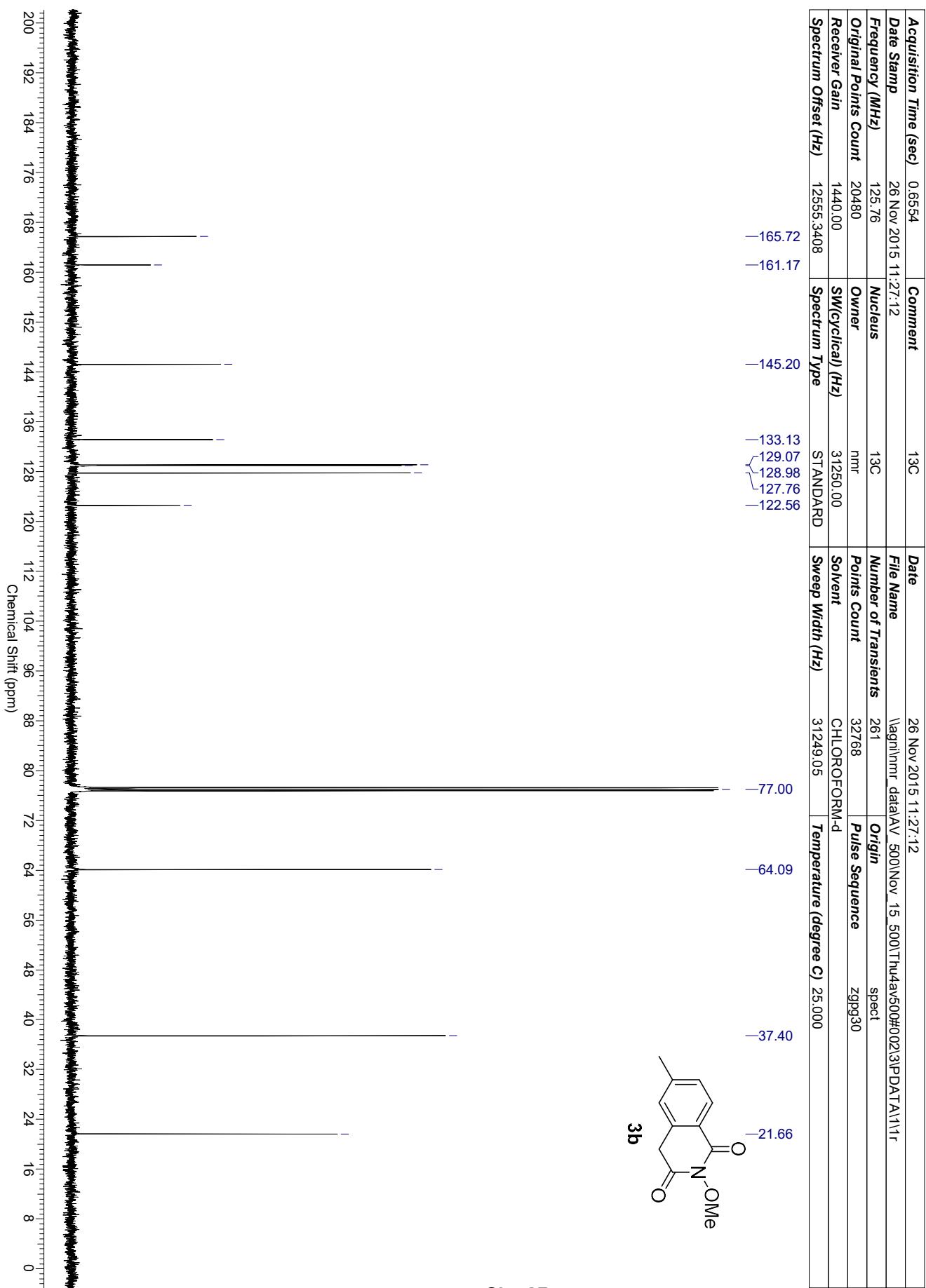


¹H NMR Spectra for Compound 3b in CDCl₃

Acquisition Time (sec)	1.6000	Comment	Ravinda 1H	Date	26 Nov 2015 11:14:24
Date Stamp	26 Nov 2015 11:14:24			File Name	\agilmr_data\AV_500\Nov_15_500\Thu4av500#021\PDAT\1\Tr
Frequency (MHz)	500.13	Nucleus	1H	Number of Transients	64
Original Points Count	16000	Owner	nmr	Points Count	32768
Receiver Gain	181.00	SW(cyclical) (Hz)	10000.00	Solvent	CHLOROFORM-d
Spectrum Offset (Hz)	2490.7729	Spectrum Type	STANDARD	Sweep Width (Hz)	9999.70
				Temperature (degree C)	24.900



¹³C NMR Spectra for Compound **3b** in CDCl₃



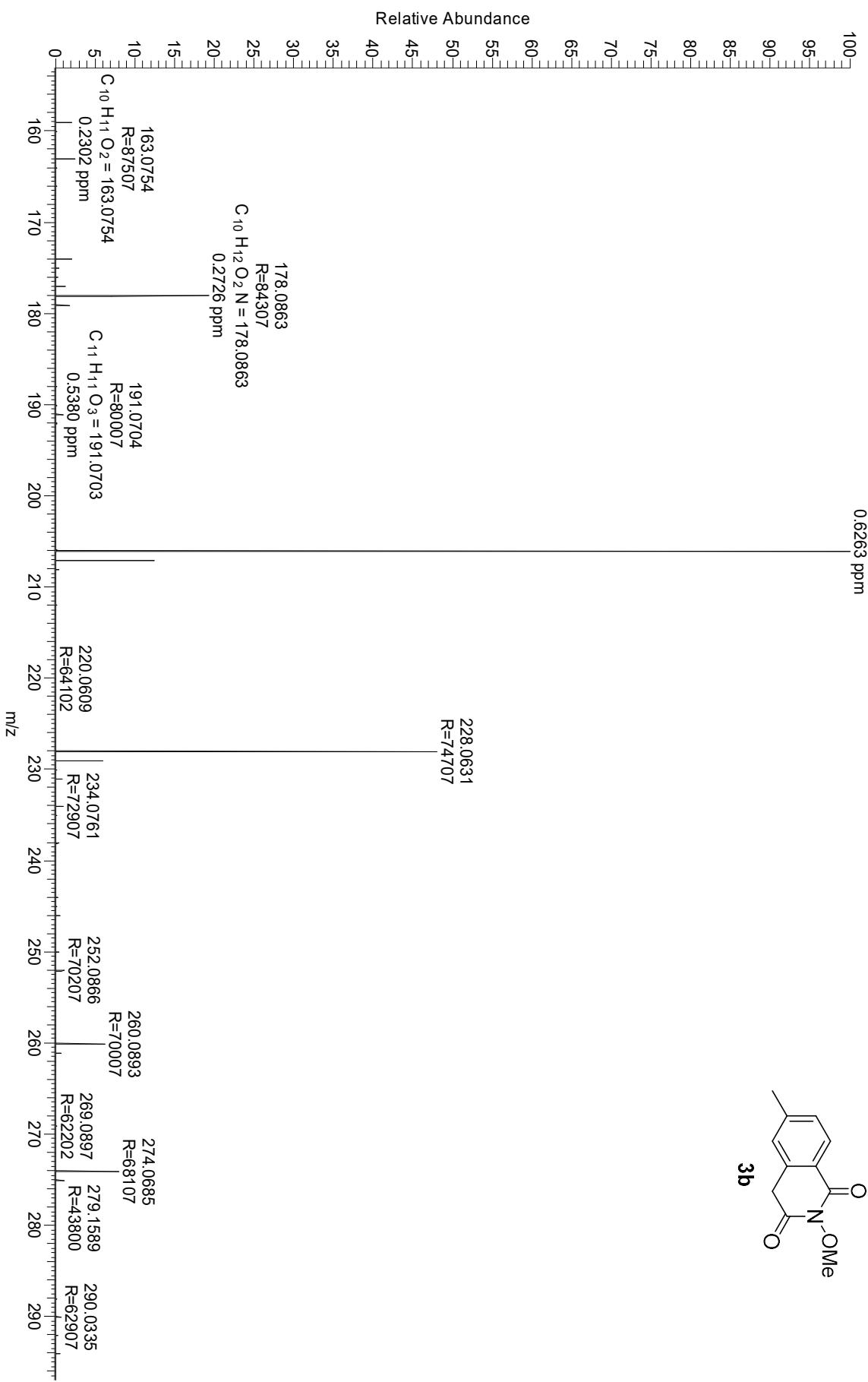
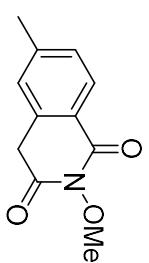
HRMS Spectra for Compound **3b** in MeOH

RSP-10#98 RT: 0.43 AV: 1 NL: 1.89E9
T:Ftms + p ESI[Full.ms [100.00-1500.00]]

226.0813

R=79107

C₁₁H₁₂O₃N = 206.0812
0.6263 ppm



¹H NMR Spectra for Compound 3c in CDCl₃

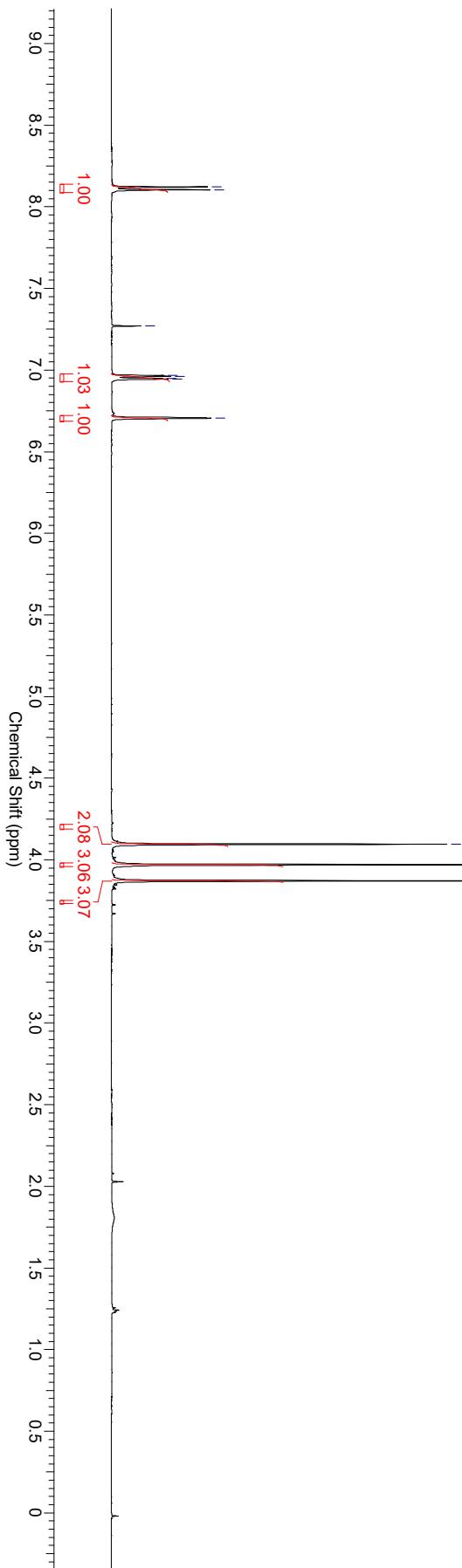
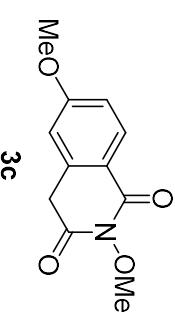
Acquisition Time (sec)	1.6000	Comment	Pitambar 1H	Date	16 Nov 2015 12:16:16
Date Stamp	16 Nov 2015 12:16:16			File Name	\agnmr\data\AV_500Nov_15_500Mon3av500#003\1PDATA\11r
Frequency (MHz)	500.13	Nucleus	1H	Number of Transients	64
Original Points Count	16000	Owner	nmr	Points Count	32768
Receiver Gain	203.00	SW(cyclical) (Hz)	10000.00	Solvent	CHLOROFORM-d
Spectrum Offset (Hz)	2492.2988	Spectrum Type	STANDARD	Sweep Width (Hz)	9999.70
				Temperature (degree C)	24.900

8.12
8.10

7.27
6.97
6.96
6.95
6.94
6.70

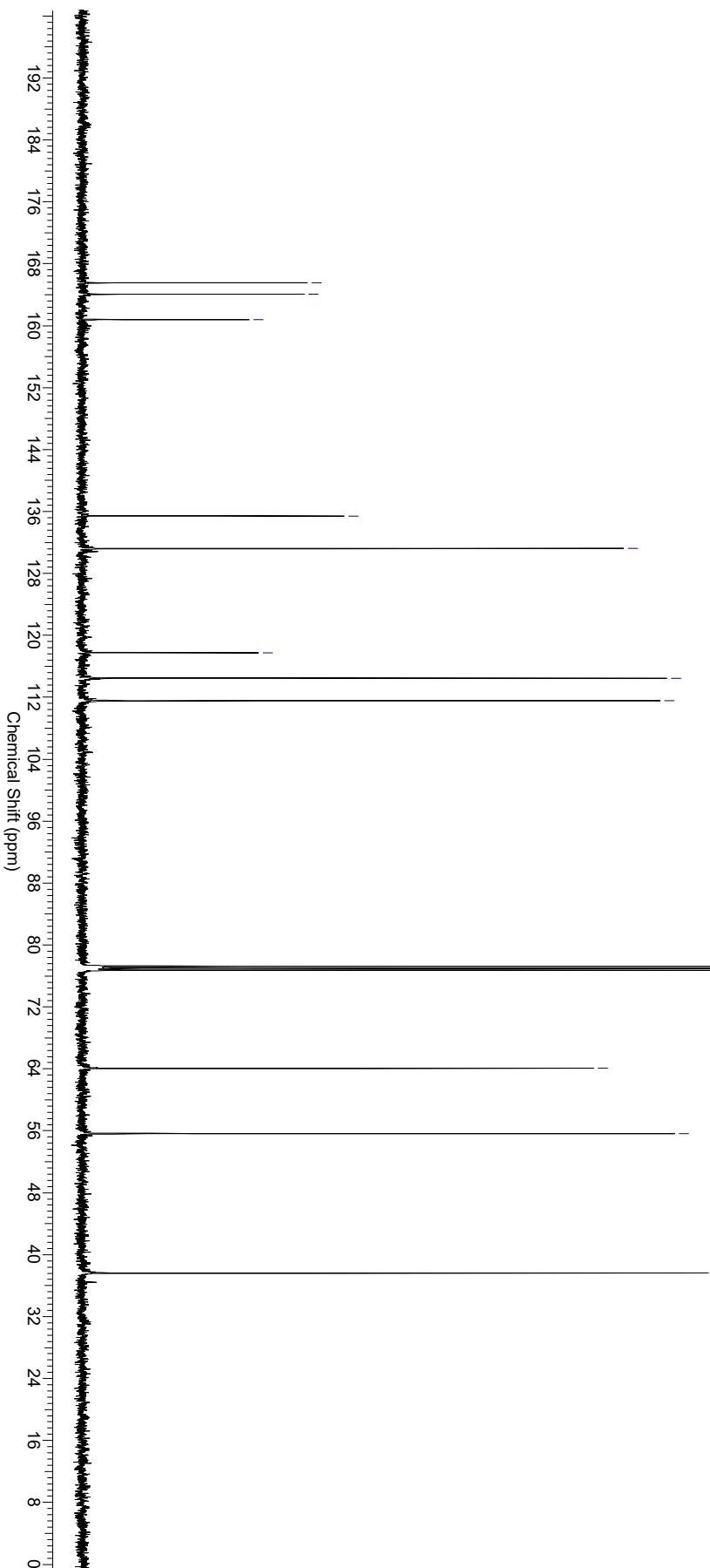
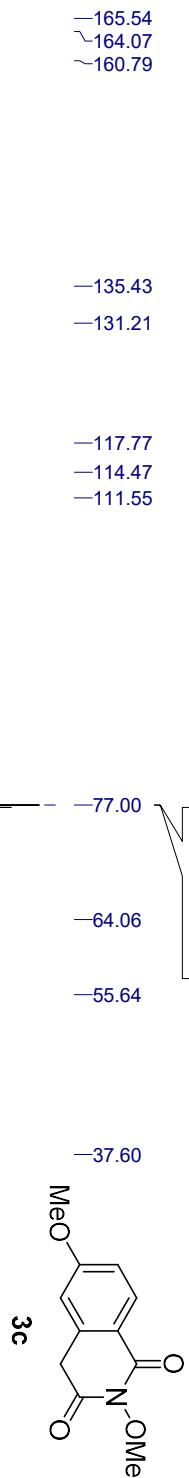
4.10
3.97
~3.87

CHLOROFORM-d



¹³C NMR Spectra for Compound **3c** in CDCl₃

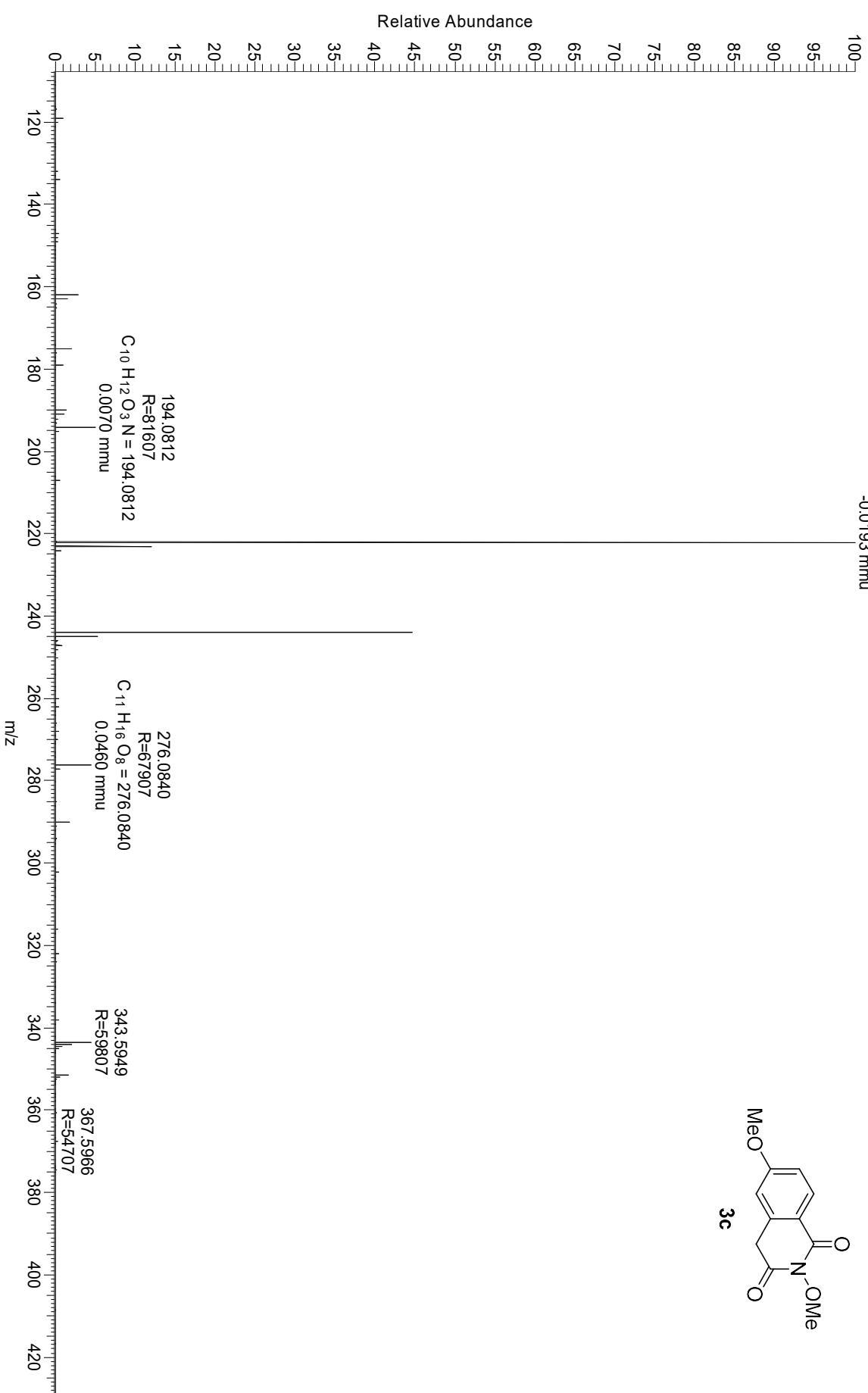
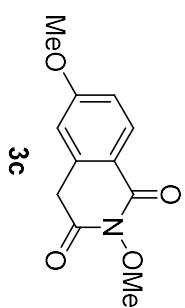
Acquisition Time (sec)	0.6554	Comment	13C	Date	16 Nov 2015 12:54:40
Date Stamp	16 Nov 2015 12:54:40			File Name	\lagnihmr\data\AV_500\Nov_15_500MHz3av500#\003\3\PDAT\1\1\
Frequency (MHz)	125.76	Nucleus	13C	Number of Transients	421
Original Points Count	20480	Owner	nmr	Points Count	32768
Receiver Gain	2050.00	SW(cyclical) (Hz)	31250.00	Solvent	CHLOROFORM-d
Spectrum Offset (Hz)	12552.4785	Spectrum Type	STANDARD	Sweep Width (Hz)	31249.05
				Temperature (degree C)	25.100



HRMS Spectra for Compound 3c in MeOH

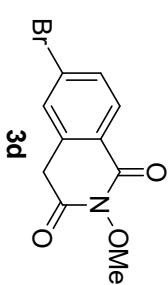
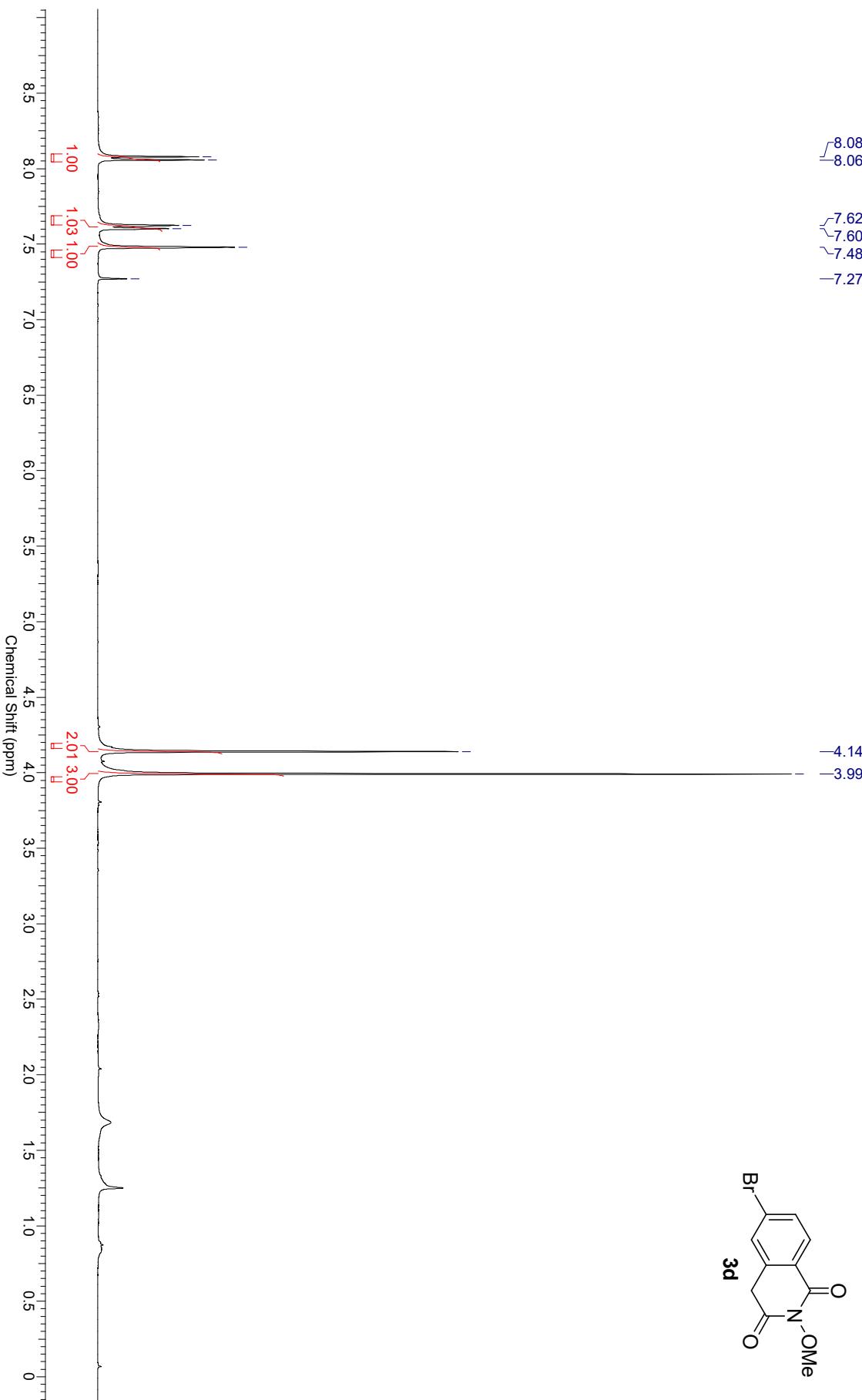
RSP-2 #99 RT: 0.44 AV: 1 NL: 3.94E9
T: FTMS + pESI Full ms [100.00-1500.00]

222.0761
R=75907
 $C_{11}H_{12}O_4N = 222.0761$
-0.0193 mmu

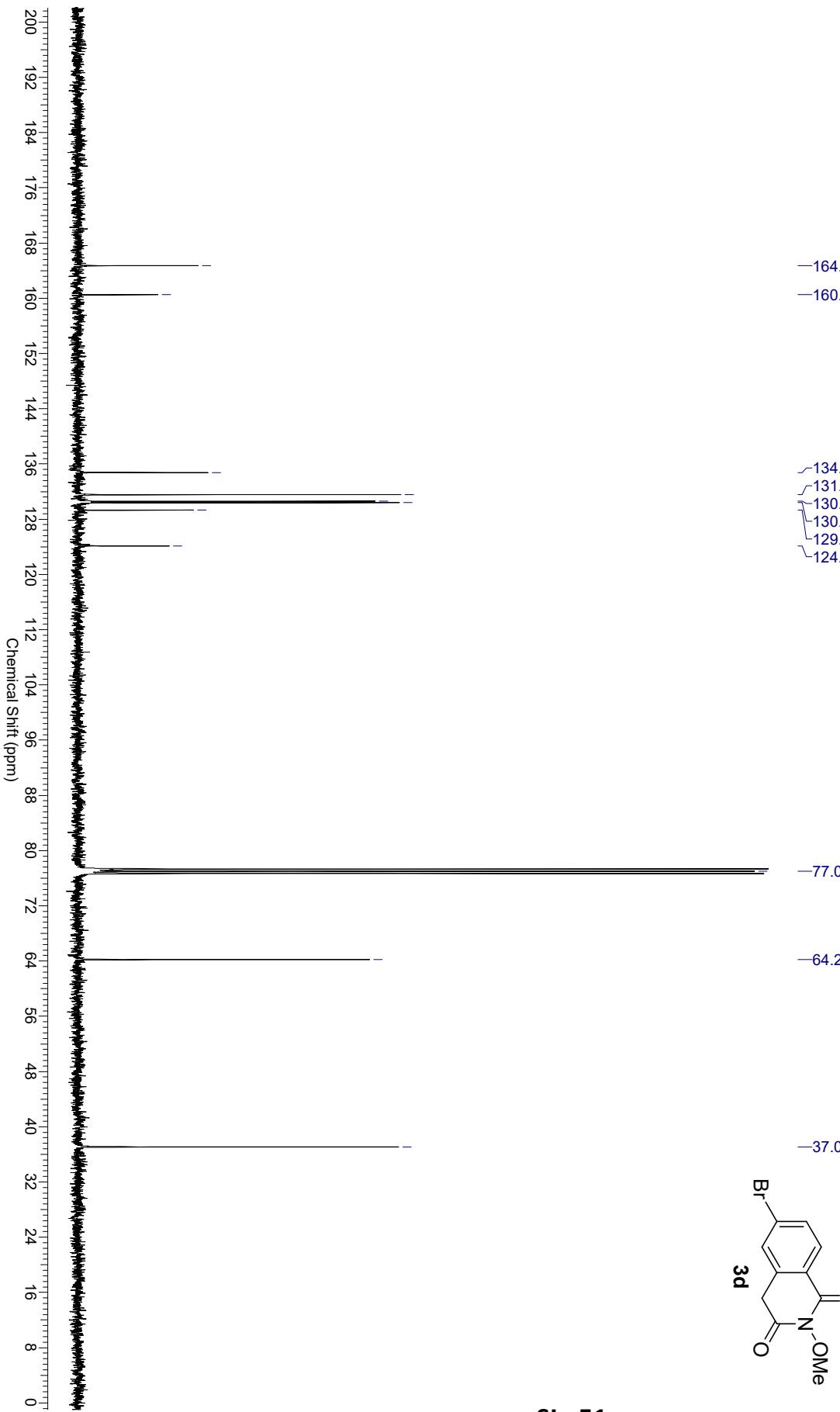


¹H NMR Spectra for Compound 3d in CDCl₃

Acquisition Time (sec)	2.0447	Comment	Ravindra 1H	Date	26 Nov 2015 10:03:52
Date Stamp	26 Nov 2015 10:03:52			File Name	\agnmr\data\AV400\Nov_15_400\Thu4av400#001\1\PDATA\1\1r
Frequency (MHz)	400.13	Nucleus	1H	Number of Transients	64
Original Points Count	16384	Owner	Administrator	Points Count	32768
Receiver Gain	406.00	SW(cyclical) (Hz)	8012.82	Solvent	CHLOROFORM-d
Spectrum Offset (Hz)	2396.0396	Spectrum Type	STANDARD	Sweep Width (Hz)	8012.58
				Temperature (degree C)	22.500

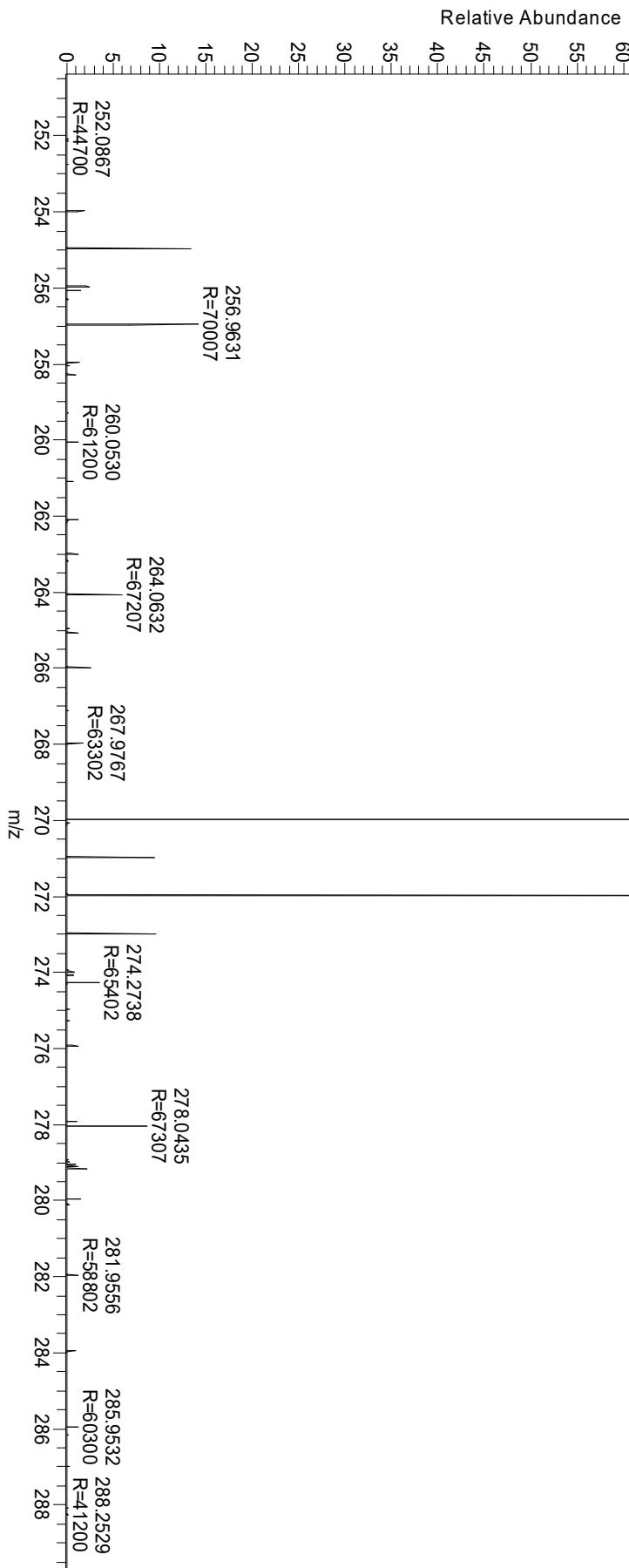
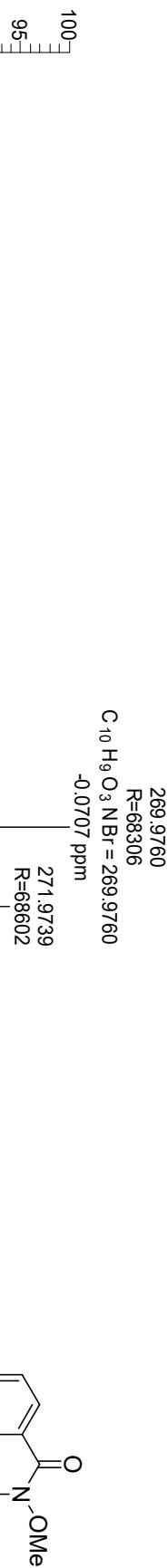


¹³C NMR Spectra for Compound **3d** in CDCl₃



HRMS Spectra for Compound **3d** in MeOH

RSP-9 #101 RT: 0.45 AV: 1 NL: 5.92E7
T: FTMS + pESI[Full ms [100.00-1500.00]



¹H NMR Spectra for Compound 3e in CDCl₃

Acquisition Time (sec)	2.0447	Comment	Pitambar 1H	Date	13 Nov 2015 14:30:32
Date Stamp	13 Nov 2015 14:30:32	File Name	\\agnlnmr_data\\v400\\Nov_15_400\\Fri2av400#005\\1\\PDATA\\111r		
Frequency (MHz)	400.13	Nucleus	1H	Number of Transients	128
Original Points Count	16384	Owner	Administrator	Points Count	32768
Receiver Gain	362.00	SW(cyclical) (Hz)	8012.82	Pulse Sequence	zg30
Spectrum Offset (Hz)	2395.7949	Spectrum Type	STANDARD	Sweep Width (Hz)	8012.58
				Temperature (degree C)	22.400

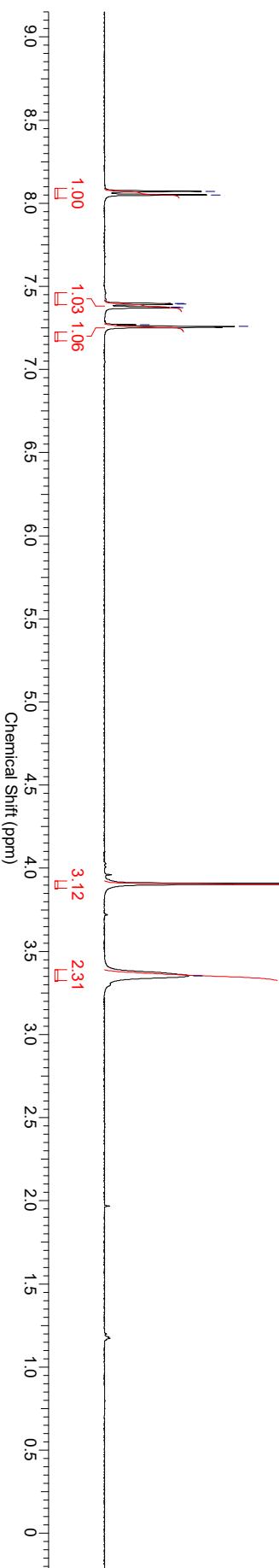
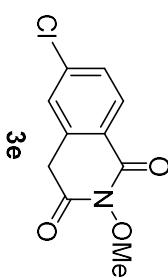
8.07
8.05

7.40
7.39
7.38
7.37
7.27
7.26

CHLOROFORM-d

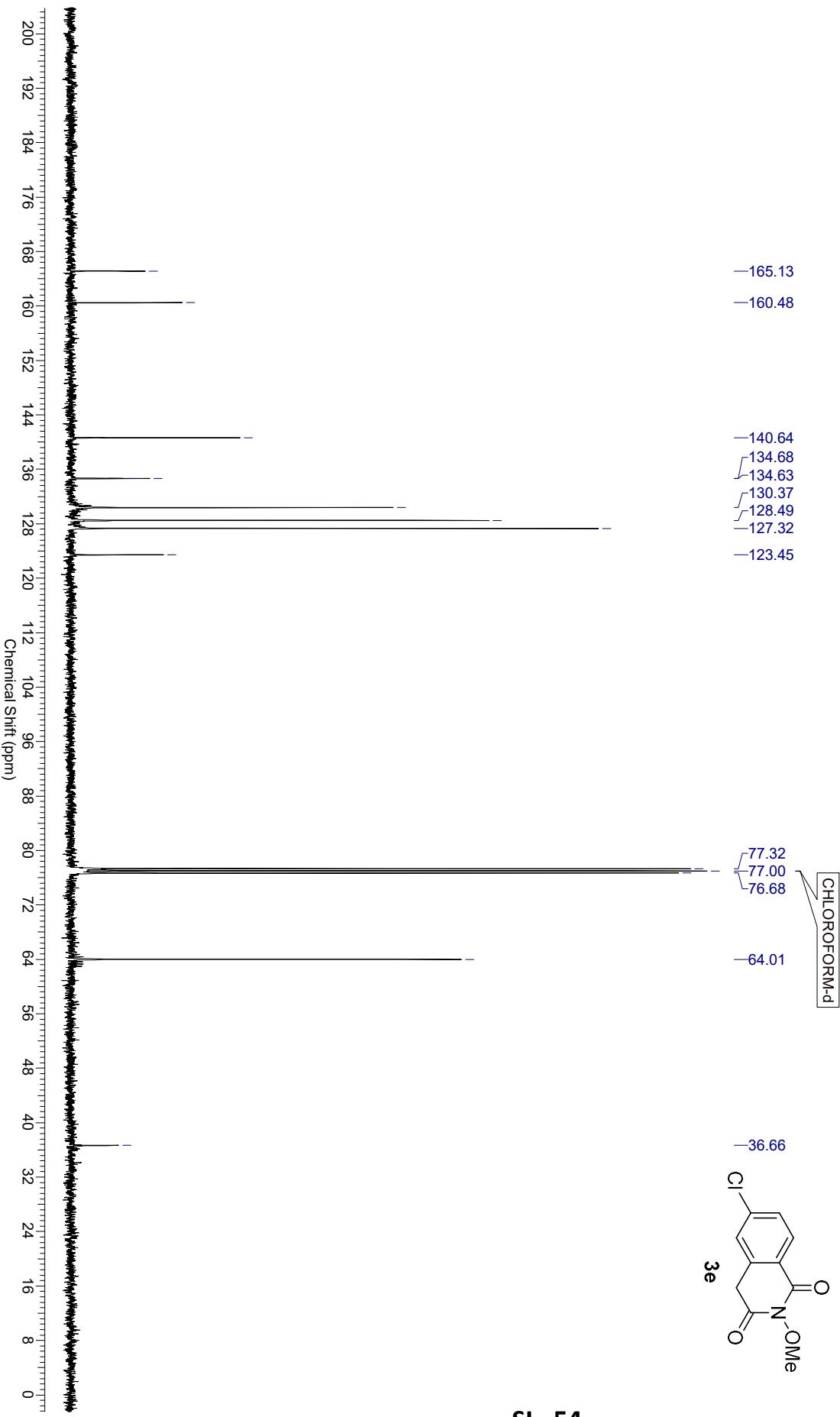
3.91

3.35



¹³C NMR Spectra for Compound 3e in CDCl₃

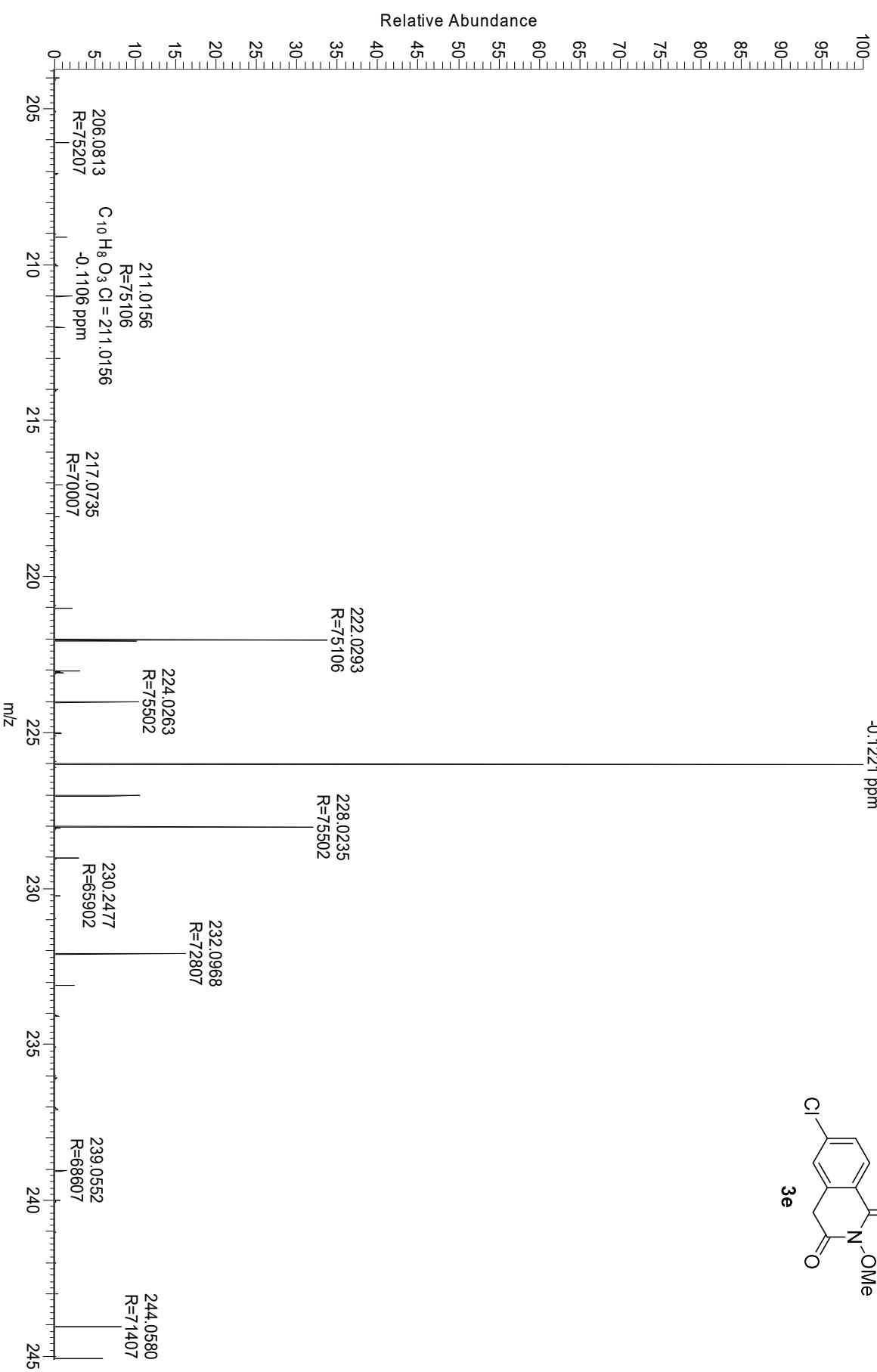
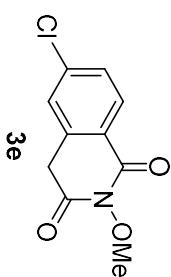
Acquisition Time (sec)	0.6488	Comment	13C	Date	13 Nov 2015 15:47:20
Date Stamp	13 Nov 2015 15:47:20			File Name	\lagnihmr\data\AV400\Nov_15_400\F12av400#005\3\PDATA\11r
Frequency (MHz)	100.61	Nucleus	13C	Number of Transients	1498
Original Points Count	16384	Owner	root	Points Count	32768
Receiver Gain	2050.00	SW(cyclical) (Hz)	25252.53	Pulse Sequence	zgpg
Spectrum Offset (Hz)	10050.2578	Spectrum Type	STANDARD	Sweep Width (Hz)	25251.75
				Temperature (degree C)	22.7.00



HRMS Spectra for Compound 3e in MeOH

RSP-3#105 RT: 0.47 AV: 1 NL: 1.13E8
T: FTMS + p ESI Full ms [100.00-1500.00]

226.0265
R=75606
 $C_{10}H_9O_3NCl = 226.0265$
-0.1221 ppm



¹H NMR Spectra for Compound 3f in CDCl₃

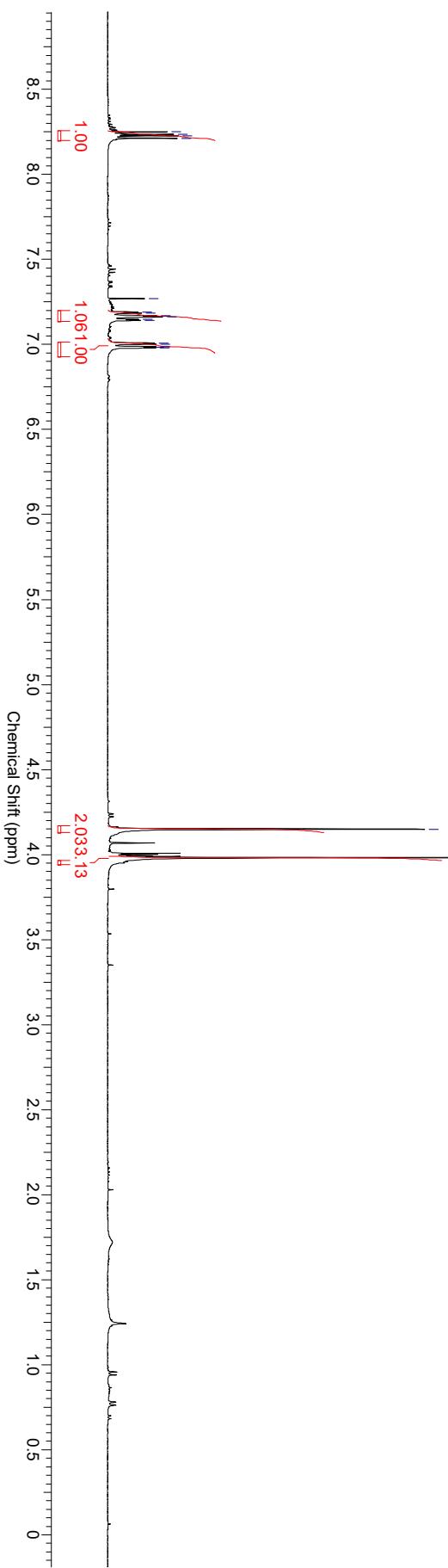
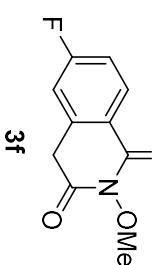
Acquisition Time (sec)	2.1838	Comment	Pitambar	Date	07 Jan 2016 08:30:41
Date Stamp	06 Jan 2016 16:35:18			File Name	\laginmr\data\JAN_LIQUID\tue2ecx400#027_PROTON-3.jdf
Frequency (MHz)	399.78	Nucleus	1H	Number of Transients	32
Original Points Count	13107	Owner	delta	Points Count	13107
Receiver Gain	38.00	Solvent	CHLOROFORM-d	Pulse Sequence	single_pulse.ex2
Spectrum Type	STANDARD	Sweep Width (Hz)	6001.85	Temperature (degree C)	23.600
				Spectrum Offset (Hz)	2007.4568

8.25
8.23
8.23
8.21

7.27
7.18
7.17
7.16
7.14
7.14
7.01
7.00
6.99
6.98
6.98

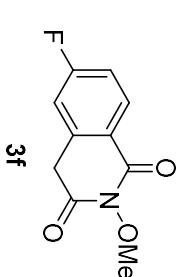
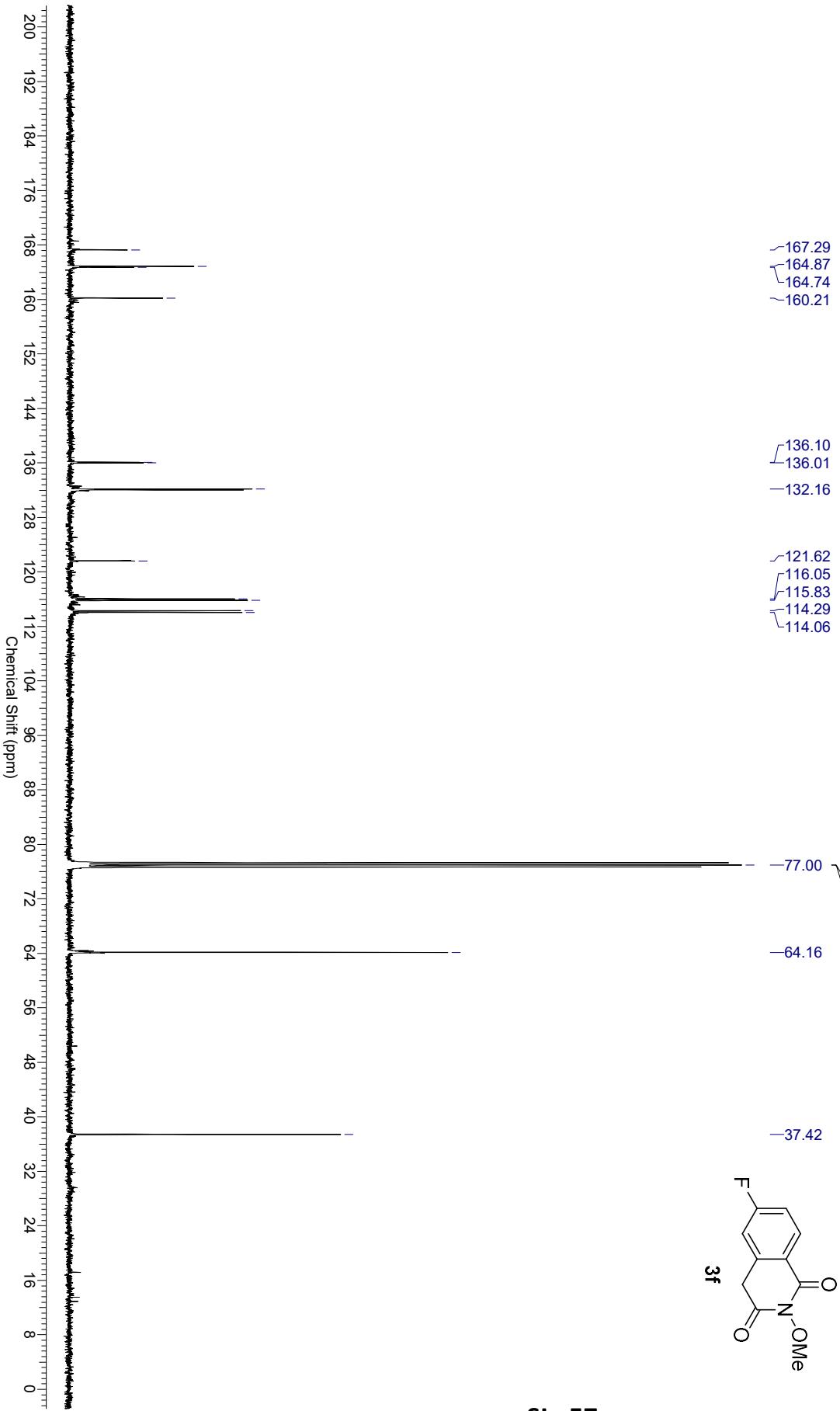
CHLOROFORM-d

4.15
3.98



¹³C NMR Spectra for Compound 3f in CDCl₃

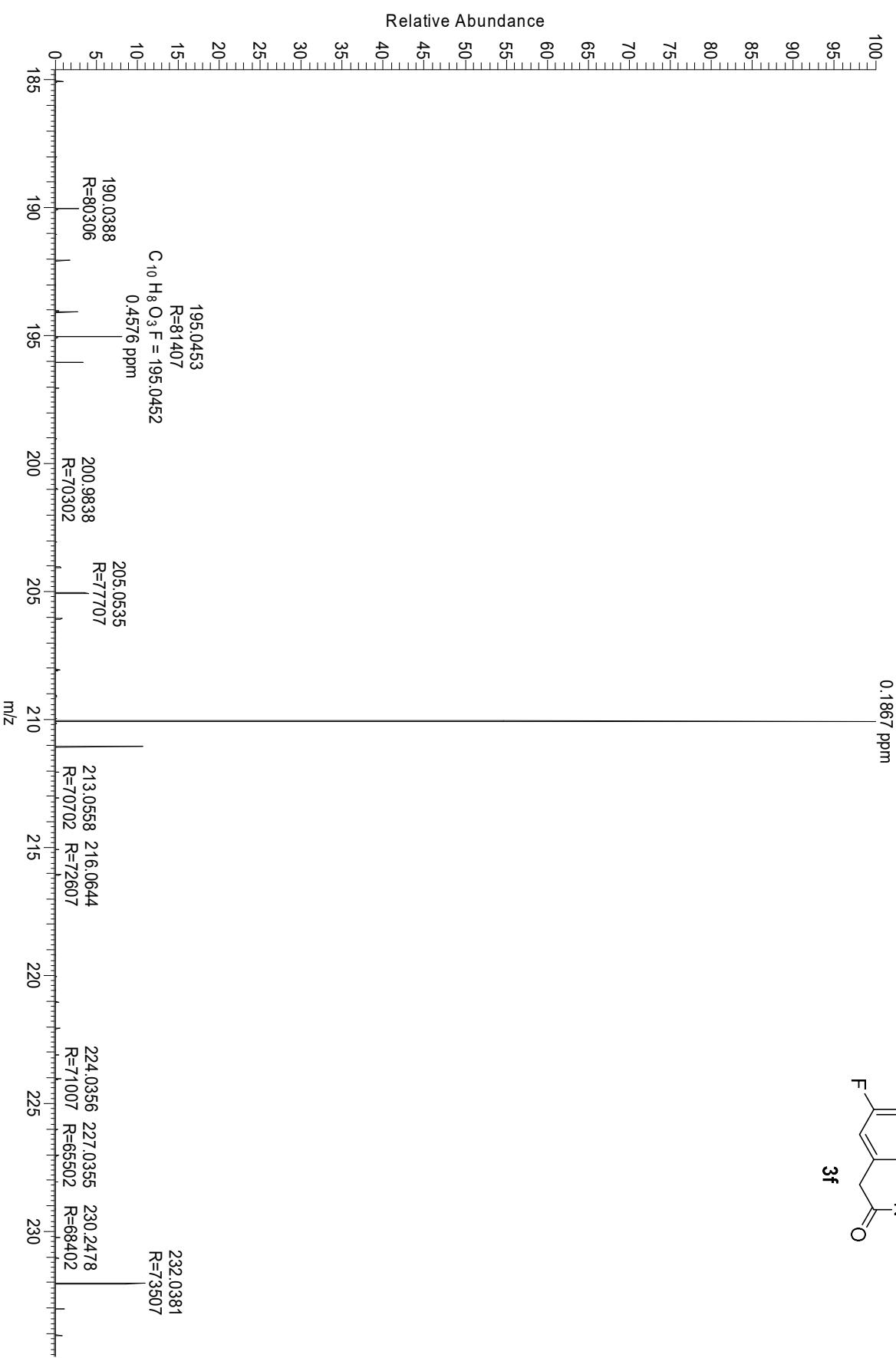
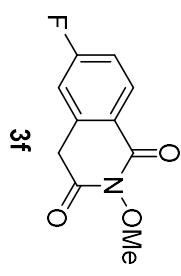
Acquisition Time (sec)	1.0434	Comment	Pitambar	Date	07-Jan-2016 09:48:40
Date Stamp	06 Jan 2016 17:21:59			File Name	\agn\inmr_data\EOL_400\2016\JAN\LIQUID\TUE2ECX400#027_CARBON-3.jdf
Frequency (MHz)	100.53	Nucleus	¹³ C	Number of Transients	1200
Original Points Count	26214	Owner	delta	Points Count	26214
Receiver Gain	60.00	Solvent	CHLOROFORM-d	Pulse Sequence	single pulse dec
Spectrum Type	STANDARD	Sweep Width (Hz)	25124.29	Temperature (degree C)	23.700



HRMS Spectra for Compound 3f in MeOH

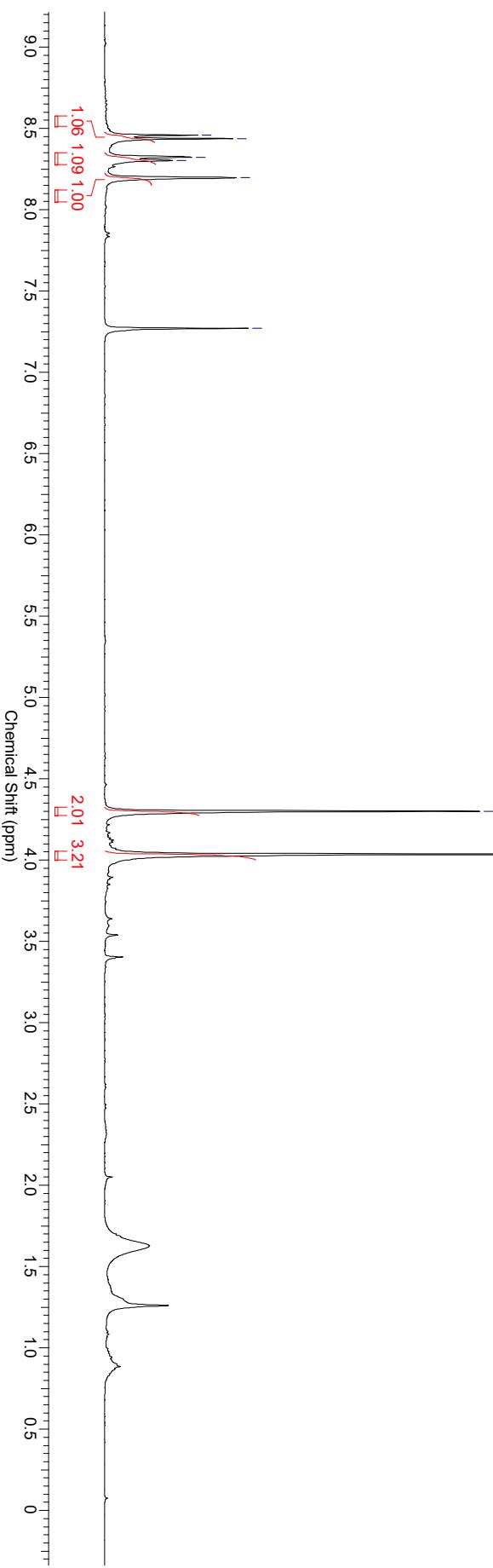
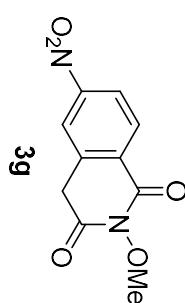
RSP-14#100 RT: 0.44 AV: 1 NL: 2.04E8
T: FTMS + pESI[Full ms] [100.00-1500.00]

210.0561
R=77707
C₁₀H₉O₃NF = 210.0561
0.1867 ppm



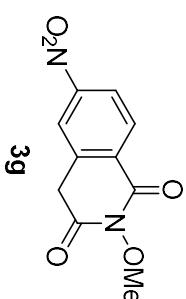
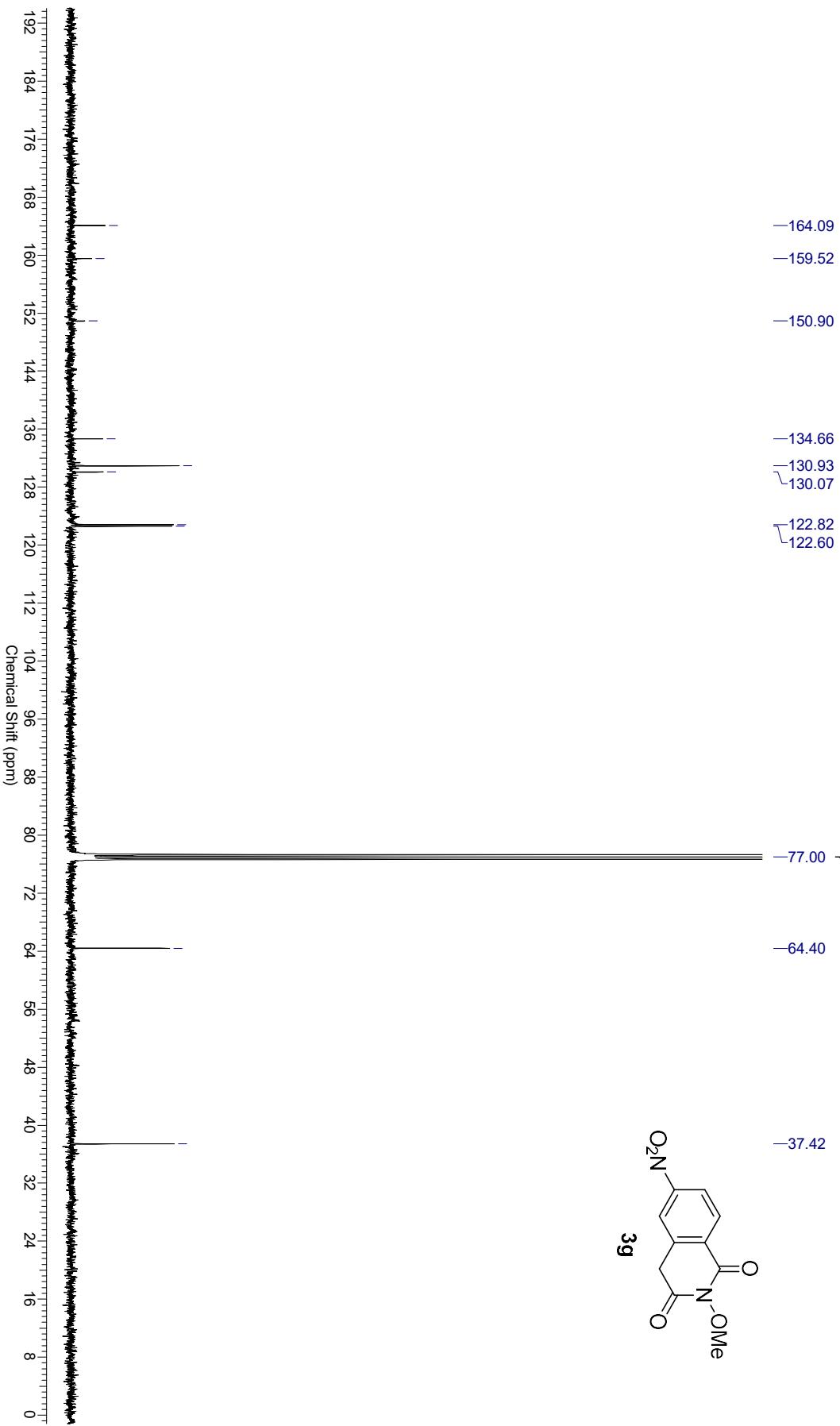
¹H NMR Spectra for Compound 3g in CDCl₃

Acquisition Time (sec)	2.0447	Comment	Ravindra 1H	Date	30 Jan 2016 12:56:40
Date Stamp	30 Jan 2016 12:56:40			File Name	\172.16.24.inmr\data\AV400\Jan_16_400\Jan_16_400\Sat5av0#0151\PDAT\A1\1r
Frequency (MHz)	400.13	Nucleus	1H	Number of Transients	128
Original Points Count	16384	Owner	root	Points Count	16384
Receiver Gain	512.00	SW(cyclical) (Hz)	8012.82	Solvent	DMSO-d6
Spectrum Type	STANDARD	Sweep Width (Hz)	8012.33	Temperature (degree C)	23.300



¹³C NMR Spectra for Compound 3g in CDCl₃

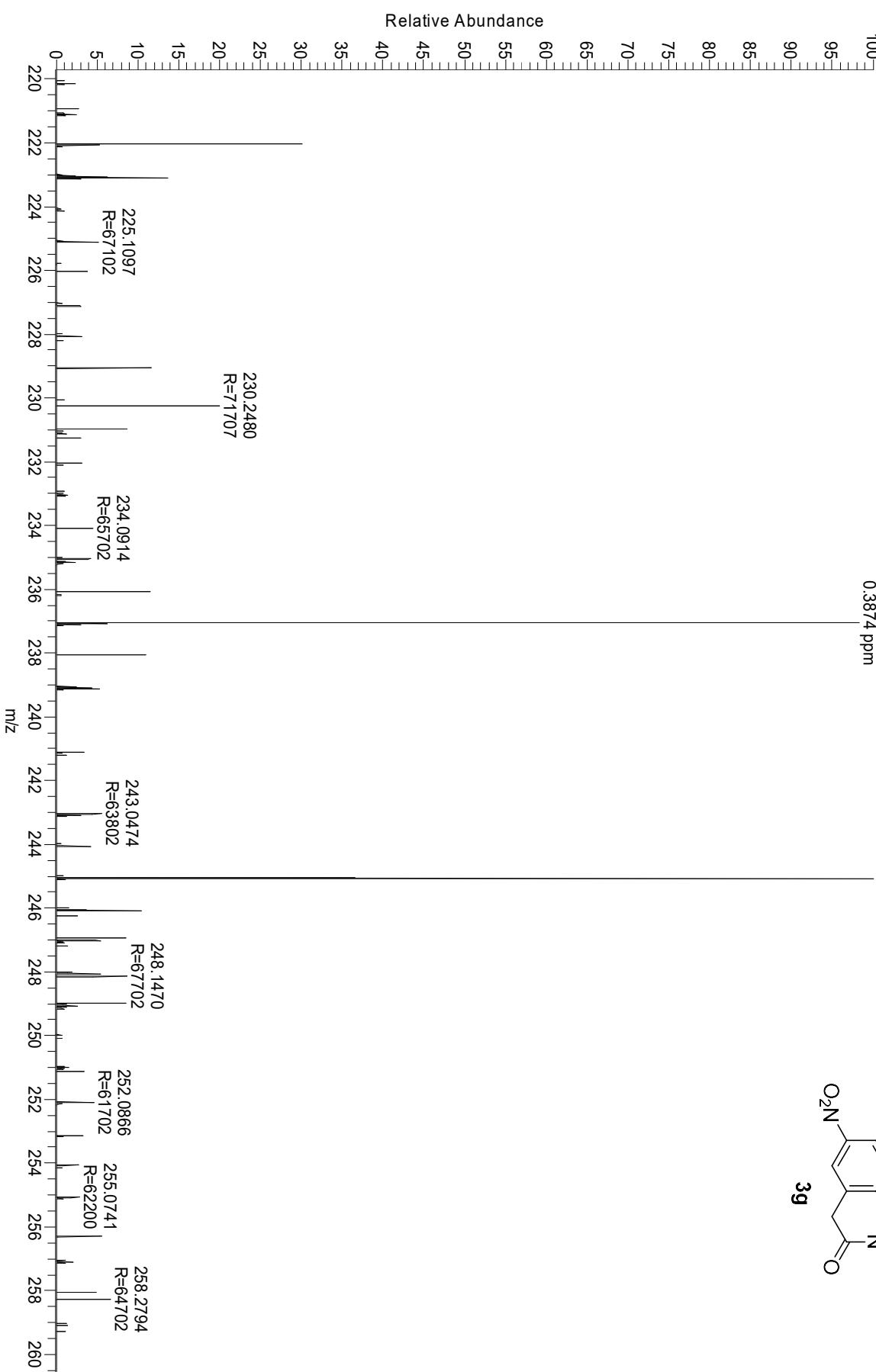
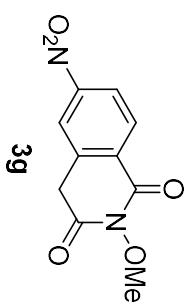
Acquisition Time (sec)	0.6488	Comment	13C	Date	30 Jan 2016 14:22:00
Date Stamp	30 Jan 2016 14:22:00	File Name	\1172.16.24nmr data\AV400\Jan_16_400\Jan_16_400\Sat\Sav400#015\3\PDAT\A\11r		
Frequency (MHz)	100.61	Nucleus	13C	Number of Transients	2232
Original Points Count	16384	Owner	root	Points Count	32768
Receiver Gain	2050.00	SW(cyclical) (Hz)	25252.53	Solvent	METHANOL-d ₄
Spectrum Offset (Hz)	10059.5068	Spectrum Type	STANDARD	Sweep Width (Hz)	25251.75
				Temperature (degree C)	23.700



HRMS Spectra for Compound 3g in MeOH

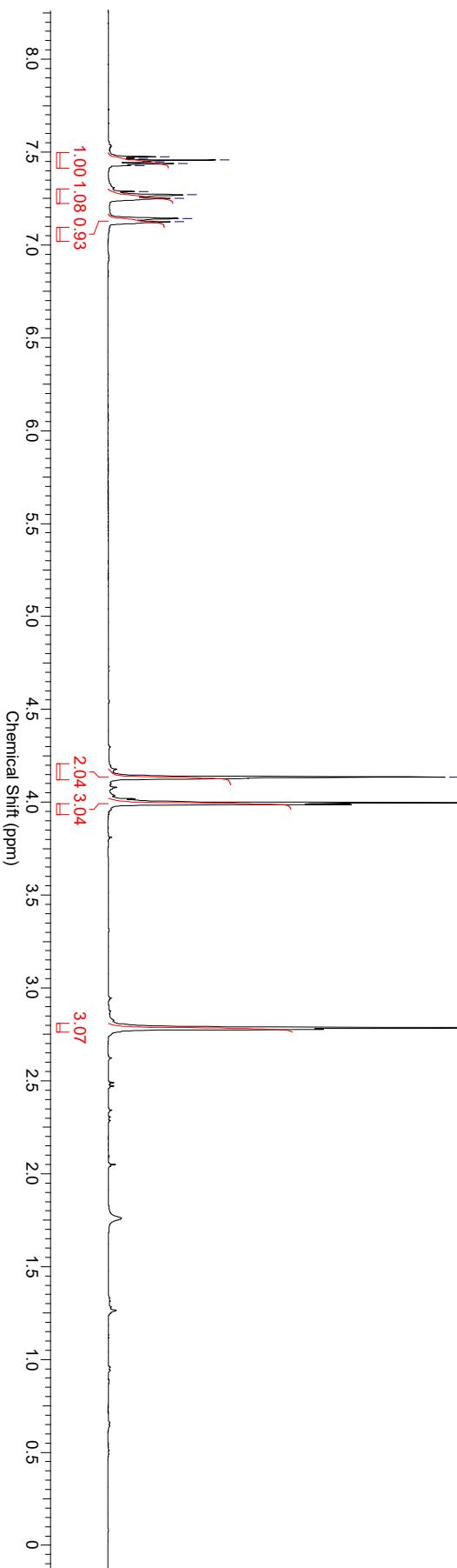
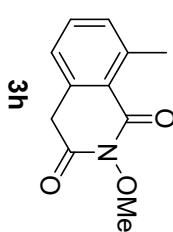
RSP-15 #101 RT: 0.45 AV: 1 NL: 7.31E6
T: FTMS + pESI[Full ms [100.00-1500.00]]

237.0507
R=73107
 $C_{10}H_9O_5N_2 = 237.0506$
0.3874 ppm
245.0785
R=71607



¹H NMR Spectra for Compound 3h in CDCl₃

Acquisition Time (sec)	2.1838	Comment	Pilambar	Date	17 Nov 2015 04:56:12
Date Stamp	16 Nov 2015 13:04:19				
File Name	F:\piptambar\Research\3rd methodology\AmideNMR\melfromMeldrom_o-toluidMon3ECX400#005.PROTON-3.jdf				
Frequency (MHz)	399.78	Nucleus	¹ H	Number of Transients	64
Original Points Count	13107	Owner	delta	Points Count	13107
Receiver Gain	32.00	Solvent	CHLOROFORM-d	Pulse Sequence	single pulse ex2
Spectrum Type	STANDARD	Sweep Width (Hz)	6001.85	Spectrum Offset (Hz)	2015.2520



¹³C NMR Spectra for Compound 3h in CDCl₃

Acquisition Time (sec)	1.0434	Comment	Pitambar	Date	17 Nov 2015 06:53:26
Date Stamp	16 Nov 2015 14:22:32				
File Name	F:\pitambar\Research\3rd methodology\Amide\NMR\meldrom\meldrom_\alpha-toluicMon3ECx400#005.CARBON-3.jdf				
Nucleus	¹³ C	Number of Transients	1200	Frequency (MHz)	100.53
Owner	delta	Points Count	26214	Original Points Count	26214
Solvent	CHLOROFORM-d	Spectrum Offset (Hz)	10039.3311	Origin	ECX 400
Temperature (degree C)	23.400	Spectrum Type	STANDARD	Receiver Gain	60.00
				Sweep Width (Hz)	25124.29

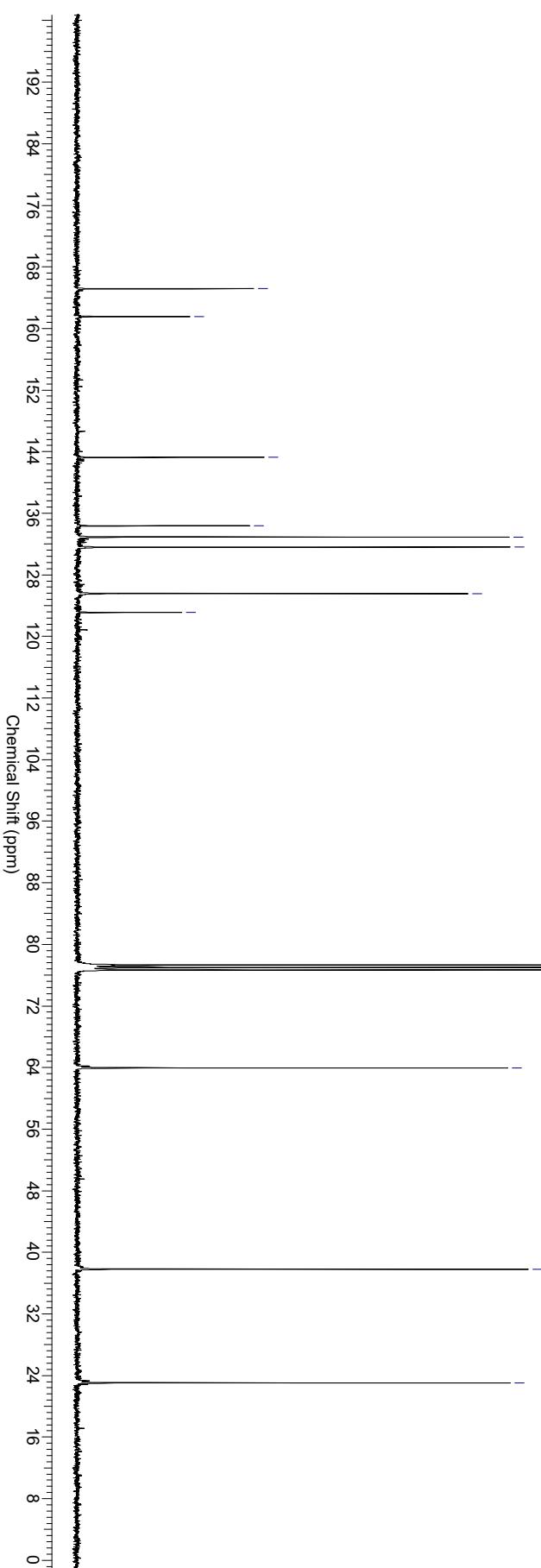
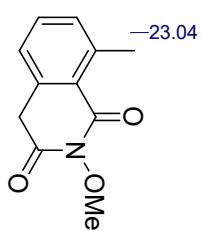
—165.16
—161.55

—143.26
—134.36
—132.92
—131.59

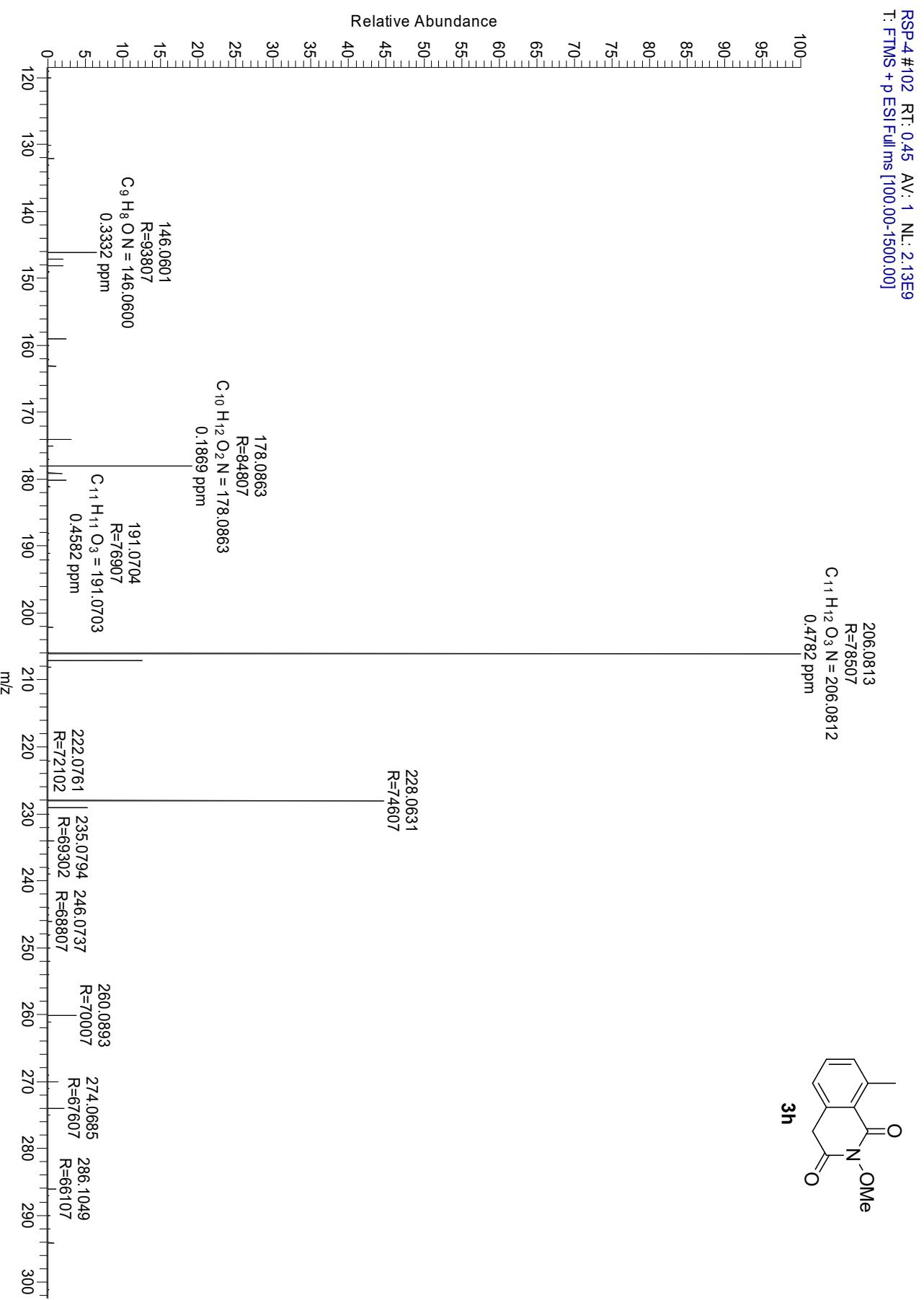
—125.57
—123.12

—77.00
—63.96

—37.80

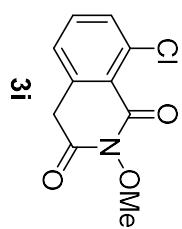
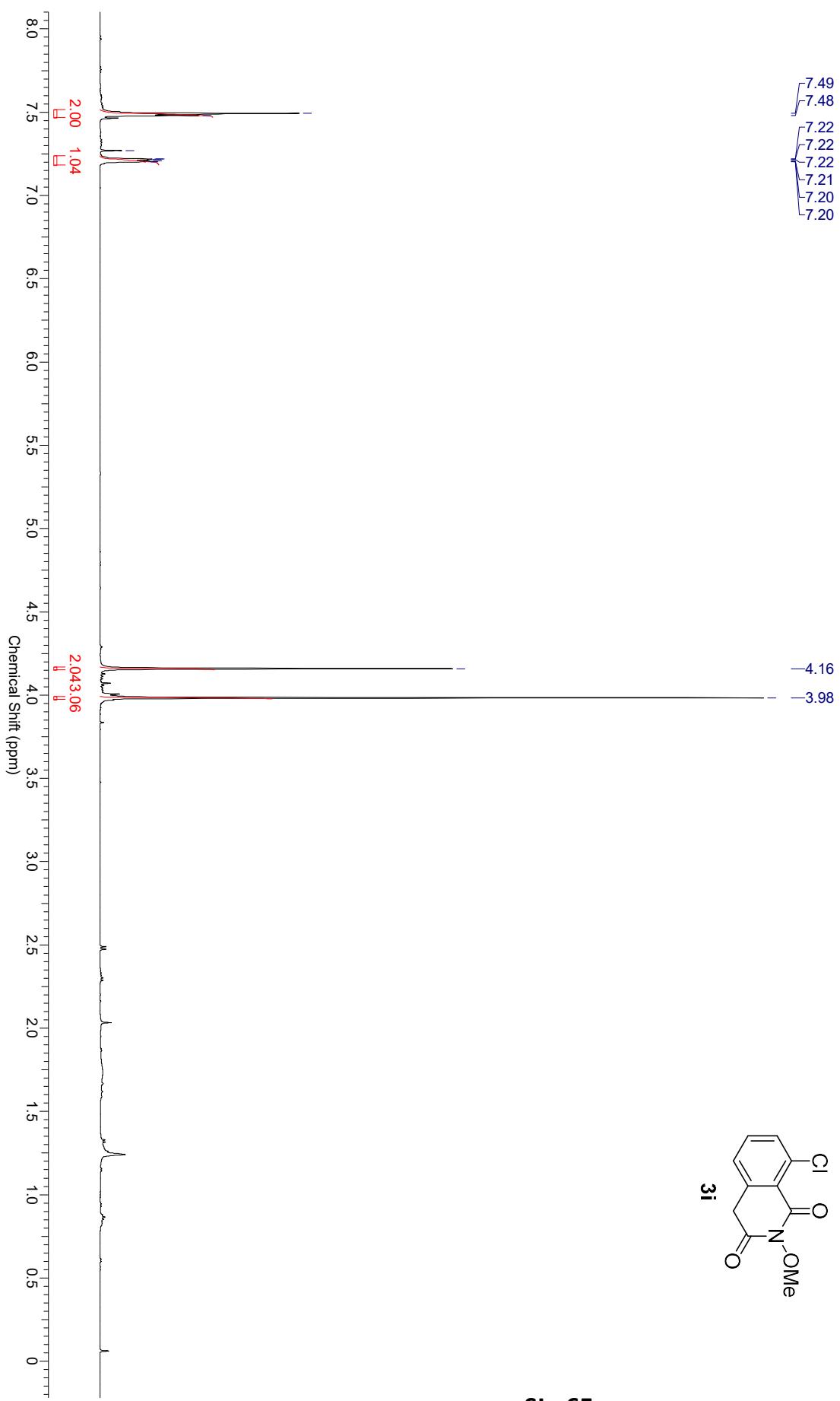


HRMS Spectra for Compound **3h** in MeOH



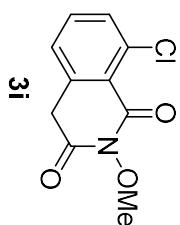
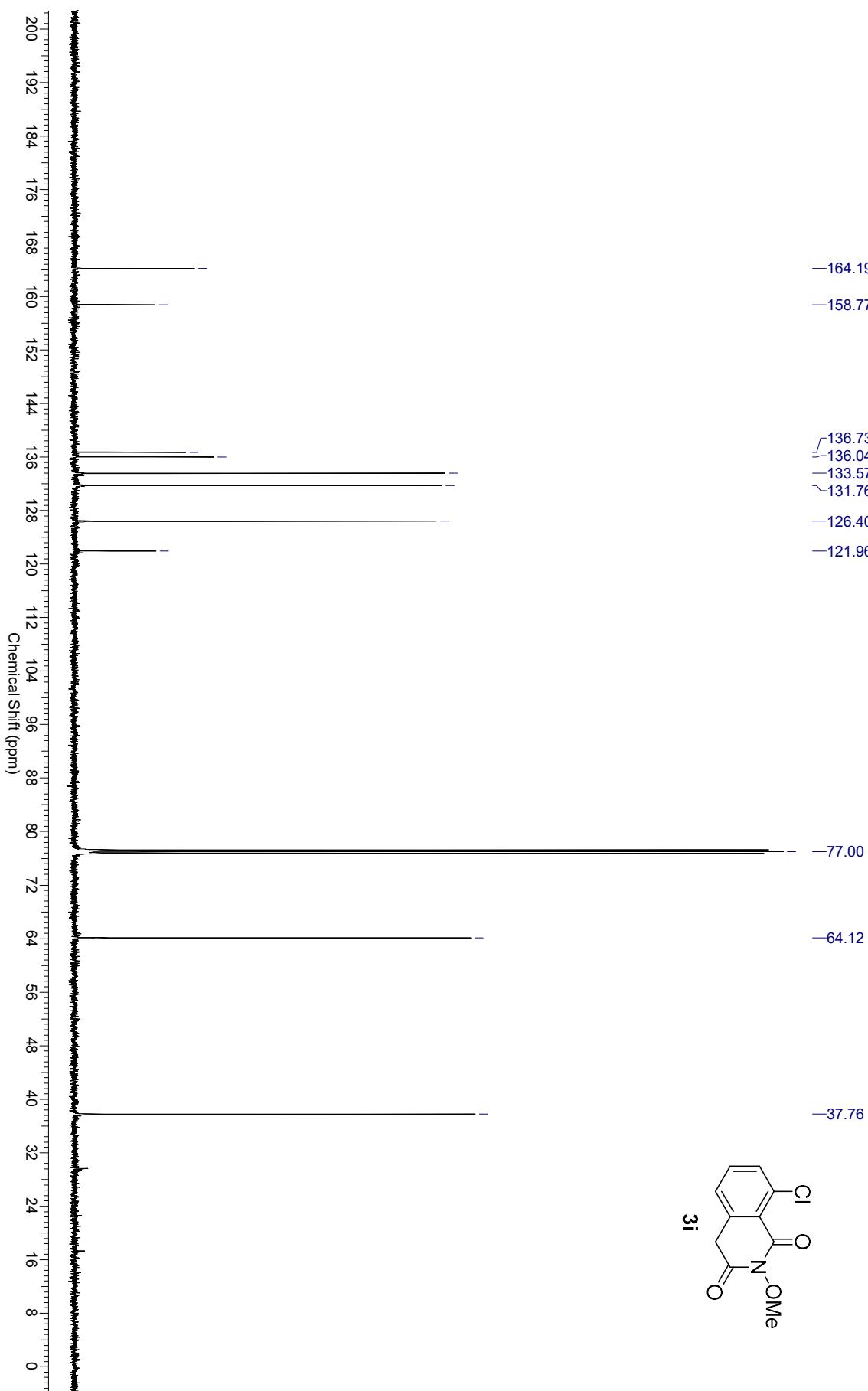
¹H NMR Spectra for Compound 3i in CDCl₃

Acquisition Time (sec)	1.6000	Comment	Ravindra 1H	Date	26 Nov 2015 11:54:56
Date Stamp	26 Nov 2015 11:54:56			File Name	\agnmr\data\AV_500\Nov_15_500\Thu4av500#004\1PDATA1\1r
Frequency (MHz)	500.13	Nucleus	1H	Number of Transients	64
Original Points Count	16000	Owner	nmr	Points Count	32768
Receiver Gain	181.00	SW(cyclical) (Hz)	10000.00	Pulse Sequence	zg30
Spectrum Offset (Hz)	2491.9937	Spectrum Type	STANDARD	Sweep Width (Hz)	9999.70
				Temperature (degree C)	25.000



¹³C NMR Spectra for Compound **3i** in CDCl₃

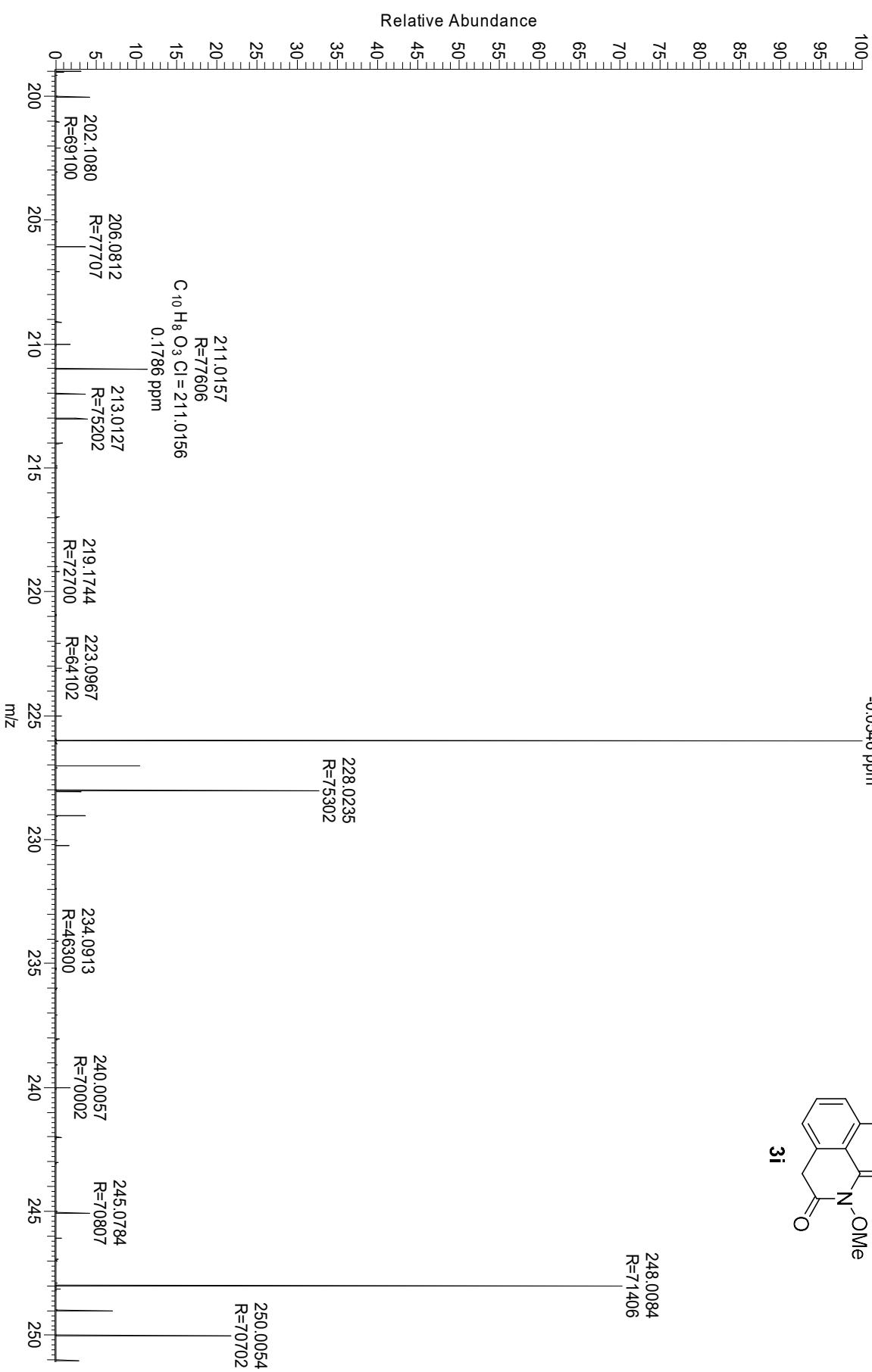
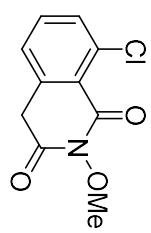
Acquisition Time (sec)	0.6554	Comment	13C	Date	26 Nov 2015 12:41:52
Date Stamp	26 Nov 2015 12:41:52	File Name	lagnlnmr_dataAV_500\Nov_15_500\Thu4av500#004\3\pdata1\1\1r		
Frequency (MHz)	125.76	Nucleus	13C	Number of Transients	695
Original Points Count	20480	Owner	nmr	Points Count	32768
Receiver Gain	1440.00	SW(cyclical) (Hz)	31250.00	Pulse Sequence	zgpg30
Spectrum Offset (Hz)	12554.3857	Spectrum Type	STANDARD	Solvent	CHLOROFORM-d
				Temperature (degree C)	25.100



HRMS Spectra for Compound 3i in MeOH

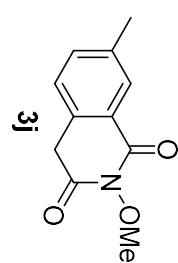
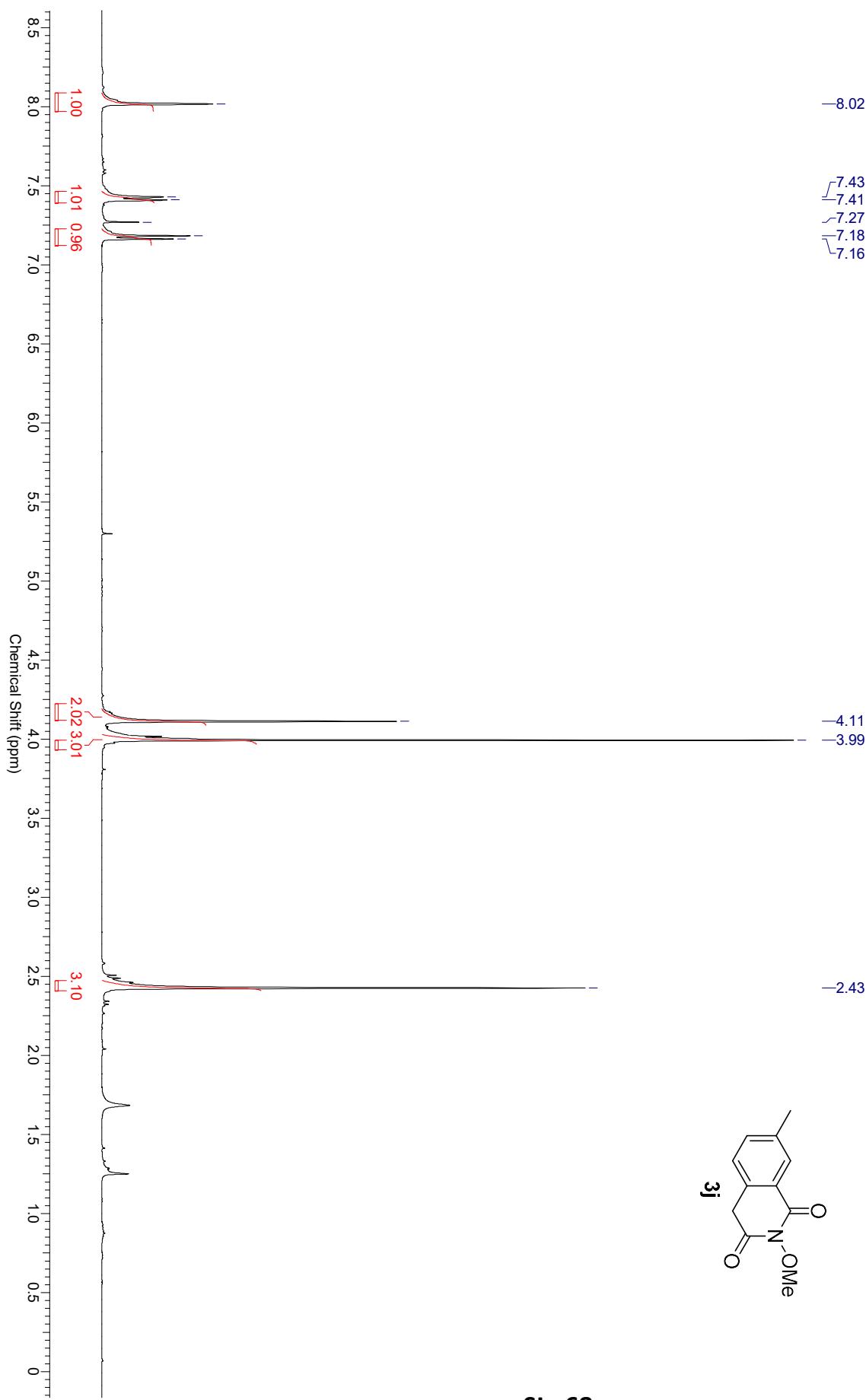
RSP-12 #102 RT: 0.45 AV: 1 NL: 1.13E8
T: FTMS + pESI Full ms [100.00-1500.00]

226.0265
R=75106
 $C_{10}H_9O_3NCl = 226.0265$
-0.0546 ppm



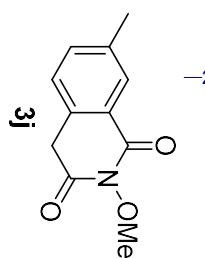
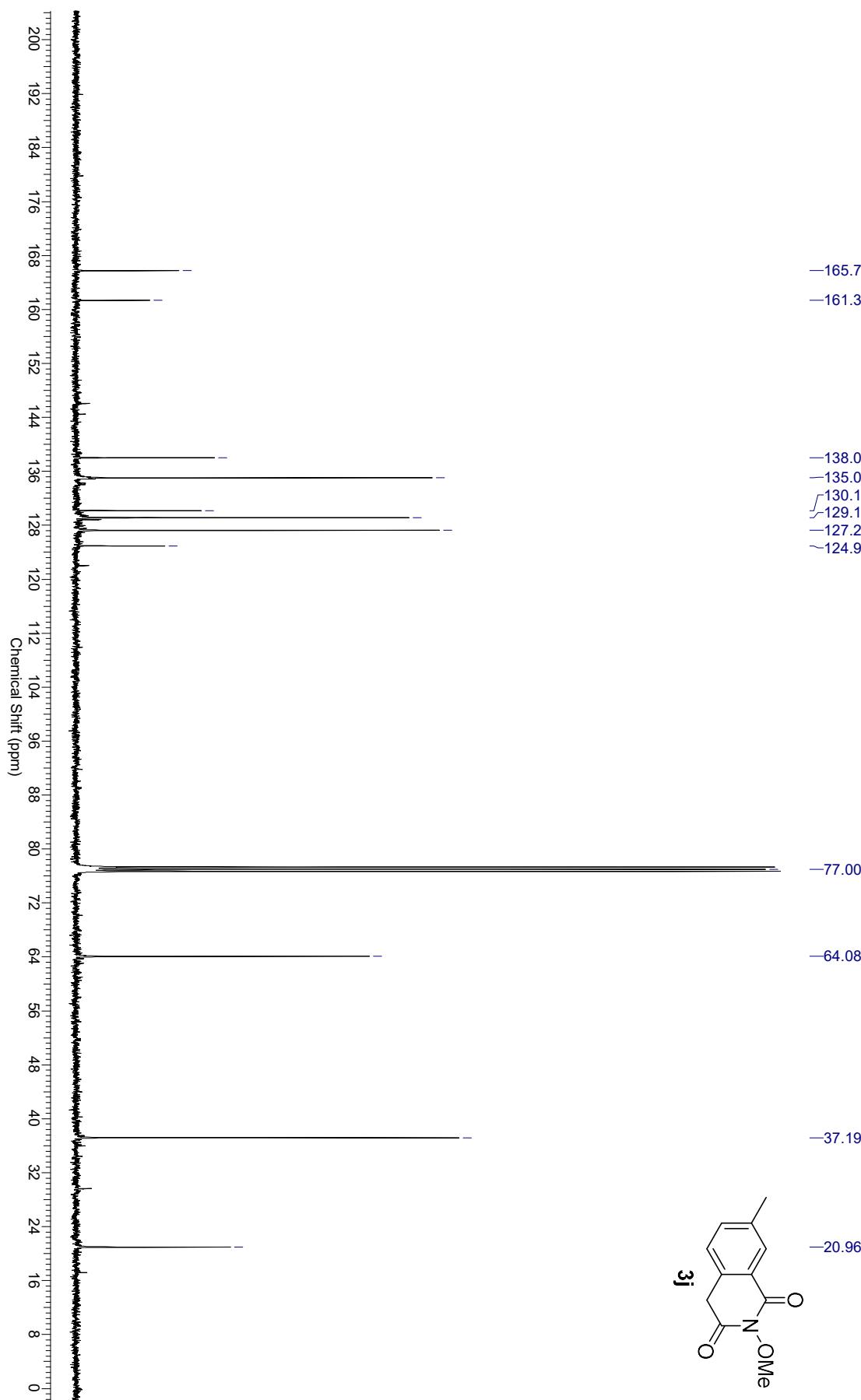
¹H NMR Spectra for Compound 3j in CDCl₃

Acquisition Time (sec)	2.0447	Comment	Ravindra 1H	Date	22 Nov 2015 11:46:16
Date Stamp	22 Nov 2015 11:46:16	File Name	\agnl\nmr\data\AV400Nov_15_400\Sun4av400#0041\PDATAl1\1r		
Frequency (MHz)	400.13	Nucleus	¹ H		
Original Points Count	16384	Owner	Administrator	Points Count	32768
Receiver Gain	406.00	SW(cyclical) (Hz)	8012.82	Pulse Sequence	zg30
Spectrum Offset (Hz)	2395.7949	Spectrum Type	STANDARD	Sweep Width (Hz)	8012.58
				Temperature (degree C)	22.900



¹³C NMR Spectra for Compound **3j** in CDCl₃

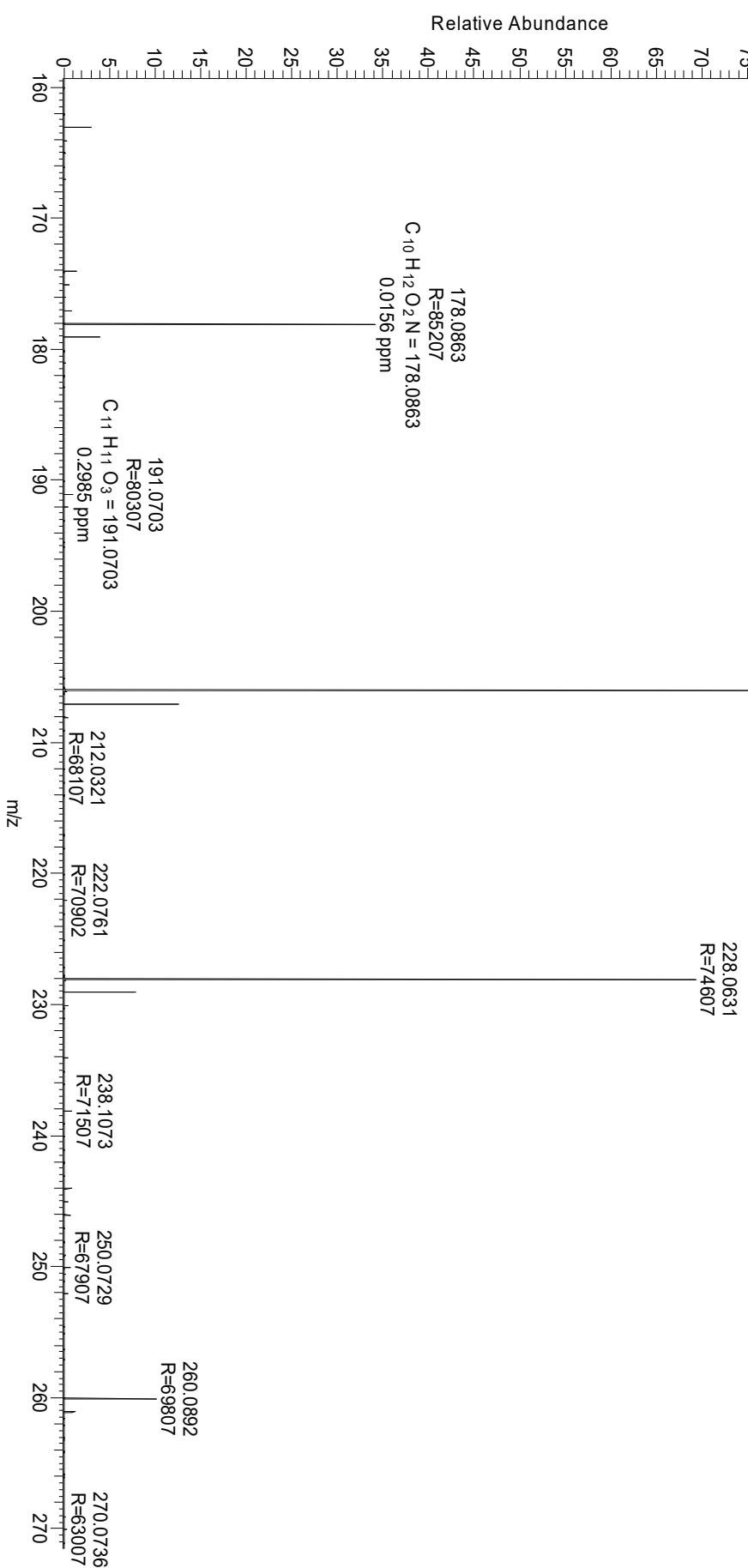
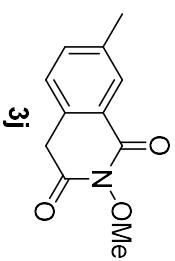
Acquisition Time (sec)	0.6488	Comment	13C	Date	22 Nov 2015 13:58:32
Date Stamp	22 Nov 2015 13:58:32	File Name	\agilmr_data\AV400Nov_15_400\Sun4a\#004\3\PDAT\11r		
Frequency (MHz)	100.61	Nucleus	13C	Number of Transients	3382
Original Points Count	16384	Owner	root	Points Count	32768
Receiver Gain	2050.00	SW(cyclical) (Hz)	25252.53	Solvent	CHLOROFORM-d
Spectrum Offset (Hz)	10056.4238	Spectrum Type	STANDARD	Sweep Width (Hz)	25251.75
				Temperature (degree C)	23.100



HRMS Spectra for Compound 3j in MeOH

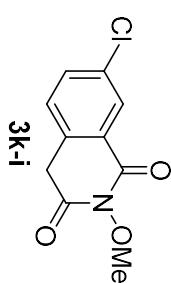
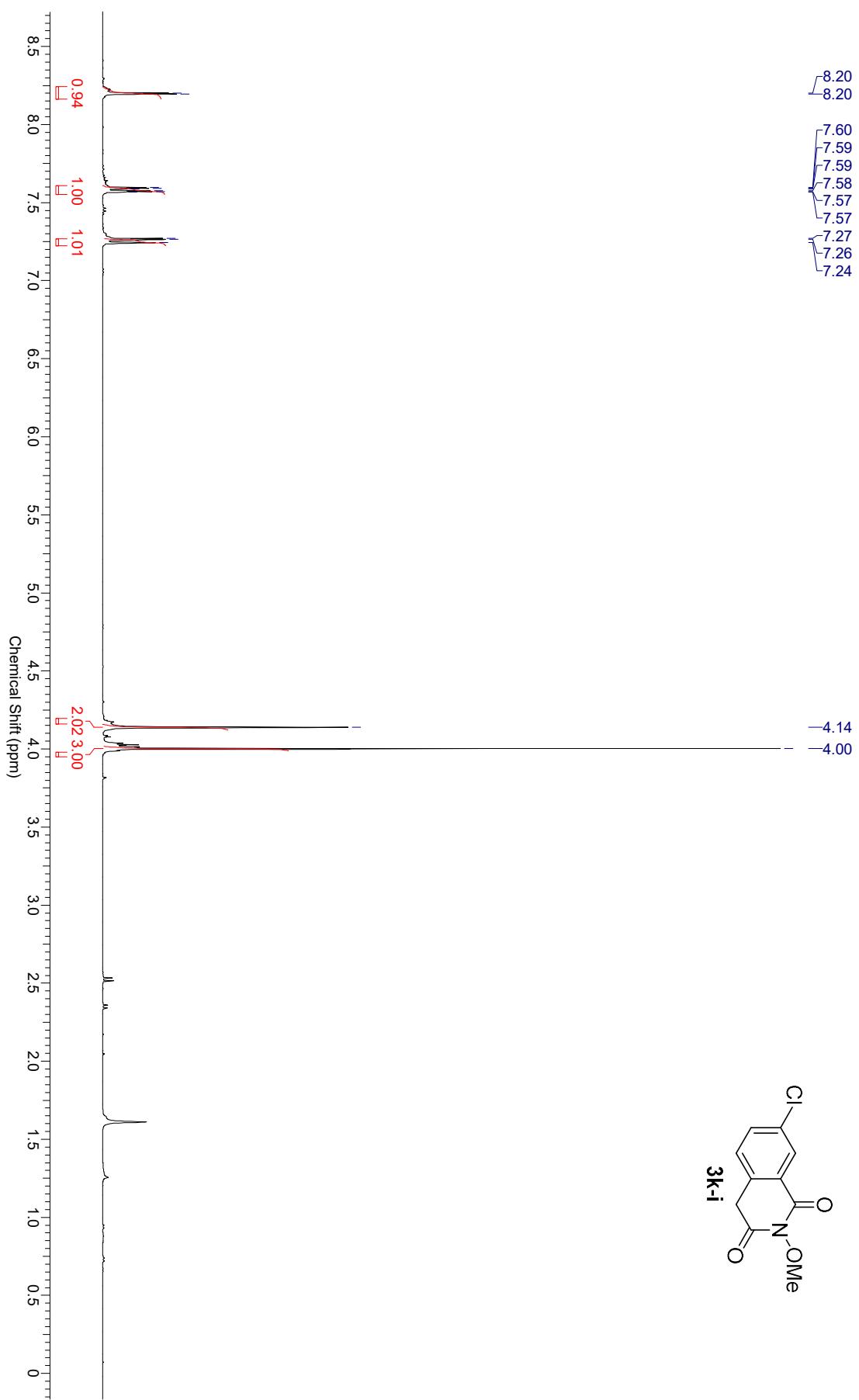
RSP-6#100 RT: 0.44 AV: 1 NL: 1.41E9
T: FTMS + p ESI Full ms [100.00-1500.00]

206.0813
R=78507
 $C_{11}H_{12}O_3N = 206.0812$
0.4782 ppm



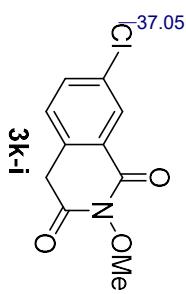
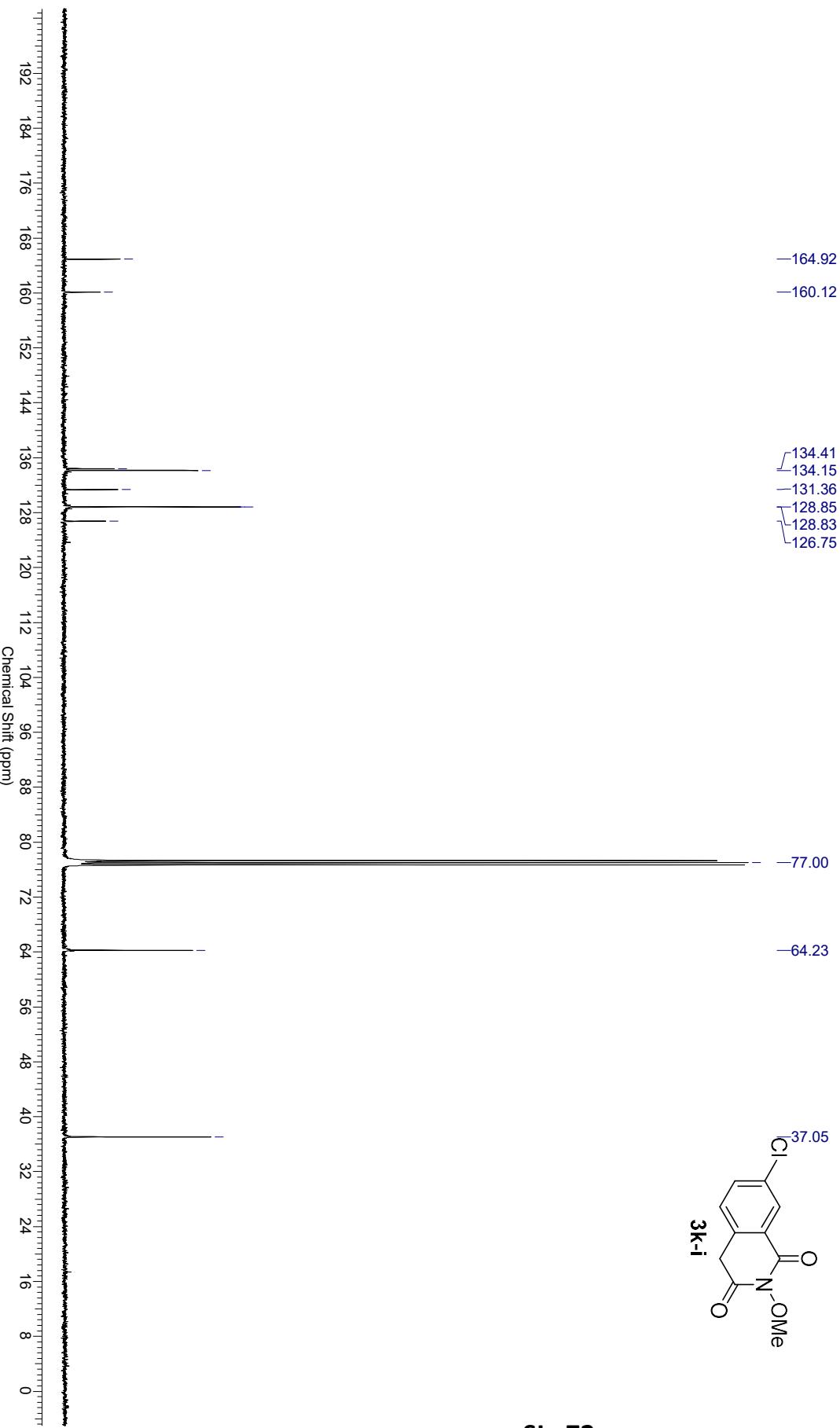
¹H NMR Spectra for Compound 3k-1 in CDCl₃

Acquisition Time (sec)	2.1838	Comment	Pitambar	Date	22 Nov 2015 16:04:01
Date Stamp	22 Nov 2015 05:59:56			File Name	\lagnihmr\data\IEOL_400\2015\Nov_2015_LiquidFlt3ECX400#021_PROTON-3.jdf
Frequency (MHz)	399.78	Nucleus	1H	Number of Transients	64
Original Points Count	13107	Owner	delta	Points Count	13107
Receiver Gain	44.00	Solvent	CHLOROFORM-d	Origin	ECX 400
Spectrum Type	STANDARD	Sweep Width (Hz)	6001.85	Pulse Sequence	single_pulse.ex2
		Temperature (degree C)	23.600	Spectrum Offset (Hz)	2007.4668



¹³C NMR Spectra for Compound 3k-1 in CDCl₃

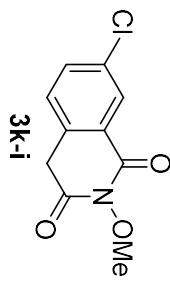
Acquisition Time (sec)	1.0434	Comment	Ptambar	Date	23 Nov 2015 00:12:42
Date Stamp	22 Nov 2015 07:34:09			File Name	\lagnmr_data\lagnmr_EOL_400\2015Nov_2015_Liquid\Fl3ECX400#021_CARBON-3.jdf
Frequency (MHz)	100.53	Nucleus	¹³ C	Number of Transients	2400
Original Points Count	26214	Owner	delta	Points Count	26214
Receiver Gain	60.00	Solvent	CHLOROFORM-d	Pulse Sequence	single pulse dec
Spectrum Type	STANDARD	Sweep Width (Hz)	25124.29	Temperature (degree C)	24.000
		Spectrum Offset (Hz)	10043.1650		



HRMS Spectra for Compound 3k-1 in MeOH

RSP-7#107 RT: 0.47 AV: 1 NL: 4.18E7
T: FTMS + p ESI[Full.ms [100.00-1500.00]

226.0267
R=76006
 $C_{10}H_9O_3NCl = 226.0265$
0.5530 ppm

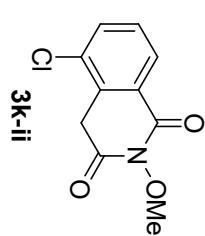
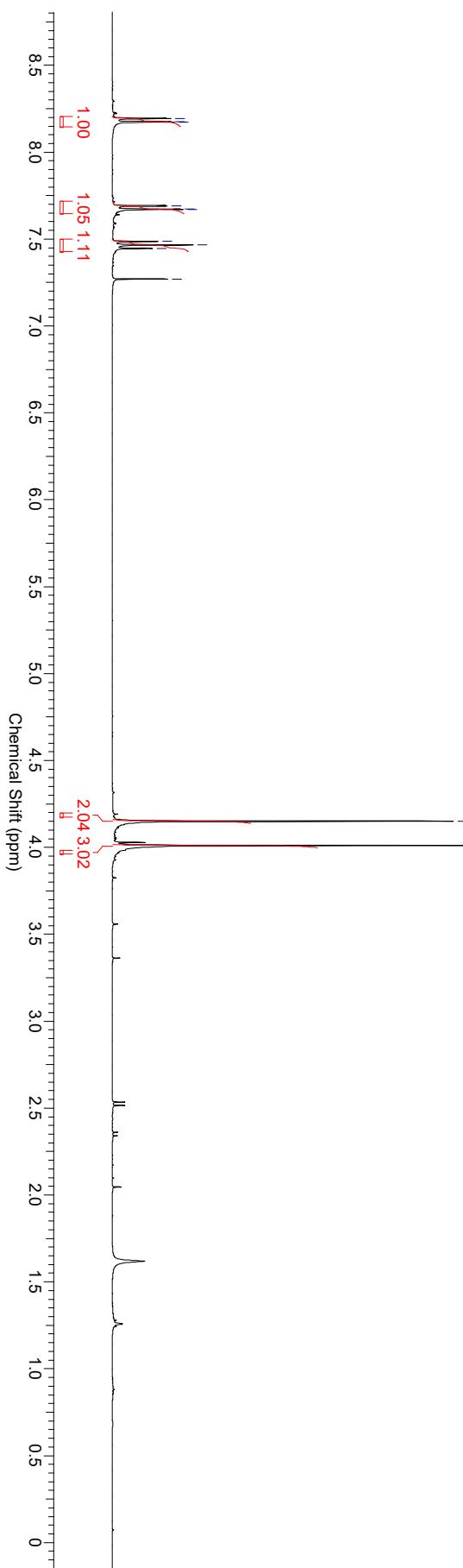


SI 73

¹H NMR Spectra for Compound 3k-2 in CDCl₃

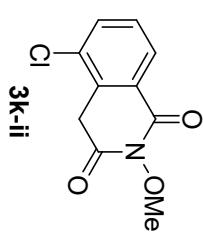
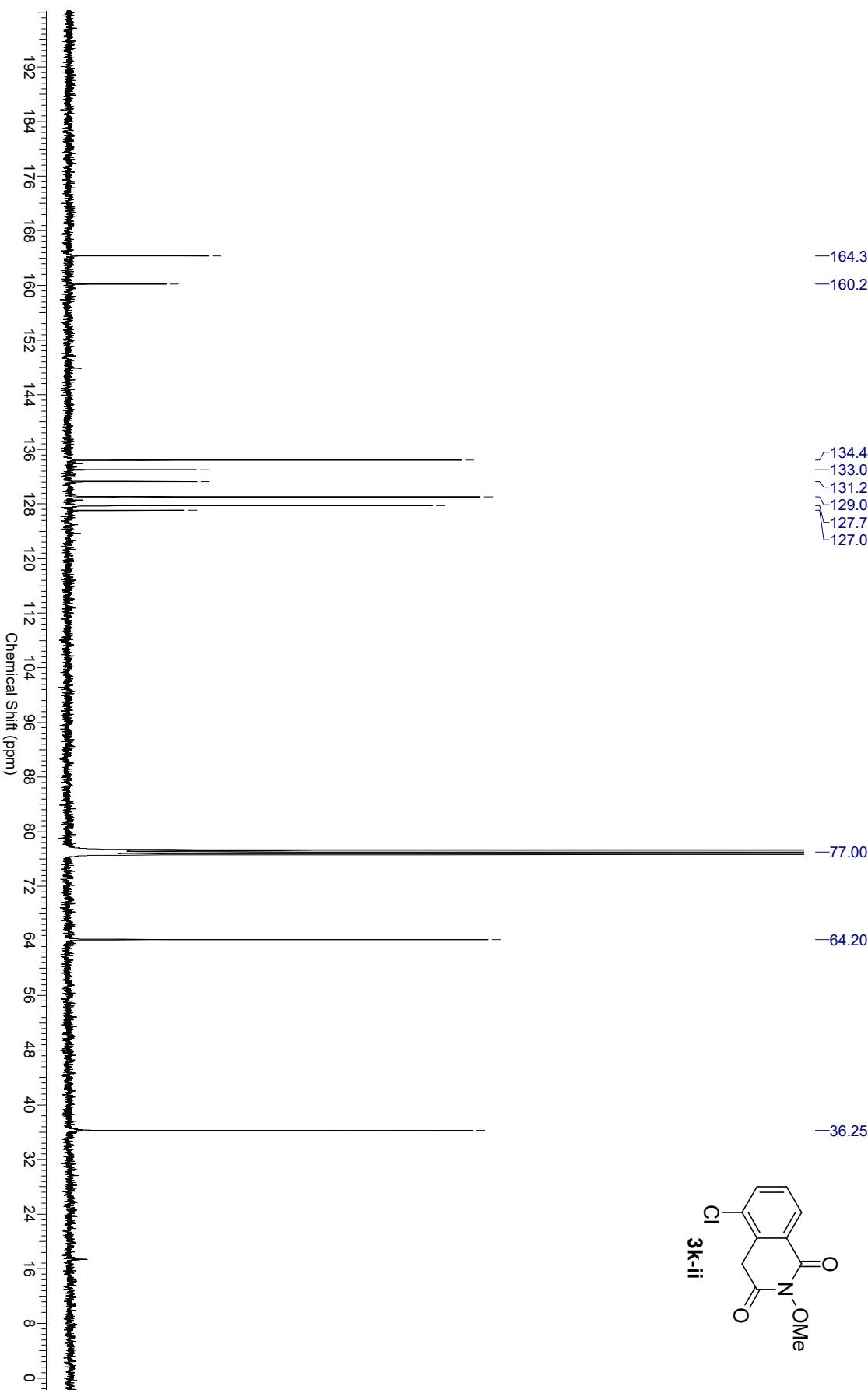
Acquisition Time (sec)	2.1838	Comment	Pitambar	Date	23 Nov 2015 05:29:05
Date Stamp	22 Nov 2015 13:37:17			File Name	\agnmr_data\EL_400\2015\Nov_2015_Liquid\TriECX400\#024_PROTON-3.jdf
Frequency (MHz)	399.78	Nucleus	1H	Number of Transients	64
Original Points Count	13107	Owner	delta	Points Count	13107
Receiver Gain	44.00	Solvent	CHLOROFORM-d	Pulse Sequence	single_pulse.ex2
Spectrum Type	STANDARD	Sweep Width (Hz)	6001.85	Temperature (degree C)	23.700
				Spectrum Offset (Hz)	2007.4668

8.19
8.18
8.17
7.69
7.67
7.67
7.49
7.47
7.45
~7.27
—4.15
—4.01



¹³C NMR Spectra for Compound 3k-2 in CDCl₃

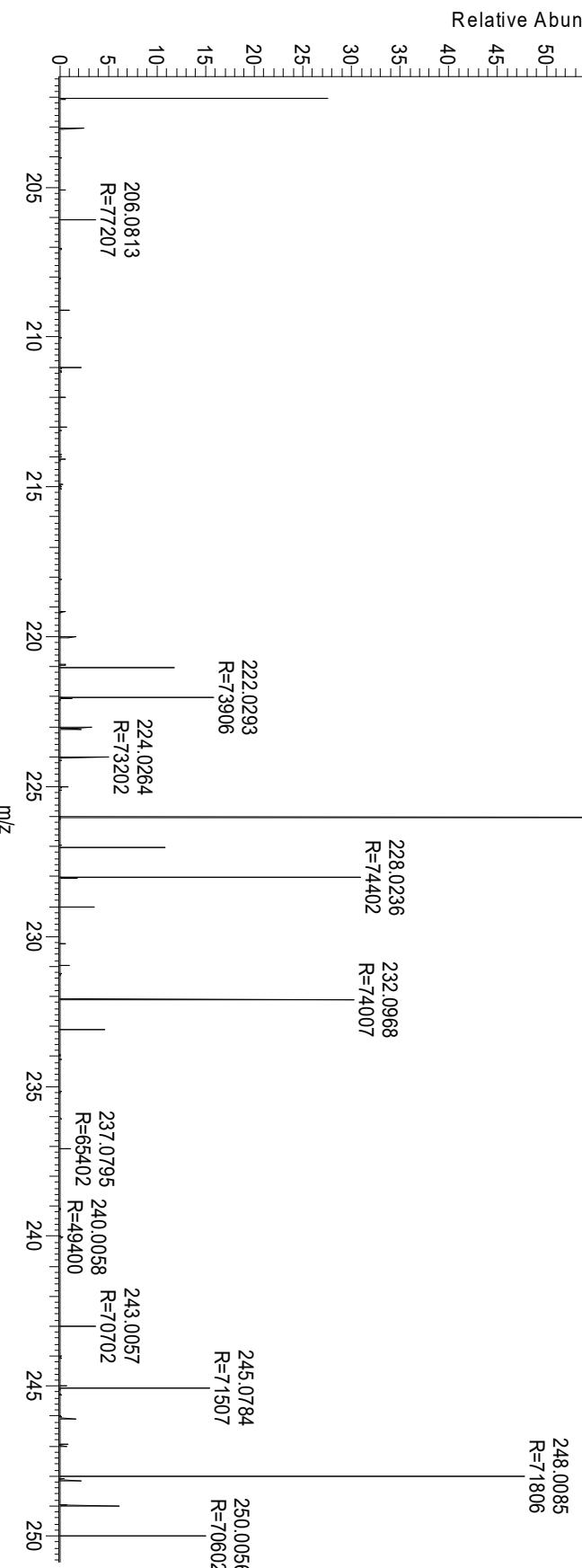
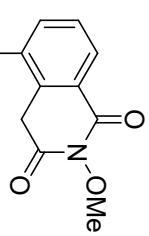
Acquisition Time (sec)	1.0434	Comment	Pitambar	Date	23 Nov 2015 08:13:10
Date Stamp	22 Nov 2015 15:34:38			File Name	\agnmr_data\JEOL_400\2015\Nov_2015_Liquid\Fl3ECX400#024_CARBON-3.jdf
Frequency (MHz)	100.53	Nucleus	¹³ C	Number of Transients	3000
Original Points Count	26214	Owner	delta	Points Count	26214
Receiver Gain	60.00	Solvent	CHLOROFORM-d	Pulse Sequence	ECX 400
Spectrum Type	STANDARD	Sweep Width (Hz)	25124.29	Temperature (degree C)	23.900
		Spectrum Offset (Hz)	10043.1650		



HRMS Spectra for Compound 3k-2 in MeOH

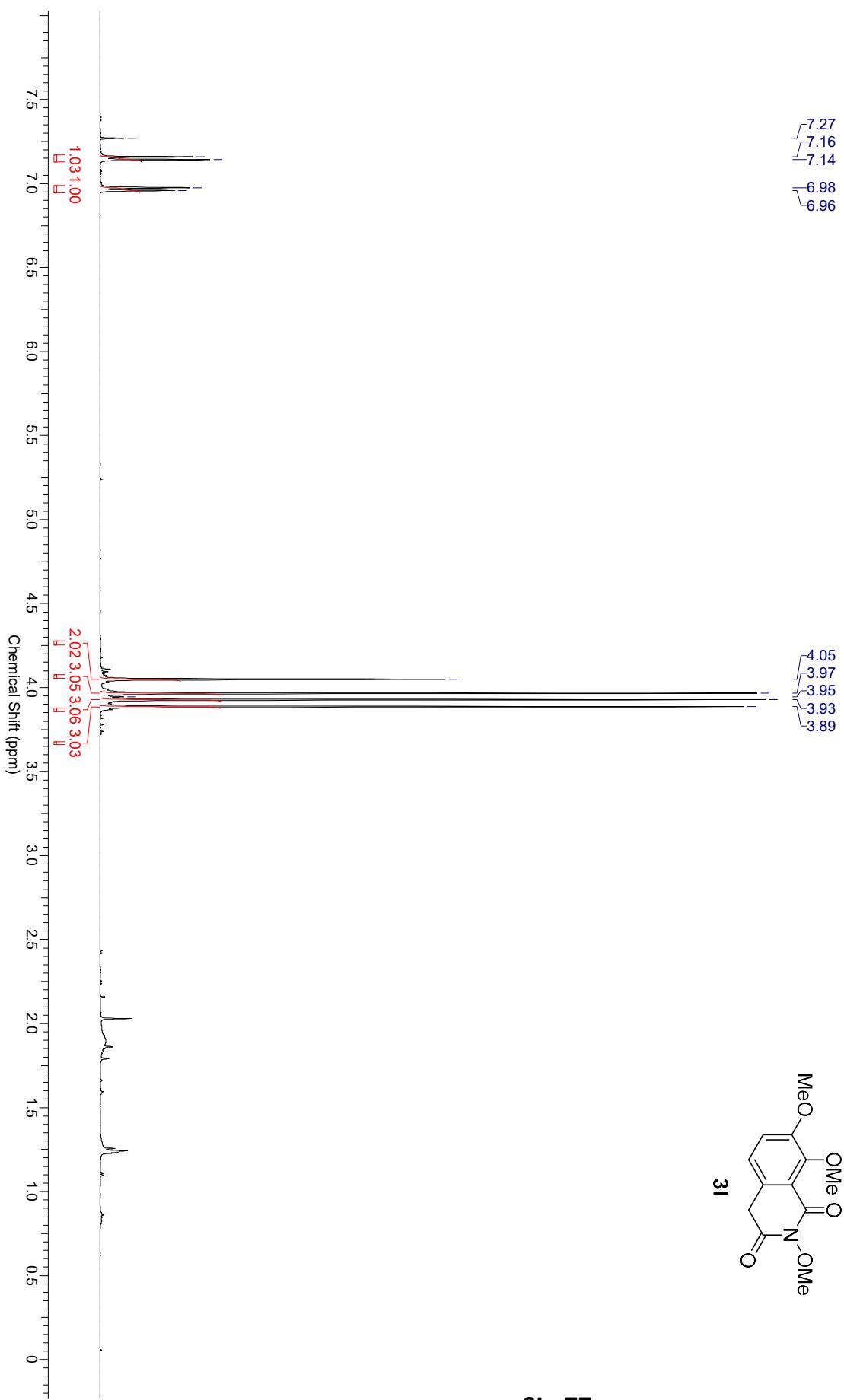
RSP-8#111 RT: 0.49 AV: 1 NL: 5.93E7
T: FTMS +p ESI Full ms [100.00-1500.00]

226.0266
R=75206
 $C_{10}H_9O_3NCl = 226.0265$
0.0804 ppm



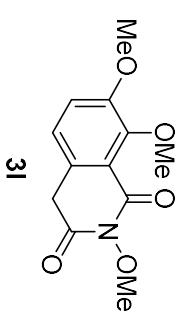
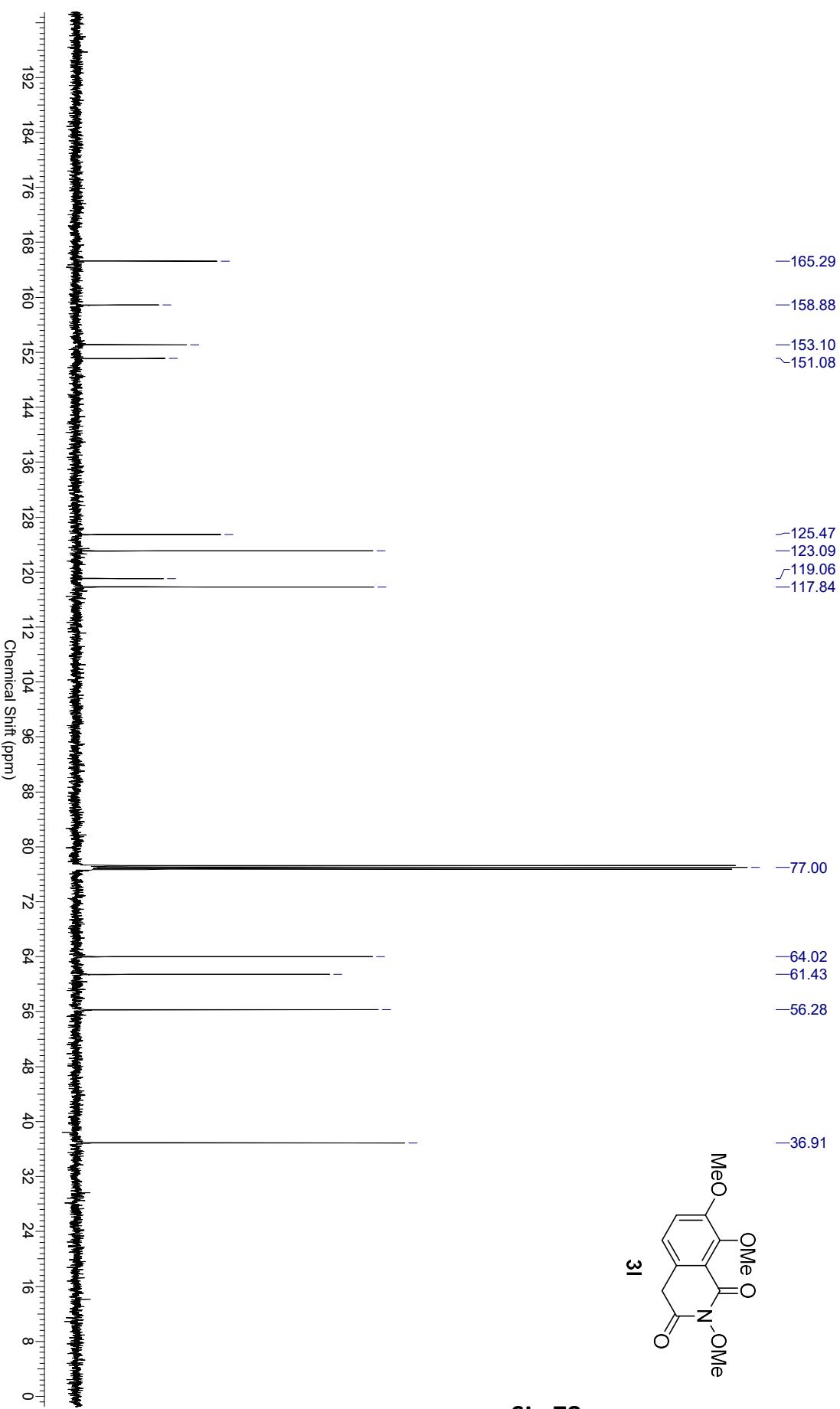
¹H NMR Spectra for Compound 3l in CDCl₃

Acquisition Time (sec)	1.6000	Comment	Ravindra 1H	Date	26 Nov 2015 11:35:44
Date Stamp	26 Nov 2015 11:35:44			File Name	\agnmr\data\AV_500Nov_15_500\Thu4av500#0031\PDAT\1\1r
Frequency (MHz)	500.13	Nucleus	1H	Number of Transients	64
Original Points Count	16000	Owner	nmr	Origin	spect
Receiver Gain	181.00	SW(cyclical) (Hz)	10000.00	Pulse Sequence	zg30
Spectrum Offset (Hz)	2491.9937	Spectrum Type	STANDARD	Sweep Width (Hz)	9999.70
				Temperature (degree C)	24.900



¹³C NMR Spectra for Compound **3l** in CDCl₃

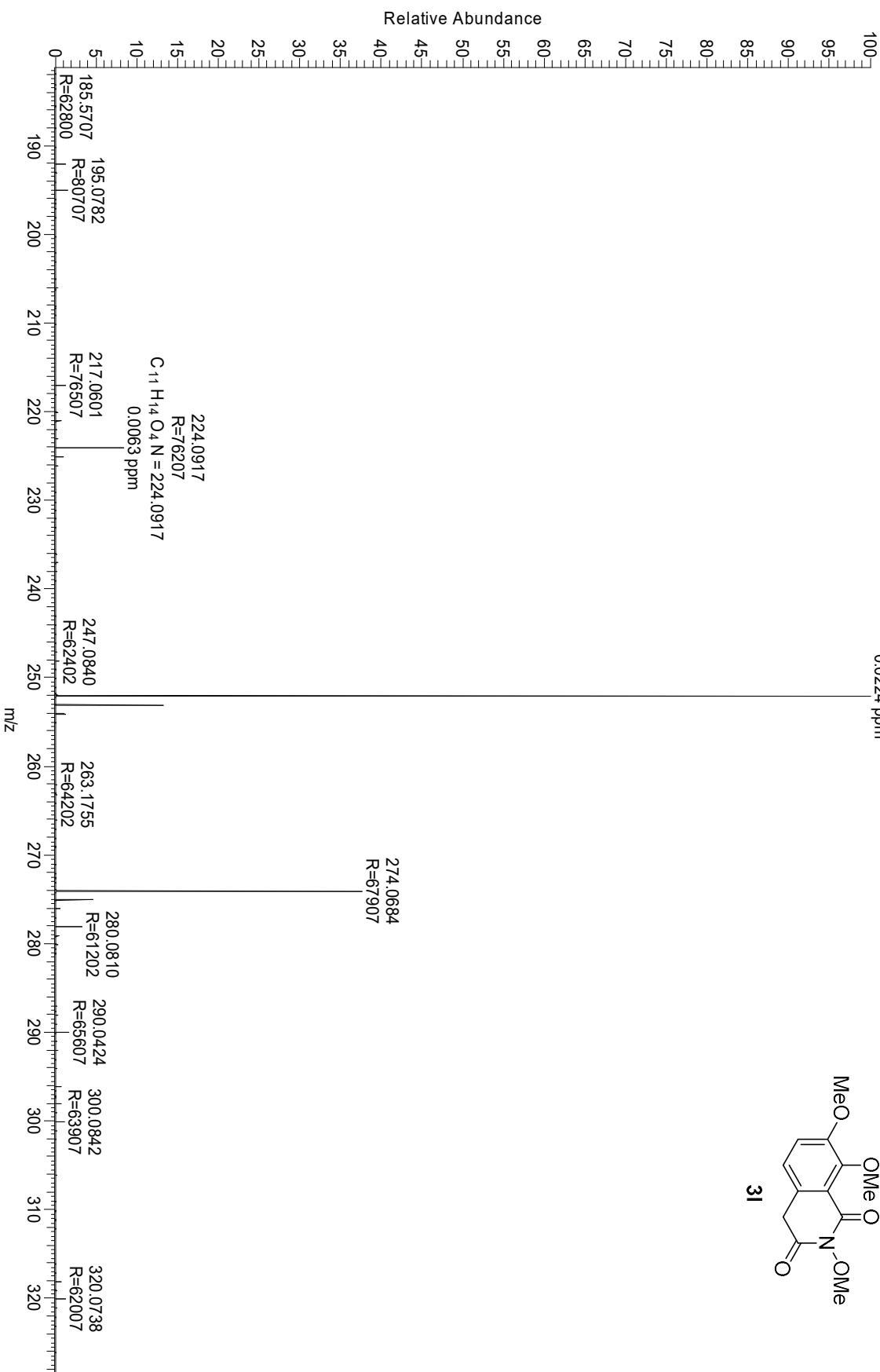
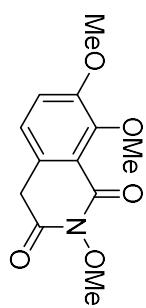
Acquisition Time (sec)	0.6554	Comment	13C	Date	26 Nov 2015 11:42:08
Date Stamp	26 Nov 2015 11:42:08	File Name	\agnmr_dataAV_500Nov_15_500\Thu4av500#003\3\PDATA\1\1r	Nucleus	13C
Frequency (MHz)	125.76	Number of Transients	161	Origin	spect
Original Points Count	20480	Owner	nmr	Pulse Sequence	zpg30
Receiver Gain	1440.00	SW(cyclical) (Hz)	31250.00	Solvent	CHLOROFORM-d
Spectrum Offset (Hz)	12553.4326	Spectrum Type	STANDARD	Sweep Width (Hz)	31249.05
				Temperature (degree C)	25.000



HRMS Spectra for Compound 3l in MeOH

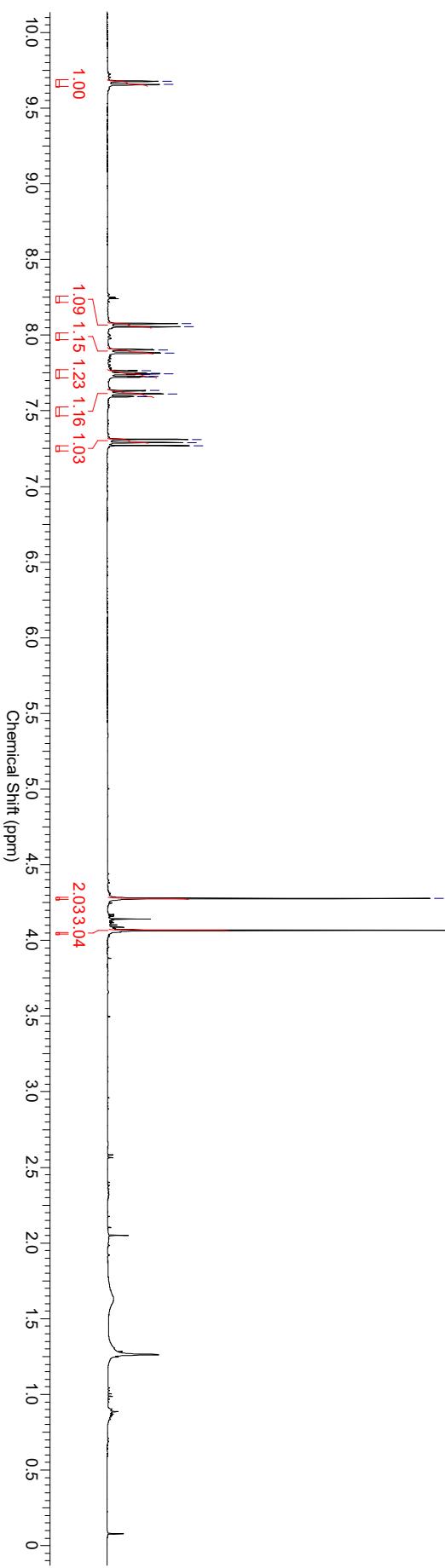
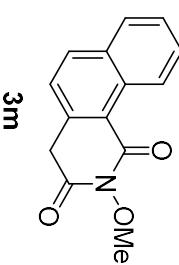
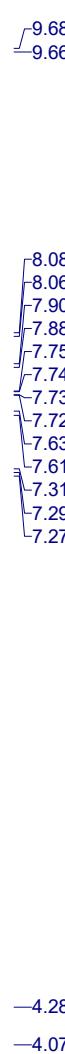
RSP-11 #95 RT: 0.42 AV: 1 NL: 3.47E9
T: FTMS + p ESI[Full ms [100.00-1500.00]]

252.0867
R=71307
 $C_{12}H_{14}O_5N = 252.0866$
0.0224 ppm



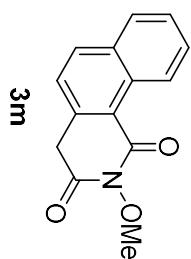
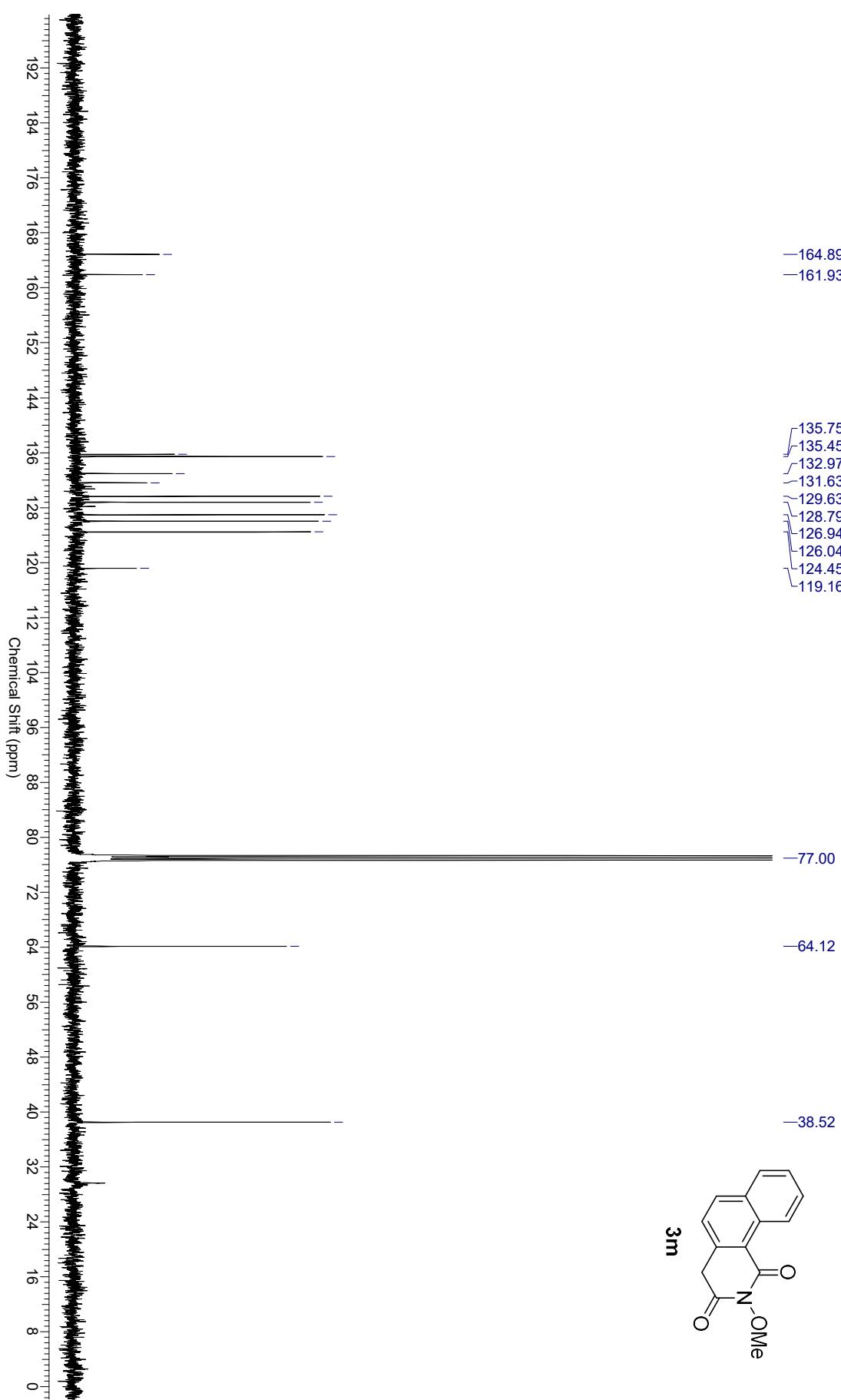
¹H NMR Spectra for Compound 3m in CDCl₃

Acquisition Time (sec)	2.1838	Comment	Milind	Date	21 Nov 2015 05:01:45
Date Stamp	20 Nov 2015 13:09:55			File Name	\agnlnmr\data\JESOL\400\2015\Nov_2015_LiquidFl3ECX400#004_PROTON-3.jdf
Frequency (MHz)	399.78	Nucleus	1H	Number of Transients	64
Original Points Count	13107	Owner	delta	Points Count	13107
Receiver Gain	42.00	Solvent	CHLOROFORM-d	Pulse Sequence	single_pulse_ex2
Spectrum Type	STANDARD	Sweep Width (Hz)	6001.85	Spectrum Offset (Hz)	2007.4668
		Temperature (degree C)	23.600		



¹³C NMR Spectra for Compound 3m in CDCl₃

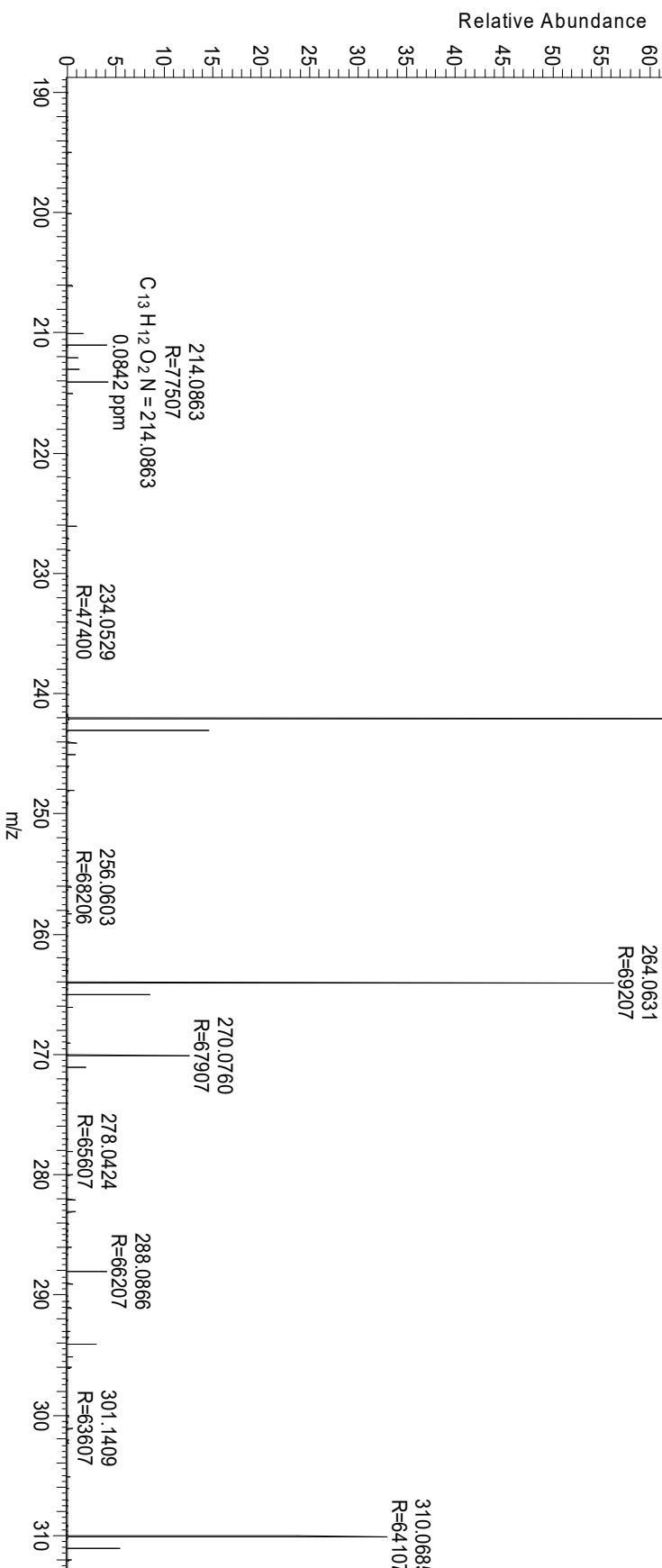
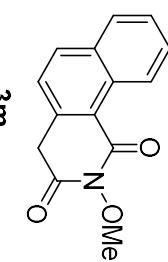
Acquisition Time (sec)	1.0434	Comment	Milind	Date	21 Nov 2015 05:48:54
Date Stamp	20 Nov 2015 13:41:13			File Name	\Vagin\NMR_data\JEOL_400\2015\Nov_2015_Liquid\fr13ECX400\#004_CARBON-3.jdf
Frequency (MHz)	100.53	Nucleus	¹³ C	Number of Transients	800
Original Points Count	26214	Owner	delta	Points Count	26214
Receiver Gain	60.00	Solvent	CHLOROFORM-d	Pulse Sequence	single_pulse_dec
Spectrum Type	STANDARD	Sweep Width (Hz)	25124.29	Temperature (degree C)	23.800



HRMS Spectra for Compound 3m in MeOH

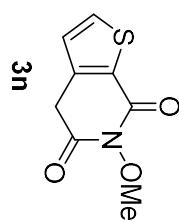
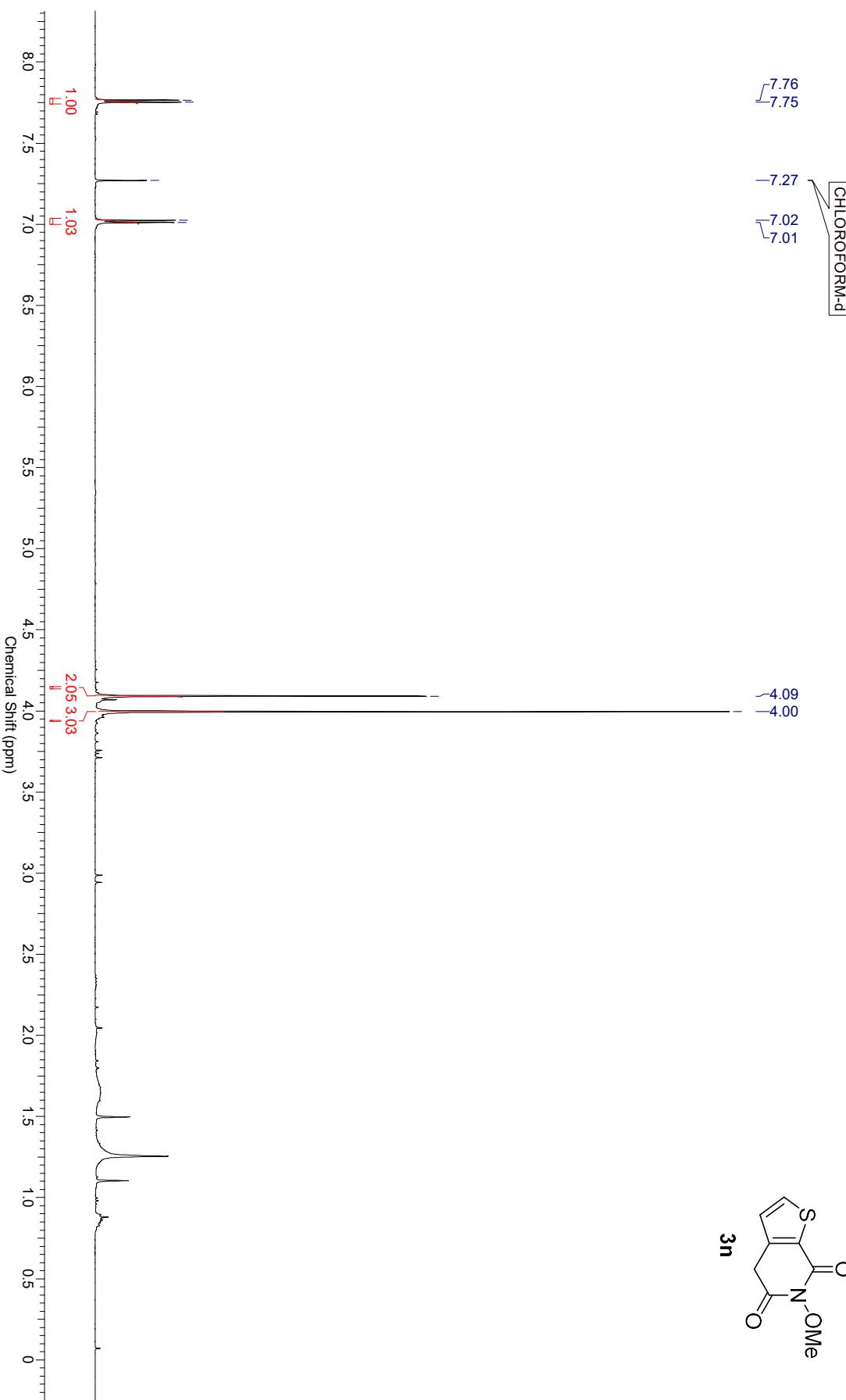
RSP-5#104 RT: 0.46 AV: 1 NL: 6.47E8
T: FTMS + p ESI Full ms [100.00-1500.00]

242.0812
R=71907
C₁₄H₁₂O₃N = 242.0812
0.0919 ppm



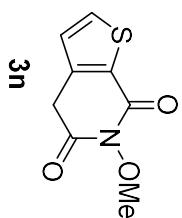
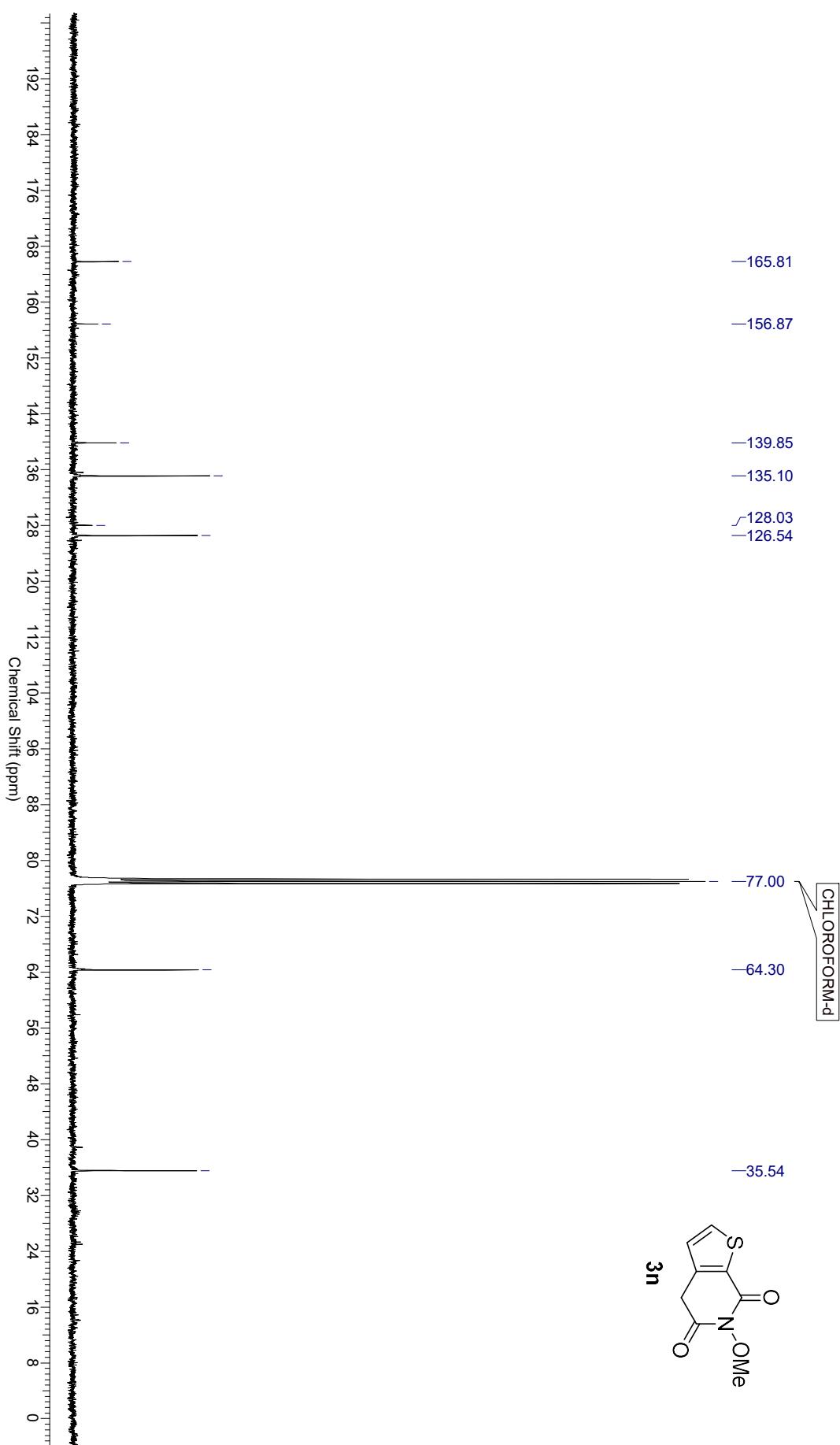
¹H NMR Spectra for Compound **3n** in CDCl₃

Acquisition Time (sec)	2.1838	Comment	Ravinda	Date	12 Jan 2016 12:26:12
Date Stamp	12.Jan.2016 02:18:38			File Name	\172.16.2.4\mr_data\Jeol_400_new\Liquid Jan 16\Mon3ECX400#002.PROTON-3.idf
Frequency (MHz)	399.78	Nucleus	1H	Number of Transients	44
Original Points Count	13107	Owner	delta	Points Count	13107
Receiver Gain	42.00	Solvent	CHLOROFORM-d	Pulse Sequence	single pulse,ex2
Spectrum Type	STANDARD	Sweep Width (Hz)	6001.85	Spectrum Offset (Hz)	2007.4668
				Temperature (degree C)	22.400



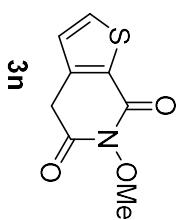
¹³C NMR Spectra for Compound 3n in CDCl₃

Acquisition Time (sec)	1.0434	Comment	Ravindra	Date	14 Jan 2016 15:54:45
Date Stamp	14 Jan 2016 05:31:22				
File Name	\172.16.240nmr\4002016_JEOL_400JAN_16_JEOL_400Liquid Jan 16\Wed3ECX400#018_CARBON-3.jdf				
Frequency (MHz)	100.53	Nucleus	13C	Number of Transients	1200
Original Points Count	26214	Owner	delta	Points Count	26214
Receiver Gain	60.00	Solvent	CHLOROFORM-d	Pulse Sequence	single_pulse_dec
Sweep Width (Hz)	25124.29	Temperature (degree C)	22.700	Spectrum Offset (Hz)	10042.2061
				Spectrum Type	STANDARD



HRMS Spectra for Compound 3n in MeOH

RS-1 151230135829 #90 RT: 0.40 AV: 1 NL: 4.34E6
 T: FTMS + pESI[Full ms [100.00-1500.00]]



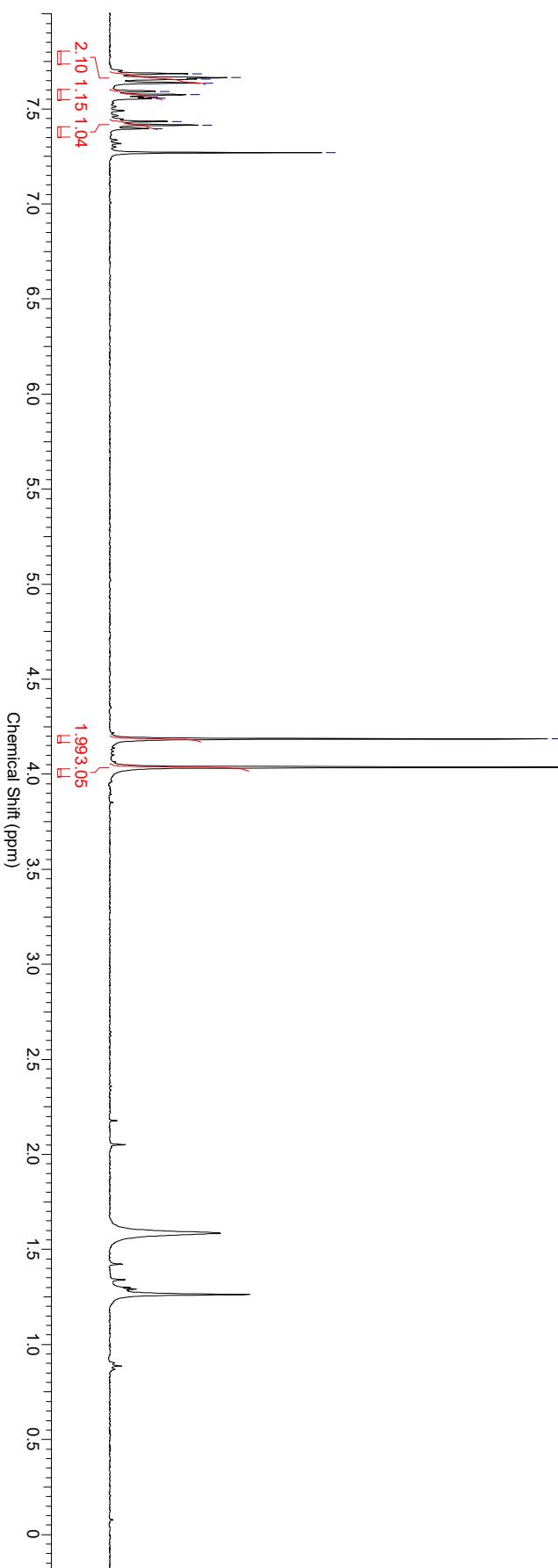
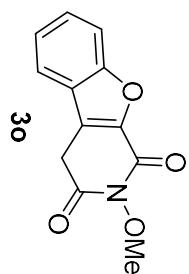
¹H NMR Spectra for Compound 3o in CDCl₃

Acquisition Time (sec)	2.0447	Comment	Revan 1H	Date	12 Feb 2016 19:07:52
Date Stamp	12 Feb 2016 19:07:52			File Name	\172.16.2.4\mr data\AV400\Feb_16_400\Tr2av400#01B1\PDAT\11r
Frequency (MHz)	400.13	Nucleus	1H	Number of Transients	2
Original Points Count	16384	Owner	root	Points Count	16384
Receiver Gain	80.60	SW(cyclical) (Hz)	8012.82	Pulse Sequence	zg30
Spectrum Offset (Hz)	2396.1619	Spectrum Type	STANDARD	Solvent	CHLOROFORM-d
				Sweep Width (Hz)	8012.33
				Temperature (degree C)	26.000

7.69
7.66
7.64
7.59
7.57
7.56
7.43
7.42
7.27

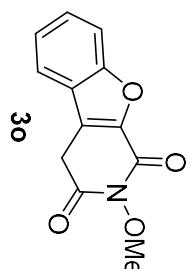
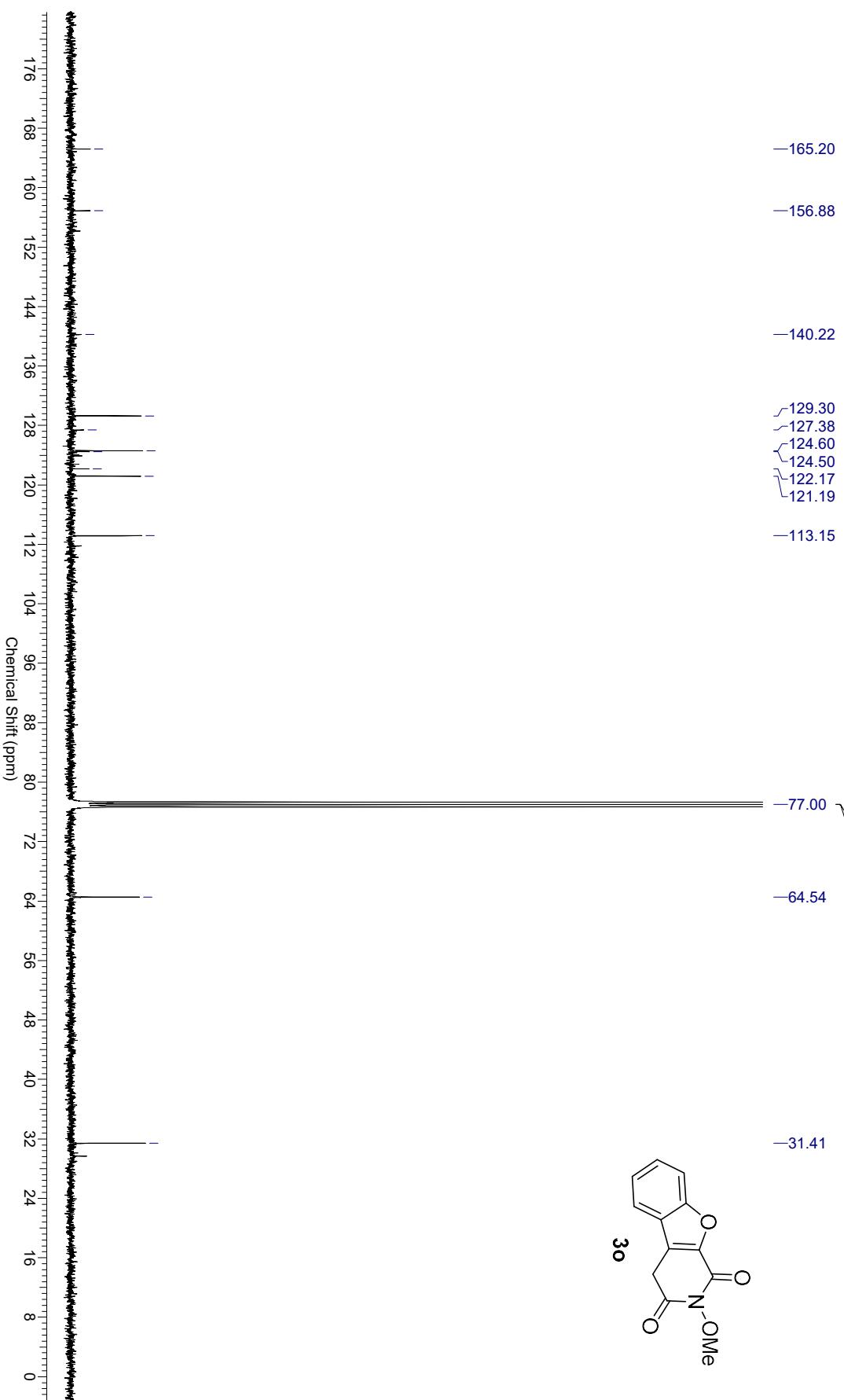
CHLOROFORM-d

—4.19
—4.04

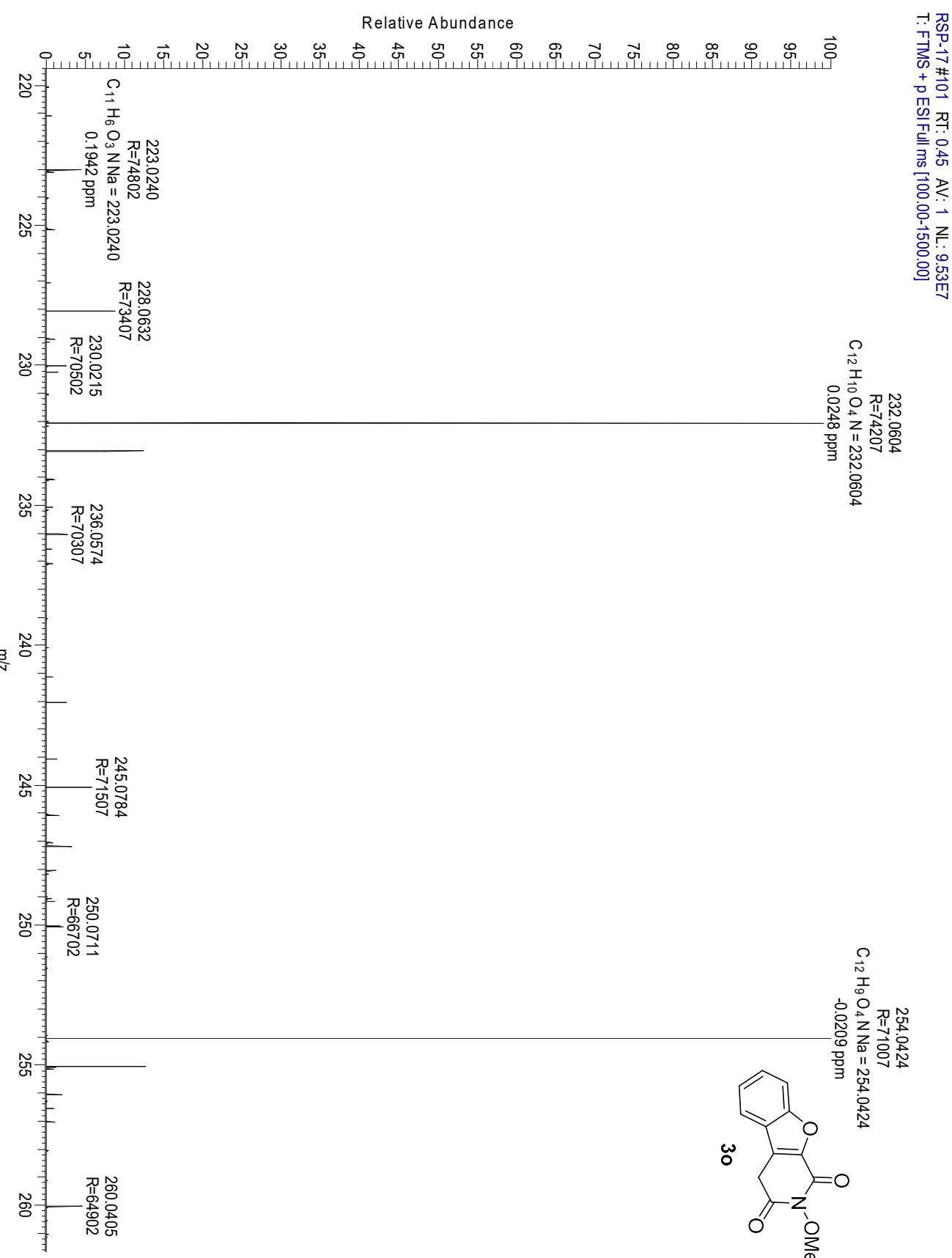


¹³C NMR Spectra for Compound **3o** in CDCl₃

Acquisition Time (sec)	0.6488	Comment	13C	Date	12 Feb 2016 19:12:08
Date Stamp	12 Feb 2016 19:12:08	File Name	\\\172.16.2.4\lmtr\data\AV400\Feb_16_400\Flr2av400#018(3)\PDATA\\1r		
Frequency (MHz)	100.61	Nucleus	13C	Number of Transients	1496
Original Points Count	16384	Owner	root	Points Count	32768
Receiver Gain	2050.00	SW(cyclical) (Hz)	25252.53	Pulse Sequence	zgpg30
Spectrum Offset (Hz)	10060.2773	Spectrum Type	STANDARD	Sweep Width (Hz)	25251.75
				Temperature (degree C)	25.900



HRMS Spectra for Compound **3o** in MeOH

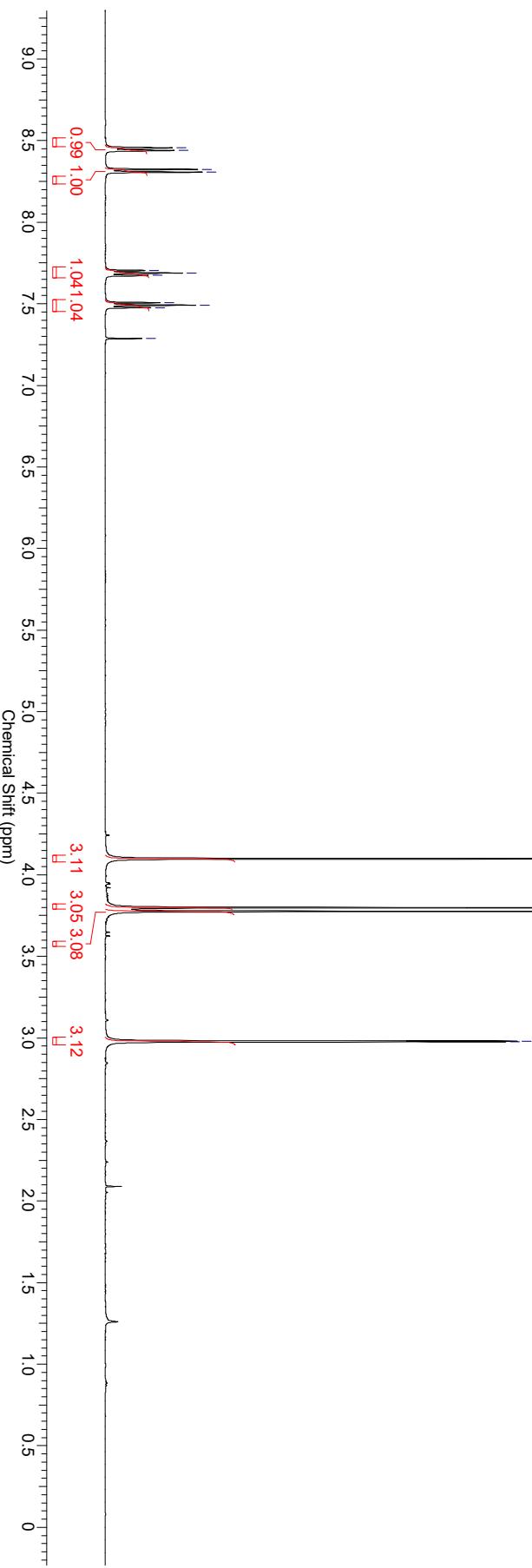
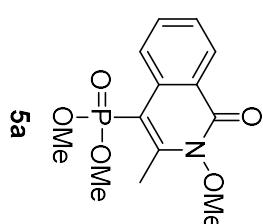


¹H NMR Spectra for Compound 5a in CDCl₃

Acquisition Time (sec)	1.6000	Comment	Venkanna 1H	Date	14 Nov 2015 10:14:40
Date Stamp	14 Nov 2015 10:14:40 <th>File Name</th> <td>\agnmr\dat\AV_500Nov_15_500\SA12av500#0111\PDAT\1\1r</td> <th>Origin</th> <td>spect</td>	File Name	\agnmr\dat\AV_500Nov_15_500\SA12av500#0111\PDAT\1\1r	Origin	spect
Frequency (MHz)	500.13	Nucleus	1H	Points Count	64
Original Points Count	16000	Owner	nmr	Pulse Sequence	zg30
Receiver Gain	203.00	SW(cyclical) (Hz)	10000.00	Solvent	CHLOROFORM-d
Spectrum Offset (Hz)	2500.6499	Spectrum Type	STANDARD	Sweep Width (Hz)	9999.70
				Temperature (degree C)	25.000

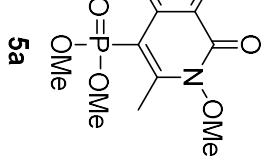
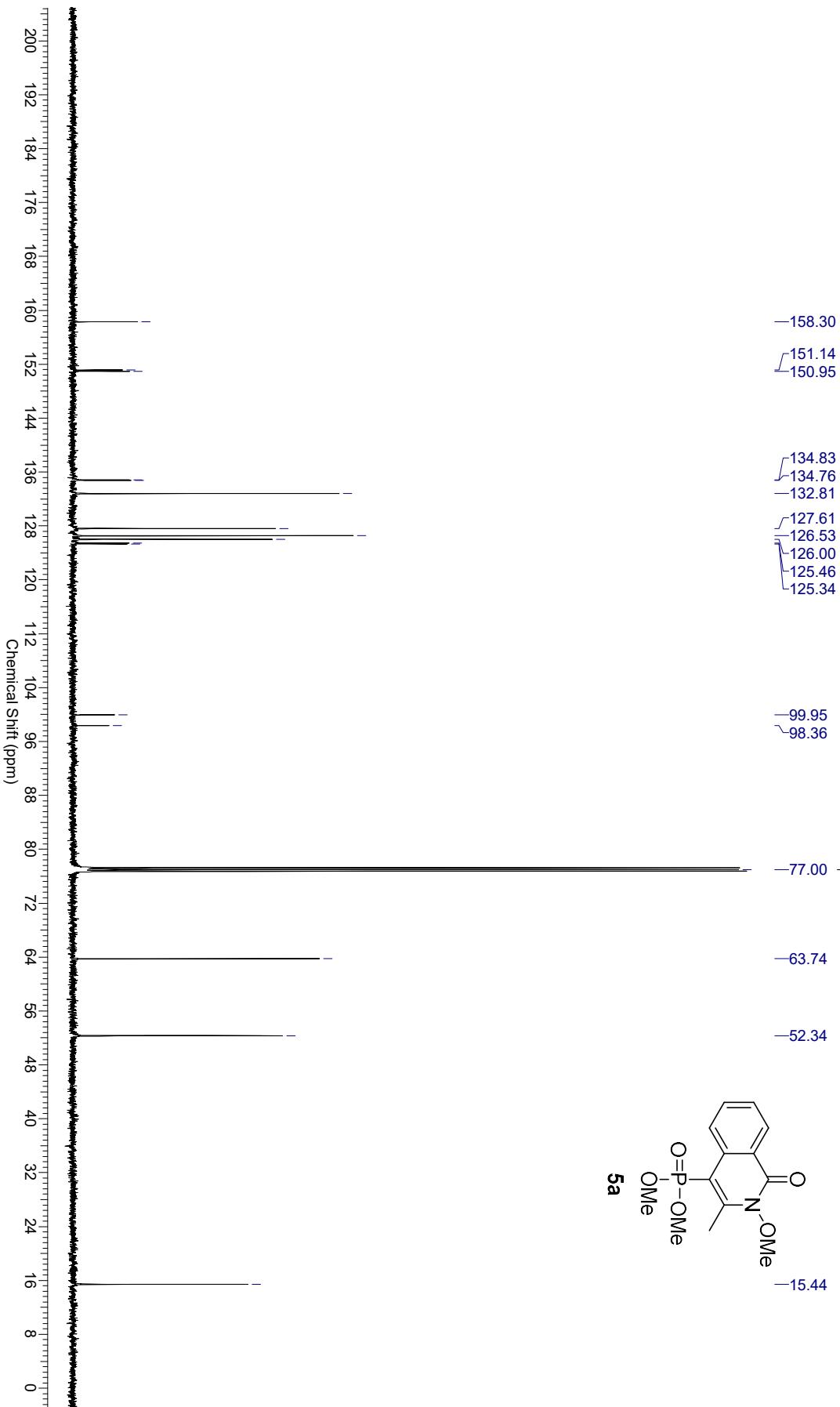
8.46
8.44
8.32
8.31
7.70
7.69
7.67
7.51
7.49
7.48
7.29

4.10
3.80
3.78
2.98
2.98



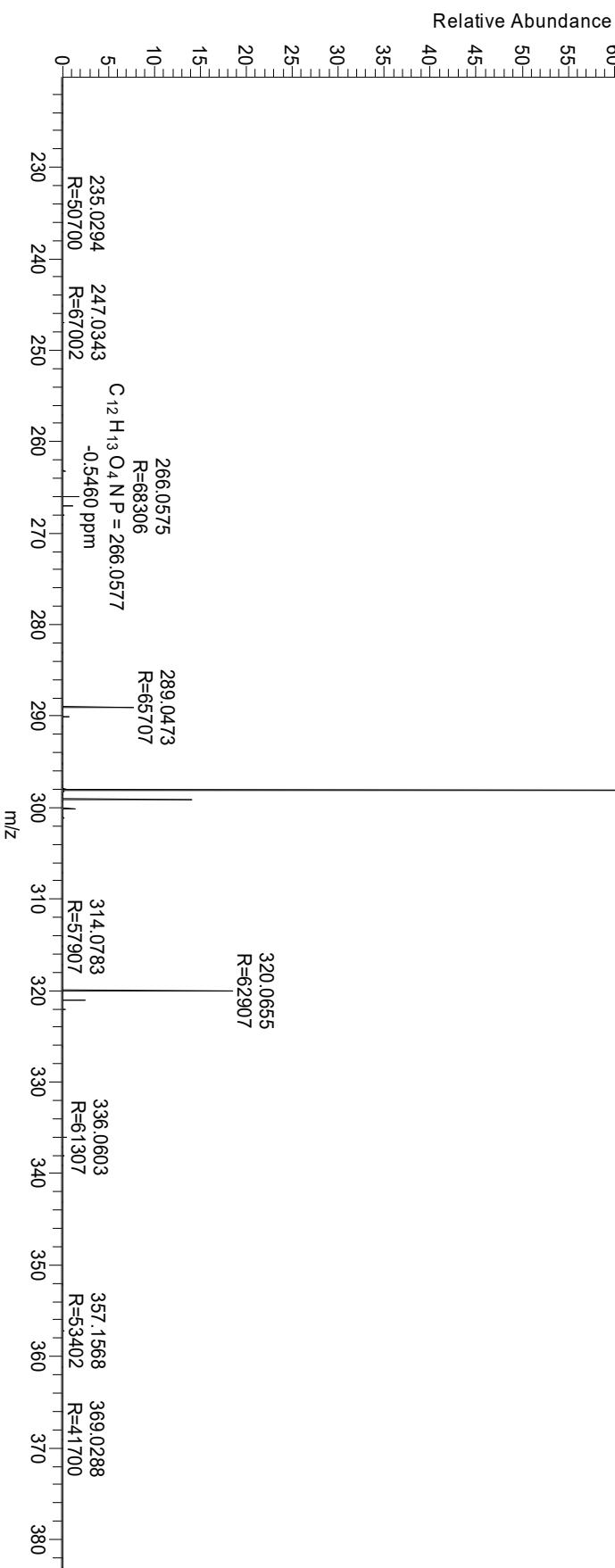
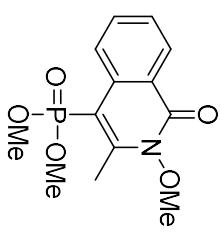
¹³C NMR Spectra for Compound 5a in CDCl₃

Acquisition Time (sec)	0.6554	Comment	13C	Date	14 Nov 2015 10:59:28
Date Stamp	14 Nov 2015 10:59:28			File Name	\agnmr_data\AV_500\Nov_15_500\Sal2av500\001\3\PDATA\111
Frequency (MHz)	125.76	Nucleus	13C	Number of Transients	636
Original Points Count	20480	Owner	nmr	Points Count	32768
Receiver Gain	2050.00	SW(cyclical) (Hz)	31250.00	Solvent	CHLOROFORM-d
Spectrum Offset (Hz)	12554.3857	Spectrum Type	STANDARD	Sweep Width (Hz)	31249.05
				Temperature (degree C)	25.000



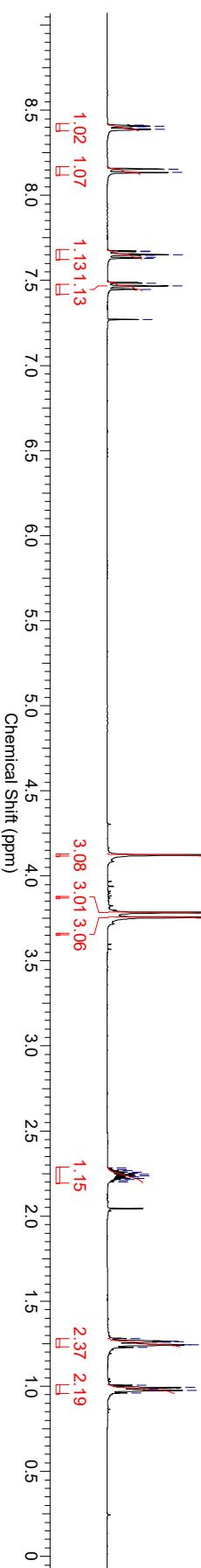
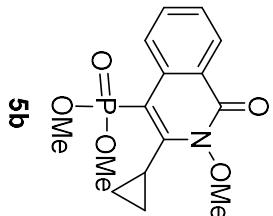
HRMS Spectra for Compound **5a** in MeOH

RSP-18#101 RT: 0.45 AV: 1 NL: 5.16E9
T: FTMS + pESI[full.ms][100.00-1500.00]



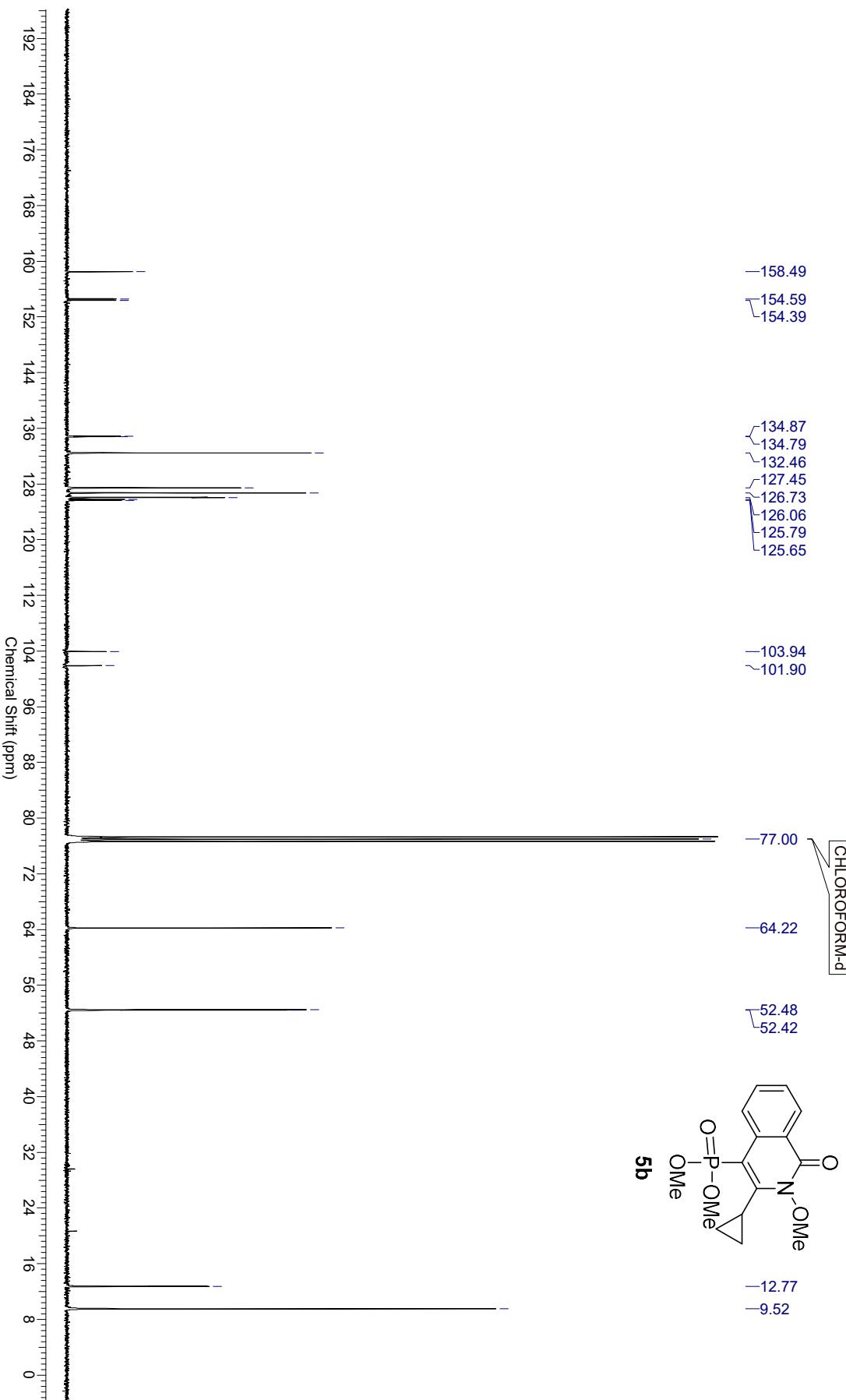
¹H NMR Spectra for Compound 5b in CDCl₃

Acquisition Time (sec)	2.1838	Comment	Ravindra	Date	17 Nov 2015 00:07:32
Date Stamp	16 Nov 2015 08:15:39				
File Name	F:\pillai\barResearch\3d methodology AmideNMRketodiazolsimple+cyclo phos\Mon3ECX400#001.PROTON-3.of				
Frequency (MHz)	399.78	Nucleus	¹ H	Number of Transients	128
Original Points Count	13107	Owner	delta	Points Count	13107
Receiver Gain	30.00	Solvent	CHLOROFORM-d	Pulse Sequence	single pulse ex2
Spectrum Type	STANDARD	Sweep Width (Hz)	6001.85	Temperature (degree C)	22.700
		Spectrum Offset (Hz)	2007.4668		



¹³C NMR Spectra for Compound 5b in CDCl₃

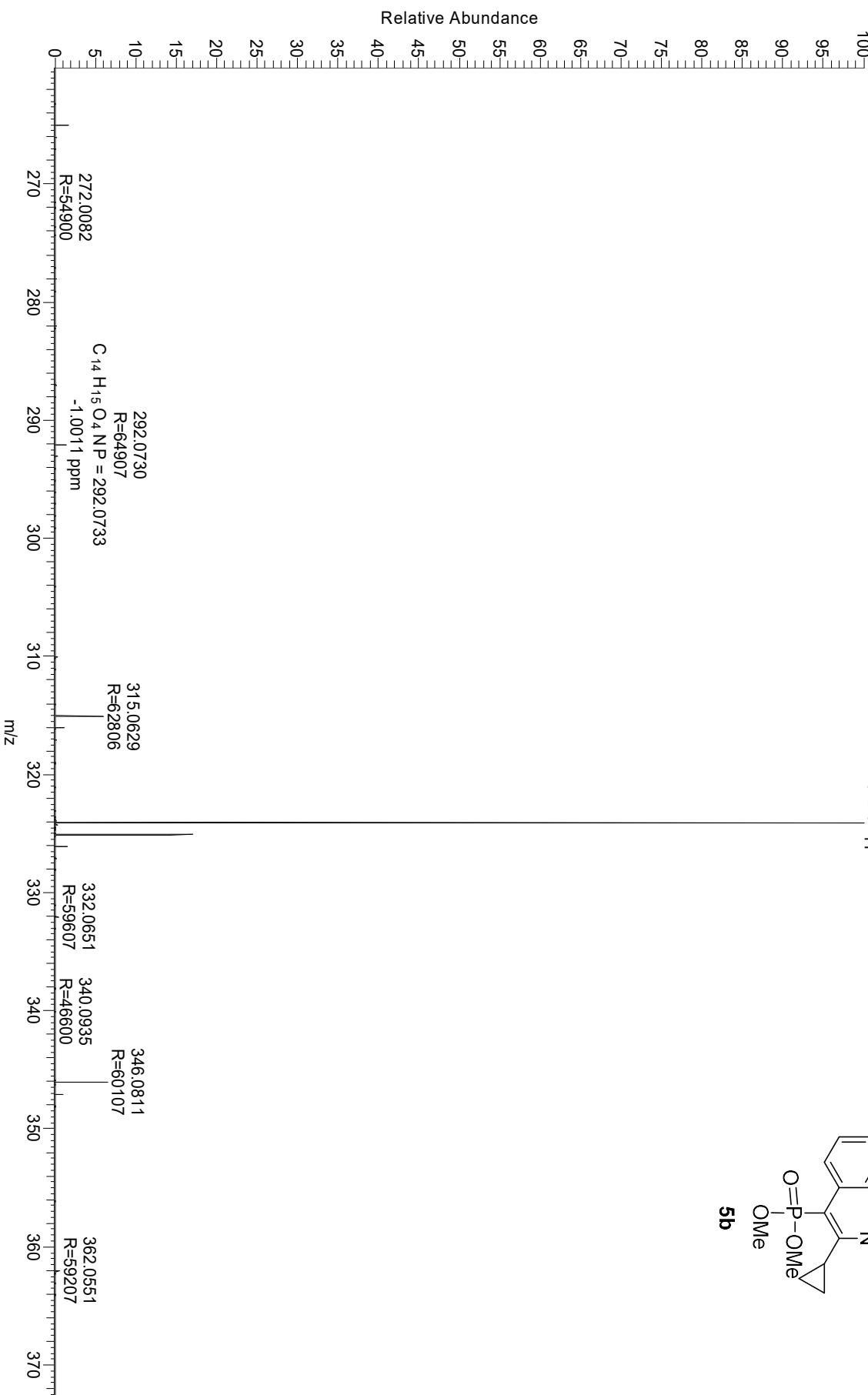
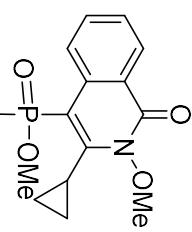
Acquisition Time (sec)	1.0434	Comment	Ravindra	Date	17 Nov 2015 02:04:10
Date Stamp	16 Nov 2015 09:33:11				
File Name	F:\pitambar\Research\3rd methodology_Amide\NMR\ketodiazol\simple+cylo phos\Mon3ECCX400#001_CARBON-3.jdf				
Frequency (MHz)	100.53	Nucleus	¹³ C	Number of Transients	2000
Original Points Count	26214	Owner	delta	Points Count	26214
Receiver Gain	60.00	Solvent	CHLOROFORM-d	Pulse Sequence	single pulse dec
Sweep Width (Hz)	25124.29	Temperature (degree C)	23.300	Spectrum Offset (Hz)	10039.3311
				Spectrum Type	STANDARD



HRMS Spectra for Compound **5b** in MeOH

RSP-19 #101 RT: 0.45 AV: 1 NL: 7.71E9
T: FTMS + pESIFull.ms [100.00-1500.00]

324.0995
R=62707
 $C_{15}H_{19}O_5NP = 324.0995$
-0.2434 ppm



¹H NMR Spectra for Compound 5c in CDCl₃

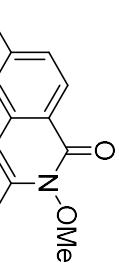
Acquisition Time (sec)	2.0447	Comment	Ravindra 1H	Date	29 Nov 2015 12:11:52
Date Stamp	29 Nov 2015 12:11:52	File Name	\agn\1nmr_data\AV400\Nov_15_400\Sun5av400#0111\PDAT\1\1r		
Frequency (MHz)	400.13	Nucleus	¹ H	Number of Transients	99
Original Points Count	16384	Owner	Administrator	Points Count	32768
Receiver Gain	228.00	SW(cyclical) (Hz)	8012.82	Pulse Sequence	zg30
Spectrum Offset (Hz)	2395.5503	Spectrum Type	STANDARD	Sweep Width (Hz)	8012.58
				Temperature (degree C)	22.400

8.32
8.31
8.30
8.29
8.11

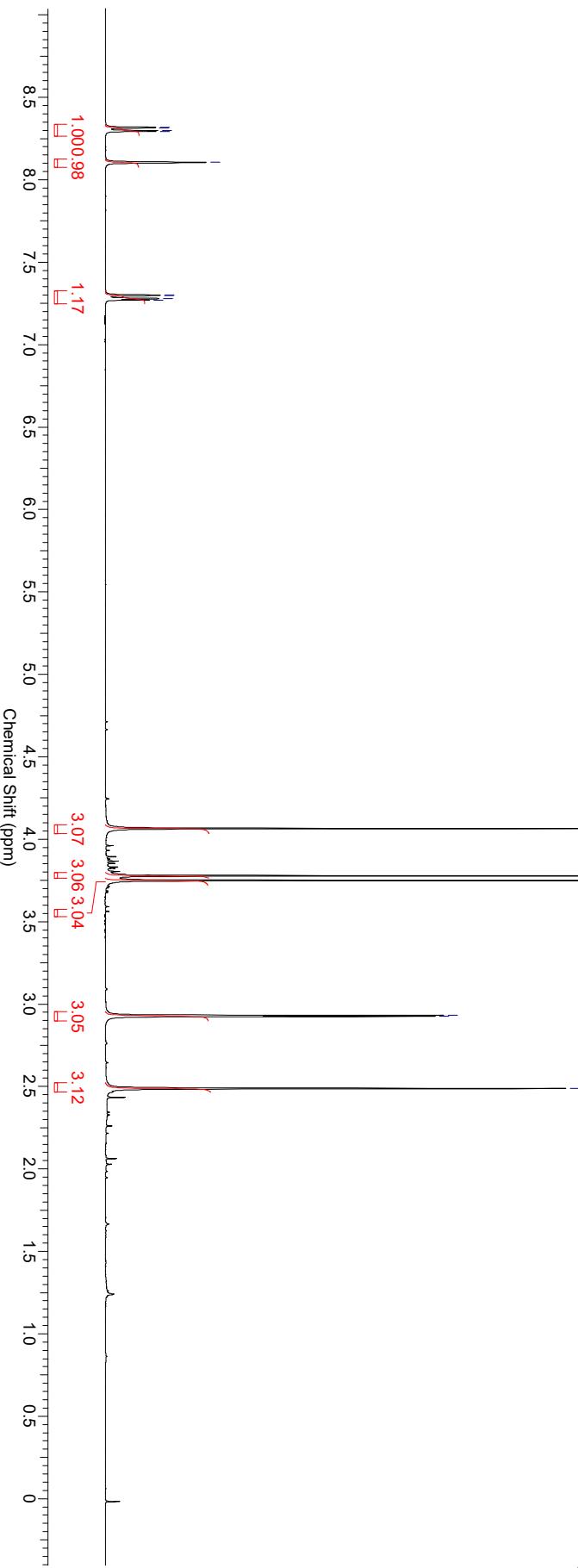
7.30
7.30
7.28
7.27

4.06
3.78
3.75

2.93
2.93



5c



¹³C NMR Spectra for Compound 5c in CDCl₃

Acquisition Time (sec)	0.6488	Comment	13C	Date	29 Nov 2015 13:03:04
Date Stamp	29 Nov 2015 13:03:04	File Name	\agnlmr_data\AV400\Nov_15_400Sun5av400#0013\PDAT\1\1r	Nucleus	13C
Frequency (MHz)	100.61	Number of Transients	2650	Origin	spect
Original Points Count	16384	Owner	root	Pulse Sequence	zgpg
Receiver Gain	2050.00	SW(cyclical) (Hz)	25252.53	Solvent	CHLOROFORM-d
Spectrum Offset (Hz)	10054.8818	Spectrum Type	STANDARD	Sweep Width (Hz)	25251.75
				Temperature (degree C)	22.900

—158.24

—151.06

—150.83

—143.46

—134.99

—134.89

—128.10

—127.55

—125.76

—123.21

—123.07

—99.84

—97.87

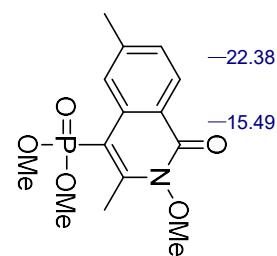
—77.00

—63.72

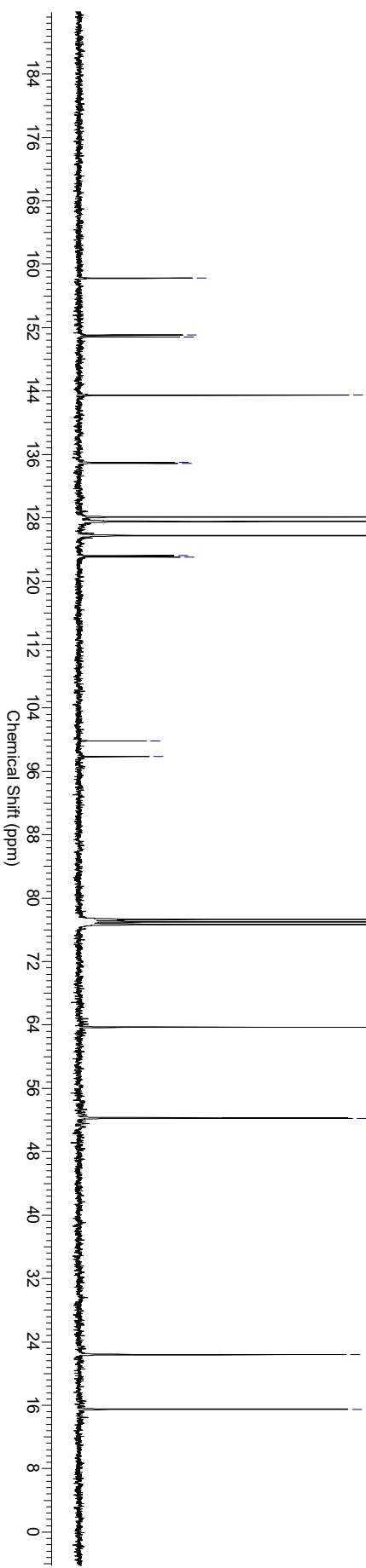
—52.23

—22.38

—15.49



5c

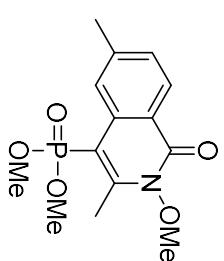


HRMS Spectra for Compound **5c** in MeOH

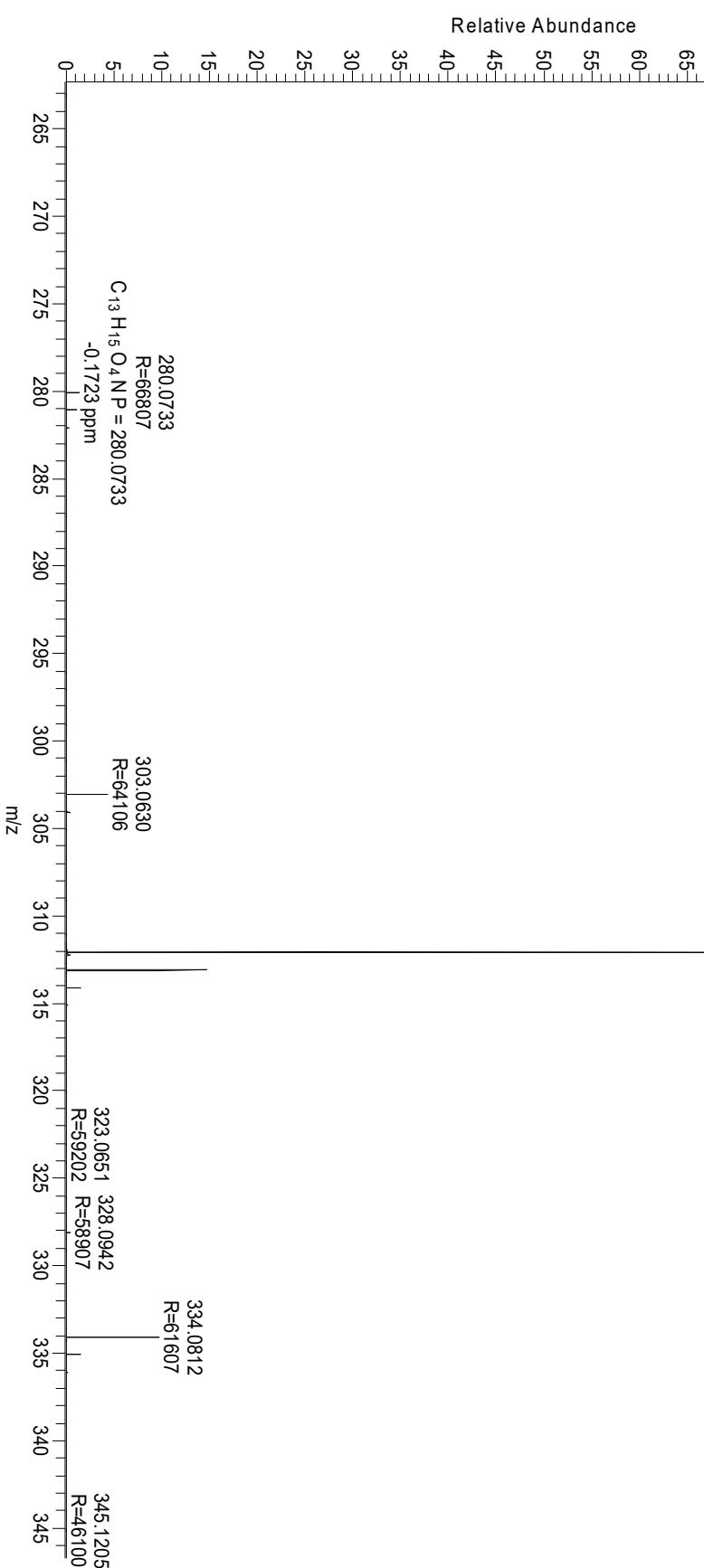
RSP-20#103 RT: 0.46 AV: 1 NL: 7.18E9
T: FTMS + p ESI[Full ms [100.00-1500.00]]

312.0995
R=63507
 $C_{14}H_{19}O_5NP = 312.0995$
-0.0572 ppm

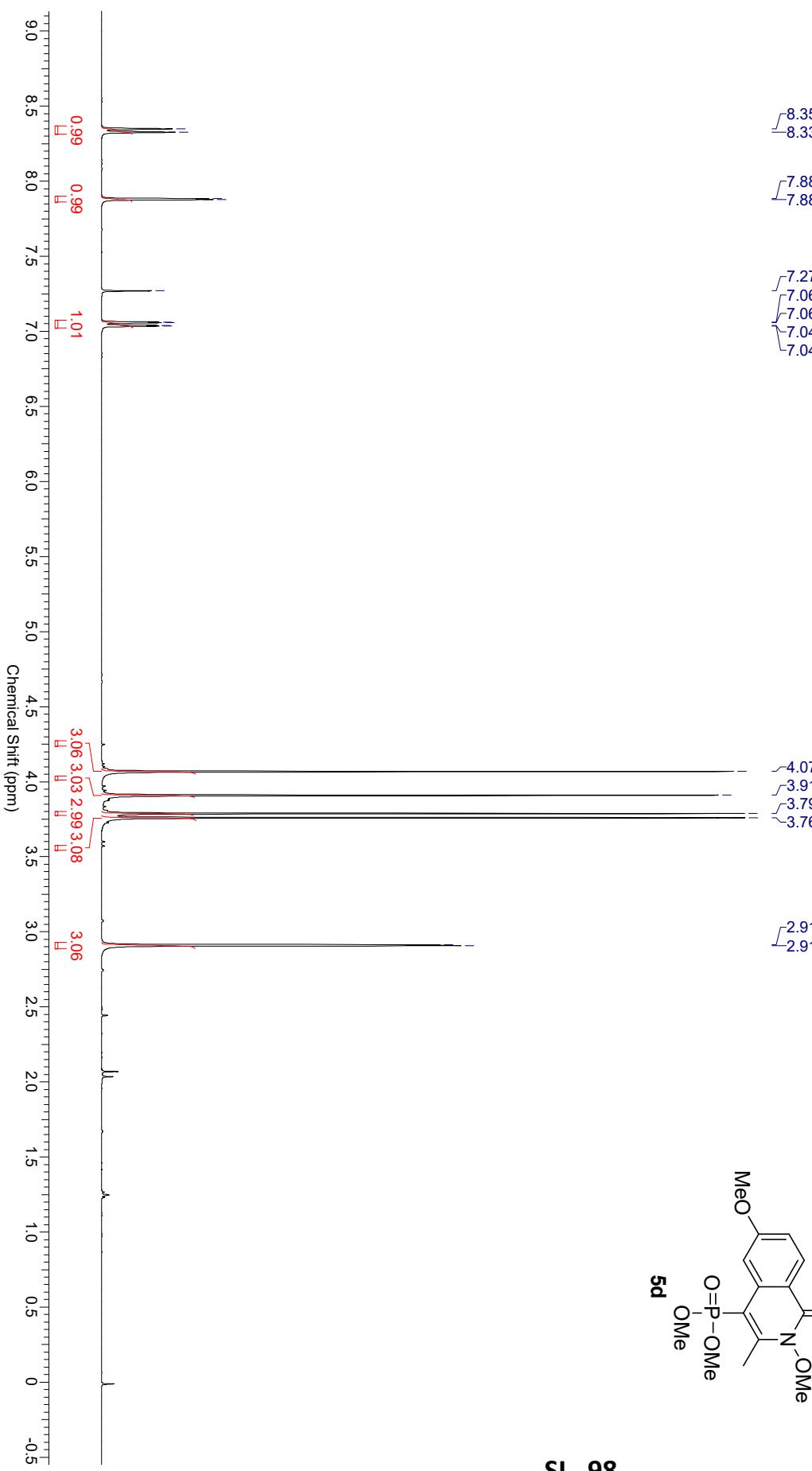
312.0995
R=63507
 $C_{14}H_{19}O_5NP = 312.0995$
-0.0572 ppm



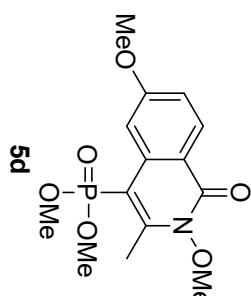
5c



¹H NMR Spectra for Compound 5d in CDCl₃

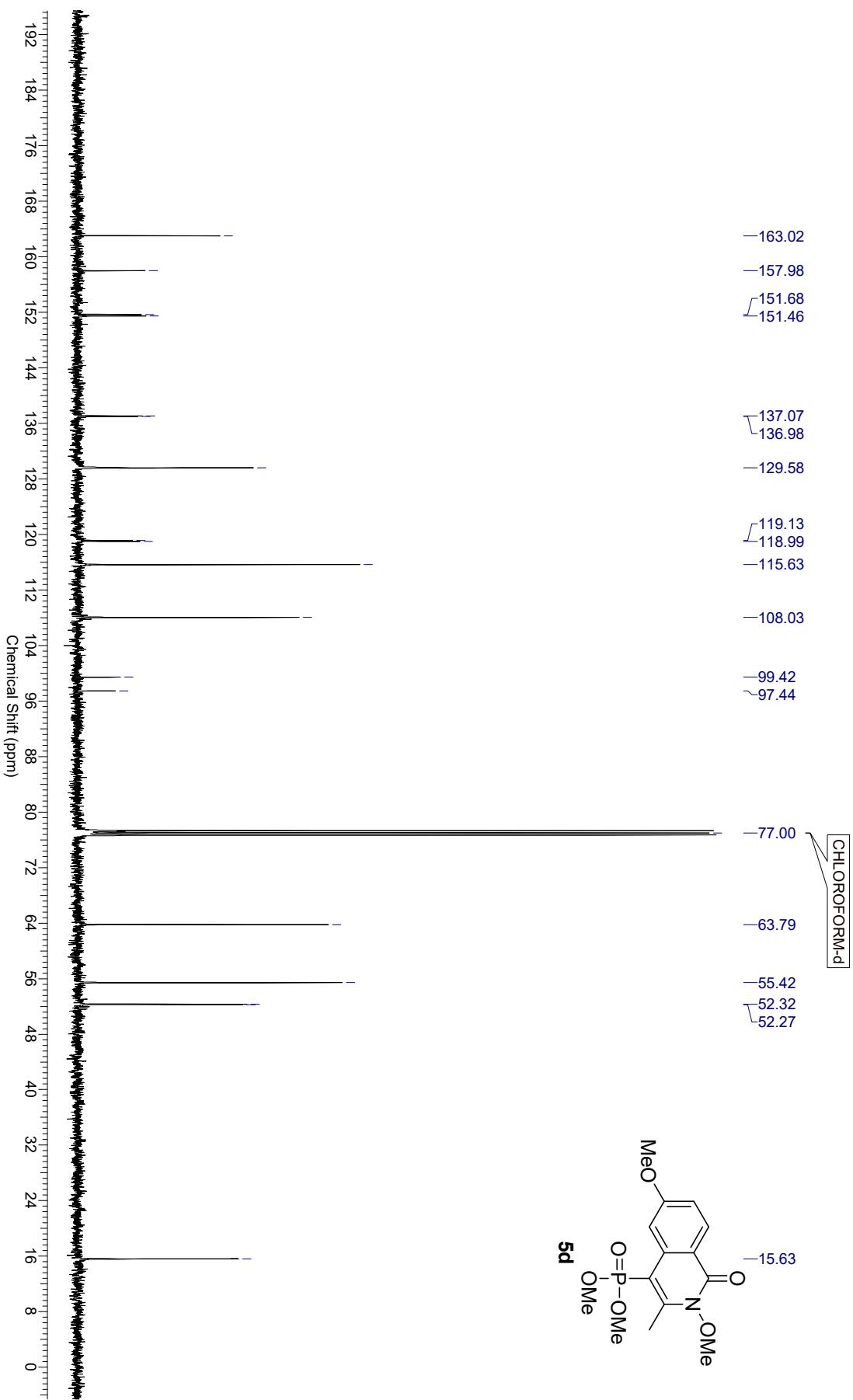


Acquisition Time (sec)	2.0447	Comment	Pitamber 1H	Date	30 Nov 2015 11:22:48
Date Stamp	30 Nov 2015 11:22:48			File Name	\agni\mr_data\400\Nov_15_400\Mon5av400\#08\1\PDATA\1\rf
Frequency (MHz)	400.13	Nucleus	1H	Number of Transients	64
Original Points Count	16384	Owner	Administrator	Points Count	32768
Receiver Gain	228.00	SW(cyclical) (Hz)	8012.82	Pulse Sequence	zg30
Spectrum Offset (Hz)	2395.0613	Spectrum Type	STANDARD	Solvent	CHLOROFORM-d
				Sweep Width (Hz)	8012.58
				Temperature (degree C)	22.500



¹³C NMR Spectra for Compound **5d** in CDCl₃

Acquisition Time (sec)	0.6488	Comment	13C	Date	30 Nov 2015 11:44:08
Date Stamp	30 Nov 2015 11:44:08			File Name	\lagnmr\data\AV400\Nov_15_400MHz\av400#0083\DATA\111r
Frequency (MHz)	100.61	Nucleus	13C	Number of Transients	823
Original Points Count	16384	Owner	root	Points Count	32768
Receiver Gain	2050.00	SW(cyclical) (Hz)	25252.53	Solvent	CHLOROFORM-d
Spectrum Offset (Hz)	10056.4233	Spectrum Type	STANDARD	Sweep Width (Hz)	25251.75
				Temperature (degree C)	22.900



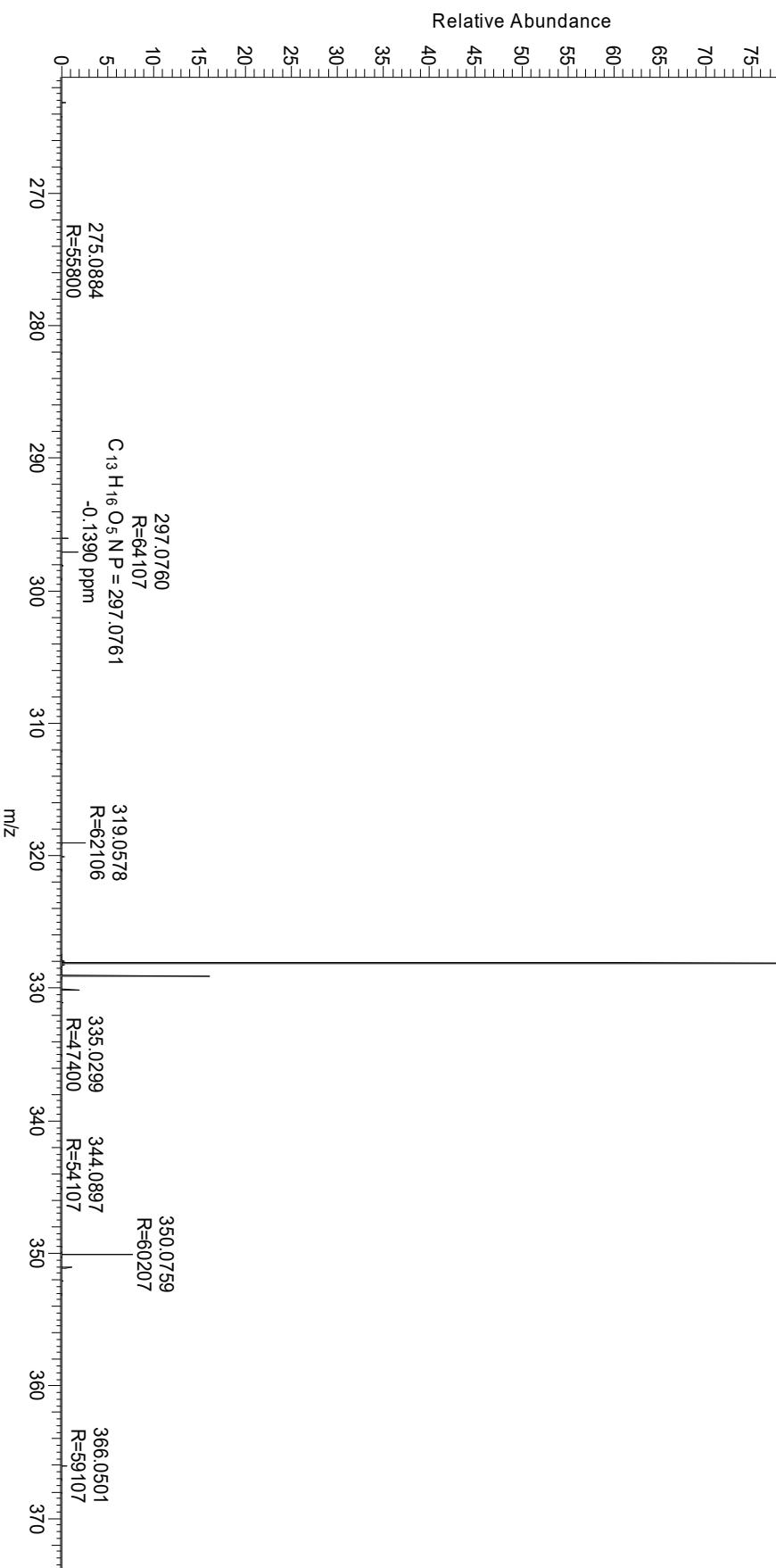
HRMS Spectra for Compound **5d** in MeOH

RSP-21 #100 RT: 0.44 AV: 1 NL: 9.52E9
T: FTMS + pESI[Full ms][100.00-1500.00]

328.0943
R=62303
 $C_{14}H_{19}O_6NP = 328.0945$
-0.4601 ppm



5d



SI 100

¹H NMR Spectra for Compound 5e in CDCl₃

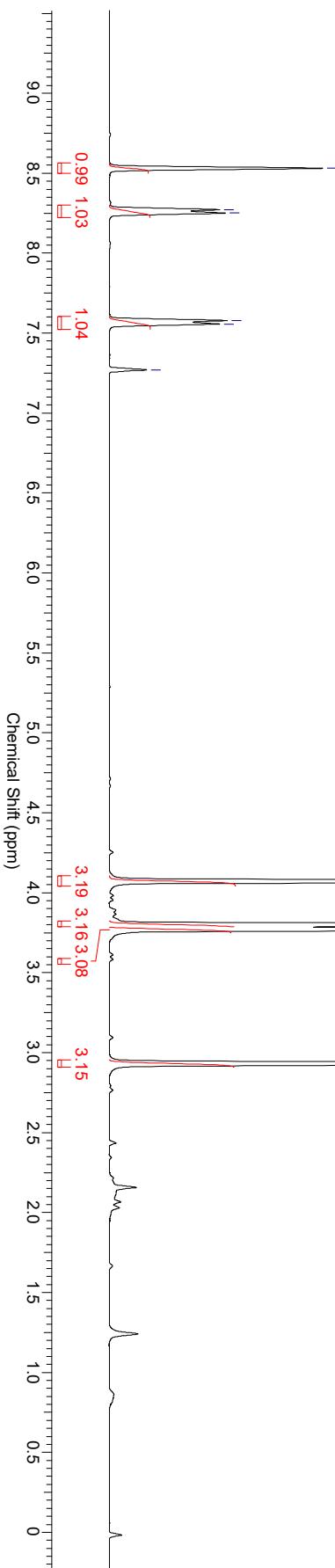
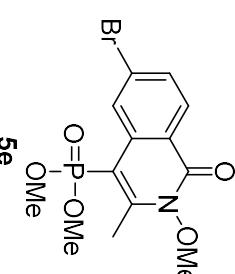
Acquisition Time (sec)	Comment	Ravindra 1H	Date	01 Dec 2015 14:30:32
Date Stamp			File Name	\agnmr_data\400\Dec_15_400Tue1av400#0111\PDATA\1\1r
Frequency (MHz)	Nucleus	1H	Number of Transients	64
400.113	Owner	Administrator	Points Count	32768
16384	SIM(cyclical) (Hz)	8012.82	Origin	spect
203.00	Solvent	CHLOROFORM-d	Pulse Sequence	zg30
2395.5503	Spectrum Type	STANDARD	Sweep Width (Hz)	8012.58
			Temperature (degree C)	22.900

-8.53
-8.27
-8.25

-7.58
-7.56
-7.27

CHLOROFORM-d

-4.07
-3.80
-3.77
-2.93



¹³C NMR Spectra for Compound 5e in CDCl₃

Acquisition Time (sec)	0.6488	Comment	13C	Date	01 Dec 2015 14:49:44
Date Stamp	01 Dec 2015 14:49:44	File Name		\\agninmr\data\AV400\Dec. 15_400\Tue1ava400#0113\PDAT\111r	
Frequency (MHz)	100.61	Nucleus	13C	Number of Transients	550
Original Points Count	16384	Owner	root	Points Count	32768
Receiver Gain	2050.00	Solvent	CHLOROFORM-d	Pulse Sequence	zgpg
Spectrum Offset (Hz)	10055.6533	Spectrum Type	STANDARD	Sweep Width (Hz)	25251.75
				Temperature (degree C)	23.200

—157.83
—152.27
—152.04

—136.36
—136.27
—129.90
—129.21
—128.78
—128.41
—124.16
—124.02

—99.52
—97.54

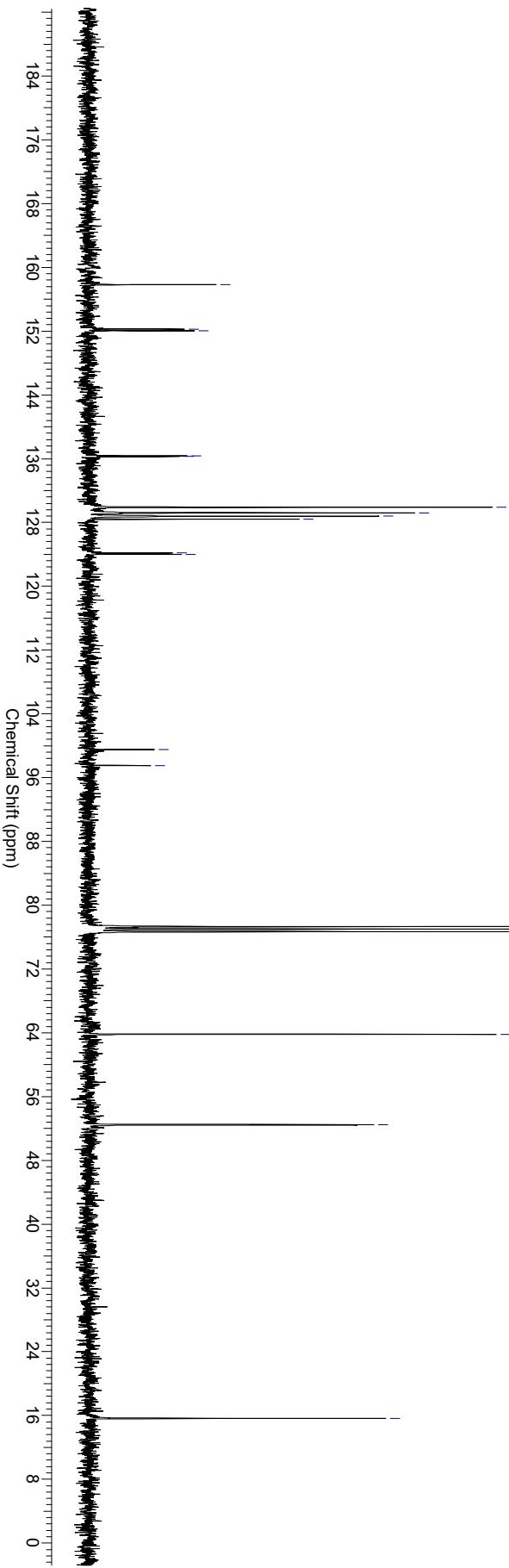
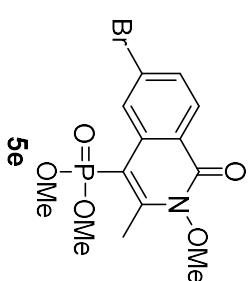
—77.00

—63.82

—52.49

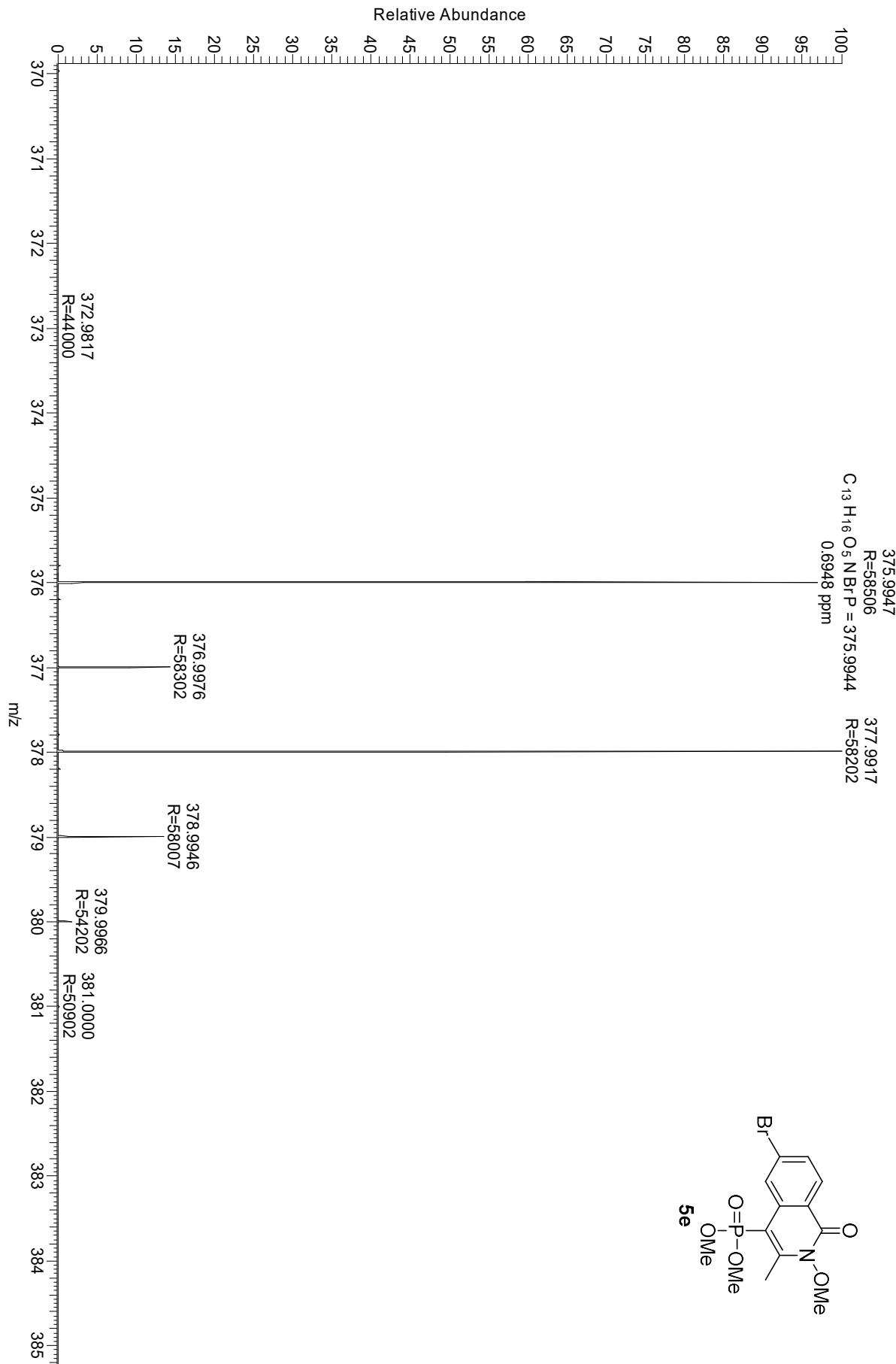
—15.64

CHLOROFORM-d



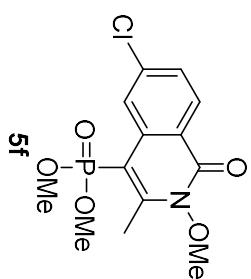
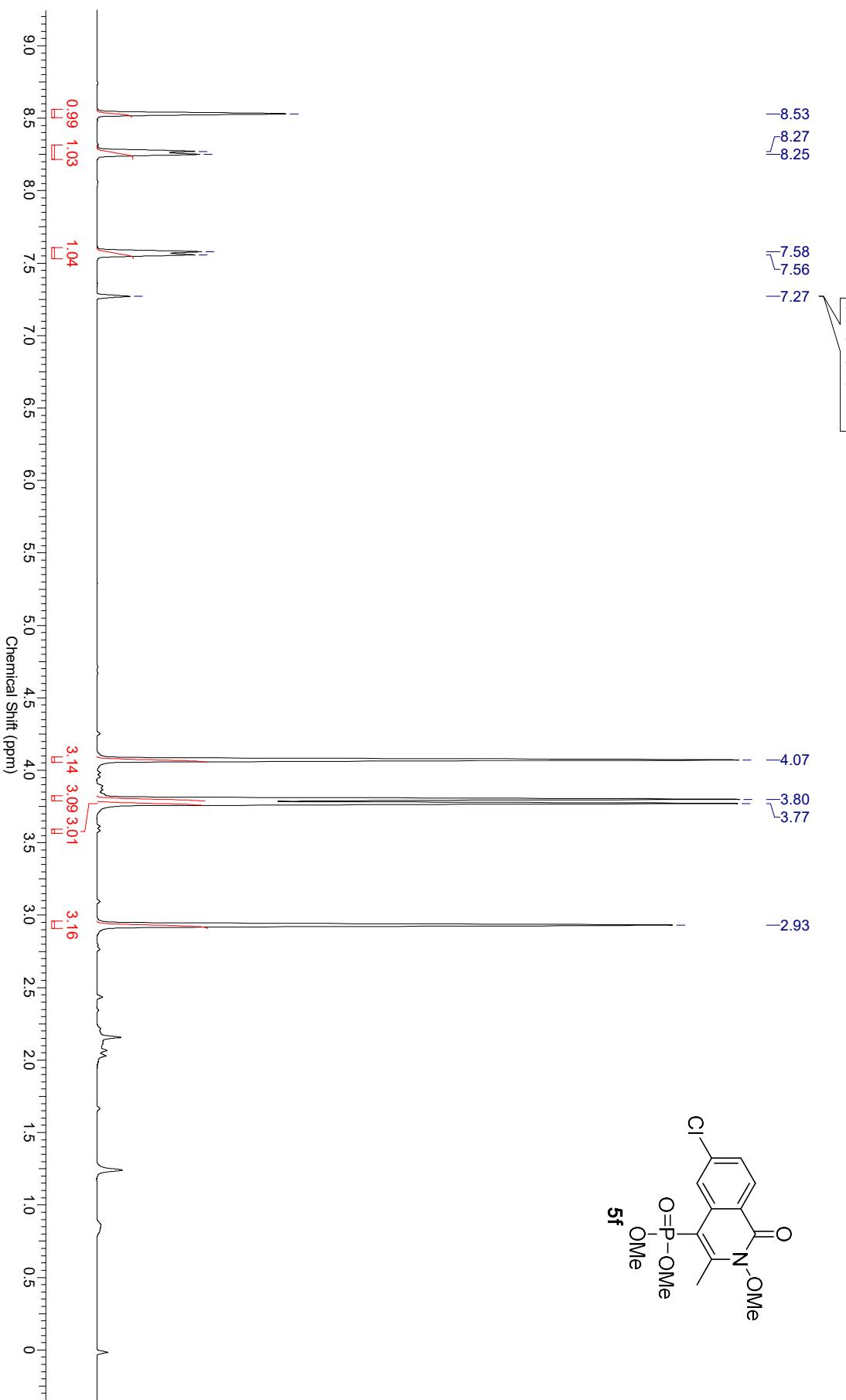
HRMS Spectra for Compound **5e** in MeOH

RSP-22 #106 RT: 0.47 AV: 1 NL: 2.62E9
 T: FTMS + pESI Full ms [100.00-1500.00]



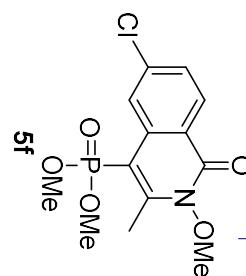
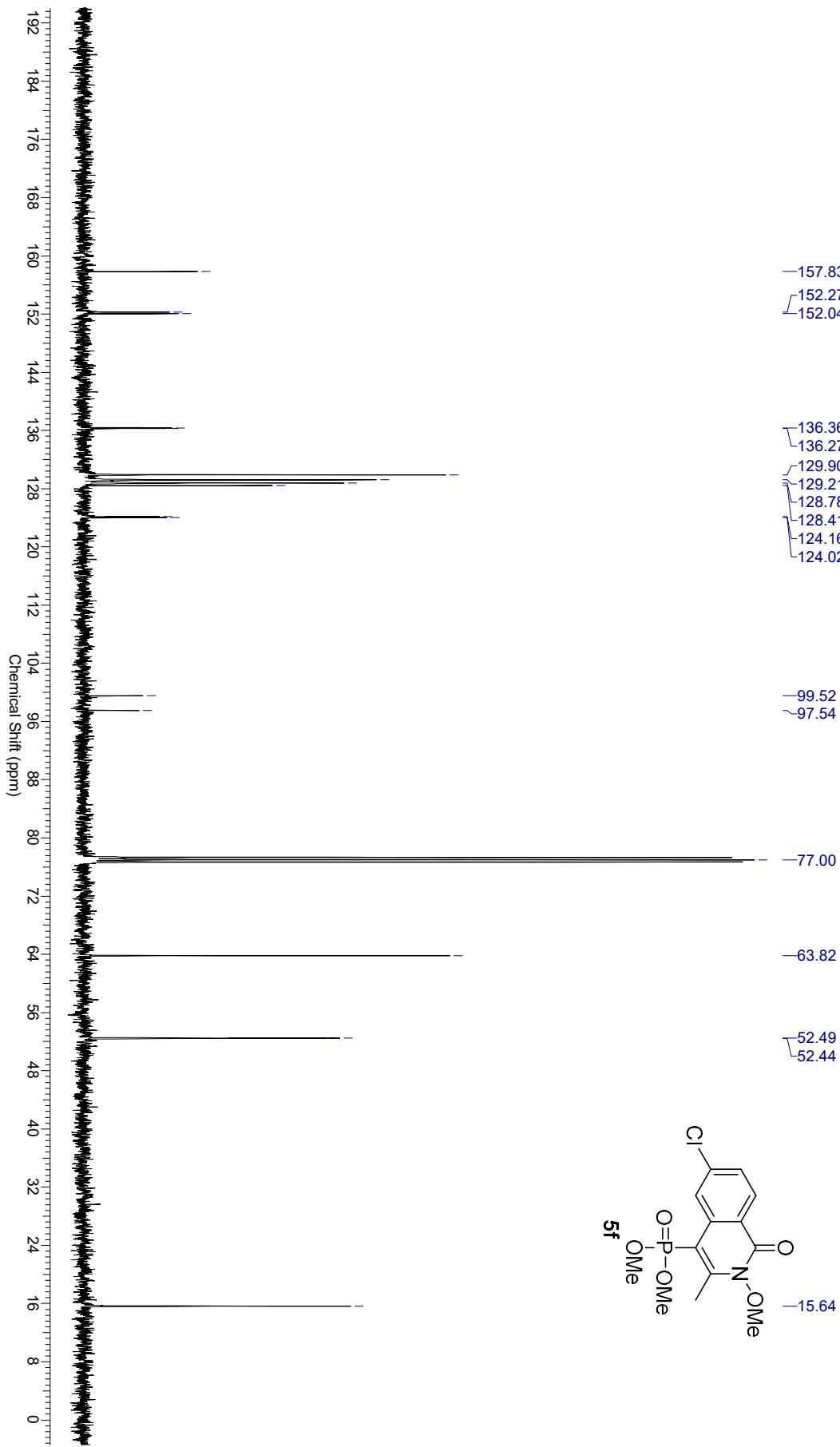
¹H NMR Spectra for Compound 5f in CDCl₃

Acquisition Time (sec)	2.0447	Comment	Ravindra 1H	Date	01 Dec 2015 14:30:32
Date Stamp	01 Dec 2015 14:30:32	File Name	lagnlnmr_data\AV400\Dec_15_400Tue1av400#0111\PDAT\1\1r		
Frequency (MHz)	400.13	Nucleus	1H	Number of Transients	64
Original Points Count	16384	Owner	Administrator	Points Count	32768
Receiver Gain	203.00	SW(cyclical) (Hz)	8012.82	Pulse Sequence	zg30
Spectrum Offset (Hz)	2395.5503	Spectrum Type	STANDARD	Sweep Width (Hz)	8012.58
				Temperature (degree C)	22.900



SI 104

¹³C NMR Spectra for Compound 5f in CDCl₃



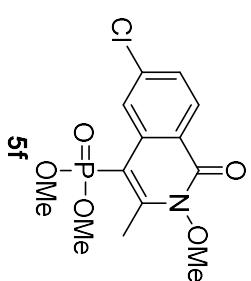
SI 105

HRMS Spectra for Compound **5f** in MeOH

RR-A-6 #106 RT: 0.47 AV: 1 NL: 1.48E9
T: FTMS + pESI[Full ms [100.00-1500.00]



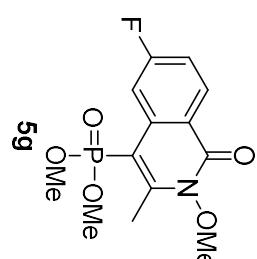
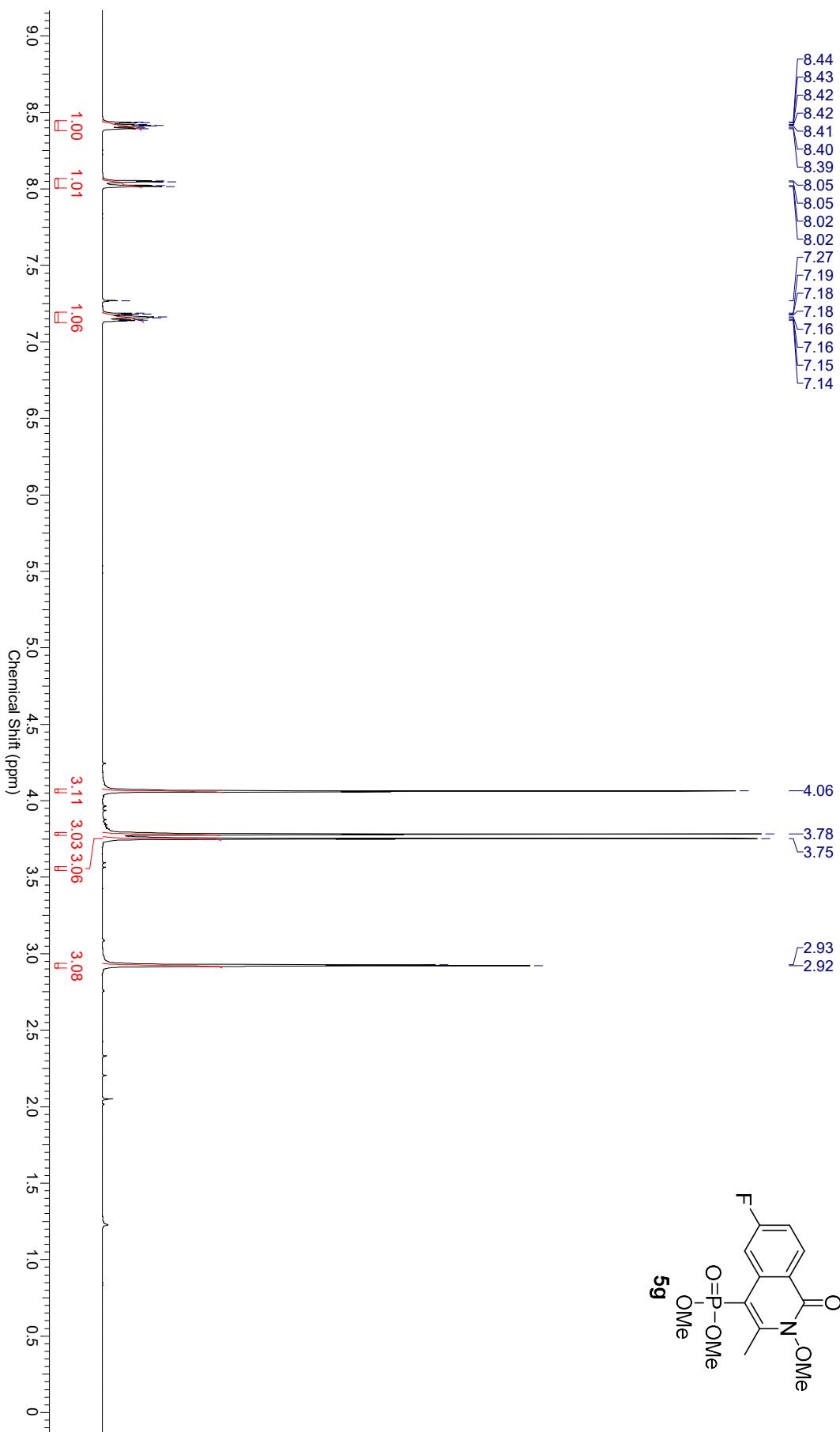
C₁₃H₁₆O₅NClP = 332.0449
R=61706
0.0259 ppm



SI 106

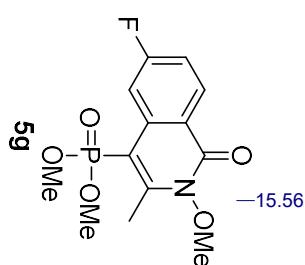
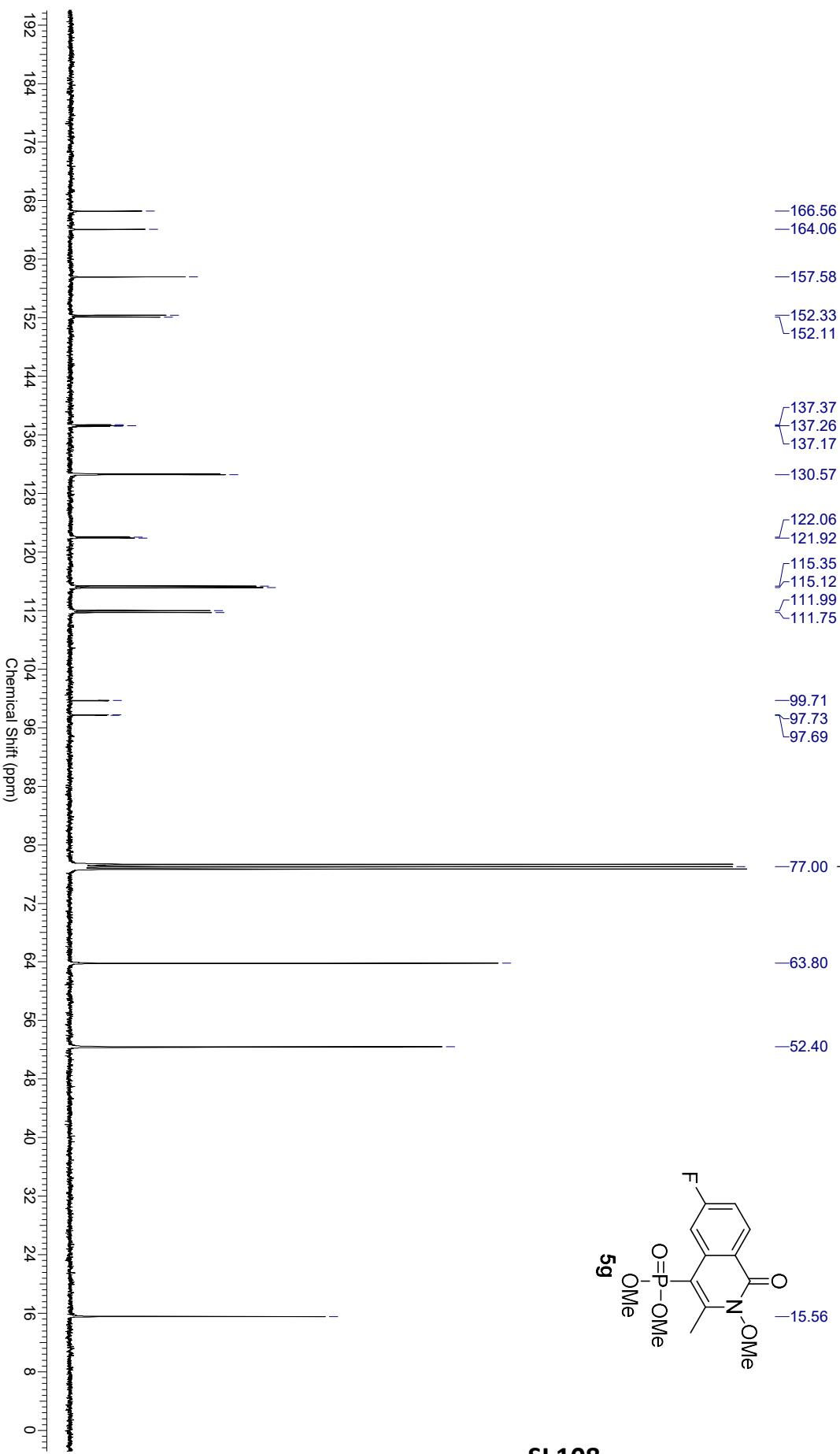
¹H NMR Spectra for Compound **5g in CDCl₃**

Acquisition Time (sec)	2.1838	Comment	Ravindra	Date	07 Jan 2016 09:56:38
Date Stamp	06-Jan-2016 18:01:16			File Name	\agnl\nmr\data\IEOL_400\2016\JAN_LIQUID\Tue2ECX400#028_PROTON-3.dif
Frequency (MHz)	399.78	Nucleus	1H	Number of Transients	32
Original Points Count	13107	Owner	delta	Points Count	13107
Receiver Gain	30.00	Solvent	CHLOROFORM-d	Pulse Sequence	single_pulse.ex2
Spectrum Type	STANDARD	Sweep Width (Hz)	6001.85	Temperature (degree C)	23.300
		Spectrum Offset (Hz)			2008.3826



¹³C NMR Spectra for Compound 5g in CDCl₃

Acquisition Time (sec)	1.0434	Comment	Ravindra	Date	07 Jan 2016 11:14:37
Date Stamp	07 Jan 2016 00:35:42			File Name	\lagin\mr_data\EOL_400\2016\JAN_LIQUID\Tus2ECX400#028_CARBON-3.jdf
Frequency (MHz)	100.53	Nucleus	13C	Number of Transients	1200
Original Points Count	26214	Owner	delta	Points Count	26214
Receiver Gain	60.00	Solvent	CHLOROFORM-d	Pulse Sequence	single pulse dec
Spectrum Type	STANDARD	Sweep Width (Hz)	25124.29	Temperature (degree C)	23.500
				Spectrum Offset (Hz)	10039.3311



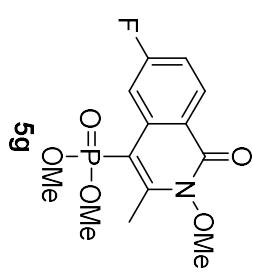
SI 108

HRMS Spectra for Compound **5g** in MeOH

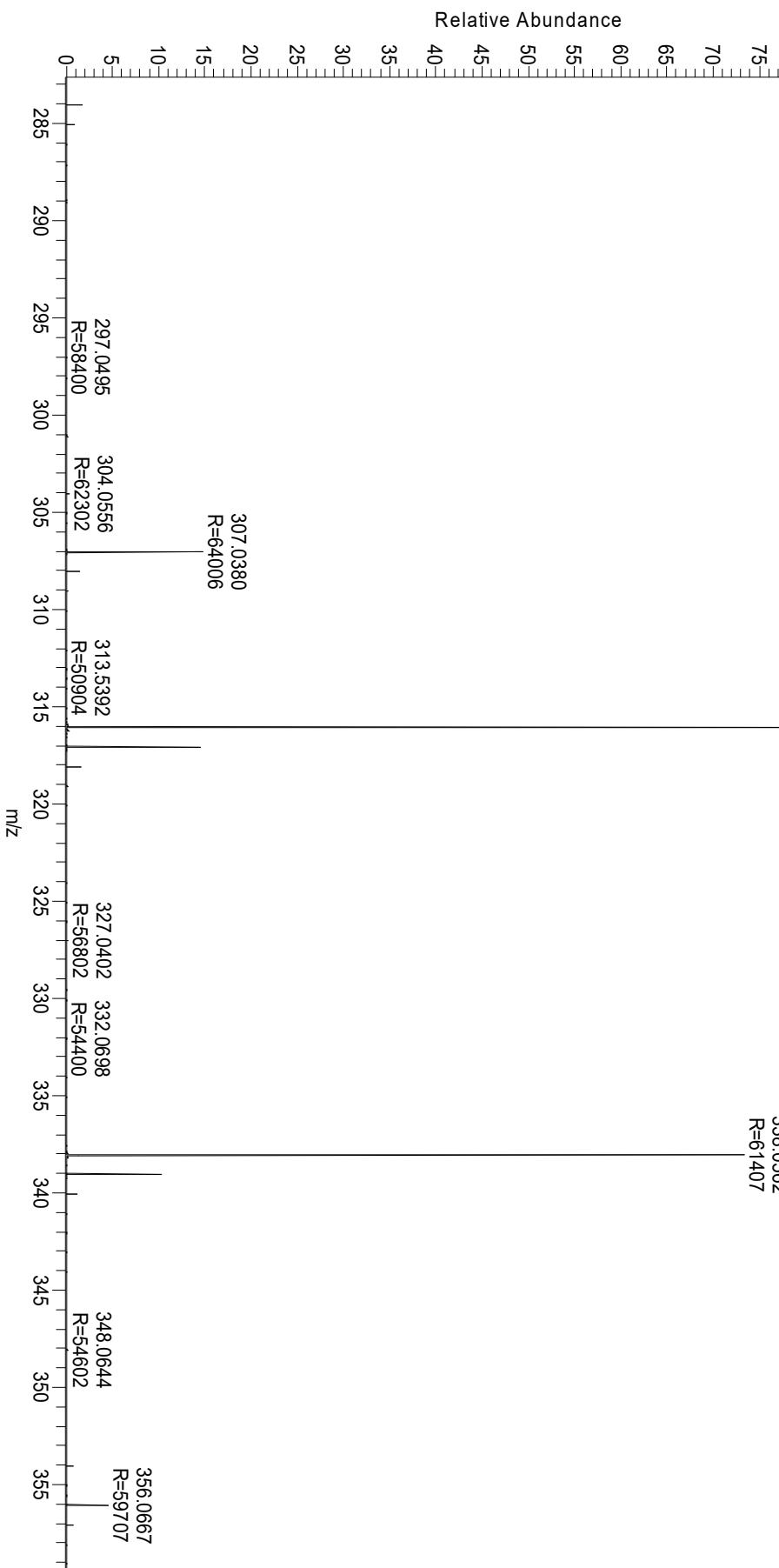
RSP-37 #100 RT: 0.44 AV: 1 NL: 3.94E9
T: FTMS + p ESI Full ms [100.00-1500.00]

316.0744
R=64007
 $C_{13}H_{16}O_5NF$ P = 316.0745
-0.0994 ppm

338.0562
R=61407

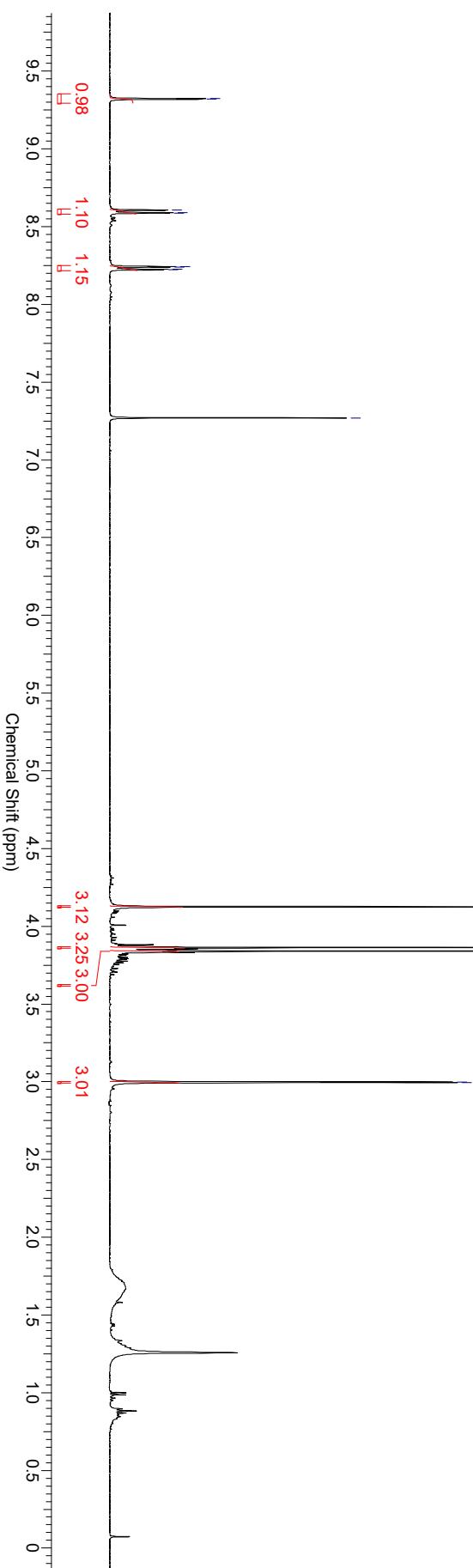
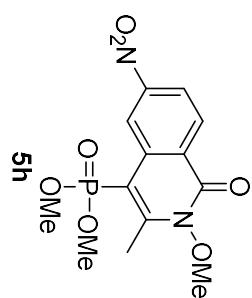


5g



¹H NMR Spectra for Compound 5h in CDCl₃

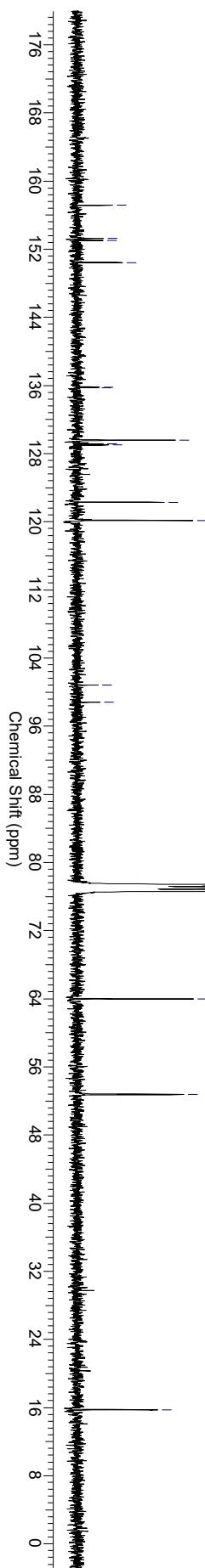
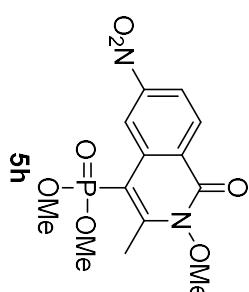
Acquisition Time (sec)	1.2800	Comment	Ravindra 1H	Date	10 Feb 2016 12:52:32	
Date Stamp	10 Feb 2016 12:52:32					
File Name	\172.16.2.4\nmr\data\AV500\2016_AV500\FEB_16_AV500\W\ed2av500#010\1\PDAT\A\1\1r					
Nucleus	1H	Number of Transients	37	Origin	spec	
Owner	nmr	Points Count	32768	Pulse Sequence	zg30	
SW(cyclical) (Hz)	12500.00	Solvent	CHLOROFORM-d	Receiver Gain	322.00	
Spectrum Type	STANDARD	Sweep Width (Hz)	12499.62	Temperature (degree C)	24.800	
					Spectrum Offset (Hz)	3492.7031



SI 110

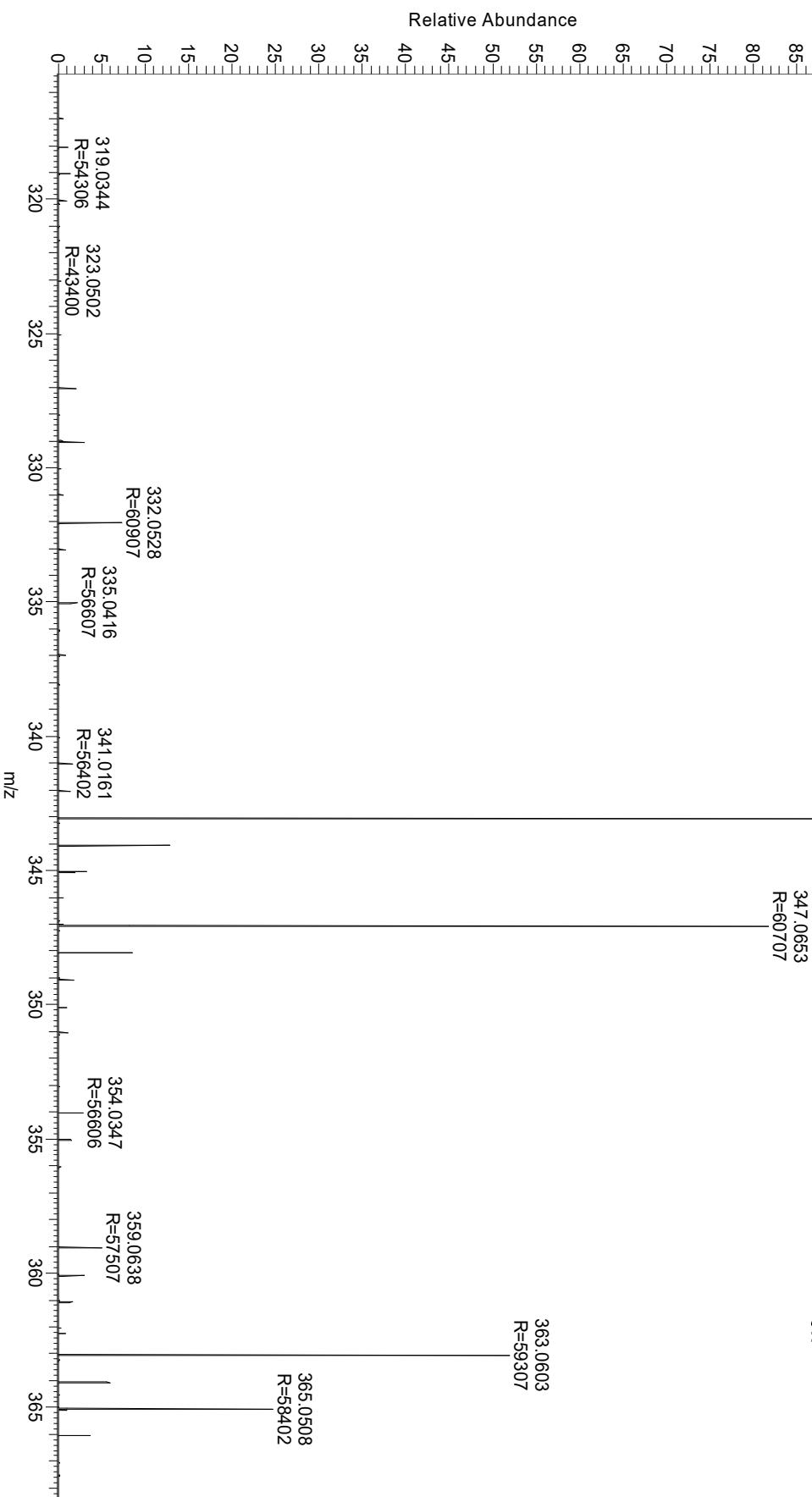
¹³C NMR Spectra for Compound 5h in CDCl₃

Acquisition Time (sec)	1.0434	Comment	Ravindra	Date	14 Feb 2016 17:03:59
Date Stamp	14 Feb 2016 16:00:57				
File Name	\172\16.2.4\nmr\400\2016_JEOL_400FEB_LIQUID_2016\Sat2ECX400#003	Number of Transients	3000	Frequency (MHz)	100.53
Nucleus	¹³ C	Origin	ECX 400	Original Points Count	26214
Owner	delta	Pulse Sequence	single pulse dec	Receiver Gain	60.00
Solvent	CHLOROFORM-d	Spectrum Offset (Hz)	10043.1660	Spectrum Type	STANDARD
Sweep Width (Hz)	25124.29	Temperature (degree C)	23.200		

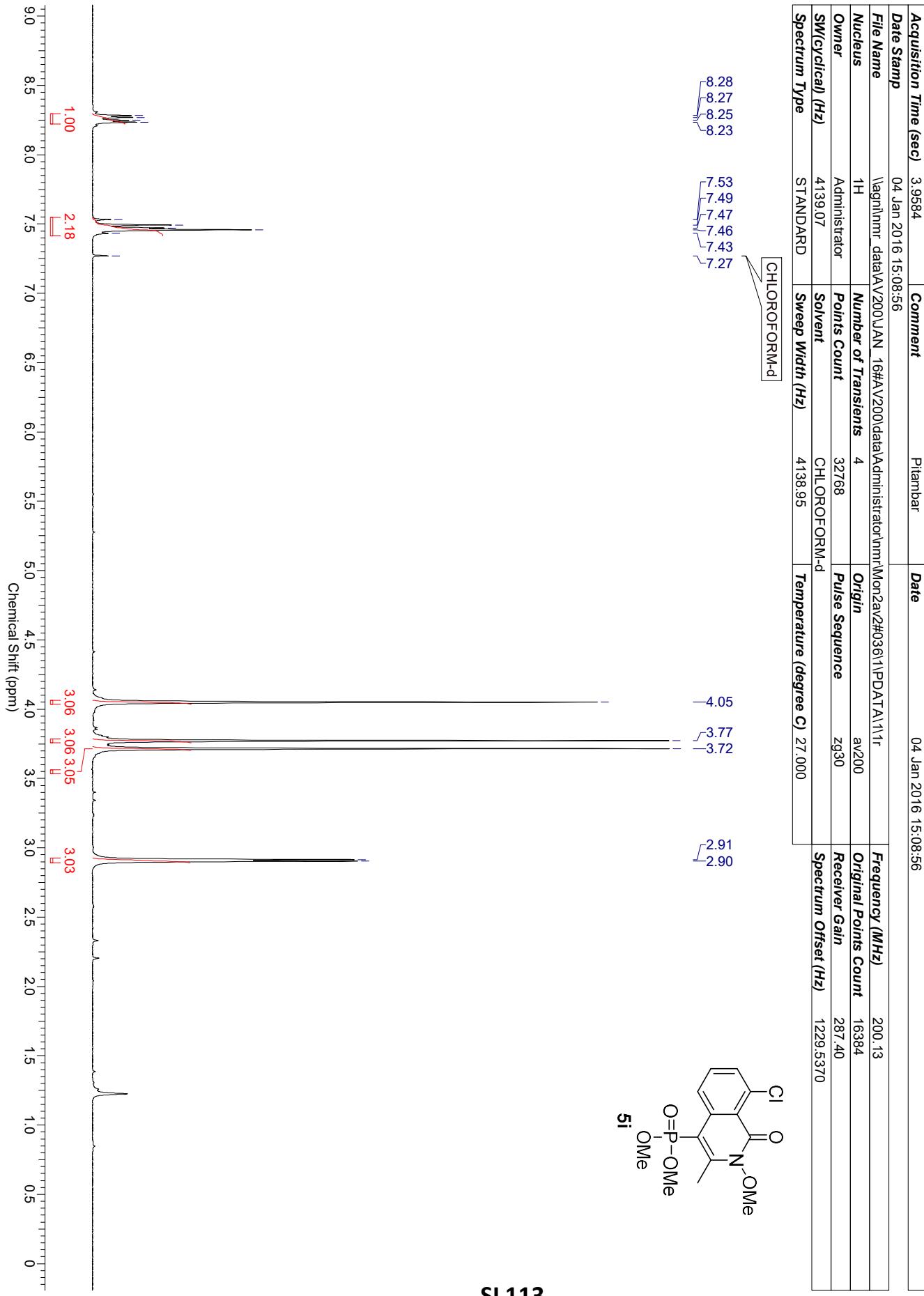


HRMS Spectra for Compound **5h** in MeOH

RSP-29 #96 RT: 0.42 AV: 1 NL: 2.29E8
T: FTMS + pESI[Full ms [[100.00-1500.00]]]



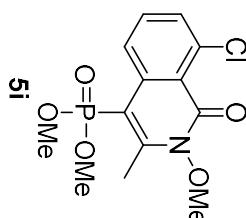
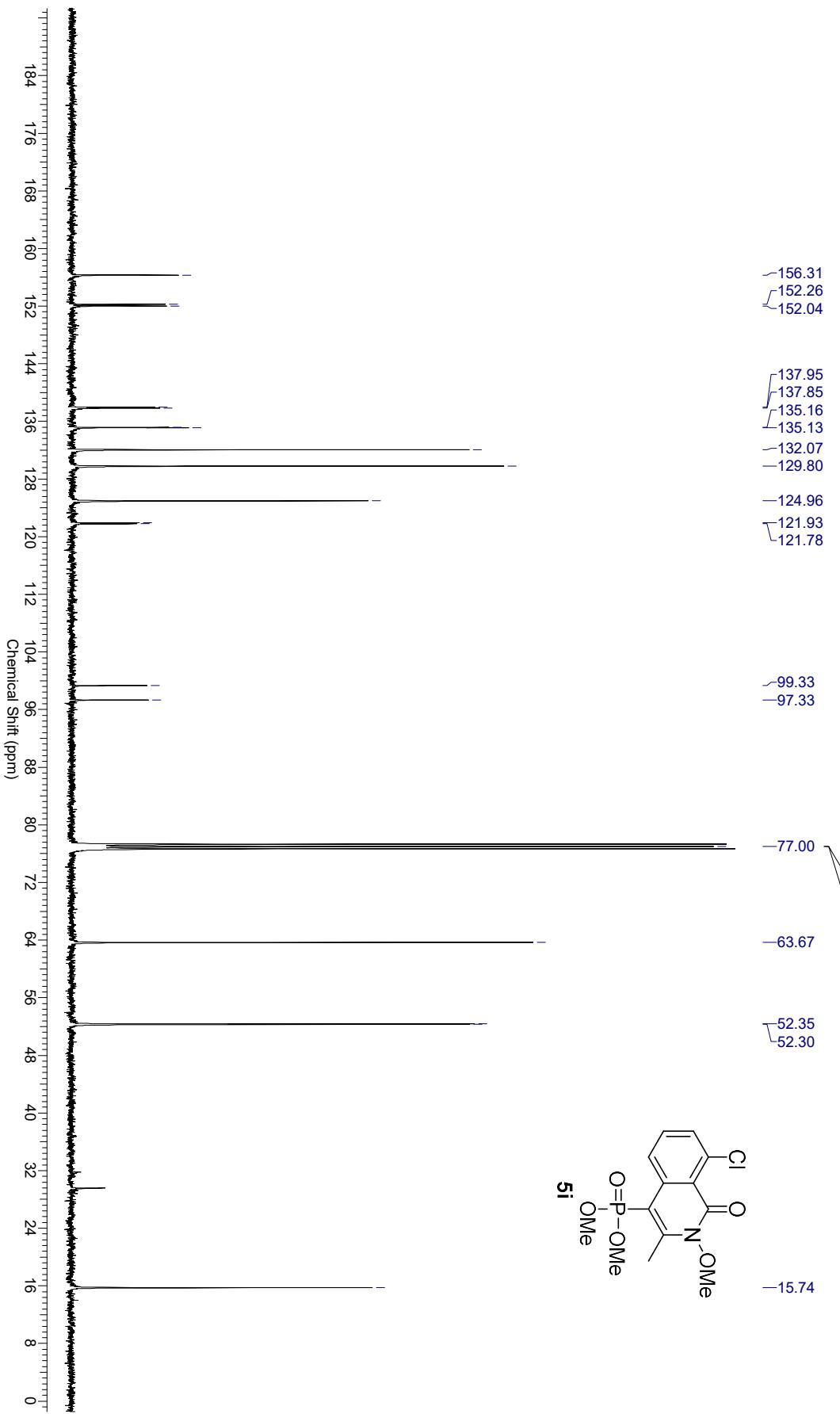
¹H NMR Spectra for Compound 5i in CDCl₃



SI 113

¹³C NMR Spectra for Compound 5i in CDCl₃

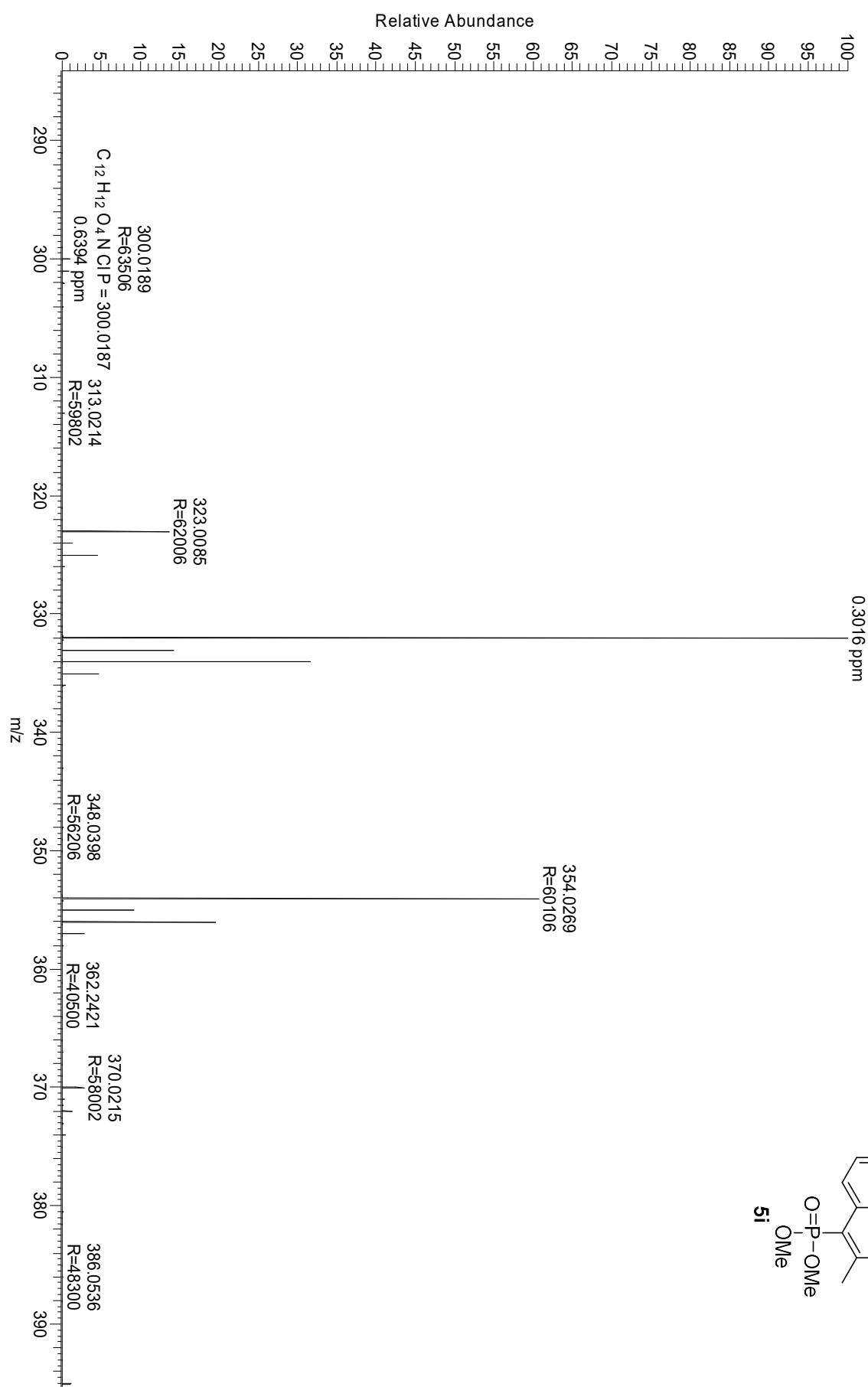
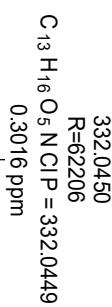
Acquisition Time (sec)	1.0434	Comment	Ravindra	Date	07.Jan.2016 08:22:31
Date Stamp	06.Jan.2016 15:55:50			File Name	\lagnmr\data\JEOL_400\2016\JAN\LIQUID\TrueECX400#026_CARBON-3.jdf
Frequency (MHz)	100.53	Nucleus	¹³ C	Number of Transients	1200
Original Points Count	26214	Owner	delta	Points Count	26214
Receiver Gain	60.00	Solvent	CHLOROFORM-d	Pulse Sequence	single pulse dec
Spectrum Type	STANDARD	Sweep Width (Hz)	25124.29	Temperature (degree C)	23.900
				Spectrum Offset (Hz)	10037.4141



SI 114

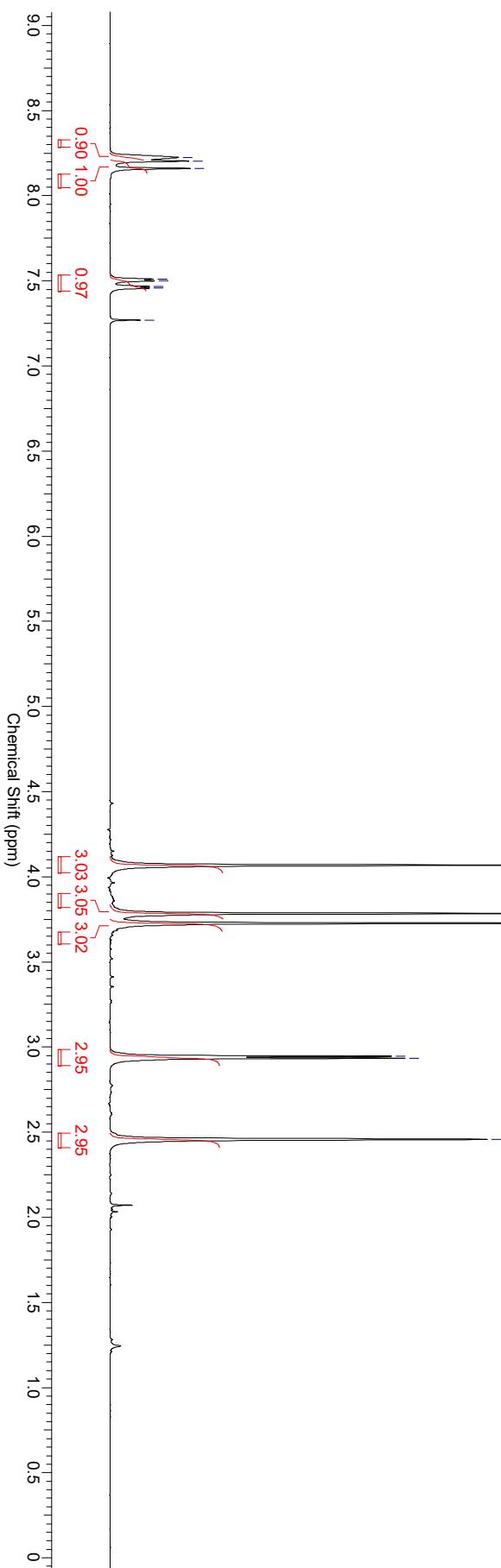
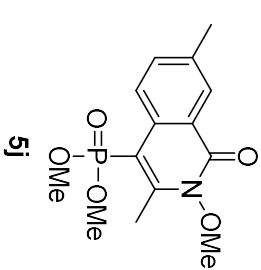
HRMS Spectra for Compound **5i** in MeOH

RSP-26_160105161903 #104 RT: 0.46 AV: 1 NL: 3.01E9
T: FTMS + p ESI|Full ms [100.00-1500.00]



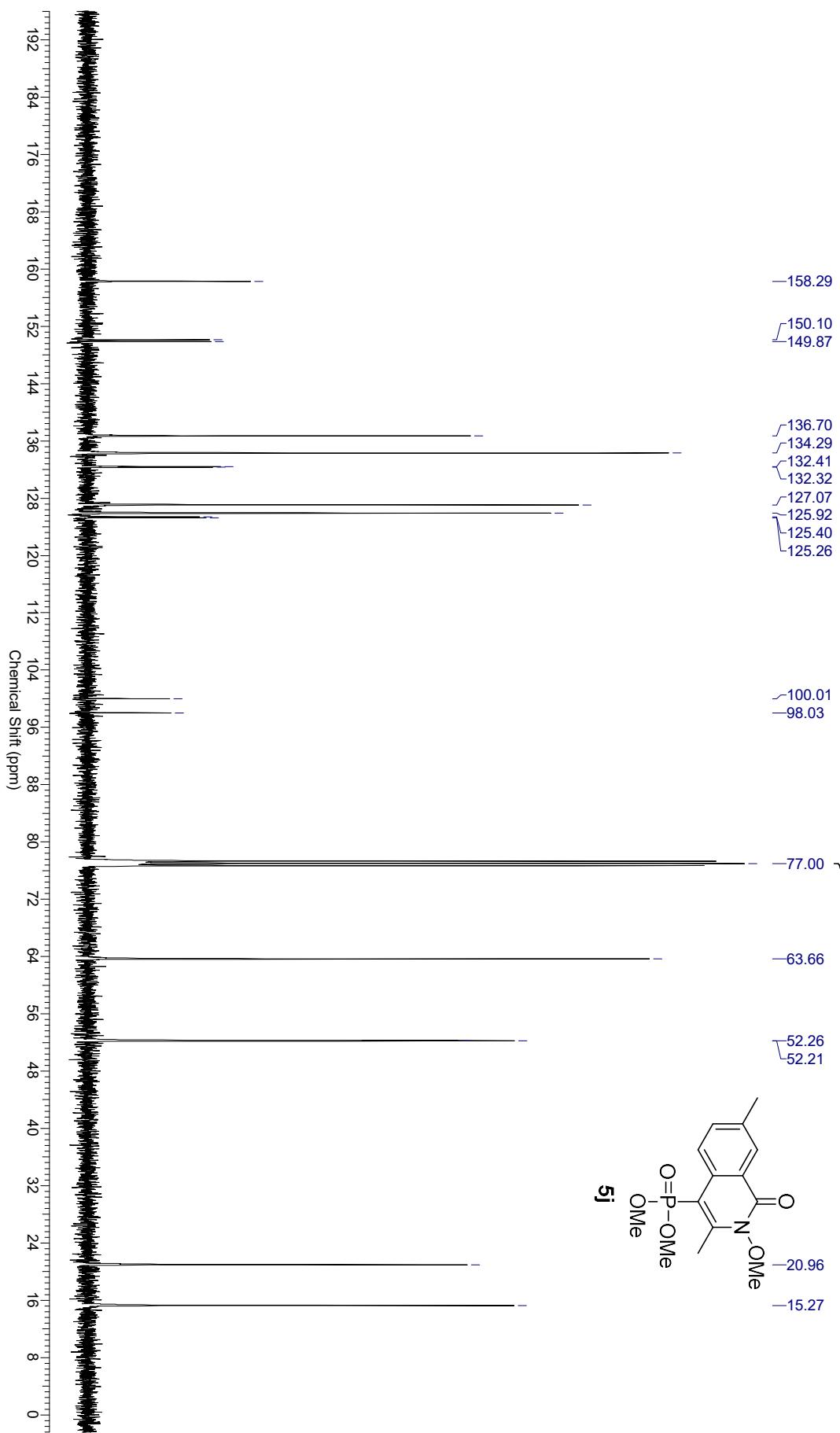
¹H NMR Spectra for Compound 5j in CDCl₃

Acquisition Time (sec)	3.9584	Comment	Ravindra	Date	07 Dec 2015 14:43:20
Date Stamp	07 Dec 2015 14:43:20				
File Name	\lagnmr\data\AV200\DEC_15#AV200\data\Administrator\1nm\Mon2a2#040\1PDATA\11r			Frequency (MHz)	200.13
Nucleus	1H			Original Points Count	16384
Owner	Administrator			Pulse Sequence	av200
SW(cyclical) (Hz)	4139.07			Receiver Gain	322.50
Spectrum Type	STANDARD			Spectrum Offset (Hz)	1229.1580
Sweep Width (Hz)	4138.95			Temperature (degree C)	27.000



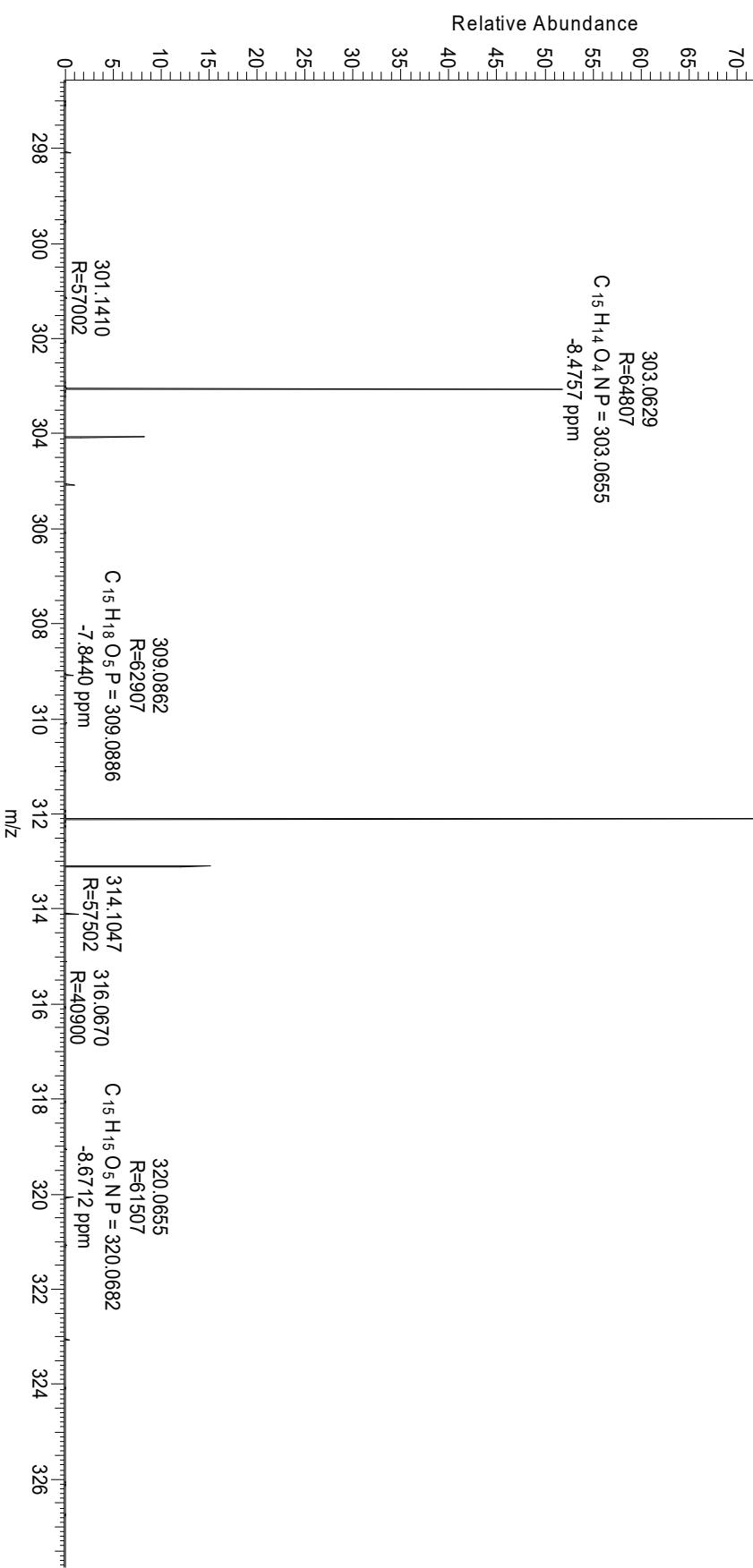
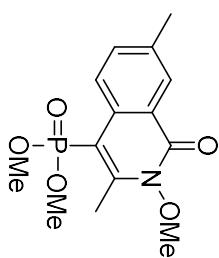
¹³C NMR Spectra for Compound 5j in CDCl₃

Acquisition Time (sec)	0.6488	Comment	13C	Date	23 Dec 2015 17:38:16
Date Stamp	23 Dec 2015 17:38:16			File Name	\agnmr_data\AV400\Dec_15_400\Wed4av400#016\b3\DATA\111r
Frequency (MHz)	100.61	Nucleus	13C	Number of Transients	633
Original Points Count	16384	Owner	root	Points Count	32768
Receiver Gain	2050.00	SW(cyclical) (Hz)	25252.53	Solvent	CHLOROFORM-d
Spectrum Offset (Hz)	10053.3408	Spectrum Type	STANDARD	Sweep Width (Hz)	25251.75
				Temperature (degree C)	22.500



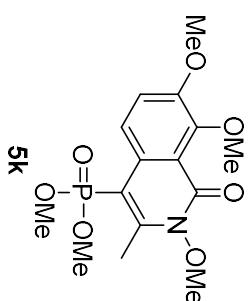
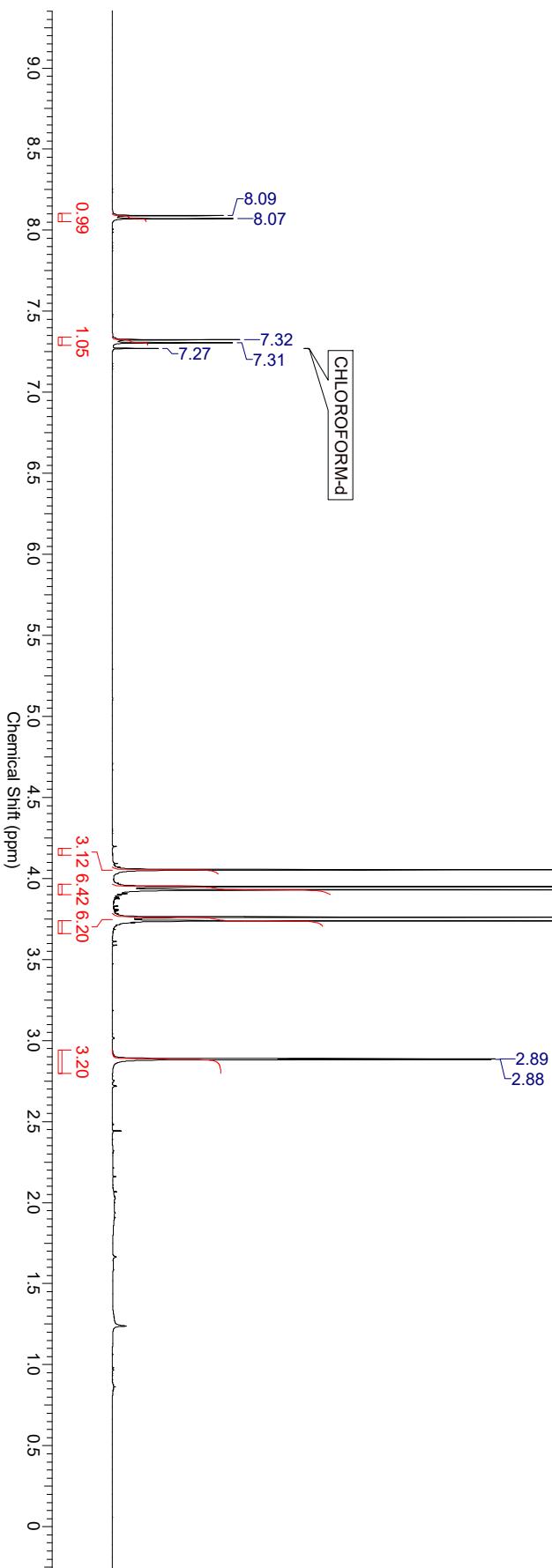
HRMS Spectra for Compound 5j in MeOH

RR-A-2#99 RT: 0.44 AV: 1 NL: 1.60E9
T: FTMS + p ESI Full ms [100.00-1500.00]

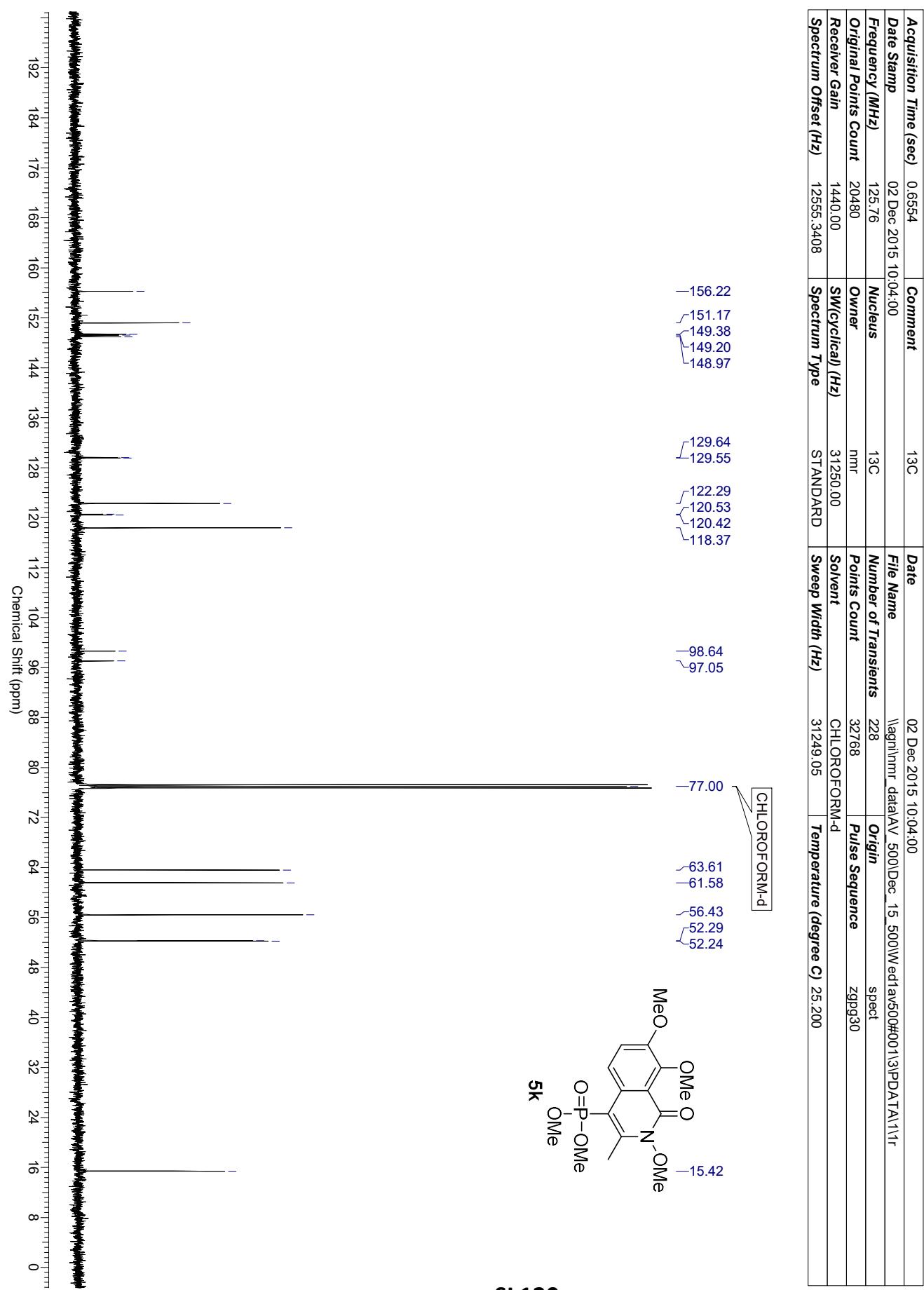


¹H NMR Spectra for Compound 5k in CDCl₃

Acquisition Time (sec)	1.6000	Comment	Pitambar 1H	Date	02 Dec 2015 09:59:44
Date Stamp	02 Dec 2015 09:59:44	File Name	\agnmr_data\AV_500\Dec_15_500\Wed\av500#001\1\PDAT\AV\1\1r		
Frequency (MHz)	500.13	Nucleus	1H	Number of Transients	64
Original Points Count	16000	Owner	nmr	Points Count	32768
Receiver Gain	161.00	SW(cyclicall) (Hz)	10000.00	Solvent	CHLOROFORM-d
Spectrum Offset (Hz)	2492.2988	Spectrum Type	STANDARD	Sweep Width (Hz)	9999.70
				Temperature (degree C)	24.900



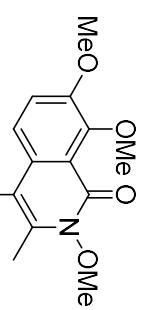
¹³C NMR Spectra for Compound 5k in CDCl₃



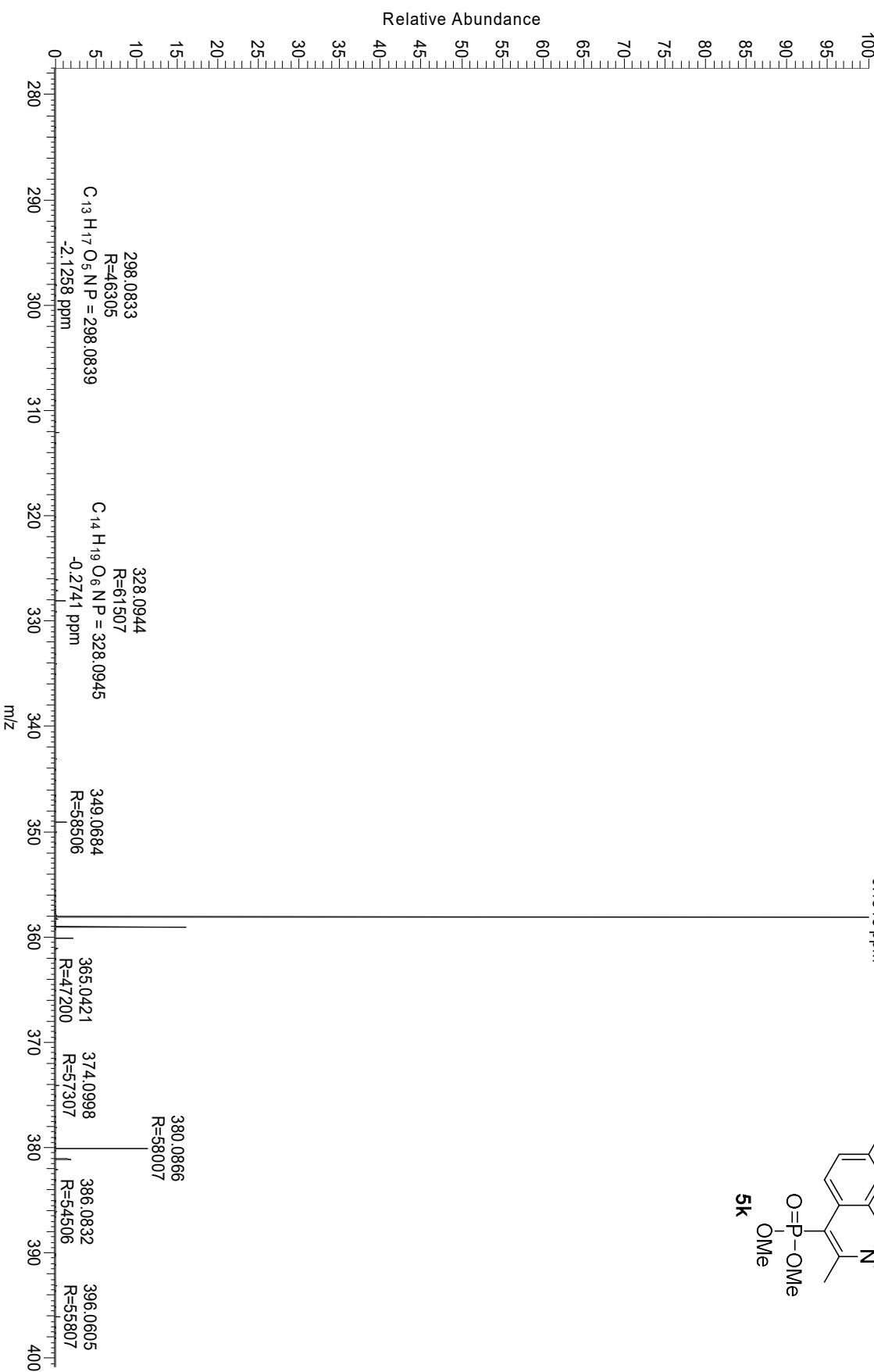
HRMS Spectra for Compound **5k** in MeOH

RSP-24 #98 RT: 0.44 AV: 1 NL: 6.52E9
T: FTMS + pESI[Full ms] [100.00-1500.00]

358.1049
R=59807
C₁₅H₂₁O₇NP = 358.1050
-0.1815 ppm

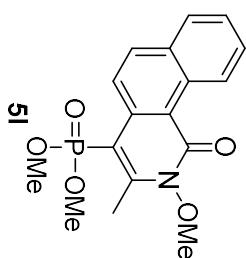
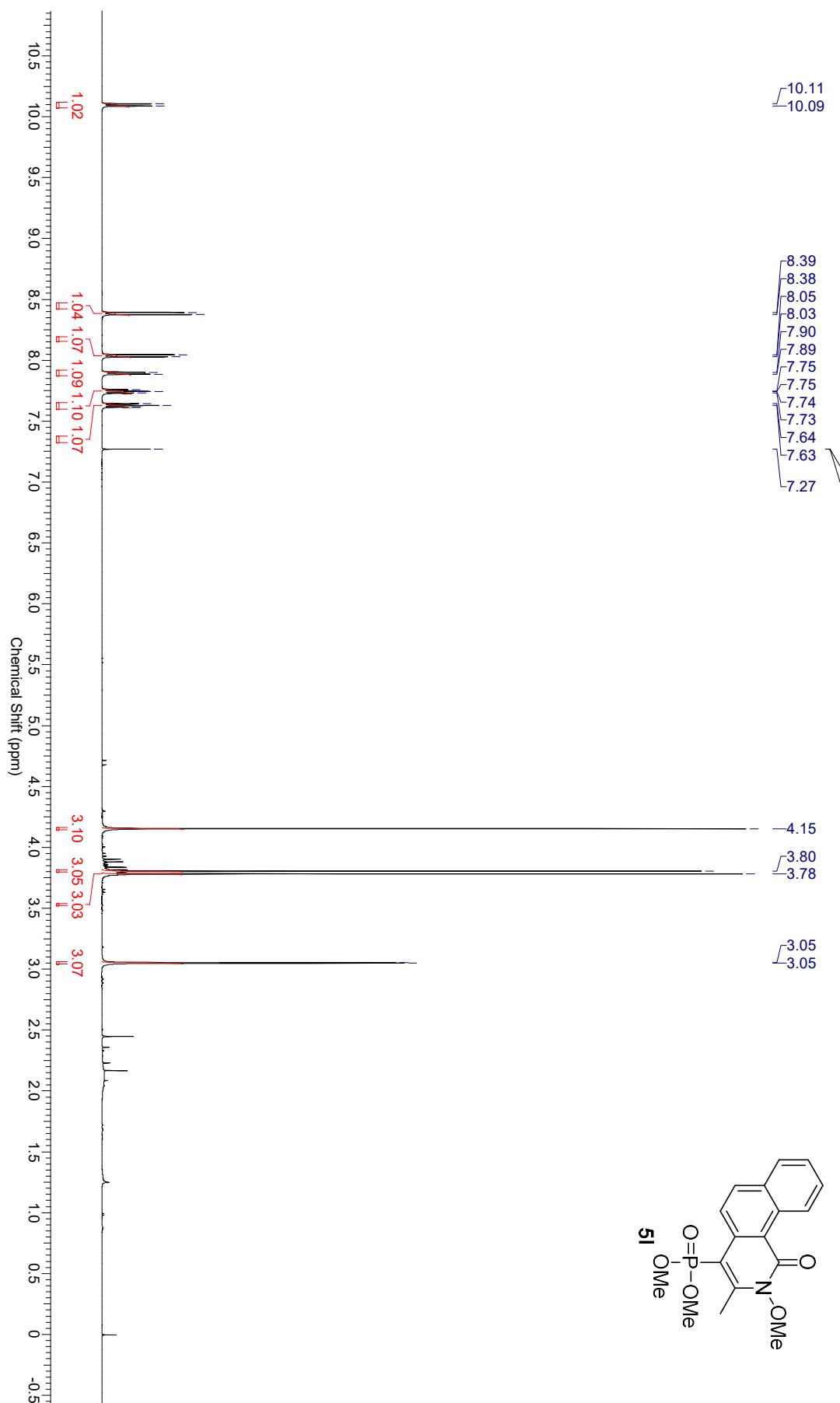


5k



¹H NMR Spectra for Compound 5l in CDCl₃

Acquisition Time (sec)	1.6000	Comment	Ravindra 1H	Date	01 Dec 2015 15:58:08
Date Stamp	01 Dec 2015 15:58:08			File Name	\agni\nmr\data\AV_500\Dec_15_500\Tue1\av500#016\1\PDAT\1\1r
Frequency (MHz)	500.13	Nucleus	¹ H	Number of Transients	64
Original Points Count	16000	Owner	nmr	Points Count	32768
Receiver Gain	144.00	SW(Cyclical) (Hz)	10000.00	Solvent	CHLOROFORM-d
Spectrum Offset (Hz)	2492.2988	Spectrum Type	STANDARD	Sweep Width (Hz)	9999.70
				Temperature (degree C)	24.900



SI 122

¹³C NMR Spectra for Compound **5l** in CDCl₃

Acquisition Time (sec)	0.6554	Comment	13C	Date	01 Dec 2015 16:19:28
Date Stamp	01 Dec 2015 16:19:28	File Name	\\agni\\nmr\\dataAV\\500\\Dec_15_500\\T1e1av500#016\\3\\PDATA\\\\1r		
Frequency (MHz)	125.76	Nucleus	13C	Number of Transients	1479
Original Points Count	20480	Owner	nmr	Points Count	32768
Receiver Gain	1440.00	SW(cyclical) (Hz)	31250.00	Pulse Sequence	zpg30
Spectrum Offset (Hz)	12554.3857	Spectrum Type	STANDARD	Solvent	CHLOROFORM-d
				Sweep Width (Hz)	31249.05
				Temperature (degree C)	25.200

—158.46

—151.95

—151.77

—137.35

—137.27

—133.81

—131.43

—128.62

—127.91

—127.17

—126.85

—123.33

—119.20

—119.09

—99.89

—98.29

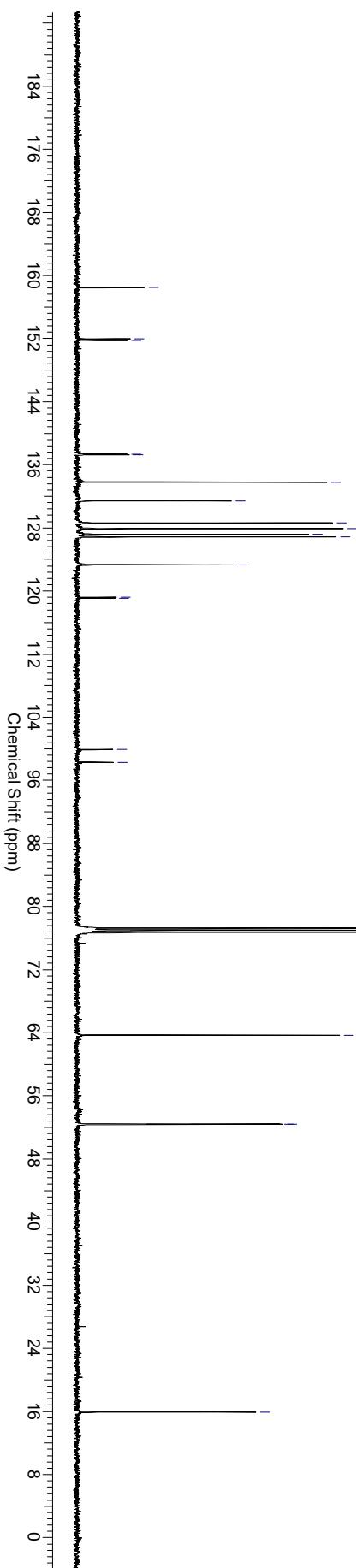
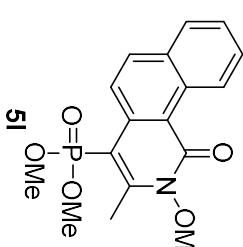
—77.00

—63.70

—52.42

—52.38

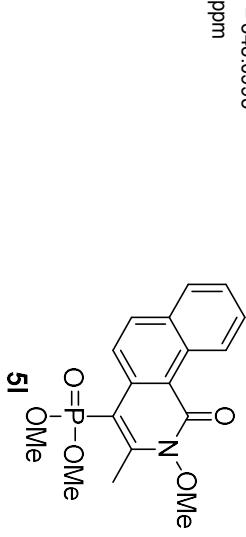
—15.92



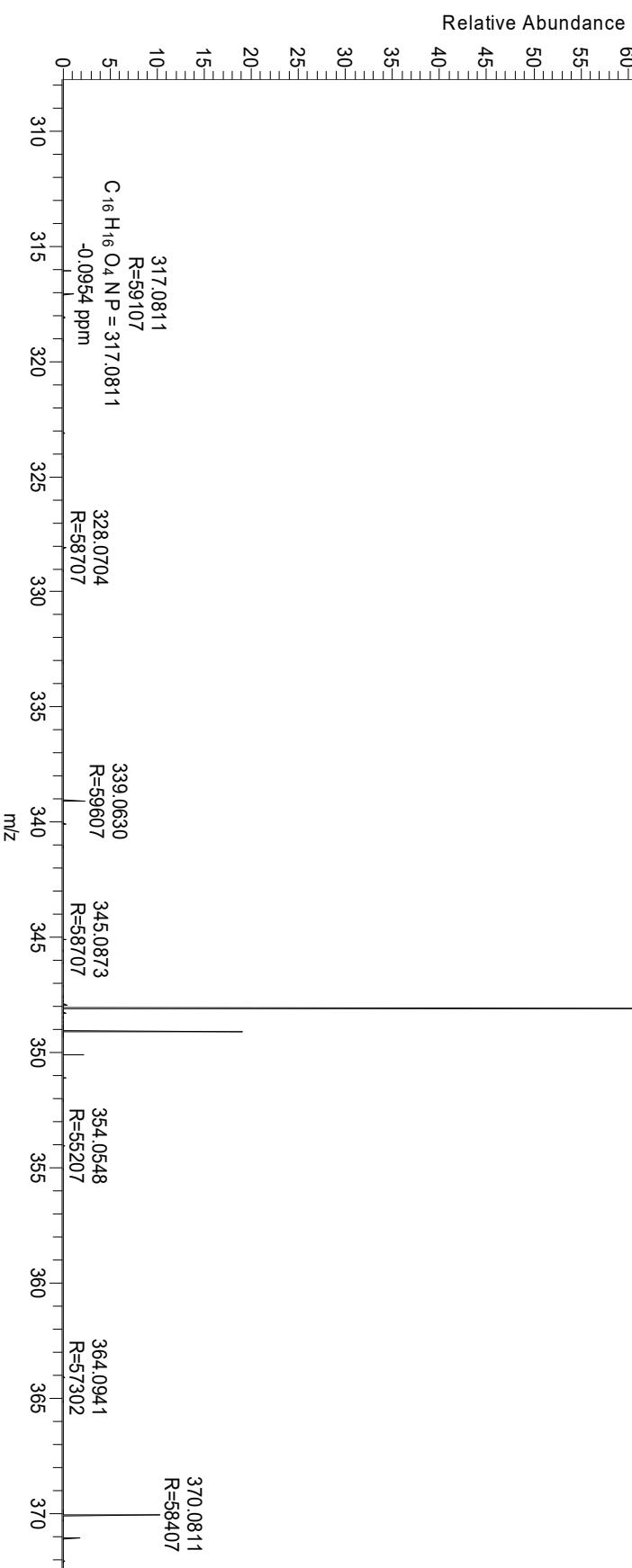
HRMS Spectra for Compound **5l** in MeOH

RSP-23 #108 RT: 0.48 AV: 1 NL: 5.10E9
T: FTMS + pESIFull.ms [100.00-1500.00]

348.0995
R=61003
 $C_{17}H_{19}O_5NP = 348.0995$
-0.2266 ppm



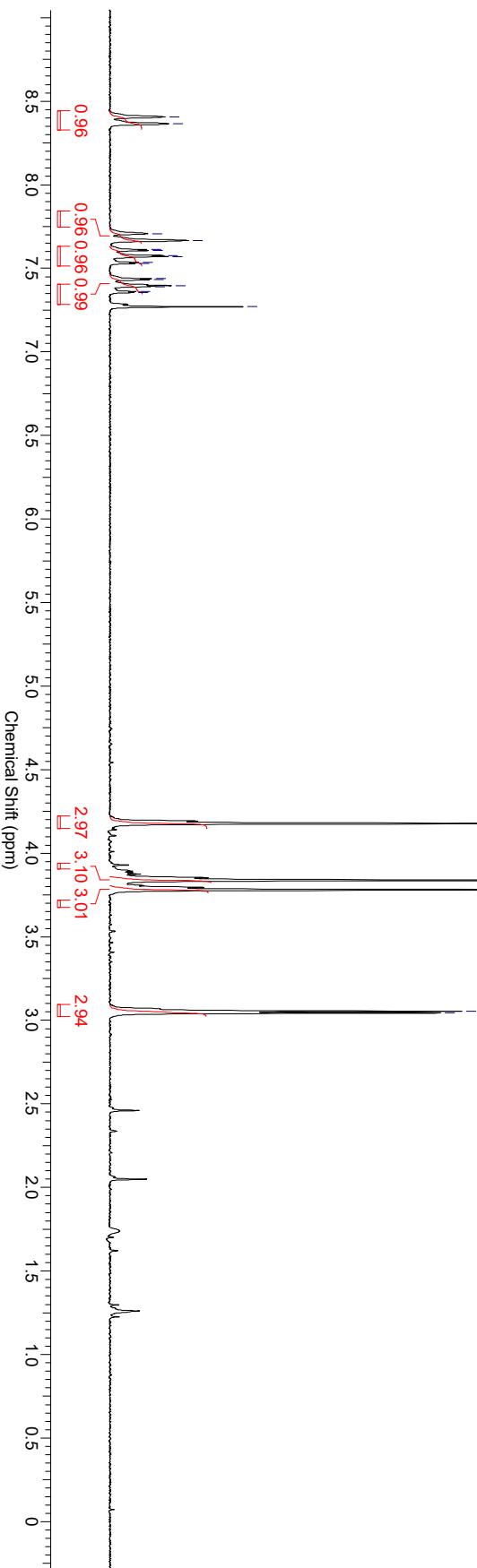
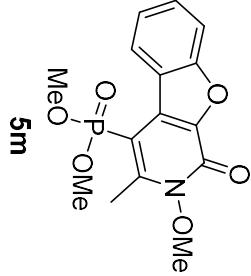
5l



SI 124

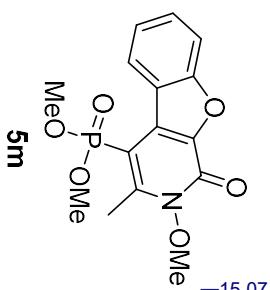
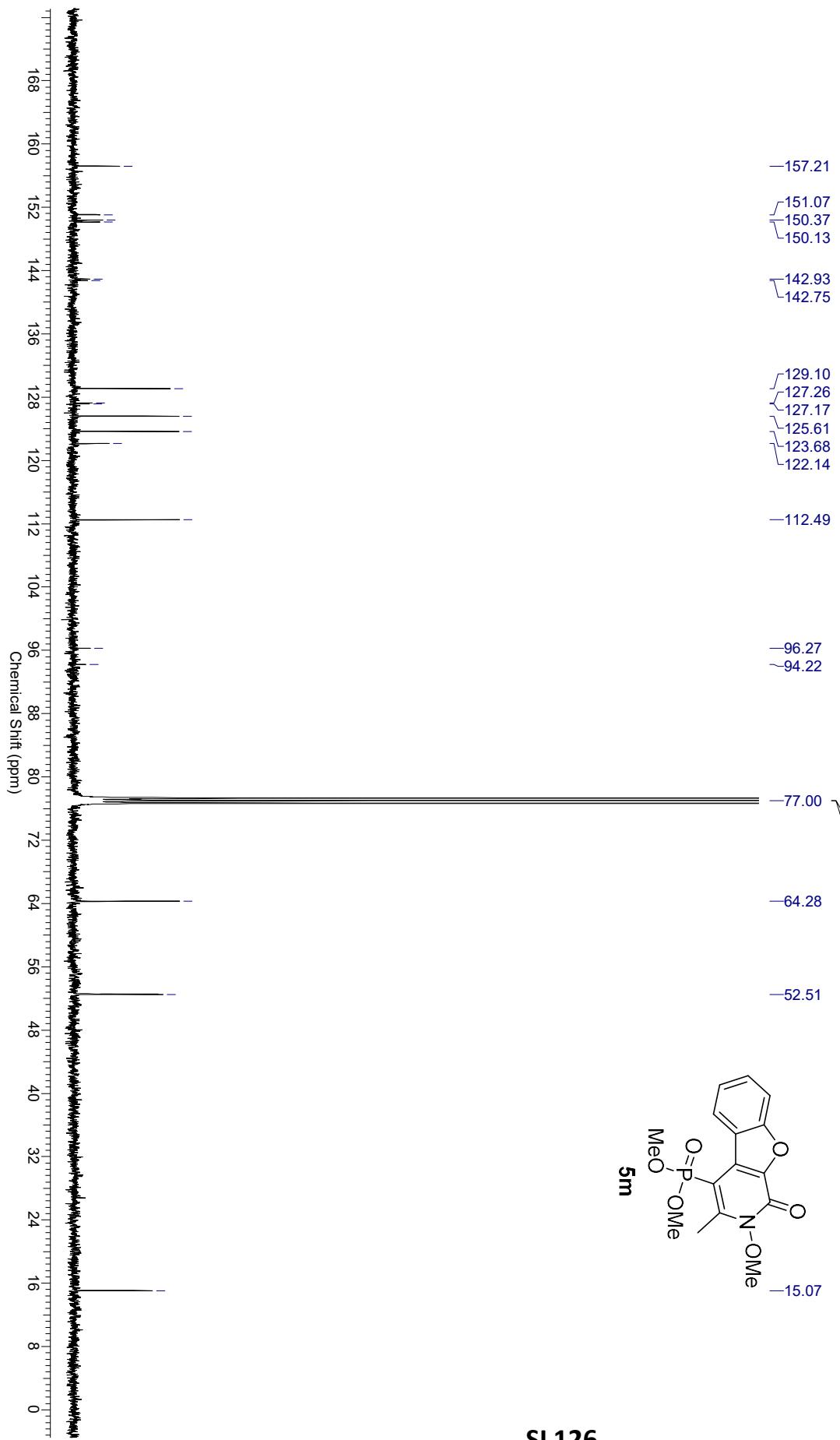
¹H NMR Spectra for Compound 5m in CDCl₃

Acquisition Time (sec)	3.9584	Comment	Ravindra	Date	06 Feb 2016 13:37:12
Date Stamp	06 Feb 2016 13:37:12				
File Name	\172.16.2.4\nmr\data\AV200\2016#AV200\FEB\16#AV200\data\Administrator\mmmlSat1ax2#\002\1\PDATA\1\1\				
Frequency (MHz)	200.13	Nucleus	1H	Number of Transients	4
Original Points Count	16384	Owner	Administrator	Points Count	32768
Receiver Gain	1149.40	SW(cyclical) (Hz)	4139.07	Solvent	CHLOROFORM-d
Spectrum Offset (Hz)	1229.1581	Spectrum Type	STANDARD	Sweep Width (Hz)	4138.95
				Temperature (degree C)	27.000



¹³C NMR Spectra for Compound 5m in CDCl₃

Acquisition Time (sec)	1.0434	Comment	Ravindra	Date	12.Jan.2016 13:59:54
Date Stamp	12.Jan.2016 03:21:03			File Name	\1172.16.24\NMR\data\jeol_400_new\Liquid Jan 16\Mon3ECX400#003_CARBON-3.jdf
Frequency (MHz)	100.53	Nucleus	¹³ C	Number of Transients	1200
Original Points Count	26214	Owner	delta	Points Count	26214
Receiver Gain	60.00	Solvent	CHLOROFORM-d	Pulse Sequence	single pulse dec
Spectrum Type	STANDARD	Sweep Width (Hz)	25124.29	Temperature (degree C)	22.600
				Origin	ECX 400
				Spectrum Offset (Hz)	10043.1650

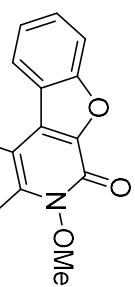


HRMS Spectra for Compound **5m** in MeOH

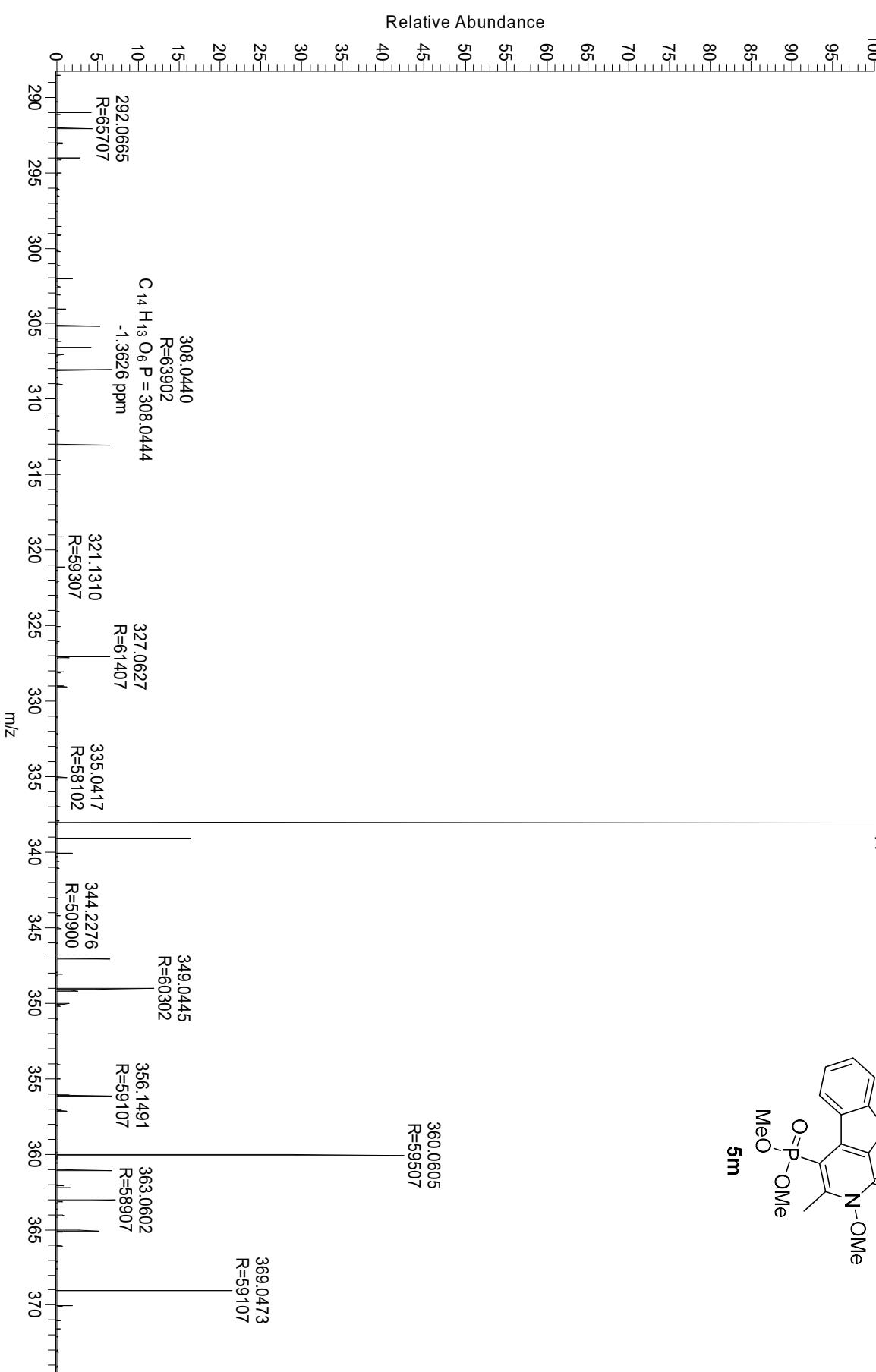
RSP-30 #101 RT: 0.45 AV: 1 NL: 3.39E8
T: FTMS + pESI[Full ms [100.00-1500.00]]

338.0786
R=61507

C₁₅H₁₇O₆NP = 338.0788
-0.7335 ppm

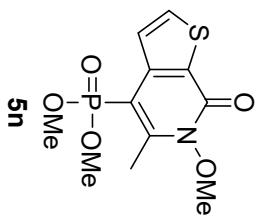
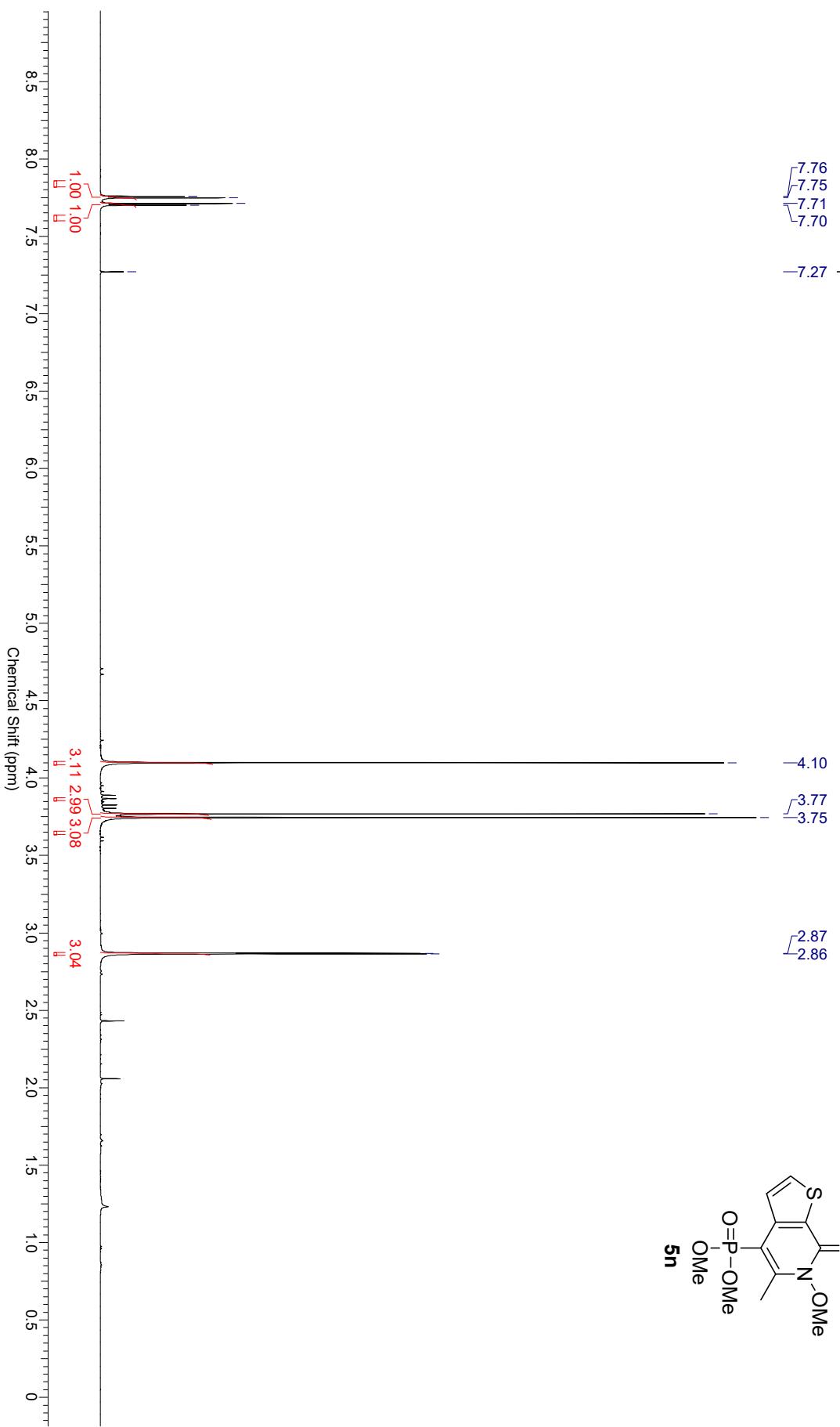


5m
R=59507



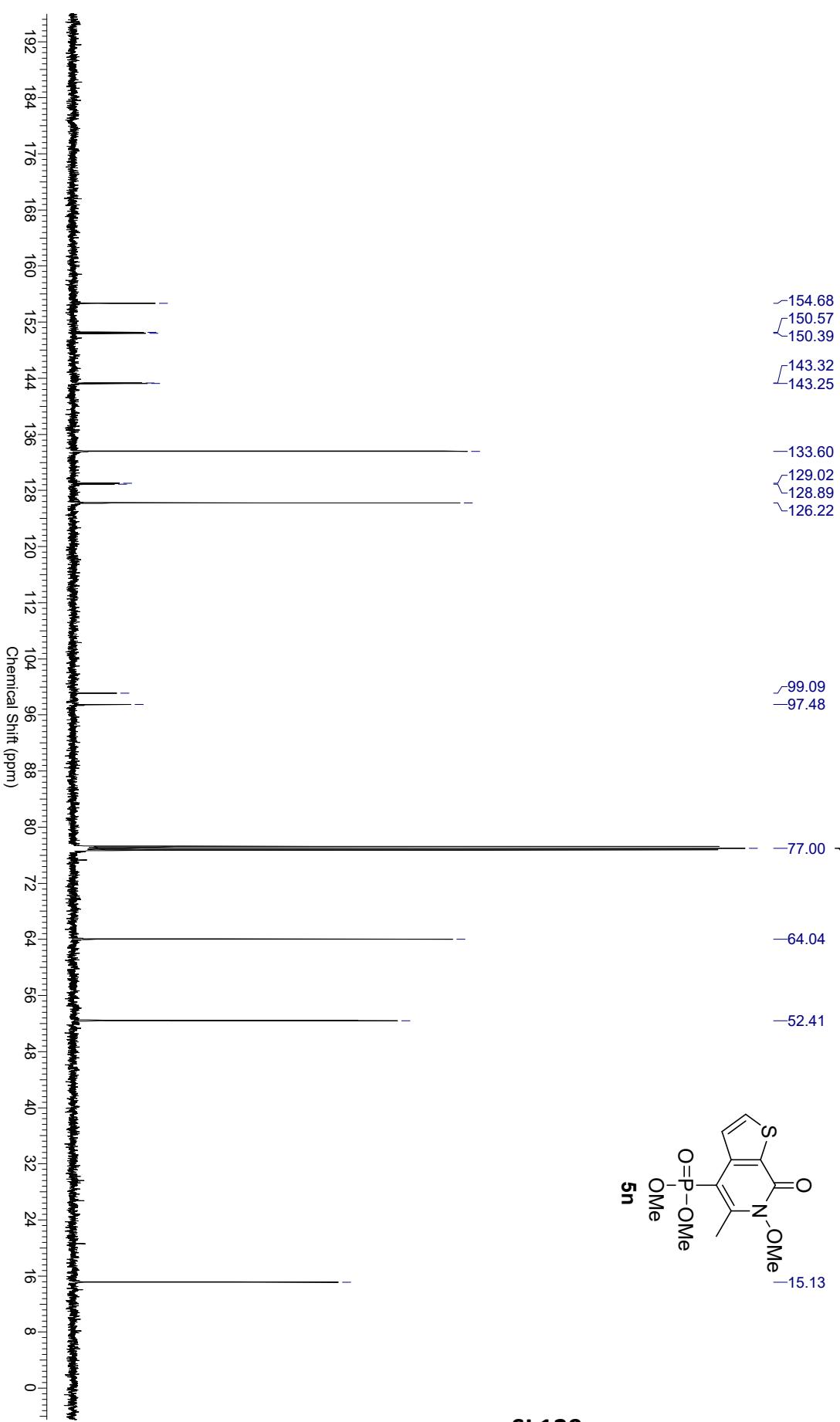
¹H NMR Spectra for Compound 5n in CDCl₃

Acquisition Time (sec)	1.6000	Comment	pitambar 1H	Date	02 Dec 2015 11:27:12
Date Stamp	02 Dec 2015 11:27:12			File Name	\laginlnmr\data\AV_500\Dec_15_500\Wed1av500#0041\PDAT\A11\1r
Frequency (MHz)	500.13	Nucleus	1H	Number of Transients	64
Original Points Count	16000	Owner	nmr	Points Count	32768
Receiver Gain	144.00	SW(cyclical) (Hz)	10000.00	Solvent	CHLOROFORM-d
Spectrum Offset (Hz)	2492.2988	Spectrum Type	STANDARD	Sweep Width (Hz)	9999.70
				Temperature (degree C)	24.900



¹³C NMR Spectra for Compound 5n in CDCl₃

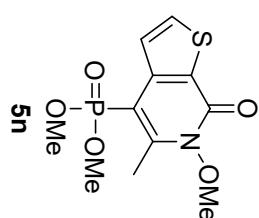
Acquisition Time (sec)	0.6554	Comment	13C	Date	02 Dec 2015 11:29:20
Date Stamp	02 Dec 2015 11:29:20			File Name	\agn\Innr\data\AV_500\Dec_15_500\Wed1av500#004\3\PDATA\11r
Frequency (MHz)	125.76	Nucleus	13C	Number of Transients	394
Original Points Count	20480	Owner	nmr	Points Count	32768
Receiver Gain	1440.00	SW(cyclical) (Hz)	31250.00	Pulse Sequence	zgpg30
Spectrum Offset (Hz)	12553.4326	Spectrum Type	STANDARD	Sweep Width (Hz)	31249.05
				Temperature (degree C)	25.100



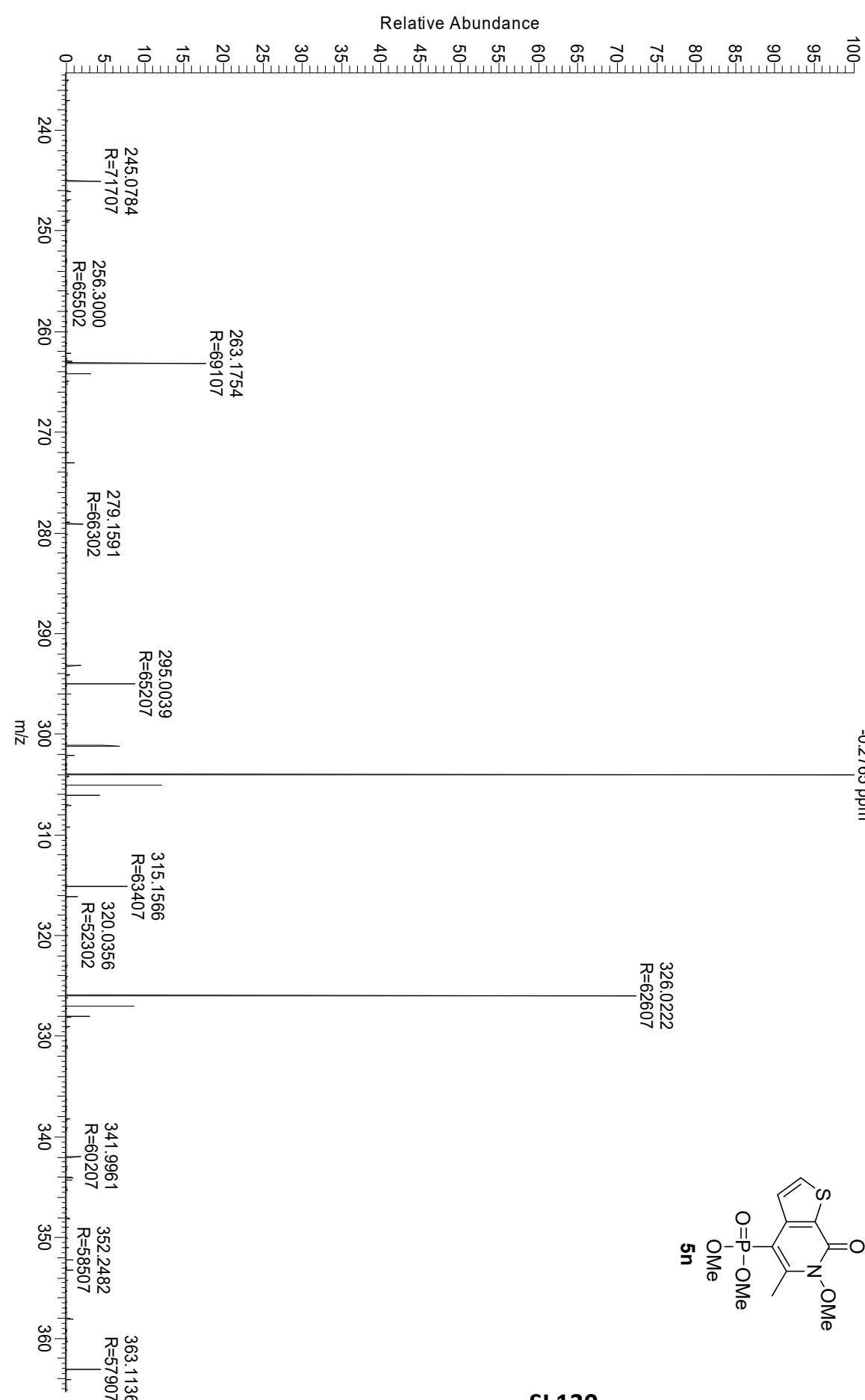
HRMS Spectra for Compound **5n** in MeOH

RSP-25#148 RT: 0.66 AV: 1 NL: 1.41E8
T: FTMS + pESI|Full ms [100.00-1500.00]

304.0402
R=64607
 $C_{11}H_{15}O_5NPS = 304.0403$
-0.2765 ppm



326.0222
R=62607



¹H NMR Spectra for Compound 5o in CDCl₃

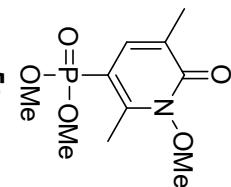
Acquisition Time (sec)	1.2800	Comment	Ravindra 1H	Date	10 Feb 2016 12:48:16
Date Stamp	10 Feb 2016 12:48:16				
File Name	\\\172.16.2.4\mmr\data\AV500\2016_AV500\FEB_16_AV500\W\ed2av\500#0091\PDAT\1\1r			Frequency (MHz)	500.13
Nucleus	1H	Number of Transients	32	Origin	spect
Owner	nmr	Points Count	32768	Pulse Sequence	zg30
SW(cyclical) (Hz)	12500.00	Solvent	CHLOROFORM-d	Receiver Gain	322.00
Spectrum Type	STANDARD	Sweep Width (Hz)	12499.62	Temperature (degree C)	25.000
				Spectrum Offset (Hz)	3492.7031

CHLOROFORM-d

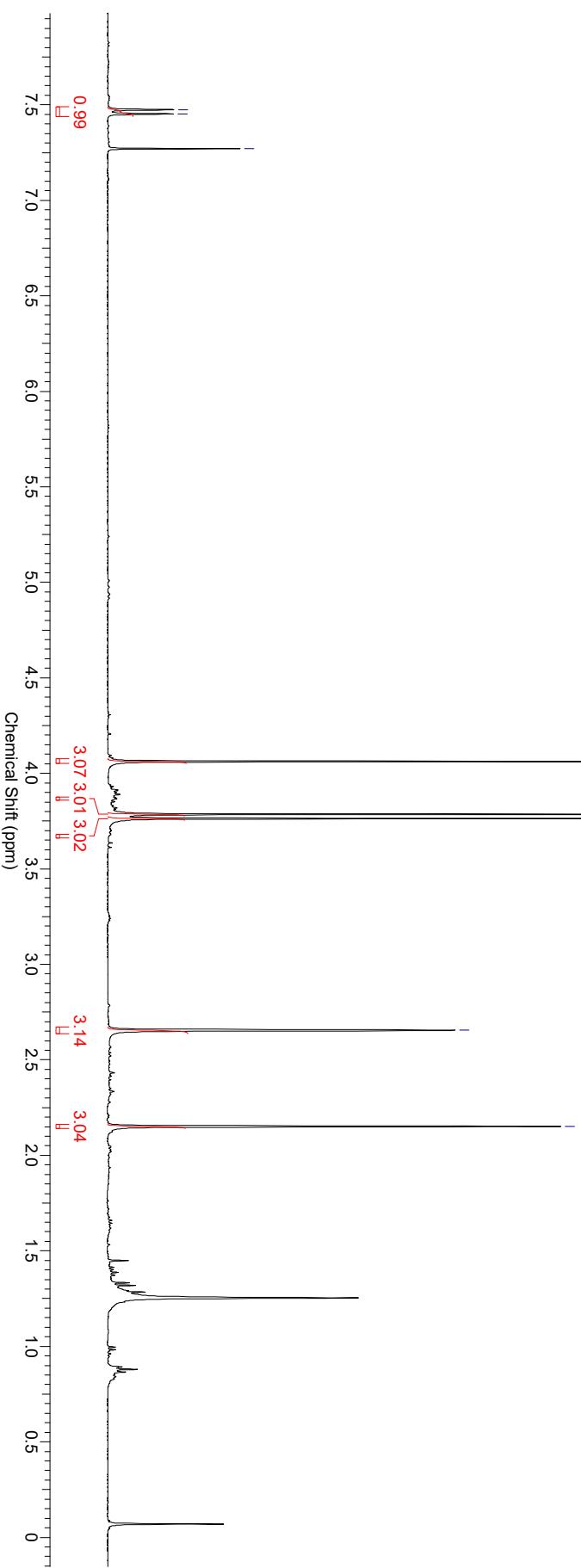
7.47
7.45
7.27

4.06
3.79
3.76

2.66
2.15

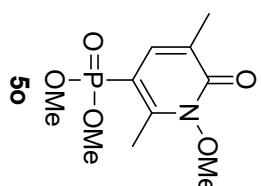
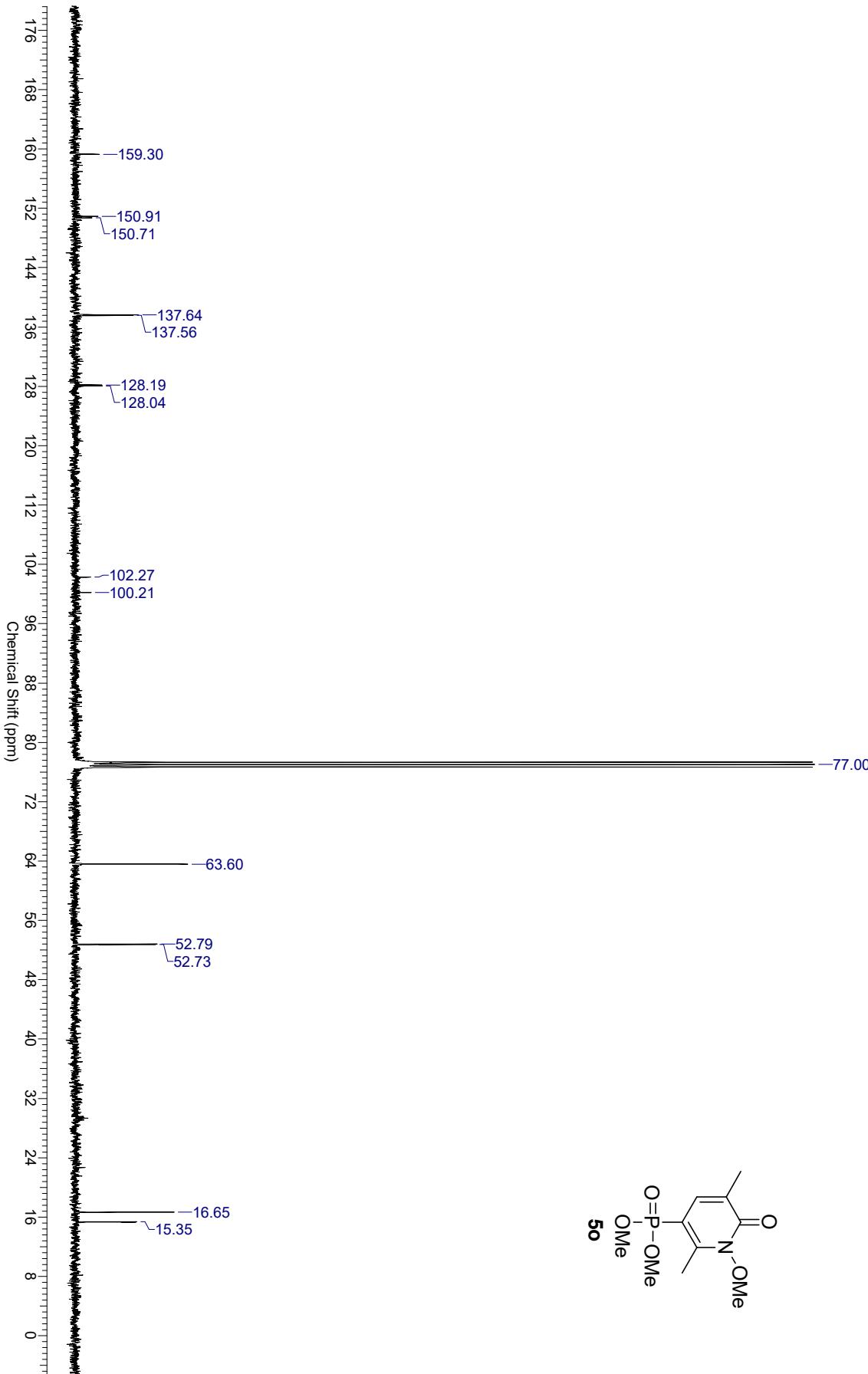


5o



¹³C NMR Spectra for Compound 5o in CDCl₃

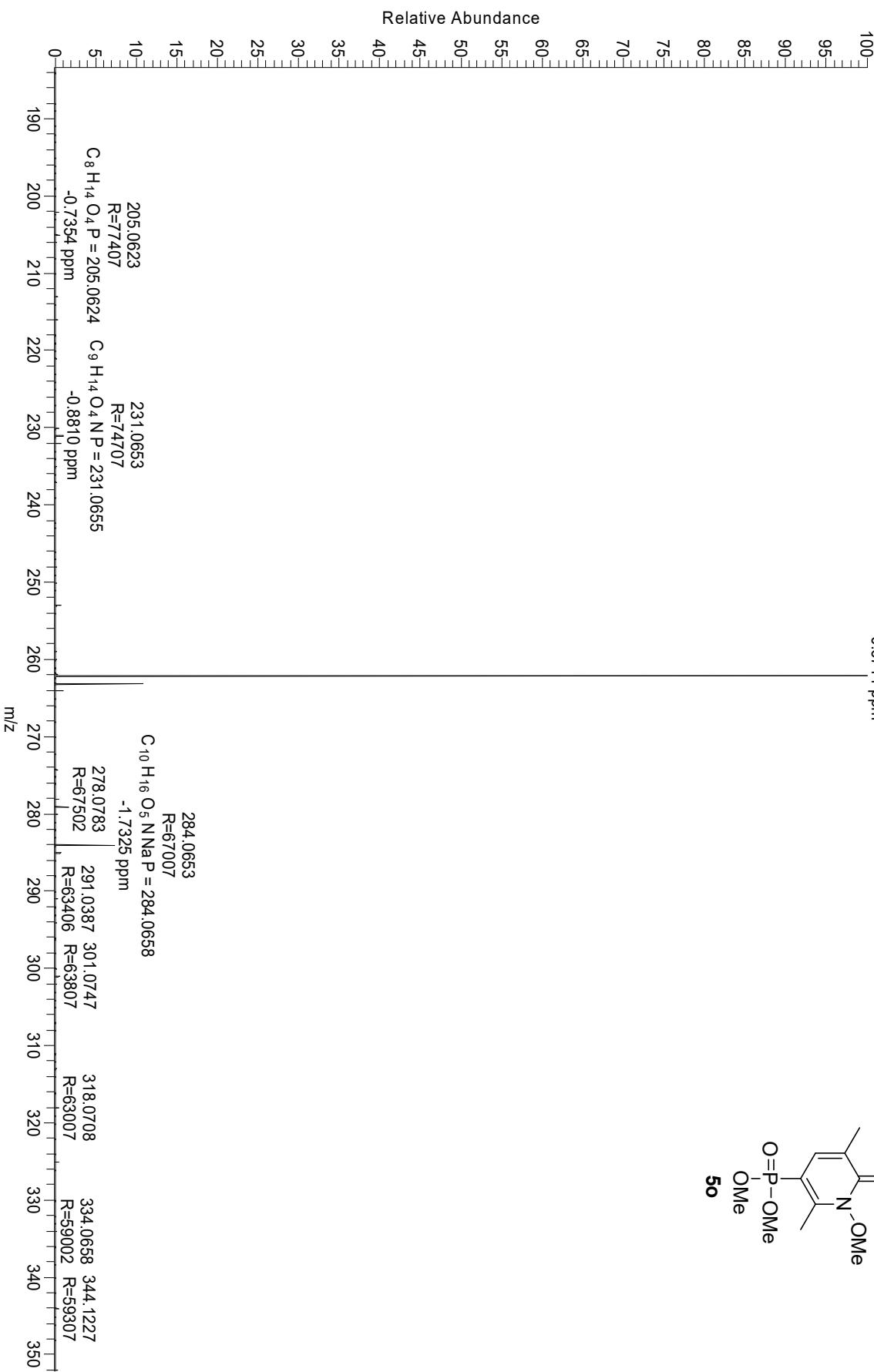
Acquisition Time (sec)	0.6488	Comment	13C	Date	12 Feb 2016 20:07:36
Date Stamp	12 Feb 2016 20:07:36	File Name	\172.16.2.4\mr data\AV400\Feb_16_400\FT2ava400#019\3\PDAT\1\1r		
Frequency (MHz)	100.61	Nucleus	13C	Number of Transients	152
Original Points Count	16384	Owner	root	Points Count	32768
Receiver Gain	2050.00	SW(Cyclical) (Hz)	25252.53	Pulse Sequence	zgpg30
Spectrum Offset (Hz)	10058.7354	Spectrum Type	STANDARD	Sweep Width (Hz)	25251.75
				Temperature (degree C)	23.900



SI 132

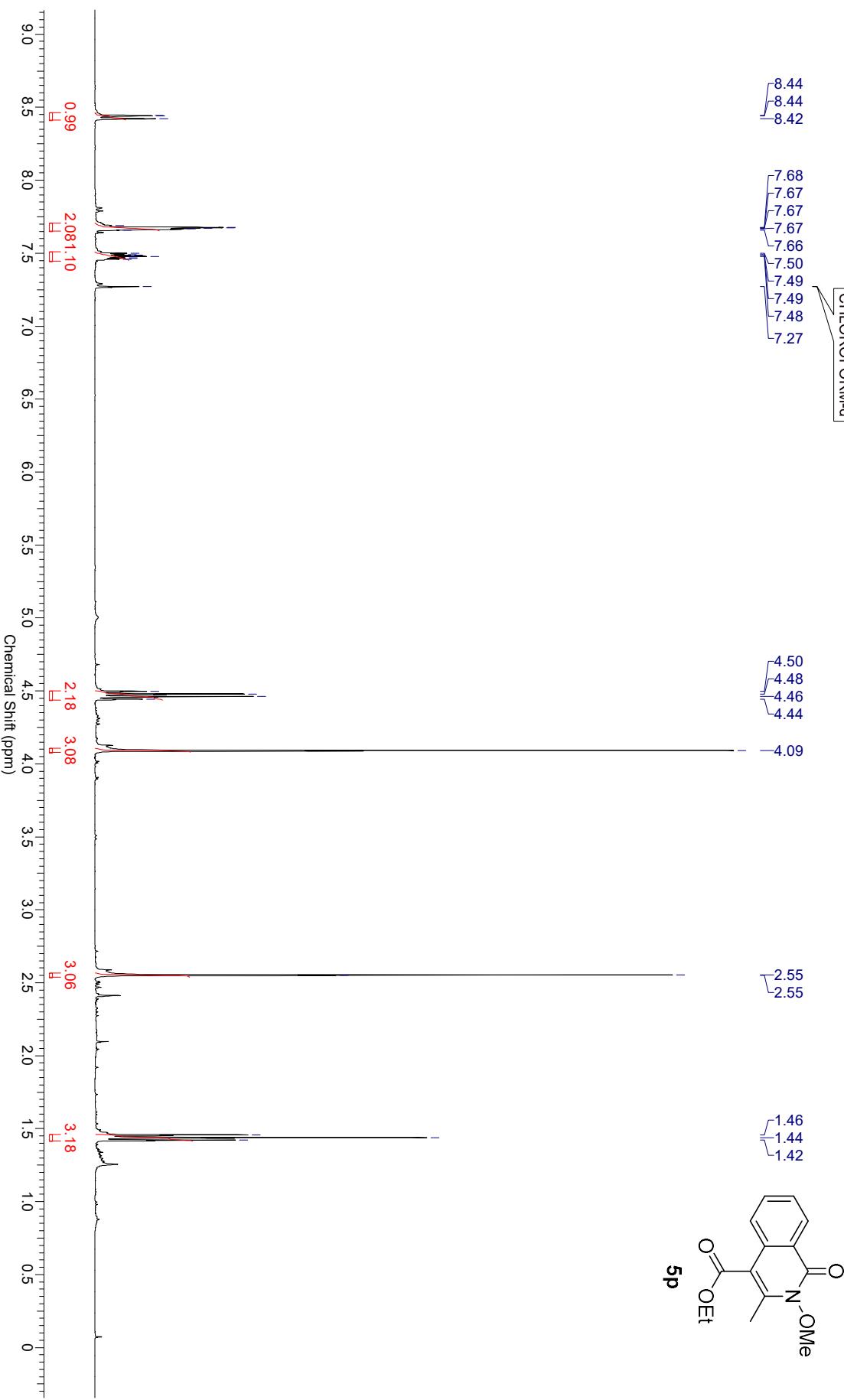
HRMS Spectra for Compound **5o** in MeOH

RR-AA-07 #111 RT: 0.49 AV: 1 NL: 7.21E9
 T: FTMS + p ESI[Full ms [100.00-1500.00]]



¹H NMR Spectra for Compound 5p in CDCl₃

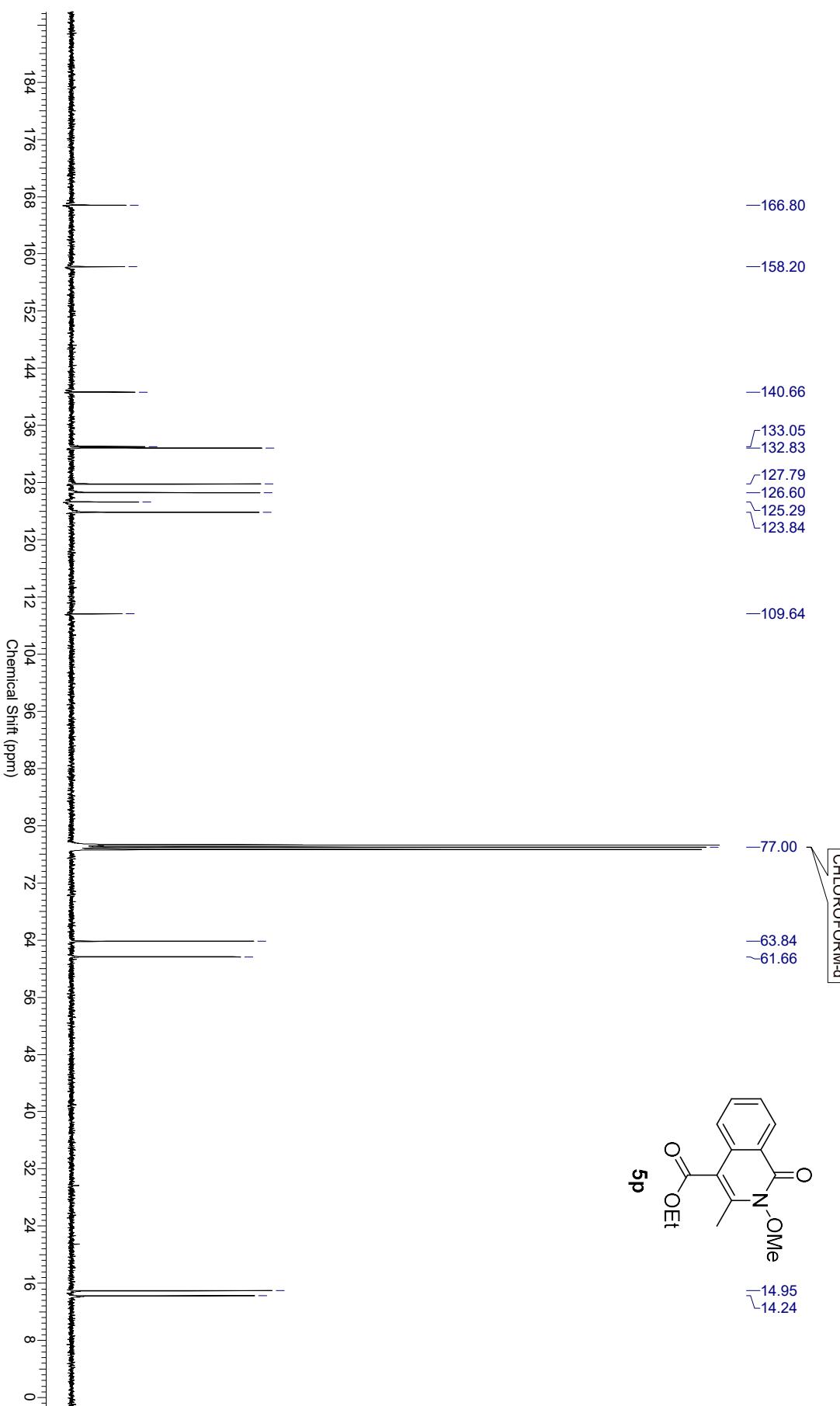
Acquisition Time (sec)	2.1838	Comment	Ravindra	Date	12 Jan 2016 06:25:42
Date Stamp	11 Jan 2016 14:30:23			File Name	\1172.16.2.4\mr data\leol_400_new\Liquid Jan 16\Mon3ECX400#001_PROTON-3.jdf
Frequency (MHz)	399.78	Nucleus	¹ H	Number of Transients	64
Original Points Count	13107	Owner	delta	Points Count	13107
Receiver Gain	38.00	Solvent	CHLOROFORM-d	Pulse Sequence	single_pulse_ex2
Spectrum Type	STANDARD	Sweep Width (Hz)	6001.85	Spectrum Offset (Hz)	2007.9249
		Temperature (degree C)	22.900		



SI 134

¹³C NMR Spectra for Compound 5p in CDCl₃

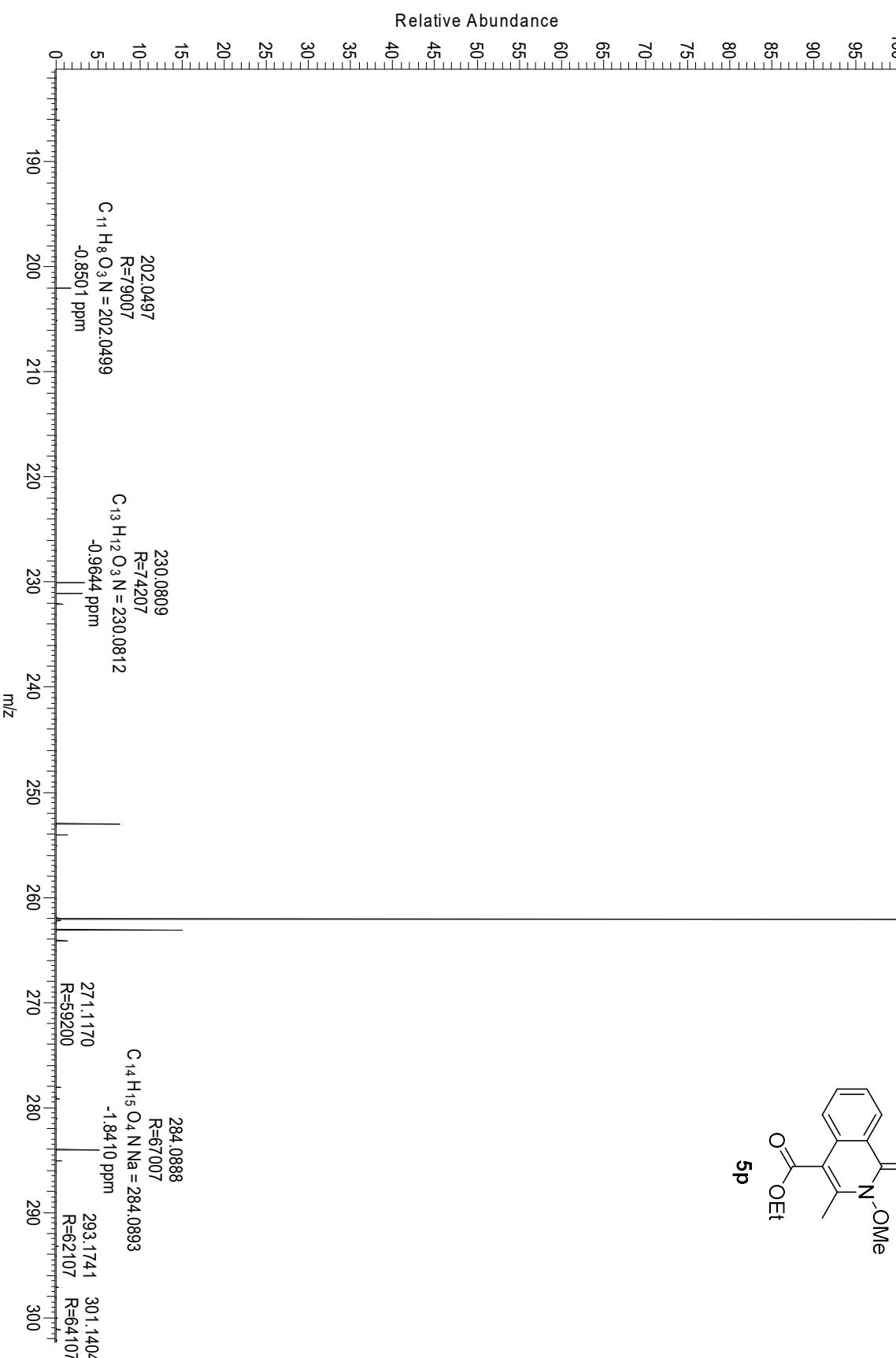
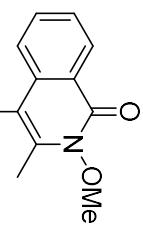
Acquisition Time (sec)	1.0434	Comment	Ravindra	Date	12 Jan 2016 07:43:45
Date Stamp	11 Jan 2016 15:17:04			File Name	\172.16.2.4\mnrr_data\eloi_400_newLiquidJan16\Mon3ECX400#001_CARBON-3.jdf
Frequency (MHz)	100.53	Nucleus	¹³ C	Number of Transients	1200
Original Points Count	26214	Owner	delta	Points Count	26214
Receiver Gain	60.00	Solvent	CHLOROFORM-d	Pulse Sequence	single pulse dec
Spectrum Type	STANDARD	Sweep Width (Hz)	25124.29	Temperature (degree C)	23.000
				Spectrum Offset (Hz)	10041.2480



HRMS Spectra for Compound 5p in MeOH

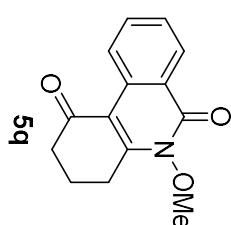
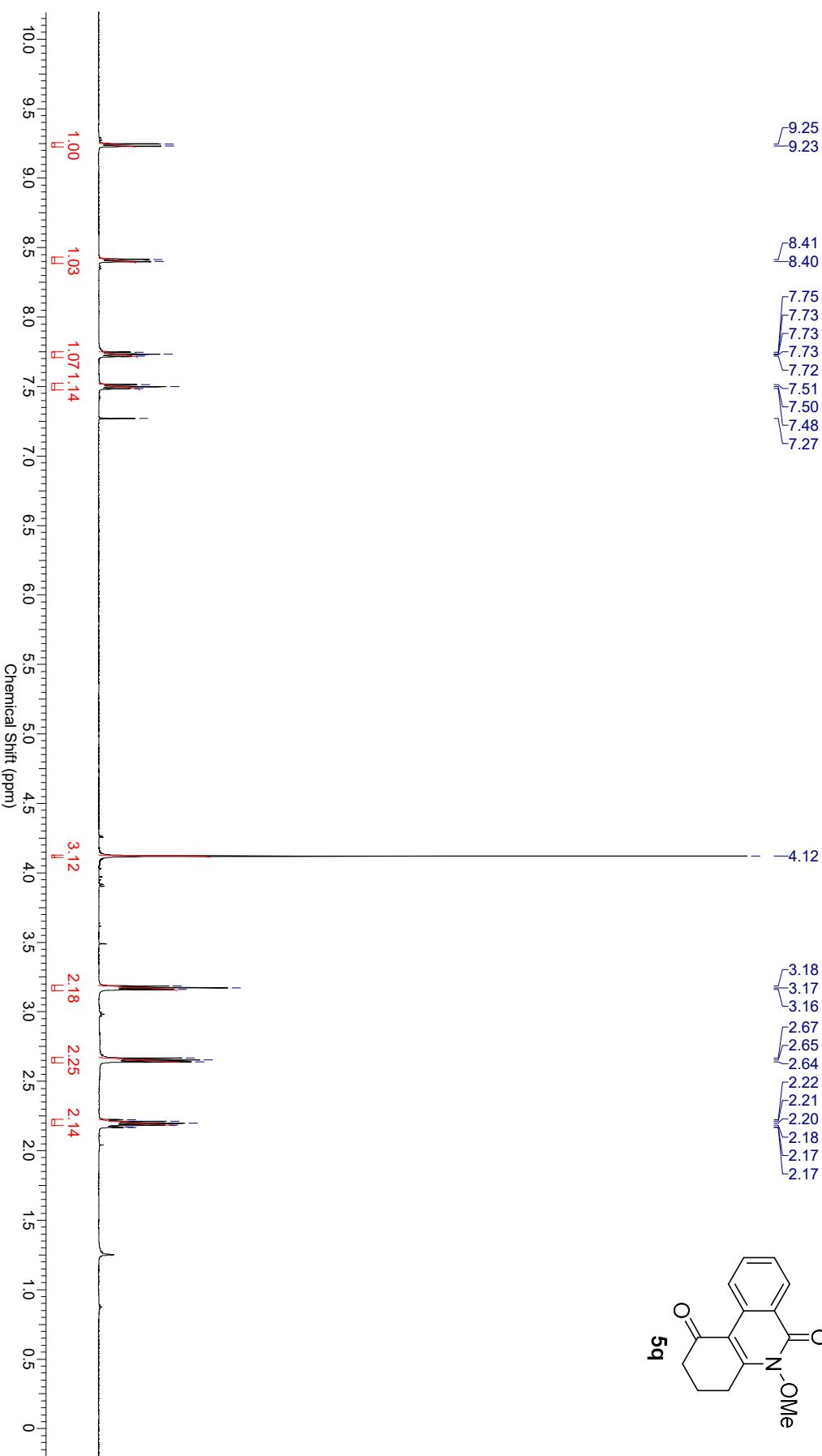
RR-AA-08 #148 RT: 0.66 AV: 1 NL: 3.87E9
T: FTMS + pESI Full ms [100.00-1500.00]

262.1071
R=70003
 $C_{14}H_{16}O_4N = 262.1074$
-0.9052 ppm



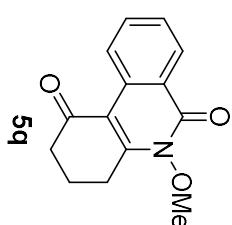
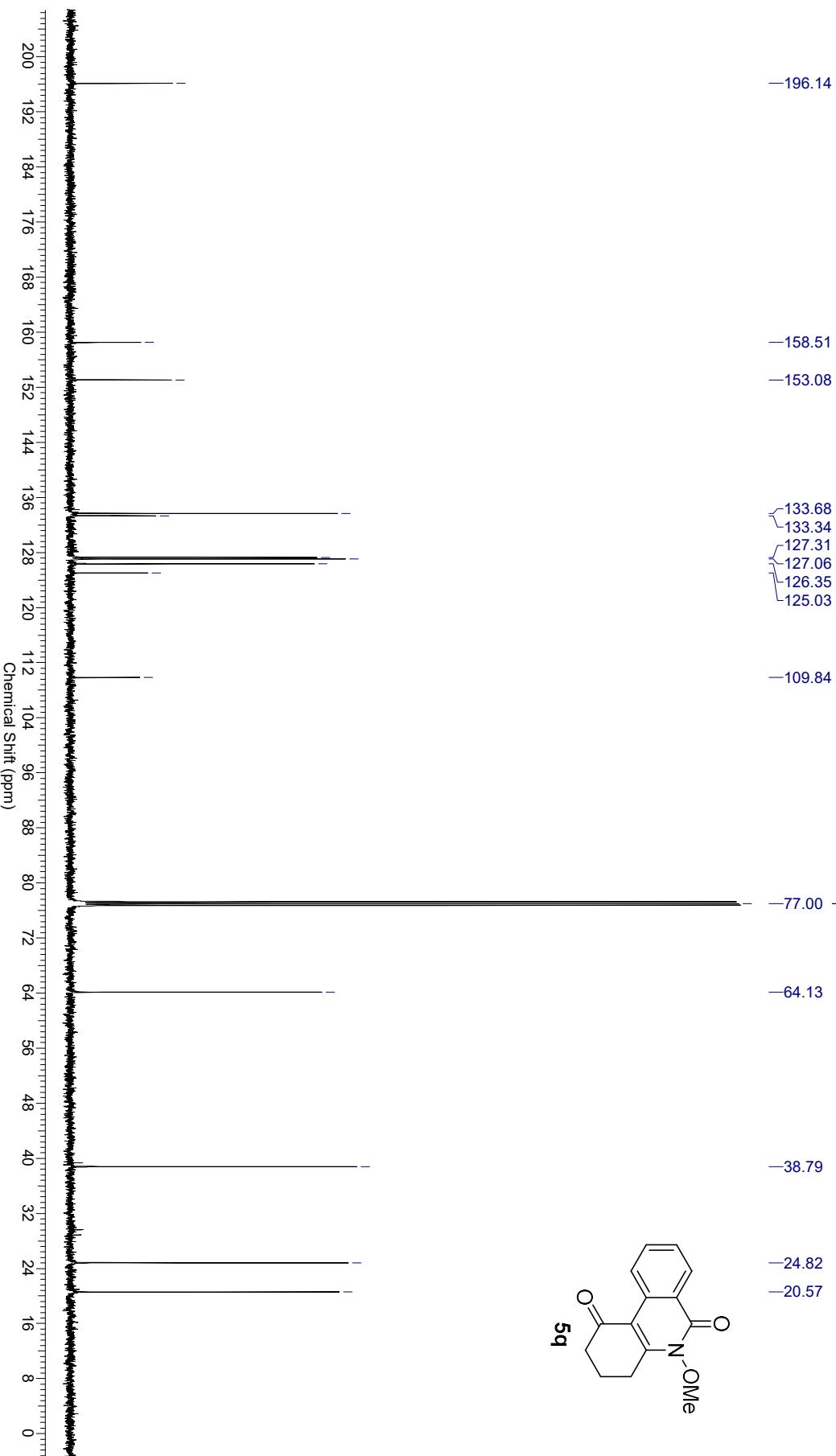
¹H NMR Spectra for Compound 5q in CDCl₃

Acquisition Time (sec)	1.2800	Comment	Ravinda 1H	Date	30 Jan 2016 14:37:04
Date Stamp	30.Jan.2016 14:37:04				
File Name	\172.16.2.4\mr\data\AV500\2016_AV500\JAN_16_AV500\SA5av500#014\1\PDAT\AV1\1r				
Nucleus	¹ H	Number of Transients	64	Origin	500.13
Owner	nmr	Points Count	32768	Pulse Sequence	16000
SW(cyclical) (Hz)	12500.00	Solvent	CHLOROFORM-d	Receiver Gain	228.00
Spectrum Type	STANDARD	Sweep Width (Hz)	12499.62	Spectrum Offset (Hz)	3492.7031



¹³C NMR Spectra for Compound 5q in CDCl₃

Acquisition Time (sec)	0.6554	Comment	13C	Date	30 Jan 2016 15:00:32
Date Stamp	30 Jan 2016 15:00:32				
File Name	\\\172.16.2.4\mmr\data\AV500\2016_AV500\JAN_16_AV500\Sa5a500#014\3PDATA\\1\1r				
Nucleus	¹³ C	Number of Transients	434	Origin	125.76
Owner	nmr	Points Count	32768	Pulse Sequence	Original Points Count
SW(cyclical) (Hz)	31250.00	Solvent	CHLOROFORM-d	Receiver Gain	20480
Spectrum Type	STANDARD	Sweep Width (Hz)	31249.05	Temperature (degree C)	1440.00
				Spectrum Offset (Hz)	26.700
					12556.2939

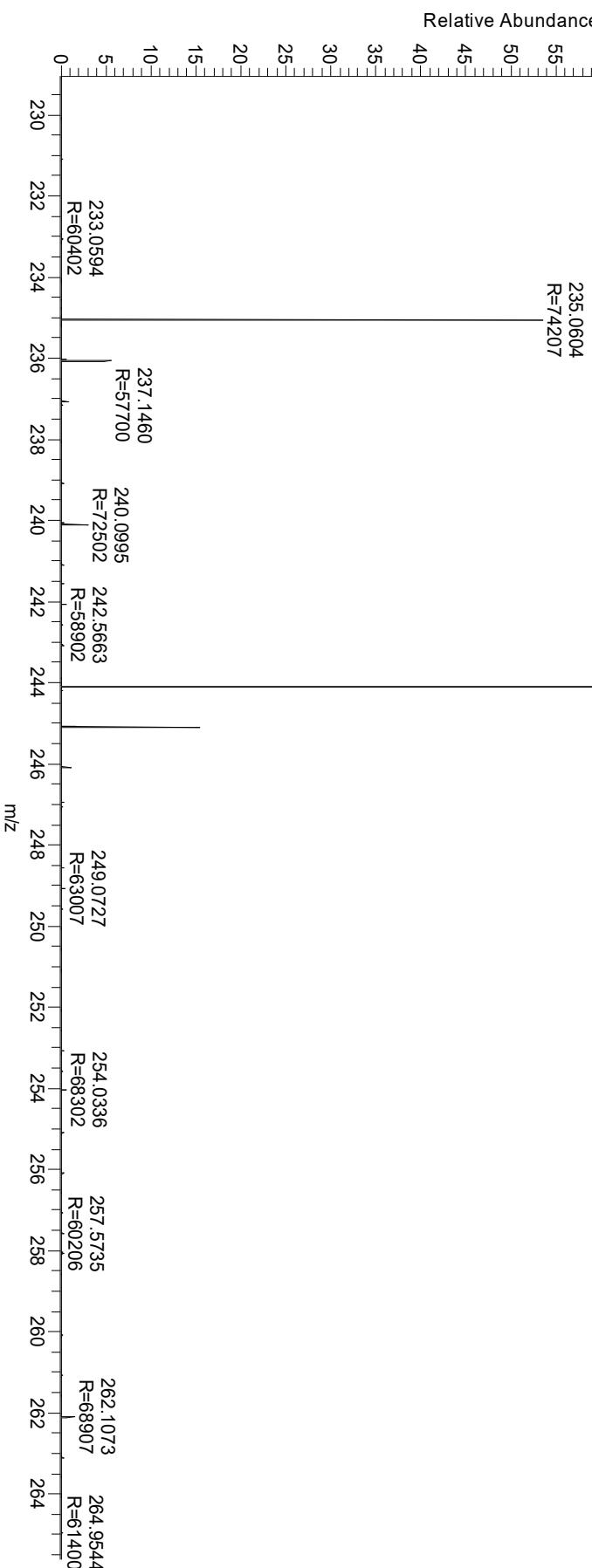
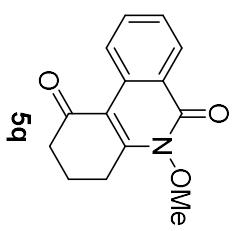


SI 138

HRMS Spectra for Compound 5q in MeOH

RR-A-4 #100 RT: 0.44 AV: 1 NL: 5.96E8
T: FTMS + p ESI Full ms[100.00-1500.00]

244.0968
R=72607
C₁₄H₁₄O₃N = 244.0968
-0.2615 ppm



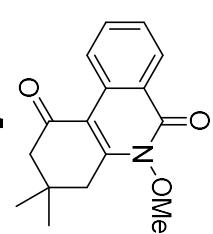
¹H NMR Spectra for Compound 5r in CDCl₃

Acquisition Time (sec)	Comment	Ravindra	Date	13 Nov 2015 22:41:12	
Date Stamp	13 Nov 2015 22:41:12				
File Name	\lagni\nmr\data\AV200\NOV_15#AV200\data\Administrator\hmri\F1\2av2# 30 1\PDAT\A1\1\fr		Frequency (MHz)	200.13	
Nucleus	1H	Number of Transients	32	Original Points Count	16384
Owner	Administrator	Points Count	32768	Pulse Sequence	av200
SW(cyclicall) (Hz)	4139.07	Solvent	CHLOROFORM-d	Receiver Gain	574.70
Spectrum Type	STANDARD	Sweep Width (Hz)	4138.95	Spectrum Offset (Hz)	1229.2844

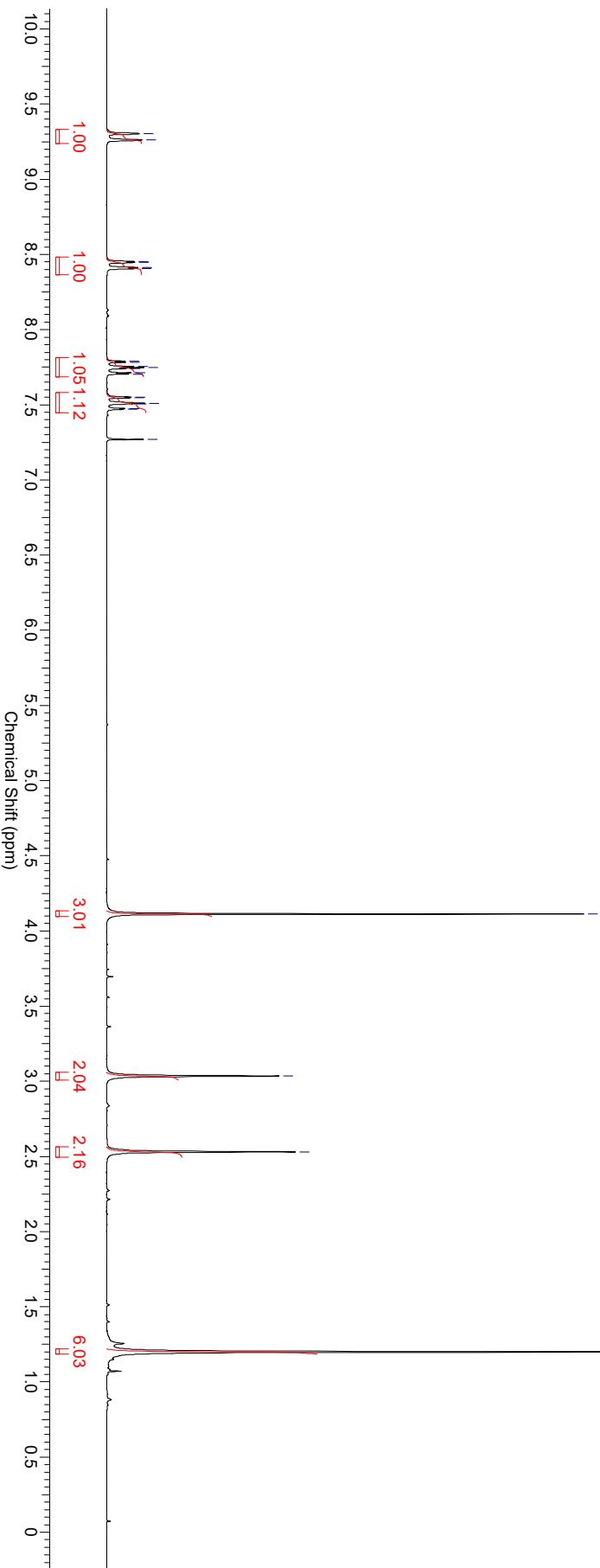
9.30
9.26
8.45
8.45
8.41
8.41
7.78
7.75
7.75
7.74
7.71
7.70
7.55
7.55
7.51
7.51
7.27

CHLOROFORM-d

—4.11
—3.03
—2.53



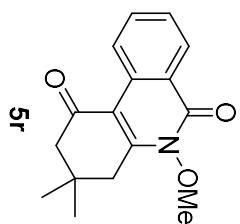
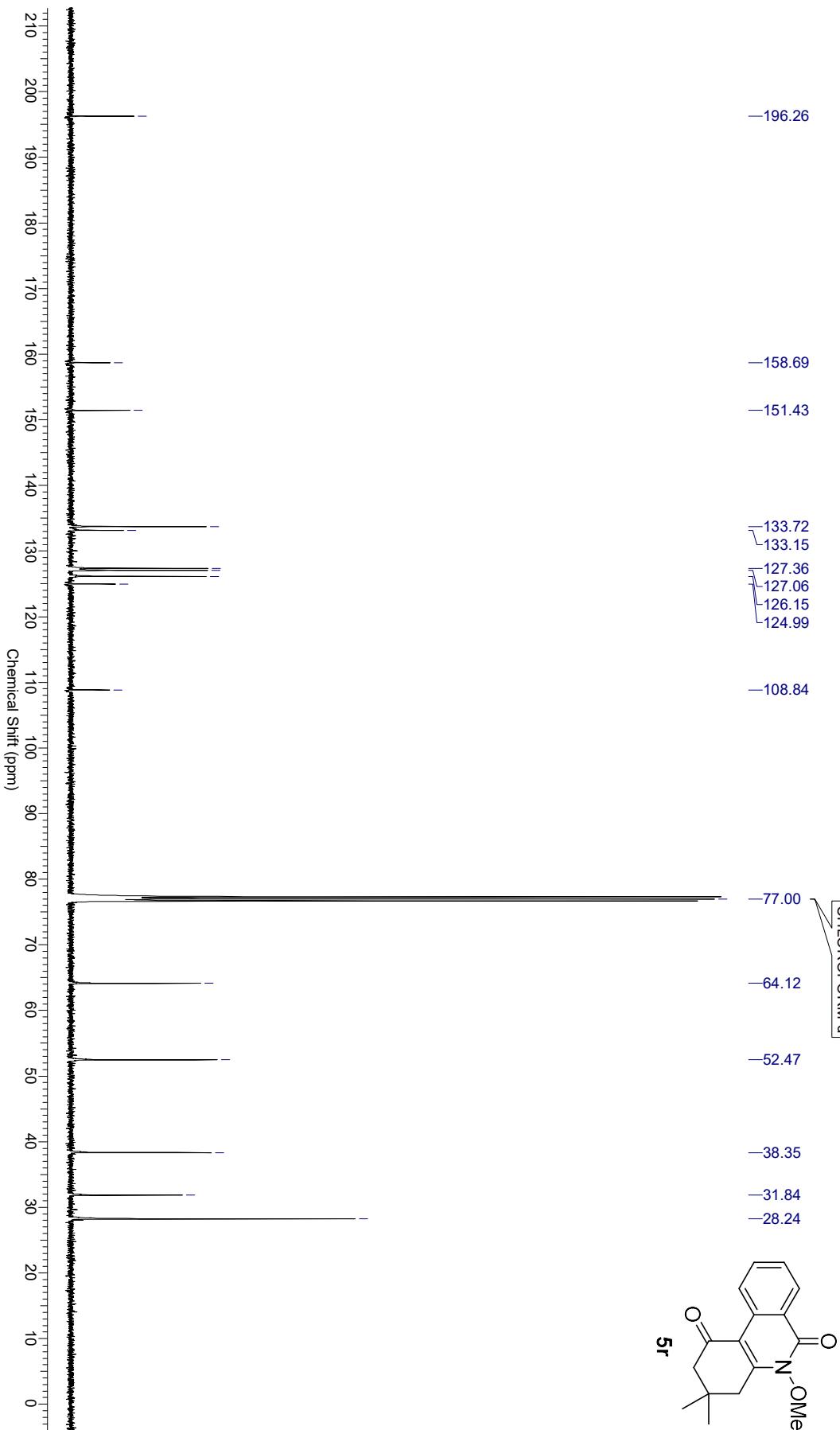
—1.20



SI 140

¹³C NMR Spectra for Compound 5r in CDCl₃

Acquisition Time (sec)	1.0434	Comment	Ravindra	Date	17 Nov 2015 04:26:58
Date Stamp	16 Nov 2015 12:16:55				
File Name	F:\jithamban\Research\3rd methodology_Amide\NMR\ketodiazoisopropyl-dimethylcyclohexanone\Mon3ECX400#002_CARBON-3.jdf				
Frequency (MHz)	100.53	Nucleus	¹³ C	Number of Transients	2000
Original Points Count	26214	Owner	delta	Points Count	26214
Receiver Gain	60.00	Solvent	CHLOROFORM-d	Pulse Sequence	single pulse dec
Sweep Width (Hz)	25124.29	Temperature (degree C)	23.400	Spectrum Offset (Hz)	10042.2061
				Spectrum Type	STANDARD



SI 141

HRMS Spectra for Compound **5r** in MeOH

RSP-31#112 RT: 0.50 AV: 1 NL: 3.06E9
T: FTMS + pESI[Full ms [100.00-1500.00]

272.1280
R=69707
 $C_{16}H_{18}O_3N = 272.1281$
-0.4748 ppm

