Electronic Supplementary Information

Gold(I)-Catalysed [1,3] O→C Rearrangement of Allenyl Ethers

Chandrababu Naidu Kona and Chepuri V. Ramana*

Division of Organic Chemistry, CSIR–National Chemical Laboratory

Dr. Homi Bhabha Road, Pune – 411 008 (India)

Email: vr.chepuri@ncl.res.in

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General information: Reactions were carried out in anhydrous solvents under an atmosphere of argon in oven-dried glassware. NMR spectra were recorded on JEOL AL-400 (400 MHz), Bruker AC 200 MHz, Bruker DRX 400 MHz and Bruker DRX 500 MHz spectrometers, and TMS was used as an internal standard of spectrometers. The chemical shifts were reported in parts per million (δ) relative to internal standard TMS (0 ppm) and for $CDCl_3$ (7.25 ppm). 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). Mass spectroscopy was carried out on PI QStar Pulsar (Hybrid Quadrupole-TOF LC/MS/MS) and High-resolution mass spectra (HRMS) were recorded on a Thermo Scientific Q-Exactive, Accela 1250 pump, and IR spectra were recorded on FT-IR PerkinElmer spectrometer by neat for oil sample and a CH_3Cl solution for solid samples. Column chromatography was performed over silica gel 100-200 mesh. All reagents were weighed and handled in air and backfilled under argon at room temperature. Unless otherwise noted, all reactions were performed under an argon atmosphere. All reagents were purchased from Aldrich and Alfa Easer and used without further purification. Compounds **1a**–**1z,** and **1a'-1h'** are prepared fllowing the procedures reported.[1–3]

General Procedure A: Synthesis of allenyl ethers:[1-3]

At room temperature, a solution of propargyl ether (2 mmol) in THF (10 mL) was treated with KO^{t}Bu (0.5 mmol) and the resulting suspension was stirred at room temperature for 1 h before quenching it with ice water. The contents were portioned between ethyl acetate (20 mL) and water (20 mL). The organic layer was separated and the aqueous layer was extracted with (2 x 10 ml ethyl acetate). The combined organic layer was concentrated under reduced pressure and the crude was subjected for the next step. For analytical purpose, the crude was purified by flash chromatography.

References:

1) B. M. Trost, J. Xie, *J. Am. Chem. Soc.* 2006, **128**, 6044.
2) B. M. Trost, J. Me, *J. Am. Chem. Soc.* 2008, **130**, 6231.
3) D.M. Cui, Z– L. Zheng, C. Zhang, *J. Org. Chem.* 2009, **74**, 1426.

Table E1. Synthesis of allenyl ethers

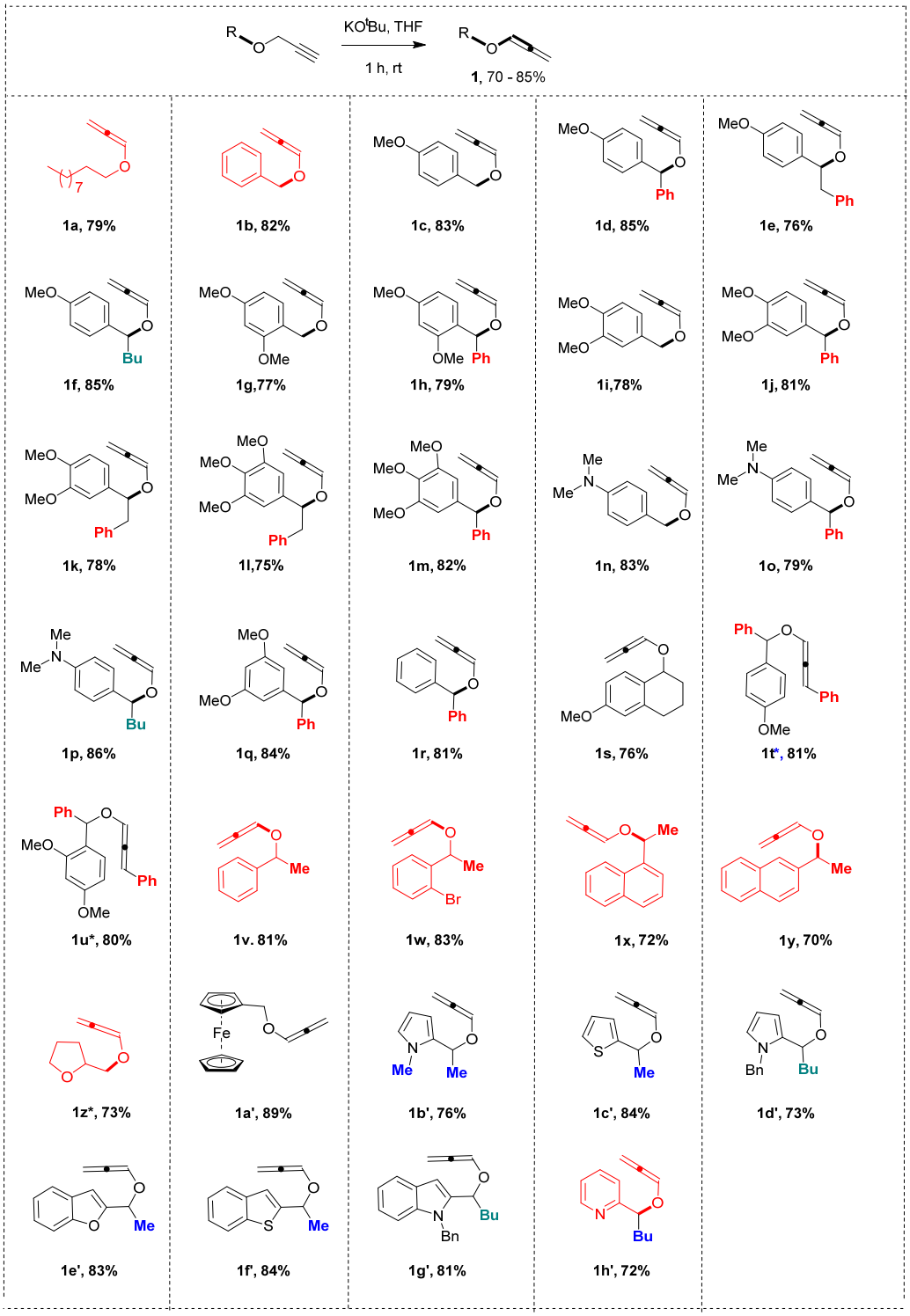
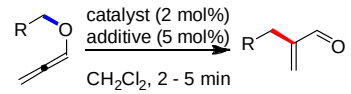


Table E2. Catalyst Optimization



1a (R = nC_9H_{17})
1b (R = Ph)
1c (R = p-$MeOC_6H_4$)

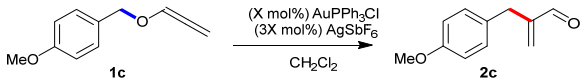
2

Entry	Substrate	Catalyst	additive	Yield
1	**1a**	$AuCl_3$	--	Hydrolysis
2	**1b**	$AuCl_3$	--	Hydrolysis
3	**1c**	$AuCl_3$	--	Hydrolysis
4	**1a**	$AuBr_3$	--	Hydrolysis
5	**1b**	$AuBr_3$	--	Hydrolysis
6	**1c**	$AuBr_3$	--	Hydrolysis
7	**1a**	$AuCl(PPh_3)$	--	No reaction
8	**1b**	$AuCl(PPh_3$	--	No reaction
9	**1c**	$AuCl(PPh_3$	--	No reaction
10	**1a**	$AuCl(PPh_3)$	$AgSbF_6$	Hydrolysis
11	**1b**	$AuCl(PPh_3)$	$AgSbF_6$	Hydrolysis
12	**1c**	$AuCl(PPh_3)$	$AgSbF_6$	70-96%
13	**1a**	$AgSbF_6$	--	Hydrolysis
14	**1b**	$AgSbF_6$	--	Hydrolysis
15	**1c**	$AgSbF_6$	--	27%
16	**1a**	AgOTf	--	No reaction
17	**1b**	AgOTf	--	No reaction
18	**1c**	AgOTf	--	No reaction
19	**1a**	$AgNTf_2$	--	Hydrolysis
20	**1b**	$AgNTf_2$	--	Hydrolysis
21	**1c**	$AgNTf_2$	--	Hydrolysis
22	**1c**	AgOAc	--	No reaction
23	**1a**	$AuCl_3$	$AgSbF_6$	Hydrolysis
24	**1b**	$AuCl_3$	$AgSbF_6$	Hydrolysis
25	**1c**	$AuCl_3$	$AgSbF_6$	Hydrolysis
26	**1a**	$AuCl(PMe_3)$	$AgSbF_6$	Hydrolysis
27	**1b**	$AuCl(PMe_3)$	$AgSbF_6$	Hydrolysis

28	**1c**	AuCl(PMe_3)	$AgSbF_6$	45-55%
29	**1a**	AuCl(PMe_3)	AgOTf	Hydrolysis
30	**1b**	AuCl(PMe_3)	AgOTf	Hydrolysis
31	**1c**	AuCl(PMe_3)	AgOTf	Hydrolysis
32	**1a**	AuCl(PPh_3)	$AgNTf_2$	Hydrolysis
33	**1b**	AuCl(PPh_3)	$AgNTf_2$	Hydrolysis
34	**1c**	AuCl(PPh_3)	$AgNTf_2$	Hydrolysis
35	**1a**	AuCl(PMe_3)	$AgNTf_2$	Hydrolysis
36	**1b**	AuCl(PMe_3)	$AgNTf_2$	Hydrolysis
37	**1c**	AuCl(PMe_3)	$AgNTf_2$	Hydrolysis
38	**1a**	AuCl(Biphenyl(^{t}Bu)$_2$)	$AgSbF_6$	Hydrolysis
39	**1b**	AuCl(Biphenyl(^{t}Bu)$_2$)	$AgSbF_6$	Hydrolysis
40	**1c**	AuCl(Biphenyl(^{t}Bu)$_2$)	$AgSbF_6$	40-45%

[Au] Complex 5 mol% and additive 15 mol%

Table E3. Optimization studies for the best Turn over frequency

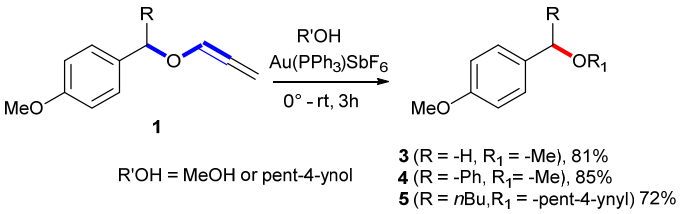


S.No	X (mol%)	temp	time	Yield[a]	S/C	TOF(h^{-1})
1	0.050	0 °C	05 min	96%	2800	4600
2	0.045	0 °C	30 min	92%	3100	4088
3	0.040	0 °C	60 min	89%	3500	2225
4	0.035	25 °C	08 h	87%	4000	310
5	0.030	25 °C	10 h	86%	4600	286
6	0.025	25 °C	16 h	86%	5600	215
7	0.0125	reflux	12 h	90%[b]	9072	600

S/C = Substrate/Catalyst Concentration

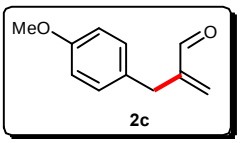
[a] isolated yields; [b]based on the 20% starting material recovered

Scheme E1. Trapping of the intermediate with alcohols

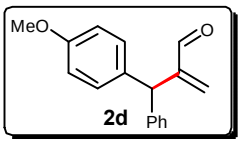


General Procedure B:

At 0 °C, to a solution of allenylether **1c** (1.0 g, 5.68 mmol) in anhydrous CH_2Cl_2 (100 ml) was added catalyst solution [(2.80 ml, 2.83 mmol) prepared by dissolving $Au(PPh_3)Cl$ (5 mg, 10.1 mmol) and $AgSbF_6$ (10 mg, 29.1 µmol) in CH_2Cl_2 (10 ml)] and allowed to stir for 5 minutes. The reaction mixture was concentrated under reduced pressure and the crude was purified by column chromatography (100-200 Silica gel) to afford **2c** (910 mg, 91%) as a colorless oil.

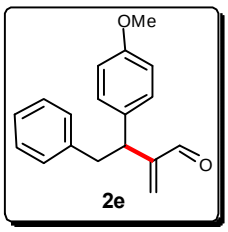


2-(4-Methoxybenzyl)acrylaldehyde (2c): Yellow oil; 91%; (R_f = 0.6, 5% ethyl acetate/pet. ether); IR ($CHCl_3$) *v*: 3361, 2998, 2954, 2836, 2700, 1690, 1611, 1509, 1464, 1300, 1248, 1178, 1035, 958, 852, 809, 769 cm^{-1}; ^1H NMR (200 MHz, $CDCl_3$): δ 3.52 (s, 2H), 3.80 (s, 3H), 6.04–6.13 (m, 2H), 6.85 (m, 2H), 7.11 (m, 2H), 9.61 (s, 1H); ^{13}C NMR (50 MHz, CDCl3): δ 33.3 (t), 55.2 (q), 113.9 (d, 2C), 130.1 (d, 2C), 135.0 (t), 150.1 (s, 2C), 158.2 (s), 194.1 (d) ppm; HRMS (ESI+): calcd. For $C_{11}H_{12}O_2Na$ $[M+Na]^+$ 199.0730; found 199.0728.

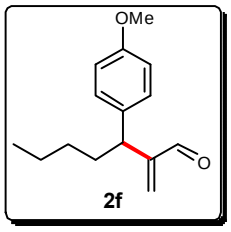


2-((4-Methoxyphenyl)(phenyl)methyl)acrylaldehyde (2d): colorless syrup; 97%; (R_f = 0.5, 5% ethyl acetate/pet. ether); IR ($CHCl_3$) *v*: 3367, 3027, 2836, 1692, 1609,1509, 1463, 1302, 1250, 1177, 1033, 966, 844, 751, 701, 545 cm^{-1}; ^1H NMR (200 MHz, $CDCl_3$): δ 3.72 (s, 3H), 5.33 (s, 1H), 5.96 (d, *J*,= 1.1 Hz, 1H), 6.23 (s, 1H) 6.78–6.86 (m, 2H), 7.02 (d, *J* = 8.8 Hz, 2H), 7.06–7.15 (m, 2H), 7.16–7.28 (m, 3H), 9.60 (s, 1H); ^{13}C NMR (50 MHz, $CDCl_3$): δ 48.4 (d), 55.2 (q), 113.9 (d, 2C), 126.6 (d), 128.4 (d, 2C), 128.8 (d, 2C), 129.9 (d, 2C), 133.1 (s), 136.6 (t), 141.5 (s), 153.0

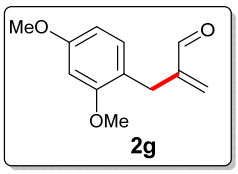
(s), 158.3 (s), 193.2 (d) ppm; HRMS (ESI+): calcd. For $C_{17}H_{16}O_2Na$ $[M+Na]^+$ 275.1043; found 275.1039.



3-(4-Methoxyphenyl)-2-methylene-4-phenylbutanal (2e): colorless syrup; 81%; (R_f = 0.6, 10% ethyl acetate/pet. ether); IR ($CHCl_3$) *v*: 3367, 3027, 2836, 1692, 1609,1509, 1463, 1302, 1250, 1177, 1033, 966, 844, 751, 701, 545 cm^{-1}; ^1H NMR (200 MHz, $CDCl_3$): δ 3.00–3.13 (m, 1H), 3.14–3.26 (m, 1H), 3.77 (s, 3H), 4.19 (t, J = 7.9 Hz, 1H), 6.10 (s, 1H), 6.38 (d, J = 0.9 Hz, 1H), 6.74–6.85 (m, 2H), 7.03–7.10 (m, 3H), 7.11–7.24 (m, 4H), 9.49 (s, 1H); ^{13}C NMR (50 MHz, $CDCl_3$): δ40.3 (t), 44.0 (d), 55.2 (q), 113.7 (d, 2C), 126.0 (d), 128.1 (d, 2C), 128.9 (d, 2C), 129.1 (d, 2C), 133.4 (s), 134.1 (t), 139.6 (s), 152.6 (s), 158.2 (s), 193.9 (d) ppm; HRMS (ESI+): calcd. For $C_{18}H_{18}O_2Na$ $[M+Na]^+$ 289.1199; found 289.1195.

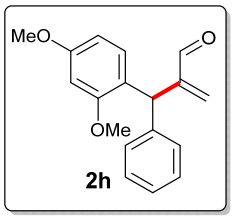


3-(4-Methoxyphenyl)-2-methyleneheptanal (2f): Yellow gum; 92%; (Rf = 0.5, 10% ethyl acetate/pet. ether); IR ($CHCl_3$) *v*: 3366, 2956, 2931, 1693, 1610, 1509, 1464, 1301, 1248, 1178, 1036, 942, 827 cm^{-1}; ^1H NMR (200 MHz, $CDCl_3$): δ 0.86 (t, J = 6.9 Hz, 3H), 1.12–1.41 (m, 4H), 1.70–1.88 (m, 2H), 3.78 (s, 3H), 3.80–3.86 (m, 1H), 6.05 (s, 1H), 6.30 (s, 1H), 6.79–6.88 (m, 2H), 7.09–7.19 (m, 2 H), 9.51 (s, 1 H); ^{13}C NMR (50 MHz, $CDCl_3$): δ 13.9 (q), 22.5 (t), 29.8 (t), 33.8 (t), 42.0 (d), 55.1 (q), 113.7 (d, 2C), 128.9 (d, 2C), 133.3 (t), 134.4 (s), 153.7 (s), 158.1 (s), 194.0 (d) ppm; HRMS (ESI+): calcd. For $C_{15}H_{20}O_2Na$ $[M+Na]^+$ 255.1356; found 255.1352.

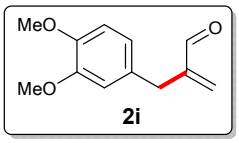


2-(2,4-Dimethoxybenzyl)acrylaldehyde (2g): Yellow gum; 91%; (R_f = 0.6, 5% ethyl acetate/pet. ether); IR ($CHCl_3$) *v*: 3359, 2998, 2935, 2835, 1909, 1690, 1590, 1512, 1464, 1418, 1333, 1262, 1155, 1029, 954, 867, 805, 771, 755 cm^{-1}; ^1H NMR (200 MHz, $CDCl_3$): δ 3.49 (s, 2H), 3.78 (s, 3H), 3.81 (s, 3H), 5.96–6.04 (m, 2H), 6.41–6.48 (m, 2H), 7.02 (s, 1H), 9.61 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 27.8 (t), 55.3 (q, 2C), 98.6 (d), 103.9 (d), 118.8 (s), 131.1 (d), 134.7 (t), 149.2 (s), 158.3 (s), 159.7 (s), 194.4 (d) ppm; HRMS (ESI+): calcd. For $C_{12}H_{14}O_3Na$ $[M+Na]^+$ 229.0835; found 229.0833.

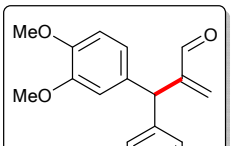
2-((2,4-Dimethoxyphenyl)(phenyl)methyl)acrylaldehyde (2h): Yellow gum; 94%; (R_f = 0.6, 5% ethyl acetate/pet. ether); IR ($CHCl_3$) *v*: 3367, 3023, 2957, 2836, 1693, 1591, 1514, 1417, 1265, 1141, 1028, 954, 754, 701 cm^{-1}; ^1H NMR (200 MHz, $CDCl_3$): δ 3.80 (s, 3H), 3.84 (s, 3H), 5.69 (s, 1H), 5.98 (s, 1H), 6.28 (s, 1H), 6.41–6.49 (m, 1H), 6.54 (d, *J* = 2.4 Hz, 1H), 6.81 (d, *J* = 8.3 Hz, 1H), 7.15–7.23 (m, 2H), 7.33 (td, *J* = 5.3, 1.8 Hz, 3H), 9.70 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 42.1 (d), 55.2 (q), 55.4 (q), 98.7 (d), 103.6 (d), 122.4 (s), 126.3 (d), 128.2 (d, 2C), 128.9 (d, 2C), 129.6 (d), 135.7 (t), 141.0 (s), 152.6 (s), 157.6 (s), 159.6 (s), 193.2 (d) ppm; HRMS (ESI+): calcd. For $C_{18}H_{18}O_3Na$ $[M+Na]^+$ 305.1148; found 305.1145.



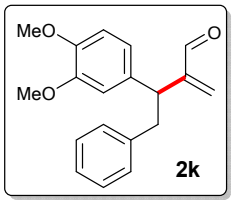
2-(3, 4-Dimethoxybenzyl)acrylaldehyde (2i): Yellow oil; 94%; (R_f = 0.4, 5% ethyl acetate/pet. ether); IR ($CHCl_3$) *v*: 3359, 2998, 2935, 2835, 1909, 1690, 1590, 1512, 1464, 1418, 1333, 1262, 1155, 1029, 954, 867, 805, 771, 755 cm^{-1}; ^1H NMR (200 MHz, $CDCl_3$): δ 3.50 (s, 2H), 3.85 (s, 6H), 6.05 (d, *J* = 0.8 Hz, 1H), 6.11 (s, 1H), 6.68–6.75 (m, 2H), 6.77–6.83 (m, 1H), 9.60 (s, 1H); ^{13}C NMR (50 MHz, $CDCl_3$): δ 33.7 (t), 55.7 (q), 55.8 (q), 111.2 (d), 112.3 (d), 121.0 (d), 130.6 (s), 134.9 (t), 147.6 (s), 148.9 (s), 149.9 (s), 194.0 (d) ppm; HRMS (ESI+): calcd. For $C_{12}H_{14}O_3Na$ $[M+Na]^+$ 229.0835; found 229.0833.



2-((3,4-Dimethoxyphenyl)(phenyl)methyl)acrylaldehyde (2j): Yellow gum; 91%; (R_f = 0.6, 5% ethyl acetate/pet. ether); IR ($CHCl_3$) *v*: 3367, 3023, 2957, 2836, 1693, 1591, 1514, 1417, 1246, 1141, 1028, 954, 754, 701 cm^{-1}; 1H NMR (200 MHz, $CDCl_3$): δ 3.85 (s, 3H), 3.90 (s, 3H), 5.38 (s, 1H), 6.06 (d, *J* = 1.0 Hz, 1H), 6.35 (s, 1H), 6.64–6.73 (m, 2H), 6.80–6.88 (m, 1H), 7.14–7.20 (m, 2H), 7.28–7.41 (m, 3H), 9.70 (s, 1H); ^{13}C NMR (50 MHz, $CDCl_3$): δ 48.6 (d), 55.7 (q, 2C), 110.9 (d), 112.4 (d), 120.7 (d), 126.5 (d), 128.3 (d, 2C), 128.7 (d, 2C), 133.5 (s), 136.5 (t), 141.2 (s), 147.7 (s), 148.9 (s), 152.8 (s), 193.0 (d) ppm; HRMS (ESI+): calcd. For $C_{18}H_{18}O_3Na$ $[M+Na]^+$ 305.1148; found 305.1144.

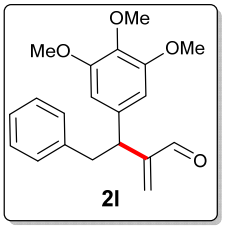


3-(3,4-Dimethoxyphenyl)-2-methylene-4-phenylbutanal (2k): Colorless gum; 82%; (R_f = 0.6, 10% ethyl acetate/pet. ether); IR ($CHCl_3$) *v*: 3362, 3025, 2934, 2835, 1692, 1591, 1515, 1454, 1419, 1260, 1141, 1028, 950,

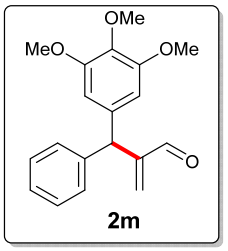


809, 757, 700 cm^{-1}; ^1H NMR (200 MHz, $CDCl_3$): δ 2.99–3.13 (m, 1H), 3.13–3.26 (m, 1H), 3.79 (s, 3H), 3.84 (s, 3H), 4.17 (t, J = 7.9 Hz, 1H), 6.13 (s, 1H), 6.37–6.41 (m, 1H), 6.63 (s, 1H), 6.71–6.77 (m, 2H), 7.02–7.09 (m, 2H), 7.13–7.23 (m, 3H), 9.51 (s, 1H); ^{13}C NMR (50 MHz, $CDCl_3$): δ 40.3 (t), 44.4 (d), 55.8 (q, 2C), 111.0 (d), 111.8 (d), 119.7 (d), 126.0 (d), 128.1 (d, 2C), 128.9 (d, 2C), 133.9 (s), 134.1 (t), 139.5 (s), 147.6 (s), 148.6 (s), 152.5 (s), 193.8 (d) ppm; HRMS (ESI+): calcd. For $C_{19}H_{20}O_3Na$ $[M+Na]^+$ 319.1305; found 319.1304.

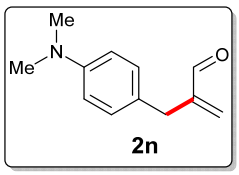
2-Methylene-4-phenyl-3-(3,4,5-trimethoxyphenyl)butanal (2l): White gum; 83%; (R_f = 0.6, 10% ethyl acetate/pet. ether); IR ($CHCl_3$) v: 3362, 2997, 2936, 2837, 1693, 1589, 1506, 1455, 1421, 1327, 1239, 1126, 1010, 700 cm^{-1}; ^1H NMR (200 MHz, $CDCl_3$): δ 2.98–3.13 (m, 1H), 3.13–3.26 (m, 1H), 3.78 (s, 6H), 3.81 (s, 3H), 4.16 (t, J = 7.8 Hz, 1H), 6.15 (s, 1H), 6.35 (s, 2H), 6.41 (s, 1H), 7.03–7.10 (m, 2H), 7.14–7.23 (m, 3H), 9.52 (s, 1H); ^{13}C NMR (101 MHz, $CDCl_3$): δ 40.3 (t), 45.0 (d), 56.1 (q, 2C), 60.8 (q), 105.1 (d, 2C), 126.2 (d), 128.2 (d, 2C), 128.9 (d, 2C), 134.5 (t), 136.6 (s), 137.0 (s), 139.4 (s), 152.1 (s), 153.0 (s, 2C), 193.9 (d) ppm; HRMS (ESI+): calcd. For $C_{20}H_{22}O_4Na$ $[M+Na]^+$ 349.1410; found 349.1404.



2-(Phenyl(3,4,5-trimethoxyphenyl)methyl)acrylaldehyde (2m): Yellow gum; 87%; (R_f = 0.5, 10% ethyl acetate/pet. ether); IR ($CHCl_3$) v: 3366, 2998, 2938, 1959, 1693, 1589, 1505, 1419, 1237, 1126, 1008, 960, 823, 702 cm^{-1}; ^1H NMR (200 MHz, $CDCl_3$): δ 3.81 (s, 6H), 3.87 (s, 3H), 5.36 (s, 1H), 6.07 (d, J = 1.1 Hz, 1H), 6.35–6.38 (m, 3H), 7.13–7.20 (m, 2H), 7.28–7.40 (m, 3H), 9.70 (s, 1H); ^{13}C NMR (50 MHz, $CDCl_3$): δ 49.2 (d), 56.0 (q, 2C), 60.8 (q), 106.1 (d, 2C), 126.7 (d), 128.4 (d, 2C), 128.8 (d, 2C), 136.6 (s), 136.7 (s), 136.8 (t), 140.9 (s), 152.6 (s), 153.1 (s, 2C), 193.1 (d) ppm; HRMS (ESI+): calcd. For $C_{19}H_{20}O_4Na$ $[M+Na]^+$ 335.1254; found 335.1248.

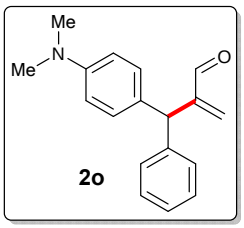


2-(4-(Dimethylamino)benzyl)acrylaldehyde (2n): Yelow oil; 92%; (R_f = 0.6, 10% ethyl acetate/pet. ether); IR ($CHCl_3$) v: 3358, 2916, 2802, 1885, 1686, 1615, 1523, 1479, 1347, 1163, 1061, 947, 857, 800 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$): δ 2.94 (s, 6H), 3.49 (s, 2H), 6.04 (d, J = 0.9 Hz, 1H),

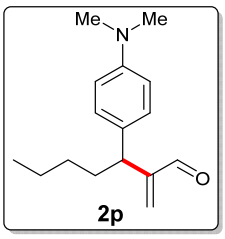


6.12 (s, 1H), 6.72 (m, J = 8.7 Hz, 2H), 7.08 (m, J = 8.7 Hz, 2H), 9.62 (s, 1H); ^{13}C NMR (50 MHz, $CDCl_3$): δ 33.0 (t), 40.7 (q, 2C), 112.9 (d, 2C), 125.8 (s), 129.7 (d, 2C), 134.7 (t), 149.3 (s), 150.4 (s), 194.2 (d) ppm; HRMS (ESI+): calcd. For $C_{12}H_{16}ON$ $[M+H]^+$ 190.1226; found 190.1225.

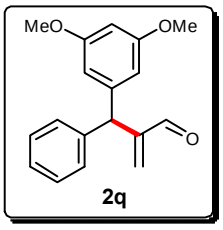
2-((4-(Dimethylamino)phenyl)(phenyl)methyl)acrylaldehyde (2o): Yellow gum; 95%; (R_f = 0.5, 10% ethyl acetate/pet. ether); IR ($CHCl_3$) v: 3365, 3026, 2885, 2803, 2696, 1884, 1692, 1612, 1520, 1449, 1350, 1221, 1162, 1061, 948, 806, 761, 701 cm^{-1}; ^{1}H NMR (200 MHz, $CDCl_3$): δ 3.00 (s, 6H), 5.37 (s, 1H), 6.08 (s, 1H), 6.34 (s, 1H), 6.71–6.80 (m, 2H), 7.01–7.10 (m, 2H), 7.16–7.24 (m, 2H), 7.29–7.42 (m, 3H), 9.72 (s, 1 H); ^{13}C NMR (50 MHz, $CDCl_3$): δ 40.6 (q, 2C), 48.2 (d), 112.7 (d, 2C), 126.4 (d), 128.3 (d, 2C), 128.8 (d, 2C), 129.6 (d, 2C), 136.4 (t), 141.9 (s), 149.2 (s), 153.2 (s, 2C), 193.4 (d) ppm; HRMS (ESI+): calcd. For $C_{18}H_{20}ON$ $[M+H]^+$ 266.1539; found 266.1539.



3-(4-(Dimethylamino)phenyl)-2-methyleneheptanal (2p): Yellow Syrup; 93%; (R_f = 0.6, 10% ethyl acetate/pet. ether); IR ($CHCl_3$) v: 3363, 2955, 2929, 1693, 1614, 1520, 1445, 1348, 1223, 1163, 1061, 812 cm^{-1}; ^{1}H NMR (200 MHz, $CDCl_3$): δ 0.84 (t, J = 6.9 Hz, 3H), 1.14–1.37 (m, 4H), 1.70–1.84 (m, 2H), 2.91 (s, 6H), 3.75 (t, J = 7.6 Hz, 1H), 6.00 (s, 1H), 6.26 (s, 1H), 6.61–6.74 (m, 2H), 7.02–7.11 (m, 2H), 9.50 (s, 1H); ^{13}C NMR (50 MHz, $CDCl_3$): δ 13.9 (q), 22.6 (t), 29.9 (t), 33.8 (t), 40.7 (q, 2C), 41.8 (d), 112.6 (d, 2C), 128.6 (d, 2C), 130.3 (s), 133.1 (t), 149.2 (s), 154.1 (s), 194.3 (d) ppm; HRMS (ESI+): calcd. For $C_{16}H_{24}ON$ $[M+H]^+$ 246.1852; found 246.1851.

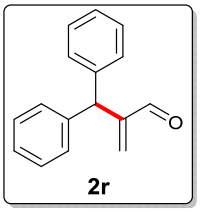


2-((3,5-Dimethoxyphenyl)(phenyl)methyl)acrylaldehyde (2q): Yellow liquid; 74%; (R_f = 0.6, 10% ethyl acetate/pet. ether); IR ($CHCl_3$) v: 3367, 3023, 2957, 2836, 1693, 1591, 1514, 1417, 1265, 1141, 1028, 954, 754, 701 cm^{-1}; ^{1}H NMR (200 MHz, $CDCl_3$): δ 3.79 (s, 6H), 5.36 (s, 1H), 6.09 (d, J = 1.0 Hz, 1H), 6.31–6.37 (m, 3H), 6.38 (d, J = 2.1 Hz, 1H), 7.14–7.21 (m, 2H), 7.29–7.36 (m, 3H), 9.69 (s, 1H); ^{13}C NMR (50 MHz, $CDCl_3$): δ 49.3 (d), 55.2 (q, 2C), 98.4 (d), 107.4 (d, 2C), 126.7 (d), 128.5 (d, 2C), 128.9 (d, 2C), 136.8 (t), 140.8 (s), 143.5 (s), 152.4

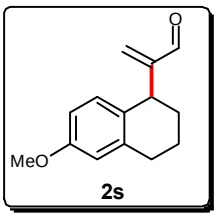


(s), 160.8 (s, 2C), 193.1 (d) ppm; HRMS (ESI+): calcd. For $C_{18}H_{18}O_3Na$ $[M+Na]^+$ 305.1148; found 305.1146.

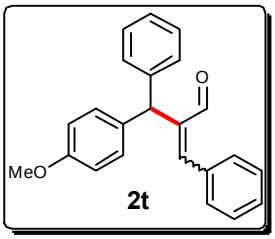
2-Benzhydrylacrylaldehyde (2r): Yellow gum; 87%; (R_f = 0.6, 10% ethyl acetate/pet. ether); IR ($CHCl_3$) *v*: 3362, 2995, 2932, 1894, 1690, 1617, 1509, 1456, 1253, 1229, 1162, 1047, 940, 830 cm^{-1}; ^1H NMR (200 MHz, CDCl3): δ 5.43 (s, 1H), 6.05 (d, J = 1.1 Hz, 1H), 6.36 (s, 1H), 7.12–7.21 (m, 4H), 7.28–7.41 (m, 6H), 9.70 (s, 1H); ^{13}C NMR (50 MHz, CDCl3): δ 49.2 (d), 126.7 (d, 2C), 128.5 (d, 4C), 128.9 (d, 4C), 136.8 (t), 141.1 (s, 2C), 152.7 (s), 193.1 (d) ppm; HRMS (ESI+): calcd. For $C_{16}H_{14}ONa$ $[M+Na]^+$ 245.0937; found 254.0933.



2-(6-Methoxy-1,2,3,4-tetrahydronaphthalen-1-yl)acrylaldehyde (2s): Yellow Syrup; 89%; (R_f = 0.6, 10% ethyl acetate/pet. ether); IR ($CHCl_3$) *v*: 3362, 2995, 2932, 1894, 1690, 1608, 1500, 1456, 1253, 1226, 1162, 1038, 940, 830 cm^{-1}; ^1H NMR (200 MHz, $CDCl_3$): δ 1.66–1.81 (m, 3H), 1.87–2.01 (m, 1H), 2.72–2.82 (m, 2H), 3.79 (s, 3H), 4.11 (br. s., 1H), 5.81 (s, 1H), 6.12 (s, 1H), 6.64–6.72 (m, 2H), 6.77–6.84 (m, 1H), 9.61 (s, 1H); ^{13}C NMR (50 MHz, $CDCl_3$): δ 19.2 (t), 28.3 (t), 29.7 (t), 36.4 (d), 55.2 (q), 112.4 (d), 113.3 (d), 129.2 (s), 130.6 (d), 137.0 (t), 139.1 (s), 155.4 (s), 157.8 (s), 194.0 (d) ppm; HRMS (ESI+): calcd. For $C_{14}H_{16}O_2Na$ $[M+Na]^+$ 239.1043; found 239.1040.

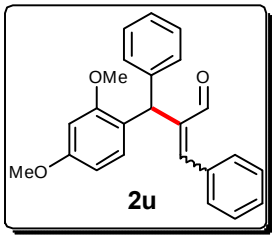


2-((4-Methoxyphenyl)(phenyl)methyl)-3-phenylacrylaldehyde (2t): Colorless syrup; 91%; (R_f = 0.4, 10% ethyl acetate/pet. ether); ^1H NMR (400 MHz, $CDCl_3$): δ 3.8 (s, 3H), 5.62 (s, 1H), 6.90 (d, J = 8.6 Hz, 2H), 7.14 (d, J = 8.6 Hz, 2H), 7.18–7.26 (m, H), 7.28–7.48 (m, 9H), 9.98 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 49.4 (d), 55.2 (q), 79.4 (d), 113.7 (d), 113.9 (d), 126.5 (d), 127.0 (d), 127.1 (d), 127.2 (d), 127.2 (d), 128.3 (d), 128.4 (d), 128.5 (d), 128.6 (d), 129.1 (d), 129.2 (d), 129.9 (d), 130.1 (d), 133.7 (s), 133.9 (s), 142.1 (s), 142.7 (s), 144.6 (s), 148.3 (d), 158.2 (s), 158.9 (s), 192.0 (d) ppm; HRMS (ESI+): calcd. For $C_{23}H_{20}O_2Na$ $[M+Na]^+$ 351.1356; found 351.1354.

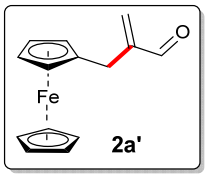


2-((2,4-Bimethoxyphenyl)(phenyl)methyl)-3-phenylacrylaldehyde (2u): Colorless syrup; 94%; (R_f = 0.4, 10% ethyl acetate/pet. ether); ^1H NMR (400 MHz, $CDCl_3$): δ 3.72 (s, 3H), 3.76

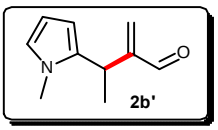
(s, 4H), 5.60 (s, 1H), 5.90 (s, 1H), 6.20 (s, 1H), 6.33–6.43 (m, 1H), 6.43–6.50 (m, 1H), 6.72 (d, J = 8.6 Hz, 1H), 7.11 (d, J = 7.1 Hz, 2H), 7.17–7.41 (m, 5H), 9.62 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 42.1 (d), 43.3 (d), 55.3 (q), 55.5 (q), 98.7 (d), 98.8 (d), 103.6 (d), 122.5 (s), 126.4 (d), 128.3 (d), 129.0(d), 129.2 (d), 129.7 (d), 129.9 (d), 130.0 (d), 134.2 (s), 135.7 (t), 141.0(s), 141.6 (s), 144.3 (s), 147.1 (d), 152.6 (s), 157.7 (s), 159.7 (s), 191.9 (s), 193.3 (d) ppm; HRMS (ESI+): calcd. For $C_{24}H_{22}O_3Na$ $[M+Na]^+$ 381.1461; found 381.1461.



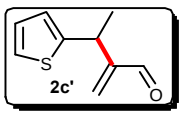
Cyclopenta-2,4-dien-1-yl(2-(2-formylallyl)cyclopenta-2,4-dien-1-yl)iron (2a'): Orange color powder; 96%; (R_f = 0.6, 5% ethyl acetate/pet. ether); IR ($CHCl_3$) v: 3362, 3093, 2924, 2853, 1894, 1693, 1627, 1464, 1340, 1245, 1105, 959, 819 cm^{-1}; ^{1}H NMR (200 MHz, $CDCl_3$): δ 3.31 (bs, 2H), 4.06–4.20 (m, 9H), 5.95 (s, 1H), 6.08 (s, 1H), 9.56 (s, 1H); ^{13}C NMR (50 MHz, $CDCl_3$): δ 28.1 (t), 67.7 (d, 2C), 68.4 (d), 68.7 (d, 3C), 68.9 (d, 2C), 69.4 (d), 84.6 (s), 134.6 (t), 150.2 (s), 194.0 (d) ppm; HRMS (ESI+): calcd. For $C_{14}H_{14}OFe$ $[M^+]$ 254.0389; found 254.0386.



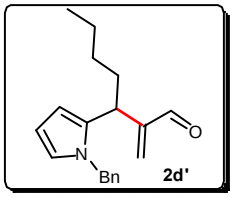
3-(1-Methyl-1H-pyrrol-2-yl)-2-methylenebutanal (2b'): Yellow oil; 89%; (R_f = 0.5, 5% ethyl acetate/pet. ether); ^{1}H NMR (200 MHz, $CDCl_3$): δ 1.40 (d, J = 7.1 Hz, 3H), 3.39 (s, 3H), 4.04 (q, J = 7.1 Hz, 1H), 5.95 (s, 1H), 6.02–6.04 (m, 1H), 6.04–6.07 (m, 1H), 6.07–6.12 (m, 1H), 6.54–6.60 (m, 1H), 9.63 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 19.6 (q), 28.9 (d), 33.4 (q), 106.1 (d), 106.5 (d), 121.8 (d), 134.4 (s), 134.8 (t), 154.0 (s), 193.7 (d) ppm; HRMS (ESI+): calcd. For $C_{10}H_{14}ON$ $[M+1]^+$ 164.1070; found 164.1070; $C_{10}H_{13}ONNa$ $[M+Na]^+$ 186.0889; found 186.0889.



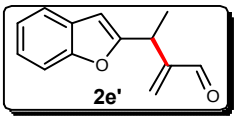
2-Methylene-3-(thiophen-2-yl)butanal (2c'): Colorless oil; 91%; (R_f = 0.5, 5% ethyl acetate/pet. ether); ^{1}H NMR (200 MHz, $CDCl_3$): δ 1.52 (d, J = 7.2 Hz, 3H), 4.34 (q, J = 7.2 Hz, 1H), 6.09 (s, 1H), 6.28 (d, J = 1.0 Hz, 1H), 6.86 (dt, J = 3.4, 1.1 Hz, 1H), 6.94 (dd, J = 5.12, 3.5 Hz, 1H), 7.16 (dd, J = 5.1, 1.3 Hz, 1H), 9.58 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 21.4 (q), 32.5 (d), 123.5 (d), 124.1 (d), 126.6 (d), 134.2 (t), 147.4 (s), 154.2 (s), 193.4 (d) ppm; HRMS (ESI+): calcd. For $C_9H_{10}ONaS$ $[M+Na]^+$ 189.0345; found 189.0344.



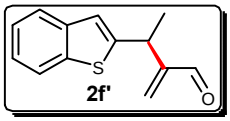
3-(1-Benzyl-1H-pyrrol-2-yl)-2-methyleneheptanal (2d'): Yellow oil; 84%; (R_f = 0.5, 5% ethyl acetate/pet. ether); ^{1}H NMR (200 MHz, $CDCl_3$): δ 0.78–0.88 (m, 3H), 1.11–1.33 (m, 4H), 1.62–1.74 (m, 2H), 3.91 (t, J = 7.3 Hz, 1H), 4.97 (s, 2H), 5.95 (s, 1H), 6.1 (s, 1H), 6.12–6.18 (m, 1H), 6.20–6.27 (m, 1H), 6.67–6.74 (m, 1H), 7.1 (dd, J = 7.4, 1.7 Hz, 2H), 7.27–7.39 (m, 3H), 9.49 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 13.9 (q), 22.4 (t), 29.9 (t), 33.6 (d), 34.7 (t), 50.5 (t), 106.6 (d), 107.0 (d), 121.4 (d), 126.7 (d, 2C), 127.3 (d), 128.5 (d, 2C), 133.7 (s), 135.4 (t), 138.2 (s), 153.0 (s), 193.8 (d) ppm; HRMS (ESI+): calcd. For $C_{19}H_{24}ON$ $[M+1]^+$ 282.1852; found 282.1851; $C_{19}H_2ONNa$ $[M+Na]^+$ 304.1672; found 304.1671.



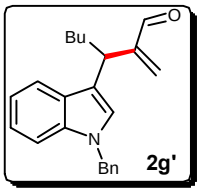
3-(Benzofuran-2-yl)-2-methylenebutanal (2e'): Yellow gum; 91%; (R_f = 0.4, 5% ethyl acetate/pet. ether); ^{1}H NMR (500 MHz, $CDCl_3$): δ 1.53 (d, J = 7.0 Hz, 3H), 4.28 (d, J = 7.0 Hz, 1H), 6.14 (s, 1H), 6.30 (s, 1H), 6.52 (s, 1H), 7.18–7.28 (m, 2H), 7.43 (d, J = 7.9 Hz, 1H), 7.53 (d, J = 7.3 Hz, 1H), 9.62 (s, 1H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 18.0 (q), 31.5 (d), 102.6 (d), 110.9 (d), 120.5 (d), 122.5 (d), 123.6 (d), 128.4 (s), 134.8 (t), 151.5 (s), 154.7 (s), 159.6 (s), 193.1 (d) ppm; HRMS (ESI+): calcd. For $C_{13}H_{13}O_2$ $[M+1]^+$ 201.0910; found 201.0910.



3-(Benzo[b]thiophen-2-yl)-2-methylenebutanal (2f'): Colorless gum; 92%; (R_f = 0.4, 5% ethyl acetate/pet. ether); ^{1}H NMR (200 MHz, $CDCl_3$): δ 1.6 (d, J = 7.1 Hz, 3H), 4.5 (d, J = 7.1 Hz, 1H), 6.01–6.15 (m, 2H), 7.27 (d, J = 1.0 Hz, 1H), 7.32–7.42 (m, 2H), 7.53–7.62 (m, 1H), 7.84–7.96 (m, 1H), 9.70 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 19.5 (q), 30.9 (d), 121.8 (d), 122.0 (d), 122.8 (d), 123.9 (d), 124.4 (d), 134.8 (t), 137.9 (s), 138.5 (s), 140.5 (s), 153.5 (s), 193.7(d) ppm; HRMS (ESI+): calcd. For $C_{13}H_{12}ONaS$ $[M+Na]^+$ 239.0501; found 239.0500.

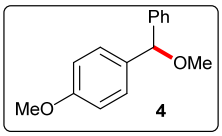


3-(1-Benzyl-1H-indol-3-yl)-2-methyleneheptanal (2g'): Yellow gum; 81%; (R_f = 0.4, 5% ethyl acetate/pet. ether); ^{1}H NMR (400 MHz, $CDCl_3$): δ 0.83–0.93 (m, 3H), 1.29–1.42 (m, 4H), 1.84–2.02 (m, 2H), 4.22 (t, J = 7.6 Hz, 1H), 5.32 (s, 2H), 6.05 (s, 1H), 6.27 (s, 1H), 7.03 (s, 1H), 7.05–7.12 (m, 3H), 7.16 (ddd, J = 8.2, 6.9, 1.4 Hz, 1H), 7.25 (d, J = 8.2 Hz, 1H), 7.27–7.34 (m, 2H), 7.54 (dd, J = 8.9, 1.1 Hz, 1H), 9.63 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 14.0 (q), 22.6 (t), 30.2 (t), 33.8(t), 34.3

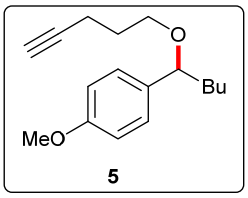


(d), 49.9 (t), 109.7 (d), 116.6 (s), 119.0 (d), 119.5 (d), 121.8 (d), 126.1 (d), 126.5 (d, 2C), 127.4 (d), 127.5 (d), 128.7 (d, 2C), 134.4 (t), 136.8 (s), 137.7 (s), 153.5 (s, 2C), 194.3 (d) ppm; HRMS (ESI+): calcd. For $C_{23}H_{26}ON$ $[M+1]^+$ 332.2009; found 232.2008; $C_{23}H_{25}ONNa$ $[M+Na]^+$ 354.1828; found 354.1828;

General Procedure C: At 0 °C, a solution of allenylether1c(100 mg, 567 µmol) and 3 equivalents of nucleophile (MeOH) in dichloromethane (5 mL) was treated with the catalyst Stock solution (1 mol % catalyst) and stirred for 3 h at room temperature. The reaction mixture was concentrated under reduced pressure and the resulting crude was purified by silica gel column chromatography to afford 3(71 mg, 81% yield) as a colorless liquid.



1-Methoxy-4-(methoxy(phenyl)methyl)benzene (4):[1] Yield: 85%; ^{1}H NMR (200 MHz, $CDCl_3$): δ 3.41 (s, 3H), 3.78 (s, 3H), 5.26 (s, 1H), 6.84–6.97 (m, 2H), 7.23–7.46 (m, 7H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 55.0 (q), 56.7 (q), 84.8 (d), 113.6 (d, 2C), 126.7 (d, 2C), 127.2 (d), 128.1 (d, 2C), 128.2 (d, 2C), 134.2 (s), 142.3 (s), 158.8 (s) ppm;

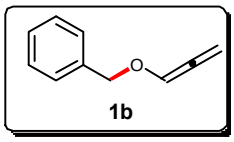


1-Methoxy-4-(1-(pent-4-yn-1-yloxy)pentyl)benzene (5): Yellow oil; 72%; (R_f = 0.5, 10% ethyl acetate/pet. ether); IR (neat) ν: 3296, 2999, 2954, 2931, 1658, 1510, 1462, 1244, 1171, 1097, 1035, 830, 634 cm^{-1}; ^{1}H NMR (200 MHz, $CDCl_3$): δ 0.88 (t, J = 6.8, 3H), 1.24–1.37 (m, 5H), 1.76 (t, J = 6.4 Hz, 3H), 1.91 (t, J = 2.6 Hz, 1H), 2.23–2.35 (m, 2H), 3.25–3.44 (m, 2H), 3.82 (s, 3H), 4.13 (dd, J = 6.3, 7.1 Hz, 1H), 6.88 (d, J = 8.6 Hz, 2H), 7.20 (d, J = 8.6 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 14.0 (q), 15.4 (t), 22.6 (t), 28.1 (t), 28.8 (t), 38.0 (t), 55.2 (q), 66.8 (t), 68.3 (s), 82.0 (d), 84.1 (d), 113.6 (d, 2C), 127.7 (d, 2C), 135.1 (s), 158.8 (s) ppm; HRMS (ESI+): calcd. For $C_{17}H_{24}O_2Na$ $[M+Na]^+$ 283.1668; found 283.1669.

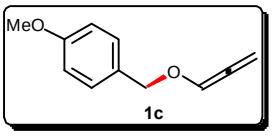
References:

[1] a) K. W. C. Poon and G. B. Dudley, *J. Org. Chem.,* 2006, **71**, 3923; b) E. O. Nwoye and G. B. Dudley, *Chem. Commun.,* 2007, 1436.

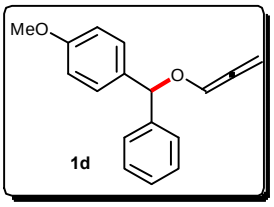
((Propa-1,2-dien-1-yloxy)methyl)benzene (1b): Light yellowish oily liquid; 82%; (R_f = 0.7, 5% ethyl acetate/pet. ether); ^{1}H NMR (200 MHz, $CDCl_3$): δ 4.60 (s, 2H), 5.46 (d, J = 5.9 Hz, 2H), 6.83 (t, J = 5.9 Hz, 1H), 7.22–7.43 (m, 5H) ppm.



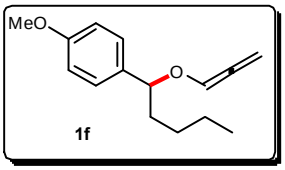
1-Methoxy-4-((propa-1,2-dien-1-yloxy)methyl)benzene (1c): colorless syrup; 83%; (R_f = 0.5, 5% ethyl acetate/pet. ether); ^{1}H NMR (200 MHz, $CDCl_3$): δ 3.79 (s, 1H), 4.53 (s, 1H), 5.46 (d, J = 5.9 Hz, 2H), 6.80 (t, J = 5.9 Hz, 1H), 6.85–6.91 (m, 2H), 7.27 (d, J = 8.6 Hz, 1H); ^{13}C NMR (50 MHz, $CDCl_3$): δ 55.2 (q), 70.3 (d), 90.9 (d), 113.8 (d, 2C), 121.4 (d), 129.3 (s), 129.4 (d), 129.5 (d), 159.3 (s), 201.3 (s) ppm.



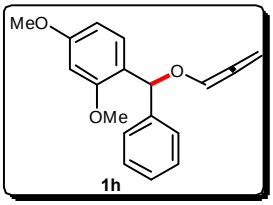
1-Methoxy-4-(phenyl(propa-1,2-dien-1-yloxy)methyl)benzene (1d): Yellow color oil; 85%; (R_f = 0.7, 5% ethyl acetate/pet. ether); ^{1}H NMR (200 MHz, $CDCl_3$): δ 3.82 (s, 3H), 5.34 (s, 1H), 5.35–5.42 (m, 1H), 5.78 (s, 1H), 6.83 (t, J = 5.9 Hz, 1H), 6.87–6.96 (m, 2H), 7.26–7.35 (m, 3H), 7.35–7.43 (m, 4H); ^{13}C NMR (50 MHz, $CDCl_3$): δ 55.1 (q), 81.5 (d), 90.6 (t), 113.7 (d, 2C), 120.4 (d), 126.8 (d, 2C), 127.4 (d), 128.3 (d, 2C), 128.4 (d, 2C), 133.5 (s), 141.5 (s), 159.0 (s), 202.0 (s) ppm.



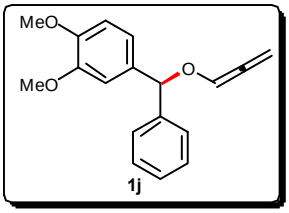
1-Methoxy-4-(1-(propa-1,2-dien-1-yloxy)pentyl)benzene (1f): Yellow color syrup; 85%; (R_f = 0.7, 5% ethyl acetate/pet. ether); ^{1}H NMR (200 MHz, $CDCl_3$): δ 0.82–0.95 (m, 3H), 1.19–1.46 (m, 5H), 1.59–1.83 (m, 1H), 1.83–2.07 (m, 1H), 3.82 (s, 3H), 4.57 (t, J = 6.7 Hz, 1H), 5.15–5.40 (m, 2H), 6.61 (t, J = 5.9 Hz, 1H), 6.84–6.96 (m, 2H), 7.18–7.28 (m, 2H); ^{13}C NMR (50 MHz, $CDCl_3$): δ 13.9 (q), 22.5 (t), 27.8 (t), 37.2 (t), 55.1 (q), 80.7 (d), 89.8 (t), 113.6 (d, 2C), 120.2 (d), 127.8 (d, 2C), 134.2 (s), 158.9 (s), 202.2 (s) ppm.



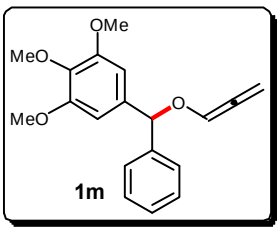
2,4-Dimethoxy-1-(phenyl(propa-1,2-dien-1-yloxy)methyl)benzene (1h): Yellow color gum; 79%; (R_f = 0.5, 5% ethyl acetate/pet. ether); ^{1}H NMR (200 MHz, $CDCl_3$): δ 3.83 (d, J = 1.3 Hz, 6H), 5.30 (d, J = 1.5 Hz, 1H), 5.33 (d, J = 1.5 Hz, 1H), 6.16 (s, 1H), 6.46–6.60 (m, 2H), 6.75–6.86 (m, 1H), 7.28–7.48 (m, 6H);



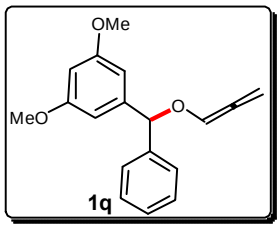
1,2-Dimethoxy-4-(phenyl(propa-1,2-dien-1-yloxy)methyl)benzene (1j): Yellow color gum; 78%; (R_f = 0.6, 5% ethyl acetate/pet. ether); ^{1}H NMR (200 MHz, $CDCl_3$): δ 3.83 (d, J = 2.0 Hz, 6H), 5.29 (dd, J = 6.0, 0.9 Hz, 2H), 5.69 (s, 1H), 6.75 (t, J = 6.0 Hz, 1H), 6.79–6.88 (m, 3H), 7.21–7.34 (m, 5H); ^{13}C NMR (50 MHz, $CDCl_3$): δ 55.8 (q, 2C), 81.7 (d), 90.6 (t), 110.2 (d), 110.8 (d), 119.7 (d), 120.4 (d), 126.9 (d, 2C), 127.5 (d), 128.3 (d, 2C), 133.8 (s), 141.4 (s), 148.5 (s), 148.9 (s), 202.0 (s) ppm.



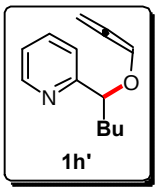
1,2,3-Trimethoxy-5-(phenyl(propa-1,2-dien-1-yloxy)methyl)benzene (1m): Yellow colr gum; 82%; (R_f = 0.4, 5% ethyl acetate/pet. ether); ^{1}H NMR (200 MHz, $CDCl_3$): δ 3.80–3.86 (m, 9H), 5.26–5.41 (m, 2H), 5.69 (s, 1H), 6.59 (s, 2H), 6.80 (t, J = 6.0 Hz, 1H), 7.24–7.40 (m, 5H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 55.9 (q, 2C), 60.7 (q), 81.8 (d), 90.7 (t), 103.9 (d, 2C), 120.3 (d), 126.8 (d, 2C), 127.6 (d), 128.2 (d, 2C), 136.8 (s), 137.1 (s), 141.0 (s), 153.0 (s, 2C), 201.8 (s) ppm.

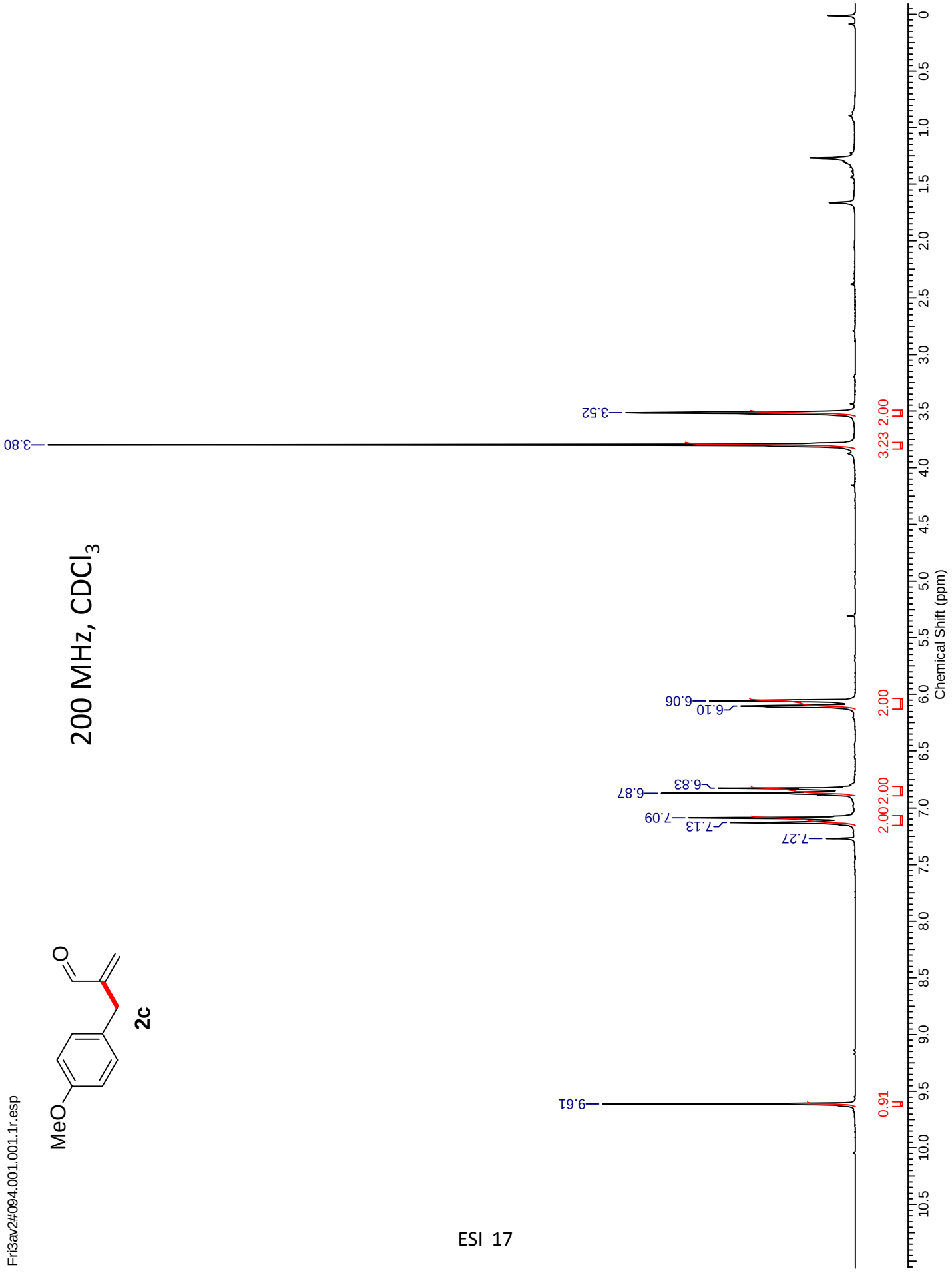


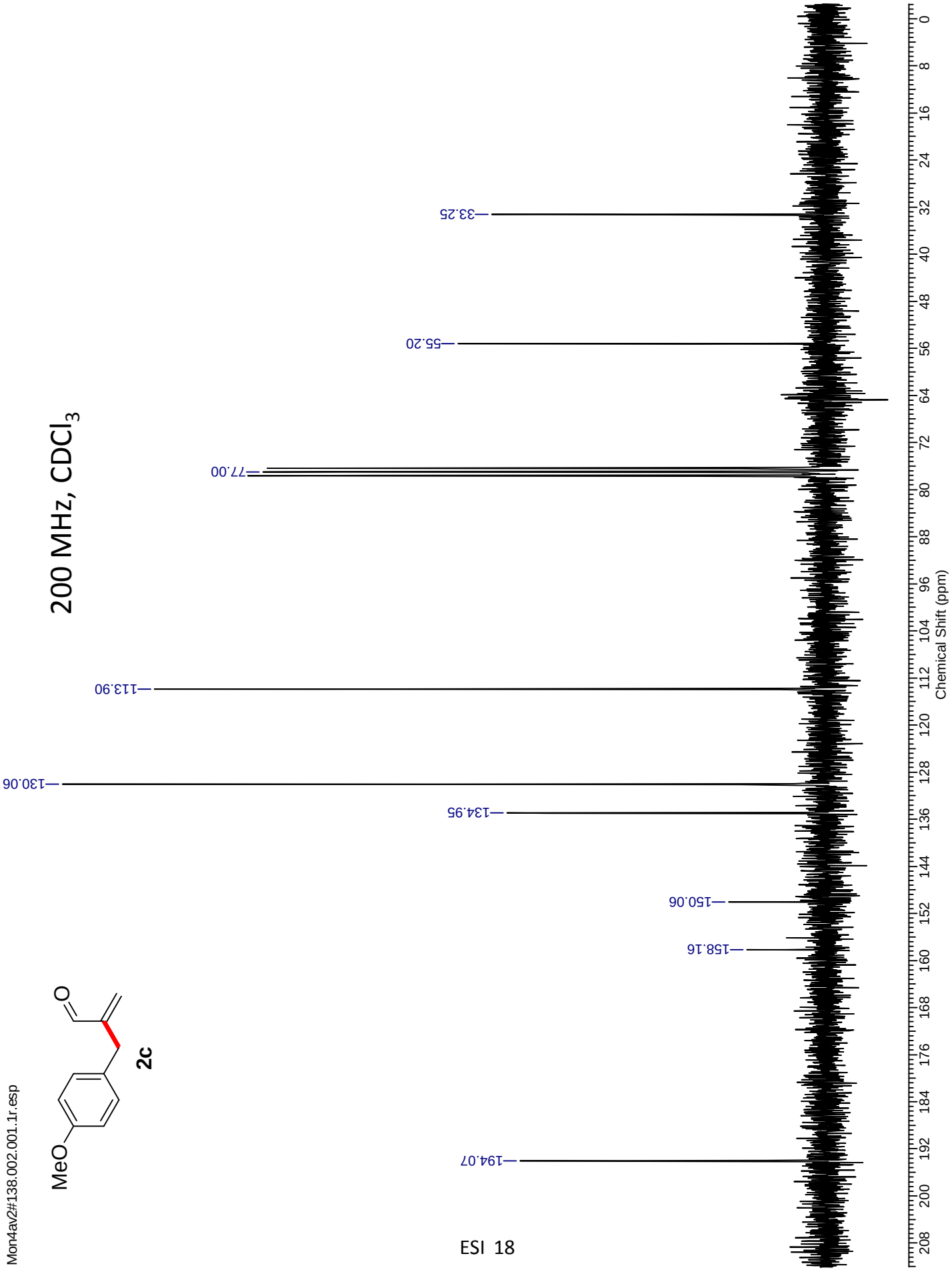
1,3-Dimethoxy-5-(phenyl(propa-1,2-dien-1-yloxy)methyl)benzene (1q): Yellow color gel; 84%; (R_f = 0.5, 5% ethyl acetate/pet. ether); ^{1}H NMR (200 MHz, $CDCl_3$): δ 3.80 (s, 6H), 5.37 (d, J = 6.0 Hz, 2H), 5.74 (s, 1H), 6.42 (t, J = 2.3 Hz, 1H), 6.59 (d, J = 2.3 Hz, 2H), 6.85 (t, J = 5.9Hz, 1H), 7.28–7.45 (m, 5H); ^{13}C NMR (50 MHz, $CDCl_3$): δ 55.2 (q, 2C), 81.8 (d), 90.8 (t), 99.3 d), 105.0 (d, 2C), 120.4 (d), 126.9 (d, 2C), 127.6(d), 128.3 (d, 2C), 141.1 (s), 143.7 (s), 160.7 (s, 2C), 201.9 (s) ppm;



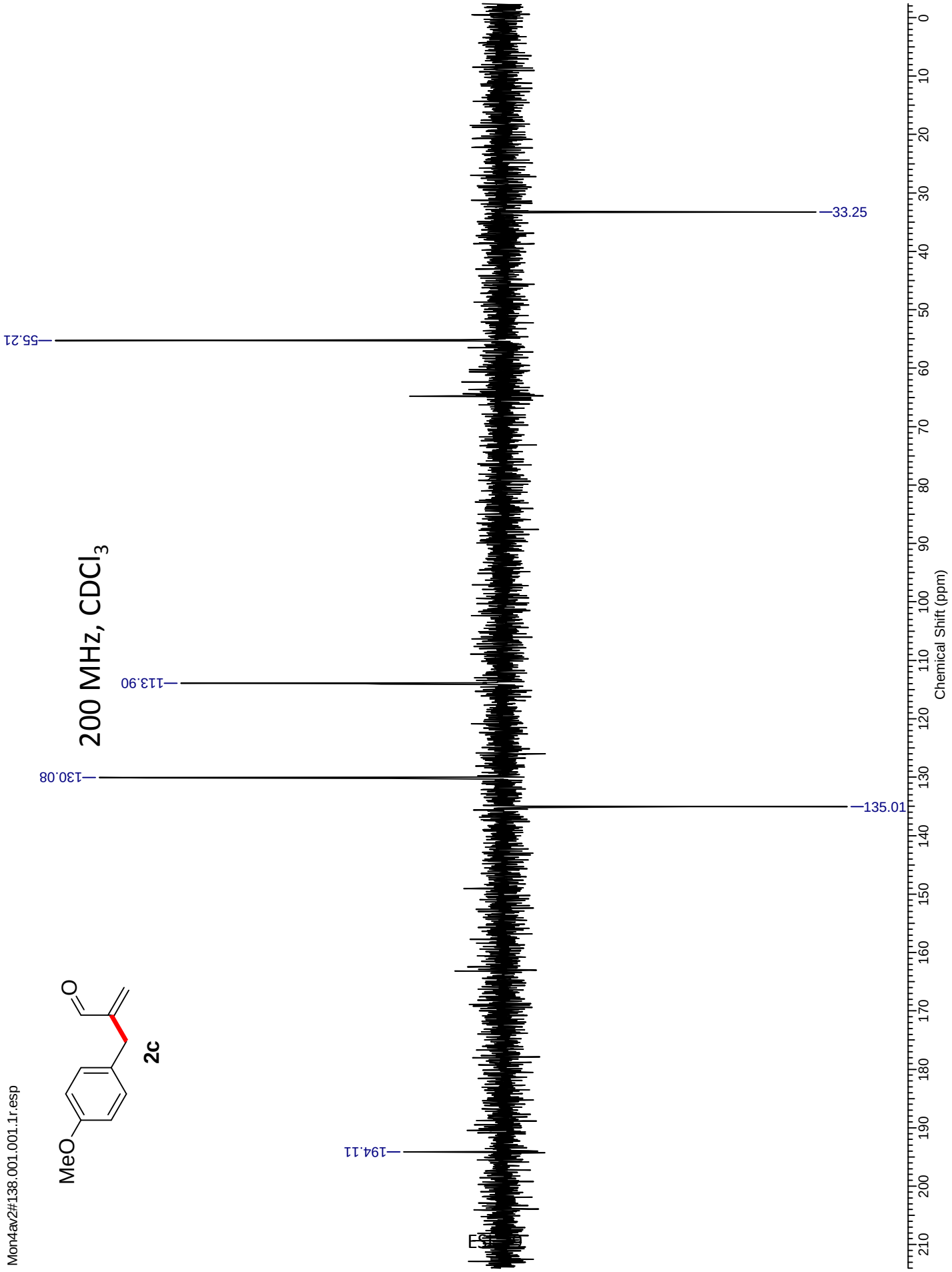
2-(Octa-1,2-dien-4-yl)pyridine (1h'): Black color oil; 72%; (R_f = 0.5, 5% ethyl acetate/pet. ether); ^{1}H NMR (200 MHz, $CDCl_3$): δ 0.79–0.92 (m, 3H), 1.21–1.47 (m, 4H), 1.76–1.94 (m, 2H), 4.73 (t, J = 6.4 Hz, 1H), 5.10 (dd, J = 8.3, 5.9 Hz, 1H), 5.3 (dd, J = 8.3, 6.0 Hz, 1H), 6.70 (t, J = 5.9 Hz, 1H), 7.1 (ddd, J = 7.5, 4.9, 1.1Hz, 1H), 7.3 (d, J = 7.8 Hz, 1H), 7.60–7.70 (m, 1H), 8.5–8.6 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 13.9 (q), 22.5 (t), 27.5 (t), 36.0 (t), 81.5 (d), 90.5 (t), 120.4 (d), 120.5 (d), 122.0 (d), 136.4 (d), 148.9 (d), 161.7 (s), 201.7 (s) ppm.

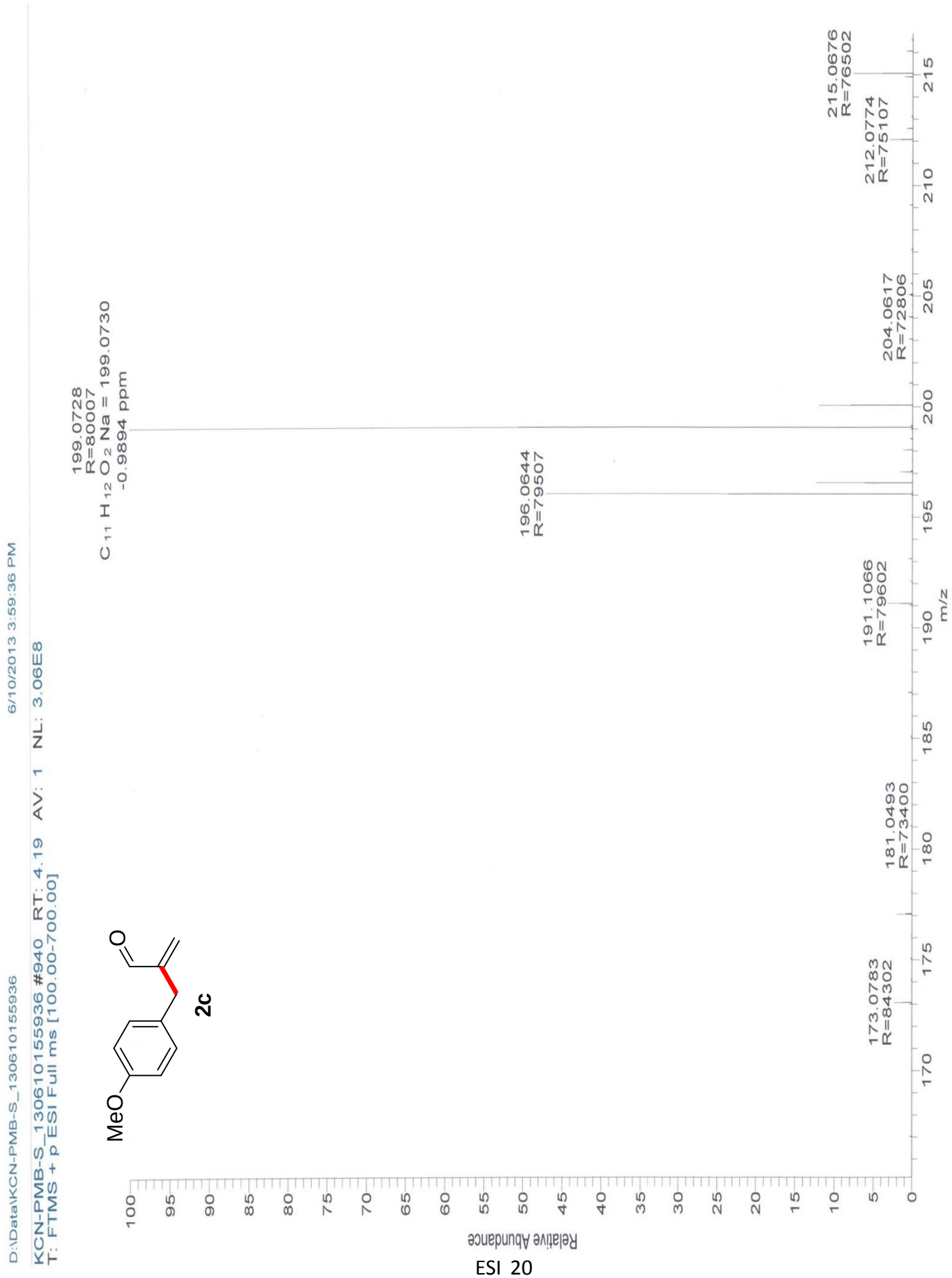


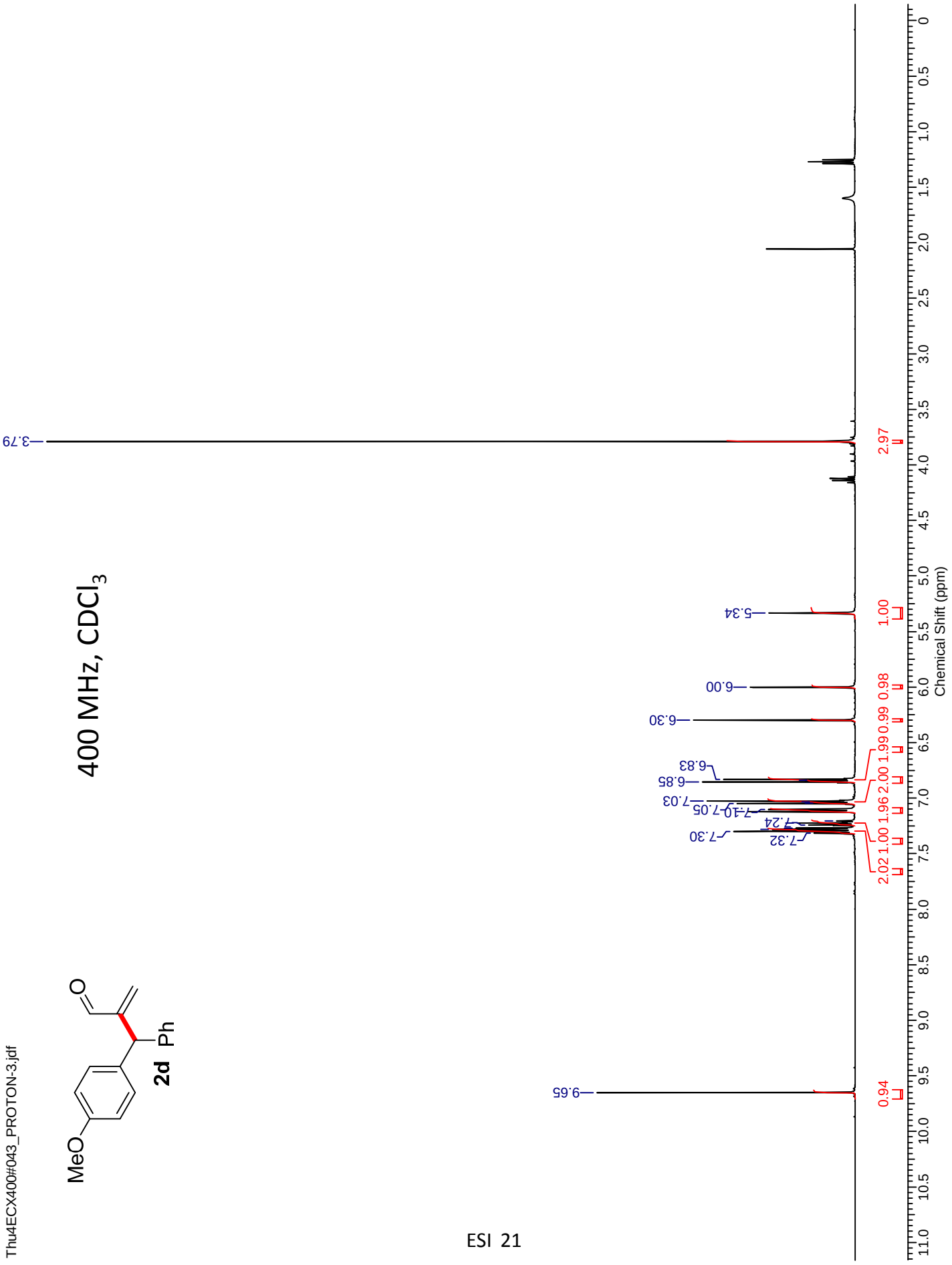


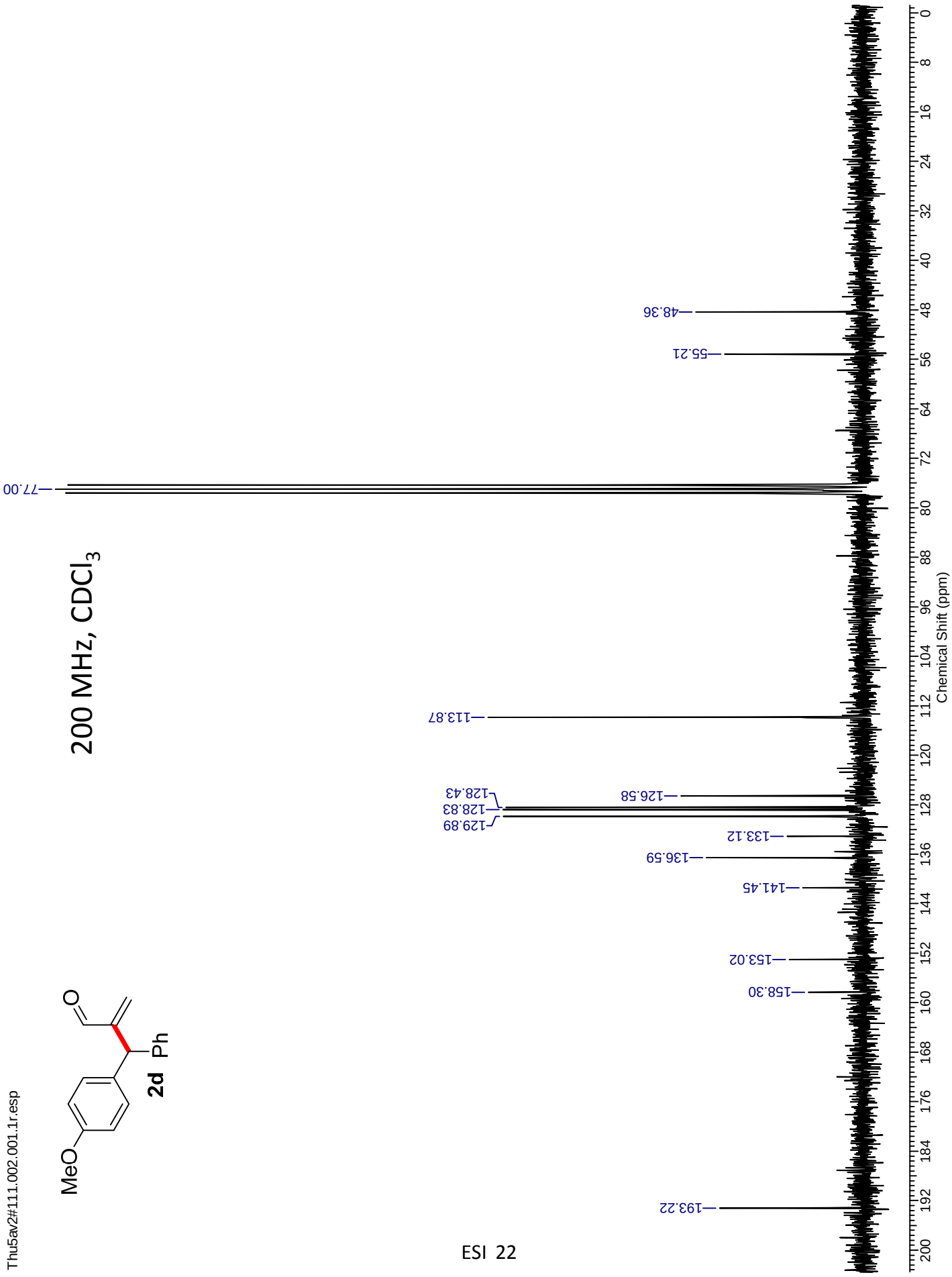

200 MHz, CDCl3
33.25
55.20
77.00
113.90
130.06
134.95
150.06
158.16
194.07
MeO
O
2c
Chemical Shift (ppm)
0
8
16
24
32
40
48
56
64
72
80
88
96
104
112
120
128
136
144
152
160
168
176
184
192
200
208
Mon4av2#138.002.001.1r.esp

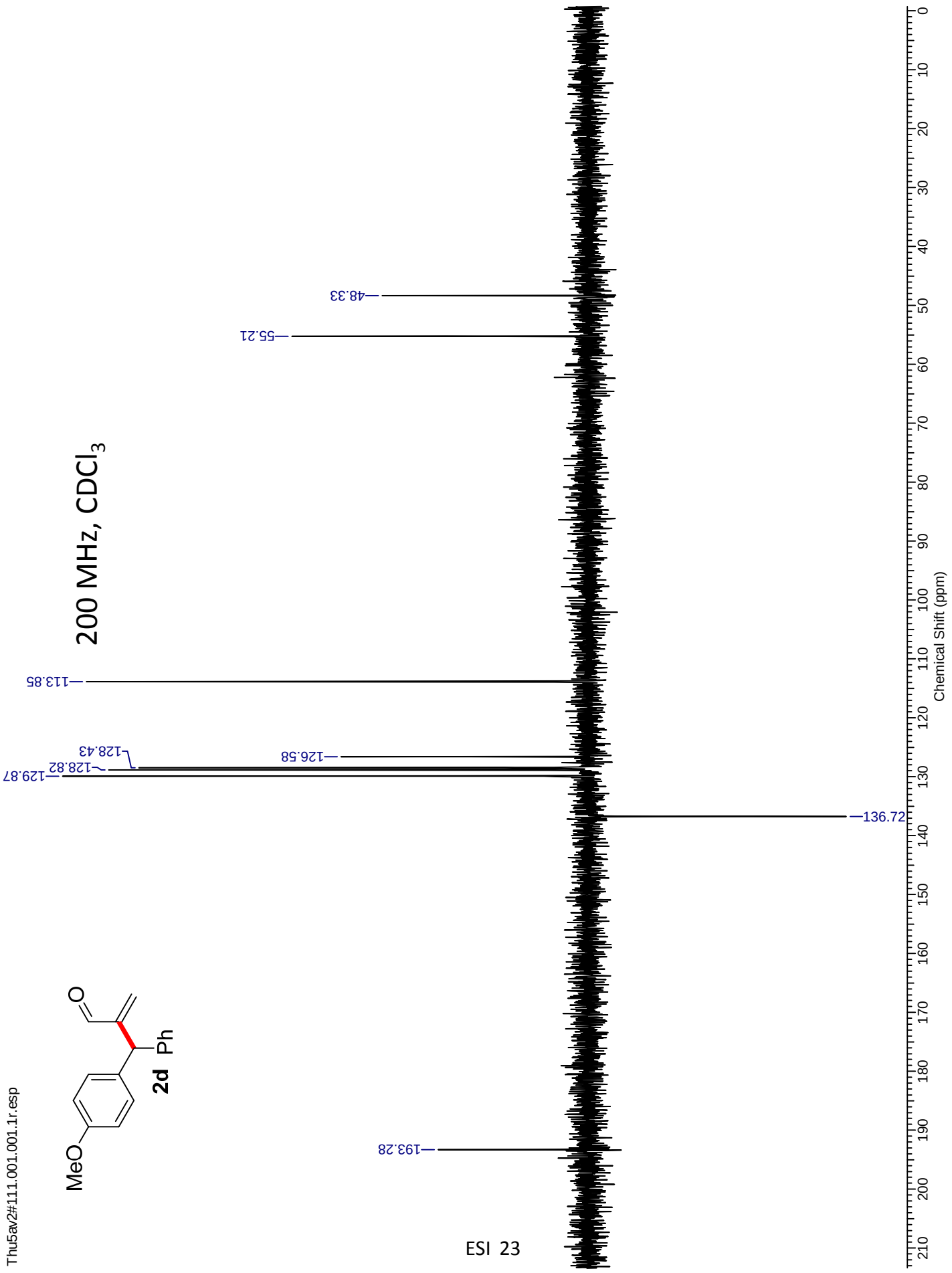
ESI 18

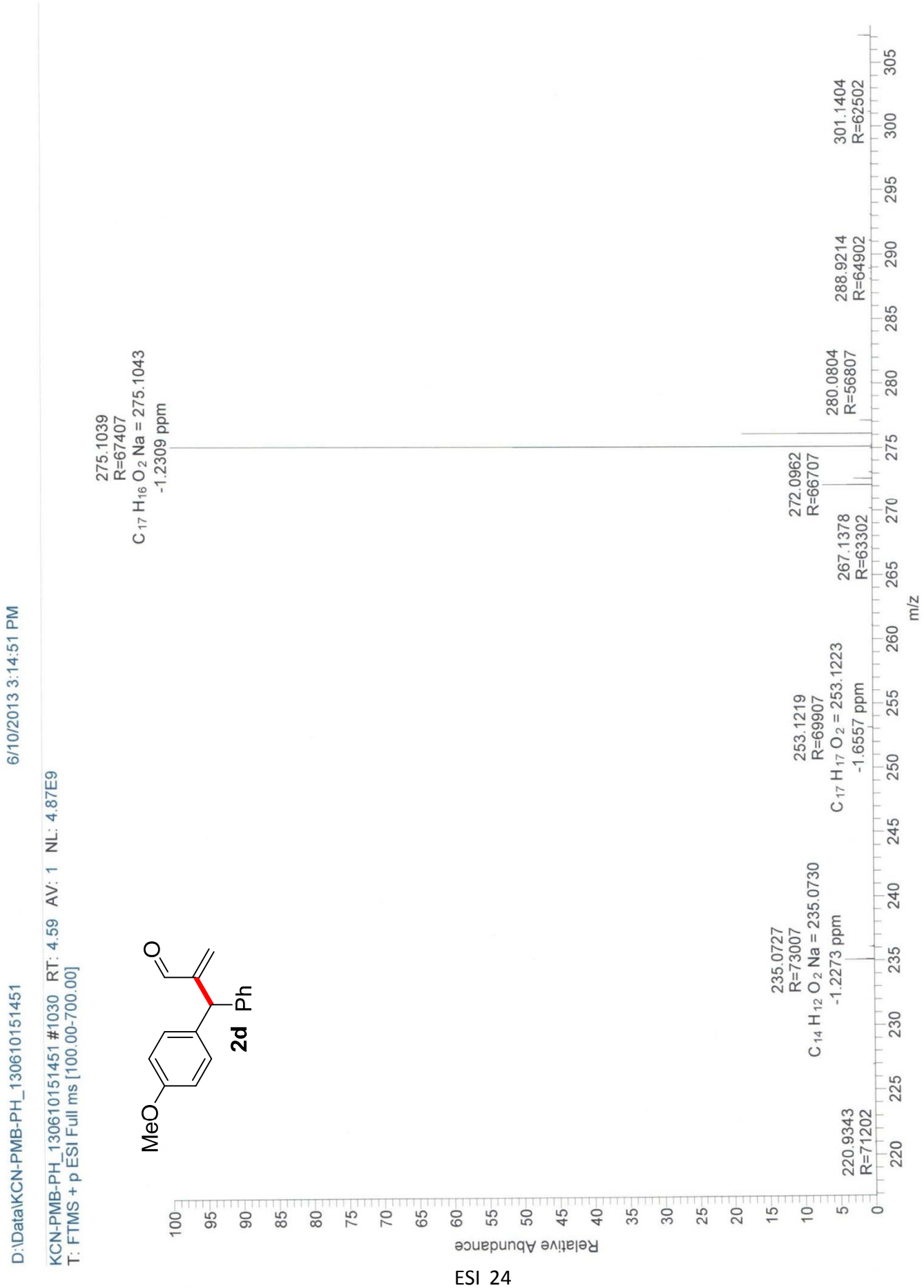



D:\Data\KCN-PMB-S_130610155936
6/10/2013 3:59:36 PM
KCN-PMB-S_130610155936 #940 RT: 4.19 AV: 1 NL: 3.06E8
T: FTMS + p ESI Full ms [100.00-700.00]
199.0728
R=80007
C 11 H 12 O 2 Na = 199.0730
-0.9894 ppm
196.0644
R=79507
215.0676
R=76502
212.0774
R=75107
204.0617
R=72806
191.1066
R=79602
181.0493
R=73400
173.0783
R=84302
MeO
O
2c
Relative Abundance
m/z


400 MHz, CDCl3
2d
Ph
MeO
O
Thu4ECX400#043_PROTON-3.jdf
9.65
7.32
7.30
7.24
7.10
7.05
7.03
6.85
6.83
6.30
6.00
5.34
3.79
0.94
2.02
1.00
1.96
2.00
1.99
0.99
0.98
1.00
2.97
Chemical Shift (ppm)


200 MHz, $CDCl_3$
77.00
48.36
55.21
113.87
126.58
128.43
128.83
129.89
133.12
136.59
141.45
153.02
158.30
193.22
Chemical Shift (ppm)
0
8
16
24
32
40
48
56
64
72
80
88
96
104
112
120
128
136
144
152
160
168
176
184
192
200
O
Ph
MeO
2d
Thu5av2#111.002.001.1r.esp


200 MHz, CDCl3
48.33
55.21
113.85
126.58
128.43
128.82
129.87
136.72
193.28
Chemical Shift (ppm)
0
10
20
30
40
50
60
70
80
90
100
110
120
130
140
150
160
170
180
190
200
210
O
Ph
MeO
2d
Thu5av2#111.001.001.1r.esp
ESI 23


D:\Data\KCN-PMB-PH_130610151451
6/10/2013 3:14:51 PM
KCN-PMB-PH_130610151451 #1030 RT: 4.59 AV: 1 NL: 4.87E9
T: FTMS + p ESI Full ms [100.00-700.00]
275.1039
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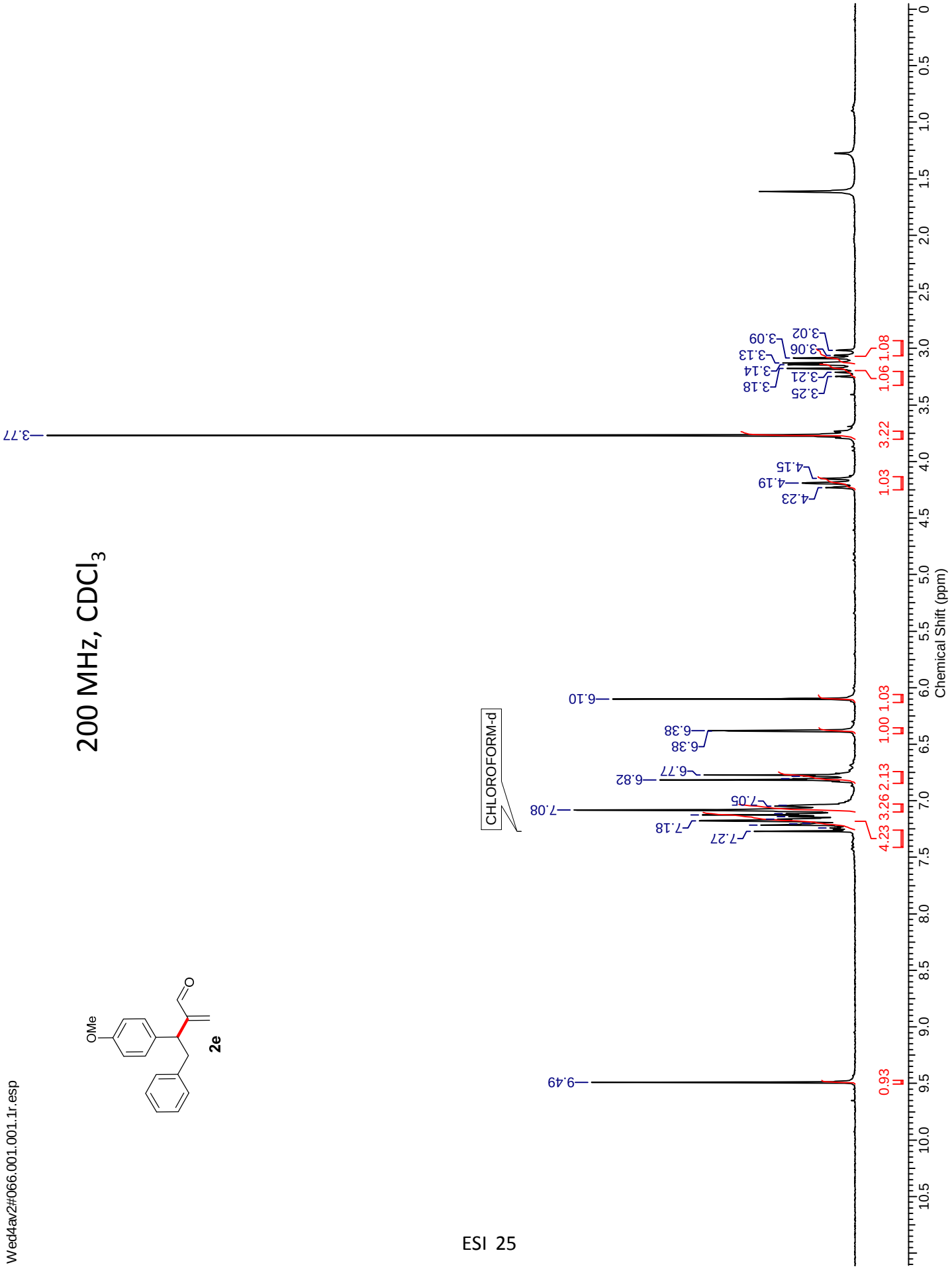
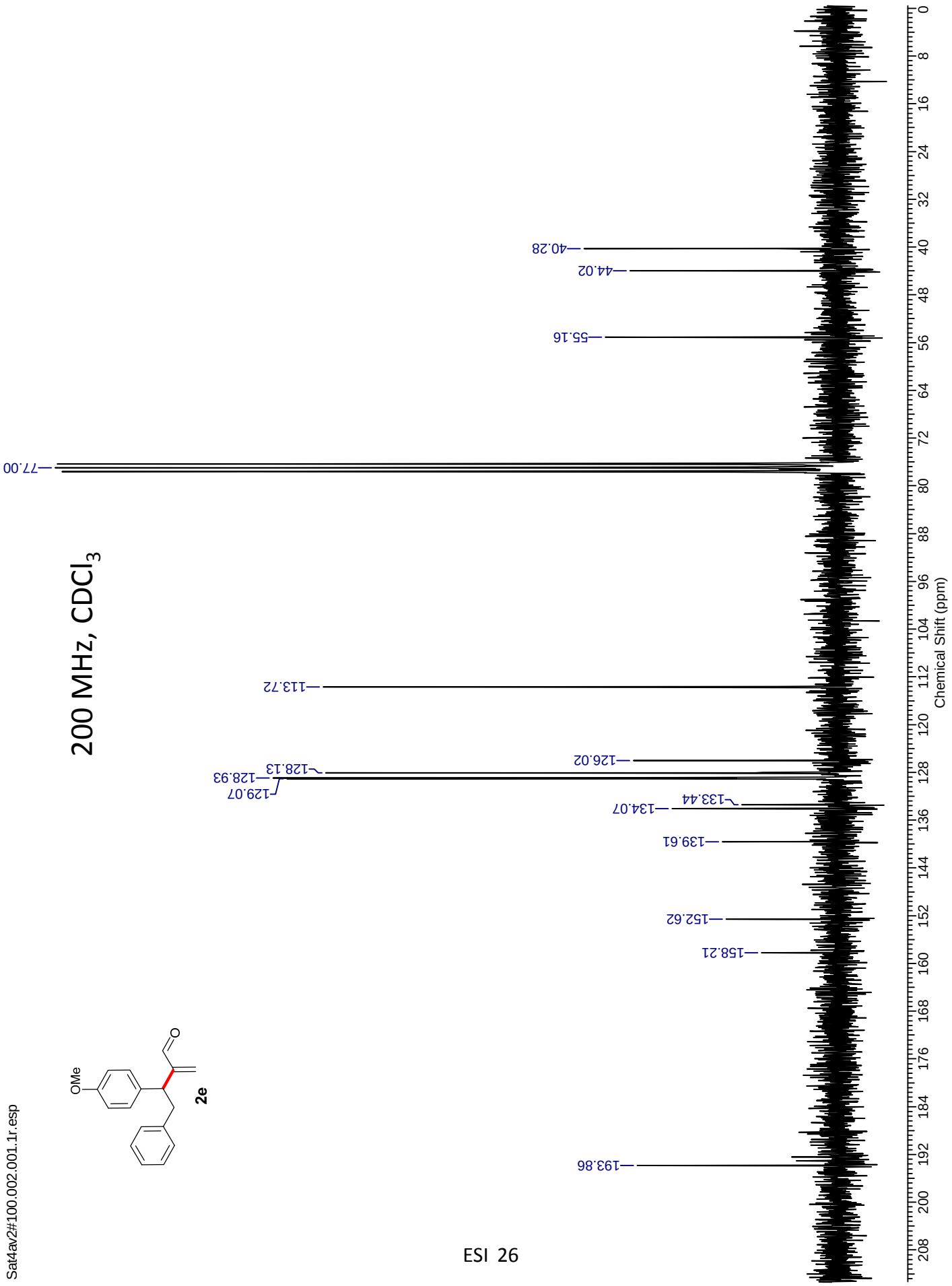
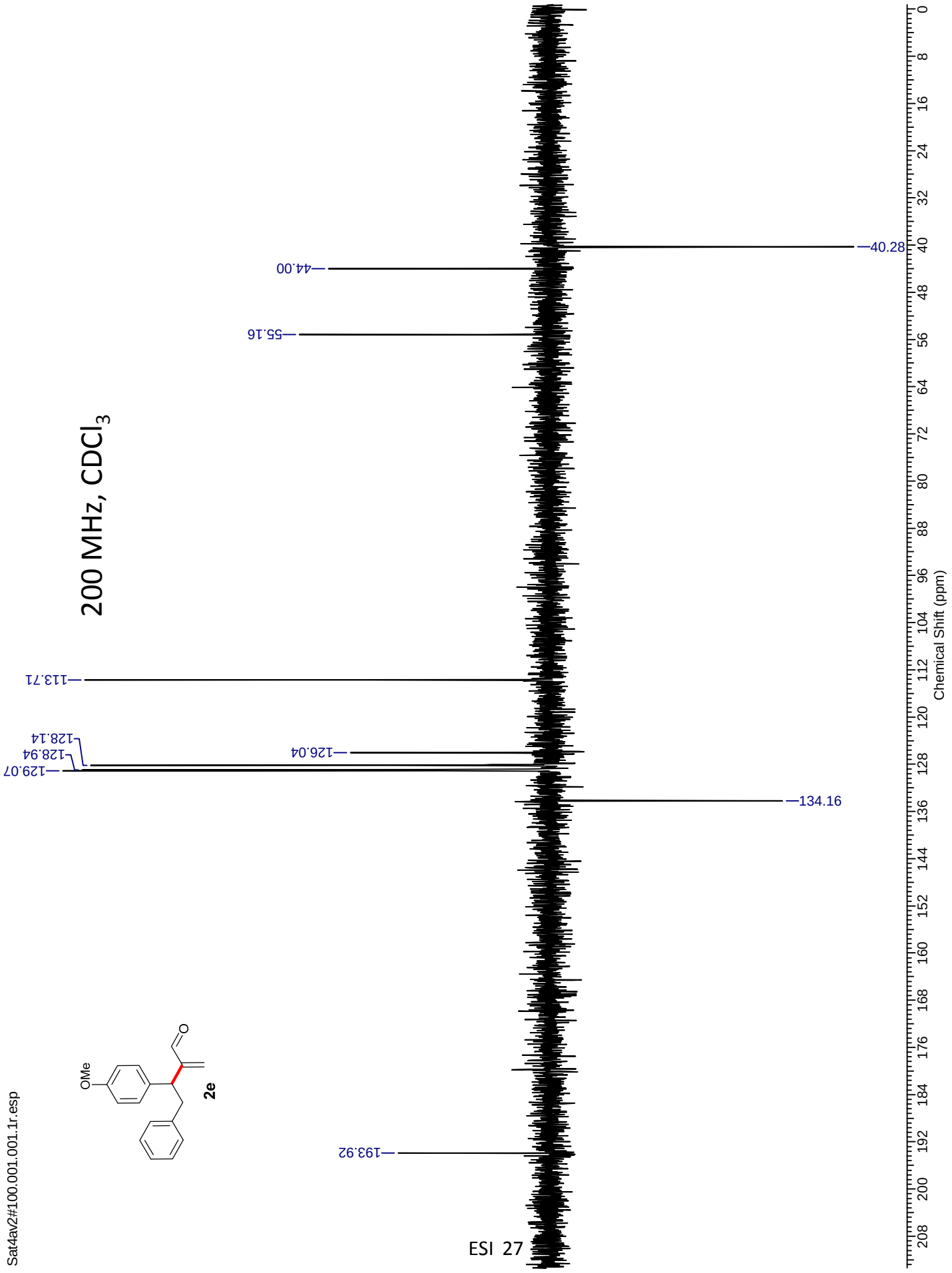
220.9343
R=71202
235.0727
R=73007
C 14 H12 O 2 Na = 235.0730
-1.2273 ppm
253.1219
R=69907
C 17 H17 O 2 = 253.1223
-1.6557 ppm
267.1378
R=63302
272.0962
R=66707
280.0804
R=56807
288.9214
R=64902
301.1404
R=62502
MeO
2d Ph
Relative Abundance
m/z


200 MHz, CDCl3
Chemical Shift (ppm)
CHLOROFORM-d
OMe
O
2e
Wed4av2#066.001.001.1r.esp
9.49
7.27
7.18
7.08
7.05
6.82
6.77
6.38
6.38
6.10
4.23
4.19
4.15
3.77
3.25
3.21
3.18
3.14
3.13
3.09
3.06
3.02
0.93
4.23
3.26
2.13
1.00
1.03
1.03
3.22
1.06
1.08

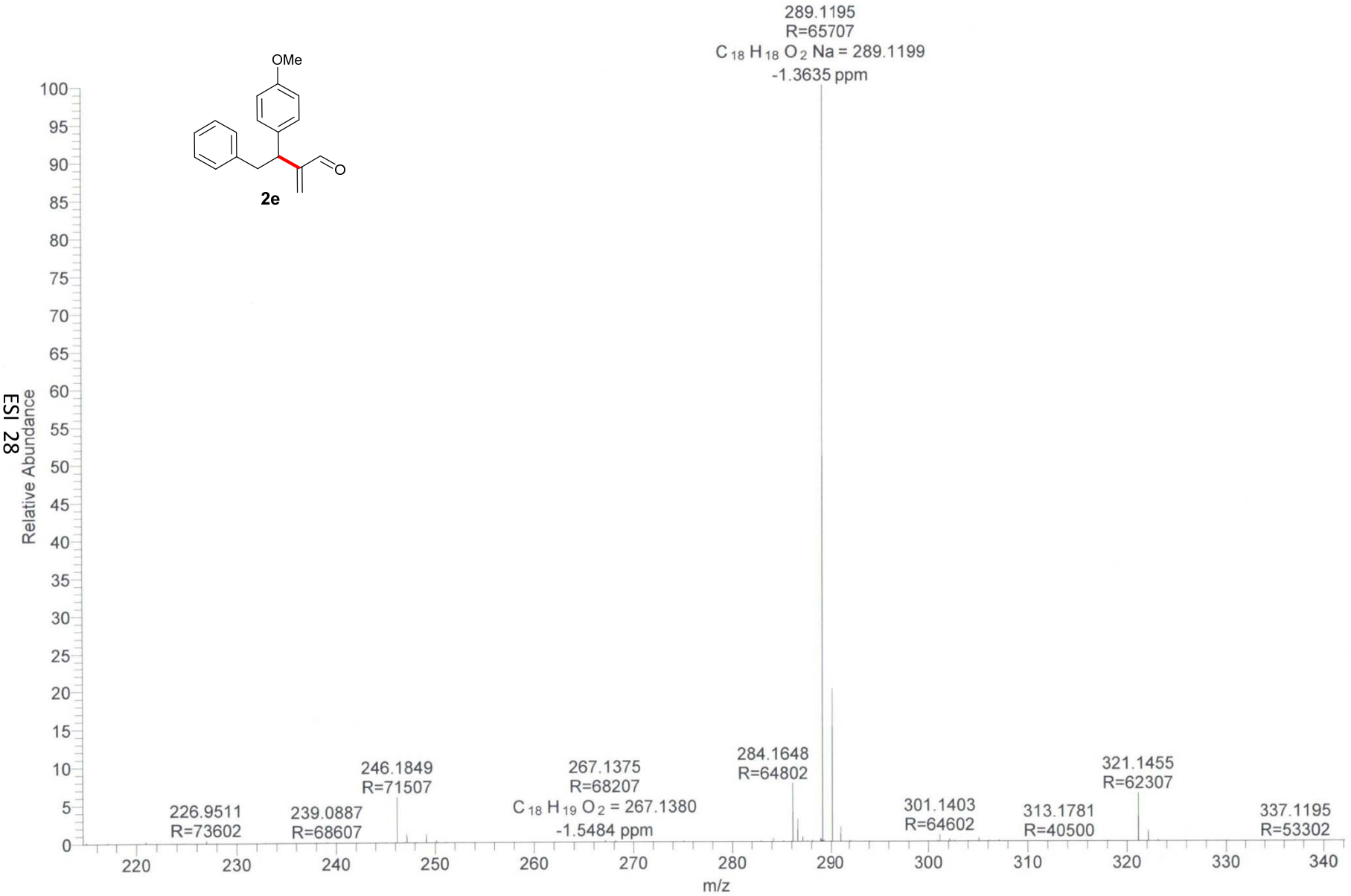

200 MHz, CDCl3
193.86
158.21
152.62
139.61
134.07
133.44
129.07
128.93
128.13
126.02
113.72
77.00
55.16
44.02
40.28
OMe
O
2e
Chemical Shift (ppm)
Sat4av2#100.002.001.1r.esp

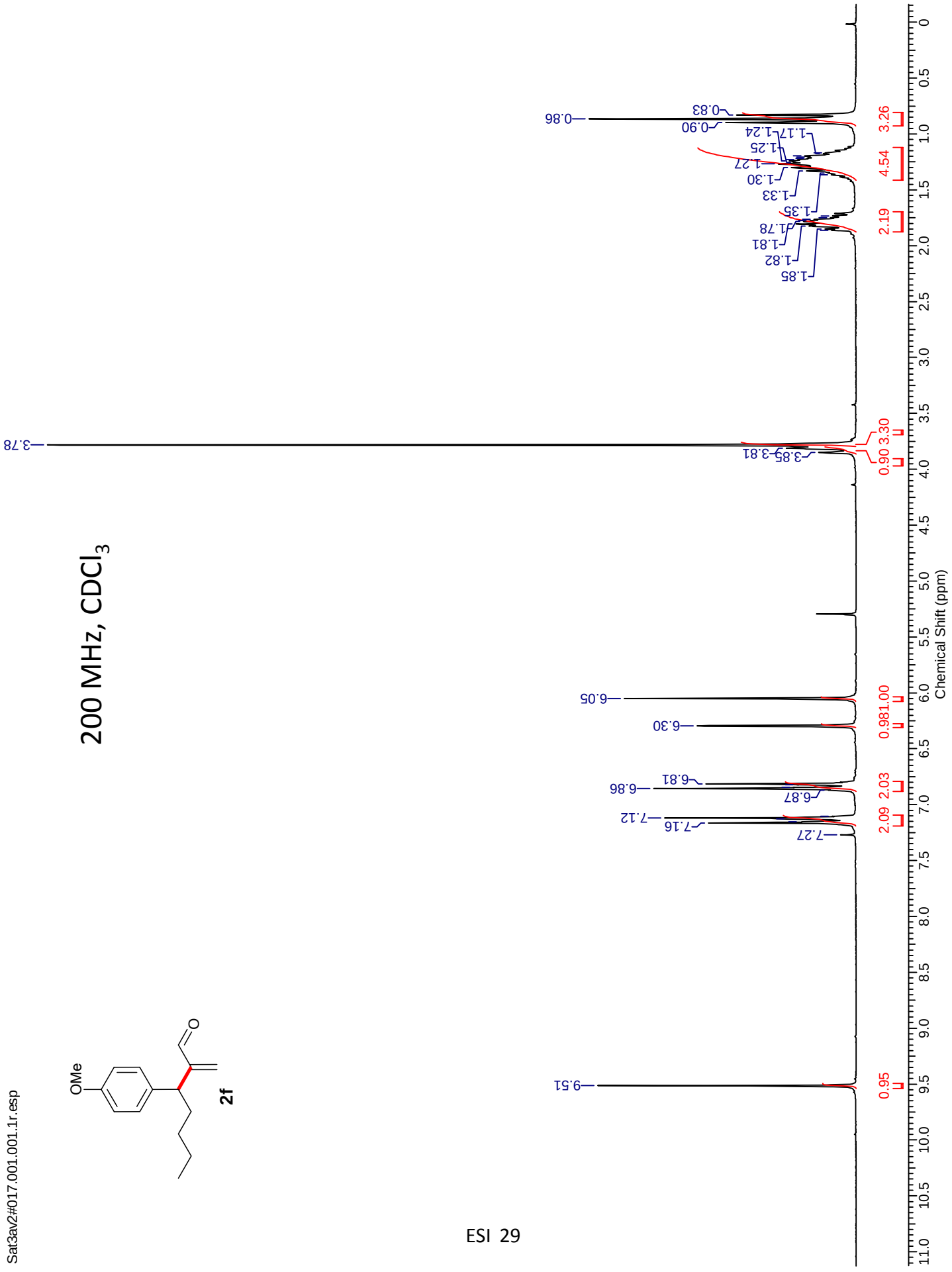


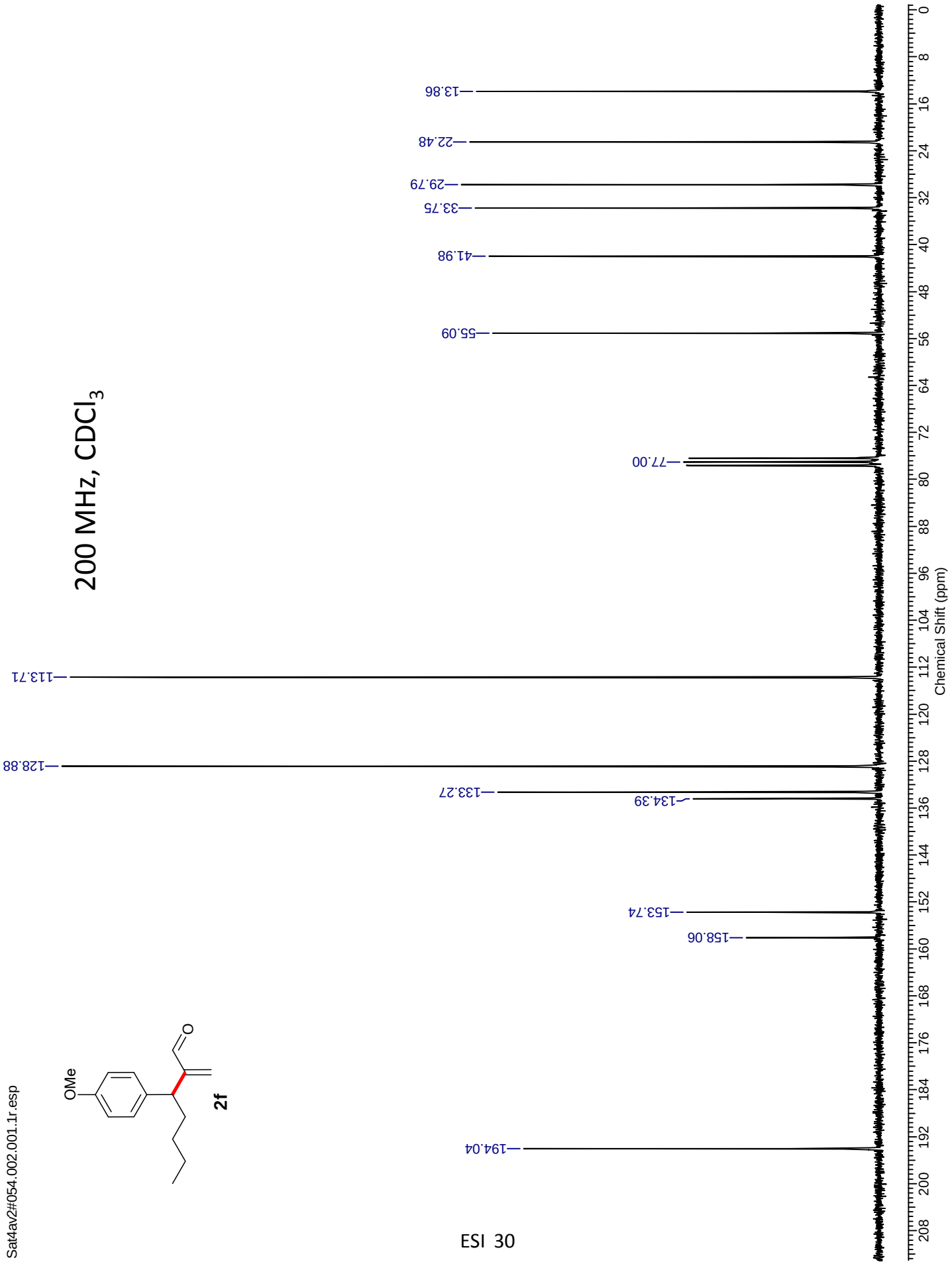
D:\Data\KCN-PMB-BN_130610150341 6/10/2013 3:03:41 PM

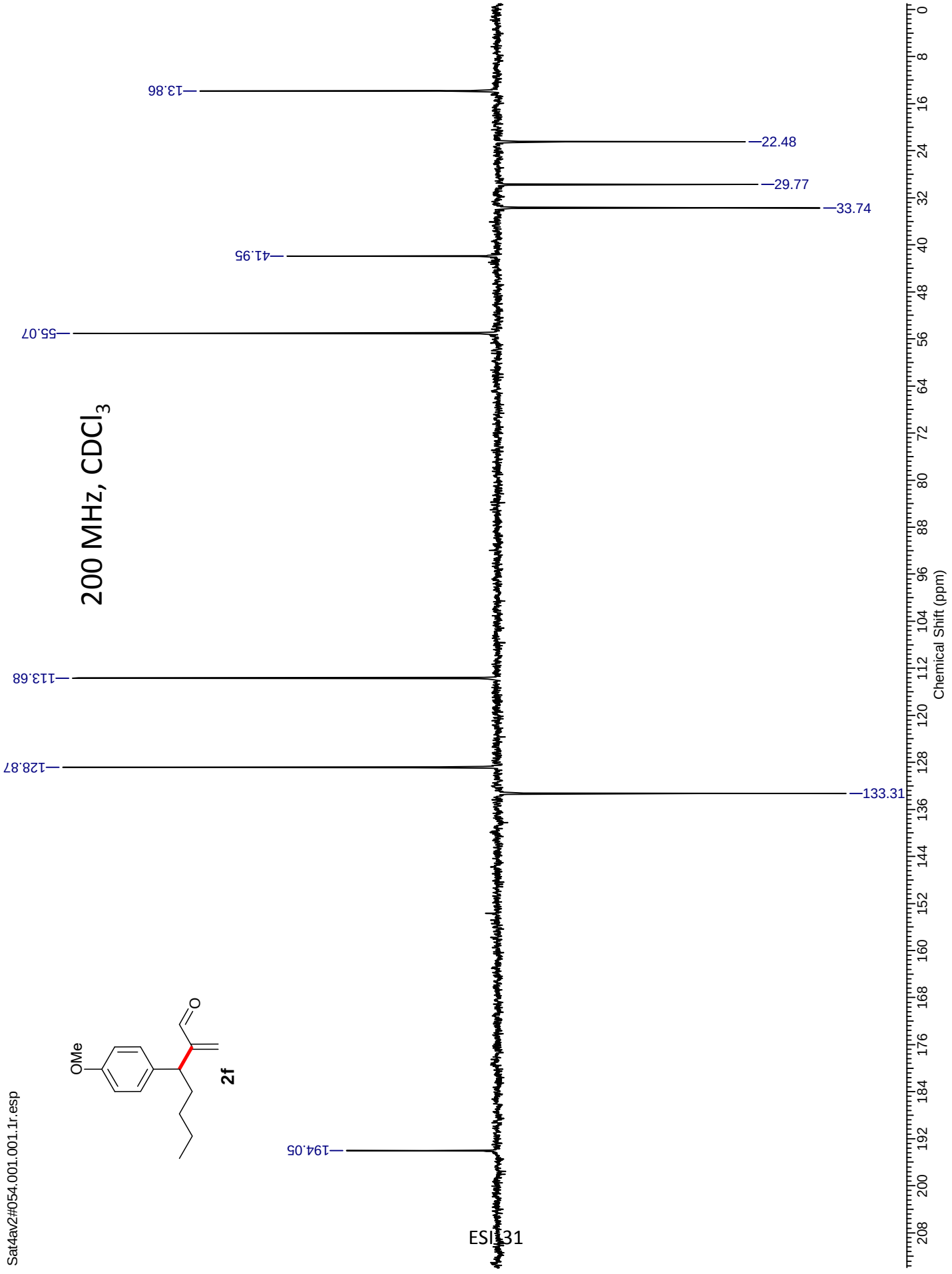
KCN-PMB-BN_130610150341 #1058 RT: 4.71 AV: 1 NL: 3.14E9
T: FTMS + p ESI Full ms [100.00-700.00]

2e




200 MHz, CDCl3
Sat3av2#017.001.001.1r.esp
OMe
O
2f
9.51
7.27
7.16
7.12
6.87
6.86
6.81
6.30
6.05
3.85
3.81
3.78
1.85
1.82
1.81
1.78
1.35
1.33
1.30
1.27
1.25
1.17
1.24
0.90
0.83
0.86
0.95
2.09
2.03
0.98
1.00
0.90
3.30
2.19
4.54
3.26
Chemical Shift (ppm)

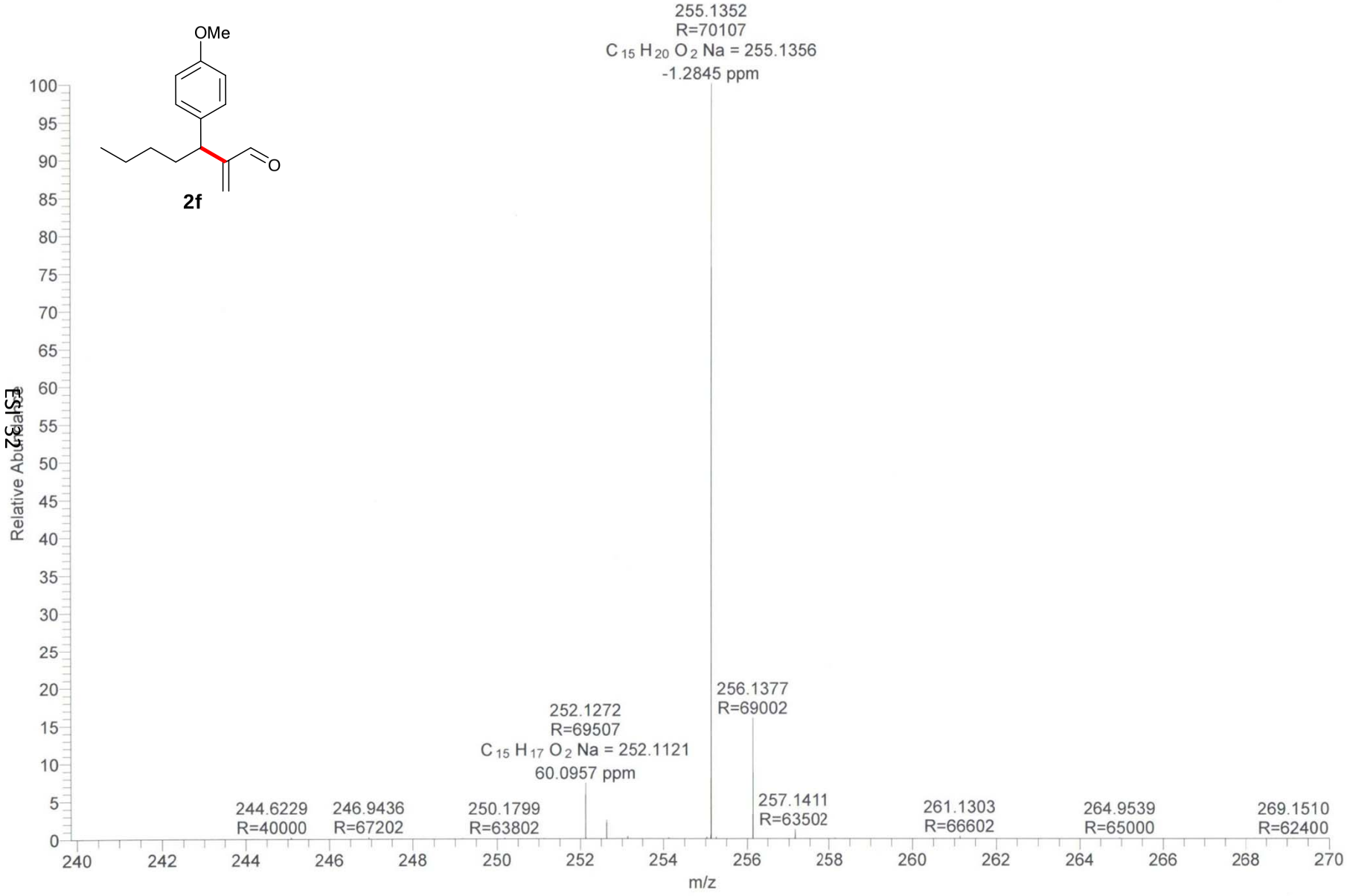

200 MHz, CDCl3
13.86
22.48
29.79
33.75
41.98
55.09
77.00
113.71
128.88
133.27
134.39
153.74
158.06
194.04
OMe
O
2f
Chemical Shift (ppm)
0
8
16
24
32
40
48
56
64
72
80
88
96
104
112
120
128
136
144
152
160
168
176
184
192
200
208
Sat4av2#054.002.001.1r.esp
ESI 30



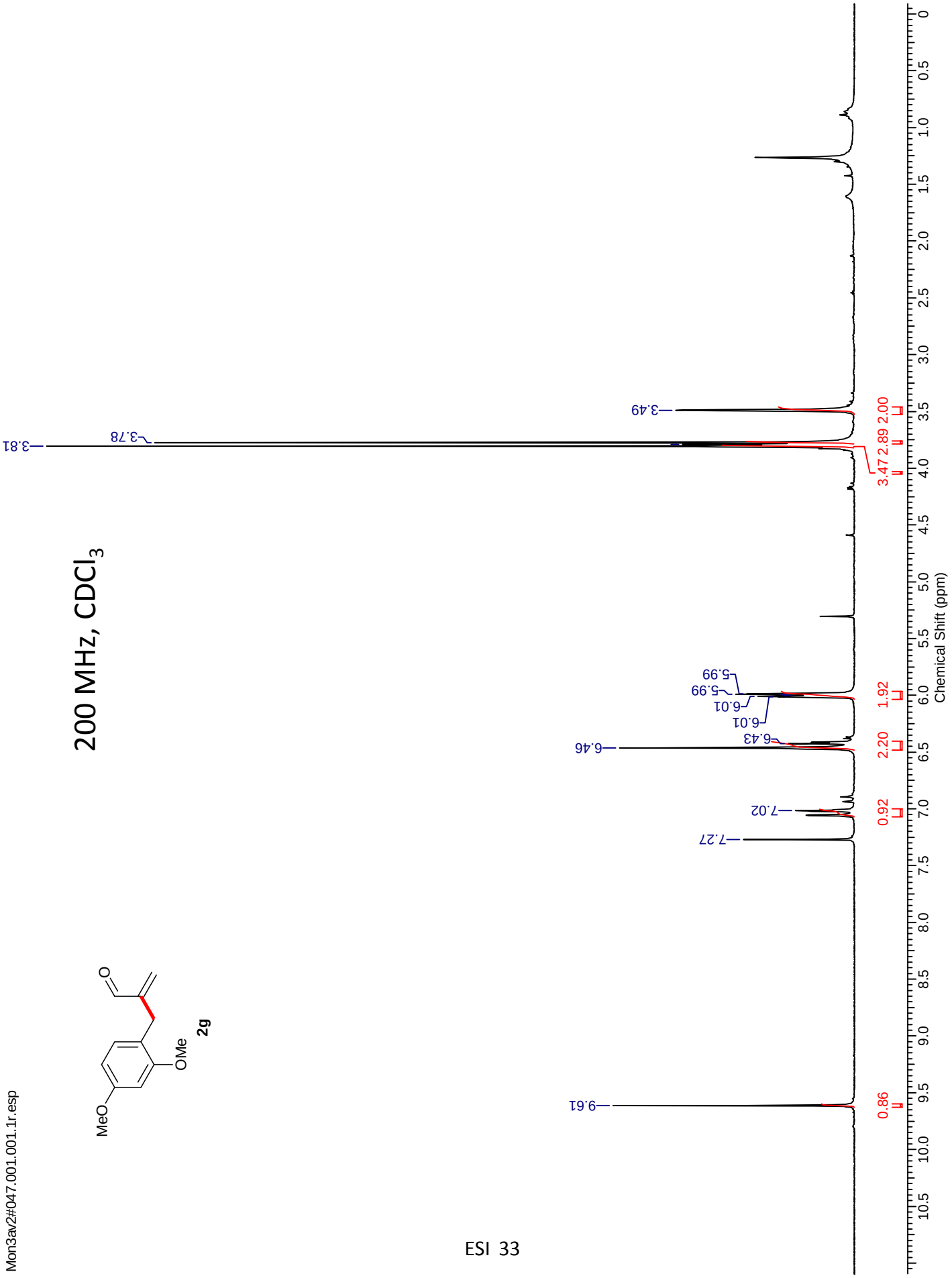
D:\Data\KCN-PMB-BU_130611135222 6/11/2013 1:52:22 PM

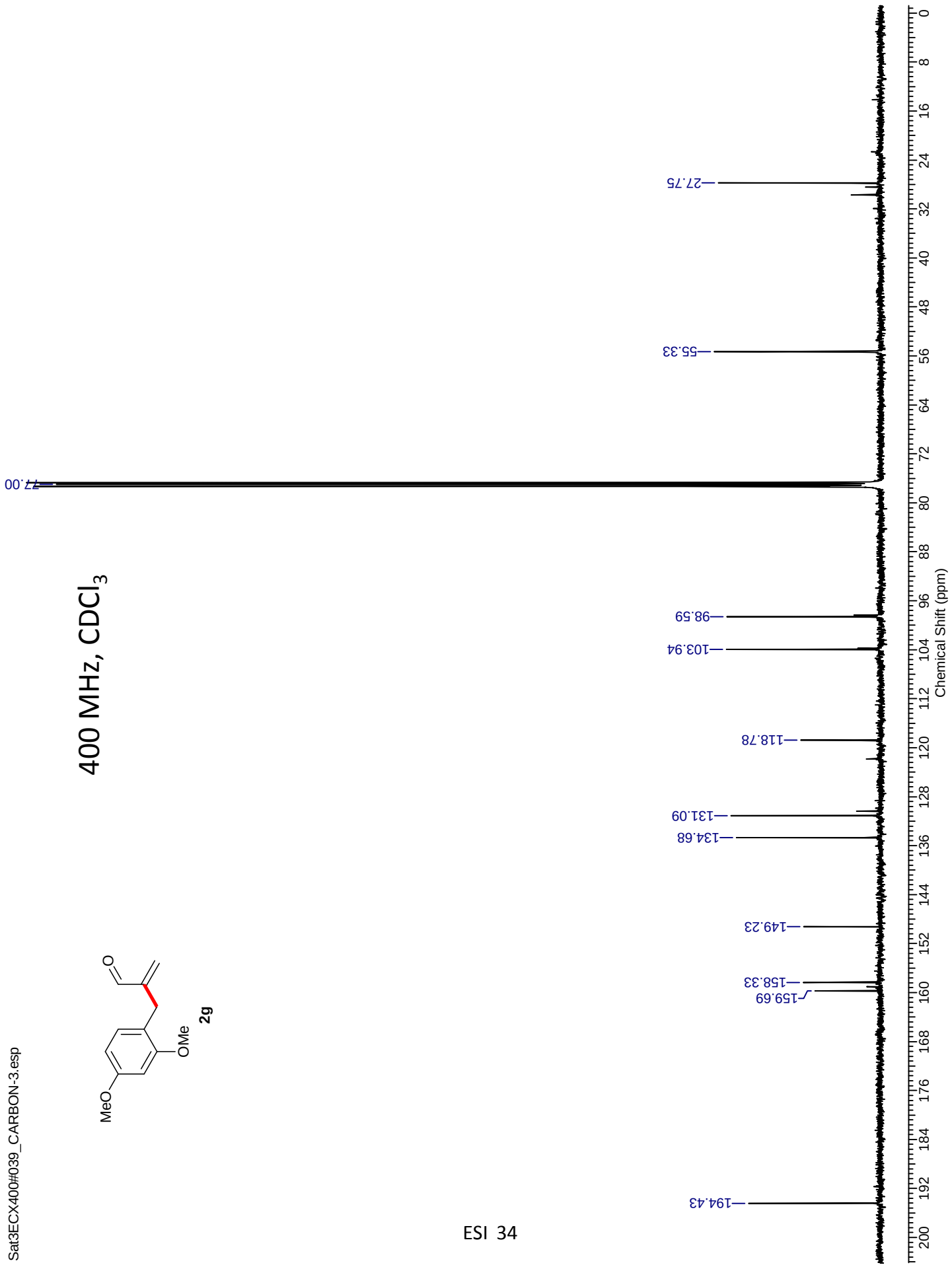
KCN-PMB-BU_130611135222 #1244 RT: 5.54 AV: 1 NL: 1.08E9
T: FTMS + p ESI Full ms [100.00-700.00]

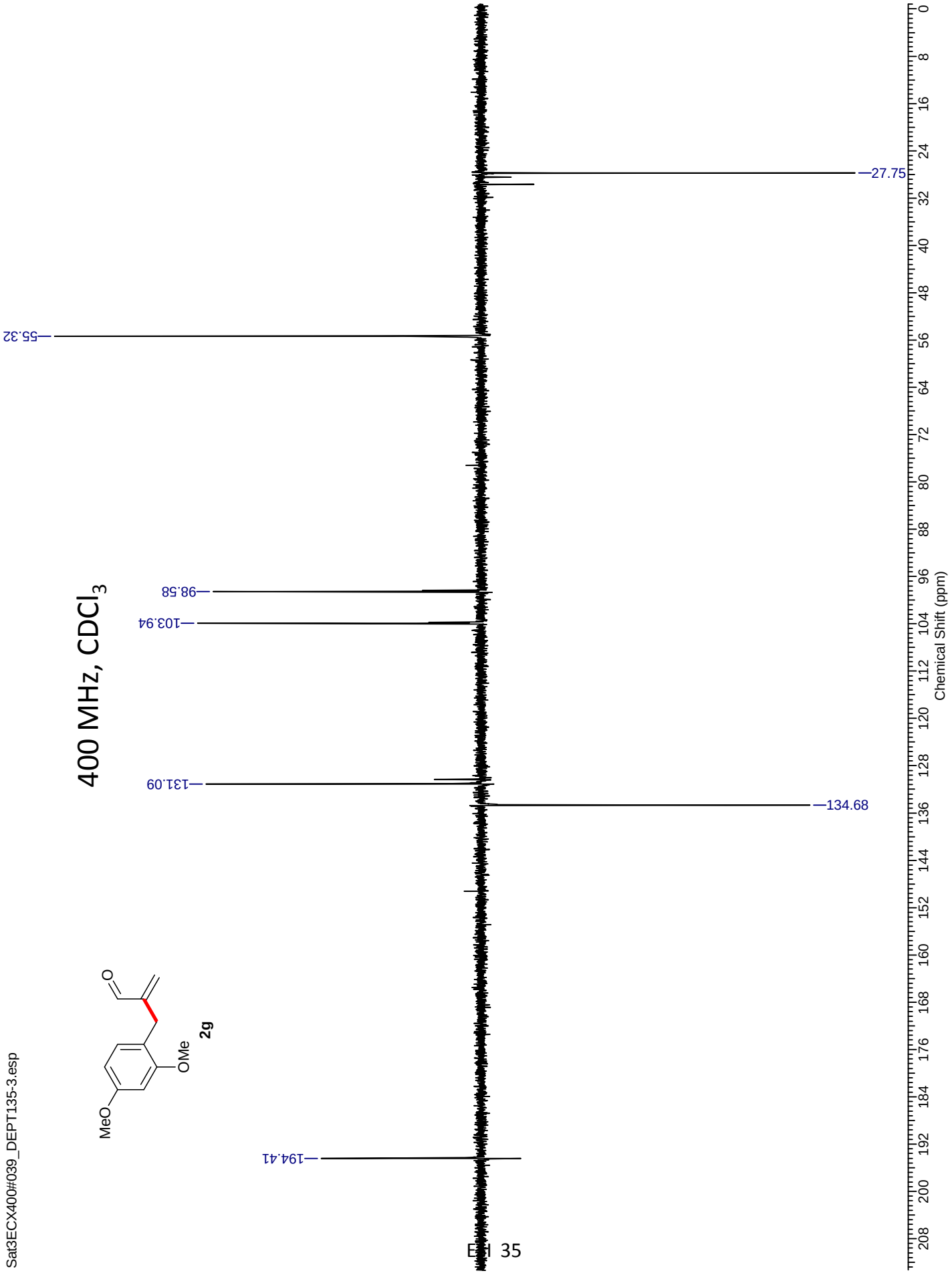
2f

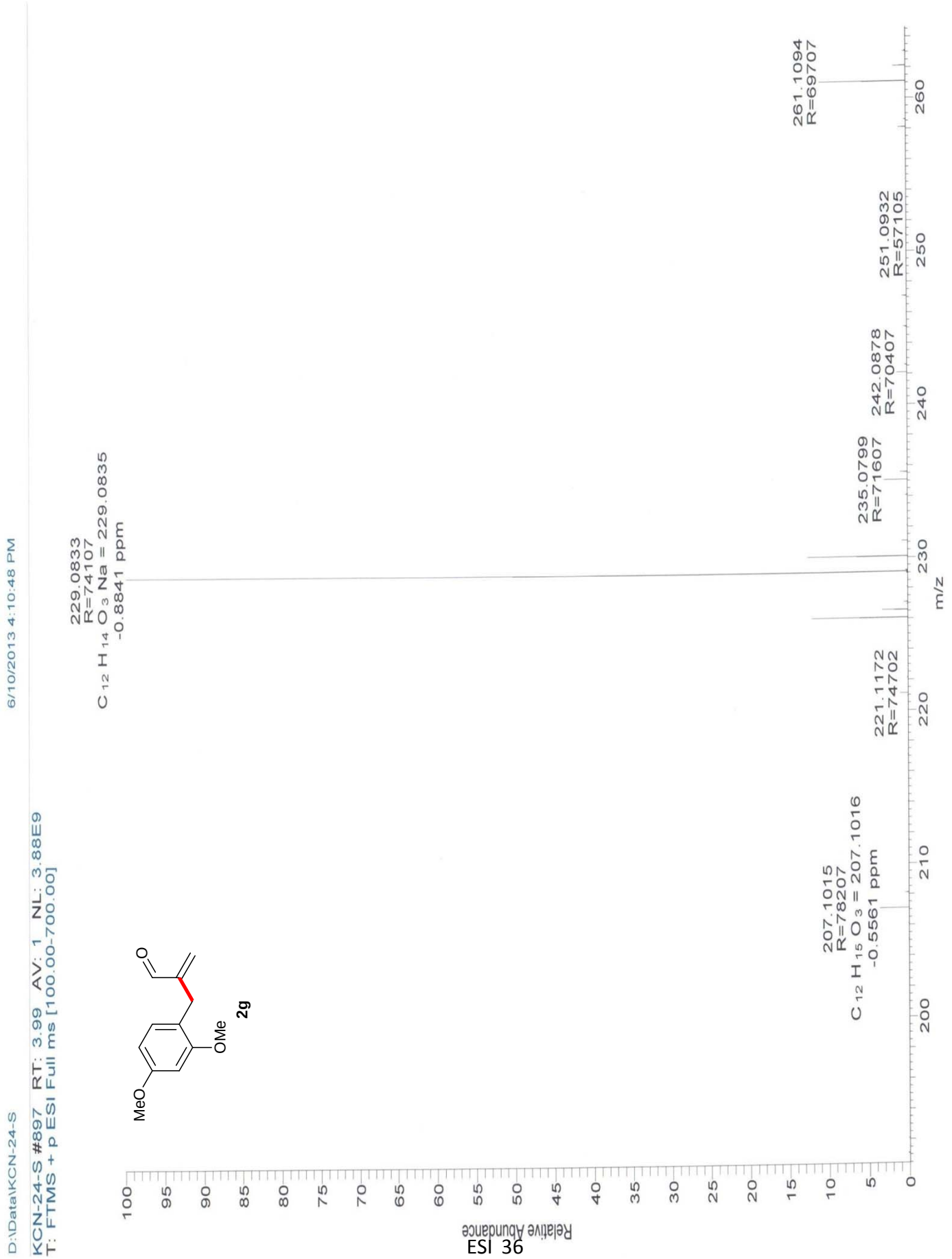


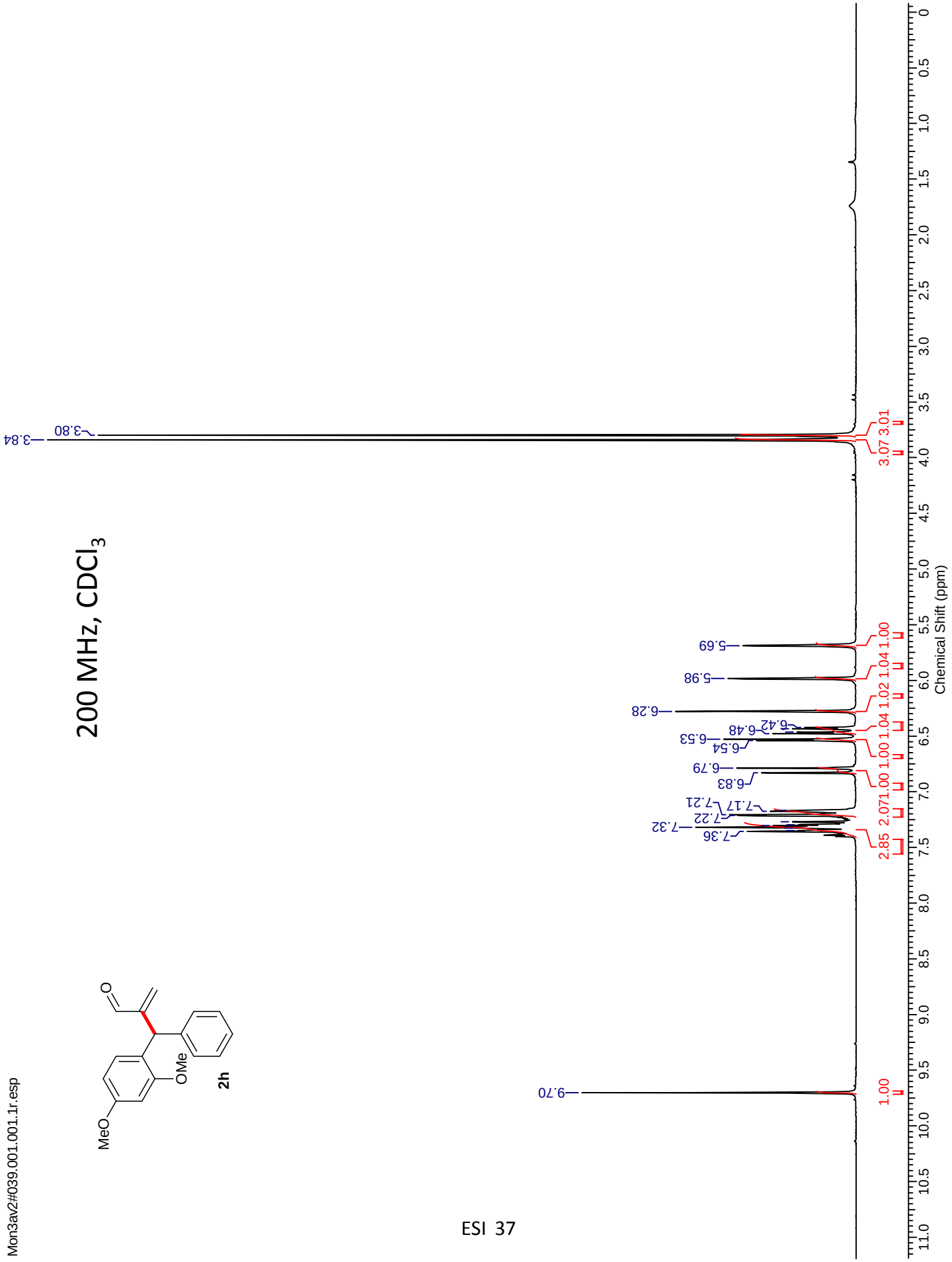
ESI 32

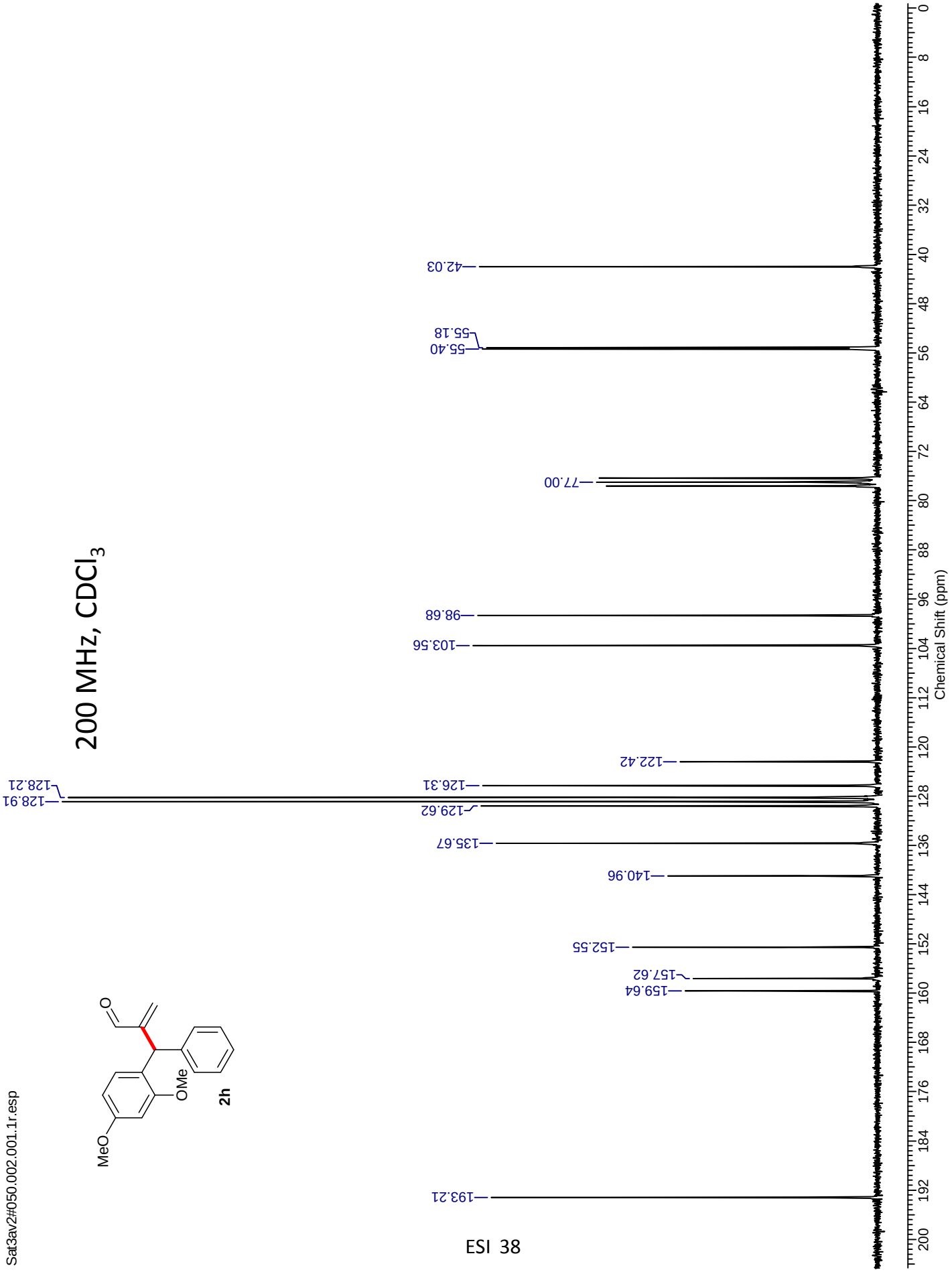

200 MHz, CDCl3
9.61
7.27
7.02
6.46
6.43
6.01
6.01
5.99
5.99
3.81
3.78
3.49
3.47
2.89
2.00
1.92
2.20
0.92
0.86
MeO
OMe
2g
Chemical Shift (ppm)
Mon3av2#047.001.001.1r.esp

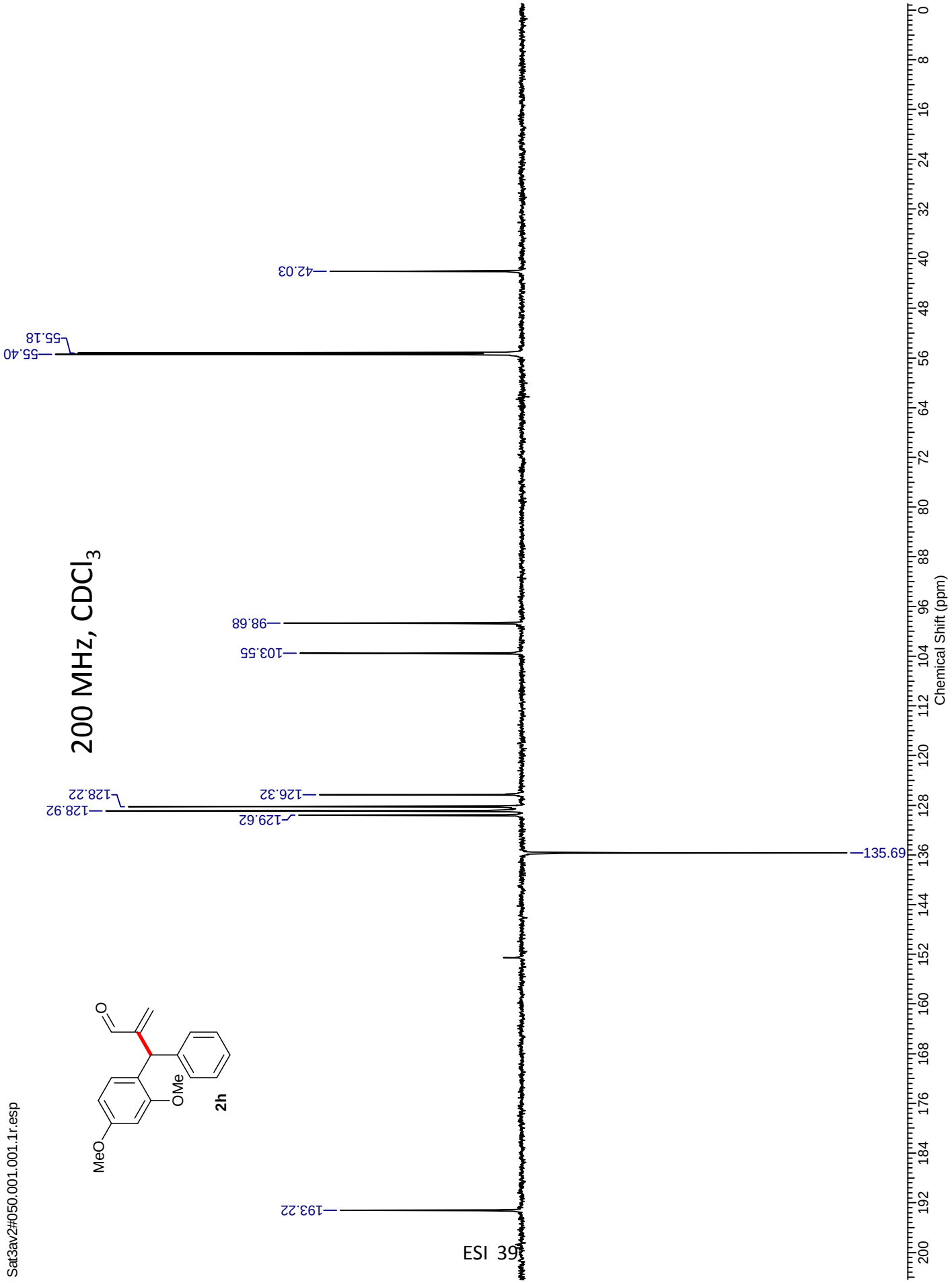

27.75
55.33
77.00
98.59
103.94
118.78
131.09
134.68
149.23
158.33
159.69
194.43
Chemical Shift (ppm)
400 MHz, CDCl3
O
OMe
MeO
2g
Sat3ECX400#039_CARBON-3.esp

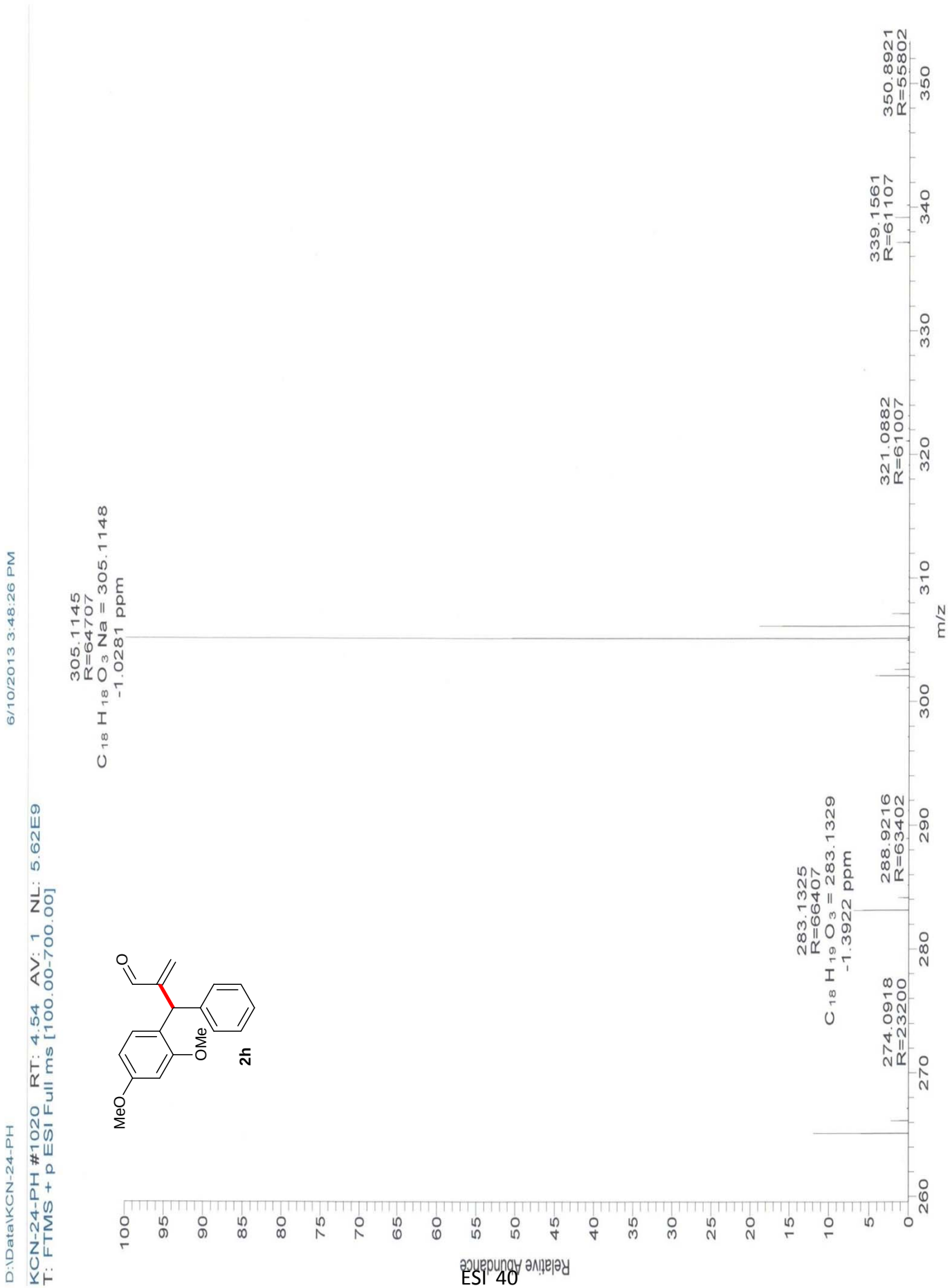

400 MHz, CDCl3
27.75
55.32
98.58
103.94
131.09
134.68
194.41
Chemical Shift (ppm)
0
8
16
24
32
40
48
56
64
72
80
88
96
104
112
120
128
136
144
152
160
168
176
184
192
200
208
O
OMe
MeO
2g
Sat3ECX400#039_DEPT135-3.esp

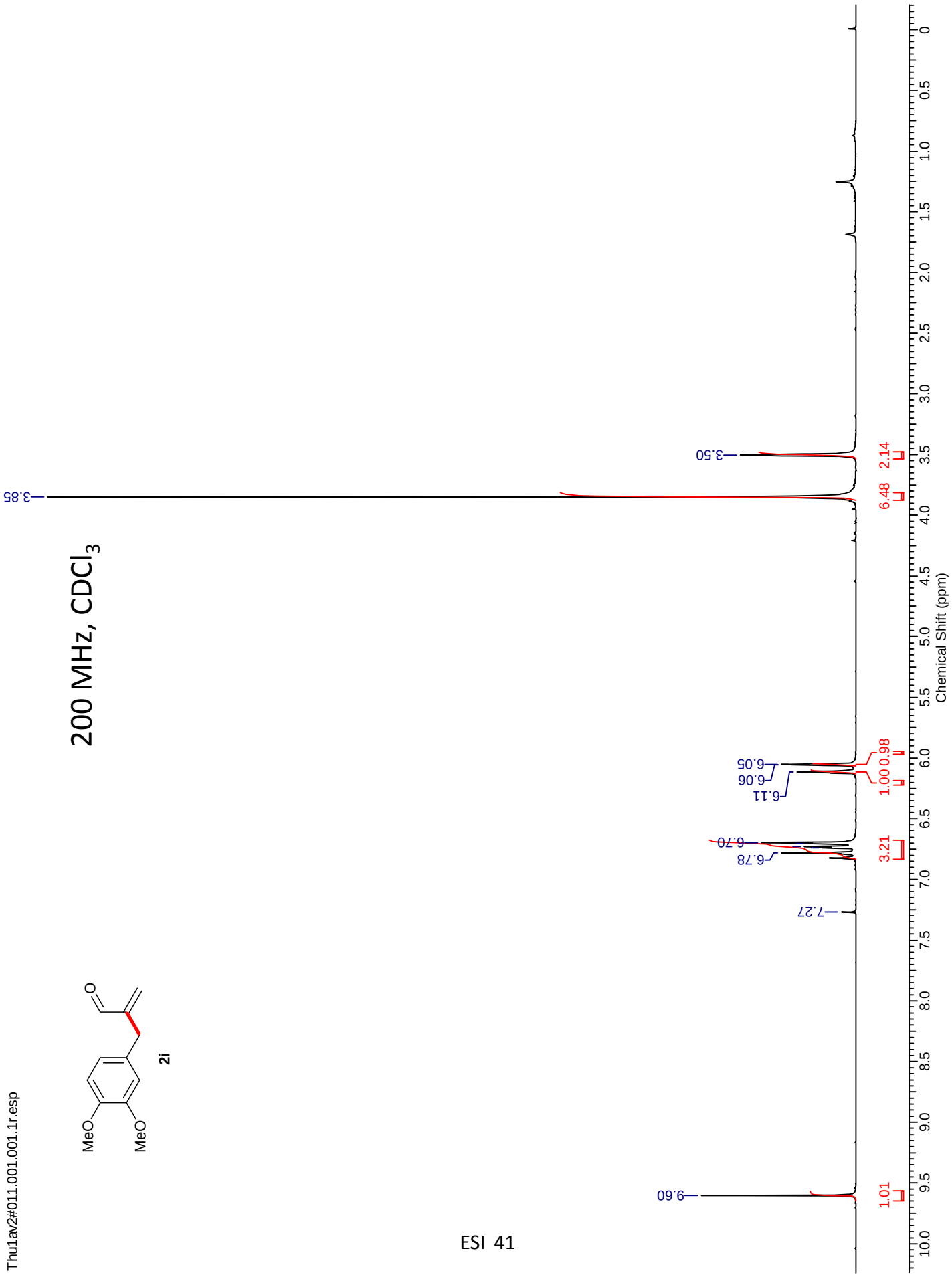

D:\Data\KCN-24-S
6/10/2013 4:10:48 PM
KCN-24-S #897 RT: 3.99 AV: 1 NL: 3.88E9
T: FTMS + p ESI Full ms [100.00-700.00]
229.0833
R=74107
C 12 H 14 O 3 Na = 229.0835
-0.8841 ppm
261.1094
R=69707
251.0932
R=57105
242.0878
R=70407
235.0799
R=71607
221.1172
R=74702
207.1015
R=78207
C 12 H 15 O 3 = 207.1016
-0.5561 ppm
MeO
OMe
O
2g
Relative Abundance
m/z

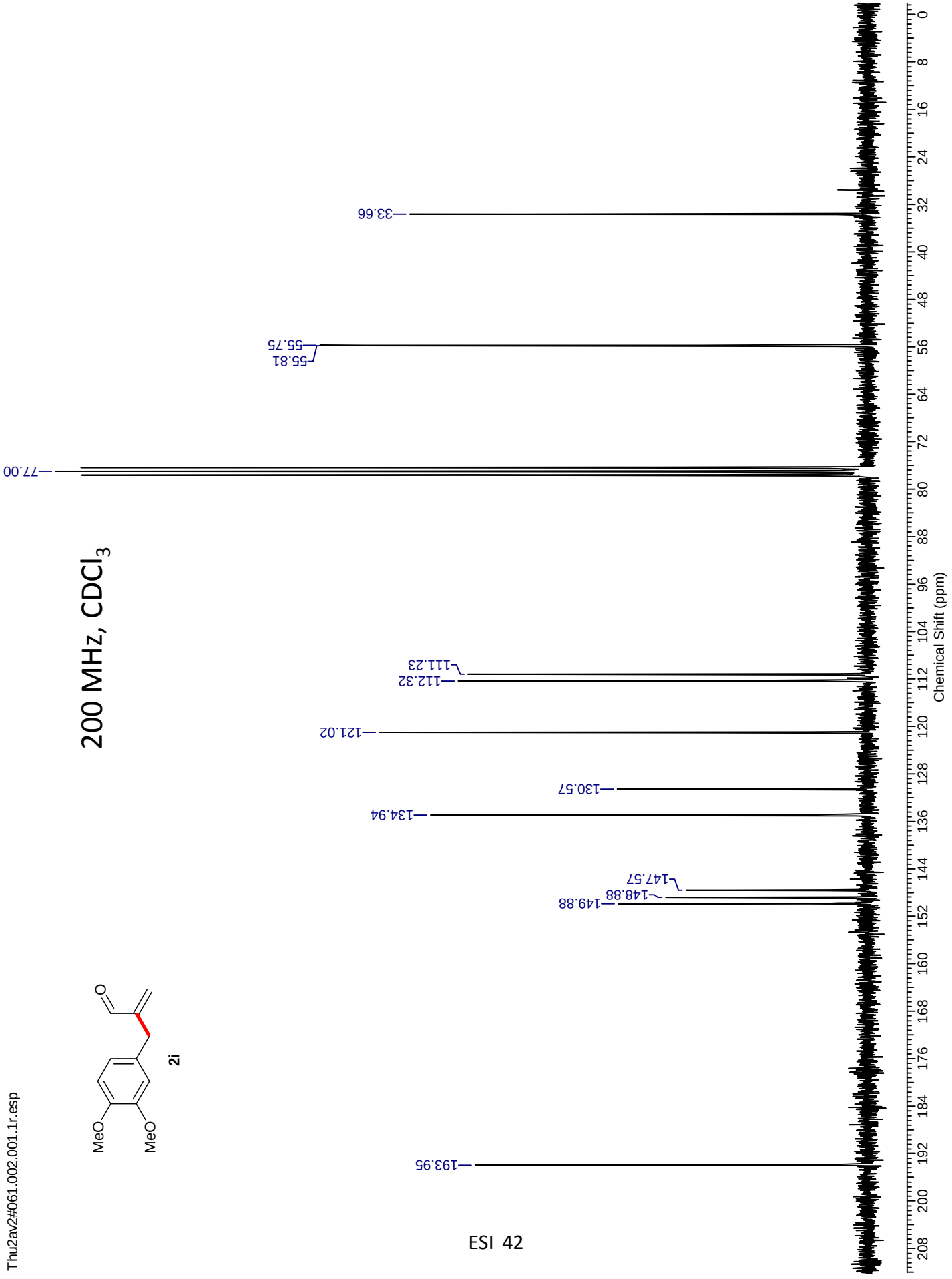

200 MHz, CDCl3
Chemical Shift (ppm)
9.70
7.36
7.32
7.22
7.21
7.17
6.83
6.79
6.54
6.53
6.48
6.42
6.28
5.98
5.69
3.84
3.80
1.00
2.85
2.07
1.00
1.00
1.04
1.02
1.04
1.00
3.07
3.01
OMe
MeO
O
2h
Mon3av2#039.001.001.1r.esp

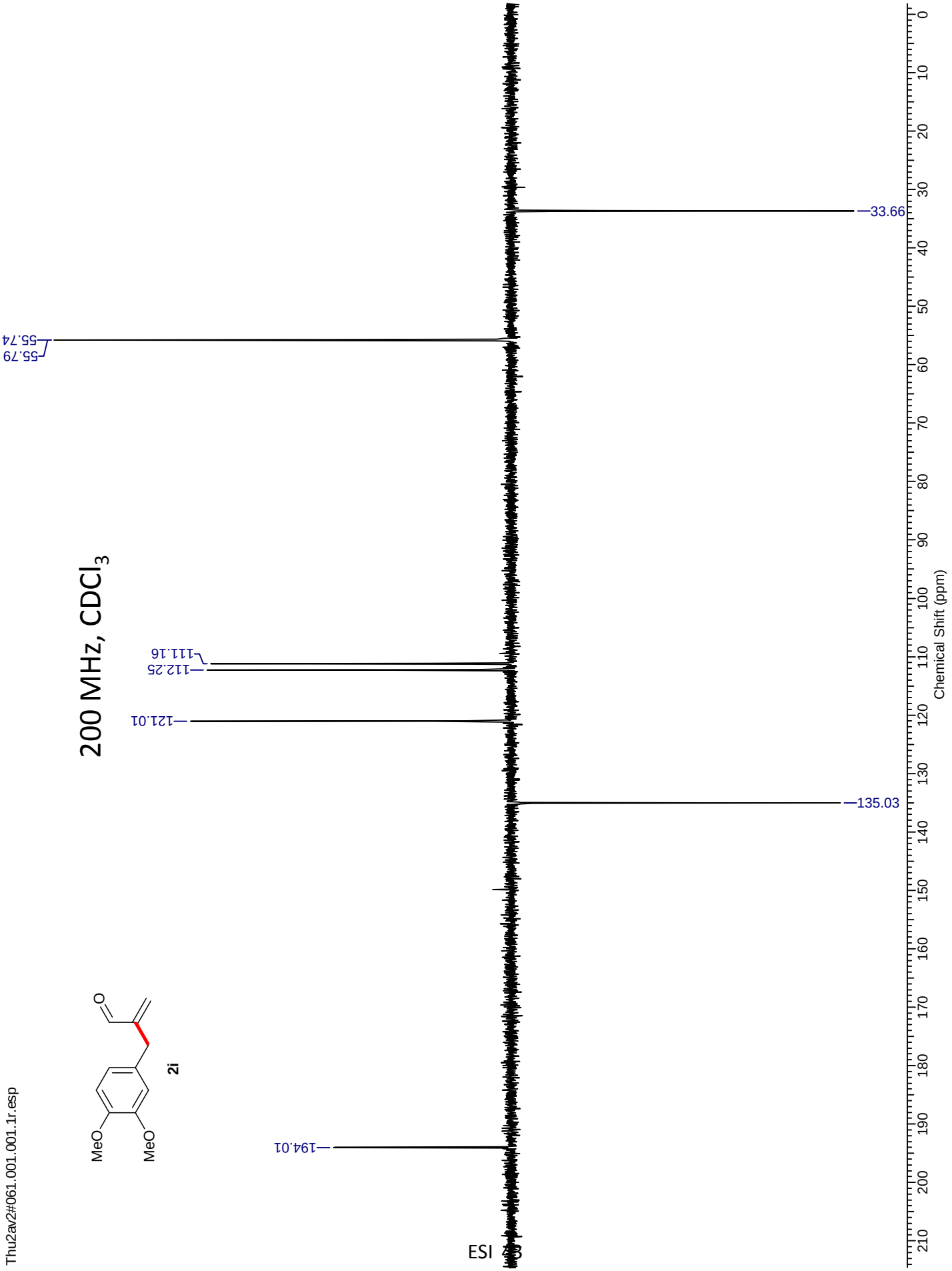

200 MHz, $CDCl_3$
42.03
55.40
55.18
77.00
98.68
103.56
122.42
126.31
128.91
128.21
129.62
135.67
140.96
152.55
157.62
159.64
193.21
O
OMe
MeO
2h
Chemical Shift (ppm)
Sat3av2#050.002.001.1r.esp

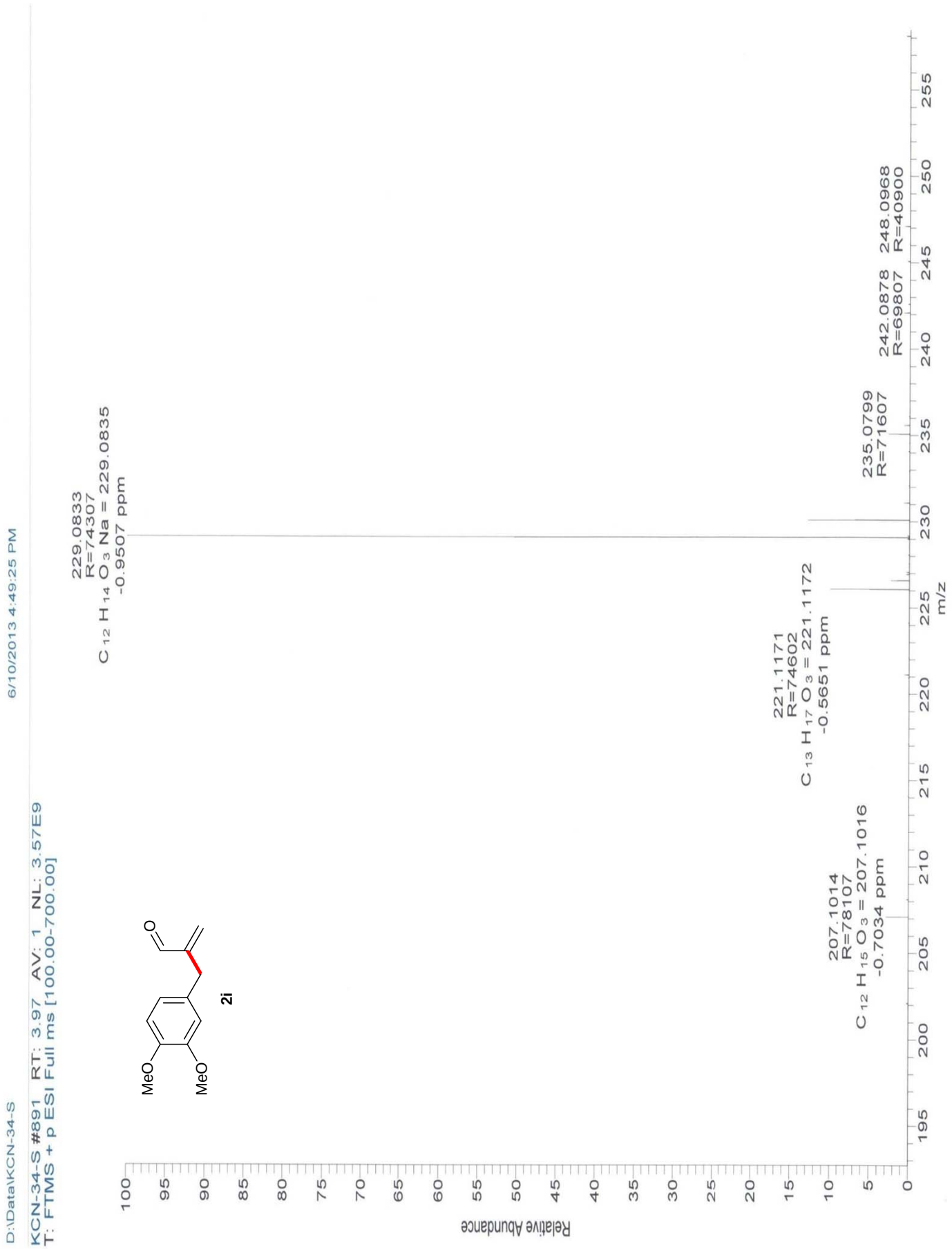

200 MHz, CDCl3
42.03
55.40
55.18
98.68
103.55
126.32
128.22
128.92
129.62
135.69
193.22
ESI 39
Chemical Shift (ppm)
0
8
16
24
32
40
48
56
64
72
80
88
96
104
112
120
128
136
144
152
160
168
176
184
192
200
O
OMe
MeO
2h
Sat3av2#050.001.001.1r.esp

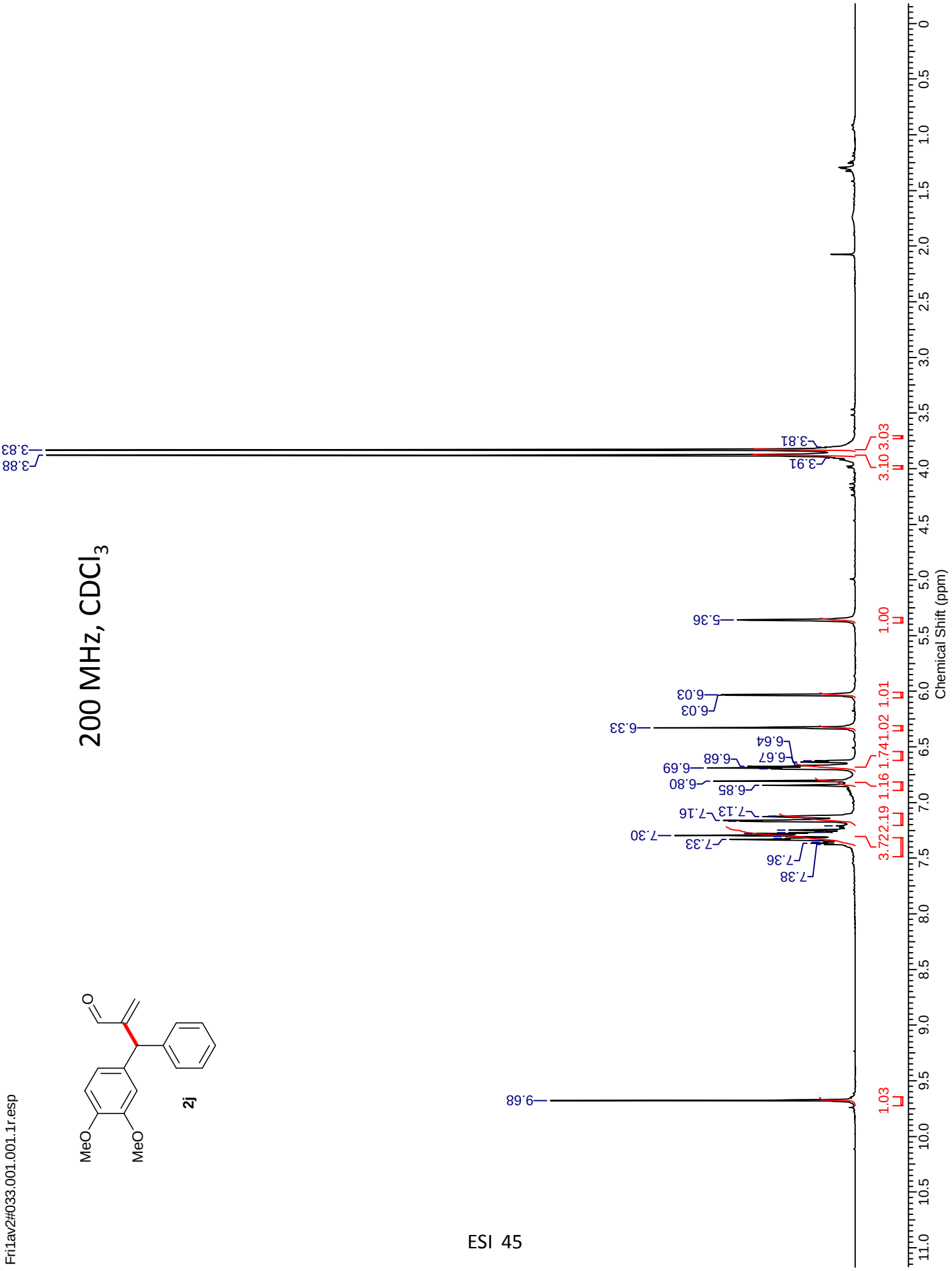

D:\Data\KCN-24-PH
6/10/2013 3:48:26 PM
KCN-24-PH #1020 RT: 4.54 AV: 1 NL: 5.62E9
T: FTMS + p ESI Full ms [100.00-700.00]
305.1145
R=64707
C 18 H 18 O 3 Na = 305.1148
-1.0281 ppm
321.0882
R=61007
339.1561
R=61107
350.8921
R=55802
283.1325
R=66407
C 18 H 19 O 3 = 283.1329
-1.3922 ppm
288.9216
R=63402
274.0918
R=23200
MeO
OMe
O
2h
Relative Abundance
m/z

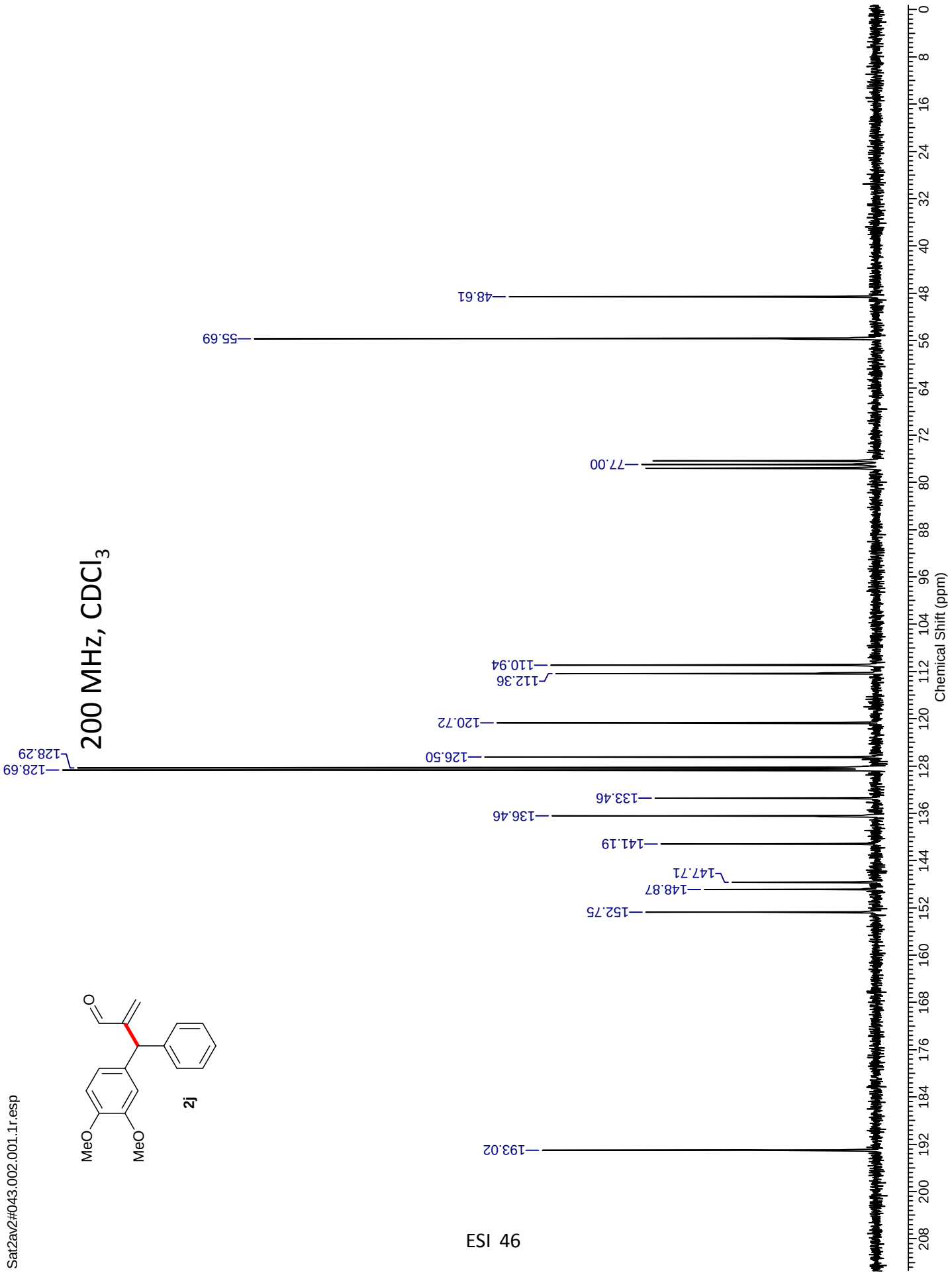

200 MHz, CDCl3
Thu1av2#011.001.001.1r.esp
2i
MeO
MeO
O
9.60
7.27
6.78
6.70
6.11
6.06
6.05
3.85
3.50
1.01
3.21
1.00
0.98
6.48
2.14
Chemical Shift (ppm)

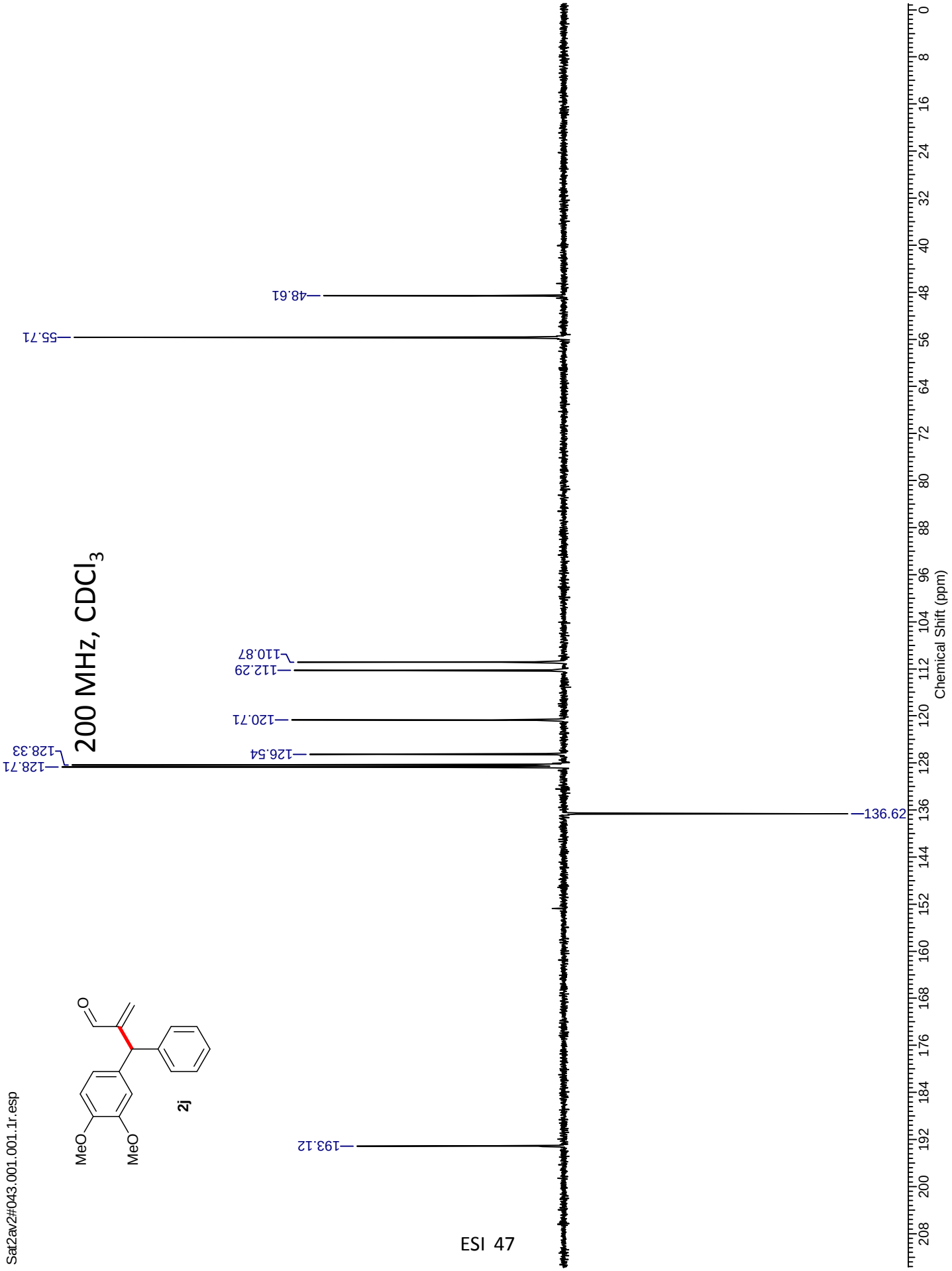

200 MHz, CDCl3
33.66
55.81
55.75
77.00
111.23
112.32
121.02
130.57
134.94
147.57
148.88
149.88
193.95
O
MeO
MeO
2i
Chemical Shift (ppm)
0
8
16
24
32
40
48
56
64
72
80
88
96
104
112
120
128
136
144
152
160
168
176
184
192
200
208
Thu2av2#061.002.001.1r.esp


200 MHz, CDCl3
33.66
55.79
55.74
111.16
112.25
121.01
135.03
194.01
MeO
MeO
O
2i
Chemical Shift (ppm)
Thu2av2#061.001.001.1r.esp
ESI

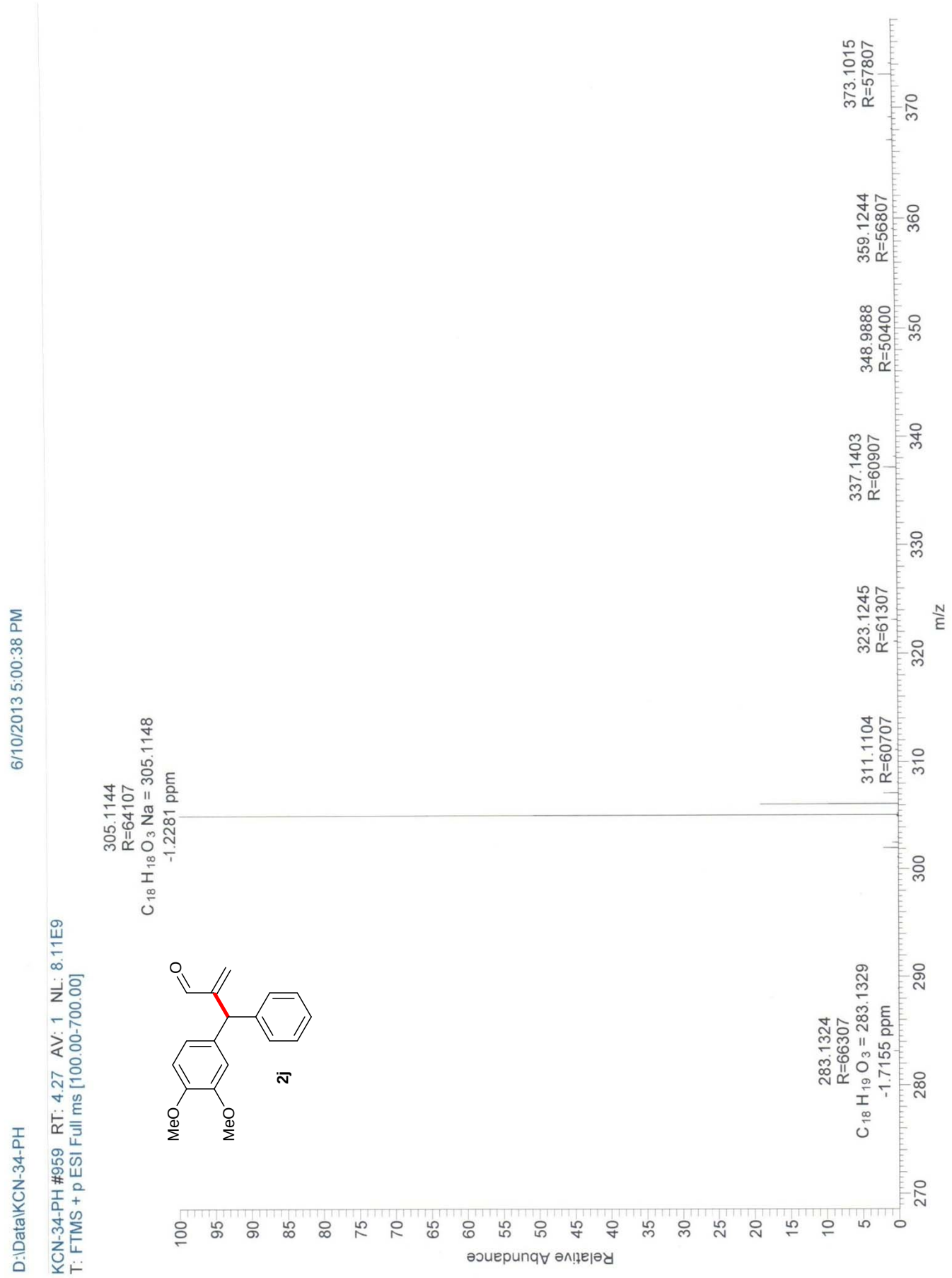

D:\Data\KCN-34-S
6/10/2013 4:49:25 PM
KCN-34-S #891 RT: 3.97 AV: 1 NL: 3.57E9
T: FTMS + p ESI Full ms [100.00-700.00]
229.0833
R=74307
C 12 H 14 O 3 Na = 229.0835
-0.9507 ppm
235.0799
R=71607
242.0878
R=69807
248.0968
R=40900
221.1171
R=74602
C 13 H 17 O 3 = 221.1172
-0.5651 ppm
207.1014
R=78107
C 12 H 15 O 3 = 207.1016
-0.7034 ppm
MeO
MeO
O
2i
Relative Abundance
m/z

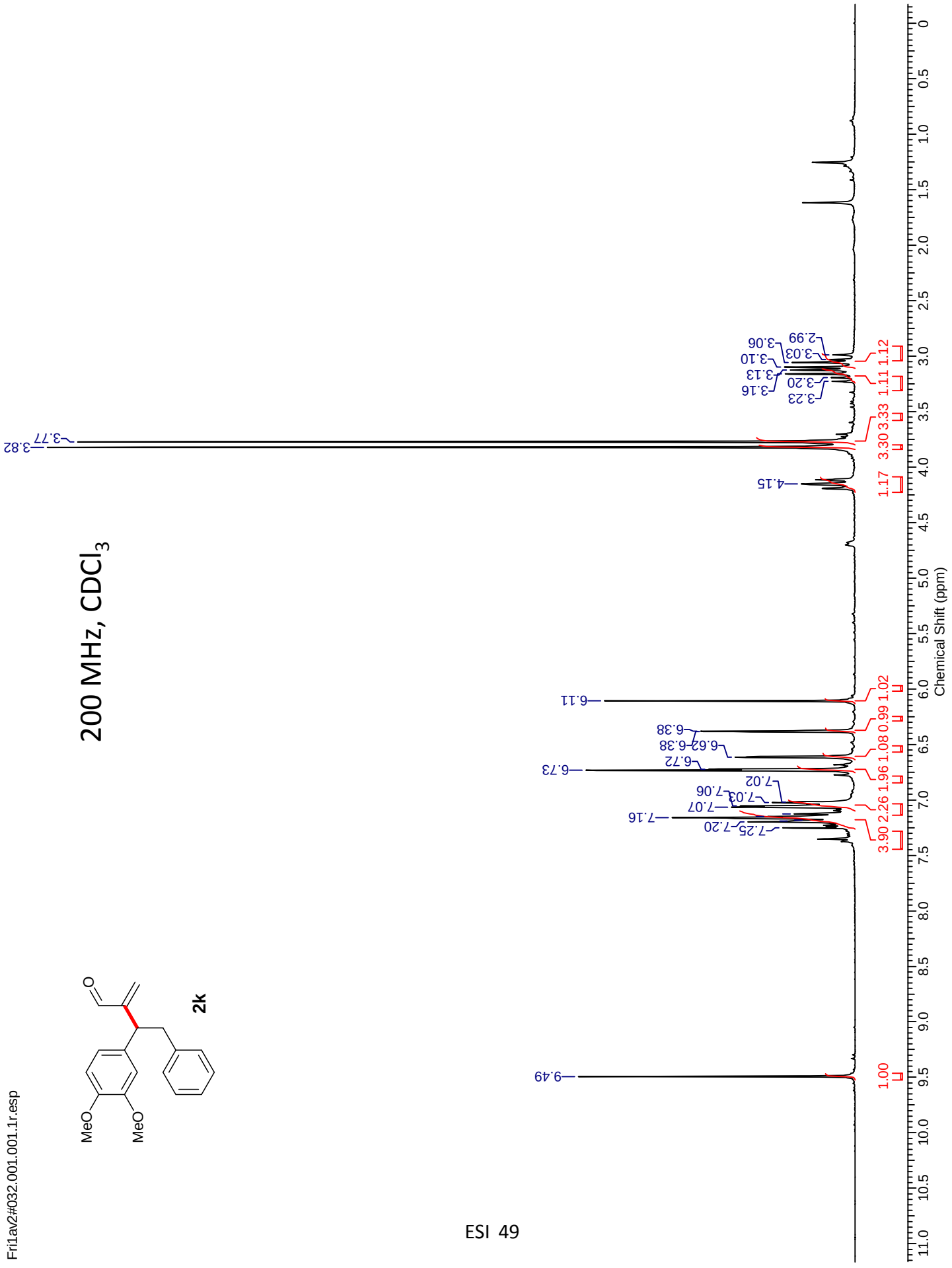

200 MHz, CDCl3
2j
MeO
MeO
O
Fri1av2#033.001.001.1r.esp
Chemical Shift (ppm)
9.68
7.38
7.36
7.33
7.30
7.16
7.13
6.85
6.80
6.69
6.68
6.67
6.64
6.33
6.03
6.03
5.36
3.91
3.88
3.83
3.81
1.03
3.72
1.19
1.16
1.74
1.02
1.01
1.00
3.10
3.03

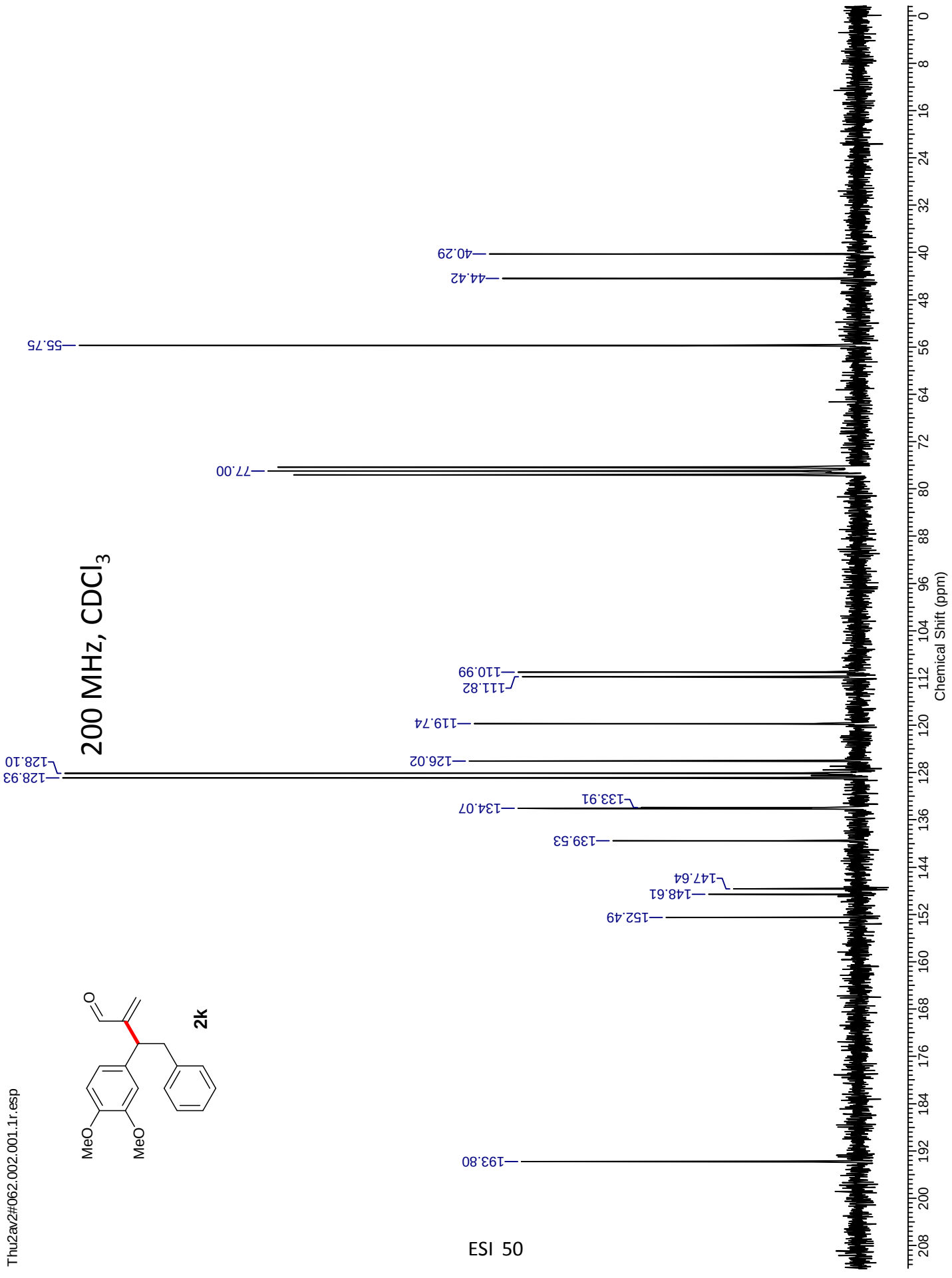

200 MHz, CDCl3
193.02
152.75
148.87
147.71
141.19
136.46
133.46
128.69
128.29
126.50
120.72
112.36
110.94
77.00
55.69
48.61
Chemical Shift (ppm)
0
8
16
24
32
40
48
56
64
72
80
88
96
104
112
120
128
136
144
152
160
168
176
184
192
200
208
O
MeO
MeO
2j
Sat2av2#043.002.001.1r.esp

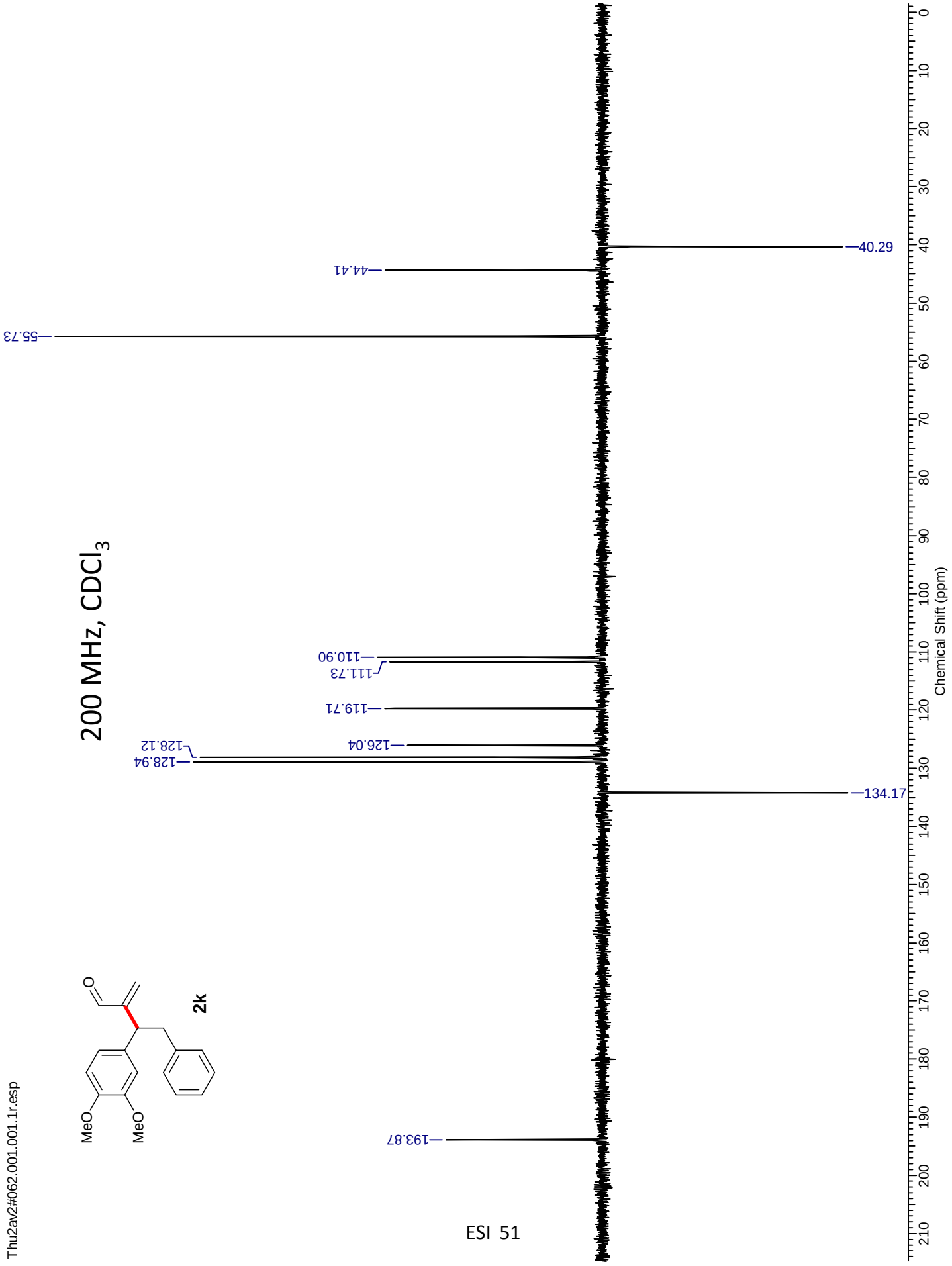

200 MHz, CDCl3
48.61
55.71
110.87
112.29
120.71
126.54
128.33
128.71
136.62
193.12
Chemical Shift (ppm)
0
8
16
24
32
40
48
56
64
72
80
88
96
104
112
120
128
136
144
152
160
168
176
184
192
200
208
O
MeO
MeO
2j
Sat2av2#043.001.001.1r.esp

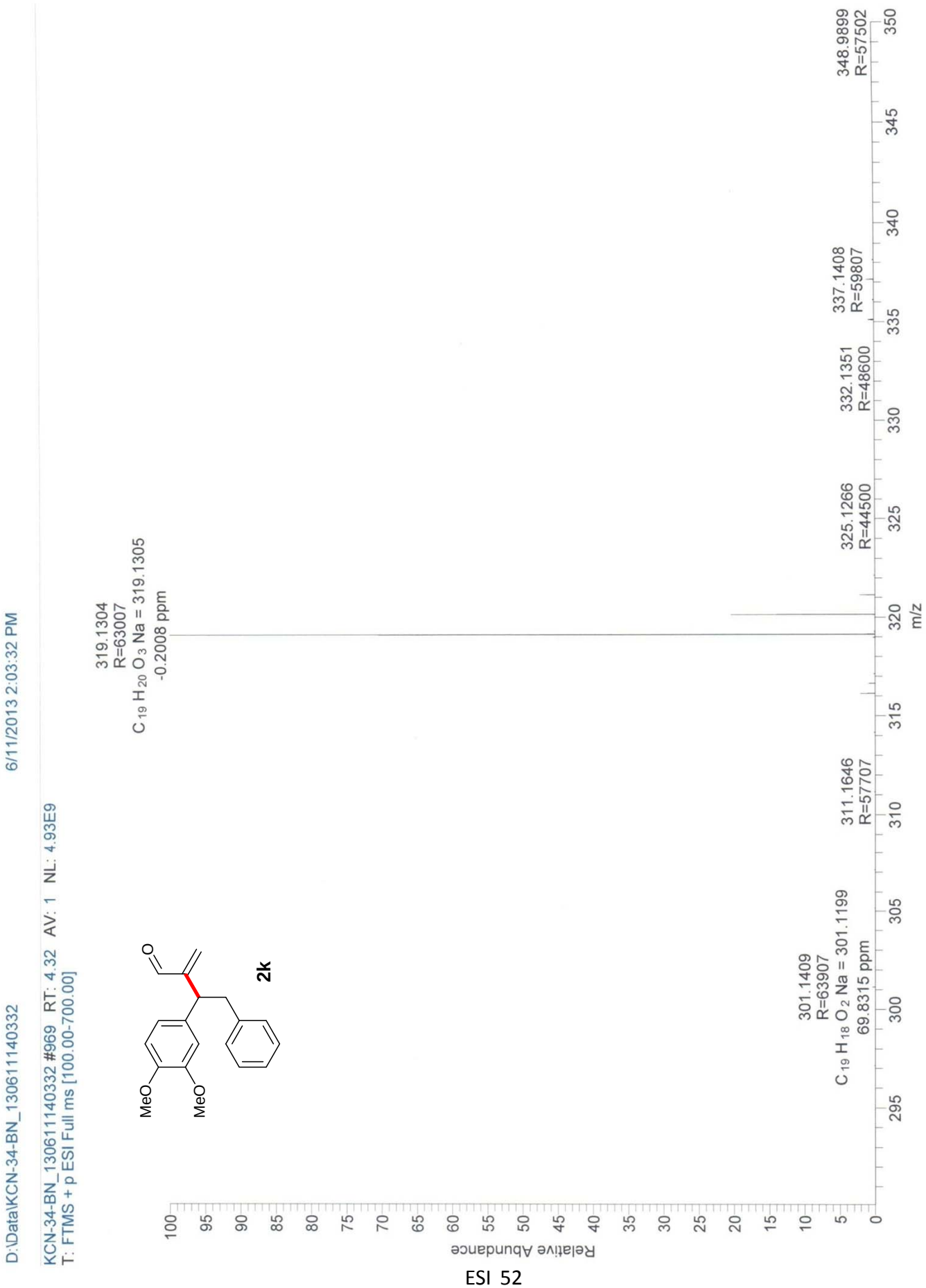
ESI 47

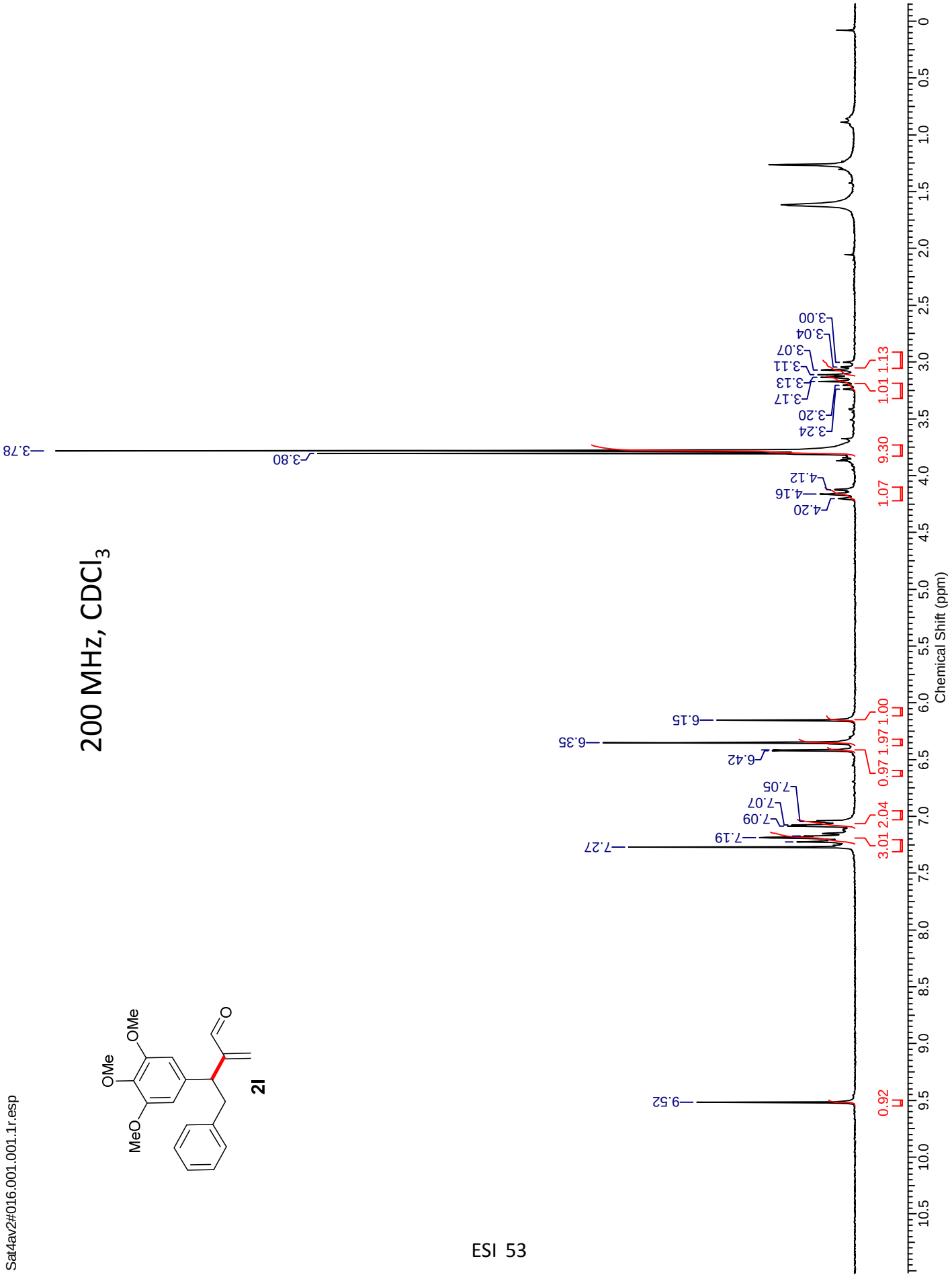

D:\Data\KCN-34-PH
6/10/2013 5:00:38 PM
KCN-34-PH #959 RT: 4.27 AV: 1 NL: 8.11E9
T: FTMS + p ESI Full ms [100.00-700.00]
305.1144
R=64107
C18 H18 O3 Na = 305.1148
-1.2281 ppm
283.1324
R=66307
C18 H19 O3 = 283.1329
-1.7155 ppm
311.1104
R=60707
323.1245
R=61307
337.1403
R=60907
348.9888
R=50400
359.1244
R=56807
373.1015
R=57807
MeO
MeO
O
2j
Relative Abundance
m/z

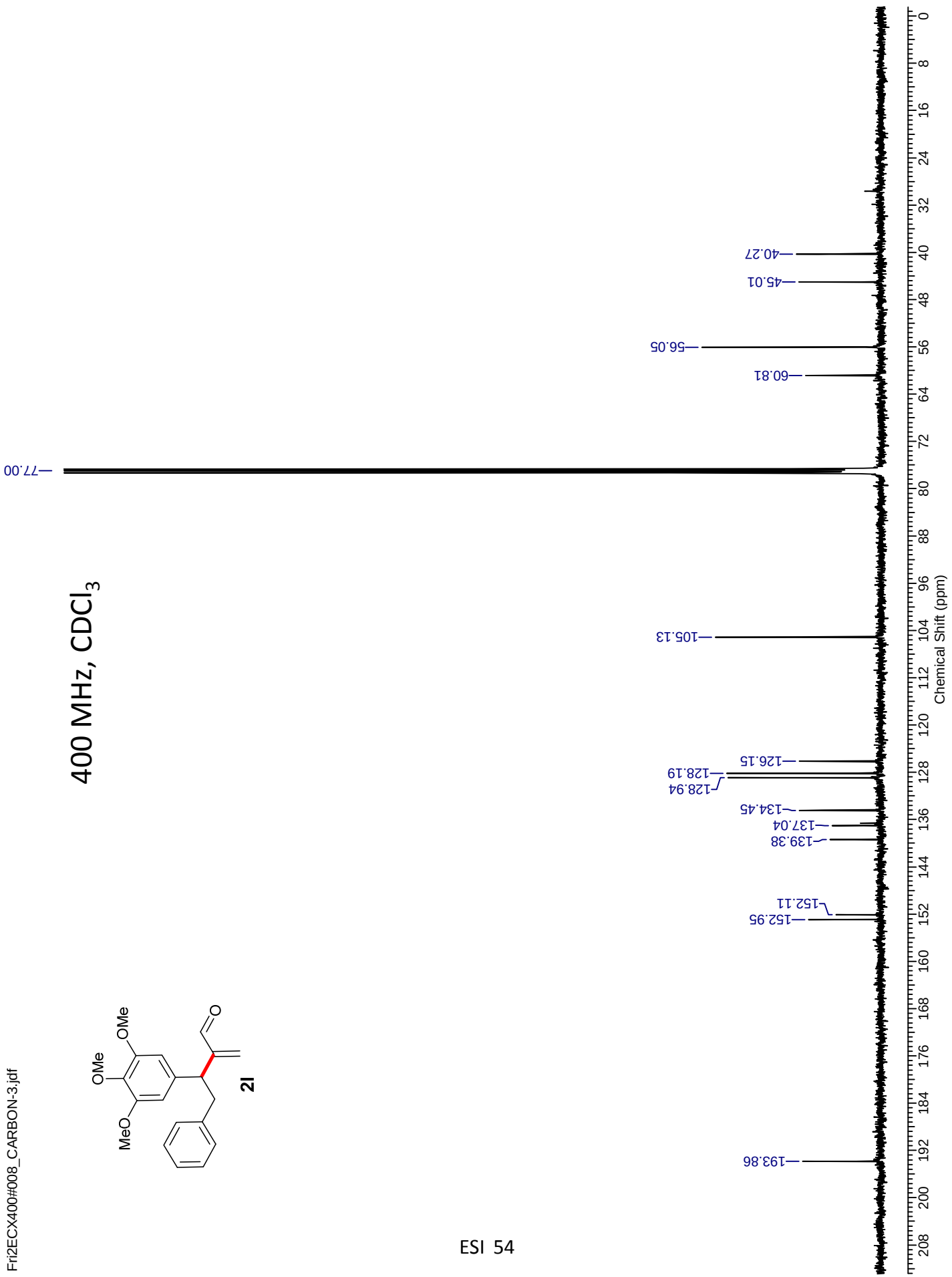

200 MHz, CDCl3
2k
MeO
MeO
O
9.49
7.25
7.20
7.16
7.07
7.03
7.06
7.02
6.73
6.72
6.62
6.38
6.38
6.11
4.15
3.82
3.77
3.23
3.20
3.16
3.13
3.10
3.06
2.99
1.00
3.90
2.26
1.96
1.08
0.99
1.02
1.17
3.30
3.33
1.11
1.12
Chemical Shift (ppm)
Fri1av2#032.001.001.1r.esp

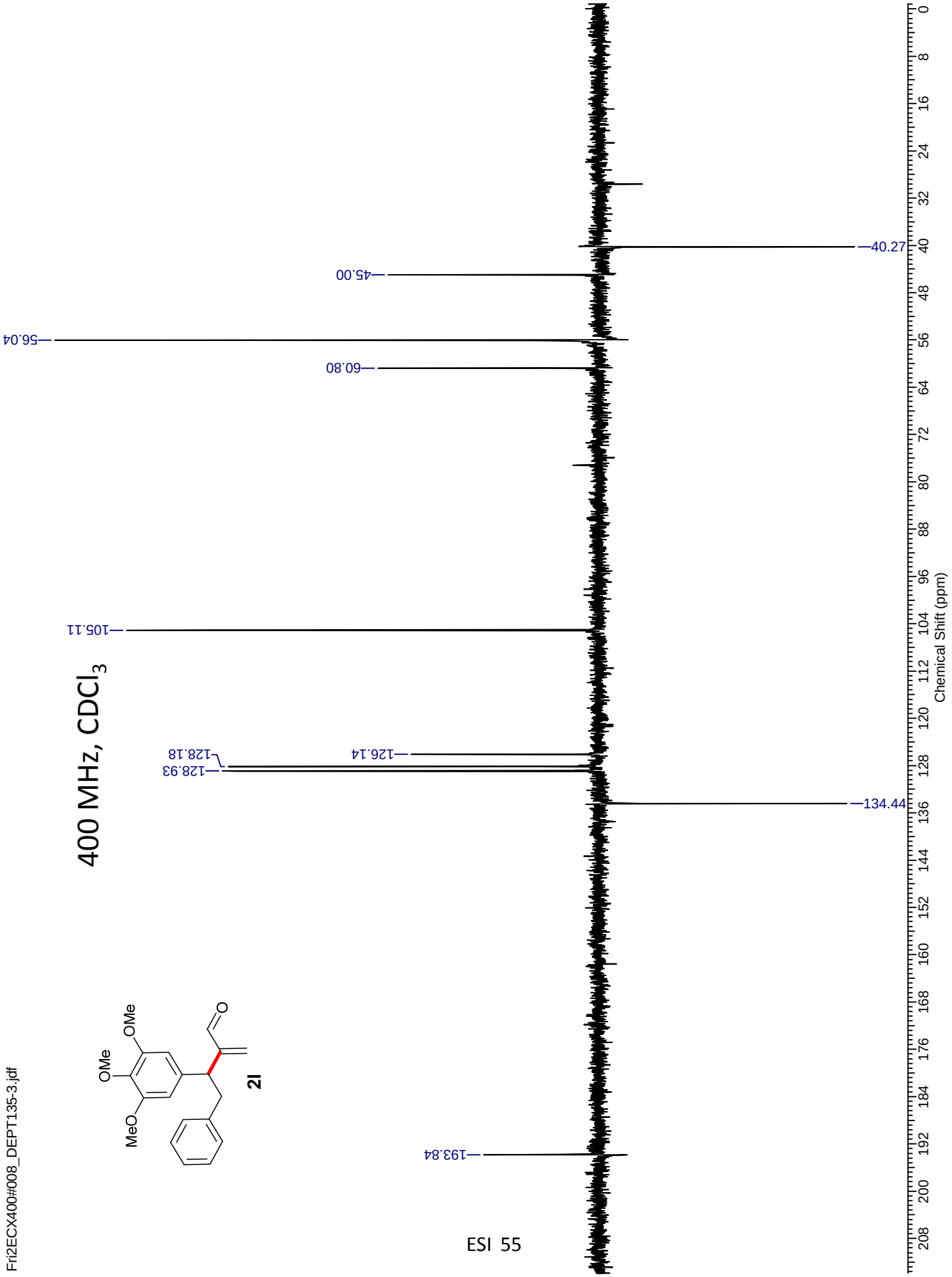


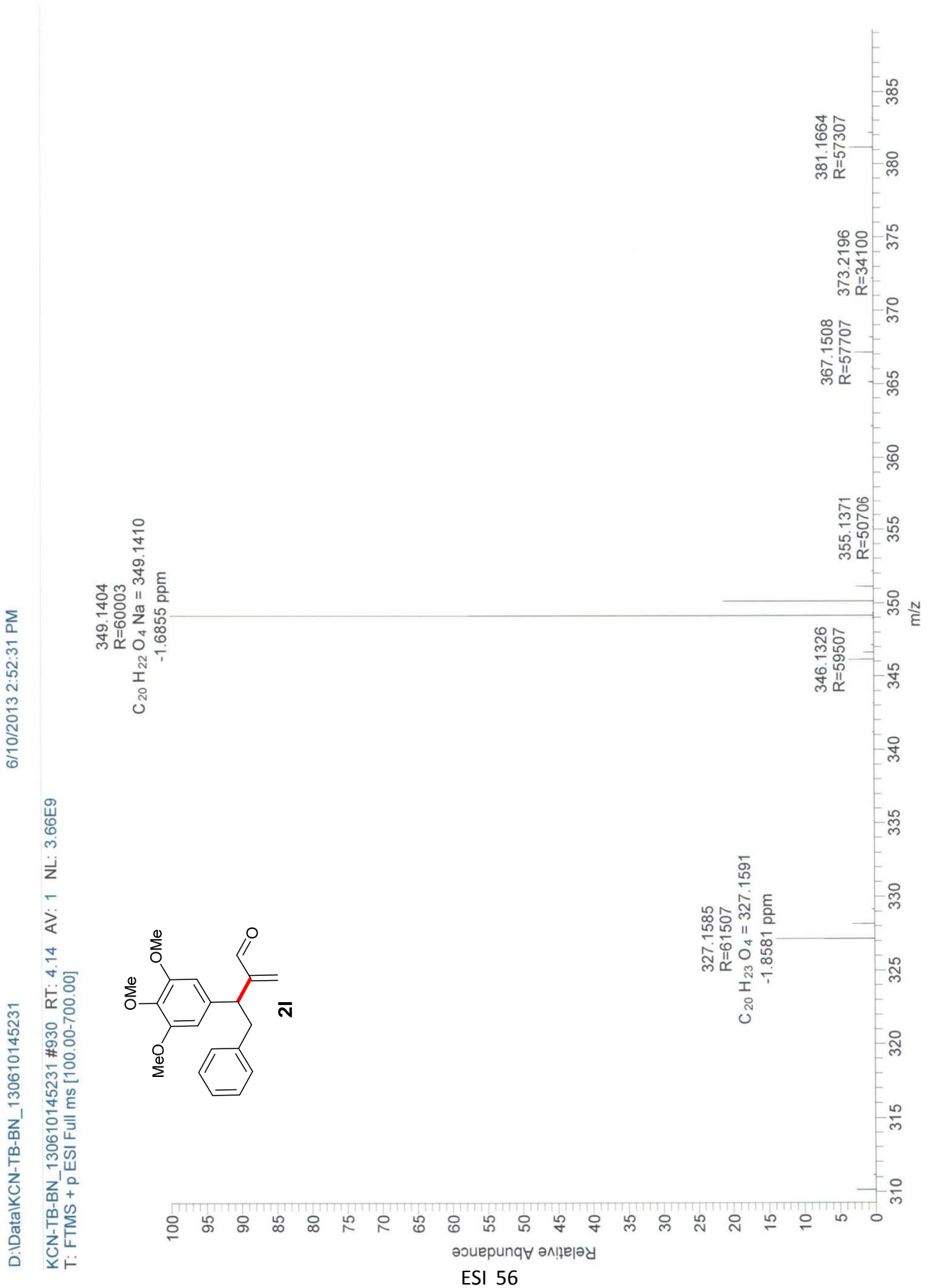

200 MHz, $CDCl_3$
40.29
44.41
55.73
110.90
111.73
119.71
126.04
128.12
128.94
134.17
193.87
Chemical Shift (ppm)
0
10
20
30
40
50
60
70
80
90
100
110
120
130
140
150
160
170
180
190
200
210
O
MeO
MeO
2k
Thu2av2#062.001.001.1r.esp
ESI 51

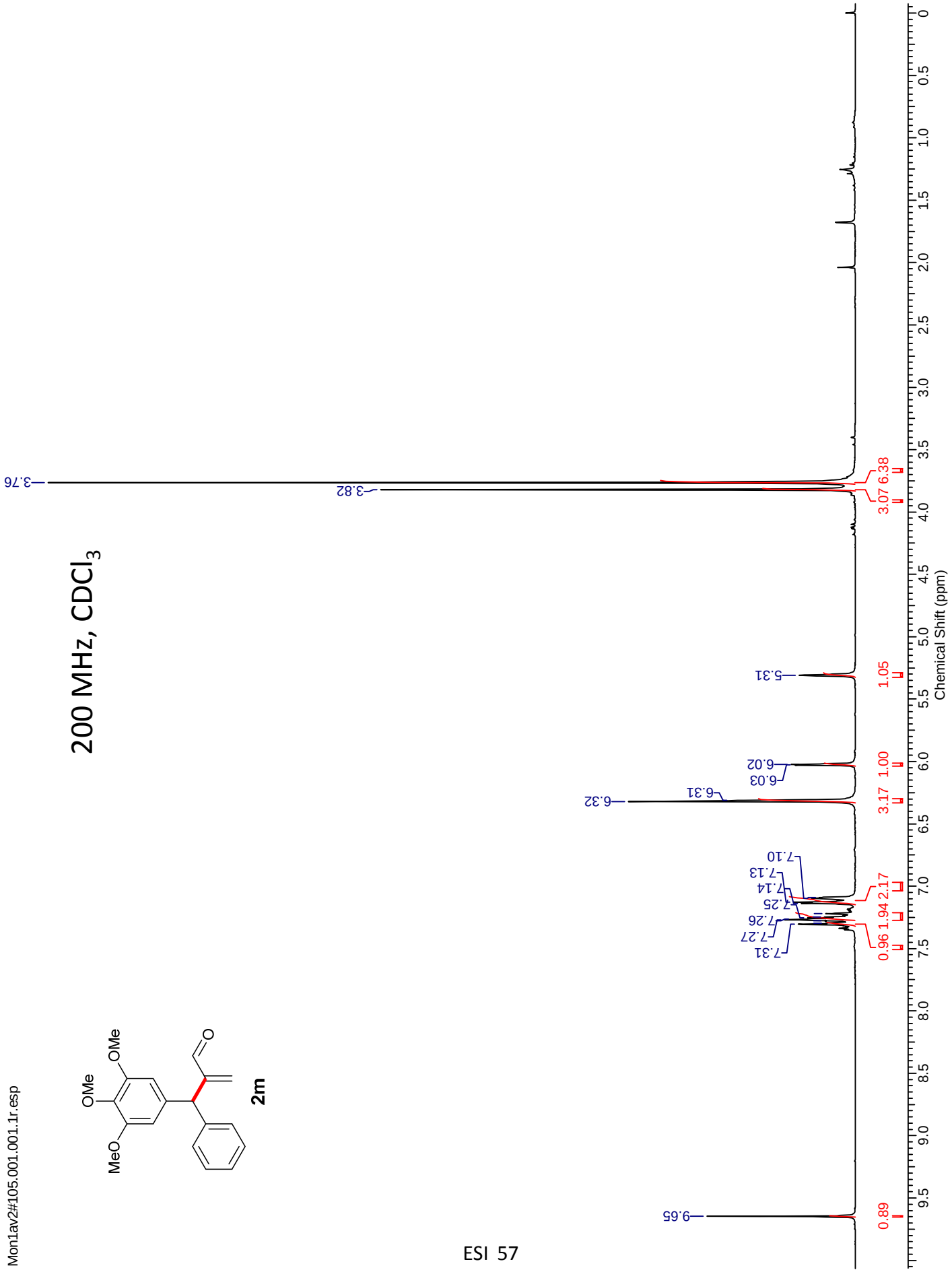

D:\Data\KCN-34-BN_130611140332
6/11/2013 2:03:32 PM
KCN-34-BN_130611140332 #969 RT: 4.32 AV: 1 NL: 4.93E9
T: FTMS + p ESI Full ms [100.00-700.00]
319.1304
R=63007
C 19 H 20 O 3 Na = 319.1305
-0.2008 ppm
301.1409
R=63907
C 19 H 18 O 2 Na = 301.1199
69.8315 ppm
311.1646
R=57707
325.1266
R=44500
332.1351
R=48600
337.1408
R=59807
348.9899
R=57502
2k
MeO
MeO
O
Relative Abundance
m/z

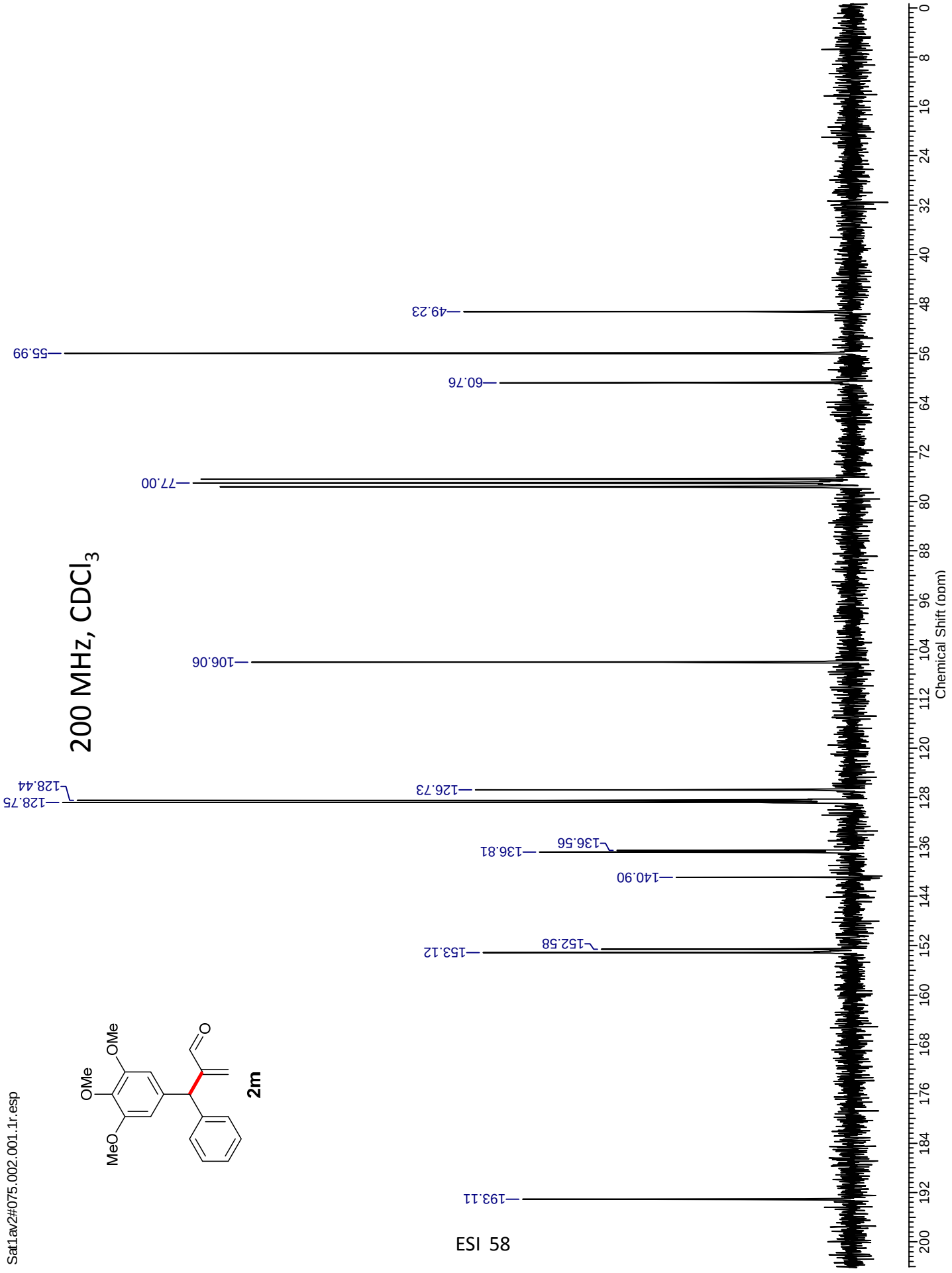

200 MHz, CDCl3
Chemical Shift (ppm)
9.52
7.27
7.19
7.09
7.07
7.05
6.42
6.35
6.15
4.20
4.16
4.12
3.80
3.78
3.24
3.20
3.17
3.13
3.11
3.07
3.04
3.00
0.92
3.01
2.04
0.97
1.97
1.00
1.01
1.13
9.30
1.07
OMe
OMe
MeO
O
2l
Sat4av2#016.001.001.1r.esp


400 MHz, $CDCl_3$
193.86
152.95
152.11
139.38
137.04
134.45
128.94
128.19
126.15
105.13
77.00
60.81
56.05
45.01
40.27
Chemical Shift (ppm)
OMe
OMe
MeO
O
2l
Fri2ECX400#008_CARBON-3.jdf

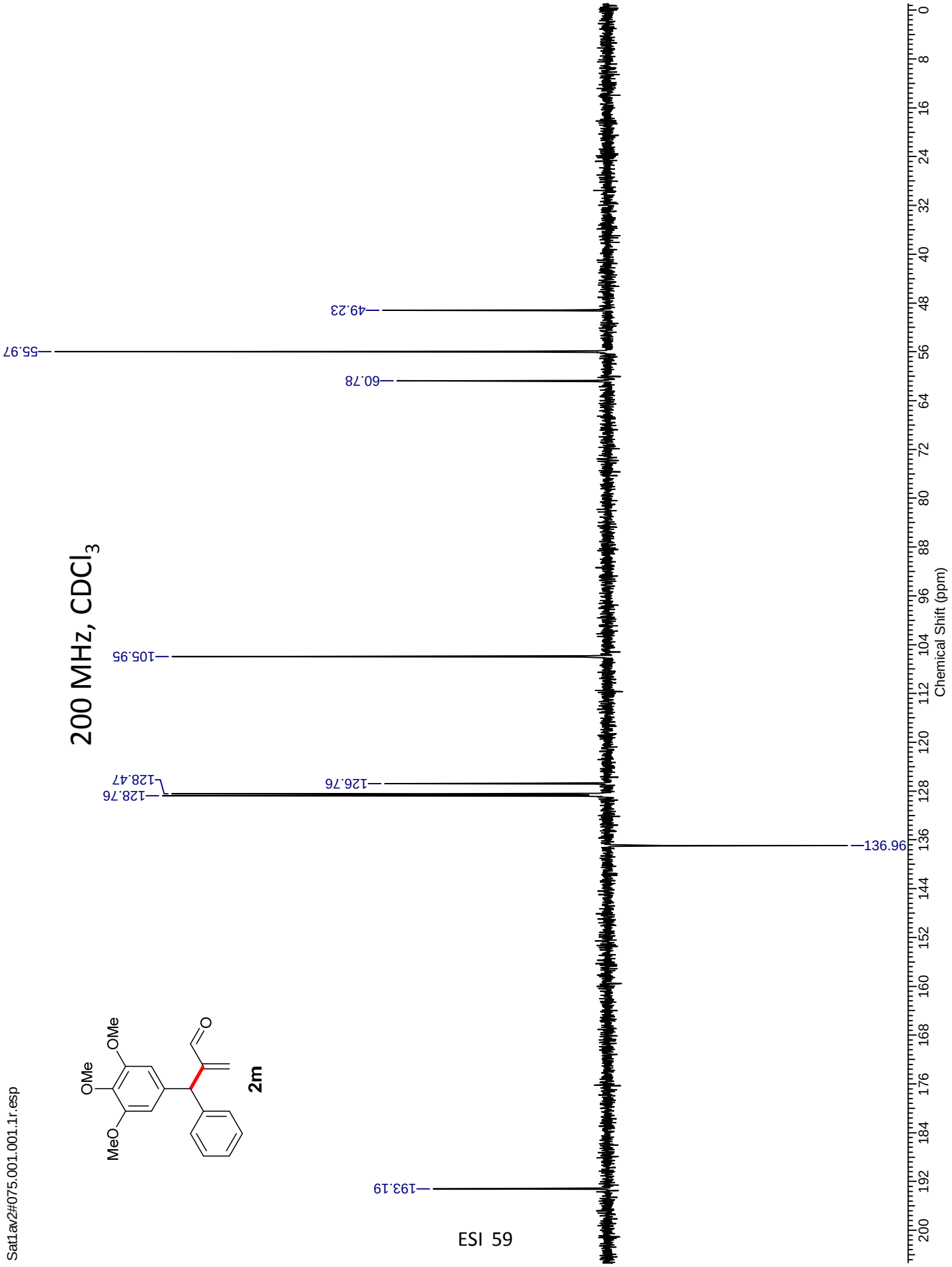

40.27
45.00
56.04
60.80
105.11
126.14
128.18
128.93
134.44
193.84
400 MHz, CDCl3
Chemical Shift (ppm)
OMe
OMe
MeO
O
2l
Fri2ECX400#008_DEPT135-3.jdf
ESI 55

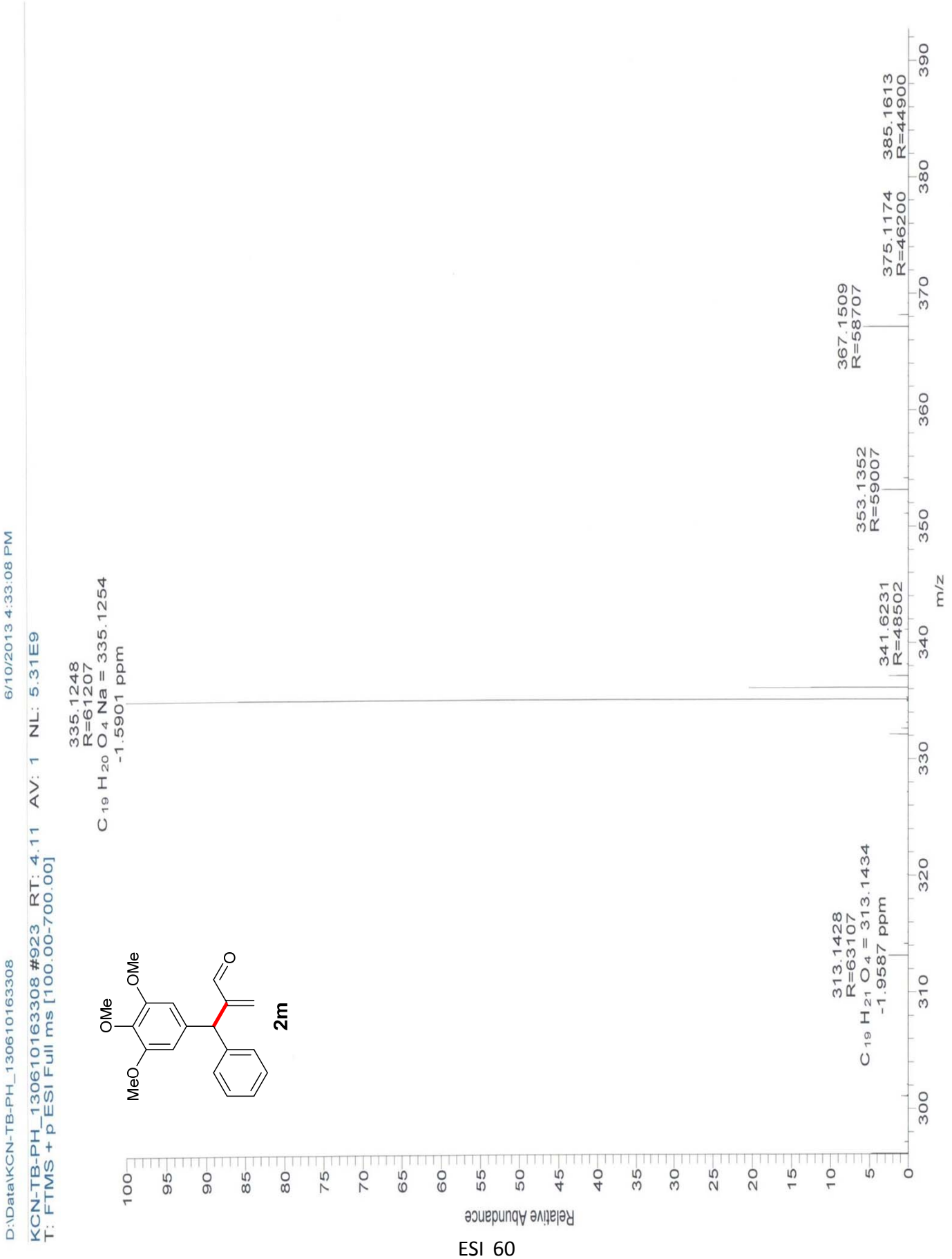

D:\Data\KCN-TB-BN_130610145231
6/10/2013 2:52:31 PM
KCN-TB-BN_130610145231 #930 RT: 4.14 AV: 1 NL: 3.66E9
T: FTMS + p ESI Full ms [100.00-700.00]
349.1404
R=60003
C 20 H 22 O 4 Na = 349.1410
-1.6855 ppm
327.1585
R=61507
C 20 H 23 O 4 = 327.1591
-1.8581 ppm
346.1326
R=59507
355.1371
R=50706
367.1508
R=57707
373.2196
R=34100
381.1664
R=57307
OMe
OMe
MeO
O
2l
Relative Abundance
m/z

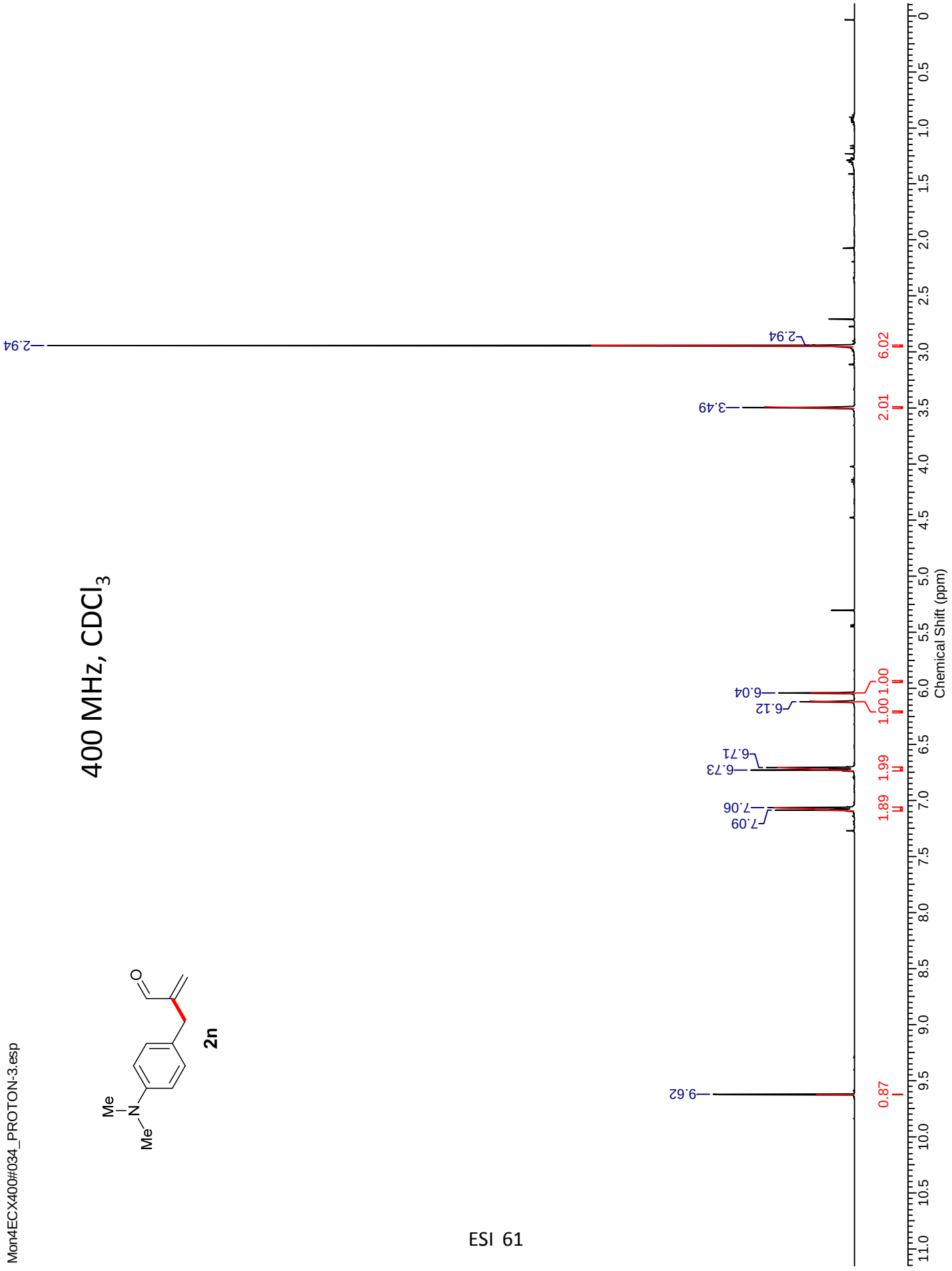

200 MHz, CDCl3
Mon1av2#105.001.001.1r.esp
OMe
OMe
MeO
O
2m
3.76
3.82
5.31
6.02
6.03
6.31
6.32
7.10
7.13
7.14
7.25
7.26
7.27
7.31
9.65
0.89
0.96
1.94
2.17
3.17
1.00
1.05
3.07
6.38
Chemical Shift (ppm)

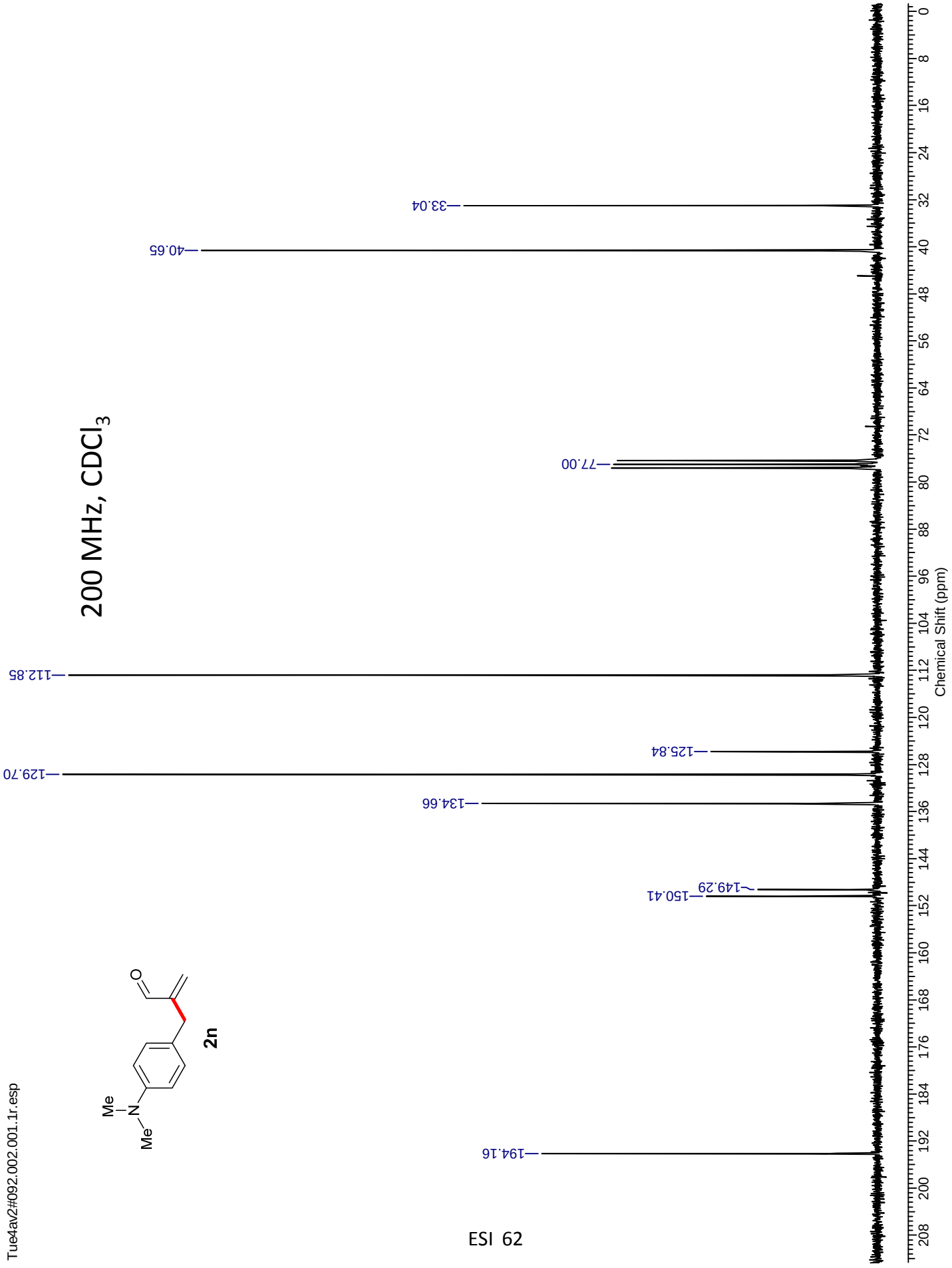


ESI 58

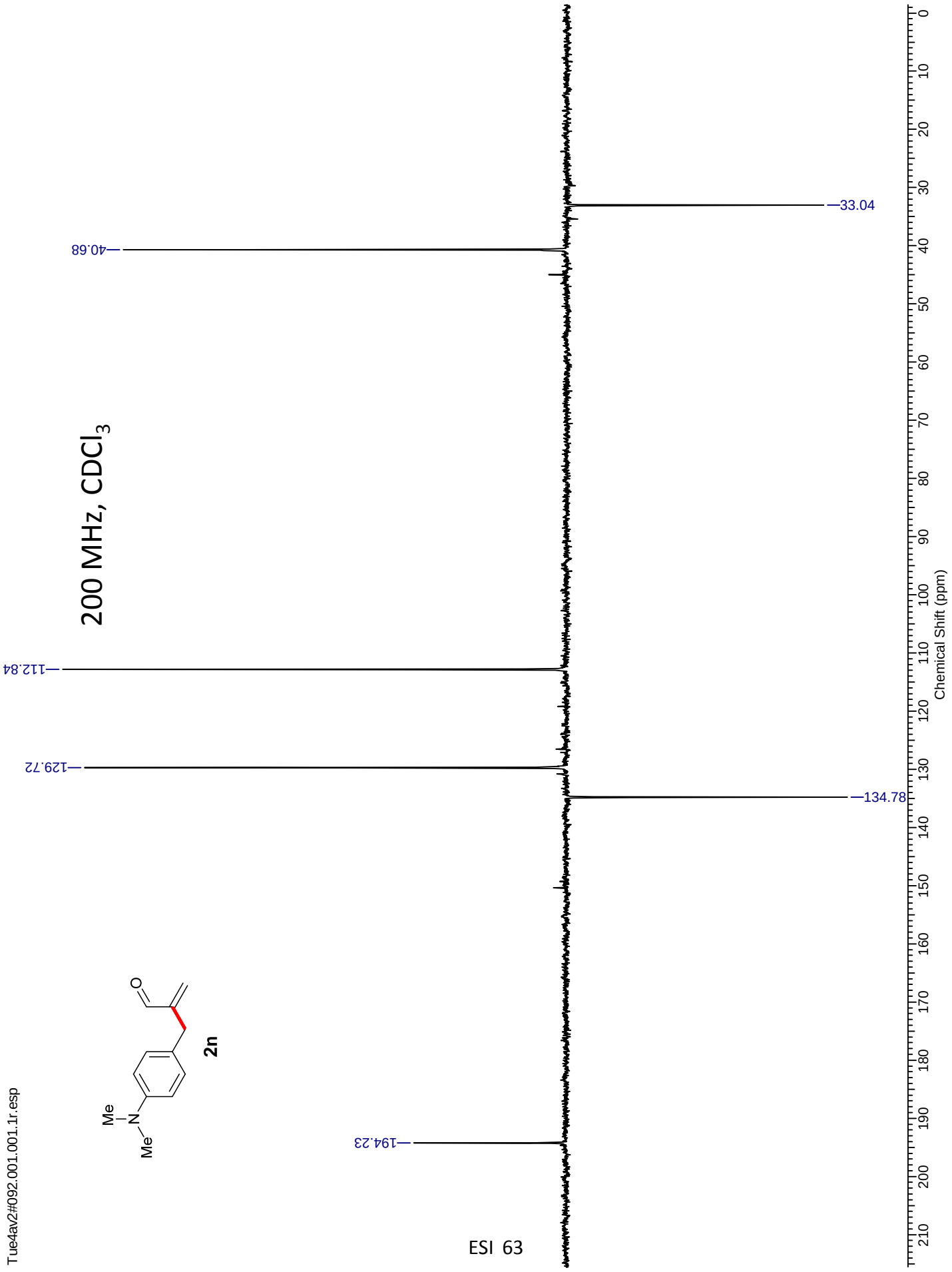

200 MHz, $CDCl_3$
193.19
136.96
128.76
128.47
126.76
105.95
60.78
55.97
49.23
OMe
OMe
MeO
O
2m
Chemical Shift (ppm)
0
8
16
24
32
40
48
56
64
72
80
88
96
104
112
120
128
136
144
152
160
168
176
184
192
200
Sat1av2#075.001.001.1r.esp
ESI 59

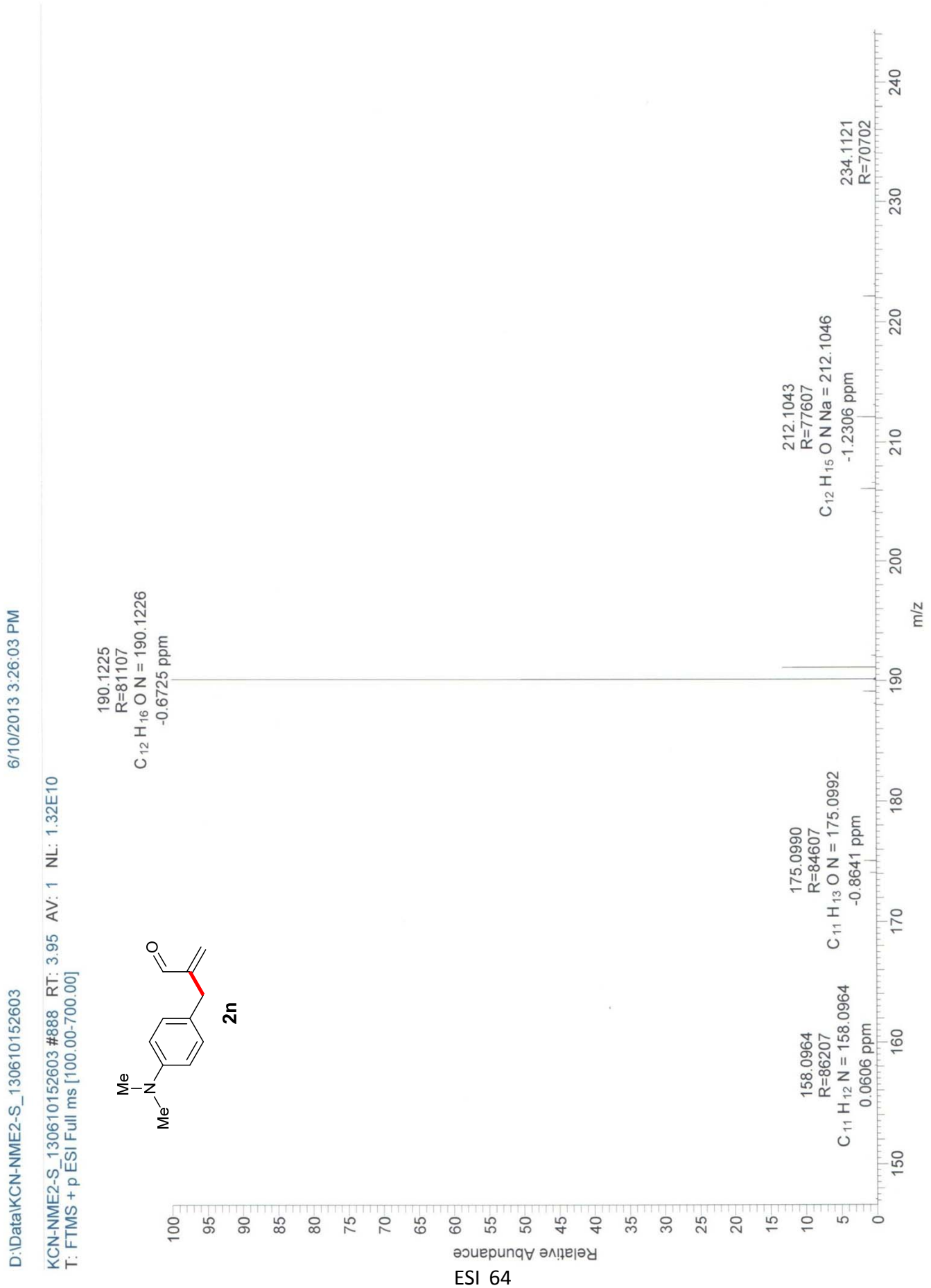

D:\Data\KCN-TB-PH_130610163308
6/10/2013 4:33:08 PM
KCN-TB-PH_130610163308 #923 RT: 4.11 AV: 1 NL: 5.31E9
T: FTMS + p ESI Full ms [100.00-700.00]
335.1248
R=61207
C19 H20 O4 Na = 335.1254
-1.5901 ppm
313.1428
R=63107
C19 H21 O4 = 313.1434
-1.9587 ppm
341.6231
R=48502
353.1352
R=59007
367.1509
R=58707
375.1174
R=46200
385.1613
R=44900
OMe
OMe
MeO
O
2m
Relative Abundance
m/z

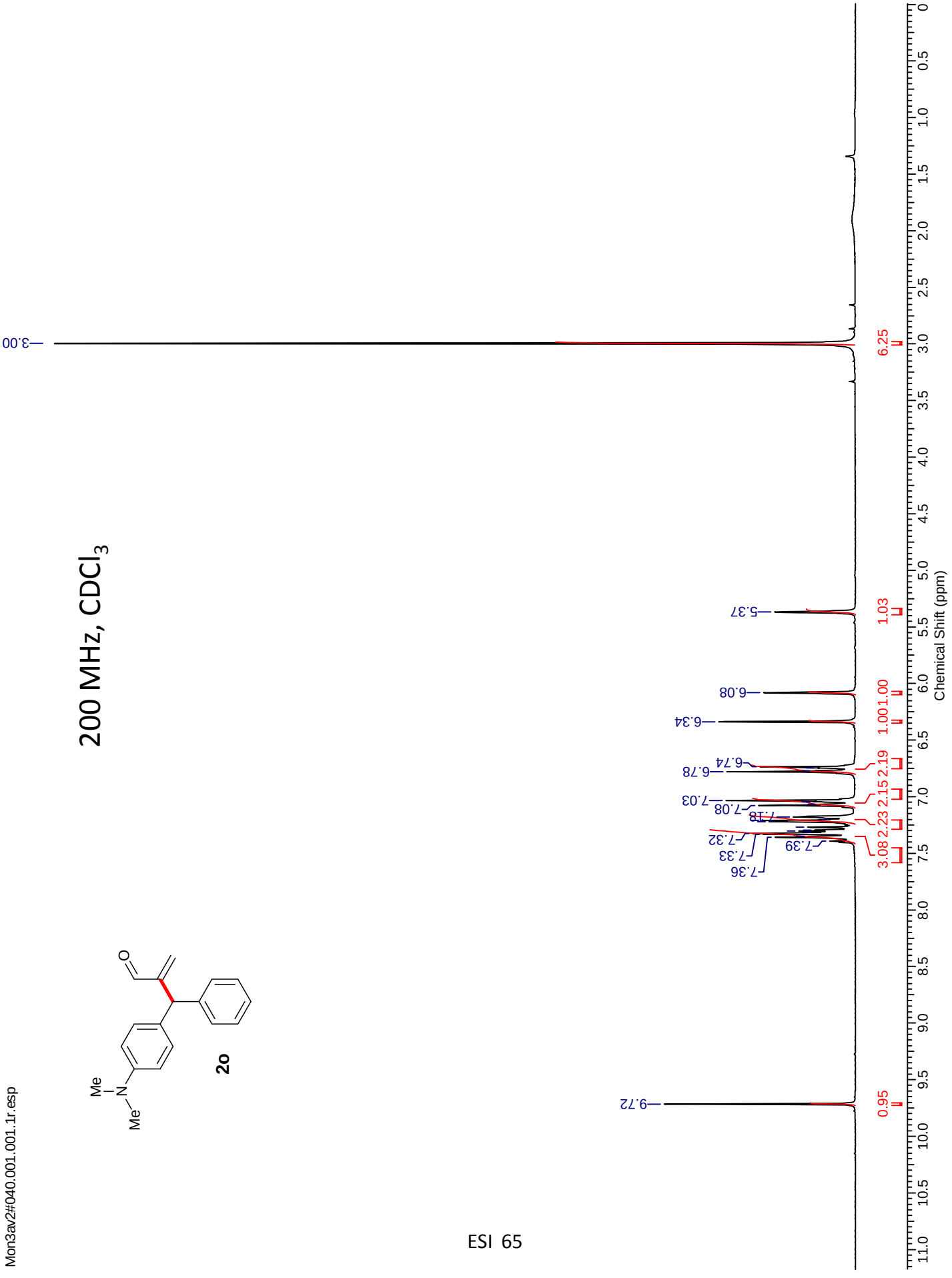

400 MHz, CDCl3
2n
Me
N
Me
O
Mon4ECX400#034_PROTON-3.esp
Chemical Shift (ppm)
9.62
7.09
7.06
6.73
6.71
6.12
6.04
3.49
2.94
0.87
1.89
1.99
1.00
1.00
2.01
6.02

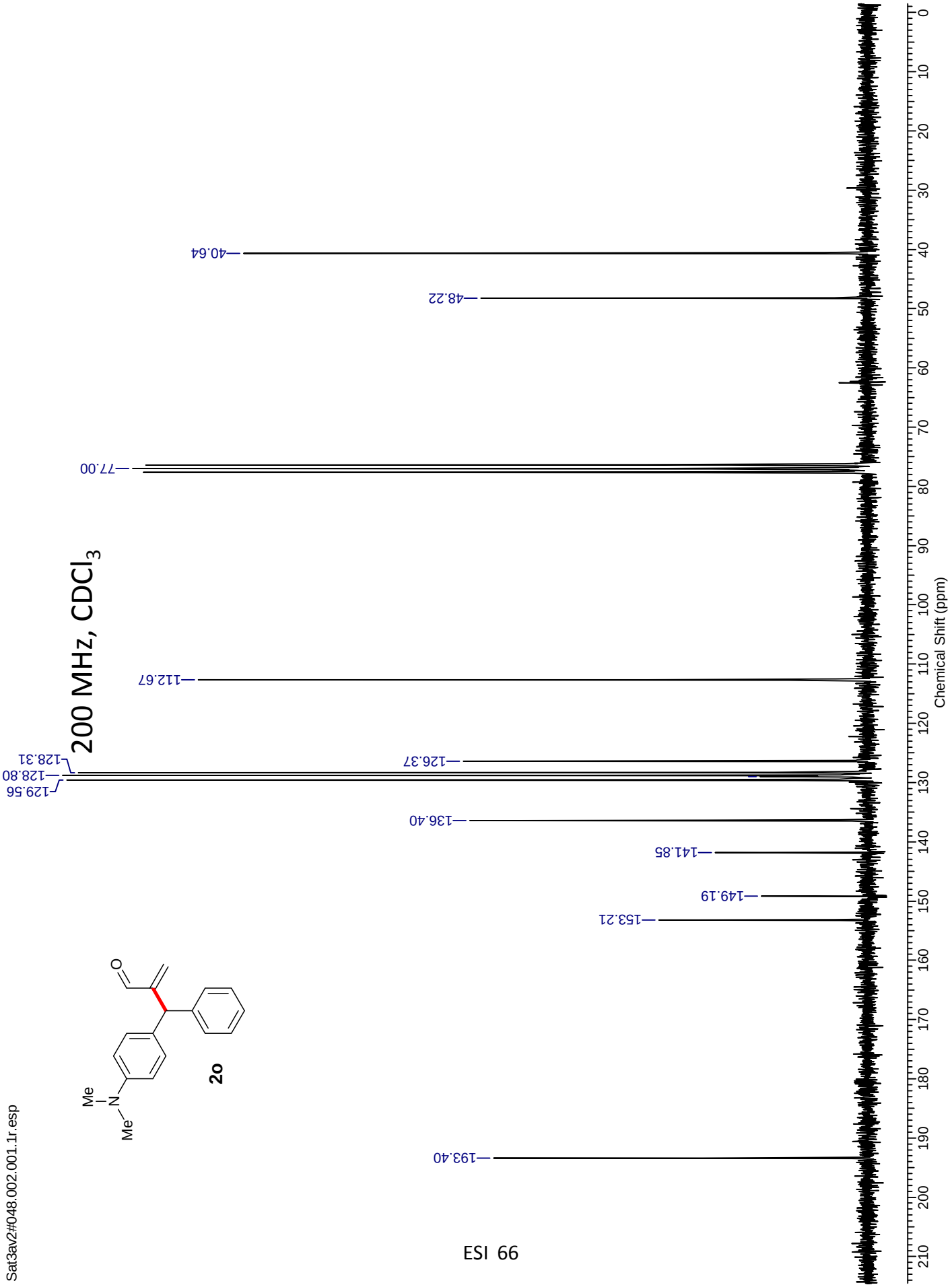

200 MHz, CDCl3
33.04
40.65
77.00
112.85
125.84
129.70
134.66
149.29
150.41
194.16
Chemical Shift (ppm)
0
8
16
24
32
40
48
56
64
72
80
88
96
104
112
120
128
136
144
152
160
168
176
184
192
200
208
O
Me
N
Me
2n
Tue4av2#092.002.001.1r.esp

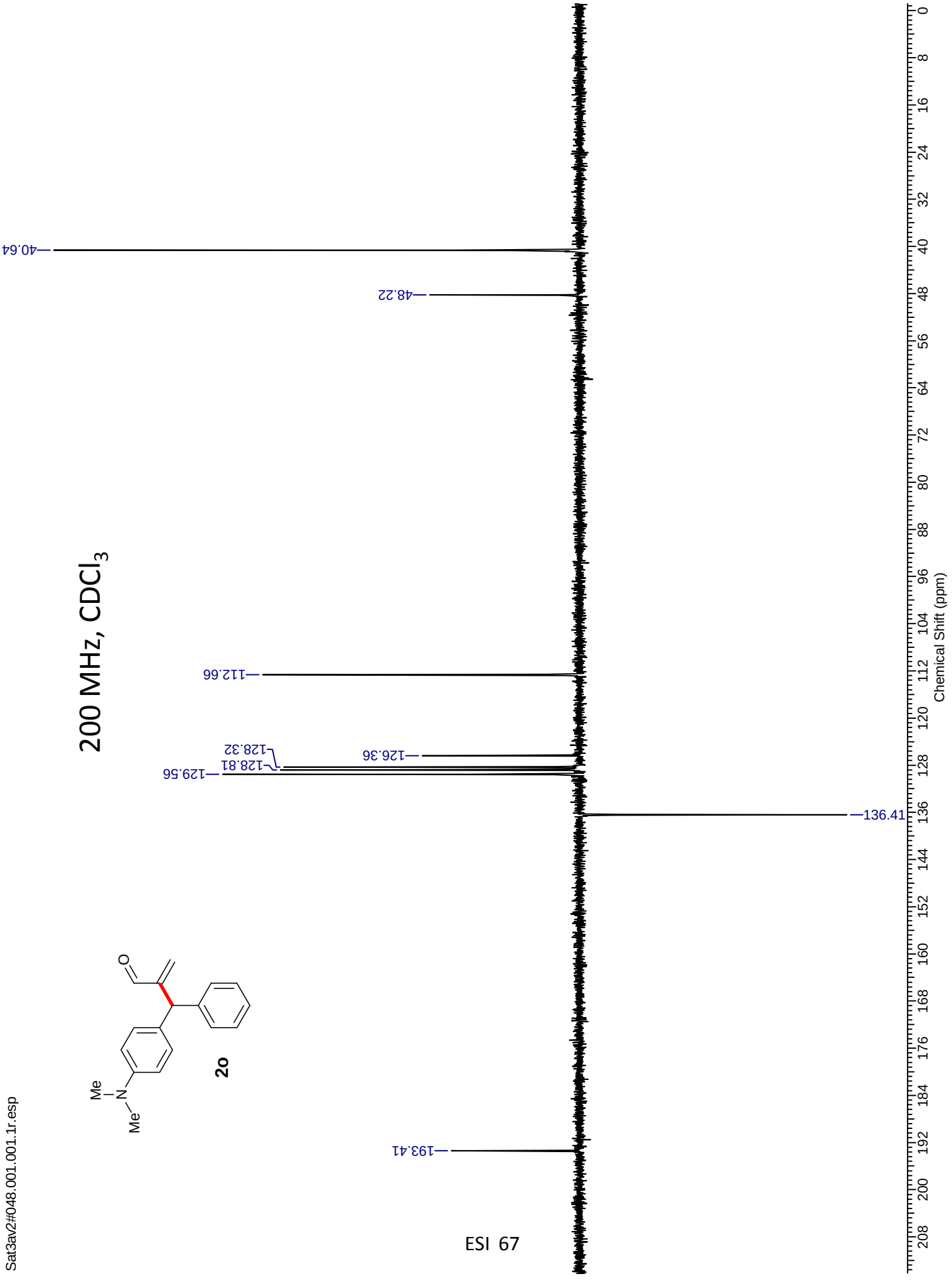
ESI 62

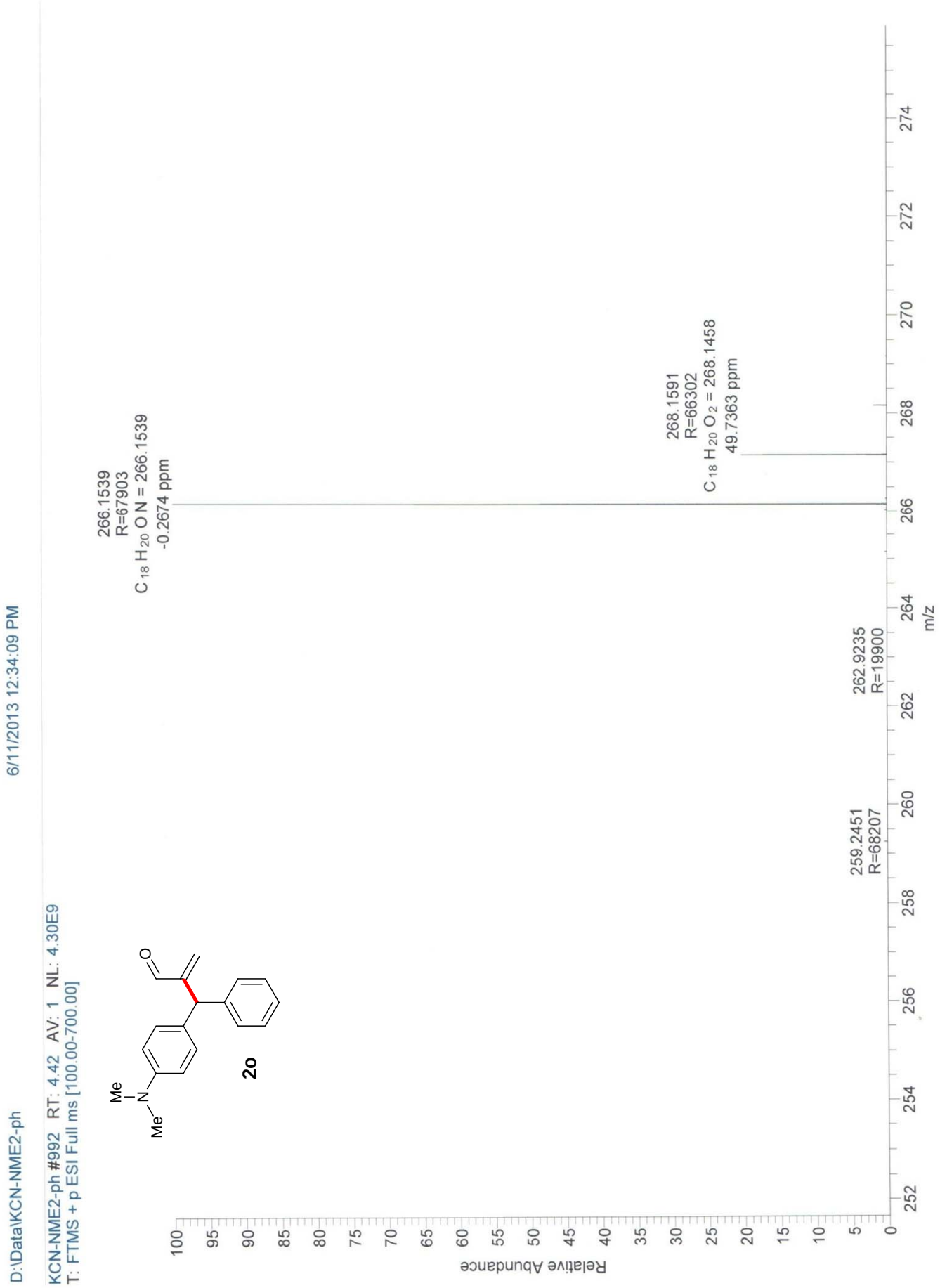

200 MHz, CDCl3
33.04
40.68
112.84
129.72
134.78
194.23
Chemical Shift (ppm)
0
10
20
30
40
50
60
70
80
90
100
110
120
130
140
150
160
170
180
190
200
210
O
Me
N
Me
2n
Tue4av2#092.001.001.1r.esp
ESI 63

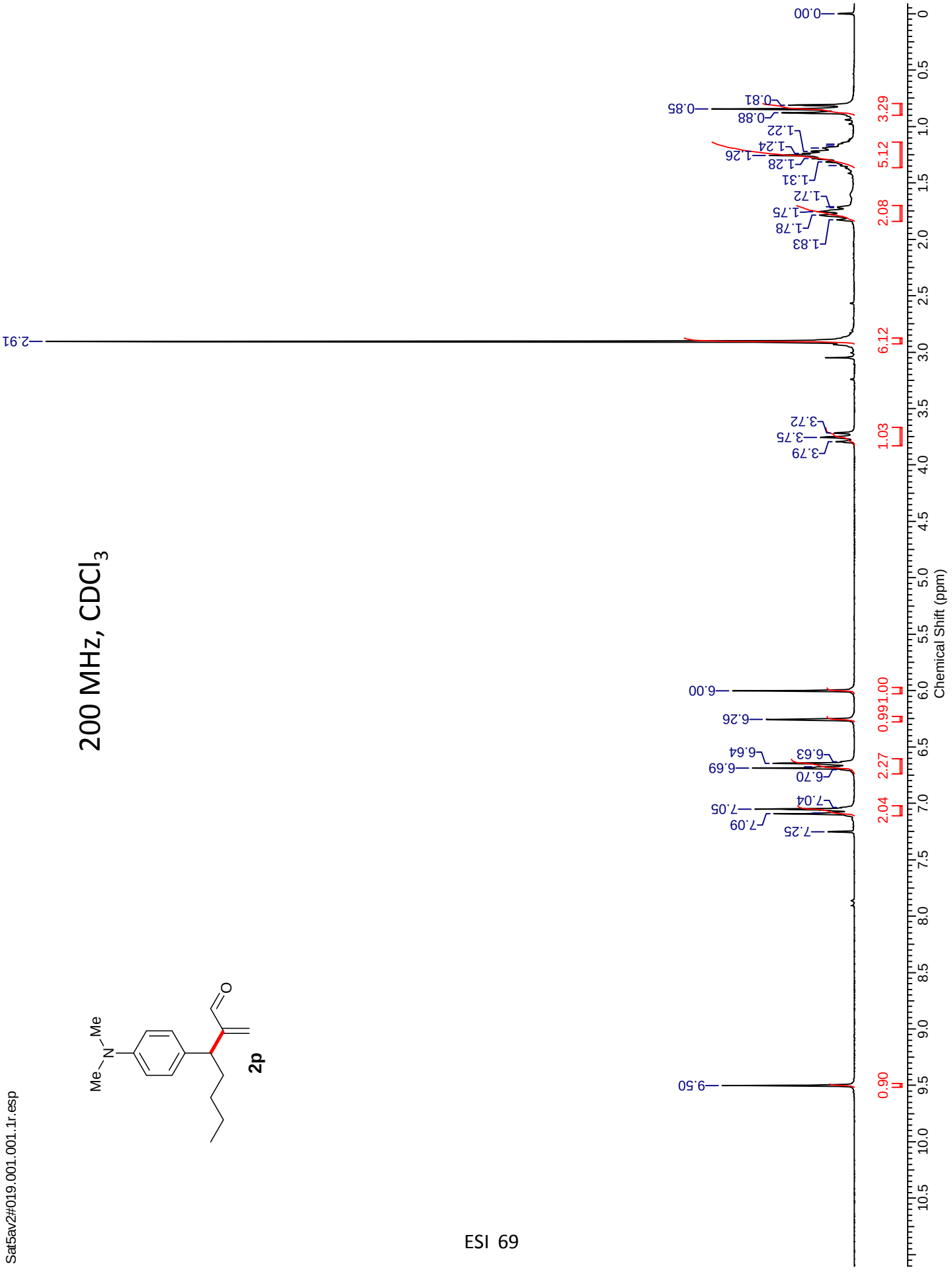

D:\Data\KCN-NME2-S_130610152603
6/10/2013 3:26:03 PM
KCN-NME2-S_130610152603 #888 RT: 3.95 AV: 1 NL: 1.32E10
T: FTMS + p ESI Full ms [100.00-700.00]
190.1225
R=81107
C 12 H 16 O N = 190.1226
-0.6725 ppm
212.1043
R=77607
C 12 H 15 O N Na = 212.1046
-1.2306 ppm
234.1121
R=70702
175.0990
R=84607
C 11 H 13 O N = 175.0992
-0.8641 ppm
158.0964
R=86207
C 11 H 12 N = 158.0964
0.0606 ppm
Me
N
Me
O
2n
Relative Abundance
m/z

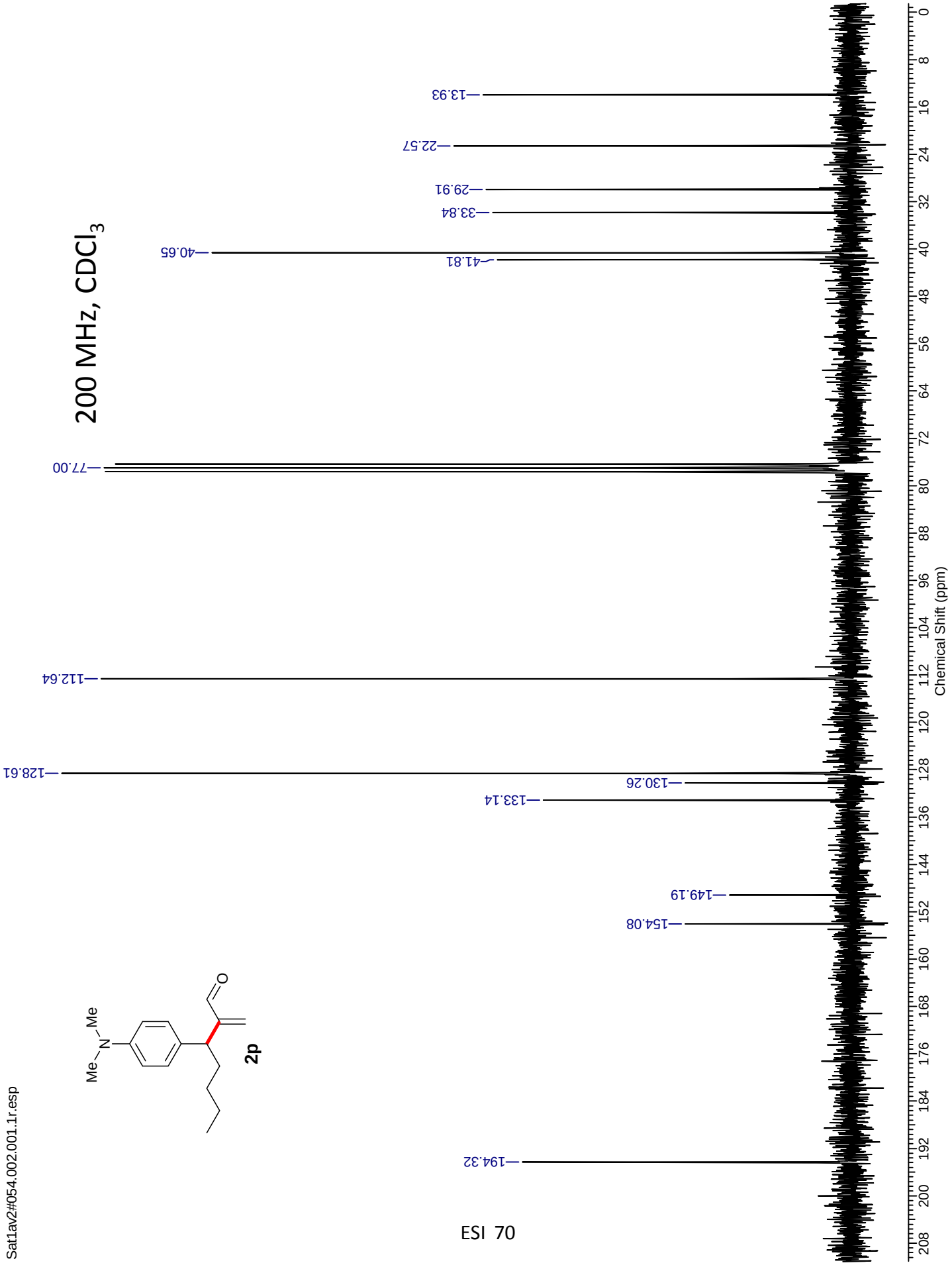

200 MHz, CDCl3
9.72
7.39
7.36
7.33
7.32
7.18
7.08
7.03
6.78
6.74
6.34
6.08
5.37
3.00
0.95
3.08
2.23
2.15
2.19
1.00
1.00
1.03
6.25
Chemical Shift (ppm)
Me
N
Me
O
2o
Mon3av2#040.001.001.1r.esp

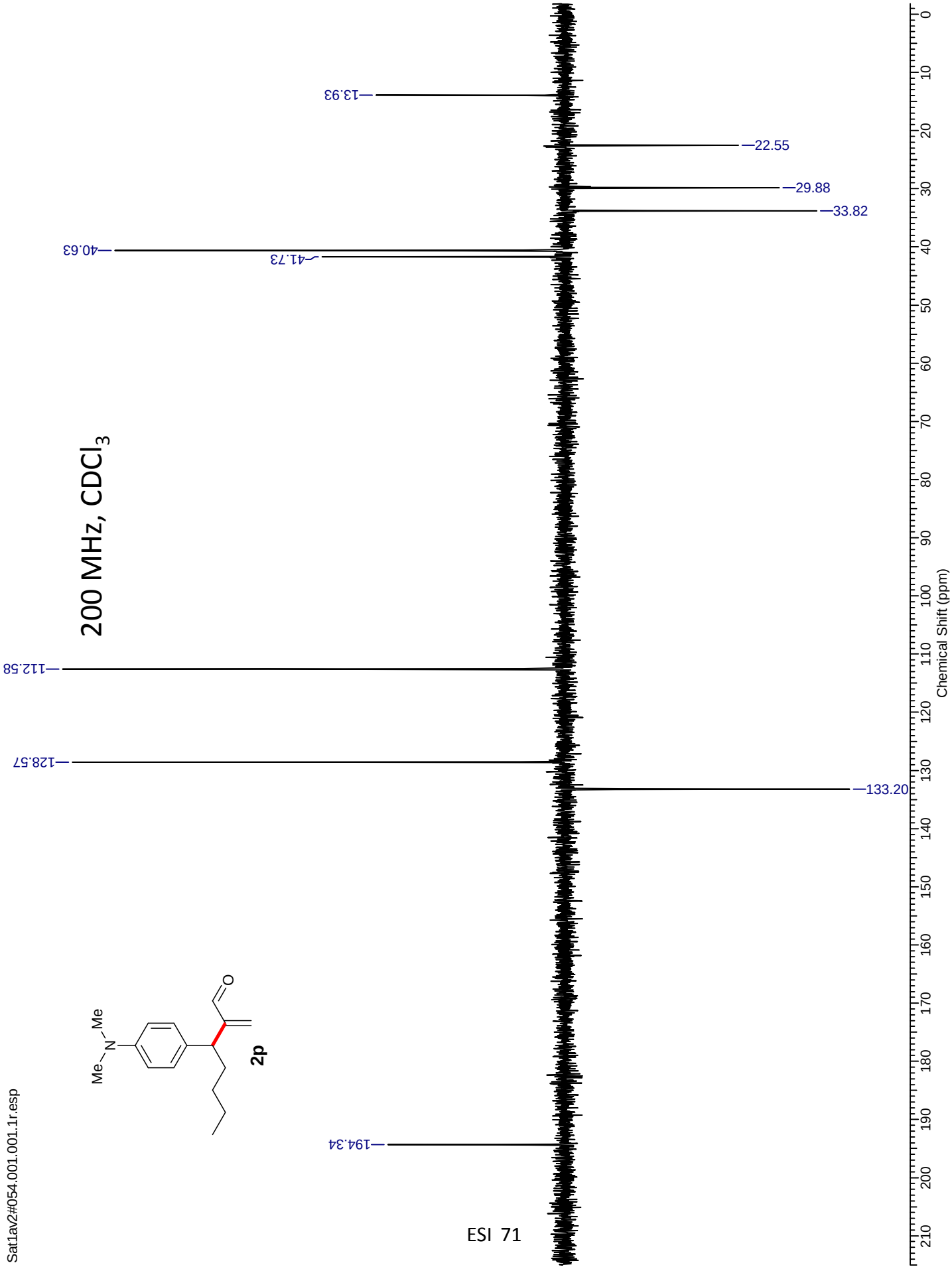

200 MHz, CDCl3
40.64
48.22
77.00
112.67
126.37
128.31
128.80
129.56
136.40
141.85
149.19
153.21
193.40
O
Me
N
Me
2o
Chemical Shift (ppm)
Sat3av2#048.002.001.1r.esp

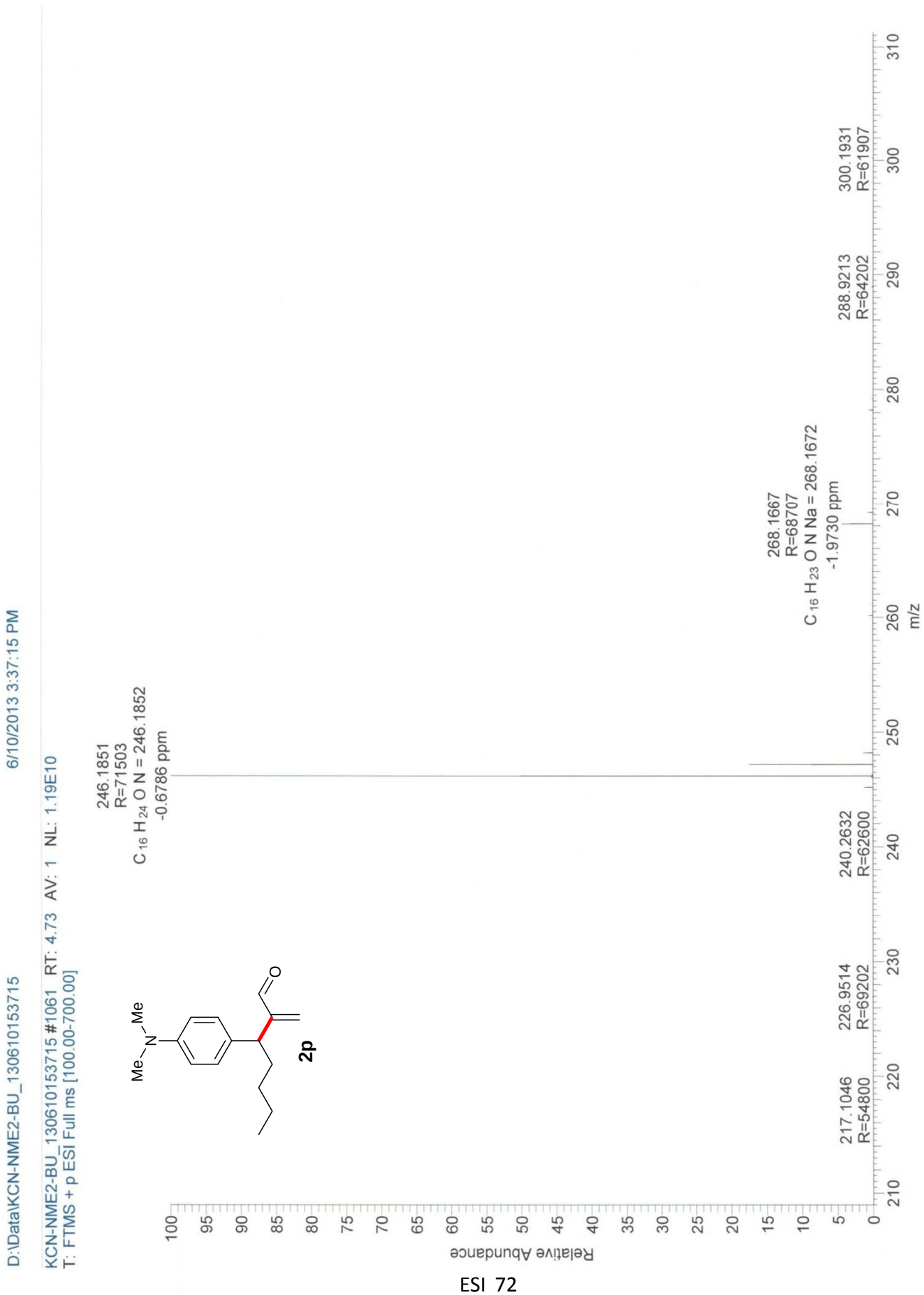

200 MHz, CDCl3
40.64
48.22
112.66
126.36
128.32
128.81
129.56
136.41
193.41
Chemical Shift (ppm)
0
8
16
24
32
40
48
56
64
72
80
88
96
104
112
120
128
136
144
152
160
168
176
184
192
200
208
O
Me
N
Me
2o
ESI 67
Sat3av2#048.001.001.1r.esp

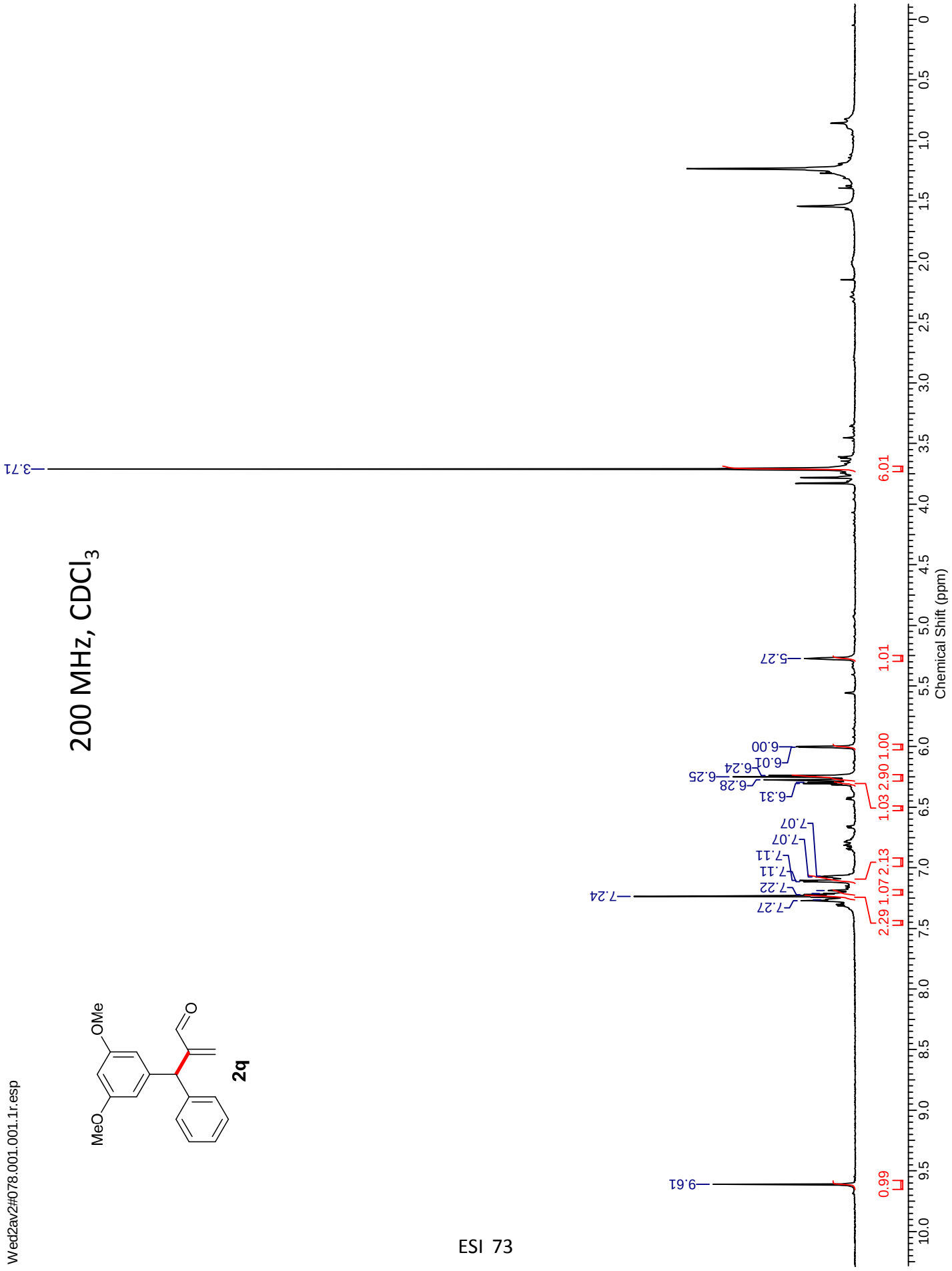

D:\Data\KCN-NME2-ph
6/11/2013 12:34:09 PM
KCN-NME2-ph #992 RT: 4.42 AV: 1 NL: 4.30E9
T: FTMS + p ESI Full ms [100.00-700.00]
266.1539
R=67903
C 18 H 20 O N = 266.1539
-0.2674 ppm
268.1591
R=66302
C 18 H 20 O 2 = 268.1458
49.7363 ppm
259.2451
R=68207
262.9235
R=19900
Me
N
Me
O
2o
Relative Abundance
m/z

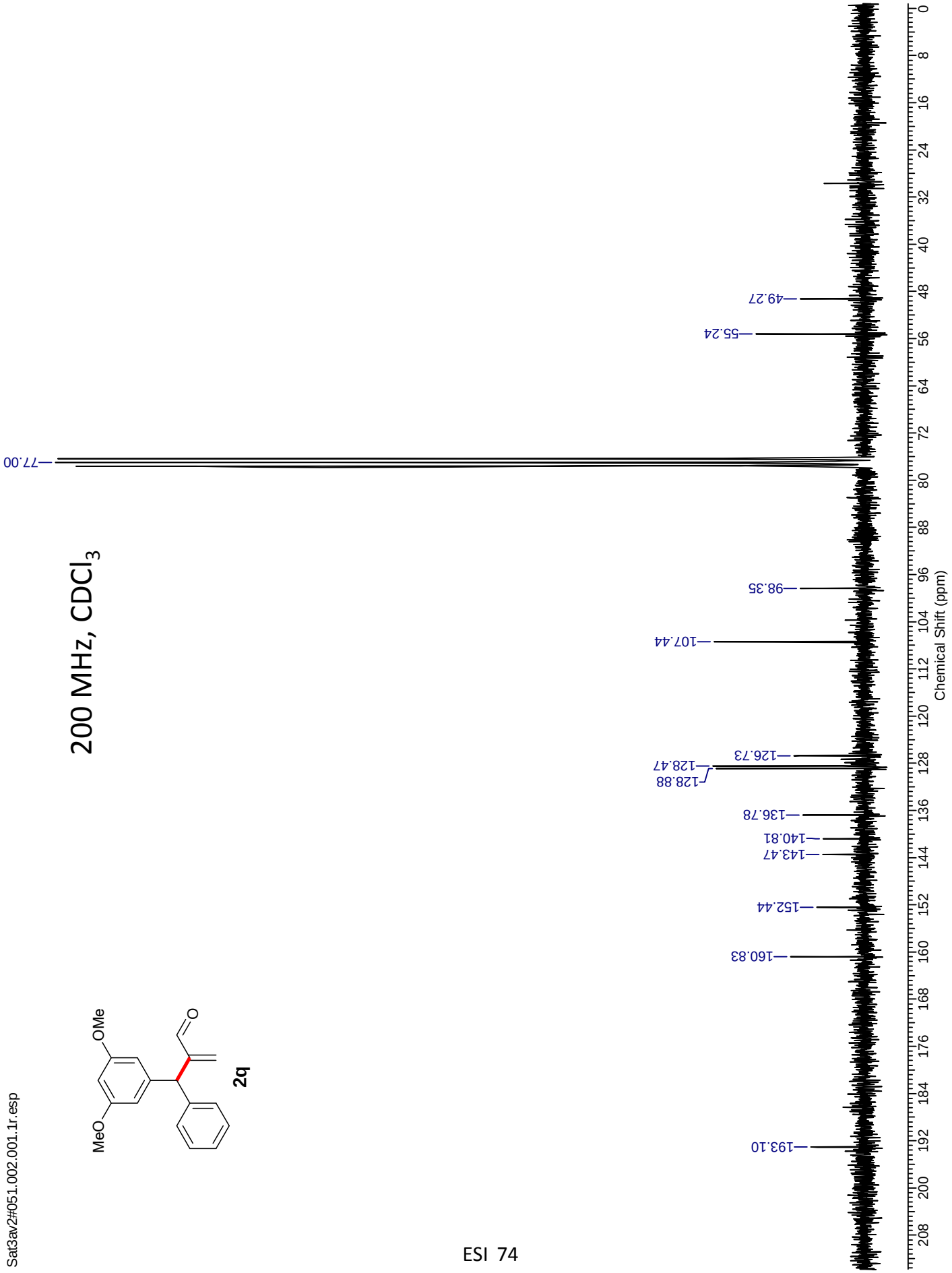

200 MHz, CDCl3
Sat5av2#019.001.001.1r.esp
Chemical Shift (ppm)
0.00
0.81
0.85
0.88
1.22
1.24
1.26
1.28
1.31
1.72
1.75
1.78
1.83
2.91
3.72
3.75
3.79
6.00
6.26
6.63
6.64
6.69
6.70
7.04
7.05
7.09
7.25
9.50
3.29
5.12
2.08
6.12
1.03
1.00
0.99
2.27
2.04
0.90
Me
N
Me
O
2p

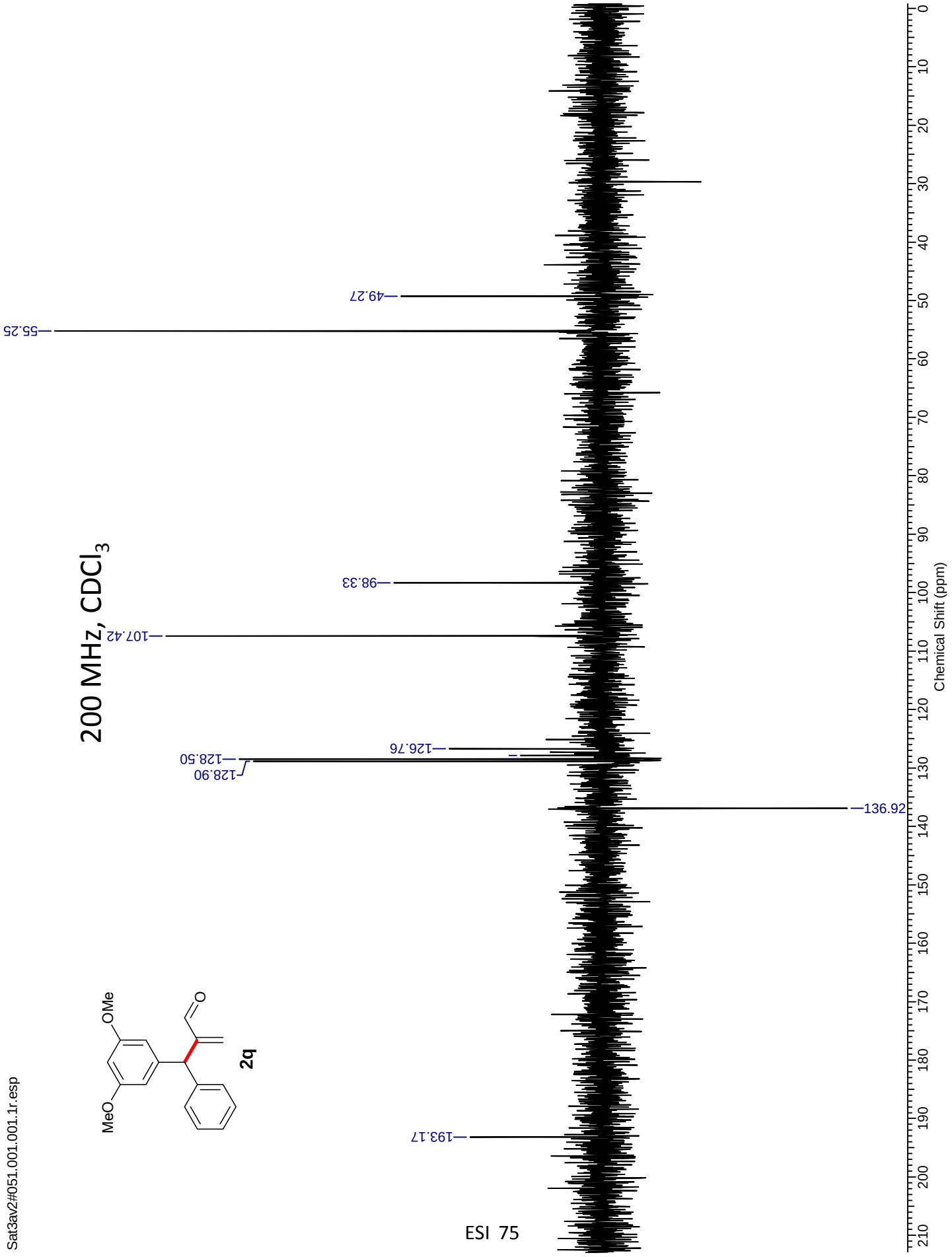

200 MHz, $CDCl_3$
13.93
22.57
29.91
33.84
40.65
41.81
77.00
112.64
128.61
130.26
133.14
149.19
154.08
194.32
Chemical Shift (ppm)
0
8
16
24
32
40
48
56
64
72
80
88
96
104
112
120
128
136
144
152
160
168
176
184
192
200
208
Me
N
Me
O
2p
Sat1av2#054.002.001.1r.esp
ESI 70

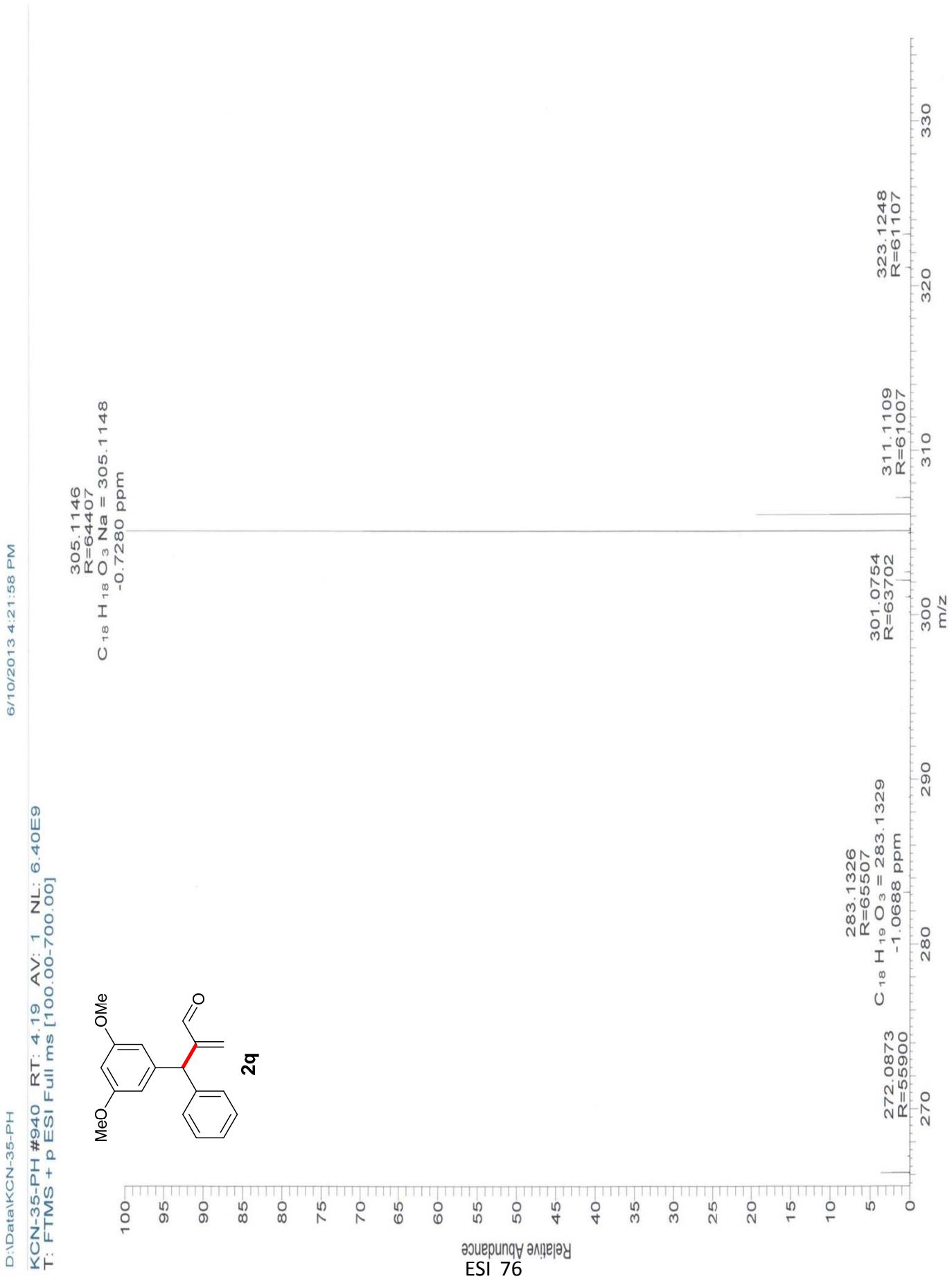

200 MHz, CDCl3
13.93
22.55
29.88
33.82
40.63
41.73
112.58
128.57
133.20
194.34
Chemical Shift (ppm)
0
10
20
30
40
50
60
70
80
90
100
110
120
130
140
150
160
170
180
190
200
210
Me
N
Me
O
2p
Sat1av2#054.001.001.1r.esp
ESI 71

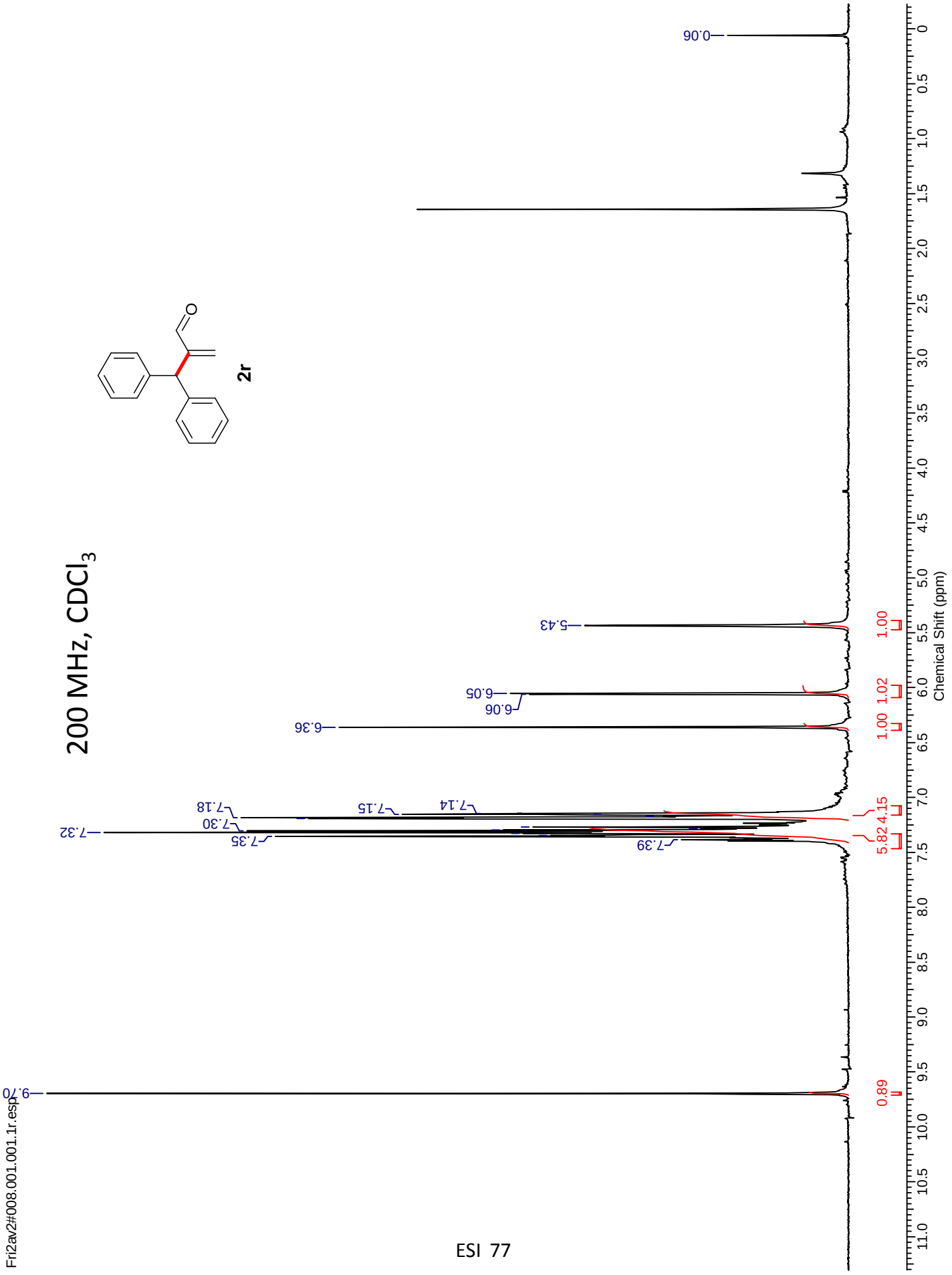

D:\Data\KCN-NME2-BU_130610153715
6/10/2013 3:37:15 PM
KCN-NME2-BU_130610153715 #1061 RT: 4.73 AV: 1 NL: 1.19E10
T: FTMS + p ESI Full ms [100.00-700.00]
246.1851
R=71503
C 16 H 24 O N = 246.1852
-0.6786 ppm
268.1667
R=68707
C 16 H 23 O N Na = 268.1672
-1.9730 ppm
217.1046
R=54800
226.9514
R=69202
240.2632
R=62600
288.9213
R=64202
300.1931
R=61907
Me
N
Me
O
2p
Relative Abundance
m/z

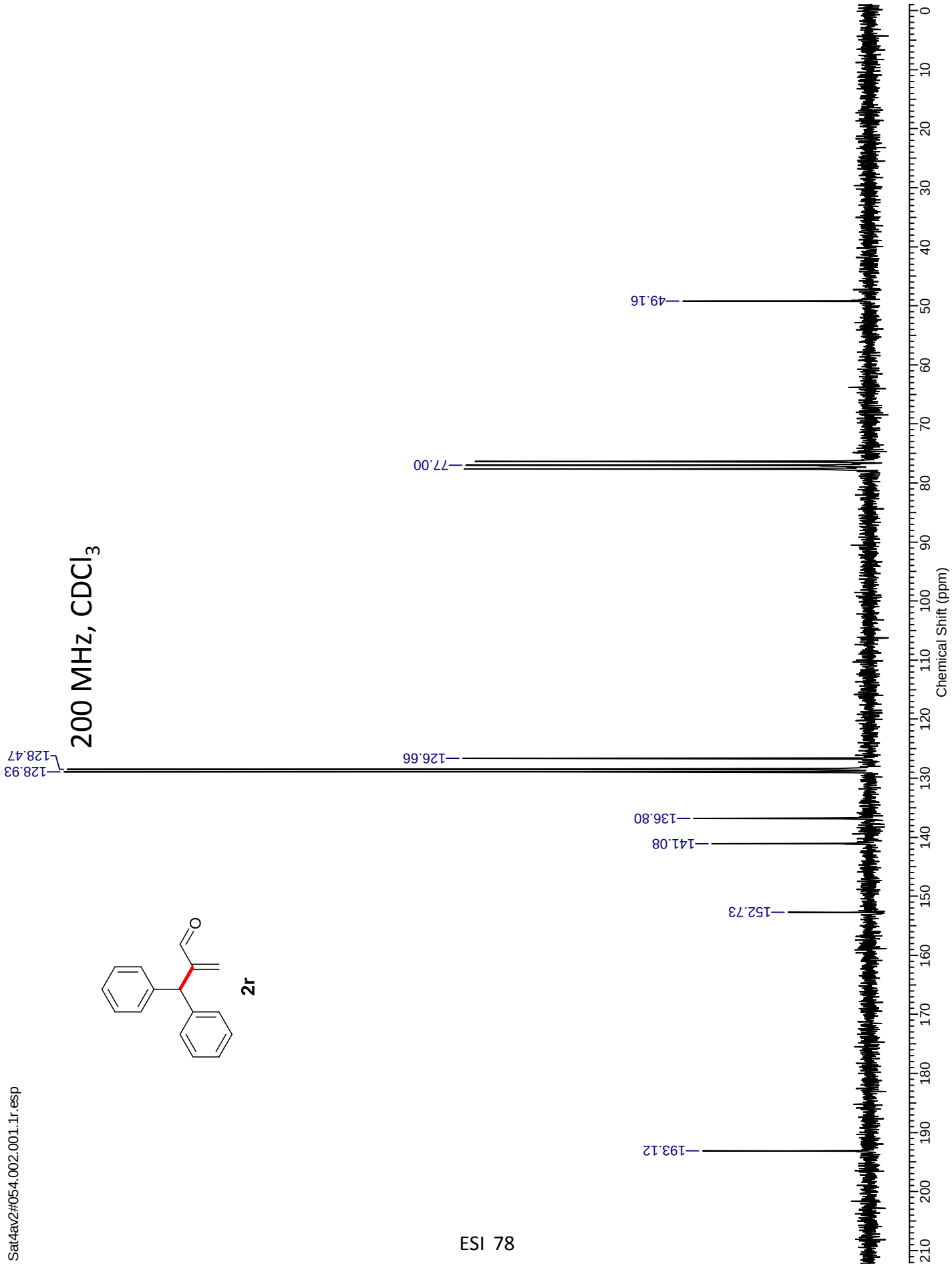

200 MHz, CDCl3
Wed2av2#078.001.001.1r.esp
OMe
MeO
O
2q
3.71
9.61
7.24
7.27
7.22
7.11
7.11
7.07
7.07
6.31
6.28
6.25
6.24
6.01
6.00
5.27
6.01
1.01
1.00
2.90
1.03
2.13
1.07
2.29
0.99
Chemical Shift (ppm)

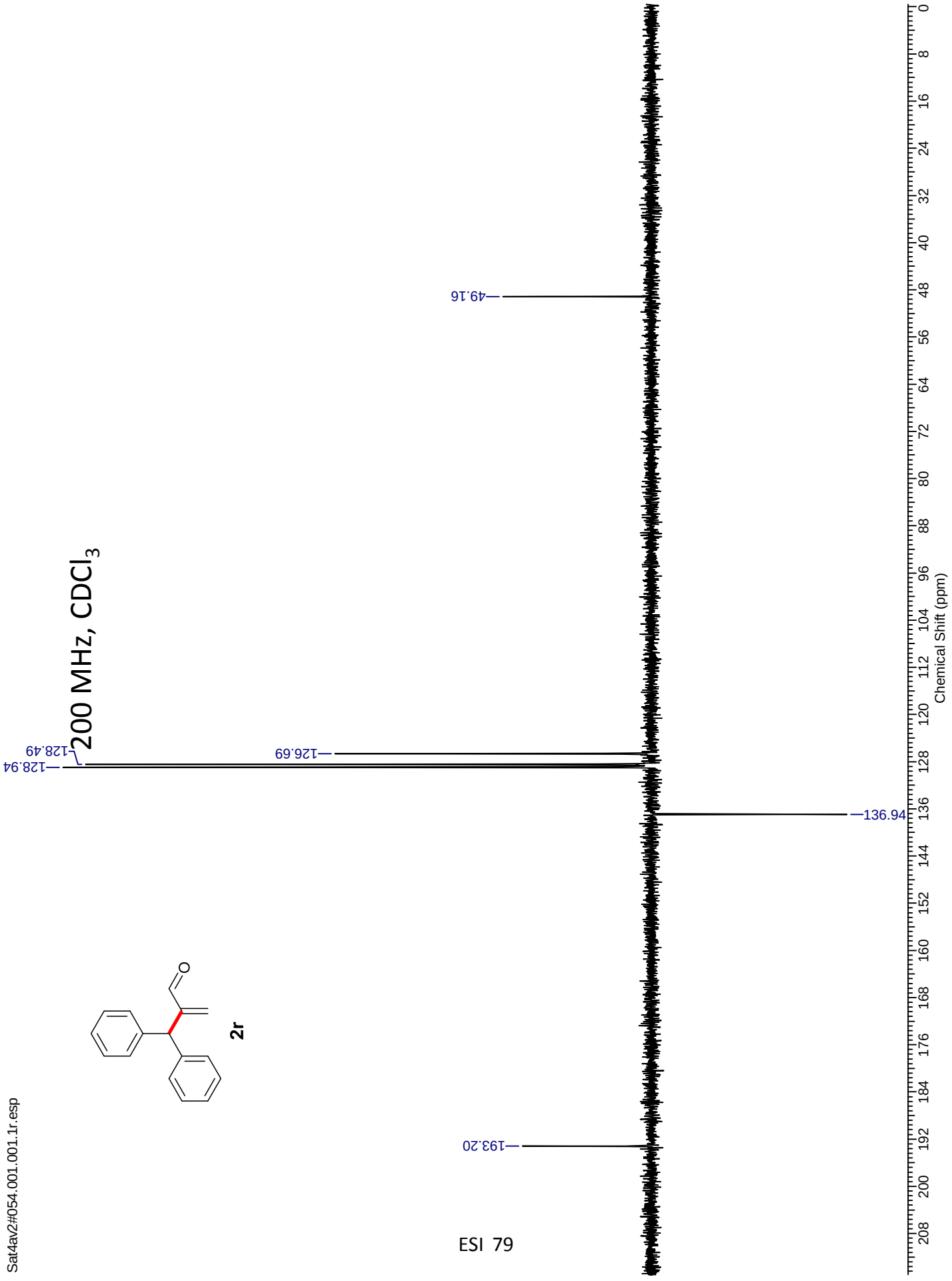

200 MHz, $CDCl_3$
77.00
49.27
55.24
98.35
107.44
126.73
128.88
128.47
136.78
140.81
143.47
152.44
160.83
193.10
Chemical Shift (ppm)
OMe
MeO
O
2q
Sat3av2#051.002.001.1r.esp

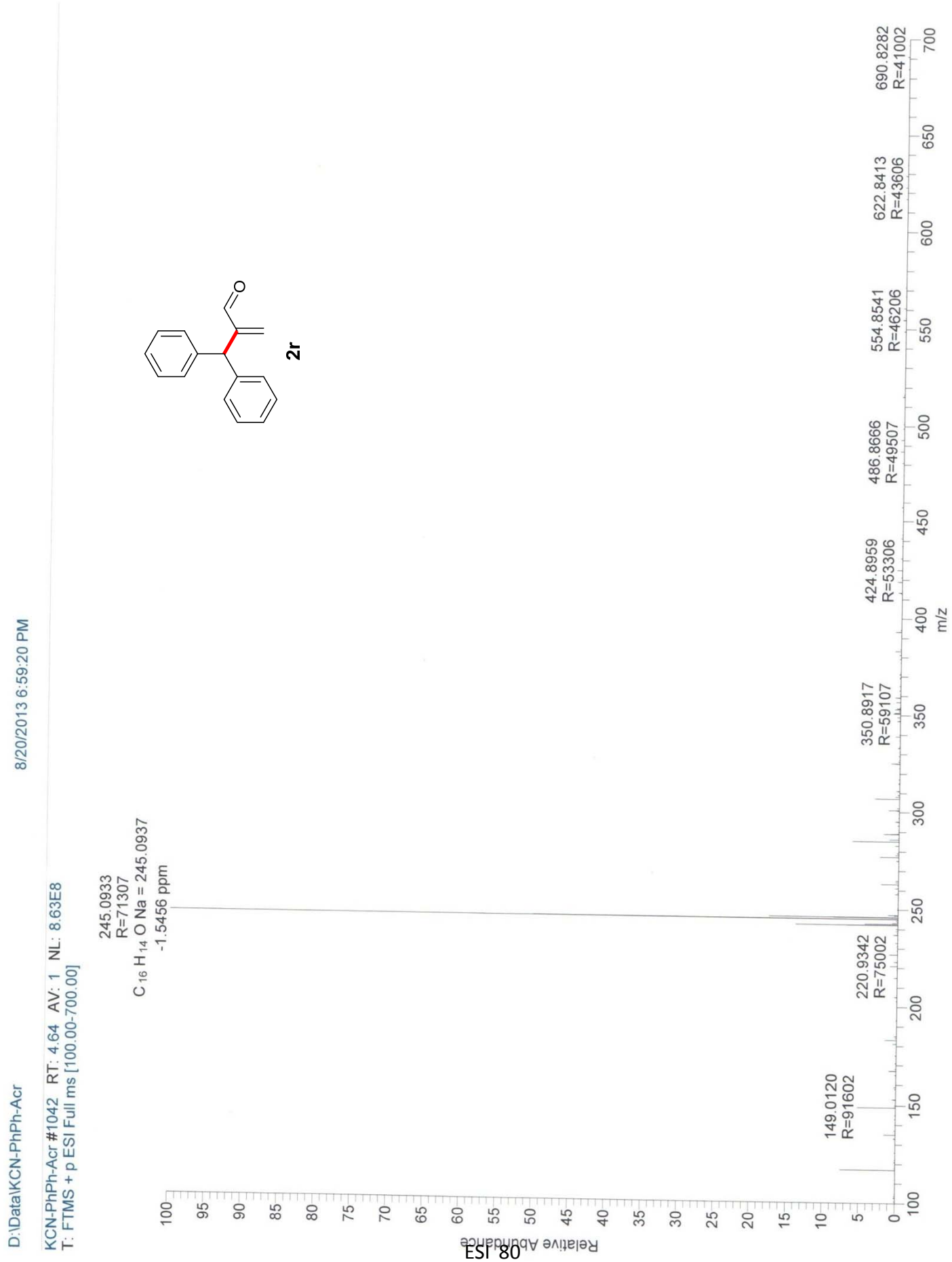

200 MHz, $CDCl_3$
55.25
49.27
98.33
107.42
128.90
128.50
126.76
136.92
193.17
OMe
MeO
O
2q
Sat3av2#051.001.001.1r.esp
ESI 75
Chemical Shift (ppm)
0
10
20
30
40
50
60
70
80
90
100
110
120
130
140
150
160
170
180
190
200
210

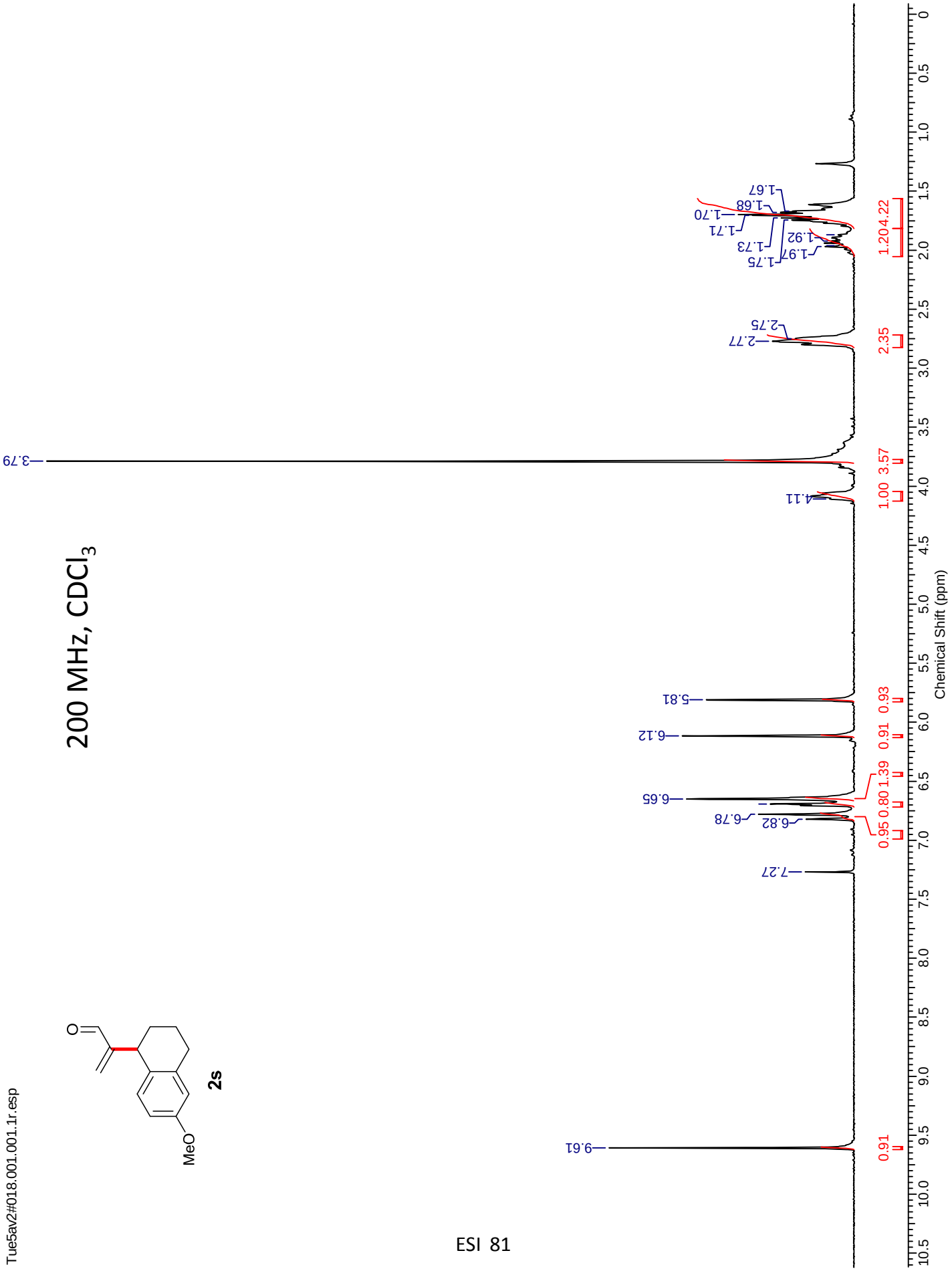

D:\Data\KCN-35-PH
6/10/2013 4:21:58 PM
KCN-35-PH #940 RT: 4.19 AV: 1 NL: 6.40E9
T: FTMS + p ESI Full ms [100.00-700.00]
305.1146
R=64407
C 18 H 18 O 3 Na = 305.1148
-0.7280 ppm
272.0873
R=55900
283.1326
R=65507
C 18 H 19 O 3 = 283.1329
-1.0688 ppm
301.0754
R=63702
311.1109
R=61007
323.1248
R=61107
OMe
MeO
O
2q
Relative Abundance
m/z

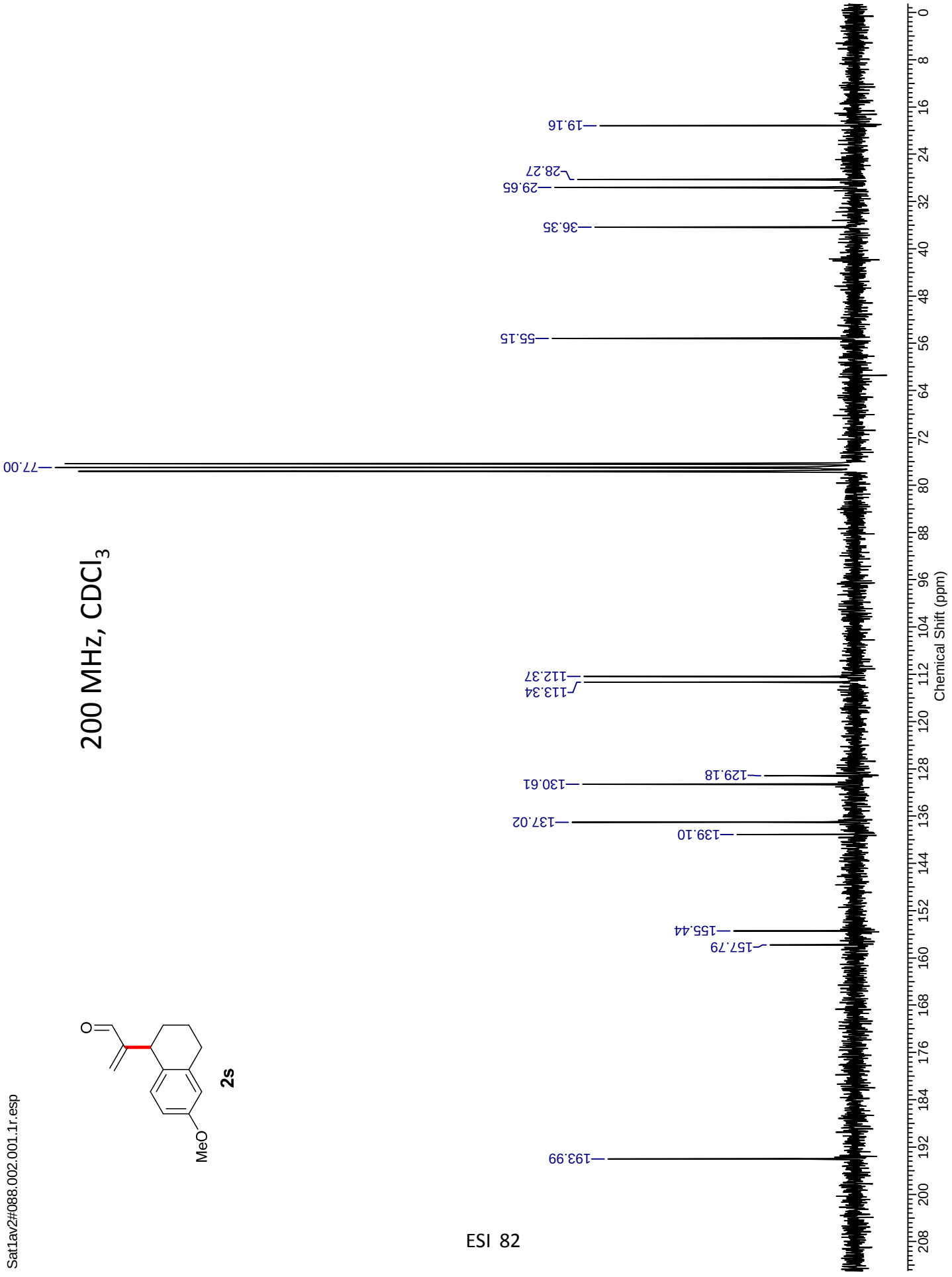

200 MHz, CDCl3
2r
O
9.70
7.32
7.35
7.30
7.18
7.15
7.14
7.39
6.36
6.06
6.05
5.43
0.06
0.89
4.15
5.82
1.02
1.00
1.00
Chemical Shift (ppm)
Fri2av2#008.001.001.1r.esp

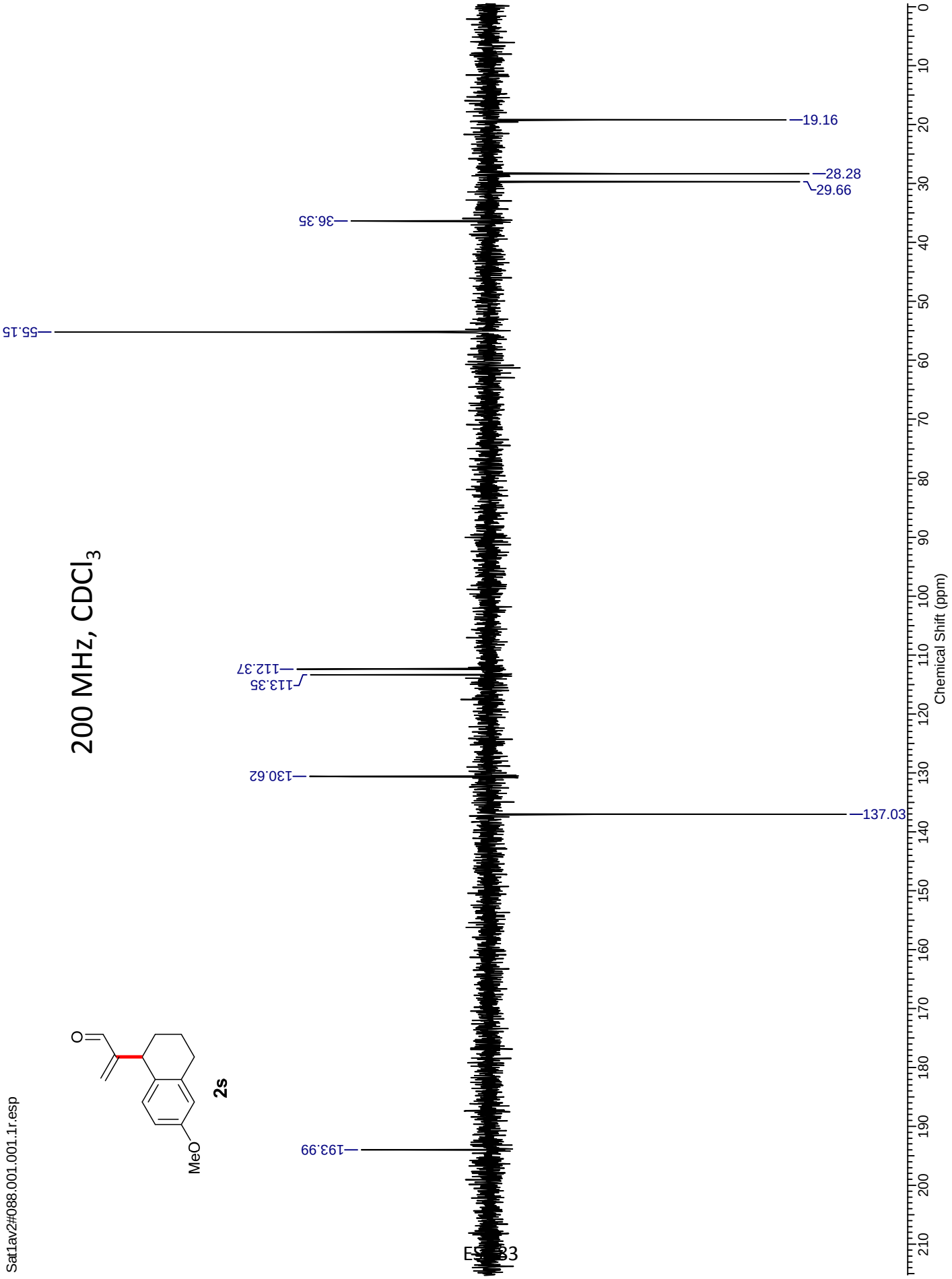

200 MHz, $CDCl_3$
128.93
128.47
126.66
77.00
49.16
136.80
141.08
152.73
193.12
O
2r
Chemical Shift (ppm)
0
10
20
30
40
50
60
70
80
90
100
110
120
130
140
150
160
170
180
190
200
210
Sat4av2#054.002.001.1r.esp

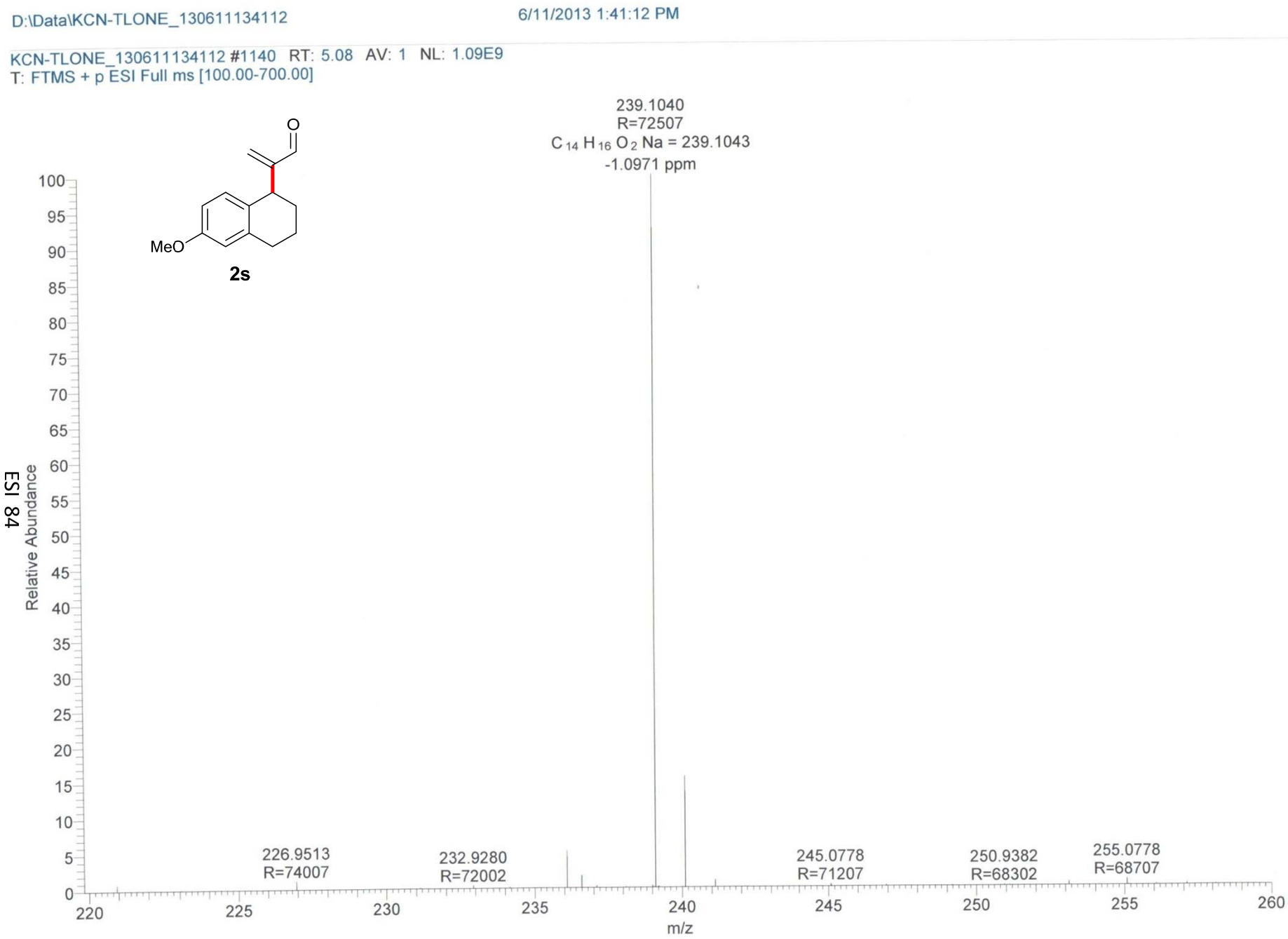

200 MHz, CDCl3
128.94
128.49
126.69
49.16
136.94
193.20
2r
Chemical Shift (ppm)
Sat4av2#054.001.001.1r.esp
ESI 79

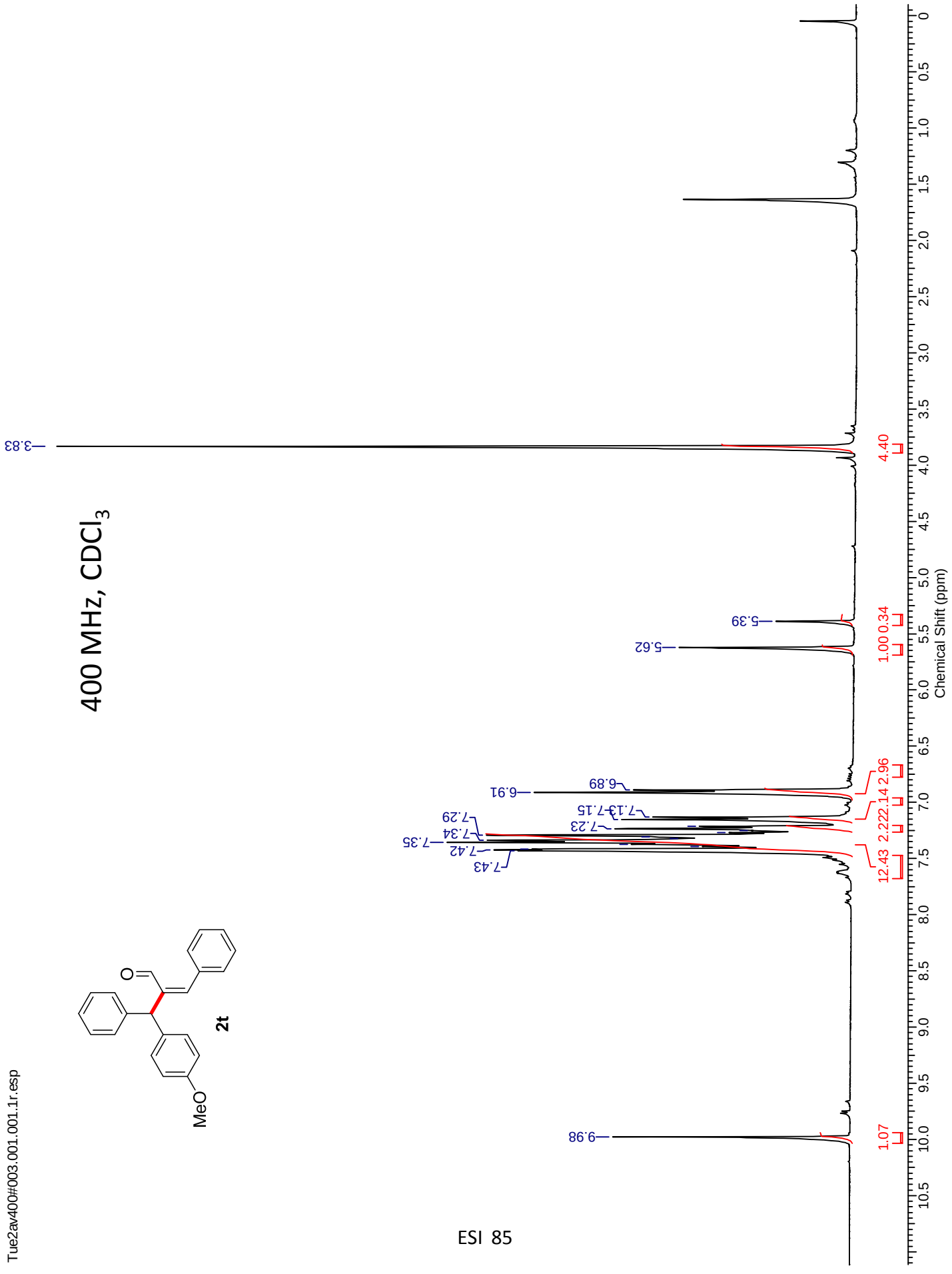

D:\Data\KCN-PhPh-Acr
8/20/2013 6:59:20 PM
KCN-PhPh-Acr #1042 RT: 4.64 AV: 1 NL: 8.63E8
T: FTMS + p ESI Full ms [100.00-700.00]
245.0933
R=71307
C 16 H 14 O Na = 245.0937
-1.5456 ppm
2r
149.0120
R=91602
220.9342
R=75002
350.8917
R=59107
424.8959
R=53306
486.8666
R=49507
554.8541
R=46206
622.8413
R=43606
690.8282
R=41002
Relative Abundance
m/z

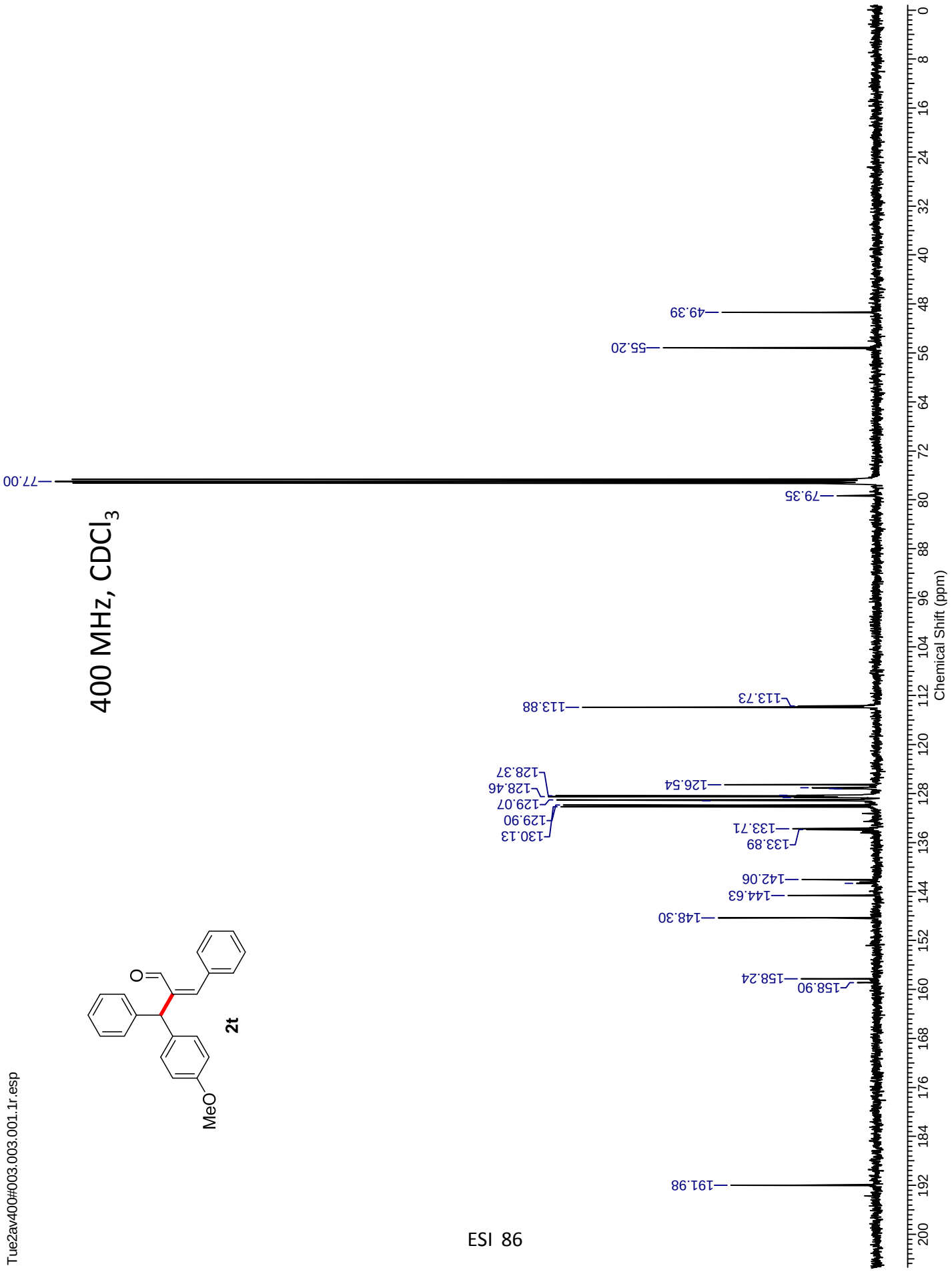

200 MHz, CDCl3
Tue5av2#018.001.001.1r.esp
2s
MeO
O
9.61
7.27
6.82
6.78
6.65
6.12
5.81
4.11
3.79
2.77
2.75
1.97
1.92
1.75
1.73
1.71
1.70
1.68
1.67
0.91
0.95
0.80
1.39
0.91
0.93
1.00
3.57
2.35
1.20
4.22
Chemical Shift (ppm)


200 MHz, $CDCl_3$
77.00
55.15
36.35
29.65
28.27
19.16
112.37
113.34
129.18
130.61
137.02
139.10
155.44
157.79
193.99
Chemical Shift (ppm)
0
8
16
24
32
40
48
56
64
72
80
88
96
104
112
120
128
136
144
152
160
168
176
184
192
200
208
O
MeO
2s
Sat1av2#088.002.001.1r.esp

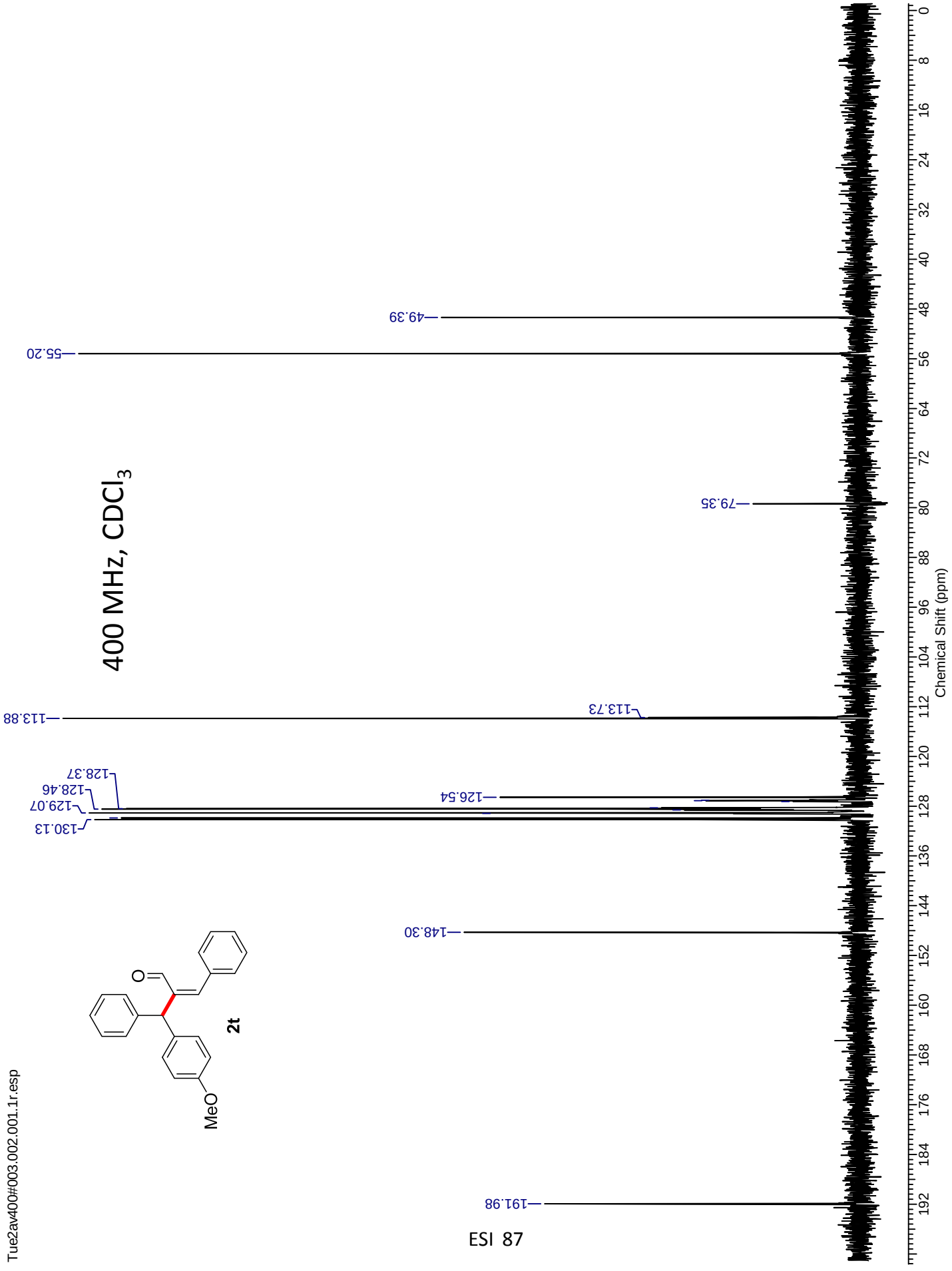




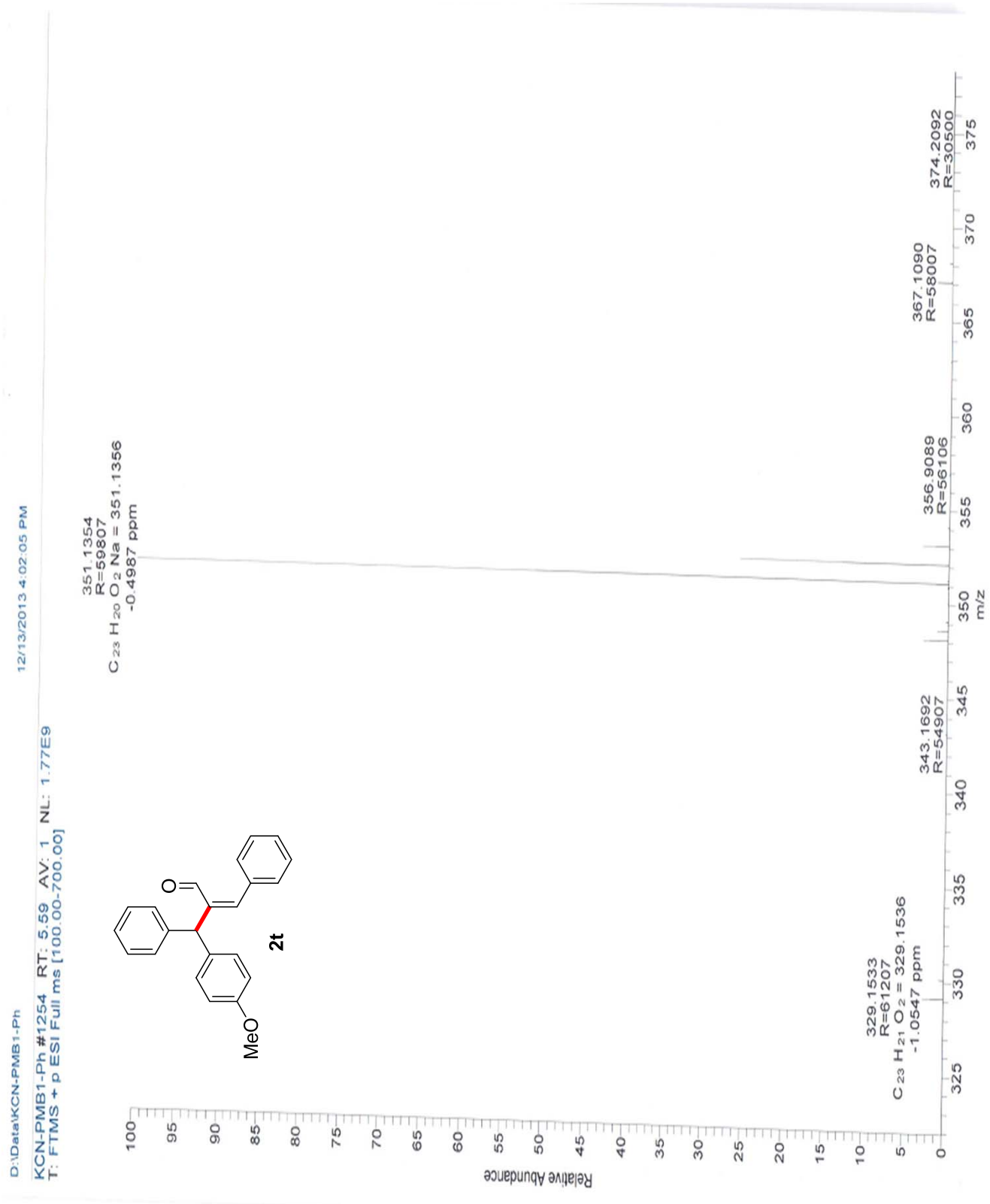


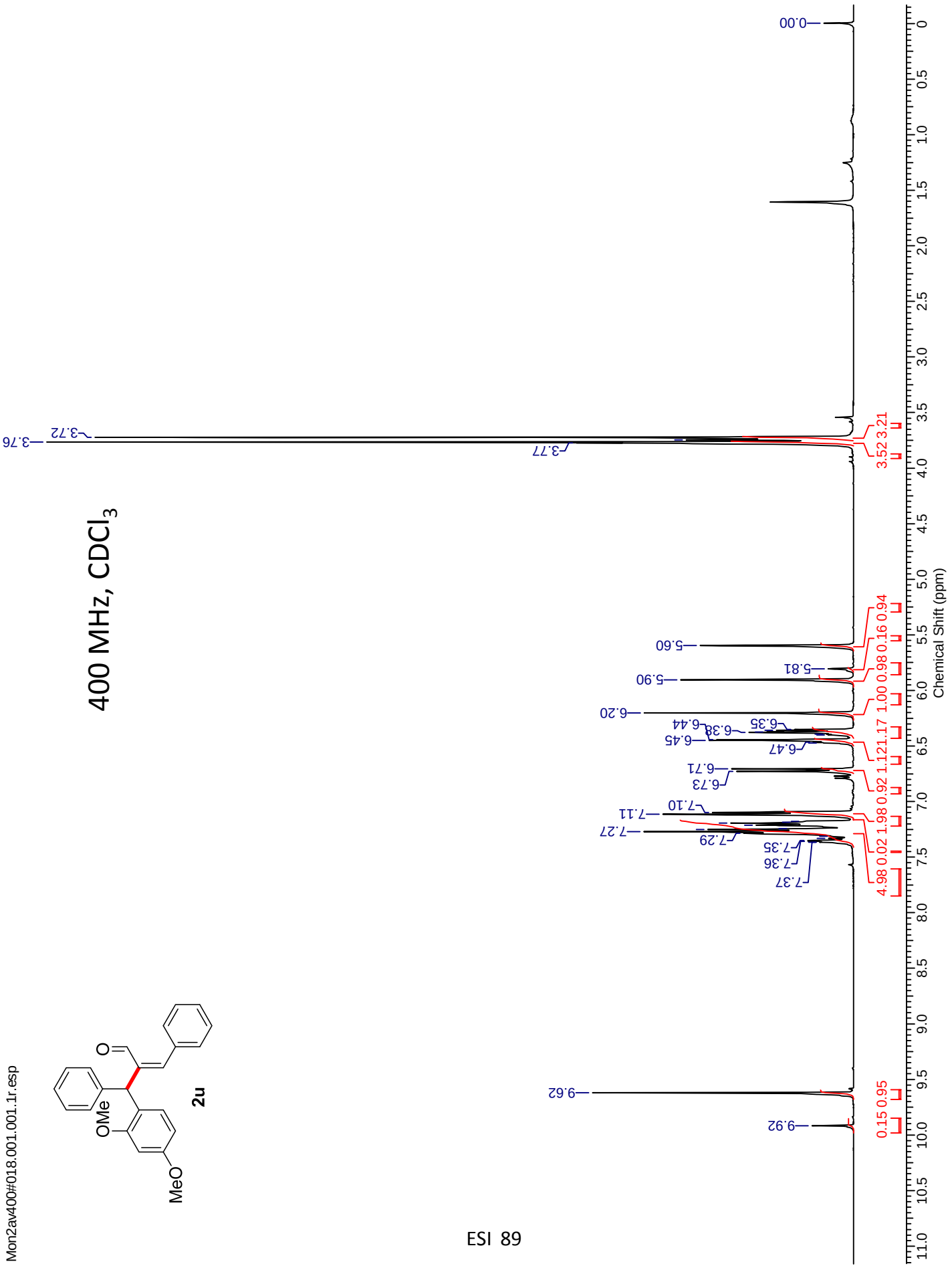


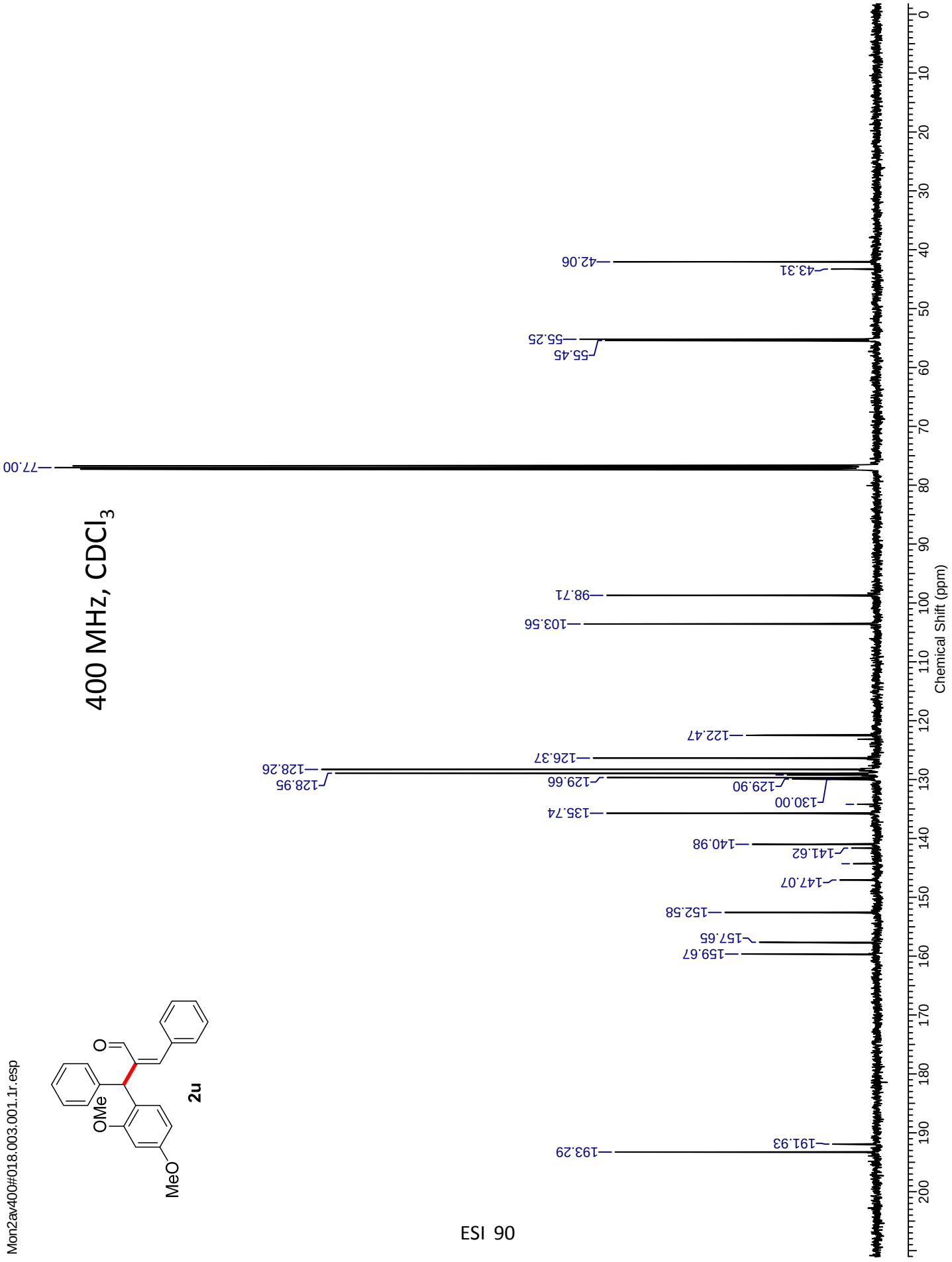
Tue2av400#003.003.001.1r.esp

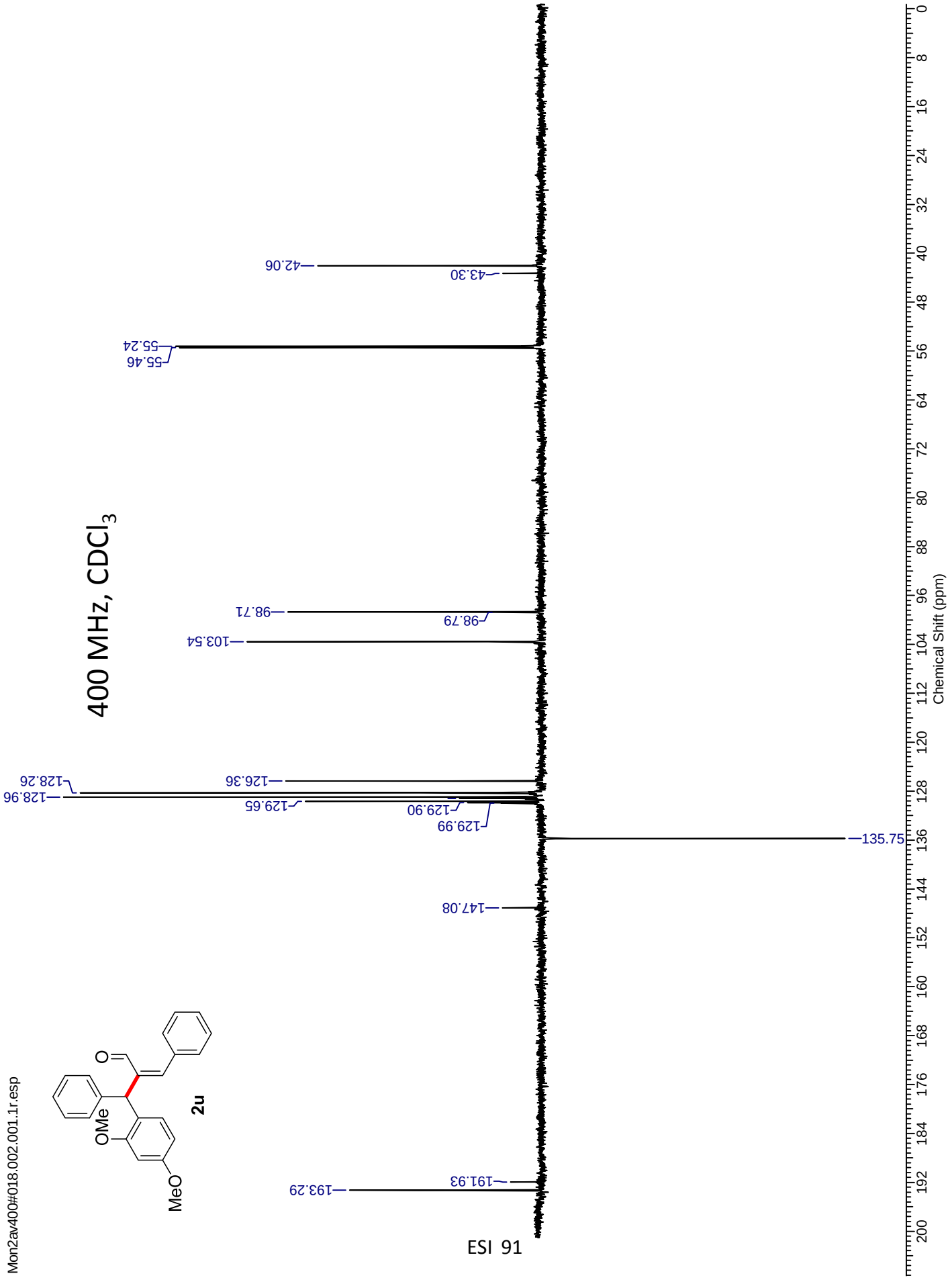

400 MHz, $CDCl_3$
191.98
148.30
130.13
129.07
128.46
128.37
126.54
113.88
113.73
79.35
55.20
49.39
MeO
O
2t
Chemical Shift (ppm)
Tue2av400#003.002.001.1r.esp

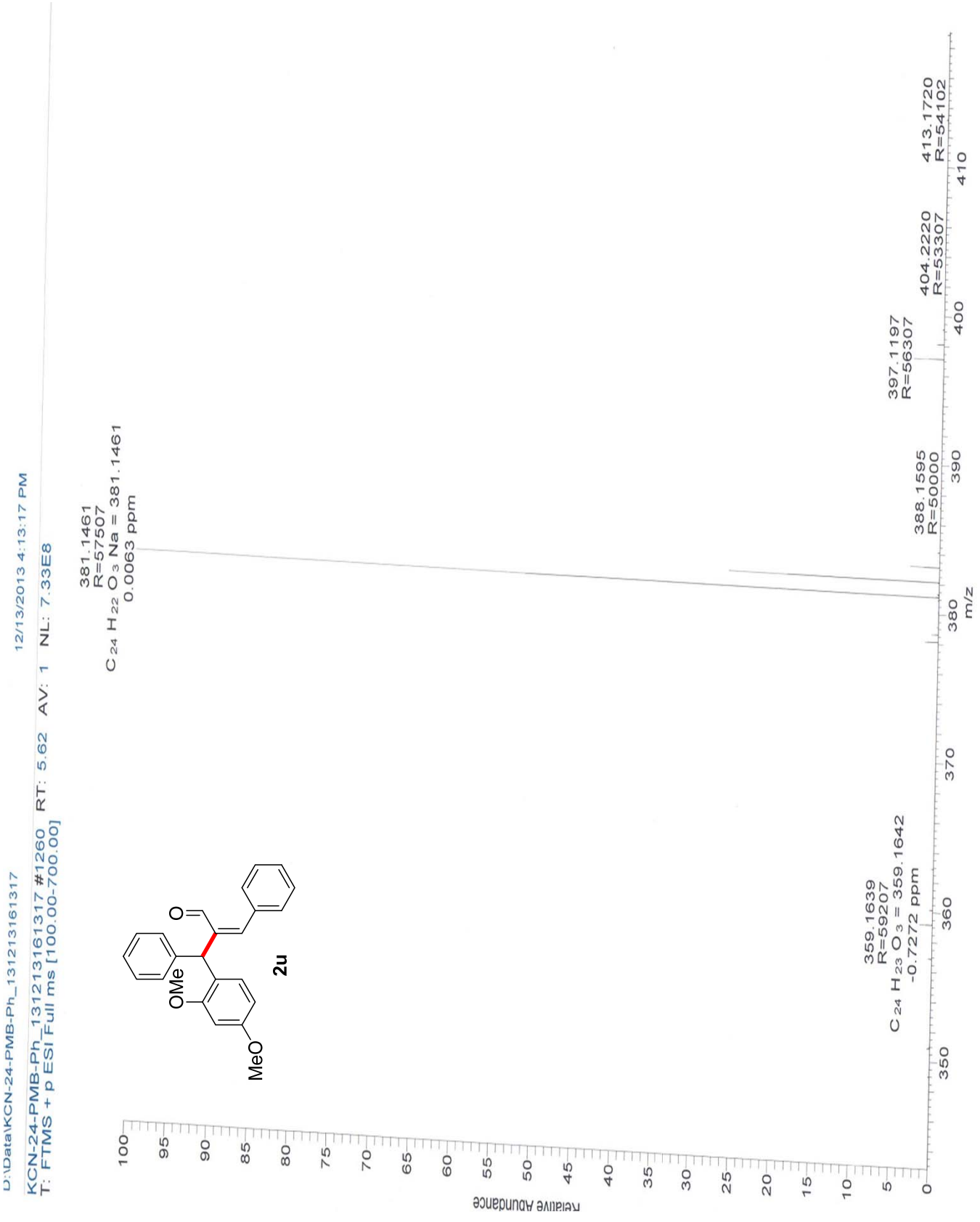
ESI 87

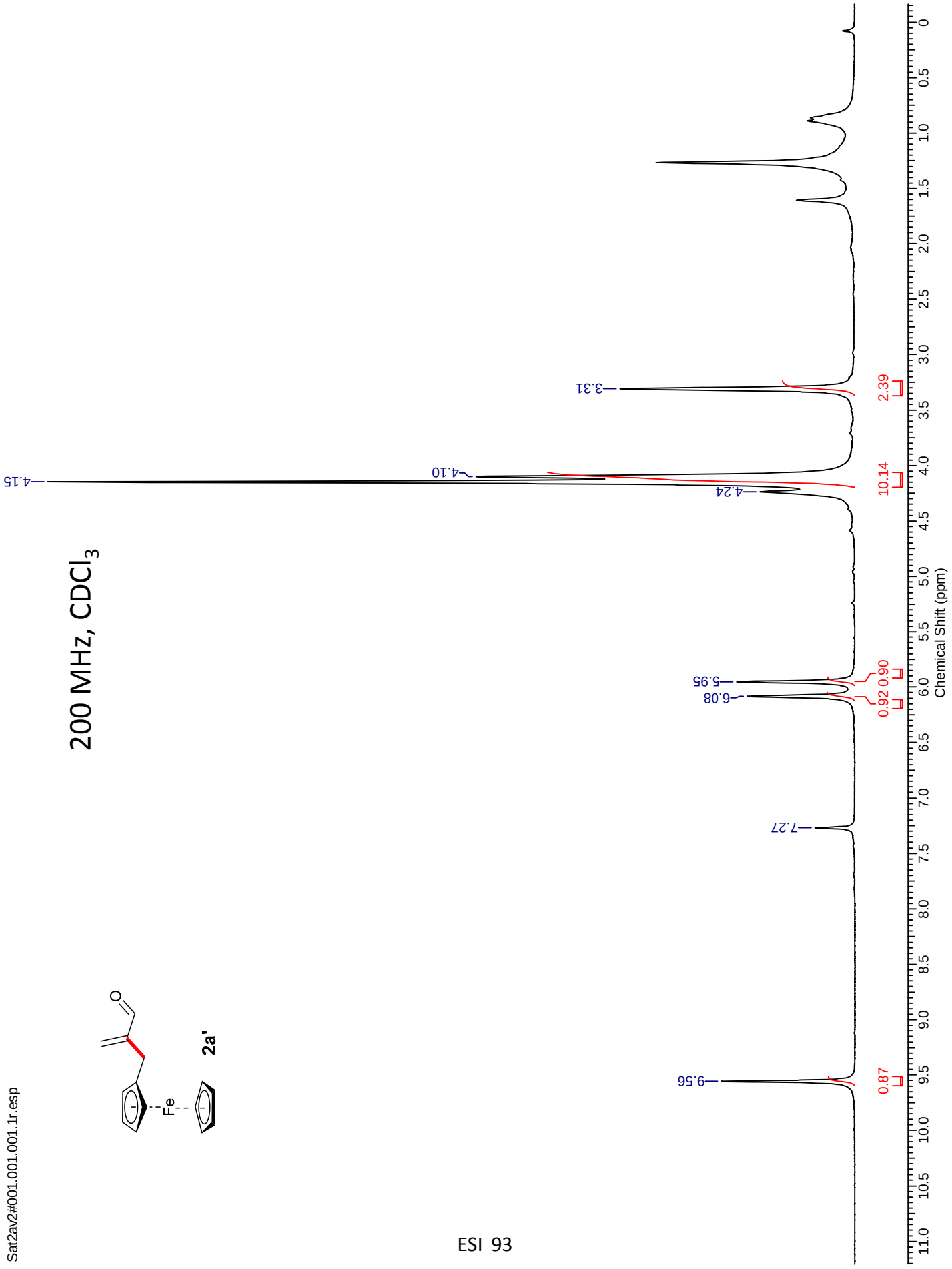

D:\Data\KCN-PMB1-Ph
12/13/2013 4:02:05 PM
KCN-PMB1-Ph #1254 RT: 5.59 AV: 1 NL: 1.77E9
T: FTMS + p ESI Full ms [100.00-700.00]
351.1354
R=59807
C 23 H 20 O 2 Na = 351.1356
-0.4987 ppm
329.1533
R=61207
C 23 H 21 O 2 = 329.1536
-1.0547 ppm
343.1692
R=54907
356.9089
R=56106
367.1090
R=58007
374.2092
R=30500
O
MeO
2t
Relative Abundance
m/z

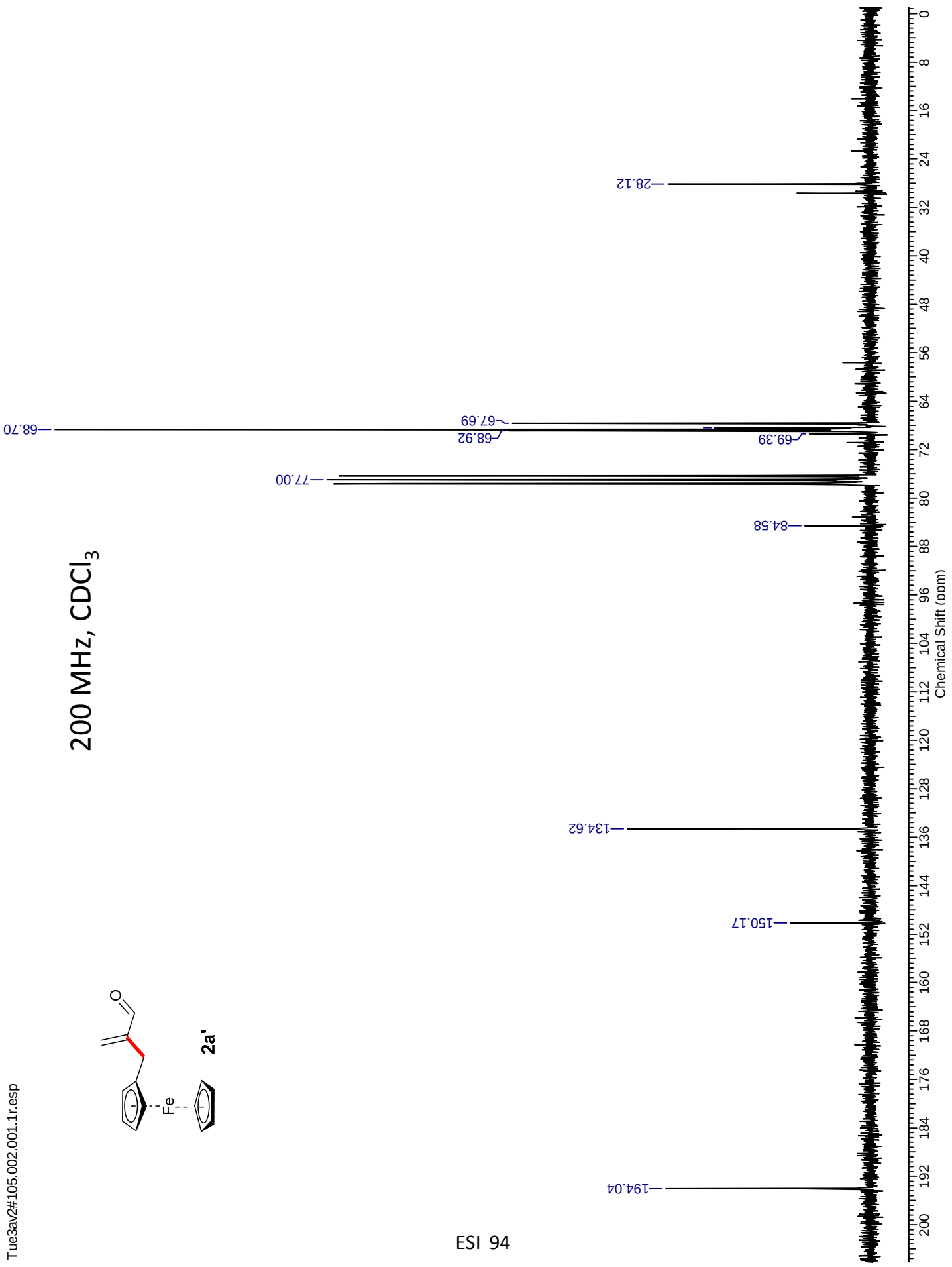

400 MHz, CDCl3
Chemical Shift (ppm)
0.00
3.72
3.76
3.77
5.60
5.81
5.90
6.20
6.35
6.38
6.44
6.45
6.47
6.71
6.73
7.10
7.11
7.27
7.29
7.35
7.36
7.37
9.62
9.92
3.52 3.21
0.16 0.94
0.98
1.00
1.12 1.17
0.92
1.98
0.02
4.98
0.15 0.95
O
OMe
MeO
2u
Mon2av400#018.001.001.1r.esp

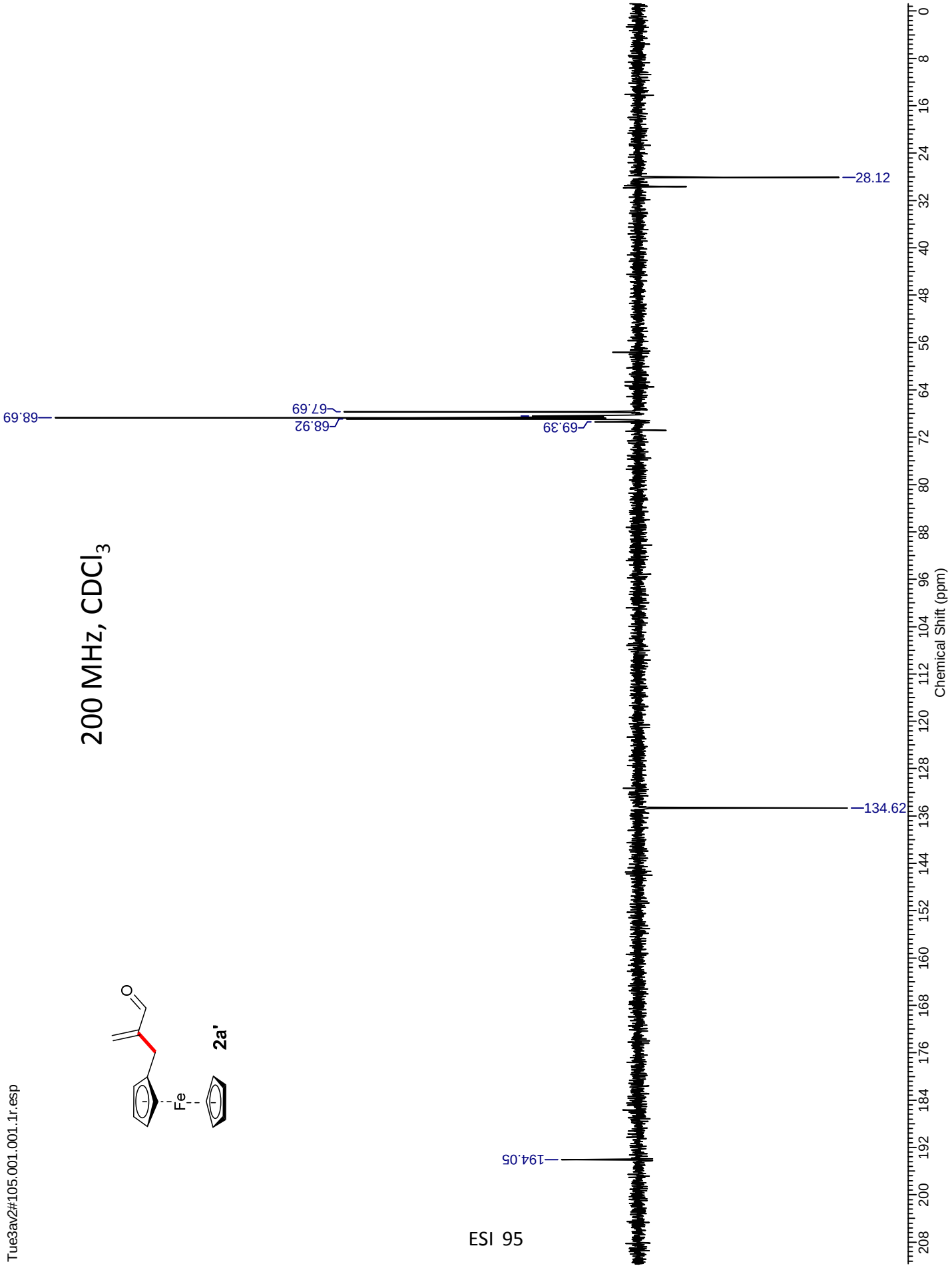

400 MHz, $CDCl_3$
77.00
42.06
43.31
55.25
55.45
98.71
103.56
122.47
126.37
128.26
128.95
129.66
129.90
130.00
135.74
140.98
141.62
147.07
152.58
157.65
159.67
191.93
193.29
Chemical Shift (ppm)
0
10
20
30
40
50
60
70
80
90
100
110
120
130
140
150
160
170
180
190
200
O
OMe
MeO
2u
Mon2av400#018.003.001.1r.esp

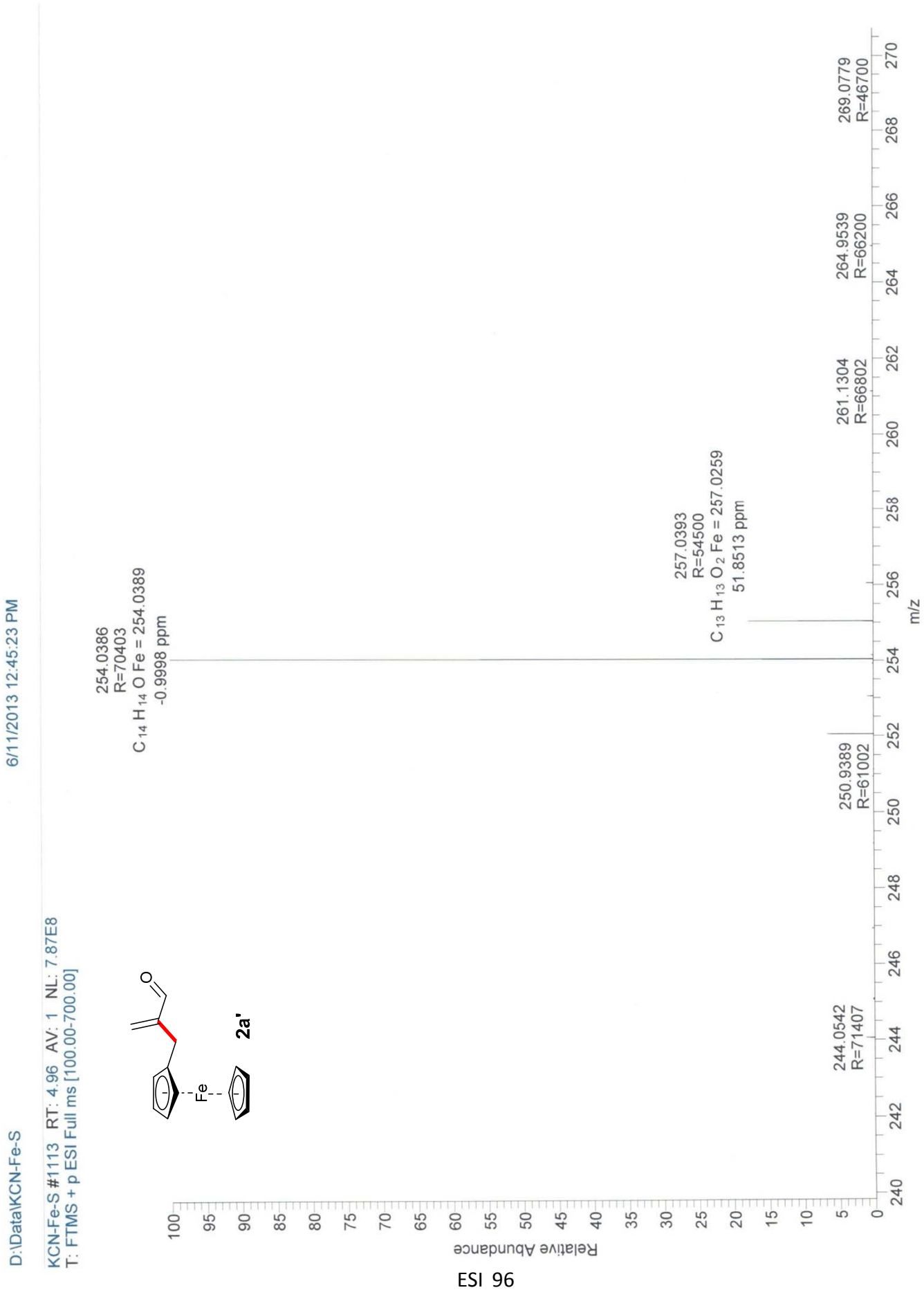


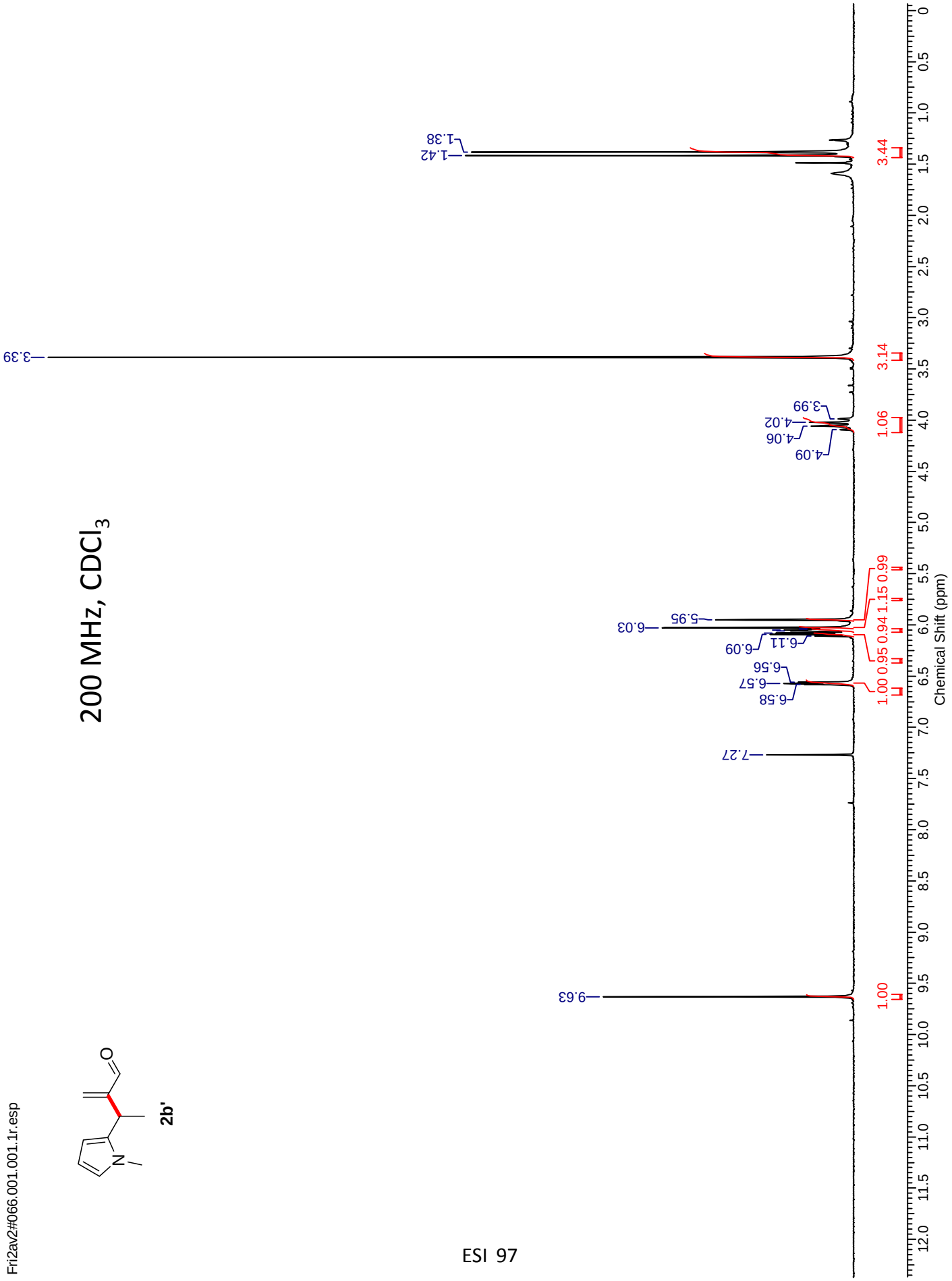

D:\Data\KCN-24-PMB-Ph_131213161317
12/13/2013 4:13:17 PM
KCN-24-PMB-Ph_131213161317 #1260 RT: 5.62 AV: 1 NL: 7.33E8
T: FTMS + p ESI Full ms [100.00-700.00]
381.1461
R=57507
C 24 H 22 O 3 Na = 381.1461
0.0063 ppm
359.1639
R=59207
C 24 H 23 O 3 = 359.1642
-0.7272 ppm
388.1595
R=50000
397.1197
R=56307
404.2220
R=53307
413.1720
R=54102
OMe
MeO
O
2u
Relative Abundance
m/z

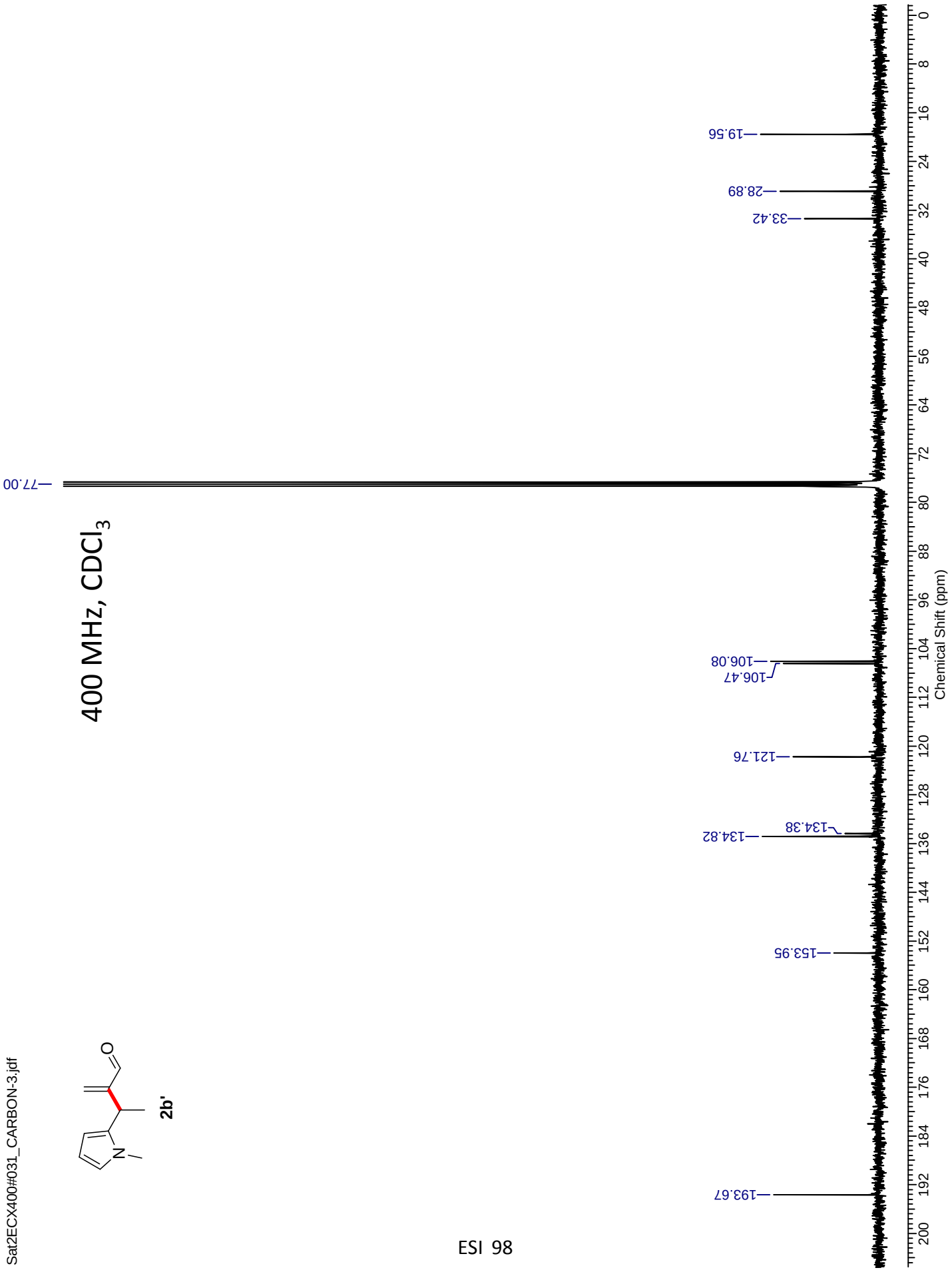

200 MHz, CDCl3
9.56
7.27
6.08
5.95
4.24
4.10
4.15
3.31
0.87
0.92
0.90
2.39
10.14
Chemical Shift (ppm)
Fe
2a'
Sat2av2#001.001.001.1r.esp

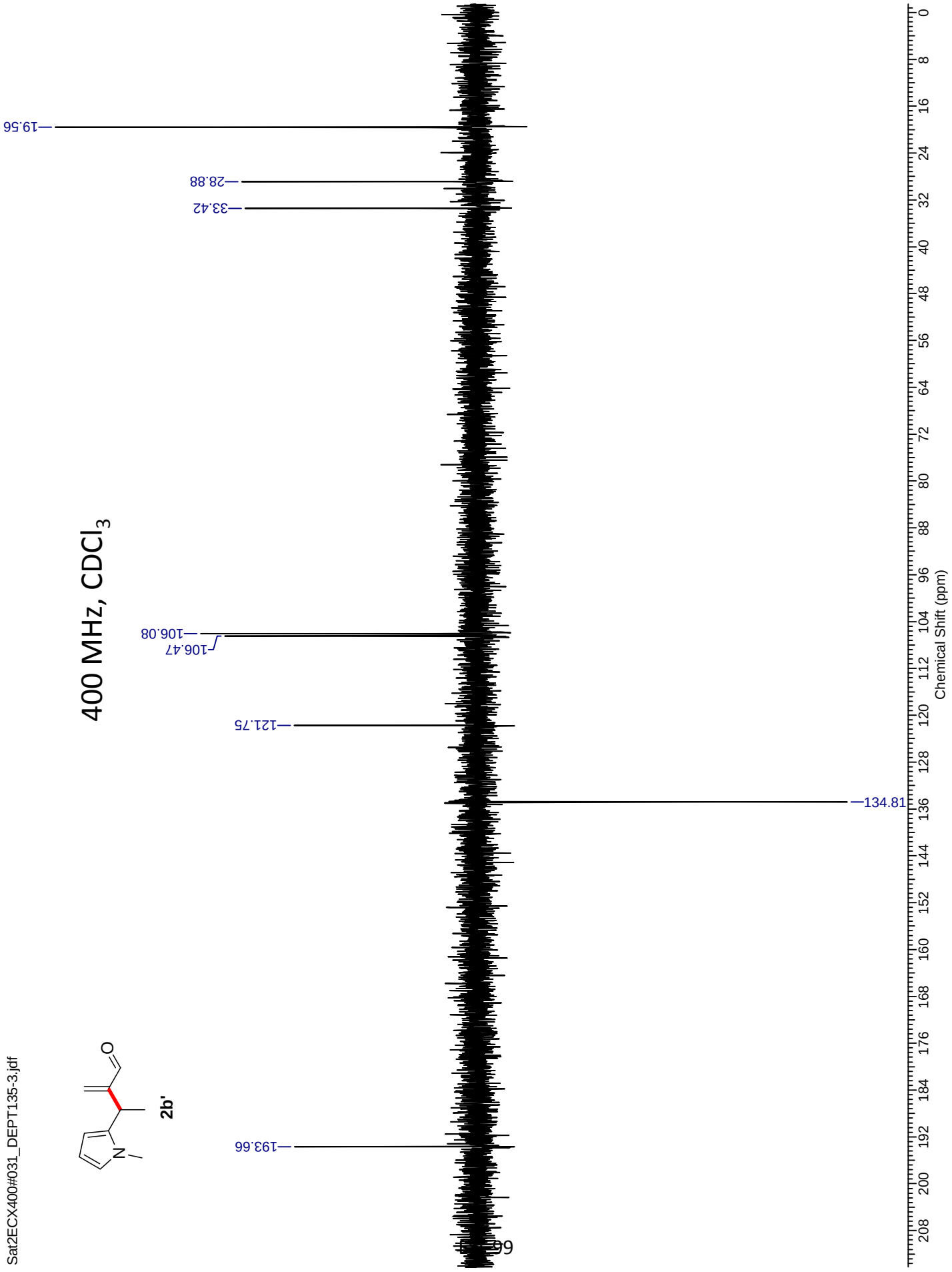


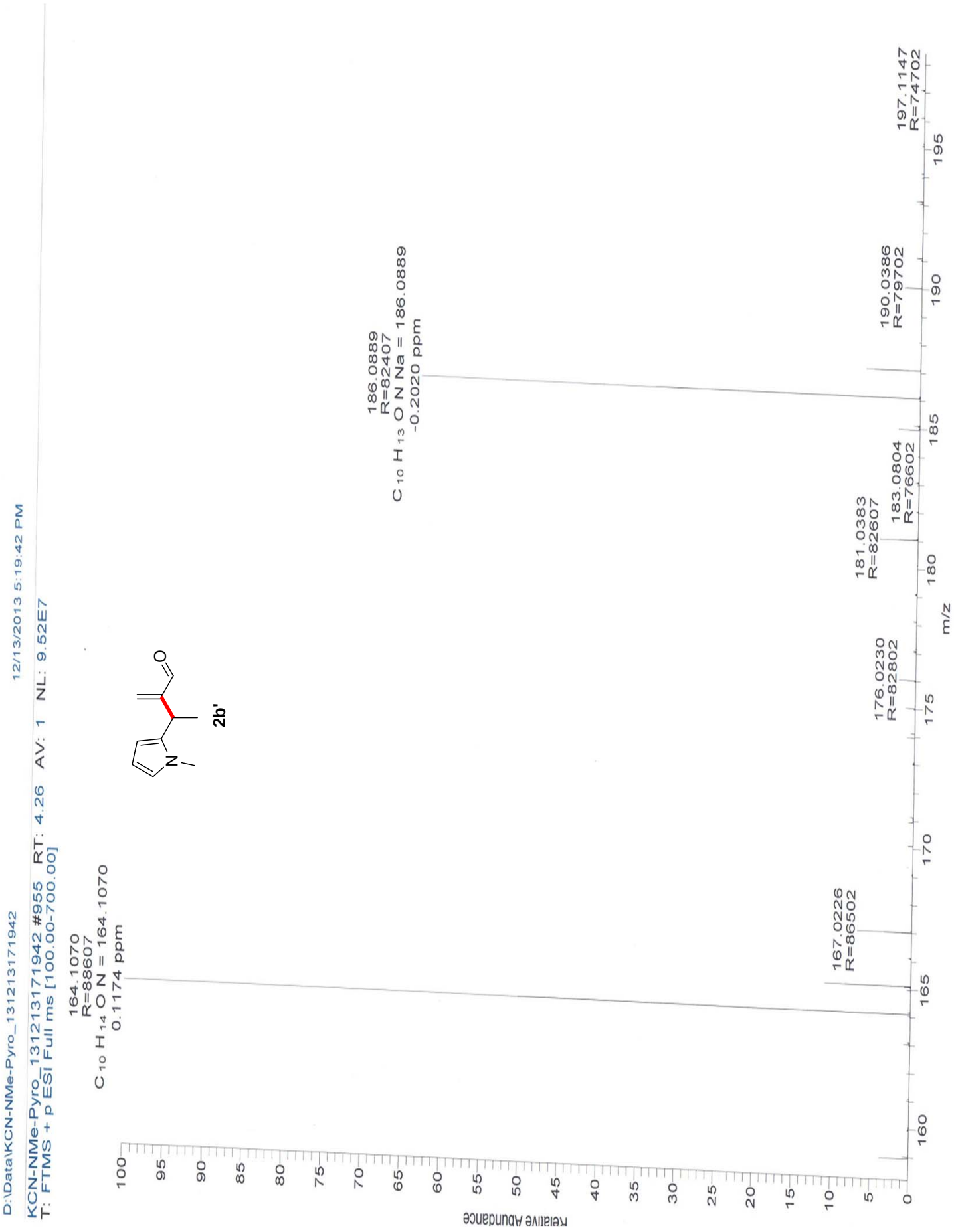


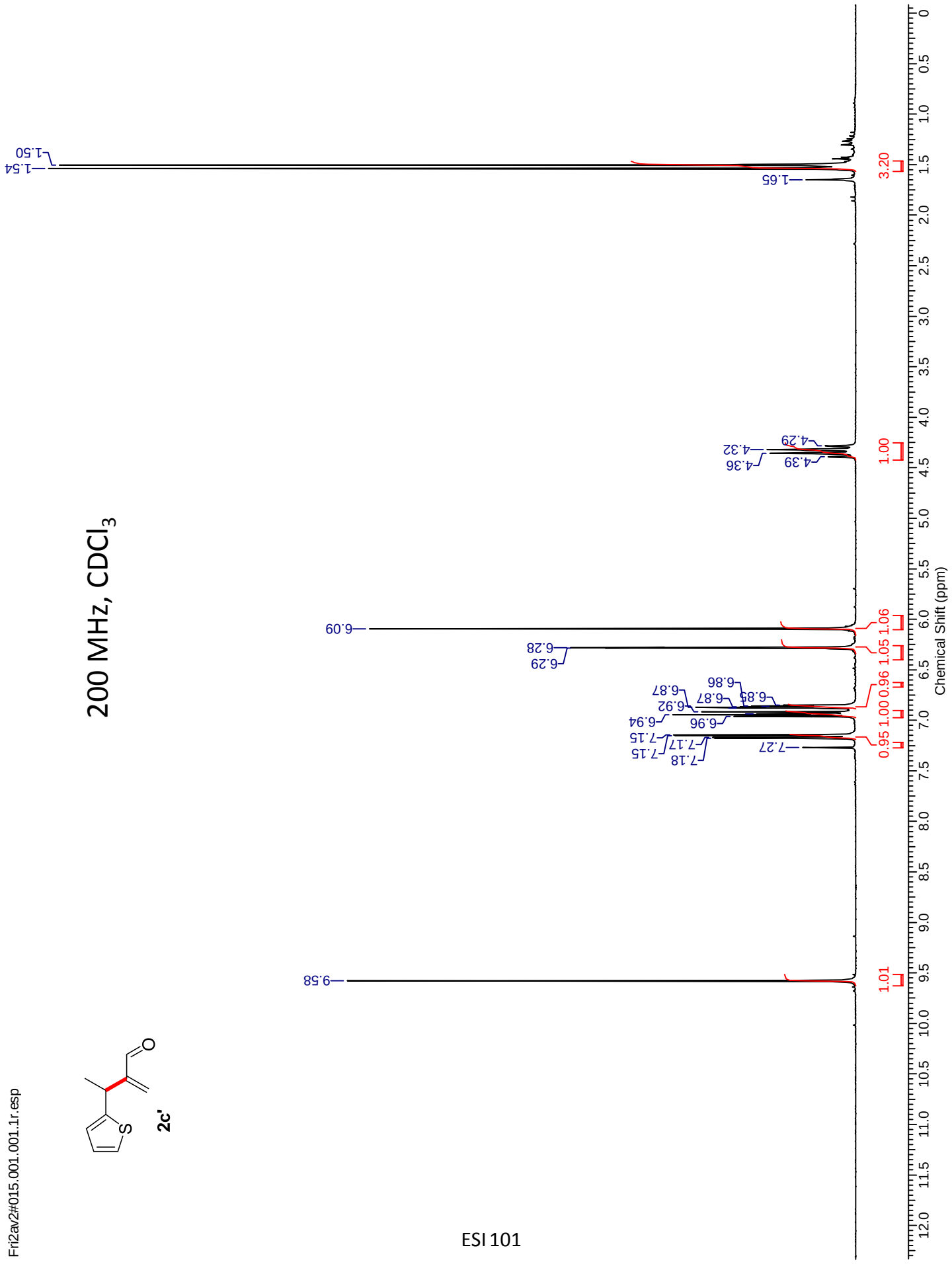

D:\Data\KCN-Fe-S
6/11/2013 12:45:23 PM
KCN-Fe-S #1113 RT: 4.96 AV: 1 NL: 7.87E8
T: FTMS + p ESI Full ms [100.00-700.00]
254.0386
R=70403
C 14 H 14 O Fe = 254.0389
-0.9998 ppm
257.0393
R=54500
C 13 H 13 O 2 Fe = 257.0259
51.8513 ppm
244.0542
R=71407
250.9389
R=61002
261.1304
R=66802
264.9539
R=66200
269.0779
R=46700
Fe
2a'
Relative Abundance
m/z

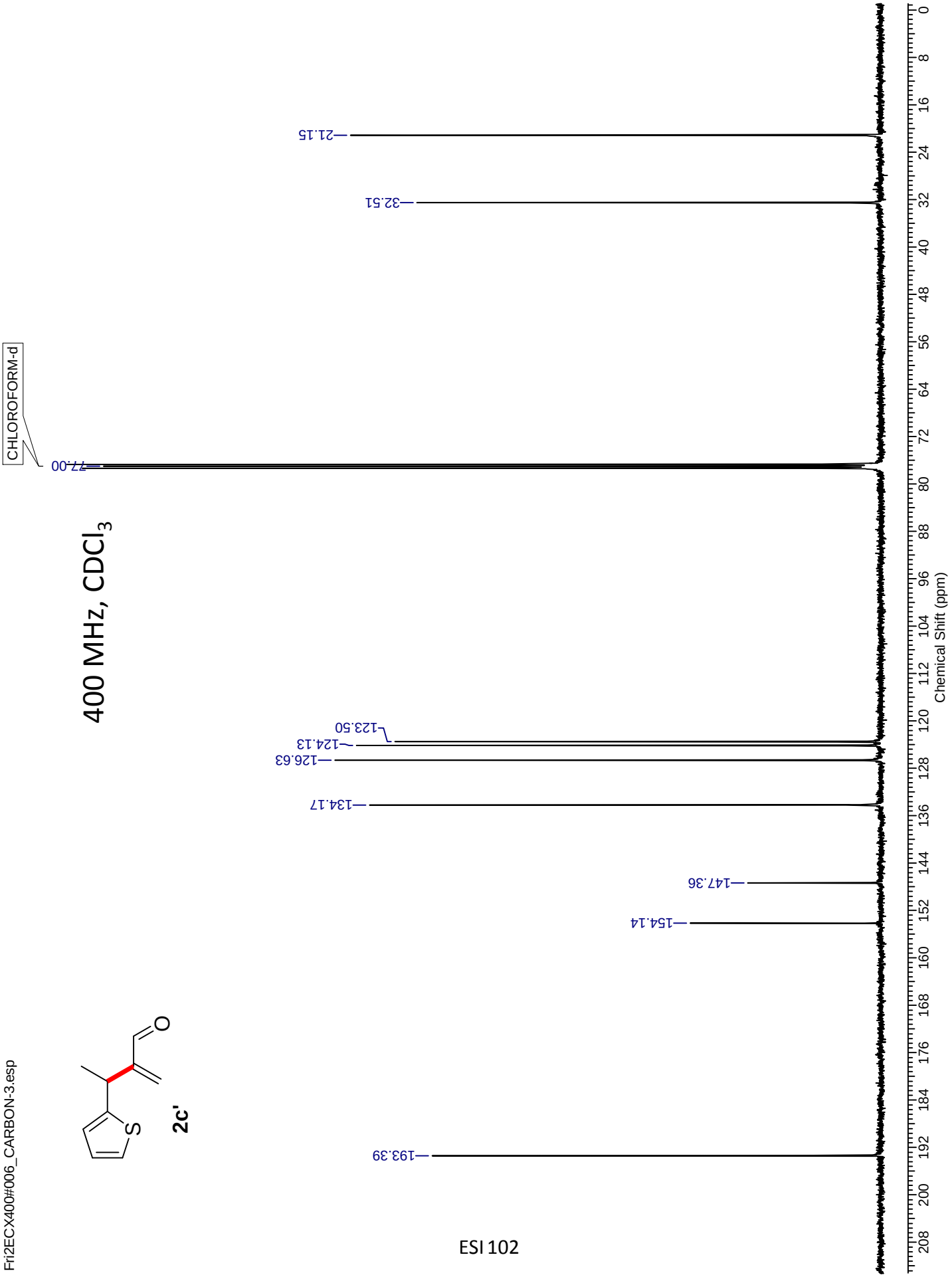

200 MHz, CDCl3
Fri2av2#066.001.001.1r.esp
2b'
O
N
9.63
7.27
6.58
6.57
6.56
6.11
6.09
6.03
5.95
4.09
4.06
4.02
3.99
3.39
1.42
1.38
1.00
0.95
0.94
1.15
0.99
1.06
3.14
3.44
Chemical Shift (ppm)

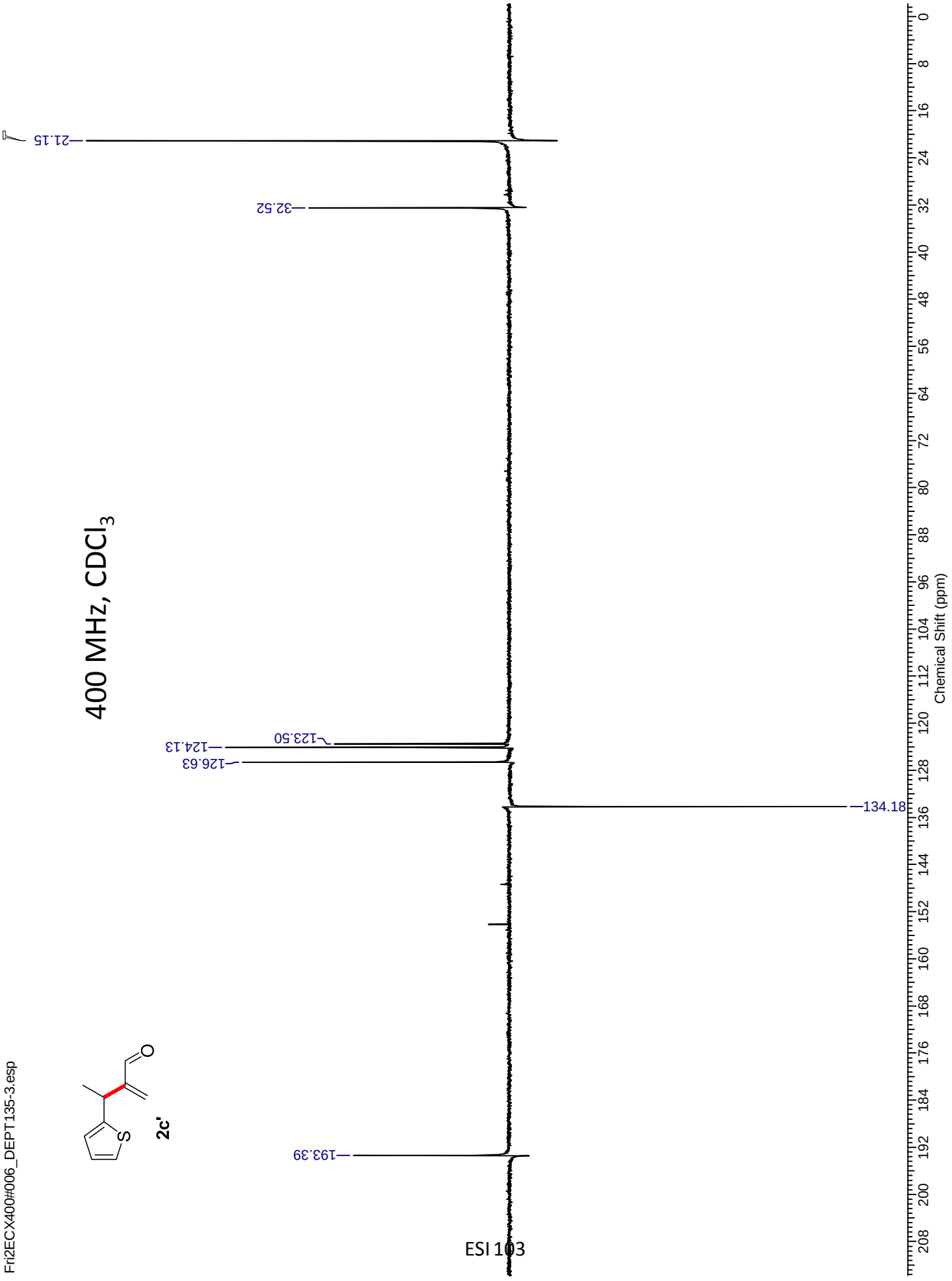

400 MHz, CDCl3
Sat2ECX400#031_CARBON-3.jdf
2b'
O
N
77.00
19.56
28.89
33.42
106.08
106.47
121.76
134.38
134.82
153.95
193.67
Chemical Shift (ppm)
0
8
16
24
32
40
48
56
64
72
80
88
96
104
112
120
128
136
144
152
160
168
176
184
192
200

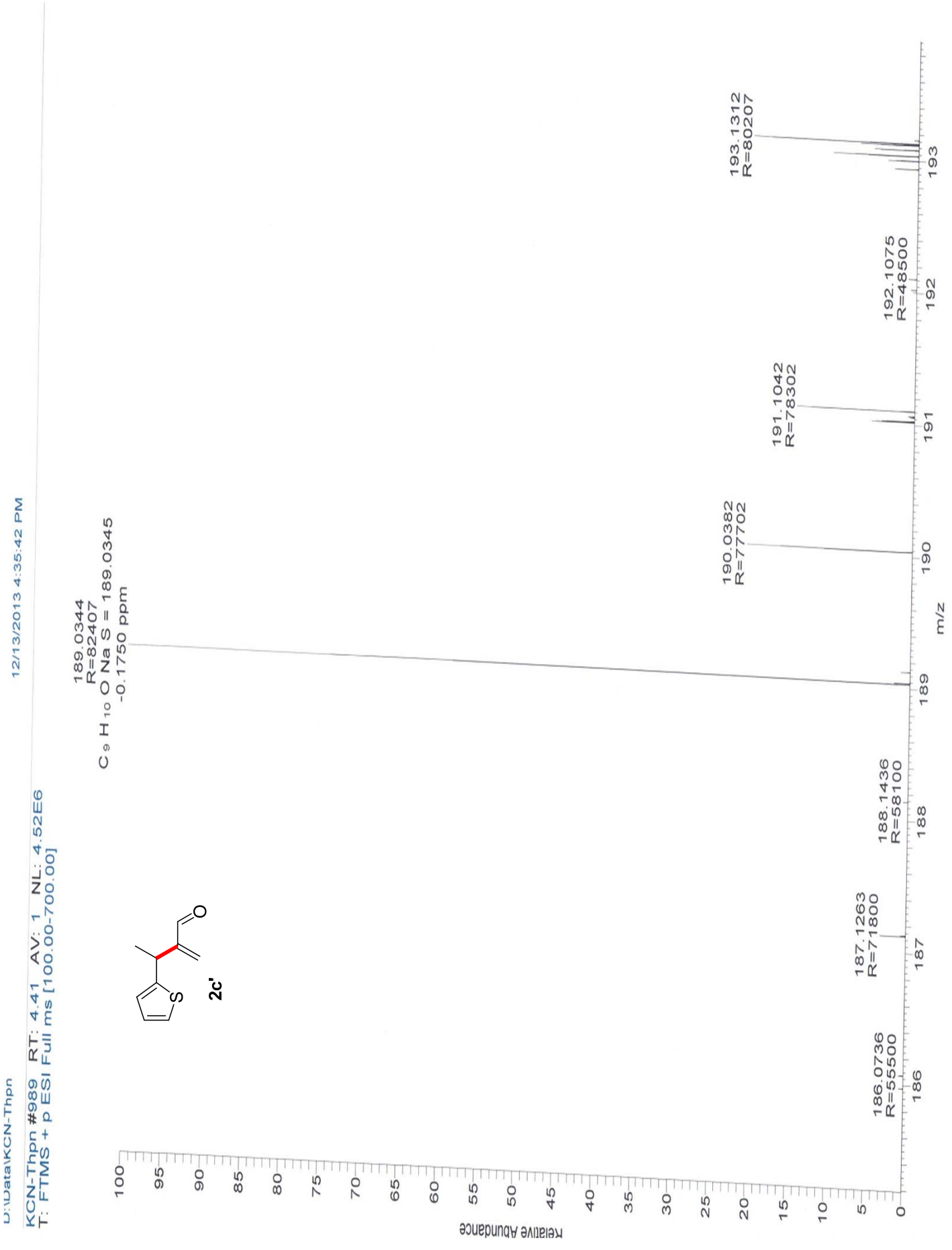

19.56
28.88
33.42
106.08
106.47
121.75
134.81
193.66
400 MHz, CDCl3
2b'
O
N
Sat2ECX400#031_DEPT135-3.jdf
Chemical Shift (ppm)
0
8
16
24
32
40
48
56
64
72
80
88
96
104
112
120
128
136
144
152
160
168
176
184
192
200
208

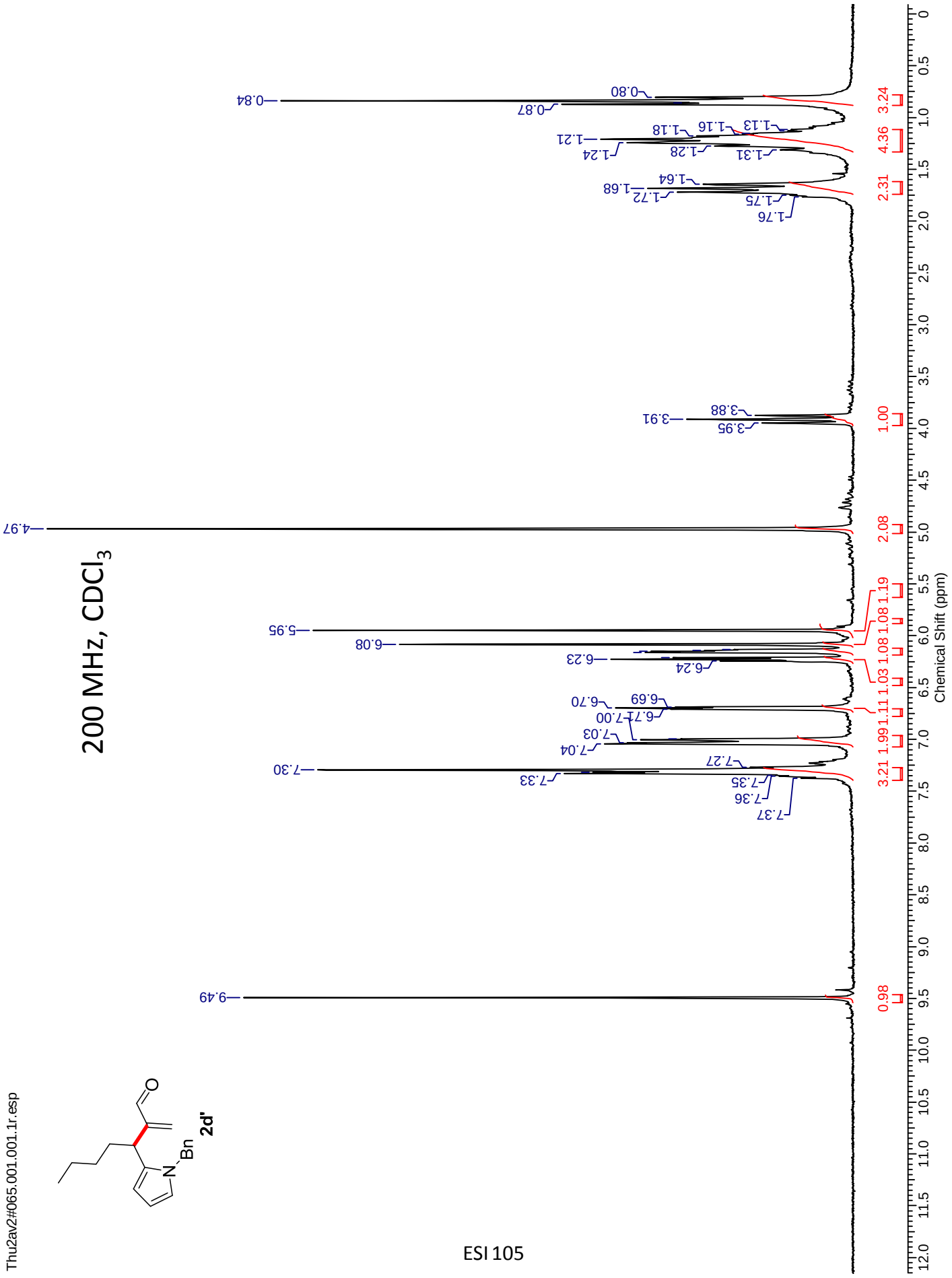

D:\Data\KCN-NMe-Pyro_131213171942
12/13/2013 5:19:42 PM
KCN-NMe-Pyro_131213171942 #955 RT: 4.26 AV: 1 NL: 9.52E7
T: FTMS + p ESI Full ms [100.00-700.00]
164.1070
R=88607
C10 H14 O N = 164.1070
0.1174 ppm
186.0889
R=82407
C10 H13 O N Na = 186.0889
-0.2020 ppm
2b'
O
N
167.0226
R=86502
176.0230
R=82802
181.0383
R=82607
183.0804
R=76602
190.0386
R=79702
197.1147
R=74702
Relative Abundance
m/z

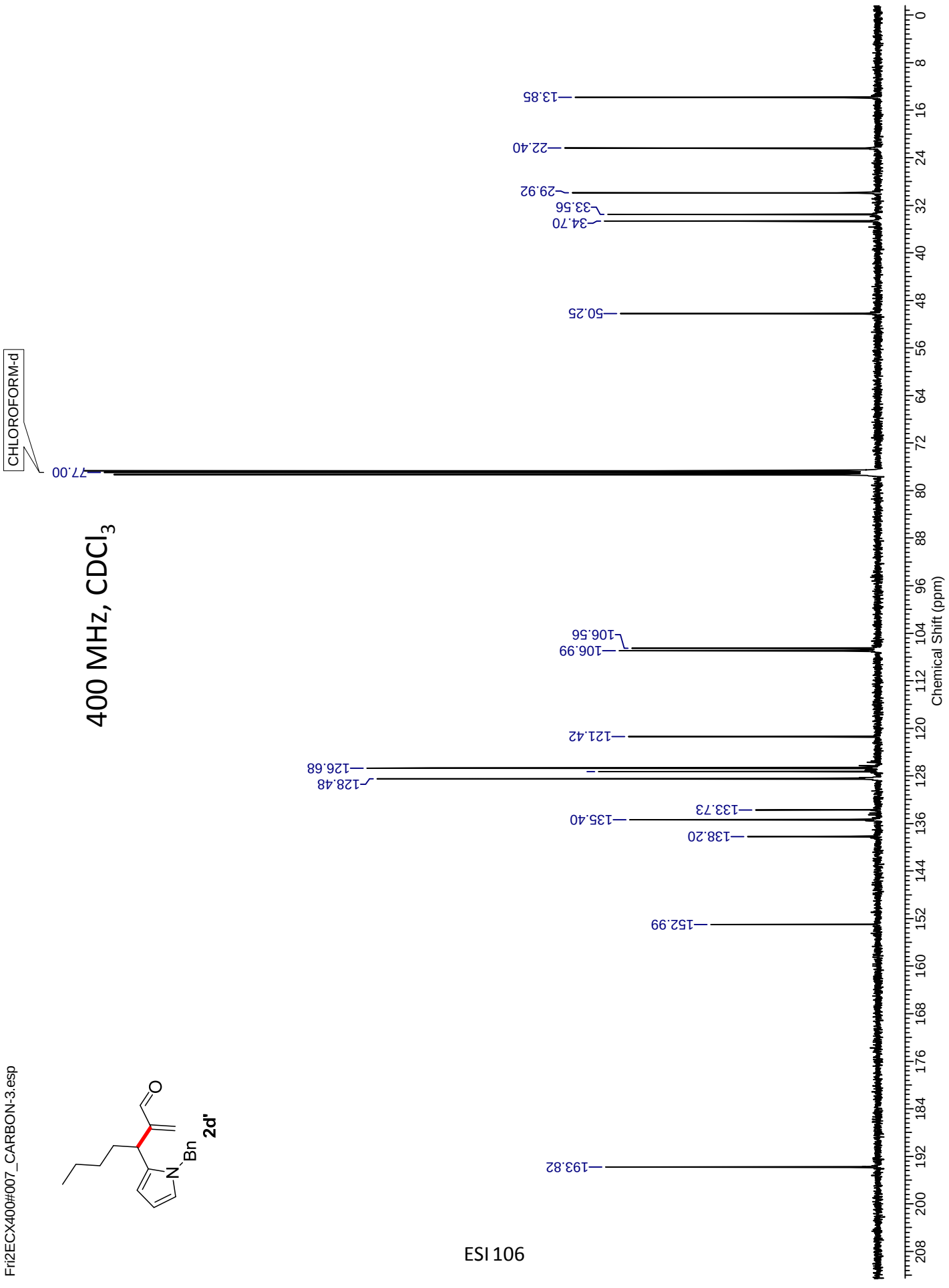

200 MHz, CDCl3
1.54
1.50
1.65
4.29
4.32
4.36
4.39
6.09
6.28
6.29
6.85
6.86
6.87
6.87
6.92
6.94
6.96
7.15
7.15
7.17
7.18
7.27
9.58
3.20
1.00
1.06
1.05
0.96
1.00
0.95
1.01
Chemical Shift (ppm)
0
0.5
1.0
1.5
2.0
2.5
3.0
3.5
4.0
4.5
5.0
5.5
6.0
6.5
7.0
7.5
8.0
8.5
9.0
9.5
10.0
10.5
11.0
11.5
12.0
O
S
2c'
Fri2av2#015.001.001.1r.esp

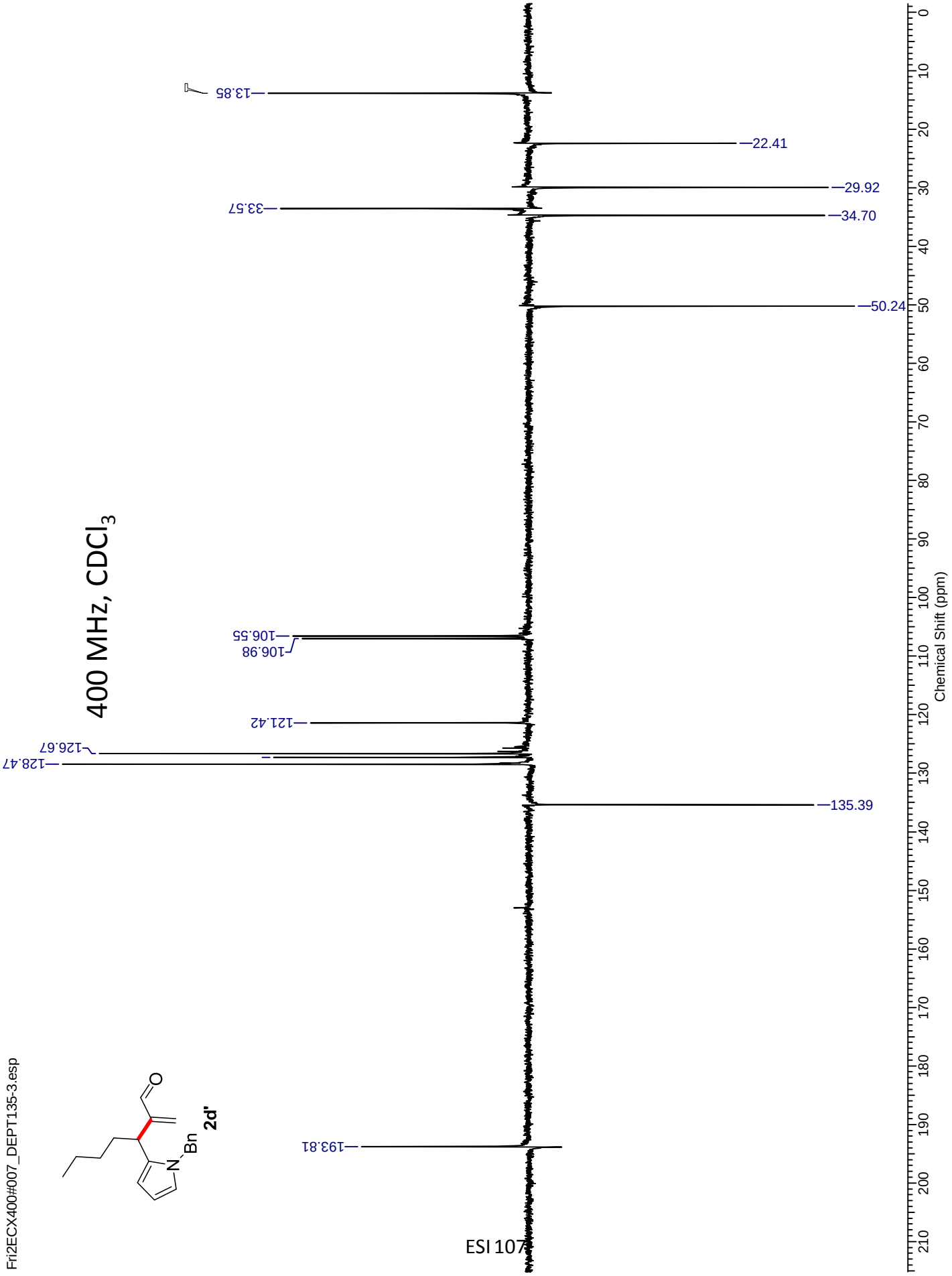

Fri2ECX400#006_CARBON-3.esp
400 MHz, $CDCl_3$
CHLOROFORM-d
77.00
21.15
32.51
123.50
124.13
126.63
134.17
147.36
154.14
193.39
O
S
2c'
ESI 102
Chemical Shift (ppm)
0
8
16
24
32
40
48
56
64
72
80
88
96
104
112
120
128
136
144
152
160
168
176
184
192
200
208

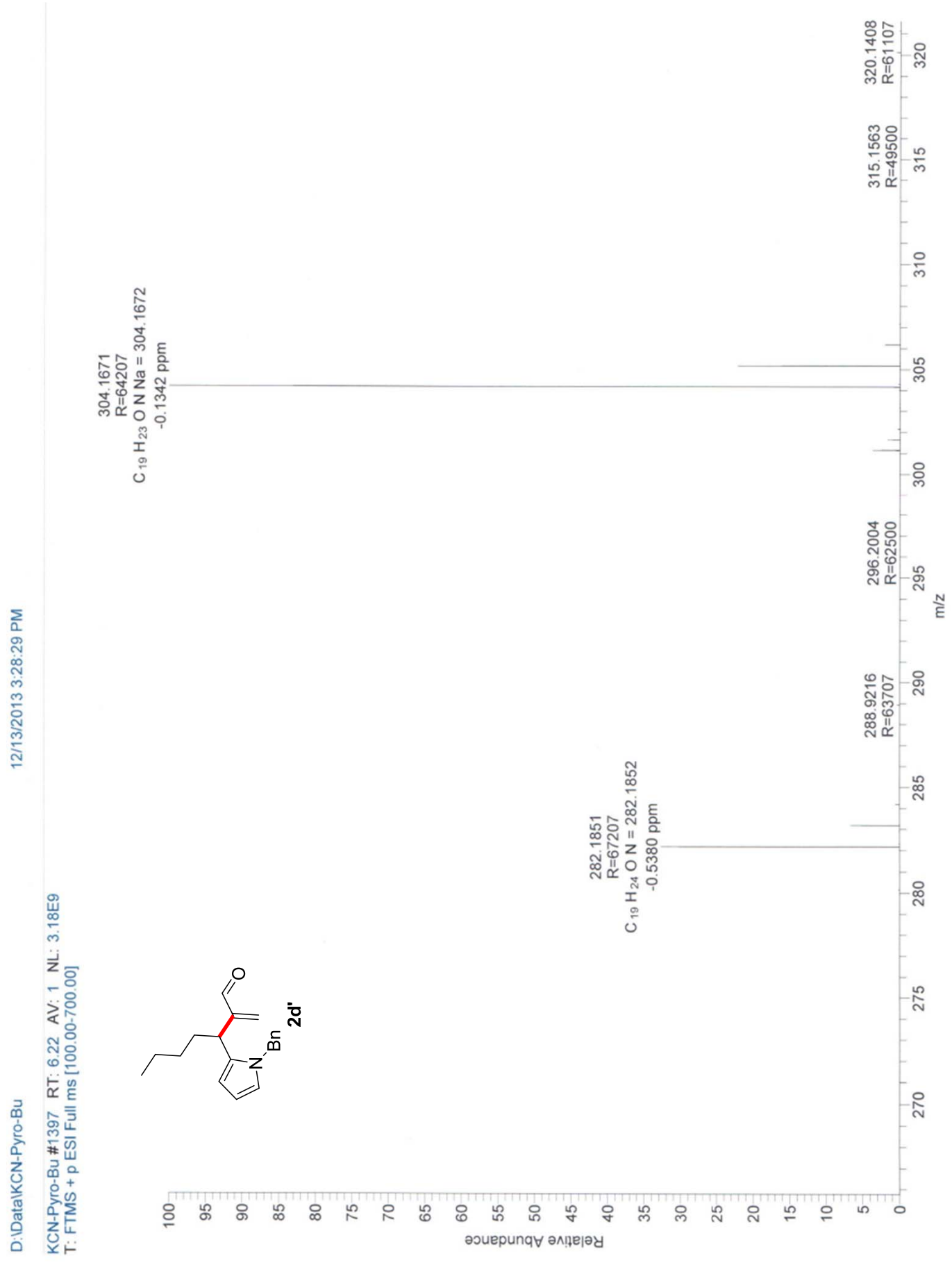

21.15
32.52
123.50
124.13
126.63
134.18
193.39
400 MHz, CDCl3
2c'
O
S
Fri2ECX400#006_DEPT135-3.esp
ESI 103
Chemical Shift (ppm)
0
8
16
24
32
40
48
56
64
72
80
88
96
104
112
120
128
136
144
152
160
168
176
184
192
200
208

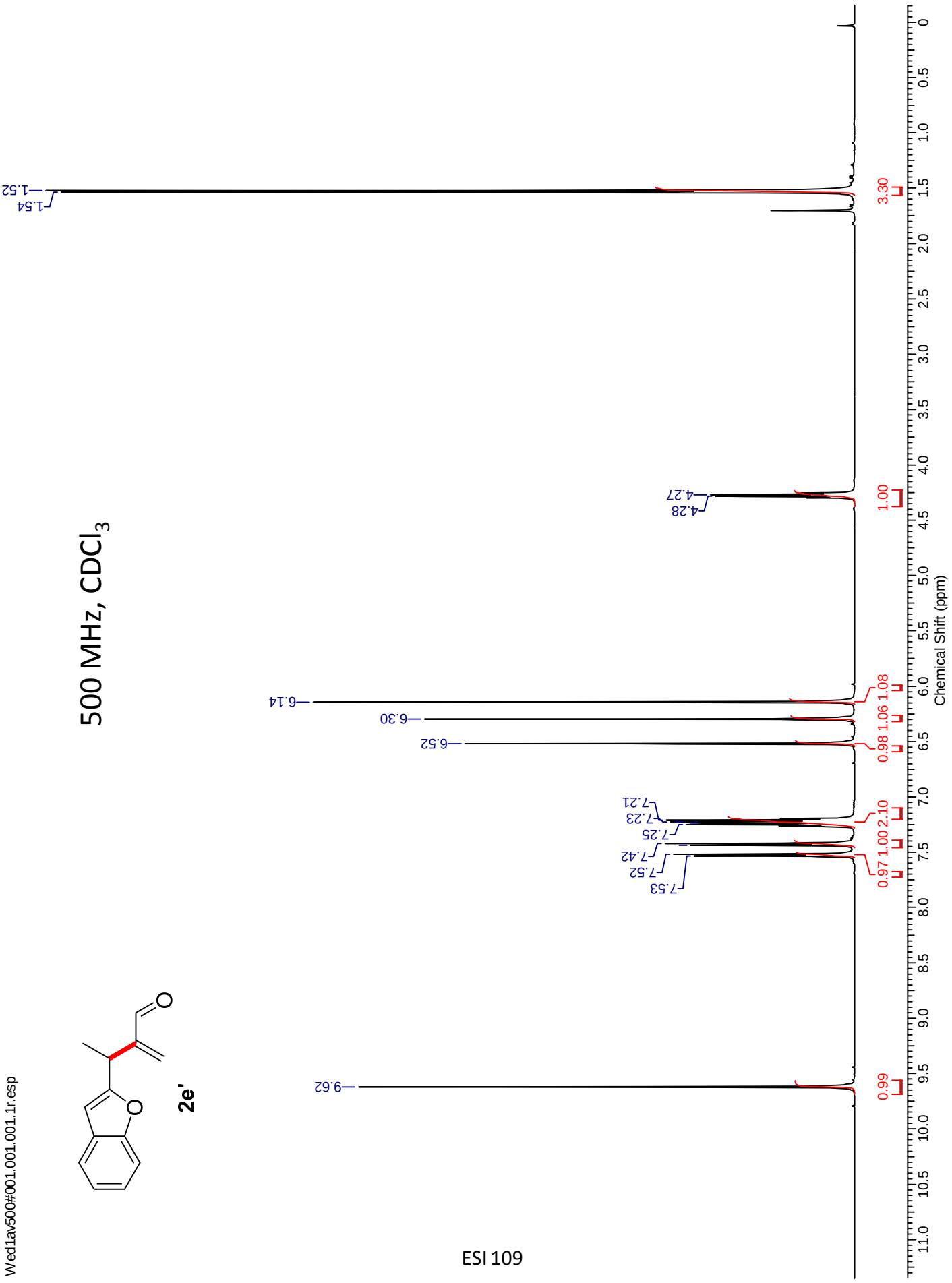

D:\Data\KCN-Thpn
12/13/2013 4:35:42 PM
KCN-Thpn #989 RT: 4.41 AV: 1 NL: 4.52E6
T: FTMS + p ESI Full ms [100.00-700.00]
189.0344
R=82407
C 9 H 10 O Na S = 189.0345
-0.1750 ppm
190.0382
R=77702
191.1042
R=78302
192.1075
R=48500
193.1312
R=80207
188.1436
R=58100
187.1263
R=71800
186.0736
R=55500
2c'
Relative Abundance
m/z

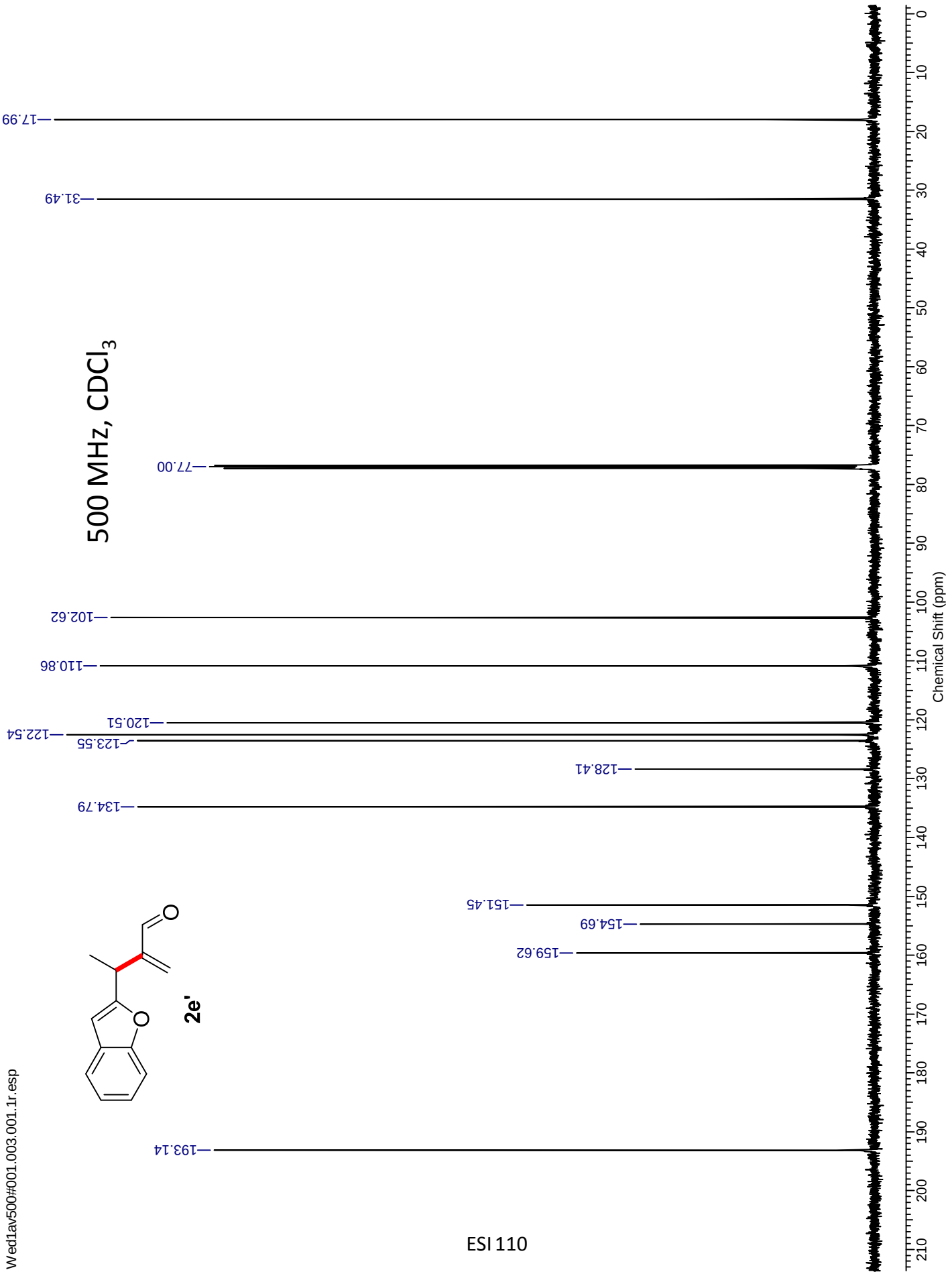


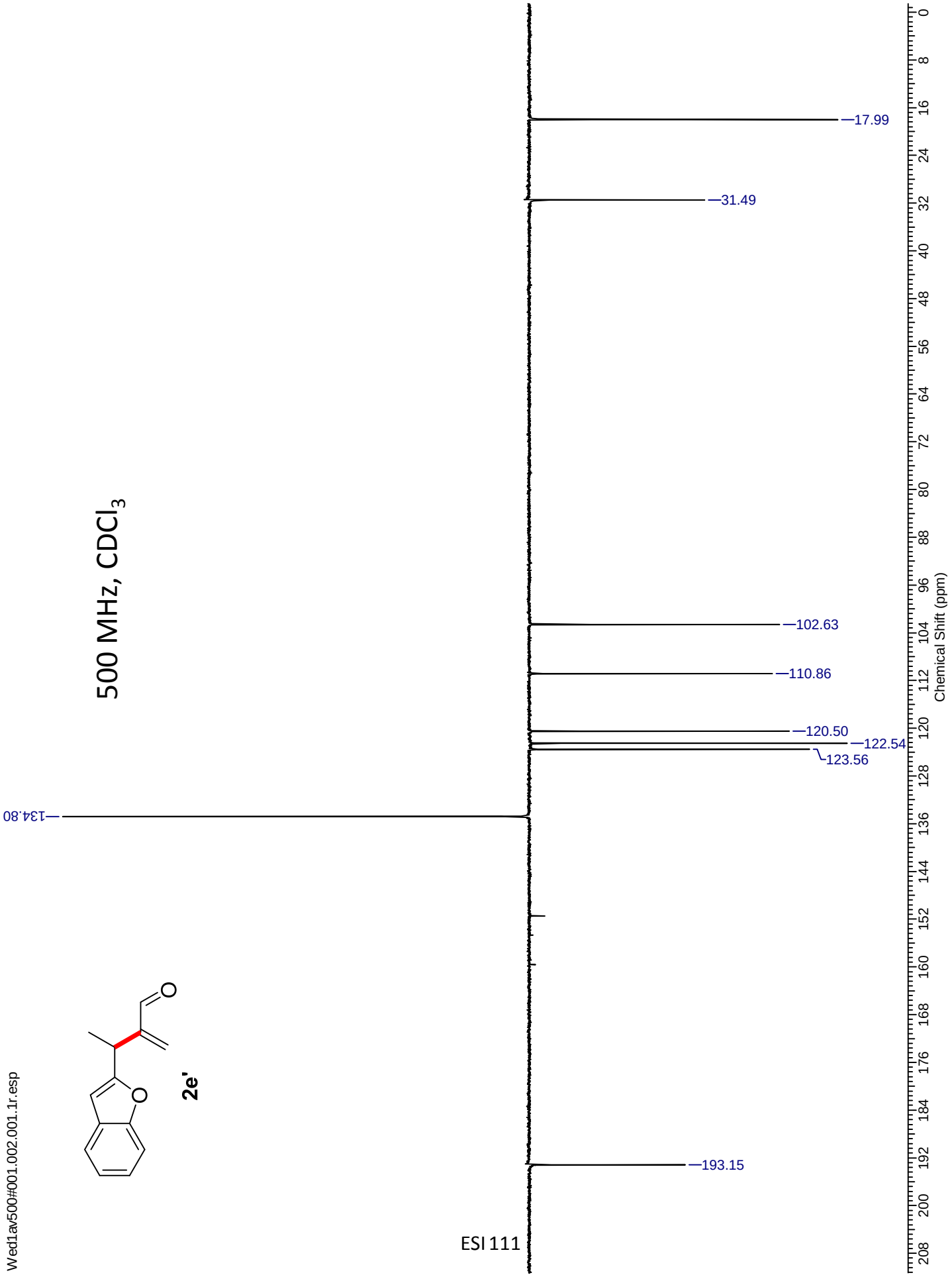


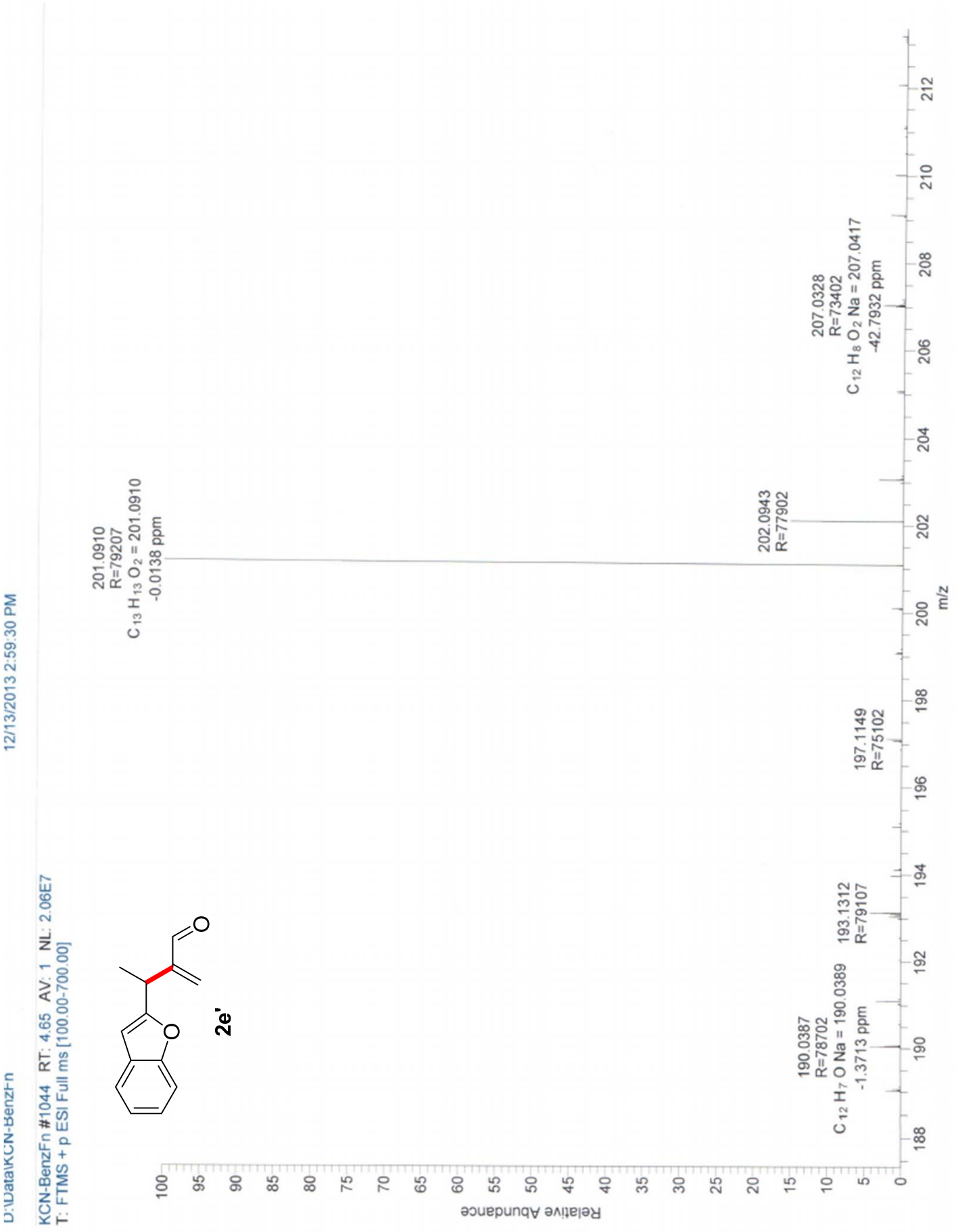

400 MHz, CDCl3
13.85
22.41
29.92
33.57
34.70
50.24
106.55
106.98
121.42
126.67
128.47
135.39
193.81
Chemical Shift (ppm)
Fri2ECX400#007_DEPT135-3.esp
ESI 107
2d'
Bn
N
O

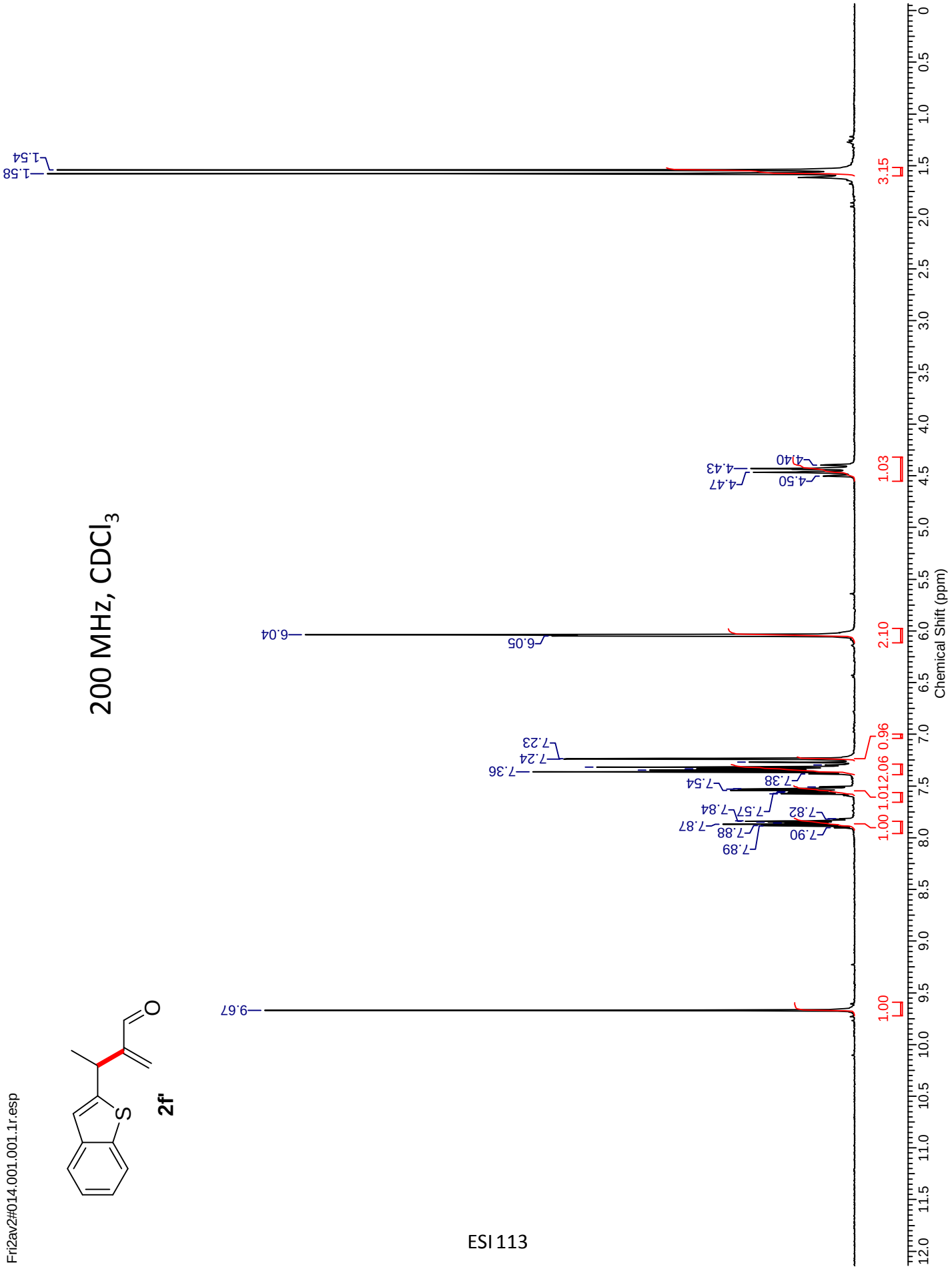

D:\Data\KCN-Pyro-Bu
12/13/2013 3:28:29 PM
KCN-Pyro-Bu #1397 RT: 6.22 AV: 1 NL: 3.18E9
T: FTMS + p ESI Full ms [100.00-700.00]
304.1671
R=64207
C 19 H 23 O N Na = 304.1672
-0.1342 ppm
282.1851
R=67207
C 19 H 24 O N = 282.1852
-0.5380 ppm
288.9216
R=63707
296.2004
R=62500
315.1563
R=49500
320.1408
R=61107
2d'
Bn
N
O
Relative Abundance
m/z

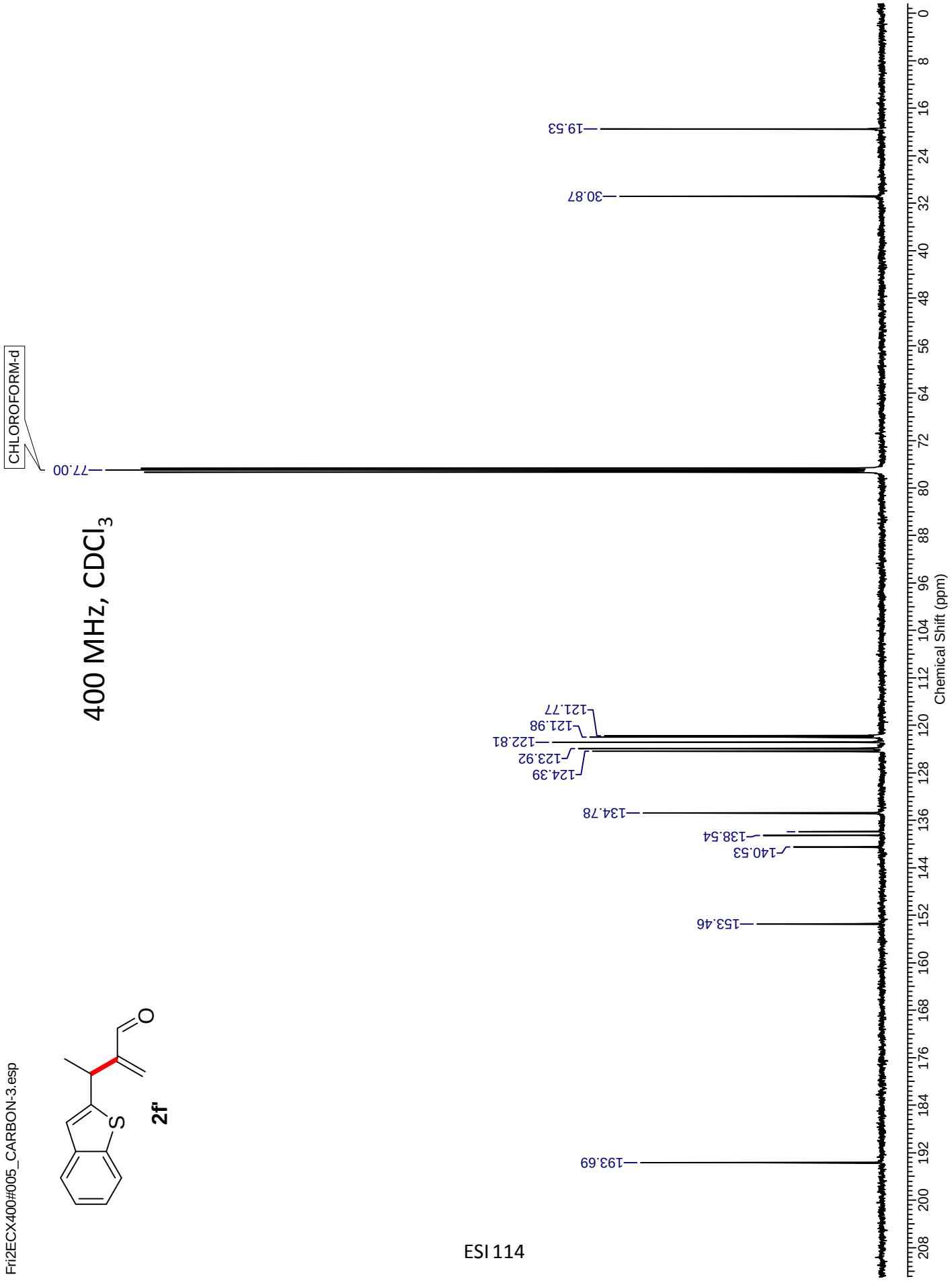


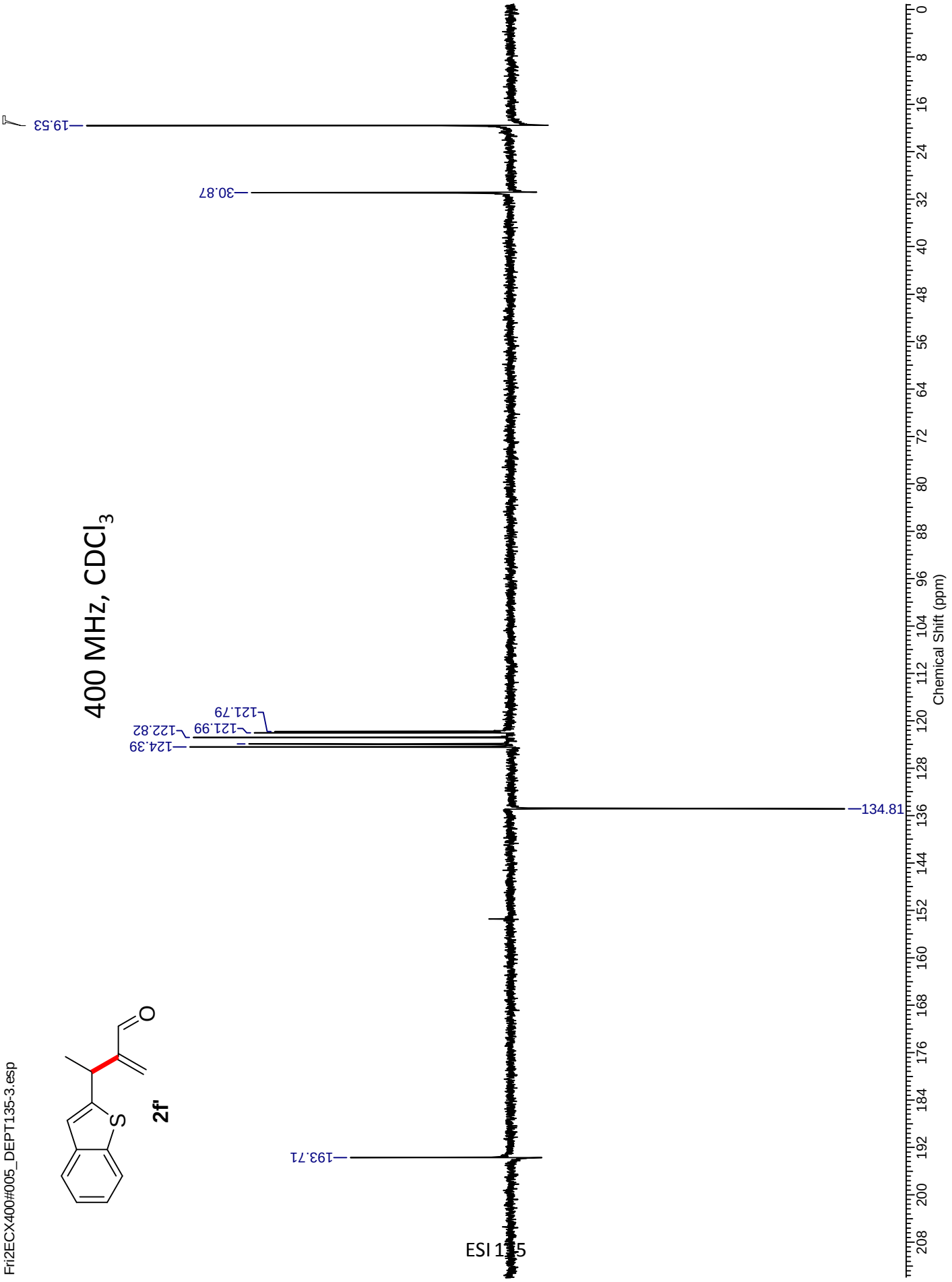

17.99
31.49
77.00
102.62
110.86
120.51
122.54
123.55
128.41
134.79
151.45
154.69
159.62
193.14
500 MHz, CDCl3
2e'
O
O
ESI 110
Wed1av500#001.003.001.1r.esp
Chemical Shift (ppm)
0
10
20
30
40
50
60
70
80
90
100
110
120
130
140
150
160
170
180
190
200
210

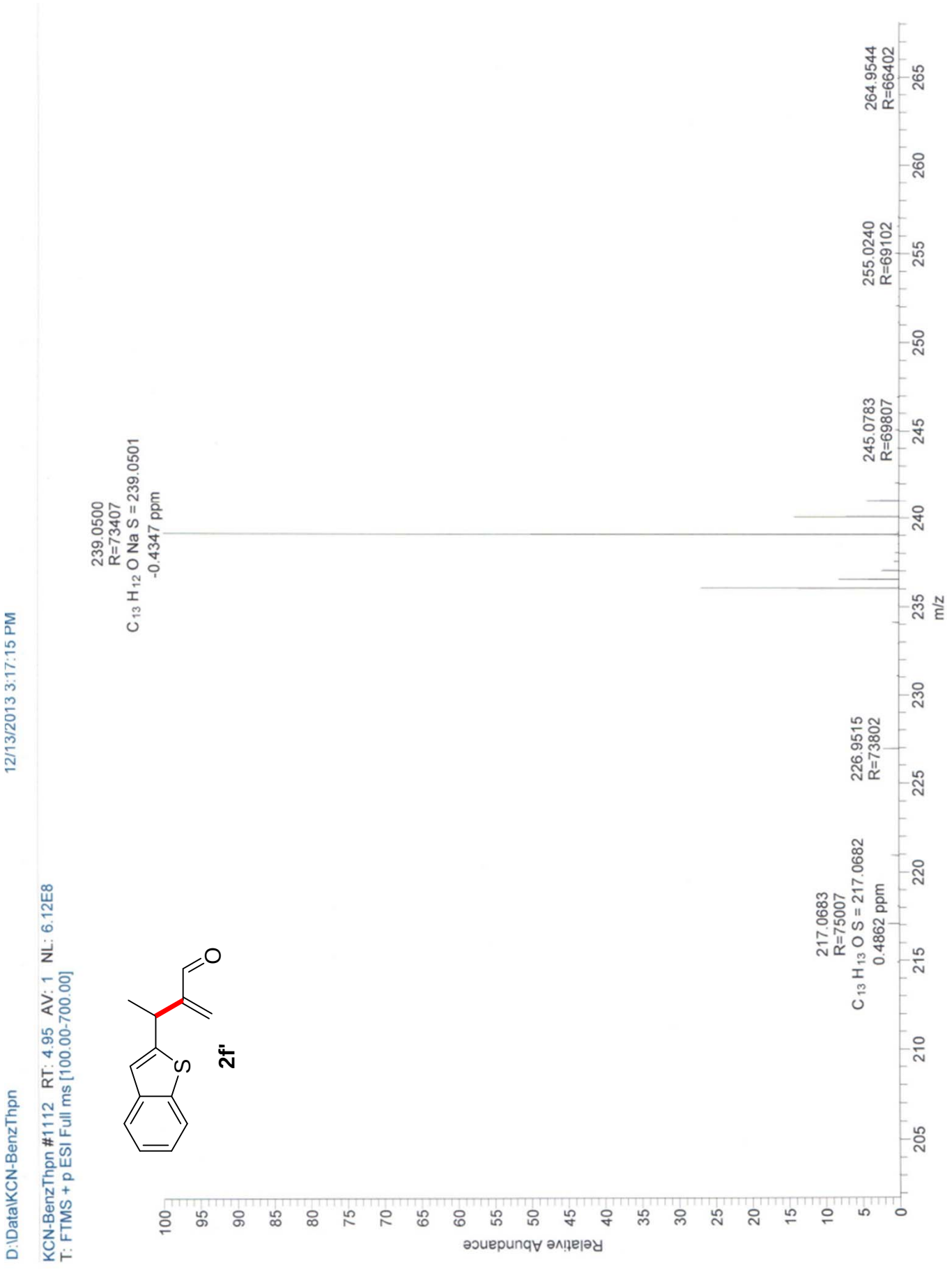


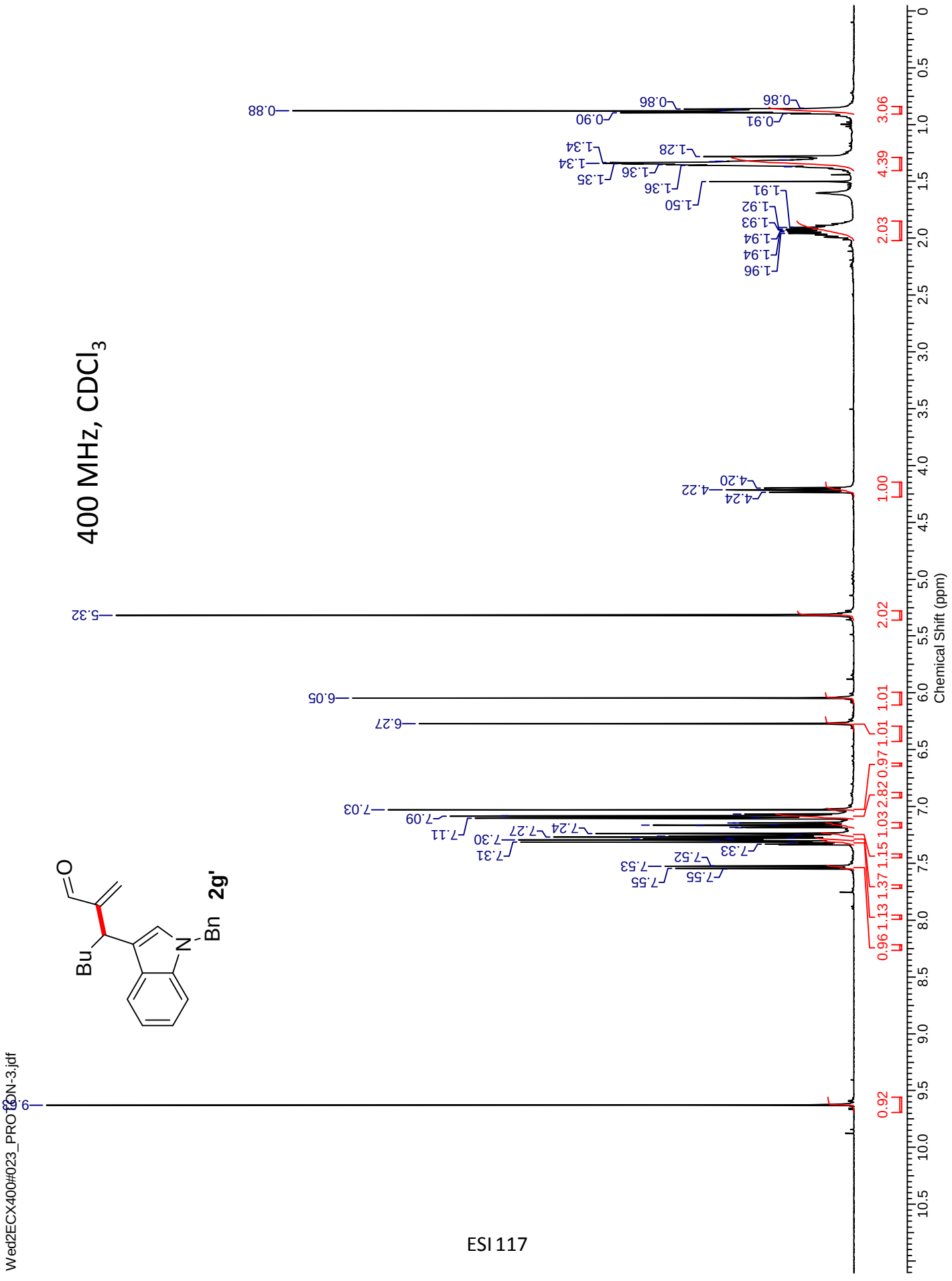

D:\Data\KCN-Benzfn
12/13/2013 2:59:30 PM
KCN-BenzFn #1044 RT: 4.65 AV: 1 NL: 2.06E7
T: FTMS + p ESI Full ms [100.00-700.00]
201.0910
R=79207
C 13 H 13 O 2 = 201.0910
-0.0138 ppm
202.0943
R=77902
207.0328
R=73402
C 12 H 8 O 2 Na = 207.0417
-42.7932 ppm
197.1149
R=75102
193.1312
R=79107
190.0387
R=78702
C 12 H 7 O Na = 190.0389
-1.3713 ppm
2e'
Relative Abundance
m/z

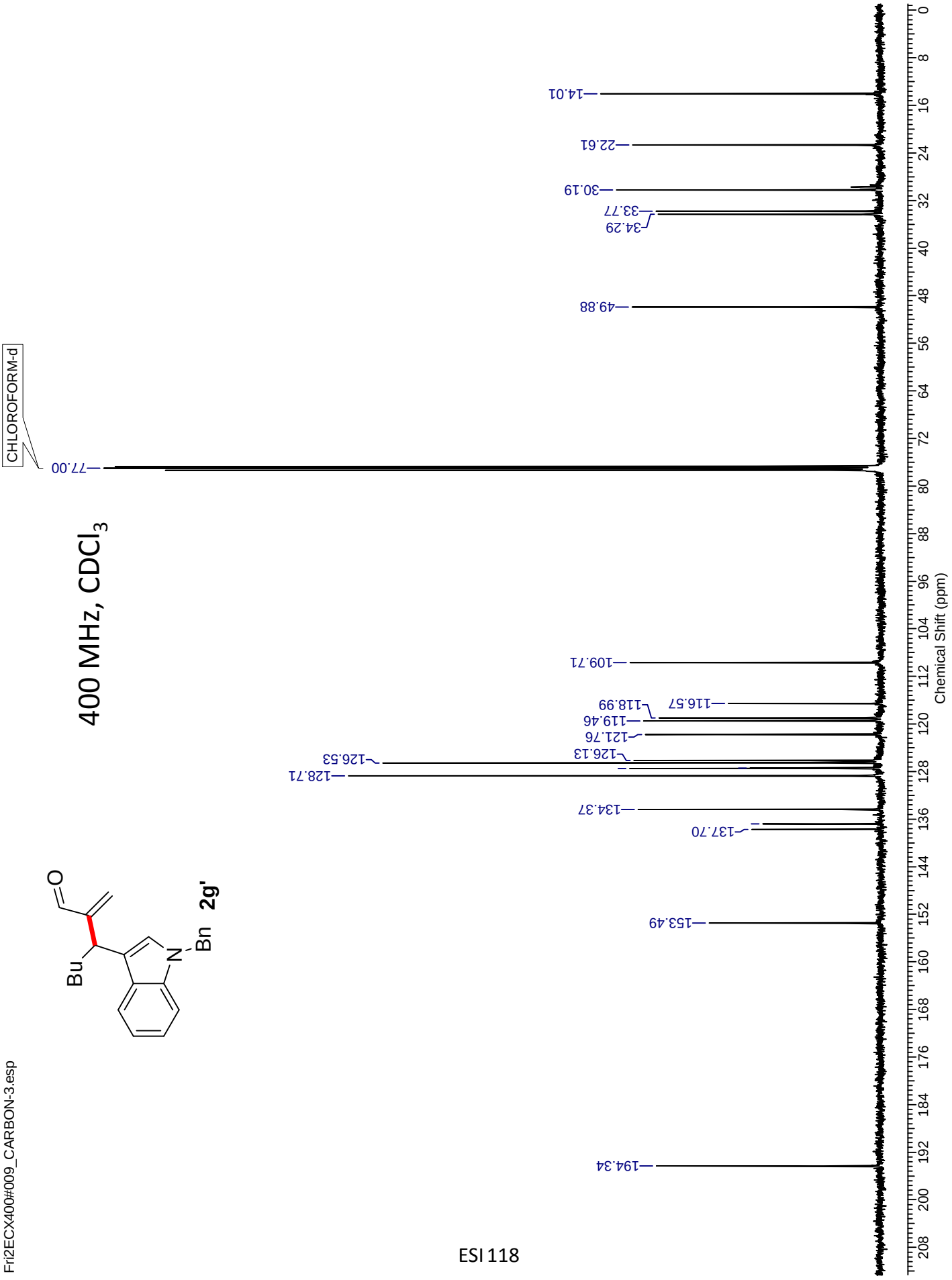

200 MHz, CDCl3
9.67
7.90
7.89
7.88
7.87
7.84
7.82
7.57
7.54
7.38
7.36
7.24
7.23
6.05
6.04
4.50
4.47
4.43
4.40
1.58
1.54
1.00
1.00
1.01
2.06
0.96
2.10
1.03
3.15
Chemical Shift (ppm)
O
S
2f'
Fri2av2#014.001.001.1r.esp

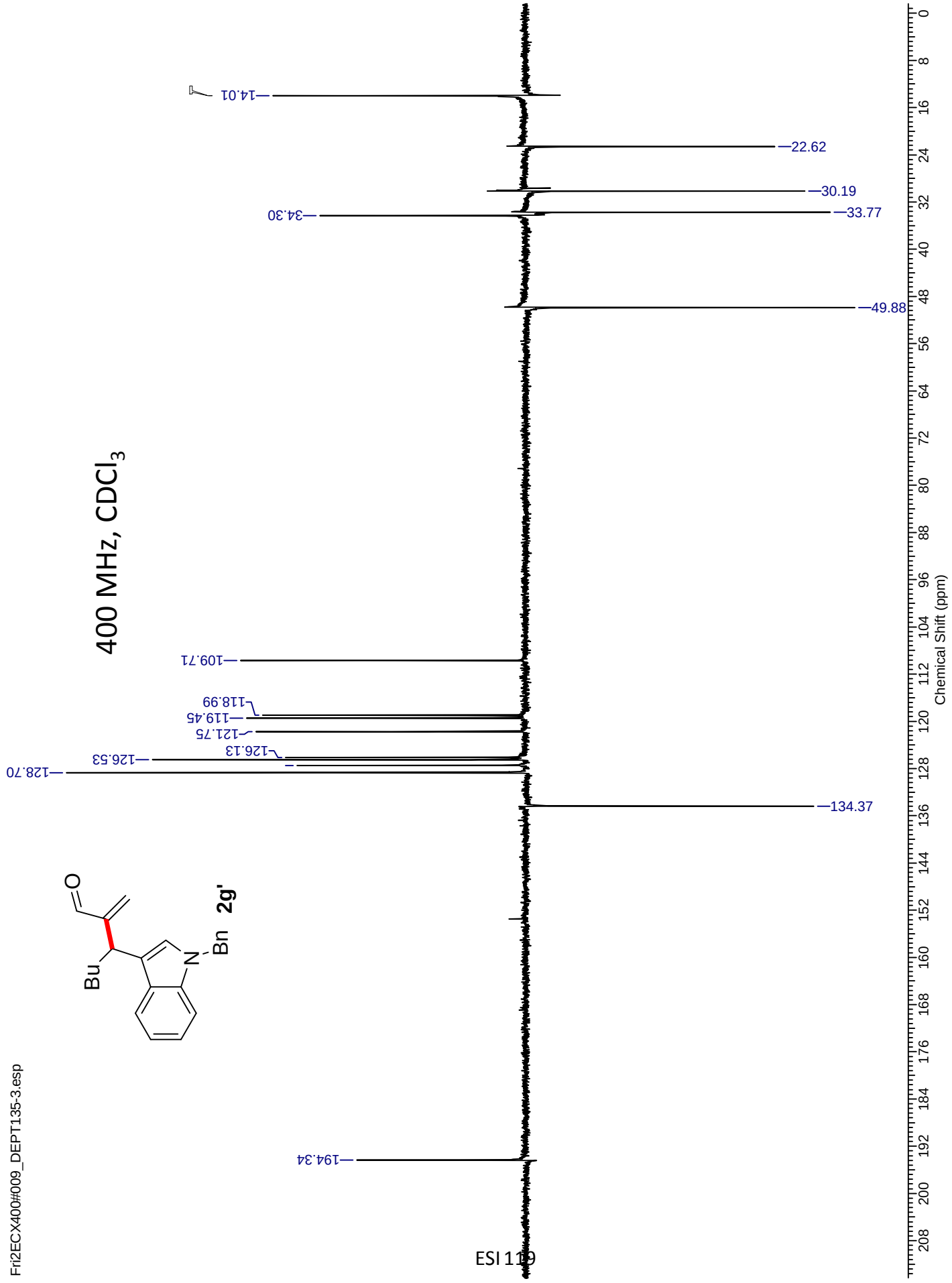

Fri2ECX400#005_CARBON-3.esp
400 MHz, $CDCl_3$
CHLOROFORM-d
77.00
19.53
30.87
121.77
121.98
122.81
123.92
124.39
134.78
138.54
140.53
153.46
193.69
O
S
2f'
Chemical Shift (ppm)
0
8
16
24
32
40
48
56
64
72
80
88
96
104
112
120
128
136
144
152
160
168
176
184
192
200
208

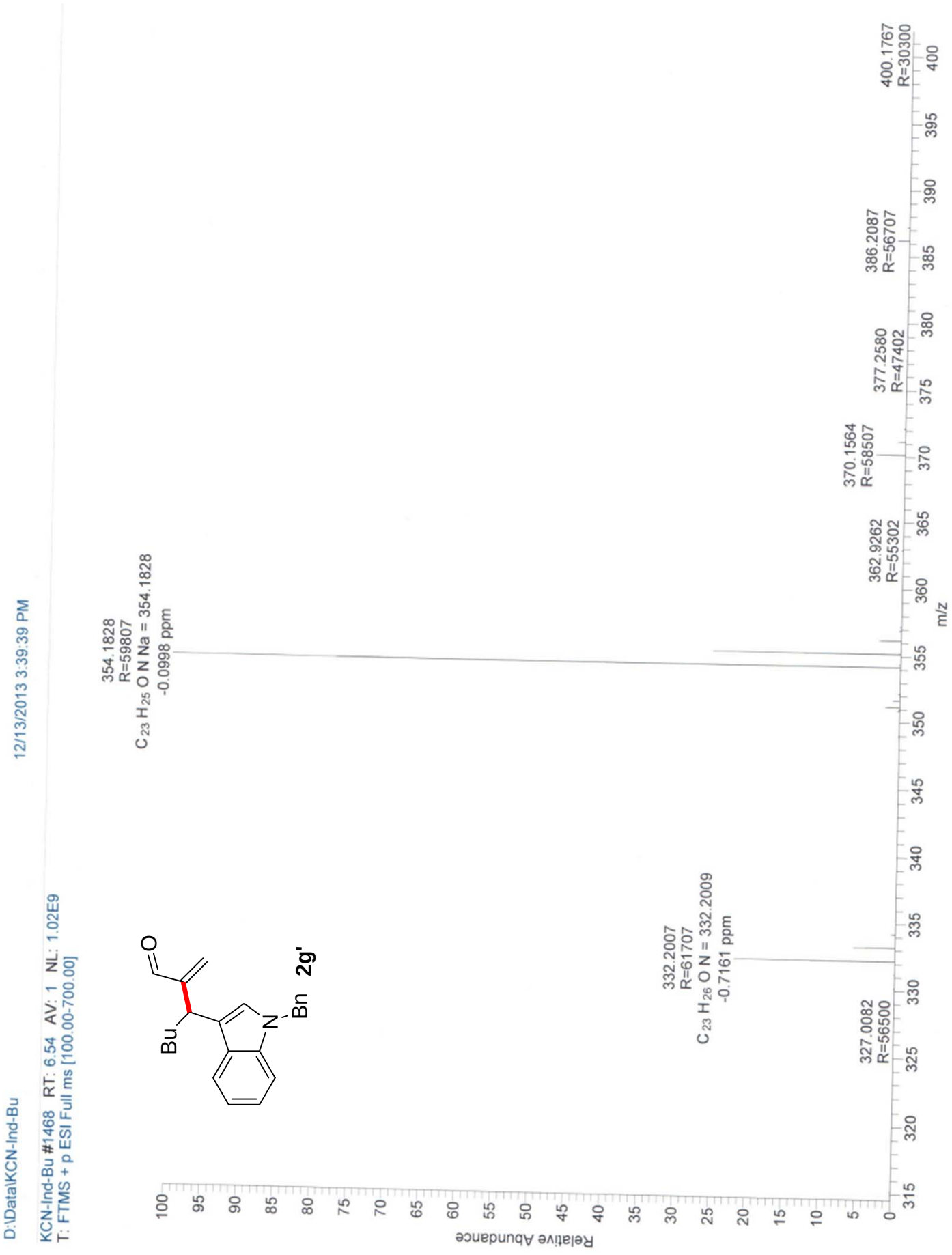

19.53
30.87
121.79
121.99
122.82
124.39
134.81
193.71
400 MHz, CDCl3
2f'
O
S
Fri2ECX400#005_DEPT135-3.esp
ESI 1 5
Chemical Shift (ppm)
0
8
16
24
32
40
48
56
64
72
80
88
96
104
112
120
128
136
144
152
160
168
176
184
192
200
208

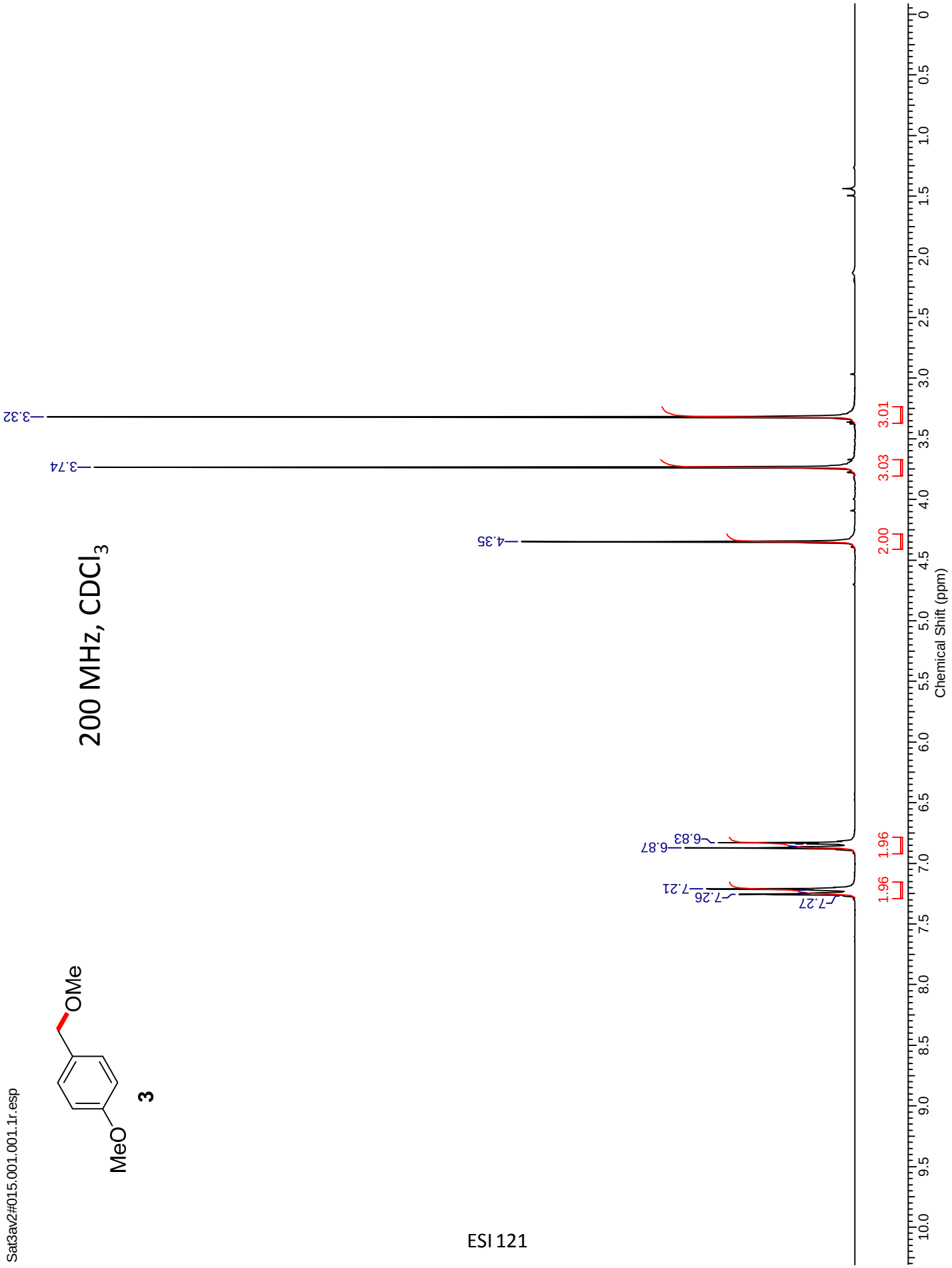

D:\Data\KCN-BenzThpn
12/13/2013 3:17:15 PM
KCN-BenzThpn #1112 RT: 4.95 AV: 1 NL: 6.12E8
T: FTMS + p ESI Full ms [100.00-700.00]
239.0500
R=73407
C 13 H 12 O Na S = 239.0501
-0.4347 ppm
245.0783
R=69807
255.0240
R=69102
264.9544
R=66402
217.0683
R=75007
C 13 H 13 O S = 217.0682
0.4862 ppm
226.9515
R=73802
2f'
O
S
Relative Abundance
m/z

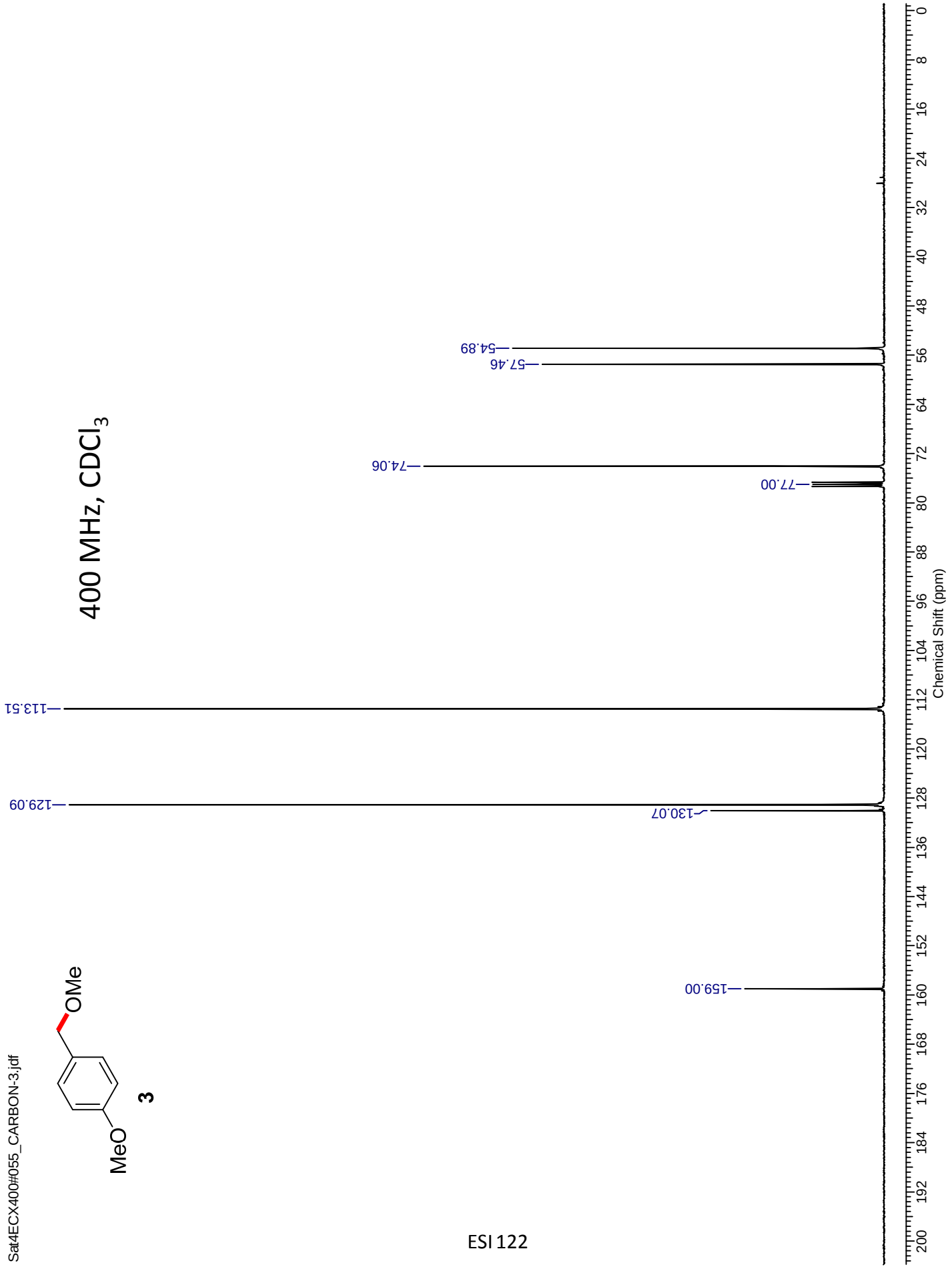


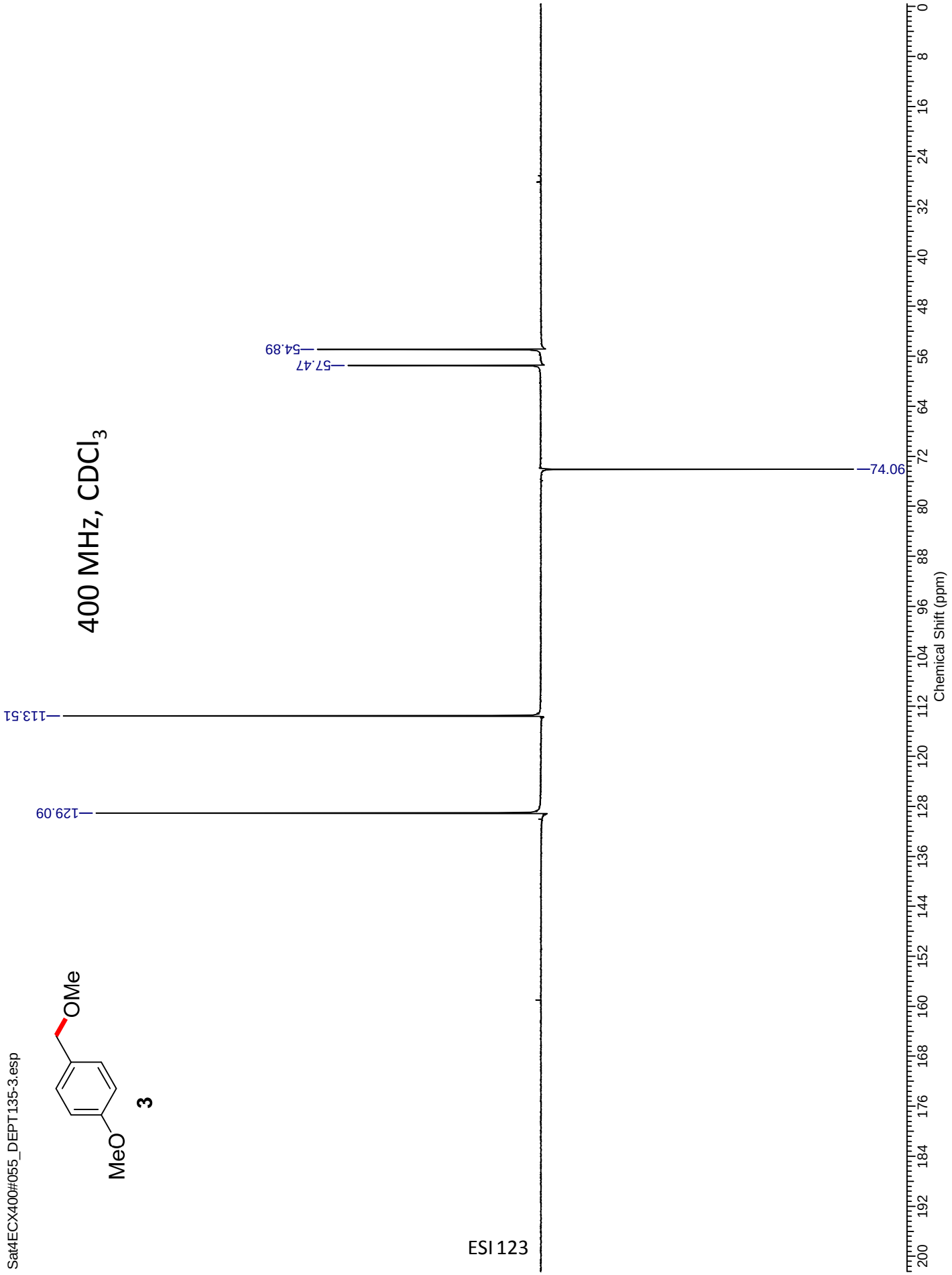


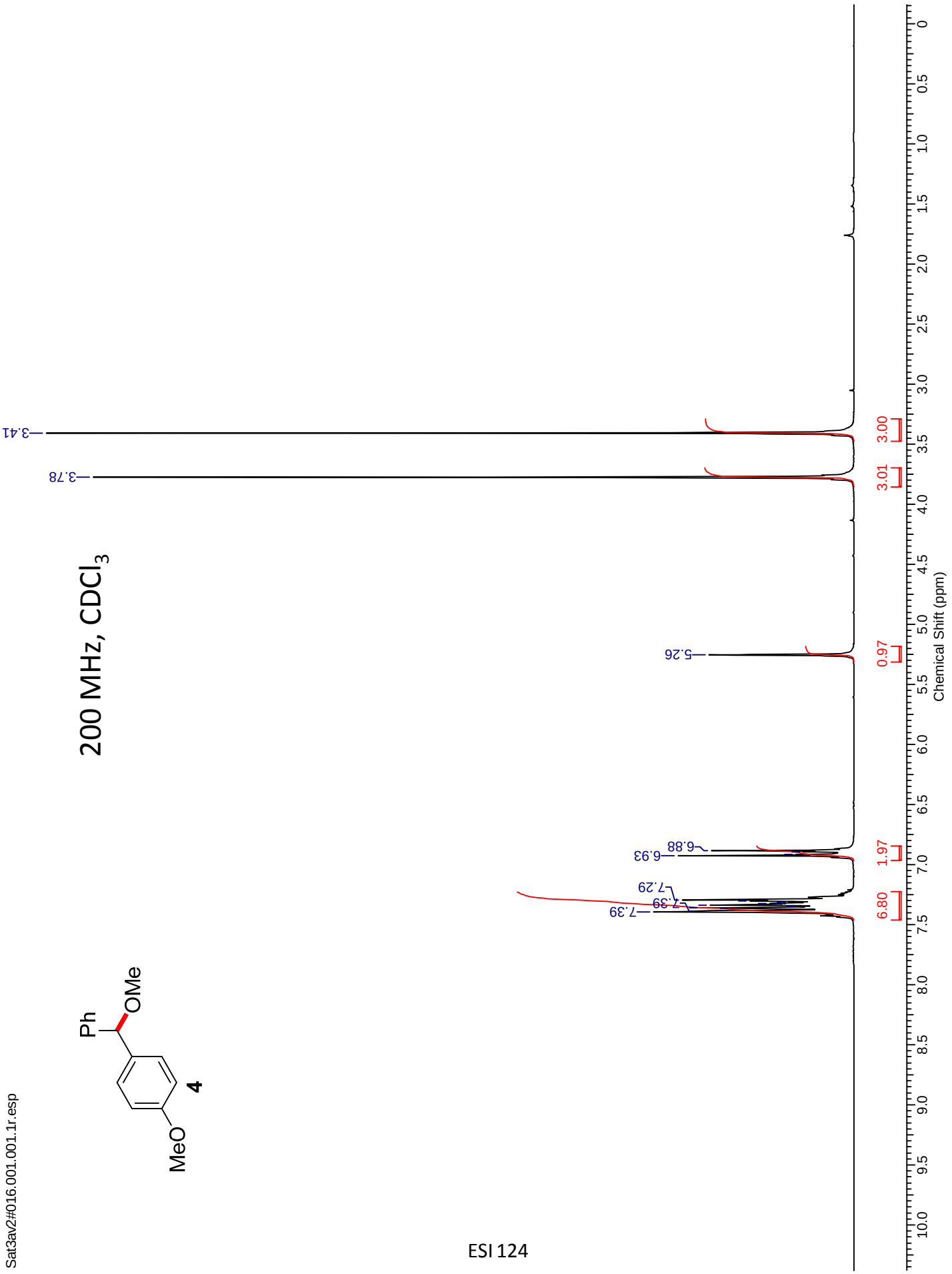

400 MHz, CDCl3
14.01
22.62
30.19
33.77
34.30
49.88
109.71
118.99
119.45
121.75
126.13
126.53
128.70
134.37
194.34
O
Bu
N
Bn
2g'
Fri2ECX400#009_DEPT135-3.esp
ESI 119
Chemical Shift (ppm)

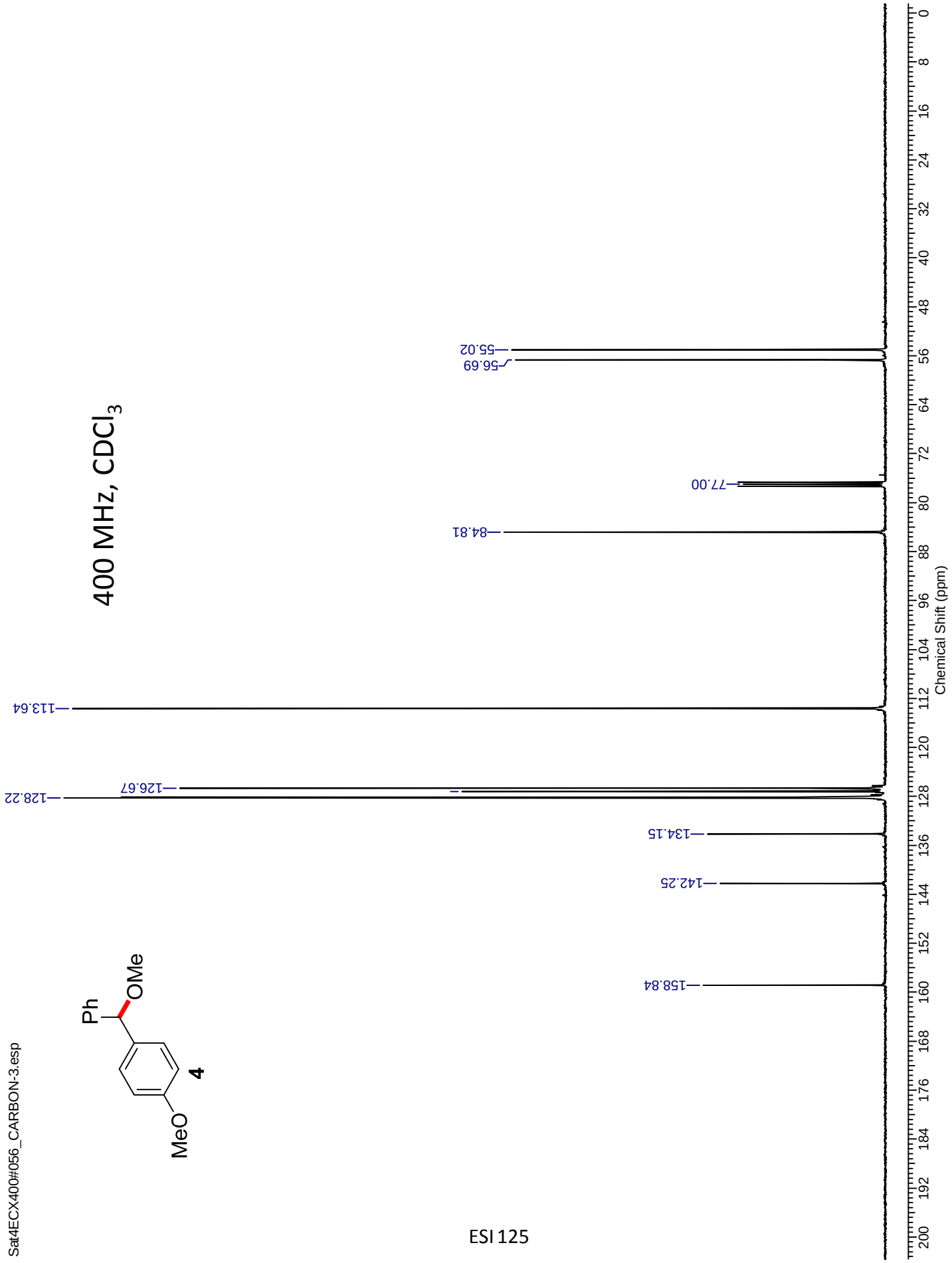

D:\Data\KCN-Ind-Bu
12/13/2013 3:39:39 PM
KCN-Ind-Bu #1468 RT: 6.54 AV: 1 NL: 1.02E9
T: FTMS + p ESI Full ms [100.00-700.00]
354.1828
R=59807
C23 H25 O N Na = 354.1828
-0.0998 ppm
332.2007
R=61707
C23 H26 O N = 332.2009
-0.7161 ppm
327.0082
R=56500
362.9262
R=55302
370.1564
R=58507
377.2580
R=47402
386.2087
R=56707
400.1767
R=30300
Relative Abundance
m/z
Bu
Bn
N
O
2g'

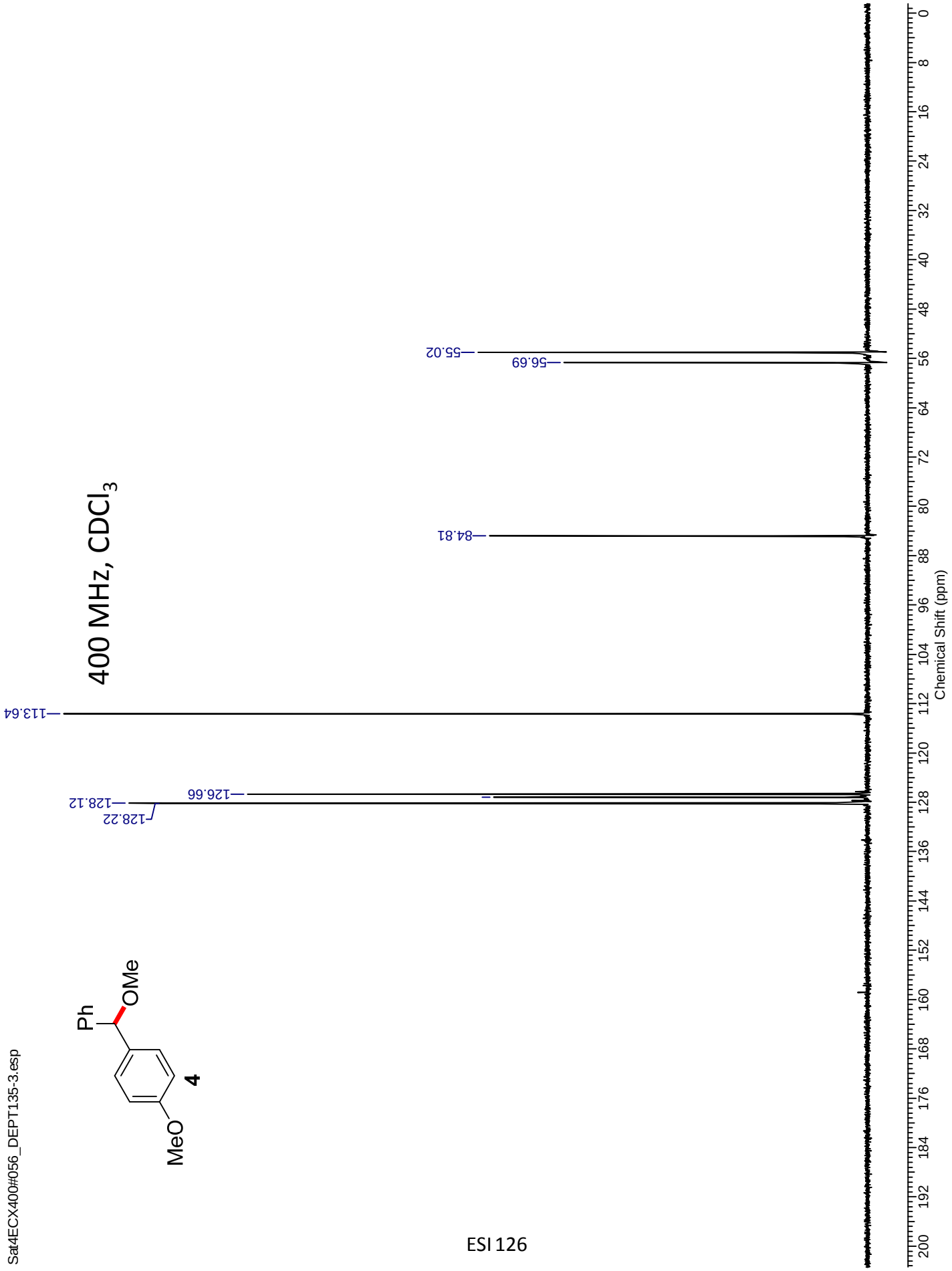

200 MHz, CDCl3
OMe
MeO
3
3.32
3.74
4.35
6.83
6.87
7.21
7.26
7.27
3.01
3.03
2.00
1.96
1.96
Chemical Shift (ppm)
Sat3av2#015.001.001.1r.esp

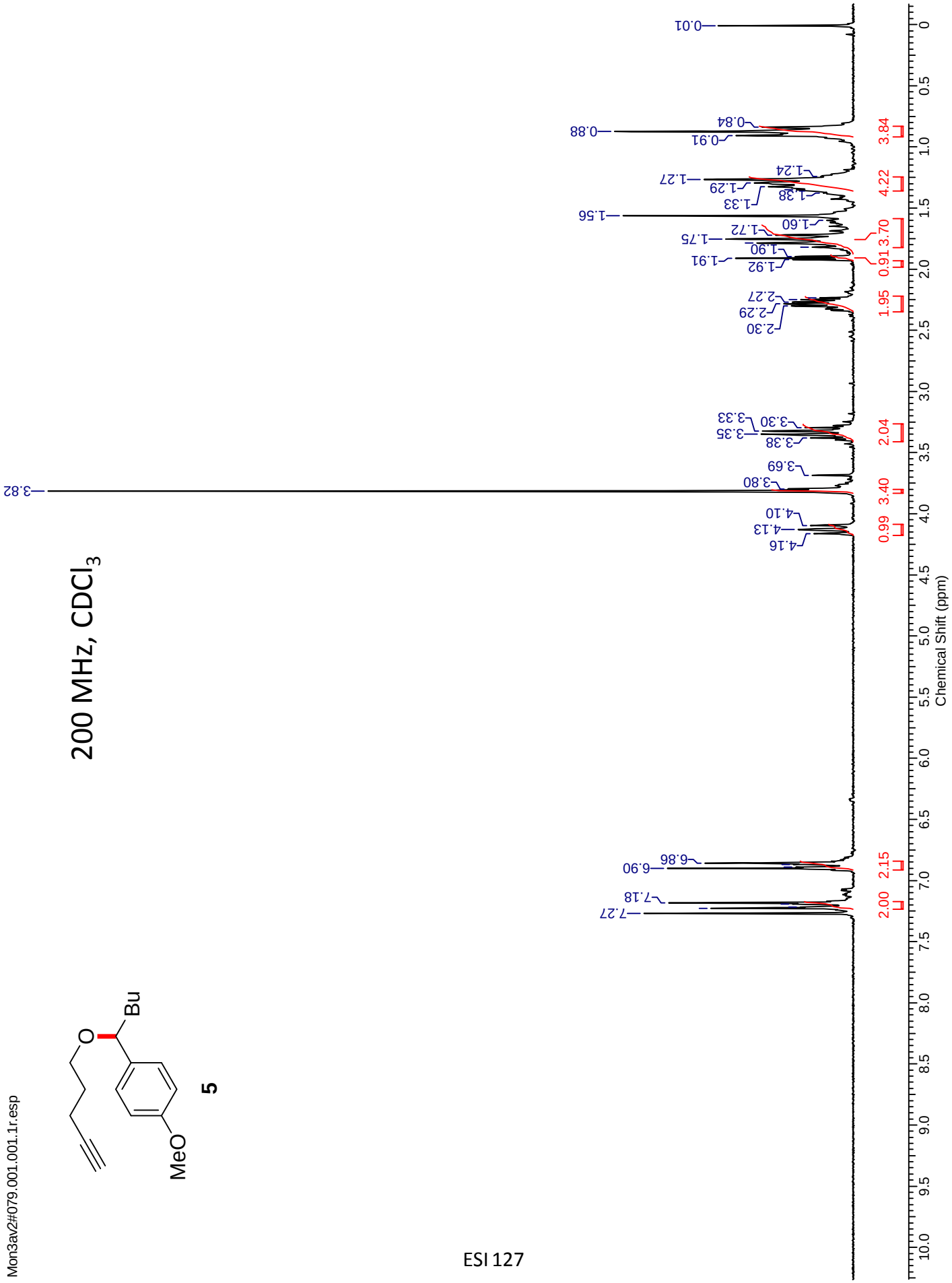

400 MHz, CDCl3
OMe
MeO
3
Sat4ECX400#055_CARBON-3.jdf
159.00
130.07
129.09
113.51
77.00
74.06
57.46
54.89
Chemical Shift (ppm)

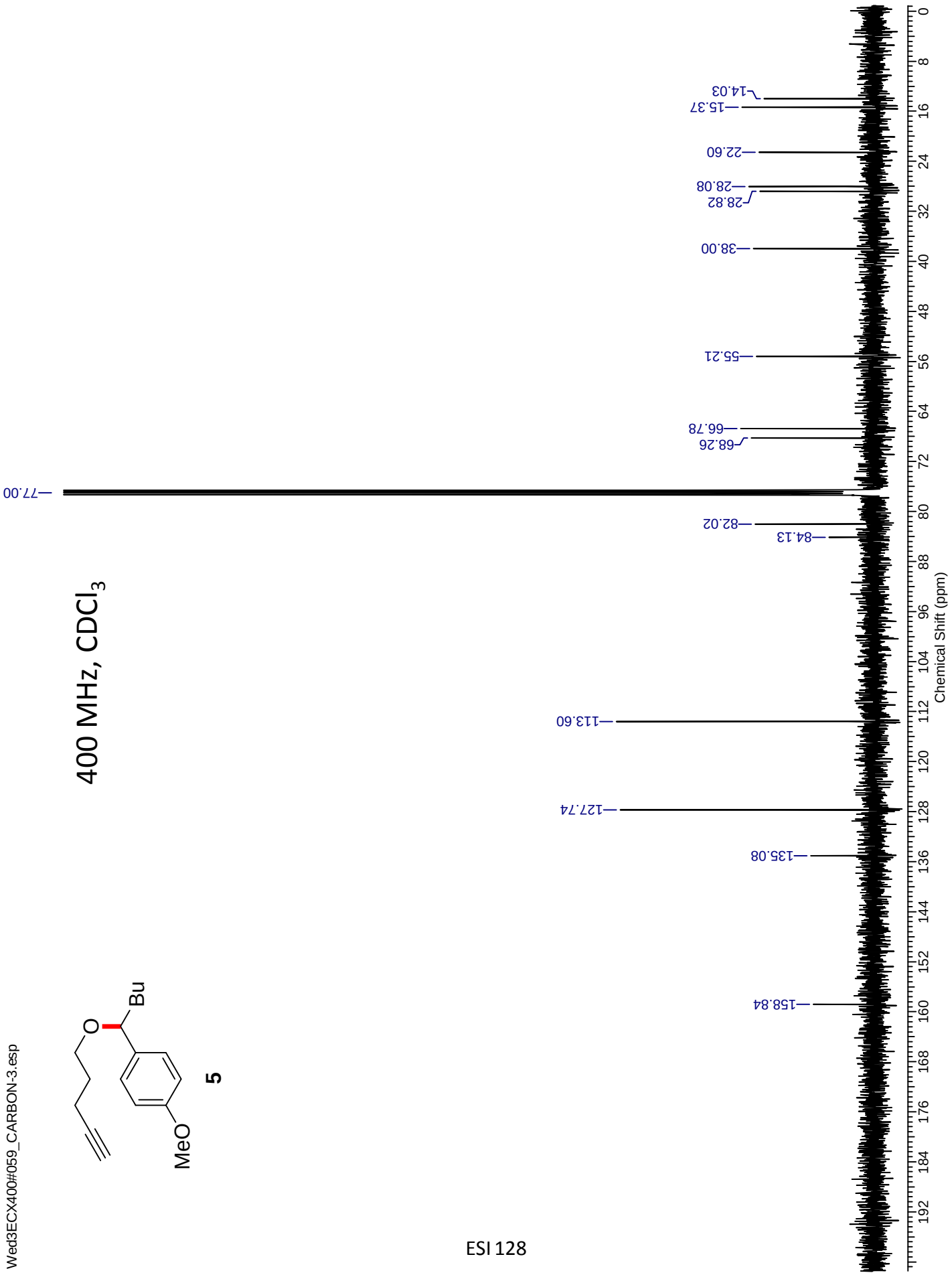

400 MHz, CDCl3
54.89
57.47
74.06
113.51
129.09
OMe
MeO
3
ESI 123
Sat4ECX400#055_DEPT135-3.esp
Chemical Shift (ppm)
0
8
16
24
32
40
48
56
64
72
80
88
96
104
112
120
128
136
144
152
160
168
176
184
192
200

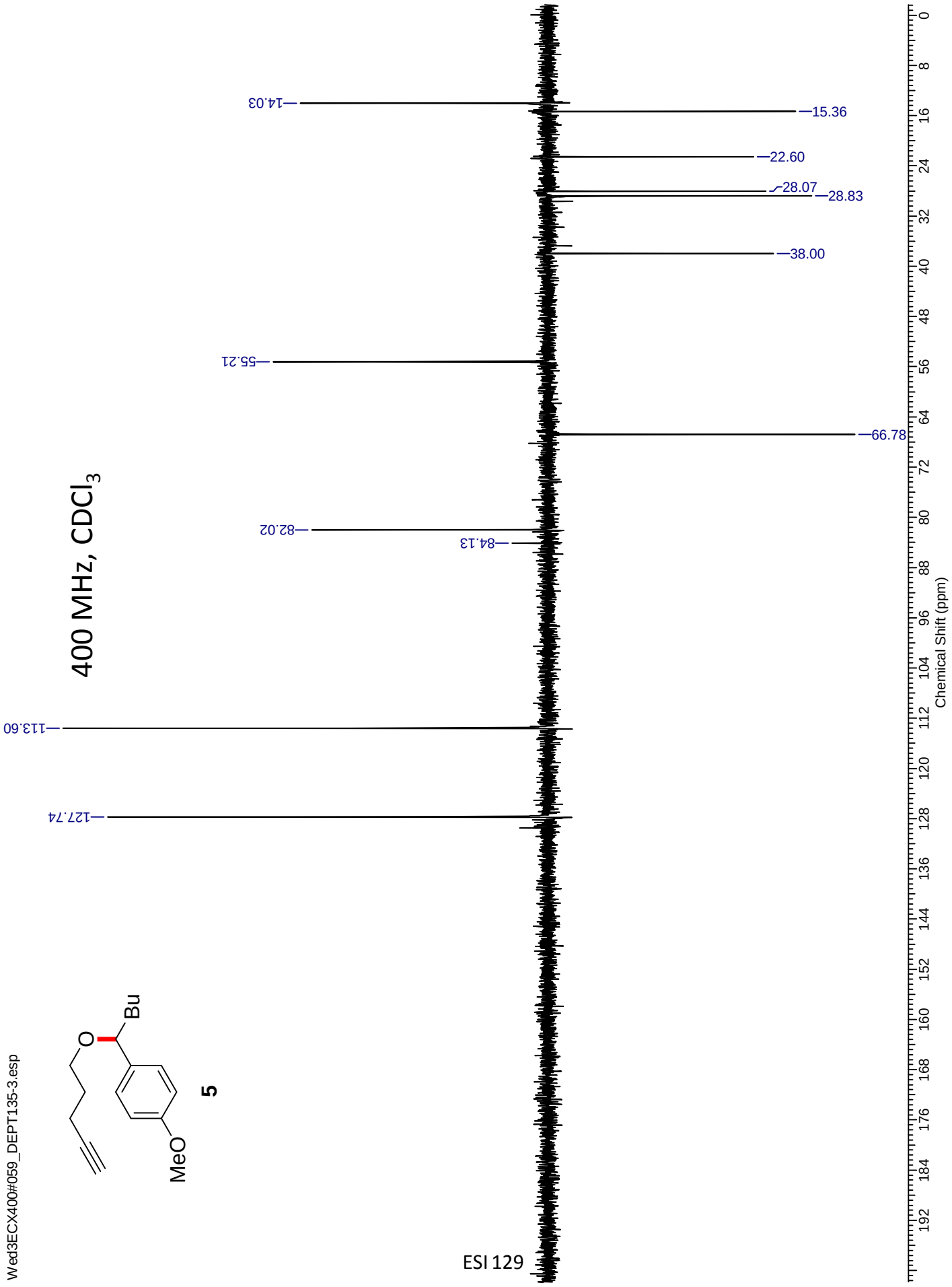

200 MHz, CDCl3
3.41
3.78
5.26
6.88
6.93
7.29
7.39
7.39
3.00
3.01
0.97
1.97
6.80
Chemical Shift (ppm)
Ph
OMe
MeO
4
Sat3av2#016.001.001.1r.esp

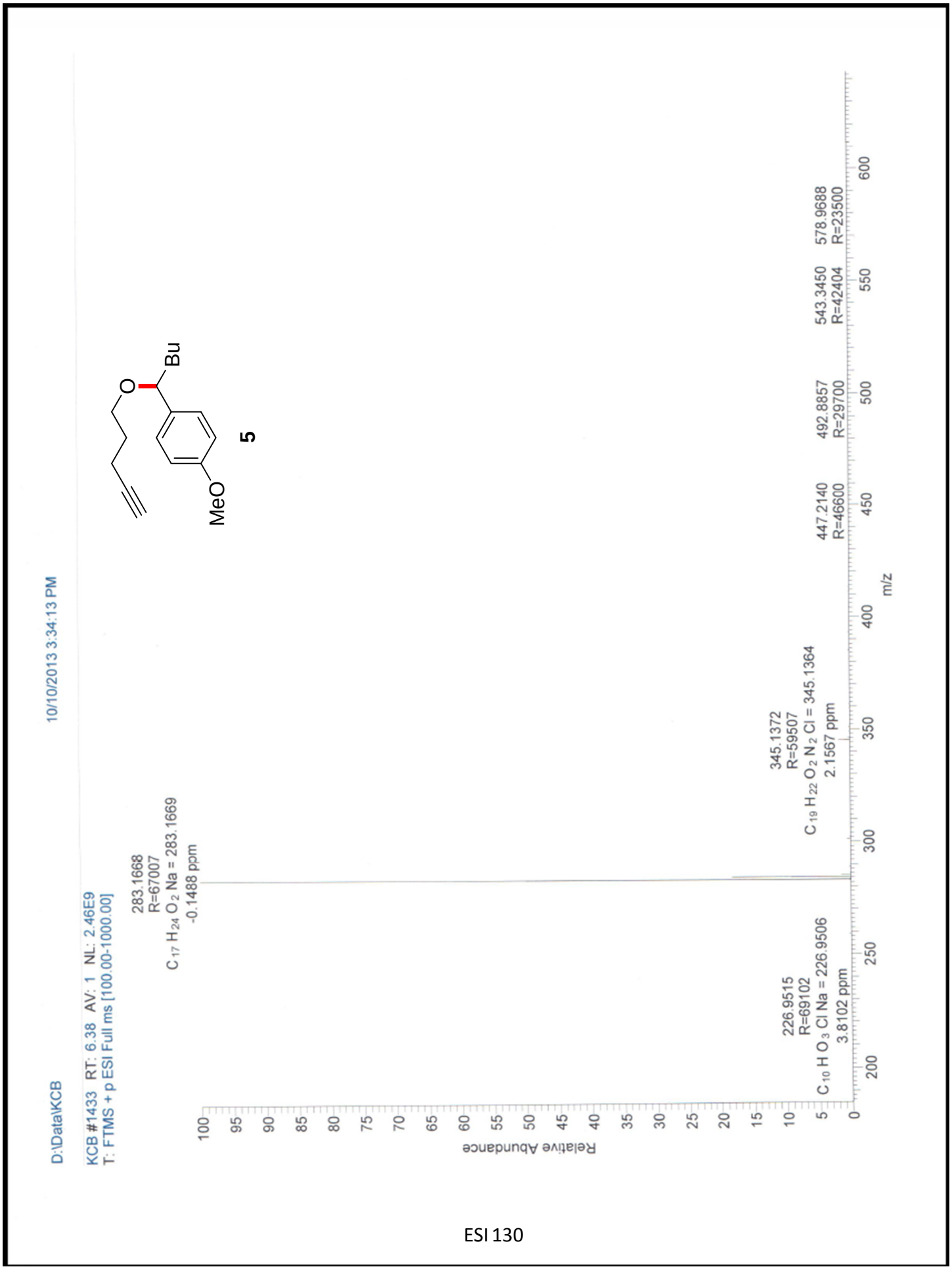

400 MHz, CDCl3
55.02
56.69
77.00
84.81
113.64
126.67
128.22
134.15
142.25
158.84
Chemical Shift (ppm)
Ph
OMe
MeO
4
Sat4ECX400#056_CARBON-3.esp

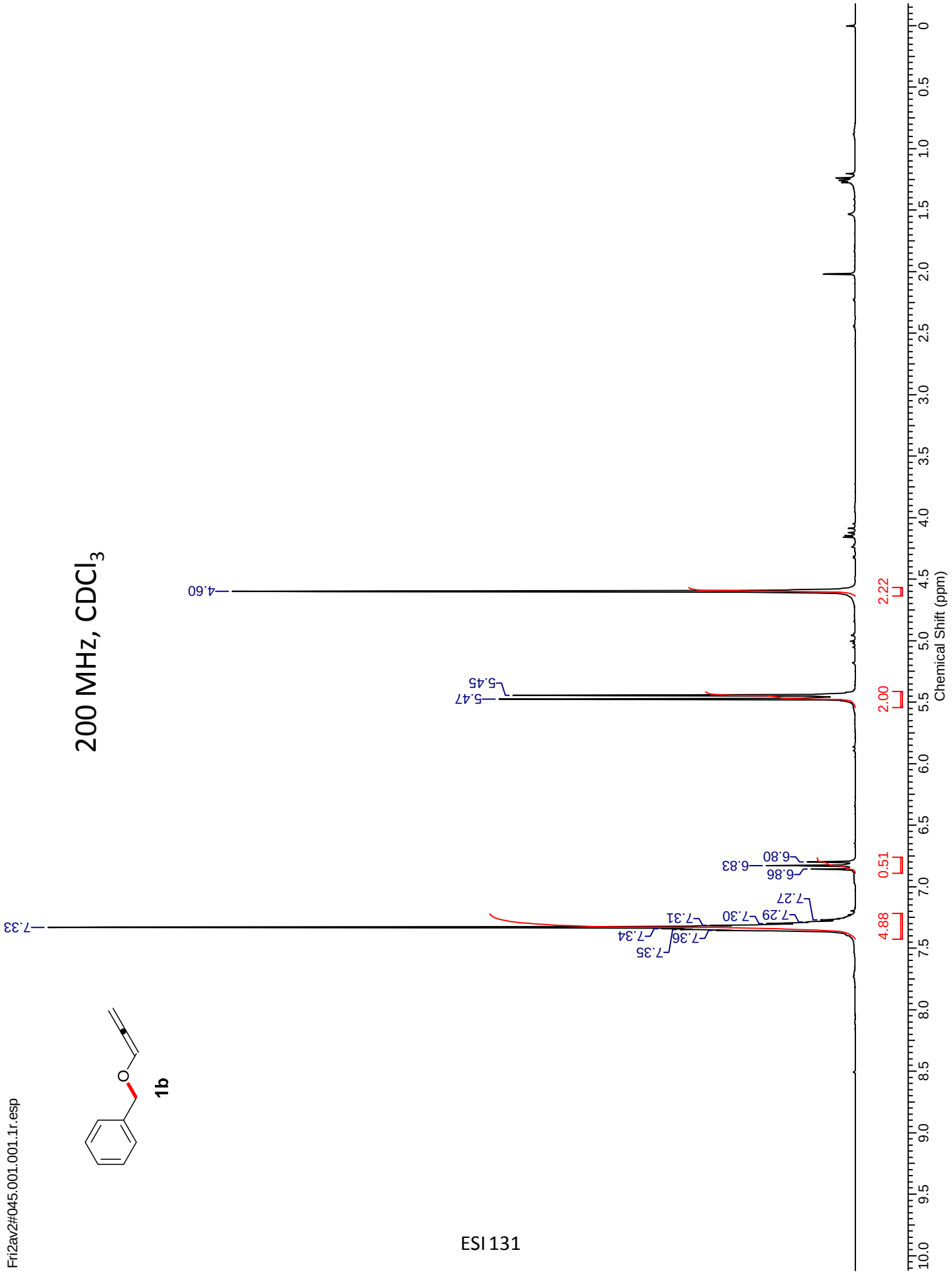

400 MHz, CDCl3
55.02
56.69
84.81
113.64
126.66
128.12
128.22
Ph
OMe
MeO
4
Chemical Shift (ppm)
Sat4ECX400#056_DEPT135-3.esp

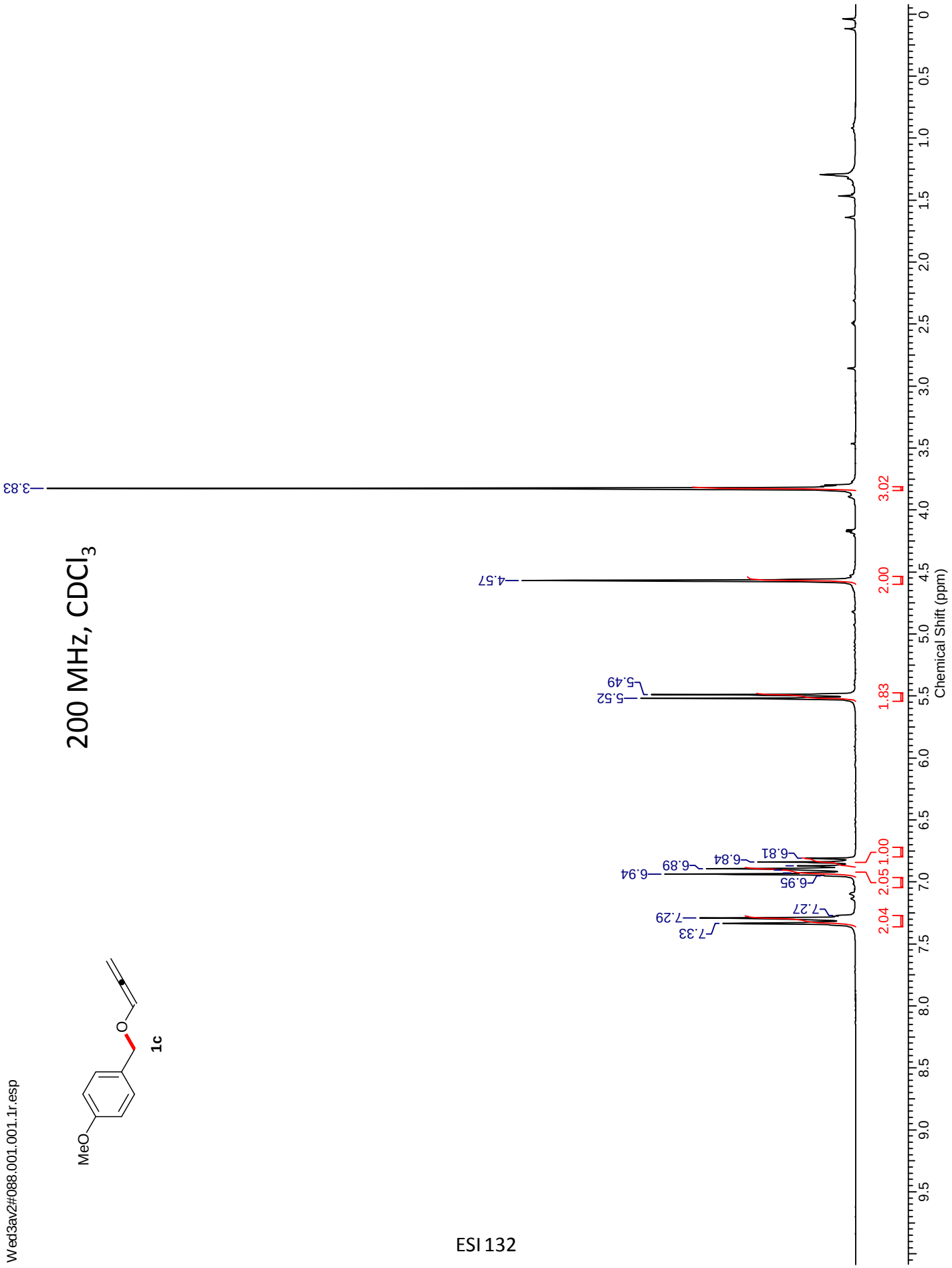

200 MHz, CDCl3
Bu
O
MeO
5
Mon3av2#079.001.001.1r.esp
Chemical Shift (ppm)
0.01
0.84
0.88
0.91
1.24
1.27
1.29
1.33
1.38
1.56
1.60
1.72
1.75
1.90
1.91
1.92
2.27
2.29
2.30
3.30
3.33
3.35
3.38
3.69
3.80
3.82
4.10
4.13
4.16
6.86
6.90
7.18
7.27
3.84
4.22
3.70
0.91
1.95
2.04
3.40
0.99
2.15
2.00

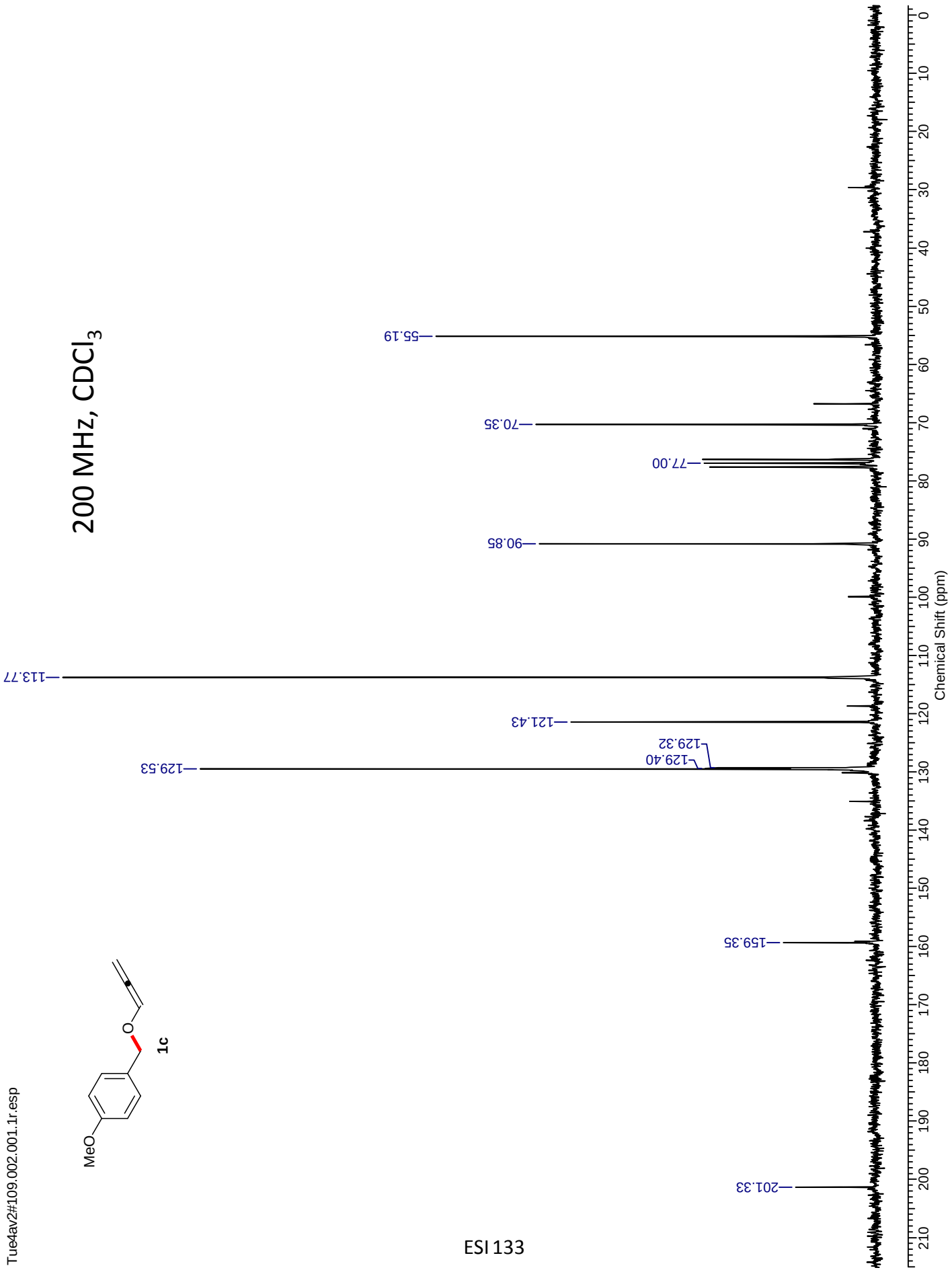

400 MHz, $CDCl_3$
Wed3ECX400#059_CARBON-3.esp
Bu
O
MeO
5
14.03
15.37
22.60
28.08
28.82
38.00
55.21
66.78
68.26
77.00
82.02
84.13
113.60
127.74
135.08
158.84
Chemical Shift (ppm)

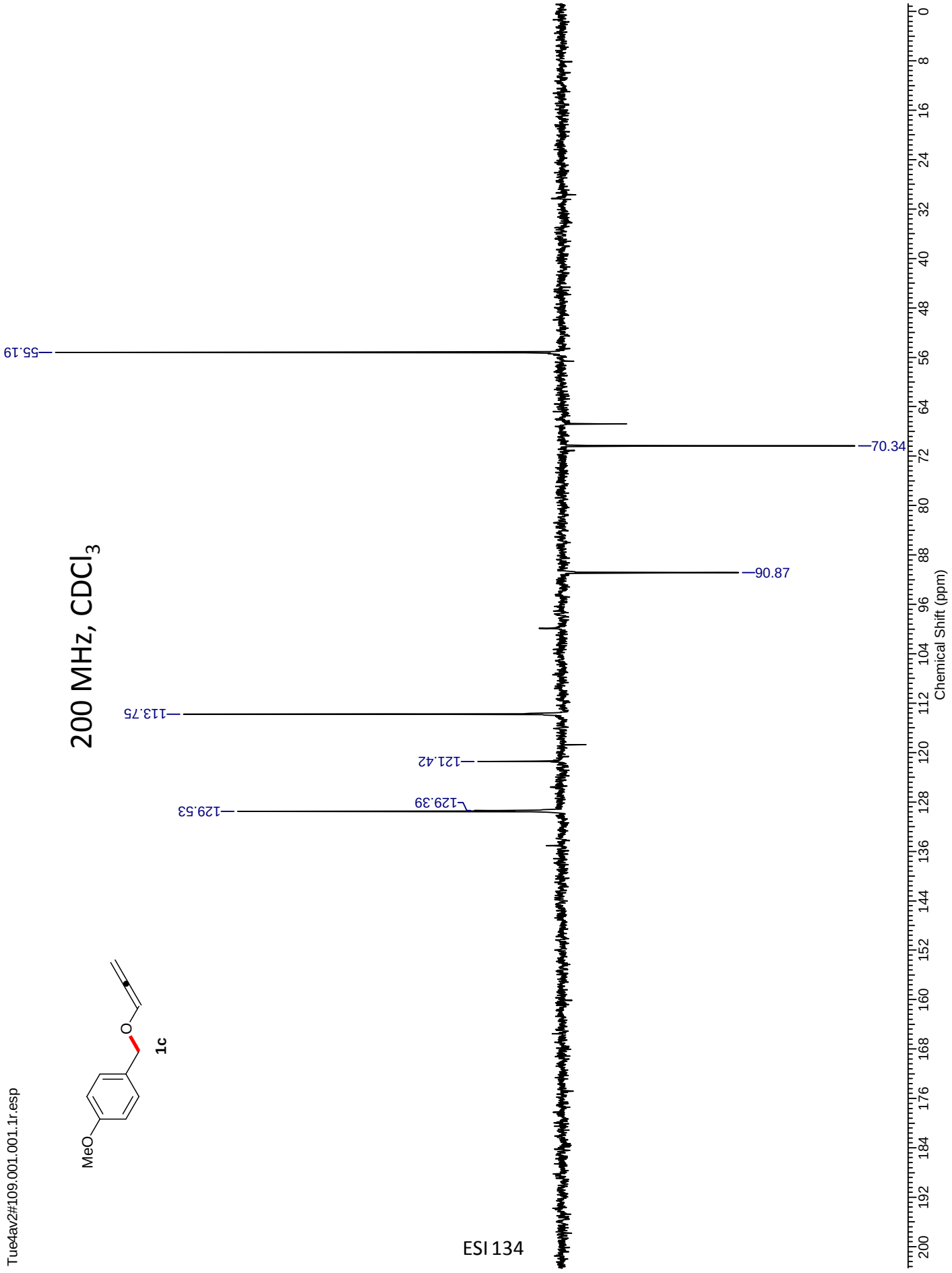

14.03
15.36
22.60
28.07
28.83
38.00
55.21
66.78
82.02
84.13
113.60
127.74
400 MHz, CDCl3
Bu
O
MeO
5
Wed3ECX400#059_DEPT135-3.esp
ESI 129
Chemical Shift (ppm)
0
8
16
24
32
40
48
56
64
72
80
88
96
104
112
120
128
136
144
152
160
168
176
184
192

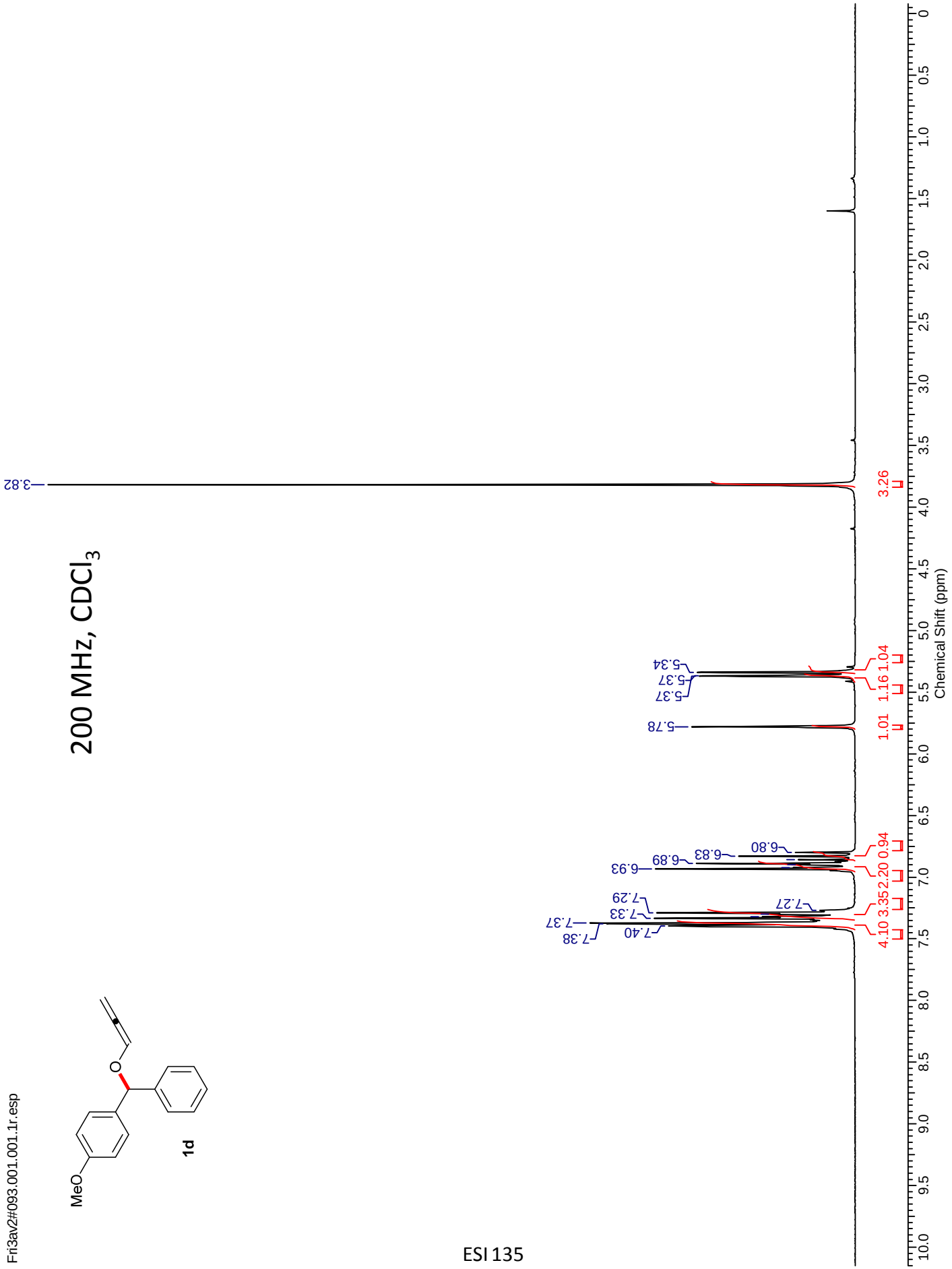

D:\Data\KCB
10/10/2013 3:34:13 PM
KCB #1433 RT: 6.38 AV: 1 NL: 2.46E9
T: FTMS + p ESI Full ms [100.00-1000.00]
283.1668
R=67007
C17 H24 O2 Na = 283.1669
-0.1488 ppm
226.9515
R=69102
C10 H O3 Cl Na = 226.9506
3.8102 ppm
345.1372
R=59507
C19 H22 O2 N2 Cl = 345.1364
2.1567 ppm
447.2140
R=46600
492.8857
R=29700
543.3450
R=42404
578.9688
R=23500
Relative Abundance
m/z
O
Bu
MeO
5

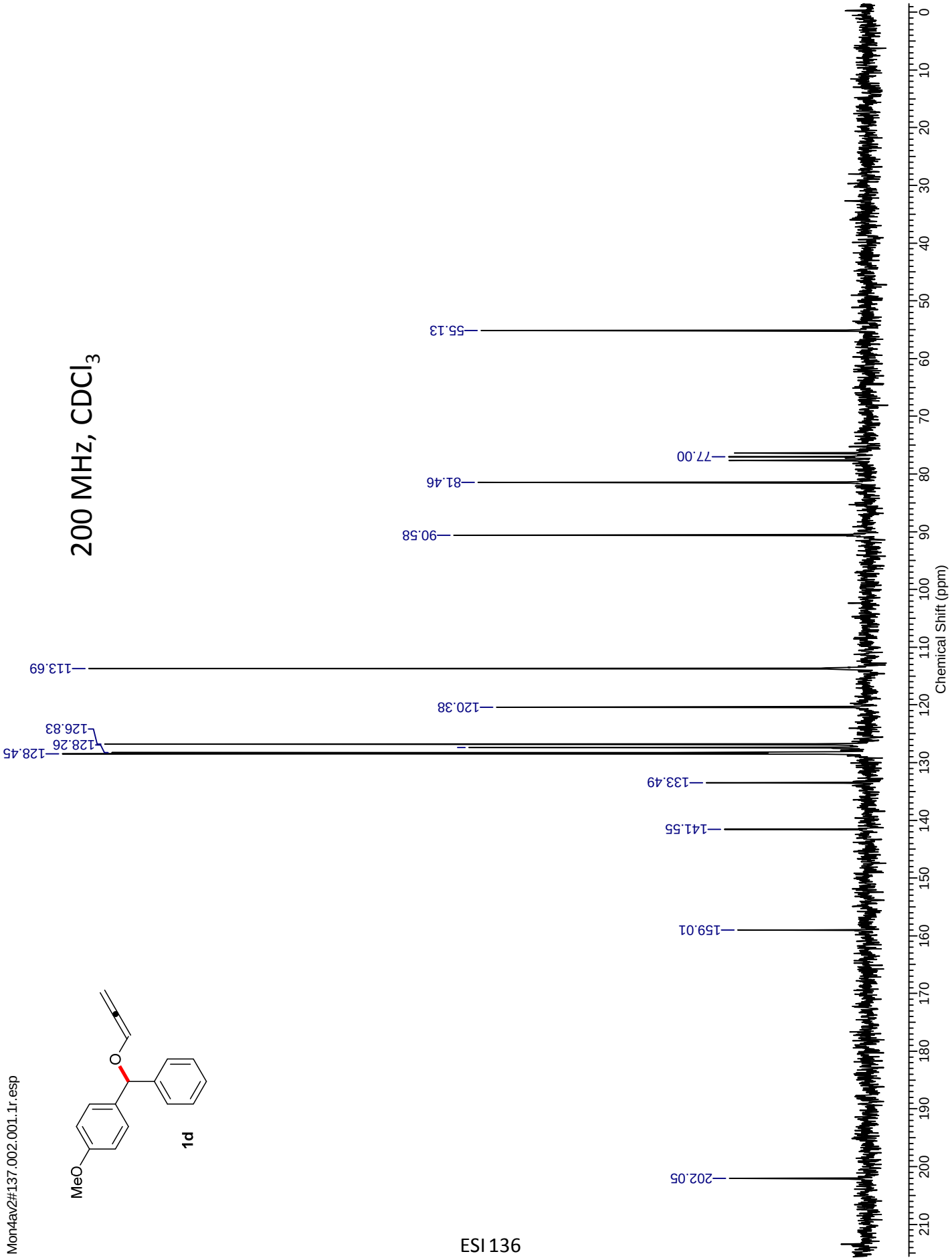

200 MHz, CDCl3
1b
Fri2av2#045.001.001.1r.esp
7.33
7.35
7.36
7.34
7.31
7.29
7.30
7.27
6.86
6.80
6.83
5.47
5.45
4.60
4.88
0.51
2.00
2.22
Chemical Shift (ppm)

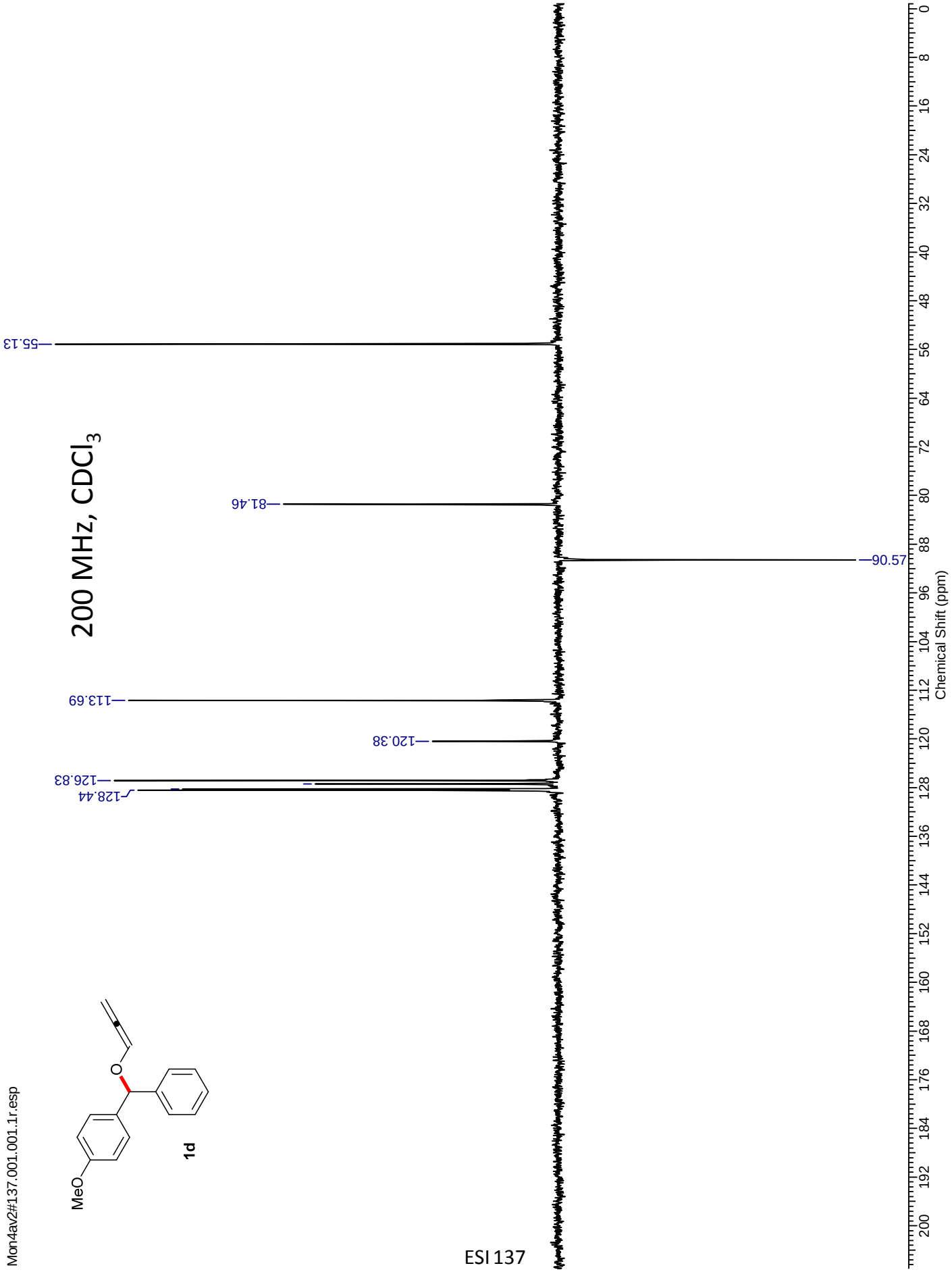

200 MHz, CDCl3
3.83
4.57
5.52
5.49
6.94
6.89
6.84
6.81
6.95
7.27
7.29
7.33
3.02
2.00
1.83
1.00
2.05
2.04
MeO
O
1c
Chemical Shift (ppm)
Wed3av2#088.001.001.1r.esp

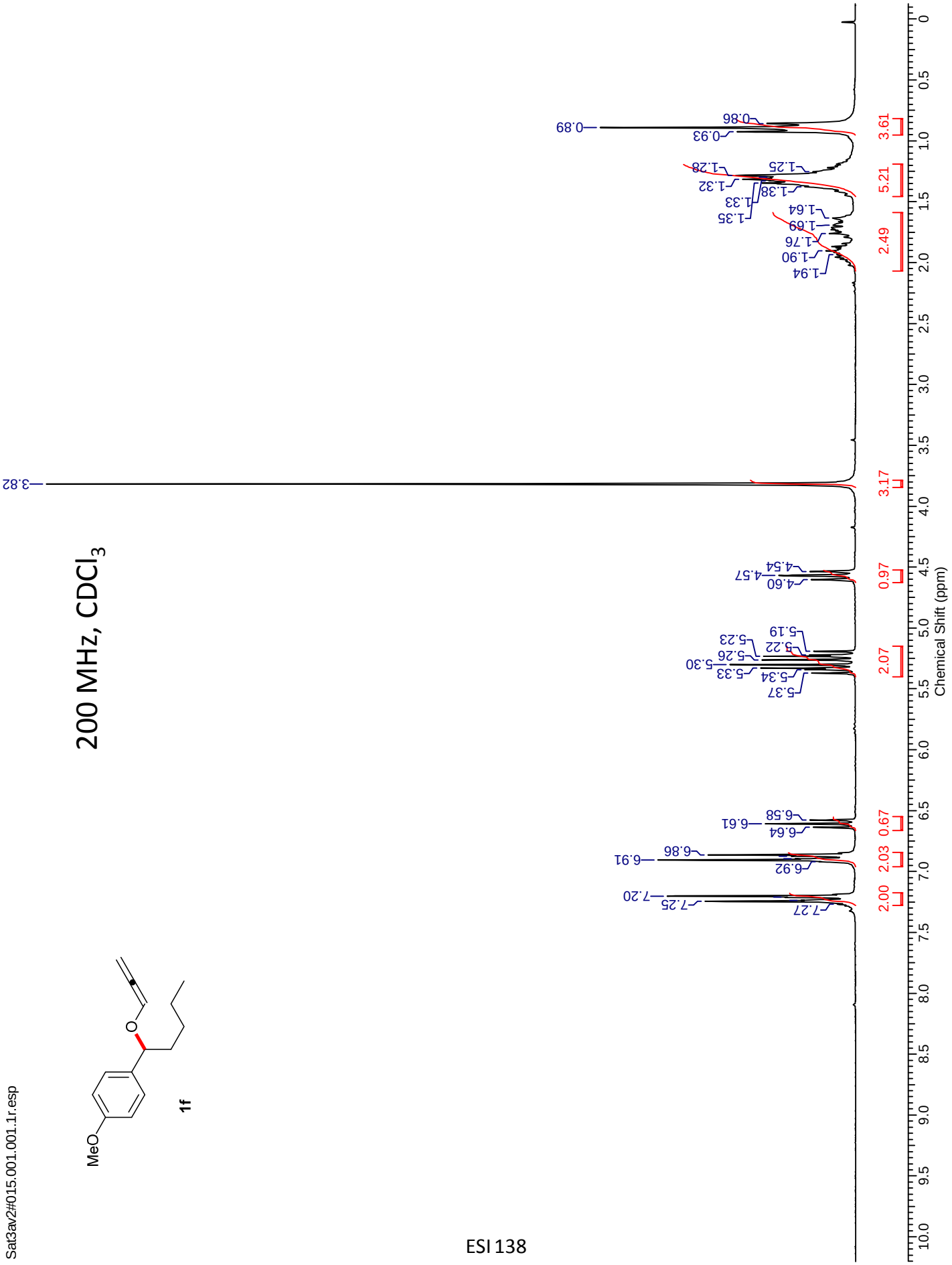

200 MHz, $CDCl_3$
55.19
70.35
77.00
90.85
113.77
121.43
129.32
129.40
129.53
159.35
201.33
Chemical Shift (ppm)
0
10
20
30
40
50
60
70
80
90
100
110
120
130
140
150
160
170
180
190
200
210
O
MeO
1c
Tue4av2#109.002.001.1r.esp

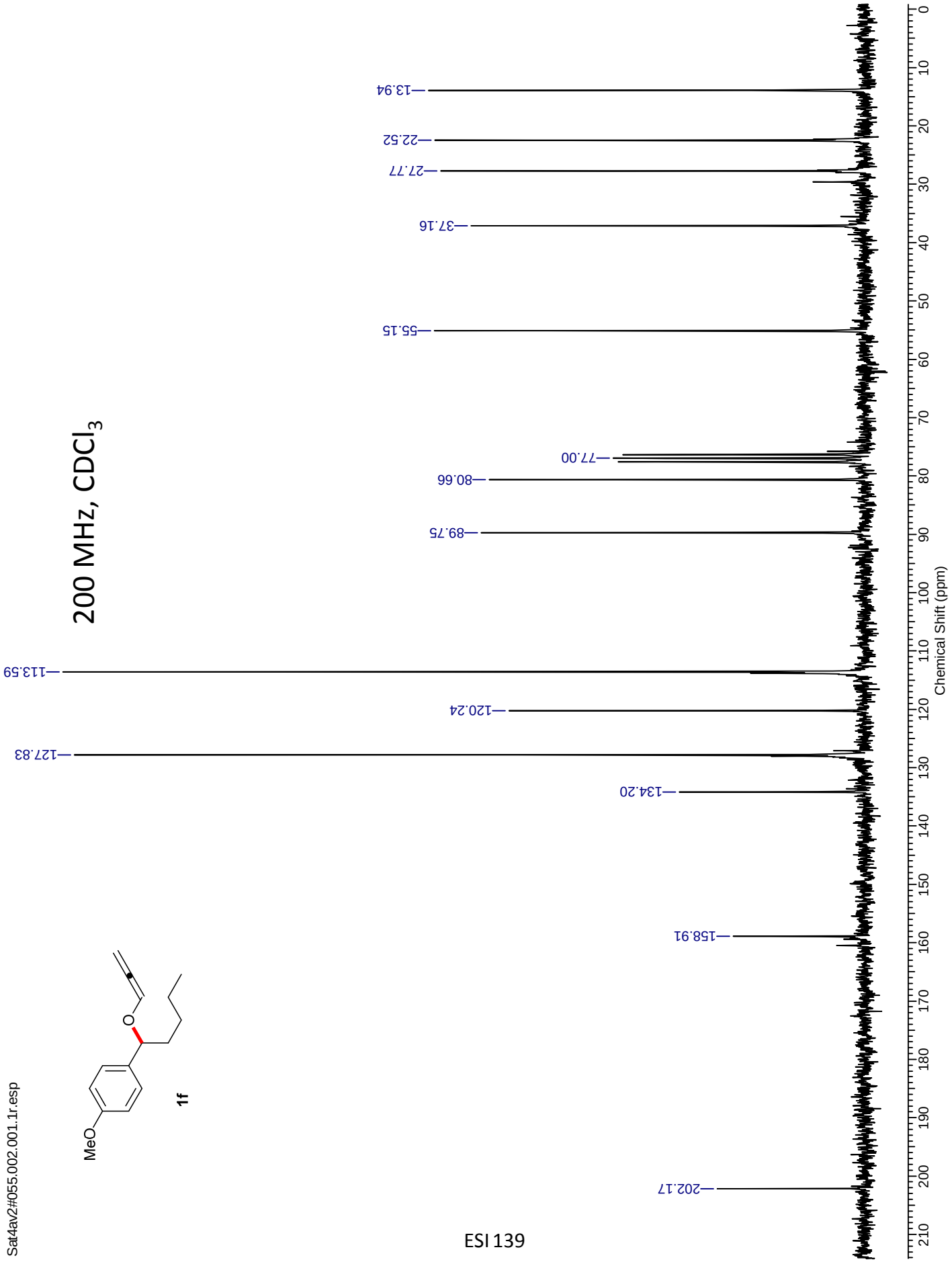

200 MHz, CDCl3
55.19
70.34
90.87
113.75
121.42
129.39
129.53
1c
MeO
O
Chemical Shift (ppm)
0
8
16
24
32
40
48
56
64
72
80
88
96
104
112
120
128
136
144
152
160
168
176
184
192
200
Tue4av2#109.001.001.1r.esp
ESI 134

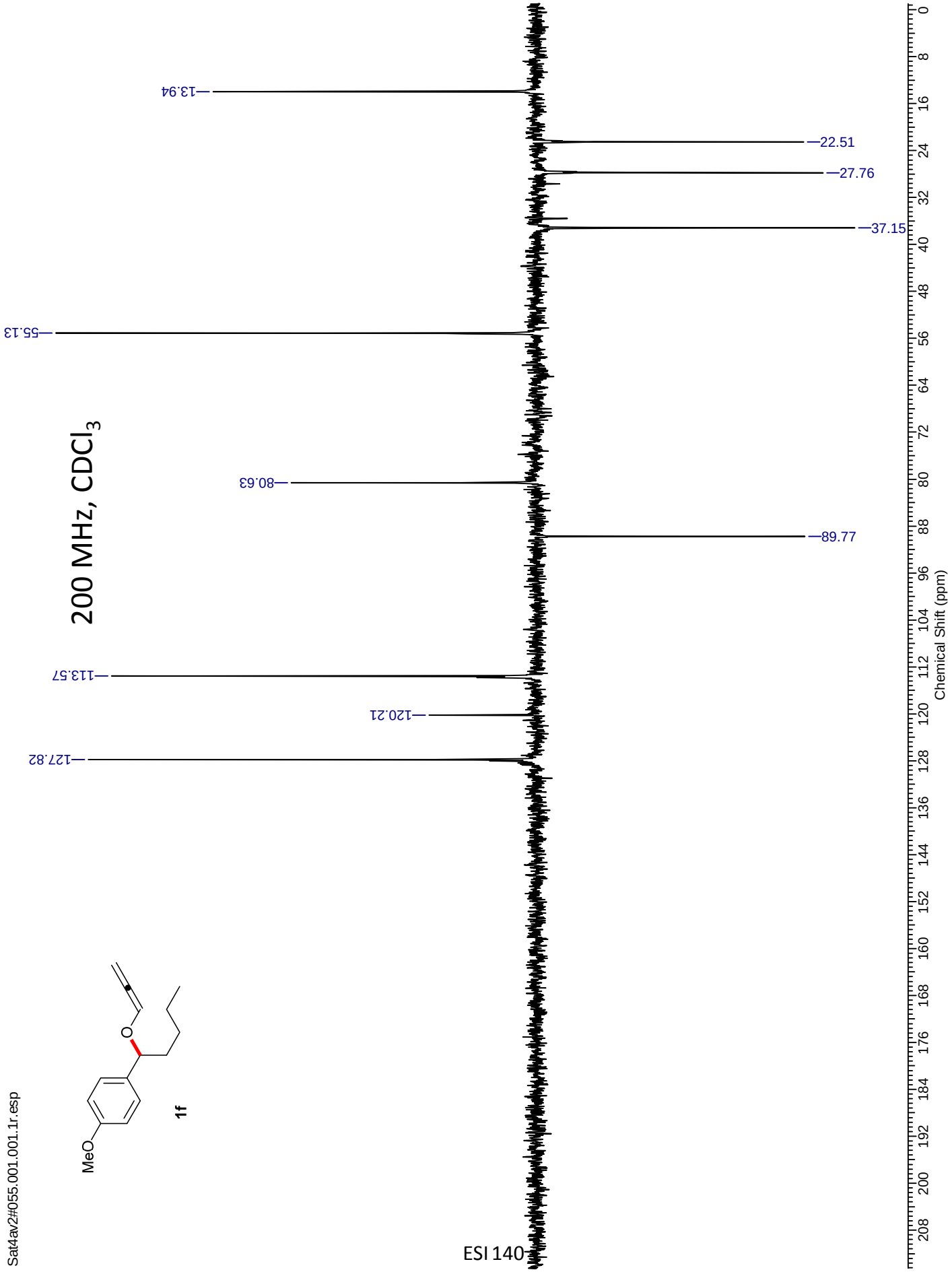

200 MHz, CDCl3
3.82
5.78
5.37
5.34
6.93
6.89
6.83
6.80
7.38
7.37
7.40
7.33
7.29
7.27
3.26
1.01
1.16
1.04
2.20
0.94
3.35
4.10
MeO
1d
Chemical Shift (ppm)
Fri3av2#093.001.001.1r.esp

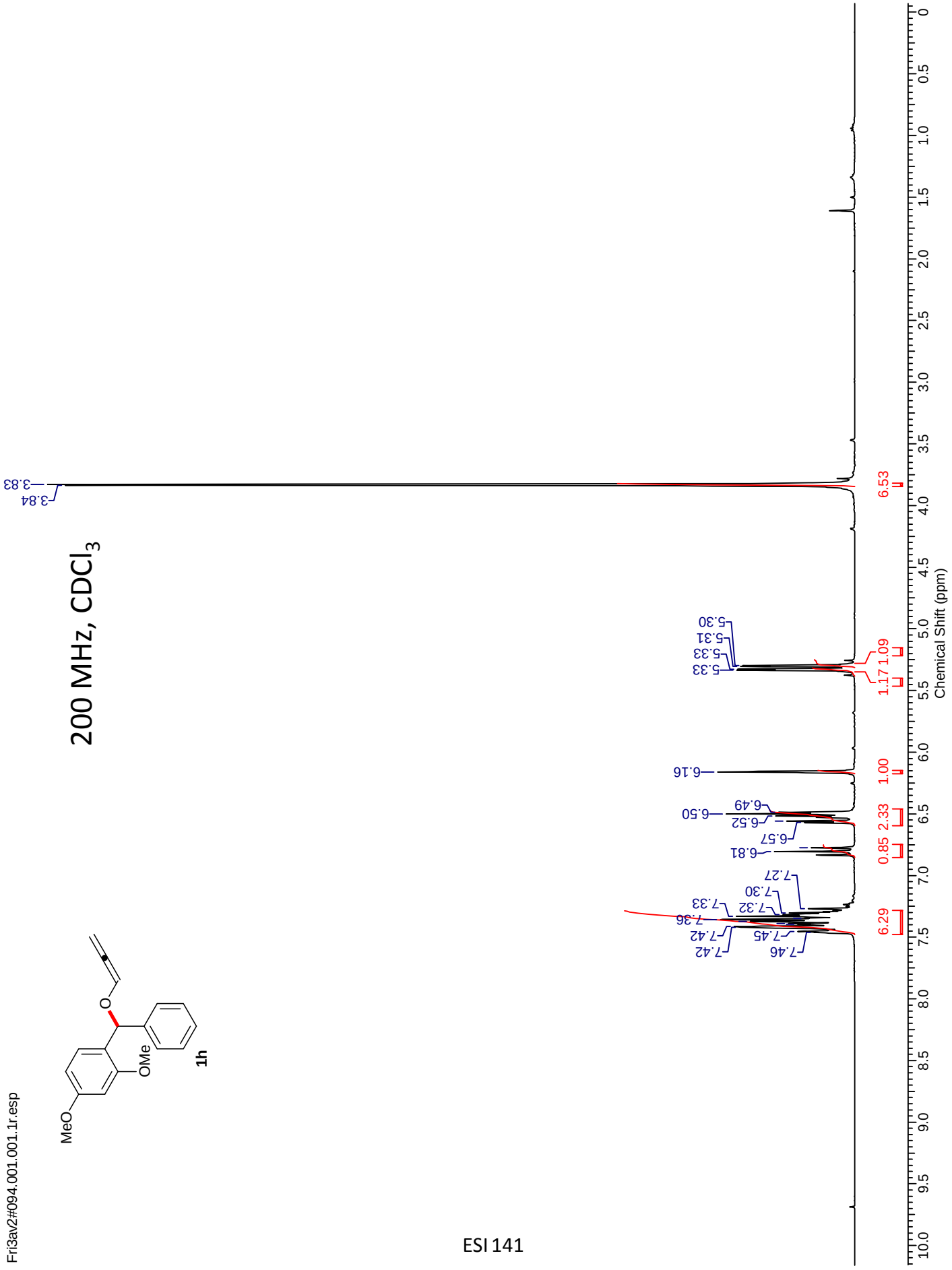

200 MHz, CDCl3
55.13
77.00
81.46
90.58
113.69
120.38
126.83
128.26
128.45
133.49
141.55
159.01
202.05
Chemical Shift (ppm)
MeO
1d
Mon4av2#137.002.001.1r.esp

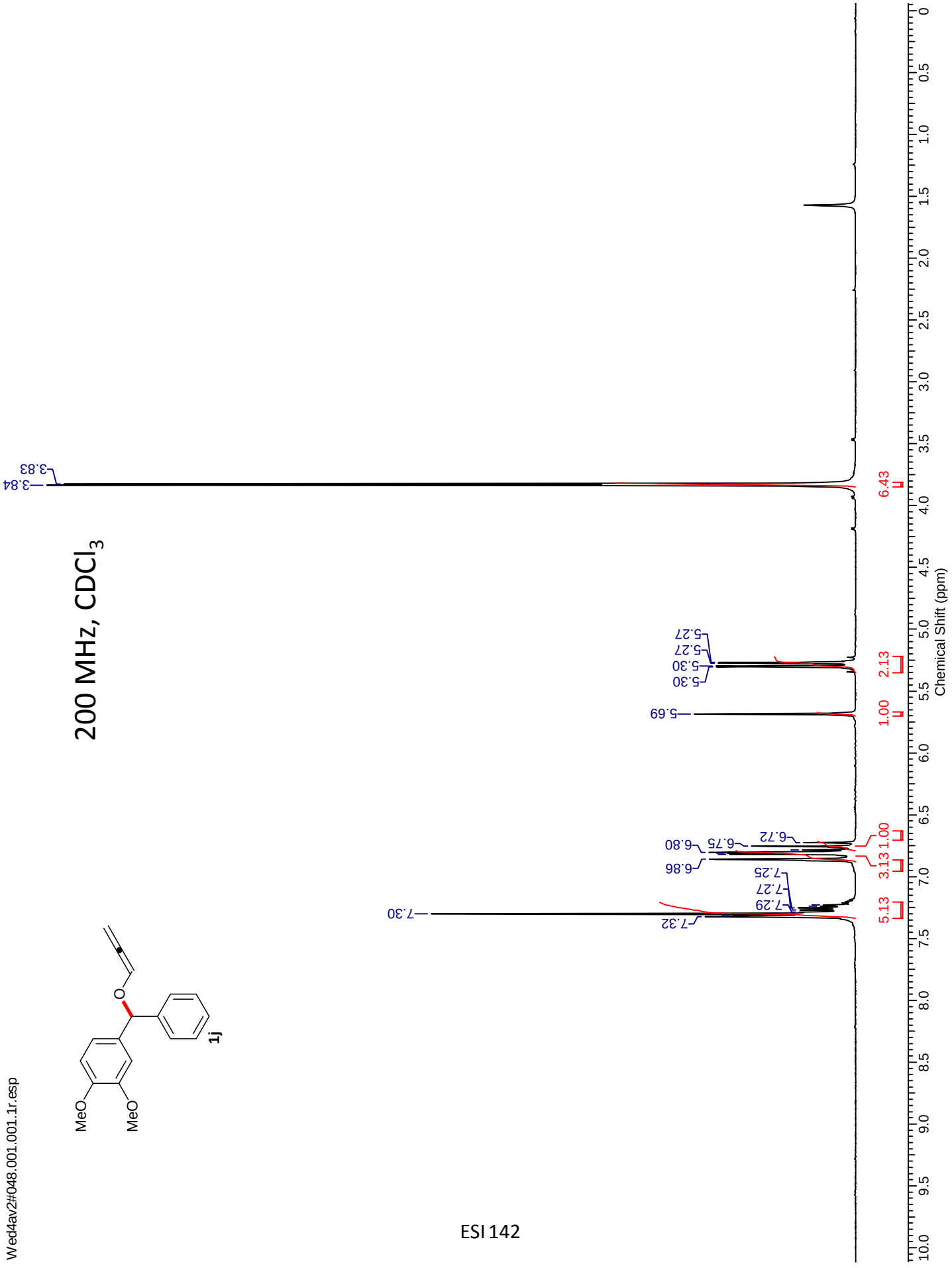

200 MHz, CDCl3
55.13
81.46
90.57
113.69
120.38
126.83
128.44
MeO
1d
Chemical Shift (ppm)
Mon4av2#137.001.001.1r.esp
ESI 137

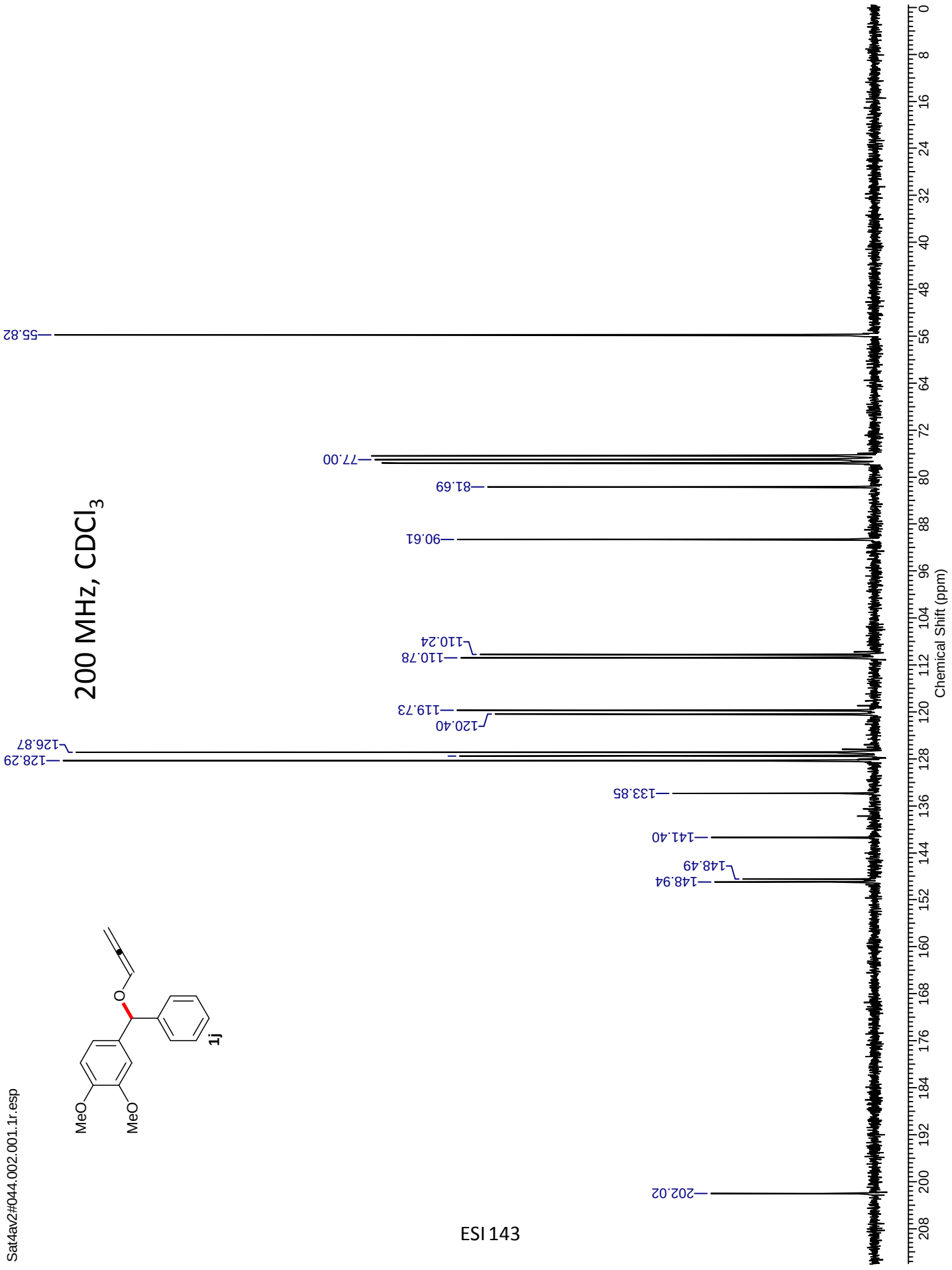

200 MHz, CDCl3
Sat3av2#015.001.001.1r.esp
MeO
O
1f
Chemical Shift (ppm)
0.89
0.93
0.86
1.25
1.28
1.32
1.38
1.33
1.35
1.64
1.69
1.76
1.90
1.94
3.82
4.54
4.57
4.60
5.19
5.22
5.23
5.26
5.30
5.33
5.34
5.37
6.58
6.61
6.64
6.86
6.91
6.92
7.20
7.25
7.27
3.61
5.21
2.49
3.17
0.97
2.07
0.67
2.03
2.00

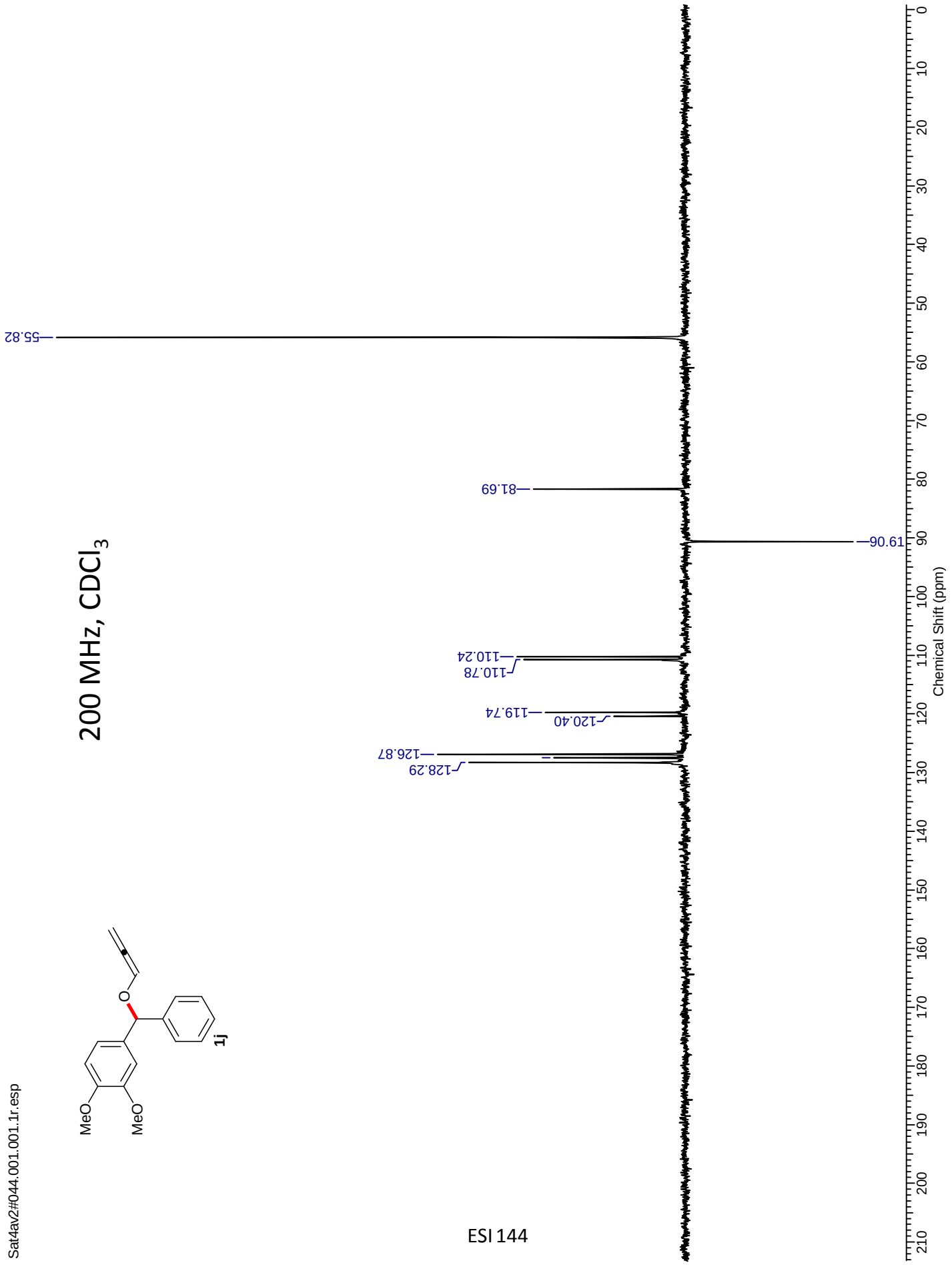

200 MHz, CDCl3
13.94
22.52
27.77
37.16
55.15
77.00
80.66
89.75
113.59
120.24
127.83
134.20
158.91
202.17
Chemical Shift (ppm)
MeO
O
1f
Sat4av2#055.002.001.1r.esp



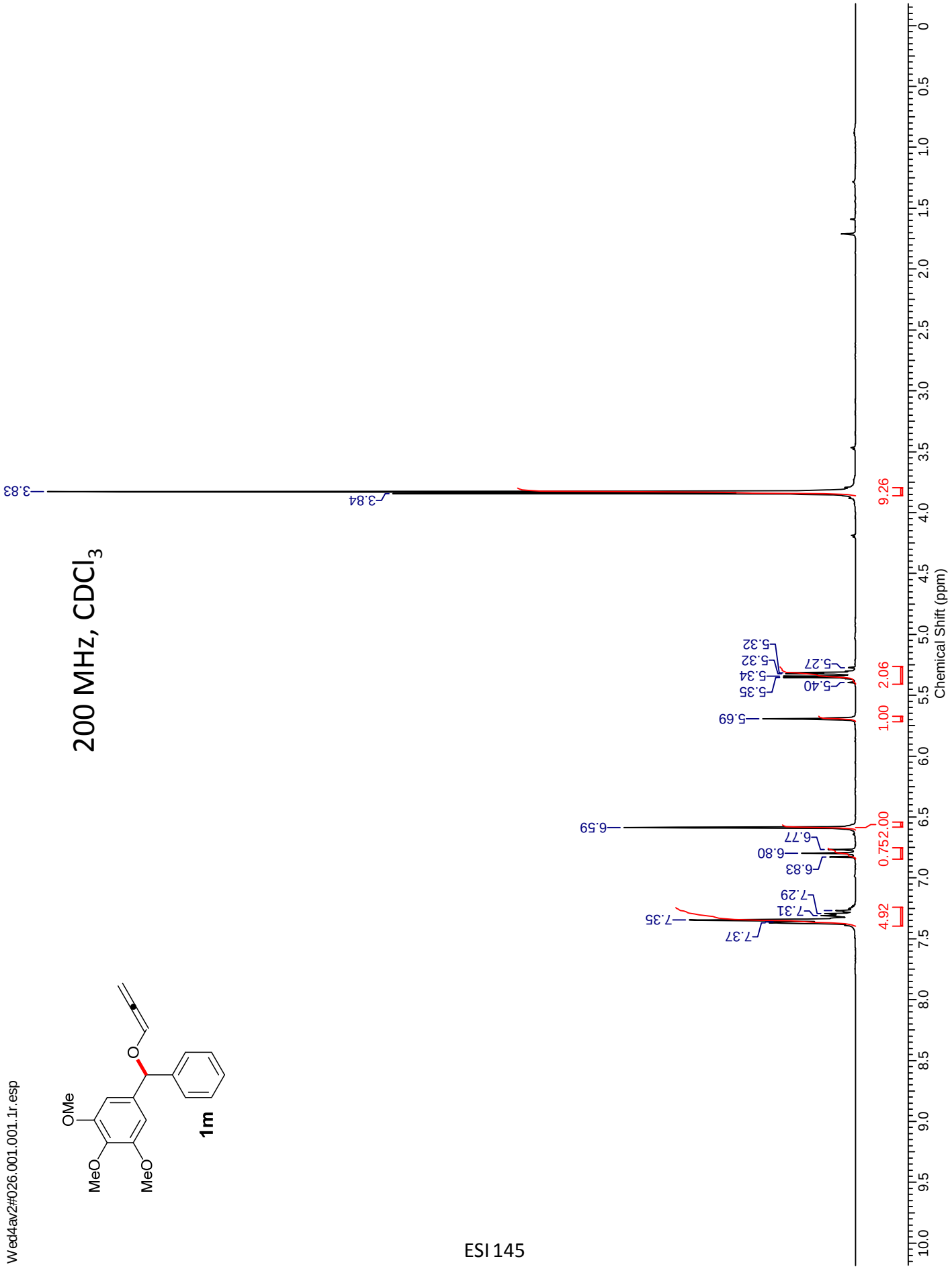

200 MHz, CDCl3
3.84
3.83
6.53
5.33
5.33
5.31
5.30
1.17
1.09
6.16
1.00
6.50
6.49
6.52
6.57
6.81
0.85
2.33
7.27
7.30
7.32
7.33
7.36
7.42
7.42
7.45
7.46
6.29
MeO
OMe
O
1h
Fri3av2#094.001.001.1r.esp
Chemical Shift (ppm)

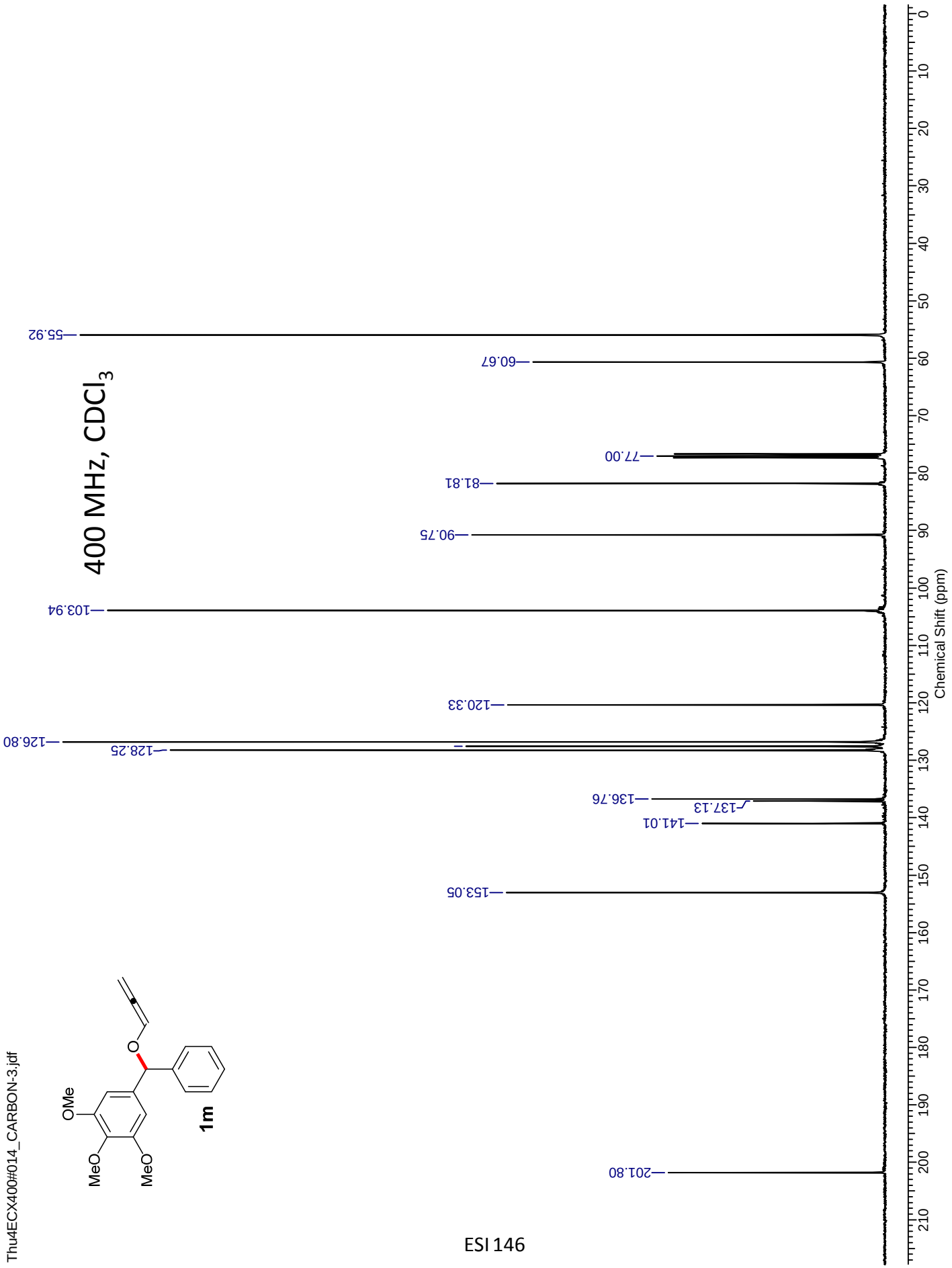

200 MHz, CDCl3
3.84
3.83
5.69
5.30
5.30
5.27
5.27
6.86
6.80
6.75
6.72
7.30
7.32
7.29
7.27
7.25
6.43
2.13
1.00
1.00
3.13
5.13
MeO
MeO
1j
Chemical Shift (ppm)
Wed4av2#048.001.001.1r.esp

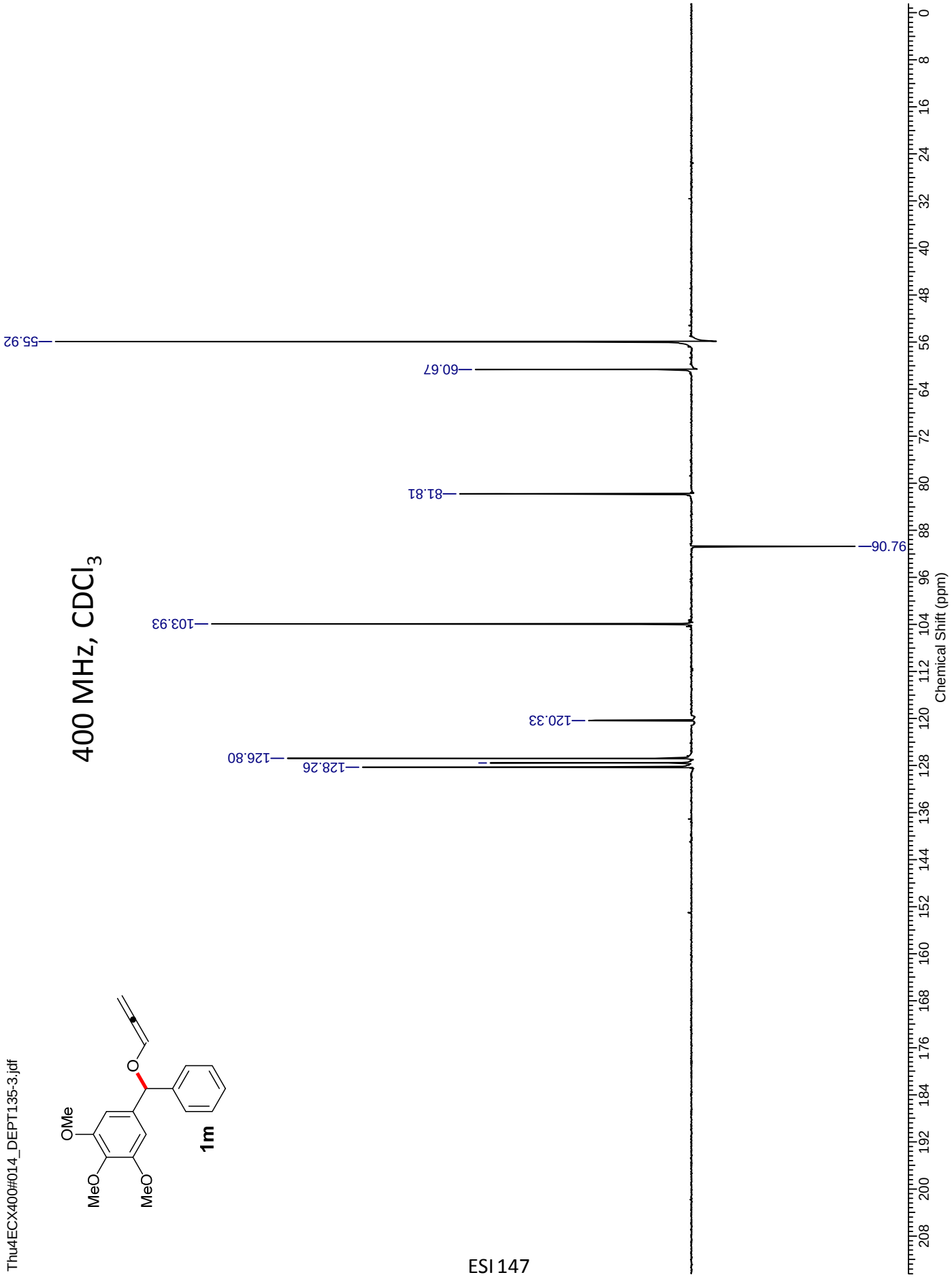

200 MHz, CDCl3
55.82
77.00
81.69
90.61
110.24
110.78
119.73
120.40
126.87
128.29
133.85
141.40
148.49
148.94
202.02
Chemical Shift (ppm)
MeO
MeO
O
1j
Sat4av2#044.002.001.1r.esp

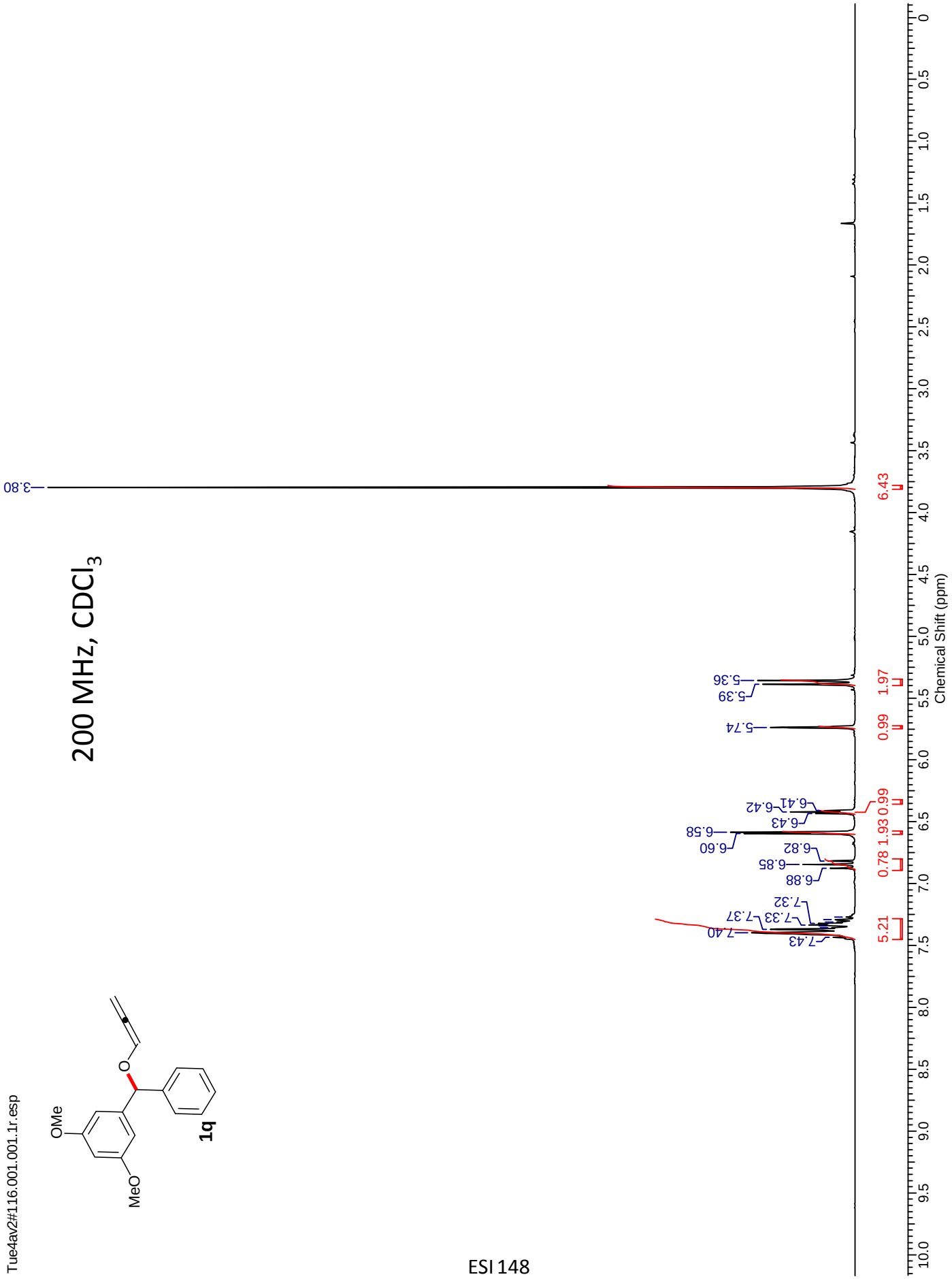

200 MHz, $CDCl_3$
55.82
81.69
90.61
110.24
110.78
119.74
120.40
126.87
128.29
Chemical Shift (ppm)
MeO
MeO
O
1j
Sat4av2#044.001.001.1r.esp

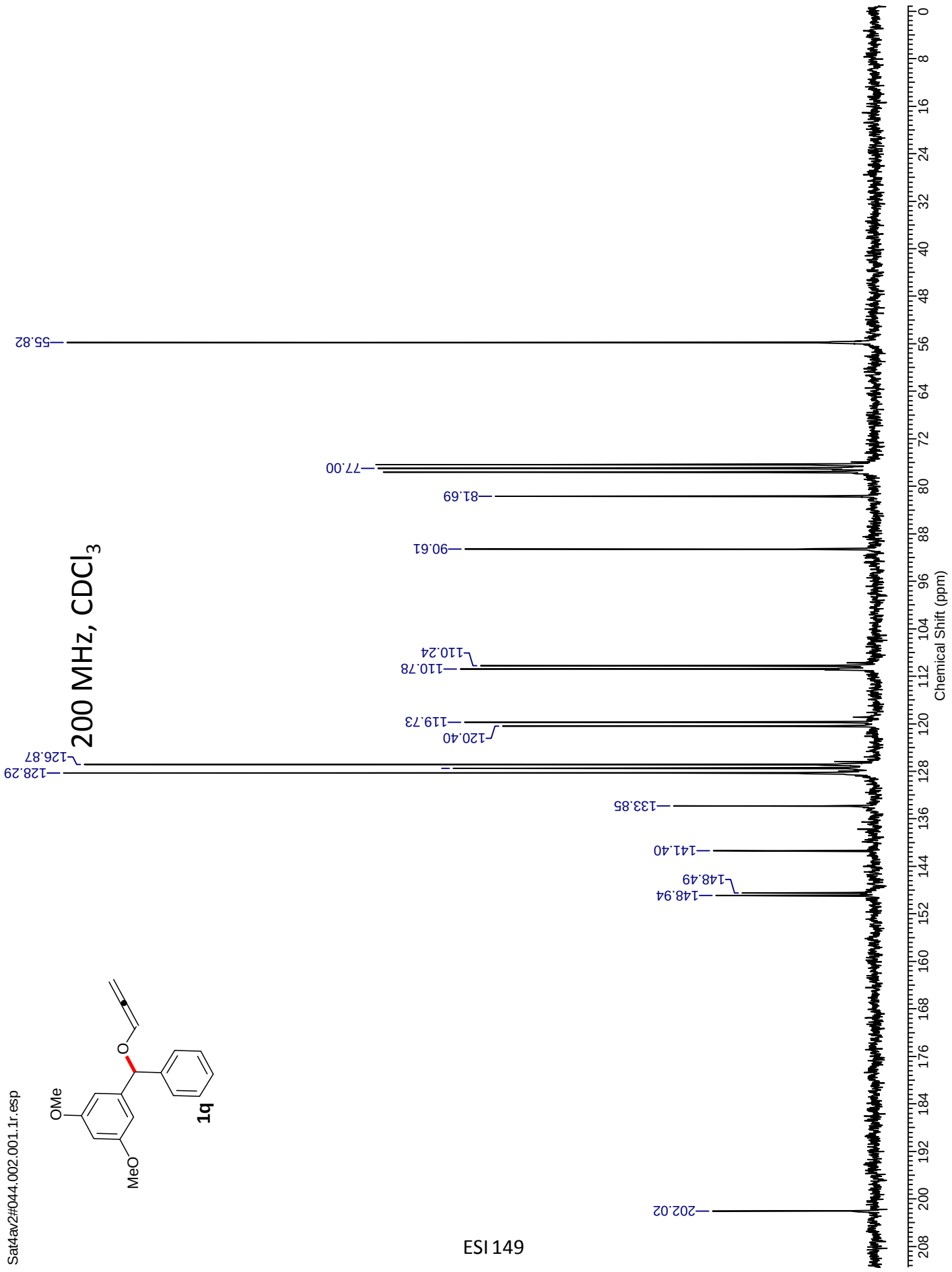
ESI 144

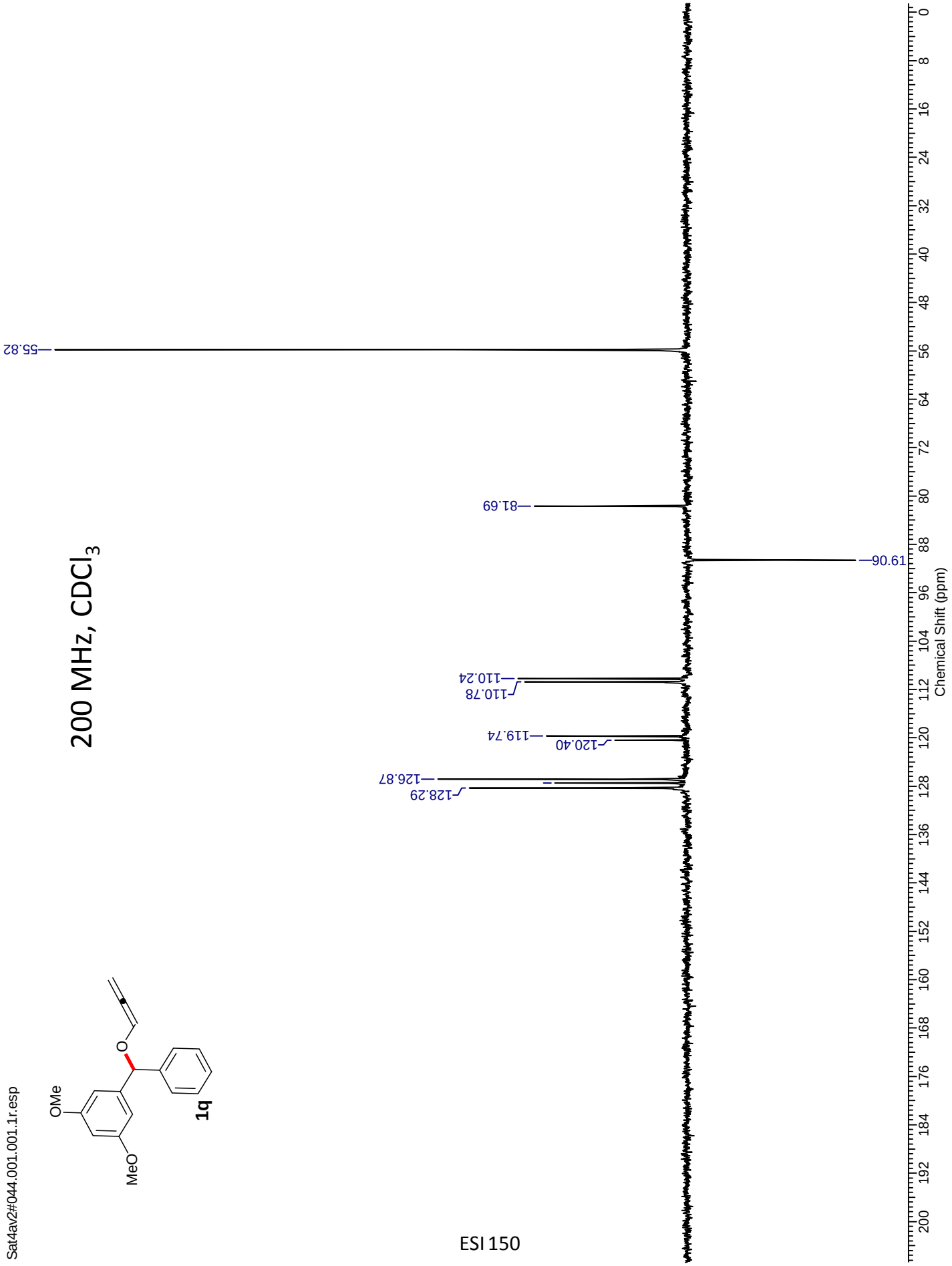

200 MHz, CDCl3
3.83
3.84
5.27
5.32
5.32
5.34
5.35
5.40
5.69
6.59
6.77
6.80
6.83
7.29
7.31
7.35
7.37
9.26
2.06
1.00
2.00
0.75
4.92
OMe
MeO
MeO
O
1m
Chemical Shift (ppm)
Wed4av2#026.001.001.1r.esp

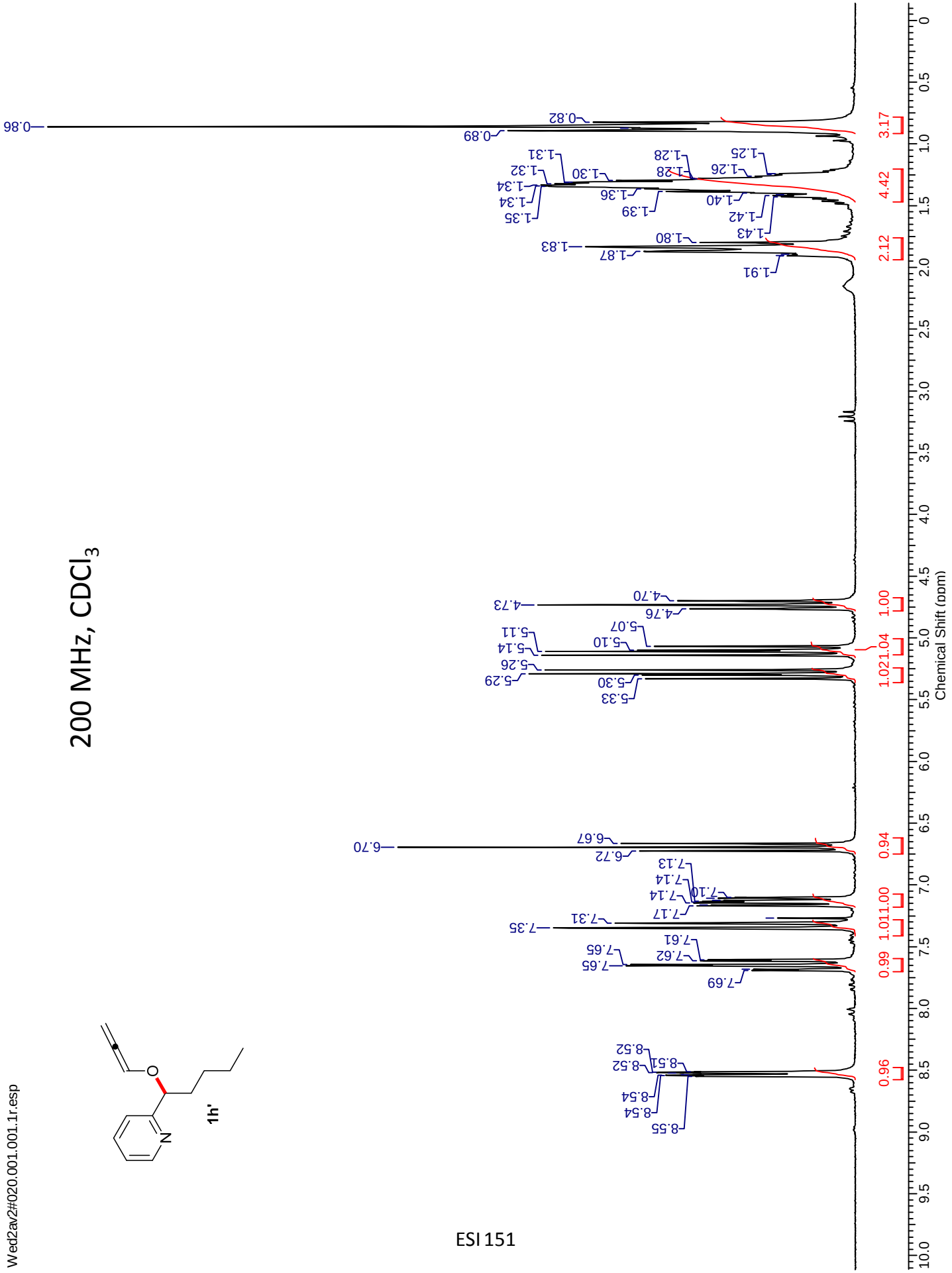

400 MHz, CDCl3
55.92
60.67
77.00
81.81
90.75
103.94
120.33
126.80
128.25
136.76
137.13
141.01
153.05
201.80
Chemical Shift (ppm)
0
10
20
30
40
50
60
70
80
90
100
110
120
130
140
150
160
170
180
190
200
210
OMe
MeO
MeO
O
1m
Thu4ECX400#014_CARBON-3.jdf

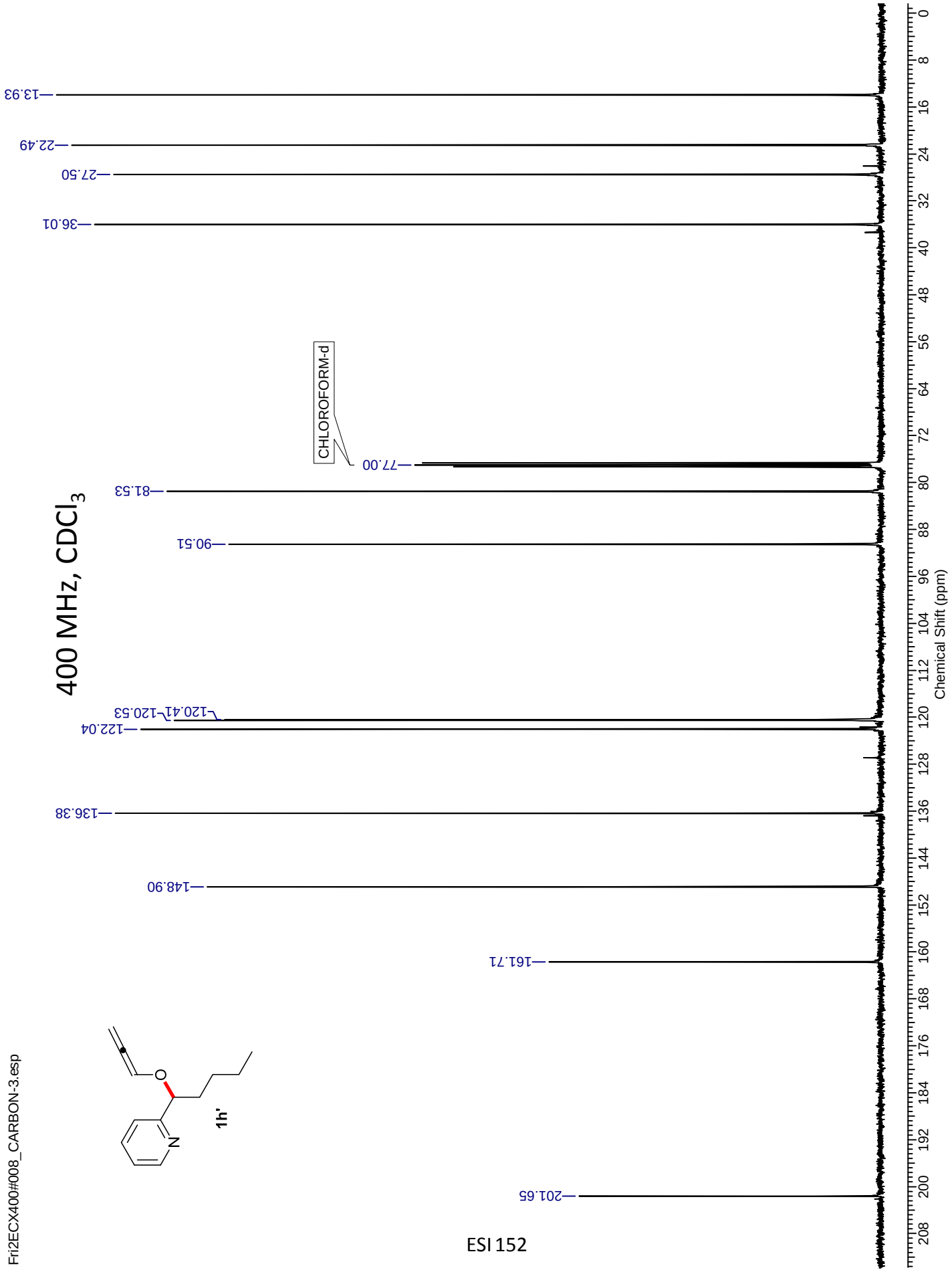

55.92
60.67
81.81
90.76
103.93
120.33
126.80
128.26
400 MHz, CDCl3
Chemical Shift (ppm)
0
8
16
24
32
40
48
56
64
72
80
88
96
104
112
120
128
136
144
152
160
168
176
184
192
200
208
OMe
MeO
MeO
O
1m
Thu4ECX400#014_DEPT135-3.jdf

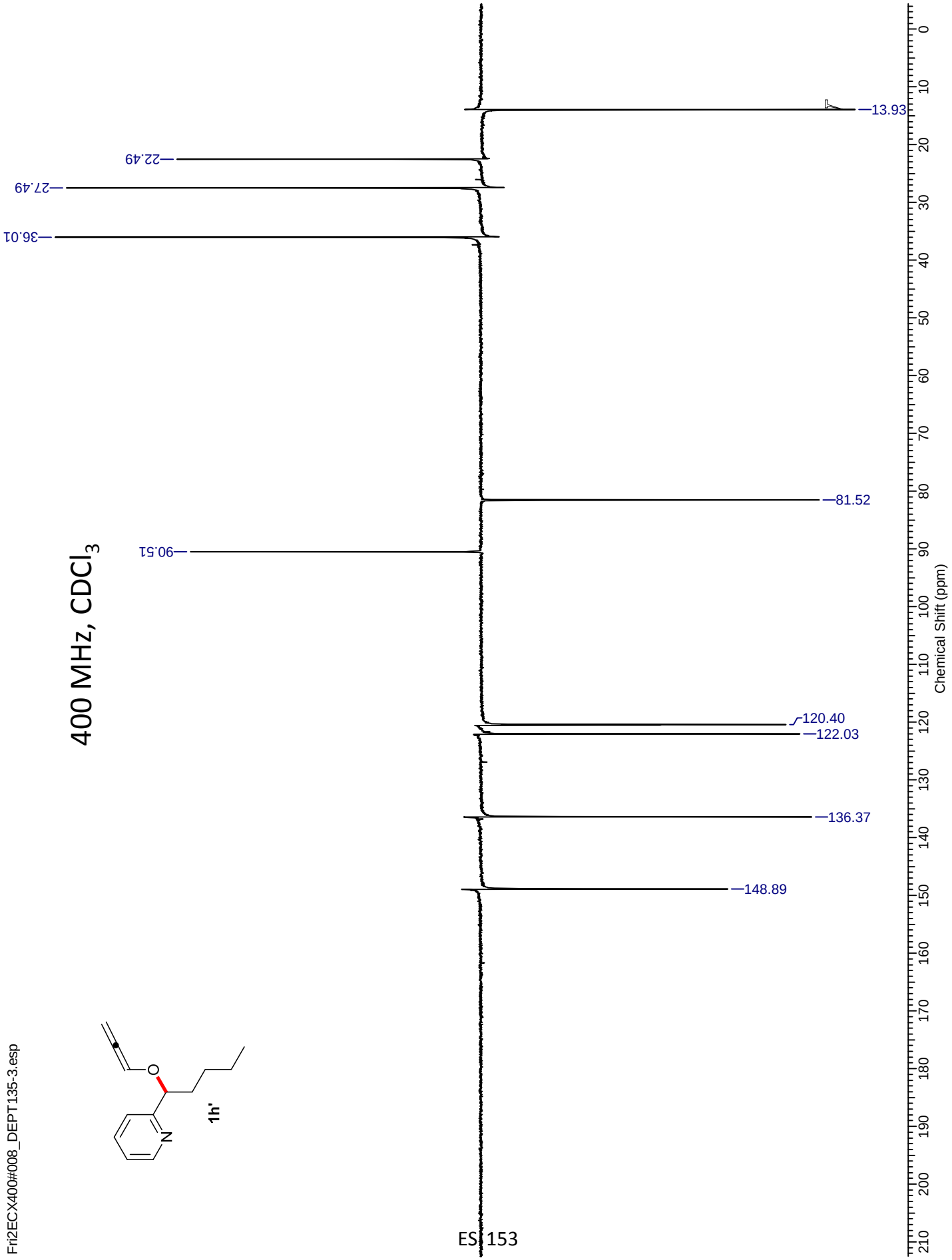

200 MHz, CDCl3
3.80
5.36
5.39
5.74
6.41
6.42
6.43
6.58
6.60
6.82
6.85
6.88
7.32
7.33
7.37
7.40
7.43
6.43
1.97
0.99
0.99
1.93
0.78
5.21
Chemical Shift (ppm)
OMe
MeO
O
1q
Tue4av2#116.001.001.1r.esp


200 MHz, CDCl3
55.82
77.00
81.69
90.61
110.24
110.78
119.73
120.40
126.87
128.29
133.85
141.40
148.49
148.94
202.02
Chemical Shift (ppm)
OMe
MeO
O
1q
Sat4av2#044.002.001.1r.esp


200 MHz, CDCl3
55.82
81.69
90.61
110.24
110.78
119.74
120.40
126.87
128.29
Chemical Shift (ppm)
0
8
16
24
32
40
48
56
64
72
80
88
96
104
112
120
128
136
144
152
160
168
176
184
192
200
OMe
MeO
O
1q
Sat4av2#044.001.001.1r.esp
ESI 150


200 MHz, CDCl3
0.86
0.82
0.89
1.25
1.26
1.28
1.28
1.30
1.31
1.32
1.34
1.34
1.35
1.36
1.39
1.40
1.42
1.43
1.80
1.83
1.87
1.91
4.70
4.73
4.76
5.07
5.10
5.11
5.14
5.26
5.29
5.30
5.33
6.67
6.70
6.72
7.13
7.14
7.14
7.17
7.31
7.35
7.61
7.62
7.65
7.65
7.69
8.51
8.52
8.52
8.54
8.54
8.55
3.17
4.42
2.12
1.00
1.04
1.02
0.94
1.00
1.01
0.99
0.96
Chemical Shift (ppm)
0
0.5
1.0
1.5
2.0
2.5
3.0
3.5
4.0
4.5
5.0
5.5
6.0
6.5
7.0
7.5
8.0
8.5
9.0
9.5
10.0
O
N
1h'
Wed2av2#020.001.001.1r.esp
ESI 151




400 MHz, CDCl3
13.93
22.49
27.49
36.01
81.52
90.51
120.40
122.03
136.37
148.89
Chemical Shift (ppm)
0
10
20
30
40
50
60
70
80
90
100
110
120
130
140
150
160
170
180
190
200
210
O
N
1h'
Fri2ECX400#008_DEPT135-3.esp