Electronic Supplementary Material (ESI) for Chemical Communications

Supporting Information

Synthesis of pseudo indoxyl derivatives via sequential Cu-catalyzed S_NAr and Smalley cyclization

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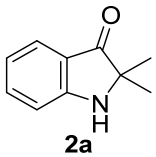
1	 **2a**	Characterization data	SI9
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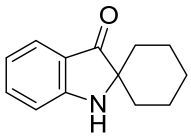
2	 **2b**	Characterization data	SI 9
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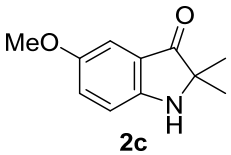
3	 **2c**	Characterization data	SI 9
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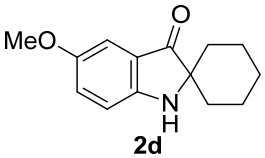
4	 **2d**	Characterization data	SI 10
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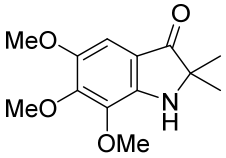
5	 **2e**	Characterization data	SI 10
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		DEPT Spectrum	SI 38
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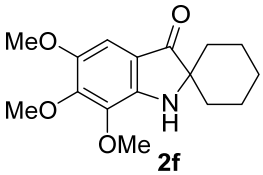
6	 **2f**	Characterization data	SI 11
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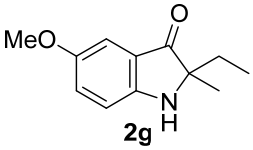
7	 **2g**	Characterization data	SI 11
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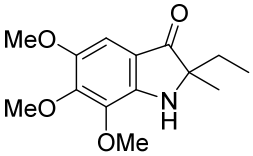
8	 **2h**	Characterization data	SI 12
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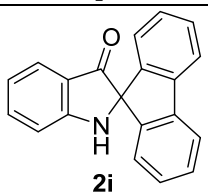
		Mass Spectrum (HRMS + ESI)	SI 54 & SI 55

S. No	Compound	Data	Page No
9	 **2i**	Characterization data	SI 12
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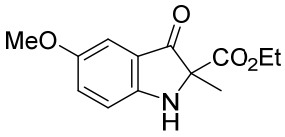
10	 **2j**	Characterization data	SI 13
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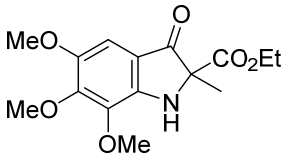
11	 **2k**	Characterization data	SI 13
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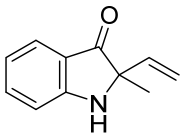
		Mass Spectrum (HRMS + ESI)	SI 69 & SI 70
12	 **2l**	Characterization data	SI 13
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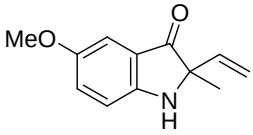
13	 **2m**	Characterization data	SI 14
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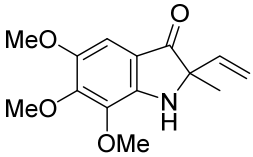
14	 **2n**	Characterization data	SI 14
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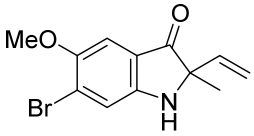
15	 **2o**	Characterization data	SI 15
		^{1}H NMR Spectrum	SI 86
		^{13}C NMR Spectrum	SI 87
		DEPT Spectrum	SI 88
		Mass Spectrum (HRMS)	SI 89

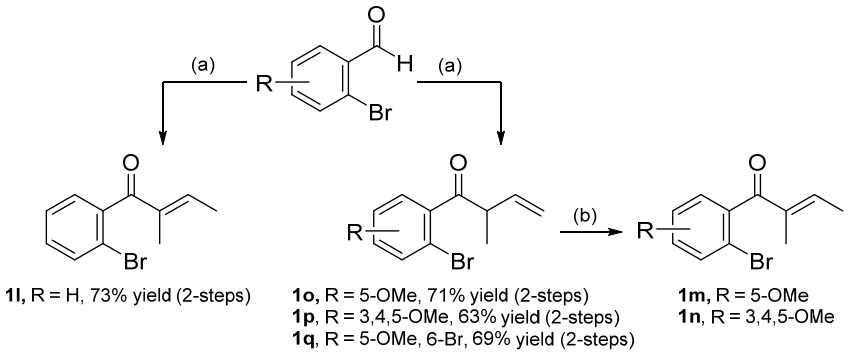
General Remarks

Reactions were carried out in anhydrous solvents under an atmosphere of argon in oven-dried glassware. Commercial reagents and solvents were used without purification. Column Chromatography was carried out by using spectrochem silica gel (60–120, 100–200, 230–400 mesh). ^{1}H and ^{13}C NMR spectroscopy measurements were carried out on Bruker AC 200 MHz or Bruker DRX 400 and Bruker DRX 500 MHz spectrometers, and TMS was used as an internal standard. ^{1}H and ^{13}C NMR chemical shifts are reported in ppm downfield from Chloroform-d (δ = 7.25) or TMS and coupling constants (*J*) are reported in Hertz (Hz). The following abbreviations are used to designate signal multiplicity: s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet, b = broad. The multiplicity of ^{13}C NMR signals was assigned with the help of DEPT spectra and the abbreviations used: s = singlet, d = doublet, t = triplet, q = quartet, represent C (quaternary), CH, CH_2 and CH_3 respectively. Mass spectroscopy was carried out on PI QStar Pulsar (Hybrid Quadrupole-TOF LC/MS/MS) and 4800 plus MALDI TOF/TOF Applied Biosystem spectrometer.

A. Synthesis of substrates 1a–1k

Compounds **1a** and **1b** have been synthesized according to the reported procedure and the same has been followed for the preparation of compounds **1c–1h**.[1] Substrate **1i** has been prepared by acylation of fluorene in the presence of LDA. Substrates **1j** and **1k** have been prepared by following a sequence of Zn-mediated Reformatsky followed by the oxidation.[2]

B. General procedure for Preparation of α-bromophenyl sec-alkenyl ketones (1l to 1q):



Scheme S1. *Reagents and conditions*: a) (i) crotyl bromide, Zn, THF, 0 °C to rt; (ii) IBX, EtOAc, reflux, 3-4h; b) DBU, DCM, 5m.

To a vigorously stirred suspension of Zn (5.0 eq.) and propargyl bromide (3.0 eq.) in THF (10 mL) was added a solution of aldehyde (1.0 eq.) in THF (10 mL) and the stirring was continued for another 30 min. The reaction mixture was cooled to 0 ºC, sat. NH_4Cl (10 mL) was added drop wise for 30 min and stirring was continued for additional 2 h. Reaction mixture was filtered through *celite* pad and the solvent was evaporated under vacuum. The crude mixtures dilute with water and extracted with ethyl acetate (3 X 25ml), washed with brine, dried (Na_2SO_4), and concentrated. The crude residue was used for next step without further purification.

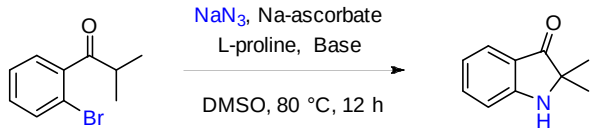
At rt, a solution of the above crude alcohol in ethyl acetate (10 mL) was treated with IBX (1.3 eq.) stirred at reflux temperature for 3 h. After complete consumption of starting material, the reaction mixture was cooled to rt and filtered through a celite pad. Solvent was evaporated under reduced pressure and the crude residue was purified over silica gel column (ethyl acetate and pet ether as eluent) to obtain the keto compounds (**1l, 1o, 1p** and **1q**) in 60–80% yields over two steps.

Compound **1o** and **1p** have been stirred with DBU in CH_2Cl_2 for 5 min to afford the required **1m** and **1n** respectively in quantitative yields.

C. Representative Procedure for synthesis of 2,2-disubstituted indolin-3-one:

To a solution of α-bromophenyl sec-alkyl\alkenyl ketones (1.0 mmol) in DMSO, were added L-proline (0.2 mmol), K_2CO_3 (1.5 mmol), $CuSO_4{:}5H_2O$ (0.2 mmol), sodium ascorbate (0.2 mmol), and NaN_3 (1.2 mmol). The mixture was stirred for 12–18 h at 70 °C (oil bath temperature). The reaction mixture was diluted with 30 mL of water and extracted with ethyl acetate (3 X 30 mL). Combined organic layer was dried (Na_2SO_4) and evaporated under reduced pressure. The crude was purified over silica gel (ethyl acetate and pet ether as eluent) to procure 2,2-disubstituted indolin-3-one in moderate to good yields.

Table S1. Optimization of Reaction Condition with various Cu–catalysts



1a **2a**

Entry[a]	Cu-catalyst	Base	Yield[c]
1	$CuSO_4\ 5H_2O$	K_2CO_3	65%
2	$CuSO_4\ 5H_2O$	KOH	23%
3	$CuSO_4\ 5H_2O$	Cs_2CO_3	57%
4	$CuSO_4\ 5H_2O$	Triton B	59%
5	$CuSO_4\ 5H_2O$	TBAOH	51%
6	$CuSO_4\ 5H_2O$	Et_3N	41%
7	$CuSO_4\ 5H_2O$	------	33%
8	CuI	K_2CO_3	36%
9	CuI	KOH	61%
10	CuI	-------	46%[b]
11	CuI	-------	36%
12	CuI	-------	53%[b, d]
13	Cu_2O	K_2CO_3	36%[b]
14	$Cu(OAc)_2$	K_2CO_3	43%

[a] all reactions performed with 1.2 eq NaN_3; 20 mol% L-proline, Na-ascorbate, Cu-cat in DMSO; [b] reaction without Na-ascorbate; [c] isolated yields [d] PEG as a solvent

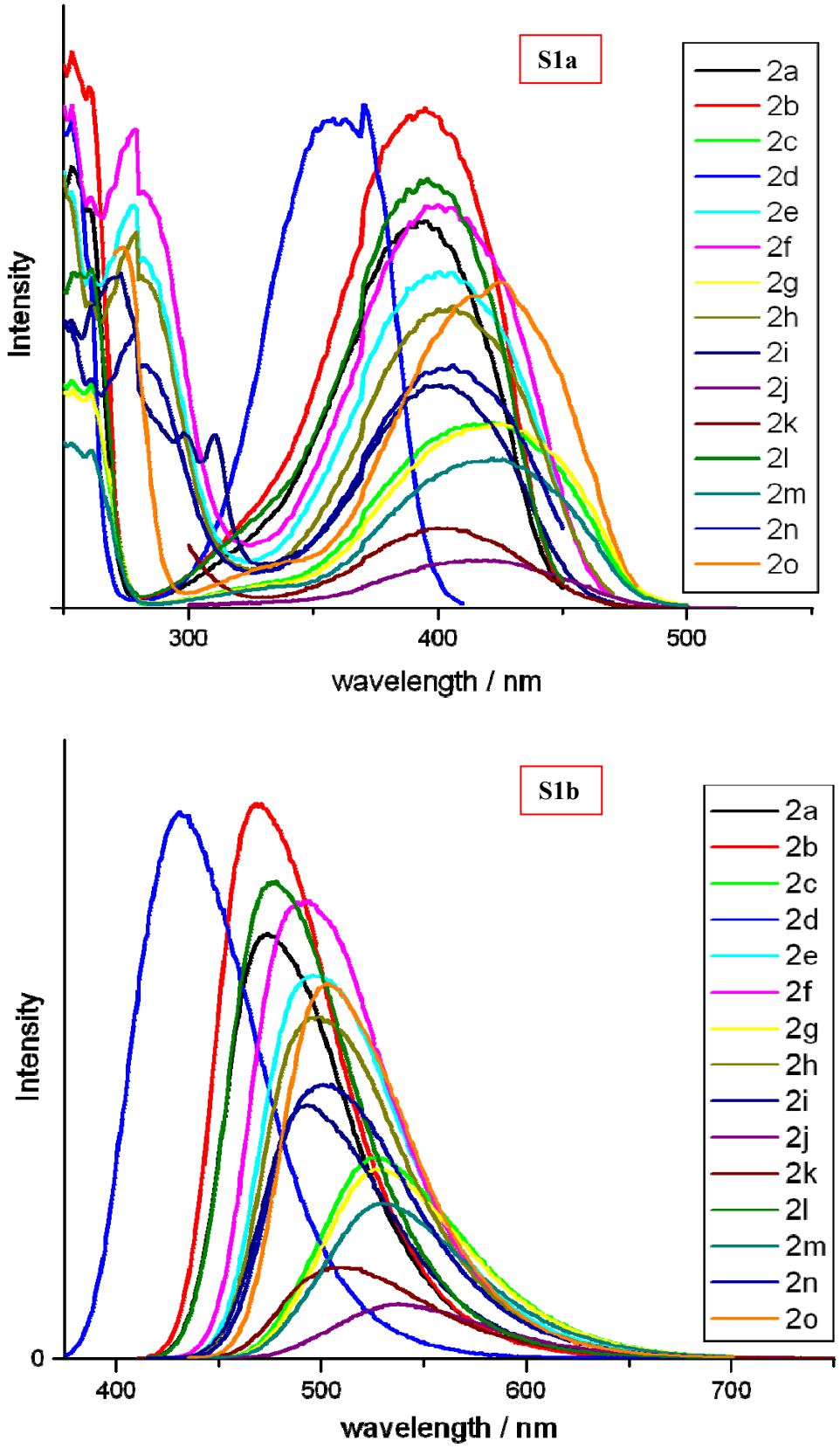


Figure S1. Absorption spectra (**S1a**) and emission spectra (**S1b**) of compound **2a-2o**

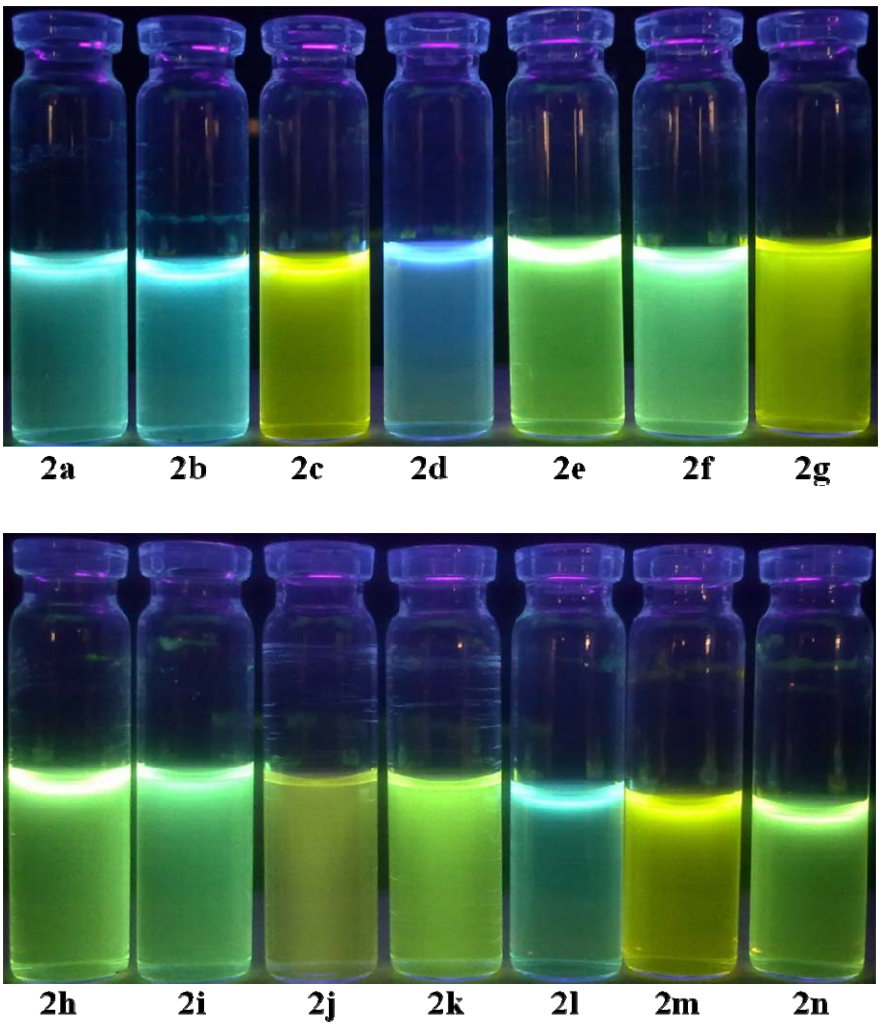
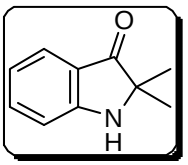


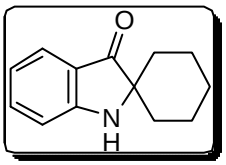
Figure S2.Photographs of compounds **2a-2n** in MeOH under 365nm irradiation.

2,2-Dimethylindolin-3-one (2a)[3]:-



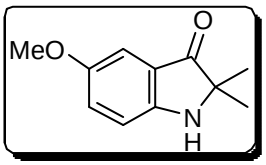
Brown solid; 65% yield; R_f = 0.4 (10% ethyl acetate/pet. ether); mp: 81–82 °C; IR (Nujol)*v*: 3363, 2924, 2855, 1681, 1619, 1464, 1375, 1142, 993, 760, 648 cm^{-1}; ^1H NMR (200 MHz, $CDCl_3$): δ 1.31 (s, 6H), 4.69 (s, 1H), 6.75–6.84 (m, 2H), 7.43 (ddd, J = 1.4, 7.1, 8.4 Hz, 1H), 7.60 (dt, J = 1.0, 7.7 Hz, 1H); ^{13}C NMR (50 MHz, $CDCl_3$): δ 24.4 (q, 2C), 63.9 (s), 112.5 (d), 118.7 (d), 119.5 (s), 125.0 (d), 137.2 (d), 159.6 (s), 205.2 (s) ppm; ESI-MS: 162.10 (65%, $[M+H]^+$); HRMS (ESI+): calcd. for $C_{10}H_{11}NOH^+$ 162.0913, found 162.0913.

Spiro[cyclohexane-1,2'-indolin]-3'-one (2b)[3]:-



Yellow solid; 71% yield; R_f = 0.5 (10% ethyl acetate/pet. ether); mp: 133–134 °C; IR (Nujol)*v*: 3330, 2924, 2854, 1669, 1620, 1463, 1377, 1141, 971, 751, 664 cm^{-1}; ^1H NMR (200 MHz, $CDCl_3$): δ 1.34–1.54 (m, 5H), 1.68–1.91 (m, 5H), 5.04 (s, 1H), 6.79 (dt, J = 0.8, 7.8 Hz, 1H), 6.86 (br d, J = 8.3 Hz, 1H), 7.42 (ddd, J = 1.3, 7.1, 8.4 Hz, 1H), 7.60 (br d, J = 7.8 Hz, 1H); ^{13}C NMR (50 MHz, $CDCl_3$): δ 22.5 (t, 2C), 24.8 (t), 32.8 (t, 2C), 66.9 (s), 112.6 (d), 118.8 (d), 120.4 (s), 125.0 (d), 137.0 (d), 159.9 (s), 204.9 (s) ppm; ESI-MS (*m/z*): 202.05 (100%, $[M+H]^+$), 224.01 (30%, $[M+Na]^+$); HRMS (ESI+): calcd. for $C_{13}H_{15}NOH^+$ 202.1226, found 202.1226**.**

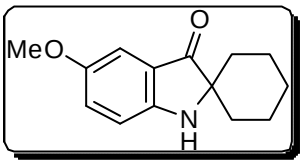
5-Methoxy-2,2-dimethylindolin-3-one (2c)[3]:-



Brown solid; 66% yield; R_f = 0.2 (10% ethyl acetate/pet. ether); mp: 56–57 °C; IR (Nujol)*v*: 3324, 2924, 2854, 1679, 1494, 1462, 1376, 1234, 1139, 1029, 913, 793 cm^{-1}; ^1H NMR (200 MHz, $CDCl_3$): δ 1.32 (s, 6H), 3.76 (s, 3H), 4.30 (s, 1H), 6.81 (dd, J = 0.5, 8.8 Hz, 1H), 7.04 (d, J = 2.6

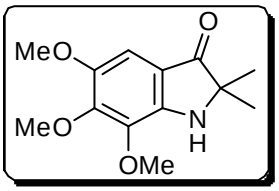
Hz, 1H), 7.14 (dd, J = 2.7, 8.8 Hz, 1H); ^{13}C NMR (50 MHz, $CDCl_3$): δ 24.6 (q, 2C), 55.8 (q), 64.9 (s), 104.7 (d), 114.3 (d), 120.0 (s), 127.8 (d), 153.4 (s), 155.4 (s), 205.5 (s) ppm; ESI-MS (*m/z*): 190.07 (100%, $[M–H]^+$), 192.10 (25%, $[M+H]^+$); HRMS (ESI+): calcd. for $C_{11}H_{13}NO_2H^+$ 192.1019, found 192.1019**.**

5'-Methoxyspiro[cyclohexane-1,2'-indolin]-3'-one (**2d**):-



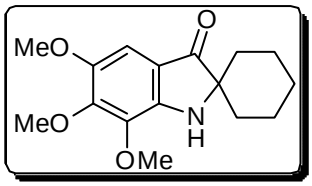
Yellow solid; 68% yield; R_f = 0.3 (15% ethyl acetate/pet. ether); mp: 63–64 °C; IR ($CHCl_3$)ν: 3404, 2925, 1714, 1601, 1489, 1460, 1377, 1269, 1217, 1118, 1029, 946, 811, 765, 721 cm^{-1}; ^{1}H NMR (200 MHz, $CDCl_3$): δ 1.35–1.50 (m, 5H), 1.68–1.85 (m, 5H), 3.75 (s, 3H), 4.71 (s, 1H), 6.84 (d, J = 8.7 Hz, 1H), 7.04 (d, J = 2.6 Hz, 1H), 7.13 (dd, J = 2.7, 8.8, 1H); ^{13}C NMR (50 MHz, $CDCl_3$): δ 21.7 (t, 2C), 24.6 (t), 31.7 (t, 2C), 55.9 (s), 90.4 (s), 104.3 (d), 114.6 (d), 120.0 (s), 128.1 (d), 154.6 (s), 166.6 (s), 204.5 (s) ppm; ESI-MS (*m/z*): 230.05 (100%, $[M–H]^+$), 232.09 (45%, $[M+H]^+$); HRMS (ESI+): calcd. for $C_{14}H_{17}NO_2H^+$ 232.1332, found 232.1331.

5,6,7-Trimethoxy-2,2-dimethylindolin-3-one (**2e**):-



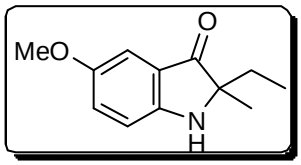
Yellow solid; 69% yield; R_f = 0.3 (20% ethyl acetate/pet. ether); mp: 74–75 °C; IR (Nujol)ν: 3360, 2854, 1704, 1617, 1459, 1376, 1297, 1133, 975, 722 cm^{-1}; ^{1}H NMR (200 MHz, $CDCl_3$): δ 1.32 (s, 6H), 3.81 (s, 3H), 3.93 (s, 3H), 3.96 (s, 3H), 4.42 (s, 1H), 6.85 (s, 1H); ^{13}C NMR (50 MHz, $CDCl_3$): δ 24.9 (q, 2C), 56.3 (q), 60.5 (q), 61.1 (q), 64.6 (s), 100.2 (d), 114.1 (s), 139.0 (s), 148.0 (s), 149.9 (s), 150.4 (s), 204.3 (s) ppm; ESI-MS: 252.07 (75%, $[M+H]^+$), 273.96 (55%, $[M+Na]^+$); HRMS (ESI+): calcd. for $C_{13}H_{17}NO_4H^+$ 252.1230, found 252.1227.

5',6',7'-Trimethoxyspiro[cyclohexane-1,2'-indolin]-3'-one (**2f**):



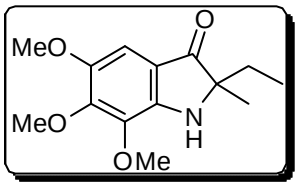
Brown solid; 74% yield; R_f = 0.4 (20% ethyl acetate/pet. ether); mp: 122–123 °C; IR (Nujol)ν: 3283, 2923, 1659, 1621, 1459, 1376, 1310, 1252, 1102, 1042, 964, 898, 783 cm^{-1}; ^1H NMR (200 MHz, $CDCl_3$): δ 1.37–1.50 (m, 5H), 1.72–1.89 (m, 5H), 3.80 (s, 3H), 3.96 (s, 6H), 4.76 (s, 1H), 6.84 (s, 1H); ^{13}C NMR (50 MHz, $CDCl_3$): δ 22.6 (t, 2C), 24.7 (t), 32.9 (t, 2C), 56.2 (q), 60.5 (q), 61.1 (q), 67.6 (s), 100.0 (d), 114.9 (s), 139.0 (s), 147.9 (s), 149.6 (s), 150.6 (s), 204.1 (s) ppm; ESI-MS: 292.17 (35%, $[M+H]^+$), 314.03 (65%, $[M+Na]^+$); HRMS (ESI+): calcd. for $C_{16}H_{21}NO_4H^+$ 292.1543, found 292.1544.

2-Ethyl-5-methoxy-2-methylindolin-3-one (**2g**):



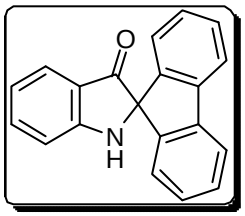
Yellow solid; 69% yield; R_f = 0.3 (15% ethyl acetate/pet. ether); mp: 76–77 °C; IR ($CHCl_3$)ν: 3341, 2967, 2927, 1670, 1496, 1455, 1262, 1228, 1140, 1029, 821, 788 cm^{-1}; ^1H NMR (200 MHz, $CDCl_3$): δ 0.79 (t, J = 7.5 Hz, 3H), 1.2 (s, 3H), 1.66 (dq, J = 7.3, 14.0 Hz, 1H), 1.73 (dq, J = 7.5, 14.9 Hz, 1H), 3.75 (s, 3H), 4.27 (s, 1H), 6.82 (d, J = 8.8 Hz, 1H), 7.02 (d, J = 2.6 Hz, 1H), 7.12 (dd, J = 2.7, 8.7 Hz, 1H); ^{13}C NMR (50 MHz, $CDCl_3$): δ 8.1 (q), 23.1 (q), 31.0 (t), 55.7 (q), 68.3 (s), 104.4 (d), 114.1 (d), 120.9 (s), 127.8 (d), 153.2 (s), 156.2 (s), 205.6 (s) ppm; ESI-MS: 203.97 (100%, $[M–H]^+$), 205.97 (25%, $[M+H]^+$); HRMS (ESI+): calcd. for $C_{12}H_{15}NO_2H^+$ 206.1176, found 206.1174.

2-Ethyl-5,6,7-trimethoxy-2-methylindolin-3-one (2h):



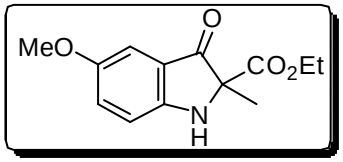
Yellow liquid; 72% yield; R_f = 0.3 (20% ethyl acetate/pet. ether); IR ($CHCl_3$)ν: 3344, 2967, 1676, 1618, 1501, 1469, 1370, 1301, 1134, 1091, 1002, 959, 792 cm^{-1}; ^1H NMR (200 MHz, $CDCl_3$): δ 0.78 (t, J = 7.4 Hz, 3H), 1.27 (s, 3H), 1.66 (dq, J = 7.3, 14.1 Hz, 1H), 1.73 (dq, J = 7.5, 14.9 Hz, 1H), 3.79 (s, 3H), 3.92 (s, 3H), 3.96 (s, 3H) 4.38 (s, 1H), 6.82 (s, 1H); ^{13}C NMR (50 MHz, $CDCl_3$): δ 8.1 (q), 23.2 (q), 31.0 (t), 56.2 (q), 60.5 (q), 61.1 (q), 67.9 (s), 100.0 (d), 115.1 (s), 138.8 (s), 147.8 (s), 149.8 (s), 151.0 (s), 204.3 (s) ppm; ESI-MS: 266.01 (100%, $[M+H]^+$), 288.03 (35%, $[M+Na]^+$); HRMS (ESI+): calcd. for $C_{14}H_{19}NO_4H^+$ 266.1387, found 266.1385.

Spiro[fluorene-9,2'-indolin]-3'-one (2i):-



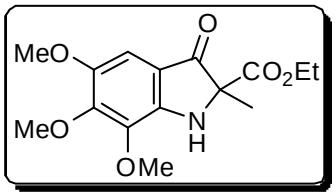
Yellow solid; 59% yield; R_f = 0.3 (20% ethyl acetate/pet. ether); mp: 211–212 °C; IR (Nujol)ν: 3386, 2924, 1699, 1614, 1463, 1377, 1152, 1028, 748, 736, 649 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$): δ 4.91 (s, 1H), 6.90 (t, J = 7.4 Hz, 1H), 7.01 (d, J = 8.3 Hz, 1H), 7.15–7.24 (m, 4H), 7.38 (dt, J = 1.2, 7.5 Hz, 2H), 7.54 (br t, J = 7.6 Hz, 1H), 7.66 (d, J = 7.8 Hz, 1H), 7.71 (d, J = 7.6 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 77.7 (s), 112.6 (d), 119.4 (d), 120.6 (d, 2C), 120.7 (s), 122.9 (d, 2C), 125.9 (d), 128.0 (d, 2C), 129.3 (d, 2C), 137.5 (d), 141.8 (s, 2C), 143.5 (s, 2C), 161.9 (s), 199.2 (s) ppm; ESI-MS (m/z): 283.97 (100%, $[M+H]^+$), 305.94 (90%, $[M+Na]^+$); HRMS (ESI+): calcd. for $C_{20}H_{13}NOH^+$ 284.1070, found 284.1071.

Ethyl 5-methoxy-2-methyl-3-oxoindoline-2-carboxylate (2j):-



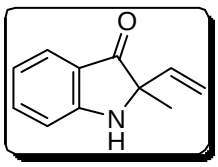
Yellow liquid; 51% yield; R_f = 0.3 (25% ethyl acetate/pet. ether); IR ($CHCl_3$)ν: 3385,2700, 2400, 1703, 1495, 1219, 1108, 933, 771 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$): δ 1.27 (t, J = 7.2 Hz, 3H), 1.63 (s, 3H), 3.76 (s, 3H), 4.14–4.28 (m, 2H), 4.95 (s, 1H), 6.93 (br d, J = 8.9 Hz, 1H), 7.03 (d, J = 2.7 Hz, 1H), 7.17 (dd, J = 2.8, 8.9 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 14.1 (q), 22.1 (q), 55.8 (q), 62.6 (t), 71.5 (s), 104.9 (d), 115.1 (d), 120.2 (s), 128.1 (d), 154.4 (s), 156.9 (s), 169.4 (s), 196.8 (s) ppm; ESI-MS (*m/z*): 271.97 (100%, $[M+Na]^+$); HRMS (ESI+): calcd. for $C_{13}H_{15}NO_4H^+$ 250.1074, found 250.1073.

Ethyl 5,6,7-trimethoxy-2-methyl-3-oxoindoline-2-carboxylate (2k):-



Yellow liquid; 61% yield; R_f = 0.2 (25% ethyl acetate/pet. ether); IR ($CHCl_3$)ν: 3356, 2932, 1741, 1697, 1499, 1469, 1369, 1297, 1091, 931, 756 cm^{-1}; ^1H NMR (200 MHz, $CDCl_3$): δ 1.26 (br t, J = 7.1 Hz, 3H), 1.63 (s, 3H), 3.81 (s, 3H), 3.95 (s, 3H), 3.99 (s, 3H), 5.02 (s, 1H), 6.83 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 14.1 (q), 21.9 (q), 56.3 (q), 60.7 (q), 61.2 (q), 62.5 (t), 71.1 (s), 100.4 (d), 114.2 (s), 139.4 (s), 148.9 (s), 150.3 (s), 151.9 (s), 169.4 (s), 195.6 (s) ppm; ESI-MS: 332.03 (100%, $[M+Na]^+$); HRMS (ESI+): calcd. for $C_{15}H_{19}NO_6H^+$ 310.1285, found 310.1285.

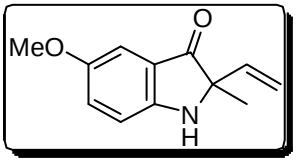
2-Methyl-2-vinylindolin-3-one (2l):



Yellow liquid; 67% yield; R_f = 0.3 (10% ethyl acetate/pet. ether); IR ($CHCl_3$)*v*: 3346, 2973, 2926, 1682, 1620, 1470, 1324, 1133,

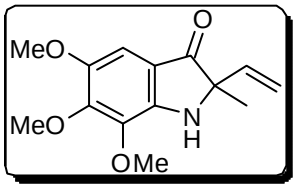
1099, 969, 752, 702 cm^{-1}; ^1H NMR (200 MHz, $CDCl_3$): δ 1.44 (s, 3H), 4.72 (s, 1H), 5.14 (dd, J = 0.8, 10.4 Hz, 1H), 5.34 (dd, J = 0.8, 17.2 Hz, 1H), 5.88 (dd, J = 10.4, 17.2 Hz, 1H), 6.77–6.89 (m, 2H), 7.45 (ddd, J = 1.3, 7.1, 8.4 Hz, 1H) 7.58 (br d, J = 7.8 Hz, 1H); ^{13}C NMR (50 MHz, $CDCl_3$): δ 22.8 (q), 68.3 (s), 112.4 (d), 114.5 (t), 119.0 (d), 119.3 (s), 125.3 (d), 137.3 (d), 137.5 (d), 159.8 (s), 202.0 (s) ppm; ESI-MS: 174.01 (40%, $[M+H]^+$); HRMS (ESI+): calcd. for $C_{11}H_{11}NOH^+$ 174.0913, found 174.0913.

5-Methoxy-2-methyl-2-vinylindolin-3-one (2m):



Yellow liquid; 71% yield; R_f = 0.4 (15% ethyl acetate/pet. ether); IR ($CHCl_3$)ν: 3346, 2927, 1681, 1495, 1440, 1270, 1227, 1125, 1028, 924, 821, 783 cm^{-1}; ^1H NMR (200 MHz, $CDCl_3$): δ 1.44 (s, 3H), 3.75 (s, 3H), 4.41 (s, 1H), 5.14 (dd, J = 0.8, 10.3 Hz, 1H), 5.34 (dd, J = 0.8, 17.2 Hz, 1H), 5.87 (dd, J = 10.4, 17.24 Hz, 1H), 6.85 (dd, J = 0.4, 8.8 Hz, 1H), 7.02 (d, J = 2.6 Hz, 1H), 7.14 (dd, J = 2.7, 8.8 Hz, 1H); ^{13}C NMR (50 MHz, $CDCl_3$): δ 22.8 (q), 55.8 (q), 69.3 (s), 105.0 (d), 114.2 (d), 114.5 (t), 119.7 (s), 127.9 (d), 137.7 (d), 153.6 (s), 155.6 (s), 202.3 (s) ppm; ESI-MS: 204.02 (100%, $[M+H]^+$); HRMS (ESI+): calcd. for $C_{12}H_{13}NO_2H^+$ 204.1019, found 204.1019.

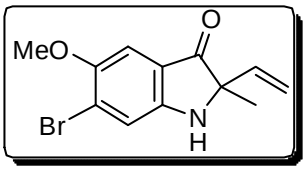
5,6,7-Trimethoxy-2-methyl-2-vinylindolin-3-one (2n):-



Yellow liquid; 74% yield; R_f = 0.3 (25% ethyl acetate/pet. ether); IR ($CHCl_3$)ν: 3344, 2932, 1684, 1618, 1500, 1469, 1304, 1123, 1090, 926, 790 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$): δ 1.44 (s, 3H), 3.80 (s, 3H), 3.95 (s, 3H), 3.98 (s, 3H), 4.53 (s, 1H), 5.14 (br d, J = 10.5 Hz, 1H), 5.34 (br d, J = 17.2 Hz, 1H), 5.87 (dd, J = 10.5, 17.2 Hz, 1H), 6.83 (s, 1H); ^{13}C NMR (100 MHz,

$CDCl_3$): δ 22.8 (q), 56.3 (q), 60.6 (q), 61.2 (q), 68.9 (s), 100.5 (d), 113.8 (s), 114.3 (t), 137.8 (d), 138.9 (s), 148.1 (s), 150.0 (s), 150.6 (s), 201.1 (s) ppm; ESI-MS (*m/z*): 263.88 (100%, $[M+H]^+$), 285.99 (30%, $[M+Na]^+$); HRMS (ESI+): calcd. for $C_{14}H_{17}NO_4H^+$ 264.1230, found 264.1230**.**

6-bromo-5-methoxy-2-methyl-2-vinylindolin-3-one (**2o**):-



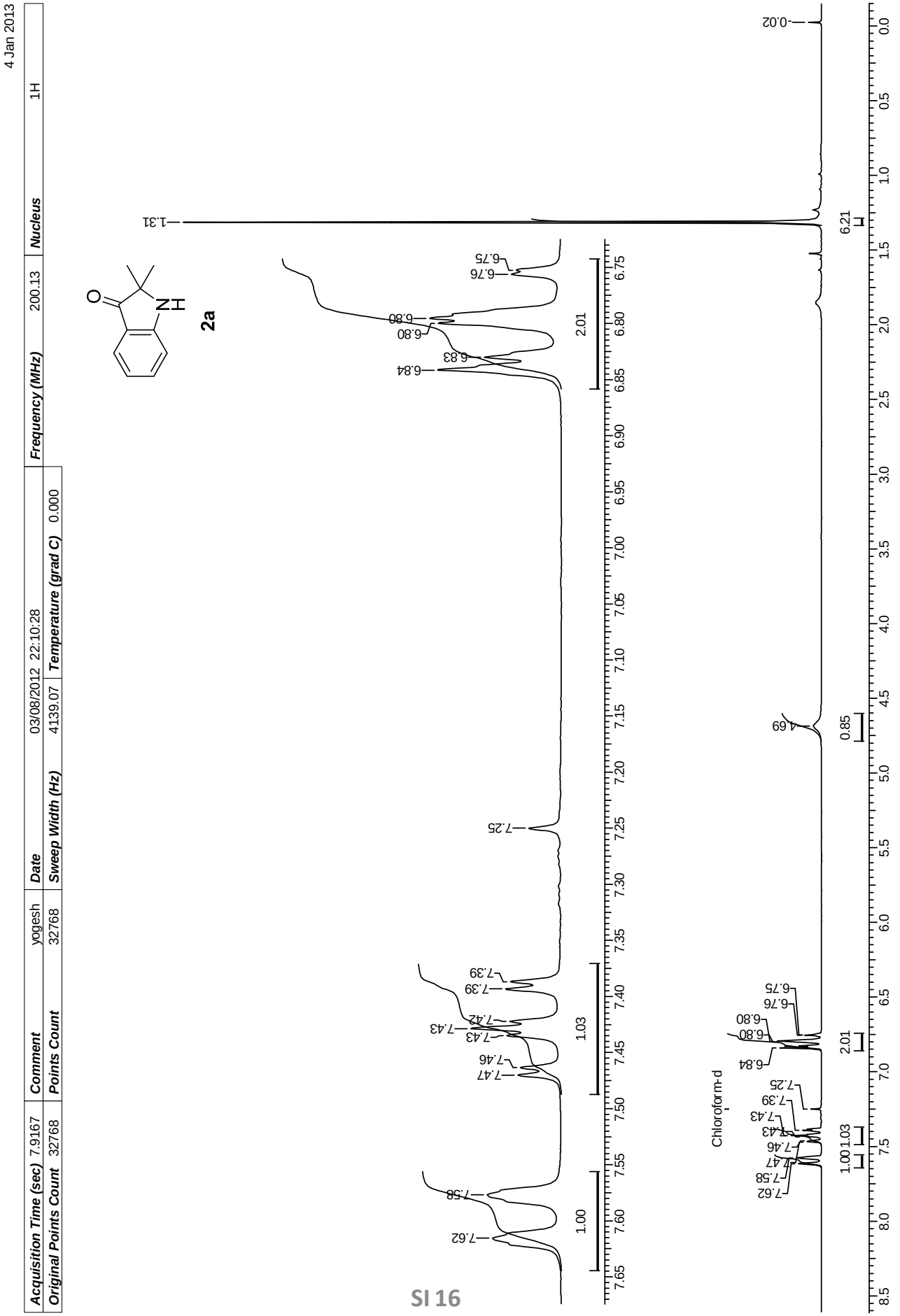
Yellow solid; 72% yield; R_f = 0.3 (15% ethyl acetate/pet. ether); mp: 154–155 °C; IR ($CHCl_3$)*v*: 3284, 2923, 1740, 1671, 1577, 1478, 1275, 1154, 1040, 848, 718 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$): δ 1.44 (s, 3H), 3.84 (s, 3H), 4.43 (s, 1H), 5.15 (br d, *J* = 10.4 Hz, 1H), 5.34 (br d, *J* = 17.1 Hz, 1H), 5.85 (dd, *J* = 10.4, 17.1 Hz, 1H), 7.05 (s, 1H), 7.20 (s, 1H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 22.8 (q), 56.7 (q), 69.2 (s), 105.7 (d), 114.7 (t), 117.8 (d), 118.9 (s), 124.2 (s), 137.3 (d), 150.0 (s), 154.9 (s), 201.4 (s) ppm; HRMS (ESI+): calcd. for $C_{12}H_{12}BrNO_2H^+$ 282.0124, found 282.0129**.**

References:-

1. L. Lunazzi, A. Mazzanti and M. Minzoni, *J. Org. Chem.*, 2006, **71**, 9297; T. J. Korn, M. A. Schade, M. N. Cheemala, S. Wirth, S. A. Guevara, G. Cahiez and P. Knochel, *Synthesis*, 2006, 3547.

2. S. P. Chavan, S. Garai and U. R. Kalkote, *Tetrahedron*, 2012, **68**, 8509

3. Azadi-Ardakani, M. A. Alkhader, J. H. Lippiatt, D. I. Patel, R. K. Smalley and S. Higson, *J. Chem. Soc., Perkin Trans. 1*, 1986, 1107; R. J. S. Beer, T. Donavanik and A. Robertson, *J. Chem. Soc.*, 1954, 4139.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7.47
7.46
7.43
7.42
7.39
7.25
6.84
6.83
6.80
6.76
6.75
4.69
-0.02
Chloroform-d
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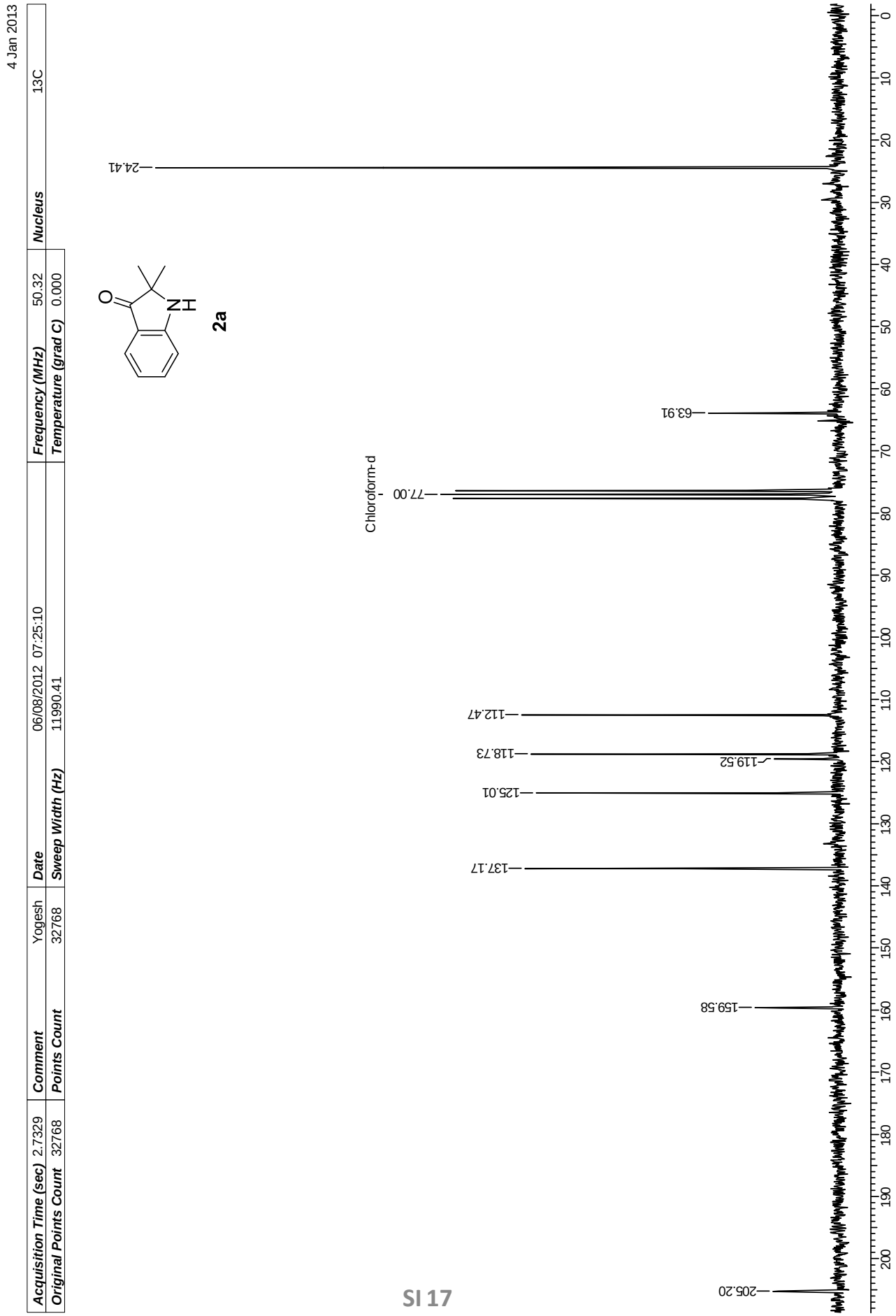
1.03
1.03
2.01
2.01
0.85
6.21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Temperature (grad C) 0.000
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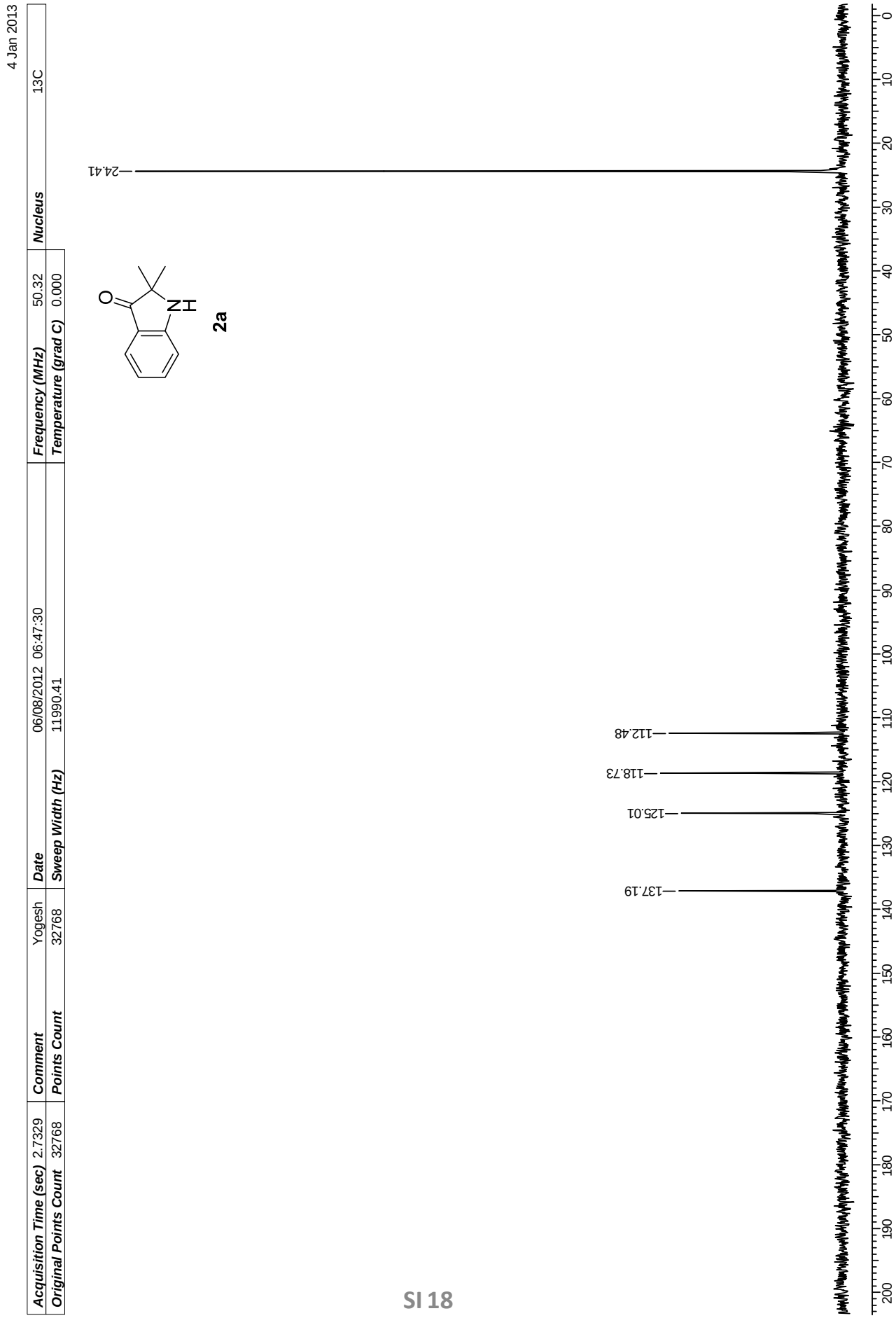
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NH
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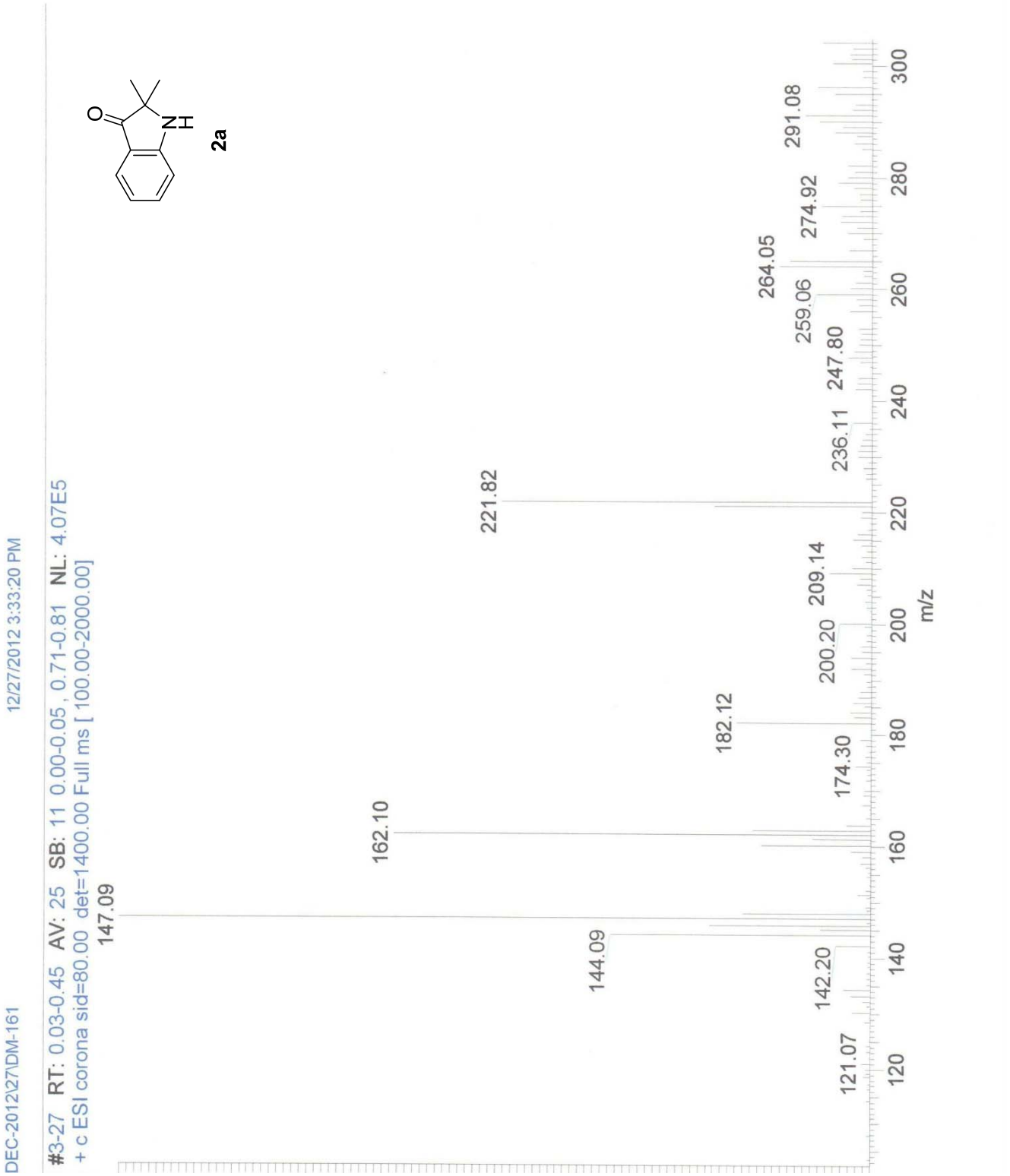
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180
200
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240
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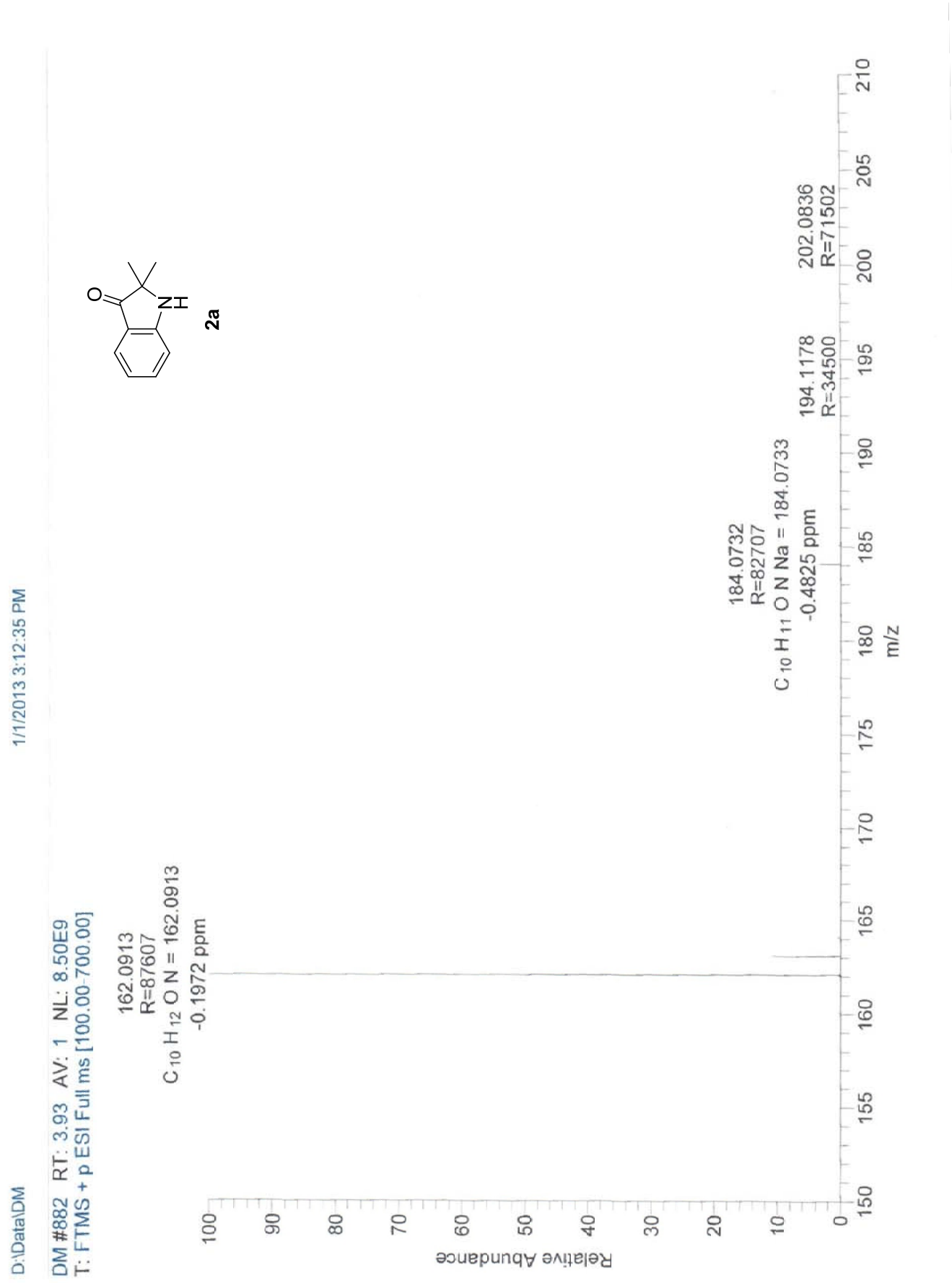
280
300
m/z
O
N
H
2a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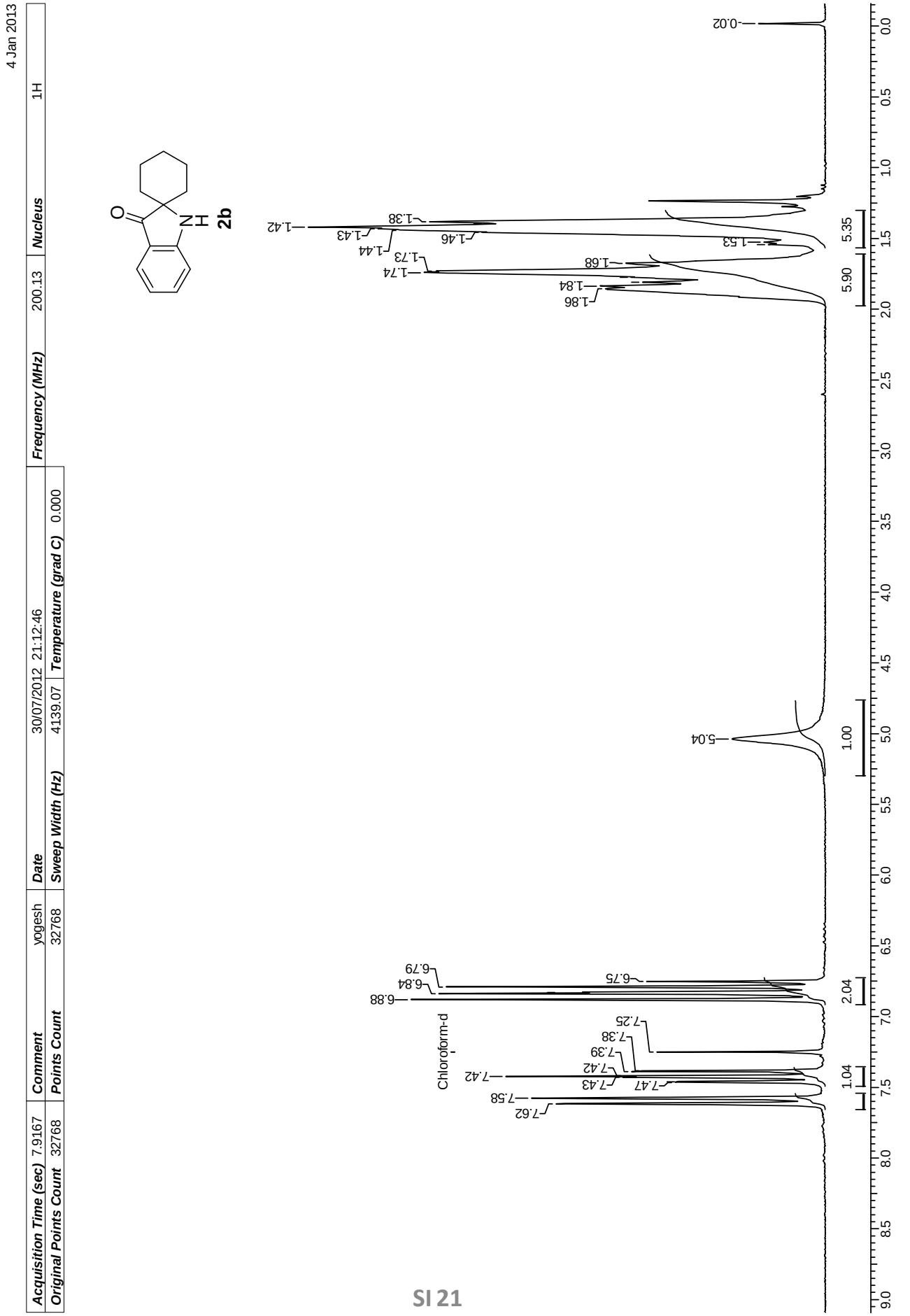
Relative Abundance
m/z
O
N
H
2a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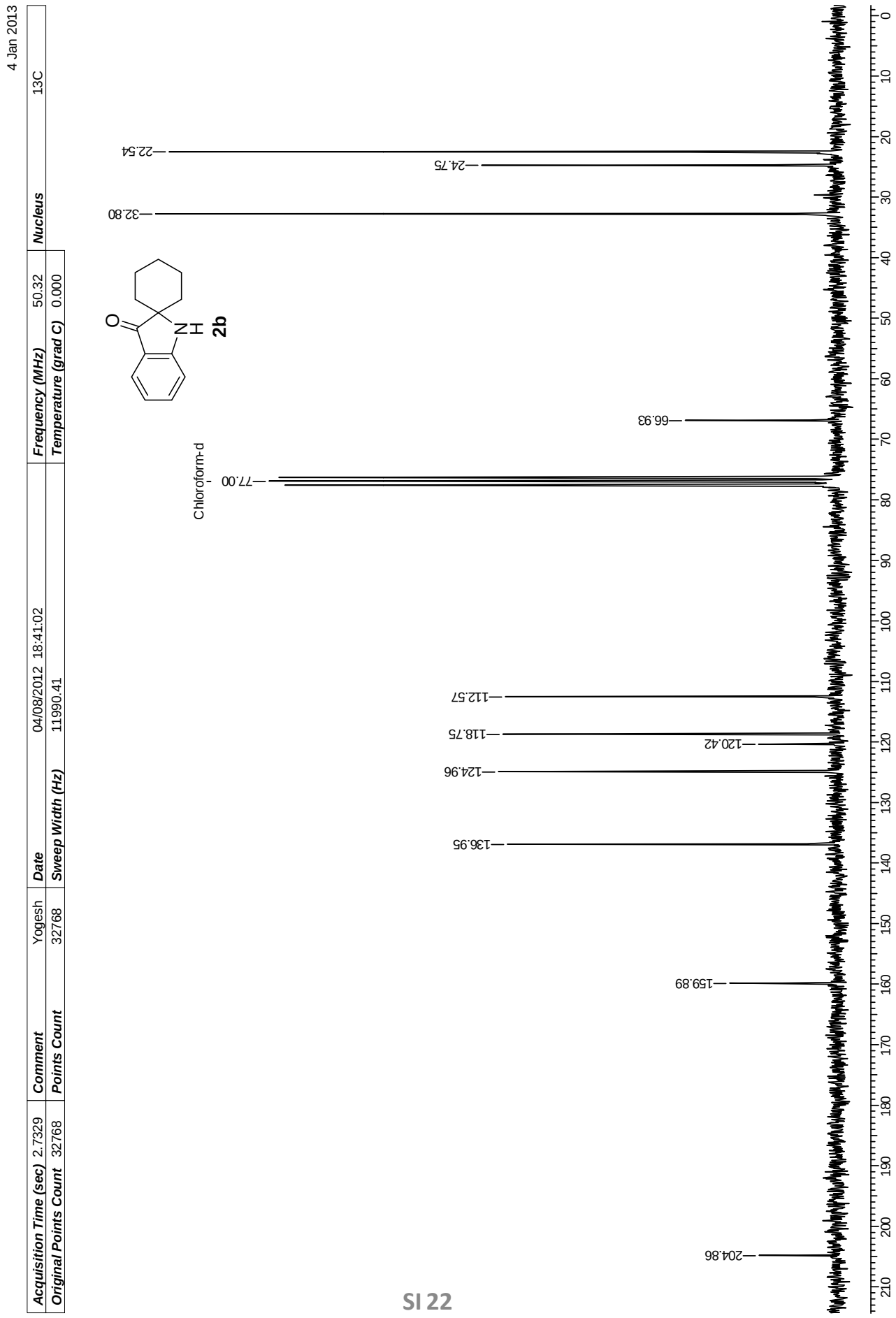
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Frequency (MHz)
Temperature (grad C)
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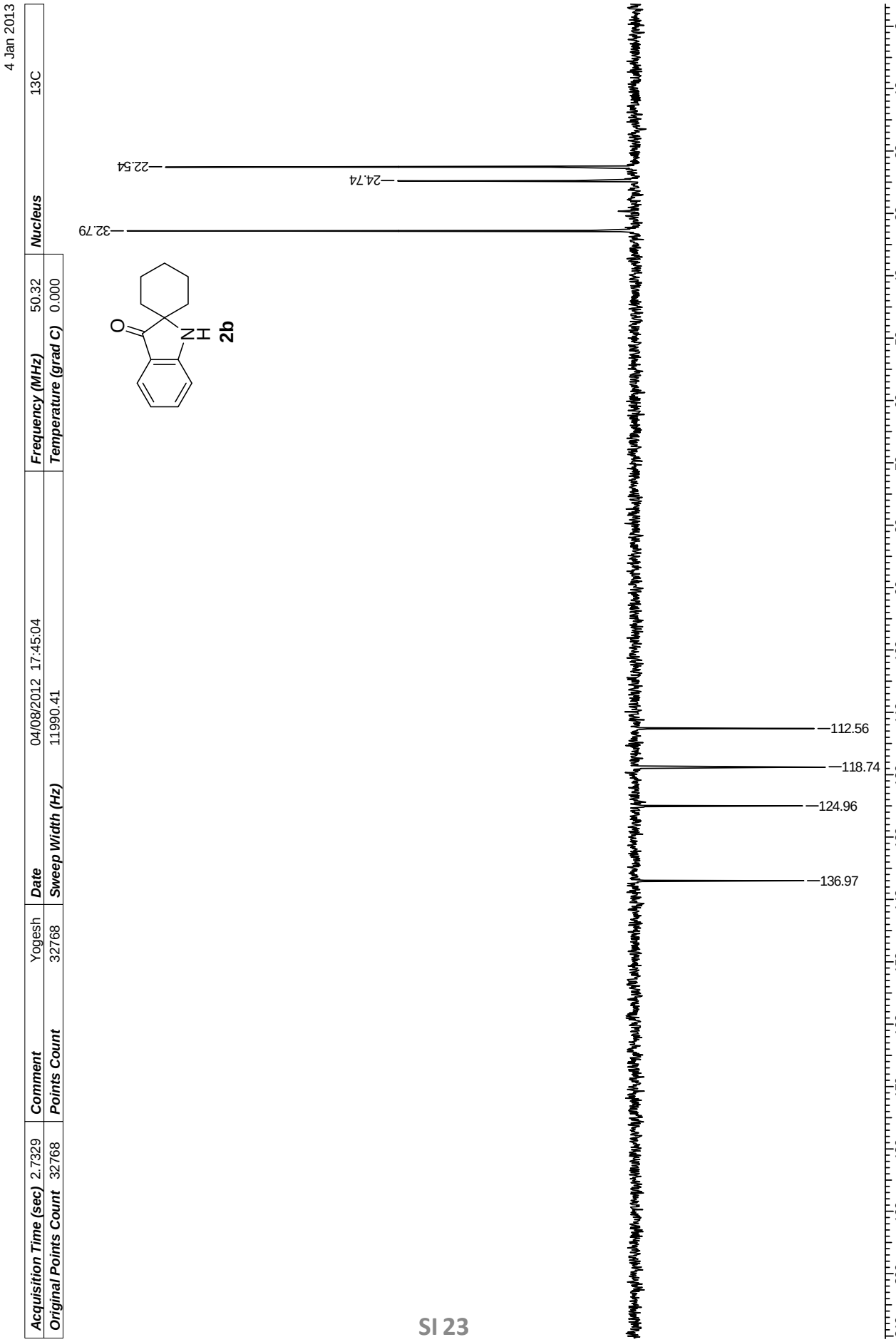
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Comment
Points Count
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Date
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Frequency (MHz)
Temperature (grad C)
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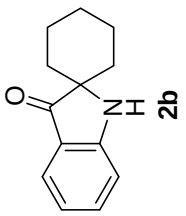
2b



2b

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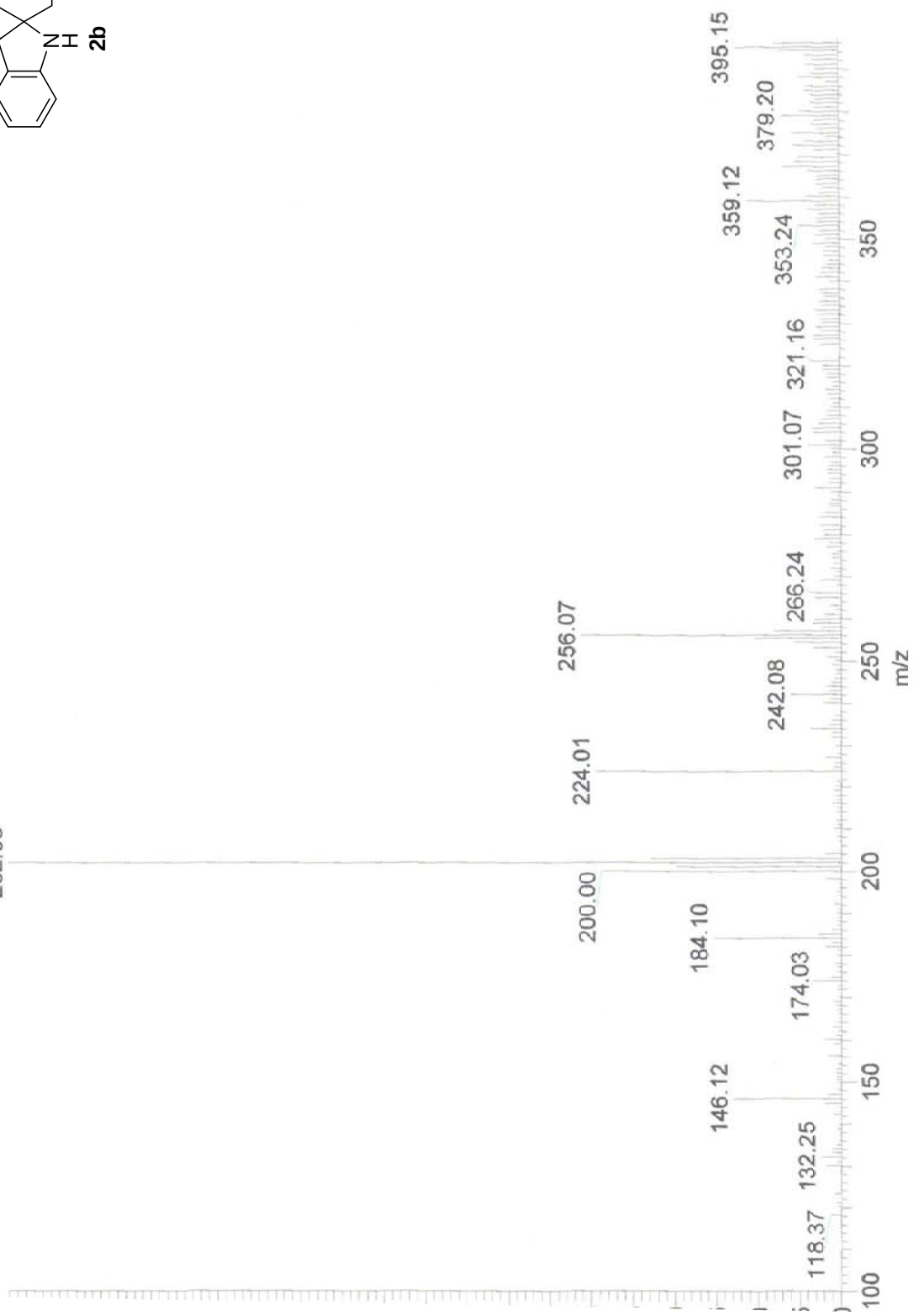
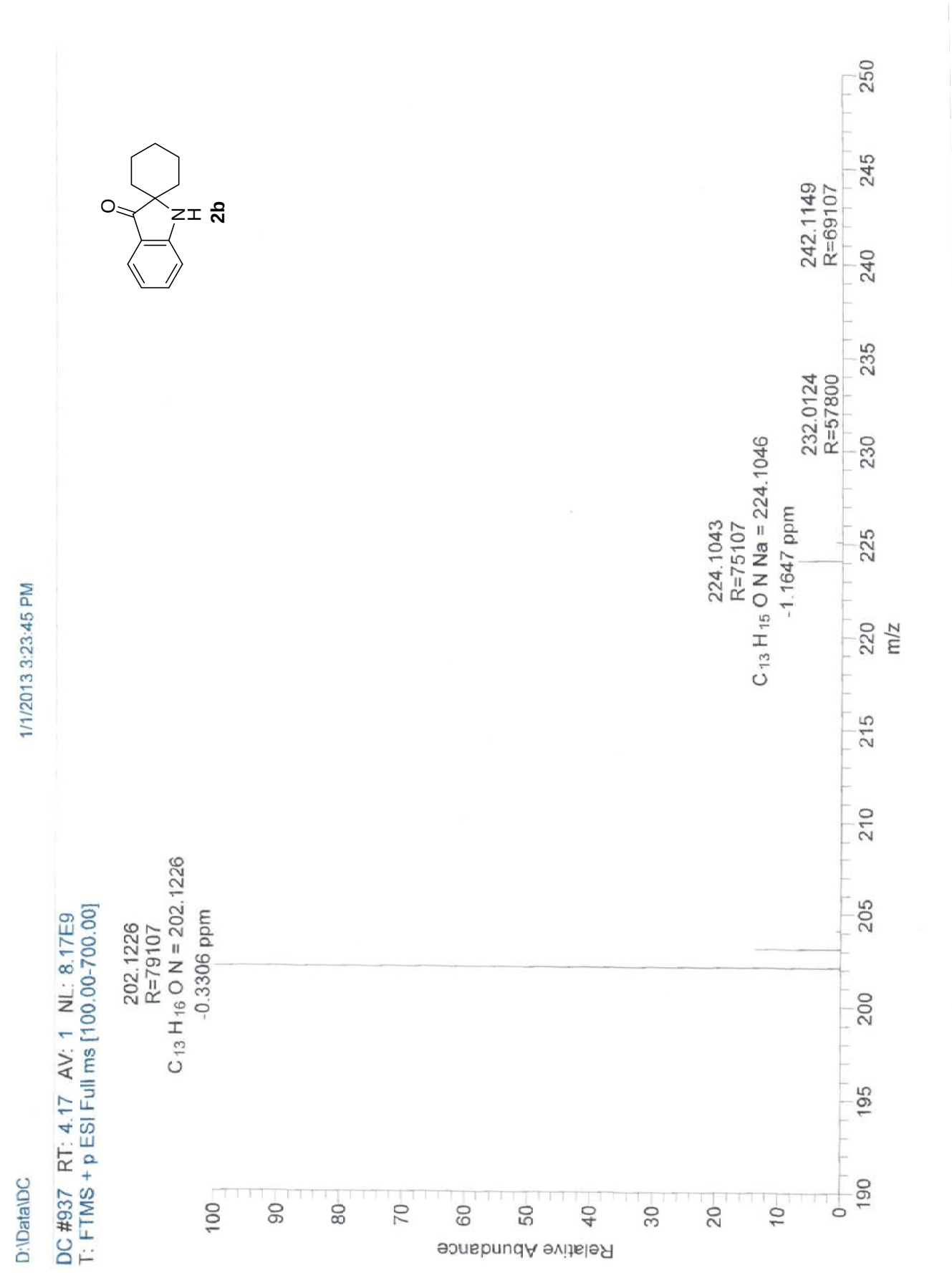
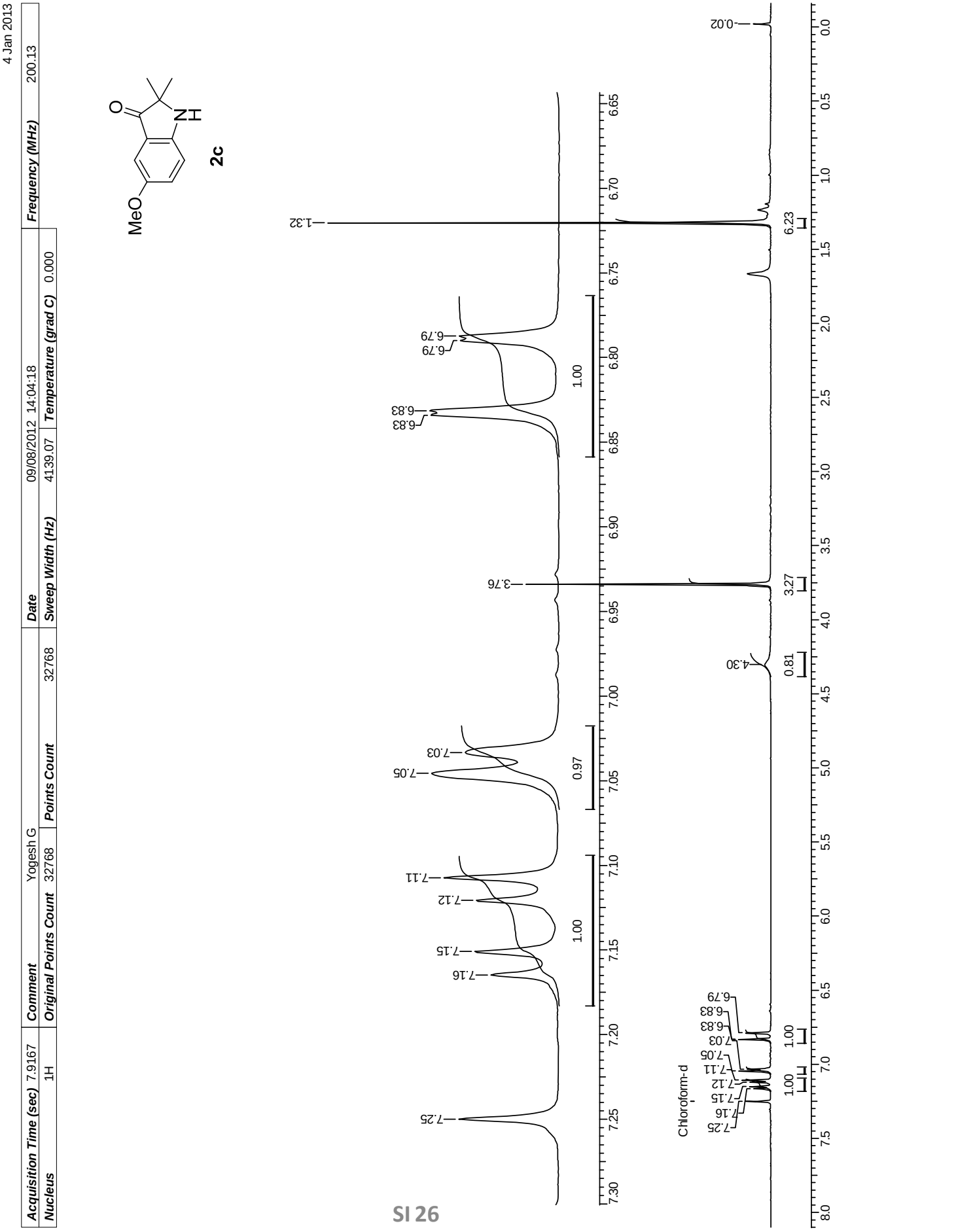
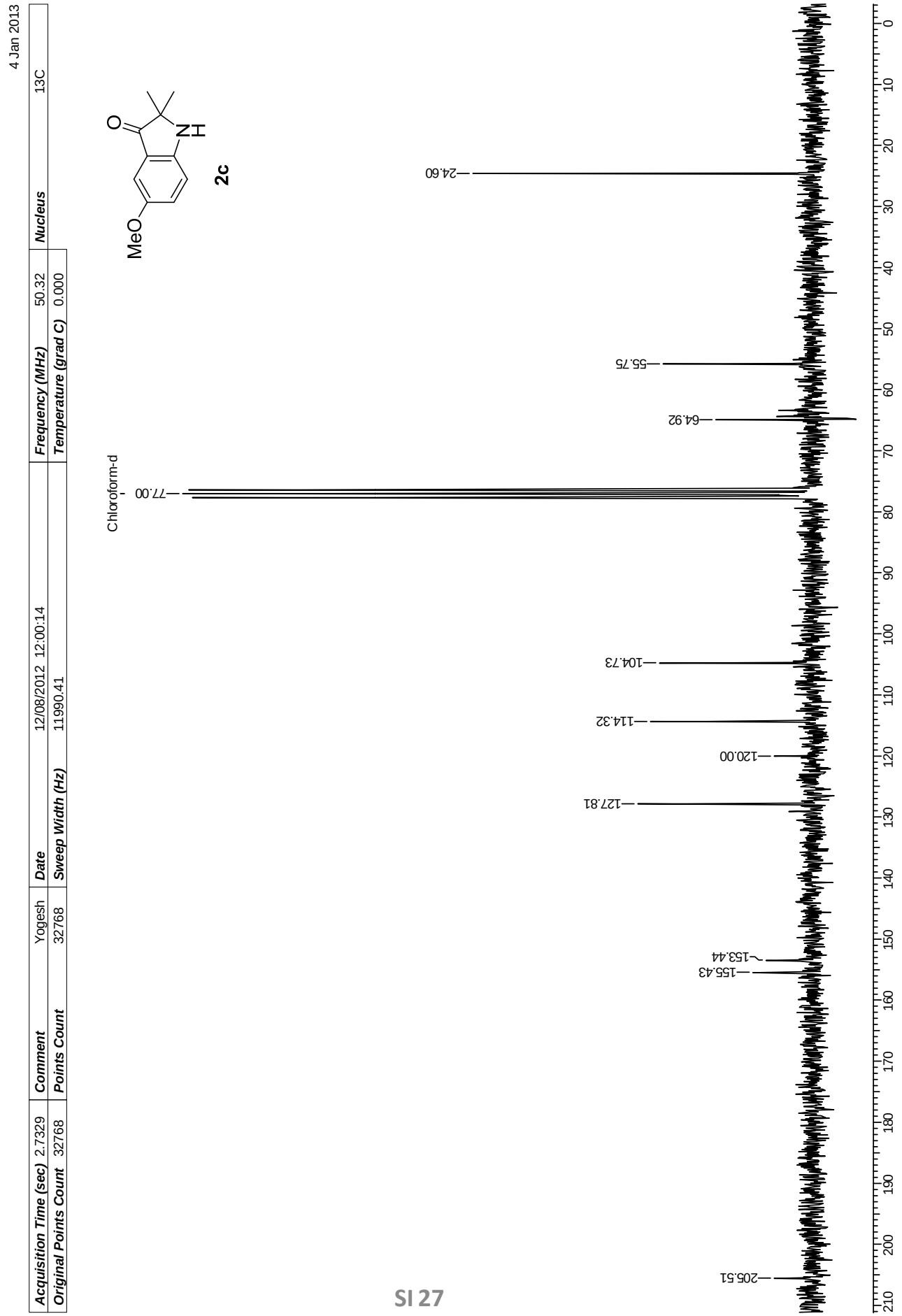
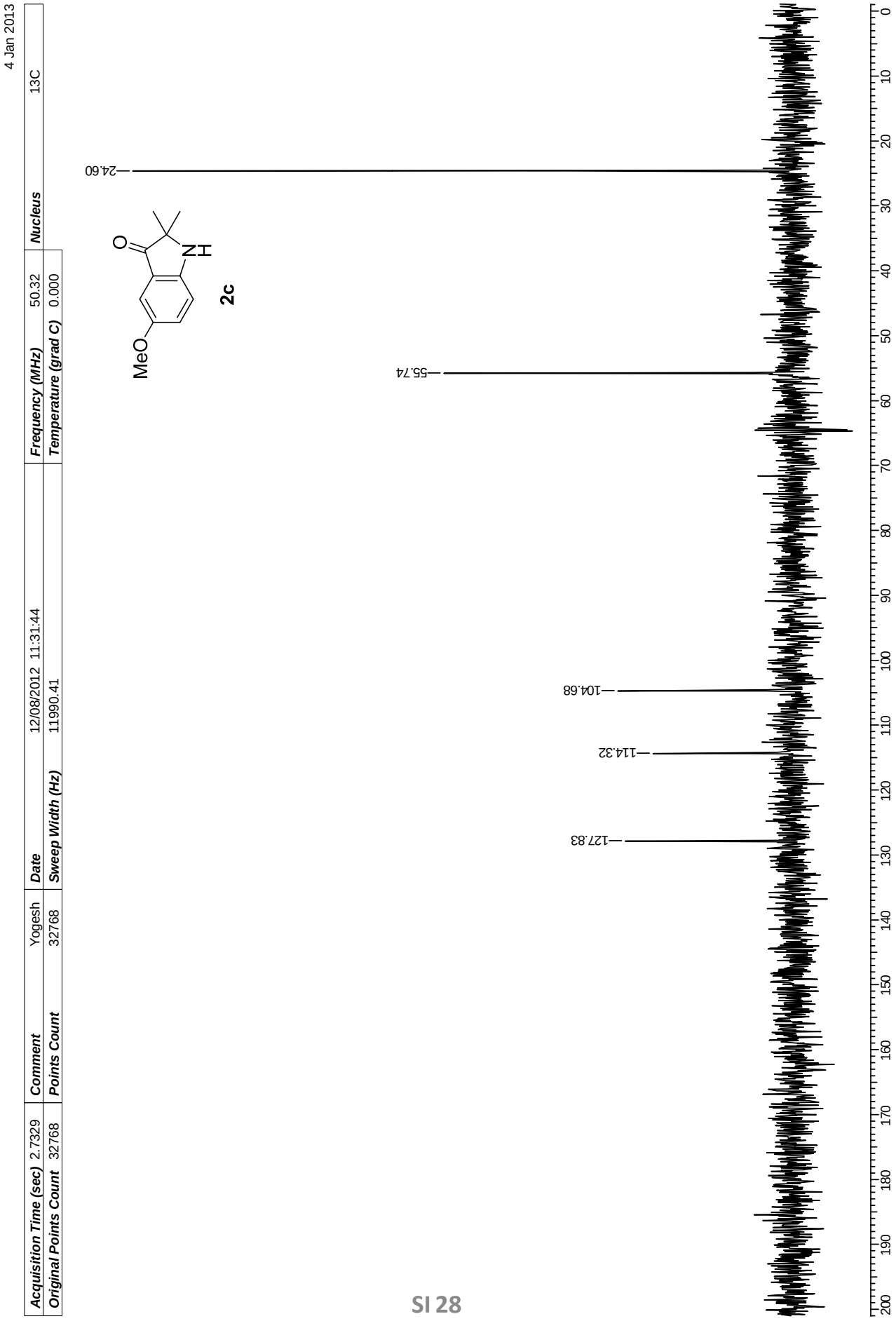
+ c ESI corona sid=80.00 det=1400.00 Full ms [100.00-2000.00]

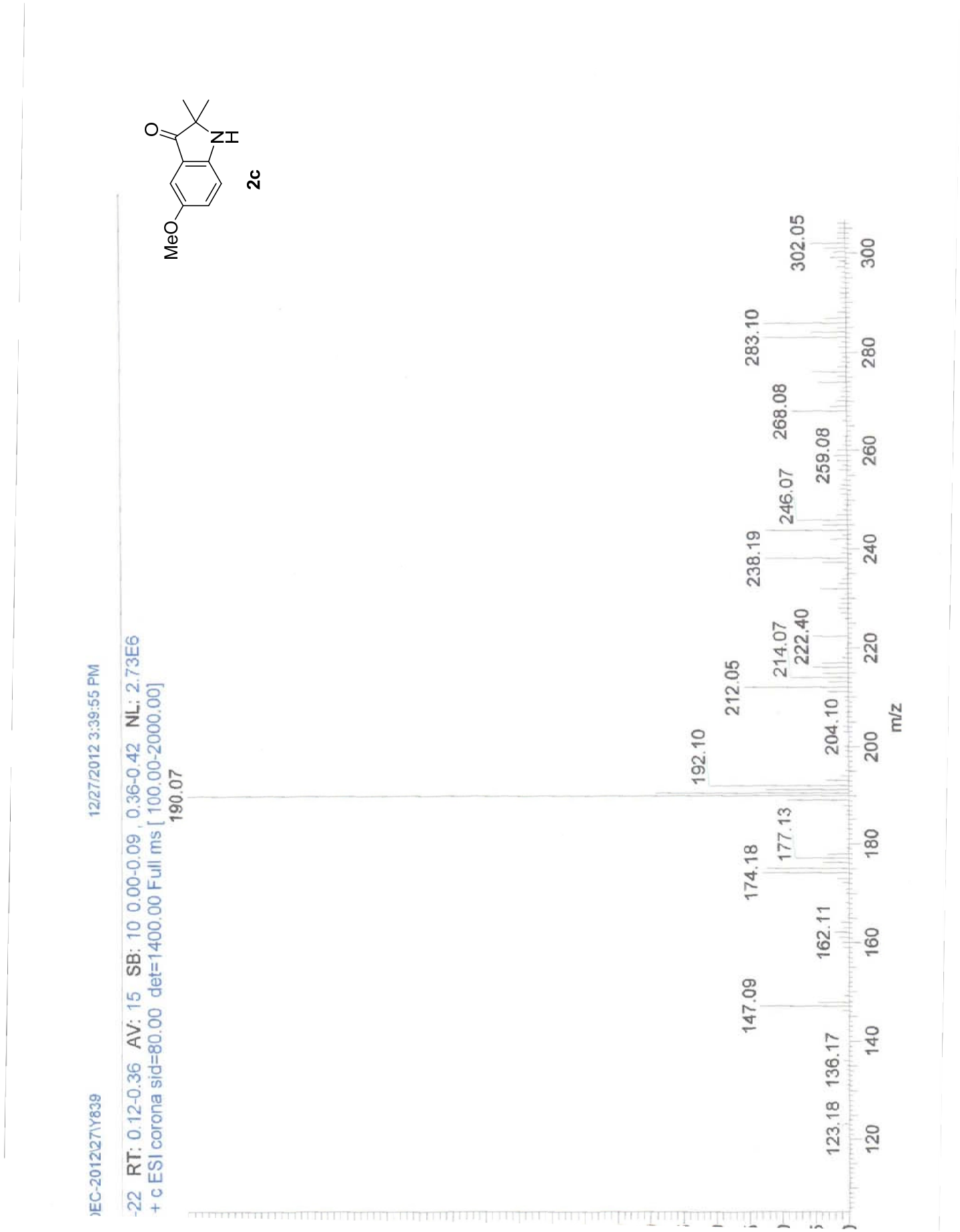


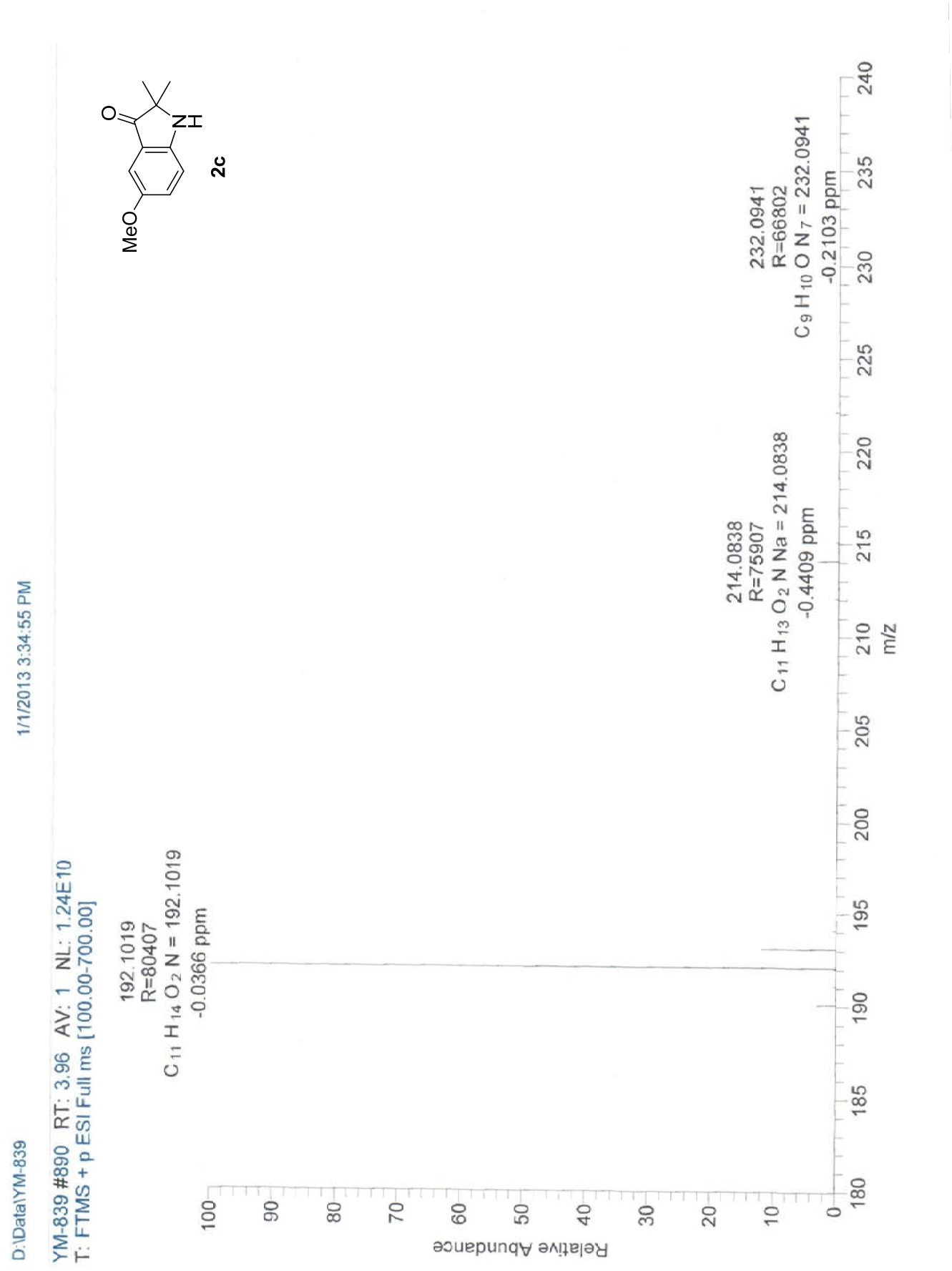

D:\Data\DC
1/1/2013 3:23:45 PM
DC #937 RT: 4.17 AV: 1 NL: 8.17E9
T: FTMS + p ESI Full ms [100.00-700.00]
202.1226
R=79107
C 13 H 16 O N = 202.1226
-0.3306 ppm
224.1043
R=75107
C 13 H 15 O N Na = 224.1046
-1.1647 ppm
232.0124
R=57800
242.1149
R=69107
Relative Abundance
m/z
2b

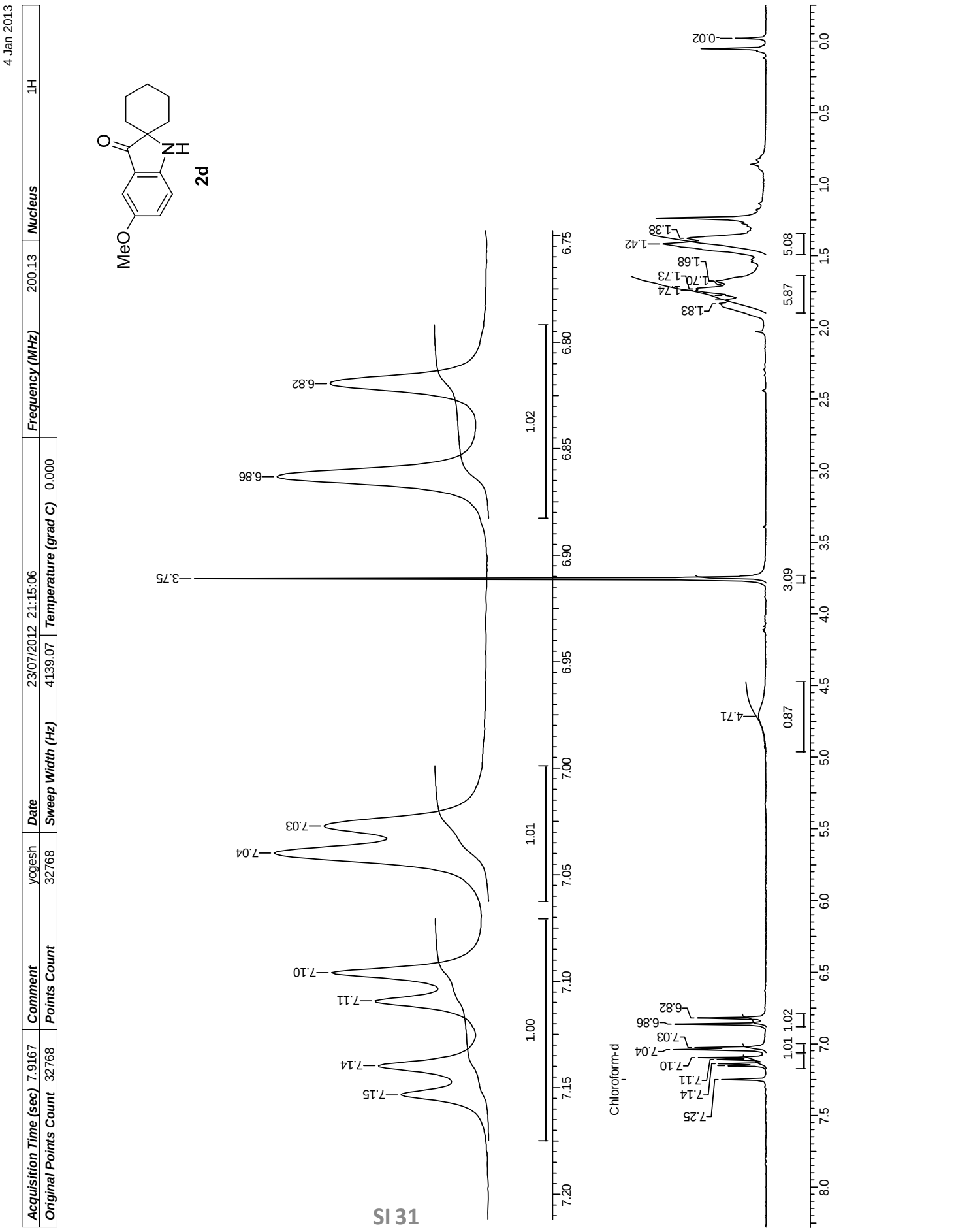

4 Jan 2013
Acquisition Time (sec) 7.9167
Comment
Date
09/08/2012 14:04:18
Frequency (MHz) 200.13
Nucleus 1H
Original Points Count 32768
Yogesh G
Points Count 32768
Sweep Width (Hz) 4139.07
Temperature (grad C) 0.000
MeO
NH
O
2c
1.32
6.79
6.79
6.83
6.83
3.76
7.03
7.05
7.11
7.12
7.15
7.16
7.25
4.30
0.02
Chloroform-d
1.00
0.97
1.00
0.81
3.27
6.23
1.00
1.00

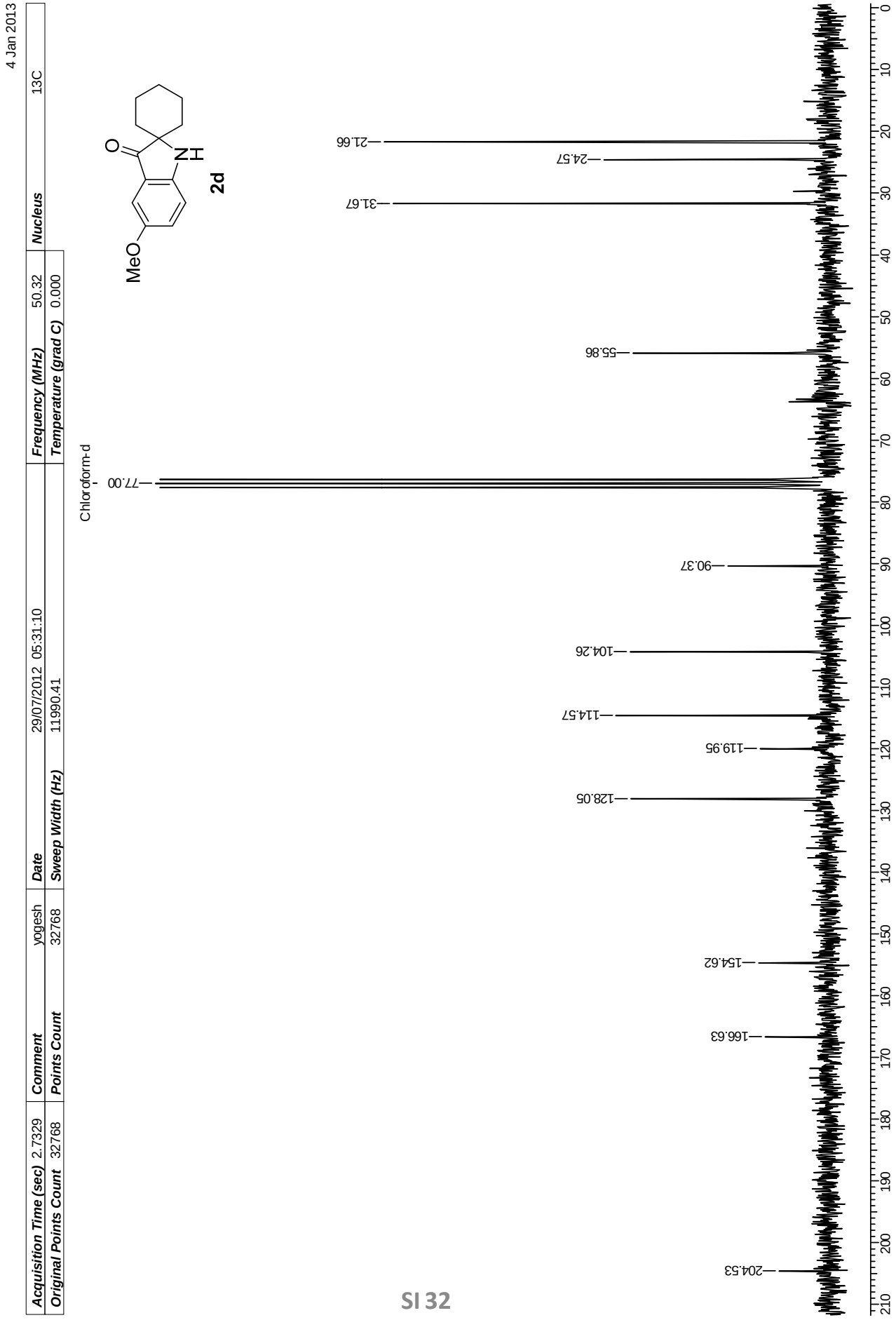

4 Jan 2013
Acquisition Time (sec) 2.7329
Original Points Count 32768
Comment
Points Count
Yogesh
32768
Date
Sweep Width (Hz)
12/08/2012 12:00:14
11990.41
Frequency (MHz)
Temperature (grad C)
50.32
0.000
Nucleus
13C
O
NH
MeO
2c
Chloroform-d
77.00
24.60
55.75
64.92
104.73
114.32
120.00
127.81
153.44
155.43
205.51

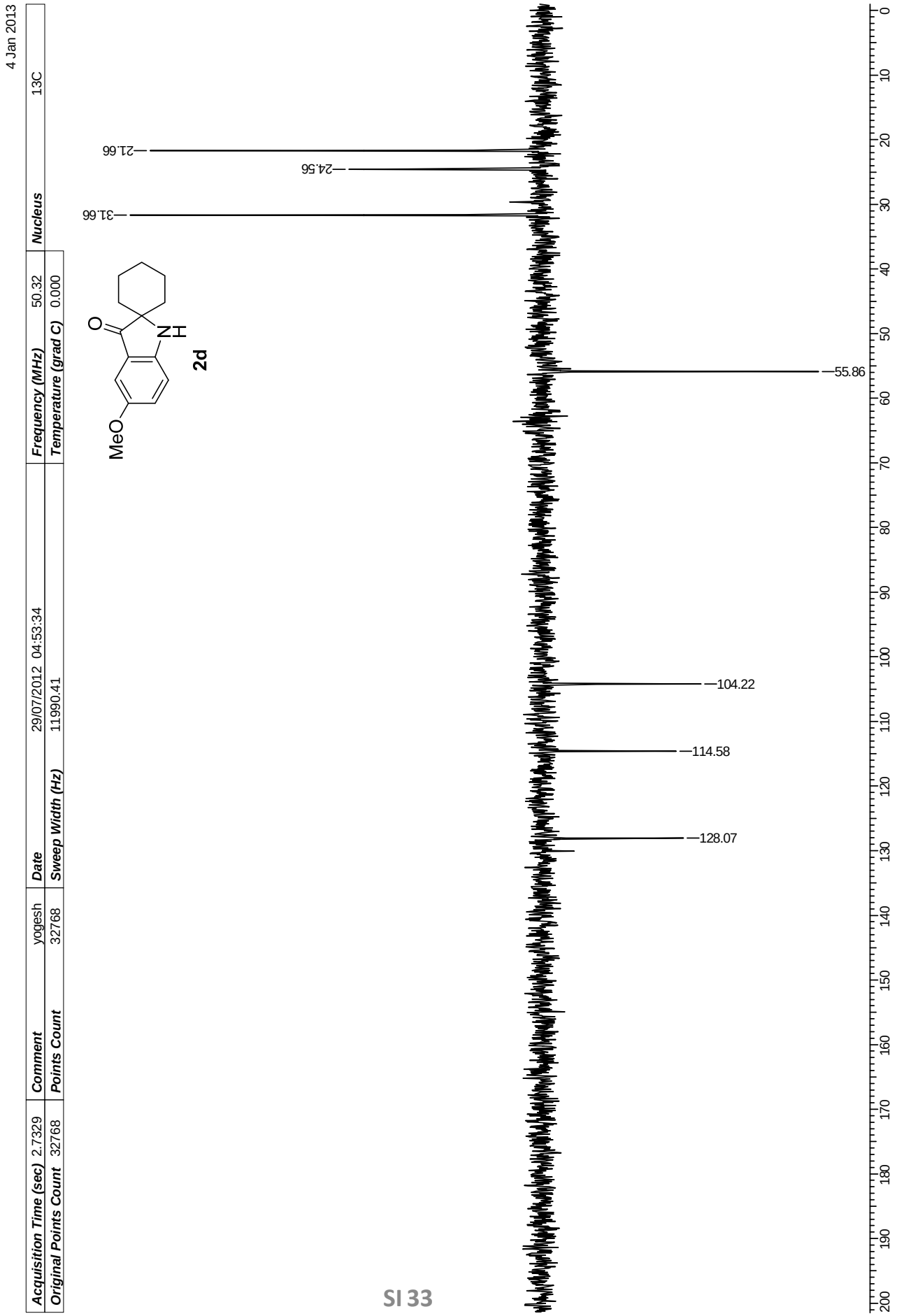

4 Jan 2013
Acquisition Time (sec) 2.7329
Original Points Count 32768
Comment
Points Count
Yogesh
32768
Date
Sweep Width (Hz)
12/08/2012 11:31:44
11990.41
Frequency (MHz)
Temperature (grad C)
50.32
0.000
Nucleus
13C
24.60
55.74
104.68
114.32
127.83
MeO
O
NH
2c
0
10
20
30
40
50
60
70
80
90
100
110
120
130
140
150
160
170
180
190
200

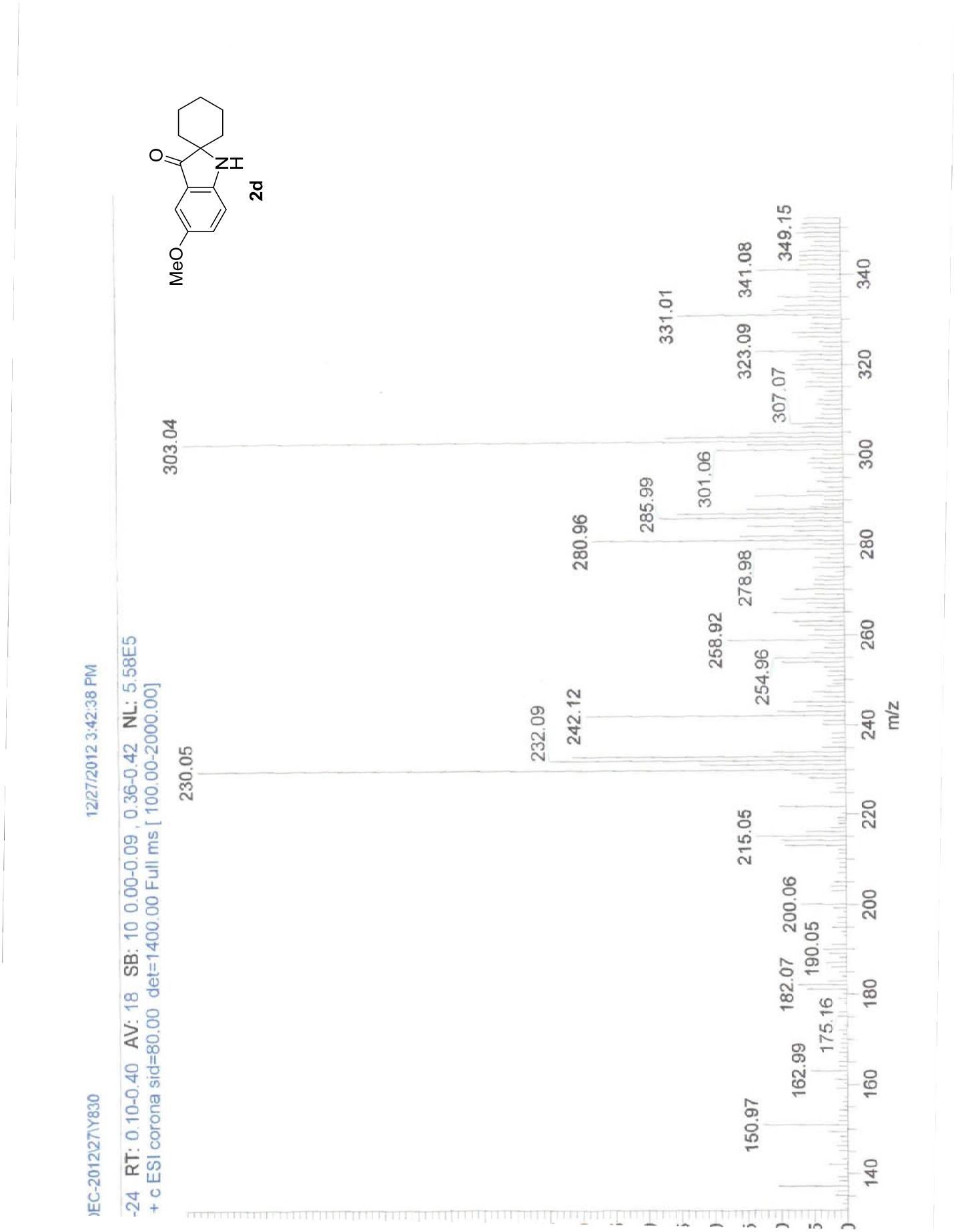

MeO
O
NH
2c
12/27/2012 3:39:55 PM
-22 RT: 0.12-0.36 AV: 15 SB: 10 0.00-0.09 , 0.36-0.42 NL: 2.73E6
+ c ESI corona sid=80.00 det=1400.00 Full ms [100.00-2000.00]
190.07
192.10
212.05
214.07
222.40
238.19
246.07
259.08
268.08
283.10
302.05
147.09
162.11
174.18
177.13
204.10
123.18
136.17
120
140
160
180
200
220
240
260
280
300
m/z

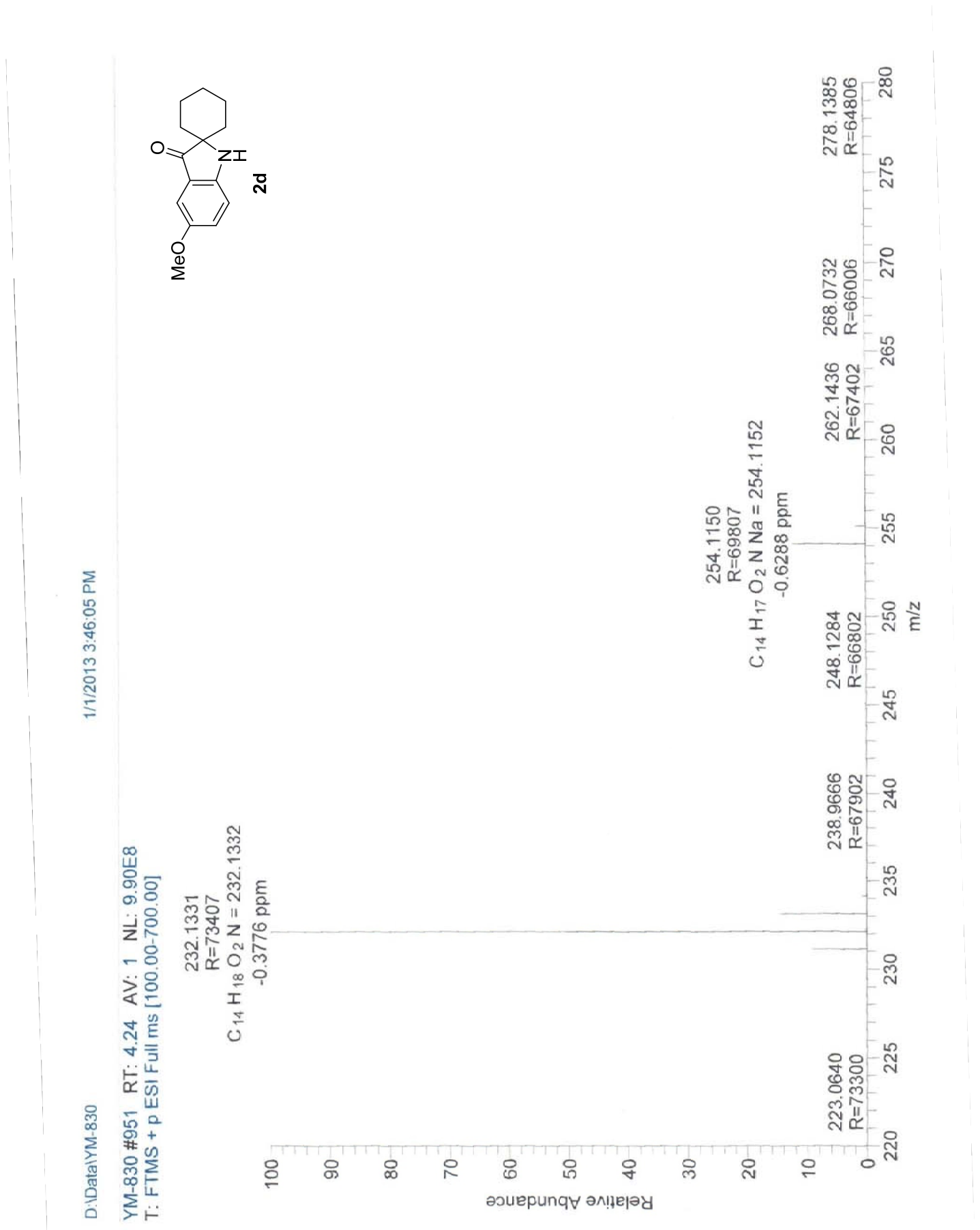

D:\Data\YM-839
1/1/2013 3:34:55 PM
YM-839 #890 RT: 3.96 AV: 1 NL: 1.24E10
T: FTMS + p ESI Full ms [100.00-700.00]
192.1019
R=80407
C 11 H 14 O 2 N = 192.1019
-0.0366 ppm
214.0838
R=75907
C 11 H 13 O 2 N Na = 214.0838
-0.4409 ppm
232.0941
R=66802
C 9 H 10 O N 7 = 232.0941
-0.2103 ppm
Relative Abundance
m/z
MeO
2c

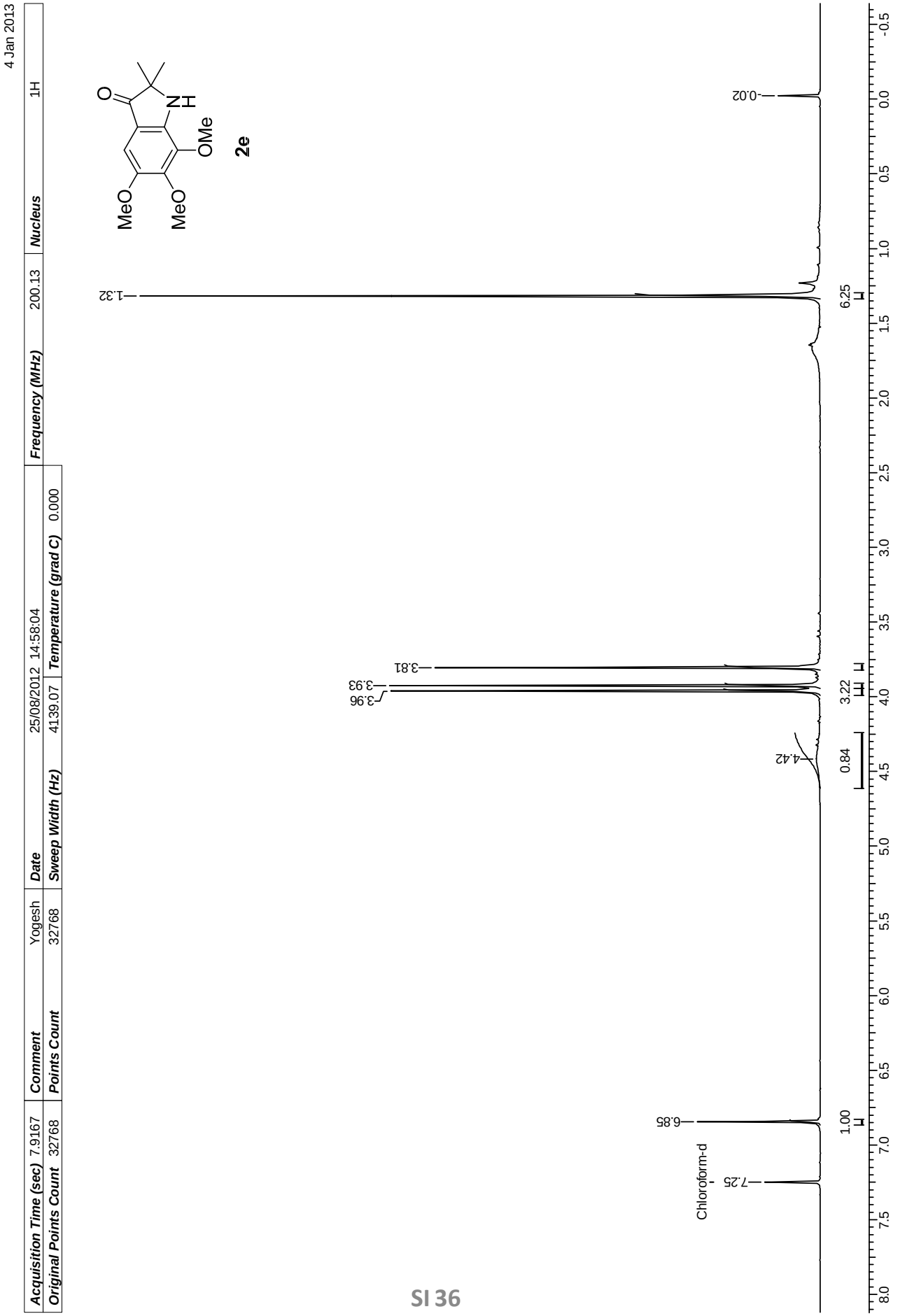

4 Jan 2013
Acquisition Time (sec) 7.9167 | Comment | yogesh | Date | 23/07/2012 21:15:06 | Frequency (MHz) | 200.13 | Nucleus | 1H
Original Points Count 32768 | Points Count | 32768 | Sweep Width (Hz) | 4139.07 | Temperature (grad C) | 0.000
MeO
NH
O
2d
3.75
6.86
6.82
7.04
7.03
7.10
7.11
7.14
7.15
4.71
-0.02
1.83
1.70
1.73
1.74
1.68
1.42
1.38
7.25
Chloroform-d
1.00
1.01
1.02
0.87
3.09
5.87
5.08

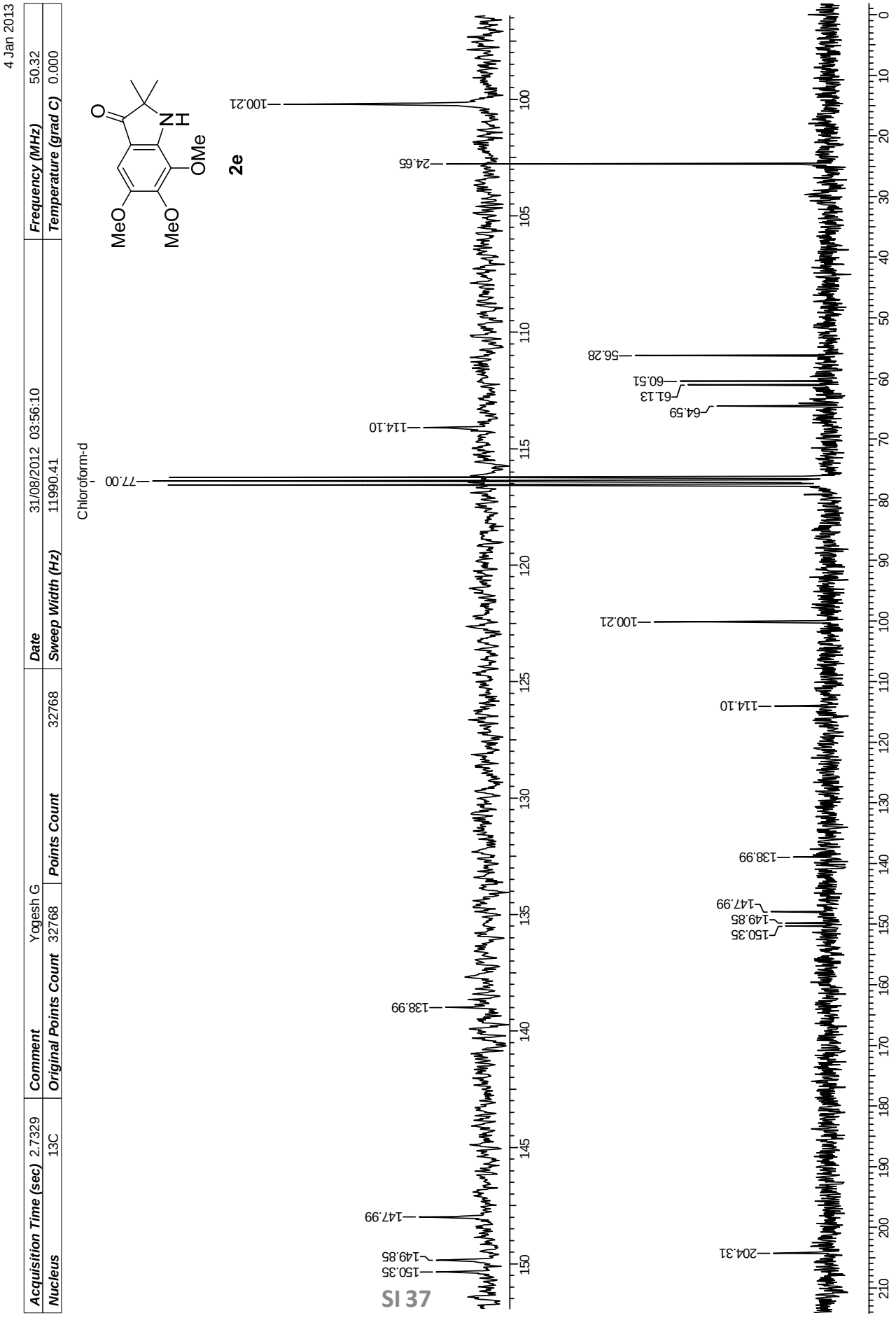

4 Jan 2013
Acquisition Time (sec) 2.7329
Original Points Count 32768
Comment
Points Count
yogesh
32768
Date
Sweep Width (Hz)
29/07/2012 05:31:10
11990.41
Frequency (MHz) 50.32
Temperature (grad C) 0.000
Nucleus 13C
2d
MeO
NH
O
Chloroform-d
77.00
21.66
24.57
31.67
55.86
90.37
104.26
114.57
119.95
128.05
154.62
166.63
204.53

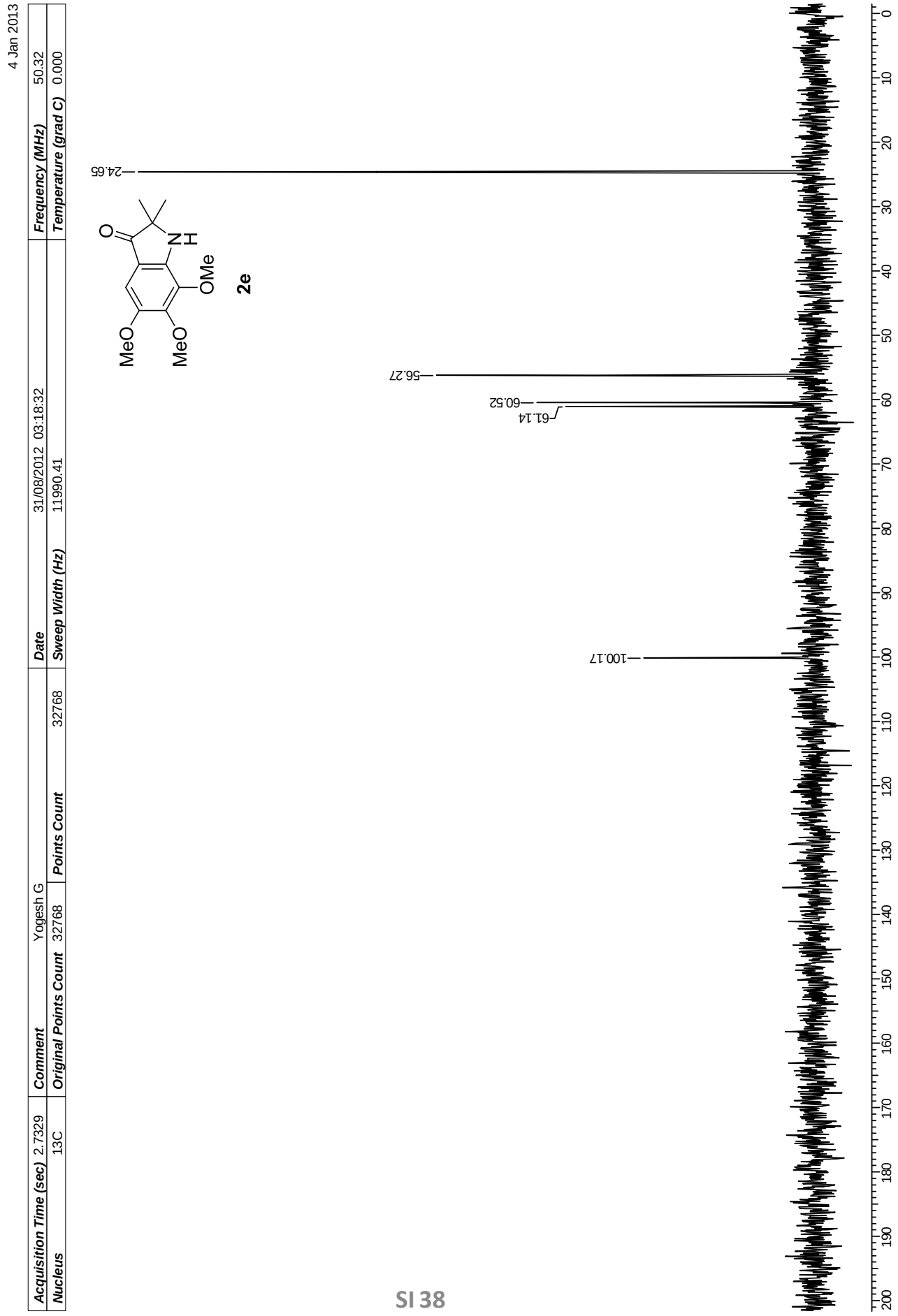

4 Jan 2013
Acquisition Time (sec) 2.7329
Original Points Count 32768
Comment
Points Count
yogesh
32768
Date
Sweep Width (Hz)
29/07/2012 04:53:34
11990.41
Frequency (MHz) 50.32
Temperature (grad C) 0.000
Nucleus 13C
21.66
24.56
31.66
55.86
104.22
114.58
128.07
MeO
NH
O
2d

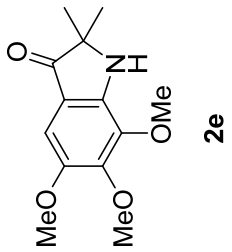

MeO
O
N
H
2d
12/27/2012 3:42:38 PM
-24 RT: 0.10-0.40 AV: 18 SB: 10 0.00-0.09 , 0.36-0.42 NL: 5.58E5
+ c ESI corona sid=80.00 det=1400.00 Full ms [100.00-2000.00]
303.04
230.05
232.09
242.12
280.96
285.99
301.06
331.01
341.08
349.15
323.09
307.07
278.98
258.92
254.96
215.05
150.97
162.99
175.16
182.07
190.05
200.06
m/z


D:\Data\YM-830
1/1/2013 3:46:05 PM
YM-830 #951 RT: 4.24 AV: 1 NL: 9.90E8
T: FTMS + p ESI Full ms [100.00-700.00]
232.1331
R=73407
C14 H18 O2 N = 232.1332
-0.3776 ppm
254.1150
R=69807
C14 H17 O2 N Na = 254.1152
-0.6288 ppm
223.0640
R=73300
238.9666
R=67902
248.1284
R=66802
262.1436
R=67402
268.0732
R=66006
278.1385
R=64806
Relative Abundance
m/z
MeO
O
N
H
2d

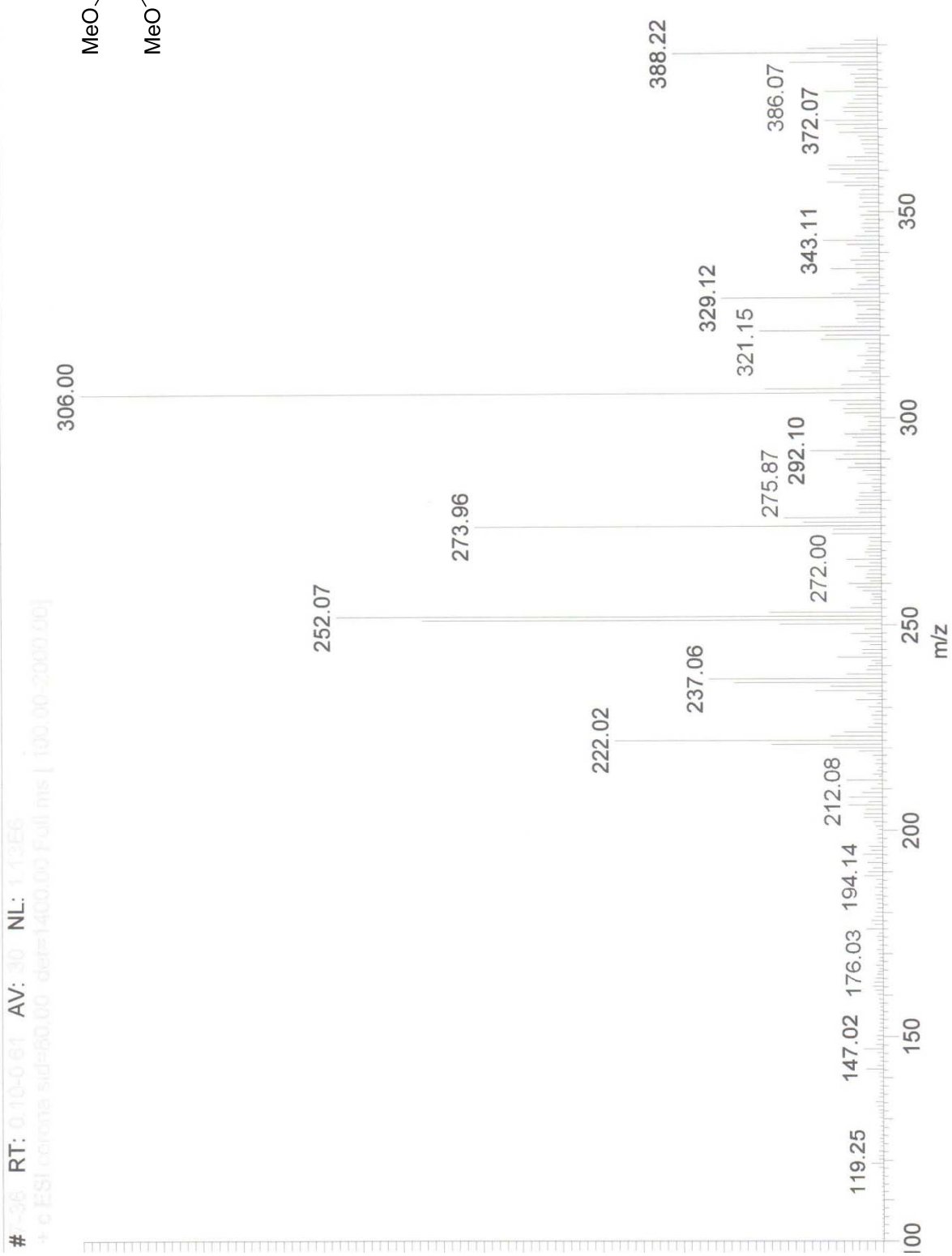

4 Jan 2013
Acquisition Time (sec) 7.9167
Original Points Count 32768
Comment
Points Count
Yogesh
32768
Date
Sweep Width (Hz)
25/08/2012 14:58:04
4139.07
Frequency (MHz)
Temperature (grad C) 0.000
200.13
Nucleus
1H
MeO
MeO
OMe
NH
O
2e
1.32
3.81
3.96
3.93
4.42
6.85
7.25
Chloroform-d
0.02
1.00
0.84
3.22
6.25


4 Jan 2013
Acquisition Time (sec) 2.7329
Comment
Date 31/08/2012 03:56:10
Frequency (MHz) 50.32
Nucleus 13C
Original Points Count 32768
Yogesh G
Points Count 32768
Sweep Width (Hz) 11990.41
Temperature (grad C) 0.000
Chloroform-d
2e
MeO
MeO
OMe
O
NH
100.21
24.65
114.10
77.00
138.99
147.99
149.85
150.35
56.28
60.51
61.13
64.59
204.31


4 Jan 2013
Acquisition Time (sec) 2.7329
Comment
Date 31/08/2012 03:18:32
Frequency (MHz) 50.32
Nucleus 13C
Original Points Count 32768
Yogesh G
Points Count 32768
Sweep Width (Hz) 11990.41
Temperature (grad C) 0.000
24.65
56.27
60.52
61.14
100.17
MeO
MeO
OMe
O
N
H
2e

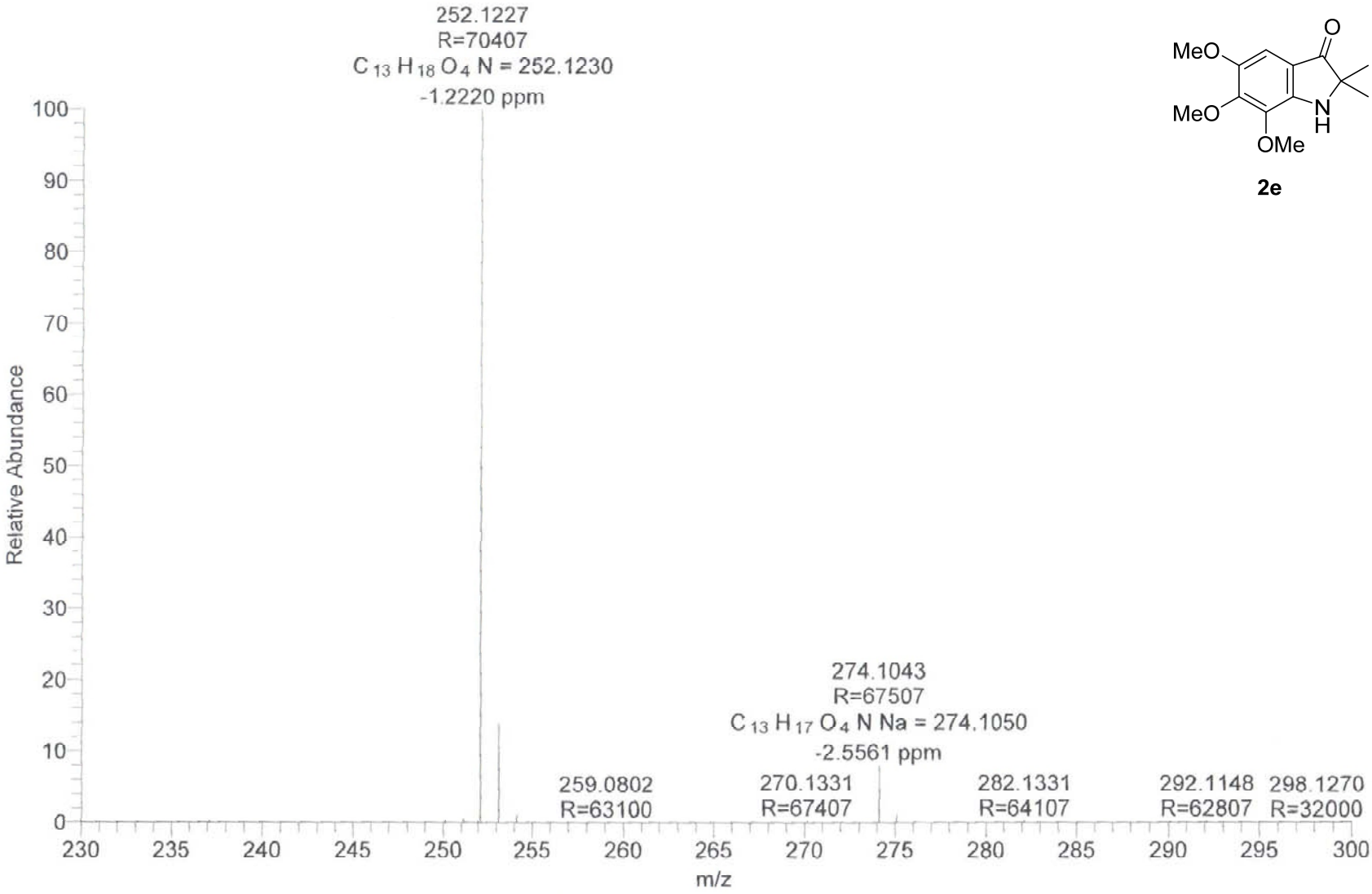

O
NH
OMe
MeO
MeO

2e

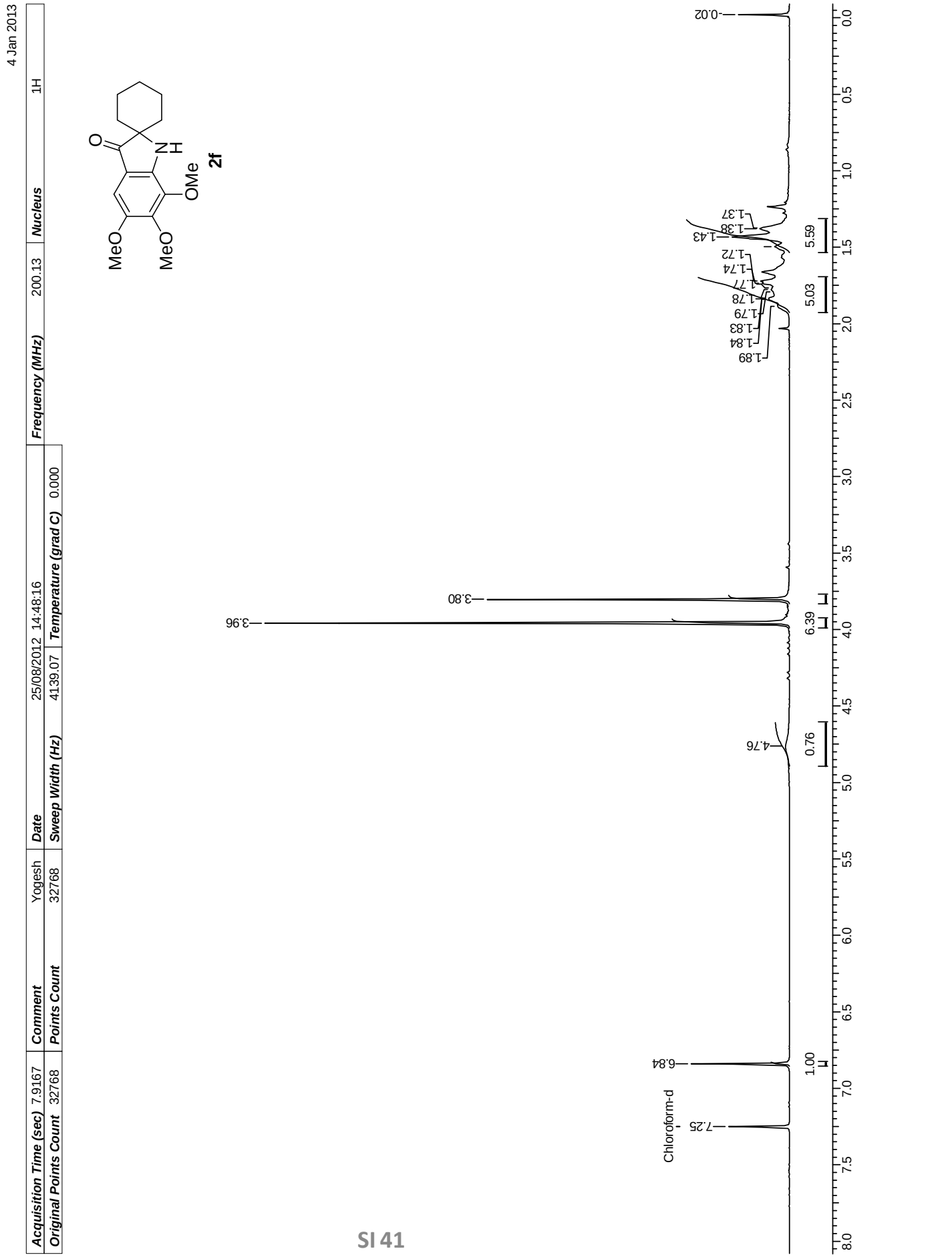

306.00
388.22
386.07
372.07
343.11
329.12
321.15
292.10
275.87
273.96
272.00
252.07
237.06
222.02
212.08
194.14
176.03
147.02
119.25
m/z
100
150
200
250
300
350

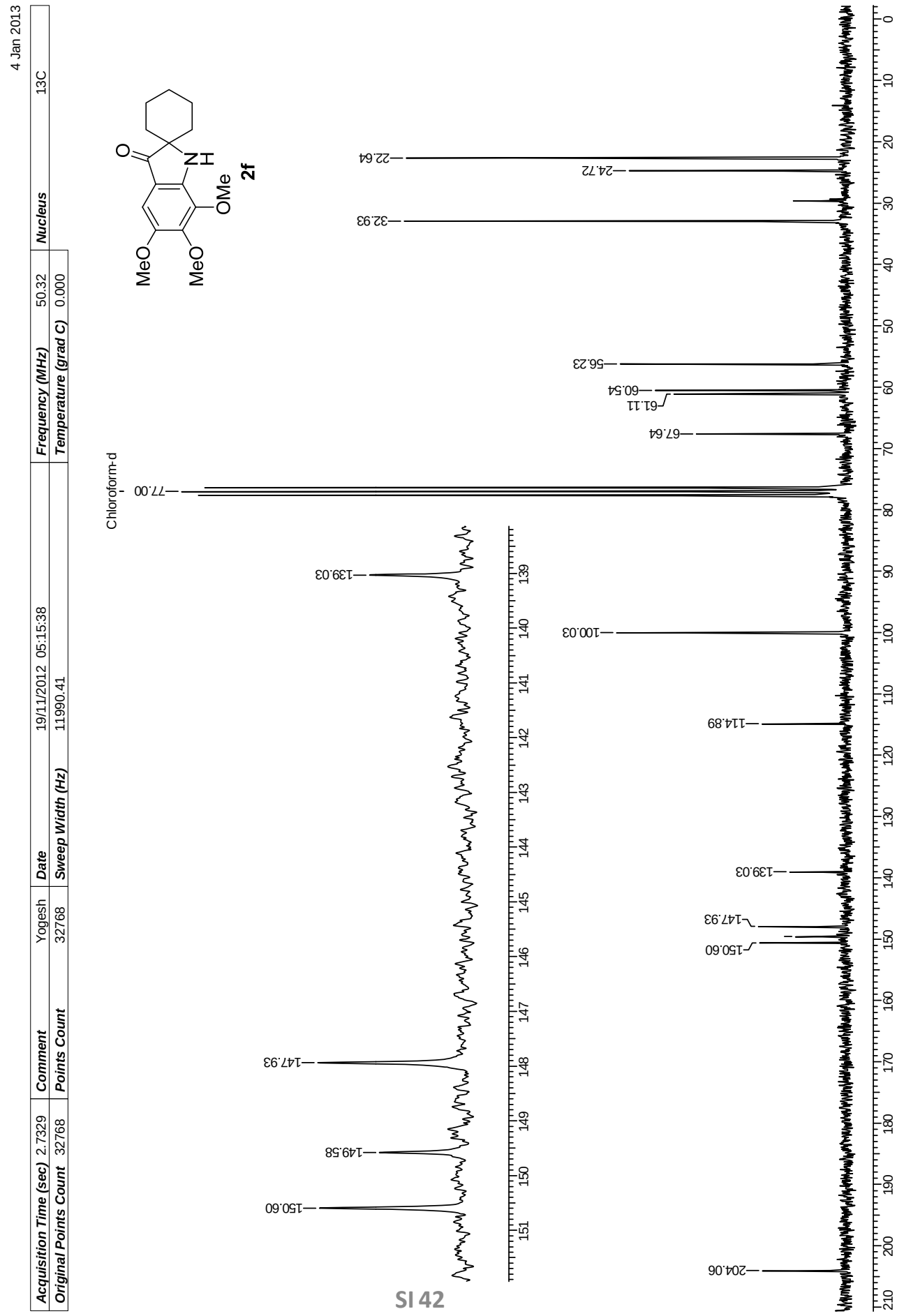
D:\Data\YM-851 1/1/2013 4:19:31 PM

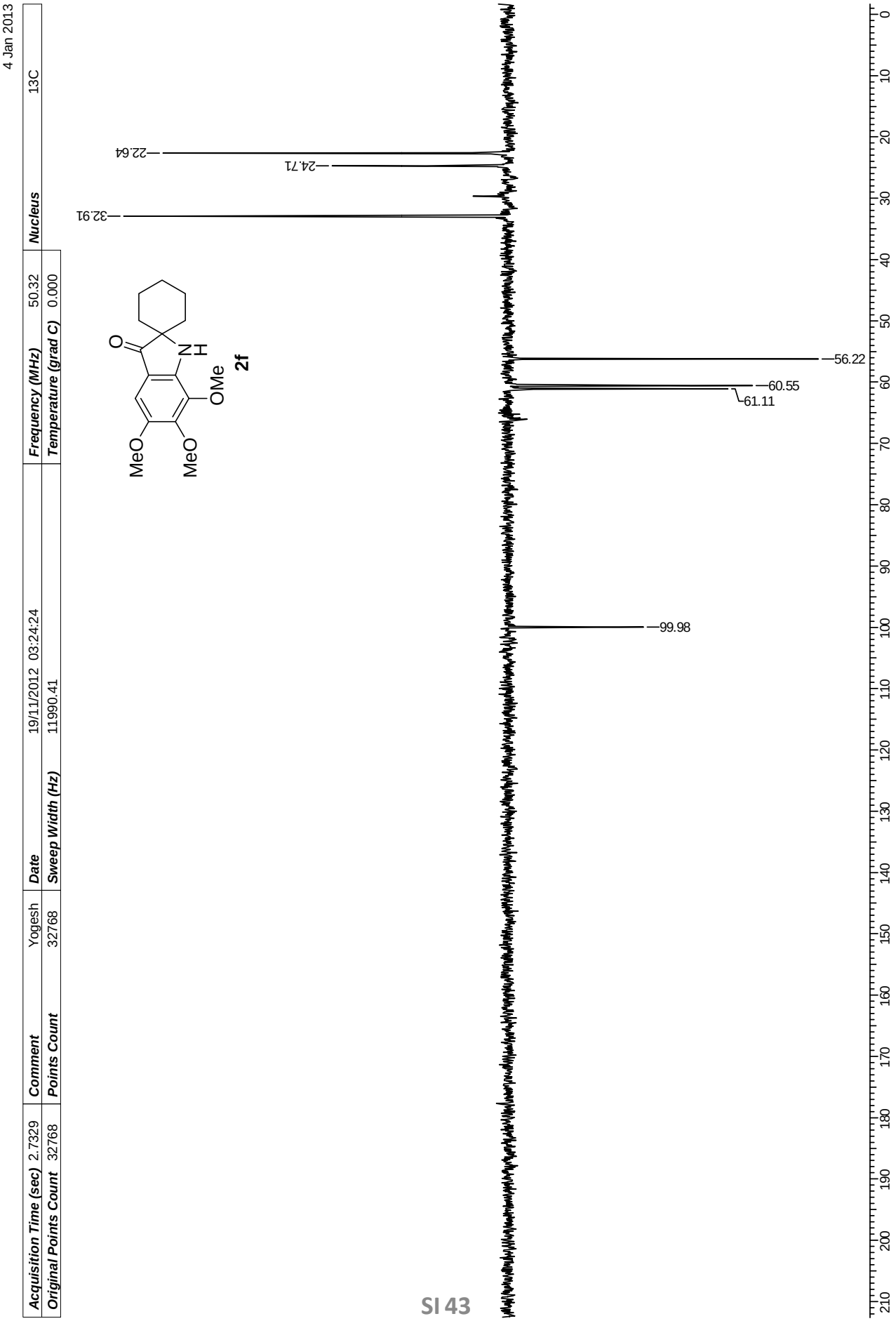
YM-851 #881 RT: 3.92 AV: 1 NL: 9.00E9
T: FTMS + p ESI Full ms [100.00-700.00]

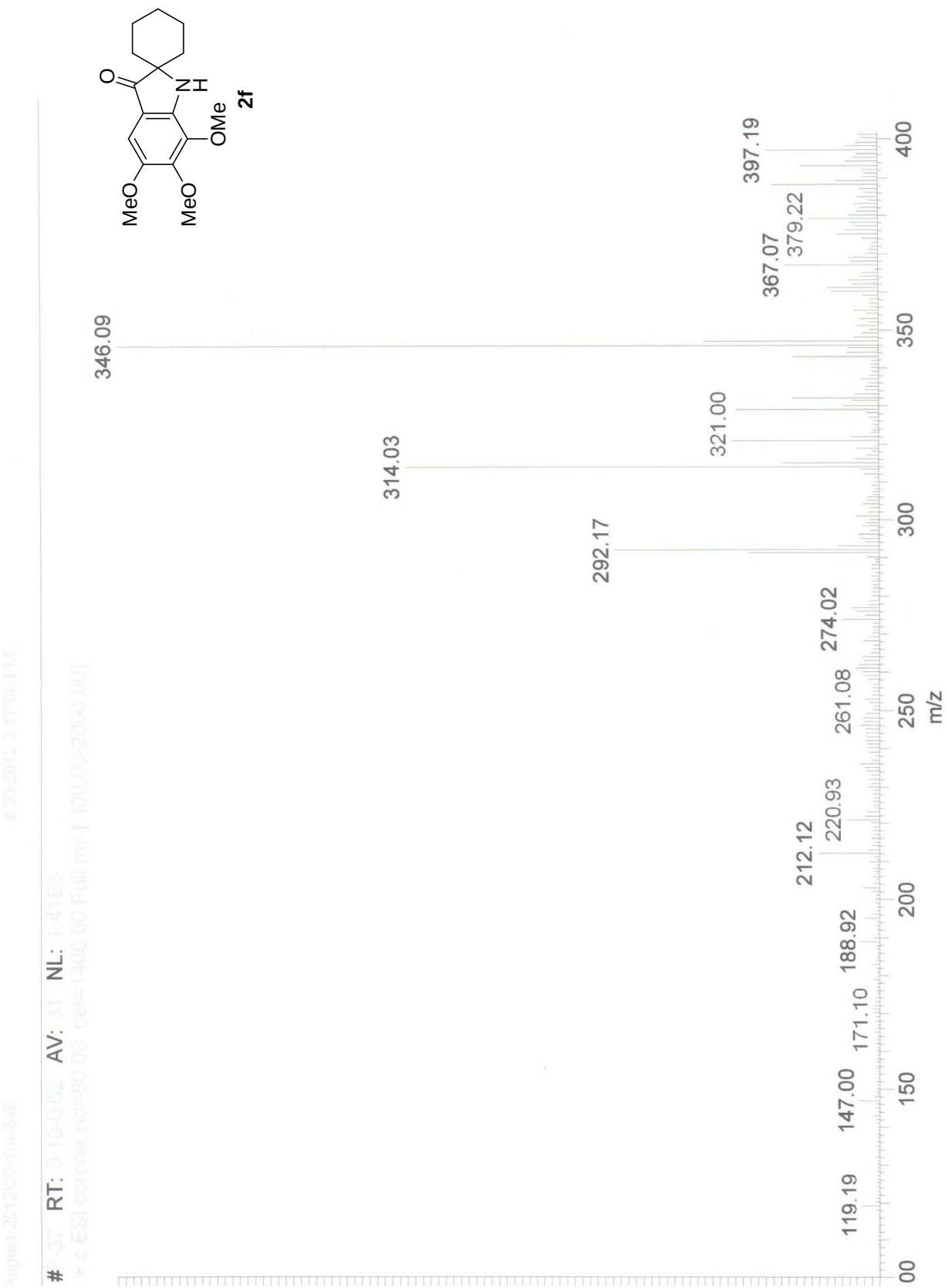


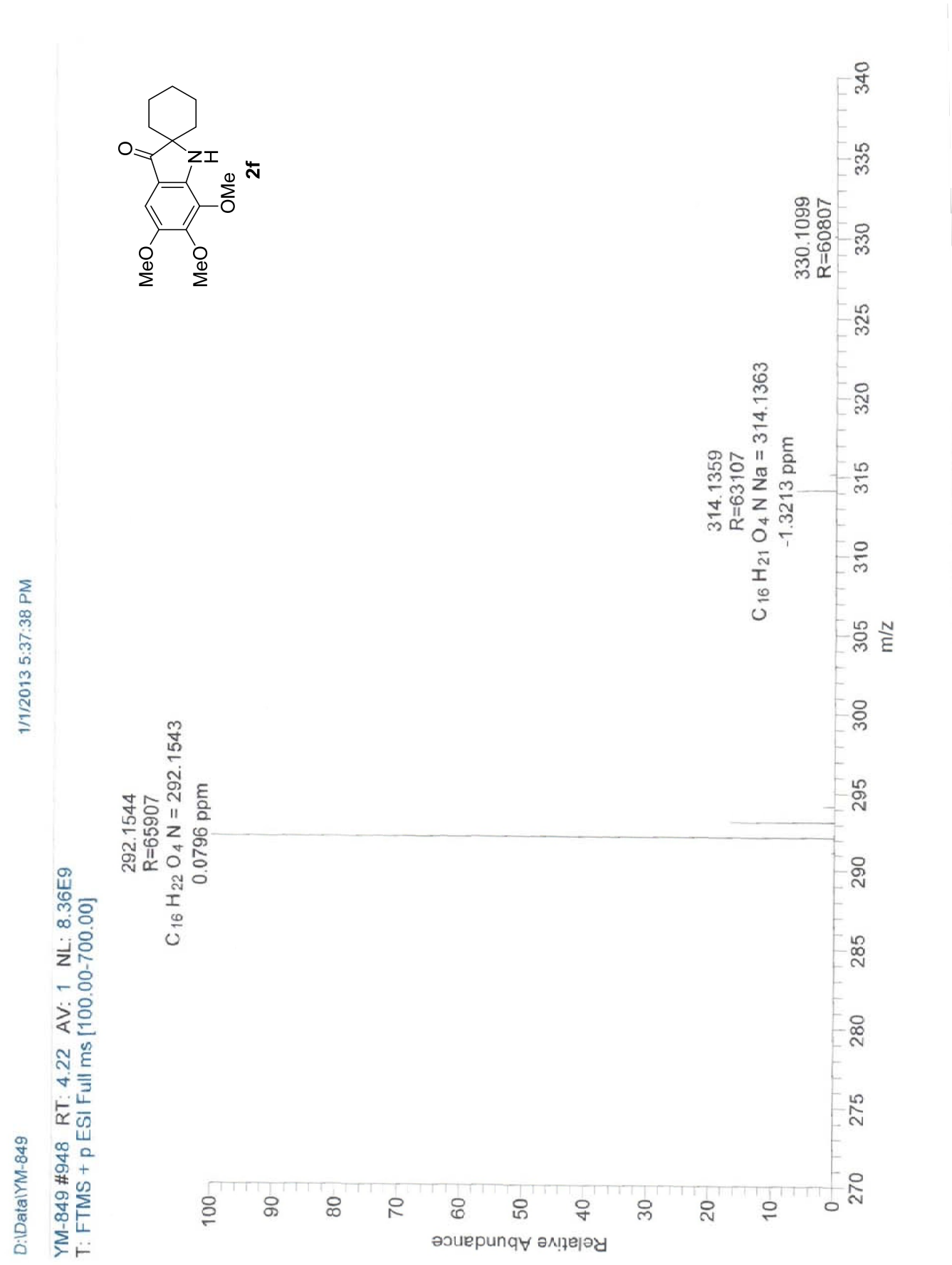
2e

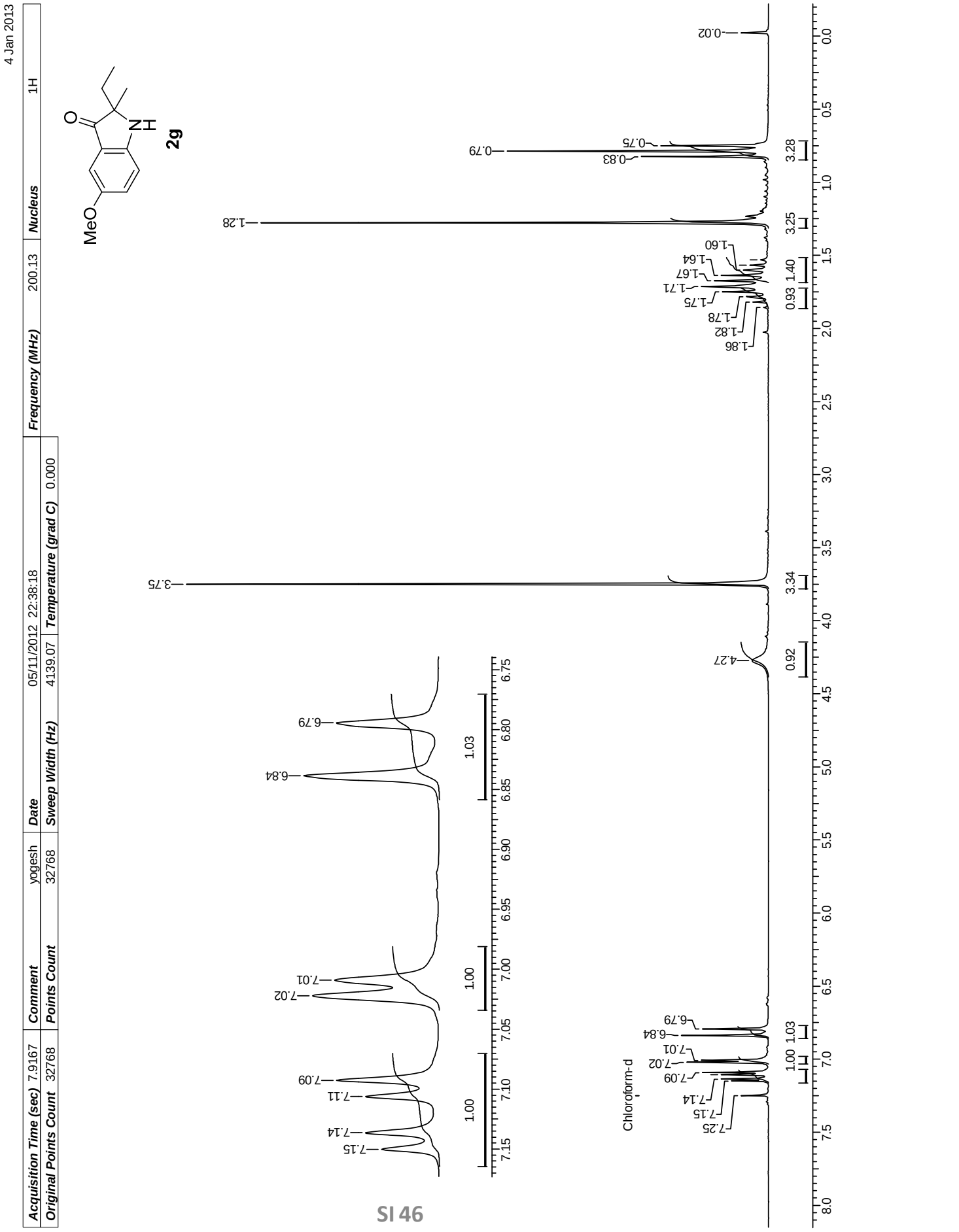

4 Jan 2013
Acquisition Time (sec) 7.9167
Original Points Count 32768
Comment
Points Count
Yogesh
32768
Date
25/08/2012 14:48:16
Sweep Width (Hz)
4139.07
Temperature (grad C)
0.000
Frequency (MHz)
200.13
Nucleus
1H
MeO
MeO
OMe
NH
O
2f
Chloroform-d
7.25
6.84
3.96
3.80
4.76
1.89
1.84
1.83
1.79
1.78
1.77
1.74
1.72
1.43
1.38
1.37
0.02
1.00
6.39
0.76
5.03
5.59

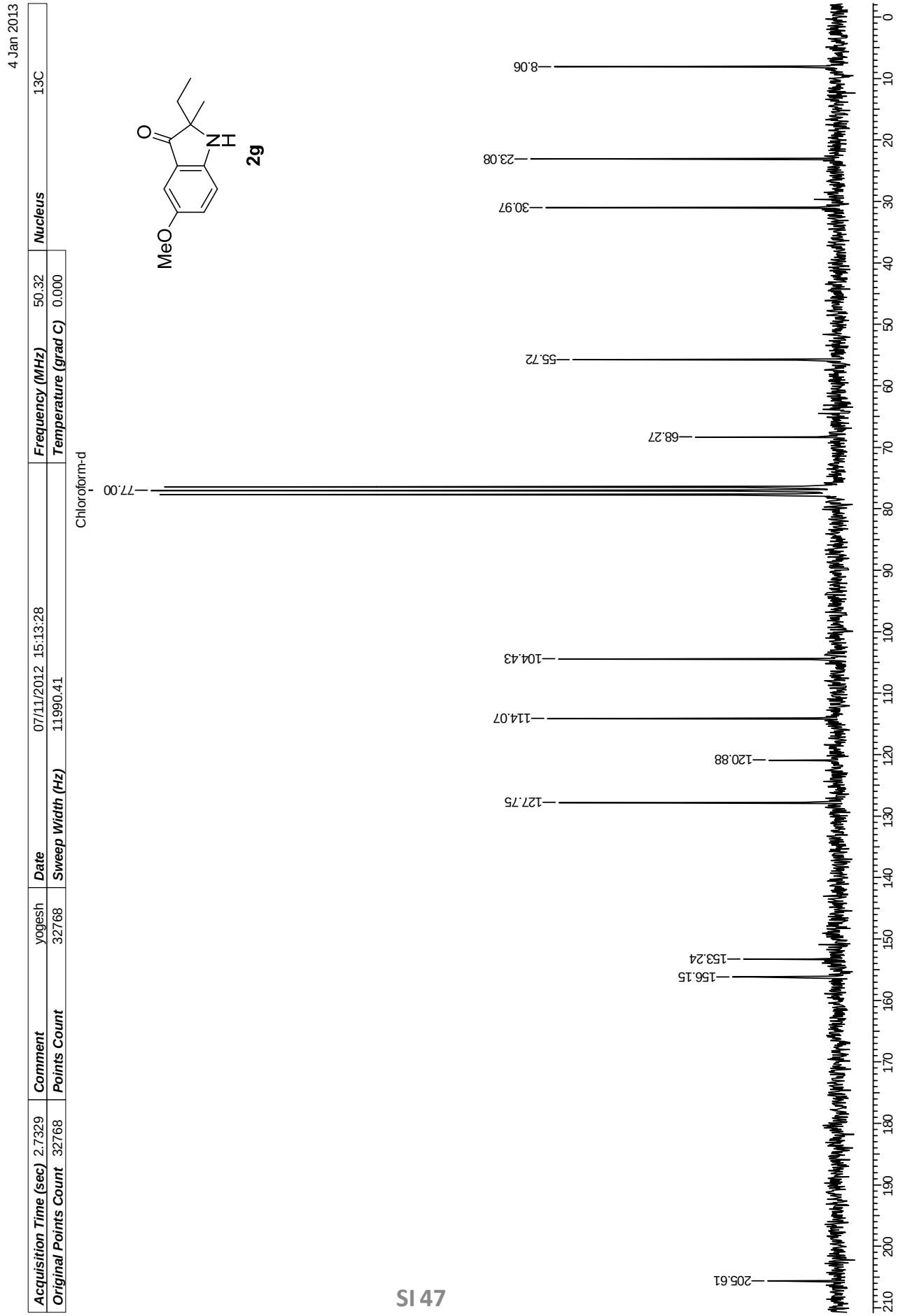

4 Jan 2013
Acquisition Time (sec) 2.7329
Original Points Count 32768
Comment
Points Count
Yogesh
32768
Date
Sweep Width (Hz)
19/11/2012 05:15:38
11990.41
Frequency (MHz) 50.32
Temperature (grad C) 0.000
Nucleus 13C
O
NH
OMe
MeO
MeO
2f
Chloroform-d
77.00
22.64
24.72
32.93
56.23
60.54
61.11
67.64
100.03
114.89
139.03
147.93
150.60
204.06
139.03
147.93
149.58
150.60

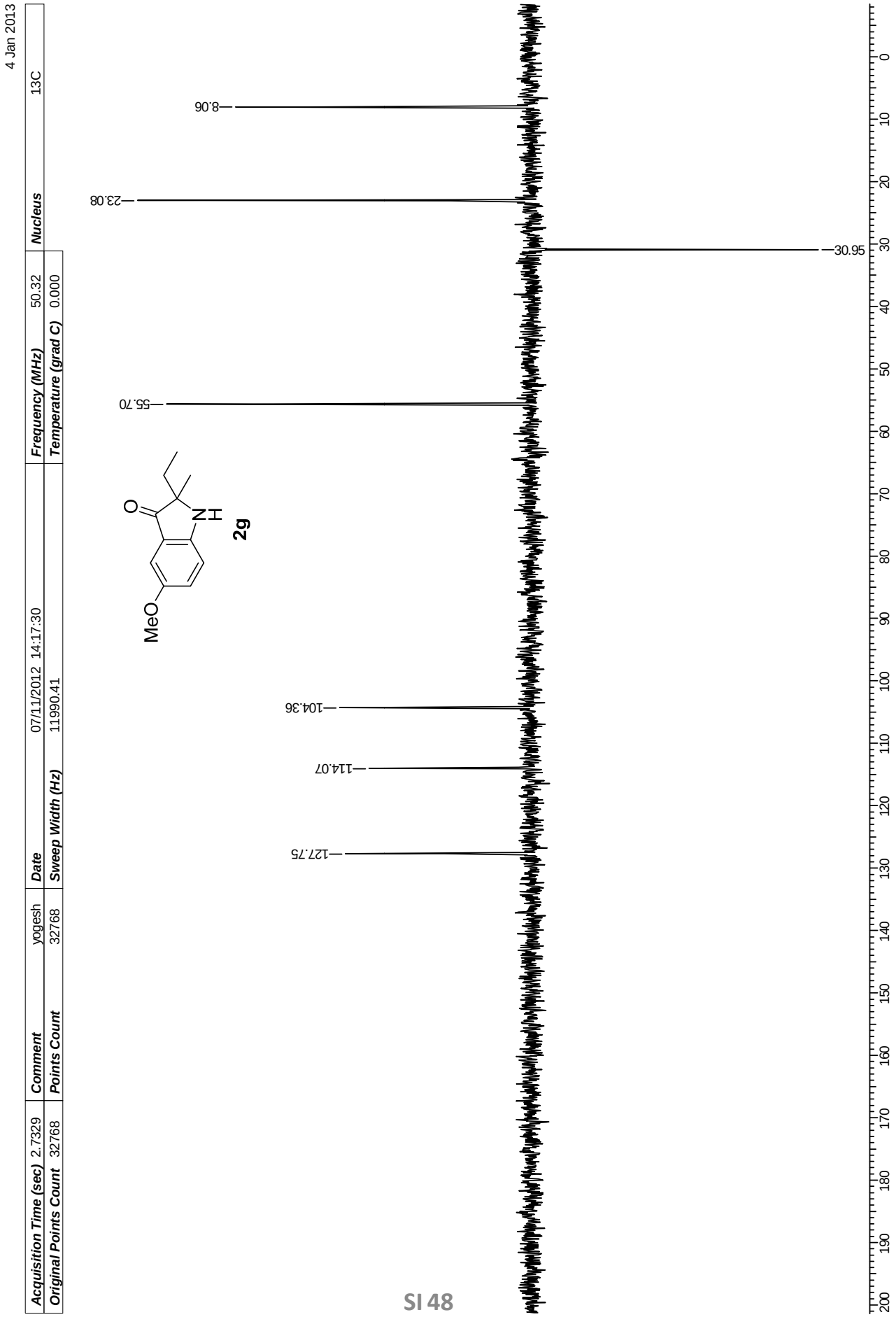

4 Jan 2013
Acquisition Time (sec) 2.7329
Original Points Count 32768
Comment
Points Count
Yogesh
32768
Date
Sweep Width (Hz)
19/11/2012 03:24:24
11990.41
Frequency (MHz) 50.32
Temperature (grad C) 0.000
Nucleus
13C
22.64
24.71
32.91
56.22
60.55
61.11
99.98
O
N
H
OMe
MeO
MeO
2f

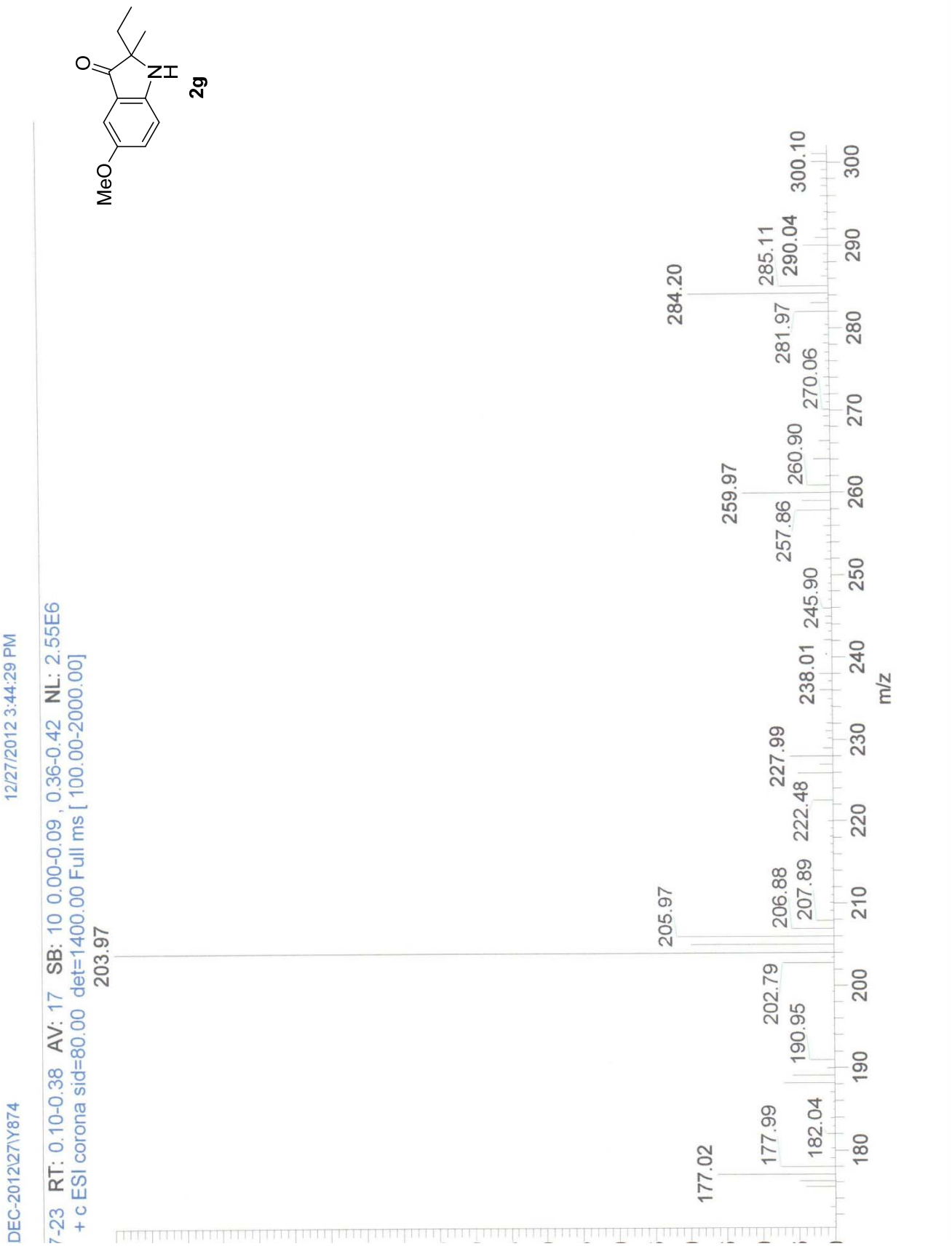

O
N
H
OMe
MeO
MeO
2f
37 RT: 0.10-0.62 AV: 31 NL: 1.41E6
+ c ESI corona sid=80.00 det=400.00 Full ms [100.00-2000.00]
346.09
314.03
292.17
321.00
397.19
367.07
379.22
274.02
261.08
220.93
212.12
188.92
171.10
147.00
119.19
m/z
00
150
200
250
300
350
400

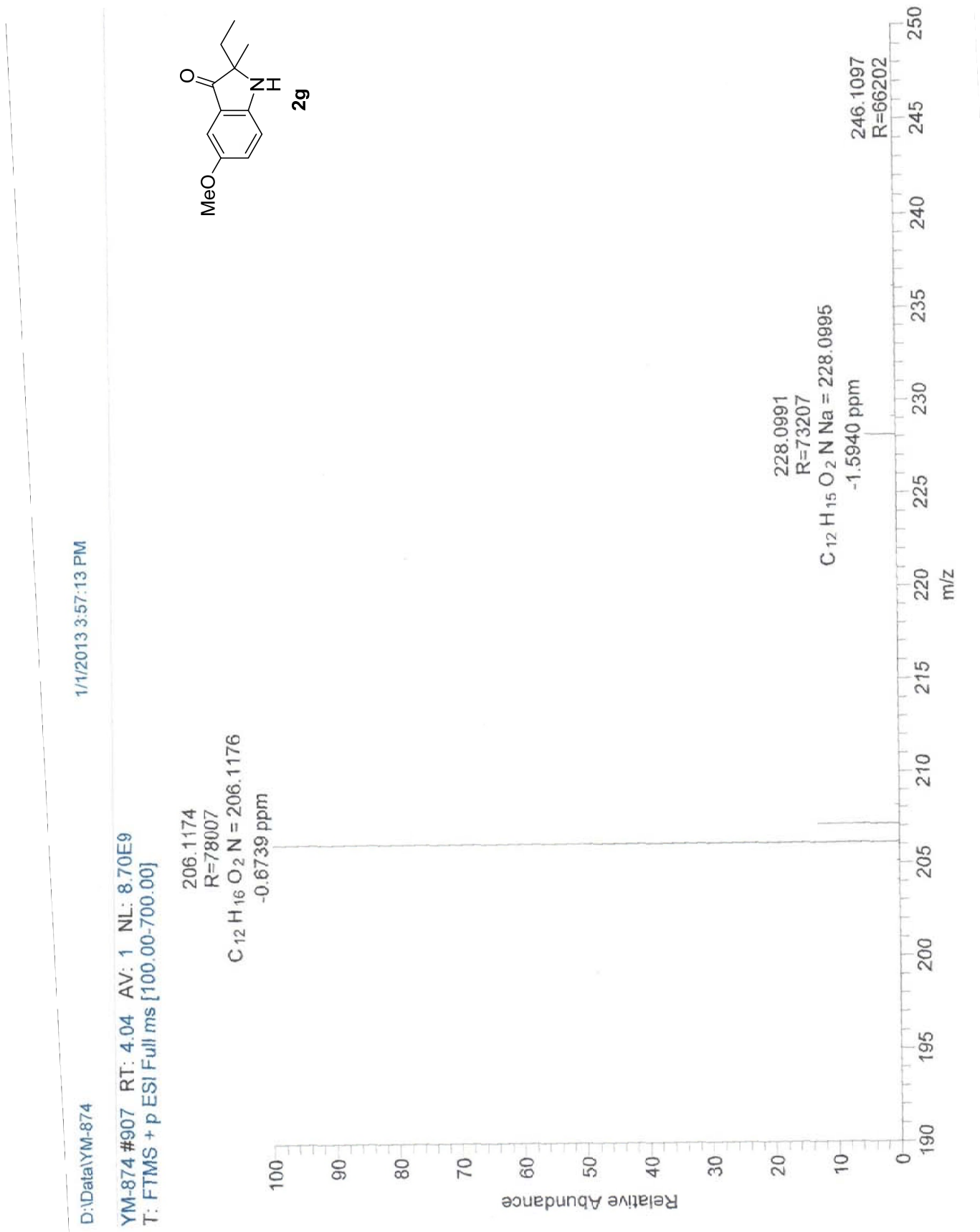

D:\Data\YM-849
1/1/2013 5:37:38 PM
YM-849 #948 RT: 4.22 AV: 1 NL: 8.36E9
T: FTMS + p ESI Full ms [100.00-700.00]
292.1544
R=65907
C 16 H 22 O 4 N = 292.1543
0.0796 ppm
314.1359
R=63107
C 16 H 21 O 4 N Na = 314.1363
-1.3213 ppm
330.1099
R=60807
MeO
MeO
OMe
2f
Relative Abundance
m/z

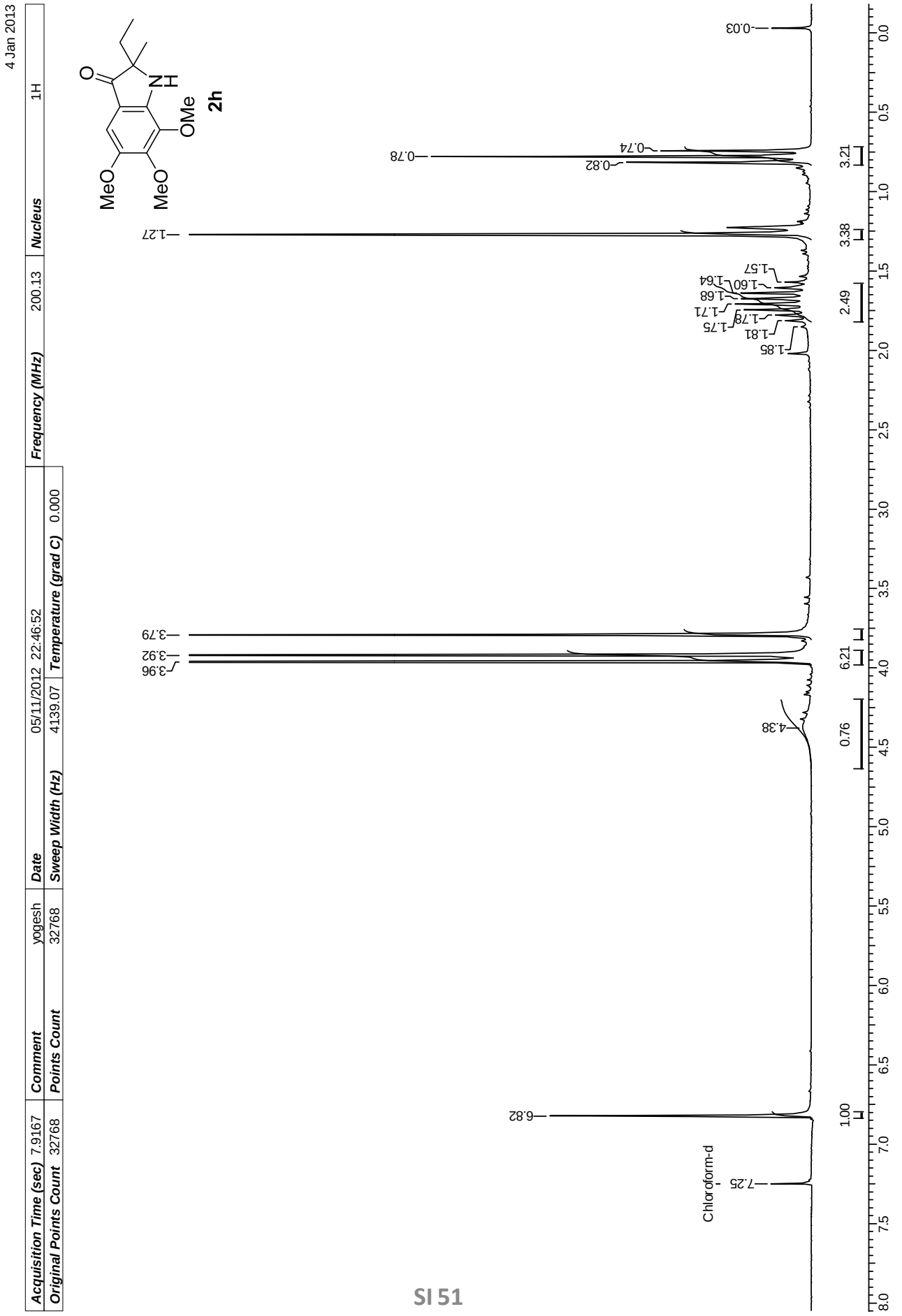

4 Jan 2013
Acquisition Time (sec) 7.9167
Original Points Count 32768
Comment
Points Count
yogesh
32768
Date
Sweep Width (Hz)
05/11/2012 22:38:18
4139.07
Temperature (grad C) 0.000
Frequency (MHz) 200.13
Nucleus 1H
MeO
NH
O
2g
3.75
1.28
0.79
0.75
0.83
0.02
1.86
1.82
1.78
1.75
1.71
1.67
1.64
1.60
4.27
7.25
7.15
7.14
7.09
7.02
7.01
6.84
6.79
Chloroform-d
7.15
7.14
7.11
7.09
7.02
7.01
6.84
6.79
1.00
1.03
1.00
1.03
0.92
3.34
0.93
1.40
3.25
3.28

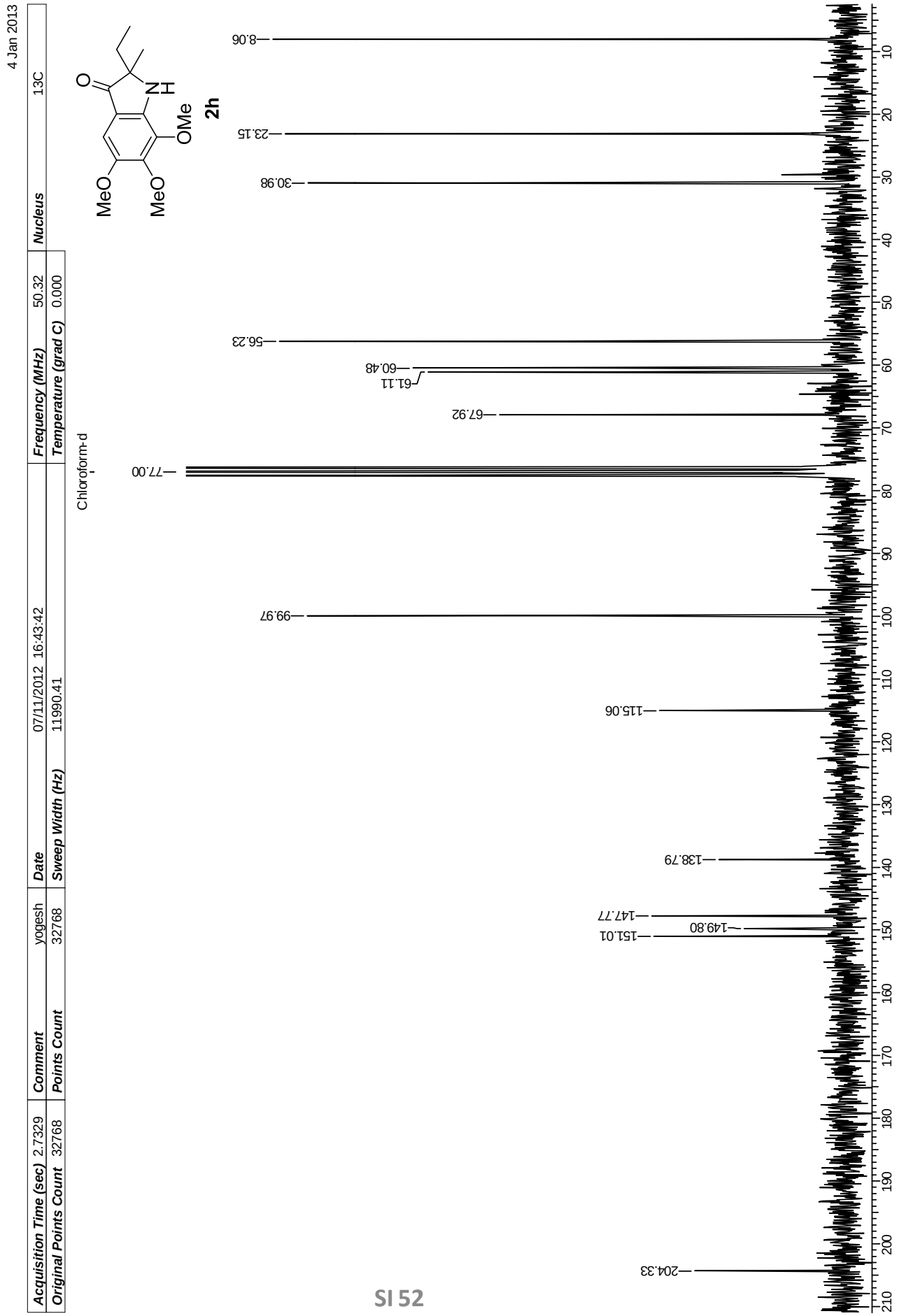

4 Jan 2013
Acquisition Time (sec) 2.7329
Original Points Count 32768
Comment
Points Count
yogesh
32768
Date
Sweep Width (Hz)
07/11/2012 15:13:28
11990.41
Frequency (MHz) 50.32
Temperature (grad C) 0.000
Nucleus 13C
MeO
NH
O
2g
Chloroform-d
77.00
8.06
23.08
30.97
55.72
68.27
104.43
114.07
120.88
127.75
153.24
156.15
205.61

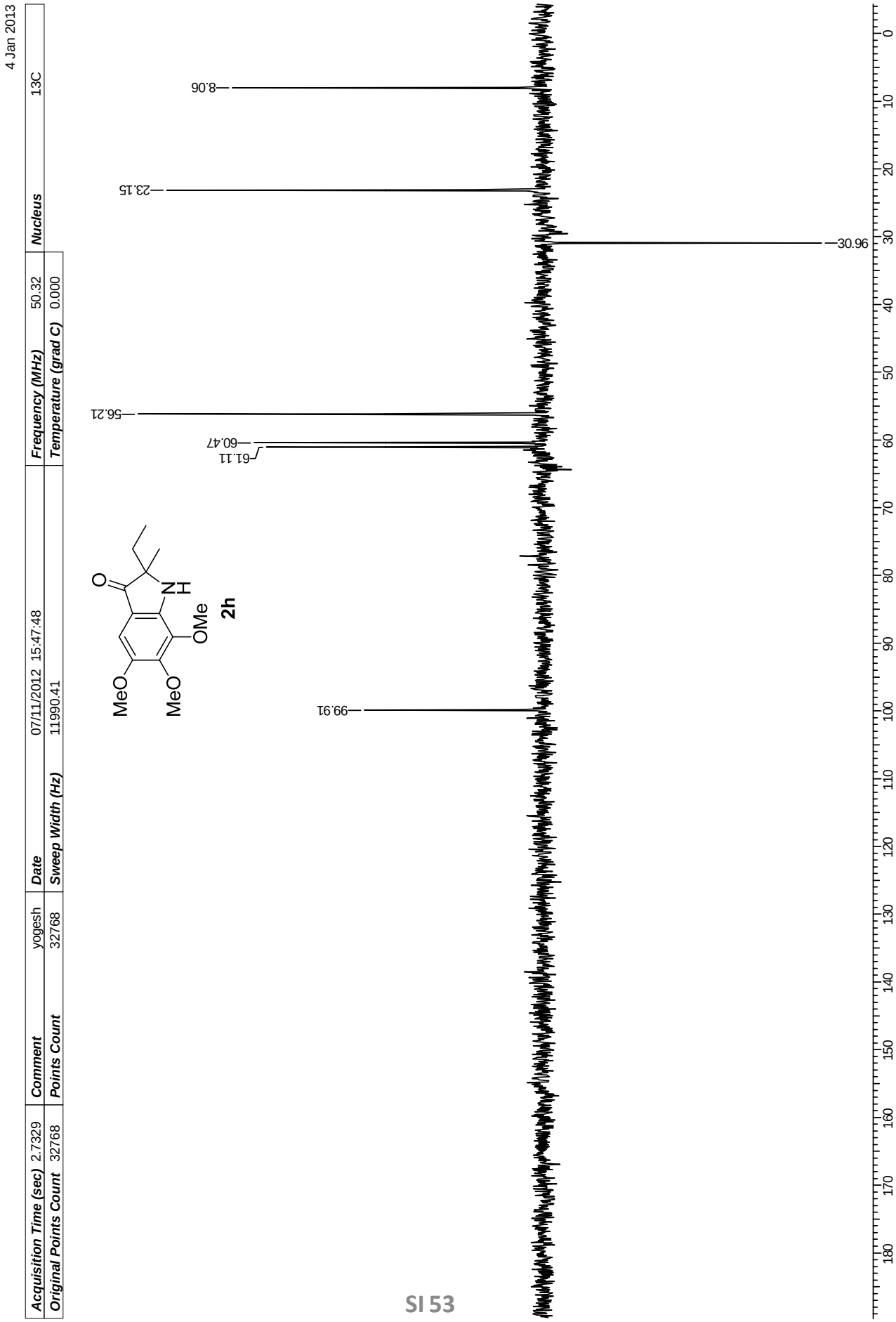

4 Jan 2013
Acquisition Time (sec) 2.7329
Original Points Count 32768
Comment
Points Count
yogesh
32768
Date
Sweep Width (Hz)
07/11/2012 14:17:30
11990.41
Frequency (MHz) 50.32
Temperature (grad C) 0.000
Nucleus 13C
8.06
23.08
30.95
55.70
104.36
114.07
127.75
MeO
O
NH
2g

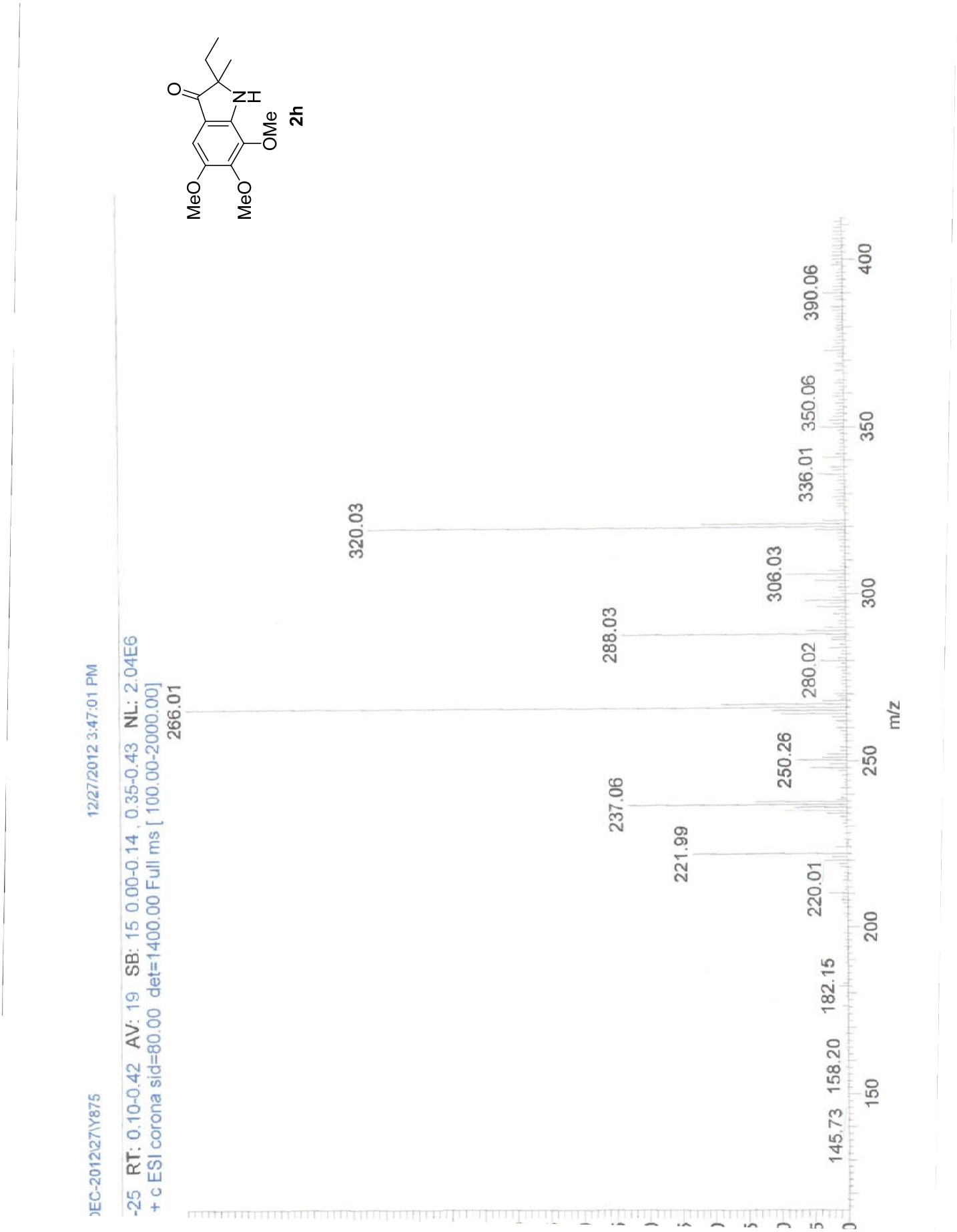


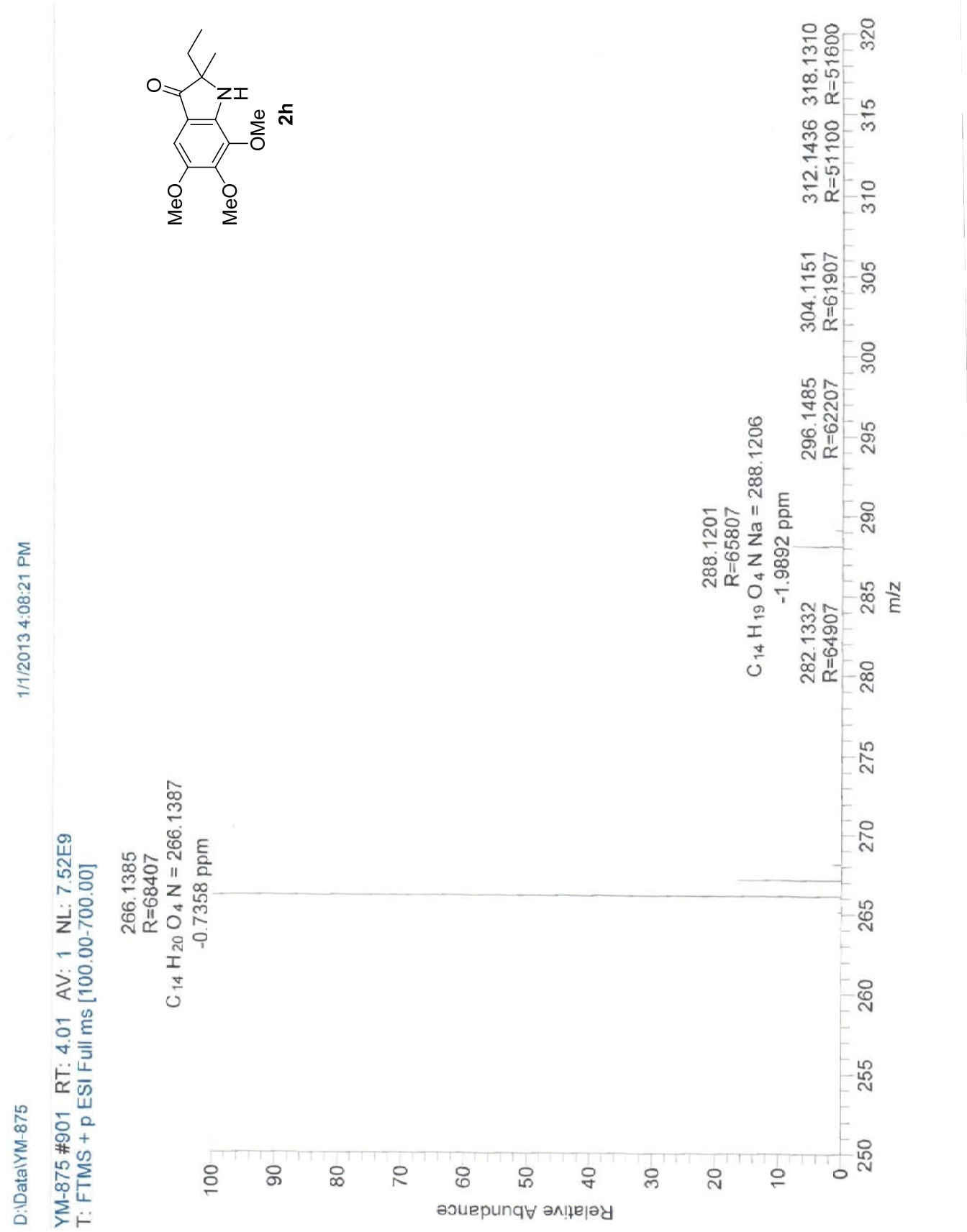

D:\Data\YM-874
1/1/2013 3:57:13 PM
YM-874 #907 RT: 4.04 AV: 1 NL: 8.70E9
T: FTMS + p ESI Full ms [100.00-700.00]
206.1174
R=78007
C 12 H 16 O 2 N = 206.1176
-0.6739 ppm
228.0991
R=73207
C 12 H 15 O 2 N Na = 228.0995
-1.5940 ppm
246.1097
R=66202
Relative Abundance
m/z
MeO
2g


4 Jan 2013
Acquisition Time (sec) 7.9167
Original Points Count 32768
Comment
Points Count
yogesh
32768
Date
Sweep Width (Hz)
05/11/2012 22:46:52
4139.07
Temperature (grad C) 0.000
Frequency (MHz) 200.13
Nucleus 1H
2h
MeO
MeO
OMe
O
NH
Chloroform-d
7.25
6.82
4.38
3.96
3.92
3.79
1.27
1.85
1.81
1.78
1.75
1.71
1.68
1.64
1.60
1.57
0.82
0.78
0.74
0.03
1.00
0.76
6.21
2.49
3.38
3.21


4 Jan 2013
Acquisition Time (sec) 2.7329
Original Points Count 32768
Comment
Points Count
yogesh
32768
Date
Sweep Width (Hz)
07/11/2012 16:43:42
11990.41
Frequency (MHz) 50.32
Temperature (grad C) 0.000
Nucleus 13C
2h
MeO
MeO
OMe
O
NH
Chloroform-d
8.06
23.15
30.98
56.23
60.48
61.11
67.92
77.00
99.97
115.06
138.79
147.77
149.80
151.01
204.33


4 Jan 2013
Acquisition Time (sec) 2.7329
Original Points Count 32768
Comment
Points Count
yogesh
32768
Date
Sweep Width (Hz)
07/11/2012 15:47:48
11990.41
Frequency (MHz) 50.32
Temperature (grad C) 0.000
Nucleus 13C
8.06
23.15
30.96
56.21
60.47
61.11
99.91
MeO
MeO
OMe
NH
O
2h

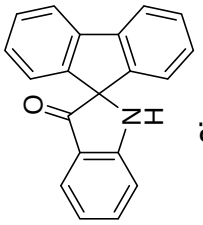

O
N
H
OMe
MeO
MeO
2h
DEC-2012\27\Y875
12/27/2012 3:47:01 PM
-25 RT: 0.10-0.42 AV: 19 SB: 15 0.00-0.14 , 0.35-0.43 NL: 2.04E6
+ c ESI corona sid=80.00 det=1400.00 Full ms [100.00-2000.00]
266.01
320.03
288.03
306.03
336.01
350.06
390.06
237.06
221.99
250.26
280.02
220.01
182.15
158.20
145.73
150
200
250
300
350
400
m/z


D:\Data\YM-875
1/1/2013 4:08:21 PM
YM-875 #901 RT: 4.01 AV: 1 NL: 7.52E9
T: FTMS + p ESI Full ms [100.00-700.00]
266.1385
R=68407
C 14 H 20 O 4 N = 266.1387
-0.7358 ppm
288.1201
R=65807
C 14 H 19 O 4 N Na = 288.1206
-1.9892 ppm
282.1332
R=64907
296.1485
R=62207
304.1151
R=61907
312.1436
R=51100
318.1310
R=51600
Relative Abundance
m/z
MeO
MeO
OMe
O
NH
2h

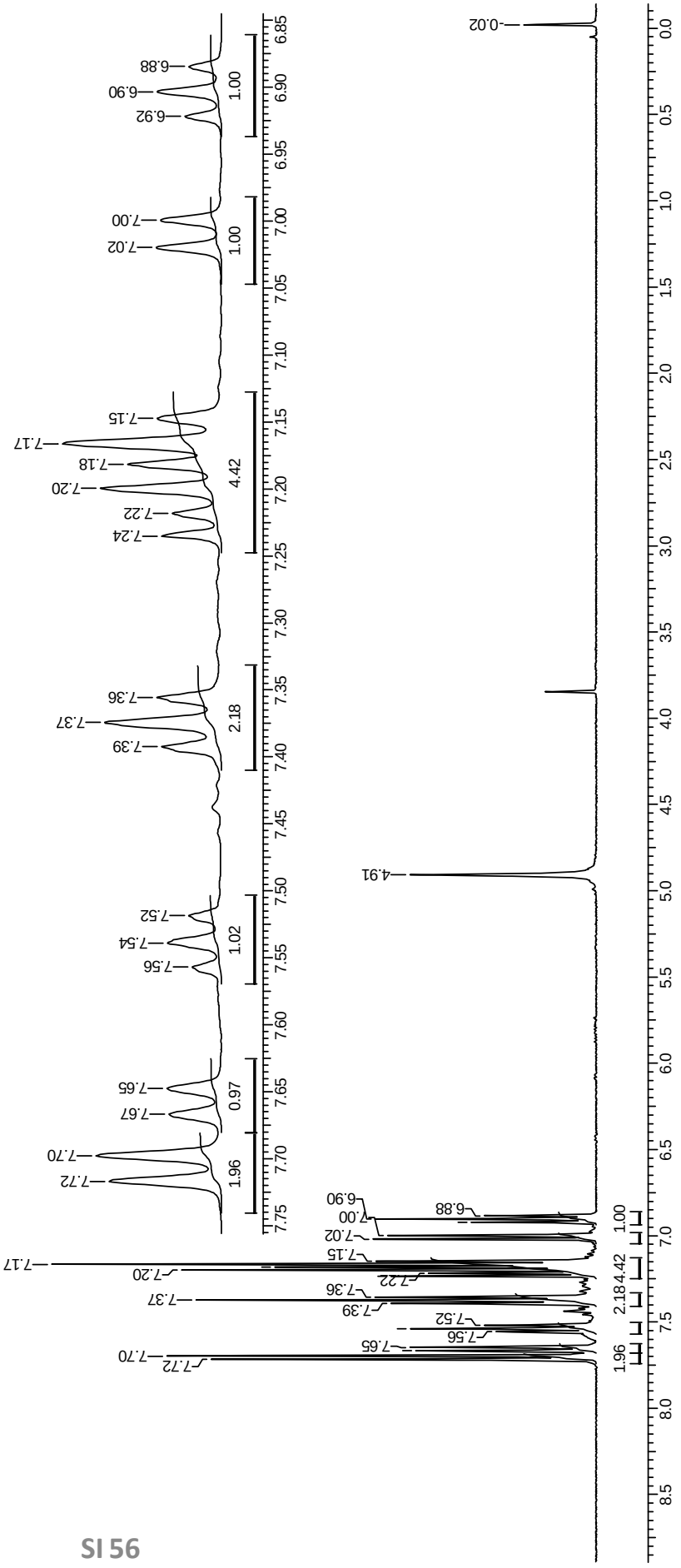
4 Jan 2013

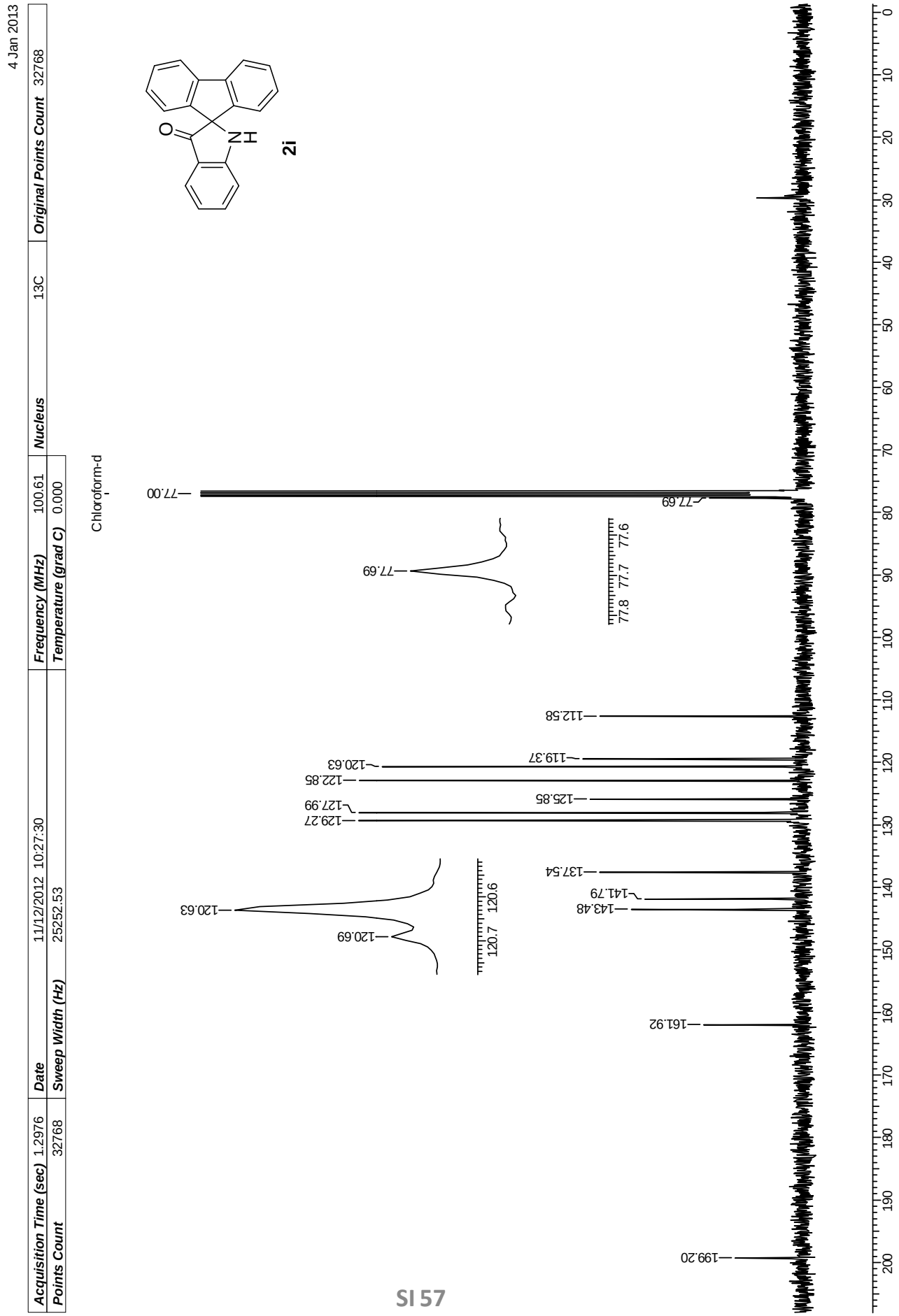
Acquisition Time (sec)	2.0447				
Comment	1H Yogesh				
Date	11 Dec 2012 08:44:56	***Frequency (MHz)***	400.13	***Nucleus***	1H
Original Points Count	16384	***Points Count***	32768	***Solvent***	CHLOROFORM-d

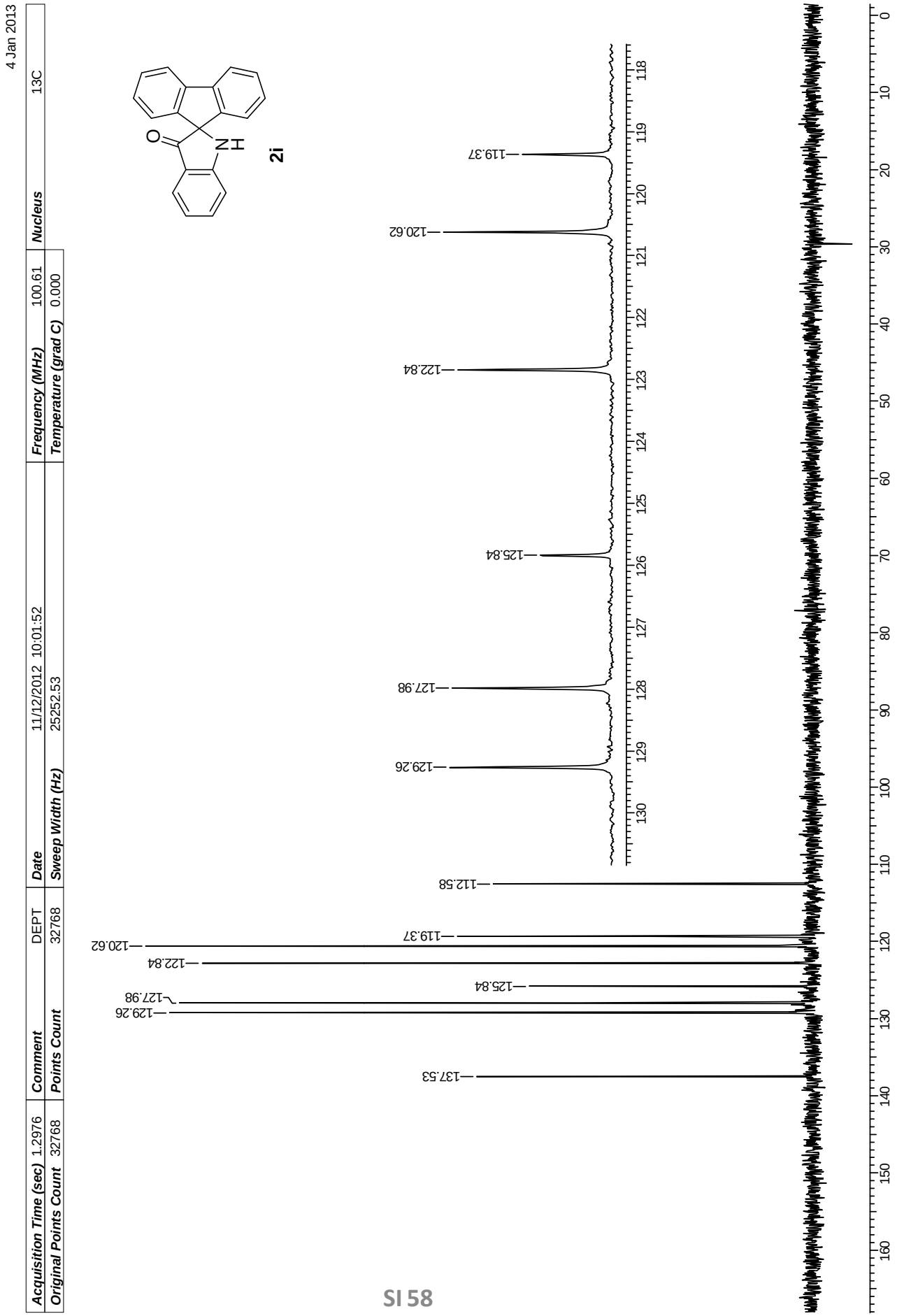
Number of Transients	16
Sweep Width (Hz)	8012.58

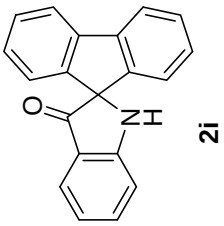


2i

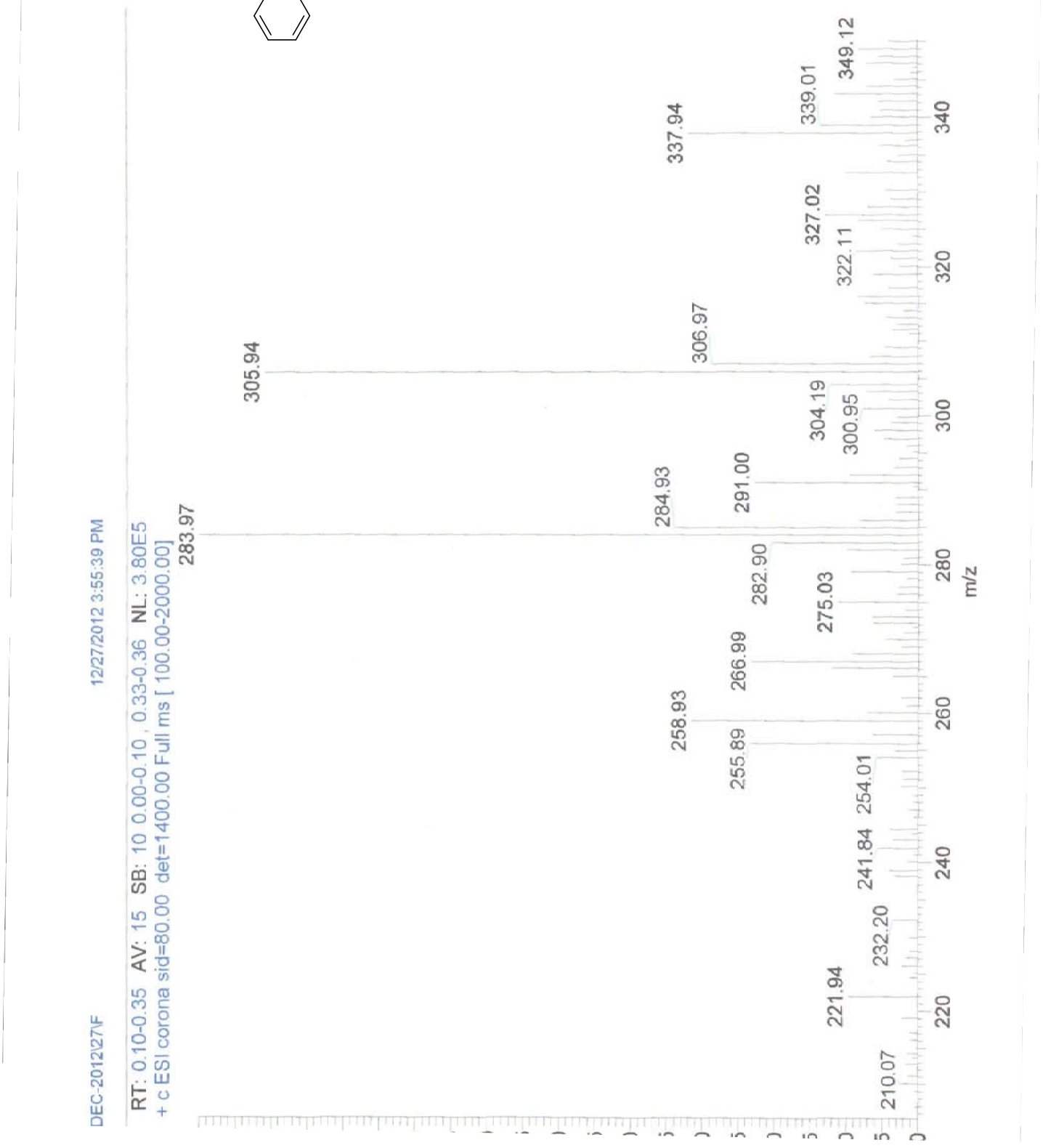


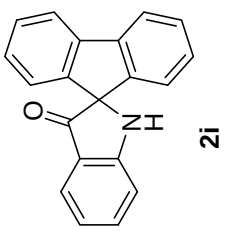

4 Jan 2013
Acquisition Time (sec) 1.2976
Points Count 32768
Date 11/12/2012 10:27:30
Sweep Width (Hz) 25252.53
Frequency (MHz) 100.61
Temperature (grad C) 0.000
Nucleus 13C
Original Points Count 32768
Chloroform-d
2i
O
N
H
77.00
77.69
77.69
77.6
77.7
77.8
112.58
119.37
120.63
122.85
125.85
127.99
129.27
120.63
120.69
120.6
120.7
137.54
141.79
143.48
161.92
199.20


4 Jan 2013
Acquisition Time (sec) 1.2976
Original Points Count 32768
Comment
Points Count
DEPT
32768
Date
Sweep Width (Hz)
11/12/2012 10:01:52
25252.53
Frequency (MHz) 100.61
Temperature (grad C) 0.000
Nucleus 13C
2i
O
N
H
119.37
120.62
122.84
125.84
127.98
129.26
112.58
137.53


O
N
H

2i


DEC-2012\27\F
12/27/2012 3:55:39 PM
RT: 0.10-0.35 AV: 15 SB: 10 0.00-0.10 , 0.33-0.36 NL: 3.80E5
+ c ESI corona sid=80.00 det=1400.00 Full ms [100.00-2000.00]
283.97
305.94
306.97
337.94
339.01
349.12
284.93
291.00
304.19
300.95
327.02
322.11
258.93
266.99
282.90
275.03
255.89
221.94
232.20
241.84
254.01
210.07
m/z



2i

D:\Data\F_130101171520 1/1/2013 5:15:20 PM

F_130101171520 #909 RT: 4.05 AV: 1 NL: 1.44E9
T: FTMS + p ESI Full ms [100.00-700.00]

284.1071
R=66507
$C_{20}H_{14}ON$ = 284.1070
0.3364 ppm

306.0887
R=63707
$C_{20}H_{13}ONNa$ = 306.0889
-0.7210 ppm

295.5916
R=39600

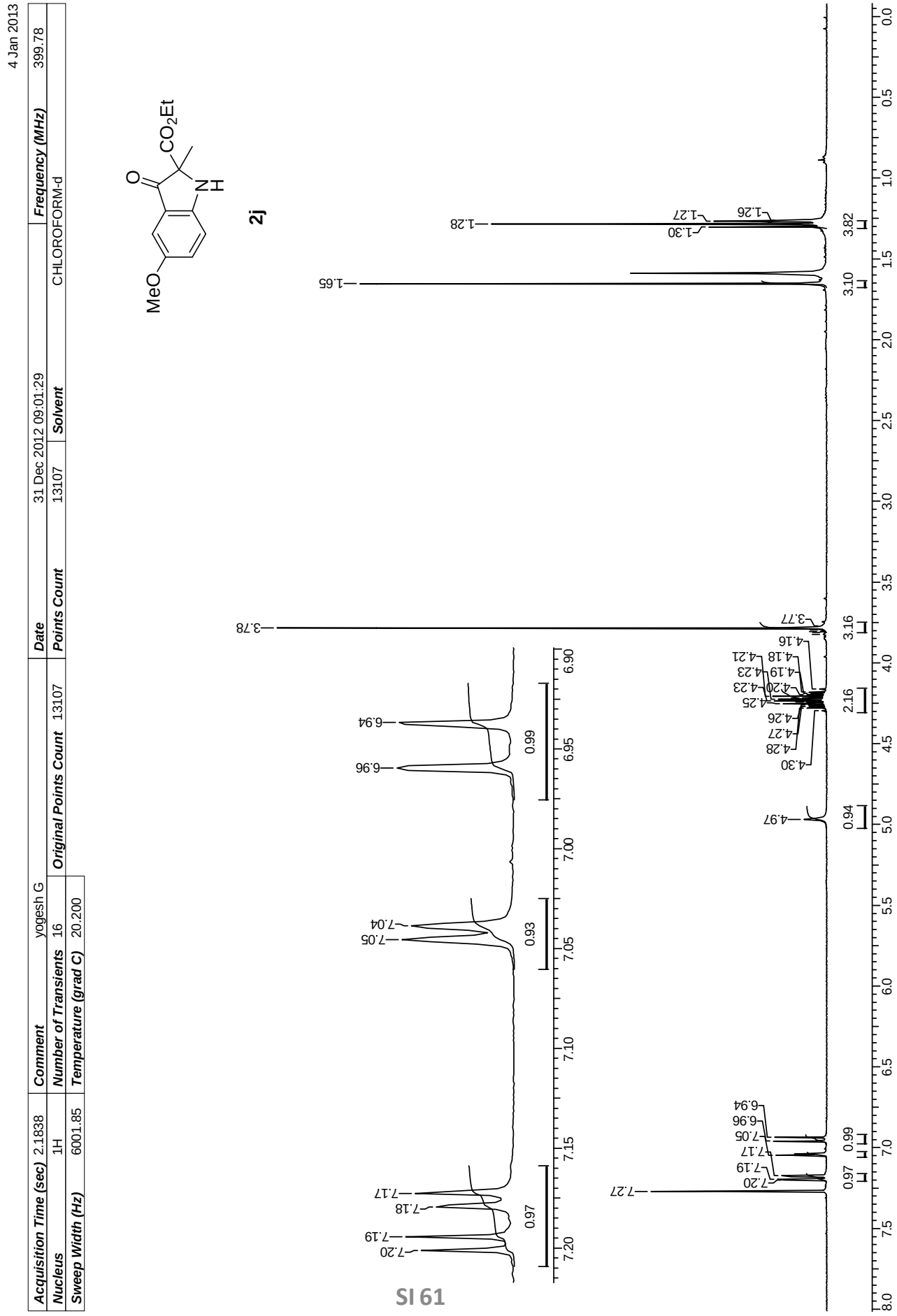
311.0663
R=59507

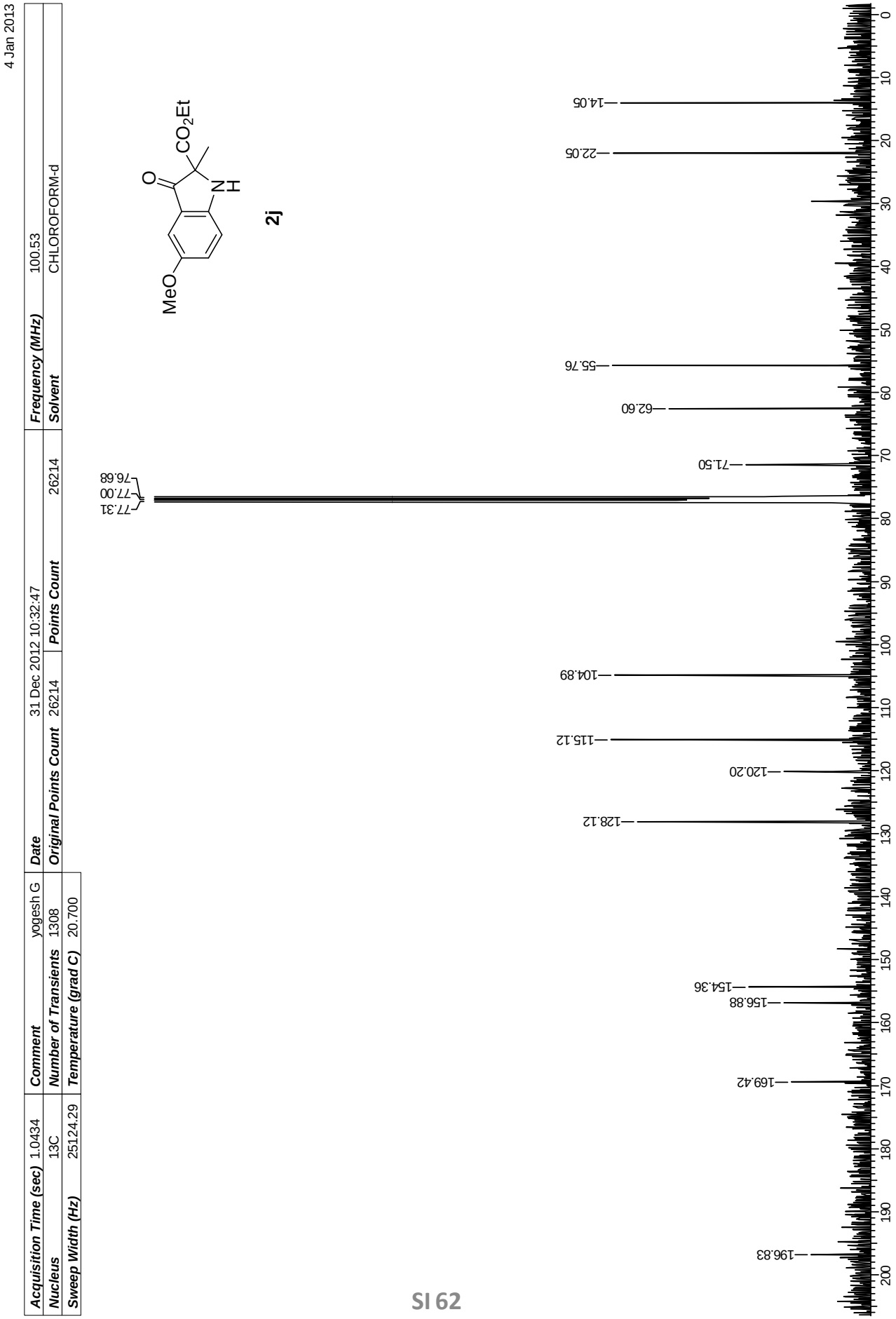
322.0627
R=62607

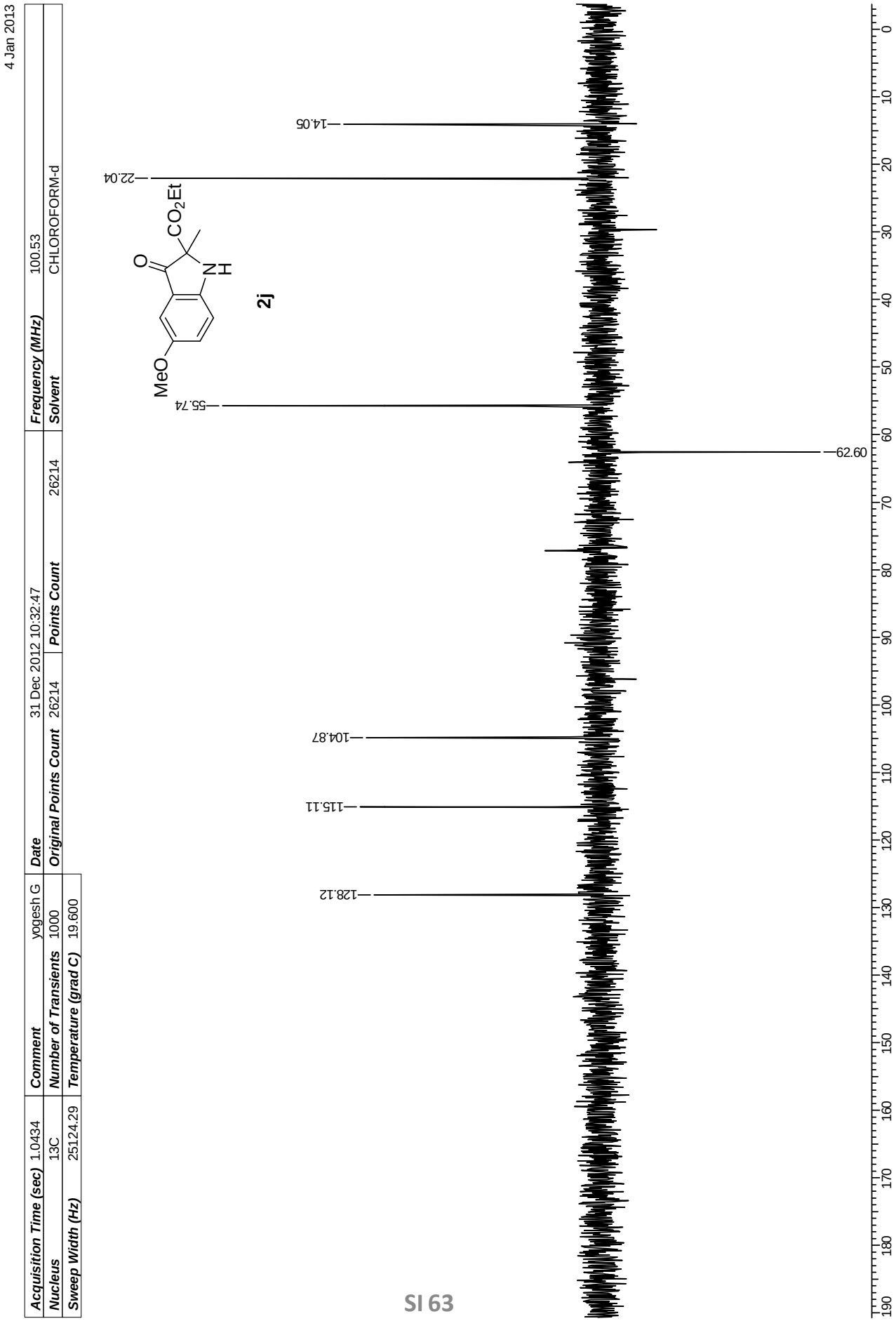
329.1646
R=60707

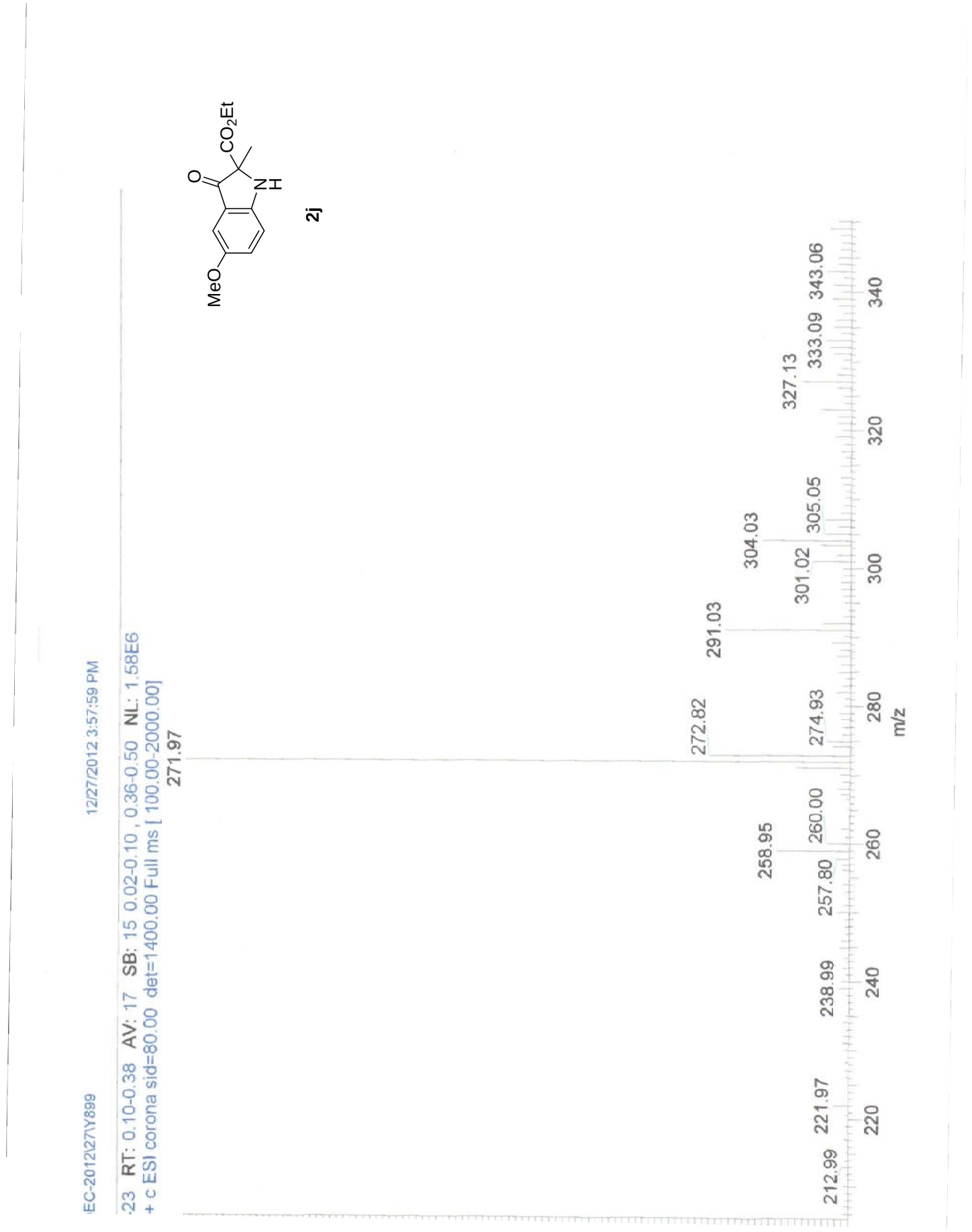
Relative Abundance

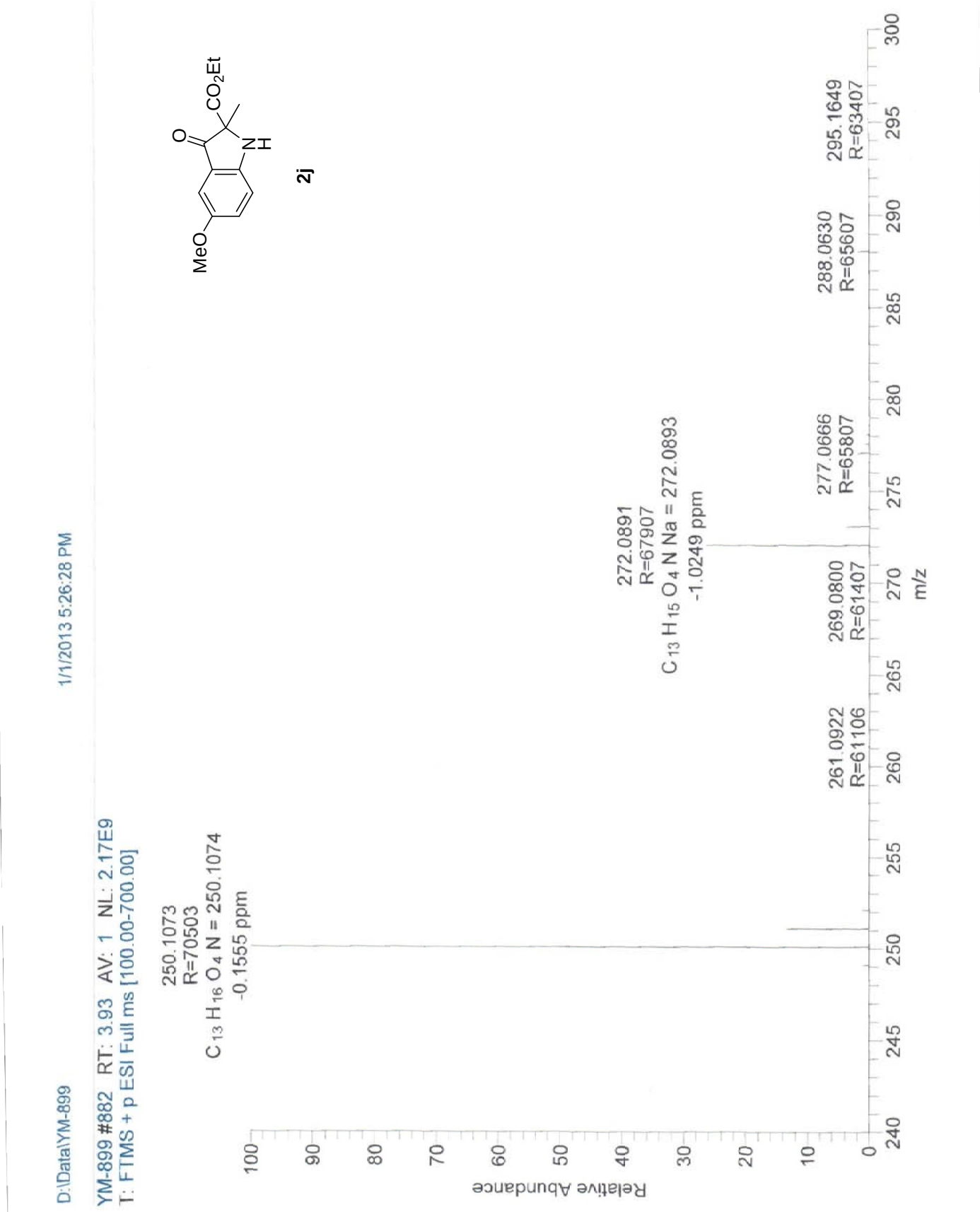
m/z

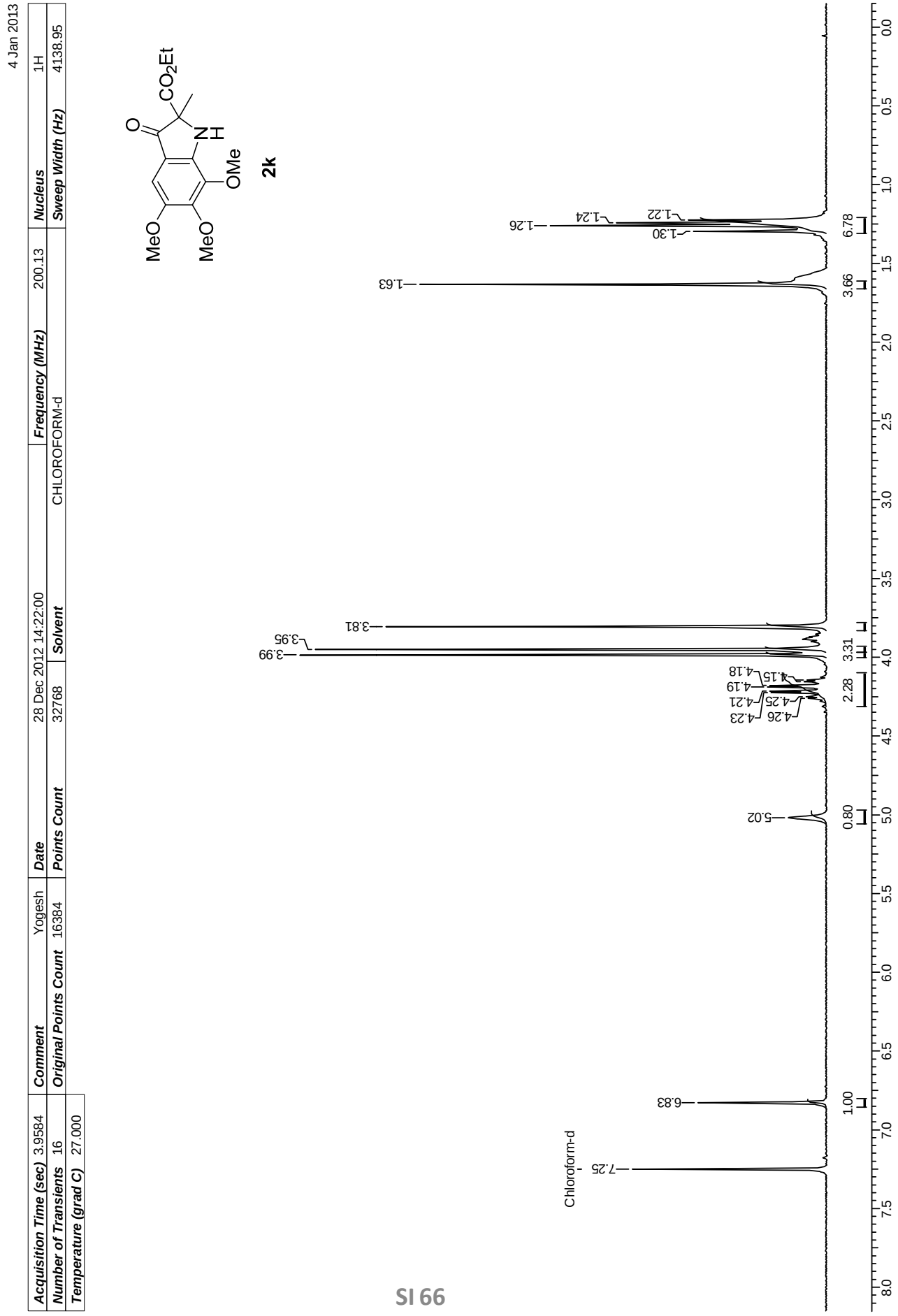

4 Jan 2013
Acquisition Time (sec) 2.1838
Comment
Date 31 Dec 2012 09:01:29
Frequency (MHz) 399.78
Nucleus 1H
Number of Transients 16
Original Points Count 13107
Points Count 13107
Solvent CHLOROFORM-d
Sweep Width (Hz) 6001.85
Temperature (grad C) 20.200
yogesh G
MeO
O
CO2Et
N
H
2j
3.78
1.65
1.28
1.30
1.27
1.26
3.77
4.16
4.18
4.21
4.19
4.23
4.20
4.23
4.25
4.26
4.27
4.28
4.30
4.97
7.27
7.20
7.19
7.17
7.05
6.96
6.94
7.20
7.19
7.18
7.17
7.05
7.04
6.96
6.94
3.82
3.10
3.16
2.16
0.94
0.99
0.97
0.99
0.93
0.97

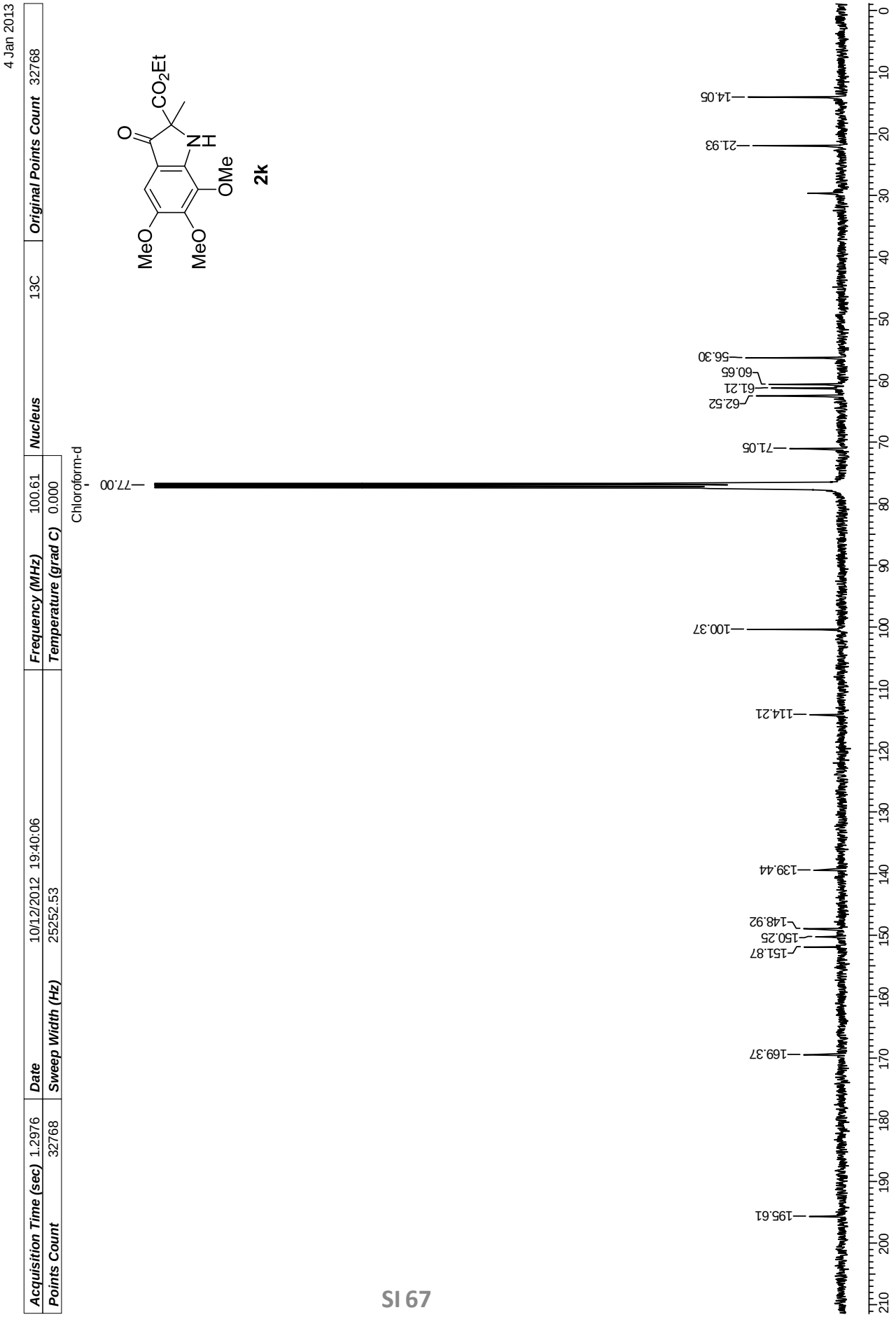

4 Jan 2013
Acquisition Time (sec) 1.0434
Comment
Date 31 Dec 2012 10:32:47
Frequency (MHz) 100.53
Nucleus 13C
Number of Transients 1308
Original Points Count 26214
Points Count 26214
Solvent CHLOROFORM-d
Sweep Width (Hz) 25124.29
Temperature (grad C) 20.700
yogesh G
CO2Et
O
N
H
MeO
2j
14.05
22.05
55.76
62.60
71.50
76.68
77.00
77.31
104.89
115.12
120.20
128.12
154.36
156.88
169.42
196.83

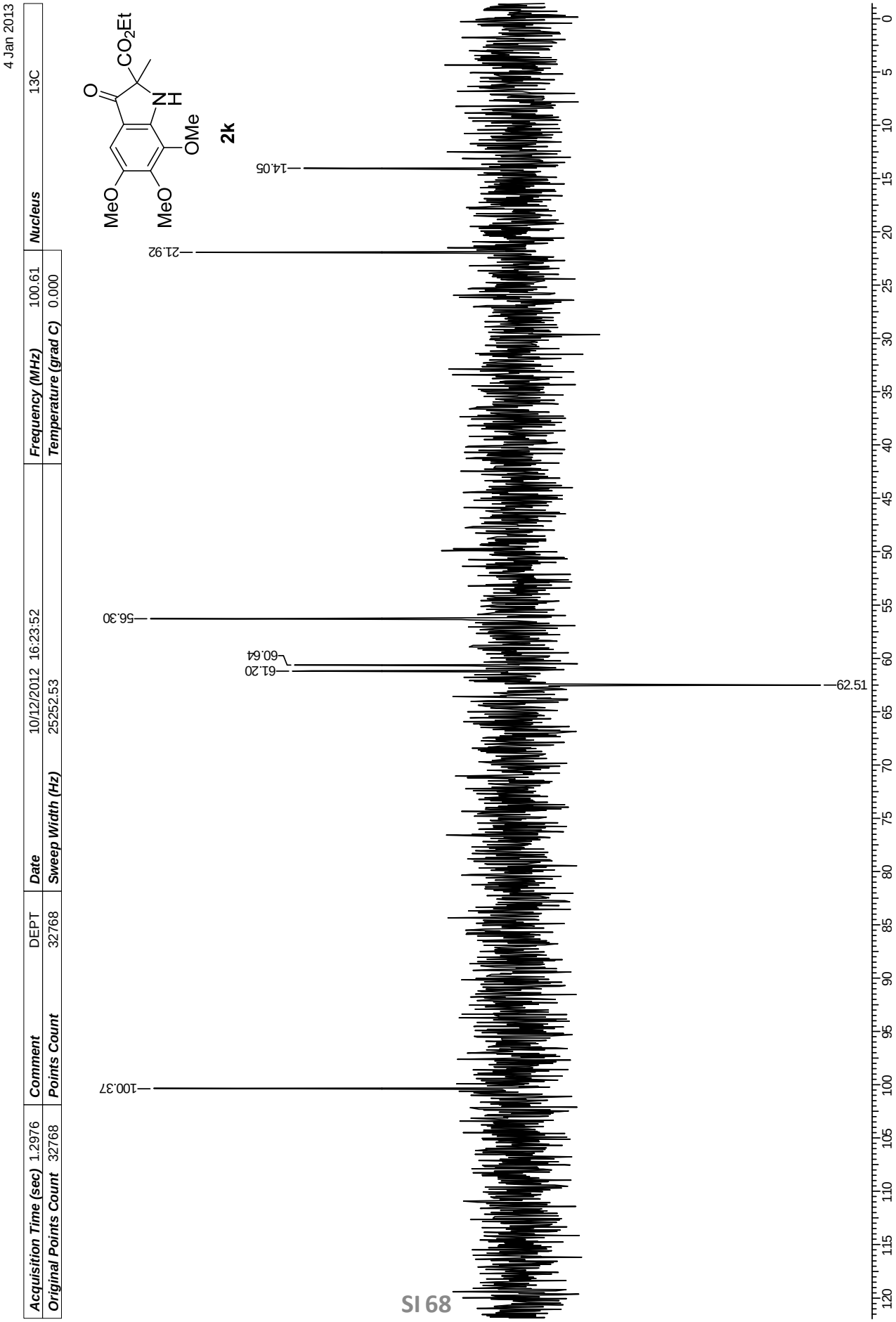

4 Jan 2013
Acquisition Time (sec) 1.0434
Comment
yogesh G
Date
31 Dec 2012 10:32:47
Frequency (MHz) 100.53
Nucleus 13C
Number of Transients 1000
Original Points Count 26214
Points Count 26214
Solvent CHLOROFORM-d
Sweep Width (Hz) 25124.29
Temperature (grad C) 19.600
14.05
22.04
55.74
62.60
104.87
115.11
128.12
MeO
O
NH
CO2Et
2j

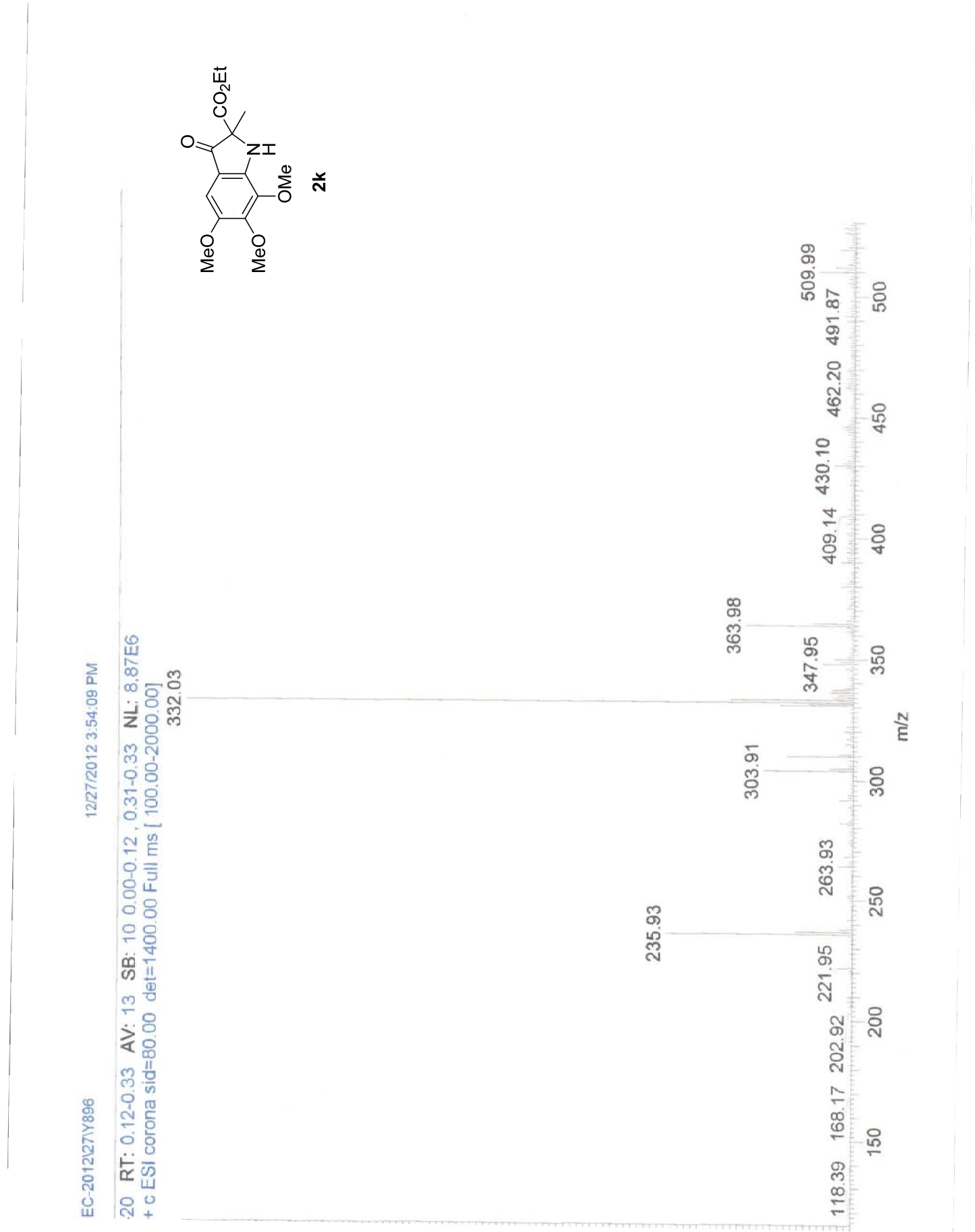

MeO
O
N
H
CO2Et
2j
EC-2012\27\Y899
12/27/2012 3:57:59 PM
23 RT: 0.10-0.38 AV: 17 SB: 15 0.02-0.10 , 0.36-0.50 NL: 1.58E6
+ c ESI corona sid=80.00 det=1400.00 Full ms [100.00-2000.00]
271.97
272.82
291.03
304.03
301.02
305.05
327.13
333.09
343.06
258.95
260.00
274.93
257.80
238.99
221.97
212.99
m/z

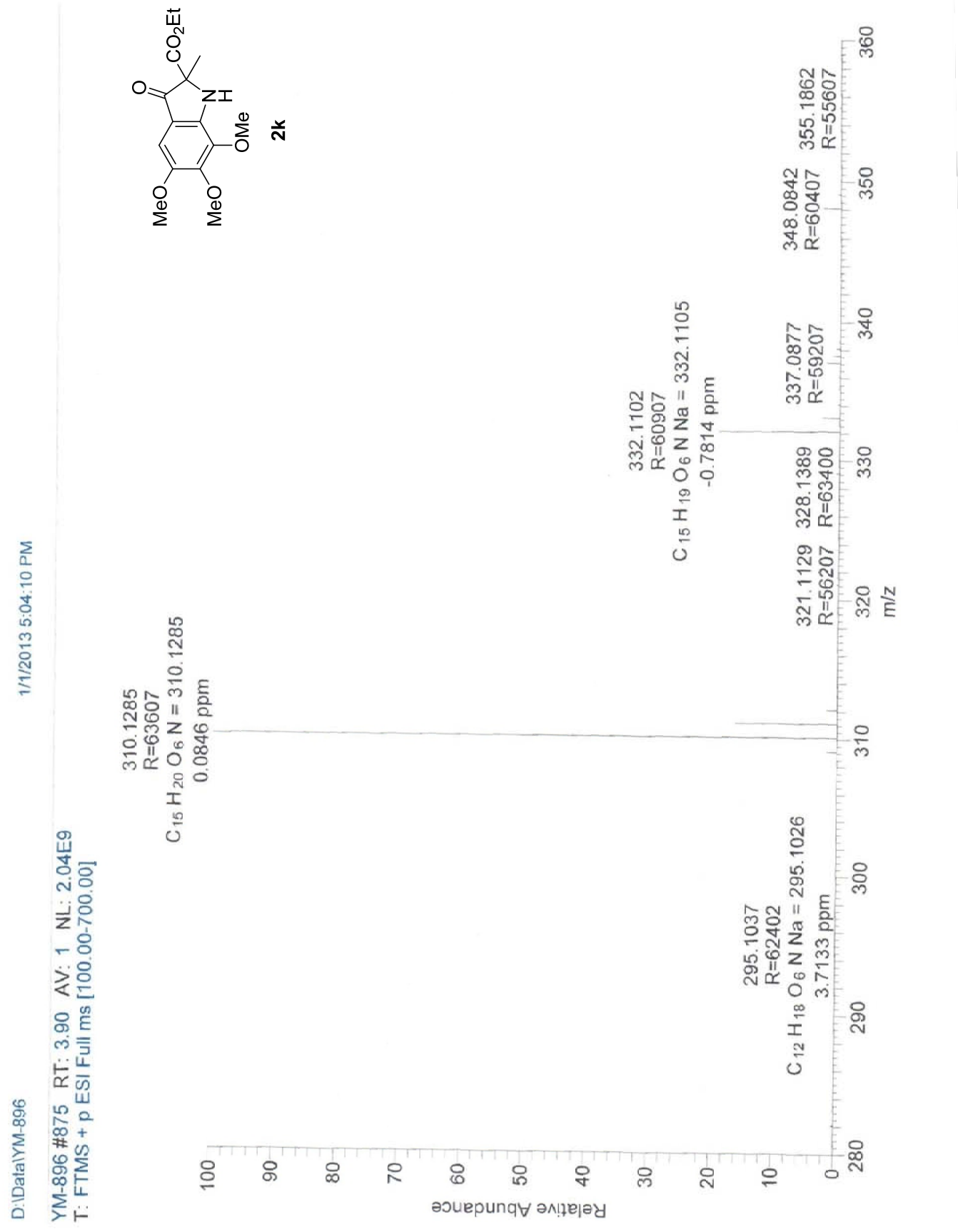

D:\Data\YM-899
1/1/2013 5:26:28 PM
YM-899 #882 RT: 3.93 AV: 1 NL: 2.17E9
T: FTMS + p ESI Full ms [100.00-700.00]
250.1073
R=70503
C 13 H 16 O 4 N = 250.1074
-0.1555 ppm
261.0922
R=61106
269.0800
R=61407
272.0891
R=67907
C 13 H 15 O 4 N Na = 272.0893
-1.0249 ppm
277.0666
R=65807
288.0630
R=65607
295.1649
R=63407
Relative Abundance
m/z
MeO
CO2Et
O
N
H
2j

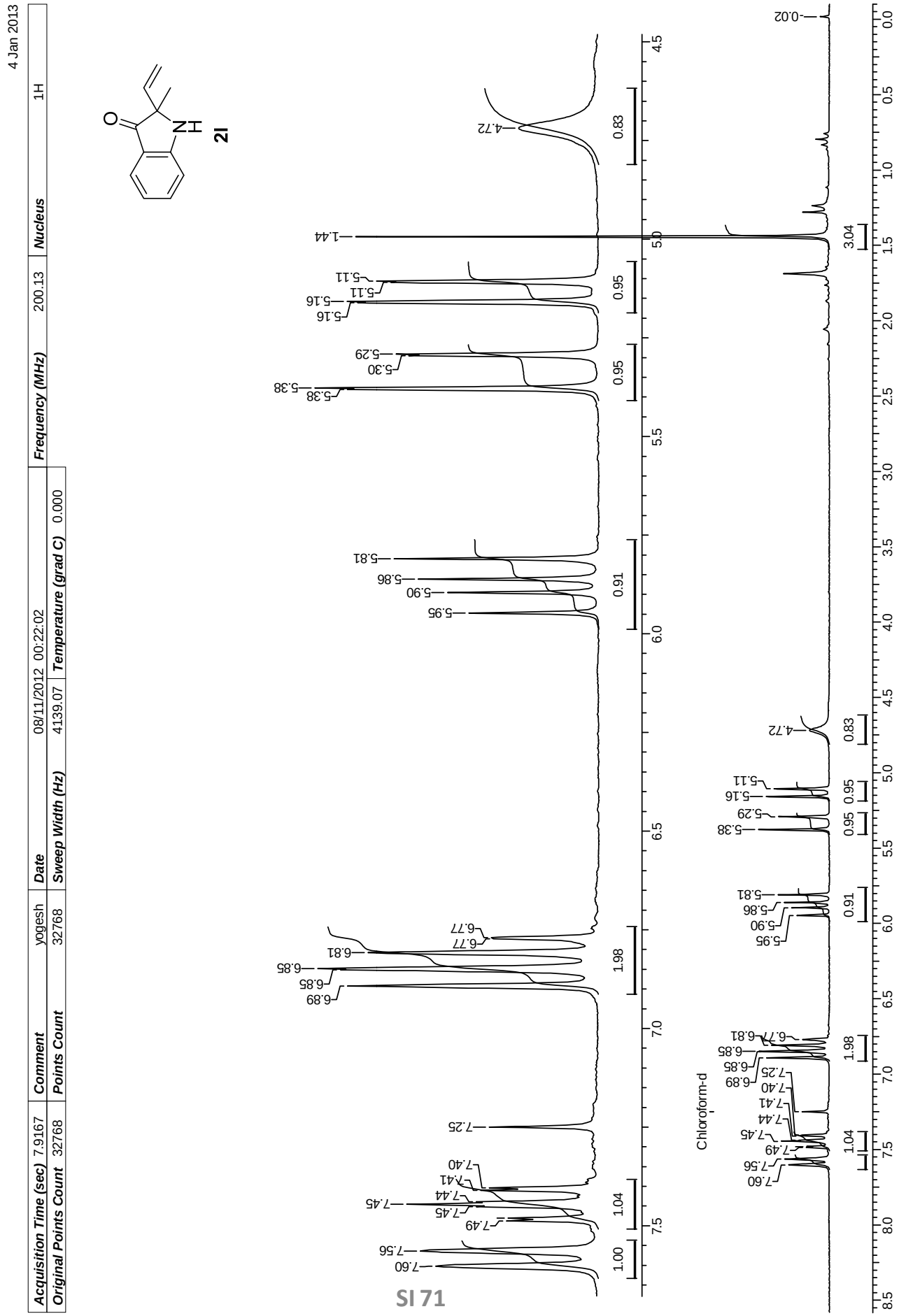

4 Jan 2013
Acquisition Time (sec) 3.9584
Comment
Frequency (MHz) 200.13
Nucleus 1H
Number of Transients 16
Original Points Count 16384
Yogesh
Date 28 Dec 2012 14:22:00
Points Count 32768
Solvent CHLOROFORM-d
Sweep Width (Hz) 4138.95
Temperature (grad C) 27.000
2k
MeO
MeO
OMe
CO2Et
O
NH
Chloroform-d
7.25
6.83
5.02
4.26
4.23
4.25
4.21
4.19
4.15
4.18
3.99
3.95
3.81
1.63
1.26
1.24
1.22
1.30
1.00
2.28
0.80
3.31
3.66
6.78

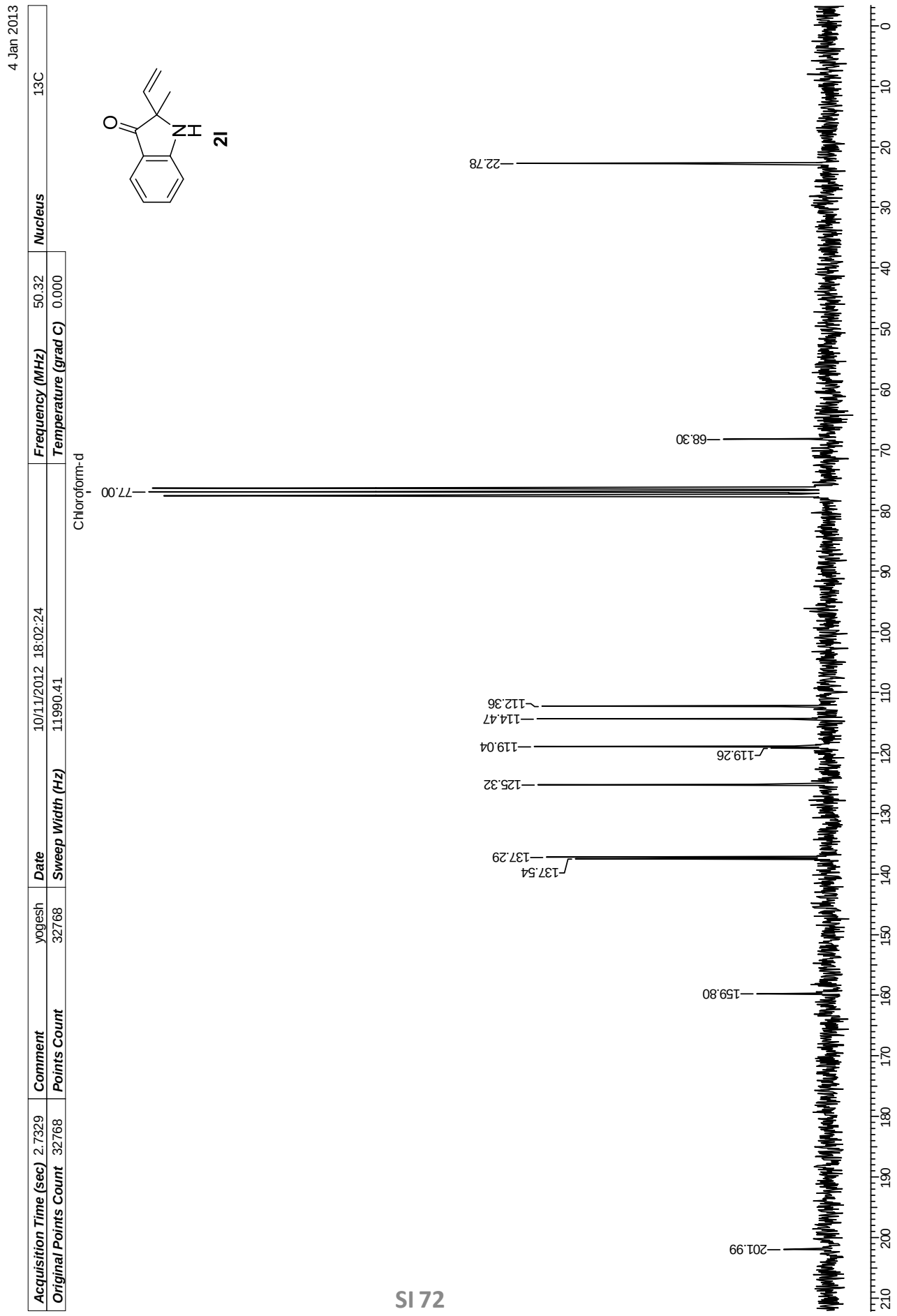

4 Jan 2013
Acquisition Time (sec) 1.2976
Points Count 32768
Date 10/12/2012 19:40:06
Sweep Width (Hz) 25252.53
Frequency (MHz) 100.61
Temperature (grad C) 0.000
Nucleus 13C
Original Points Count 32768
Chloroform-d
2k
MeO
MeO
OMe
O
NH
CO2Et
77.00
14.05
21.93
56.30
60.65
61.21
62.52
71.05
100.37
114.21
139.44
148.92
150.25
151.87
169.37
195.61

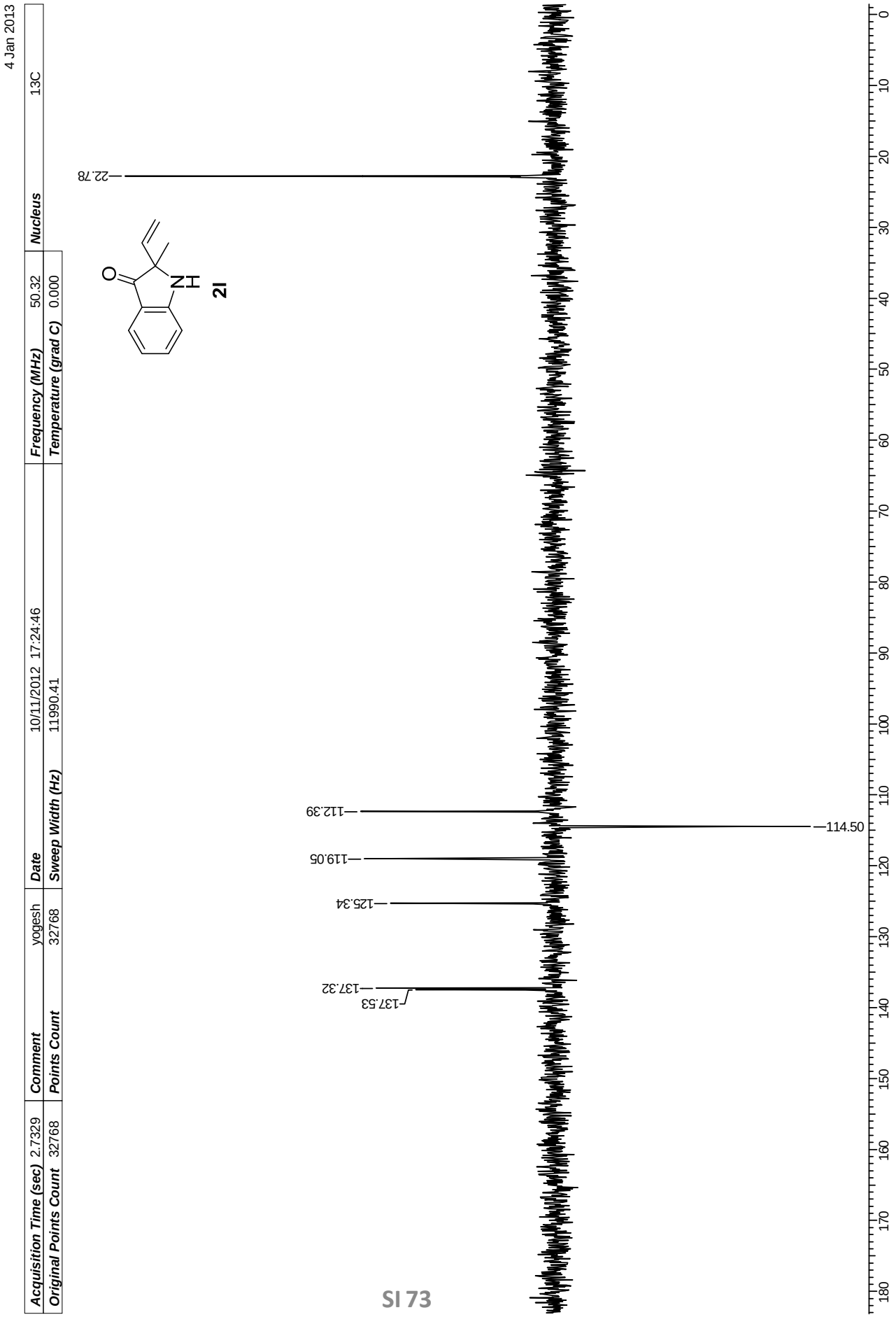

4 Jan 2013
Acquisition Time (sec) 1.2976
Original Points Count 32768
Comment
Points Count
DEPT
32768
Date
Sweep Width (Hz)
10/12/2012 16:23:52
25252.53
Frequency (MHz) 100.61
Temperature (grad C) 0.000
Nucleus 13C
2k
MeO
MeO
OMe
CO2Et
O
NH
14.05
21.92
56.30
60.64
61.20
62.51
100.37

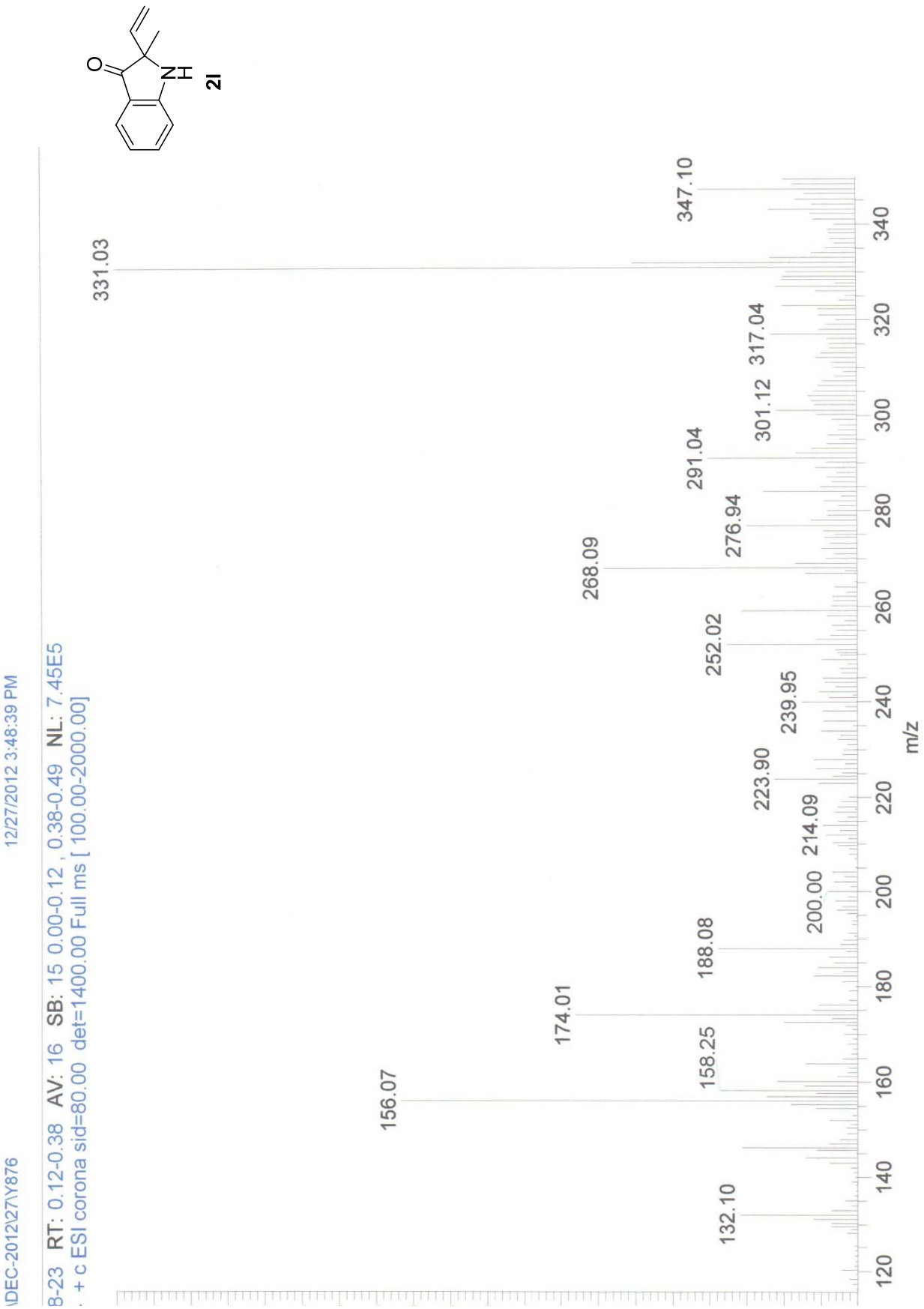

EC-2012\27\Y896
12/27/2012 3:54:09 PM
:20 RT: 0.12-0.33 AV: 13 SB: 10 0.00-0.12 , 0.31-0.33 NL: 8.87E6
+ c ESI corona sid=80.00 det=1400.00 Full ms [100.00-2000.00]
332.03
363.98
347.95
303.91
235.93
263.93
221.95
409.14
430.10
462.20
491.87
509.99
118.39
168.17
202.92
150
200
250
300
350
400
450
500
m/z
MeO
MeO
OMe
O
N
H
CO2Et
2k

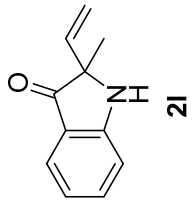

D:\Data\YM-896
1/1/2013 5:04:10 PM
YM-896 #875 RT: 3.90 AV: 1 NL: 2.04E9
T: FTMS + p ESI Full ms [100.00-700.00]
310.1285
R=63607
C15 H20 O6 N = 310.1285
0.0846 ppm
295.1037
R=62402
C12 H18 O6 N Na = 295.1026
3.7133 ppm
321.1129
R=56207
328.1389
R=63400
332.1102
R=60907
C15 H19 O6 N Na = 332.1105
-0.7814 ppm
337.0877
R=59207
348.0842
R=60407
355.1862
R=55607
Relative Abundance
m/z
MeO
MeO
OMe
O
CO2Et
N
H
2k


4 Jan 2013
Acquisition Time (sec) 7.9167
Original Points Count 32768
Comment
Points Count
yogesh
32768
Date
Sweep Width (Hz)
08/11/2012 00:22:02
4139.07
Temperature (grad C) 0.000
Frequency (MHz) 200.13
Nucleus 1H
2l
Chloroform-d


4 Jan 2013
Acquisition Time (sec) 2.7329
Original Points Count 32768
Comment
Points Count
yogesh
32768
Date
Sweep Width (Hz)
10/11/2012 18:02:24
11990.41
Frequency (MHz)
Temperature (grad C)
50.32
0.000
Nucleus
13C
Chloroform-d
2l
77.00
22.78
68.30
112.36
114.47
119.04
119.26
125.32
137.29
137.54
159.80
201.99


4 Jan 2013
Acquisition Time (sec) 2.7329
Original Points Count 32768
Comment
Points Count
yogesh
32768
Date
Sweep Width (Hz)
10/11/2012 17:24:46
11990.41
Frequency (MHz) 50.32
Temperature (grad C) 0.000
Nucleus 13C
22.78
112.39
114.50
119.05
125.34
137.32
137.53
2I


2l
\DEC-2012\27\Y876
12/27/2012 3:48:39 PM
B-23 RT: 0.12-0.38 AV: 16 SB: 15 0.00-0.12 , 0.38-0.49 NL: 7.45E5
. + c ESI corona sid=80.00 det=1400.00 Full ms [100.00-2000.00]
331.03
347.10
317.04
301.12
291.04
276.94
268.09
252.02
239.95
223.90
214.09
200.00
188.08
174.01
158.25
156.07
132.10
120
140
160
180
200
220
240
260
280
300
320
340
m/z



2l

D:\Data\YM-876 1/1/2013 4:30:39 PM

YM-876 #893 RT: 3.98 AV: 1 NL: 1.94E9
T: FTMS + p ESI Full ms [100.00-700.00]

174.0913
R=85307
$C_{11}H_{12}ONa$ = 174.0913
-0.4466 ppm

196.0732
R=80207
$C_{11}H_{11}ONNa$ = 196.0733
-0.6865 ppm

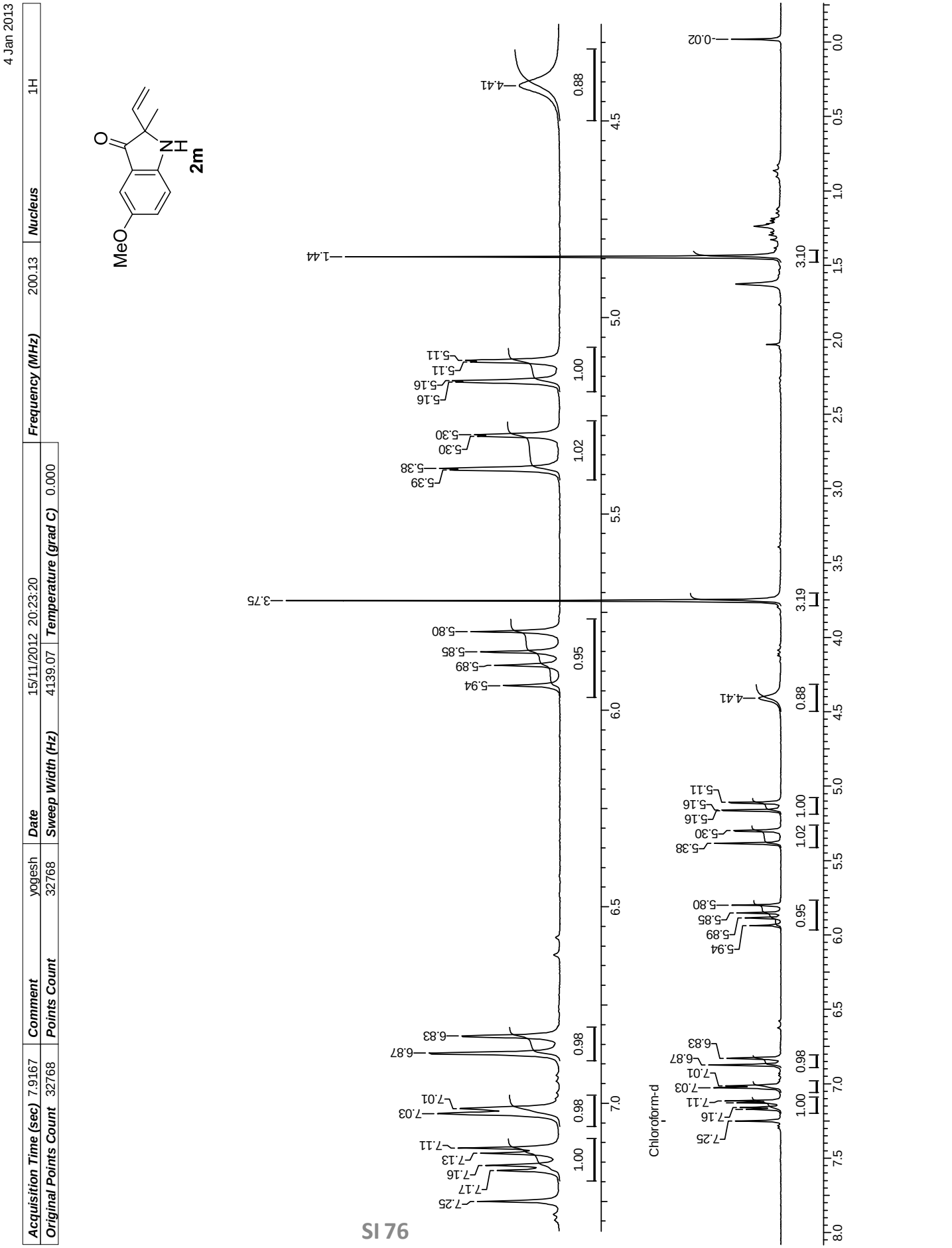
164.9845
R=86802

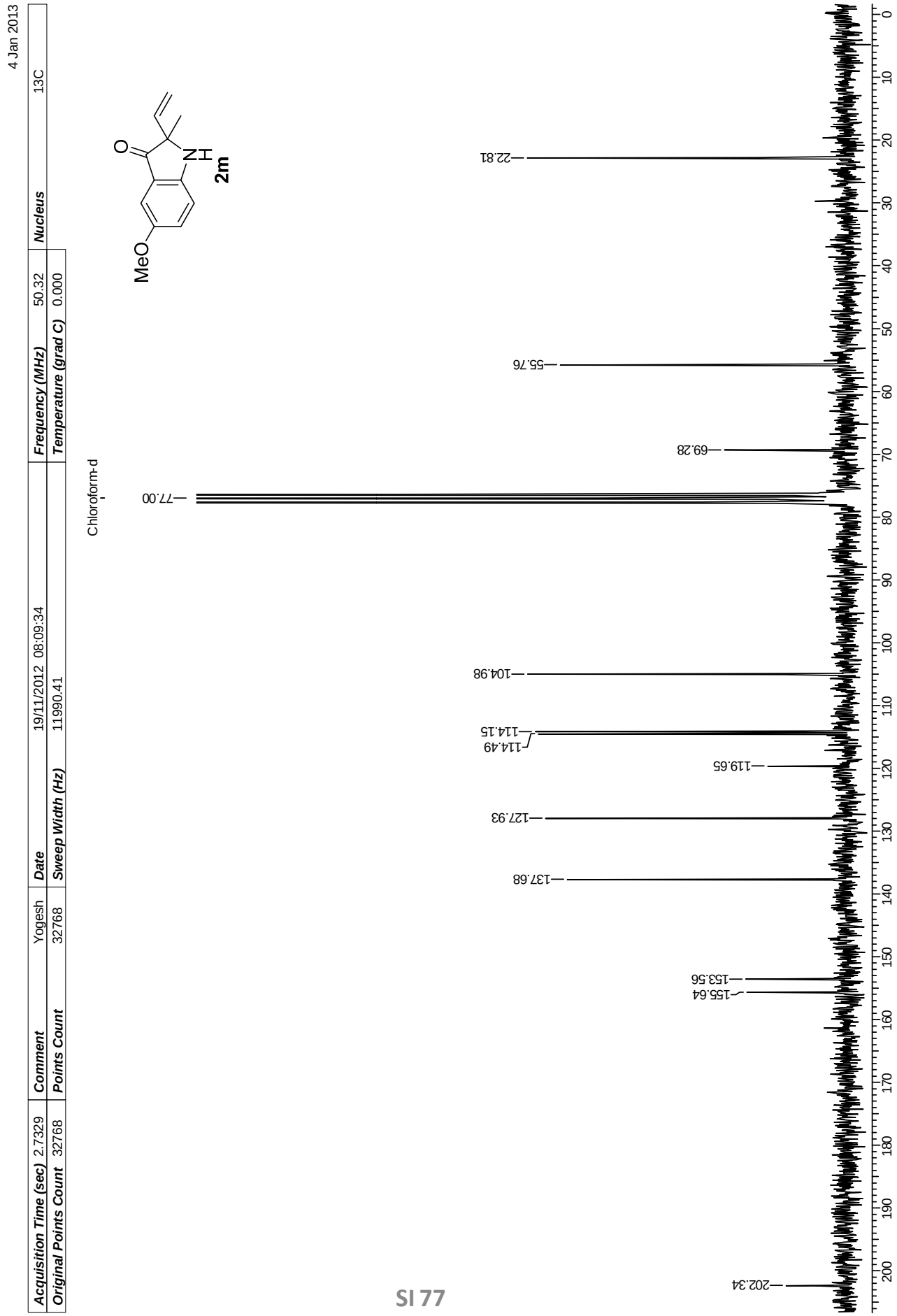
214.0837
R=74007

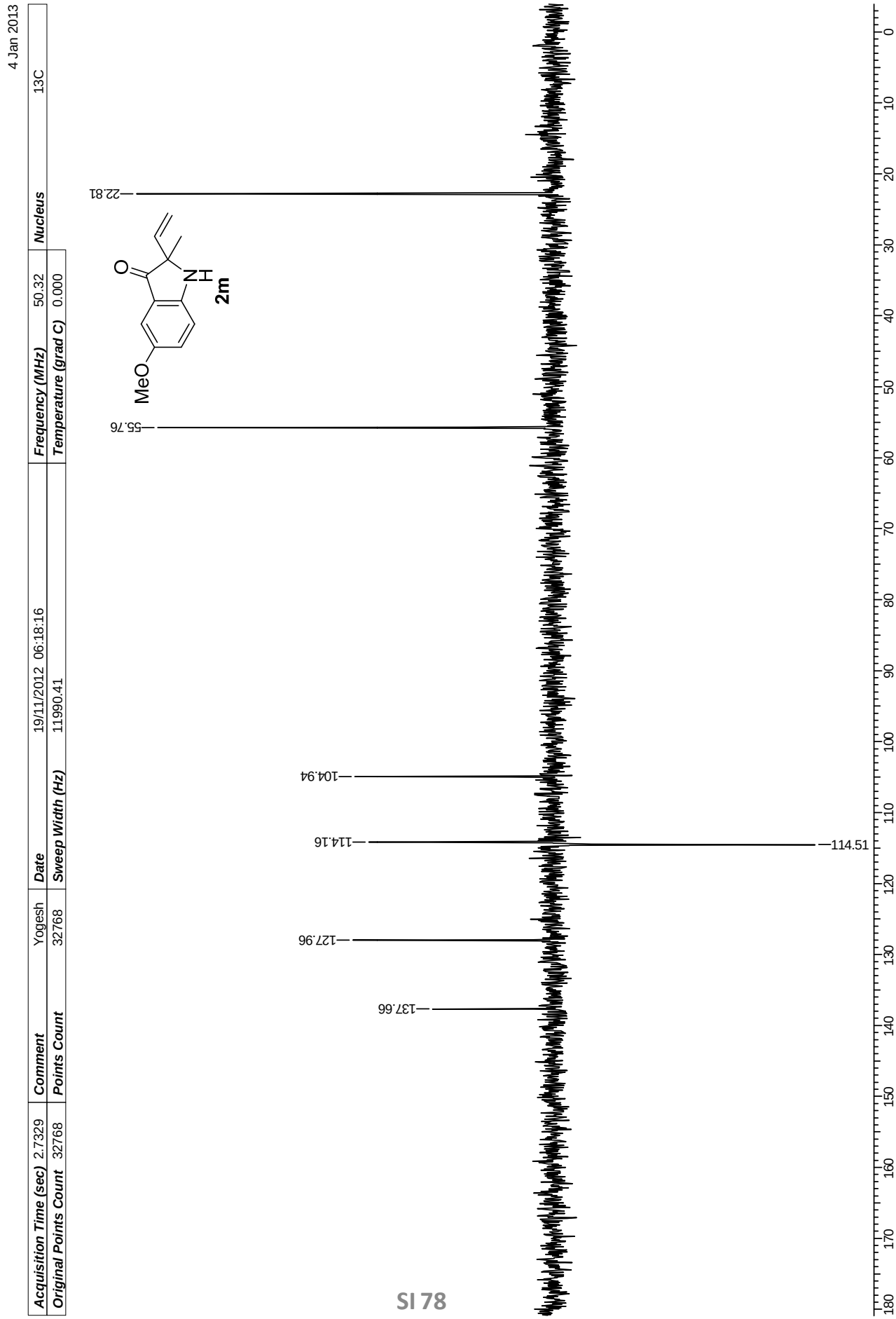
226.0836
R=71007

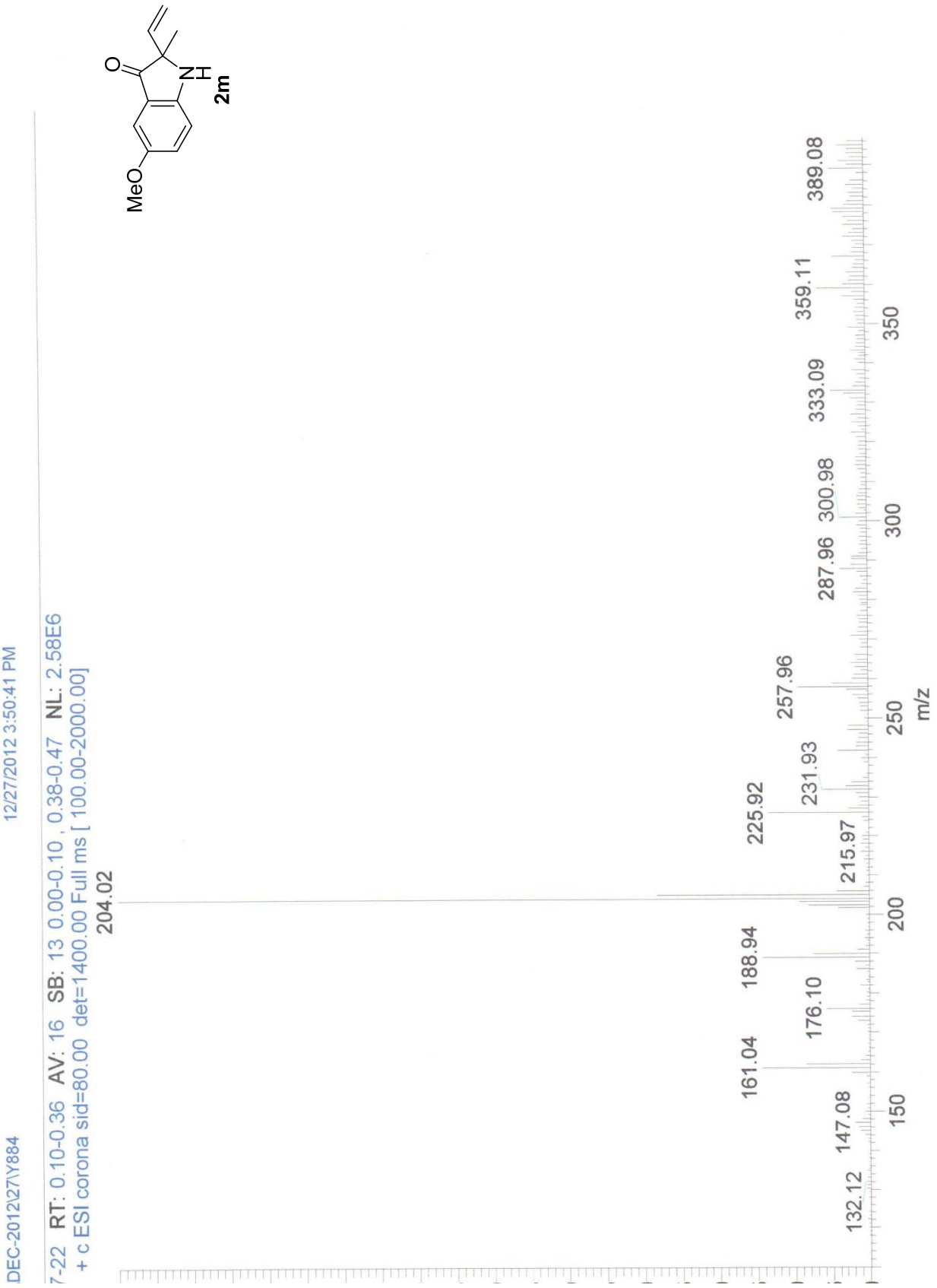
Relative Abundance

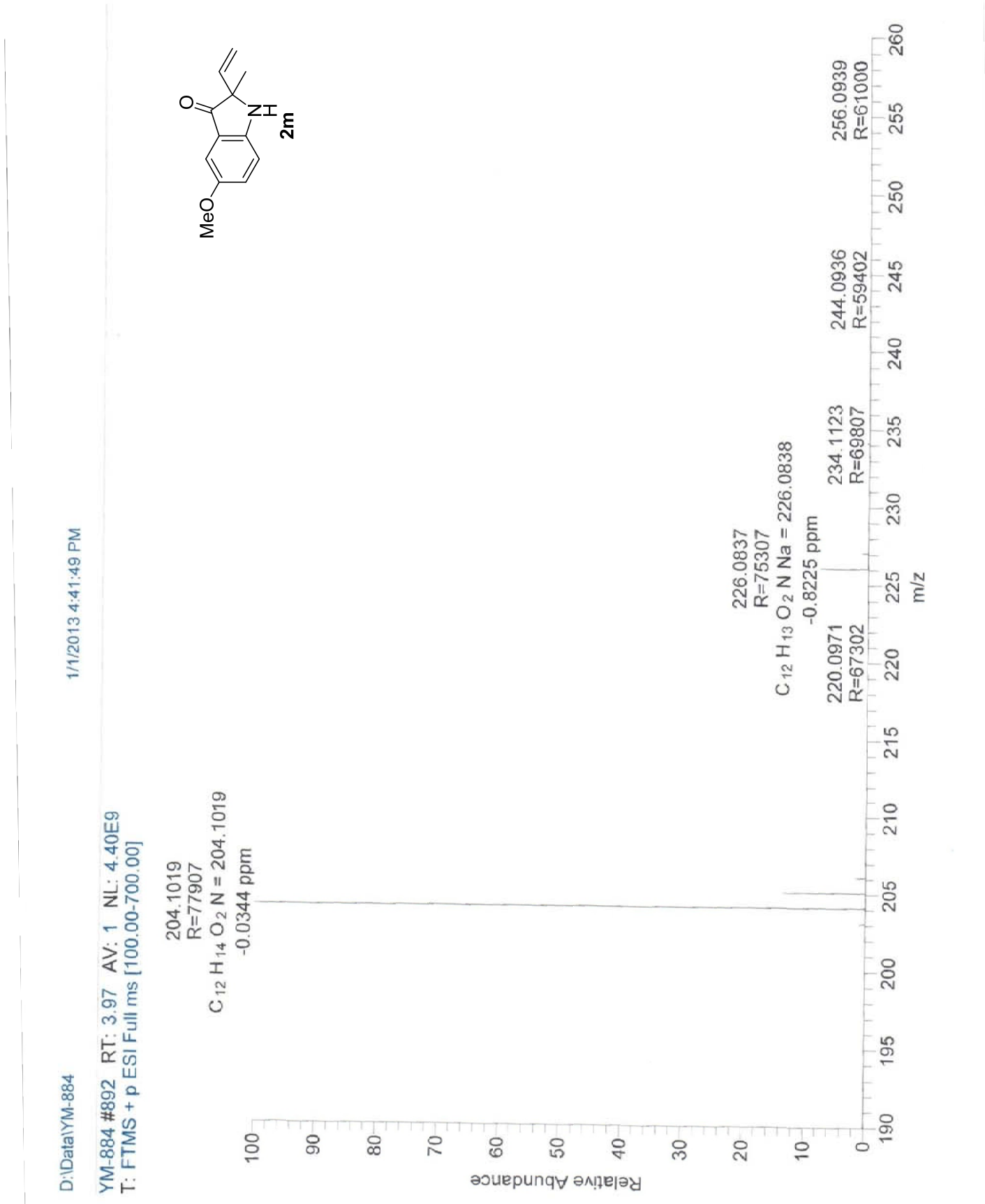
m/z

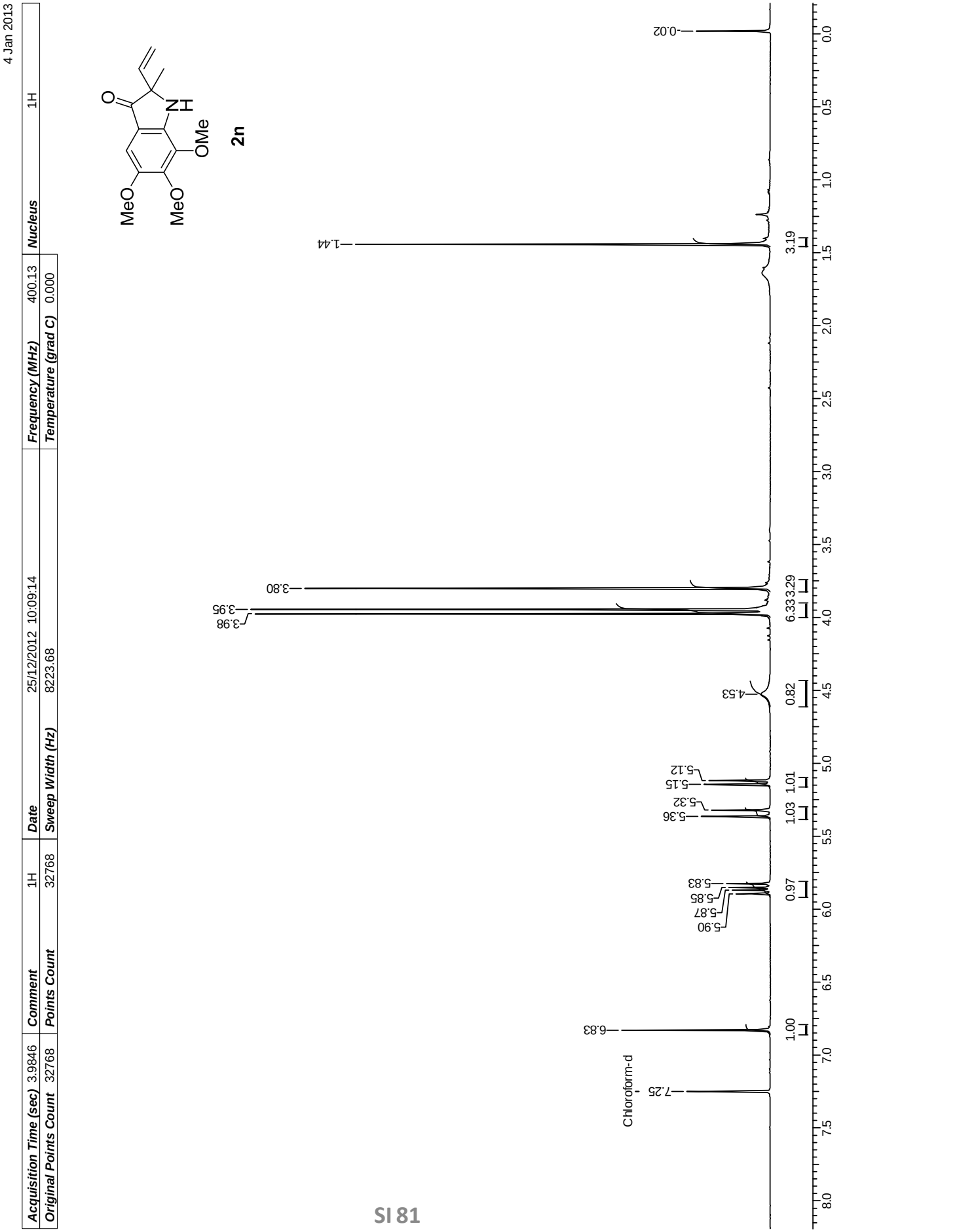

4 Jan 2013
Acquisition Time (sec) 7.9167
Original Points Count 32768
Comment
Points Count
yogesh
32768
Date
Sweep Width (Hz)
15/11/2012 20:23:20
4139.07
Temperature (grad C) 0.000
Frequency (MHz) 200.13
Nucleus 1H
MeO
NH
O
2m
Chloroform-d

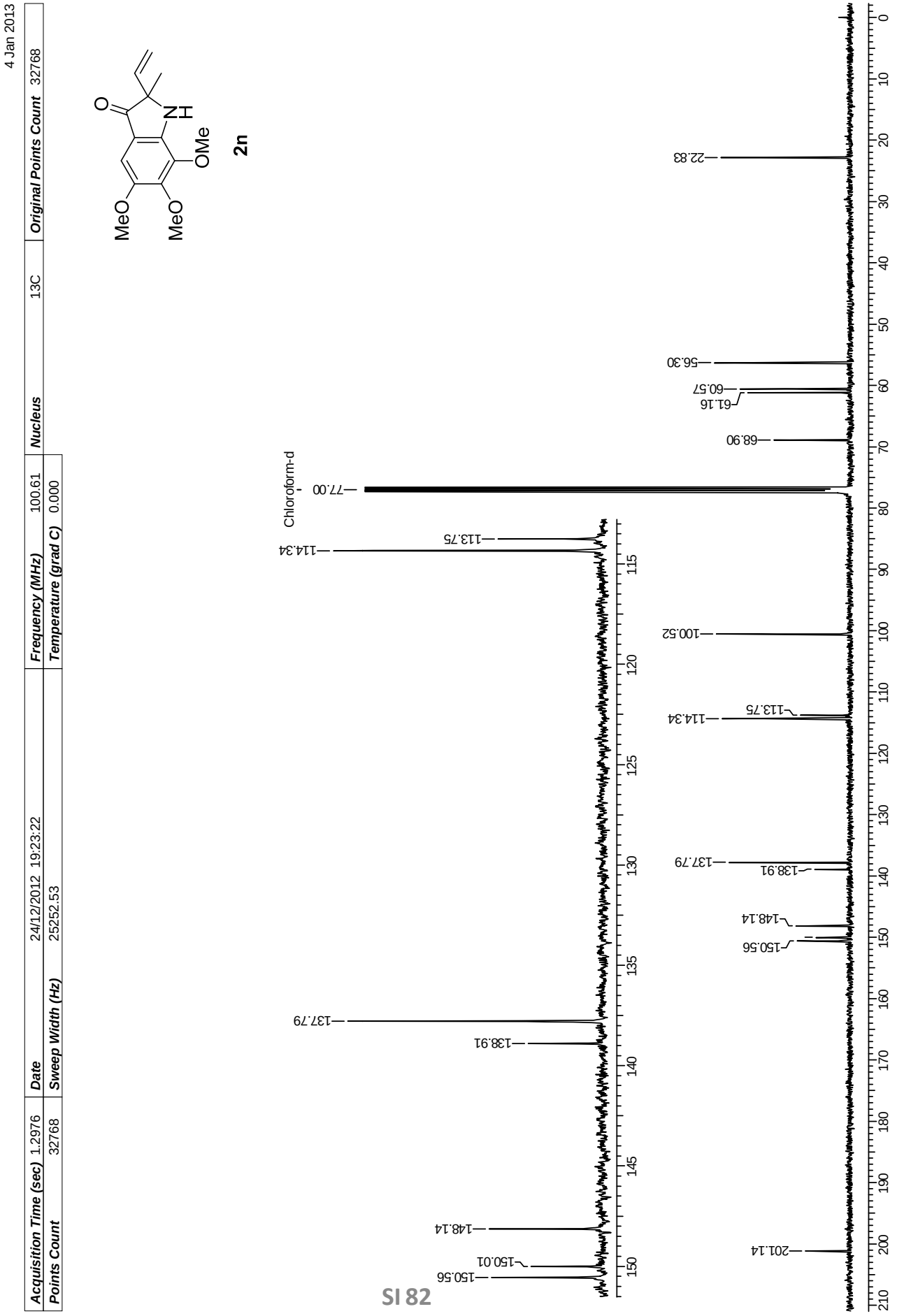

4 Jan 2013
Acquisition Time (sec) 2.7329
Original Points Count 32768
Comment
Points Count
Yogesh
32768
Date
Sweep Width (Hz)
19/11/2012 08:09:34
11990.41
Frequency (MHz)
Temperature (grad C)
50.32
0.000
Nucleus
13C
2m
MeO
NH
O
Chloroform-d
77.00
22.81
55.76
69.28
104.98
114.49
114.15
119.65
127.93
137.68
153.56
155.64
202.34

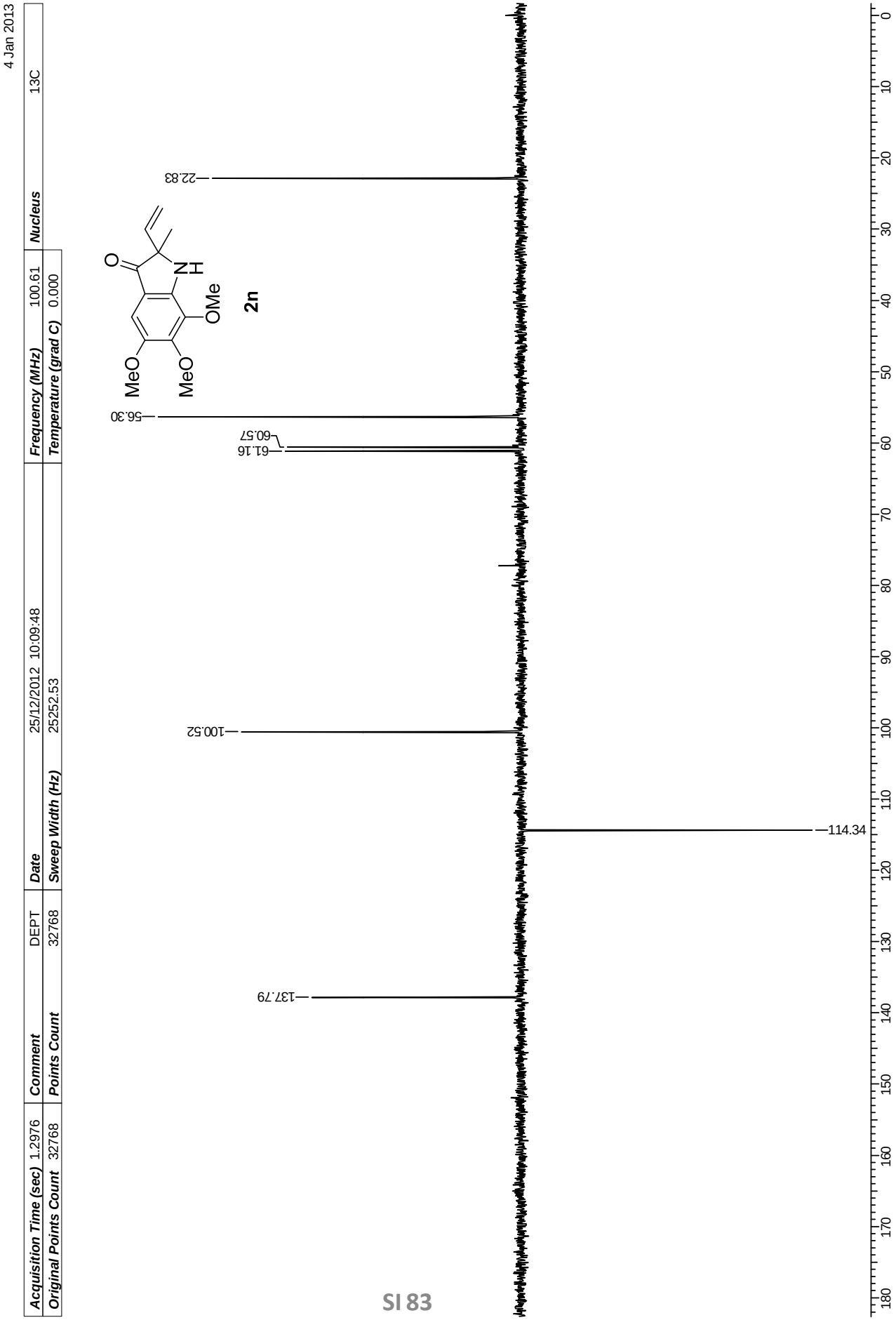

4 Jan 2013
Acquisition Time (sec) 2.7329
Original Points Count 32768
Comment
Points Count
Yogesh
32768
Date
Sweep Width (Hz)
19/11/2012 06:18:16
11990.41
Frequency (MHz) 50.32
Temperature (grad C) 0.000
Nucleus 13C
22.81
55.76
104.94
114.16
114.51
127.96
137.66
O
NH
MeO
2m
0
10
20
30
40
50
60
70
80
90
100
110
120
130
140
150
160
170
180

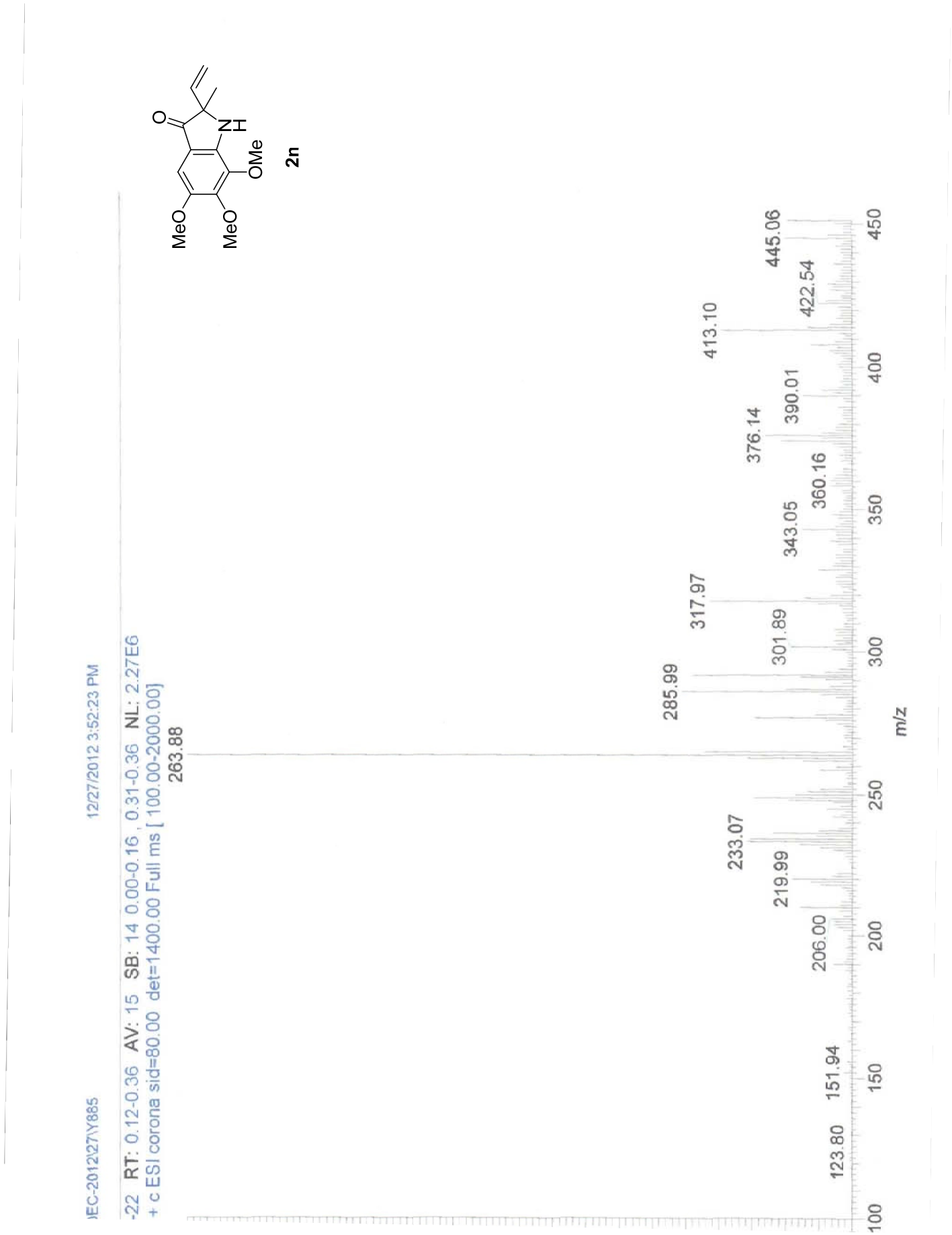

2m
MeO
O
N
H
DEC-2012\27\Y884
12/27/2012 3:50:41 PM
7-22 RT: 0.10-0.36 AV: 16 SB: 13 0.00-0.10 , 0.38-0.47 NL: 2.58E6
+ c ESI corona sid=80.00 det=1400.00 Full ms [100.00-2000.00]
204.02
225.92
257.96
231.93
215.97
161.04
188.94
176.10
147.08
132.12
287.96
300.98
333.09
359.11
389.08
150
200
250
300
350
m/z

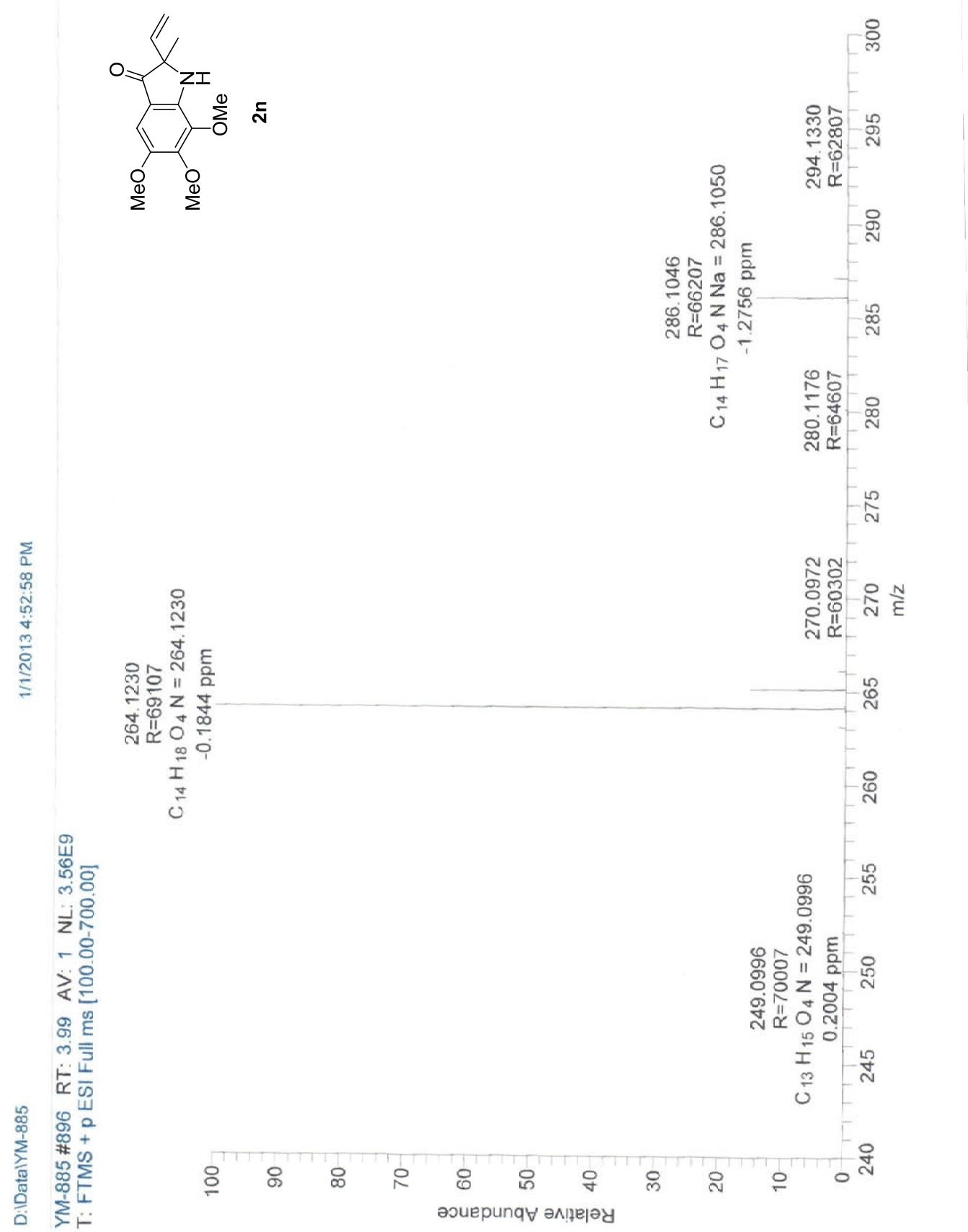

D:\Data\YM-884
1/1/2013 4:41:49 PM
YM-884 #892 RT: 3.97 AV: 1 NL: 4.40E9
T: FTMS + p ESI Full ms [100.00-700.00]
204.1019
R=77907
C 12 H 14 O 2 N = 204.1019
-0.0344 ppm
220.0971
R=67302
226.0837
R=75307
C 12 H 13 O 2 N Na = 226.0838
-0.8225 ppm
234.1123
R=69807
244.0936
R=59402
256.0939
R=61000
Relative Abundance
m/z
MeO
2m

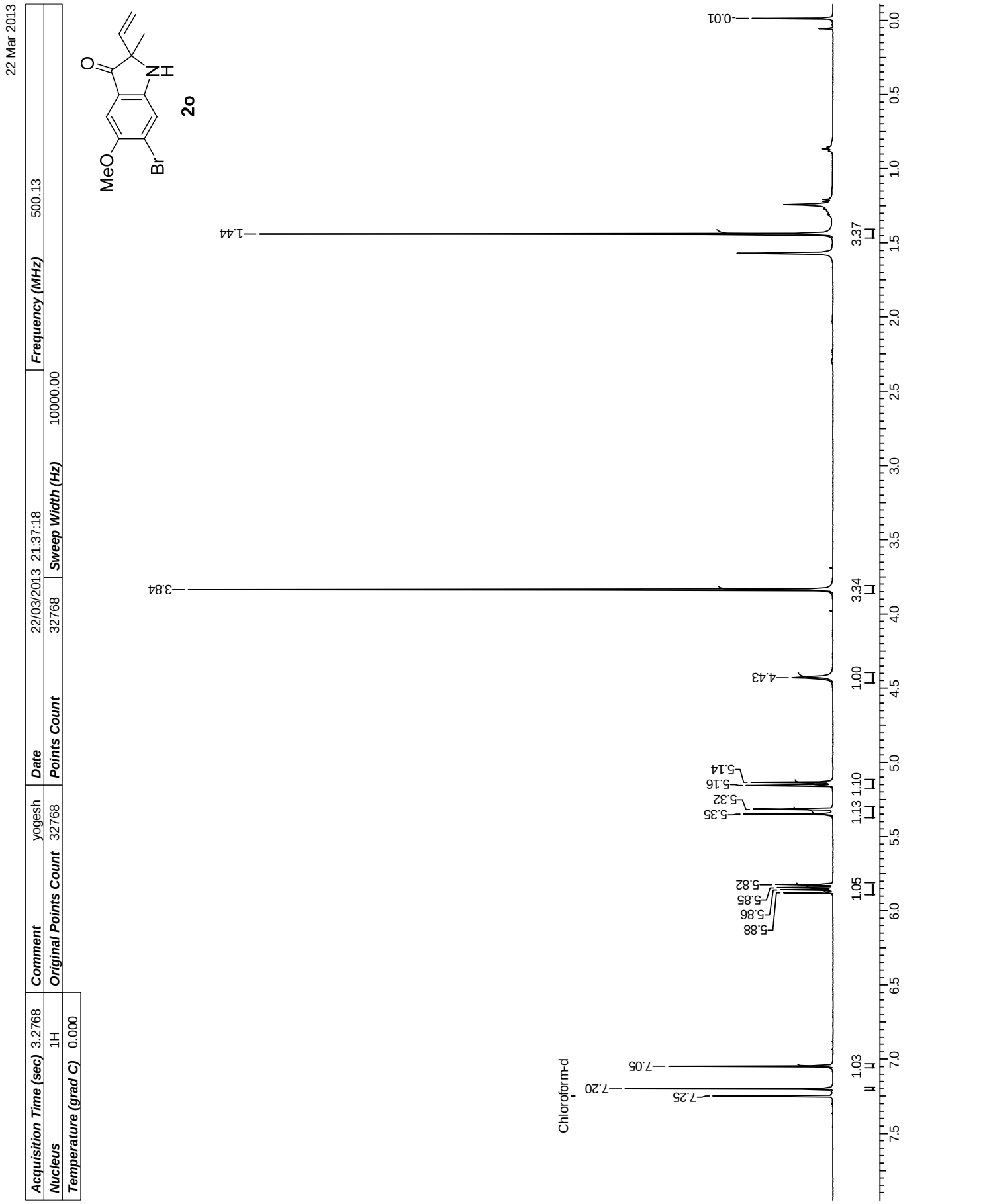

4 Jan 2013
Acquisition Time (sec) 3.9846
Original Points Count 32768
Comment
Points Count
1H
32768
Date
Sweep Width (Hz)
25/12/2012 10:09:14
8223.68
Frequency (MHz)
Temperature (grad C)
400.13
0.000
Nucleus
1H
MeO
MeO
OMe
O
N
H
2n
Chloroform-d
7.25
6.83
5.90
5.87
5.85
5.83
5.36
5.32
5.15
5.12
4.53
3.98
3.95
3.80
1.44
0.02
1.00
0.97
1.03
1.01
0.82
6.33
3.29
3.19

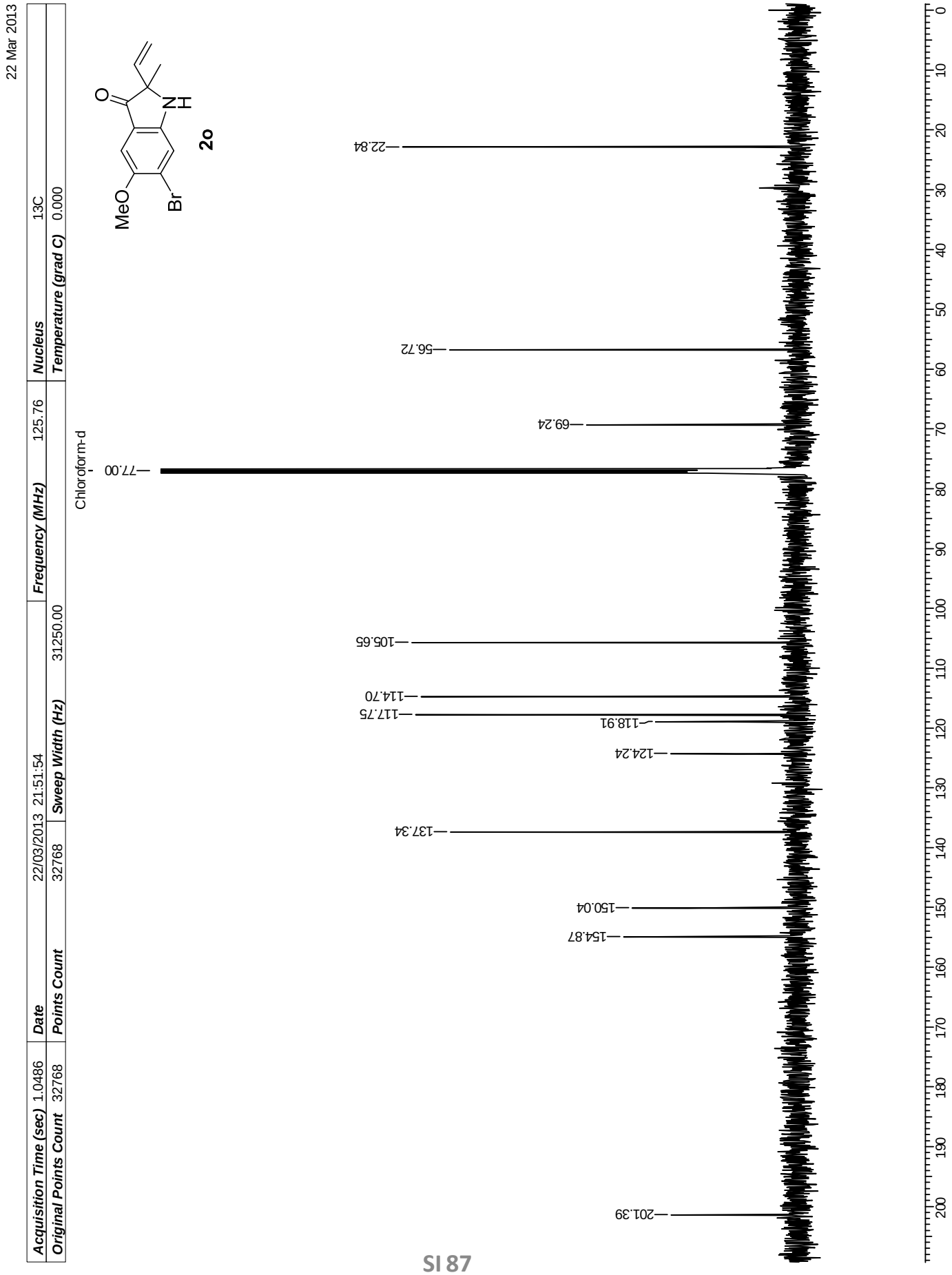

4 Jan 2013
Acquisition Time (sec) 1.2976
Points Count 32768
Date 24/12/2012 19:23:22
Sweep Width (Hz) 25252.53
Frequency (MHz) 100.61
Temperature (grad C) 0.000
Nucleus 13C
Original Points Count 32768
2n
MeO
MeO
OMe
O
NH
Chloroform-d
77.00
114.34
113.75
137.79
138.91
148.14
150.01
150.56
22.83
56.30
60.57
61.16
68.90
100.52
201.14

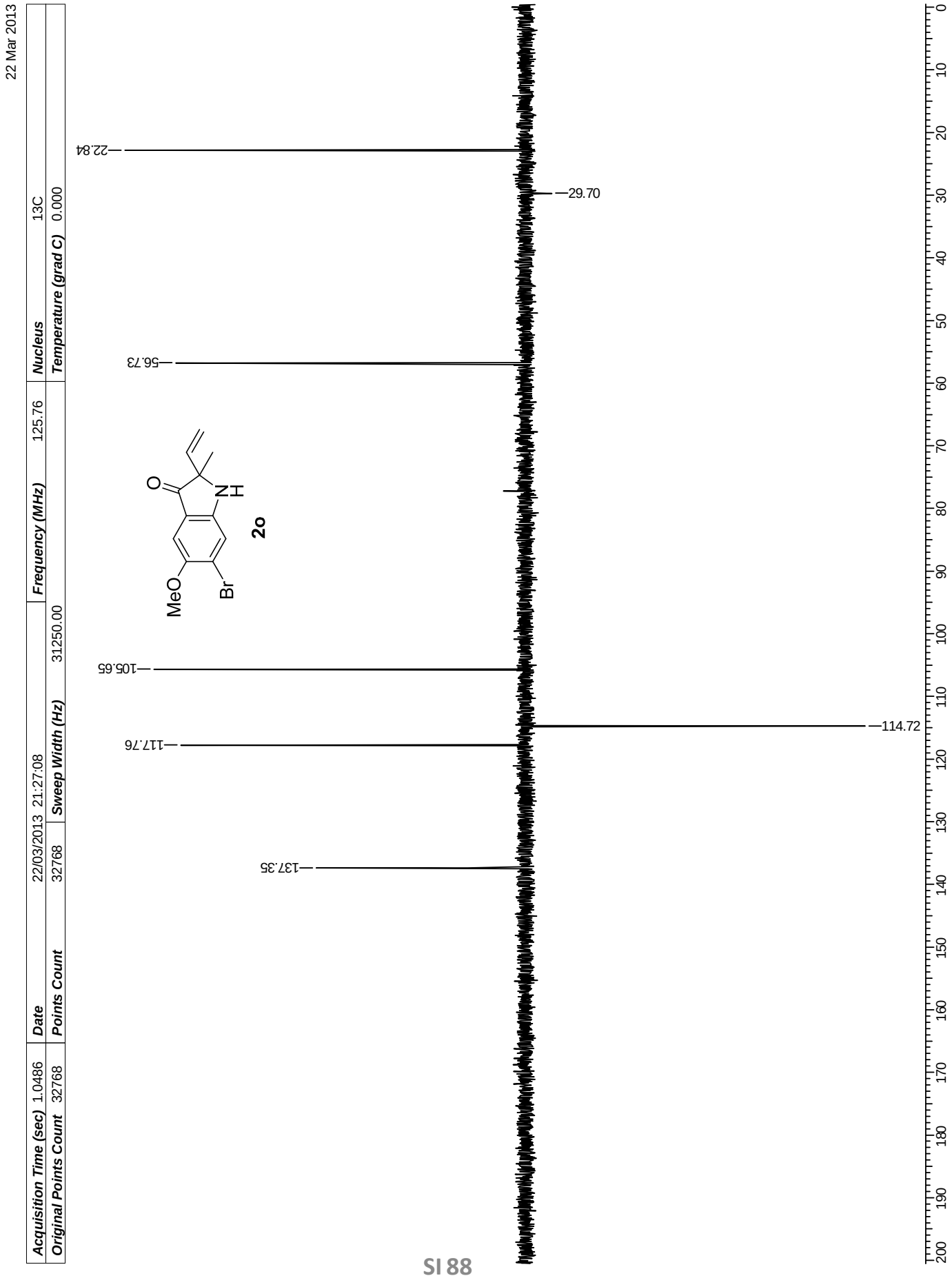

4 Jan 2013
Acquisition Time (sec) 1.2976
Original Points Count 32768
Comment
Points Count
DEPT
32768
Date
25/12/2012 10:09:48
Sweep Width (Hz)
25252.53
Frequency (MHz) 100.61
Temperature (grad C) 0.000
Nucleus
13C
22.83
56.30
60.57
61.16
100.52
114.34
137.79
MeO
MeO
OMe
O
NH
2n

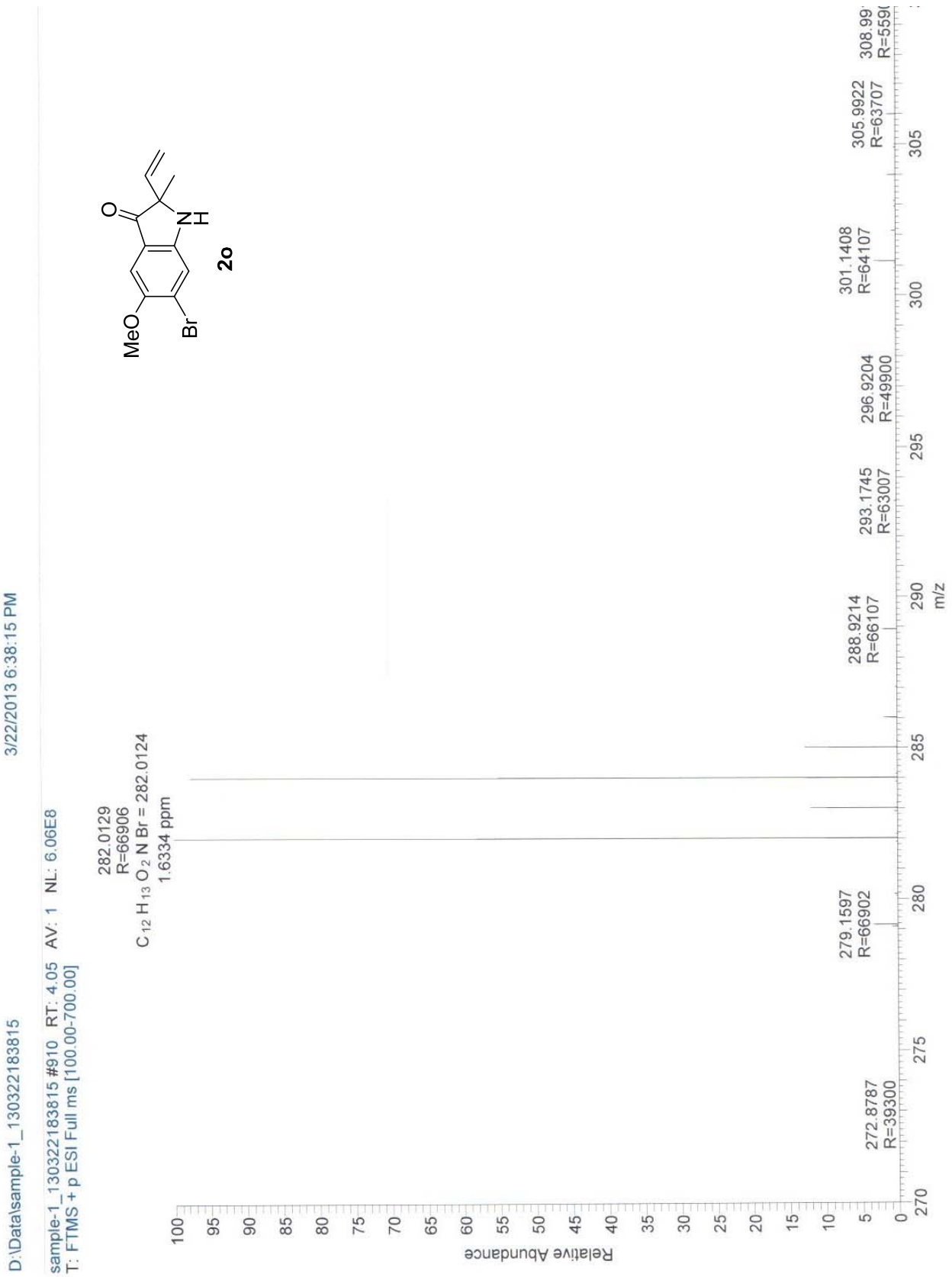

O
N
H
OMe
MeO
MeO
2n
)EC-2012\27\Y885
12/27/2012 3:52:23 PM
-22 RT: 0.12-0.36 AV: 15 SB: 14 0.00-0.16 , 0.31-0.36 NL: 2.27E6
+ c ESI corona sid=80.00 det=1400.00 Full ms [100.00-2000.00]
263.88
285.99
317.97
301.89
343.05
360.16
376.14
390.01
413.10
422.54
445.06
233.07
219.99
206.00
151.94
123.80
100
150
200
250
300
350
400
450
m/z


D:\Data\YM-885
1/1/2013 4:52:58 PM
YM-885 #896 RT: 3.99 AV: 1 NL: 3.56E9
T: FTMS + p ESI Full ms [100.00-700.00]
264.1230
R=69107
C 14 H 18 O 4 N = 264.1230
-0.1844 ppm
249.0996
R=70007
C 13 H 15 O 4 N = 249.0996
0.2004 ppm
270.0972
R=60302
280.1176
R=64607
286.1046
R=66207
C 14 H 17 O 4 N Na = 286.1050
-1.2756 ppm
294.1330
R=62807
Relative Abundance
m/z
MeO
MeO
OMe
NH
O
2n


22 Mar 2013
Acquisition Time (sec) 3.2768
Nucleus 1H
Temperature (grad C) 0.000
Comment yogesh
Original Points Count 32768
Date 22/03/2013 21:37:18
Points Count 32768
Sweep Width (Hz) 10000.00
Frequency (MHz) 500.13
MeO
Br
O
NH
2o
Chloroform-d
7.25
7.20
7.05
5.88
5.86
5.85
5.82
5.35
5.32
5.16
5.14
4.43
3.84
1.44
0.01
1.03
1.05
1.13
1.10
1.00
3.34
3.37


22 Mar 2013
Acquisition Time (sec) 1.0486
Original Points Count 32768
Date 22/03/2013 21:51:54
Points Count 32768
Sweep Width (Hz) 31250.00
Frequency (MHz) 125.76
Nucleus 13C
Temperature (grad C) 0.000
Chloroform-d
2o
O
NH
MeO
Br
22.84
56.72
69.24
77.00
105.65
114.70
117.75
118.91
124.24
137.34
150.04
154.87
201.39


22 Mar 2013
Acquisition Time (sec) 1.0486
Original Points Count 32768
Date 22/03/2013 21:27:08
Points Count 32768
Sweep Width (Hz) 31250.00
Frequency (MHz) 125.76
Nucleus 13C
Temperature (grad C) 0.000
22.84
29.70
56.73
105.65
114.72
117.76
137.35
O
N
H
MeO
Br
2o


D:\Data\sample-1_130322183815
3/22/2013 6:38:15 PM
sample-1_130322183815 #910 RT: 4.05 AV: 1 NL: 6.06E8
T: FTMS + p ESI Full ms [100.00-700.00]
282.0129
R=66906
C 12 H 13 O 2 N Br = 282.0124
1.6334 ppm
272.8787
R=39300
279.1597
R=66902
288.9214
R=66107
293.1745
R=63007
296.9204
R=49900
301.1408
R=64107
305.9922
R=63707
308.99
R=559(
MeO
Br
O
NH
2o
Relative Abundance
m/z