Electronic Supplementary Information

A Modular Total Synthesis of Trigonoliimine C

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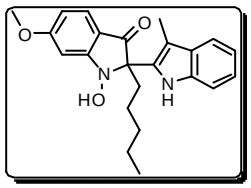
General Remarks

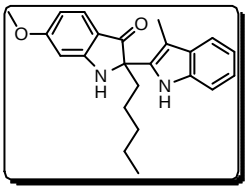
Reactions were carried out in anhydrous solvents under an atmosphere of argon in oven-dried glassware. Commercial reagents and solvents were used without purification. Column Chromatography was carried out by using spectrochem silica gel (60–120, 100–200, 230–400 mesh). ^{1}H and ^{13}C NMR spectroscopy measurements were carried out on Bruker AV 200 MHz AV 400, AV 500 MHz and and JEOL 400 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, dt = doublet of triplet, td = triplet of doublet, bs = 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 a API QStar Pulsar (Hybrid Quadrupole-TOF LC/MS/MS) spectrometer or UPLC coupled Mass Spectrometer (Waters) and HRMS mass spectra were recorded on a Thermo Scientific Q-Exactive, Accela 1250 pump.

***General procedure A:* Addition of 3-methyl indole to isatogen:** To a solution of isatogen **7a** (60 mg, 0.24 mmol) and 3-methyl indole (38 mg, 0.29 mmol) in CH_2Cl_2 (2 mL) was added, $Au[PPh_3]Cl$ (12 mg, 0.6 mmol, 10 mol%), $AgSbF_6$ (20 mg, 0.6 mmol, 25 mol%) at 0 °C. The reaction mixture was stirred for overnight at room temperature. After completion of the reaction as indicated by TLC, the volatiles are removed under reduced pressure and the resulting crude was purified by column chromatography to afford compound **8a** (69 mg, 75%) as a brown red liquid.



Spectral data of compound 8a: R_f = 0.3 (10% ethyl acetate/pet. ether); IR ($CHCl_3$) *v*: 3392, 3255, 2953, 2927, 1670, 1620, 1579, 1458, 1290, 1229, 1149, 1099, 823, 741 cm^{-1}; **^{1}H NMR (Acetone-D_6, 200 MHz):** δ 0.84 (bs, 3H), 1.31 (bs, 6H), 2.18 (s, 3H), 2.46–2.50 (m, 2H), 3.96 (s, 3H), 6.57 (d, *J* = 8.5, 1.5 Hz, 1H), 6.67 (d, *J* = 1.6 Hz, 1H), 7.05 (m, 2H), 7.33 (d, *J* = 7.3 Hz, 1H), 7.41 (d, *J* = 9.1 Hz, 1H), 7.50 (d, *J* = 8.4 Hz, 1H), 8.98 (s, 1H), 9.98 (bs, 1H); **^{13}C NMR (CD_3OD, Jeol, 100 MHz):** δ 9.7 (q), 14.4 (q), 23.6 (t), 24.9 (t), 33.3 (t), 34.9 (t), 56.6 (q), 78.5 (s), 95.4 (d), 109.3 (s), 112.2 (d), 112.0 (d), 115.2 (s), 119.1 (d), 119.7 (d), 122.4 (d), 126.1 (d), 130.9 (s), 132.5 (s), 136.9 (s), 166.9 (s), 170.2 (s), 199.2 (s) ppm. ESI-MS (*m/z*): 401.08 (100%, $[M+Na]^+$), 417.18 (5%, $[M+K]^+$), HRMS (ESI+): calcd. for $C_{23}H_{27}O_3N_2$, $[M+H]^+$: 379.2016, found 379.2014.

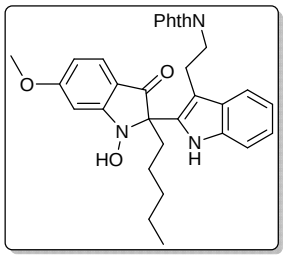
General procedure B: ***N-hydroxy indoxyl reduction*****:** A solution of indoxyl **8a** (60 mg, 0.16 mmol) and hydrazine monohydrate (79 mg, 1.6 mmol) in MeOH (6 ml) was heated to reflux for 2 h. After completion of the reaction, the volatiles are removed under reduced pressure and the crude purified by column chromatography to afford compound **9** (51 mg, 89%) as a brown liquid.



Spectral data of compound 9: R_f = 0.3 (10% ethyl acetate/pet. ether); IR ($CHCl_3$) *v*: 3368, 3056, 1738, 1649, 1371, 1252, 1116, 882, 723 cm^{-1}; **^{1}H NMR ($CDCl_3$, 200 MHz):** δ 0.81 (t, *J* = 6.3 Hz, 3H), 1.23 (bs, 6H), 1.19–2.13 (m, 2H), 2.45 (s, 3H), 3.86 (s, 3H), 5.28 (s, 1H), 6.37 (d, *J* = 2.0 Hz, 1H), 6.67 (dd, *J* = 8.6, 2.0 Hz, 1H), 7.06 (td, *J* = 7.1,1.3 Hz, 1H), 7.14 (td, *J* = 7.1,1.3, Hz, 1H), 7.28 (dd, *J* = 7.2 Hz, 1H), 7.48 (d, *J* = 7.1 Hz, 1H), 7.53 (d, *J* = 8.6 Hz, 1H), 9.02 (s, 1H); **^{13}C NMR (CD_3OD,50 MHz):** 9.7 (q), 14.0 (q), 22.4 (t), 23.5 (t), 31.8 (t), 39.7 (t), 55.6 (q), 70.2 (s), 94.7 (d), 107.4 (s),

109.5 (d), 110.8 (d), 113.5 (s), 118.1 (d), 119.0 (d), 121.8 (d), 126.6 (d), 129.5 (s), 130.9 (s), 134.7 (s), 162.8 (s), 168.2 (s), 200.0 (s) ppm. ESI-MS (*m/z*): 385.01 (100%, $[M+Na]^+$), HRMS (ESI+): calcd. for $C_{23}H_{27}O_2N_2$, $[M+H]^+$: 363.2067, found 363.2056.

2-(2-(2-(1-Hydroxy-6-methoxy-3-oxo-2-pentylindolin-2-yl)-1H-indol-3-yl)ethyl)isoindoline-1,3-dione (10a): The addition of tryptamine **4** (183 mg, 0.63 mmol) to isatogen **7a** (130 mg, 0.52 mmol), was carried out following the general procedure A to obtain indoxyl derivative **10a** (194 mg, 68%) as a brown red liquid; R_f = 0.3 (20% ethyl acetate/pet. ether); IR ($CHCl_3$) *v*: 3262, 3045, 2882, 1765, 1648, 1329, 1268, 1123, 843, 768 cm^{-1};

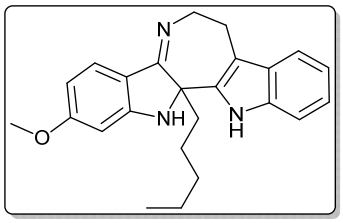


^{1}H NMR ($CDCl_3$, 500 MHz): δ 0.78 (t, *J* = 6.4 Hz, 3H), 1.18 (bs, 4H), 1.24–1.32 (m, 2H), 2.24 (dt, *J* = 12.2, 4.3, 3.7 Hz, 1H), 2.36 (dt, *J* = 12.2, 3.7, 2.7 Hz, 1H), 3.27 (t, *J* = 9.2 Hz, 2H), 3.86–3.92 (m, 1H), 3.96 (s, 3H), 3.98–4.05 (m, 1H), 6.63 (dd, *J* = 8.5, 1.2 Hz, 1H), 6.89 (d, *J* = 1.2 Hz, 1H), 7.10 (t, *J* = 7.6 Hz, 1H), 7.16 (t, *J* = 7.3 Hz, 1H), 7.35 (d, *J* = 7.9 Hz, 1H), 7.58 (d, *J* = 8.5 Hz, 1H), 7.64 (d, *J* = 7.6 Hz, 1H), 7.77 (dd, *J* = 5.2, 2.1 Hz, 2H), 7.90 (dd, *J* = 5.2, 2.1 Hz, 2H), 8.97 (s, 1H), 9.47 (bs, 1H); **^{13}C NMR ($CDCl_3$, 125 MHz):** 14.0 (q), 22.2 (t), 23.7 (t), 24.2 (t), 31.8 (t), 38.5 (t), 39.0 (t), 55.9 (q), 76.2 (s), 96.9 (d), 107.8 (s),111.1 (d), 112.5 (d), 114.7 (s), 117.9 (d), 119.5 (d), 122.0 (d), 123.6 (d, 2C), 125.2 (d), 128.3 (s), 132.0 (s), 133.4 (s), 134.3 (d, 2C), 135.1 (s), 166.7 (s), 168.3 (s), 169.3 (s), 198.2 (s) ppm; ESI-MS (*m/z*): 560.17 (100%, $[M+Na]^+$), HRMS (ESI+): calcd. for $C_{32}H_{32}O_5N_3$, $[M+H]^+$: 538.2336, found 538.2336.

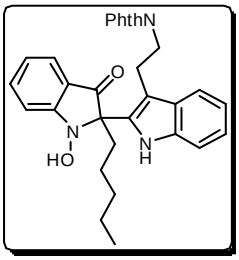
General Procedure C: ***N-Phth deprotection and cyclization*****:** A solution of indoxyl **10a** (110 mg, 0.20 mmol) and hydrazine monohydrate (102 mg, 2.1 mmol) in methanol (5 ml) was heated to reflux for 2 h and then the reaction mixture was concentrated. The resulting crude mixture was dissolved in THF (7 mL) and transferred to a flame-dried flask. After the flask was degassed with purging argon gas and Ti(O*i*-Pr)$_4$ (116 mg, 0.12 ml, 0.41 mmol) was added drop wise at

room temperature. The reaction mixture was stirred at 75 °C for 2 h. The reaction mixture was concentrated under reduced pressure and the resulting residue was subjected for purification by flash chromatography on neutral silica gel (treated with triethyl amine) to afford the compound **11a** (38 mg, 48%) as a pale yellow liquid.

***Spectra data of compound 11a*:** IR ($CHCl_3$) *v*: 3052, 2929, 2850, 1754, 1566, 1349, 1243, 1182, 1017, 884, 763 cm^{-1}; **^{1}H NMR(500MHz, $CDCl_3$):** δ 0.82 (t, *J* = 6.7 Hz, 3H), 1.24 (bs, 4H), 1.30–1.46 (m, 2H), 2.12 (dt, *J* = 12.5, 2.7 Hz, 1H), 2.53 (dt, *J* = 13.1, 4.6 Hz, 1H), 3.06 (dt, *J* = 16.5, 3.7 Hz, 1H), 3.12 (td, *J* = 16.8, 3.0 Hz, 1H), 3.76 (s, 3H), 4.23 (td, *J* = 11.9, 3.7 Hz, 1H), 4.43 (dt, *J* = 12.8, 3.7, 2.4 Hz, 1H), 6.30 (bs, 1H), 6.42 (d, *J* = 7.6 Hz, 1H), 7.07 (t, *J* = 7.3 Hz, 1H), 7.14 (t, *J* = 7.6 Hz, 1H), 7.30 (d, *J* = 8.2 Hz, 1H), 7.46 (d, *J* = 7.6 Hz, 1H), 7.52 (d, *J* = 8.5 Hz, 1H), 8.27 (bs, 1H); **^{13}C NMR(125MHz, $CDCl_3$):** 14.0 (q), 22.4 (t), 23.3 (t), 24.0 (t), 31.8 (t), 40.9 (t), 47.0 (t), 55.4 (q), 68.4 (s),96.6 (d), 108.3 (d), 110.0 (s), 110.7 (d), 118.1 (d), 119.4 (d), 122.0 (d), 124.2 (d), 129.3 (s), 132.5 (s), 132.6 (s), 135.1 (s), 157.0 (s), 164.7 (s), 172.5 (s) ppm ppm. ESI-MS (*m/z*): 374.15 (60%, $[M+H]^+$), HRMS (ESI+): calcd. for $C_{24}H_{28}N_3O$, $[M+H]^+$: 374.2227, found 374.2225.

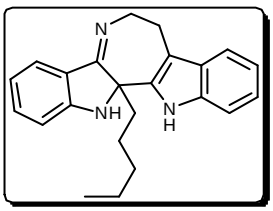


2-(2-(2-(1-Hydroxy-3-oxo-2-pentylindolin-2-yl)-1H-indol-3-yl)ethyl)isoindoline-1,3-dione (10b): The addition of *N*-phthalimido tryptamine **4** (290 mg, 1.0 mmol) to isatogen **7b** (180 mg, 0.83 mmol) has been carried out according to the general procedure **A** to obtain the N-OH indoxyl derivative **10b** (268 mg, 63%) as a pale yellow solid. R_f = 0.2 (10% ethyl acetate/pet. ether); M.P =179 °C; IR ($CHCl_3$) *v*: 3368, 2929, 1707, 1605, 1494, 1452, 1397,



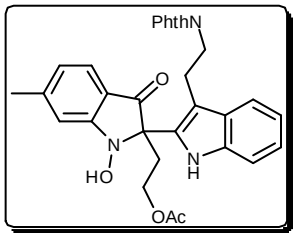
1290, 1289, 1102, 1024, 745, 718 cm^{-1};**^{1}H NMR ($CDCl_3$, 200 MHz):** δ 0.77 (t, *J* = 3.5 Hz, 2H), 1.17–1.29 (m, 6H), 2.26–2.28 (m, 2H), 3.29 (t, *J* = 8.7 Hz, 3H), 3.88–4.07 (m, 2H), 7.06–7.21 (m, 3H), 7.33 (d, *J* = 7.7 Hz, 1H), 7.54 (d, *J* = 7.9 Hz, 1H), 7.65–7.68 (app d, *J* = 6.7 Hz, 3H), 7.70 (dd, *J* = 5.2, 3.0 Hz, 2H), 7.91 (dd, *J* = 5.0, 3.3 Hz 2H), 9.06 (s, 1H), 9.41 (s, 1H); **^{13}C NMR ($CDCl_3$, 50 MHz):** δ 13.9 (q), 22.1 (t), 23.7 (t), 24.3 (t), 31.8 (t), 38.5 (t), 39.0 (t), 76.6 (s), 108.1 (s), 111.0 (d), 115.0 (d), 118.0 (d), 119.5 (d), 122.1 (d), 122.7 (d), 123.5 (d, 2C), 125.6 (d), 128.3 (s), 131.9 (s, 2C), 132.9 (s), 134.3 (d, 2C), 135.1 (s), 135.9 (s), 137.8 (d), 164.2 (s), 169.2 (s, 2C), 200.9 (s) ppm. ESI-MS (*m/z*): 530.24 (100%, $[M+Na]^+$), 546.17 (25%, $[M+K]^+$), HRMS (ESI+): calcd. for $C_{31}H_{30}O_4N_3$, $[M+H]^+$: 508.2231, found 508.2229.

12b-Pentyl-7,12,12b,13-tetrahydro-6H-azepino[3,2-b:4,5-b']diindole (11b): According to the general procedure C, the treatment of indoxyl **10b** (140 mg, 0.28 mmol) with hydrazine monohydrate (140 mg, 2.8 mmol) followed by $Ti(Oi\text{-}Pr)_4$ (160 mg, 0.55 mmol) gave the **11b** (48 mg, 51%) as a yellow liquid; R_f = 0.3 (10%/0.2% MeOH/Et_3N/CH_2Cl_2); R_f = 0.3 (70% ethyl acetate/pet ether); IR ($CHCl_3$) *v*: 3273, 2955, 2925, 2854, 1739, 1615, 1462, 1377, 1294, 1211, 1165, 1024, 824, 743 cm^{-1}; **^{1}H NMR (400MHz, $CDCl_3$):** δ 0.82 (t, *J* = 6.8 Hz, 3H), 1.24 (m, 6H), 1.40 (m, 2H), 2.15 (qd, *J* = 4.5 Hz, 1H), 2.55 (qd, *J* = 5.0, 4.8 Hz, 1H), 3.09 (td, *J* = 13.1, 3.8 Hz, 1H), 3.15 (dt, *J* = 13.1, 3.8 Hz, 1H), 4.23 (dt, *J* = 11.8, 3.5 Hz, 1H), 4.43 (td, *J* = 12.5, 4.0, 3.5 Hz, 1H), 6.79 (d, *J* = 8.0 Hz, 1H), 6.84 (td, *J* = 7.5, 0.5 Hz, 1H), 7.08 (td, *J* = 6.8, 1.0 Hz,1H), 7.14 (td, *J* = 7.0, 1.0 Hz, 1H), 7.25 (td, *J* = 1.2 Hz, 1H), 7.31 (d, *J* = 8.0 Hz, 1H), 7.48 (d, *J* = 7.8 Hz, 1H), 7.66 (d, *J* = 7.5 Hz 1H), 8.46 (s, 1H); **^{13}C NMR (100 MHz, $CDCl_3$):** 13.1 (q), 22.4 (t), 23.1 (t), 24.0 (t), 31.8 (t), 40.7 (t), 47.2 (t), 67.8 (s), 109.9 (s), 110.7 (d), 112.2 (d), 118.1 (d), 119.4 (d), 120.6 (d), 122.0 (d), 123.0

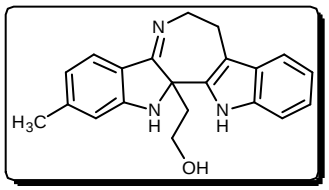


(d), 125.9 (s), 129.2 (s), 132.4 (s), 133.3 (d), 135.1 (s), 155.1 (s), 173.7 (s) ppm. ESI-MS (*m/z*): 344.16 (60%, $[M+H]^+$), HRMS (ESI+): calcd. for $C_{23}H_{26}N_3$, $[M+H]^+$: 344.2121, found 344.2120.

2-(3'-(2-(1,3-Dioxoisoindolin-2-yl)ethyl)-6-methyl-3-oxo-2,2'-biindolin-2-yl)ethyl acetate (10c): The addition of tryptamine **4** (295 mg, 1.02 mmol) to isatogen **7c** (210 mg, 0.85 mmol) was carried out following the general procedure A to obtain indoxyl derivative **10c** (324 mg, 71%) as a yellow solid; R_f = 0.3 (30% ethyl acetate/pet. ether); M.P = 118–120 °C; IR ($CHCl_3$) *v*: 3399, 3225, 2952,1701, 1628, 1316, 1215, 1108, 821, 745 cm^{-1};**^{1}H NMR ($CDCl_3$, 500 MHz):** δ 1.88 (s, 3H), 2.51 (s, 3H), 2.69 (bs, 2H), 3.29 (app t, 2H), 3.90 (m, 1H), 4.04–4.10 (m, 1H), 4.17 (m, 2H), 6.96 (d, *J* = 7.3 Hz, 1H), 7.12 (t, *J* = 6.4 Hz, 1H), 7.19 (t, *J* = 6.7 Hz,1H), 7.35 (s, 1H), 7.39 (d, *J* = 7. 3 Hz, 1H), 7.59 (d, *J* = 7.3 Hz, 1H), 7.66 (d, *J* = 7.3 Hz, 1H), 7.77 (bs, 2H), 7.92 (bs, 2H), 9.16 (s, 1H), 9.55 (s, 1H); **^{13}C NMR ($CDCl_3$, 125 MHz):** 20.6 (q), 22.5 (q), 23.5 (t), 36.6 (t), 38.8 (t), 60.5 (t), 74.8 (s), 96.0 (d), 108.6 (s), 111.1 (d), 115.1 (d), 117.7 (d), 118.8 (s), 119.5 (d), 122.2 (d), 123.4 (d, 2C), 124.8 (d), 128.2 (s), 131.7 (s), 131.8 (s, 2C), 134.2 (d, 2C), 135.2 (s), 149.8 (s), 163.9 (s), 169.1 (s, 2C), 170.6 (s), 198.6 (s) ppm. ESI-MS (*m/z*): 560.17 (100%, $[M+Na]^+$), 576.15 (50%, $[M+K]^+$), HRMS (ESI+): calcd. for $C_{31}H_{27}O_6N_3$, $[M+Na]^+$: 560.1792, found 560.1791.

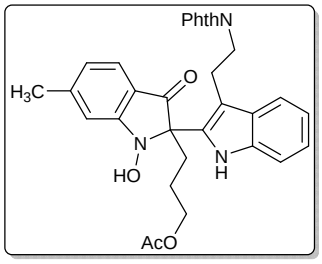


2-(2-Methyl-6,7,12,13-tetrahydro-12bH-azepino[3,2-b:4,5-b']diindol-12b-yl)ethan-1-ol (11c): According to the general procedure C, the treatment of indoxyl **10c** (135 mg, 0.25 mmol) with hydrazine monohydrate (125 mg, 2.51 mmol) followed by Ti(O*i*-Pr)$_4$ (142 mg, 0.5 mmol) gave the **11c** (36 mg, 43%) as a yellow liquid; R_f = 0.2 (10%/0.2%



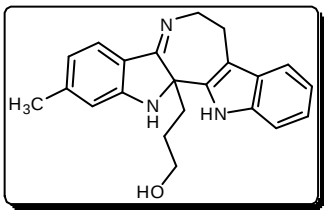
MeOH/Et_3N/CH_2Cl_2); IR ($CHCl_3$) *v*: 3276, 2950, 2729, 1734, 1522, 1360, 1232, 1192, 1021, 893, 723 cm^{-1}; **^{1}H NMR [500MHz, CD_3OD : $CDCl_3$ (3:1)]:** δ 2.29 (s, 3H), 2.48 (m, 2H), 3.03 (td, *J* = 16.5, 3.1 Hz, 1H), 3.16 (app dt, *J* = 12.5, Hz, 1H), 3.69 (t, *J* = 5.8 Hz, 2H), 4.02 (d, *J* = 12.5, Hz, 1H), 4.35 (t, *J* = 12.5 Hz, 1H), 6.61 (d, *J* = 7.9 Hz, 1H), 6.63 (bs, 1H), 7.01 (t, *J* = 7.6 Hz, 1H), 7.11 (t, *J* = 7.6 Hz, 1H), 7.33 (d, *J* = 7.9 Hz, 1H), 7.43 (d, *J* = 7.9 Hz, 1H), 7.50 (d, *J* = 7.9 Hz, 1H); **^{13}C NMR [125MHz, CD_3OD:$CDCl_3$ (3:1)]:** 22.4 (q), 24.3 (t), 42.0 (t), 47.3 (t), 59.1 (t), 68.7 (s), 110.6 (d), 111.5 (d), 112.8 (d), 118.5 (d), 119.7 (s), 120.7 (s), 121.8 (d), 122.7 (d), 123.9 (d), 129.1 (s), 130.5 (s), 136.3 (s), 147.1 (s), 157.6 (s), 177.4 (s) ppm. ESI-MS (*m/z*): 332.10 (100%, $[M+H]^+$), HRMS (ESI+): calcd. for $C_{21}H_{22}N_3O$, $[M+H]^+$: 332.1757, found 332.1757.

3-(2-(3-(2-(1,3-Dioxoisoindolin-2-yl)ethyl)-1H-indol-2-yl)-1-hydroxy-6-methyl-3-oxoindolin-2-yl)propyl acetate (10d): The addition of tryptamine **4** (146 mg, 0.51 mmol) to isatogen **7d** (110 mg, 0.42 mmol), was carried out following the general procedure A to obtain indoxyl derivative **10d** (142 mg, 61%) as a yellow solid; R_f = 0.3 (30% ethyl acetate/pet. ether); M.P=130–133 °C; IR ($CHCl_3$) *v*: 3392, 3245, 2968, 1721, 1638, 1331, 1256, 1123, 843, 768 cm^{-1};**^{1}H NMR ($CDCl_3$, 200 MHz):** δ 1.66–1.78 (m, 2H), 2.00 (s, 3H), 2.39 (m, 2H), 2.51 (s, 3H), 3.28–3.35 (m, 2H), 3.98–4.08 (m, 4H), 6.96 (d, *J* = 8.8 Hz, 1H), 7.17 (bs, 2H), 7.35 (bs, 2H), 7.59 (d, *J* = 7.7 Hz, 1H), 7.69 (d, *J* = 6.7 Hz, 1H), 7.79 (bs, 2H), 7.93 (bs, 2H), 9.11 (s, 1H), 9.46 (bs, 1H); **^{13}C NMR ($CDCl_3$:CD_3OD, 100 MHz):** 20.2 (q), 22.0 (q), 23.5 (t), 23.6 (t), 32.3 (t), 38.3 (t), 64.0 (t), 76.3 (s), 108.3 (s), 110.6 (d), 113.3 (d), 117.7 (d), 118.1 (s), 118.8 (d), 121.4 (d), 122.8 (d, 2C), 123.1 (d), 123.6 (d), 128.4 (s), 131.5 (s), 133.8 (d, 2C), 133.9 (s), 135.1 (s), 150.1 (s), 163.7 (s), 169.1 (s, 2C), 172.0 (s), 198.8 (s); ESI-

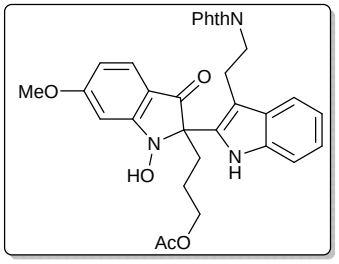


MS (*m/z*): 573.97 (100%, $[M+Na]^+$), HRMS (ESI+): calcd. for $C_{32}H_{29}O_6N_3$, $[M+Na]^+$: 574.1949, found 574.1943.

3-(2-Methyl-6,7,12,13-tetrahydro-12bH-azepino[3,2-b:4,5-b']diindol-12b-yl)propan-1-ol (11d): According to the general procedure C, the treatment of indoxyl **10d** (122 mg, 0.22 mmol), with hydrazine monohydrate (110 mg, 2.21 mmol) followed by Ti(O*i*-Pr)$_4$ (125 mg, 2ml, 0.44 mmol) gave **11d** (36 mg, 47%) as a yellow liquid; R_f = 0.2 (10%/0.2% MeOH/Et_3N/CH_2Cl_2); IR ($CHCl_3$) *v*: 3356, 2829, 1721, 1648, 1478, 1423, 1397, 1290, 1252, 1110, 1079, 868, 728 cm^{-1}; **^{1}H NMR(500MHz, CD_3OD):** δ 1.55–1.63 (m, 2H), 2.29 (s, 3H), 2.25–2.31 (m, 1H), 2.52–2.59 (m, 1H), 3.09 (td, *J* = 13.4, 3.7 Hz, 1H), 3.15 (dt, *J* = 16.8, 3.1 Hz, 1H), 3.51 (t, *J* = 6.4 Hz, 2H), 4.00 (dt, *J* = 12.2, 3.3 Hz, 1H), 4.42 (td, *J* = 12.5, 2.7, 1.8 Hz, 1H), 6.58 (d, *J* = 7.9 Hz, 1H), 6.62 (s, 1H), 7.00 (td, *J* = 7.9, 0.6 Hz, 1H), 7.10 (td, *J* = 7.9, 0.9 Hz, 1H), 7.32 (d, *J* = 8.2 Hz, 1H), 7.42 (d, *J* = 7.9 Hz, 1H), 7.48 (d, *J* = 8.2 Hz, 1H); **^{13}C NMR [125MHz, CD_3OD:$CDCl_3$(3:1)]:** 22.5 (q), 24.4 (t), 28.1 (t), 37.8 (t), 47.3 (t), 62.2 (t), 69.4 (s), 110.1 (s), 111.6 (d), 112.3 (d), 118.6 (d), 119.8 (d), 120.7 (s), 121.6 (d), 122.8 (d), 123.9 (d), 129.3 (s), 131.7 (s), 136.4 (s), 147.6 (s), 158.3 (s), 177.5 (s) ppm. ESI-MS (*m/z*): 346.11 (100%, $[M+H]^+$), HRMS (ESI+): calcd. for $C_{22}H_{24}N_3O$, $[M+H]^+$: 346.1914, found 346.1914.

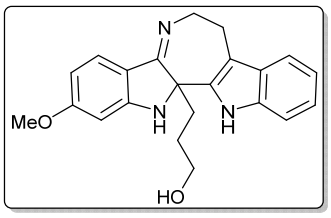


3-(2-(3-(2-(1,3-Dioxoisoindolin-2-yl)ethyl)-1H-indol-2-yl)-1-hydroxy-6-methoxy-3-oxoindolin-2-yl)propyl acetate (10e): The addition of tryptamine **4** (251 mg, 0.86 mmol) to isatogen **7e** (200 mg, 0.72 mmol), was carried out following the general procedure A to obtain N-OH indoxyl derivative **10e** (305 mg, 74%)

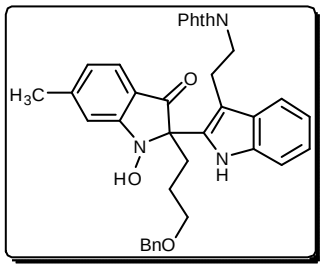


as a pale yellow solid M.P=193–195 °C; R_f = 0.2 (30% ethyl acetate/pet. ether); IR ($CHCl_3$) *v*: 3368, 2929, 1707, 1605, 1494, 1452, 1397, 1290, 1289, 1102, 1024, 745, 718 cm^{-1}; **^{1}H NMR ($CDCl_3$, 200 MHz):** δ 1.47–1.75 (m, 2H), 1.99 (s, 3H), 2.39 (t, *J* = 5.9 Hz, 2H), 3.28 (t, *J* = 8.7 Hz, 2H), 3.96 (s, 3H), 3.86–3.99 (m, 4H), 6.65 (d, *J* = 8.5 Hz, 1H), 6.9 (s, 1H), 7.11 (app td, *J* = 7.0, 6.8 Hz, 1H), 7.18 (app td, *J* = 6.8, 6.4 Hz,1H), 7.36 (d, *J* = 7.4 Hz, 1H), 7.59 (d, *J* = 8.6 Hz, 1H), 7.65 (d, *J* = 7.7 Hz, 1H), 7.75 (dd, *J* = 5.2, 3.0 Hz, 2H), 7.90 (dd, *J* = 5.5, 3.0 Hz, 2H), 9.08 (s, 1H), 9.48 (s, 1H); **^{13}C NMR ($CDCl_3$,50 MHz):** 20.8 (q), 23.6 (t), 24.0 (t), 34.8 (t), 38.9 (t), 55.8 (q), 64.0 (t), 96.0 (s), 97.0 (d), 108.2 (s), 111.1 (d), 112.7 (d), 114.4 (s), 117.9 (d), 119.5 (d), 122.8 (d), 123.5 (d, 2C), 125.3 (s), 128.2 (s), 131.9 (s, 2C), 132.6 (s), 134.2 (d, 2C), 135.1 (s), 166.5 (s), 168.3 (s), 169.1 (s, 2C), 170.9 (s), 197.5 (s) ppm. ESI-MS (*m/z*): 590.16 (100%, $[M+Na]^+$), 606.12 (5%, $[M+K]^+$), HRMS (ESI+): calcd. for $C_{32}H_{30}O_7N_3$, $[M+H]^+$: 568.2078, found 568.2078.

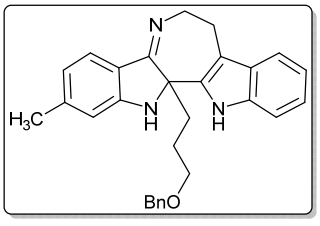
3-(2-Methoxy-6,7,12,13-tetrahydro-12bH-azepino[3,2-b:4,5-b']diindol-12b-yl)propan-1-ol (11e): According to the general procedure C, the treatment of indoxyl **10e** (210 mg, 0.37 mmol), with hydrazine monohydrate (185 mg, 3.70 mmol) followed by Ti(O*i*-Pr)$_4$ (210 mg, 0.74 mmol) gave **11e** (52 mg, 39%) as a yellow liquid; R_f = 0.2 (10%/0.2% MeOH/Et_3N/CH_2Cl_2); IR ($CHCl_3$) *v*: 3272, 2925, 2854, 1734, 1621, 1457, 1338, 1289, 1166, 1121, 825, 744 cm^{-1}; **^{1}H NMR [500MHz, CD_3OD : $CDCl_3$ (3:1)]:** δ 1.55–1.63 (m, 2H), 2.25–2.31 (m, 1H), 2.48–2.54 (m, 1H), 3.03 (td, *J* = 16.8, 3.1 Hz, 1H), 3.14 (app dt, *J* = 16.5 Hz, 1H), 3.51 (t, *J* = 6.1 Hz, 2H), 3.79(s, 3H), 3.96 (d, *J* = 12.5 Hz, 1H), 4.37 (t, *J* = 12.8 Hz, 1H), 6.27 (bs, 1H), 6.31 (dd, *J* = 8.8, 1.8 Hz, 1H), 7.01 (t, *J* = 7.6 Hz, 1H), 7.10 (t, *J* = 7.6 Hz, 1H), 7.32 (d, *J* = 8.2 Hz, 1H), 7.42 (d, *J* = 7.9 Hz, 1H), 7.49 (d, *J* = 8.8 Hz, 1H), 8.56 (bs, 1H); **^{13}C NMR**



[125MHz, CD_3OD:$CDCl_3$, (3:1)]: 24.4 (t), 27.8 (t), 37.6 (t), 47.0 (t), 55.9 (q), 62.1 (t), 69.4 (s), 94.9 (d), 108.6 (d), 109.9 (s), 111.4 (d), 116.0 (s), 118.4 (d), 119.6 (d), 122.5 (d), 125.2 (d), 129.0 (s), 131.6 (s), 136.1 (s), 159.5 (s), 166.9 (s), 175.9 (s) ppm. ESI-MS (*m/z*): 362.15 (100%, $[M+H]^+$), HRMS (ESI+): calcd. for $C_{22}H_{24}N_3O_2$, $[M+H]^+$: 362.1863, found 362.1865.



2-(2-(2-(2-(3-(Benzyloxy)propyl)-1-hydroxy-6-methyl-3-oxoindolin-2-yl)-1H-indol-3-yl)ethyl)isoindoline-1,3-dione (10f): The addition of tryptamine **4** (123 mg, 0.43 mmol) to isatogen **7f** (110 mg, 0.35 mmol), was carried out following the general procedure A to obtain indoxyl derivative **10f** (127 mg, 60%) as a brown red liquid; R_f = 0.4 (20% ethyl acetate/pet. ether); IR ($CHCl_3$) *v*: 3243, 3051, 2863, 1728, 1654, 1369, 1243, 1021, 823, 754 cm^{-1};**^{1}H NMR ($CDCl_3$, 500 MHz):** δ 1.51–1.56 (m, 1H), 1.64–1.71 (m, 1H), 2.41 (t, *J* = 8.3 Hz, 2H), 2.47 (s, 3H), 3.22–3.28 (m, 2H), 3.36–3.42 (m, 2H), 3.83–3.91 (m, 1H), 4.0–4.06 (m, 1H), 4.42 (s, 2H), 6.90 (d, *J* = 8.0 Hz, 1H), 7.07 (dt, *J* = 7.8 Hz, 1H), 7.13 (dt, *J* = 8.0, 1.0 Hz, 1H), 7.25–7.30 (m, 7H), 7.56 (d, *J* = 8.0 Hz, 1H), 7.62 (d, *J* = 7.9 Hz, 1H), 7.74 (dd, *J* = 5.5, 3.0 Hz, 1H), 7.89 (dd, *J* = 5.2, 3.0 Hz, 2H), 8.97 (bs, 1H), 9.48 (bs, 1H); **^{13}C NMR ($CDCl_3$, 100 MHz):** 22.6 (q), 23.6 (t), 25.0 (t), 34.7 (t), 38.9 (t), 69.9 (t), 72.8 (t), 76.5 (s), 108.4 (s), 111.1 (d), 114.9 (d), 117.9 (d), 119.1 (d), 119.5 (s), 122.0 (d), 123.5 (d), 123.5 (d), 124.5 (d), 127.5 (d), 127.7 (d), 128.3 (d), 128.3 (s), 131.9 (s), 132.7 (s), 134.3 (d), 135.1 (s), 138.3 (s), 149.8 (s), 164.2 (s), 169.2 (s), 199.5(s), ppm; ESI-MS (*m/z*): 560.17 (100%, $[M+Na]^+$), HRMS (ESI+): calcd. for $C_{37}H_{33}O_5N_3$, $[M+Na]^+$: 622.2312, found 538.2308.

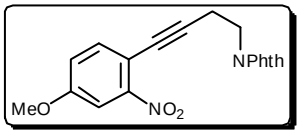


12b-(3-(Benzyloxy)propyl)-2-methyl-7,12,12b,13-tetrahydro-6H-azepino[3,2-b:4,5-b']diindole (11f): According to the general procedure C, the treatment of indoxyl **10f** (52 mg, 0.09 mmol) with hydrazine monohydrate (43 mg, 0.9 mmol) followed by $Ti(Oi\text{-}Pr)_4$ (49 mg, 0.17 mmol) gave the **11a** (19 mg, 52%) as a yellow liquid; R_f = 0.4 (80% ethyl acetate/pet. ether); IR ($CHCl_3$)

v: 3063, 2832, 1654, 1552, 1321, 1289, 1093, 1045, 864, 735 cm^{-1}; **^{1}H NMR(500MHz, $CDCl_3$):** δ 1.67–1.84 (m, 2H), 1.28 (s, 3H), 2.40–2.49 (m, 2H), 3.04–3.11 (m, 2H), 3.50 (t, *J* = 5.9 Hz, 2H), 4.18 (app td, *J* = 11.7, 3.6 Hz, 1H), 4.37 (dt, *J* = 11.5, 5.3 Hz, 1H), 4.52 (s, 2H), 6.46 (bs, 1H), 6.65 (dd, *J* = 7.8, 0.6 Hz, 1H), 7.06 (dt, *J* = 7.3, 1.3 Hz, 1H), 7.14 (dt, *J* = 7.6, 1.3 Hz, 1H), 7.22 (bs, 1H), 7.36 (bs, 5H), 7.44 (bs, 1H), 7.50 (d, *J* = 8.1 Hz, 1H), 8.36 (bs, 1H); **^{13}C NMR(125MHz, $CDCl_3$):** 22.1 (q), 23.6 (t), 24.8 (t), 29.3 (t), 31.9 (t), 37.2 (t), 69.7 (t), 71.3 (s), 73.3 (t), 105.1 (s), 109.4 (s), 109.7 (s), 110.3 (s), 110.9 (d), 111.8 (d), 117.8 (d), 119.4 (d), 121.7 (d), 122.4 (d), 127.8 (2C, d), 127.9 (d), 128.3 (d), 128.5 (2C, d), 135.1 (s), 137.7 (s), 140.9 (s), 161.6 (s), 172.7 (s) ppm. ESI-MS (*m/z*): 436.21 (100%, $[M+H]^+$), HRMS (ESI+): calcd. for $C_{24}H_{28}N_3O_2$, $[M+H]^+$: 436.2383, found 436.2384.

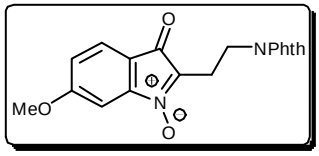
Synthesis of Compound (11g): Compound **11g** (45 mg, 27%) was prepared by the addition of tryptamine **4** (175 mg, 0.61 mmol) to isatogen **7g** (120 mg, 0.51 mmol) followed by subjecting the resulting crude indoxyl derivative **10g** with hydrazine monohydrate (134 mg, 2.7 mmol) and then with Ti(O*i*-Pr)$_4$(152 mg, 0.54 mmol). Pale yellow solid; R_f = 0.4 (10%/0.2% MeOH/Et_3N/CH_2Cl_2); M.P=153–155 °C; IR ($CHCl_3$) *v*: 3273, 2925, 2851, 1658, 1610, 1458, 1320, 1290, 1289, 1151, 1024, 744, 718 cm^{-1}; **^{1}H NMR ($CDCl_3$, 400 MHz):** δ 2.13–2.18 (m, 1H), 2.23–2.32 (m, 2H), 2.35 (s, 3H), 2.62–2.66 (m, 1H), 3.0–3.08 (m, 1H), 3.1–3.16 (dt, *J* = 16.9, 3.4 Hz, 1H), 3.28 (dt, *J* = 9.3, 9.1 Hz, 1H), 3.85–3.9 (m, 1H), 4.22–4.26 (m, 2H), 6.68 (s, 1H), 6.76 (d, *J* = 7.8 Hz 1H), 7.07 (app td, *J* = 7.1, 7.8 Hz, 1H), 7.14 (td, *J* = 7.1, 7.8 Hz, 1H), 7.29 (d, *J* = 8.1 Hz, 1H), 7.46 (d, *J* = 7.8 Hz, 1H), 7.51 (d, *J* = 7.8 Hz, 1H), 8.13 (s, 1H) ; **^{13}C NMR ($CDCl_3$, 100 MHz):** 22.1 (q). 23.0 (t), 28.0 (t), 37.1 (t), 48.5 (t), 53.6 (t), 74.6 (s), 109.5 (s), 110.7 (d), 112.4 (s), 114.3 (d), 118.1 (d), 119.5 (d), 122.0 (d), 122.6 (d), 123.0 (d), 129.5 (s), 133.4 (s), 134.9 (s), 144.4 (s), 159.8 (s), 175.1 (s) ppm. ESI-MS (*m/z*): 328.16 (100%, $[M+H]^+$), HRMS (ESI+): calcd. for $C_{22}H_{22}N_3$ $[M+H]^+$: 328.1808, found 328.1808.

***N*-Phthalimido 4-(4-methoxy-2-nitrophenyl)but-3-yn-1-amine (12):**



To a solution of the alkyne **6** (1.8 g, 9.05 mmol) and an aryl iodide **5** (3.028 g, 10.85 mmol) in THF (8 mL) and Et_3N (8 mL), were added triphenylphosphine (475 mg, 1.81 mmol) and $Pd(PPh_3)_2Cl_2$ (315 mg, 0.45 mmol) and the mixture was degassed with argon for 10 min. To this CuI (345 mg, 1.81 mmol) was introduced and the mixture was degassed with argon for 10 min and stirred at room temperature for 6 h under argon atmosphere. After completion of the reaction as indicated by TLC, the volatiles are removed under reduced pressure and residue was purified by column chromatography to yield **12** (2.6 g, 82%) as pale yellow solid. R_f = 0.10 (20% ethyl acetate/pet ether); M.P = 118–120 °C; IR ($CHCl_3$) *v*: 3399, 3225, 2952, 1701, 1628, 1316, 1215, 1108, 821, 745 cm^{-1}; **^{1}H NMR ($CDCl_3$, 200 MHz):** δ 2.87 (t, *J* = 7.0 Hz, 2H), 3.84 (s, 3H), 3.96 (t, *J* = 7.1 Hz, 2H), 7.05 (dd, *J* = 8.7, 2.6 Hz, 1H), 7.43 (d, *J* = 2.6 Hz, 1H), 7.45 (d, *J* = 8.6 Hz, 1H), 7.71 (dd, *J* = 5.6, 3.0 Hz, 2H), 7.85 (dd, *J* = 5.7, 3.1 Hz, 2H); **^{13}C NMR ($CDCl_3$,50 MHz):** 19.8 (t), 36.5 (t), 55.9 (q), 77.5 (s), 92.3 (s), 109.0 (d), 110.8 (s), 119.7 (d), 123.4 (d), 132.0 (s), 134.1 (d), 135.8 (d), 150.7 (s), 159.1 (s), 168.2 (s) ppm. ESI-MS (*m/z*): 372.92 (100%, $[M+Na]^+$), HRMS (ESI+): calcd. for $C_{19}H_{14}N_2O_5$, $[M+Na]^+$: 373.0795, found 373.0792.

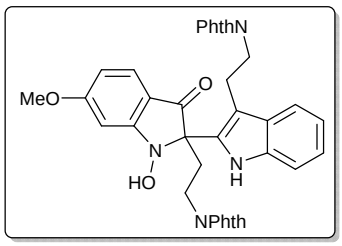
Synthesis of isatogen 3: $PdCl_2$ (13 mg, 0.073 mmol, 5 mol%) was added to a solution of an alkyne **12** (300 mg, 1.5 mmol) in CH_3CN (30 mL), and the mixture was stirred under argon at room temp. for 11 h.



The reaction mixture was concentrated, and the residue obtained was purified by column chromatography (ethyl acetate in petroleum ether) to afford a compound **3** (159 mg, 53% yield) as yellow solid. R_f (40% ethyl acetate/pet. ether) 0.50 ; IR ($CHCl_3$) *v*: 3399, 3225, 2952, 1701, 1628, 1316, 1215, 1108, 821, 745 cm^{-1}; **^{1}H NMR ($CDCl_3$, 200 MHz):** δ 2.87 (t, *J* = 6.1 Hz, 2H),

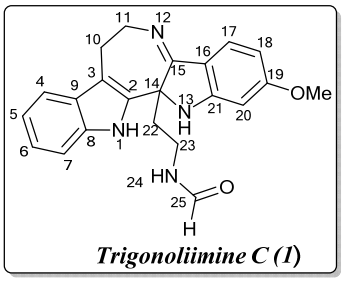
3.90 (s, 3H), 4.02 (t, *J* = 6.1 Hz, 2H), 6.90 (dd, *J* = 8.2, 2.1 Hz, 1H), 7.12 (d, *J* = 2.1 Hz, 1H), 7.45 (d, *J* = 8.1 Hz, 1H), 7.69 (dd, *J* = 5.4, 3.1 Hz, 2H), 7.78 (dd, *J* = 5.7, 3.0 Hz, 2H); **^{13}C NMR ($CDCl_3$, 125 MHz):** 21.3 (t), 34.1 (t), 56.3 (q), 101.5 (d), 114.9 (d), 115.3 (d), 123.3 (d, 2C), 123.4 (d), 132.0 (s, 2C), 133.9 (d, 2C), 137.1 (s), 150.1 (s), 165.3 (s), 168.2 (s, 2C), 185.3 (s) ppm. ESI-MS (*m/z*): 373.07 (100%, $[M+Na]^+$), 389.08 (5%, $[M+K]^+$), HRMS (ESI+): calcd. for $C_{19}H_{14}N_2O_5$, $[M+Na]^+$: 373.0795, found 373.0791.

Preparation of indoxyl (2): The addition of tryptamine **4** (140 mg, 0.48 mmol) to isatogen **3** (140 mg, 0.40 mmol) was carried out following the general procedure **A** to obtain indoxyl **2** as yellow solid (182 mg, 71%); R_f = 0.3 (40% ethyl acetate/pet. ether); M.P = 227–228 °C; IR ($CHCl_3$) *v*: 3389, 3235, 2963, 1701, 1628, 1361, 1223, 1118, 825, 725 cm^{-1}; **^{1}H NMR ($CDCl_3$, 400 MHz):** δ 2.61–2.68 (m, 1H), 2.70–2.78 (m, 1H), 3.11 (m, 1H), 3.22–3.25 (m, 1H), 3.84–3.88 (m, 3H), 3.96 (s, 3H), 4.07 (m, 1H), 6.65 (d, *J* = 8.3 Hz, 1H), 6.92 (s, 1H), 6.99 (app td, *J* = 7.3 Hz, 1H), 7.08 (app td, *J* = 7.3 Hz, 1H), 7.35 (d, *J* = 8.1 Hz, 1H), 7.53 (d, *J* = 9.0 Hz, 1H), 7.56 (d, *J* = 9.3 Hz, 1H), 7.63 (dd, *J* = 5.1, 3.0 Hz, 2H), 7.72 (dd, *J* = 5.1, 3.0 Hz, 2H), 7.74 (dd, *J* = 5.1, 3.1 Hz, 2H), 7.84 (dd, *J* = 5.1, 3.4 Hz, 2H), 9.01 (s, 1H), 9.69 (s, 1H) ; **^{13}C NMR ($CDCl_3$,100 MHz):** 23.6 (t), 34.4 (t), 34.7 (t), 38.7 (t), 55.9 (q), 75.4 (s), 97.2 (d), 109.1 (s), 111.2 (d), 112.8 (d), 113.8 (s), 117.9 (d), 119.5 (d), 122.1 (d), 123.1 (d, 2C), 123.4 (d, 2C), 125.7 (d), 128.4 (s), 131.0 (s), 131.9 (s, 4C), 133.8 (d, 2C), 134.2 (d, 2C), 135.4 (s), 165.4 (s), 168.0 (s, 2C), 168.3 (s), 169.2 (s, 2C), 195.9 (s) ppm. ESI-MS (*m/z*): 663.23 (100%, $[M+Na]^+$), 679.19 (50%, $[M+K]^+$), HRMS (ESI+): calcd. for $C_{37}H_{29}N_4O_7$ $[M+H]^+$: 641.2031, found 641.2030.



Synthesis of (±)-Trigonoliimine C (1): The general procedure B has been followed for the phthalimide deprotection/N–O reduction of indoxyl **2** (100 mg, 0.15 mmol) with hydrazine monohydrate (78 mg, 1.56 mmol) and then procedure D was followed for the cyclization of resulting amine using $Ti(O^iPr)_4$ (88 mg, 0.31 mmol) followed by usual workup and purification column chromatography (neutral silica gel, 10% MeOH and 0.2% NH_4OH in CH_2Cl_2 as eluent) gave the cyclized compound (20 mg) which was subjected for *N*-formylation[1,2] immediately using freshly prepared *N*-formyl benzotriazole (8.5 mg, 0.06 mmol, 1eq) and THF (1ml) as a solvent to provide (±)-Trigonoliimine C (18 mg, 31% yield over 3 steps); IR ($CHCl_3$) *v*: 3272, 2925, 2854, 1734, 1621, 1457, 1338, 1289, 1166, 1121, 825, 744 cm^{-1}; **^{1}H NMR [(500MHz, CD_3OD:$CDCl_3$ (3:1)]:** δ 2.45–2.5 (m, 1H), 2.61–2.67 (m, 1H), 3.03 (td, *J* = 16.8, 13.4, 3.4 Hz, 1H), 3.12 (app dt, *J* = 16.9 Hz, 1H), 3.20–3.29 (m, 2H), 3.79 (s, 3H), 3.98 (app dt, *J* = 12.7, 3.4 Hz, 1H), 4.33 (app td, *J* = 12.7, 2.4 Hz, 1H), 6.29 (d, *J* = 2.0 Hz, 1H), 6.31 (dd, *J* = 8.6, 1.5 Hz, 1H),7.02 (t, *J* = 7.1 Hz, 1H), 7.12 (app td, *J* = 7.1 Hz, 1H), 7.32 (d, *J* = 8.1 Hz, 1H), 7.41 (d, *J* = 8.3 Hz, 1H), 7.49 (d, *J* = 8.8 Hz, 1H),7.92 (s, 1H); **^{13}C NMR [(100MHz, CD_3OD:$CDCl_3$ (3:1)]:** δ 24.7 (t), 35.0 (t), 40.4 (t), 47.2 (t), 56.2 (q), 68.9 (s), 95.3 (d), 109.6 (s), 110.5 (d), 111.8 (d), 115.6 (d), 118.8 (d), 120.0 (d), 123.1 (d), 125.7 (s), 129.5 (s), 131.0 (s), 136.7 (s), 160.4 (s), 163.8 (d), 168.0 (s), 173.2 (s), ppm; ESI-MS (*m/z*): 375.02 (100%, $[M+H]^+$), HRMS (ESI+): calcd. for $C_{22}H_{23}N_4O_2$, $[M+H]^+$: 375.1816, found 375.1814.

1. R. Katritzky, H. X. Chang and B. Z. Yang, *Synthesis*, 1995, 503–505.
2. S. Han and M. Movassaghi, *J. Am. Chem. Soc.*, 2011, **133**, 10768–10771.



Trigonoliimine C (1)

Comparison of ^{13}C NMR Spectra for Synthetic Trigonoliimine C and Natural Trigonoliimine C

Carbon	δ_C Synthesized CD_3OD:$CDCl_3$(3:1)	δ_C reported by UK Tambar[3] CD_3OD:$CDCl_3$	δ_C reported by Hao[4] CD_3OD:$CDCl_3$
1 (*NH*)	-	-	-
2	131.03	131.7	131.4
3	110.49	110.5	110.3
4	118.77	118.8	118.5
5	120.05	119.96	119.7
6	123.07	122.93	122.7
7	111.83	111.73	111.5
8	136.72	136.85	136.3
9	129.51	129.69	129.2
10	24.68	24.59	24.3
11	47.19	47.68	47.5
13(*NH*)	-	-	-
14	68.87	68.62	68.1
15	173.2	175.57	174.9
16	115.61	116.62	116.5
17	125.75	125.5	125.2
18	109.65	108.93	108.4
19	167.98	167.45	166.8
20	95.28	95.57	95.3
21	160.40	159.73	159.0
22	40.38	40.53	40.2
23	35.03	35.08	34.9
24(*NH*)	-	-	-
25	163.76	163.78	163.4
OMe	56.17	55.98	55.8

3) X. B. Qi, H. L. Bao, and U. K. Tambar, *J. Am. Chem. Soc.*, 2011, **133**, 10050
4) C. J. Tan, Y. T. Di, Y. H. Wang, Y. Zhang, Y. K. Si, Q. Zhang, S. Gao, X. J. Hu, X. Fang, S. F. Li and X. J. Hao, *Org. Lett.*, 2010, **12**, 2370.

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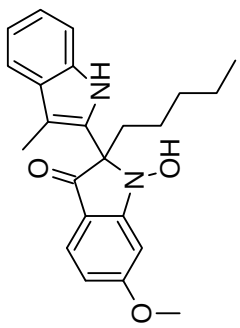
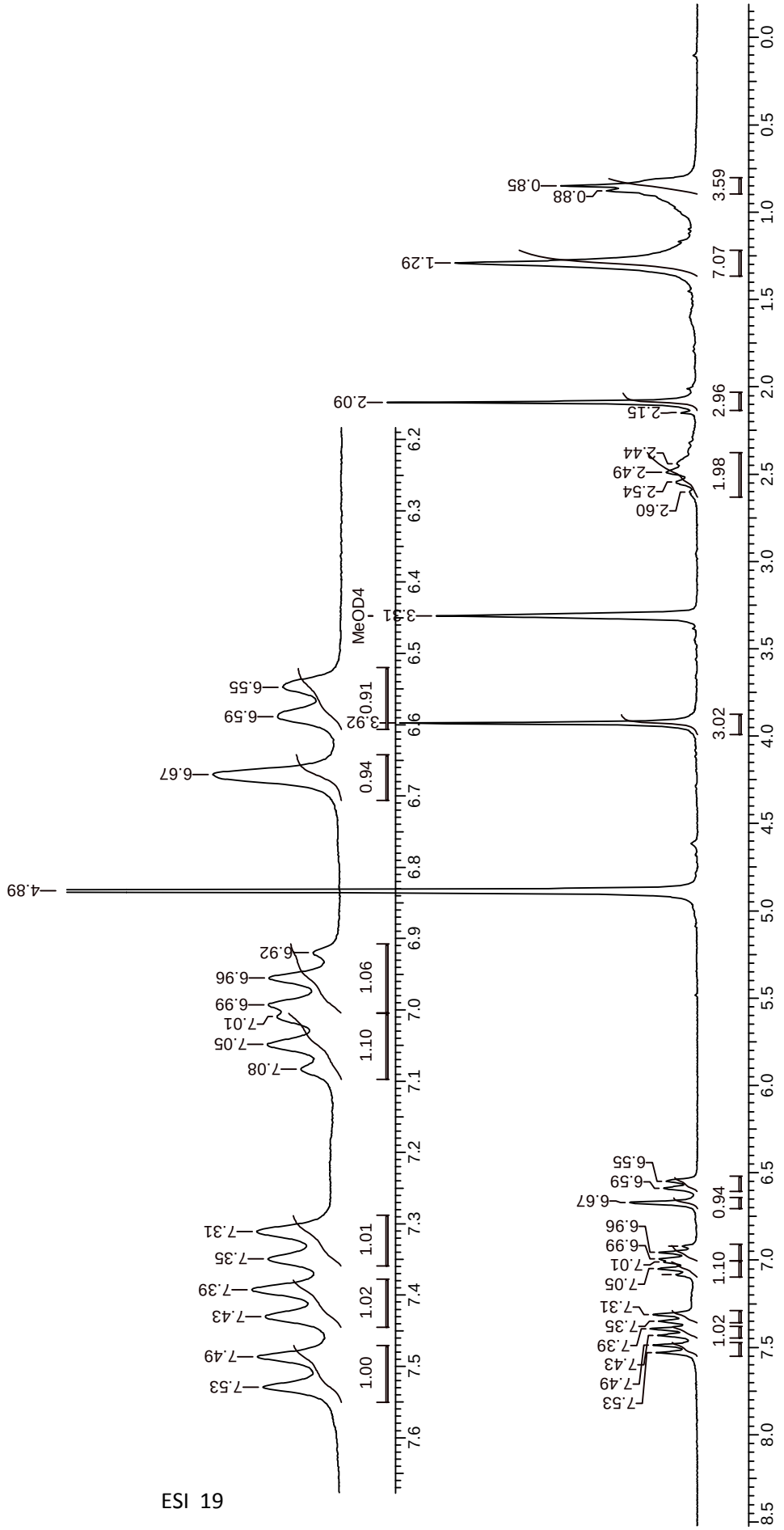
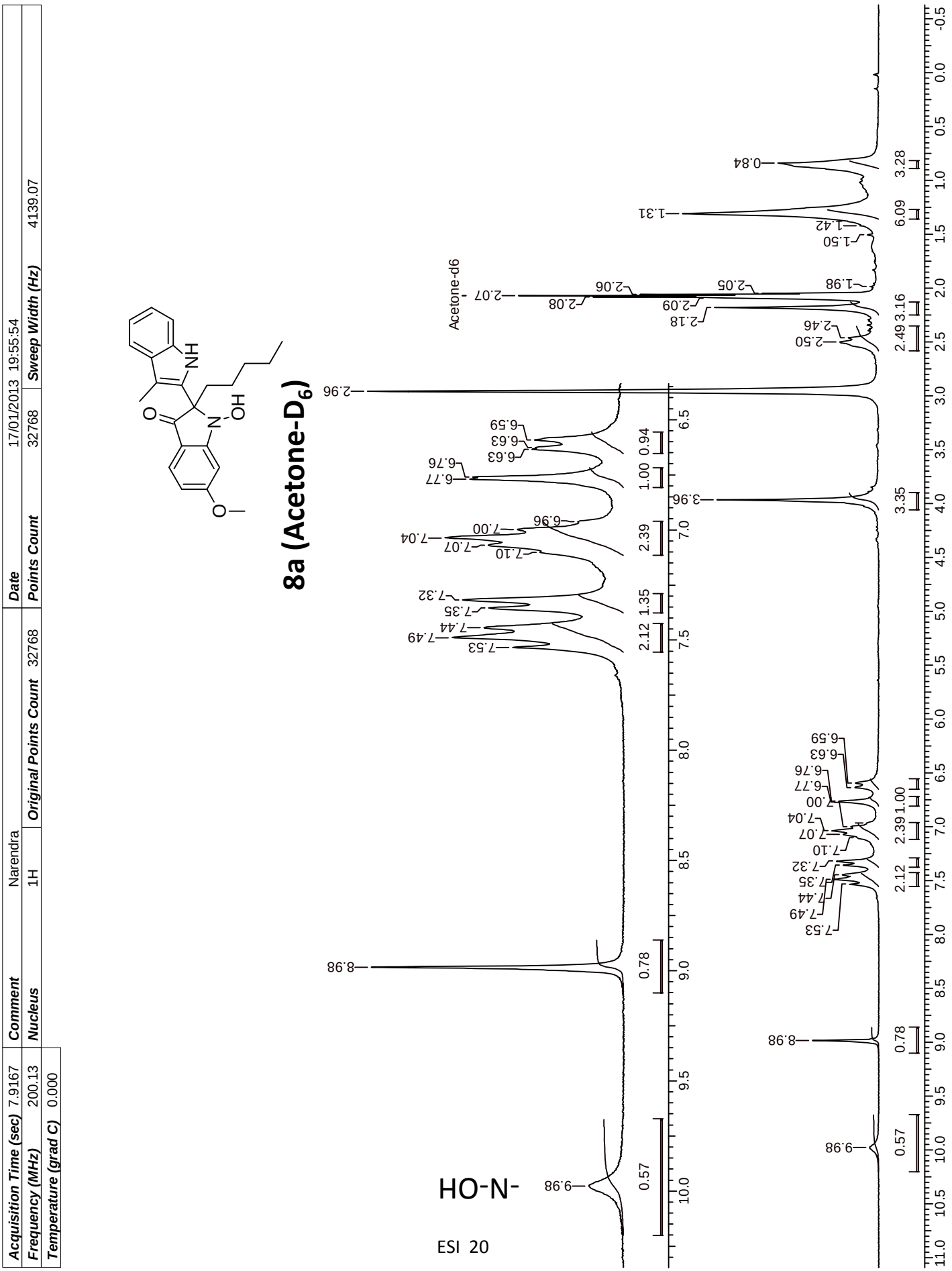


8a (CD_3OD)

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Narendra
Original Points Count 32768
Date 17/01/2013 19:55:54
Points Count 32768
Sweep Width (Hz) 4139.07
8a (Acetone-D6)
HO-N-
ESI 20
Acetone-d6

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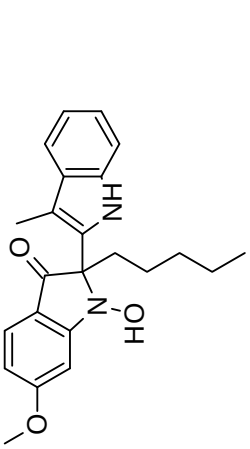
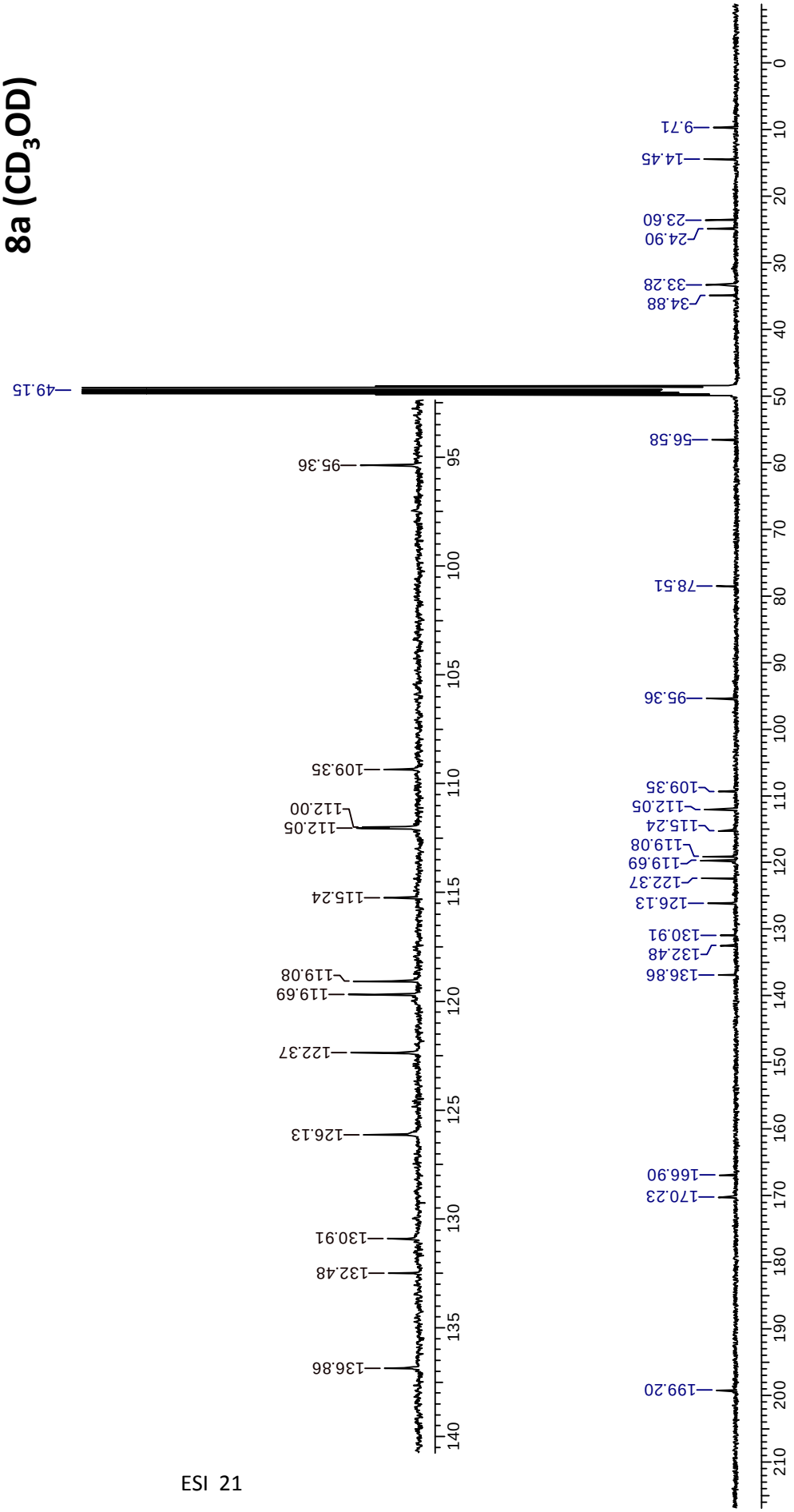
Frequency (MHz)	100.53		
Solvent	METHANOL-D3	***Points Count***	26214



8a (CD_3OD)



Acquisition Time (sec)	1.0434	*Comment*	n reddy	*Date*	16 Jan 2013 13:59:37
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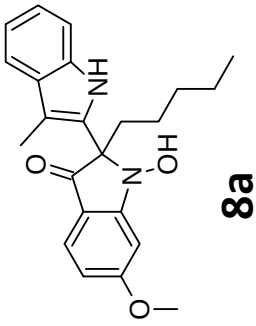
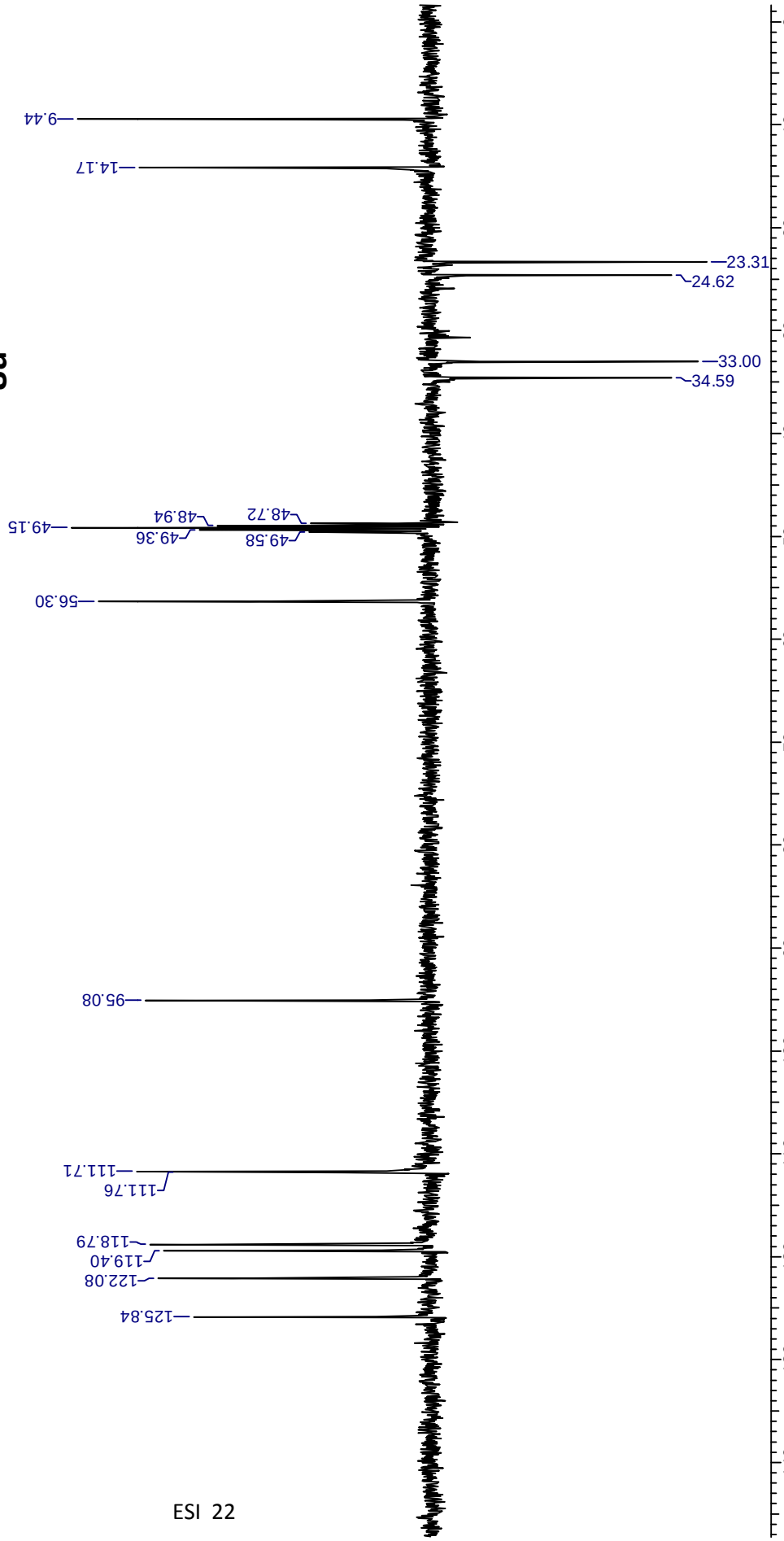
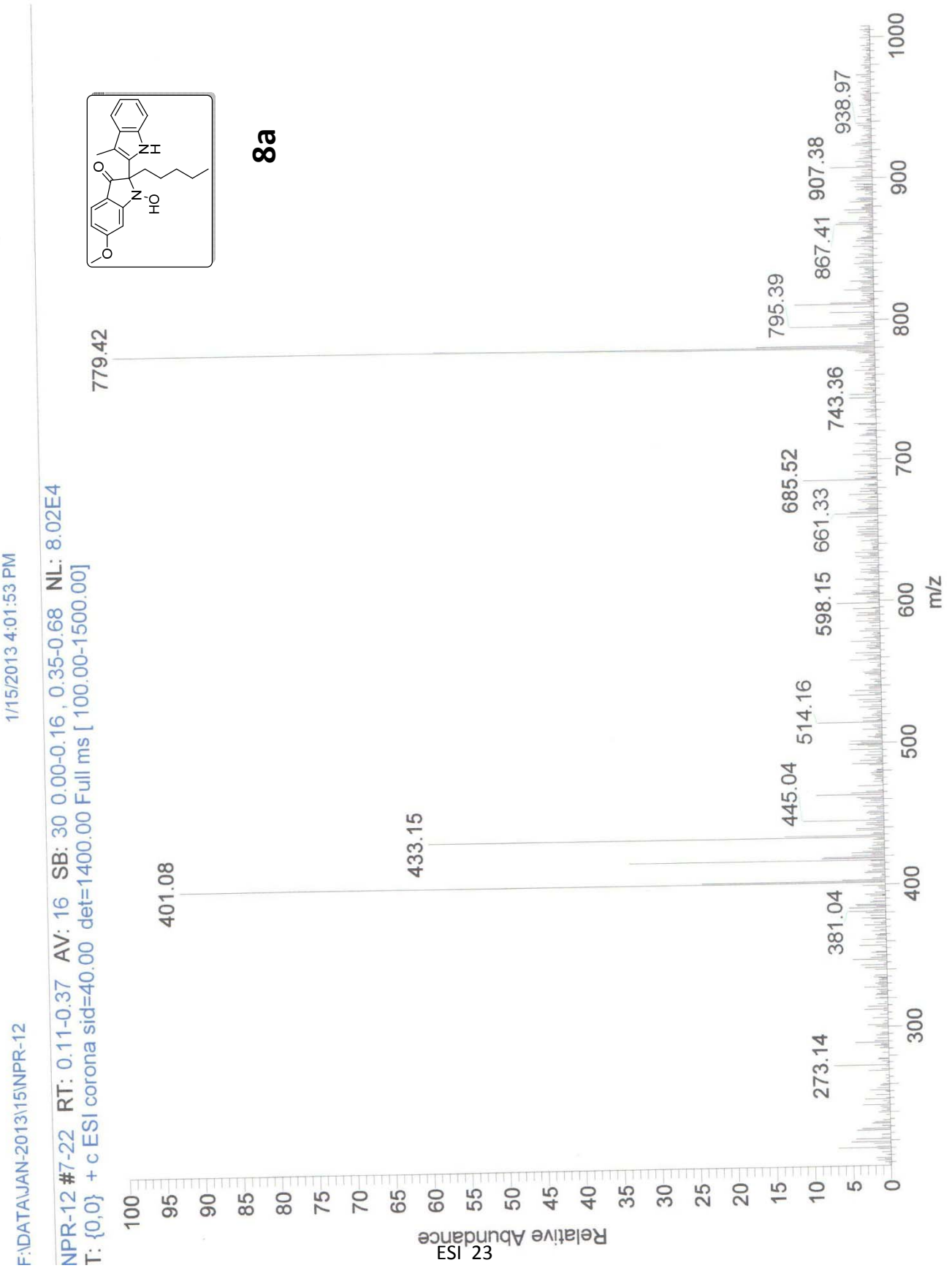
Sweep Width (Hz)	25124.29			*Temperature (grad C)*	20.300

Frequency (MHz)	100.53		
Solvent	METHANOL-D3		
Points Count	26214		



8a

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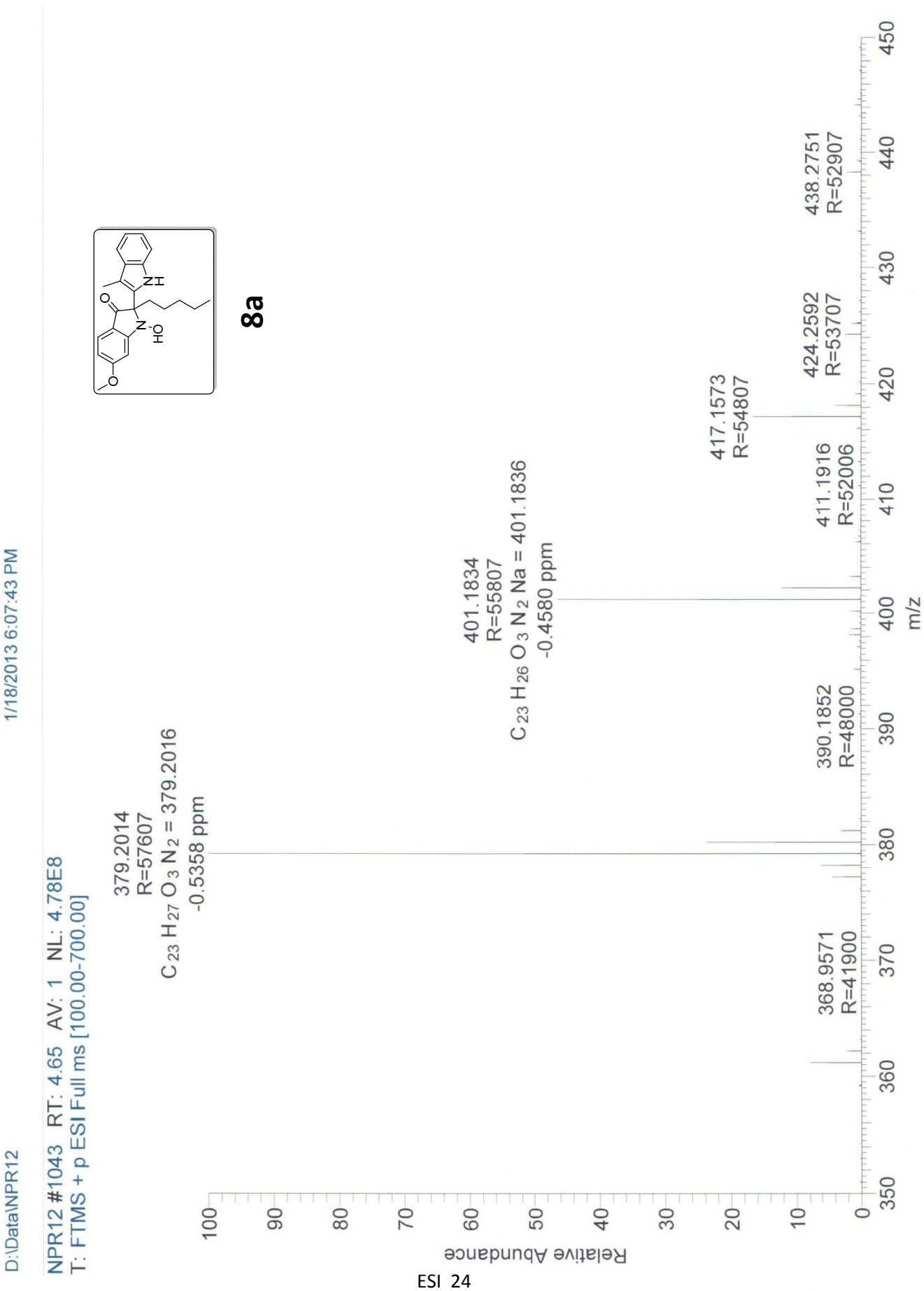
T: {0,0} + c ESI corona sid=40.00 det=1400.00 Full ms [100.00-1500.00]
8a
Relative Abundance
m/z
273.14
381.04
401.08
433.15
445.04
514.16
598.15
661.33
685.52
743.36
779.42
795.39
867.41
907.38
938.97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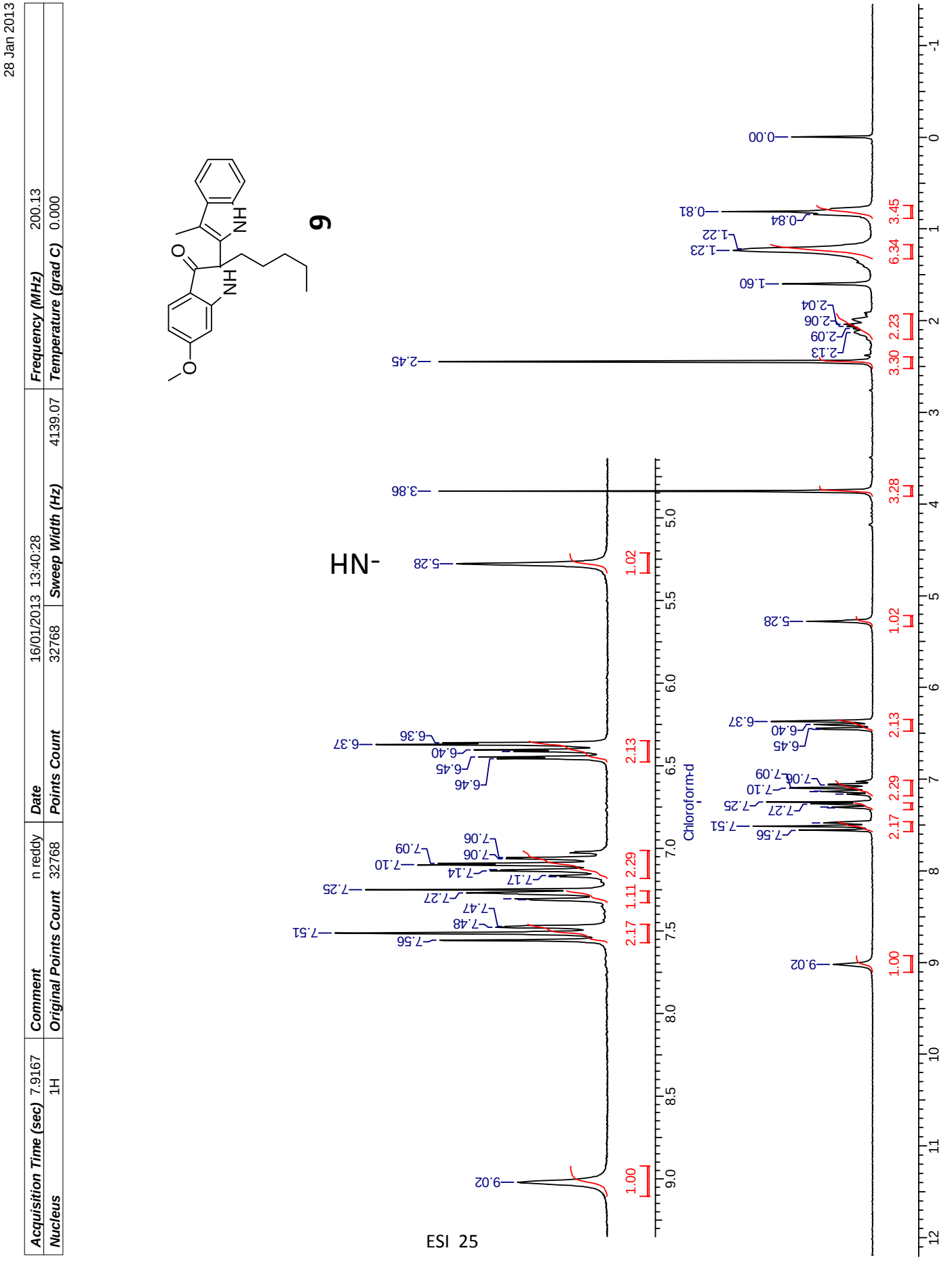
C 23 H 27 O 3 N 2 = 379.2016
-0.5358 ppm
401.1834
R=55807
C 23 H 26 O 3 N 2 Na = 401.1836
-0.4580 ppm
368.9571
R=41900
390.1852
R=48000
411.1916
R=52006
417.1573
R=54807
424.2592
R=53707
438.2751
R=52907
8a
Relative Abundance
m/z


28 Jan 2013
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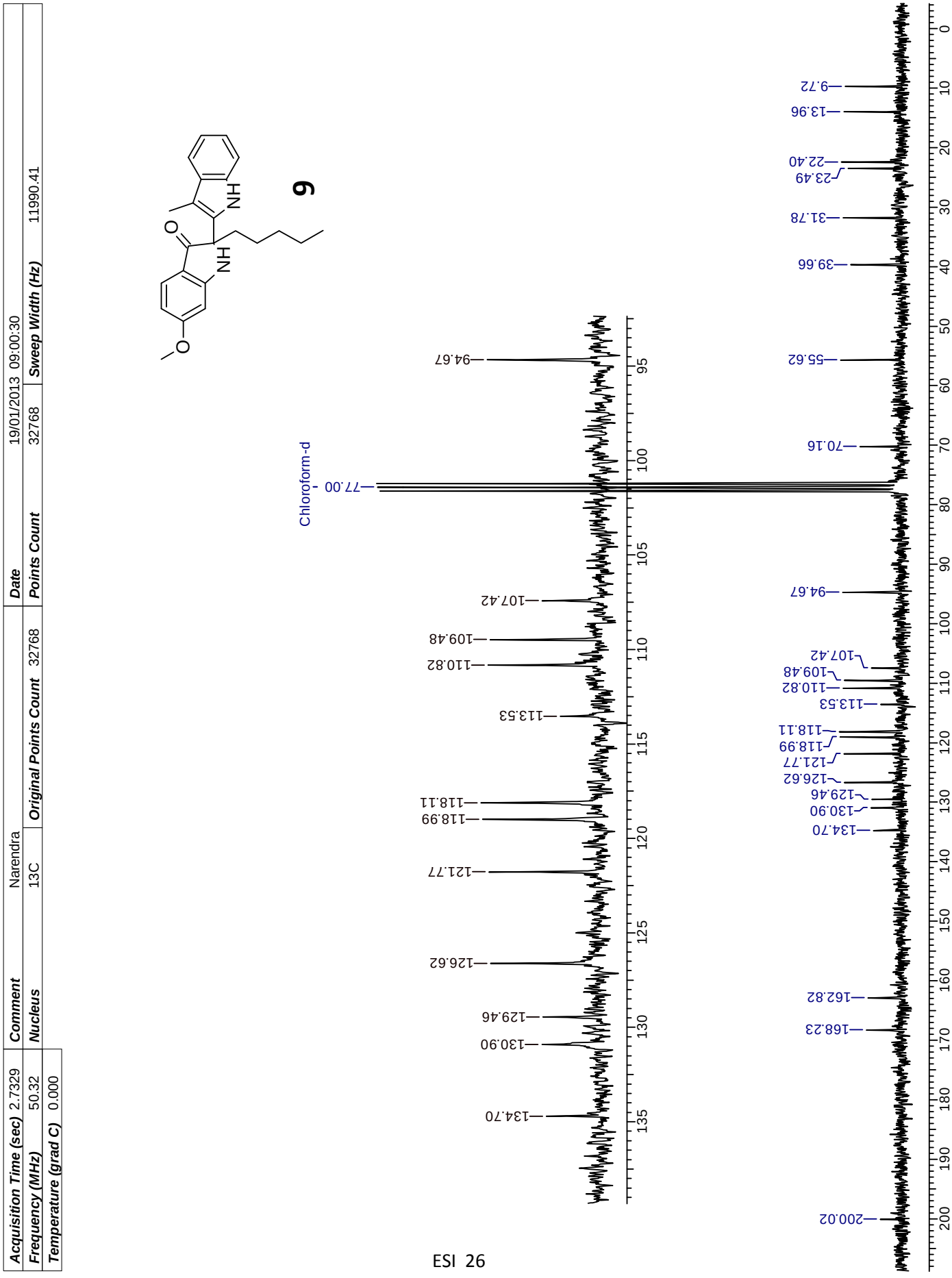
Comment n reddy
Original Points Count 32768
Date 16/01/2013 13:40:28
Points Count 32768
Sweep Width (Hz) 4139.07
Frequency (MHz) 200.13
Temperature (grad C) 0.000
NH
NH
O
O
6
HN
Chloroform-d
ESI 25


Acquisition Time (sec) 2.7329
Frequency (MHz) 50.32
Temperature (grad C) 0.000
Comment
Nucleus
Narendra
13C
Original Points Count 32768
Date
Points Count
19/01/2013 09:00:30
32768
Sweep Width (Hz)
11990.41
9
NH
NH
O
O
Chloroform-d
77.00
94.67
107.42
109.48
110.82
113.53
118.11
118.99
121.77
126.62
129.46
130.90
134.70
9.72
13.96
22.40
23.49
31.78
39.66
55.62
70.16
94.67
107.42
109.48
110.82
113.53
118.11
118.99
121.77
126.62
129.46
130.90
134.70
162.82
168.23
200.02

28 Jan 2013

Acquisition Time (sec) 2.7329	***Comment***	Narendra	***Date***	19/01/2013 08:21:46	
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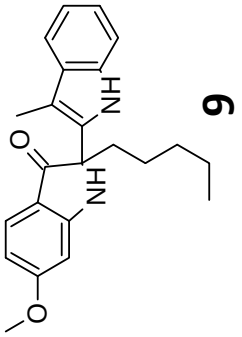
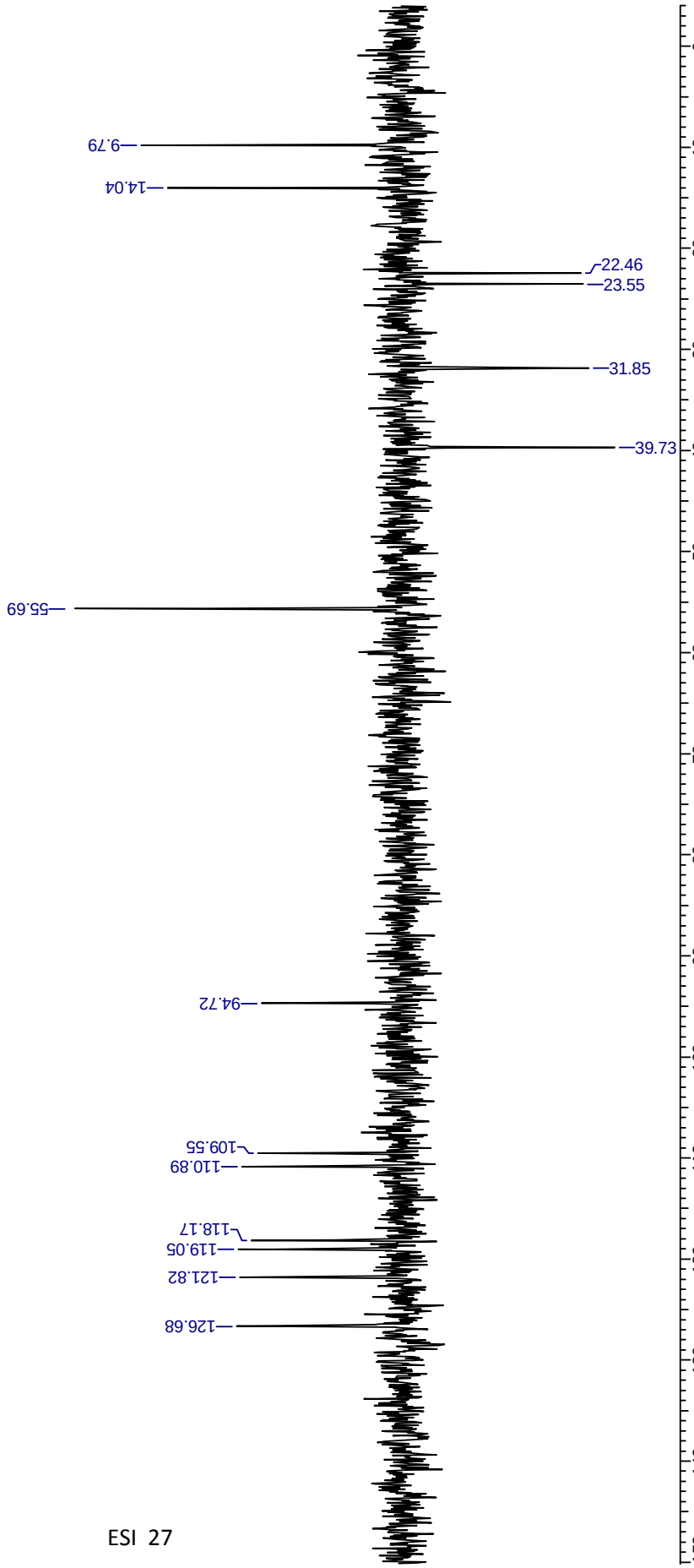
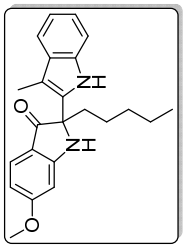
Temperature (grad C) 0.000					



6





9

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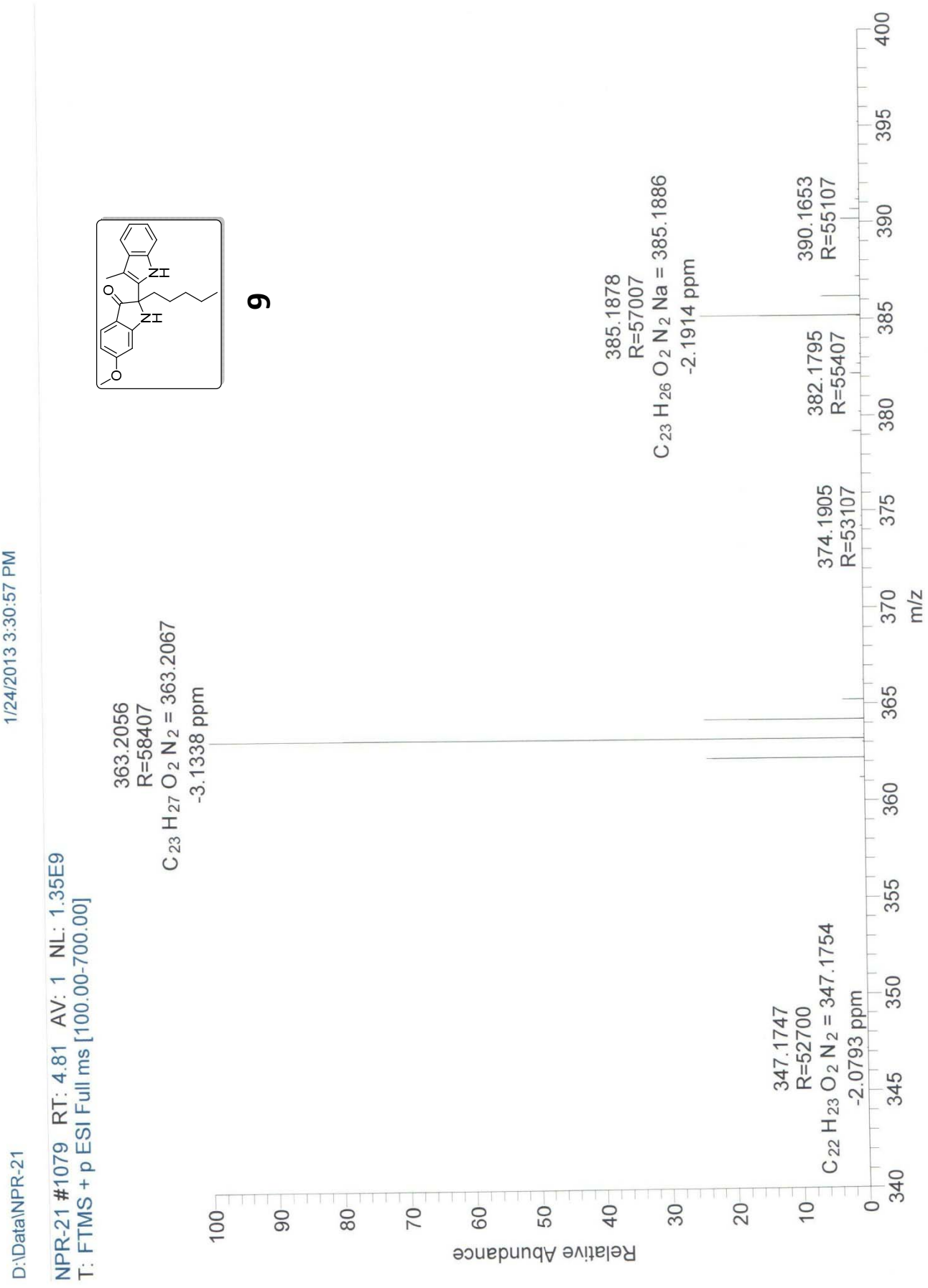
Relative Abundance
m/z
385.01
417.09
433.10
442.13
449.10
512.11
544.16
632.21
685.36
763.41
779.33
829.70
892.42
232.02
279.00
365.04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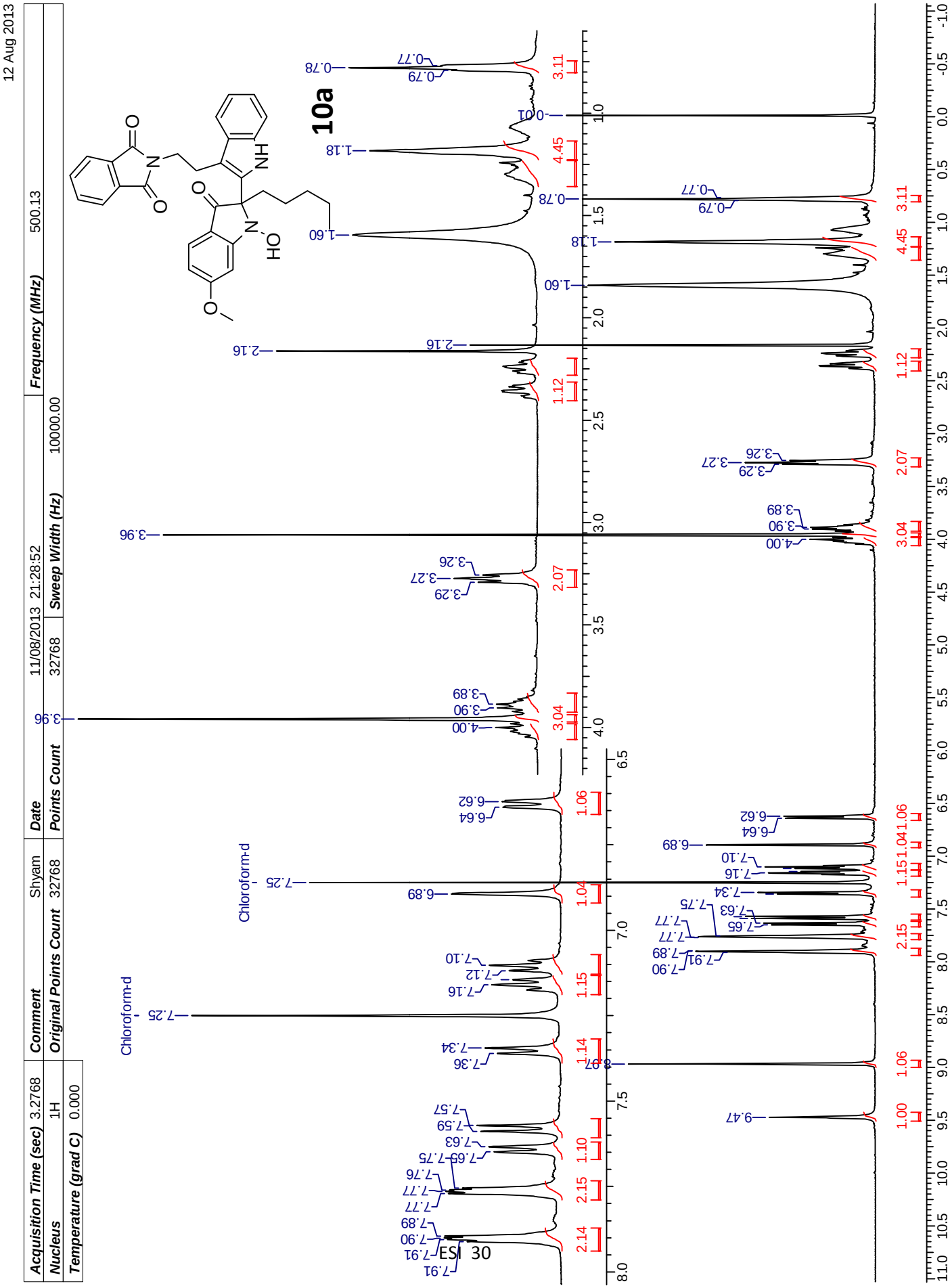
T: FTMS + p ESI Full ms [100.00-700.00]
363.2056
R=58407
C 23 H 27 O 2 N 2 = 363.2067
-3.1338 ppm
347.1747
R=52700
C 22 H 23 O 2 N 2 = 347.1754
-2.0793 ppm
374.1905
R=53107
382.1795
R=55407
385.1878
R=57007
C 23 H 26 O 2 N 2 Na = 385.1886
-2.1914 ppm
390.1653
R=55107
Relative Abundance
m/z
9


12 Aug 2013
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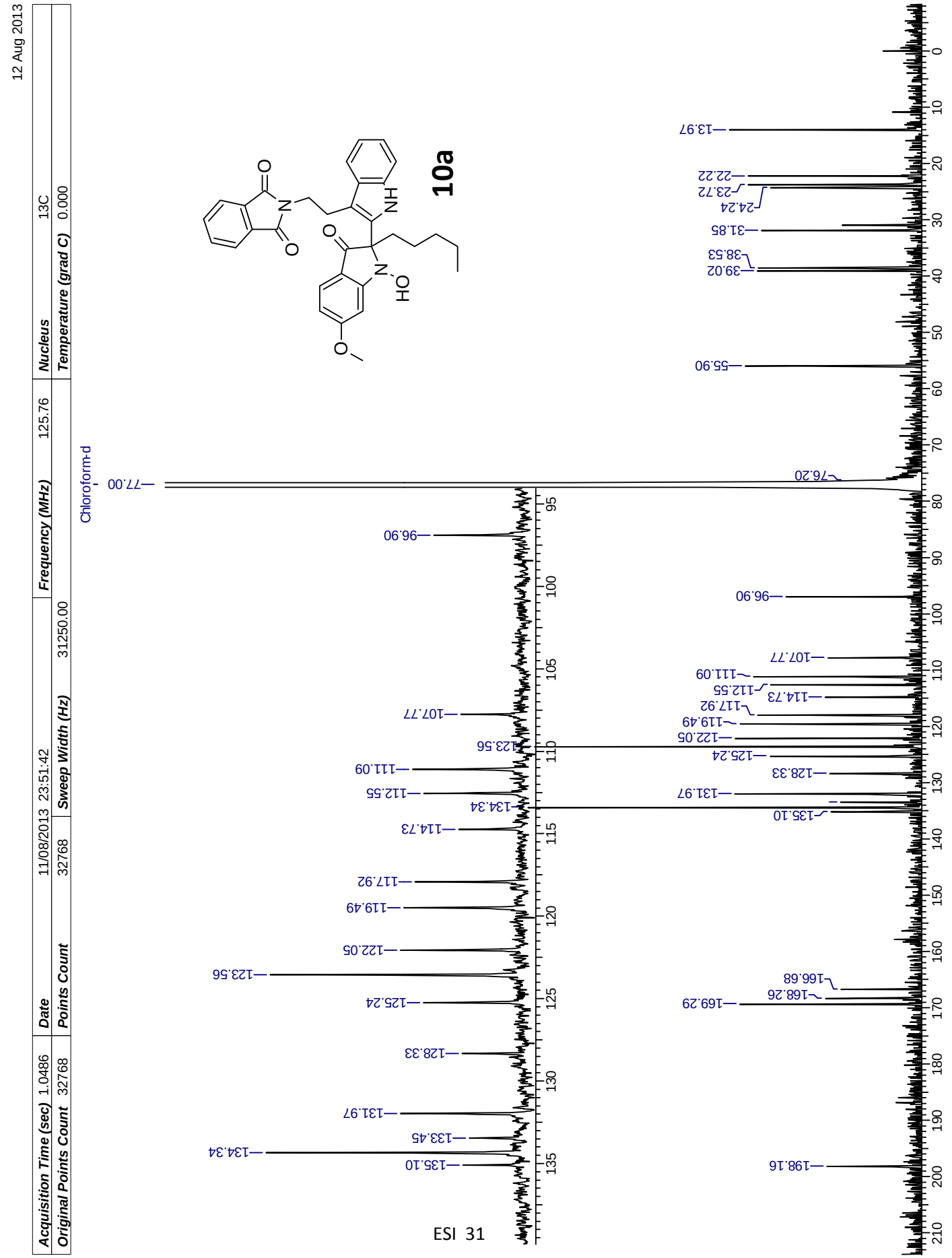
Nucleus 1H
Temperature (grad C) 0.000
Comment
Original Points Count 32768
Shyam
Date 11/08/2013 21:28:52
Points Count 32768
Sweep Width (Hz) 10000.00
Frequency (MHz) 500.13
10a
Chloroform-d
Chloroform-d
ESI 30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Date 11/08/2013 23:51:42
Points Count 32768
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Frequency (MHz) 125.76
Nucleus 13C
Temperature (grad C) 0.000
10a
Chloroform-d
77.00
13.97
22.22
23.72
24.24
31.85
38.53
39.02
55.90
76.20
96.90
107.77
111.09
112.55
114.73
117.92
119.49
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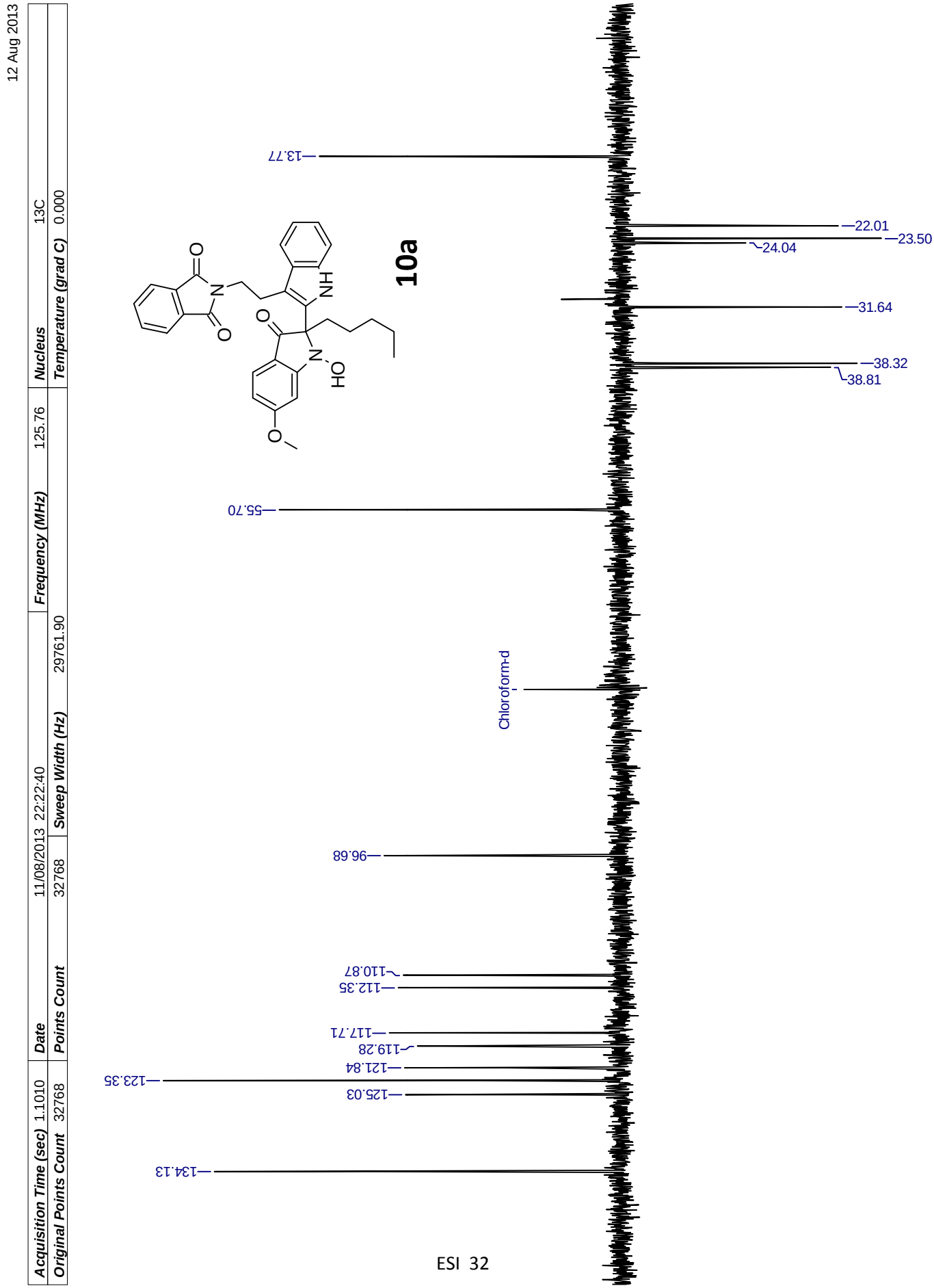
134.34
135.10
166.68
168.26
169.29
198.16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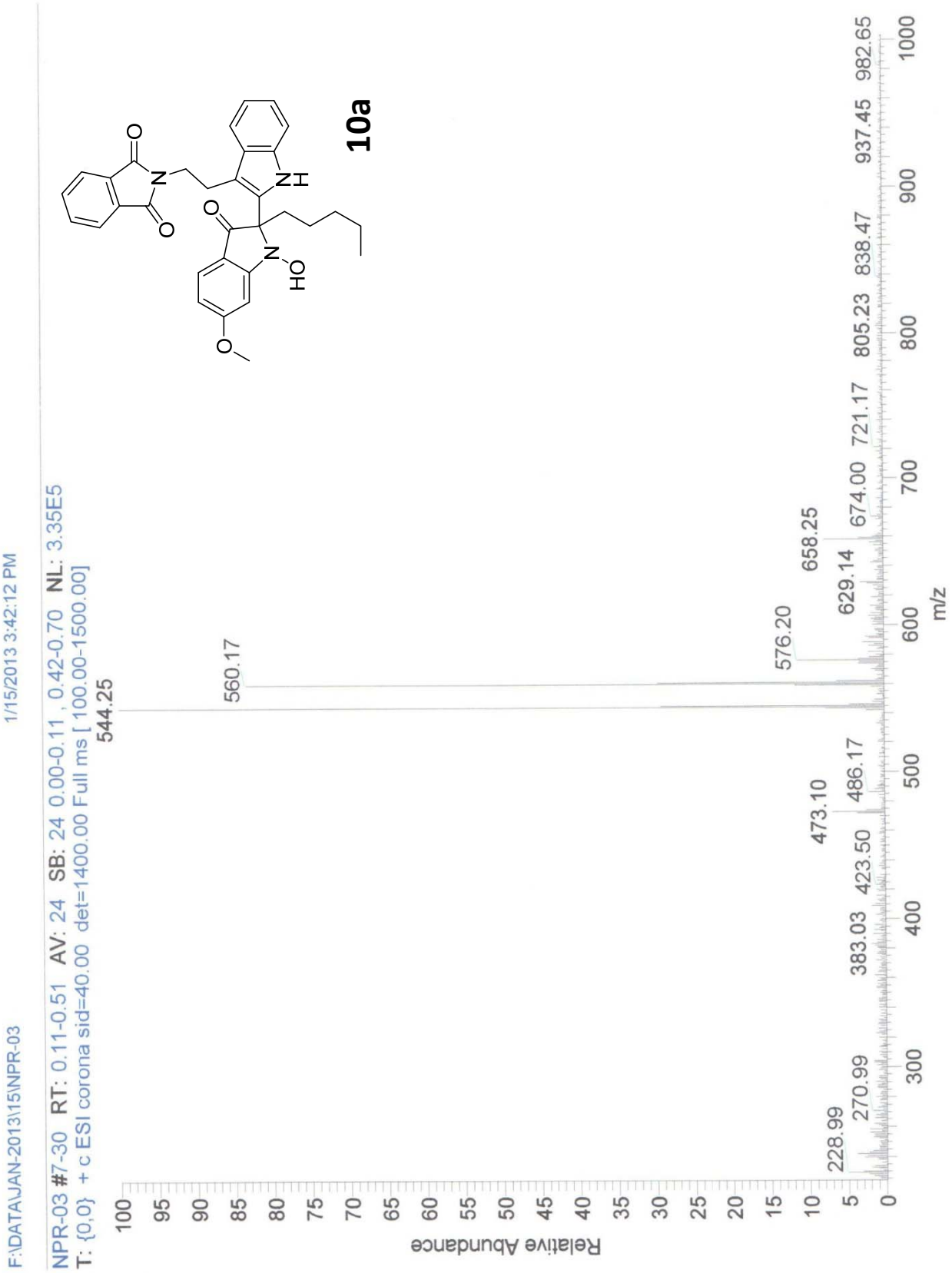
Date 11/08/2013 22:22:40
Points Count 32768
Sweep Width (Hz) 29761.90
Frequency (MHz) 125.76
Nucleus 13C
Temperature (grad C) 0.000
10a
13.77
22.01
23.50
24.04
31.64
38.32
38.81
55.70
Chloroform-d
96.68
110.87
112.35
117.71
119.28
121.84
123.35
125.03
134.13
0
10
20
30
40
50
60
70
80
90
100
110
120
130
140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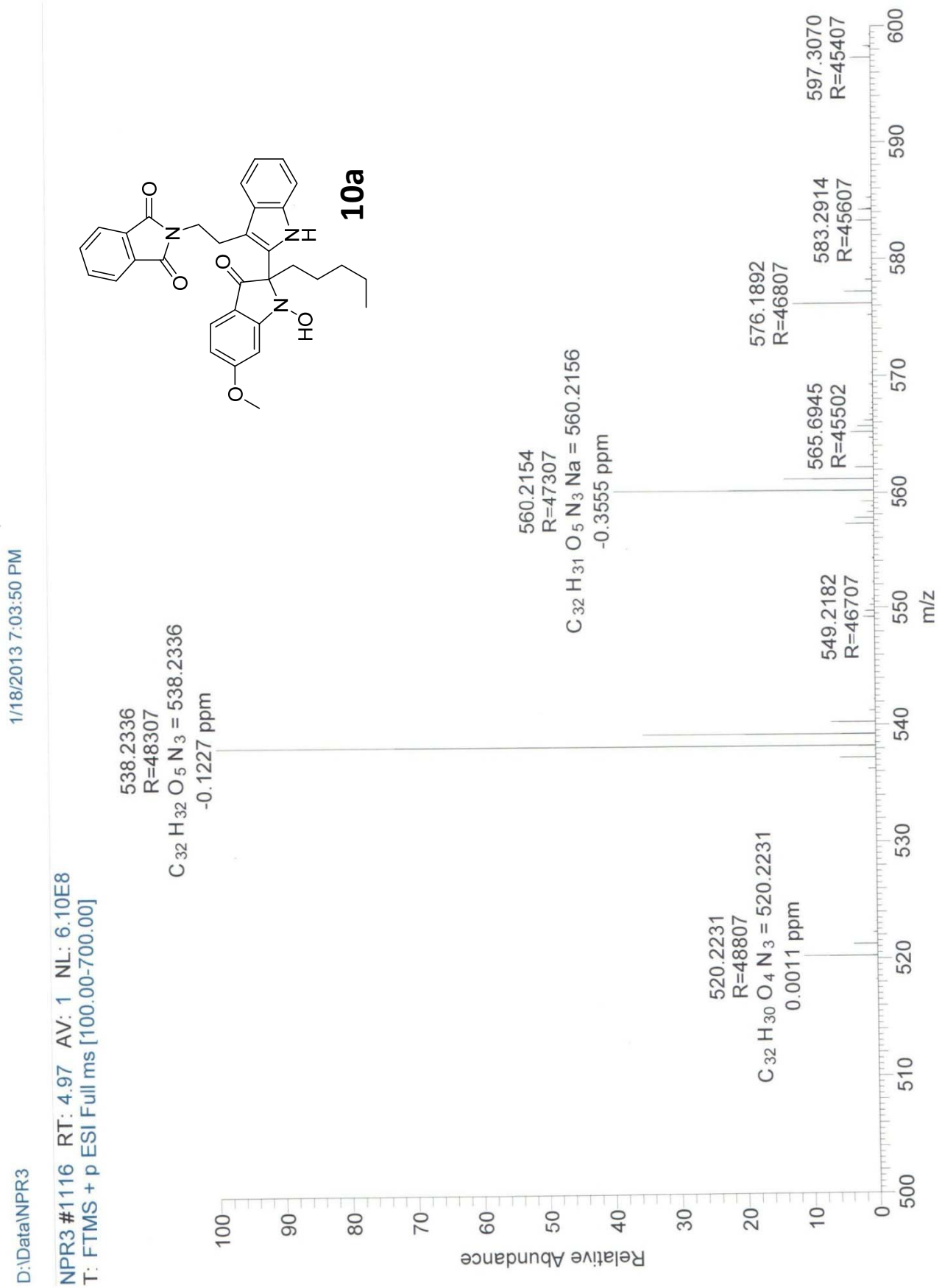
T: {0,0} + c ESI corona sid=40.00 det=1400.00 Full ms [100.00-1500.00]
544.25
560.17
576.20
658.25
629.14
674.00
721.17
805.23
838.47
937.45
982.65
473.10
486.17
423.50
383.03
270.99
228.99
Relative Abundance
m/z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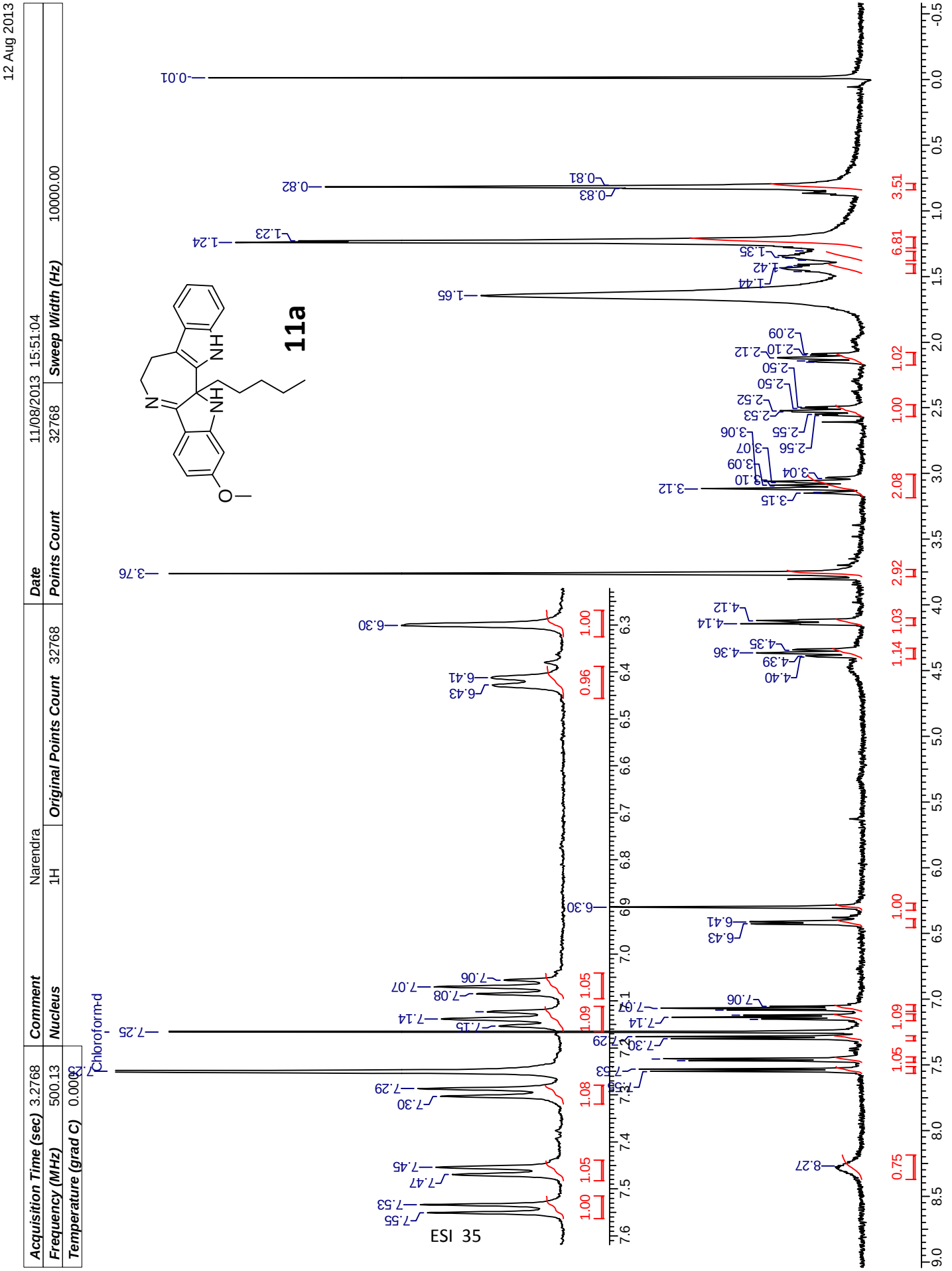
538.2336
R=48307
C 32 H 32 O 5 N 3 = 538.2336
-0.1227 ppm
560.2154
R=47307
C 32 H 31 O 5 N 3 Na = 560.2156
-0.3555 ppm
520.2231
R=48807
C 32 H 30 O 4 N 3 = 520.2231
0.0011 ppm
549.2182
R=46707
565.6945
R=45502
576.1892
R=46807
583.2914
R=45607
597.3070
R=45407
Relative Abundance
m/z


12 Aug 2013
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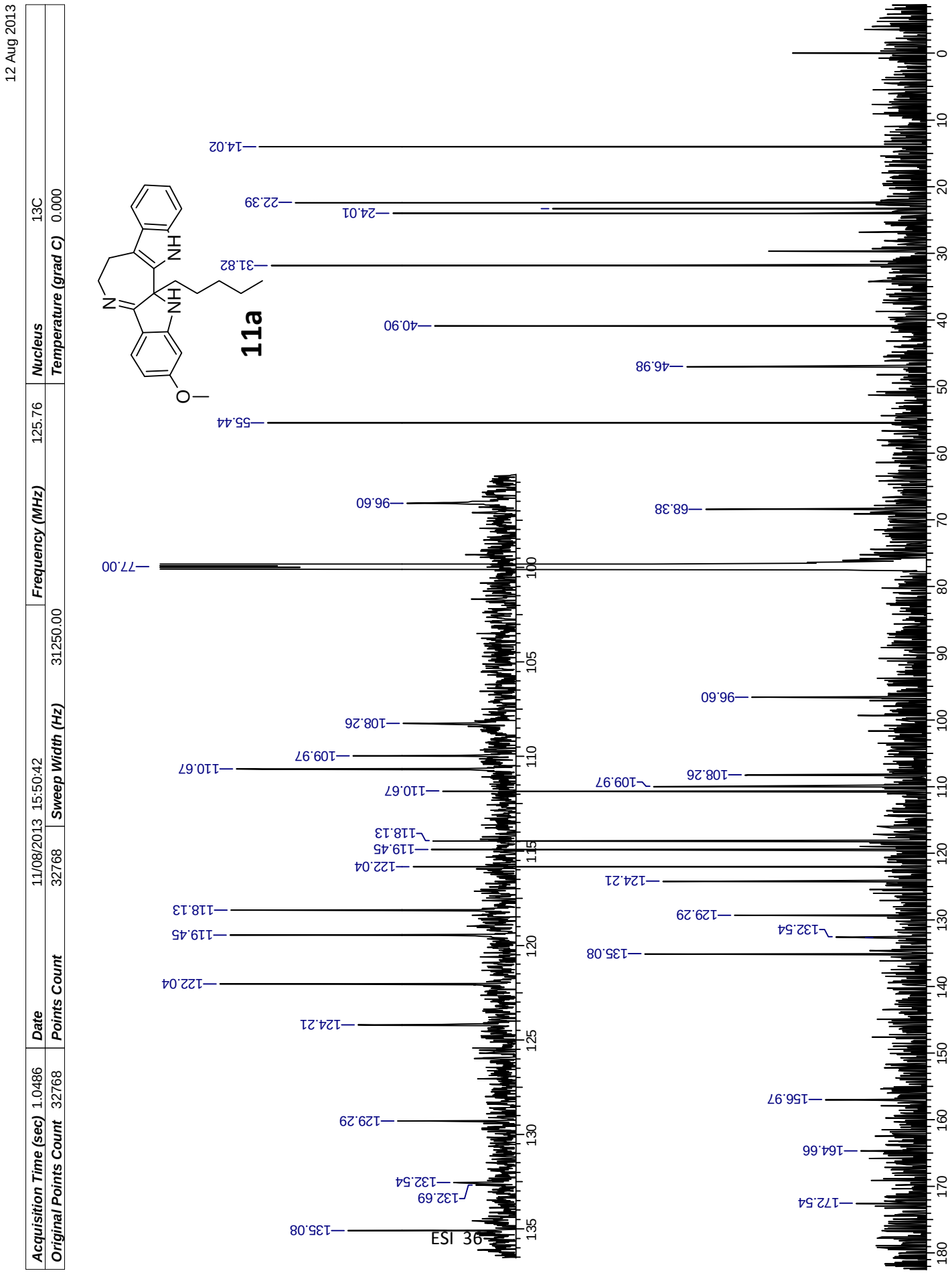
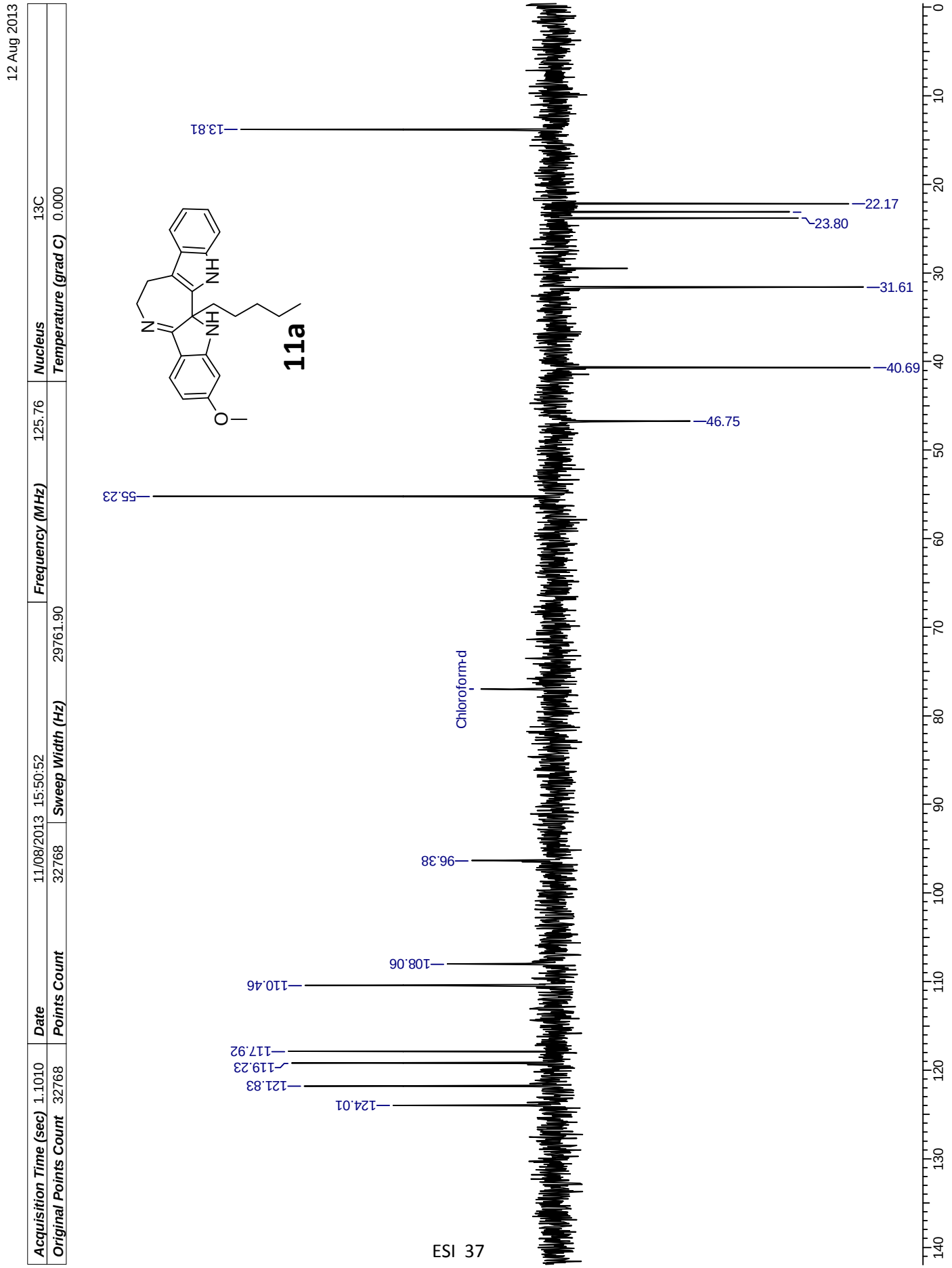
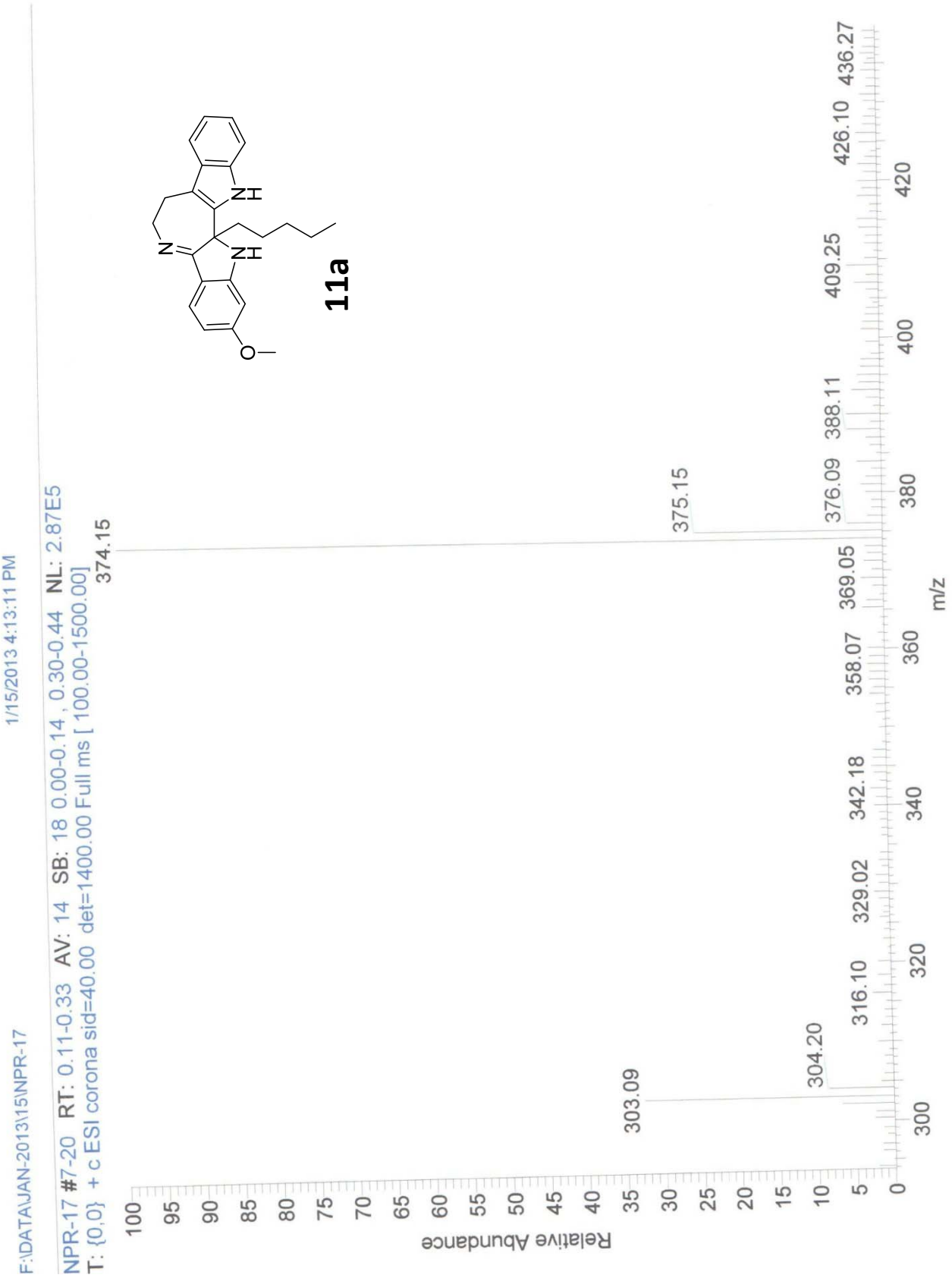
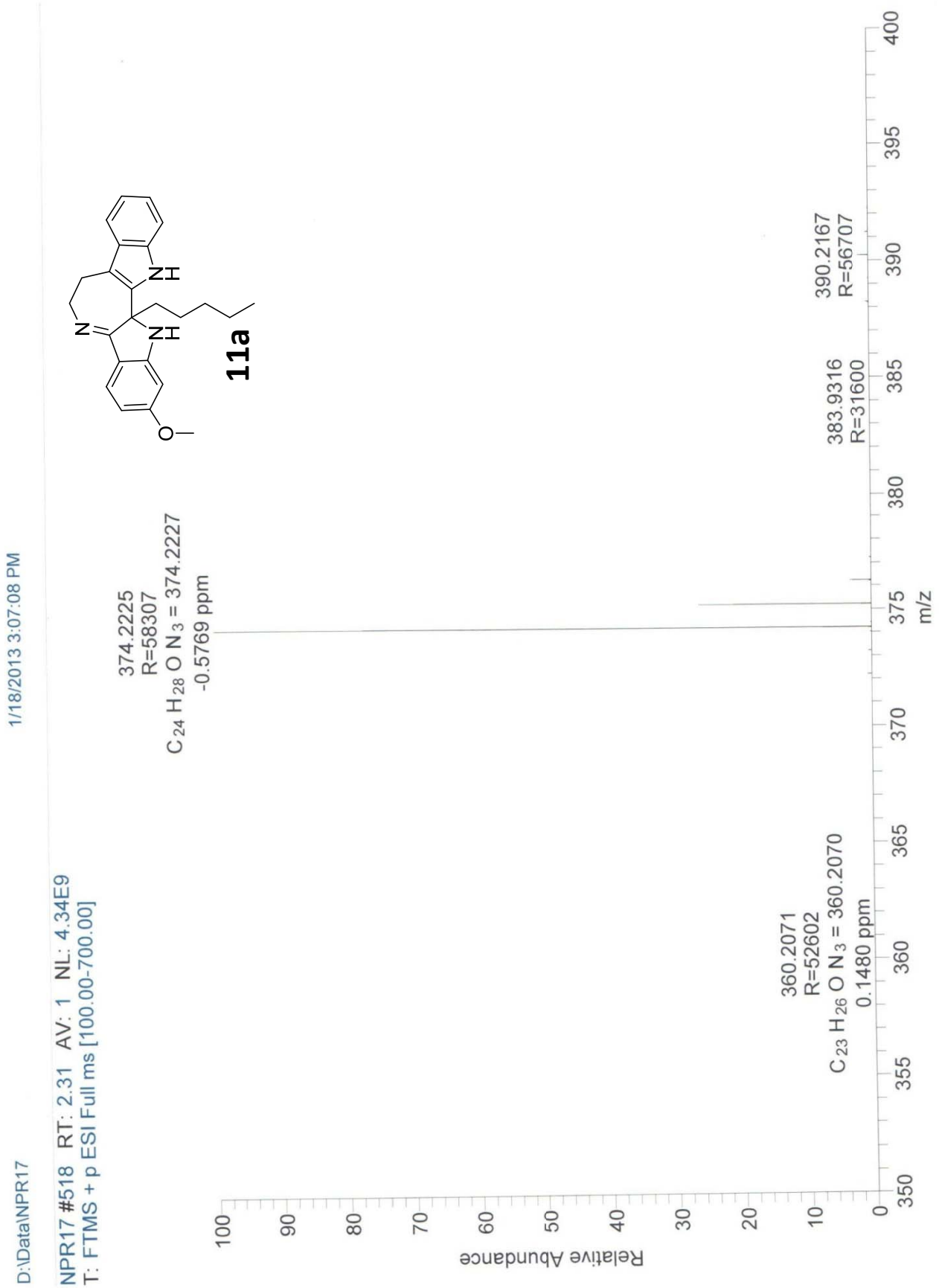
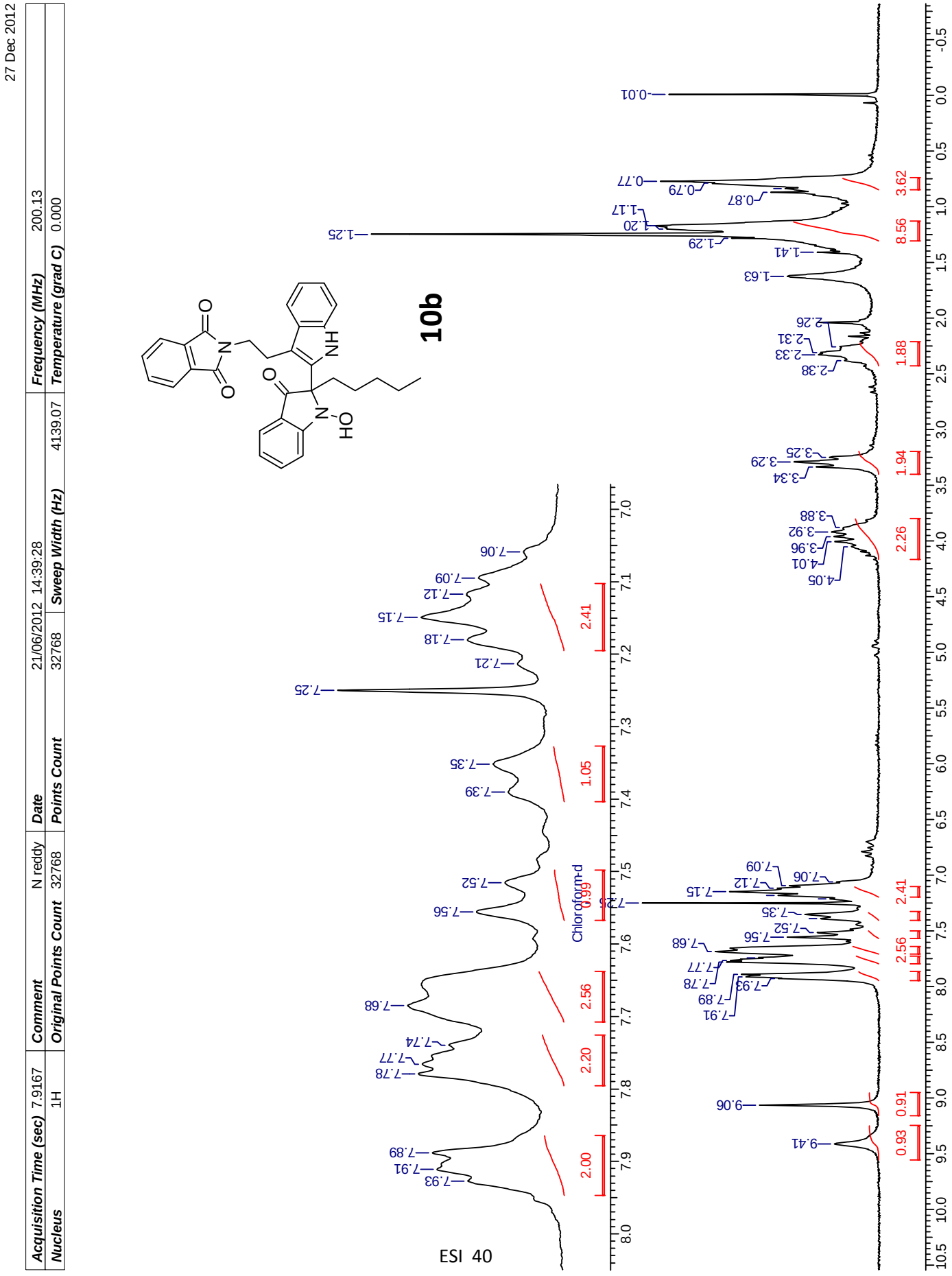
Frequency (MHz) 500.13
Temperature (grad C) 0.0000
Comment
Nucleus
Narendra
1H
Original Points Count 32768
Date
11/08/2013 15:51:04
Points Count 32768
Sweep Width (Hz) 10000.00
11a
Chloroform-d
ESI 35

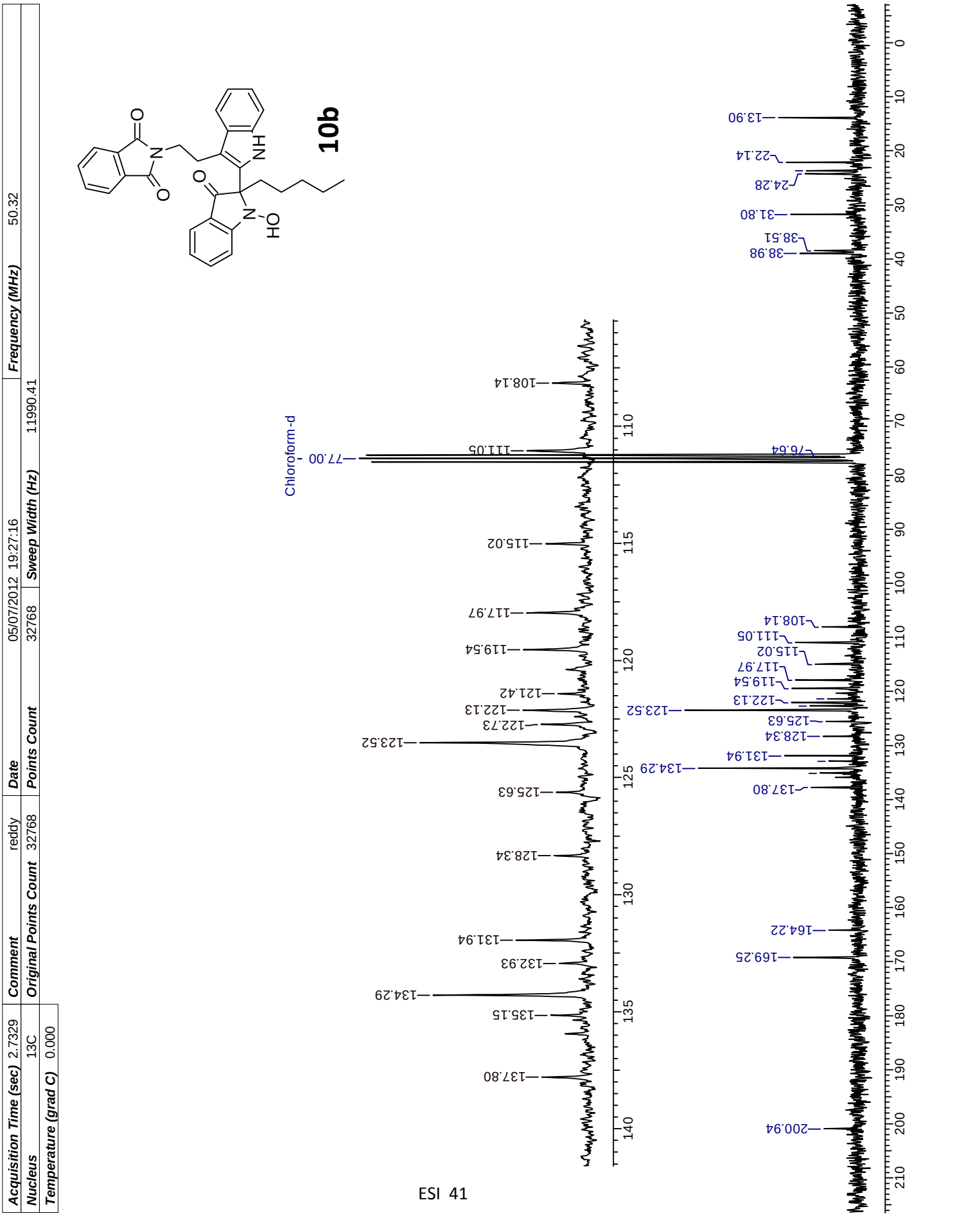


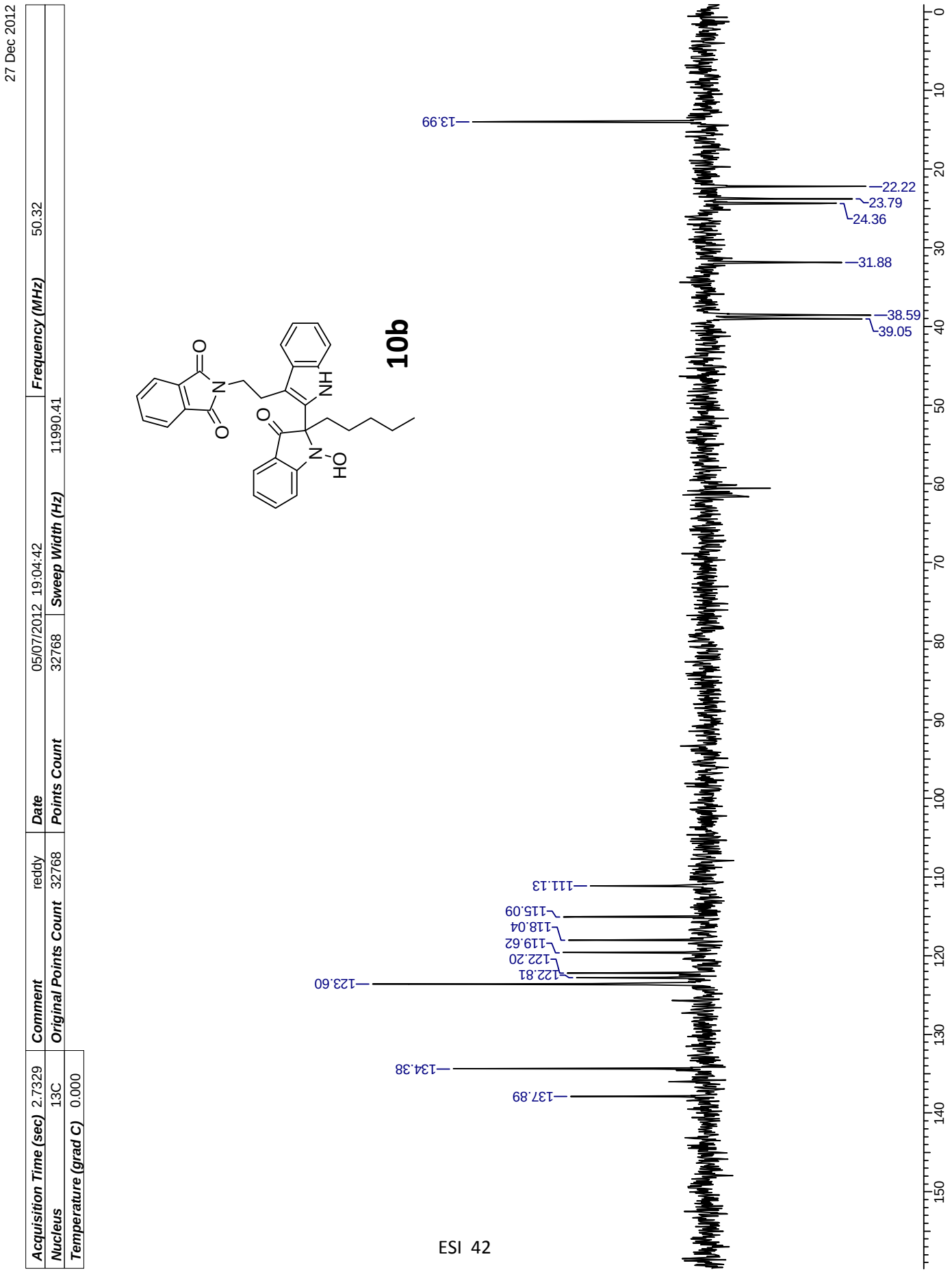

12 Aug 2013
Acquisition Time (sec) 1.1010
Original Points Count 32768
Date 11/08/2013 15:50:52
Points Count 32768
Sweep Width (Hz) 29761.90
Frequency (MHz) 125.76
Nucleus 13C
Temperature (grad C) 0.000
13.81
22.17
23.80
31.61
40.69
46.75
55.23
Chloroform-d
96.38
108.06
110.46
117.92
119.23
121.83
124.01
NH
NH
N
O
11a

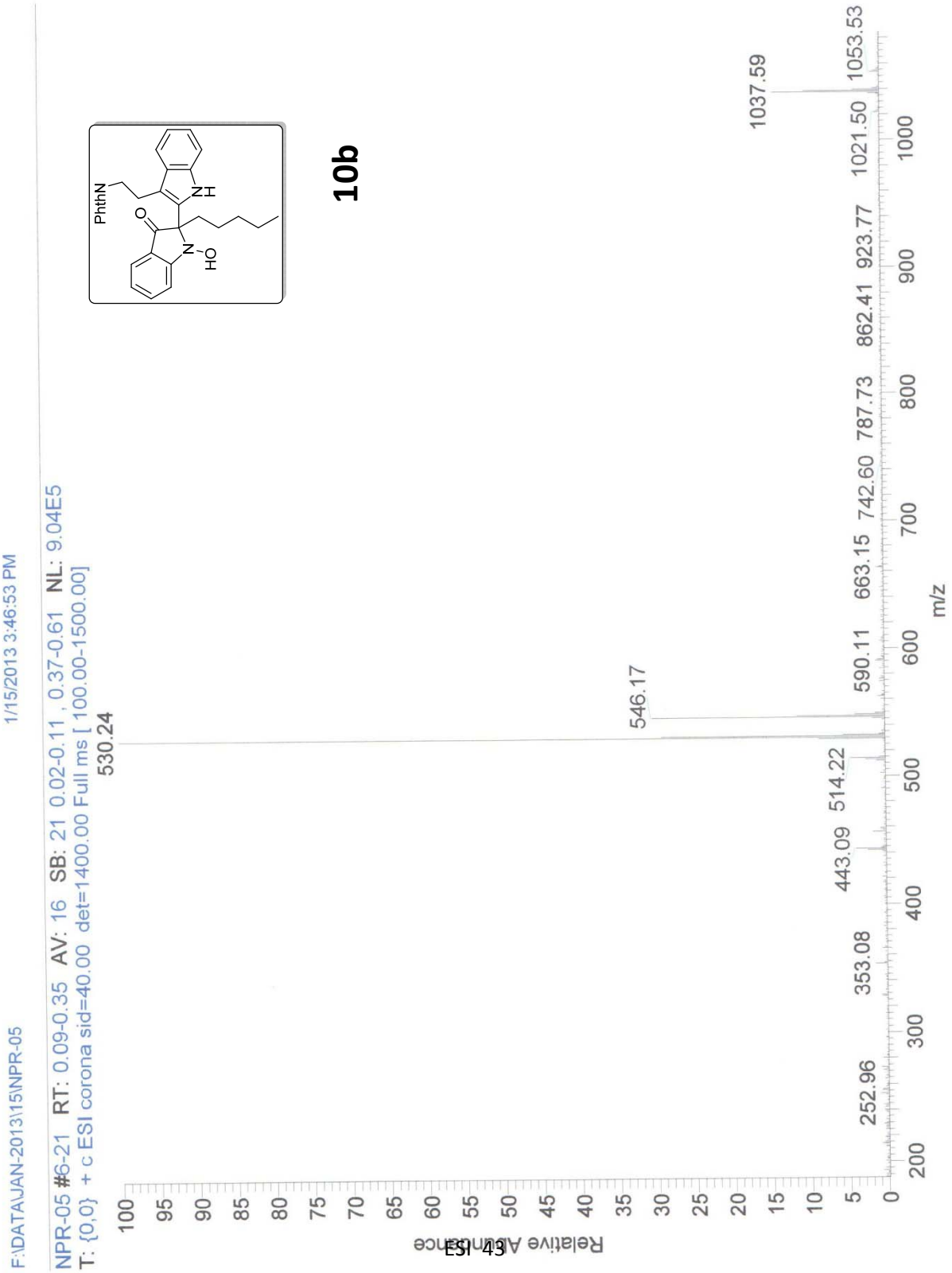

F:\DATA\JAN-2013\15\NPR-17
1/15/2013 4:13:11 PM
NPR-17 #7-20 RT: 0.11-0.33 AV: 14 SB: 18 0.00-0.14 , 0.30-0.44 NL: 2.87E5
T: {0,0} + c ESI corona sid=40.00 det=1400.00 Full ms [100.00-1500.00]
374.15
375.15
303.09
304.20
316.10
329.02
342.18
358.07
369.05
376.09
388.11
409.25
426.10
436.27
Relative Abundance
m/z
11a

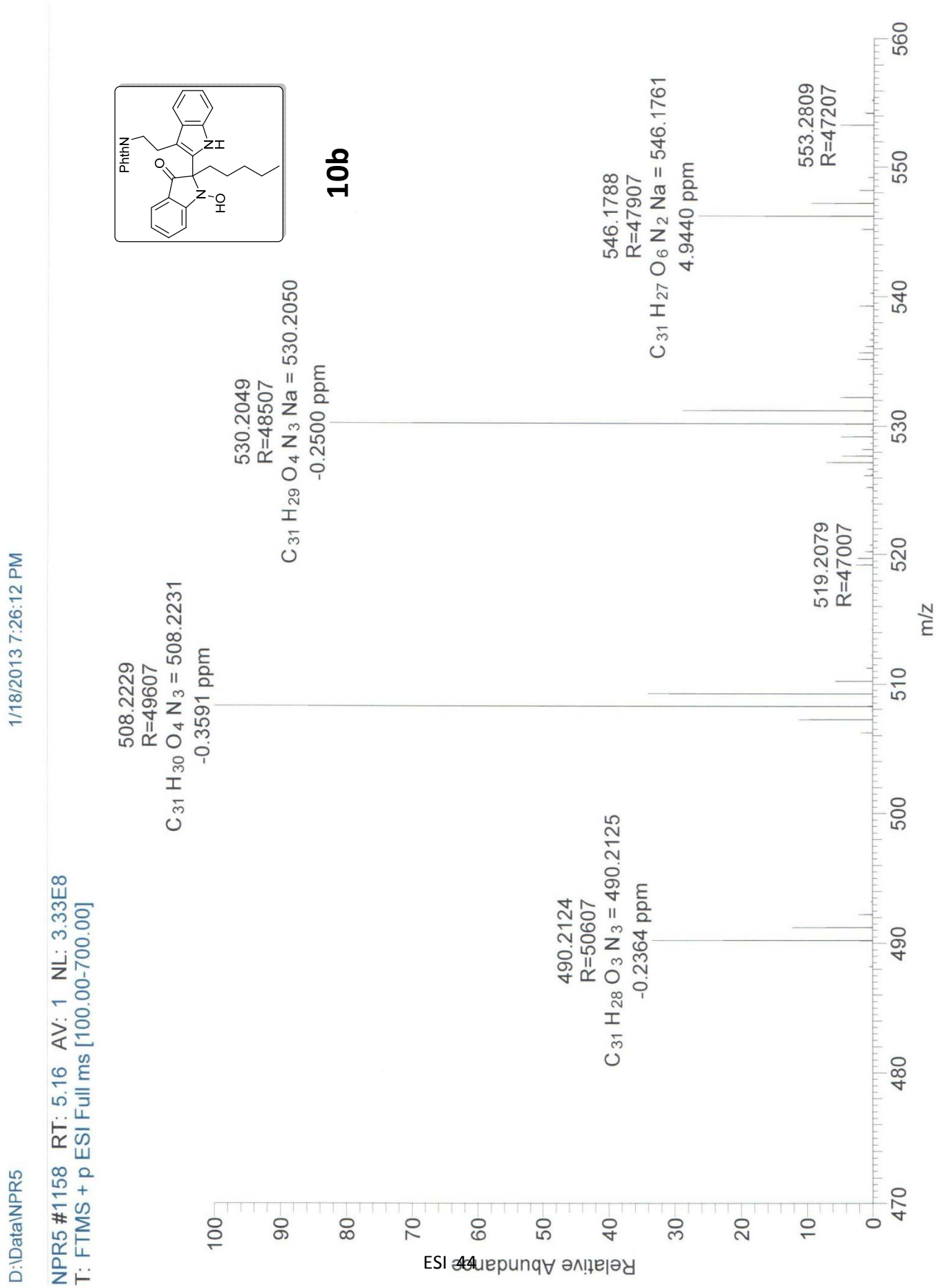

D:\Data\NPR17
1/18/2013 3:07:08 PM
NPR17 #518 RT: 2.31 AV: 1 NL: 4.34E9
T: FTMS + p ESI Full ms [100.00-700.00]
374.2225
R=58307
C 24 H 28 O N 3 = 374.2227
-0.5769 ppm
360.2071
R=52602
C 23 H 26 O N 3 = 360.2070
0.1480 ppm
383.9316
R=31600
390.2167
R=56707
11a
Relative Abundance
m/z

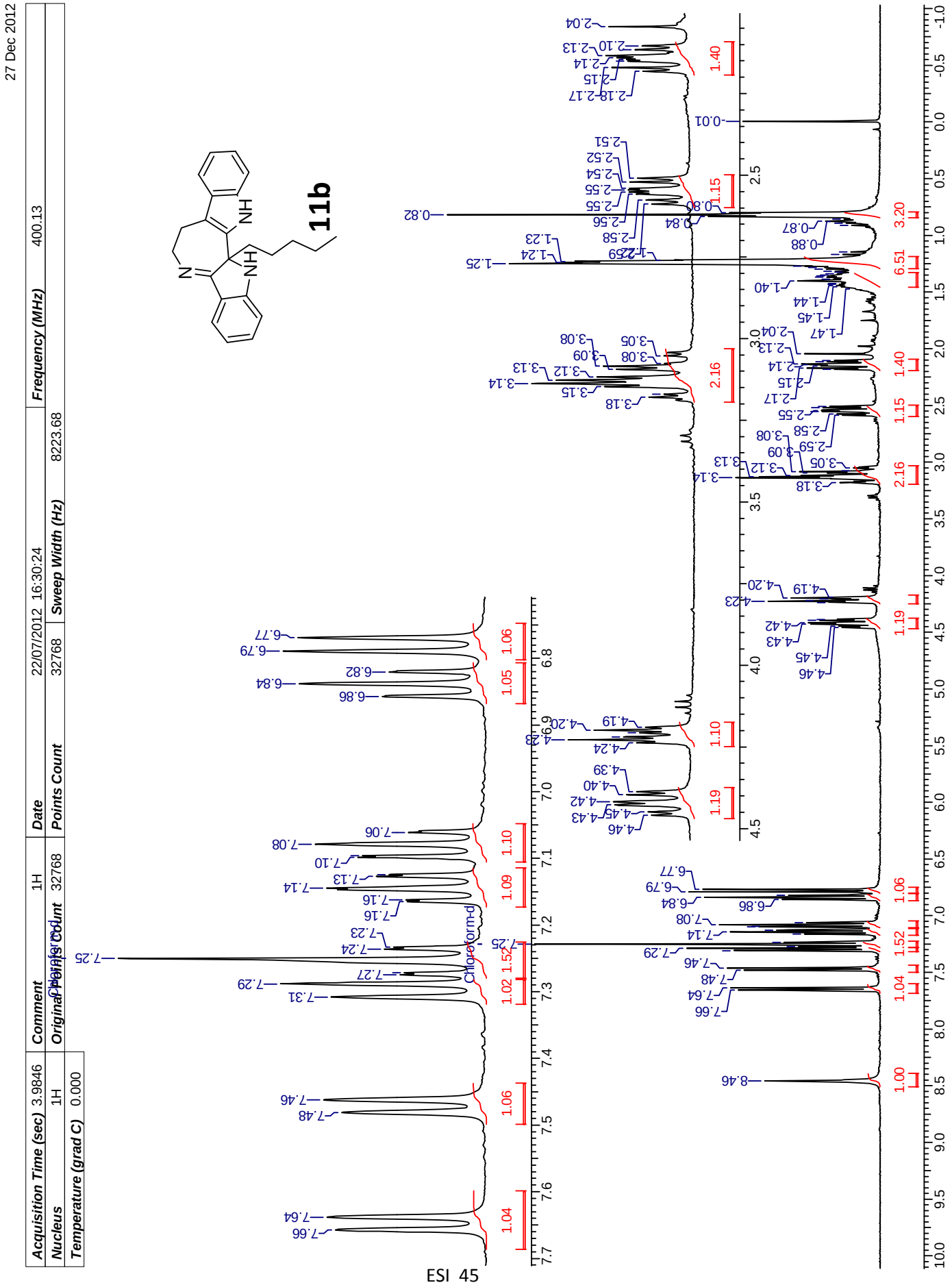

27 Dec 2012
Acquisition Time (sec) 7.9167
Nucleus 1H
Comment
Original Points Count 32768
N reddy
Date
Points Count 32768
21/06/2012 14:39:28
Sweep Width (Hz) 4139.07
Frequency (MHz) 200.13
Temperature (grad C) 0.000
10b
Chloroform-d
ESI 40


Acquisition Time (sec) 2.7329
Comment
Date 05/07/2012 19:27:16
Frequency (MHz) 50.32
Nucleus 13C
Original Points Count 32768
reddy
Points Count 32768
Sweep Width (Hz) 11990.41
Temperature (grad C) 0.000
10b
Chloroform-d
77.00
108.14
111.05
115.02
117.97
119.54
121.42
122.13
122.73
123.52
125.63
128.34
131.94
132.93
134.29
135.15
137.80
164.22
169.25
200.94
13.90
22.14
24.28
31.80
38.51
38.98
76.64


27 Dec 2012
Acquisition Time (sec) 2.7329
Comment
reddy
Date
05/07/2012 19:04:42
Frequency (MHz) 50.32
Nucleus 13C
Original Points Count 32768
Points Count 32768
Sweep Width (Hz) 11990.41
Temperature (grad C) 0.000
10b
13.99
22.22
23.79
24.36
31.88
38.59
39.05
111.13
115.09
118.04
119.62
122.20
122.81
123.60
134.38
137.89

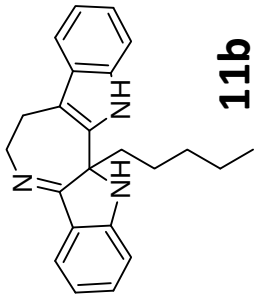

F:\DATA\JAN-2013\15\NPR-05
1/15/2013 3:46:53 PM
NPR-05 #6-21 RT: 0.09-0.35 AV: 16 SB: 21 0.02-0.11 , 0.37-0.61 NL: 9.04E5
T: {0,0} + c ESI corona sid=40.00 det=1400.00 Full ms [100.00-1500.00]
PhthN
10b
Relative Abundance
m/z
530.24
546.17
1037.59
252.96
353.08
443.09
514.22
590.11
663.15
742.60
787.73
862.41
923.77
1021.50
1053.53


D:\Data\NPR5
1/18/2013 7:26:12 PM
NPR5 #1158 RT: 5.16 AV: 1 NL: 3.33E8
T: FTMS + p ESI Full ms [100.00-700.00]
PhthN
10b
508.2229
R=49607
C 31 H 30 O 4 N 3 = 508.2231
-0.3591 ppm
530.2049
R=48507
C 31 H 29 O 4 N 3 Na = 530.2050
-0.2500 ppm
490.2124
R=50607
C 31 H 28 O 3 N 3 = 490.2125
-0.2364 ppm
519.2079
R=47007
546.1788
R=47907
C 31 H 27 O 6 N 2 Na = 546.1761
4.9440 ppm
553.2809
R=47207
Relative Abundance
m/z

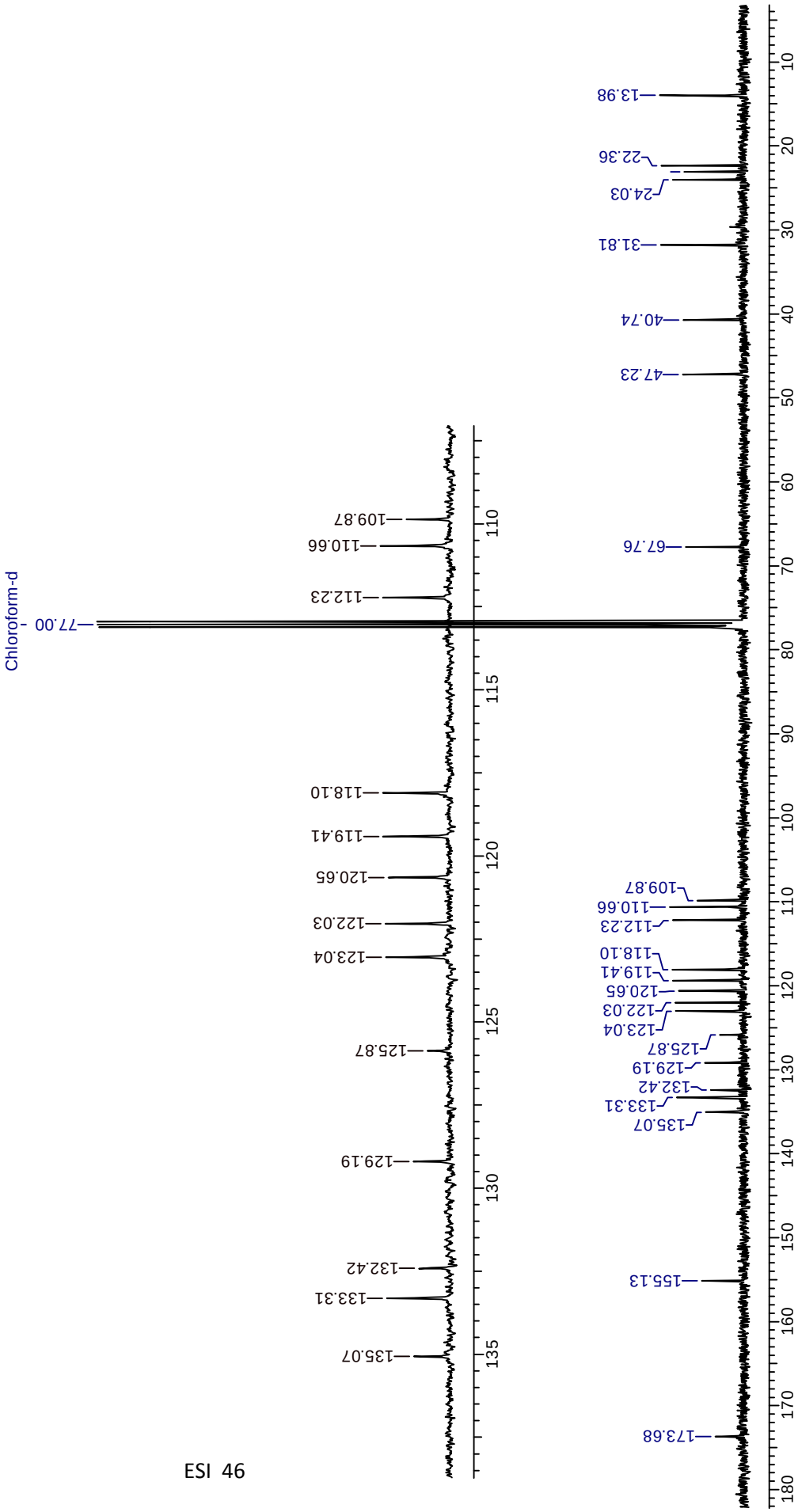


Acquisition Time (sec)	1.2976	***Date***	22/07/2012 16:30:14	***Frequency (MHz)***	100.61
Original Points Count	32768	***Points Count***	32768	***Sweep Width (Hz)***	25252.53

Nucleus	13C		
Temperature (grad C)	0.000		

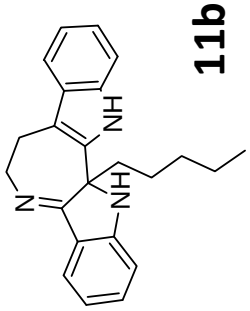


11b

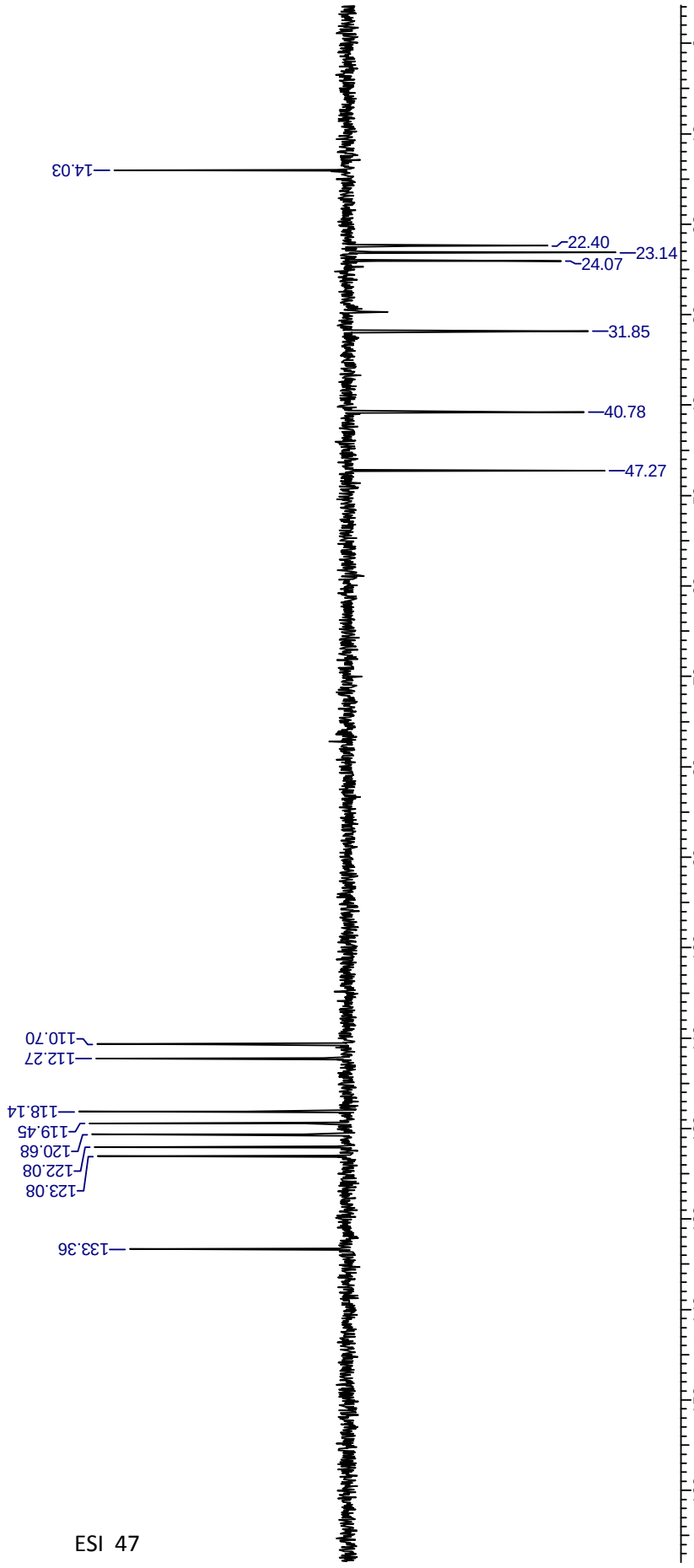


27 Dec 2012

Acquisition Time (sec) 1.2976	*Comment*	DEPT	*Date* 22/07/2012 16:30:36	*Frequency (MHz)*	100.61
Nucleus 13C	*Original Points Count* 32768		*Points Count* 32768	*Sweep Width (Hz)*	25252.53
Temperature (grad C) 0.000					

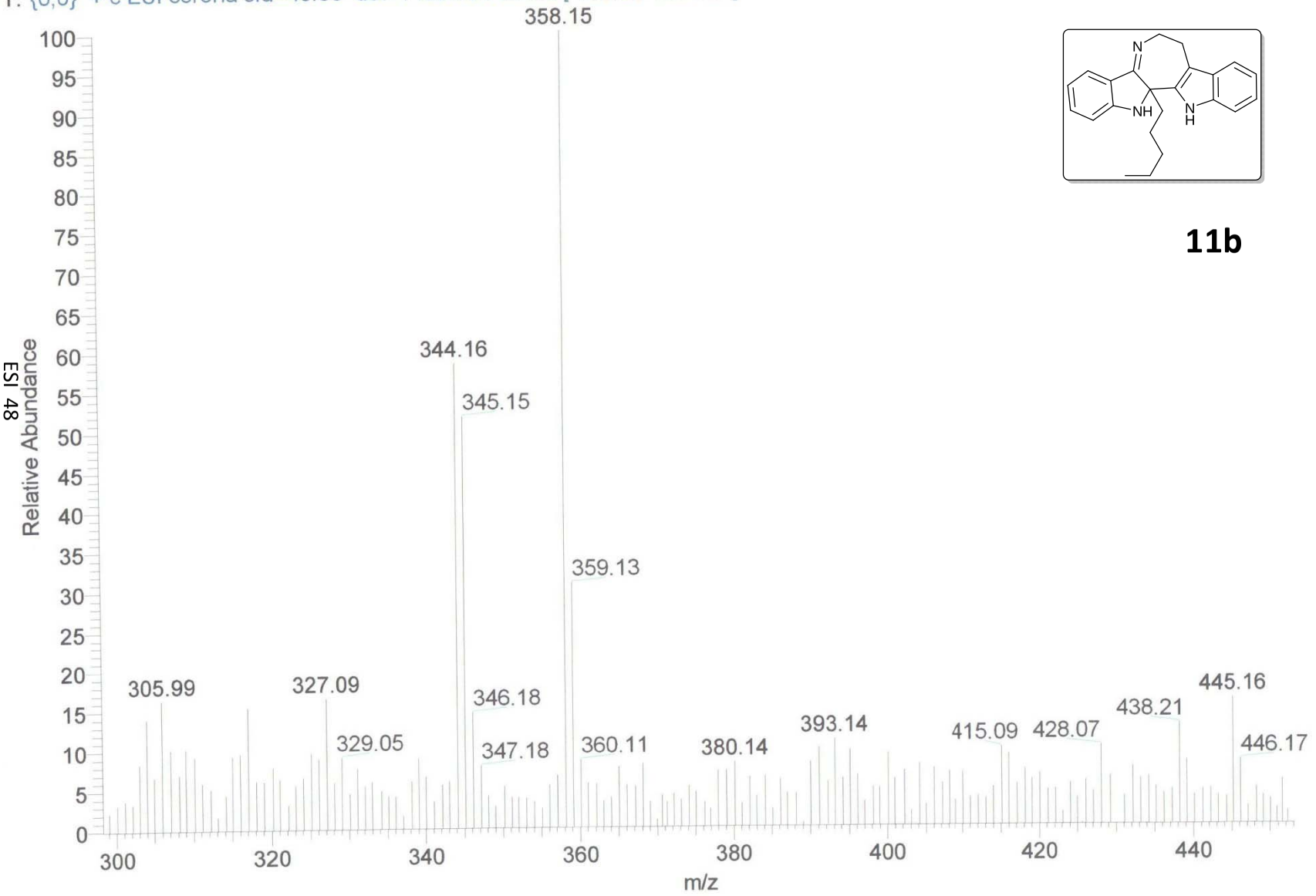


11b

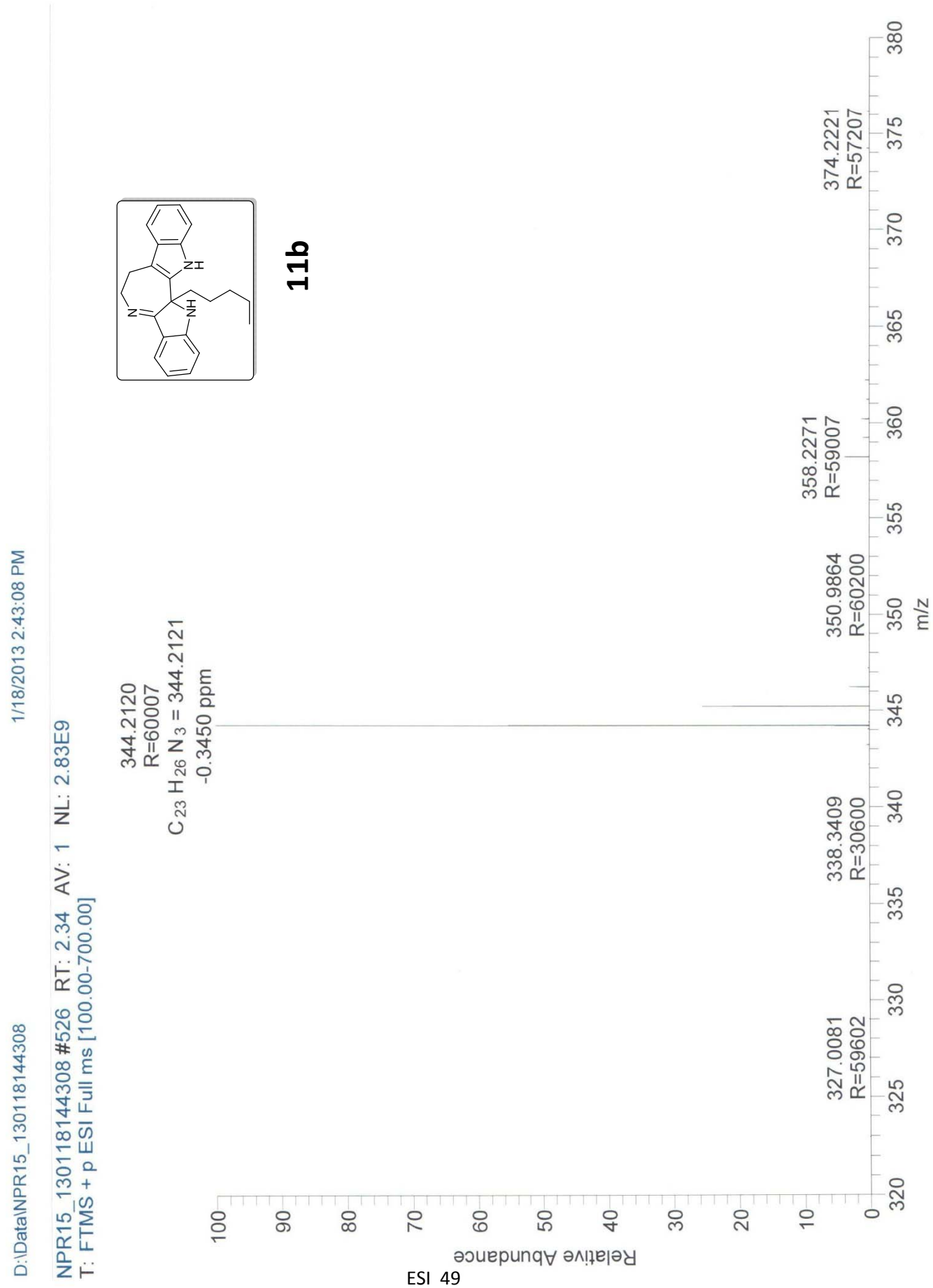


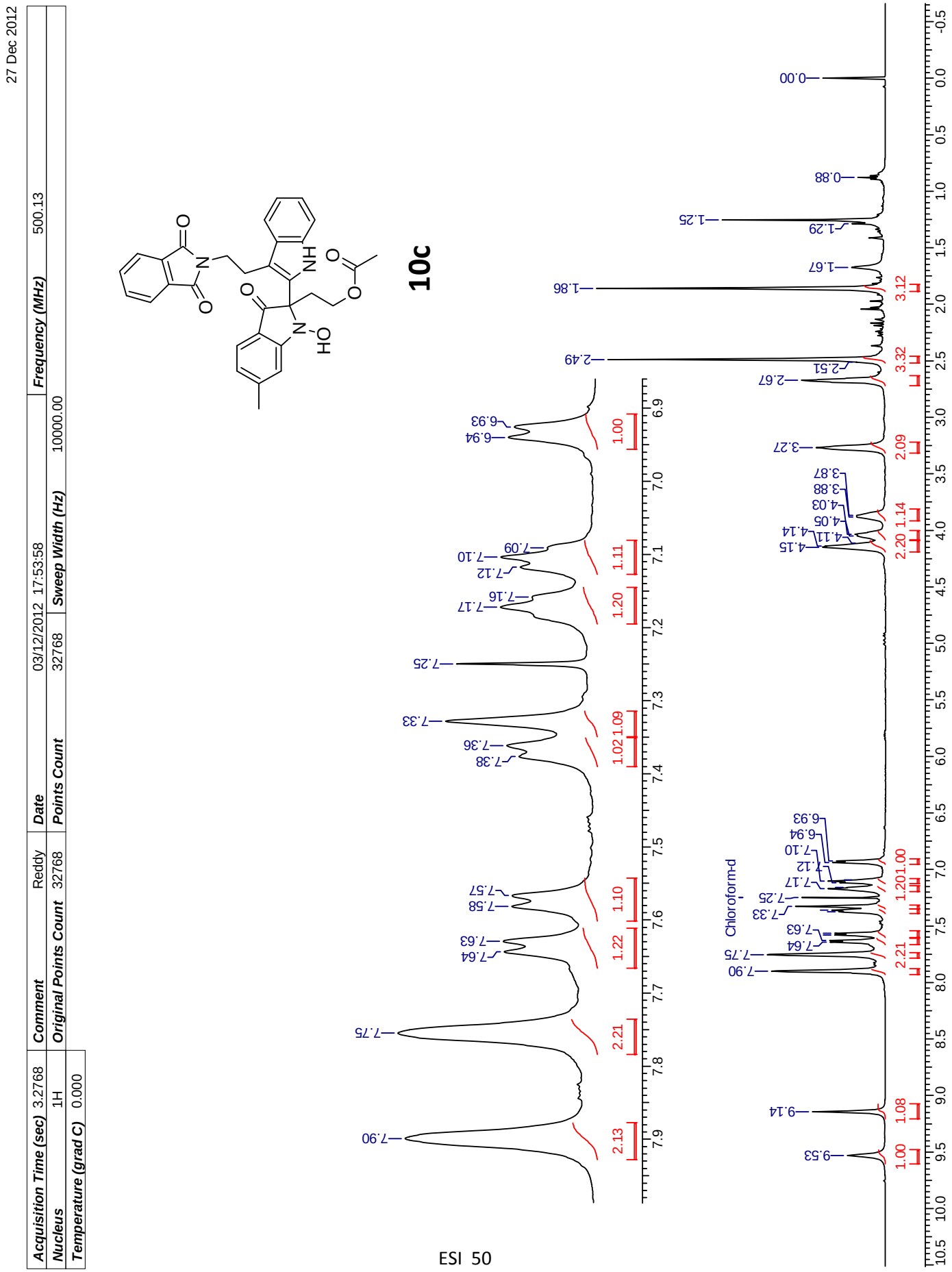
F:\DATA\JAN-2013\15\NPR-15 1/15/2013 4:07:54 PM

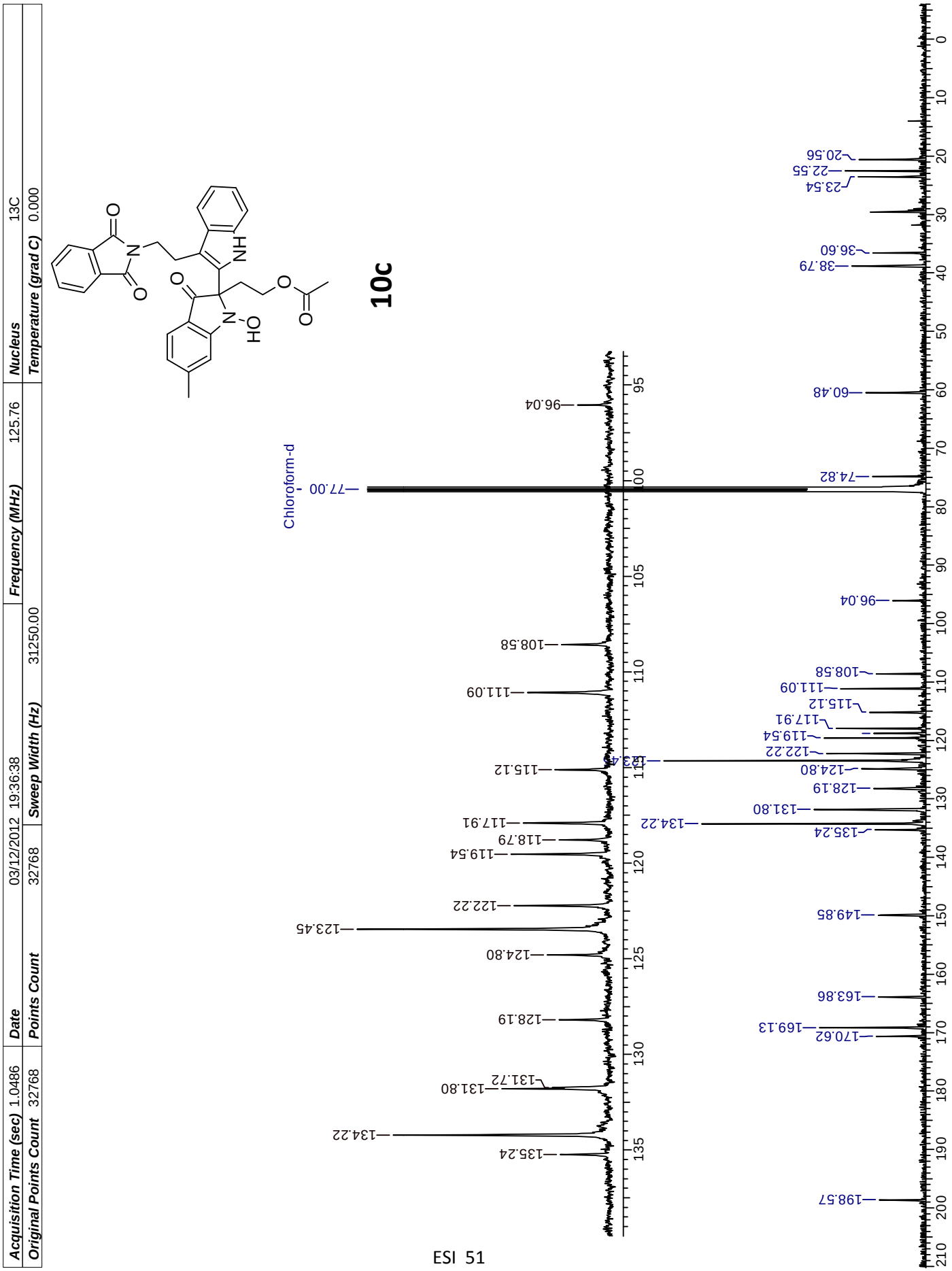
NPR-15 #8-19 RT: 0.12-0.32 AV: 12 SB: 19 0.02-0.14 , 0.30-0.47 NL: 1.55E5
T: {0,0} + c ESI corona sid=40.00 det=1400.00 Full ms [100.00-1500.00]

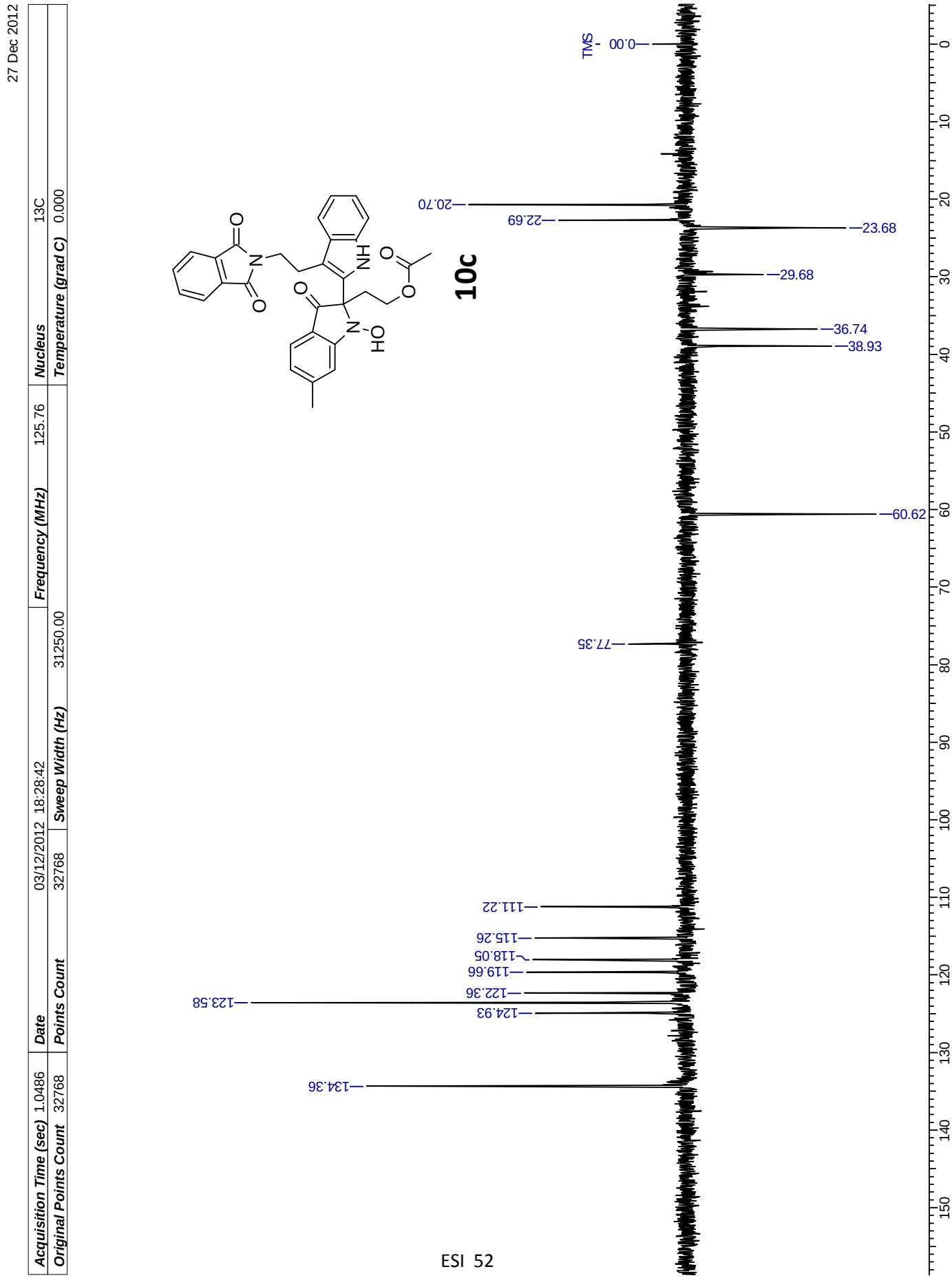


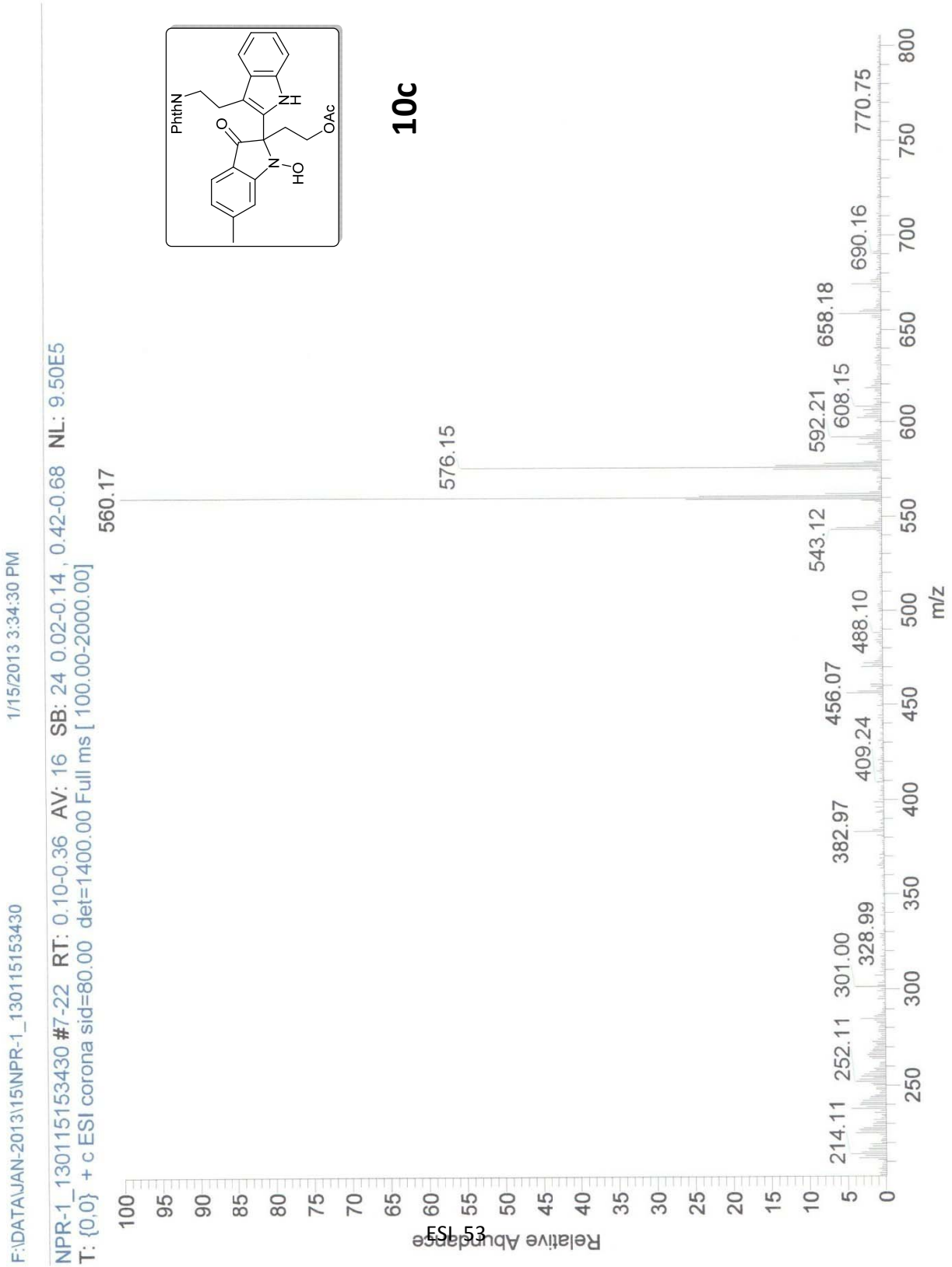
11b

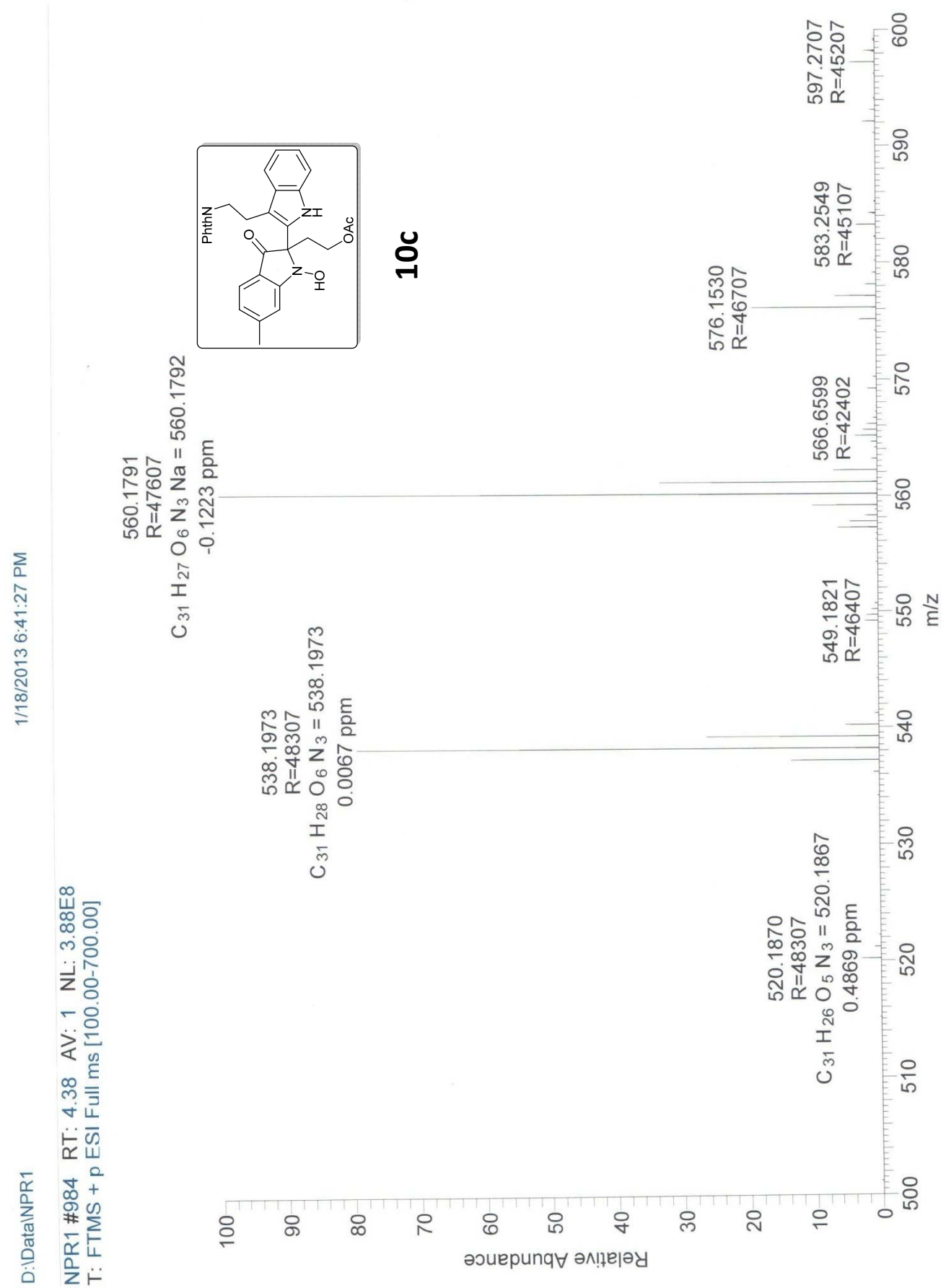

D:\Data\NPR15_130118144308
1/18/2013 2:43:08 PM
NPR15_130118144308 #526 RT: 2.34 AV: 1 NL: 2.83E9
T: FTMS + p ESI Full ms [100.00-700.00]
344.2120
R=60007
C23 H26 N3 = 344.2121
-0.3450 ppm
327.0081
R=59602
338.3409
R=30600
350.9864
R=60200
358.2271
R=59007
374.2221
R=57207
Relative Abundance
m/z
11b

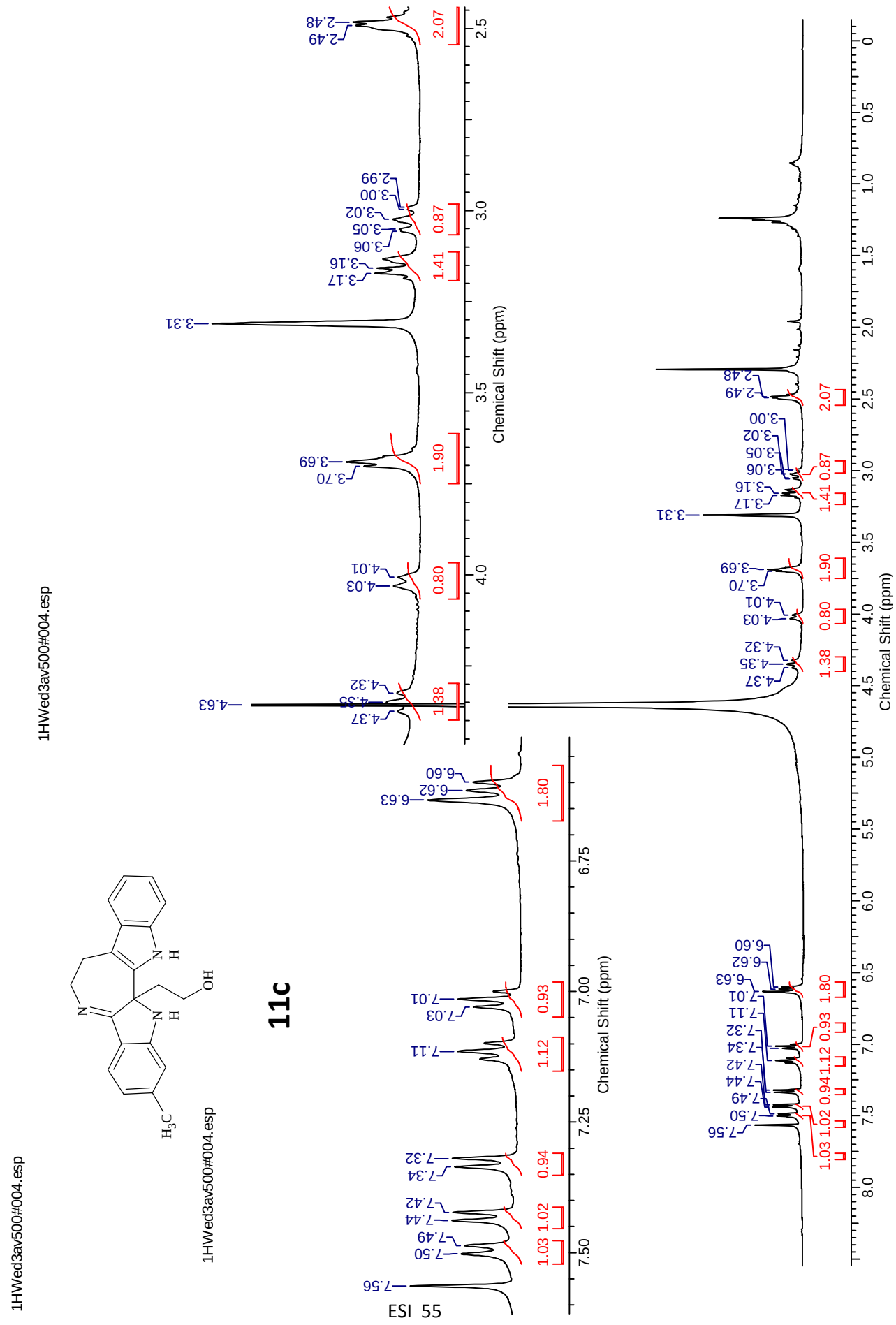

27 Dec 2012
Acquisition Time (sec) 3.2768
Nucleus 1H
Temperature (grad C) 0.000
Comment Reddy
Original Points Count 32768
Date 03/12/2012 17:53:58
Points Count 32768
Frequency (MHz) 500.13
Sweep Width (Hz) 10000.00
10c
Chloroform-d

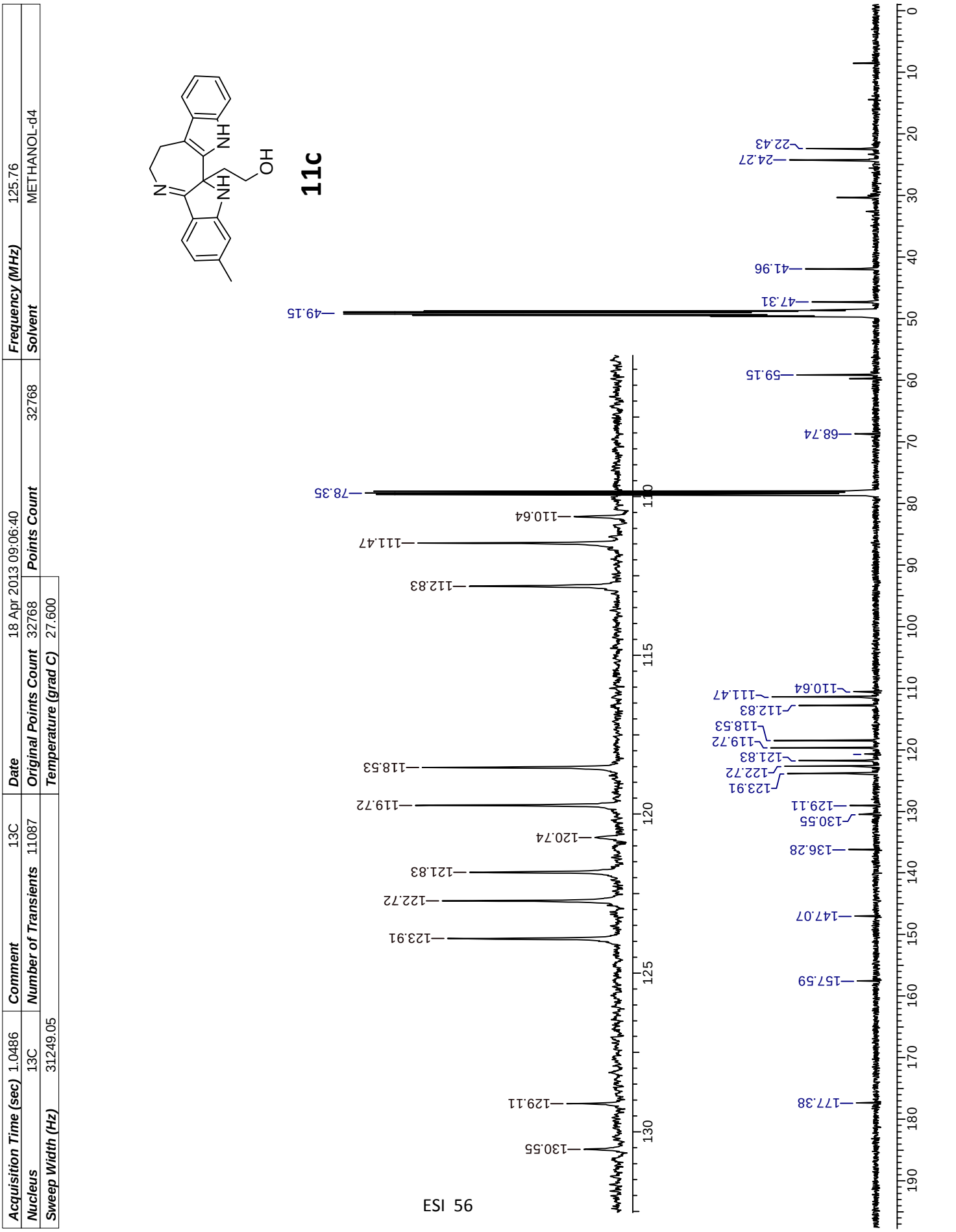

Acquisition Time (sec) 1.0486
Original Points Count 32768
Date 03/12/2012 19:36:38
Points Count 32768
Sweep Width (Hz) 31250.00
Frequency (MHz) 125.76
Nucleus 13C
Temperature (grad C) 0.000
10c
Chloroform-d
77.00
96.04
108.58
111.09
115.12
117.91
118.79
119.54
122.22
123.45
124.80
128.19
131.72
131.80
134.22
135.24
20.56
22.55
23.54
36.60
38.79
60.48
74.82
96.04
108.58
111.09
115.12
117.91
119.54
122.22
123.45
124.80
128.19
131.80
134.22
135.24
149.85
163.86
169.13
170.62
198.57

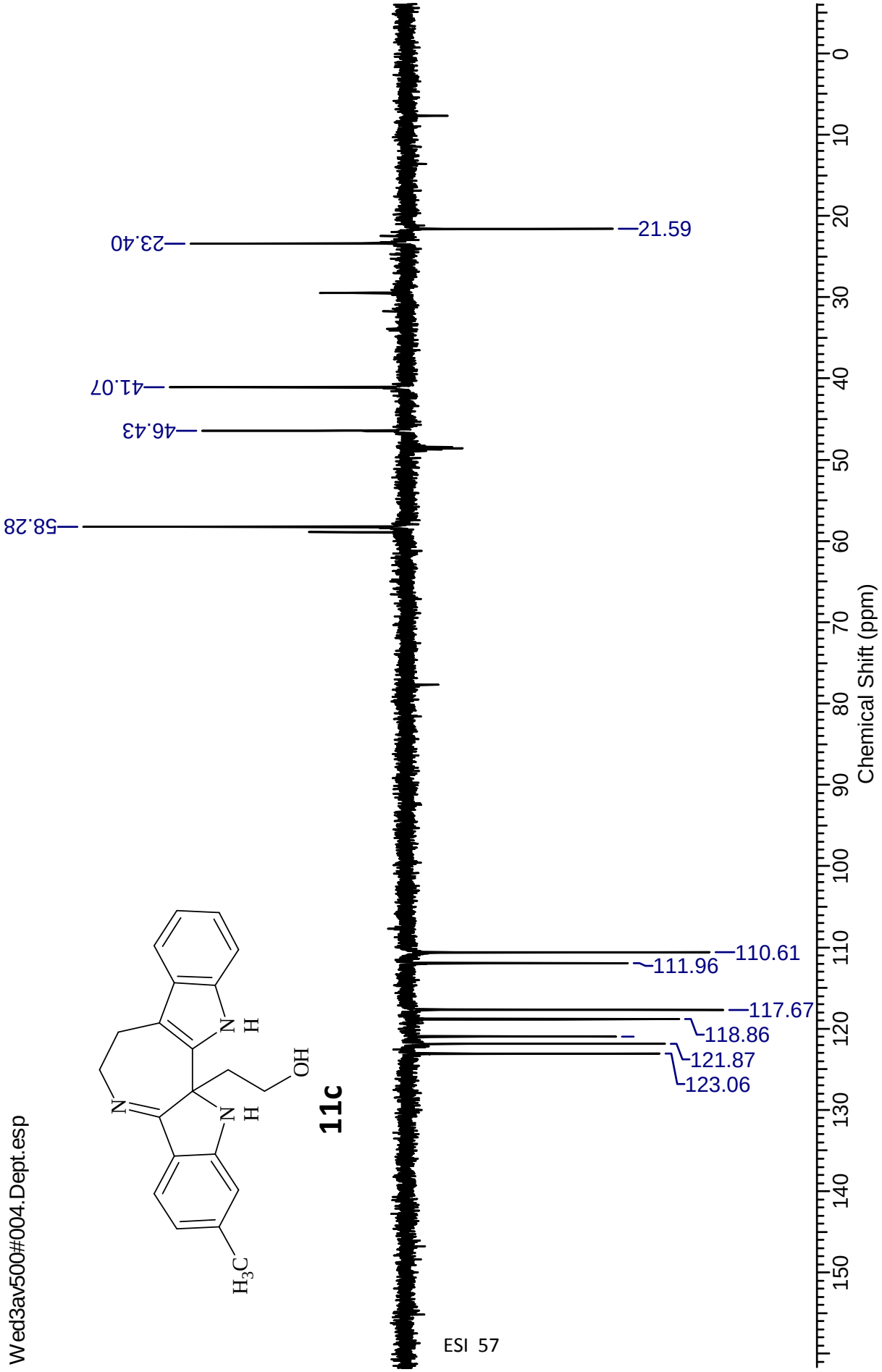

27 Dec 2012
Acquisition Time (sec) 1.0486
Original Points Count 32768
Date 03/12/2012 18:28:42
Points Count 32768
Sweep Width (Hz) 31250.00
Frequency (MHz) 125.76
Nucleus 13C
Temperature (grad C) 0.000
10c
TMS
0.00
20.70
22.69
23.68
29.68
36.74
38.93
60.62
77.35
111.22
115.26
118.05
119.66
122.36
123.58
124.93
134.36

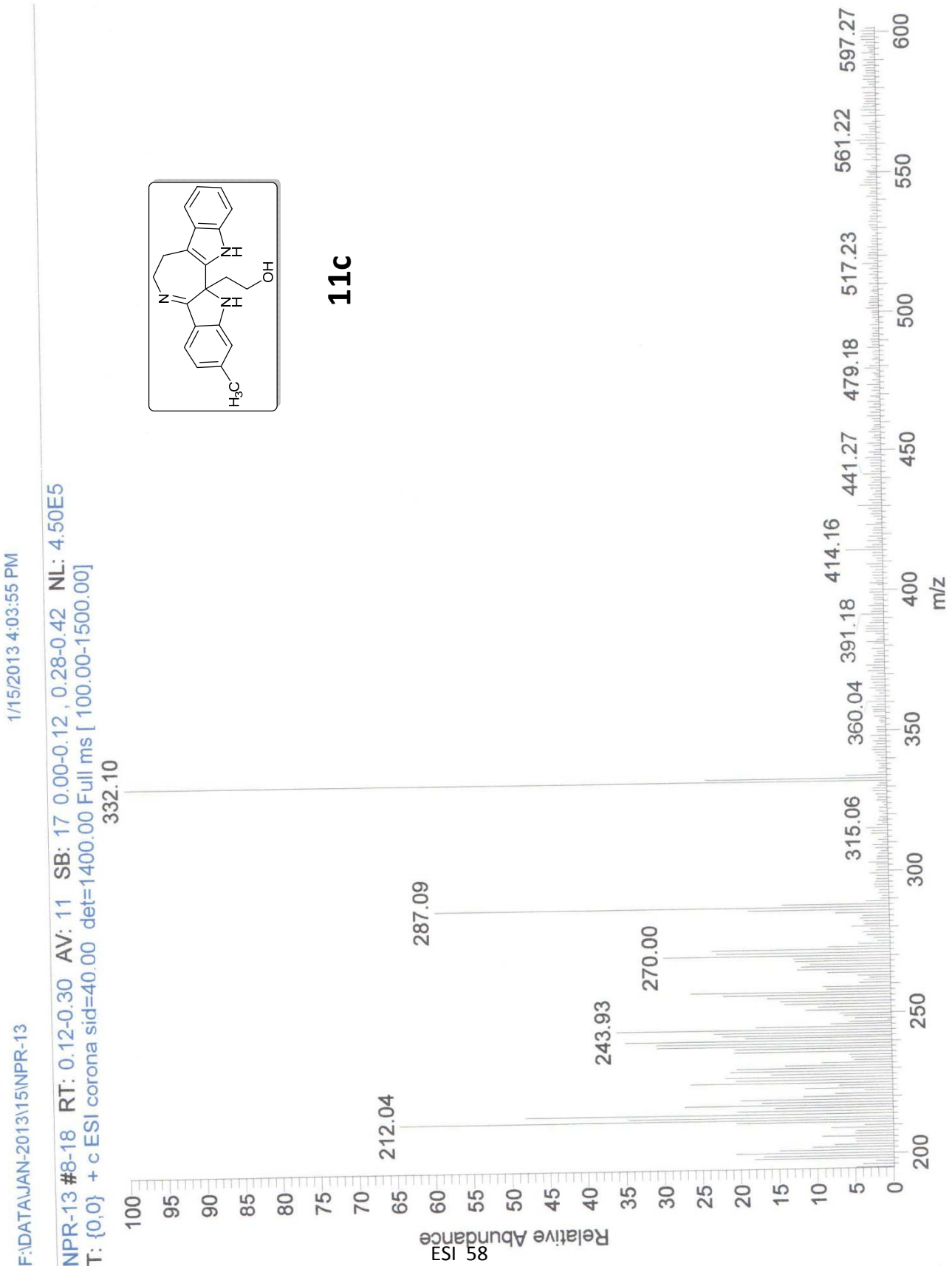

F:\DATA\JAN-2013\15\NPR-1_130115153430
1/15/2013 3:34:30 PM
NPR-1_130115153430 #7-22 RT: 0.10-0.36 AV: 16 SB: 24 0.02-0.14 , 0.42-0.68 NL: 9.50E5
T: {0,0} + c ESI corona sid=80.00 det=1400.00 Full ms [100.00-2000.00]
PhthN
O
N
H
N
HO
OAc
10c
560.17
576.15
543.12
592.21
608.15
658.18
690.16
770.75
456.07
488.10
409.24
382.97
328.99
301.00
252.11
214.11
Relative Abundance
m/z


D:\Data\NPR1
1/18/2013 6:41:27 PM
NPR1 #984 RT: 4.38 AV: 1 NL: 3.88E8
T: FTMS + p ESI Full ms [100.00-700.00]
10c
PhthN
OAc
HO
560.1791
R=47607
C31 H27 O6 N3 Na = 560.1792
-0.1223 ppm
538.1973
R=48307
C31 H28 O6 N3 = 538.1973
0.0067 ppm
520.1870
R=48307
C31 H26 O5 N3 = 520.1867
0.4869 ppm
549.1821
R=46407
566.6599
R=42402
576.1530
R=46707
583.2549
R=45107
597.2707
R=45207
Relative Abundance
m/z

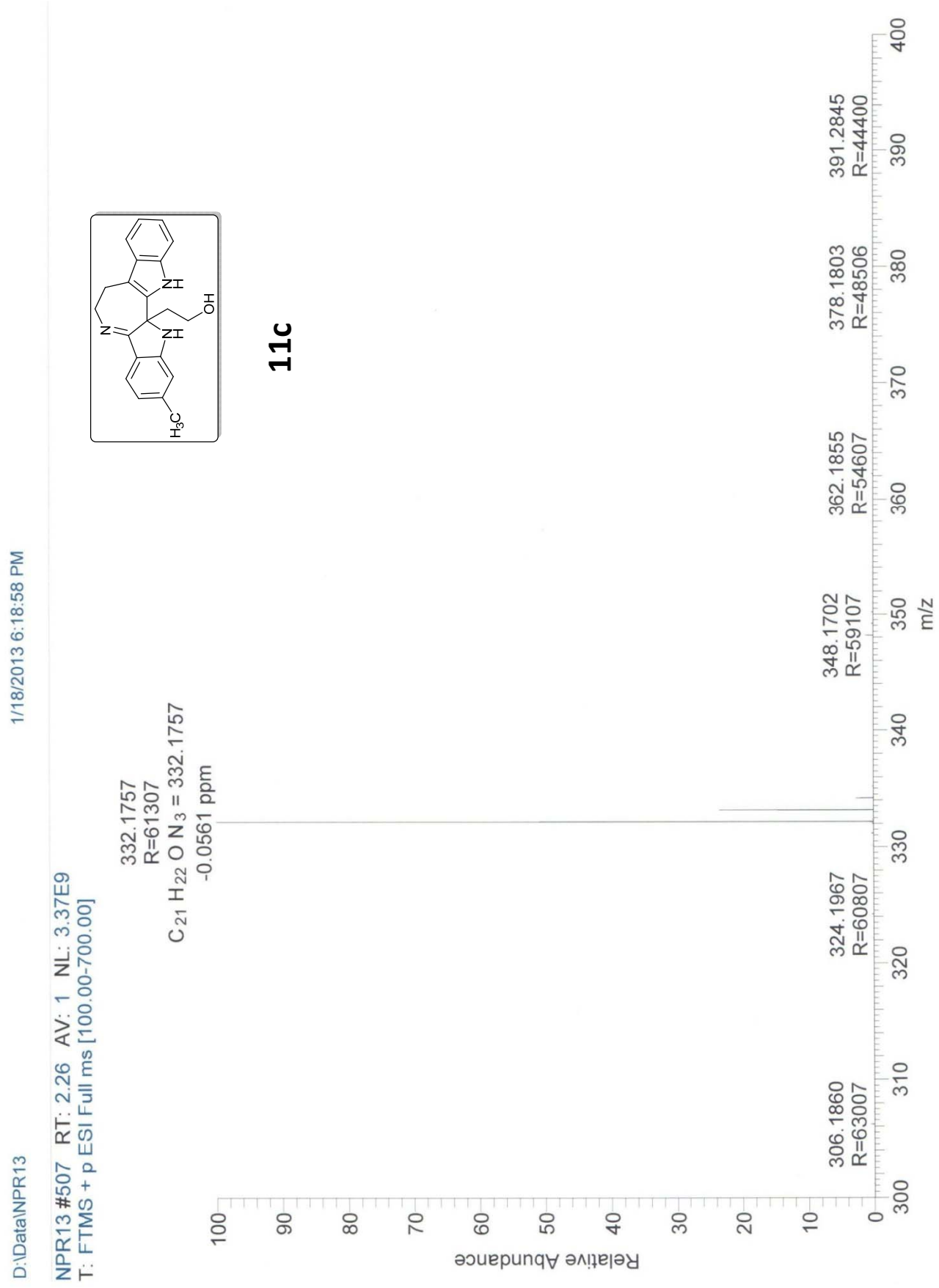

1HWed3av500#004.esp
1HWed3av500#004.esp
1HWed3av500#004.esp
11c
ESI 55
Chemical Shift (ppm)
Chemical Shift (ppm)
Chemical Shift (ppm)

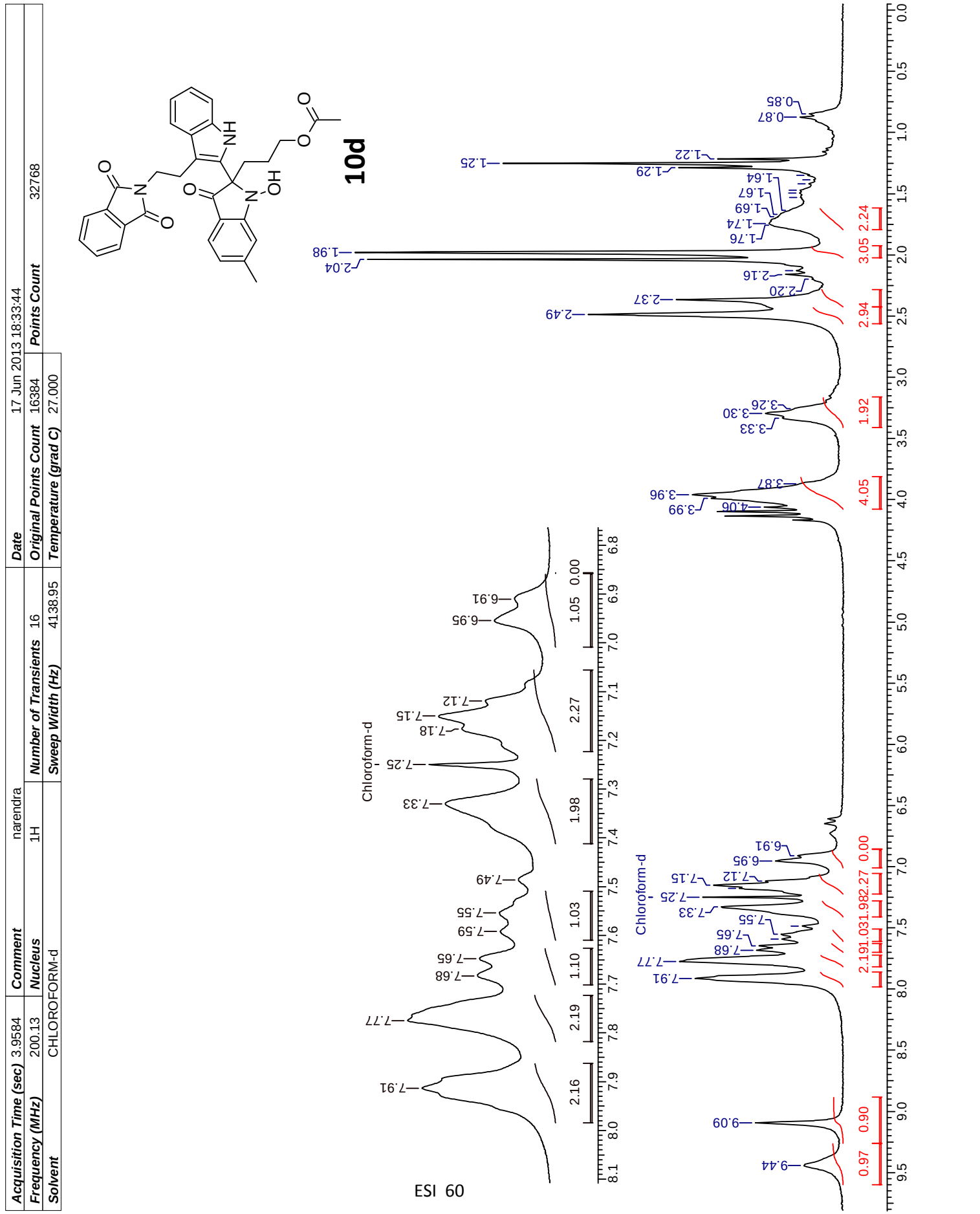

Acquisition Time (sec) 1.0486
Comment
Date 18 Apr 2013 09:06:40
Frequency (MHz) 125.76
Nucleus 13C
Number of Transients 11087
Original Points Count 32768
Points Count 32768
Solvent METHANOL-d4
Sweep Width (Hz) 31249.05
Temperature (grad C) 27.600
11c
NH
NH
N
OH
22.43
24.27
41.96
47.31
49.15
59.15
68.74
78.35
110.64
111.47
112.83
118.53
119.72
120.74
121.83
122.72
123.91
129.11
130.55
136.28
147.07
157.59
177.38




F:\DATA\JAN-2013\15\NPR-13
1/15/2013 4:03:55 PM
NPR-13 #8-18 RT: 0.12-0.30 AV: 11 SB: 17 0.00-0.12 , 0.28-0.42 NL: 4.50E5
T: {0,0} + c ESI corona sid=40.00 det=1400.00 Full ms [100.00-1500.00]
Relative Abundance
m/z
332.10
287.09
212.04
243.93
270.00
315.06
360.04
391.18
414.16
441.27
479.18
517.23
561.22
597.27
N
H
N
H
OH
H3C

11c

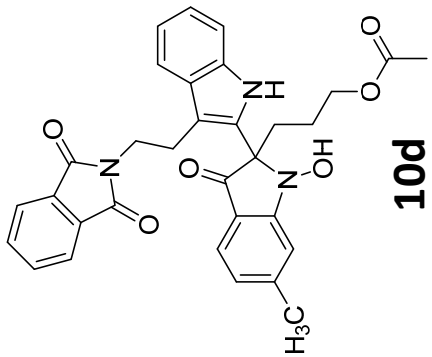

D:\Data\NPR13
1/18/2013 6:18:58 PM
NPR13 #507 RT: 2.26 AV: 1 NL: 3.37E9
T: FTMS + p ESI Full ms [100.00-700.00]
332.1757
R=61307
C 21 H 22 O N 3 = 332.1757
-0.0561 ppm
306.1860
R=63007
324.1967
R=60807
348.1702
R=59107
362.1855
R=54607
378.1803
R=48506
391.2845
R=44400
Relative Abundance
m/z
11c



Acquisition Time (sec)	3.9584	***Comment***	narendra	***Date***	17 Jun 2013 18:33:44
Frequency (MHz)	200.13	***Nucleus***	1H	***Number of Transients***	16	***Original Points Count***	16384	***Points Count***	32768
Solvent	CHLOROFORM-d			***Sweep Width (Hz)***	4138.95	***Temperature (grad C)***	27.000		

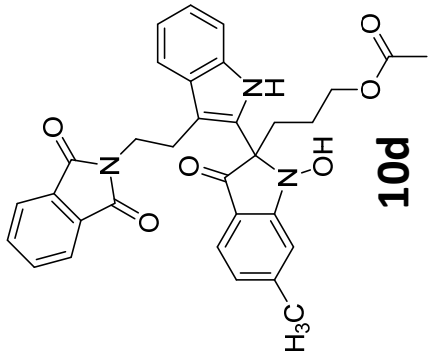
26 Jun 2013
AC38 Wed2av500#020
narendra

Acquisition Time (sec)	1.0434	***Comment***	narendra	***Date***	26 Jun 2013 00:13:28	***Frequency (MHz)***	100.53
Nucleus	13C	***Number of Transients***	600	***Original Points Count***	26214	***Points Count***	26214	***Solvent***	METHANOL-D3
Sweep Width (Hz)	25124.29	***Temperature (grad C)***	19.200				

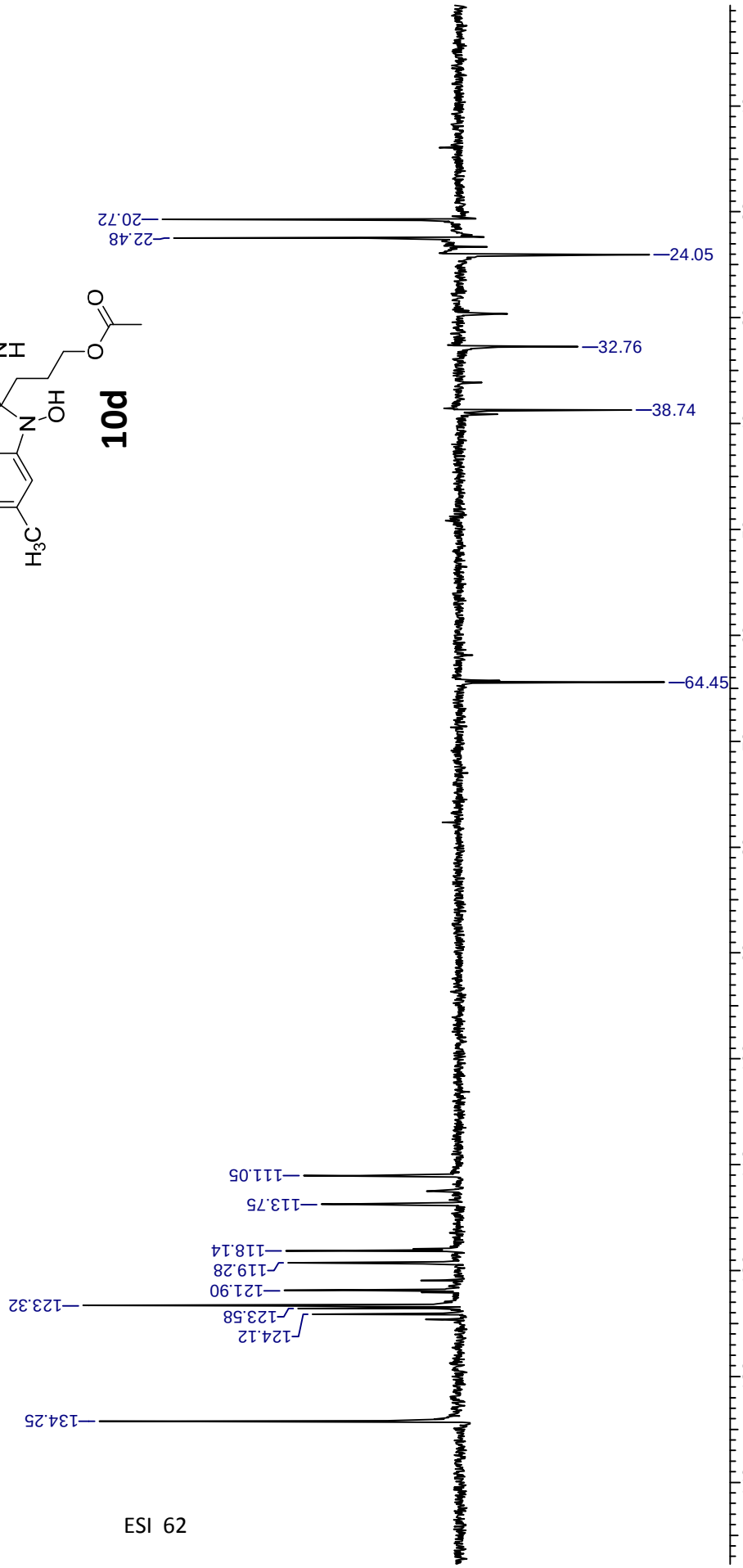


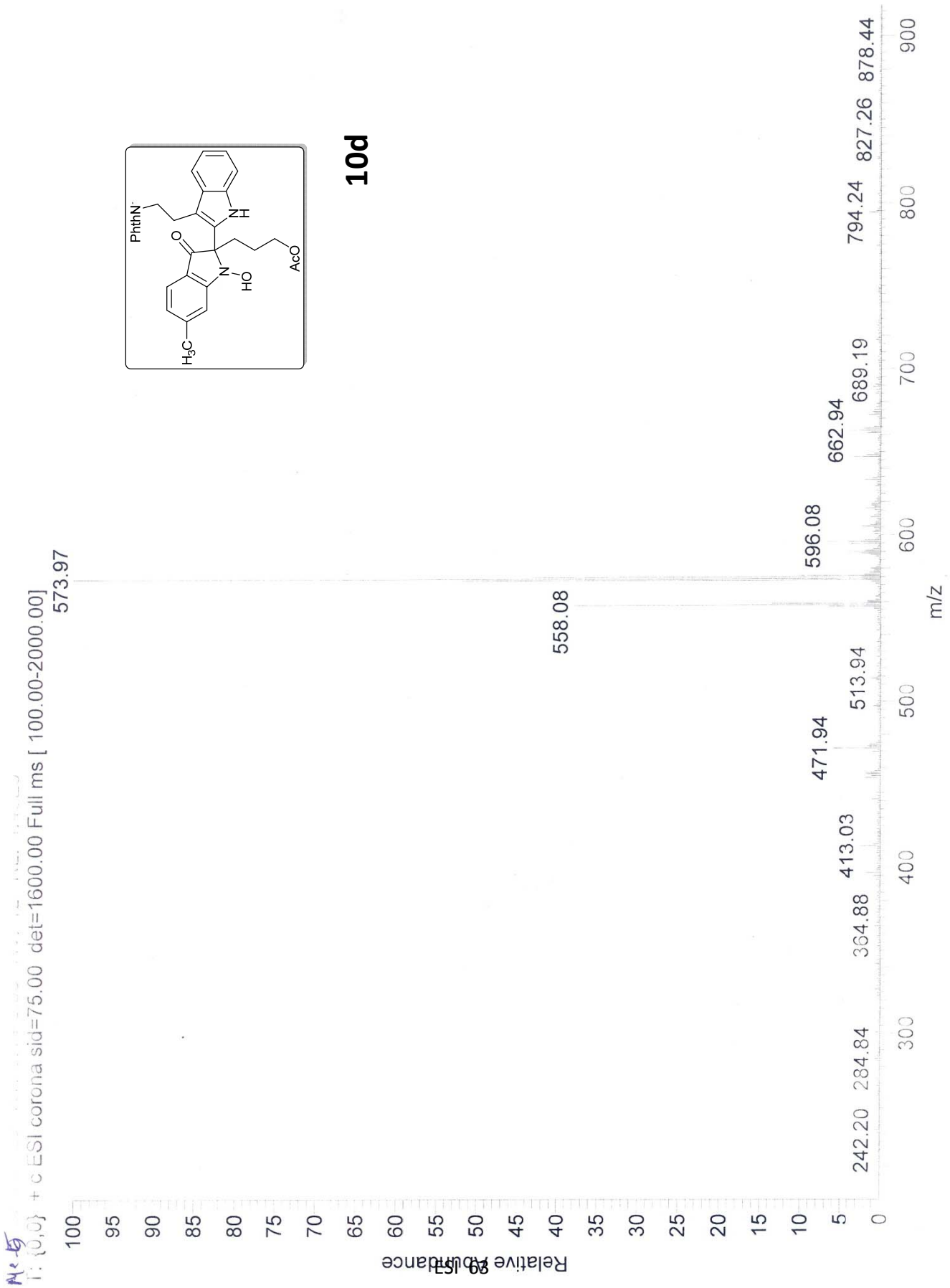
26 Jun 2013
AC38 Wed2av500#020
narendra

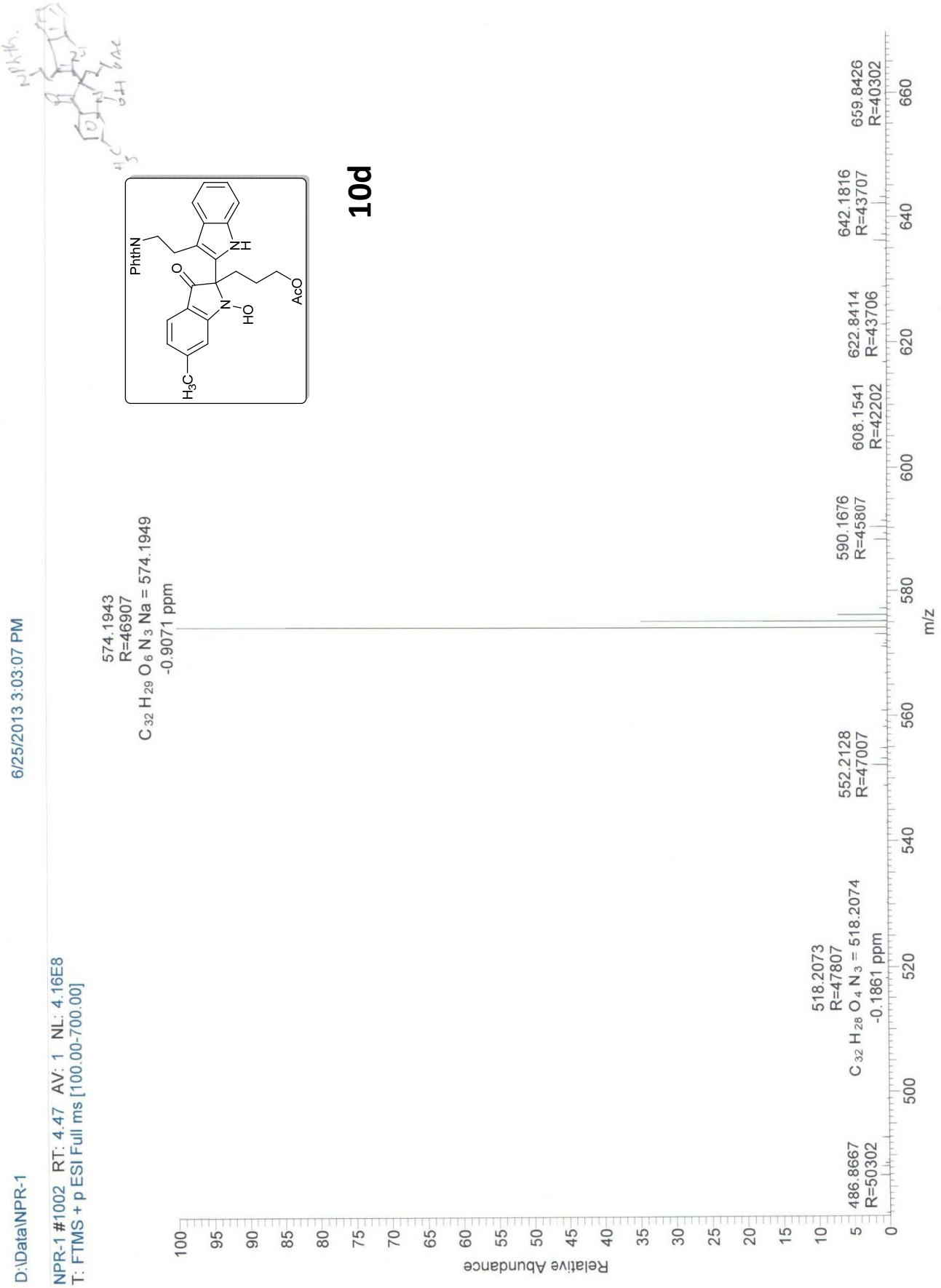
Acquisition Time (sec)	1.0434	***Comment***	narendra	***Date***	26 Jun 2013 00:13:28
Nucleus	13C	***Number of Transients***	400	***Original Points Count***	26214
Sweep Width (Hz)	25124.29	***Temperature (grad C)***	19.200	***Points Count***	26214
				Frequency (MHz)	100.53
				Solvent	METHANOL-D3

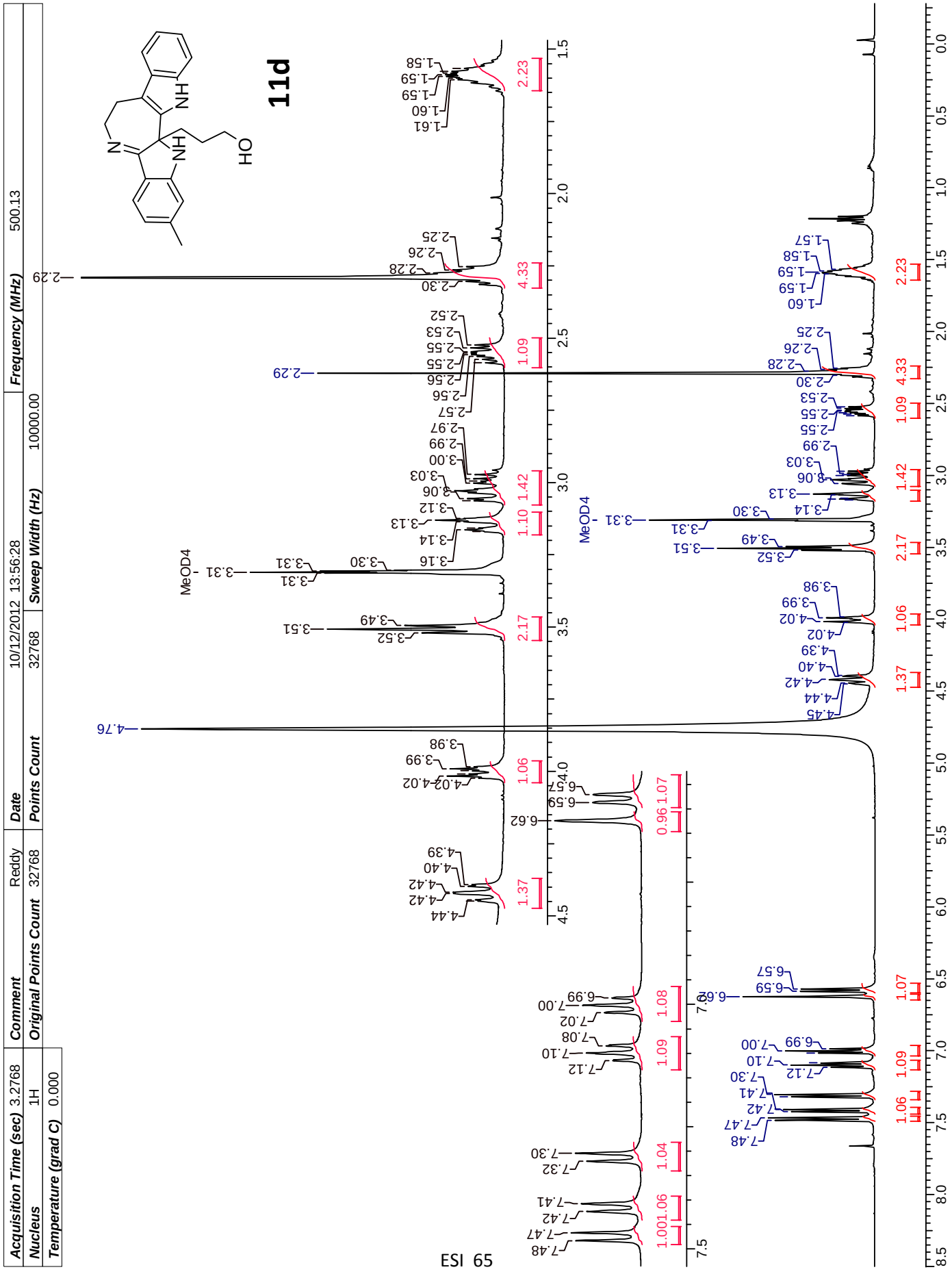


10d

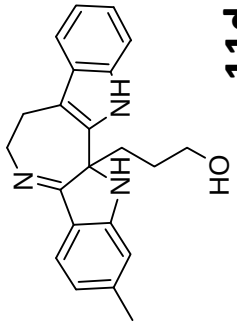



10d
PhthN
O
N
H
N
HO
AcO
H3C
T: {0,0} + c ESI corona sid=75.00 det=1600.00 Full ms [100.00-2000.00]
573.97
558.08
596.08
662.94
471.94
513.94
413.03
364.88
242.20
284.84
689.19
794.24
827.26
878.44
Relative Abundance
m/z
ESI 63

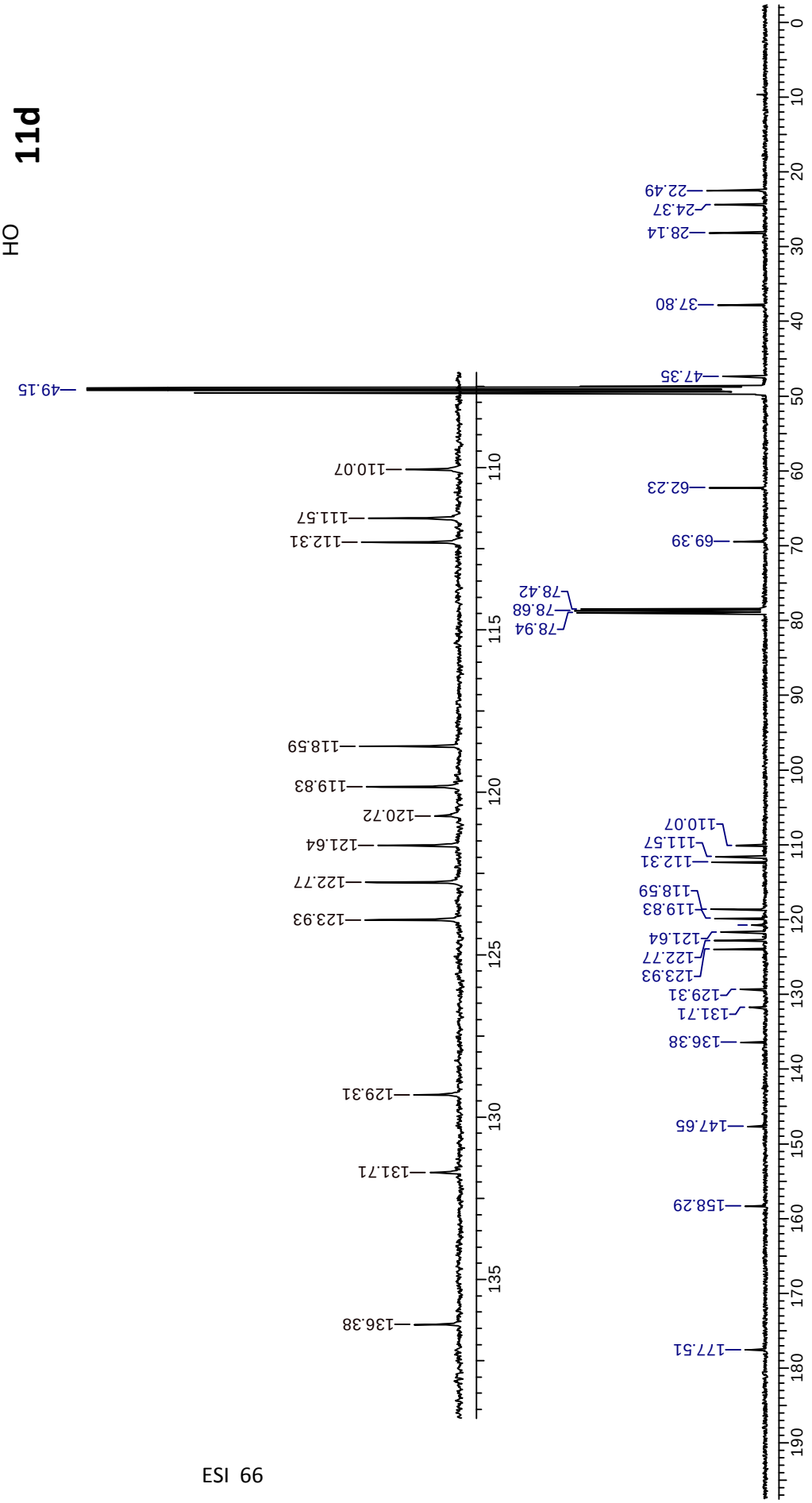

D:\Data\NPR-1
6/25/2013 3:03:07 PM
NPR-1 #1002 RT: 4.47 AV: 1 NL: 4.16E8
T: FTMS + p ESI Full ms [100.00-700.00]
574.1943
R=46907
C 32 H 29 O 6 N 3 Na = 574.1949
-0.9071 ppm
518.2073
R=47807
C 32 H 28 O 4 N 3 = 518.2074
-0.1861 ppm
486.8667
R=50302
552.2128
R=47007
590.1676
R=45807
608.1541
R=42202
622.8414
R=43706
642.1816
R=43707
659.8426
R=40302
Relative Abundance
m/z
PhthN
AcO
HO
10d

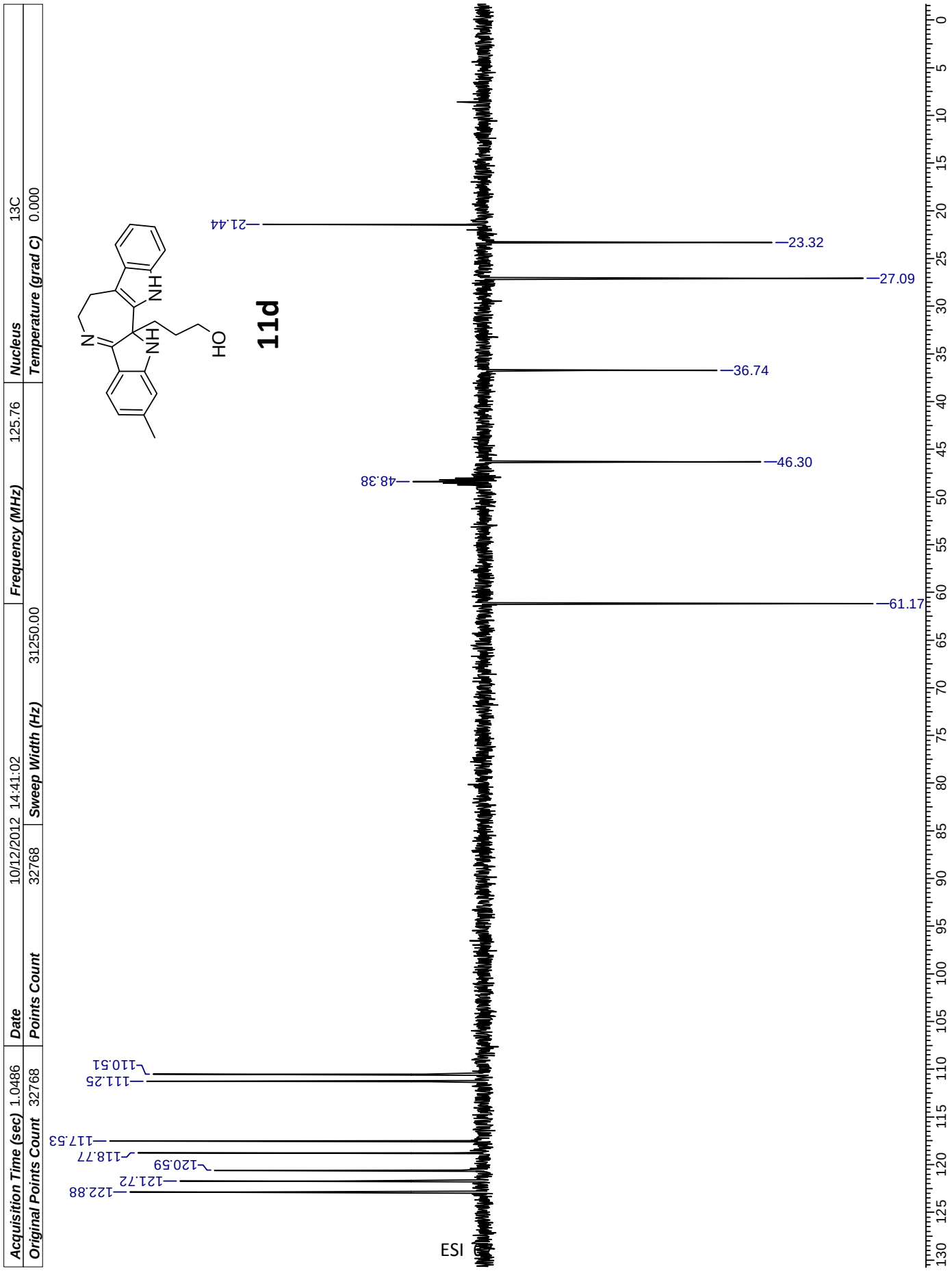

11d
Acquisition Time (sec) 3.2768
Nucleus 1H
Temperature (grad C) 0.000
Comment Reddy
Original Points Count 32768
10/12/2012 13:56:28
Date
Points Count 32768
Frequency (MHz) 500.13
Sweep Width (Hz) 10000.00
MeOD4
MeOD4
ESI 65

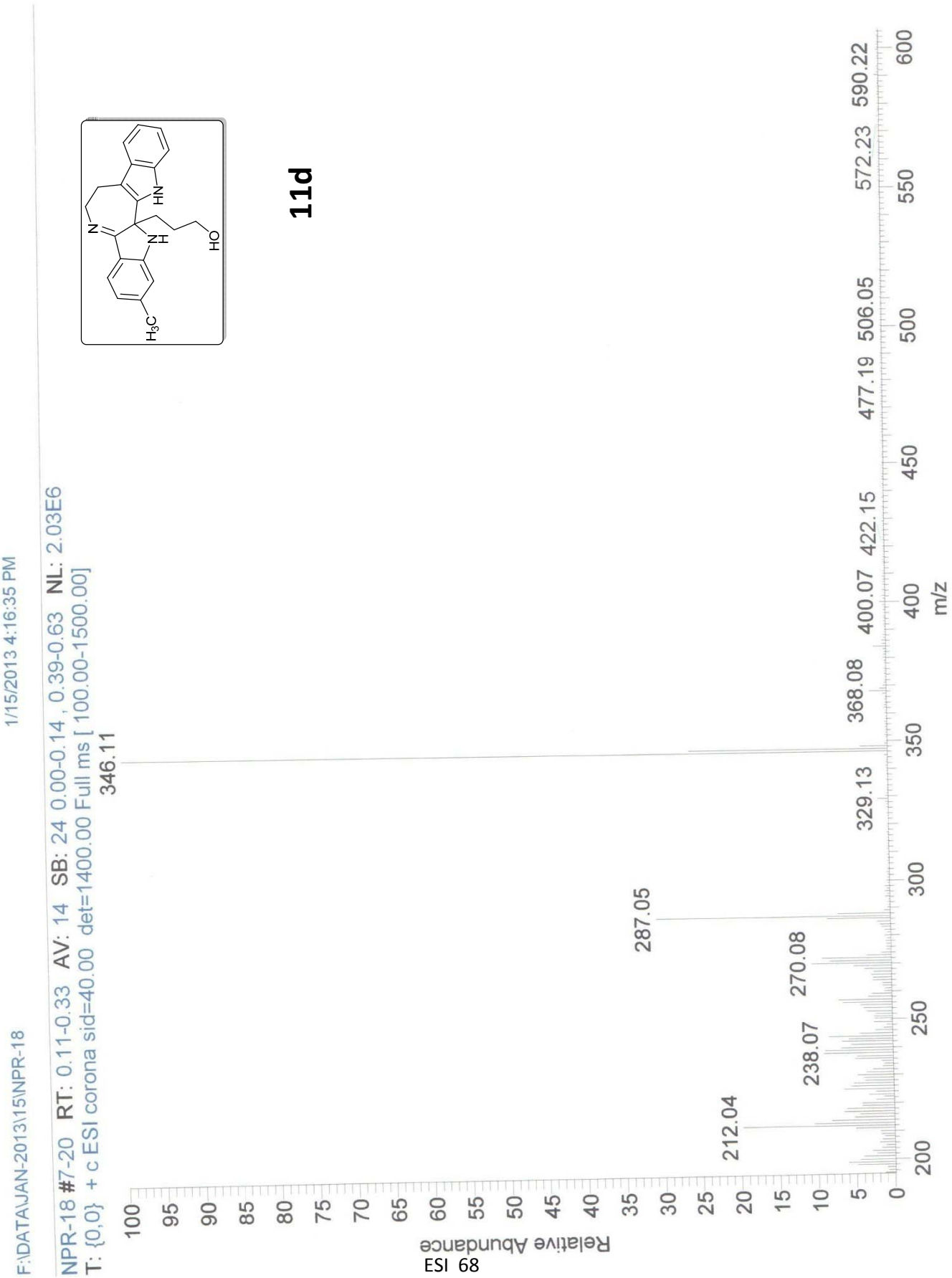
Acquisition Time (sec)	1.0486	*Date*	10/12/2012 15:55:06	*Frequency (MHz)*	125.76	*Nucleus*	13C
Original Points Count	32768	*Points Count*	32768	*Sweep Width (Hz)*	31250.00	*Temperature (grad C)*	0.000



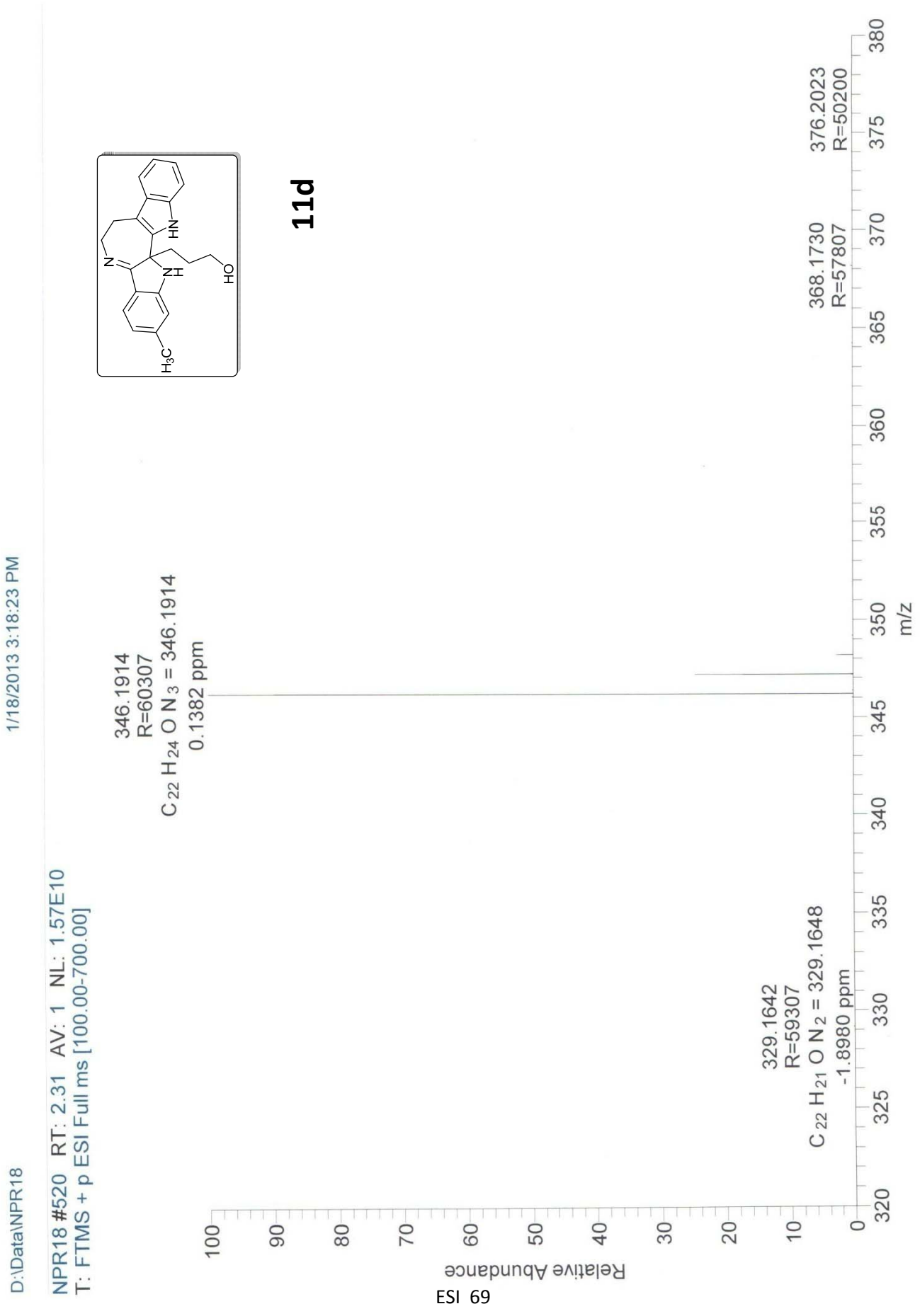
11d

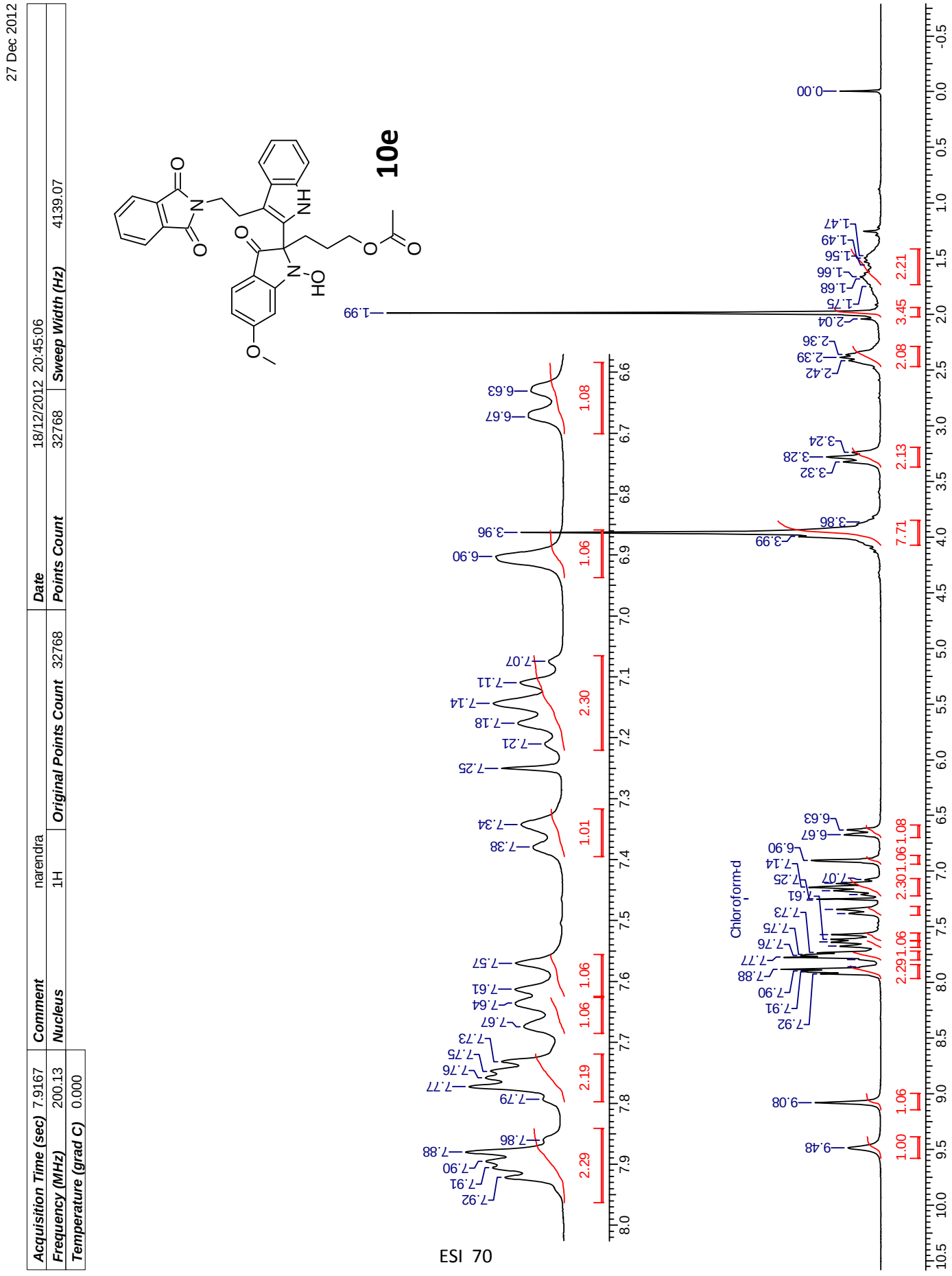


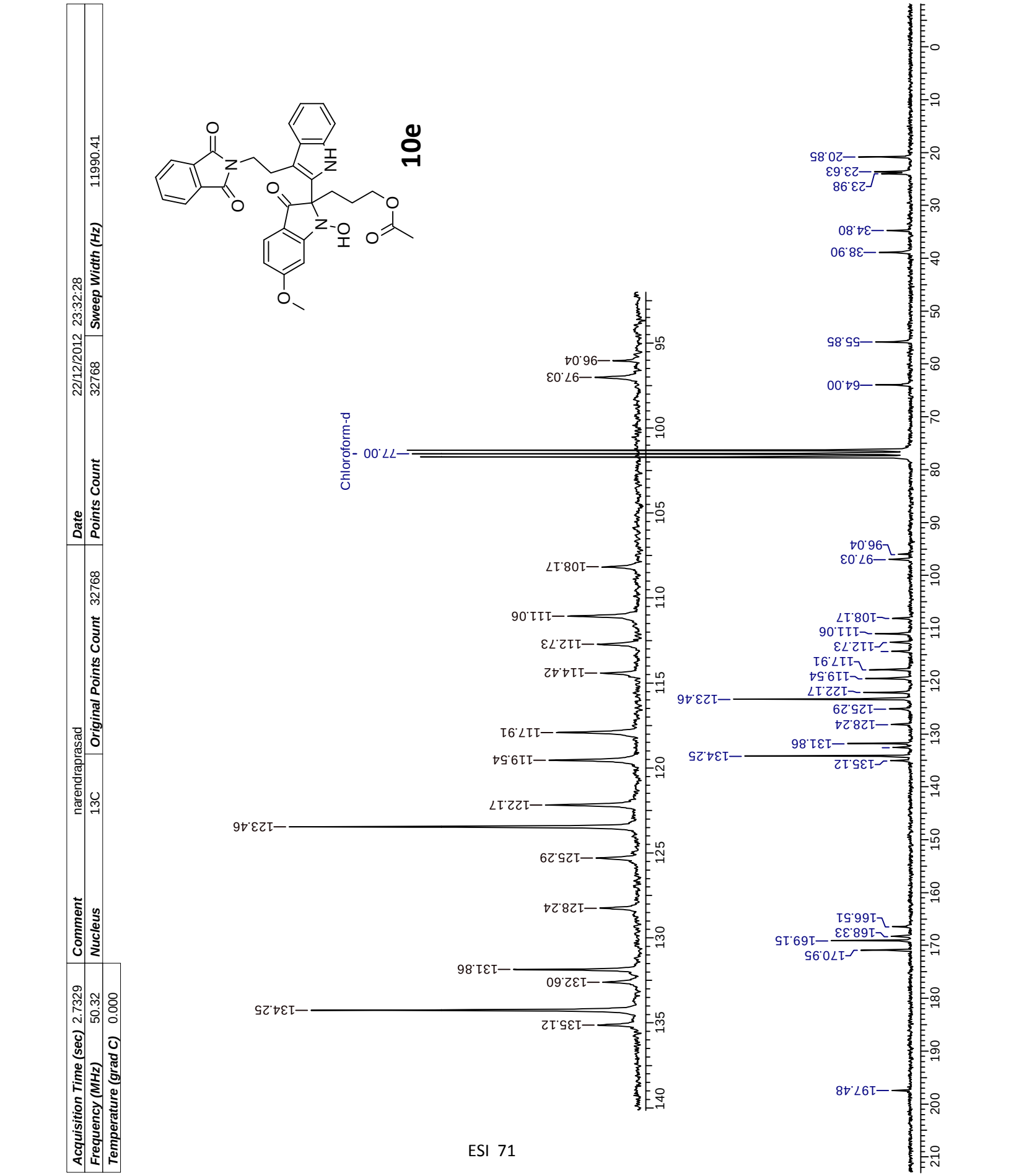

Acquisition Time (sec) 1.0486
Original Points Count 32768
Date 10/12/2012 14:41:02
Points Count 32768
Sweep Width (Hz) 31250.00
Frequency (MHz) 125.76
Nucleus 13C
Temperature (grad C) 0.000
11d
ESI
21.44
23.32
27.09
36.74
46.30
48.38
61.17
110.51
111.25
117.53
118.77
120.59
121.72
122.88


F:\DATA\JAN-2013\15\NPR-18
1/15/2013 4:16:35 PM
NPR-18 #7-20 RT: 0.11-0.33 AV: 14 SB: 24 0.00-0.14 , 0.39-0.63 NL: 2.03E6
T: {0,0} + c ESI corona sid=40.00 det=1400.00 Full ms [100.00-1500.00]
346.11
287.05
212.04
238.07
270.08
329.13
368.08
400.07
422.15
477.19
506.05
572.23
590.22
Relative Abundance
m/z
HN
N
N
H
HO
H3C

11d


D:\Data\NPR18
1/18/2013 3:18:23 PM
NPR18 #520 RT: 2.31 AV: 1 NL: 1.57E10
T: FTMS + p ESI Full ms [100.00-700.00]
346.1914
R=60307
C 22 H 24 O N 3 = 346.1914
0.1382 ppm
329.1642
R=59307
C 22 H 21 O N 2 = 329.1648
-1.8980 ppm
368.1730
R=57807
376.2023
R=50200
Relative Abundance
m/z
11d

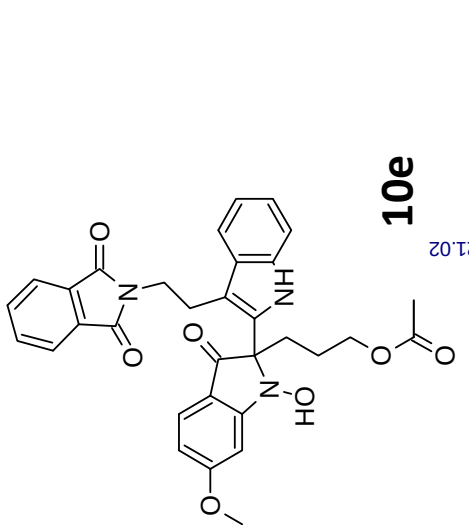

27 Dec 2012
Acquisition Time (sec) 7.9167
Frequency (MHz) 200.13
Temperature (grad C) 0.000
Comment
Nucleus 1H
narendra
Original Points Count 32768
Date 18/12/2012 20:45:06
Points Count 32768
Sweep Width (Hz) 4139.07
10e
ESI 70

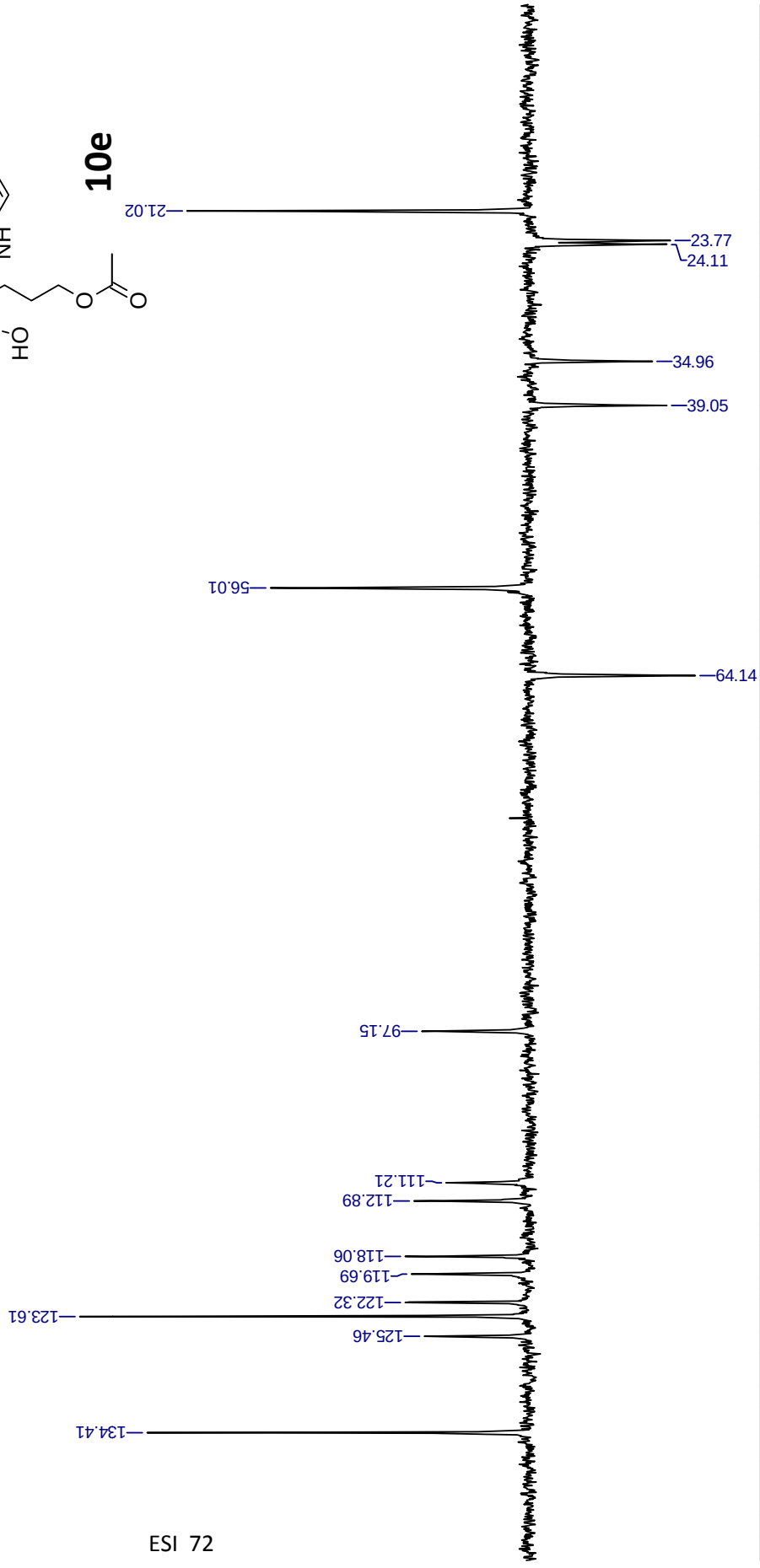


Acquisition Time (sec)	2.7329	*Comment*		*Date*	22/12/2012 23:32:28
Frequency (MHz)	50.32	*Nucleus*	13C	*Original Points Count*	32768
Temperature (grad C)	0.000			*Points Count*	32768
		narendraprasad		*Sweep Width (Hz)*	11990.41

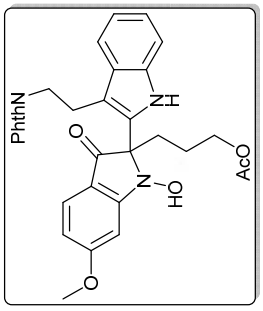
27 Dec 2012

Acquisition Time (sec)	2.7329	*Comment*	narendraprasad	*Date*	22/12/2012 20:28:22
Frequency (MHz)	50.32	*Nucleus*	13C	*Original Points Count*	32768
Temperature (grad C)	0.000			*Points Count*	32768
				Sweep Width (Hz)	11990.41

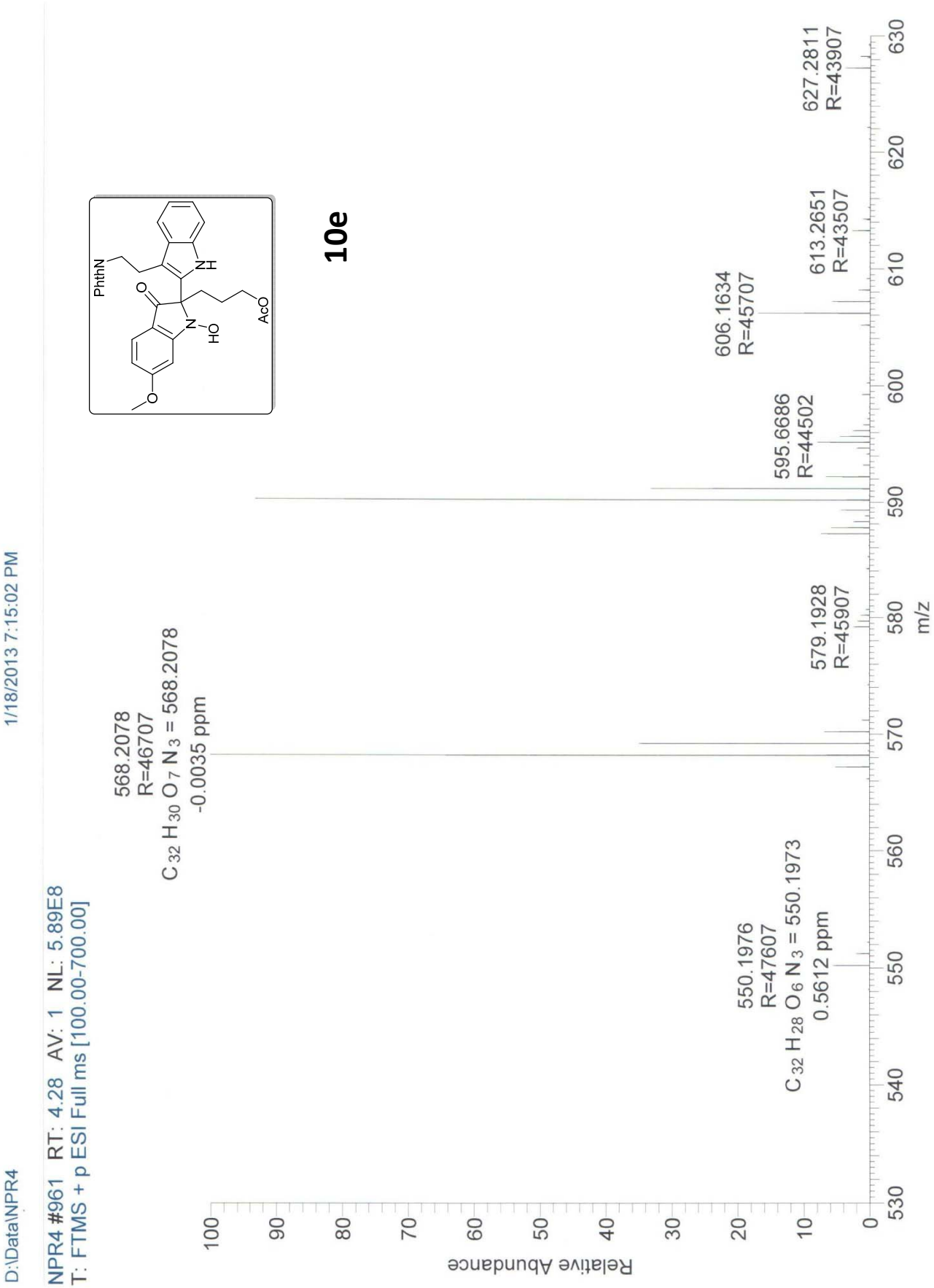


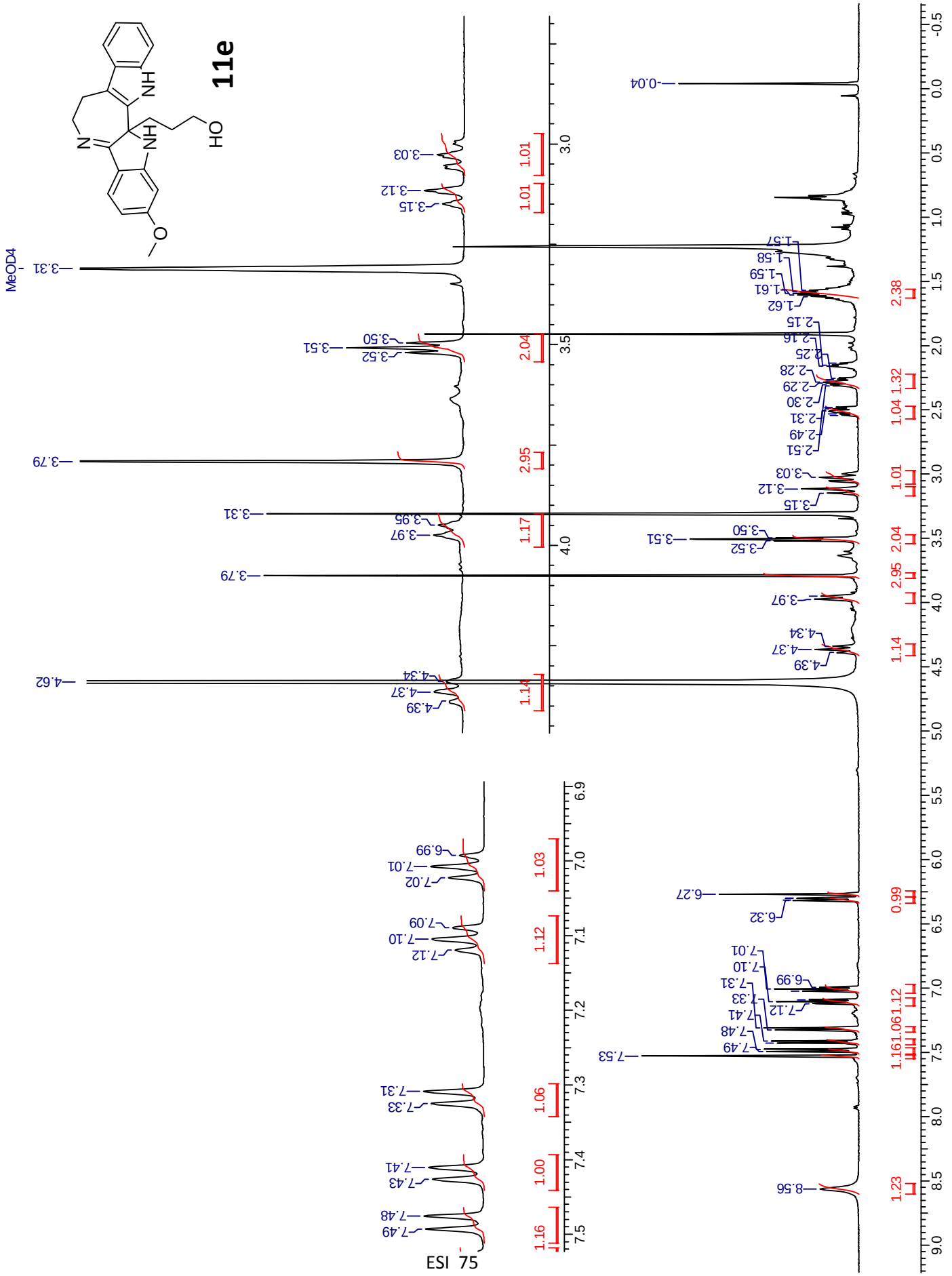


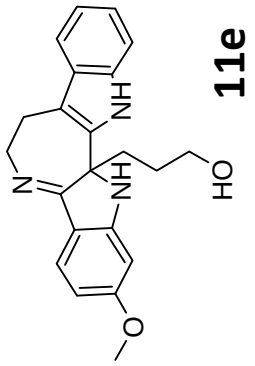
10e


PhthN
O
N
H
N
HO
AcO
O

F:\DATA\JAN-2013\15\NPR-04
1/15/2013 3:44:56 PM
NPR-04 #7-25 RT: 0.11-0.42 AV: 19 SB: 26 0.00-0.11 , 0.44-0.76 NL: 1.75E6
T: {0,0} + c ESI corona sid=40.00 det=1400.00 Full ms [100.00-1500.00]
590.16
606.12
214.09
301.09
413.09
472.10
573.16
634.13
688.09
769.33
826.14
894.36
928.32
Relative Abundance
m/z

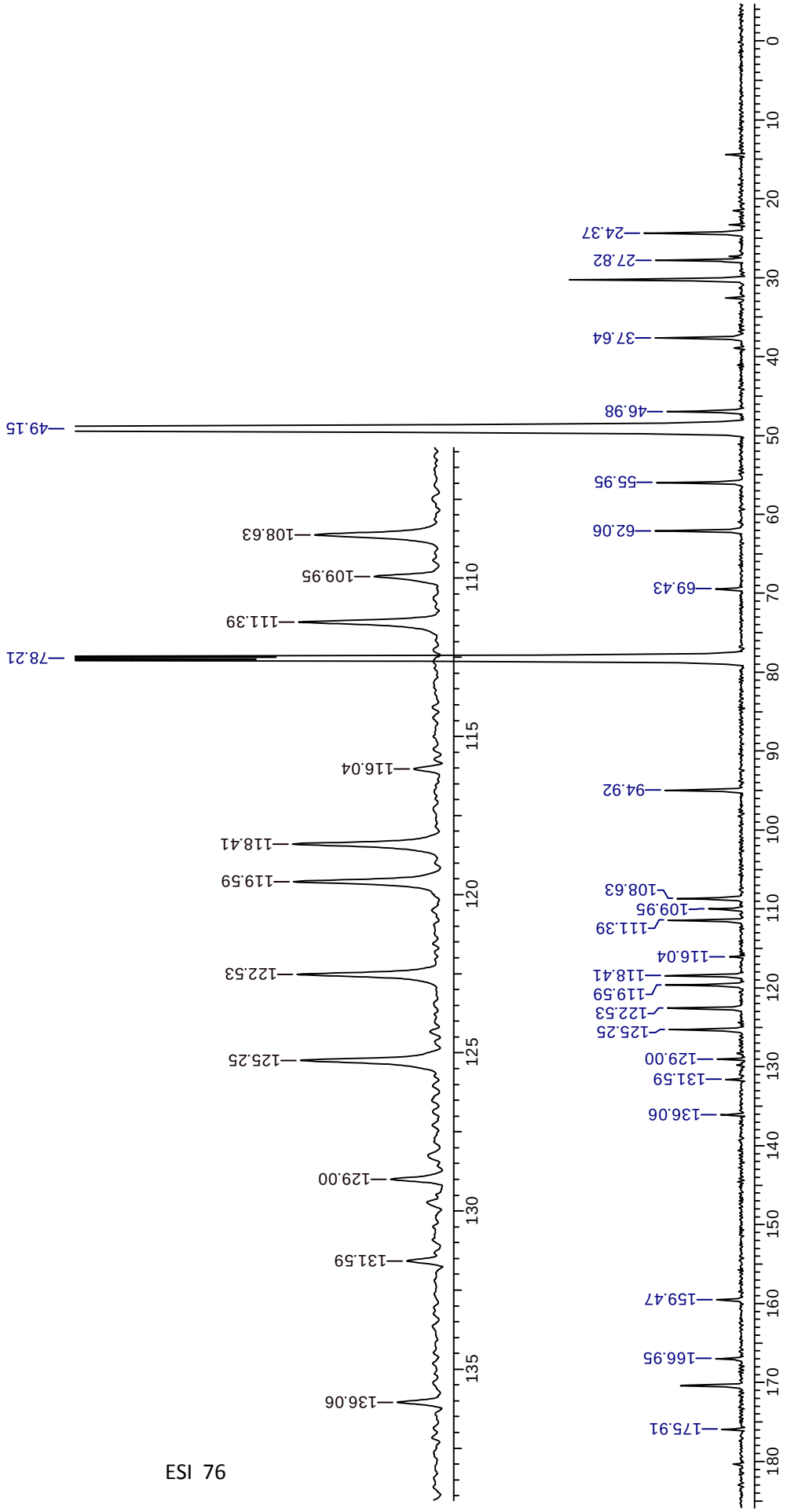

D:\Data\NPR4
1/18/2013 7:15:02 PM
NPR4 #961 RT: 4.28 AV: 1 NL: 5.89E8
T: FTMS + p ESI Full ms [100.00-700.00]
10e
PhthN
AcO
HO
568.2078
R=46707
C 32 H 30 O 7 N 3 = 568.2078
-0.0035 ppm
550.1976
R=47607
C 32 H 28 O 6 N 3 = 550.1973
0.5612 ppm
579.1928
R=45907
595.6686
R=44502
606.1634
R=45707
613.2651
R=43507
627.2811
R=43907
Relative Abundance
m/z

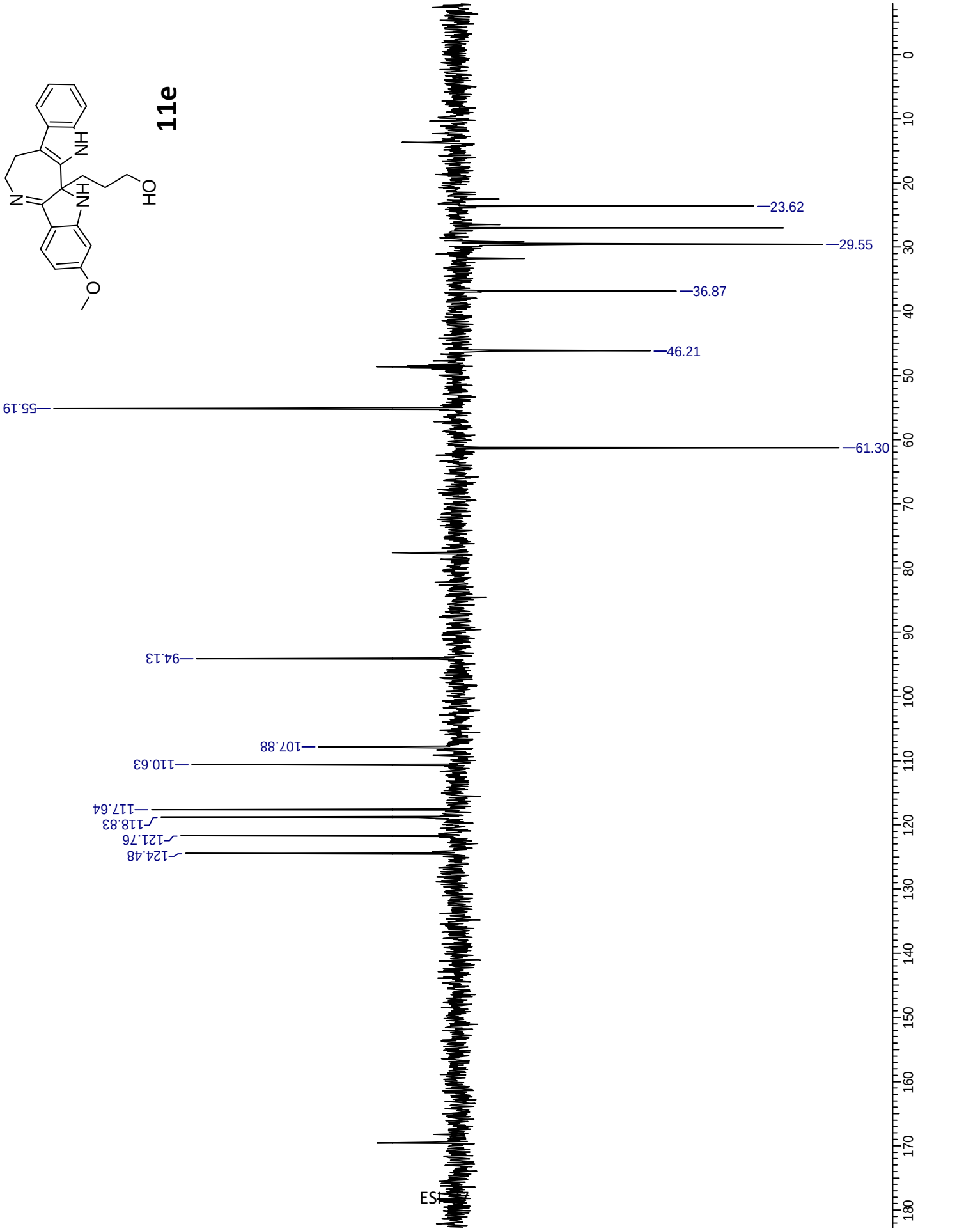

11e
NH
NH
N
HO
O
MeOD4
ESI 75



11e

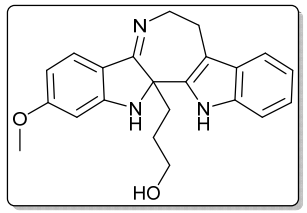
Acquisition Time (sec)	1.0486	***Comment***		***Frequency (MHz)***	125.76
Nucleus	13C	***Number of Transients***	12029	***Solvent***	METHANOL-d4
Sweep Width (Hz)	31249.05				
		Date	29 Jan 2013 09:40:32		
		Original Points Count	32768	***Points Count***	32768
		Temperature (grad C)	23.400		



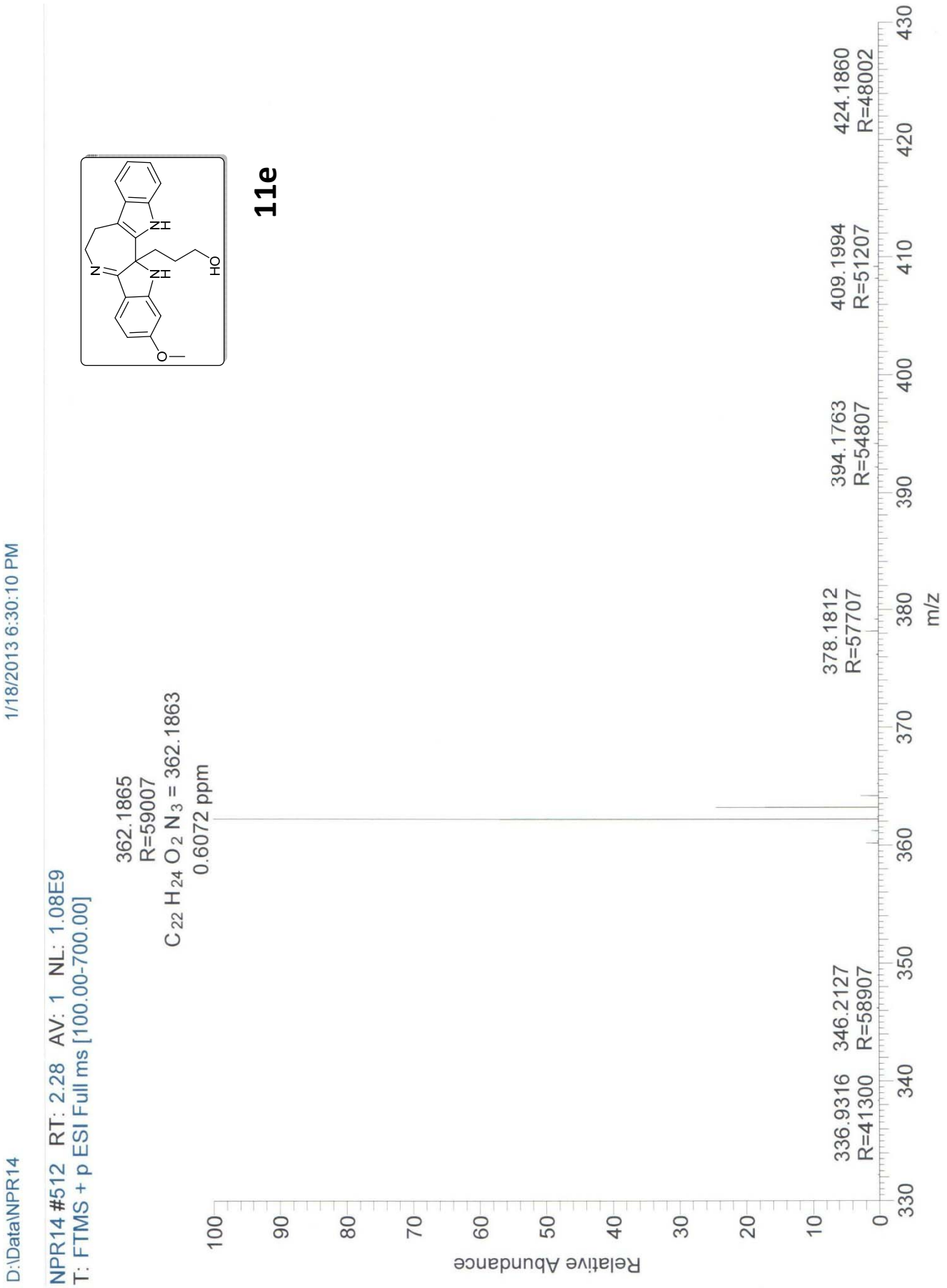


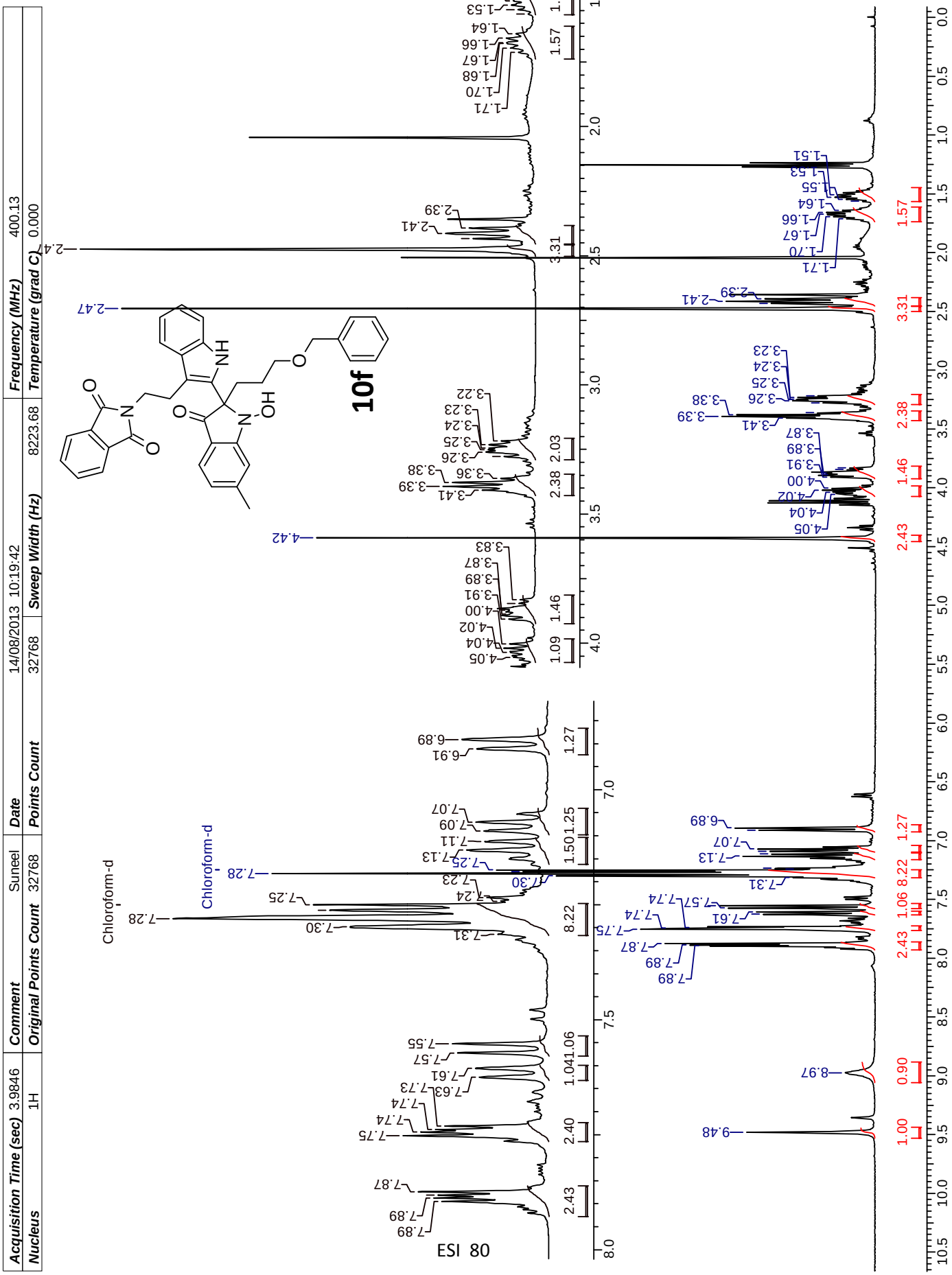
F:\DATA\JAN-2013\15\NPR-14 1/15/2013 4:05:55 PM

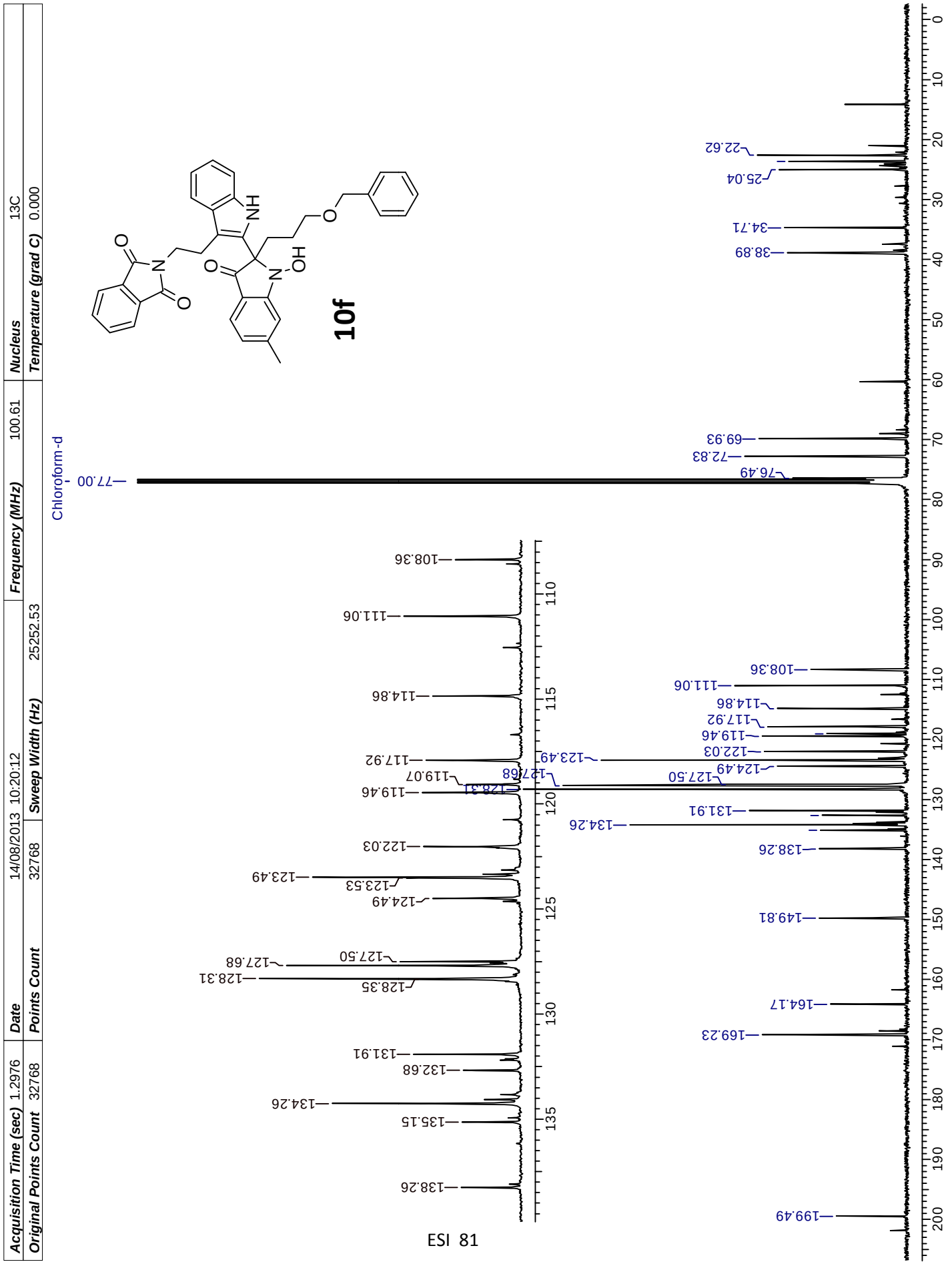
NPR-14 #7-20 RT: 0.11-0.33 AV: 14 SB: 25 0.00-0.11 , 0.37-0.67 NL: 7.47E4
T: {0,0} + c ESI corona sid=40.00 det=1400.00 Full ms [100.00-1500.00]

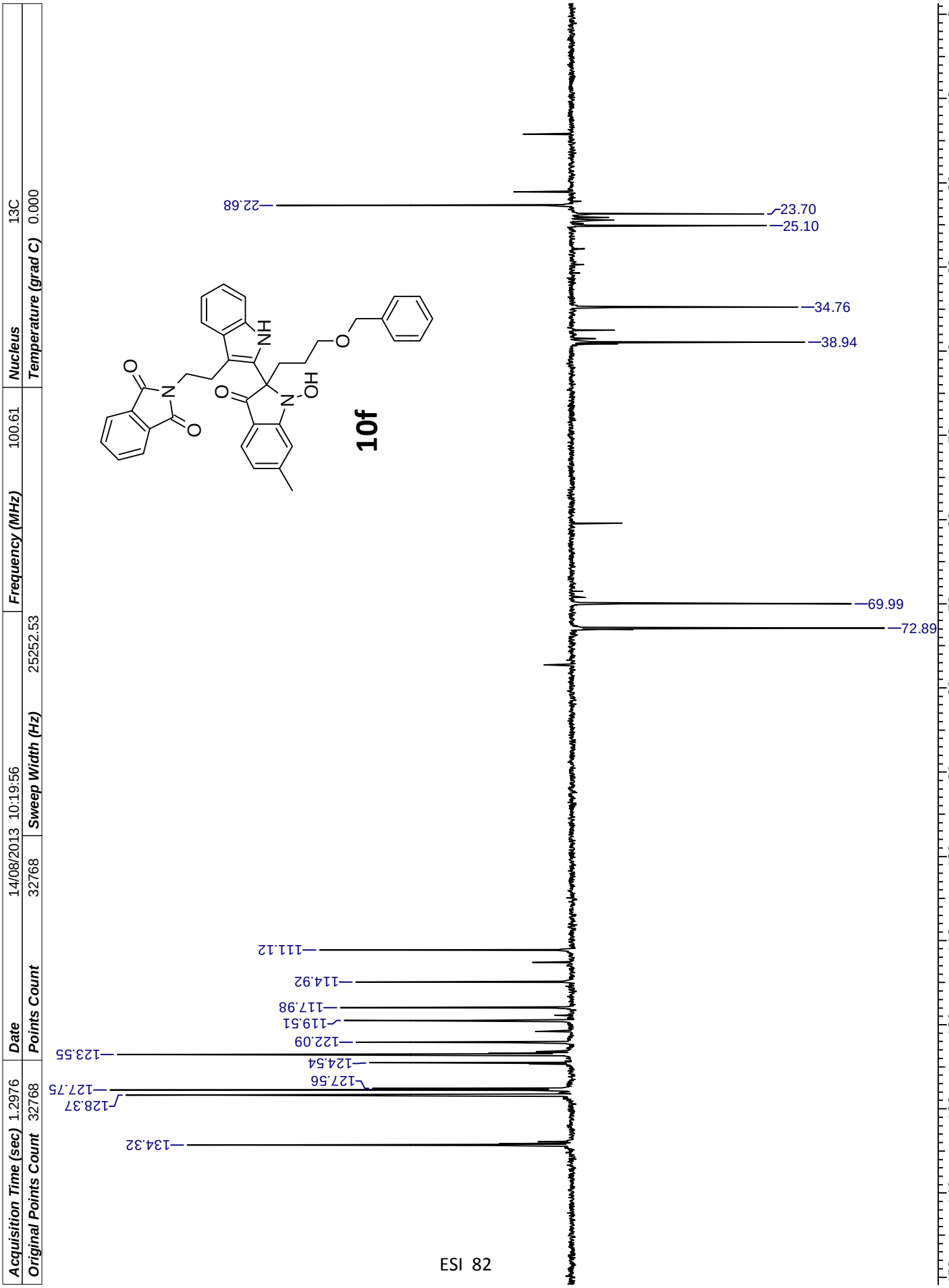


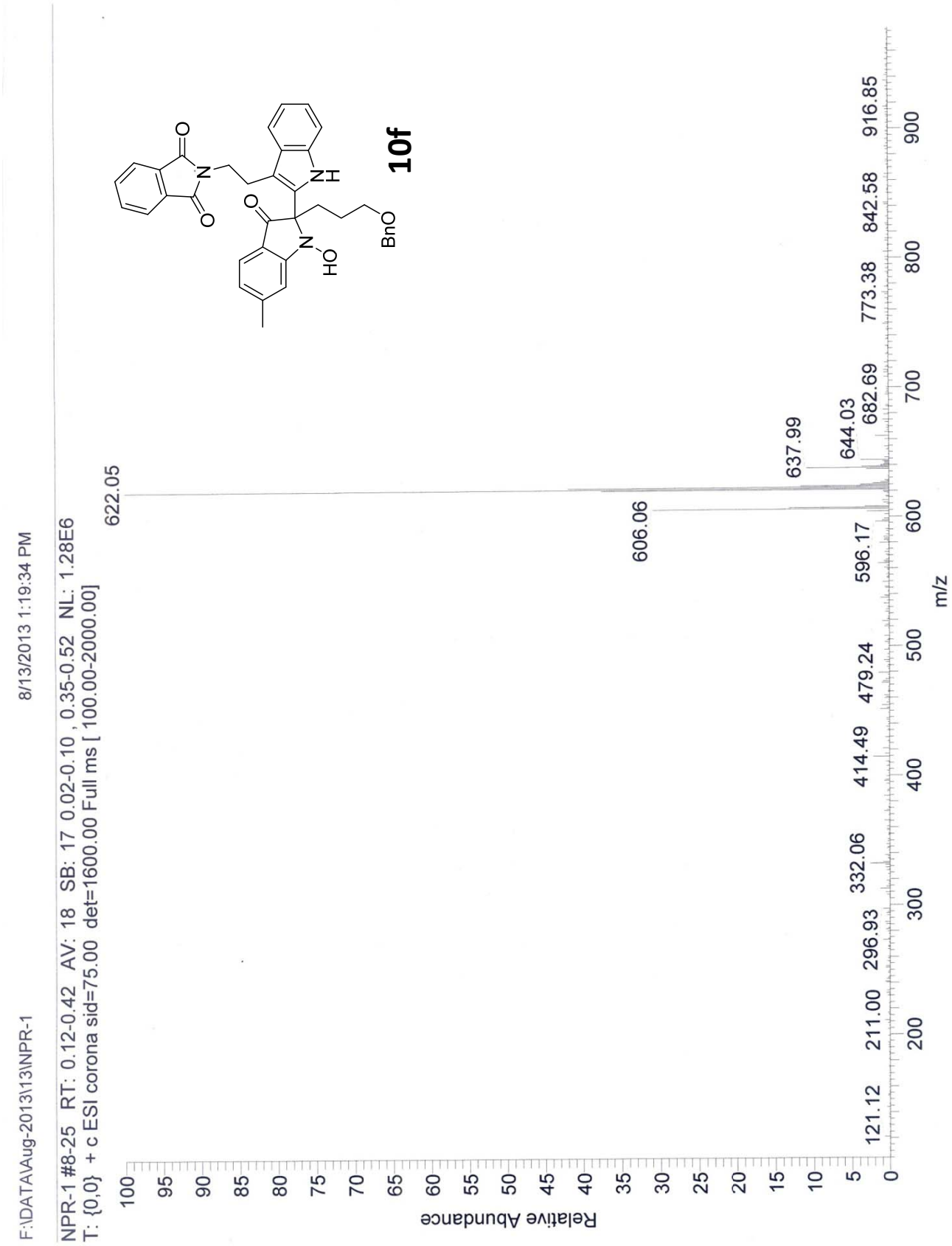
11e

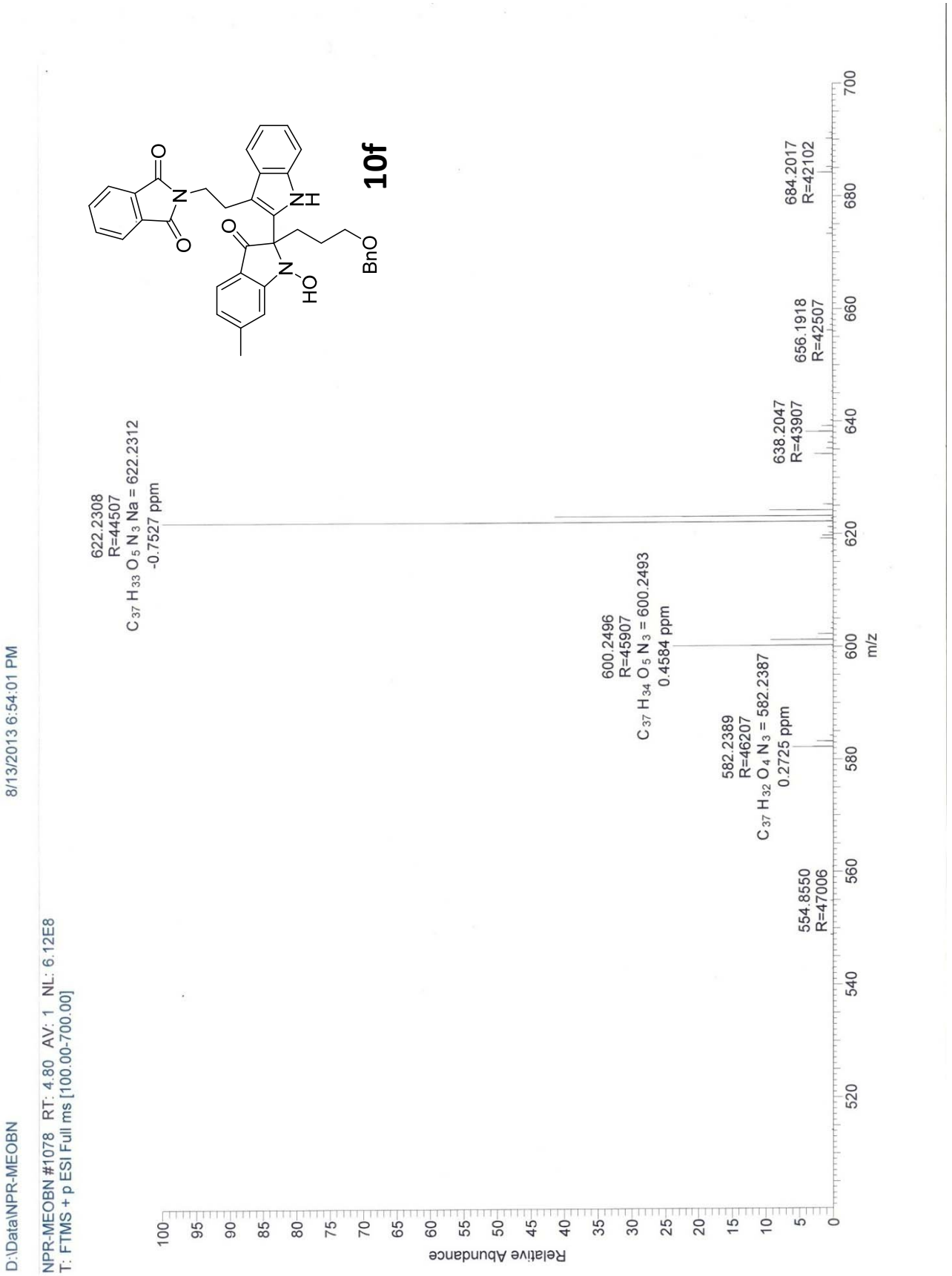

D:\Data\NPR14
1/18/2013 6:30:10 PM
NPR14 #512 RT: 2.28 AV: 1 NL: 1.08E9
T: FTMS + p ESI Full ms [100.00-700.00]
362.1865
R=59007
C 22 H 24 O 2 N 3 = 362.1863
0.6072 ppm
336.9316
R=41300
346.2127
R=58907
378.1812
R=57707
394.1763
R=54807
409.1994
R=51207
424.1860
R=48002
Relative Abundance
m/z
11e

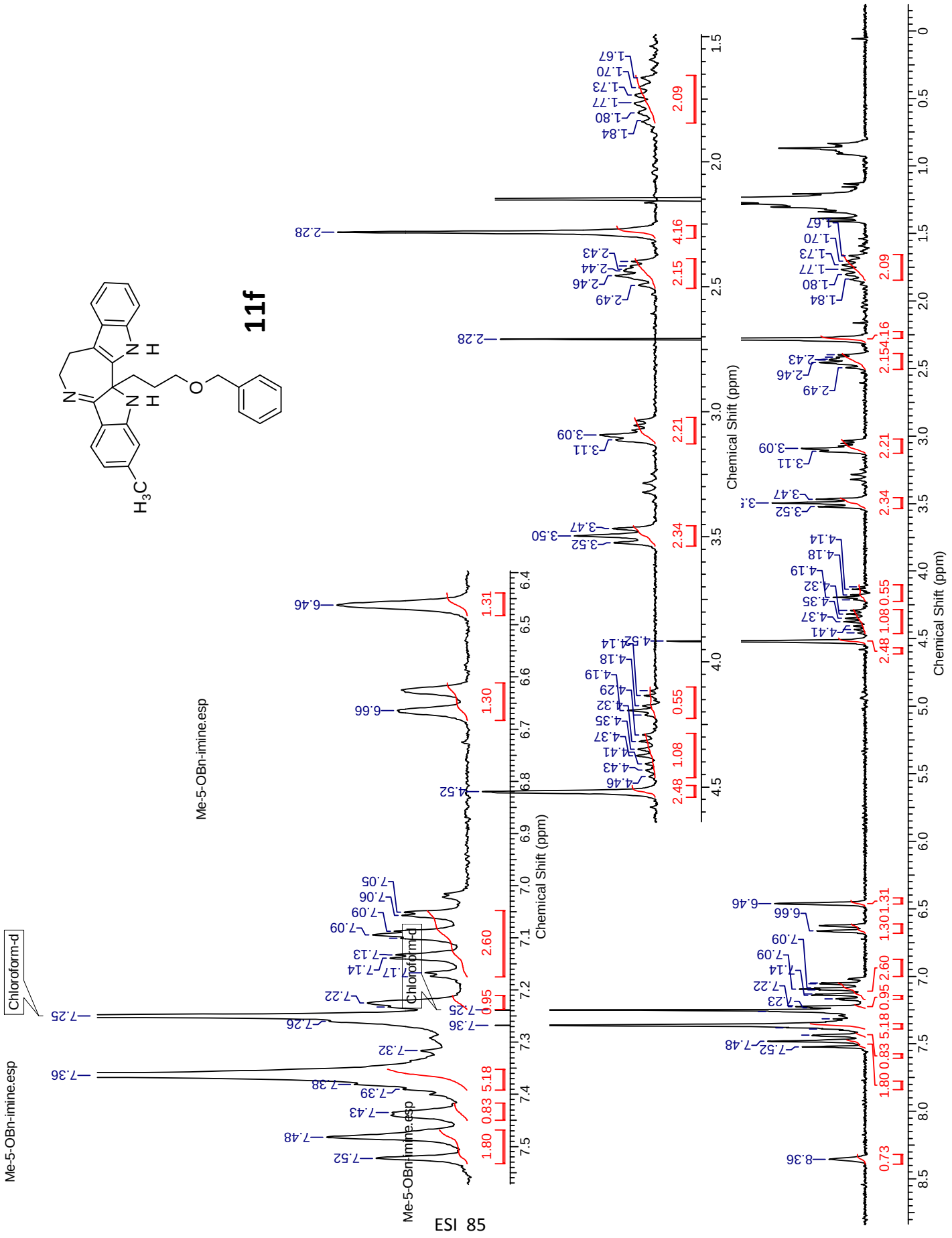

Acquisition Time (sec) 3.9846
Nucleus 1H
Comment
Original Points Count 32768
Suneel
Date 14/08/2013 10:19:42
Points Count 32768
Sweep Width (Hz) 8223.68
Frequency (MHz) 400.13
Temperature (grad C) 0.000
10f
Chloroform-d
ESI 80

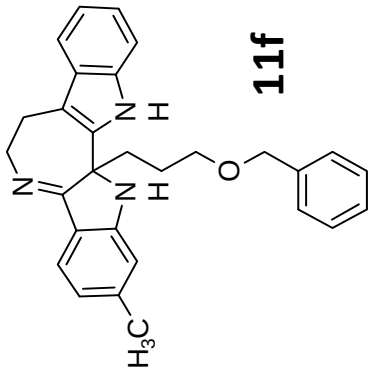

Acquisition Time (sec) 1.2976
Original Points Count 32768
Date 14/08/2013 10:20:12
Points Count 32768
Sweep Width (Hz) 25252.53
Frequency (MHz) 100.61
Nucleus 13C
Temperature (grad C) 0.000
Chloroform-d
77.00
10f
ESI 81


Acquisition Time (sec) 1.2976
Original Points Count 32768
Date 14/08/2013 10:19:56
Points Count 32768
Sweep Width (Hz) 25252.53
Frequency (MHz) 100.61
Nucleus 13C
Temperature (grad C) 0.000
22.68
23.70
25.10
34.76
38.94
69.99
72.89
111.12
114.92
117.98
119.51
122.09
123.55
124.54
127.56
127.75
128.37
134.32
10f

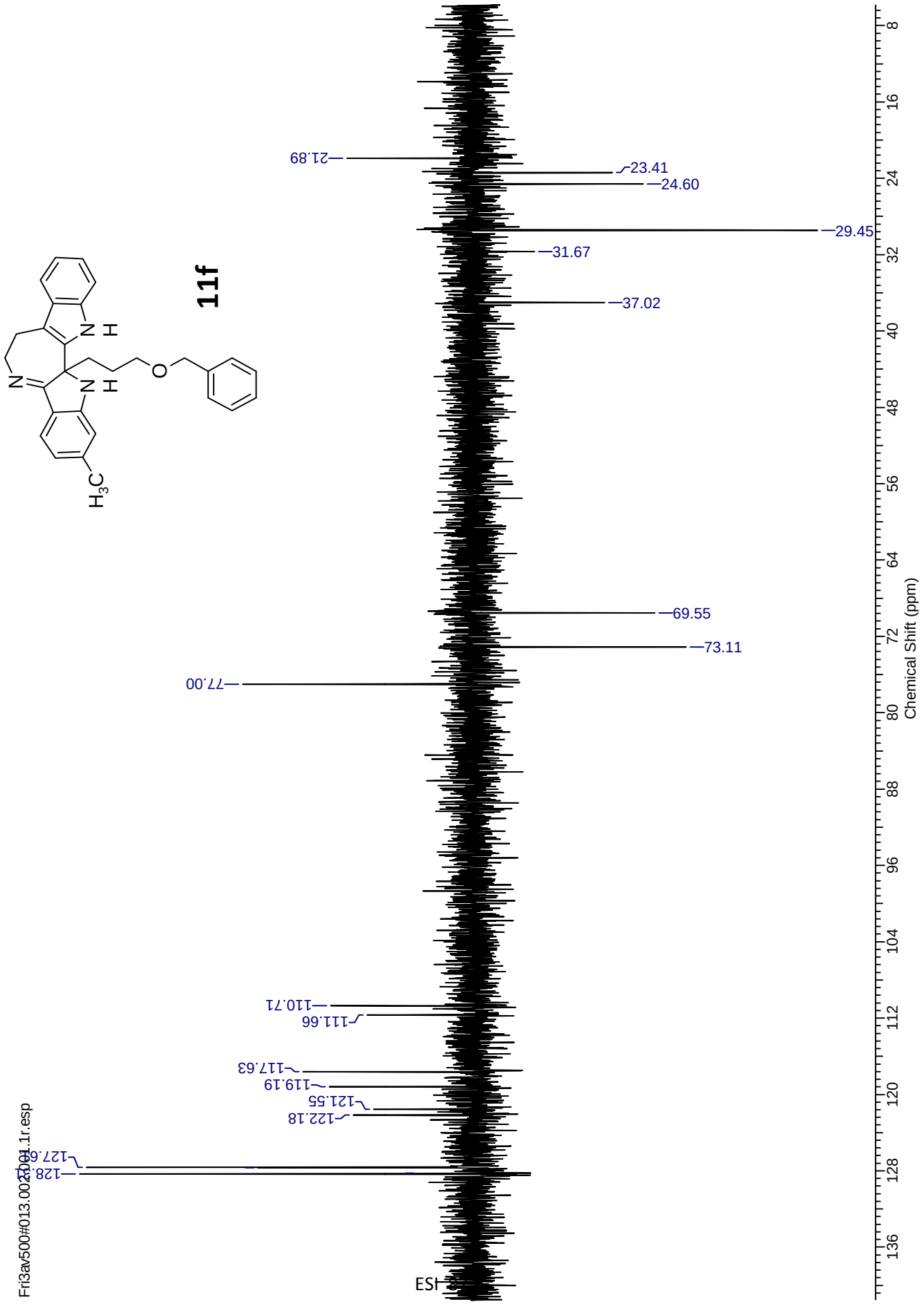

F:\DATA\Aug-2013\13\NPR-1
8/13/2013 1:19:34 PM
NPR-1 #8-25 RT: 0.12-0.42 AV: 18 SB: 17 0.02-0.10 , 0.35-0.52 NL: 1.28E6
T: {0,0} + c ESI corona sid=75.00 det=1600.00 Full ms [100.00-2000.00]
10f
BnO
HO
N
H
O
Relative Abundance
m/z
622.05
606.06
637.99
644.03
682.69
773.38
842.58
916.85
596.17
479.24
414.49
332.06
296.93
211.00
121.12

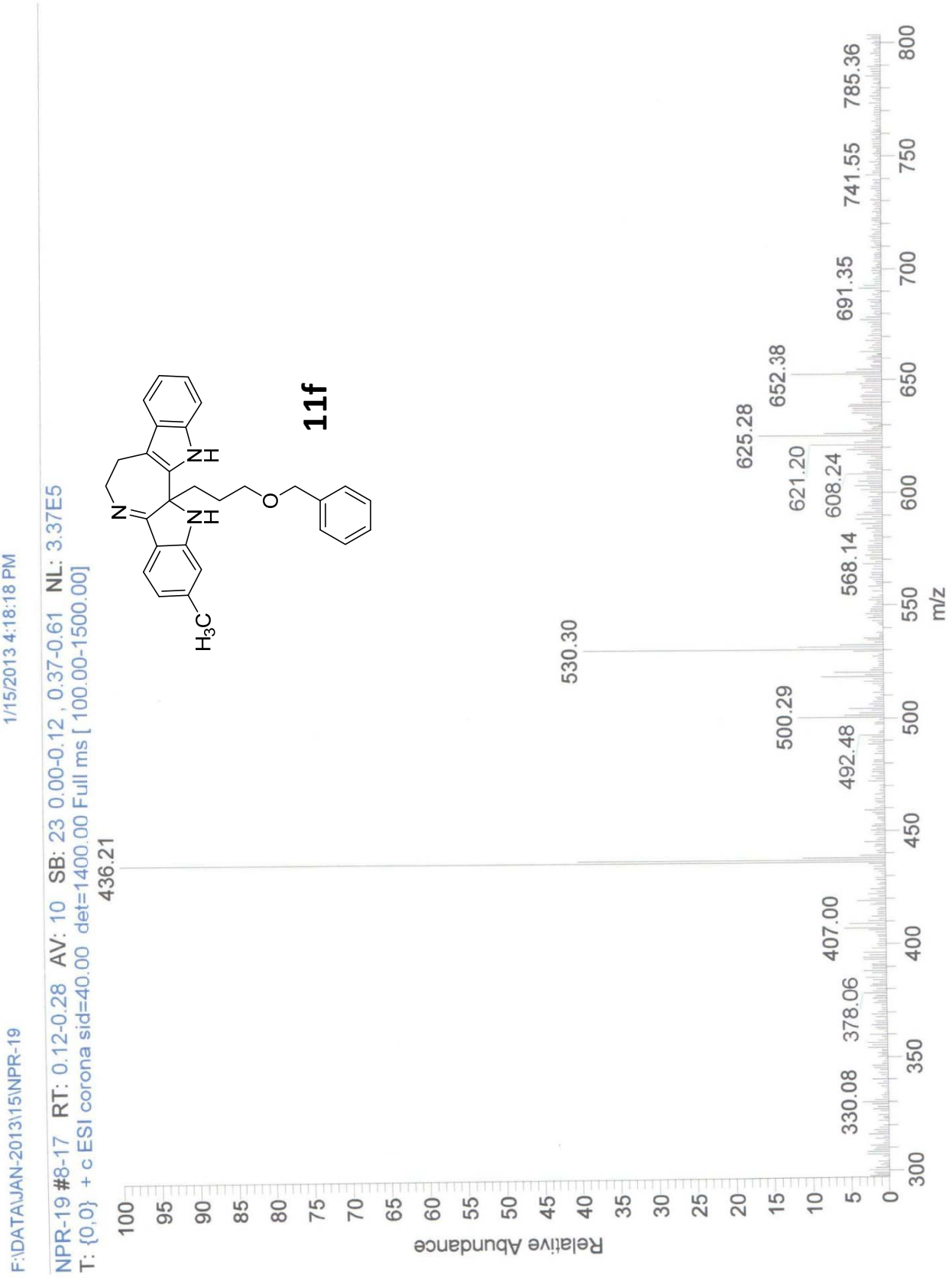

D:\Data\NPR-MEOBN
8/13/2013 6:54:01 PM
NPR-MEOBN #1078 RT: 4.80 AV: 1 NL: 6.12E8
T: FTMS + p ESI Full ms [100.00-700.00]
622.2308
R=44507
C 37 H 33 O 5 N 3 Na = 622.2312
-0.7527 ppm
600.2496
R=45907
C 37 H 34 O 5 N 3 = 600.2493
0.4584 ppm
582.2389
R=46207
C 37 H 32 O 4 N 3 = 582.2387
0.2725 ppm
554.8550
R=47006
638.2047
R=43907
656.1918
R=42507
684.2017
R=42102
Relative Abundance
m/z
BnO
HO
10f

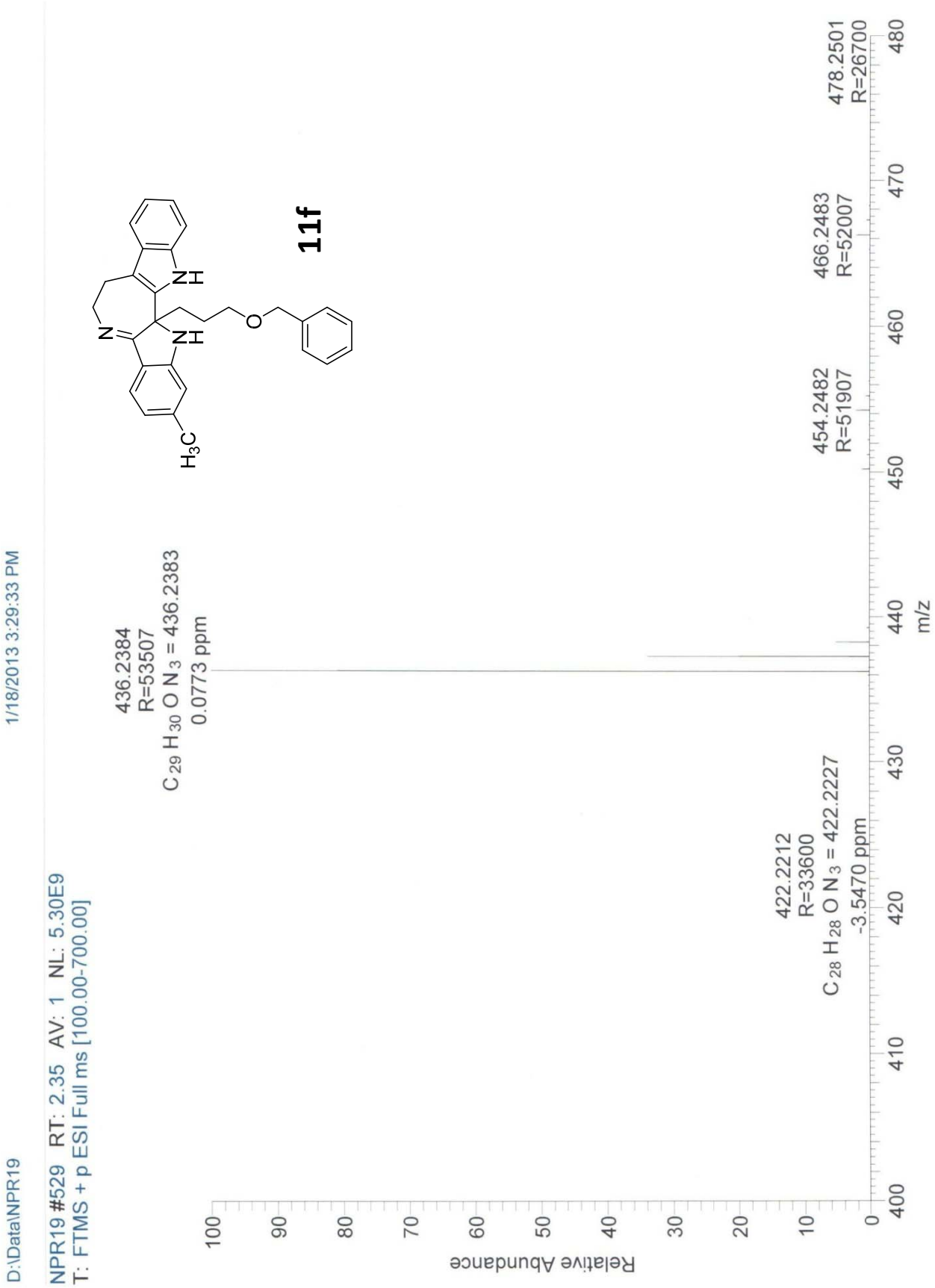

11f
Me-5-OBn-imine.esp
Chloroform-d
Chemical Shift (ppm)

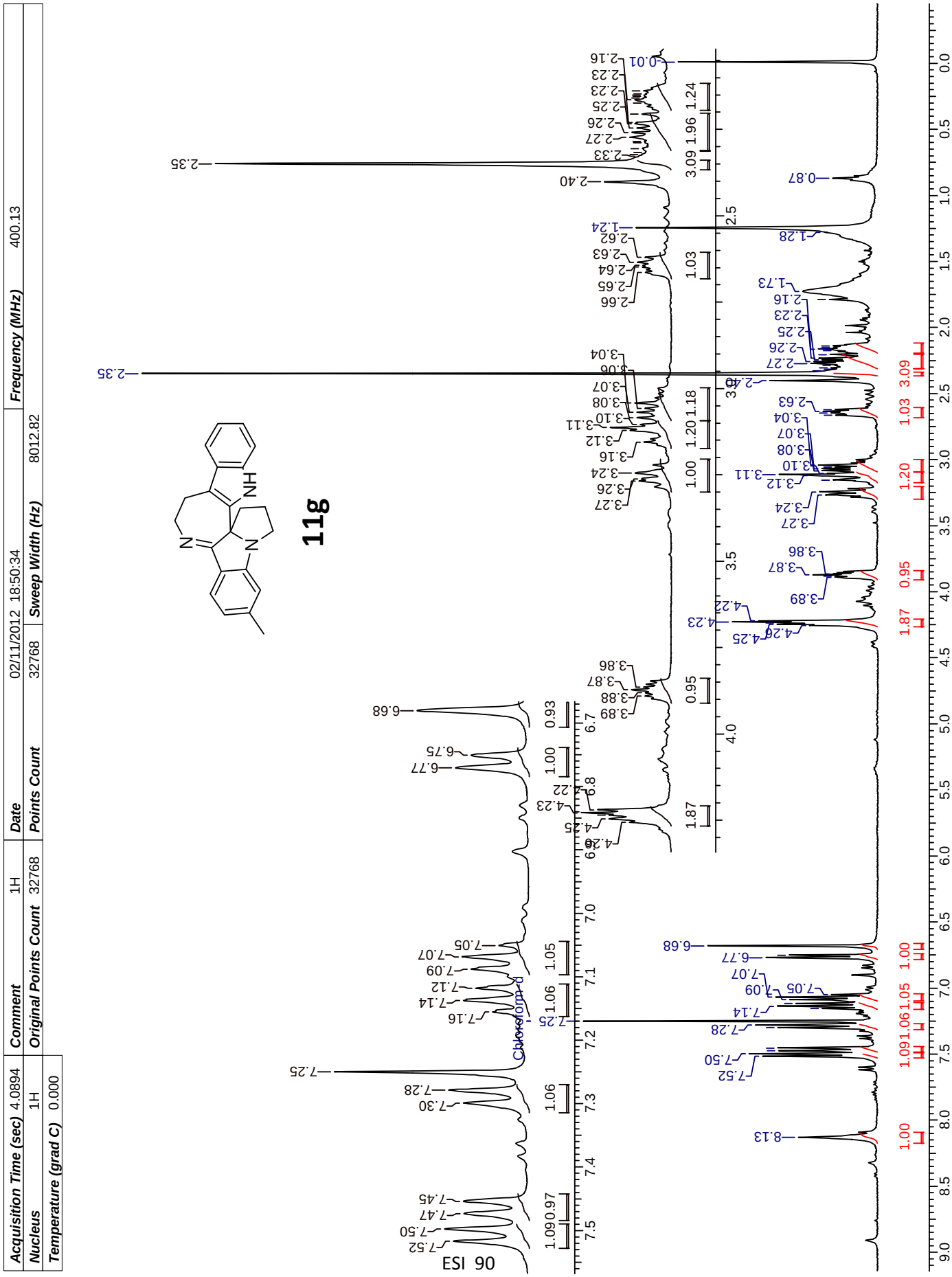

11f
N
H
N
H
O
N
H3C

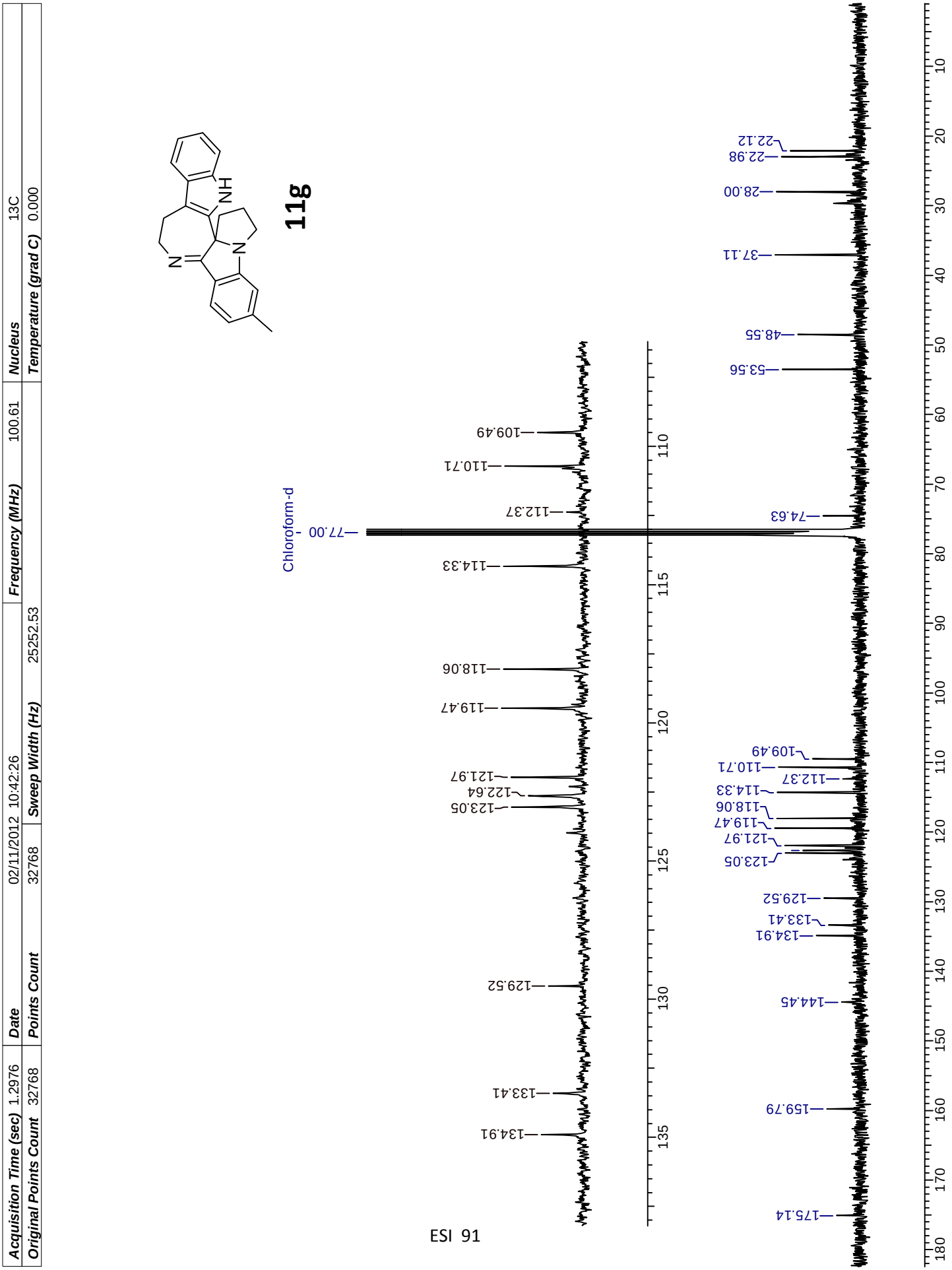
Fri3av500#013.003.001.1r.esp
Fri3av500#013.003.001.1r.esp
77.00
109.45
109.75
110.34
110.92
111.85
117.84
119.40
121.75
122.38
127.82
128.51
135.11
137.75
140.92
22.09
23.62
24.80
29.32
31.88
37.22
69.75
71.28
73.31
105.07
109.45
109.75
110.34
110.92
111.85
117.84
119.40
121.75
122.38
127.82
128.51
135.11
137.75
140.92
161.63
172.71
Chemical Shift (ppm)
Chemical Shift (ppm)

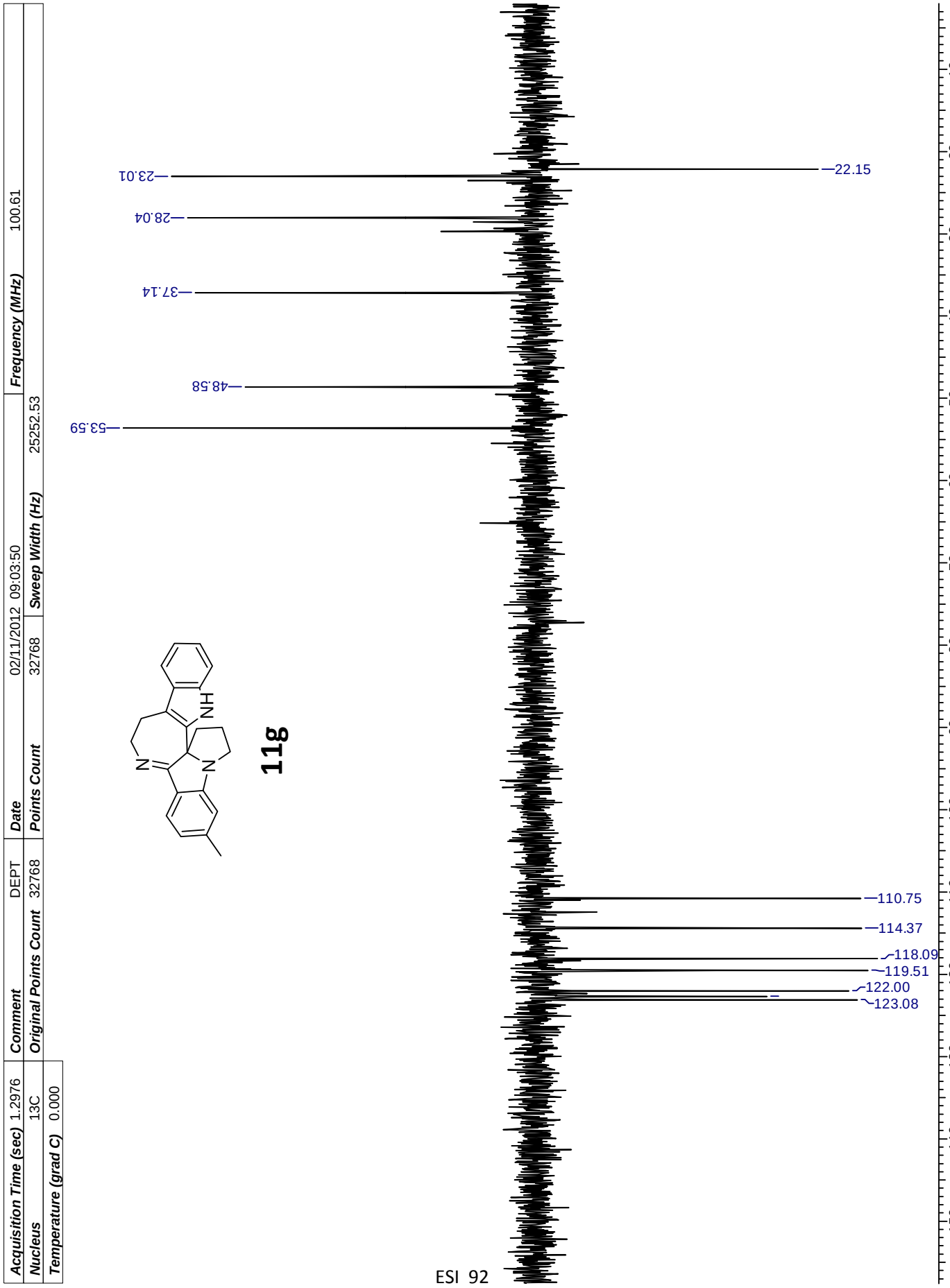

11f
H
N
N
H
O
H3C
21.89
23.41
24.60
29.45
31.67
37.02
69.55
73.11
77.00
110.71
111.66
117.63
119.19
121.55
122.18
127.68
128.12
ESI
Fri3av500#013.002.001.1r.esp
8
16
24
32
40
48
56
64
72
80
88
96
104
112
120
128
136
Chemical Shift (ppm)

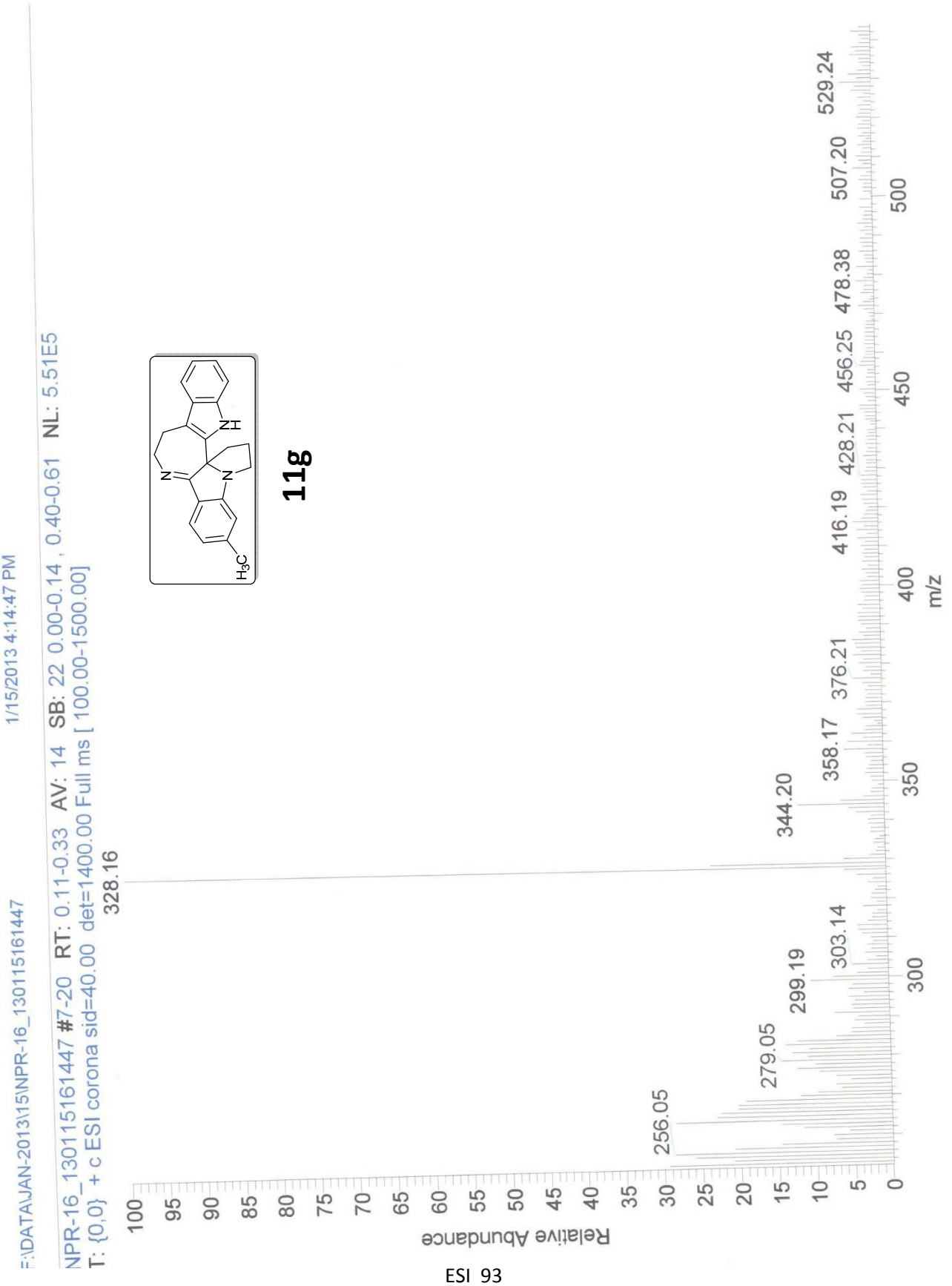

F:\DATA\JAN-2013\15\NPR-19
1/15/2013 4:18:18 PM
NPR-19 #8-17 RT: 0.12-0.28 AV: 10 SB: 23 0.00-0.12 , 0.37-0.61 NL: 3.37E5
T: {0,0} + c ESI corona sid=40.00 det=1400.00 Full ms [100.00-1500.00]
11f
Relative Abundance
m/z
436.21
530.30
500.29
492.48
625.28
652.38
621.20
608.24
568.14
691.35
741.55
785.36
330.08
378.06
407.00

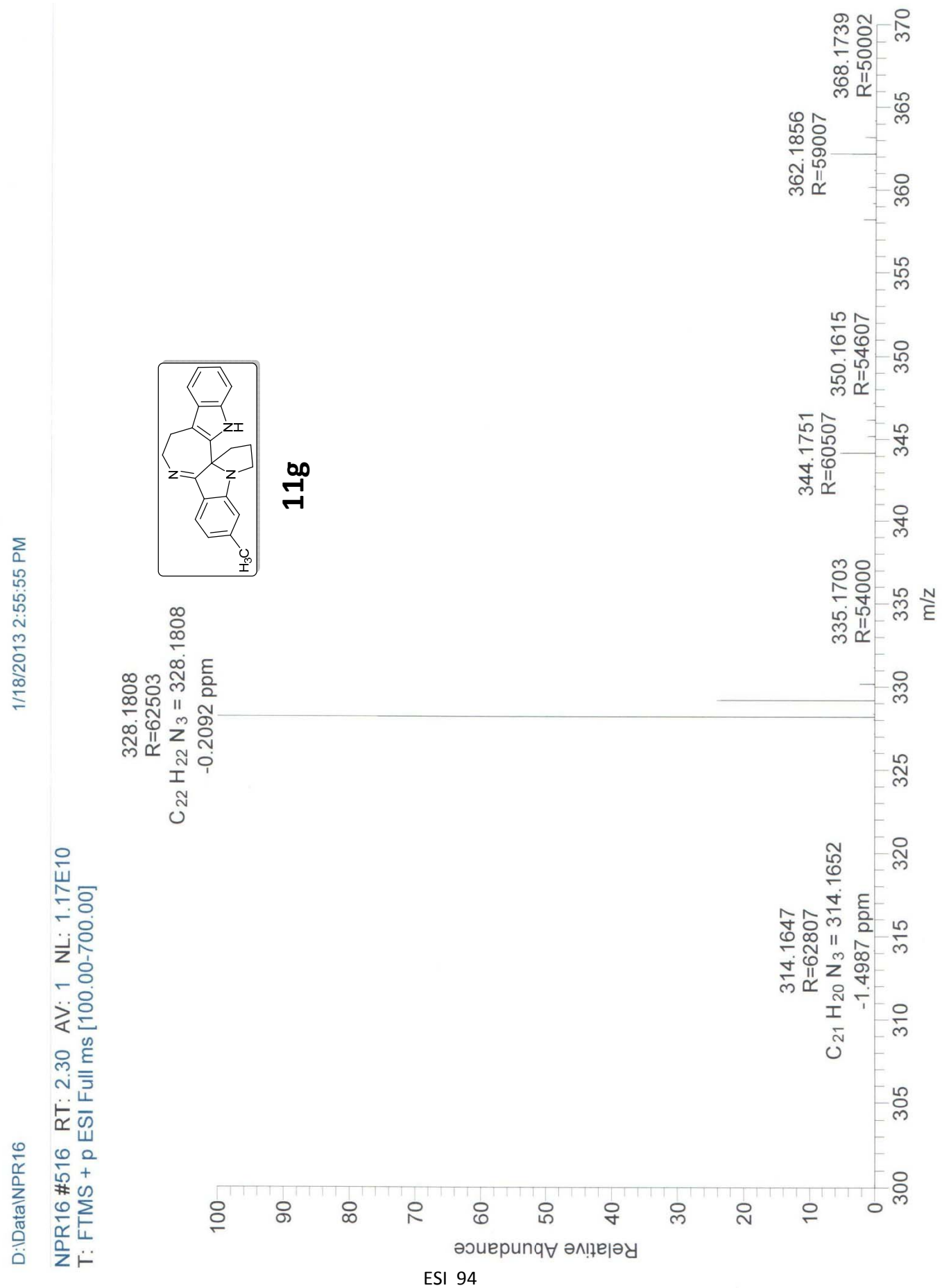

D:\Data\NPR19
1/18/2013 3:29:33 PM
NPR19 #529 RT: 2.35 AV: 1 NL: 5.30E9
T: FTMS + p ESI Full ms [100.00-700.00]
11f
436.2384
R=53507
C 29 H 30 O N 3 = 436.2383
0.0773 ppm
422.2212
R=33600
C 28 H 28 O N 3 = 422.2227
-3.5470 ppm
454.2482
R=51907
466.2483
R=52007
478.2501
R=26700
Relative Abundance
m/z

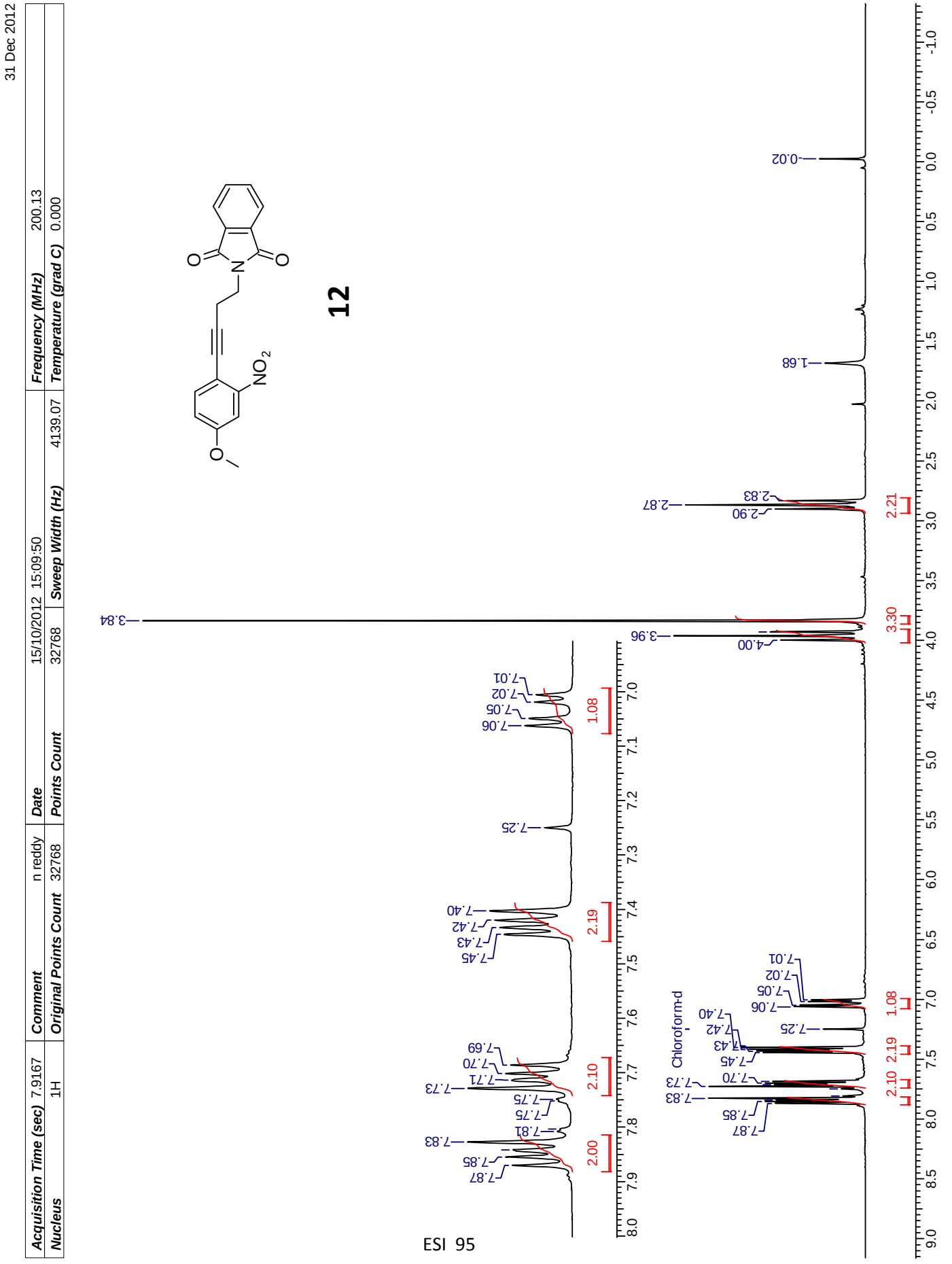


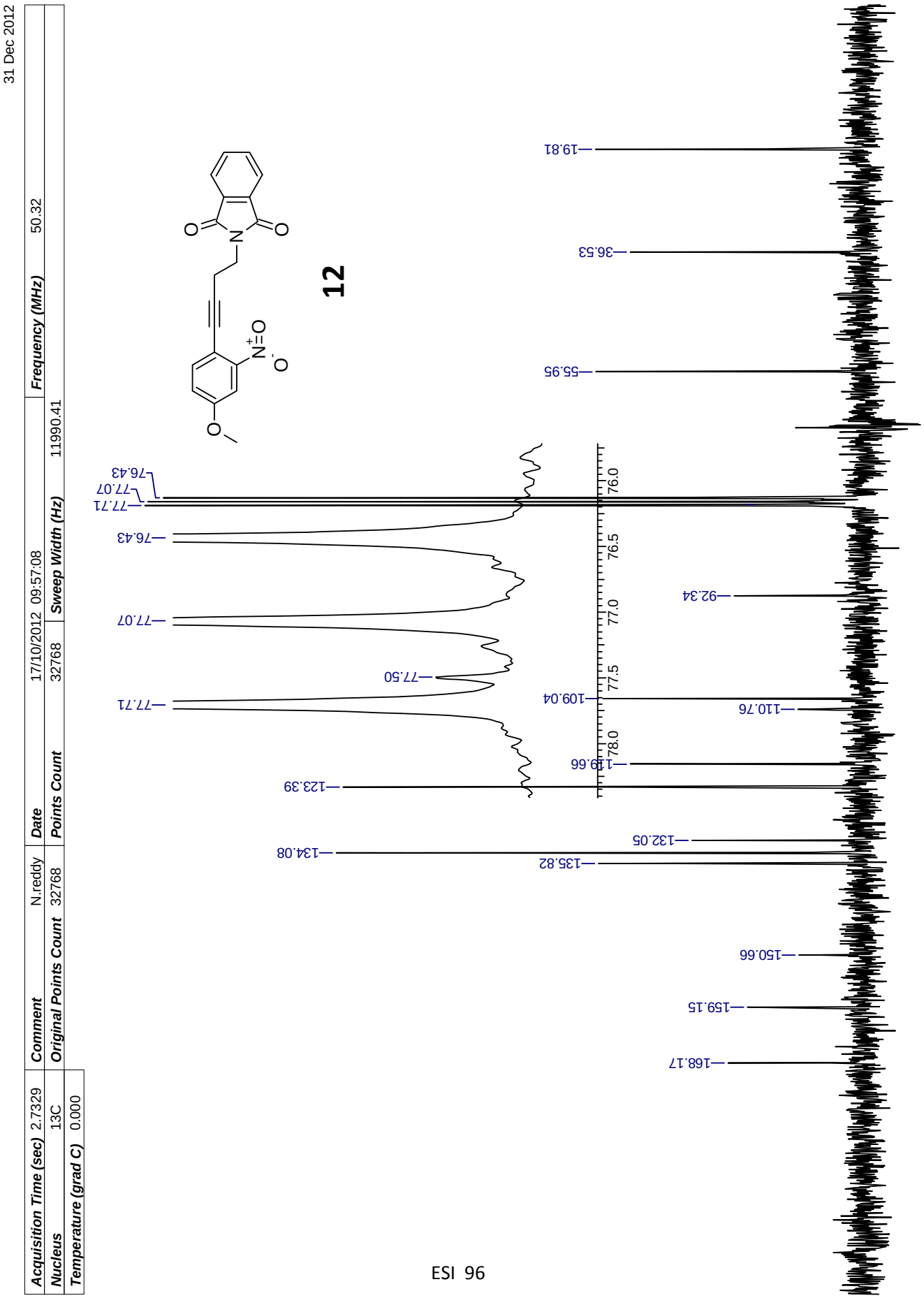

Acquisition Time (sec) 1.2976
Original Points Count 32768
Date 02/11/2012 10:42:26
Points Count 32768
Sweep Width (Hz) 25252.53
Frequency (MHz) 100.61
Nucleus 13C
Temperature (grad C) 0.000
11g
NH
N
N
Chloroform-d
77.00
109.49
110.71
112.37
114.33
118.06
119.47
121.97
122.64
123.05
129.52
133.41
134.91
144.45
159.79
175.14
74.63
48.55
53.56
37.11
28.00
22.98
22.12
ESI 91

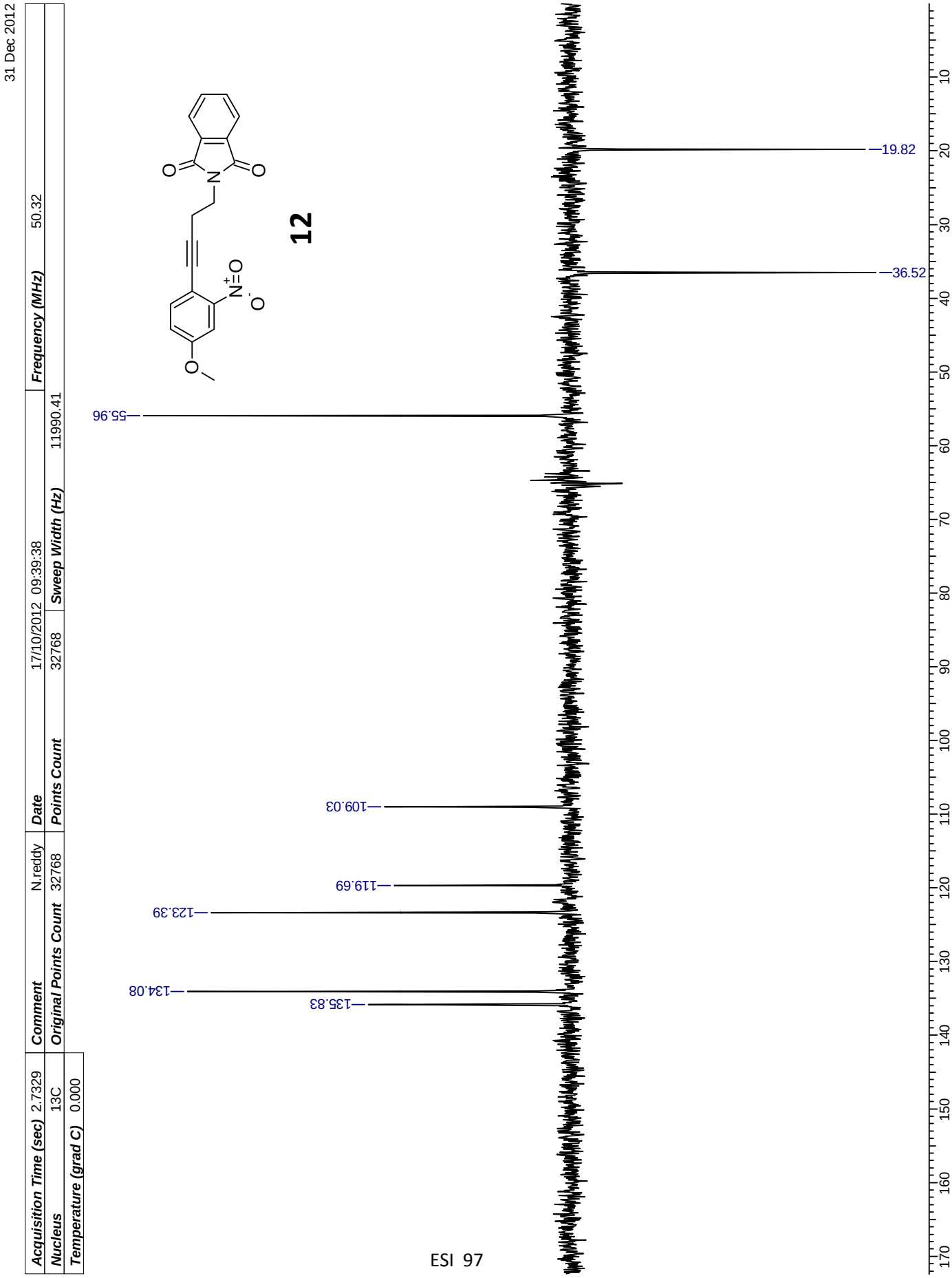

Acquisition Time (sec) 1.2976
Comment DEPT
Date 02/11/2012 09:03:50
Frequency (MHz) 100.61
Nucleus 13C
Original Points Count 32768
Points Count 32768
Sweep Width (Hz) 25252.53
Temperature (grad C) 0.000
11g
NH
N
N
22.15
23.01
28.04
37.14
48.58
53.59
110.75
114.37
118.09
119.51
122.00
123.08
10
20
30
40
50
60
70
80
90
100
110
120
130
140
150

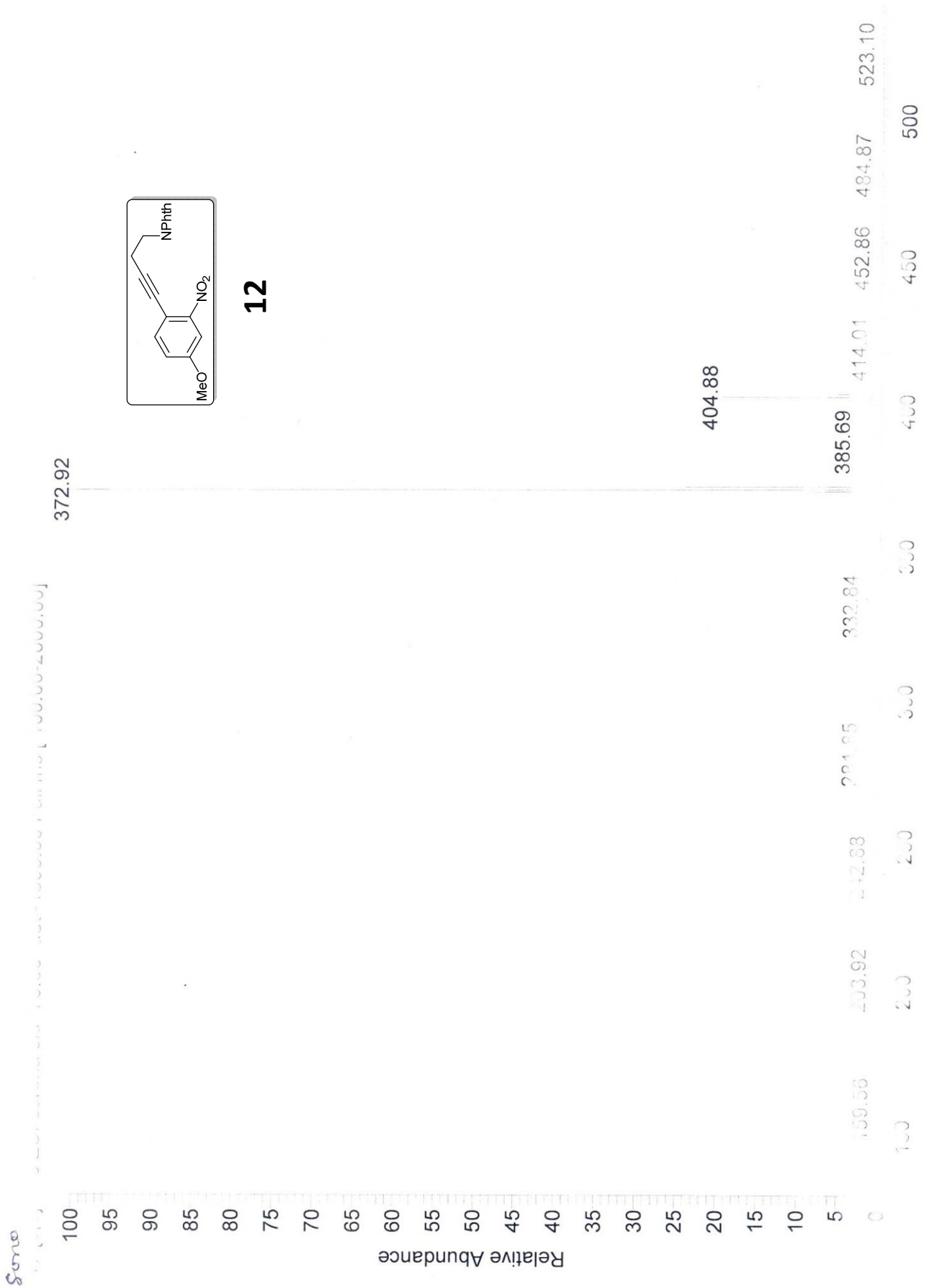

F:\DATA\JAN-2013\15\NPR-16_130115161447
1/15/2013 4:14:47 PM
NPR-16_130115161447 #7-20 RT: 0.11-0.33 AV: 14 SB: 22 0.00-0.14 , 0.40-0.61 NL: 5.51E5
T: {0,0} + c ESI corona sid=40.00 det=1400.00 Full ms [100.00-1500.00]
328.16
256.05
279.05
299.19
303.14
344.20
358.17
376.21
416.19
428.21
456.25
478.38
507.20
529.24
Relative Abundance
m/z
H3C
11g

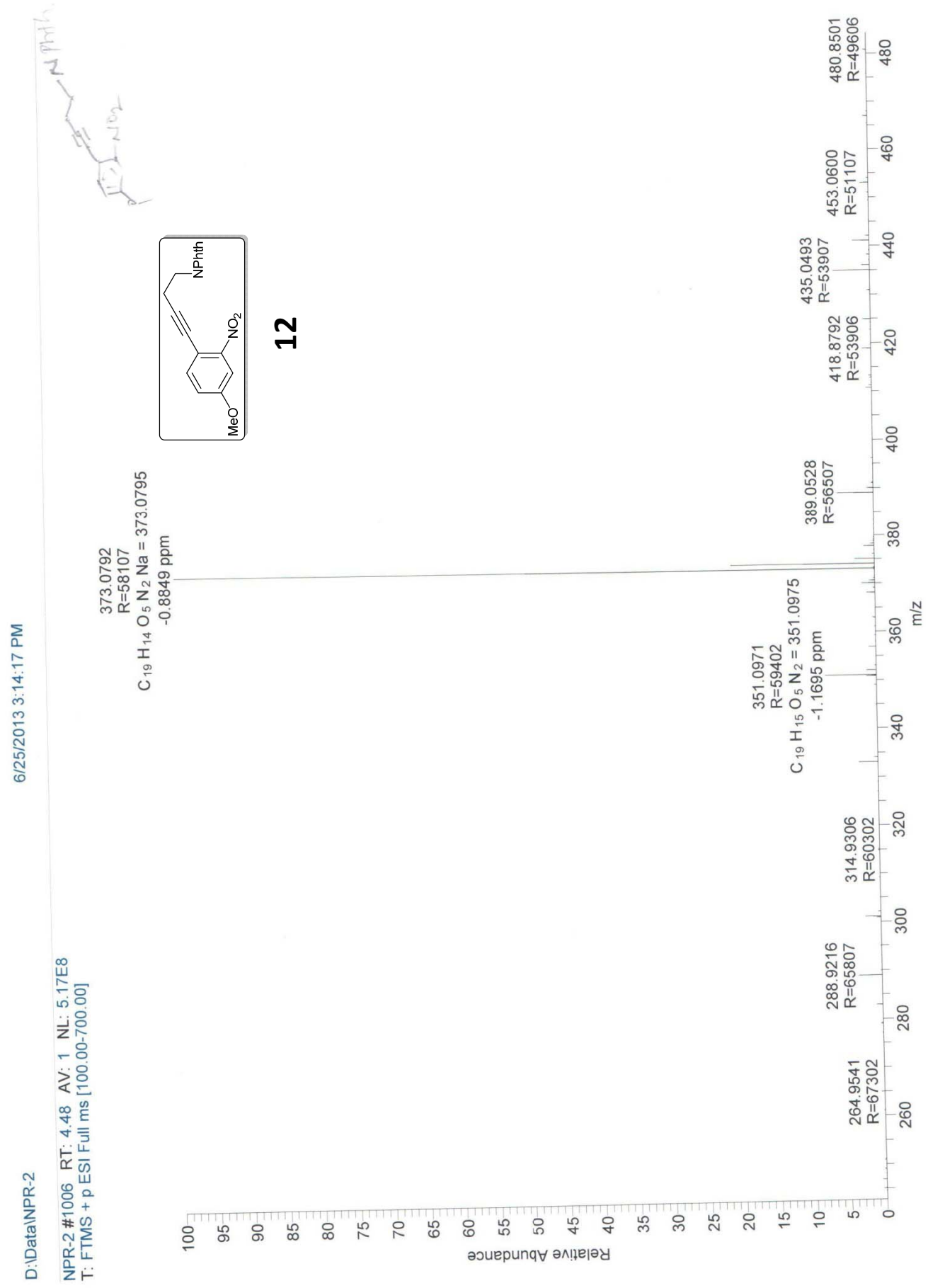

D:\Data\NPR16
1/18/2013 2:55:55 PM
NPR16 #516 RT: 2.30 AV: 1 NL: 1.17E10
T: FTMS + p ESI Full ms [100.00-700.00]
328.1808
R=62503
C 22 H 22 N 3 = 328.1808
-0.2092 ppm
314.1647
R=62807
C 21 H 20 N 3 = 314.1652
-1.4987 ppm
335.1703
R=54000
344.1751
R=60507
350.1615
R=54607
362.1856
R=59007
368.1739
R=50002
Relative Abundance
m/z
11g

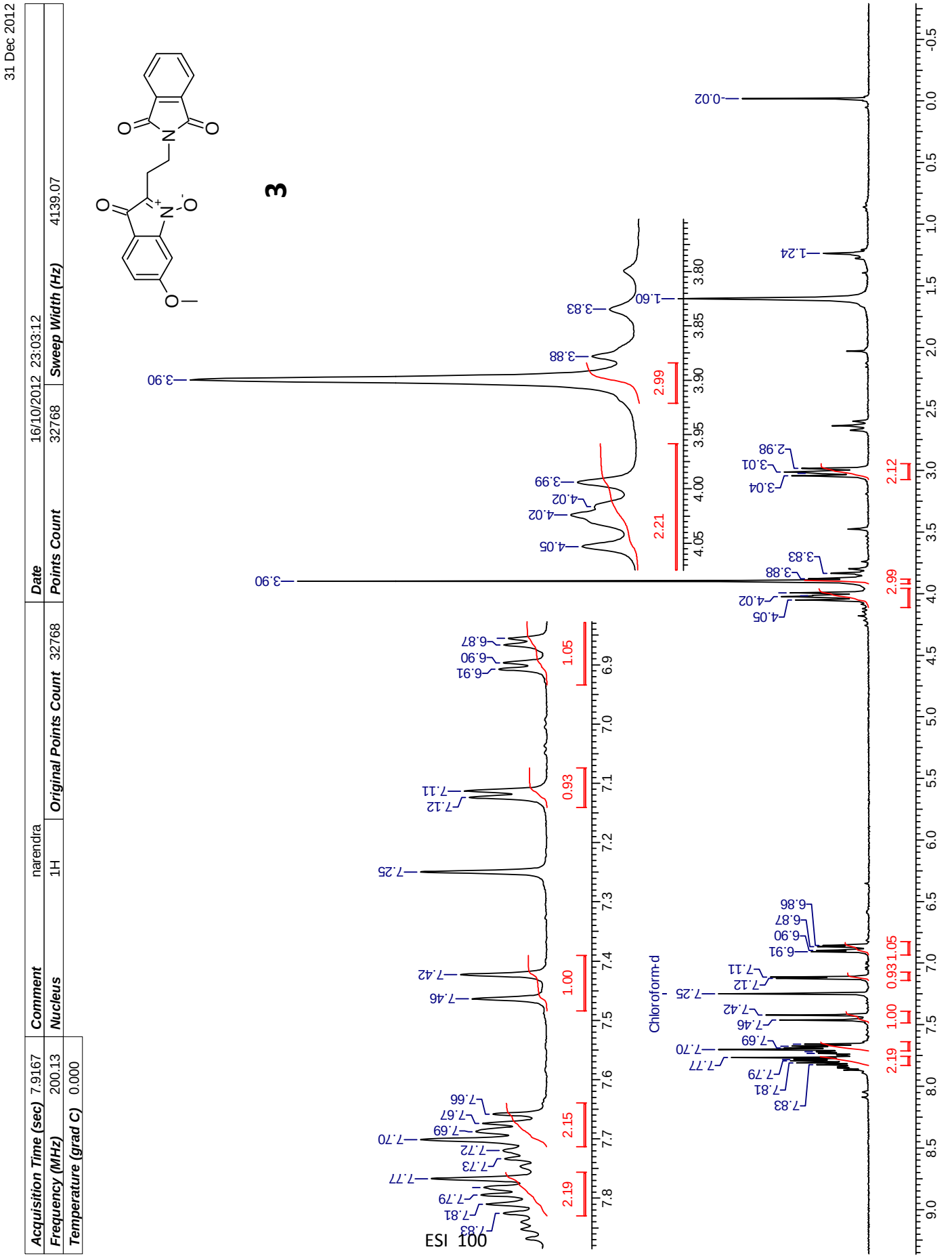

31 Dec 2012
Acquisition Time (sec) 7.9167
Comment
n reddy
Date
15/10/2012 15:09:50
Frequency (MHz) 200.13
Nucleus 1H
Original Points Count 32768
32768
Points Count
Sweep Width (Hz) 4139.07
Temperature (grad C) 0.000
12
ESI 95
Chloroform-d

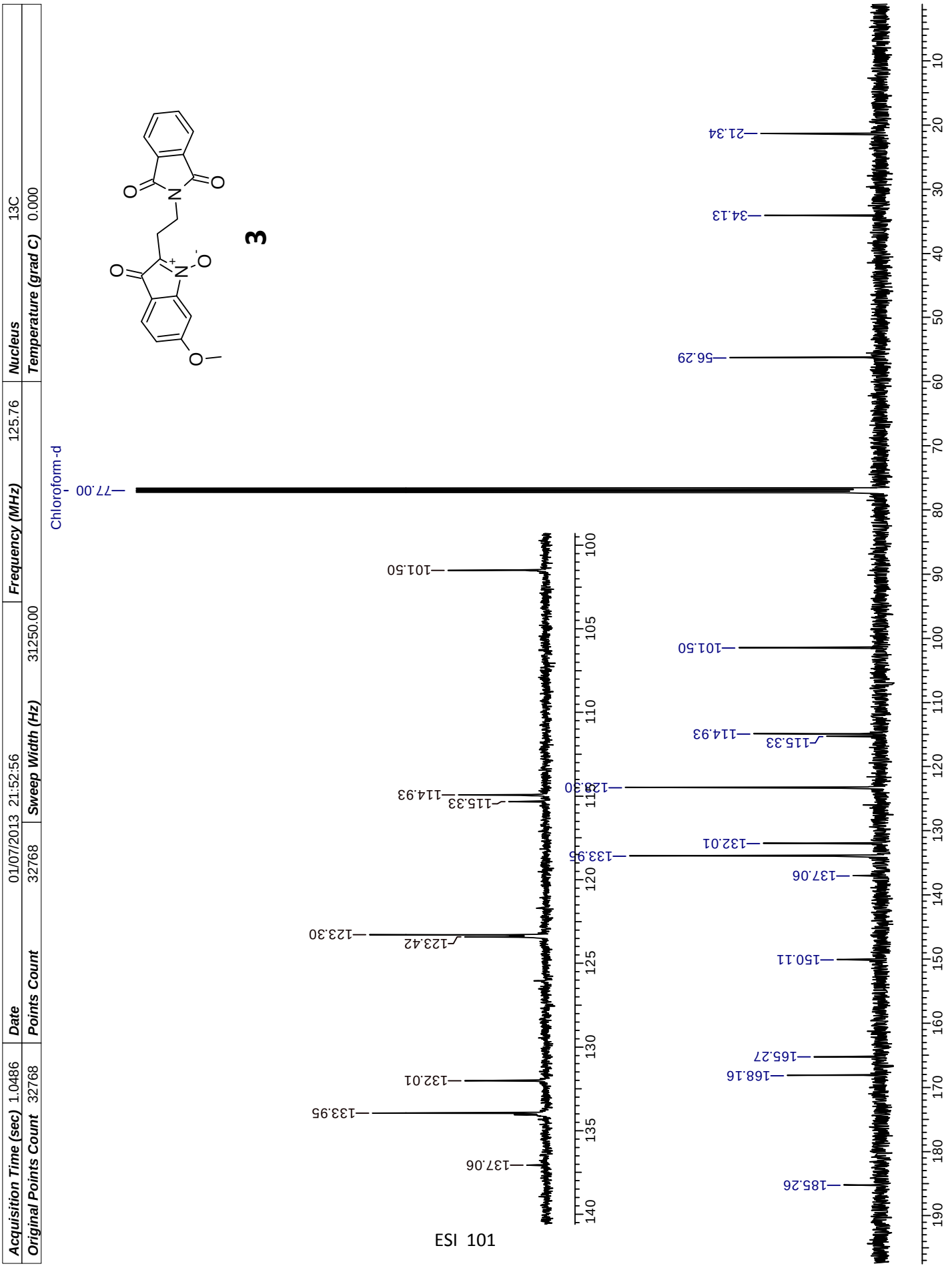

31 Dec 2012
Acquisition Time (sec) 2.7329
Comment
Date 17/10/2012 09:57:08
Frequency (MHz) 50.32
Nucleus 13C
Original Points Count 32768 N.reddy
Points Count 32768
Sweep Width (Hz) 11990.41
Temperature (grad C) 0.000
12
19.81
36.53
55.95
76.43
77.07
77.71
92.34
109.04
110.76
119.66
123.39
132.05
134.08
135.82
150.66
159.15
168.17

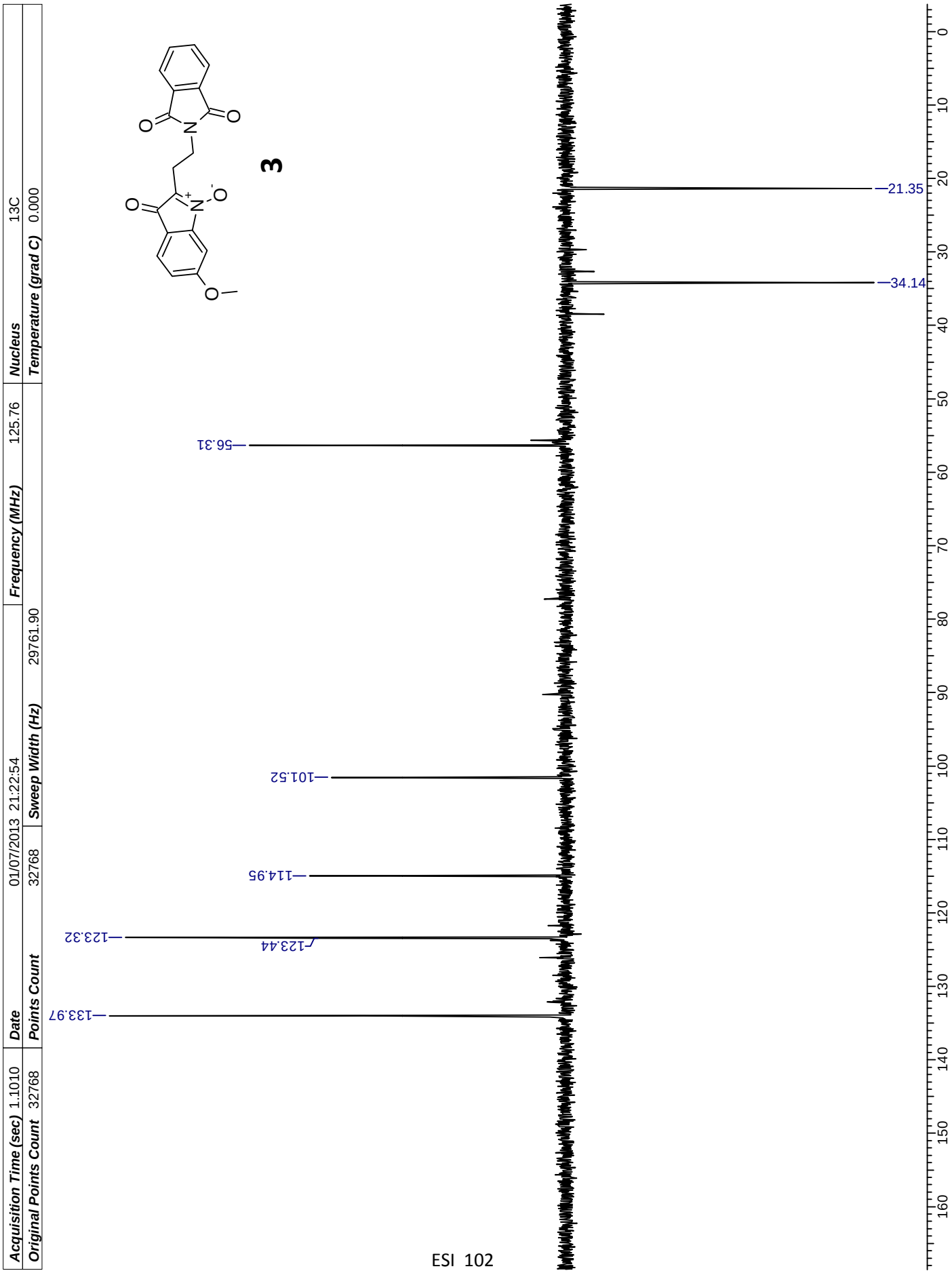

31 Dec 2012
Acquisition Time (sec) 2.7329
Nucleus 13C
Temperature (grad C) 0.000
Comment N.reddy
Original Points Count 32768
Date 17/10/2012 09:39:38
Points Count 32768
Sweep Width (Hz) 11990.41
Frequency (MHz) 50.32
12
55.96
134.08
135.83
123.39
119.69
109.03
36.52
19.82

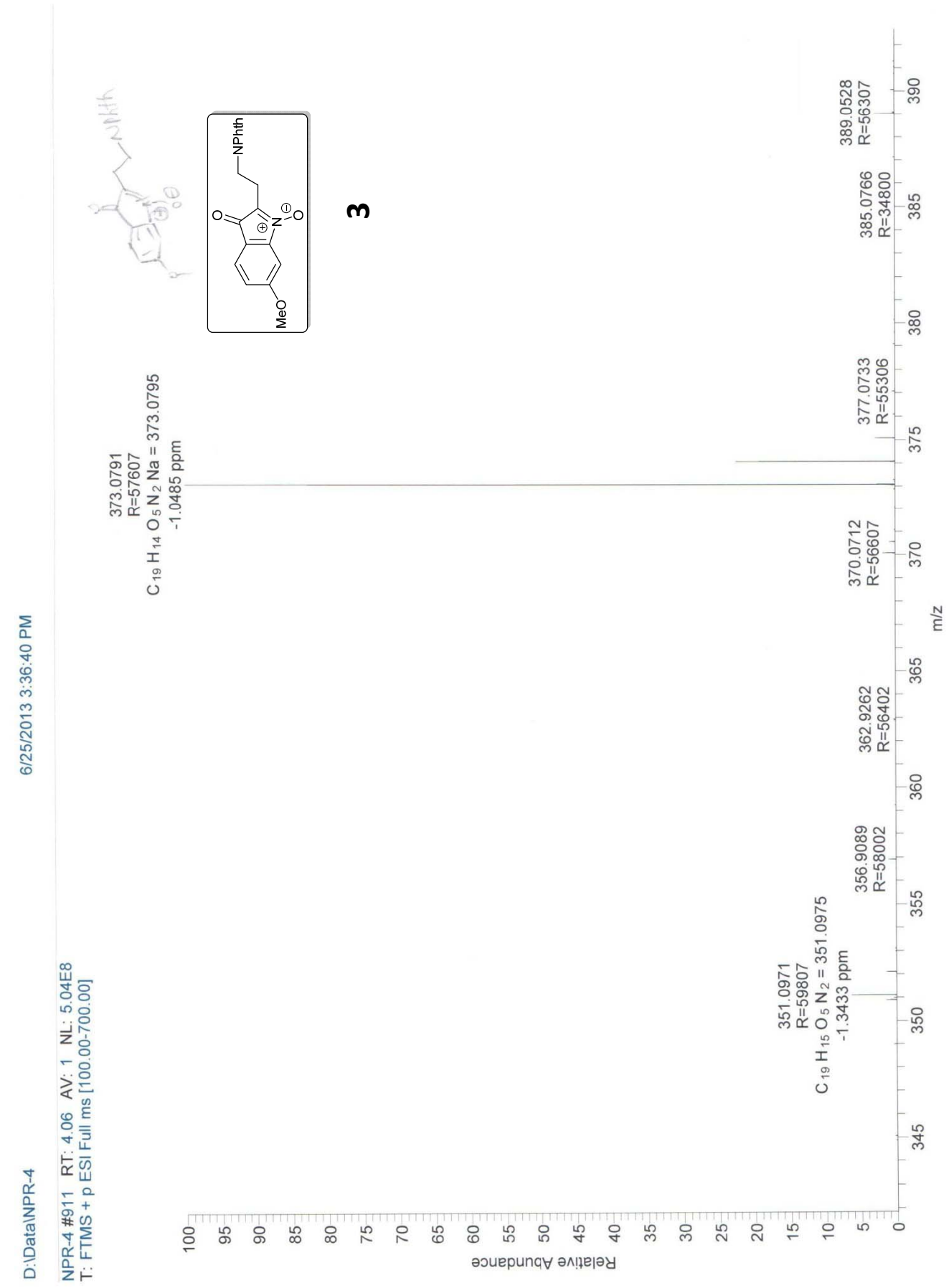

NPhth
NO2
MeO
12
372.92
385.69
404.88
Relative Abundance

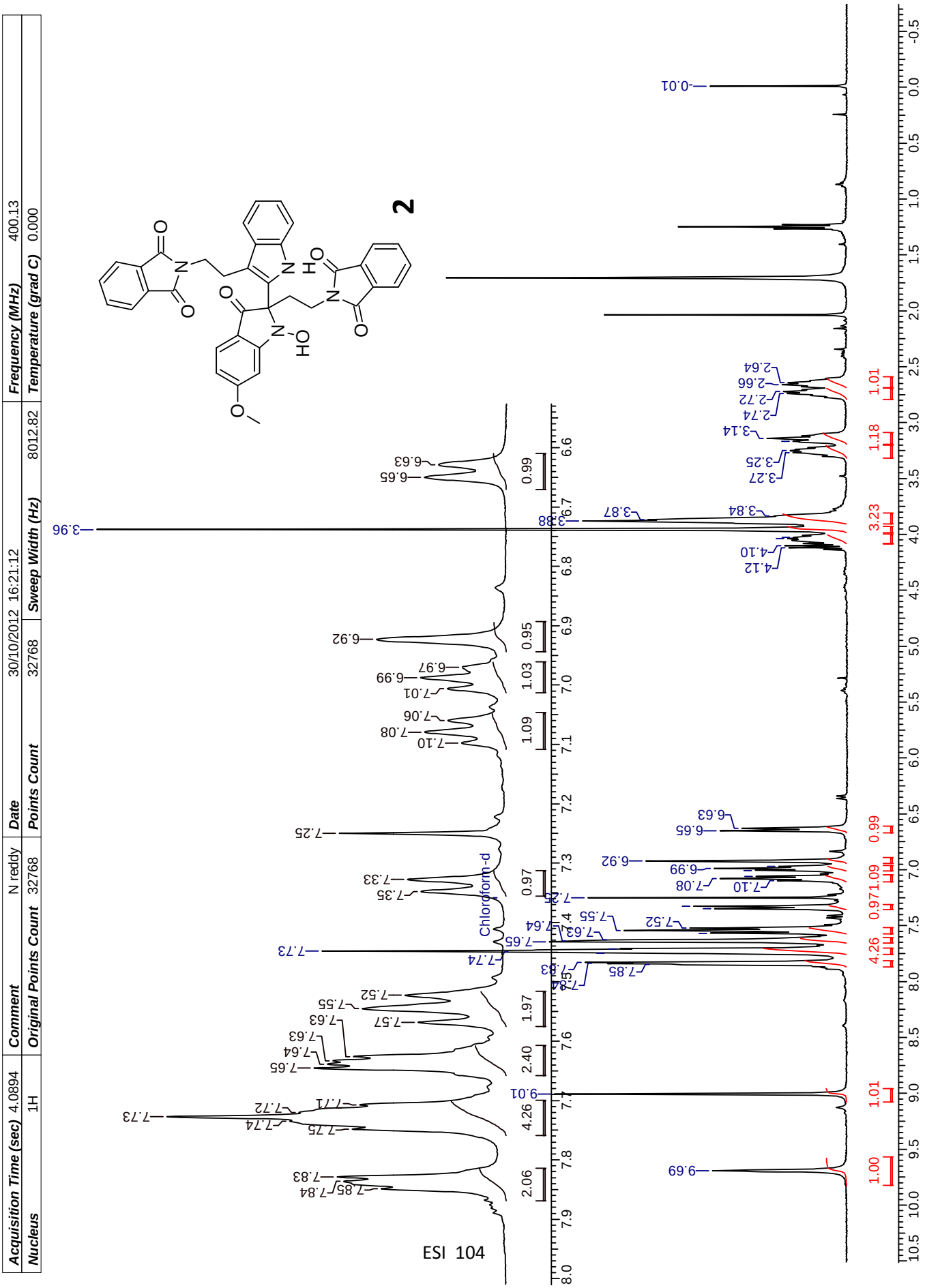

D:\Data\NPR-2
6/25/2013 3:14:17 PM
NPR-2 #1006 RT: 4.48 AV: 1 NL: 5.17E8
T: FTMS + p ESI Full ms [100.00-700.00]
373.0792
R=58107
C 19 H 14 O 5 N 2 Na = 373.0795
-0.8849 ppm
351.0971
R=59402
C 19 H 15 O 5 N 2 = 351.0975
-1.1695 ppm
264.9541
R=67302
288.9216
R=65807
314.9306
R=60302
389.0528
R=56507
418.8792
R=53906
435.0493
R=53907
453.0600
R=51107
480.8501
R=49606
NPhth
NO2
MeO
12
Relative Abundance
m/z

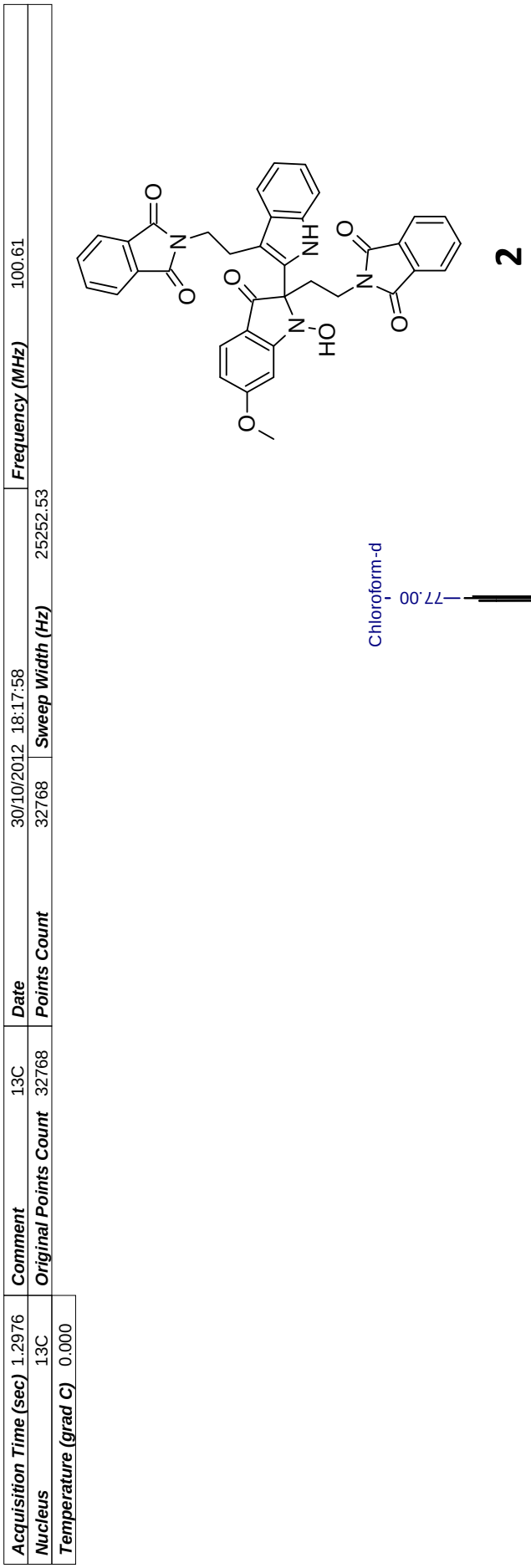

31 Dec 2012
Acquisition Time (sec) 7.9167
Frequency (MHz) 200.13
Temperature (grad C) 0.000
Comment
Nucleus 1H
narendra
Original Points Count 32768
Date 16/10/2012 23:03:12
Points Count 32768
Sweep Width (Hz) 4139.07
3
ESI 100
Chloroform-d

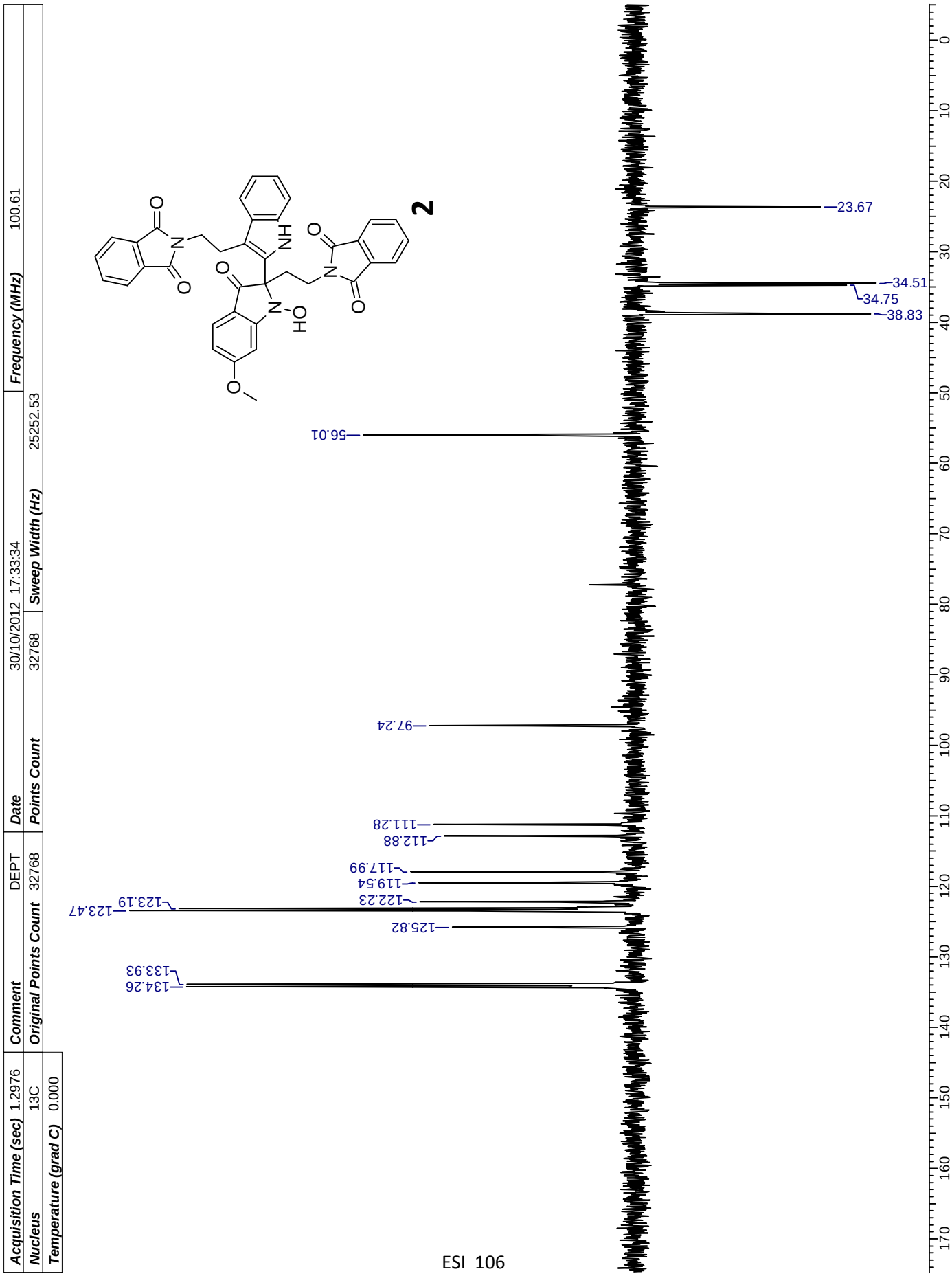

Acquisition Time (sec) 1.0486
Original Points Count 32768
Date 01/07/2013 21:52:56
Points Count 32768
Sweep Width (Hz) 31250.00
Frequency (MHz) 125.76
Nucleus 13C
Temperature (grad C) 0.000
3
Chloroform-d
77.00
21.34
34.13
56.29
101.50
114.93
115.33
123.30
132.01
133.95
137.06
150.11
165.27
168.16
185.26
ESI 101

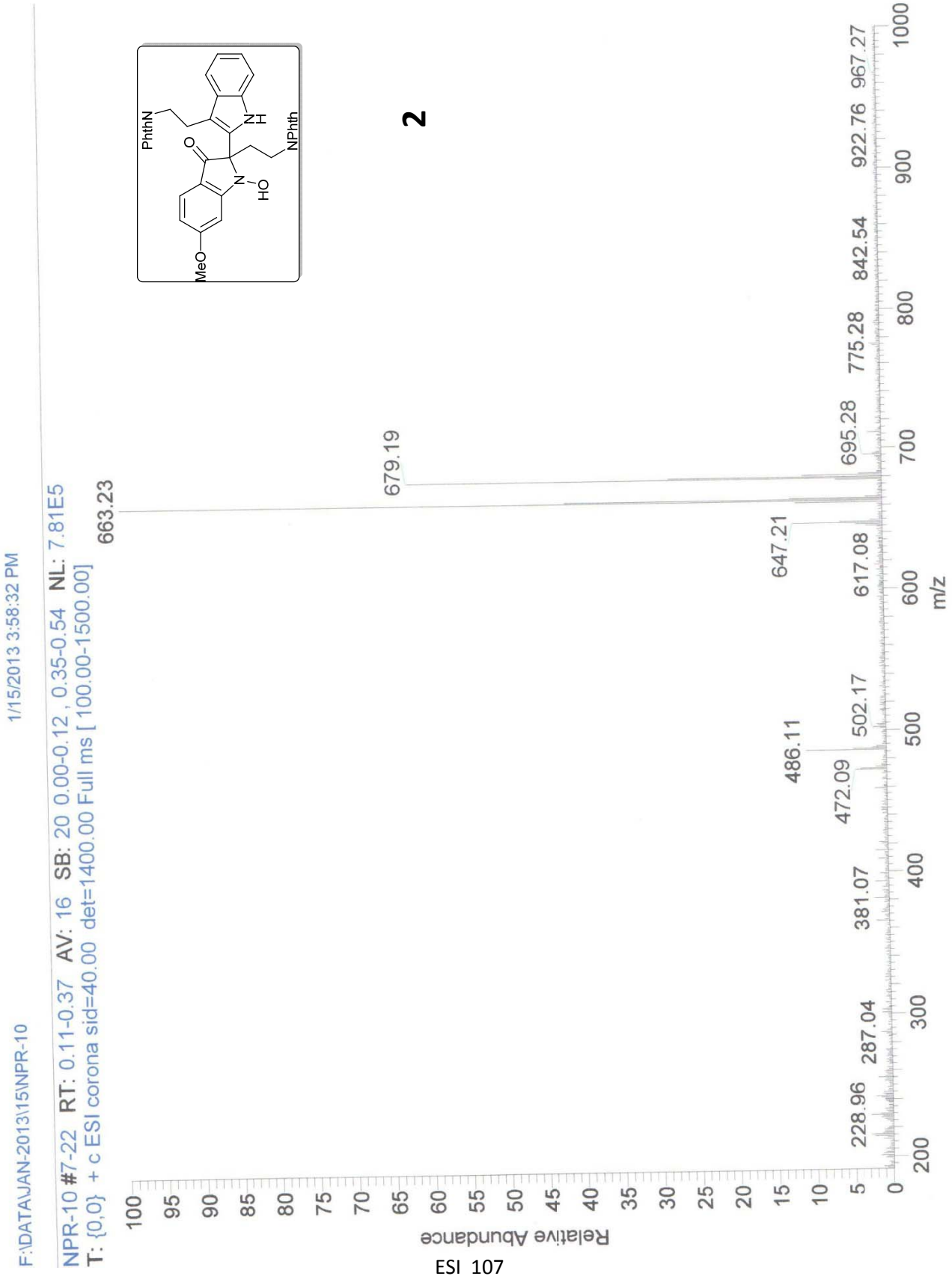

Acquisition Time (sec) 1.1010
Original Points Count 32768
Date 01/07/2013 21:22:54
Points Count 32768
Sweep Width (Hz) 29761.90
Frequency (MHz) 125.76
Nucleus 13C
Temperature (grad C) 0.000
3
133.97
123.32
123.44
114.95
101.52
56.31
34.14
21.35
ESI 102

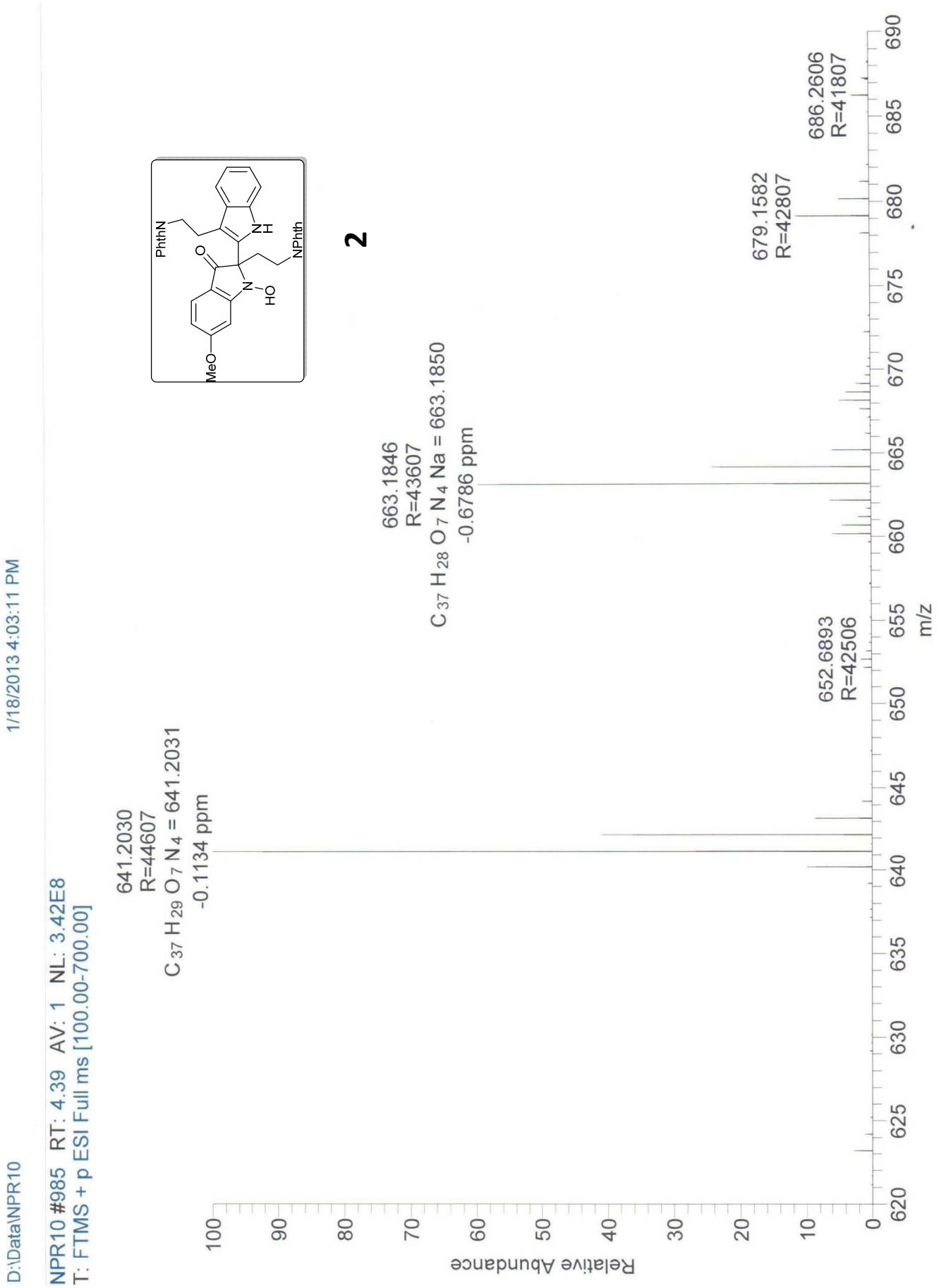

D:\Data\NPR-4
6/25/2013 3:36:40 PM
NPR-4 #911 RT: 4.06 AV: 1 NL: 5.04E8
T: FTMS + p ESI Full ms [100.00-700.00]
373.0791
R=57607
C 19 H 14 O 5 N 2 Na = 373.0795
-1.0485 ppm
351.0971
R=59807
C 19 H 15 O 5 N 2 = 351.0975
-1.3433 ppm
356.9089
R=58002
362.9262
R=56402
370.0712
R=56607
377.0733
R=55306
385.0766
R=34800
389.0528
R=56307
NPhth
MeO
3
Relative Abundance
m/z

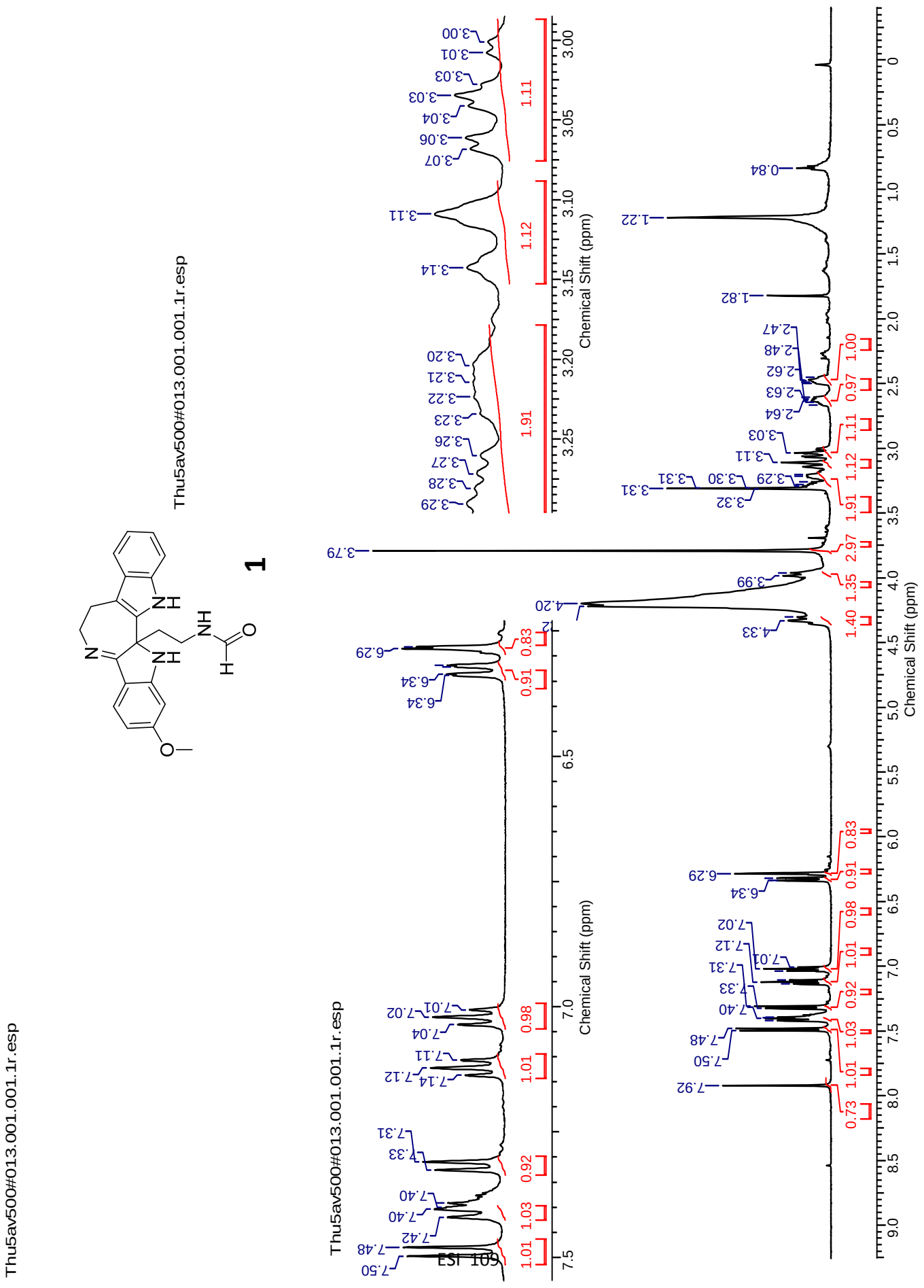

Acquisition Time (sec) 4.0894
Nucleus 1H
Comment N reddy
Original Points Count 32768
Date 30/10/2012 16:21:12
Points Count 32768
Sweep Width (Hz) 8012.82
Frequency (MHz) 400.13
Temperature (grad C) 0.000
2
ESI 104
Chloroform-d

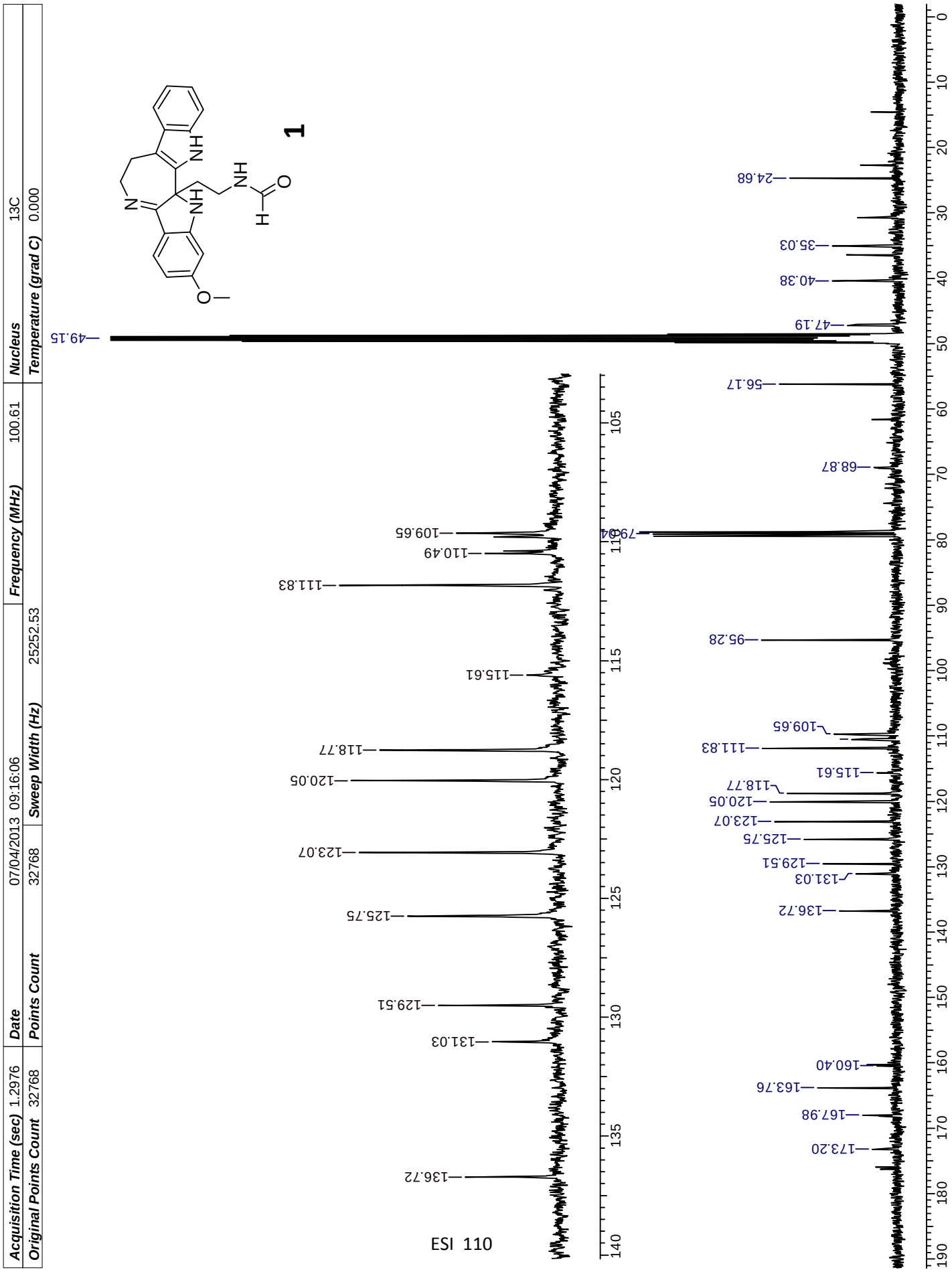

Acquisition Time (sec) 1.2976
Comment
Date 30/10/2012 18:17:58
Frequency (MHz) 100.61
Nucleus 13C
Original Points Count 32768
Points Count 32768
Sweep Width (Hz) 25252.53
Temperature (grad C) 0.000
2
Chloroform-d
77.00
97.16
109.09
111.20
112.79
113.76
117.90
119.46
122.15
123.11
123.40
125.72
128.37
131.03
131.87
133.85
134.18
135.42
165.42
168.02
168.34
169.18
195.93
75.41
55.93
38.75
34.67
34.42
23.60

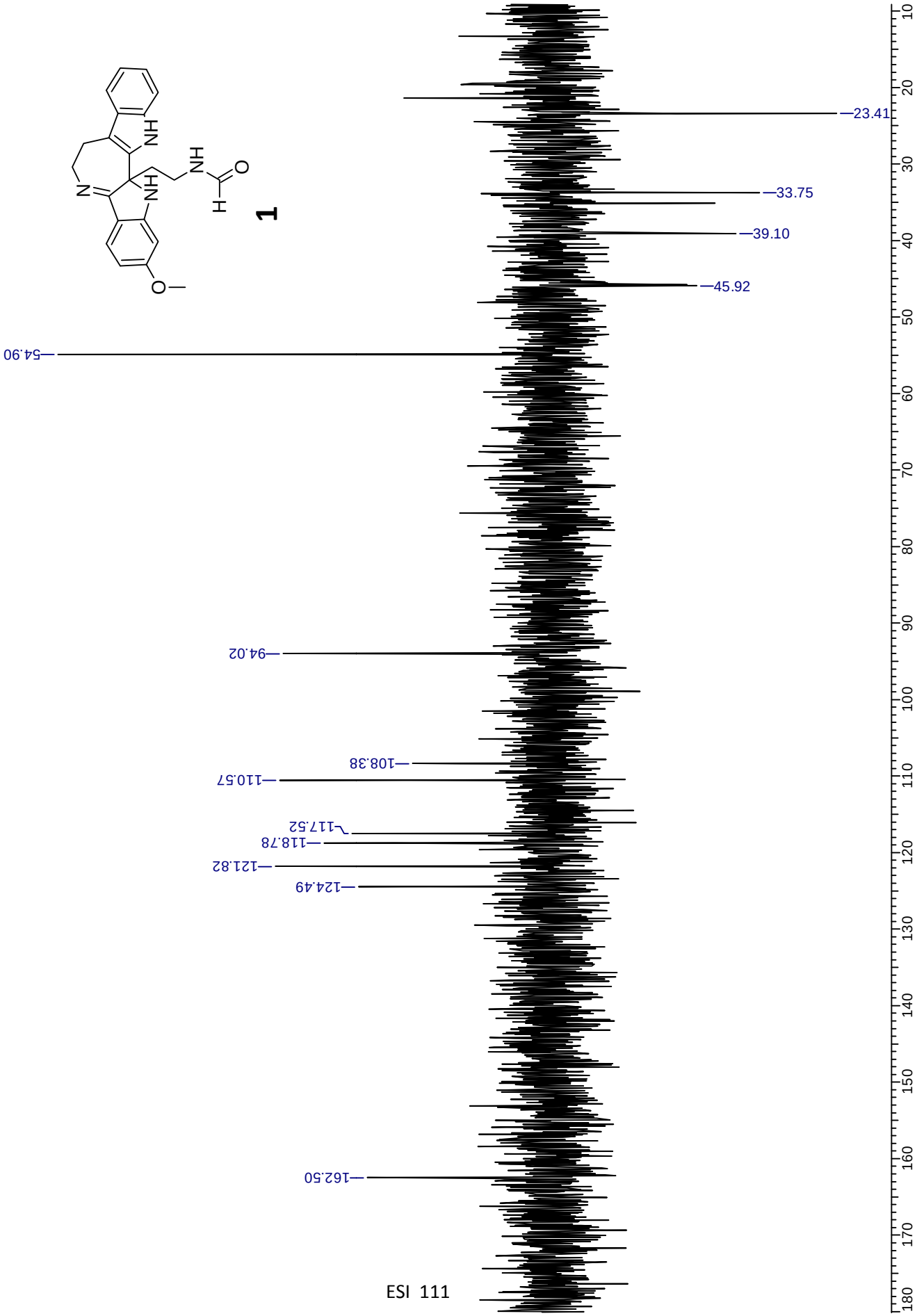

Acquisition Time (sec) 1.2976
Nucleus 13C
Temperature (grad C) 0.000
Comment DEPT
Original Points Count 32768
Date 30/10/2012 17:33:34
Points Count 32768
Sweep Width (Hz) 25252.53
Frequency (MHz) 100.61
2
23.67
34.51
34.75
38.83
56.01
97.24
111.28
112.88
117.99
119.54
122.23
123.19
123.47
125.82
133.93
134.26

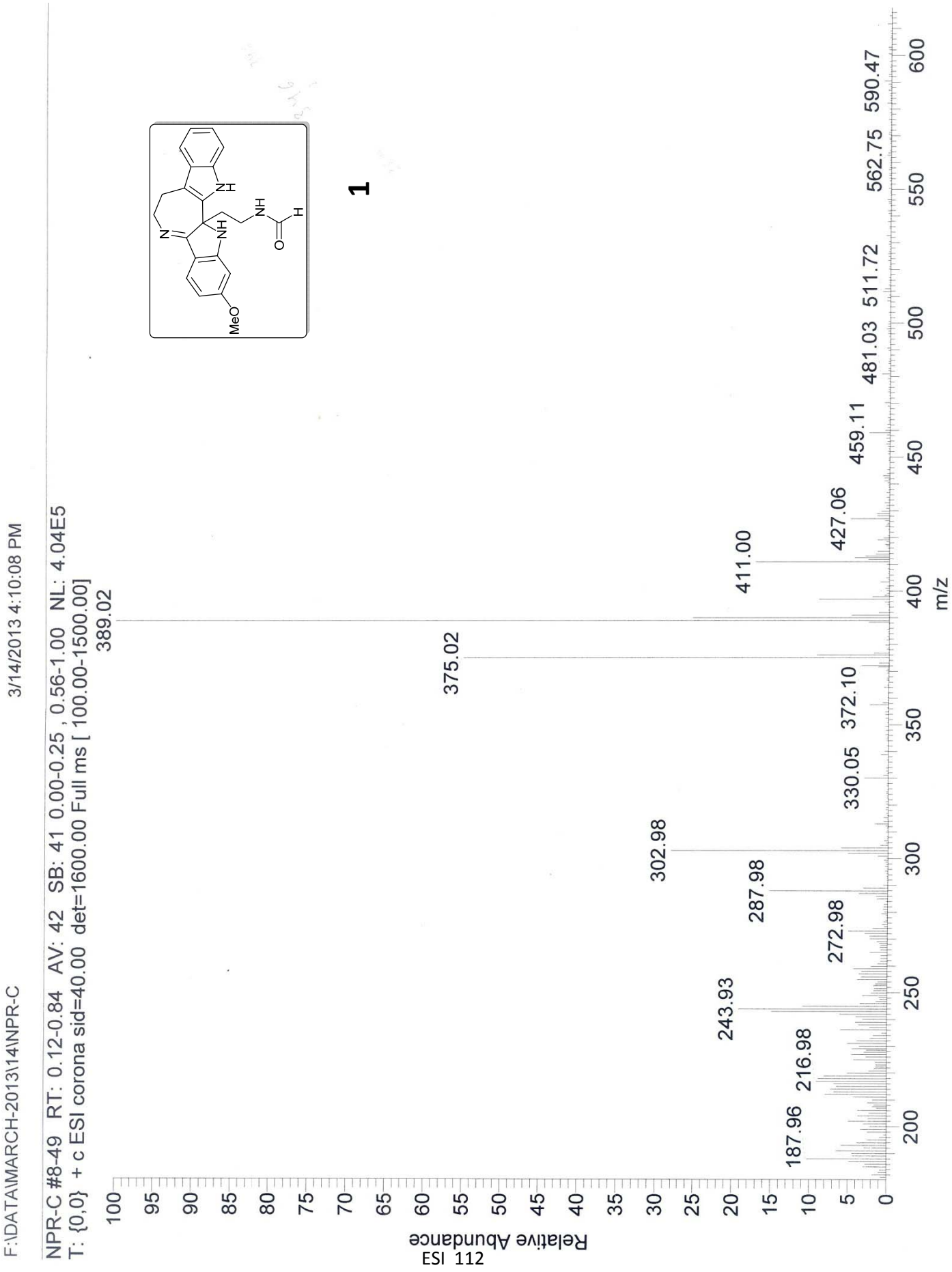

F:\DATA\JAN-2013\15\NPR-10
1/15/2013 3:58:32 PM
NPR-10 #7-22 RT: 0.11-0.37 AV: 16 SB: 20 0.00-0.12 , 0.35-0.54 NL: 7.81E5
T: {0,0} + c ESI corona sid=40.00 det=1400.00 Full ms [100.00-1500.00]
PhthN
NPhth
MeO
HO
2
Relative Abundance
m/z
663.23
679.19
647.21
695.28
486.11
472.09
502.17
617.08
775.28
228.96
287.04
381.07
842.54
922.76
967.27


D:\Data\NPR10
1/18/2013 4:03:11 PM
NPR10 #985 RT: 4.39 AV: 1 NL: 3.42E8
T: FTMS + p ESI Full ms [100.00-700.00]
641.2030
R=44607
C 37 H 29 O 7 N 4 = 641.2031
-0.1134 ppm
663.1846
R=43607
C 37 H 28 O 7 N 4 Na = 663.1850
-0.6786 ppm
652.6893
R=42506
679.1582
R=42807
686.2606
R=41807
Relative Abundance
m/z
PhthN
NPhth
MeO
HO
2



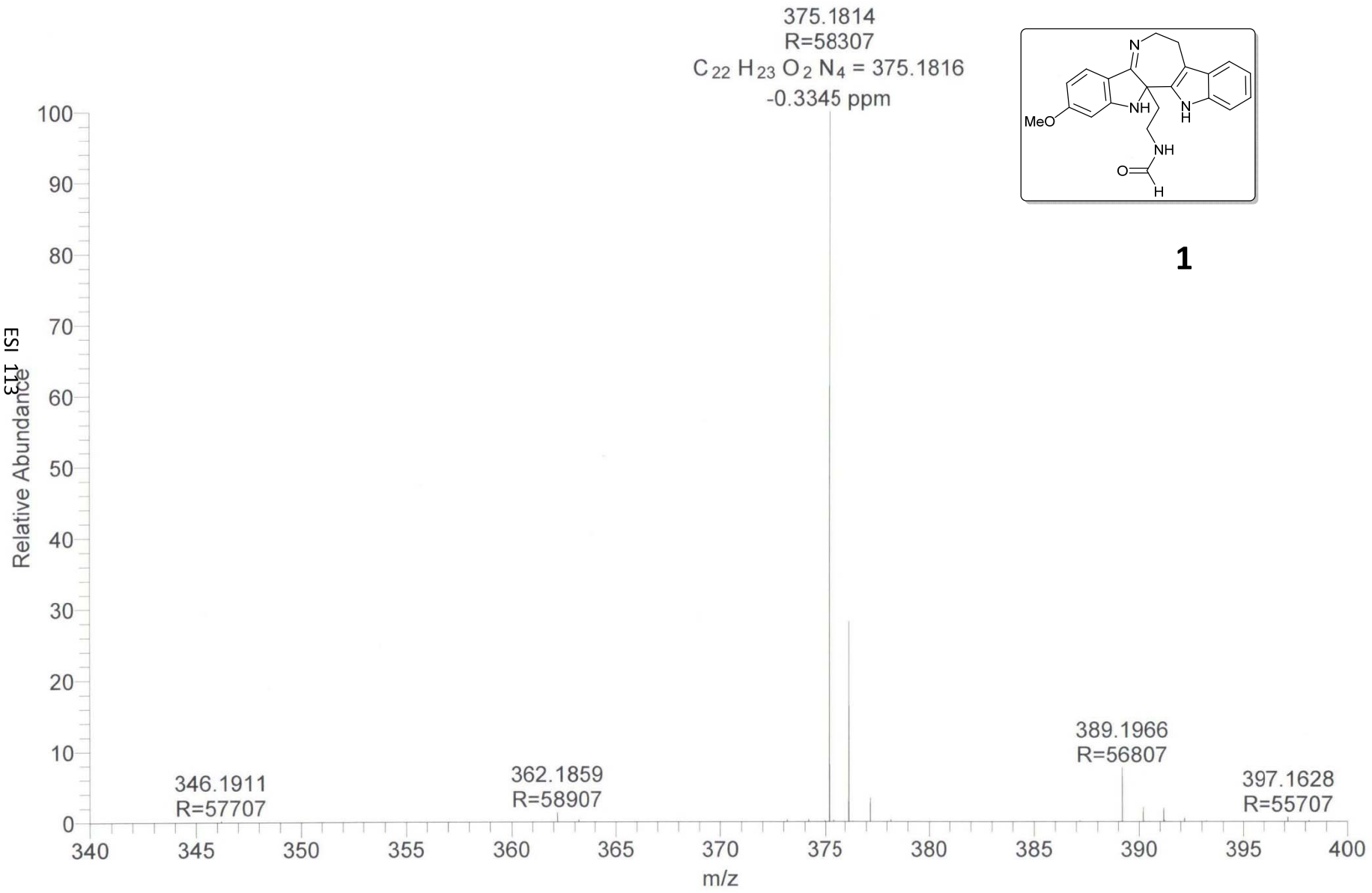



Acquisition Time (sec) 1.3631
Comment
Date
Frequency (MHz) 100.61
Nucleus 13C
Original Points Count 32768
DEPT
Points Count 32768
07/04/2013 09:16:44
Sweep Width (Hz) 24038.46
Temperature (grad C) 0.000
1
23.41
33.75
39.10
45.92
54.90
94.02
108.38
110.57
117.52
118.78
121.82
124.49
162.50
ESI 111


F:\DATA\MARCH-2013\14\NPR-C
3/14/2013 4:10:08 PM
NPR-C #8-49 RT: 0.12-0.84 AV: 42 SB: 41 0.00-0.25 , 0.56-1.00 NL: 4.04E5
T: {0,0} + c ESI corona sid=40.00 det=1600.00 Full ms [100.00-1500.00]
MeO
NH
N
O
H
1
ESI 112
Relative Abundance
m/z
389.02
375.02
411.00
427.06
459.11
481.03
511.72
562.75
590.47
302.98
287.98
272.98
243.93
216.98
187.96
330.05
372.10

D:\Data\NPR20 1/18/2013 3:40:47 PM

NPR20 #516 RT: 2.30 AV: 1 NL: 2.61E9
T: FTMS + p ESI Full ms [100.00-700.00]



1