Electronic Supplementary Material (ESI) for Chemical Communications

Diazaheterocyclic Analogues of Tetracene: Synthesis and Properties of a Naphtho-Fused Cinnoline and a Naphtho-Fused Isoindazole

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Experimental Details

General Information. ^{1}H and ^{13}C NMR spectra were recorded in $CDCl_3$ using a Varian Inova 300 (^{1}H: 299.93 MHz, ^{13}C: 75.42 MHz), 500 (^{1}H: 500.11 MHz, ^{13}C: 125.75 MHz), or 600 (^{1}H: 599.90 MHz, ^{13}C: 150.88 MHz) spectrometer. Chemical shifts (δ) are expressed in ppm relative to the residual chloroform (^{1}H: 7.26 ppm, ^{13}C: 77.16 ppm) reference. IR spectra were recorded on a Nicolet Magna FTIR 550 spectrometer. UV-Vis spectra were recorded on a HP 8453 UV-Vis spectrometer. Fluorescence spectra were recorded on a Horiba-Jobin Yvon Fluoromax-4 spectrofluorimeter. High resolution mass spectra were recorded on a Waters LCT Premier ESI-MS in positive mode. Dry THF was distilled from a sodium and benzophenone still under N_2. All reagents were purchased from commercial suppliers and used as received unless otherwise indicated.

Bromide 5. N-Bromosuccinimide (16.74 g, 94 mmol) was added to a solution of 2-aminoanthraquinone (10 g, 45 mmol) in DMF (700 mL) at 0 °C under Ar. The solution was warmed to rt and stirred overnight. Water (500 mL) was added to the mixture, and the resultant precipitate was collected by vacuum filtration. The solid was washed with water and oven-dried yielding 2-amino-1,3-dibromoanthraquinone (15.43 g, 90%) as a brown solid. ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.47 (s, 1H), 8.24 (br s, 2H), 7.76 (br s, 2H), 5.61 (s, 2H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 182.5, 180.5, 148.3, 134.5, 134.2, 134.0, 132.5, 132.0, 131.0, 127.7, 126.7, 125.9, 113.4, 106.6; HRMS (ESI+) for $C_{14}H_7Br_2NO_2$ $(M+H)^+$: calcd 379.8922, found 379.8916.

2-Amino-1,3-dibromoanthraquinone (15 g, 39 mmol), Sn powder (4.91 g, 41 mmol), conc. HCl (54 mL) and glacial AcOH (72 mL) were stirred at 110 °C in a sealed screwtop, pressure reaction vessel overnight. The reaction was cooled to rt and diluted with cold water. The brown precipitate was collected by vacuum filtration, washed with cold water, and oven-dried yielding **5** (11.64 g, 97%) as a light brown solid. ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.40 (s, 1H), 8.31-8.24 (m, 2H), 7.80-7.74 (m, 2H), 7.53 (s, 1H), 4.87 (s, 2H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 183.3, 181.1, 149.3, 134.4, 134.4, 133.9, 133.8, 133.6, 133.0, 127.3, 127.2, 125.0, 114.8, 111.9; HRMS (ESI+) for $C_{14}H_8BrNO_2$ $(M+H)^+$: calcd 301.9817, found 301.9825.

Ethynylamine 6. 2-Amino-3-bromoanthraquinone (**5**, 4.0 g, 13.2 mmol), $Pd(PPh_3)_2Cl_2$ (0.186 g, 0.26 mmol) and CuI (0.101 g, 0.5 mmol) were dissolved in THF (75 mL) and *i*-Pr_2NH (75 mL). The mixture was purged with Ar for 45 min after which (triisopropylsilyl)acetylene (TIPSA, 11.9 mL, 53 mmol) was added. The reaction was stirred overnight at 85 °C. After cooling to rt, the mixture was passed over a short pad of silica (CH_2Cl_2) and then concentrated. The crude material was purified by flash chromatography (1:1 hexanes:CH_2Cl_2) to yield ethynylamine **6** (3.88 g, 73%) as an orange solid. ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.31-8.22 (m, 2H), 8.27 (s, 1H), 7.80-7.69 (m, 2H), 7.49 (s, 1H), 5.02 (s, 2H), 1.16 (br s, 21H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 183.4, 181.41, 152.6, 134.7, 134.2, 134.1, 133.8, 133.6, 133.0, 127.3, 127.2, 123.9, 113.0, 110.6, 101.7, 101.3, 18.9, 11.3; HRMS (ESI+) for $C_{25}H_{29}NO_2Si$ $(M+H)^+$: calcd 404.2046, found 404.2037.

Triazene 7. To a flame-dried flask containing BF_3•OEt_2 (8.6 mL, 69 mmol) cooled to –20 °C under an Ar atmosphere was added dropwise a solution of ethynylamine **6** (3.4 g, 8.7 mmol) in dry THF (20 mL). A solution of *t*-BuONO (7.2 mL, 61 mmol) in dry THF (15 mL) was then added dropwise while maintaining the –20 °C temperature. After stirring an additional 30 min at –20 °C, the reaction mixture was then cannulated into a suspension of K_2CO_3 (24.0 g, 173 mmol) and $HNEt_2$ (18 mL, 173 mmol) in DMF (90 mL) at 0 °C and stirred for 1.5 h. The reaction was diluted with EtOAc, and washed with aq. NH_4Cl (3X) and brine (3X). The organic layer was dried ($MgSO_4$) and filtered, and the solvent removed in vacuo. The crude material was purified by flash chromatography (1:1:hexanes:CH_2Cl_2) to yield triazene **7** (2.37 g, 57%) as an orange solid. ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.39 (s, 1H), 8.34 (s, 1H), 8.32-8.25 (m, 2H), 7.81-7.72 (m, 2H), 3.90 (q, J = 7.2 Hz, 4H), 1.40 (t, J = 7.2 Hz, 3H), 1.26 (t, J = 7.2 Hz, 3H), 1.16 (s, 21H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 183.5, 182.3, 157.0, 134.3, 134.2, 134.1, 134.0, 133.9, 133.3, 129.2, 127.4, 127.3, 123.8, 115.0, 104.1, 99.8, 49.9, 42.3, 18.9, 14.6, 11.5, 11.2; HRMS (ESI+) for $C_{29}H_{37}N_3O_2Si$ $(M+H)^+$: calcd, 488.2709, found 488.2713.

Triazene 3. To a solution of triazene **7** (2.3 g, 4.7 mmol) in THF (60 mL) and MeOH (3 mL) was added TBAF (1 M, 23.6 mL). The mixture was stirred for 10 min, and then diluted

with EtOAc and washed with aq. NH_4Cl (2X), H_2O (3X) and brine (3X). The organic layer was dried ($MgSO_4$), filtered and concentrated in vacuo to yield the terminal acetylene **3** (1.5 g, 96%) as a yellow solid. ^{1}H NMR (600 MHz, $CDCl_3$) δ 8.44 (s, 1H), 8.32-8.28 (m, 2H), 8.31 (s, 1H), 7.81-7.75 (m, 2H), 3.90 (q, J = 7.2 Hz, 4H), 3.49 (s, 1H), 1.41 (t, J = 7.2 Hz, 3H), 1.32 (t, J = 7.2 Hz, 3H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 183.4, 182.1, 157.4, 134.3, 134.0, 134.0, 133.9, 133.9, 133.7, 129.3, 127.4, 127.4, 122.5, 115.2, 85.0, 80.8, 50.1, 42.8, 14.6, 10.9; HRMS (ESI+) for $C_{20}H_{17}N_3O_2$ $(M+H)^+$: calcd 332.1399, found 332.1390.

Cinnoline 8. A solution of ethynyltriazene **3** (0.83 g, 2.5 mmol) in ODCB (170 mL) was sealed in a screwtop pressure reaction vessel and stirred overnight in a preheated 200 °C sand bath. After cooling to rt, the solvent was removed in vacuo and the resultant material purified by column chromatography (7:3 CH_2Cl_2:EtOAc) yielded cinnoline **8** (0.39 g, 60%) as a brown solid. ^{1}H NMR (300 MHz, $CDCl_3$) δ 9.58 (s, 1H), 9.54 (d, J = 6.3 Hz, 1H), 8.91 (s, 1H), 8.49-8.41 (m, 2H), 8.11 (d, J = 6.3 Hz, 1H), 7.93-7.89 (m, 2H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 182.0, 181.7, 146.7, 135.2, 135.0, 134.5, 134.2, 134.0, 133.6, 131.9, 131.8, 129.3, 129.2, 128.1, 127.9, 123.6; HRMS (ESI+) for $C_{16}H_8N_2O_2$ $(M+H)^+$: calcd 261.0664, found 261.0661.

Diazatetracene 1. To a solution of TIPSA (1.5 mL, 6.6 mmol) in THF (6 mL) at 0 °C was added BuLi (3.75 mL, 6 mmol) and the mixture was stirred for 1.5 h. In a separate flask, a solution of cinnoline **8** (0.39 g, 1.5 mmol) in THF (3 mL) was cooled to 0 °C and then the lithiate solution was cannulated into the solution of **8**, which as warmed to rt and stirred overnight. The reaction mixture was diluted with EtOAc, washed with water, dried ($MgSO_4$), filtered through a short pad of silica, and evaporated to dryness to give the crude diol (0.742 g). To a portion of the crude diol (48.4 mg, 0.077 mmol) in AcOH (10 mL) was added KI (38.5 mg, 0.23 mmol) and NaH_2PO_2 (24.6 mg, 0.23 mmol), and the reaction was stirred overnight at rt. The mixture was diluted with water (10 mL), and extracted with CH_2Cl_2 (3 x 15 mL). The organic layer was dried ($MgSO_4$), filtered, and evaporated to dryness. Purification by preparative TLC (1:39 EtOAc:CH_2Cl_2) yielded diazatetracene **1** (14.4 mg, 25%) as a purple solid. ^{1}H NMR (300 MHz, $CDCl_3$) δ 10.13 (s, 1H), 9.31 (s, 1H), 9.24 (d, J = 6.3 Hz, 1H), 8.71-8.60 (m, 2H), 7.96 (d, J = 6.3

Hz, 1H), 7.66-7.58 (m, 2H), 1.32 (br s, 42H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 146.6, 141.5, 134.3, 133.8, 132.4, 132.4, 130.9, 128.3, 127.9, 127.8, 127.6, 126.8, 122.5, 121.4, 120.9, 119.0, 108.7, 107.6, 103.3, 103.0, 19.1, 19.1, 11.7, 11.7; UV-Vis ($CHCl_3$) λ_{max} (ε): 539 (11,200), 576 (11,900) nm; HRMS (ESI+) for $C_{38}H_{50}N_2Si_2$ $(M+H)^+$: calcd 591.3591, found 591.3576.

Aldehyde 9. A solution of ethynyltriazene **3** (0.5 g, 1.5 mmol) in DCE (150 mL) was bubbled with O_2 for 1 h. CuCl (0.749 g, 7.5 mmol) was added, and the reaction stirred at rt for 3 h. The mixture was filtered through a short pad of silica and evaporated to dryness. Column chromatography (1:19 acetone:CH_2Cl_2) gave aldehyde **9** (0.186 g, 35%) as a yellow solid. ^{1}H NMR (300 MHz, $CDCl_3$) δ 10.49 (s, 1H), 9.30 (s, 1H), 8.86 (s, 1H), 8.47-8.37 (m, 2H), 7.86-7.81 (m, 2H), 3.46 (q, J = 6.9 Hz, 4H), 0.94 (t, J = 6.9 Hz, 6H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 183.0, 182.5, 181.0, 147.0, 135.3, 134.6, 134.5, 134.4, 134.3, 132.1, 130.8, 127.9, 127.7, 124.5, 122.0, 120.0, 53.1, 12.2; HRMS (ESI+) for $C_{20}H_{17}N_3O_3$ $(M+H)^+$: calcd 348.1348, found 348.1345.

Ether 10. Aldehyde (0.25 g, 0.7 mmol) was dissolved in THF (40 mL) and cooled in an ice bath. $NaHB(OAc)_3$ (0.61 g, 2.8 mmol) was added, the reaction mixture removed from the ice bath, and stirred over night. The reaction mixture was quenched with aq NH_4Cl and extracted with DCM (4 x 20 mL). The organic layer was dried ($MgSO_4$), filtered, and evaporated to dryness to give the alcohol (0.25 g, 99%) as a yellow solid which was used without further purification: ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.86 (s, 1H), 8.71 (s, 1H), 8.41-8.35 (m, 2H), 7.83-7.80 (m, 2H), 5.22 (d, J = 6.1 Hz, 2H), 3.40 (br s, 4H), 3.10 (t, J = 6.1 Hz, 1H), 0.93 (t, J = 7.2 Hz, 6H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 183.3, 182.9, 147.0, 139.0, 134.7, 134.2, 134.1, 131.3, 127.7, 127.6, 126.9, 124.1, 120.3, 119.6, 119.5, 55.3, 52.9, 12.2.

To an ice-cooled flask charged with NaH (0.034 g, 1.4 mmol) and THF (10 mL) was added a solution of alcohol (0.25 g, 0.72 mmol) in THF (10 mL) dropwise over a period of 20 min. After the addition was complete, a solution of MeI (0.22 mL, 3.6 mmol) in THF (5 mL) was added over a period of 10 min. The reaction was warmed to rt and stirred overnight. The mixture was quenched with aq NH_4Cl and extracted with CH_2Cl_2 (4 x 20 mL). The organic layer was dried

($MgSO_4$), filtered, and evaporated to dryness. Column chromatography (1:4 EtOAc:hexanes) of the crude material gave ether **10** (0.196 g, 75%) as a yellow solid. ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.93 (s, 1H), 8.72 (s, 1H), 8.42-8.35 (m, 2H), 7.83-7.78 (m, 2H), 4.97 (s, 2H), 3.50 (s, 3H), 3.35 (br s, 4H), 0.97 (t, *J* = 7.2 Hz, 6H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 183.4, 182.9, 147.1, 137.4, 134.8, 134.8, 134.2, 134.0, 131.3, 127.6, 127.6, 127.0, 124.7, 121.6, 119.6, 64.2, 59.0, 52.7, 12.1; HRMS (ESI+) for $C_{21}H_{21}N_3O_3$ $(M+H)^+$: calcd 364.1661, found 364.1646.

Diazatetracene 2. To a solution of TIPSA (0.53 mL, 2.4 mmol) in THF (3 mL) at 0 °C was added BuLi (1.3 mL, 2.2 mmol) and the mixture was stirred for 1.5 h. In a separate flask, a solution of ether **10** (0.19 g, 0.5 mmol) in THF (3 mL) was cooled to 0 °C and then the lithiate solution was cannulated into the solution of **10**, which was warmed to rt and stirred overnight. The reaction mixture was diluted with EtOAc, washed with water, dried ($MgSO_4$), filtered through a short pad of silica, and evaporated to dryness to give the crude diol (0.136 g). To a portion of the crude diol (45 mg) in THF (10 mL) was added $SnCl_2$ (35 mg, 0.18 mmol). After stirring the reaction at 60 °C for 30 min, the mixture was diluted with water (10 mL) and then extracted with CH_2Cl_2 (3 x 15 mL). The organic layer was dried ($MgSO_4$), filtered, and evaporated to dryness affording ethynylisoindazole **2** (42 mg, 34%) as a purple solid. ^{1}H NMR (300 MHz, $CDCl_3$) δ 9.4 (d, *J* = 1.4 Hz, 1H), 9.13 (d, *J* = 1.4 Hz, 1H) 8.56-8.47 (m, 2H), 7.43-7.44-7.35 (m, 2H), 5.11 (s, 2H), 3.48 (s, 3H), 3.44 (br s, 4H), 1.30 (br s, 42H), 0.88 (t, *J* = 7.1 Hz); ^{13}C NMR (150 MHz, $CDCl_3$) δ 145.4, 133.2, 132.4, 132.0, 131.4, 128.5, 127.63, 127.6, 126.5, 126.1, 122.8, 119.1, 118.6, 113.1, 105.7, 105.3, 104.6, 104.4, 63.8, 58.5, 52.9, 19.2, 19.1, 12.2, 11.8, 11.7; UV-Vis ($CHCl_3$) λ_{max} (ε): 544 (6,800), 584 (9,900) nm; HRMS (ESI+) for $C_{43}H_{63}N_3OSi_2$ calcd 694.4588, found 694.4566.

Calculations

DFT calculations were performed using the Gaussian09 suite of programs[1]. Harmonic frequency analysis at the same level of theory as the minimization were used to confirm minimized structures. To reduce computational time, simplified structures **1'** and **2'** were used (Figure S1).

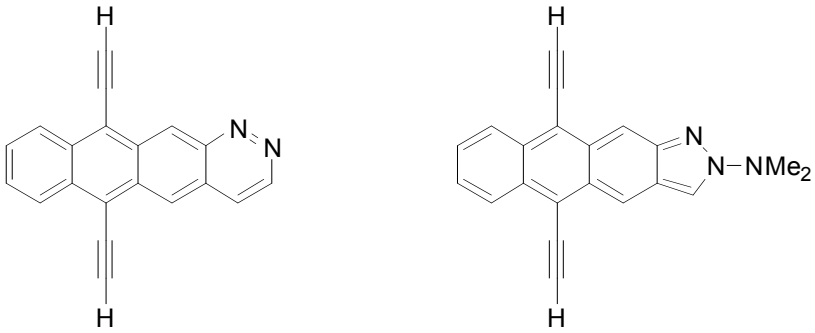

Figure S1 Structures of simplified molecules **1'** and **2'**.

Cartesian Coordinates

1'

B3LYP/6-311+G** = -973.581224445 au
B3LYP/6-311+G** Zero Point Corrected Energy = -973.281256 au
NIMAG = 0

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C	-4.67320300	0.67815900	0.00005000
C	-3.50341900	1.38130700	-0.00002200
C	-2.23767400	0.71374300	-0.00001800
C	-2.22413100	-0.73067200	0.00000000
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C	0.23961600	-0.70715900	-0.00008000
C	-0.99754900	-1.42326000	-0.00006100
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C	2.66319800	0.75970600	0.00004400
C	2.67207400	-0.67475000	-0.00004600
C	1.47327400	-1.37814600	-0.00012100
C	3.94348700	1.37744900	0.00013800
C	5.04173400	0.57154900	0.00012800
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C	-0.95577800	-4.05053300	-0.00004000
C	-1.03916800	2.85332100	-0.00005400

C	-1.04589600	4.05981300	-0.00009800
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H	-5.62114800	1.20400500	0.00007600
H	-3.51342800	2.46357300	-0.00004300
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H	6.05015700	0.96656200	0.00019300
H	-0.92977600	-5.11321600	0.00011300
H	-1.05543200	5.12266100	-0.00028300

2'

B3LYP/6-311+G** = -877.705925347
B3LYP/6-311+G** Zero Point Corrected Energy = -877.473117
NIMAG = 0

C	5.38194800	-0.95840200	0.00008700
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C	-5.83127400	-0.34597200	-1.22392100
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H	6.45329400	0.93558800	0.00007400
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H	-6.91330900	-0.20499500	-1.23267700
H	-5.59988800	-1.41634000	-1.27951100

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1. Gaussian 09, Revision A.1, Frisch, M. J.; Trucks, G. W.; Schlegel, H. B.; Scuseria, G. E.; Robb, M. A.; Cheeseman, J. R.; Scalmani, G.; Barone, V.; Mennucci, B.; Petersson, G. A.; Nakatsuji, H.; Caricato, M.; Li, X.; Hratchian, H. P.; Izmaylov, A. F.; Bloino, J.; Zheng, G.; Sonnenberg, J. L.; Hada, M.; Ehara, M.; Toyota, K.; Fukuda, R.; Hasegawa, J.; Ishida, M.; Nakajima, T.; Honda, Y.; Kitao, O.; Nakai, H.; Vreven, T.; Montgomery, Jr., J. A.; Peralta, J. E.; Ogliaro, F.; Bearpark, M.; Heyd, J. J.; Brothers, E.; Kudin, K. N.; Staroverov, V. N.; Kobayashi, R.; Normand, J.; Raghavachari, K.; Rendell, A.; Burant, J. C.; Iyengar, S. S.; Tomasi, J.; Cossi, M.; Rega, N.; Millam, N. J.; Klene, M.; Knox, J. E.; Cross, J. B.; Bakken, V.; Adamo, C.; Jaramillo, J.; Gomperts, R.; Stratmann, R. E.; Yazyev, O.; Austin, A. J.; Cammi, R.; Pomelli, C.; Ochterski, J. W.; Martin, R. L.; Morokuma, K.; Zakrzewski, V. G.; Voth, G. A.; Salvador, P.; Dannenberg, J. J.; Dapprich, S.; Daniels, A. D.; Farkas, Ö.; Foresman, J. B.; Ortiz, J. V.; Cioslowski, J.; Fox, D. J. Gaussian, Inc., Wallingford CT, **2009**.

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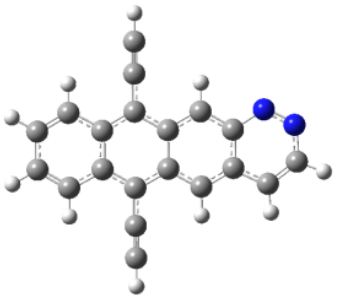
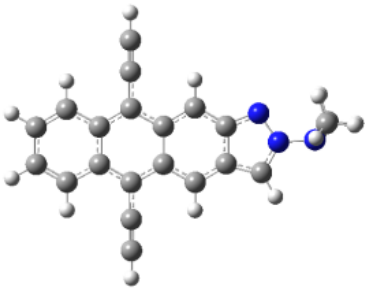
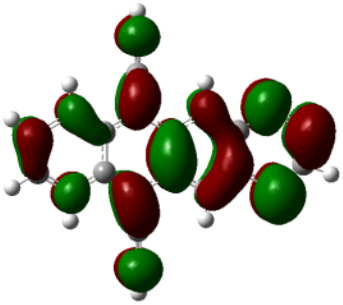
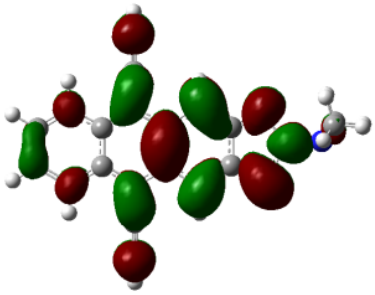
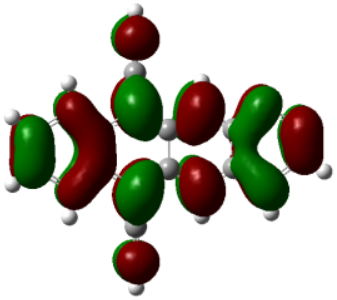
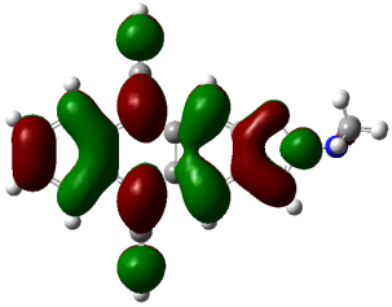
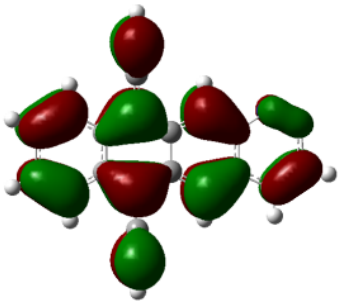
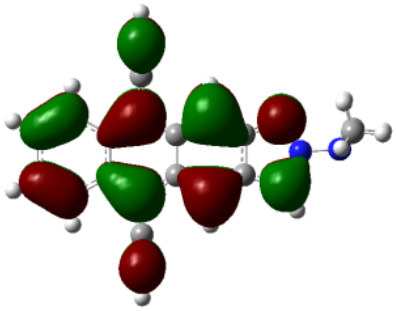
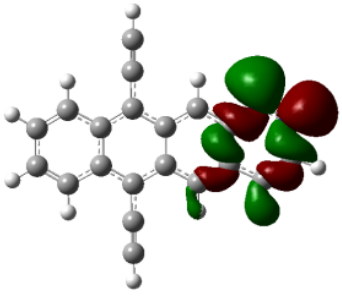
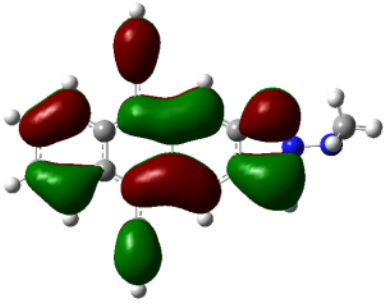
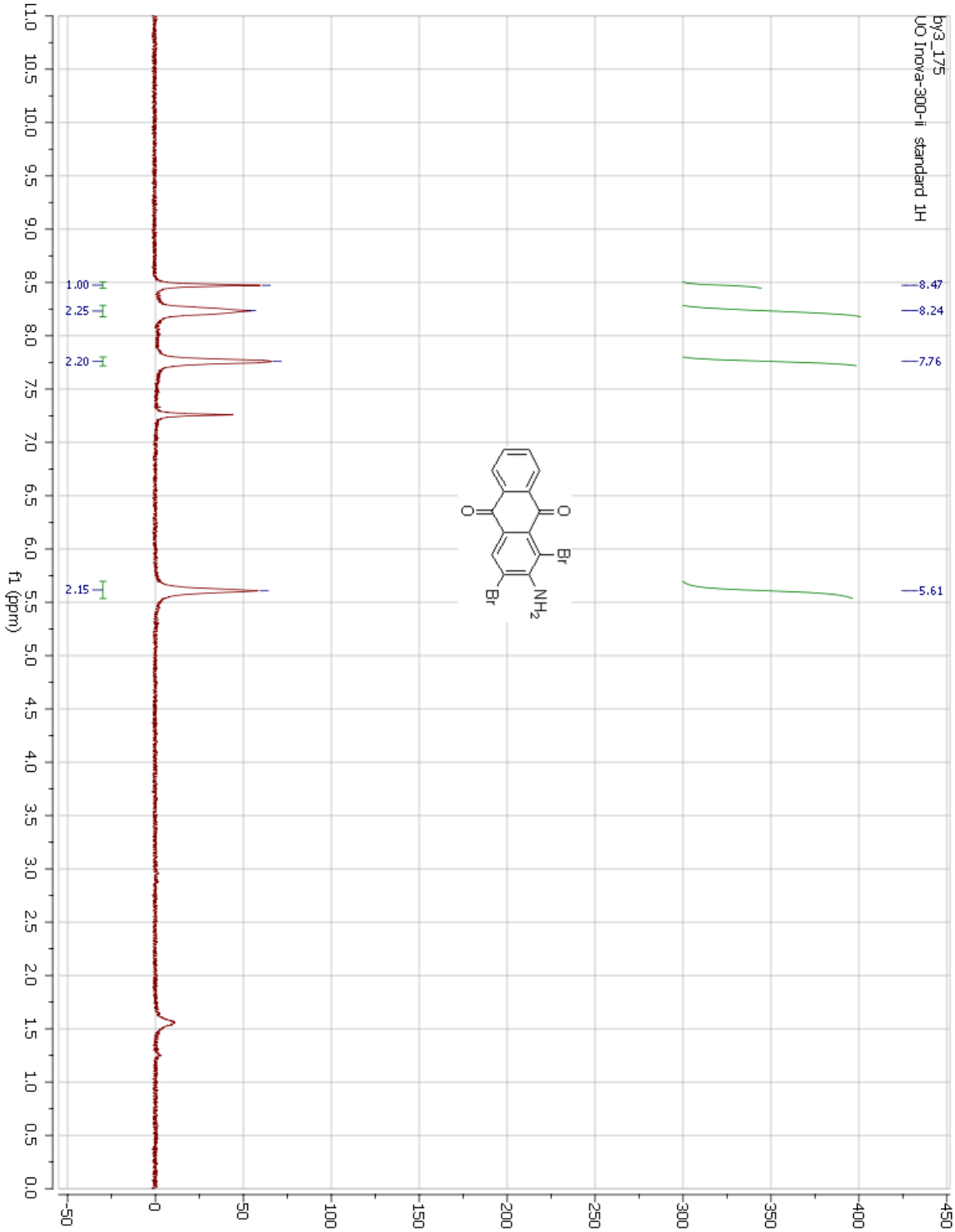
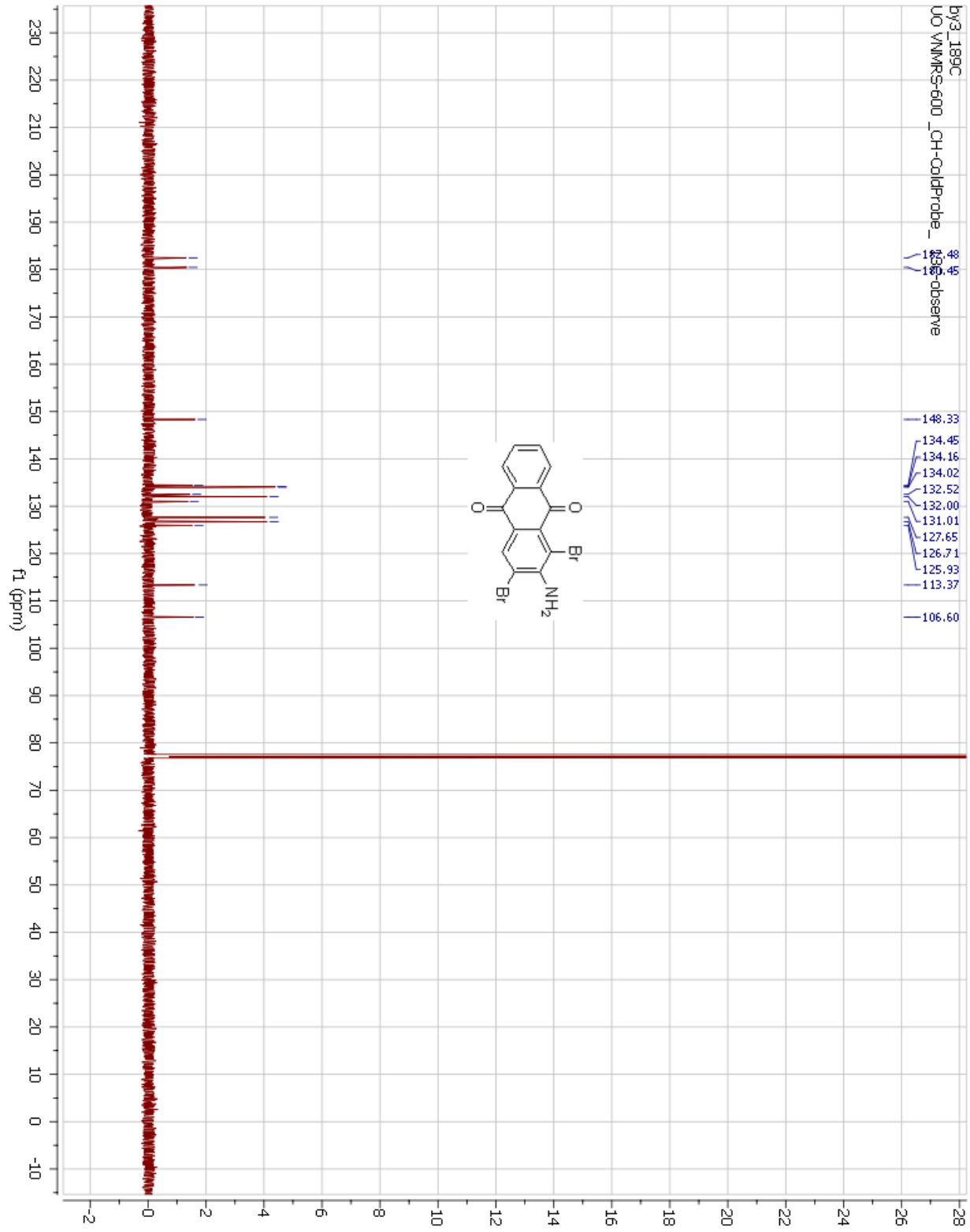
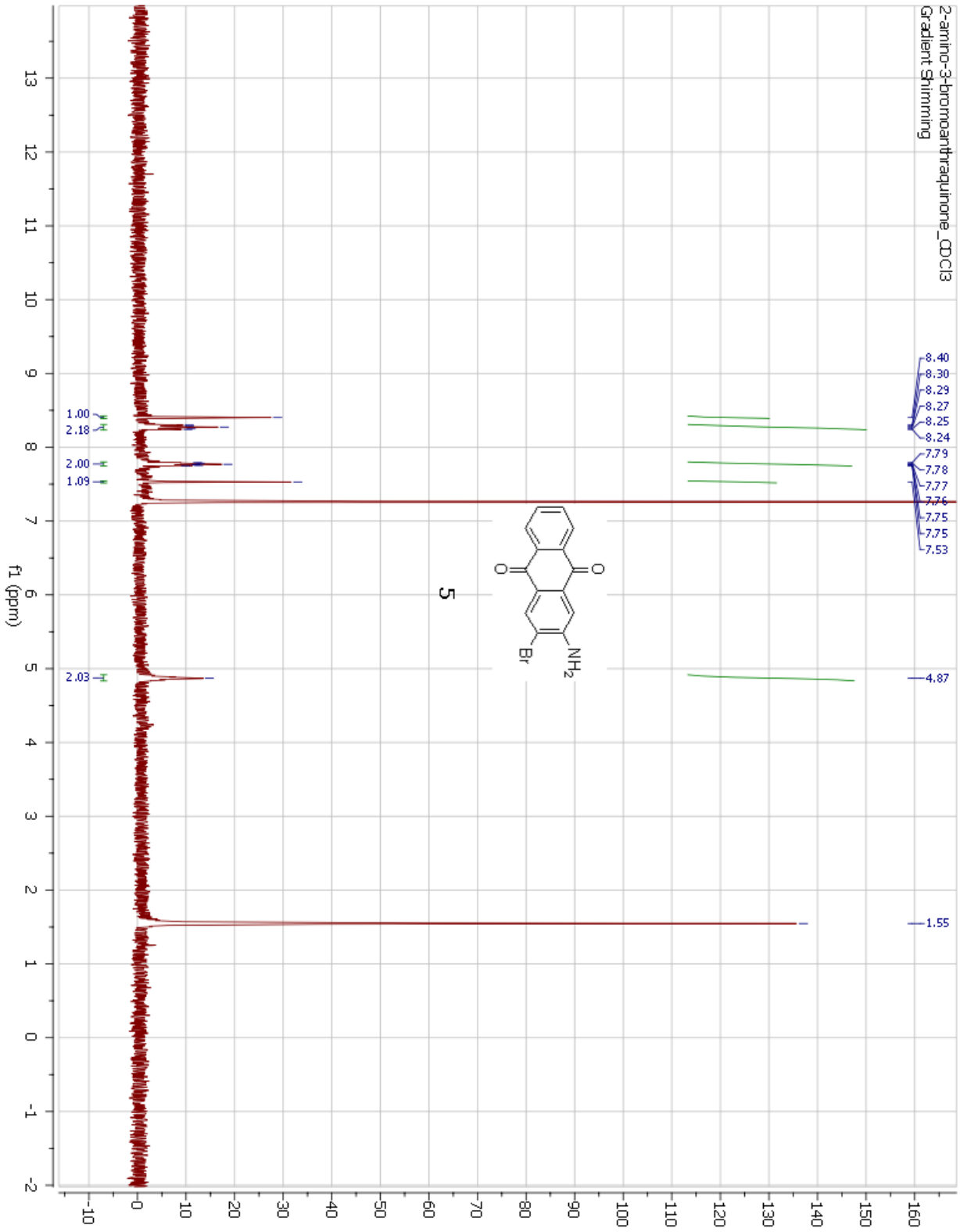
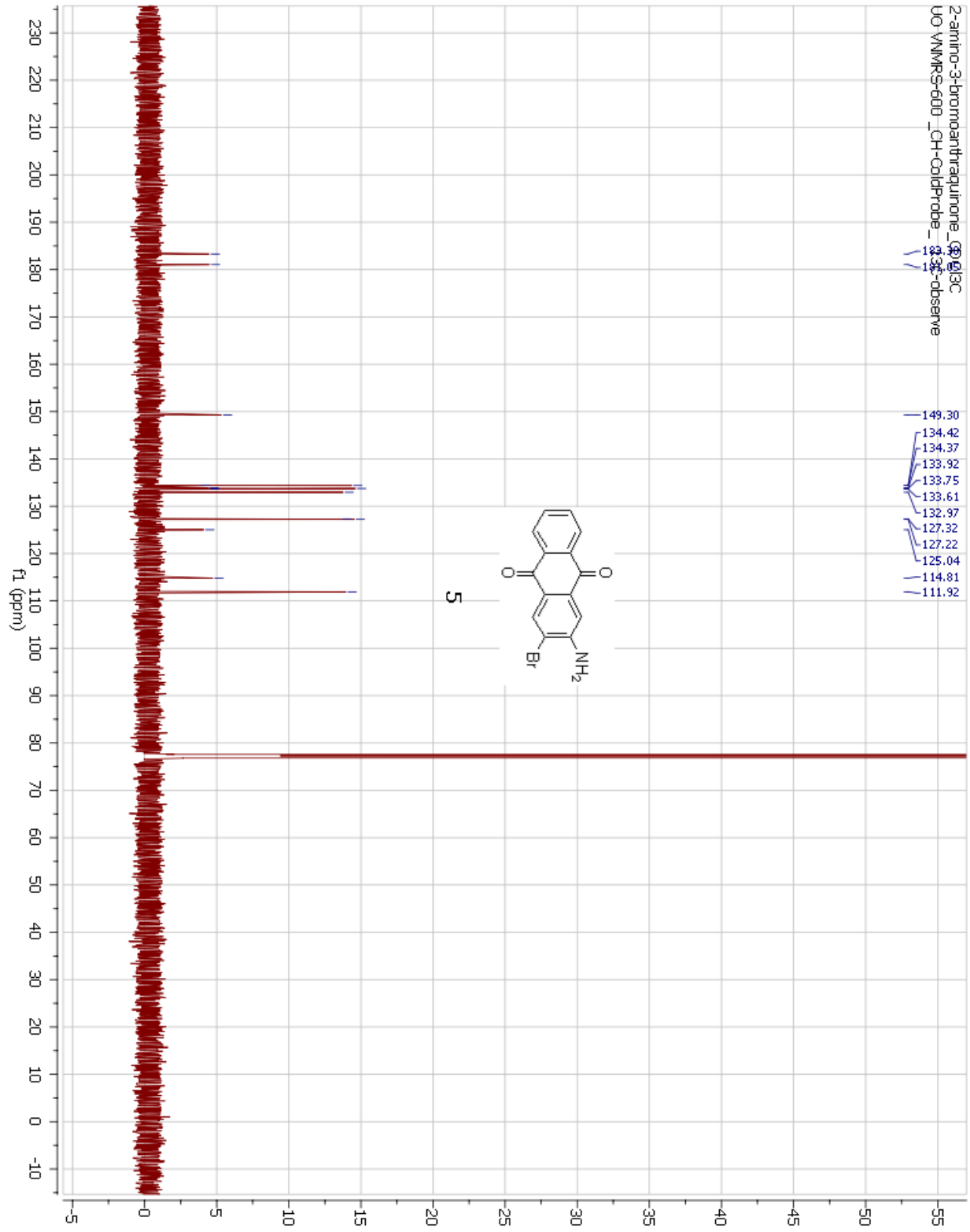
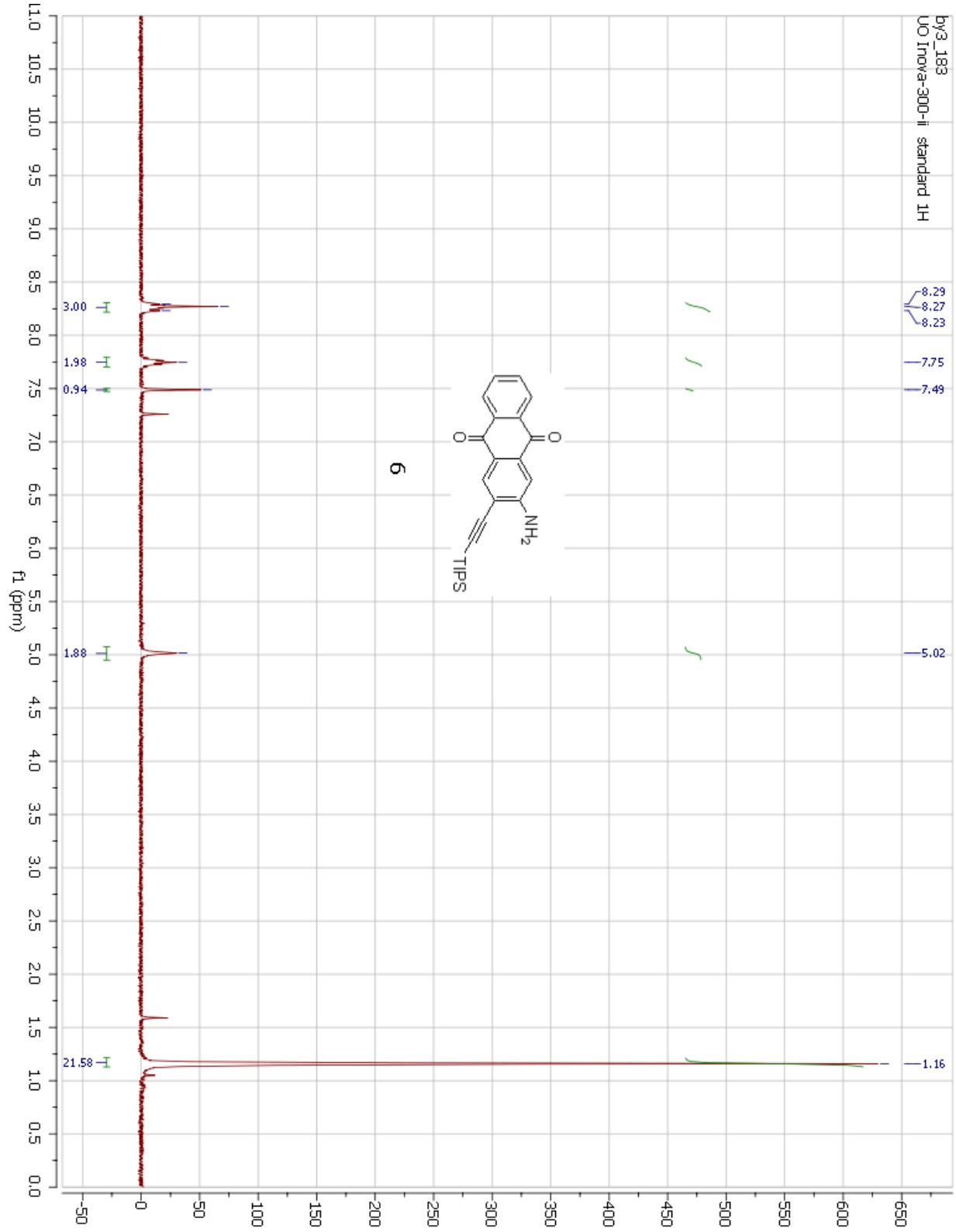
		DFT Minimized Structures
		LUMO+1
		LUMO
		HOMO
		HOMO–1

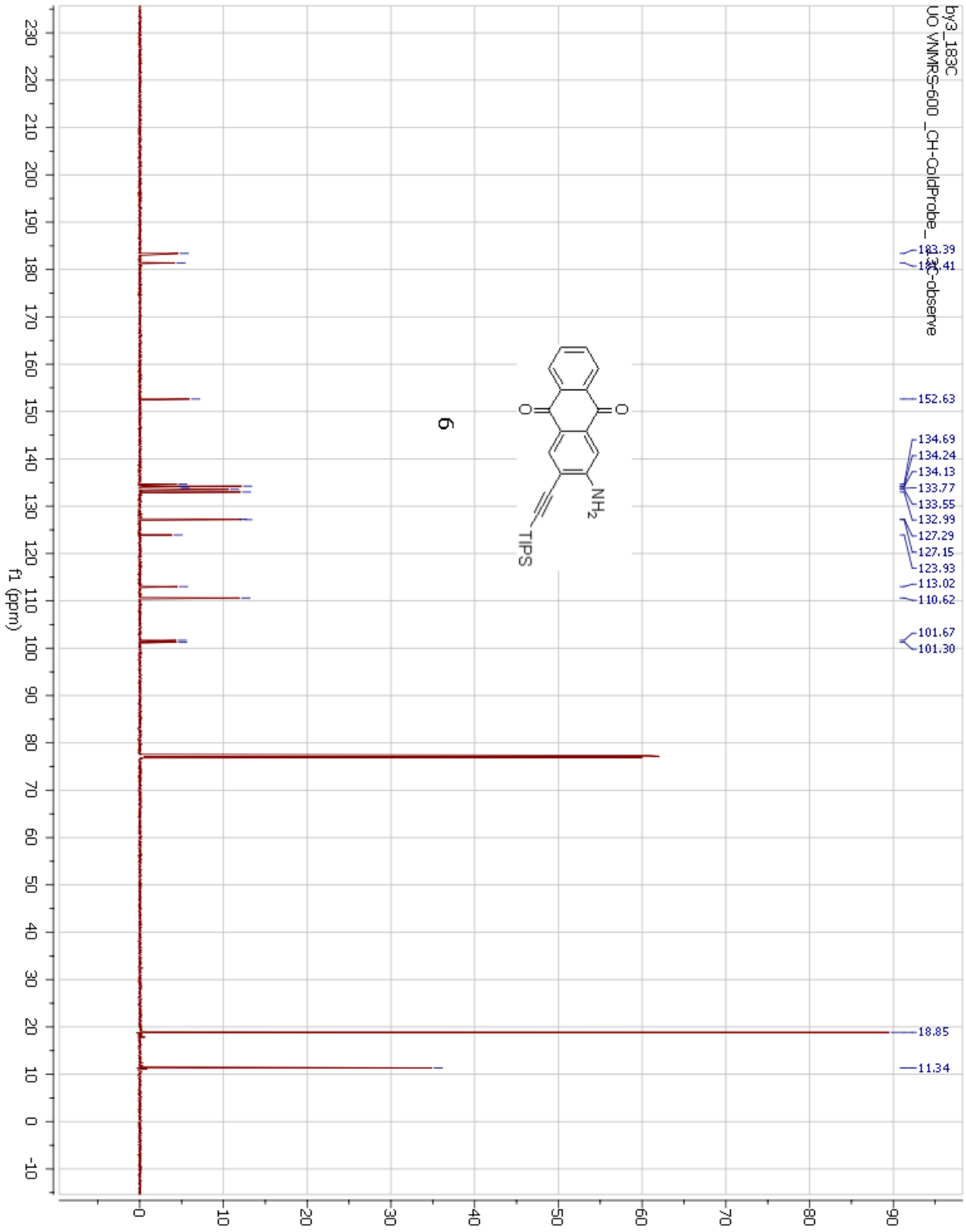

by3_175
UO Inova-300-ii standard 1H
1.00
2.25
2.20
2.15
8.47
8.24
7.76
5.61
O
O
Br
Br
NH2
f1 (ppm)

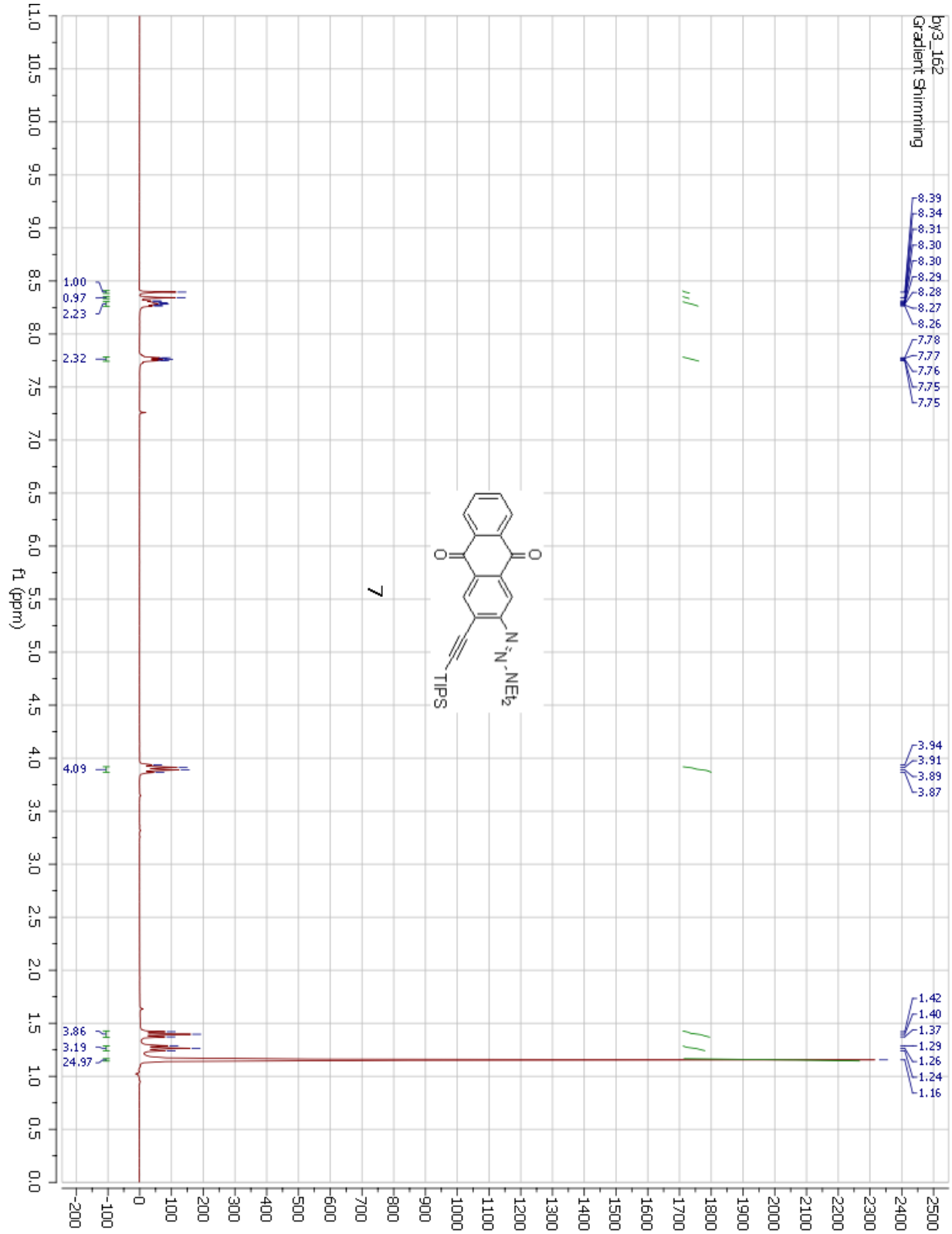

by3_189C
UO VNMRS-600 _CH-ColdProbe_
189.48
189.45
148.33
134.45
134.16
134.02
132.52
132.00
131.01
127.65
126.71
125.93
113.37
106.60
O
O
Br
Br
NH2
f1 (ppm)

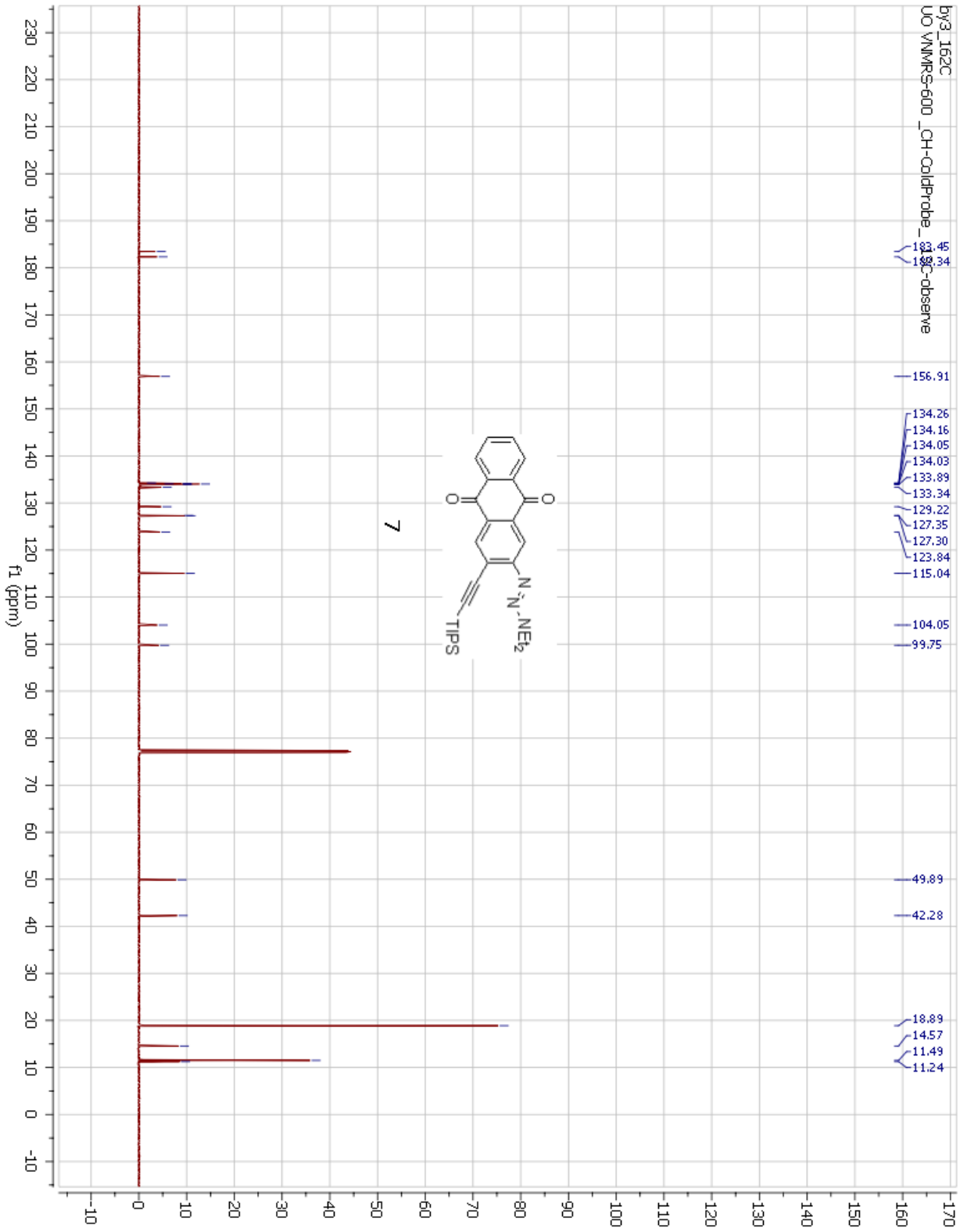

2-amino-3-bromoanthraquinone_CDCl3
Gradient Shimming
f1 (ppm)
1.00
2.18
2.00
1.09
2.03
8.40
8.30
8.29
8.27
8.25
8.24
7.79
7.78
7.77
7.76
7.75
7.75
7.53
4.87
1.55
5
O
O
Br
NH2

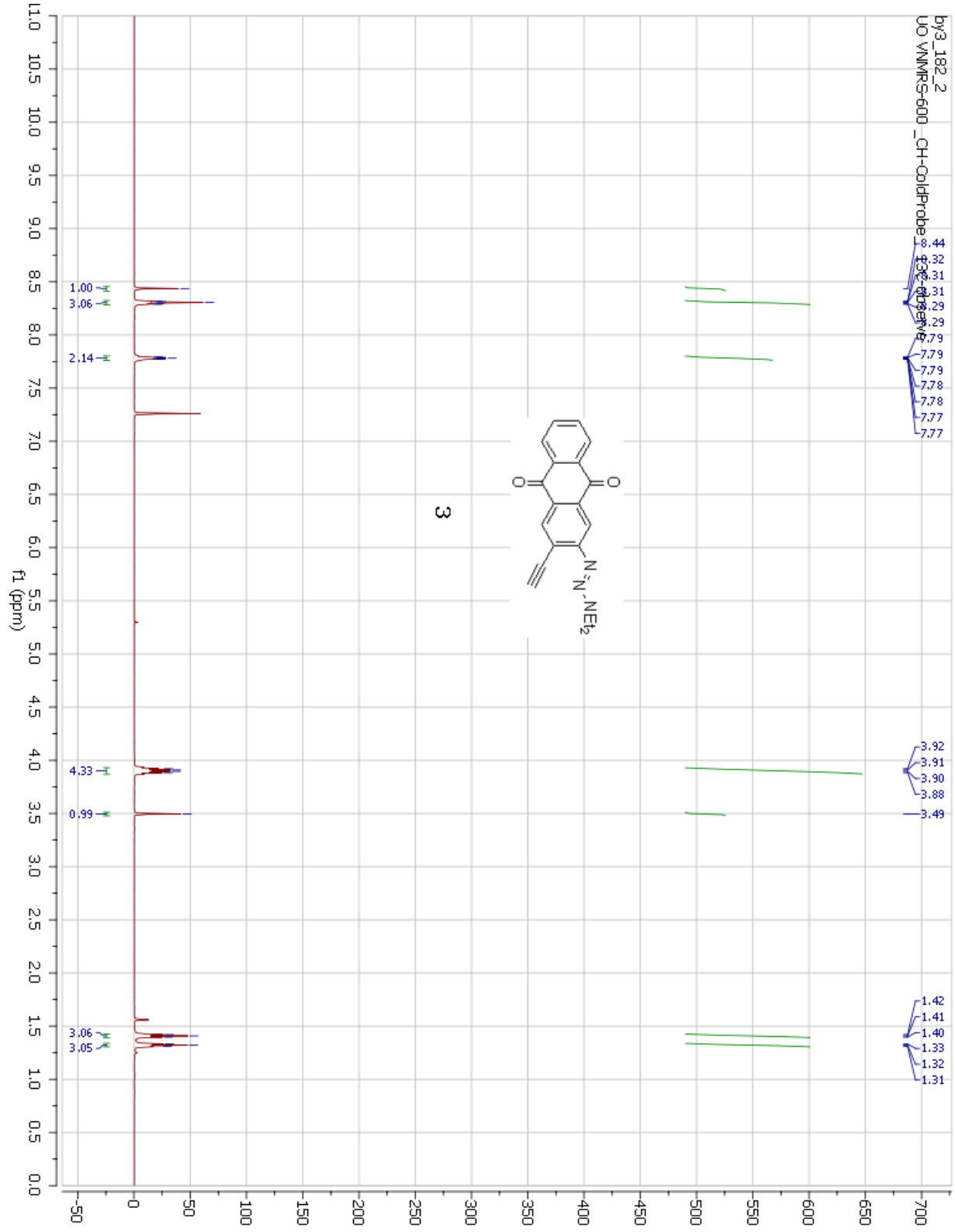

2-amino-3-bromoanthraquinone_CDCl3C
UO vNMRS-600 _CH-ColdProbe_ 13C-observe
149.30
134.42
134.37
133.92
133.75
133.61
132.97
127.32
127.22
125.04
114.81
111.92
5
O
O
Br
NH2
f1 (ppm)

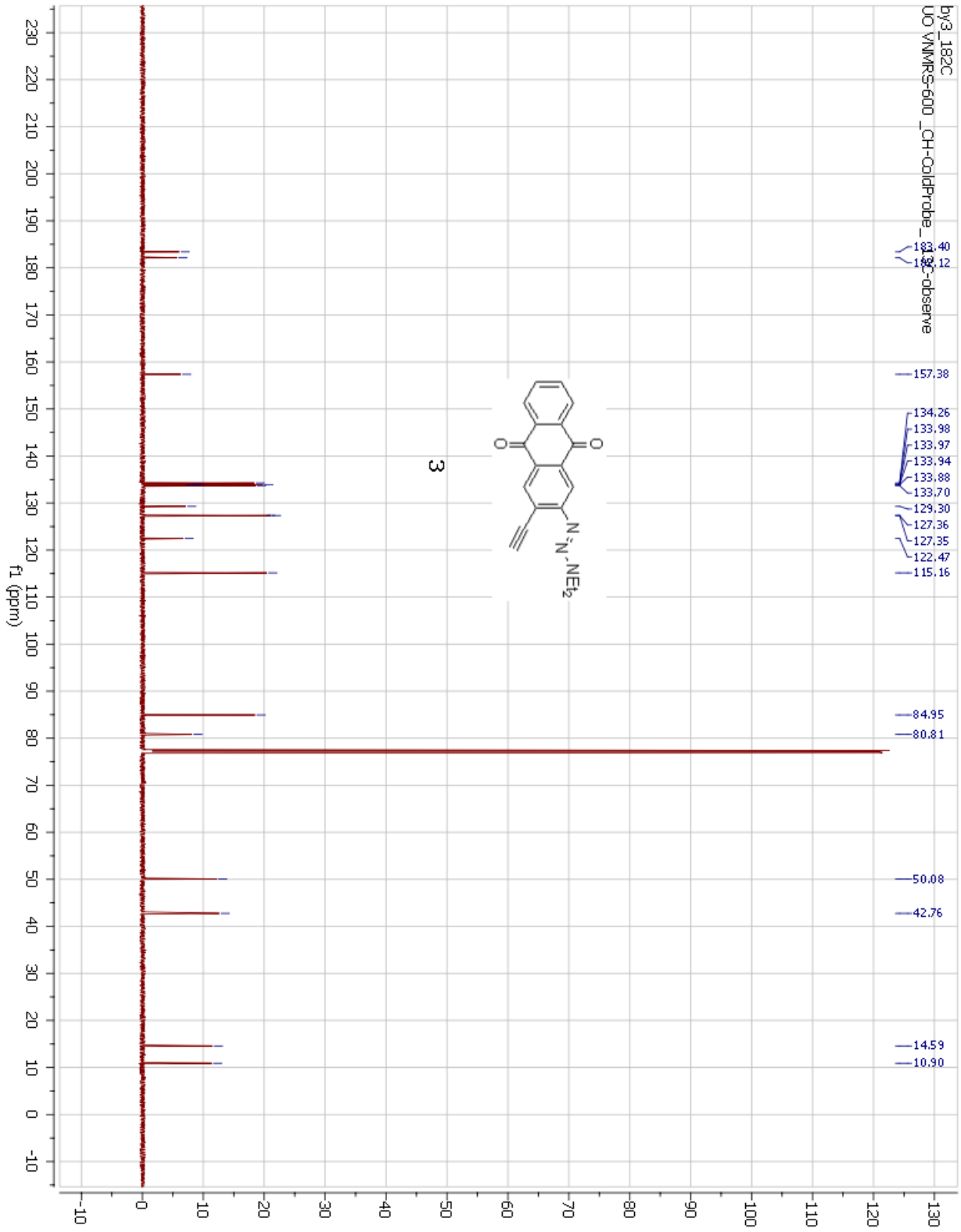

by3_183
UO Inova-300-ii standard 1H
8.29
8.27
8.23
7.75
7.49
5.02
1.16
3.00
1.98
0.94
1.88
21.58
6
O
O
NH2
TIPS
f1 (ppm)

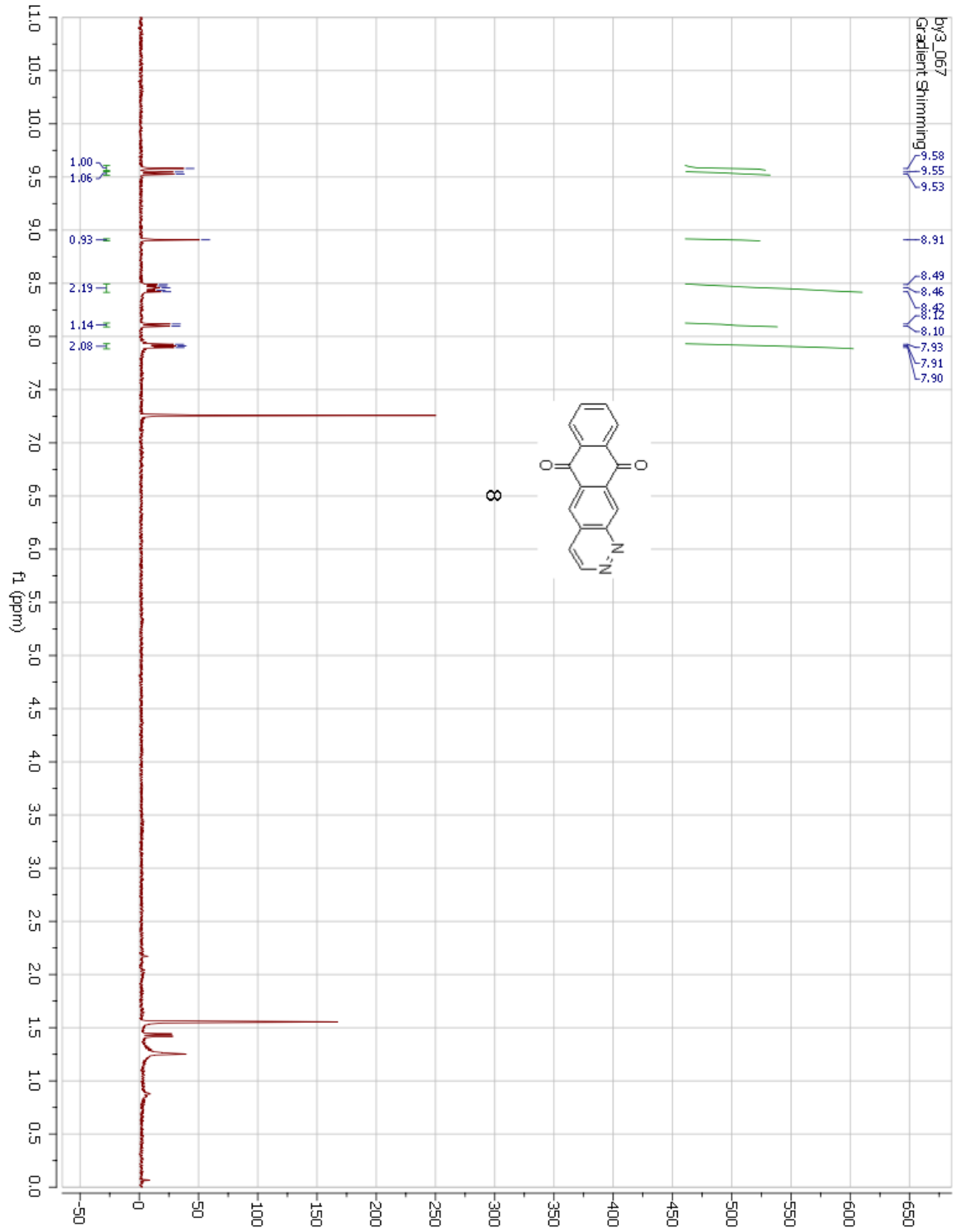

by3_183C
UO VNMRS-600 _CH-ColdProbe_
13C-observe
183.39
182.41
152.63
134.69
134.24
134.13
133.77
133.55
132.99
127.29
127.15
123.93
113.02
110.62
101.67
101.30
18.85
11.34
6
NH2
TIPS
f1 (ppm)

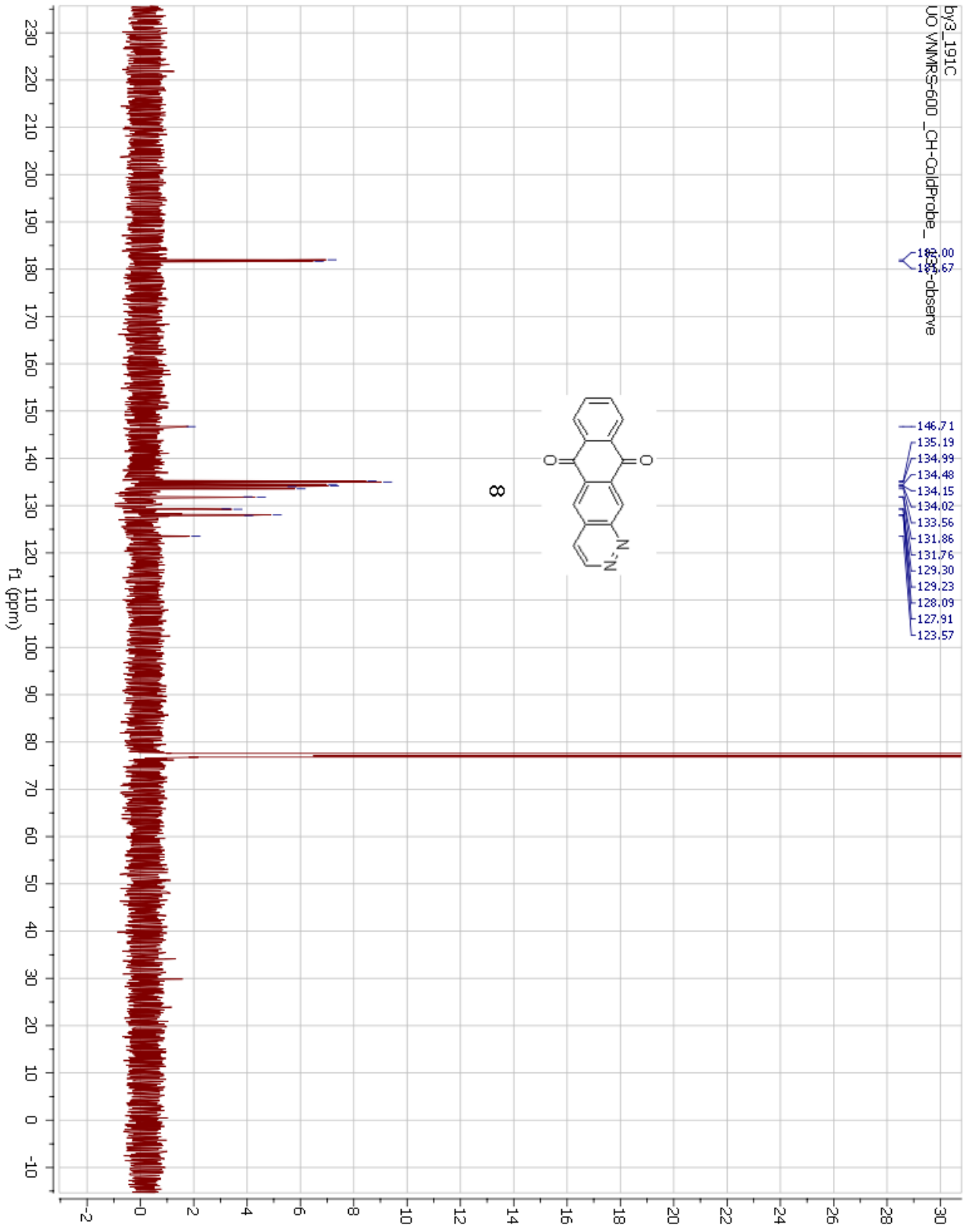

by3_162
Gradient Shimming
f1 (ppm)
1.00
0.97
2.23
2.32
4.09
3.86
3.19
24.97
8.39
8.34
8.31
8.30
8.30
8.29
8.28
8.27
8.26
7.78
7.77
7.76
7.75
7.75
3.94
3.91
3.89
3.87
1.42
1.40
1.37
1.29
1.26
1.24
1.16
7
TIPS
NEt2

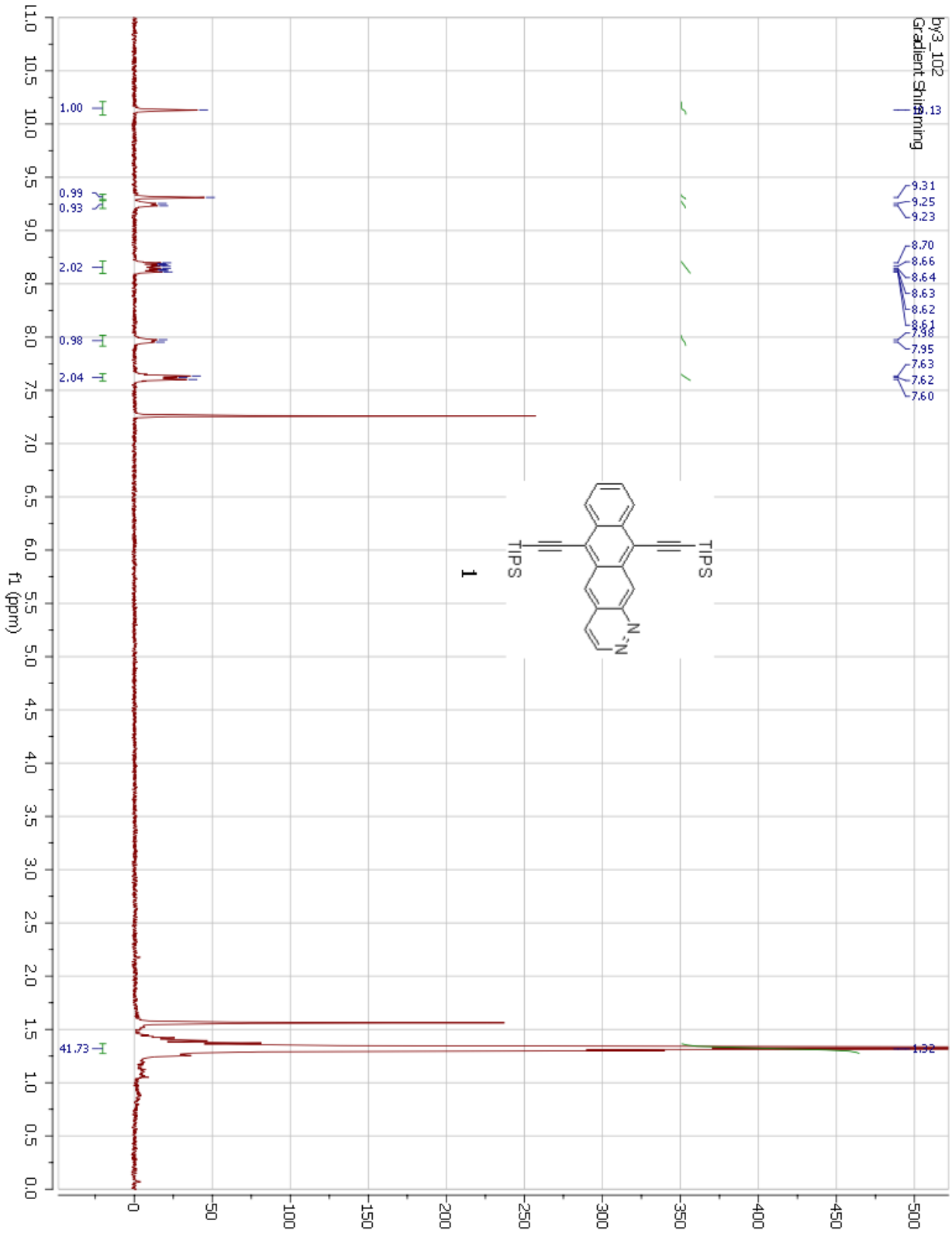

by3_162C
UO VNMRS-600 _CH-ColdProbe_
13C-observe
183.45
183.34
156.91
134.26
134.16
134.05
134.03
133.89
133.34
129.22
127.35
127.30
123.84
115.04
104.05
99.75
49.89
42.28
18.89
14.57
11.49
11.24
7
TIPS
NEt2
f1 (ppm)

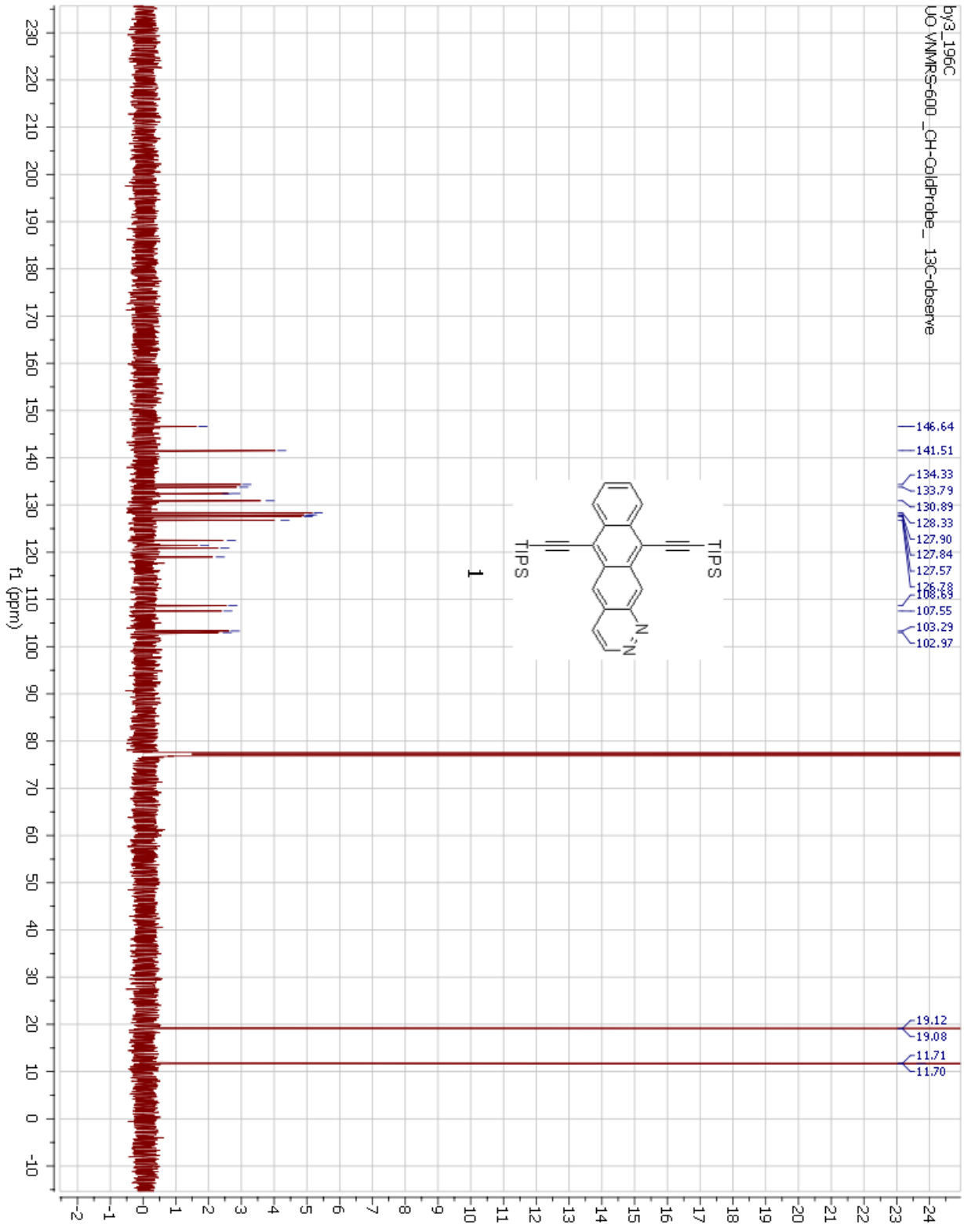

by3_182_2
UO vNMRS-600 _CH-ColdProbe_
f1 (ppm)
1.00
3.06
2.14
4.33
0.99
3.06
3.05
8.44
8.32
8.31
8.31
8.29
8.29
7.79
7.79
7.79
7.78
7.78
7.77
7.77
3.92
3.91
3.90
3.88
3.49
1.42
1.41
1.40
1.33
1.32
1.31
3
O
O
N
N
NEt2

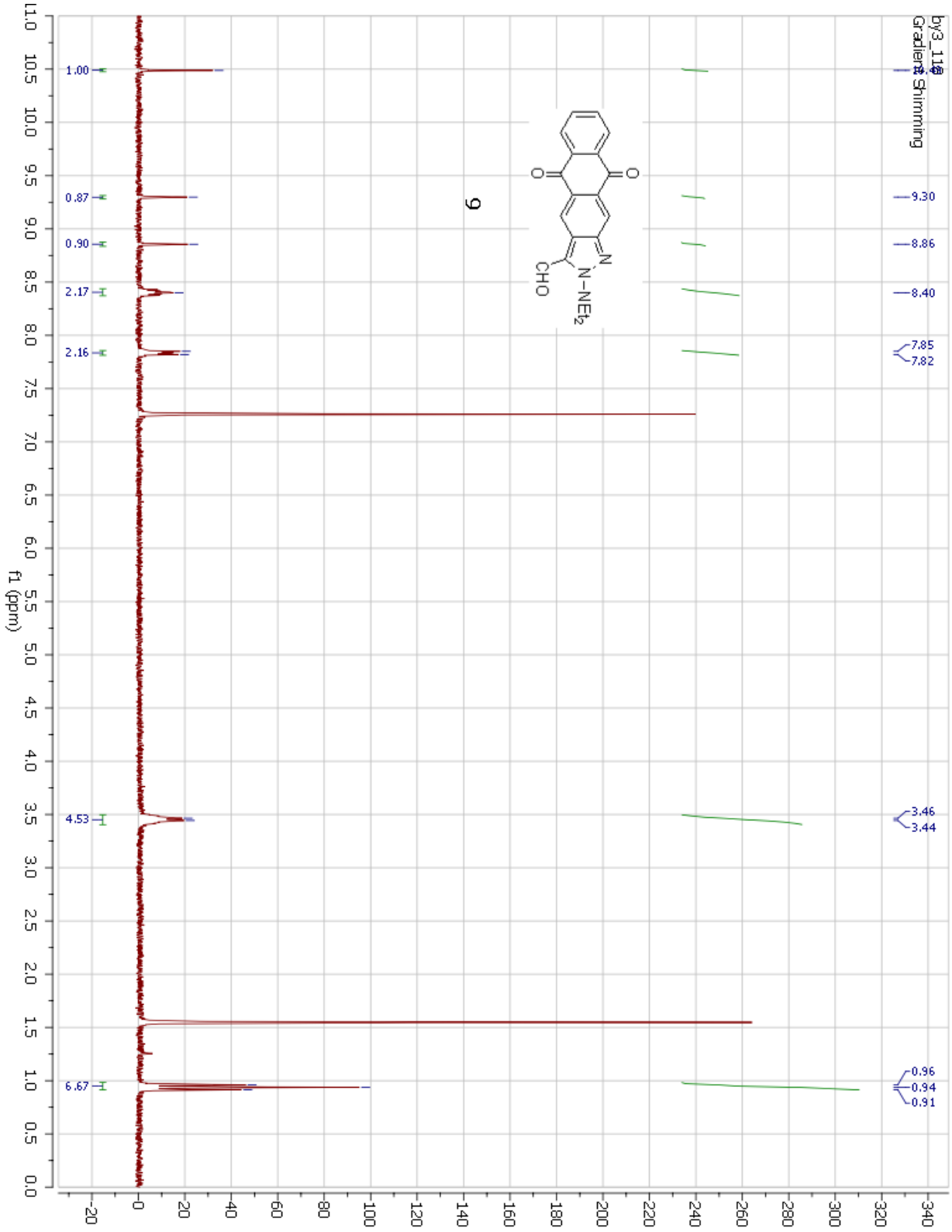

by3_182C
UO VNMRS-600 _CH-ColdProbe_
13C-observe
183.40
182.12
157.38
134.26
133.98
133.97
133.94
133.88
133.70
129.30
127.36
127.35
122.47
115.16
84.95
80.81
50.08
42.76
14.59
10.90
f1 (ppm)
3
O
O
N
N
NEt2

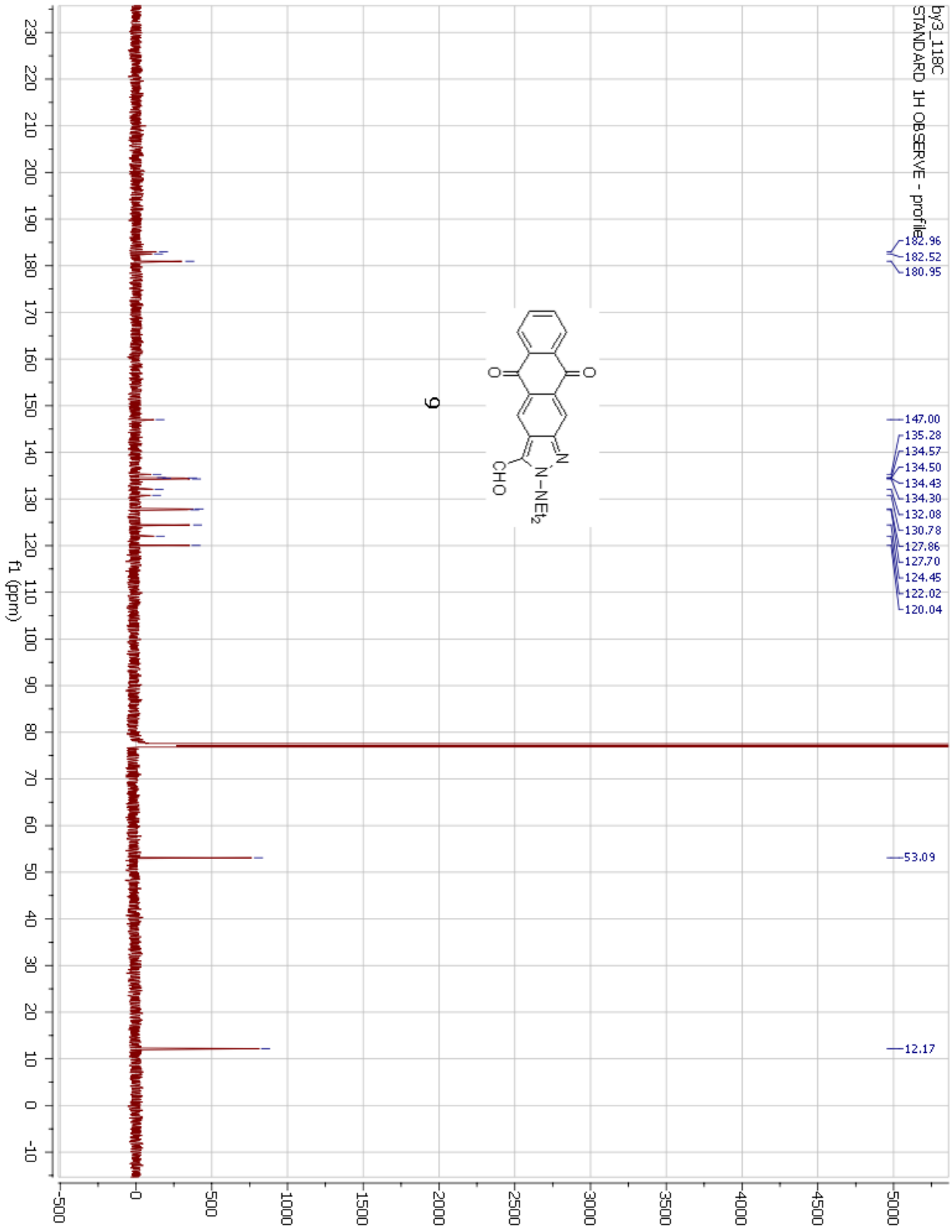

by3_067
Gradient Shimming
9.58
9.55
9.53
8.91
8.49
8.46
8.42
8.12
8.10
7.93
7.91
7.90
1.00
1.06
0.93
2.19
1.14
2.08
f1 (ppm)
8

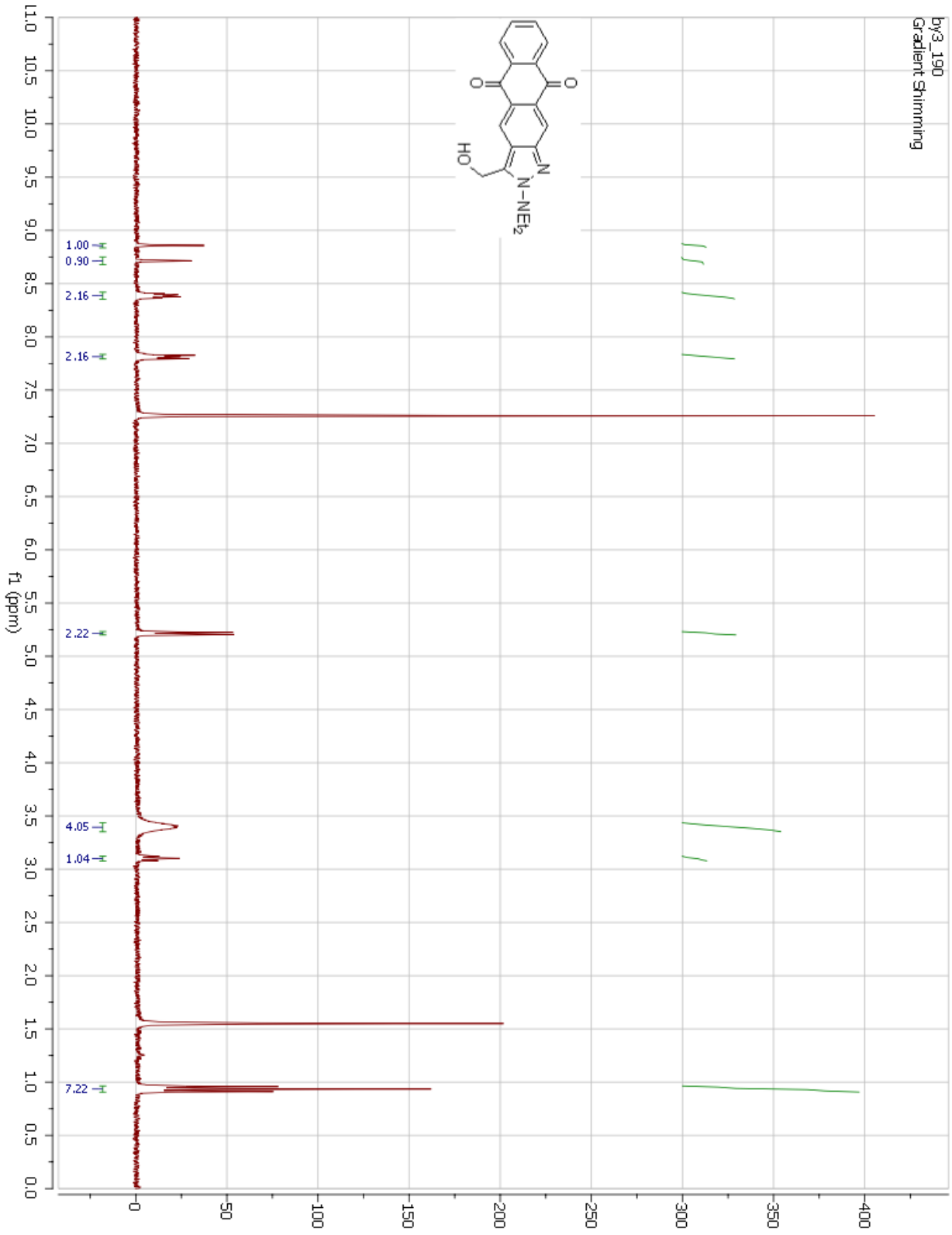

by3_191C
UO VNMRS-600 _CH-ColdProbe_
13C-observe
183.00
182.67
146.71
135.19
134.99
134.48
134.15
134.02
133.56
131.86
131.76
129.30
129.23
128.09
127.91
123.57
8
O
O
N
N
f1 (ppm)

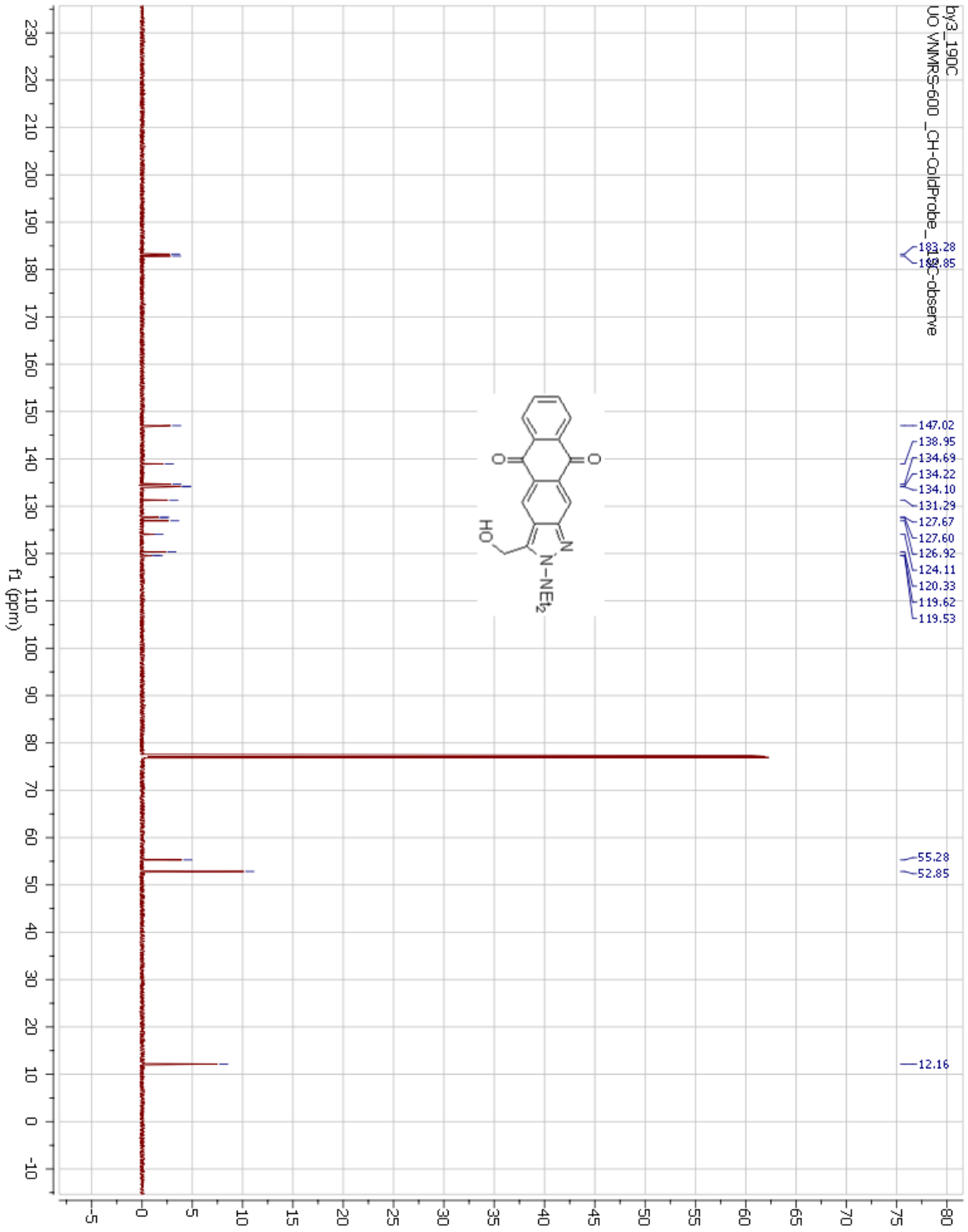

by3_102
Gradient Shimming
9.31
9.25
9.23
8.70
8.66
8.64
8.63
8.62
8.61
7.98
7.95
7.63
7.62
7.60
1.00
0.99
0.93
2.02
0.98
2.04
41.73
1.32
TIPS
TIPS
N
N
1
f1 (ppm)

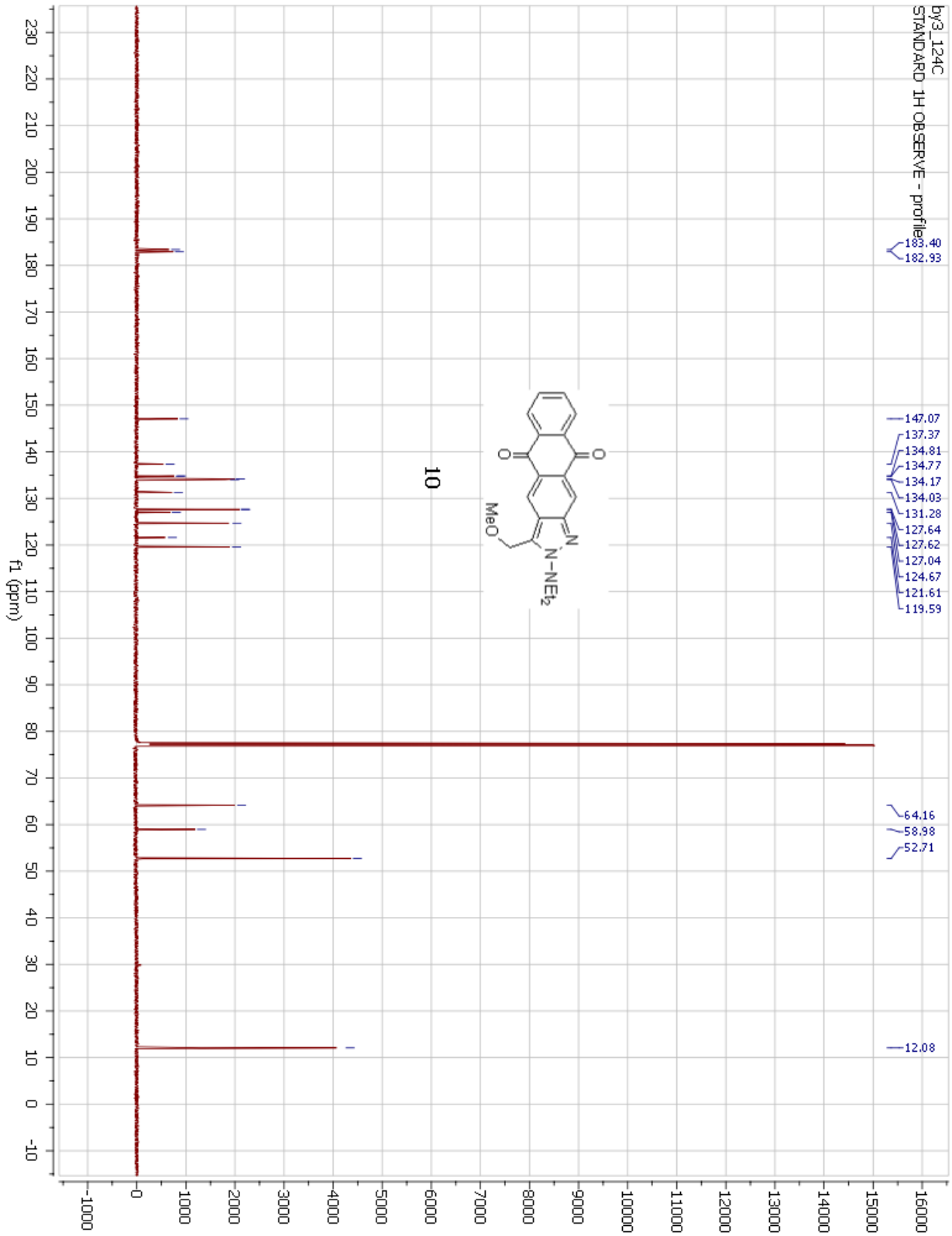

by3_196C
UO vNMRS-600 _CH-ColdProbe_ 13C-observe
146.64
141.51
134.33
133.79
130.89
128.33
127.90
127.84
127.57
126.78
108.69
107.55
103.29
102.97
19.12
19.08
11.71
11.70
TIPS
TIPS
1
f1 (ppm)

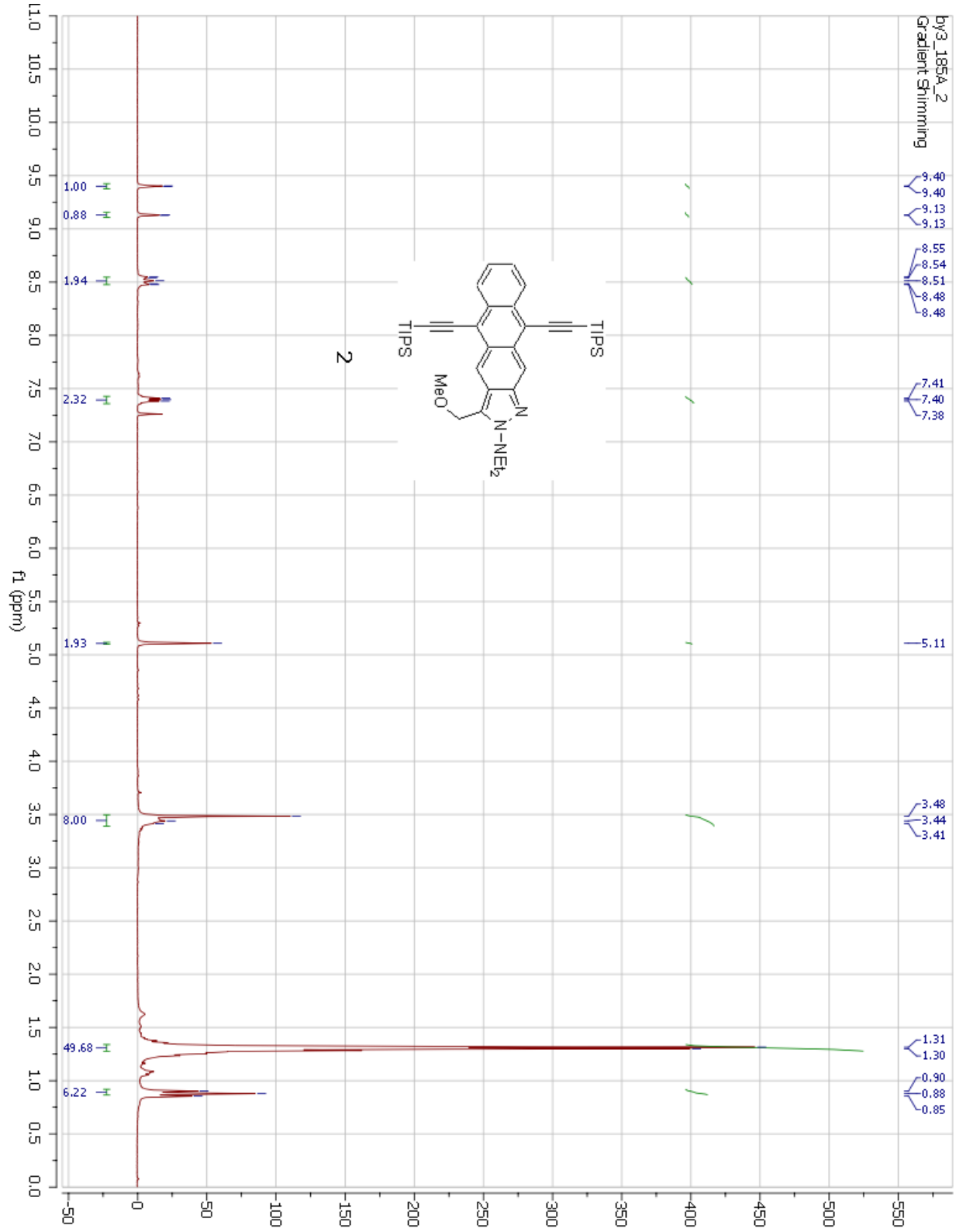

by3_11
Gradient Shimming
9
f1 (ppm)
1.00
0.87
0.90
2.17
2.16
4.53
6.67
9.30
8.86
8.40
7.85
7.82
3.46
3.44
0.96
0.94
0.91

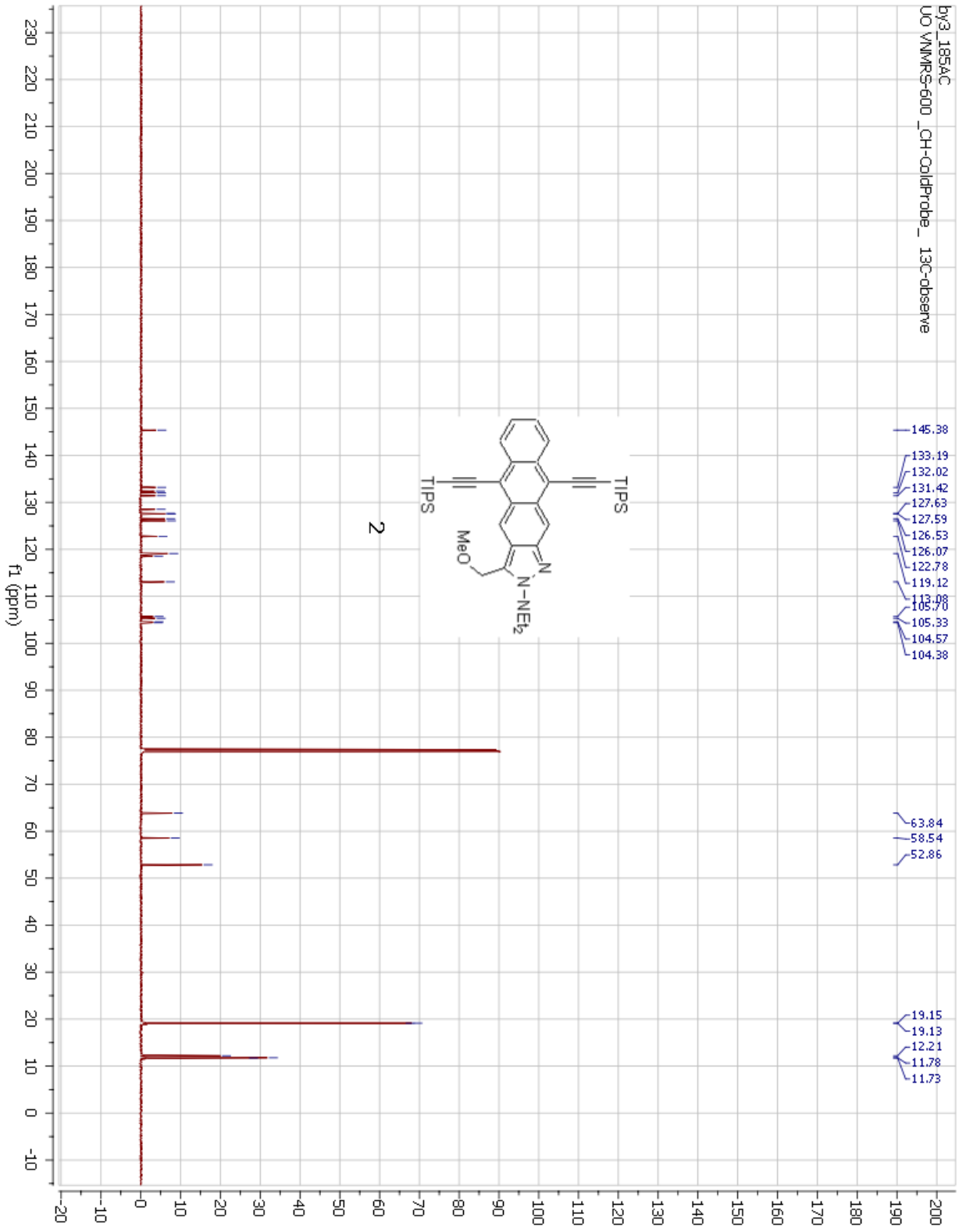

by3_118C
STANDARD 1H OBSERVE - profile
182.96
182.52
180.95
147.00
135.28
134.57
134.50
134.43
134.30
132.08
130.78
127.86
127.70
124.45
122.02
120.04
53.09
12.17
f1 (ppm)
9
CHO
N-NEt2


by3_190
Gradient Shimming
f1 (ppm)
1.00
0.90
2.16
2.16
2.22
4.05
1.04
7.22
HO
N
N–NEt2
O
O


by3_190C
UO VNMRS-600 _CH-ColdProbe_13C-observe
183.28
147.02
138.95
134.69
134.22
134.10
131.29
127.67
127.60
126.92
124.11
120.33
119.62
119.53
55.28
52.85
12.16
HO
O
O
N
N
N-NEt2
f1 (ppm)


by3_124C
STANDARD 1H OBSERVE - profile
183.40
182.93
147.07
137.37
134.81
134.77
134.17
134.03
131.28
127.64
127.62
127.04
124.67
121.61
119.59
64.16
58.98
52.71
12.08
10
MeO
N
N-NEt2
O
O
f1 (ppm)


by3_195A_2
Gradient Shimming
f1 (ppm)
1.00
0.88
1.94
2.32
1.93
8.00
49.68
6.22
2
TIPS
TIPS
MeO
N
N–NEt2
9.40
9.40
9.13
9.13
8.55
8.54
8.51
8.48
8.48
7.41
7.40
7.38
5.11
3.48
3.44
3.41
1.31
1.30
0.90
0.88
0.85


by3_185AC
UO VNMRS-600 _CH-ColdProbe_ 13C-observe
f1 (ppm)
145.38
133.19
132.02
131.42
127.63
127.59
126.53
126.07
122.78
119.12
113.08
105.70
105.33
104.57
104.38
63.84
58.54
52.86
19.15
19.13
12.21
11.78
11.73
TIPS
TIPS
MeO
N
N-NEt2
2