Electronic Supplementary Material (ESI) for Organic & Biomolecular Chemistry.

Supporting Information

Telescoped Synthesis of C3-Functionalized (*E*)-Arylamidines Using Ugi-Mumm and Regiospecific Quinazolinone Rearrangements

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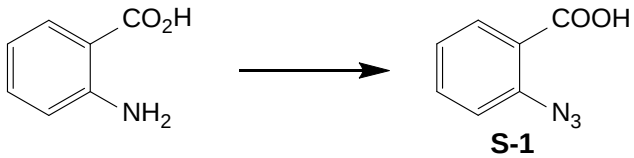
1. Experimental Procedures

General Methods

Compounds not described below were purchased from commercial vendors. Purity of all final compounds was confirmed by HPLC/MS analysis and determined to be ≥ 95%.

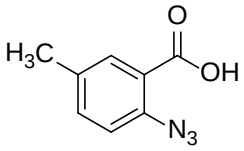
Analytical TLC experiments were performed on aluminum-backed Silica Gel plates (TLC Silica gel 60 F_{254}) from EMD Millipore and analyzed with 254 nm UV light using diluted samples. All compounds were characterized with the following instrumentation: Varian Unity-Inova 400 MHz NMR spectrometer (operating at 400 and 101 MHz, respectively) or a Varian Unity-Inova 500 MHz NMR spectrometer (operating at 500 and 126 MHz, respectively) in $CDCl_3$ ($CDCl_3$: ^1H = δ 7.26 ppm, ^{13}C = δ 77.16 ppm). The chemical shifts (δ) reported are given in parts per million (ppm) and the coupling constants (J) are in Hertz (Hz). The spin multiplicities are reported as s = singlet, br s = broad singlet, d = doublet, t = triplet, q = quartet, p = pentuplet, dd = doublet of doublet, ddd = doublet of doublet of doublet, and m = multiplet. The LC-MS analysis was performed on an Agilent 1290 Infinity II HPLC system with 1290 Infinity II Diode Array Detector and an Agilent 6120 Quadrupole LC-MS system. The analytical chromatography method utilized the following parameters: Poroshell 120 EC-C18, 1.9 µm column, UV detection wavelength = 254 nm, Flow rate = 1.0 mL/min, Gradient = 5-100% LC-MS grade Methanol over 4 min; The organic mobile phase and aqueous mobile phase contained 0.1% LC-MS grade formic acid. The mass spectrometer utilized the following parameters: an Agilent multimode source that simultaneously acquires ESI+/APCI+; Final compounds were determined to be > 95% purity by UV-LCMS at 254 nm. Microwave irradiated (MWI) reactions were carried out using an Anton Paar Monowave 300 Microwave Synthesis Reactor. Flash chromatography separations were carried out using a Teledyne Isco CombiFlash Rf 200 purification system with silica gel columns (normal-phase). High resolution mass spectra (HRMS) were performed by Analytical Instrument Center at the School of Pharmacy on an Electron Spray Injection (ESI) mass spectrometer. Melting points were measured on OptiMelt MPA100 Automated Melting Point System.

1.1 General procedure A: Representative Procedure for the Synthesis of 2-azidobenzoic acid derivatives[1]



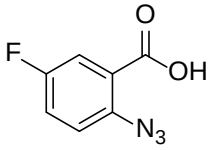
S-1

2-Azidobenzoic acid S-1. To a flame-dried, 250 mL round bottom flask under nitrogen was added anthranilic acid (1.4 g, 10.0 mmol, 1.0 equiv.) and dry acetonitrile (100 mL, 0.1 M). The solution was cooled to 0 °C in an ice bath. After 20 min *tert*-butyl nitrite (1.8 mL, 15.0 mmol, 1.5 equiv.) was added dropwise followed by azidotrimethylsilane (1.6 mL, 12.0 mmol, 1.2 equiv.). The reaction mixture was allowed to warm slowly to rt under nitrogen for 12 h. The reaction mixture was concentrated *in vacuo*, diluted with EtOAc (50 mL) and washed saturated brine solution (2x 50 mL). After separation, the organic layer was dried over $MgSO_4$, filtered and concentrated *in vacuo* to yield 2-azidobenzoic acid, **S-1** (1.47 g, 90%) as a light brown solid. Material carried forward without further purification. Characterization matched literature reference.[1]



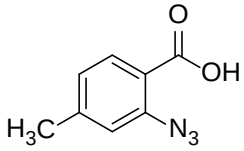
S-2

2-Azido-5-methylbenzoic acid S-2. Obtained using **general procedure A** as a light brown solid (467 mg, 82%). Material carried forward without further purification. Characterization matched literature reference.[2]



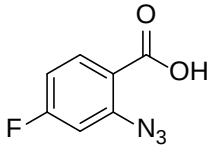
S-3

2-Azido-5-fluorobenzoic acid S-3 Obtained using **general procedure A** as an off-white solid (513 mg, 88%). Material carried forward without further purification. Characterization matched literature reference.[2]



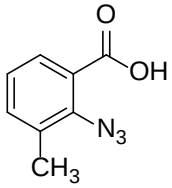
S-4

2-Azido-4-methylbenzoic acid S-4. Obtained using **general procedure A** as a light brown solid (201 mg, 86%). Material carried forward without further purification. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.01 (d, *J* = 8.4 Hz, 1H), 7.05 (d, *J* = 6.5 Hz, 1H), 2.44 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 168.3, 145.8, 140.0, 133.4, 126.0, 119.9, 118.0, 21.6.



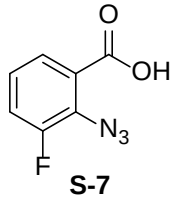
S-5

2-Azido-4-fluorobenzoic acid S-5. Obtained using **general procedure A** as a light brown solid (491 mg, 84%). Material carried forward without further purification. Characterization matched literature reference.[1]



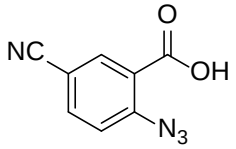
S-6

2-Azido-3-methylbenzoic acid S-6. Obtained using **general procedure A** as a light brown solid (283 mg, 97%). Material carried forward without further purification. Characterization matched literature reference.[3]



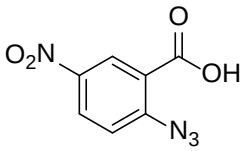
S-7

2-Azido-3-fluorobenzoic acid S-7. Obtained using **general procedure A** as a light brown solid (231 mg, 79%). Crude material carried forward. ^{1}H NMR (400 MHz, DMSO-d_6) δ 13.50 (s, 1H), 7.61 (dt, *J* = 7.8, 1.3 Hz, 1H), 7.53 (ddd, *J* = 11.4, 8.3, 1.5 Hz, 1H), 7.30 (td, *J* = 8.1, 5.1 Hz, 1H). ^{19}F NMR (376 MHz, DMSO) δ -123.87. ^{13}C NMR (101 MHz, DMSO) δ 166.2, 166.2, 157.7, 155.3, 127.3, 127.2, 127.2, 127.0, 127.0, 126.6, 126.5, 120.2, 119.9.



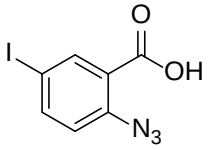
S-8

2-Azido-5-cyanobenzoic acid S-8. Obtained using **general procedure A**. The crude residue was purified by normal-phase chromatography (0.5% MeOH/CH_2Cl_2) to yield 2-azido-5-cyanobenzoic acid, **S-8** (171 mg, 45%) as orange solid. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.37 (d, *J* = 2.1 Hz, 1H), 7.86 (dd, *J* = 8.5, 2.0 Hz, 1H), 7.39 (d, *J* = 8.5 Hz, 1H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 166.7, 145.1, 137.2, 137.1, 121.6, 120.7, 117.2, 108.7.



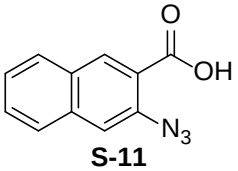
S-9

2-Azido-5-nitrobenzoic acid S-9. Obtained using **general procedure A** as an orange solid (476 mg, 71%). Material carried forward without further purification. Characterization matched literature reference.[4]



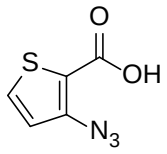
S-10

2-Azido-5-iodobenzoic acid S-10. Obtained using **general procedure A** as a light orange solid (859 mg, 92%). Crude material carried forward. Characterization matched literature reference.[1]



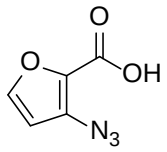
S-11

3-Azido-2-naphthanoic acid S-11. Obtained using **general procedure A** as a red solid (120 mg, 53%). Crude material carried forward. Characterization matched literature reference.[2]



S-12

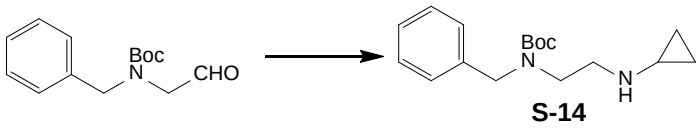
3-Azidothiophene-2-carboxylic acid S-12. Obtained using **general procedure A** as a brown solid (541 mg, 92%). Crude material carried forward. Characterization matched literature reference.[5]



S-13

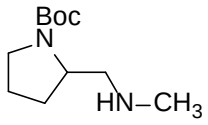
3-Azidofuran-2-carboxylic acid S-13. Obtained using literature procedure[5] as a brown solid (122 mg, 54%). Crude material carried forward. Characterization matched literature reference.[5]

1.2 Procedure for the Synthesis of *tert*-butyl benzyl(2-cyclopropylamino)ethyl) carbamate



S-14

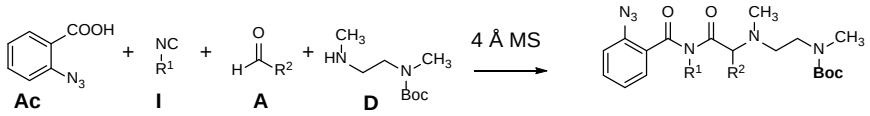
***tert*-Butyl benzyl(2-cyclopropylamino)ethyl) carbamate S-14.** To a flame-dried, 50 mL three-neck flask containing activated 3 Å molecular sieves under nitrogen was added aldehyde[6] (1.45 g, 5.8 mmol, 1.0 equiv.) in MeOH (23 mL, 0.25 M). Cyclopropylamine (0.42 mL, 6.1 mmol, 1.05 equiv.) was added neat dropwise while stirring at rt. The reaction mixture stirred at rt for 3 h and then cooled to 0 °C in ice bath for 20 min. $NaBH_4$ was slowly added, and the resulting reaction mixture was stirred at rt for 14 h under nitrogen. The reaction mixture was diluted with CH_2Cl_2 (15 mL) and then filtered through celite. The collected filtrate was concentrated *in vacuo*. Crude mixture was diluted with CH_2Cl_2 (20 mL), washed 2 X with saturated brine solution (20 mL). Organic layer was dried over $MgSO_4$ and concentrated in vacuo. Purified by normal phase chromatography (10-50% EtOAc/hexane) to yield diamine **S-14** as a clear oil (772 mg, 46%). ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.37 – 7.09 (m, 5H), 4.46 (s, 2H), 3.31 (d, *J* = 26.3 Hz, 2H), 2.80 (s, 2H), 2.10 (s, 1H), 1.56 – 1.34 (m, 9H), 0.40 (td, *J* = 6.5, 4.3 Hz, 2H), 0.30 – 0.22 (m, 2H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 156.0, 138.5, 128.7, 128.5, 127.7, 127.1, 79.8, 47.5, 46.7, 30.1, 28.4, 6.4.



S-15

***tert*-Butyl 2-((methylamino)methyl)pyrrolidine-1-carboxylate S-15.** Obtained using literature procedure[7] as a clear oil (844 mg, 95%). Characterization matched literature reference.[8]

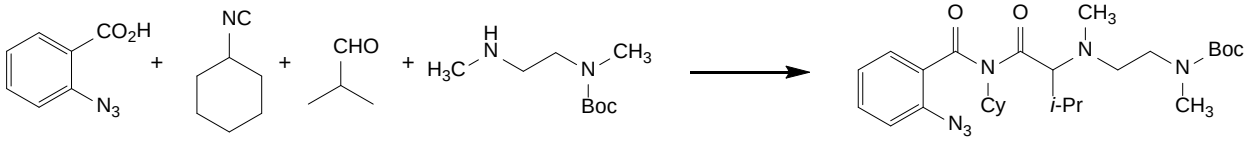
1.3 Ugi-Mumm Imide (10) Optimization Table S1



Entry	stoichiometry (D:A:Ac:I)	Conditions of premixing D and A or not before addition of Ac and I	Final Reaction Conc	Solvent	Isocyanide (I)	Aldehyde (A)	Imide Yield	Acid SM Consumed	Passerini
					R^1	R^2			
1	(1:1:1:1)	no premix	0.1 M	CH_2Cl_2	4-MeO-phenyl	i-Pr	58%	N	N
2	(1:1:1:1)	no premix	0.1 M	CH_2Cl_2:CH_3OH	4-MeO-phenyl	i-Pr	68%	N	N
3	(1.5:1.5:1:1)	no premix	0.1 M	CH_2Cl_2:CH_3OH	4-MeO-phenyl	i-Pr	73%	N	2% (LCMS)
4	(1.5:1.5:1:1.5)	no premix	0.1 M	CH_2Cl_2:CH_3OH	4-MeO-phenyl	i-Pr	85%	N	7% (LCMS)
5	(1.5:1.5:1:1.5)	no premix	0.2 M	CH_2Cl_2:CH_3OH	4-MeO-phenyl	i-Pr	82%	N	N
6	(1.5:1.5:1:1.5)	premix 9 min	0.2 M	CH_2Cl_2:CH_3OH	4-MeO-phenyl	i-Pr	91%	N	N
7	(2:4:1:4)	no premix	0.2 M	CH_2Cl_2:CH_3OH	4-MeO-phenyl	i-Pr	85%	Y	N
8	(1:1:1:1)	no premix	0.1 M	CH_2Cl_2	cyclohexyl	i-Bu	55%	N	N
9	(2:2:1:2)	premix 8 min	0.2 M	CH_2Cl_2:CH_3OH	cyclohexyl	i-Bu	60%	N	N
10	(2:3:1:3)	premix 6 min	0.2 M	CH_2Cl_2:CH_3OH	cyclohexyl	i-Bu	80%	N	N
11	(2:4:1:4)	premix 6 min	0.2 M	CH_2Cl_2:CH_3OH	cyclohexyl	i-Bu	98%	Y	N
12	(1:1:1:1)	no premix	0.1 M	CH_2Cl_2	4-MeO-phenyl	i-Bu	61%	N	
13	(2:2:1:2)	premix 10 min	0.2 M	CH_2Cl_2:CH_3OH	4-MeO-phenyl	i-Bu	62%	Y	N
14	(2:3:1:3)	premix 17 min	0.2 M	CH_2Cl_2:CH_3OH	4-MeO-phenyl	i-Bu	65%	Y	N
15	(2:4:1:4)	premix 12 min	0.2 M	CH_2Cl_2:CH_3OH	4-MeO-phenyl	i-Bu	78%	Y	N
16	(1:1:1:1)	no premix	0.1 M	CH_2Cl_2	cyclohexyl	i-Pr	83%	N	N
17	(2:4:1:4)	premix 10 min	0.2 M	CH_2Cl_2:CH_3OH	cyclohexyl	i-Pr	89%	Y	N
18	(1:1:1:1)	no premix	0.1 M	CH_2Cl_2	cyclohexyl	phenyl	86%	Y	N
19	(2:4:1:4)	premix 6 min	0.2 M	CH_2Cl_2:CH_3OH	cyclohexyl	phenyl	97%	Trace	N
20	(1:1:1:1)	no premix	0.1 M	CH_2Cl_2	cyclohexyl	H	0%	N	30 % Isolated
21	(2:4:1:4)	premix 10 min	0.2 M	CH_2Cl_2	cyclohexyl	H	45%	N	N

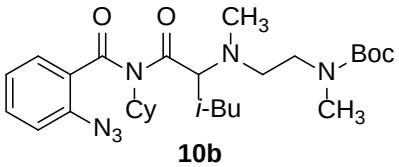
D = diamine; A = aldehyde; Ac = Acid; I = isocyanide; SM = starting material

1.4 General procedure B: Representative Procedure for the Synthesis of Imides:



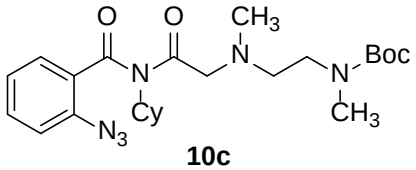
1 **2** **3** **4** **10a**

***tert*-Butyl (2-((1-(2-azido-*N*-cyclohexylbenzamido)-3-methyl-1-oxobutan-2-yl)(methyl)amino) ethyl)(methyl)carbamate 10a.** To a flame-dried, 25 mL three-necked round bottom flask under nitrogen was added activated, powdered, 4Å molecular sieves (50 mg) followed by amine, **4** (115 mg, 0.61 mmol, 2.0 equiv.) in dry CH_2Cl_2 (0.3 mL, 0.1 M). A solution of aldehyde, **3** (112 µL, 1.2 mmol, 4.0 equiv.) in dry CH_2Cl_2 (0.3 mL, 0.1M) was added with stirring followed by carboxylic acid, **1** (50 mg, 0.31 mmol, 1.0 equiv.) in 3:1 dry CH_2Cl_2/MeOH (0.4 mL, 0.1 M) and finally isocyanide, **2** (152 µL, 1.2 mmol, 4.0 equiv.) in dry CH_2Cl_2 (0.4 mL, 0.1 M). The reaction mixture was stirred at rt for 12 h after which the reaction was complete as judged by complete consumption of carboxylic acid on TLC (20% MeOH/CH_2Cl_2). The crude reaction mixture was filtered through a pad of celite and the filtrate was concentrated *in vacuo.* The crude residue was purified by normal-phase chromatography (0-20% EtOAc/hexanes) to yield the imide, **10a** (141 mg, 89%) as pale yellow oil. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.56 – 7.48 (m, 1H), 7.38 (d, *J* = 7.6 Hz, 1H), 7.25 (d, *J* = 8.1 Hz, 1H), 7.22 – 7.16 (m, 1H), 3.95 (s, 1H), 3.28 – 3.12 (m, 3H), 2.83 (d, *J* = 8.8 Hz, 4H), 2.73 – 2.52 (m, 1H), 2.35 (d, *J* = 7.7 Hz, 3H), 2.31 – 2.20 (m, 1H), 2.06 (ddt, *J* = 13.3, 10.1, 6.7 Hz, 2H), 1.88 – 1.68 (m, 5H), 1.62 – 1.53 (m, 1H), 1.44 (s, 8H), 1.30 – 1.05 (m, 3H), 0.82 (d, *J* = 6.6 Hz, 3H), 0.68 (d, *J* = 6.6 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 175.4, 172.1, 155.7, 138.0, 132.3, 129.5, 125.0, 119.2, 79.2, 74.4, 60.3, 52.2, 52.0, 47.5, 47.0, 37.9, 37.1, 34.5, 32.3, 29.5, 28.4, 27.6, 26.6, 26.4, 25.2, 19.7, 19.5. HRMS (ESI): Calculated for $C_{27}H_{42}N_6O_4$ (M^++H): 515.33403; Found: 515.33594.



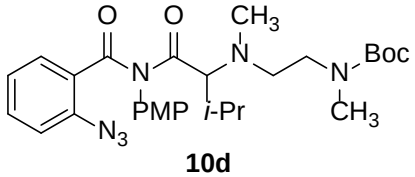
10b

***tert*-Butyl (2-((1-(2-azido-*N*-cyclohexylbenzamido)-4-methyl-1-oxopentan-2-yl)(methyl)amino) ethyl)(methyl)carbamate 10b.** Obtained using **general procedure B**. Purified by normal-phase chromatography (0-10% EtOAc/hexanes) to yield the imide, **10b** (158 mg, 98%) as a pale-yellow oil. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.56 – 7.49 (m, 1H), 7.45 (d, *J* = 7.6 Hz, 1H), 7.25 (d, *J* = 8.3 Hz, 1H), 7.20 (td, *J* = 7.5, 1.0 Hz, 1H), 4.18 (s, 1H), 3.33 – 3.15 (m, 1H), 2.81 (s, 3H), 2.60 – 2.42 (m, 2H), 2.23 – 2.08 (m, 4H), 2.00 (q, *J* = 12.2 Hz, 1H), 1.80 (t, *J* = 8.5 Hz, 4H), 1.70 (d, *J* = 12.1 Hz, 1H), 1.64 – 1.49 (m, 2H), 1.43 (s, 9H), 1.36 (dt, *J* = 13.8, 6.5 Hz, 1H), 1.31 – 1.15 (m, 4H), 0.73 (d, *J* = 6.6 Hz, 3H), 0.66 (d, *J* = 6.6 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 176.6, 171.0, 155.6, 138.5, 132.6, 130.2, 129.0, 124.8, 119.4, 79.2, 66.4, 59.0, 50.8, 47.0, 46.5, 37.9, 35.0, 34.7, 34.5, 31.8, 29.5, 28.4, 26.5, 26.4, 25.3, 24.4, 22.7, 21.8. HRMS (ESI): Calculated for $C_{28}H_{44}N_6O_4$ (M^++H): 529.34968; Found: 529.35162.



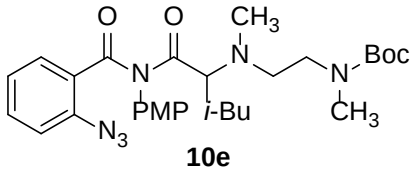
10c

***tert*-Butyl (2-((2-(2-azido-*N*-cyclohexylbenzamido)-2-oxoethyl)(methyl)amino)ethyl)(methyl) carbamate 10c.** Obtained using **general procedure B**. Purified by normal-phase chromatography (0-20% EtOAc/hexanes) to yield the imide, **10c** (65 mg, 45%) as a pale-yellow oil. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.53 – 7.45 (m, 2H), 7.26 – 7.21 (m, 1H), 7.19 (td, *J* = 7.6, 1.1 Hz, 1H), 4.24 (s, 1H), 3.24 (d, *J* = 13.3 Hz, 2H), 3.03 (s, 2H), 2.81 (s, 3H), 2.41 – 2.31 (m, 2H), 2.06 (dd, *J* = 12.6, 4.0 Hz, 2H), 2.02 (s, 4H), 1.85 – 1.71 (m, 4H), 1.62 (dt, *J* = 13.5, 3.5 Hz, 1H), 1.43 (s, 9H), 1.36 – 1.09 (m, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 173.9, 170.2, 155.4, 138.7, 132.4, 130.4, 128.5, 124.5, 119.6, 79.5, 63.0, 58.2, 53.4, 52.8, 45.6, 45.5, 40.7, 34.6, 30.3, 28.4, 26.4, 25.3. HRMS (ESI): Calculated for $C_{24}H_{36}N_6O_4$ (M^++H): 473.28708; Found: 473.28737.



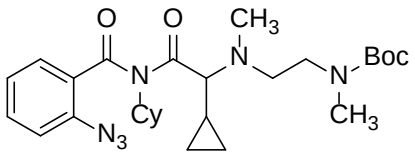
10d

***tert*-Butyl (2-((1-(2-azido-*N*-(4-methoxyphenyl)benzamido)-3-methyl-1-oxobutan-2-yl) (methyl)amino)ethyl)(methyl)carbamate 10d**. Obtained using **general procedure B** . Purified by normal-phase chromatography (0-15% EtOAc/hexanes) to yield the imide, **10d** (140 mg, 85%) as a pale-yellow oil. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.38 – 7.28 (m, 2H), 7.14 – 7.03 (m, 4H), 6.84 (d, *J* = 8.5 Hz, 2H), 3.77 (s, 4H), 3.51 – 3.16 (m, 2H), 2.83 (d, *J* = 12.5 Hz, 4H), 2.42 (s, 3H), 2.15 (dp, *J* = 10.4, 6.6 Hz, 1H), 1.45 (s, 10H), 0.99 (d, *J* = 6.9 Hz, 3H), 0.94 (d, *J* = 6.6 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 175.0, 170.6, 159.4, 155.7, 136.4, 131.0, 130.8, 130.3, 129.4, 128.5, 124.6, 118.5, 114.3, 79.3, 72.36, 55.4, 52.0, 51.6, 47.5, 47.0, 38.2, 38.0, 34.6, 28.5, 28.2, 28.0, 20.1, 19.6. HRMS (ESI): Calculated for $C_{28}H_{38}N_6O_5$ (M^++H): 539.29765; Found: 539.29880.



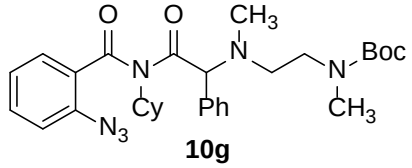
10e

***tert*-Butyl (2-((1-(2-azido-*N*-(4-methoxyphenyl)benzamido)-4-methyl-1-oxopentan-2-yl) (methyl)amino)ethyl)(methyl)carbamate 10e.** Obtained using **general procedure B**. Purified by normal-phase chromatography (0-15% EtOAc/hexanes) to yield the imide, **10e** (132 mg, 78%) as a pale-yellow oil . ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.36 (td, *J* = 7.8, 1.5 Hz, 2H), 7.21 – 7.04 (m, 4H), 6.94 – 6.76 (m, 2H), 4.05 – 3.85 (m, 1H), 3.77 (s, 3H), 3.44 – 3.09 (m, 2H), 2.81 (d, *J* = 5.4 Hz, 3H), 2.75 – 2.56 (m, 2H), 2.35 (s, 3H), 1.63 (dd, *J* = 14.0, 7.4 Hz, 2H), 1.44 (s, 9H), 0.89 (d, *J* = 5.7 Hz, 3H), 0.80 (d, *J* = 6.0 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 176.8, 170.5, 159.4, 155.6, 136.5, 131.2, 130.8, 130.0, 129.2, 128.7, 124.7, 118.4, 114.4, 79.2, 64.4, 55.4, 51.4, 47.5, 47.0, 38.1, 37.7, 36.1, 35.5, 34.6, 28.5, 24.5, 23.0, 21.8. HRMS (ESI): Calculated for $C_{29}H_{40}N_6O_5$ (M^++H): 553.31330; Found: 553.31416.



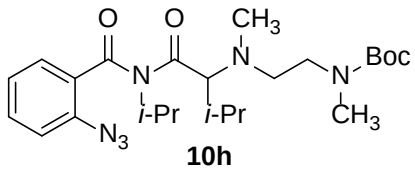
10f

***tert*-Butyl (2-((2-(2-azido-*N*-cyclohexylbenzamido)-1-cyclopropyl-2-oxoethyl)(methyl)amino) ethyl)(methyl)carbamate 10f.** Obtained using **general procedure B**. Purified by normal-phase chromatography (0-50% EtOAc/hexanes) to yield the imide, **10f** (140 mg, 89%) as a yellow oil. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.50 (t, *J* = 7.8 Hz, 1H), 7.45 (d, *J* = 7.5 Hz, 1H), 7.23 (d, *J* = 8.1 Hz, 1H), 7.18 (t, *J* = 7.6 Hz, 1H), 4.24 – 4.05 (m, 1H), 3.38 – 3.09 (m, 1H), 2.83 (s, 3H), 2.71 – 2.52 (m, 2H), 2.45 (dd, *J* = 30.8, 8.5 Hz, 1H), 2.17 (s, 4H), 2.02 (d, *J* = 11.9 Hz, 1H), 1.90 – 1.66 (m, 5H), 1.68 – 1.54 (m, 1H), 1.44 (s, 9H), 1.31 – 1.11 (m, 3H), 1.00 (dddd, *J* = 13.1, 9.7, 8.1, 5.0 Hz, 1H), 0.56 (d, *J* = 8.1 Hz, 2H), 0.14 (d, *J* = 10.4 Hz, 1H). ^{13}C NMR (101 MHz, $CDCl_3$) δ ^{13}C NMR (101 MHz, $CDCl_3$) δ 176.3, 176.0, 170.8, 155.6, 138.5, 132.5, 130.2, 128.7, 124.7, 119.3, 79.2, 73.6, 59.0, 51.2, 50.5, 46.9, 46.5, 38.7, 34.8, 34.6, 31.1, 29.7, 28.4, 26.5, 26.4, 25.3, 8.6, 8.1, 4.4, 2.7. HRMS (ESI): Calculated for $C_{27}H_{40}N_6O_4$ (M^++H): 513.31838; Found: 513.31920.



10g

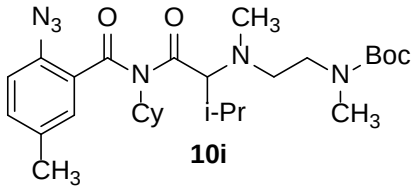
***tert*-Butyl (2-((2-(2-azido-*N*-cyclohexylbenzamido)-2-oxo-1-phenylethyl)(methyl)amino)ethyl) (methyl)carbamate 10g.** Obtained using **general procedure B**. Purified by normal-phase chromatography (0-20% EtOAc/hexanes) to yield the imide, **10g** (163 mg, 97%) as a yellow oil. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.41 (td, *J* = 7.8, 1.6 Hz, 1H), 7.37 – 7.27 (m, 1H), 7.26 – 7.18 (m, 5H), 7.12 – 7.04 (m, 2H), 4.49 (s, 1H), 3.86 (d, *J* = 13.3 Hz, 1H), 3.36 – 3.05 (m, 2H), 2.66 (s, 3H), 2.45 (t, *J* = 7.0 Hz, 2H), 2.16 (d, *J* = 7.0 Hz, 3H), 2.11 – 1.80 (m, 2H), 1.76 – 1.58 (m, 3H), 1.52 , 1.47 – 1.31 (m, 13H), 1.15 – 1.04 (m, 2H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 175.8, 171.2, 155.6, 138.0, 135.5, 132.3, 129.7, 129.6, 129.5, 128.8, 128.1, 124.8, 119.0, 79.1, 74.4, 59.6, 51.3, 46.8, 46.2, 39.6, 39.4, 34.4, 30.6, 29.6, 28.4, 26.4, 26.3, 25.2. HRMS (ESI): Calculated for $C_{30}H_{40}N_6O_4$ (M^++H): 549.31838; Found: 549.31889.



10h

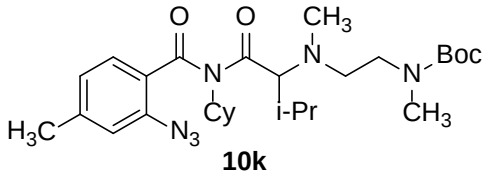
***tert*-Butyl (2-((1-(2-azido-*N*-isopropylbenzamido)-3-methyl-1-oxobutan-2-yl)(methyl)amino) ethyl)(methyl)carbamate 10h.** Obtained using **general procedure B**. Purified by normal-phase chromatography (0-40% EtOAc/hexanes) to yield the imide, **10h** (128 mg, 88%) as a yellow oil. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.57 – 7.47 (m, 1H), 7.38 (d, *J* = 7.6 Hz, 1H), 7.25 (d, *J* = 8.2 Hz, 1H), 7.21 (t, *J* = 7.6 Hz, 1H), 4.26 (d, *J* = 13.7 Hz, 1H), 3.39 (dd, *J* = 20.1, 10.1 Hz, 2H), 3.21 (ddd, *J* = 13.8, 7.6, 5.9 Hz, 1H), 2.84 (d, *J* = 8.8 Hz, 4H), 2.65 (dd, *J* = 35.8, 11.9 Hz, 1H), 2.38 (d, *J* = 5.5 Hz, 3H), 2.08 (dq, *J* = 10.1, 6.6 Hz, 1H), 1.45 (d, *J* = 5.3 Hz, 12H), 1.40 (d, *J* = 6.8 Hz, 3H),

0.85 (d, *J* = 6.6 Hz, 3H), 0.76 (d, *J* = 6.9 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 175.8, 171.9, 155.7, 137.8, 132.1, 129.2, 129.1, 125.0, 119.1, 79.2, 74.0, 52.1, 52.0, 47.6, 47.0, 37.8, 37.1, 34.5, 28.4, 27.6, 22.0, 19.9, 19.8, 19.6. HRMS (ESI): Calculated for $C_{24}H_{38}N_6O_4$ (M^++H): 475.30273; Found: 475.30264.



10i

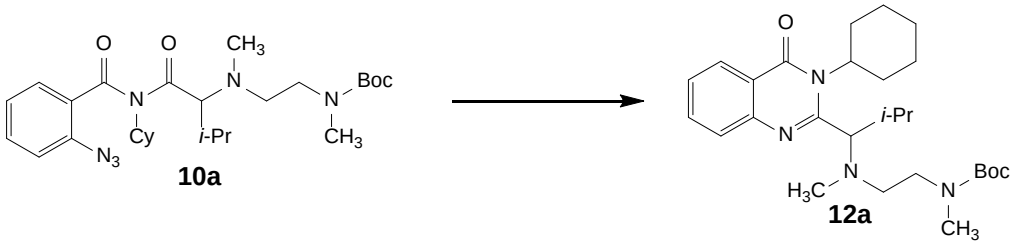
***tert*-Butyl (2-((1-(2-azido-*N*-cyclohexyl-5-methylbenzamido)-3-methyl-1-oxobutan-2-yl) (methyl)amino)ethyl)(methyl)carbamate 10i.** Obtained using **general procedure B**. Purified by normal-phase chromatography (0-40% EtOAc/hexanes) to yield the imide, **10i** (175 mg, 78%) as yellow oil. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.32 (dd, *J* = 8.2, 2.0 Hz, 1H), 7.20 – 7.04 (m, 2H), 4.04 (t, *J* = 11.7 Hz, 1H), 3.48 – 3.23 (m, 1H), 3.21 – 3.07 (m, 2H), 2.83 (d, *J* = 9.8 Hz, 4H), 2.74 – 2.47 (m, 1H), 2.34 (d, *J* = 8.9 Hz, 6H), 2.30 – 2.19 (m, 1H), 2.11 – 1.94 (m, 2H), 1.78 (dtd, *J* = 22.0, 12.5, 12.0, 3.6 Hz, 5H), 1.60 (dd, *J* = 7.1, 3.5 Hz, 1H), 1.44 (s, 9H), 1.32 – 1.10 (m, 3H), 0.79 (d, *J* = 6.6 Hz, 3H), 0.63 (d, *J* = 6.9 Hz, 3H).^{13}C NMR (101 MHz, $CDCl_3$) δ 175.2, 172.1, 155.6, 135.3, 135.0, 133.0, 130.2, 129.3, 119.1, 79.2, 74.7, 60.0, 52.0, 47.5, 46.9, 37.8, 36.9, 34.4, 32.4, 29.4, 28.4, 27.5, 26.6, 26.4, 25.2, 20.7, 19.6, 19.5. HRMS (ESI): Calculated for $C_{28}H_{44}N_6O_4$ (M^++H): 529.34968; Found: 529.35027.



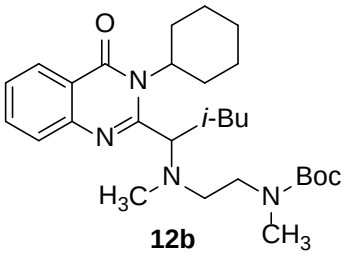
10k

***tert*-Butyl (2-((1-(2-azido-*N*-cyclohexyl-4-methylbenzamido)-3-methyl-1-oxobutan-2-yl) (methyl)amino)ethyl)(methyl)carbamate 10k.** Obtained using **general procedure B**. Purified by normal-phase chromatography (0-40% EtOAc/hexanes) to yield the imide, **10k** (143 mg, 67%) as a yellow oil. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.25 (d, *J* = 6.5 Hz, 2H), 7.00 (s, 1H), 6.96 (d, *J* = 7.9 Hz, 1H), 3.98 (d, *J* = 12.4 Hz, 1H), 3.26 – 3.09 (m, 3H), 2.80 (d, *J* = 8.3 Hz, 4H), 2.39 (s, 3H), 2.31 (s, 3H), 2.21 (dt, *J* = 12.3, 5.9 Hz, 1H), 2.02 (ddd, *J* = 13.6, 10.3, 6.8 Hz, 2H), 1.82 – 1.67 (m, 4H), 1.41 (s, 9H), 1.27 – 1.08 (m, 4H), 0.78 (d, *J* = 6.6 Hz, 3H), 0.65 (d, *J* = 6.7 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 175.31, 172.41, 155.75, 143.64, 138.20, 129.94, 126.89, 125.87, 119.76, 79.32, 74.48, 60.19, 52.04, 47.56, 38.03, 34.53, 32.41, 29.59, 28.51, 27.64, 26.66, 26.51, 25.32, 21.61, 19.89, 19.65. HRMS (ESI): Calculated for $C_{28}H_{44}N_6O_4$ (M^++H): 529.34968; Found: 529.35153.

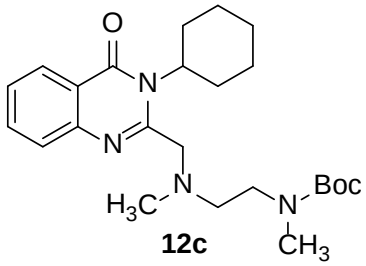
1.5 General procedure C: Representative Procedure for the Synthesis of Quinazolinones:



***tert*-Butyl (2-((1-(3-cyclohexyl-4-oxo-3,4-dihydroquinazolin-2-yl)-2-methylpropyl)(methyl) amino)ethyl)(methyl)carbamate 12a**. To a flame-dried, 25 mL, three-necked round bottom flask equipped with a reflux condenser, under nitrogen was added triphenylphosphine (101 mg, 0.38 mmol, 1.5 equiv.) followed by dry toluene (1.0 mL). A solution of imide **10a** (132 mg, 0.26 mmol, 1.0 equiv.) in dry toluene (1.6 mL) was added and the reaction mixture was stirred at rt for 30 min. Next, the reaction mixture was heated to 110 °C and stirred at reflux for 12h, after which the reaction was complete as judged by complete consumption of imide **10a** on TLC (30% EtOAc/hexanes). The crude reaction mixture was filtered through a pad of celite and the filtrate was concentrated *in vacuo.* The crude residue was purified by normal-phase chromatography (0-50% EtOAc/hexanes) to yield the quinazolinone, **12a** (92 mg, 76%) as clear colorless oil. ^{1}H NMR (400 MHz, Chloroform-d) δ 8.21 (dd, J = 8.0, 1.4 Hz, 1H), 7.67 (t, J = 7.7 Hz, 1H), 7.58 (d, J = 8.1 Hz, 1H), 7.40 (t, J = 7.5 Hz, 1H), 4.19 (t, J = 12.1 Hz, 1H), 3.51 – 3.45 (m, 2H), 3.15 (d, J = 38.2 Hz, 2H), 2.90 (dd, J = 23.8, 11.6 Hz, 2H), 2.77 (s, 3H), 2.67 (s, 1H), 2.58 (d, J = 12.9 Hz, 3H), 1.94 (d, J = 9.5 Hz, 2H), 1.77 – 1.60 (m, 4H), 1.40 (s, 10H), 1.36 (s, 1H), 1.21 (t, J = 7.0 Hz, 1H), 1.12 (d, J = 6.7 Hz, 3H), 0.79 (d, J = 6.4 Hz, 3H). HRMS (ESI): Calculated for $C_{27}H_{42}N_4O_3$ (M^++H): 471.33297; Found: 471.33369.

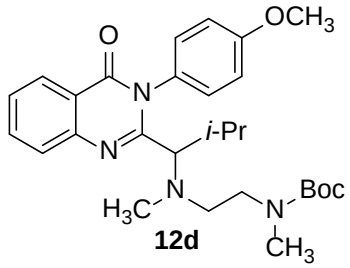


***tert*-Butyl (2-((1-(3-cyclohexyl-4-oxo-3,4-dihydroquinazolin-2-yl)-3-methylbutyl)(methyl) amino)ethyl)(methyl)carbamate 12b.** Obtained using **general procedure C**. Purified by normal-phase chromatography (0-20% EtOAc/hexanes) to yield the quinazolinone, **12b** (81 mg, 60%) as yellow oil. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.20 (d, *J* = 7.9 Hz, 1H), 7.66 (t, *J* = 7.5 Hz, 1H), 7.61 (d, *J* = 8.1 Hz, 1H), 7.40 (t, *J* = 7.4 Hz, 1H), 4.35 (s, 1H), 3.77 – 3.69 (m, 1H), 3.65 (t, *J* = 7.1 Hz, 0H), 3.54 (s, 1H), 3.42 – 3.06 (m, 3H), 2.84 (d, *J* = 10.7 Hz, 3H), 2.76 – 2.60 (m, 5H), 2.52 – 2.31 (m, 5H), 2.26 (s, 1H), 1.91 (d, *J* = 11.0 Hz, 3H), 1.81 – 1.54 (m, 5H), 1.44 (s, 5H), 1.40 – 1.22 (m, 14H), 0.92 (d, *J* = 6.2 Hz, 3H), 0.87 (t, *J* = 7.1 Hz, 6H).^{13}C NMR (101 MHz, $CDCl_3$) δ 163.4, 155.5, 154.5, 146.2, 133.6, 127.3, 126.5, 126.2, 122.2, 79.2, 64.0, 58.9, 58.3, 56.2, 51.2, 49.5, 47.7, 47.1, 38.9, 35.3, 34.6, 29.7, 29.2, 28.5, 28.3, 27.0, 25.8, 25.3, 24.8, 24.8, 23.7, 22.9, 22.8, 21.8. HRMS (ESI): Calculated for $C_{28}H_{44}N_4O_3$ (M^++H): 485.34862; Found: 485.34931.



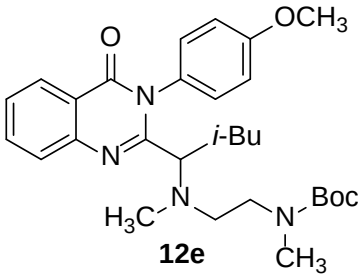
12c

***tert*-Butyl (2-(((3-cyclohexyl-4-oxo-3,4-dihydroquinazolin-2-yl)methyl)(methyl)amino)ethyl) (methyl)carbamate 12c** Obtained using **general procedure C**. Purified by normal-phase chromatography Purified by normal-phase chromatography (0-5% MeOH/CH_2Cl_2) to yield the quinazolinone, **12c** (29 mg, 89%) as a yellow oil.. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.23 (d, *J* = 7.9 Hz, 1H), 7.70 (t, *J* = 7.6 Hz, 1H), 7.63 (d, *J* = 8.1 Hz, 1H), 7.44 (t, *J* = 7.5 Hz, 1H), 4.57 – 4.30 (m, 1H), 3.68 (s, 2H), 3.42 – 3.26 (m, 2H), 2.83 (d, *J* = 8.1 Hz, 3H), 2.76 (dd, *J* = 12.0, 3.6 Hz, 1H), 2.68 (d, *J* = 7.2 Hz, 3H), 2.38 (s, 3H), 1.98 – 1.87 (m, 2H), 1.82 – 1.62 (m, 3H), 1.39 (d, *J* = 29.0 Hz, 14H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 162.8, 155.5, 153.9, 146.5, 133.8, 126.9, 126.8, 126.4, 122.5, 79.4, 77.4, 63.9, 60.4, 54.5, 46.8, 46.1, 42.0, 41.9, 34.8, 28.8, 28.4, 26.8, 25.2. HRMS (ESI): Calculated for $C_{24}H_{36}N_4O_3$ (M^++H): 429.28602; Found: 429.28666.



12d

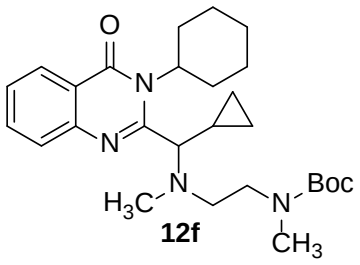
***tert*-Butyl (2-((1-(3-(4-methoxyphenyl)-4-oxo-3,4-dihydroquinazolin-2-yl)-2-methylpropyl) (methyl)amino)ethyl)(methyl)carbamate 12d.** Obtained using **general procedure C**. Purified by normal-phase chromatography (0-15% EtOAc/hexanes) to yield the quinazolinone, **12d** (88mg, 81%) as an off-white foam. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.28 (dd, *J* = 8.0, 1.4 Hz, 1H), 7.74 (t, *J* = 7.6 Hz, 1H), 7.69 (dd, *J* = 8.2, 1.3 Hz, 1H), 7.49 – 7.41 (m, 1H), 7.33 – 7.18 (m, 1H), 7.05 (d, *J* = 7.6 Hz, 3H), 3.88 (s, 3H), 3.20 (d, *J* = 75.7 Hz, 1H), 3.03 (d, *J* = 12.6 Hz, 3H), 2.66 (d, *J* = 69.6 Hz, 5H), 2.34 – 2.24 (m, 3H), 1.40 (s, 9H), 0.93 (d, *J* = 6.7 Hz, 3H), 0.84 (d, *J* = 7.3 Hz, 3H).^{13}C NMR (101 MHz, $CDCl_3$) δ 163.0, 159.8, 156.2, 155.7, 147.1, 134.2, 130.8, 129.9, 129.6, 127.5, 127.0, 126.5, 121.0, 114.6, 79.1, 70.9, 55.5, 50.0, 49.0, 47.6, 46.6, 39.8, 3.4, 29.6, 29.4, 28.4, 20.8, 20.3. HRMS (ESI): Calculated for $C_{28}H_{38}N_4O_4$ (M^++H): 495.29658; Found: 495.29764.



12e

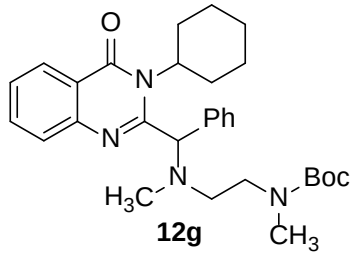
***tert*-Butyl (2-((1-(3-(4-methoxyphenyl)-4-oxo-3,4-dihydroquinazolin-2-yl)-3-methylbutyl) (methyl)amino)ethyl)(methyl)carbamate 12e.** Obtained using **general procedure C**. Purified by normal-phase chromatography (0-15% EtOAc/hexanes) to yield quinazolinone **12e** (86mg, 88%)

as off-white foam. ^{1}H NMR (400 MHz, Chloroform-d) δ 8.37 – 8.10 (m, 1H), 7.74 (qd, *J* = 8.2, 1.6 Hz, 2H), 7.46 (ddd, J = 8.2, 6.4, 1.8 Hz, 1H), 7.41 – 7.26 (m, 1H), 7.15 – 6.95 (m, 3H), 3.87 (s, 3H), 3.42 (s, 1H), 3.19 – 2.75 (m, 2H), 2.61 (d, *J* = 65.4 Hz, 3H), 2.40 – 2.21 (m, 1H), 2.13 (s, 3H), 2.05 (d, *J* = 6.9 Hz, 1H), 1.48 (s, 1H), 1.43 – 1.31 (m, 10H), 0.80 (d, *J* = 6.5 Hz, 3H), 0.77 (d, *J* = 6.2 Hz, 4H). ^{13}C NMR (101 MHz, CDCl3) δ 163.1, 159.8, 147.0, 134.2, 130.9, 129.5, 128.9, 127.7, 127.0, 126.7, 121.2, 114.5, 79.1, 62.4, 55.5, 38.6, 34.2, 34.1, 33.9, 33.2, 28.4, 25.1, 23.1, 22.2. HRMS (ESI): Calculated for $C_{27}H_{42}N_6O_4$ (M^++H): 509.31223; Found: 509.31305.



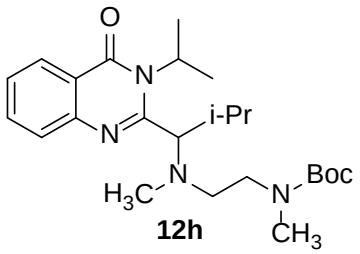
12f

***tert*-Butyl (2-(((3-cyclohexyl-4-oxo-3,4-dihydroquinazolin-2-yl)(cyclopropyl)methyl)(methyl) amino)ethyl)(methyl)carbamate 12f.** Obtained using **general procedure C**. Purified by normal-phase chromatography (0-30% EtOAc/hexanes) to yield the quinazolinone, **12f** (95mg, 70%) as a yellow oil. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.21 (d, *J* = 7.7 Hz, 1H), 7.66 (d, *J* = 7.5 Hz, 1H), 7.59 (d, *J* = 8.1 Hz, 1H), 7.41 (t, *J* = 7.5 Hz, 1H), 3.28 (d, *J* = 26.4 Hz, 2H), 3.14 – 3.01 (m, 1H), 2.91 – 2.68 (m, 6H), 2.52 (s, 1H), 2.44 (s, 3H), 1.88 (t, *J* = 6.0 Hz, 2H), 1.74 – 1.59 (m, 3H), 1.48 – 1.21 (m, 16H), 0.86 (qd, *J* = 11.6, 11.2, 6.2 Hz, 2H), 0.49 (dq, *J* = 9.9, 5.8 Hz, 2H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 161.1, 153.4, 144.4, 131.5, 124.9, 124.4, 124.2, 119.9, 77.2, 56.8, 49.5, 45.2, 44.3, 38.2, 32.5, 27.5, 26.9, 26.5, 26.2, 24.6, 23.2. HRMS (ESI): Calculated for $C_{27}H_{40}N_4O_3$ (M^++H): 469.31732; Found: 469.31814.



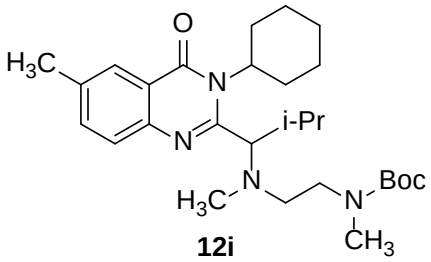
12g

***tert*-Butyl (2-(((3-cyclohexyl-4-oxo-3,4-dihydroquinazolin-2-yl)(phenyl)methyl)(methyl)amino) ethyl)(methyl)carbamate 12g.** Obtained using **general procedure C**. Purified by normal-phase chromatography (0-25% EtOAc/hexanes) to yield the quinazolinone, **12g** (99mg, 82%) as a colorless oil. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.20 (d, *J* = 8.0 Hz, 1H), 7.73 – 7.69 (m, 2H), 7.46 – 7.41 (m, 1H), 7.38 – 7.27 (m, 5H), 5.06 (d, *J* = 32.8 Hz, 1H), 4.36 – 4.05 (m, 1H), 3.69 (s, 1H), 3.36 (d, *J* = 45.0 Hz, 3H), 3.12 – 2.95 (m, 1H), 2.87 (s, 1H), 2.72 (d, *J* = 6.1 Hz, 4H), 2.53 (s, 5H), 2.33 (s, 1H), 1.81 – 1.51 (m, 3H), 1.42 (d, *J* = 21.3 Hz, 13H), 1.20 – 1.11 (m, 2H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 163.2, 155.8, 146.6, 137.4, 133.9, 129.2, 128.8, 128.72, 128.2, 127.5, 126.8, 126.5, 122.3, 79.3, 59.3, 51.8, 47.5, 46.9, 40.7, 34.8, 28.6, 28.3, 26.6, 26.5, 25.2. HRMS (ESI): Calculated for $C_{30}H_{40}N_4O_3$ (M^++H): 505.31732; Found: 505.31802.



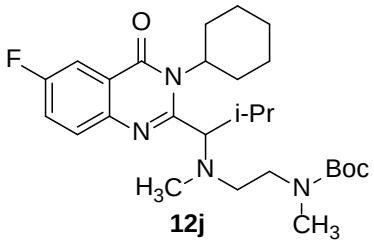
12h

***tert*-Butyl (2-((1-(3-isopropyl-4-oxo-3,4-dihydroquinazolin-2-yl)-2-methylpropyl)(methyl) amino)ethyl)(methyl)carbamate 12h.** Obtained using **general procedure C**. Purified by normal-phase chromatography (0-40% EtOAc/hexanes) to yield the quinazolinone, **12h** (75 mg, 71%) as a colorless oil. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.22 (dd, *J* = 8.0, 1.4 Hz, 1H), 7.73 – 7.64 (m, 1H), 7.59 (dd, *J* = 8.2, 1.2 Hz, 1H), 7.47 – 7.36 (m, 1H), 4.68 (p, *J* = 6.7 Hz, 1H), 3.52 (d, *J* = 10.1 Hz, 1H), 3.24 (dt, *J* = 14.2, 7.2 Hz, 1H), 3.18 – 2.86 (m, 1H), 2.84 – 2.63 (m, 5H), 2.54 (d, *J* = 7.5 Hz, 3H), 1.71 (d, *J* = 6.7 Hz, 3H), 1.65 (d, *J* = 6.8 Hz, 3H), 1.42 (s, 9H), 1.11 (d, *J* = 6.7 Hz, 3H), 0.81 (d, *J* = 6.4 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 163.1, 156.1, 155.7, 146.6, 133.7, 127.2, 126.4, 126.3, 122.0, 79.3, 76.8, 71.5, 71.3, 50.7, 47.8, 47.2, 39.4, 39.1, 34.7, 30.0, 29.8, 28.5, 21.1, 20.6, 19.9. HRMS (ESI): Calculated for $C_{24}H_{38}N_4O_3$ (M^++H): 431.30167; Found: 431.30255.



12i

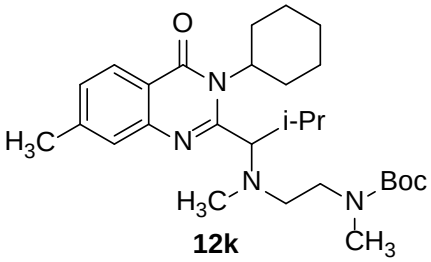
***tert*-Butyl (2-((1-(3-cyclohexyl-6-methyl-4-oxo-3,4-dihydroquinazolin-2-yl)-2-methylpropyl) (methyl)amino)ethyl)(methyl)carbamate 12i.** Obtained using **general procedure C**. Purified by normal-phase chromatography (0-15% EtOAc/hexanes) to yield the quinazolinone, **12i** (114 mg, 69%) as a colorless oil. 1H NMR (400 MHz, Chloroform-d) δ 7.99 (s, 1H), 7.48 (d, J = 1.2 Hz, 2H), 4.17 (t, J = 12.1 Hz, 1H), 3.45 (d, J = 10.1 Hz, 1H), 3.14 (d, J = 45.9 Hz, 2H), 2.95 – 2.53 (m, 12H), 2.45 (s, 3H), 1.93 (d, J = 9.8 Hz, 2H), 1.73 – 1.61 (m, 4H), 1.40 (s, 9H), 1.33 (d, J = 8.6 Hz, 2H), 1.11 (d, J = 6.7 Hz, 3H), 0.78 (d, J = 6.4 Hz, 3H). ^{13}C NMR (101 MHz, CDCl3) δ 163.3, 155.8, 155.4, 144.6, 136.5, 135.2, 127.1, 125.8, 121.8, 79.4, 71.5, 59.5, 40.3, 34.6, 30.2, 29.3, 28.7, 28.6, 27.1, 26.9, 25.4, 21.5, 21.2, 20.7. HRMS (ESI): Calculated for $C_{28}H_{44}N_4O_3$ (M^++H): 485.34862; Found: 485.34952.



12j

tert-butyl (2-((1-(3-cyclohexyl-6-fluoro-4-oxo-3,4-dihydroquinazolin-2-yl)-2-methylpropyl) (methyl)amino)ethyl)(methyl)carbamate 12j. Obtained using crude material from **general procedure B** carried forward in **general procedure C.** Purified by normal-phase chromatography (0-15% EtOAc/hexanes) to yield the quinazolinone, **12j** (176 mg, 84% over both steps) as pale-yellow oil. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.83 (dd, *J* = 8.7, 3.0 Hz, 1H), 7.59 (dd, *J* = 8.9, 4.9

Hz, 1H), 7.39 (td, *J* = 8.5, 3.0 Hz, 1H), 4.19 (t, *J* = 12.0 Hz, 1H), 3.46 (d, *J* = 10.0 Hz, 1H), 3.30 – 3.03 (m, 3H), 3.03 – 2.83 (m, 3H), 2.80 – 2.64 (m, 3H), 2.58 (d, *J* = 9.9 Hz, 3H), 1.94 (d, *J* = 9.8 Hz, 2H), 1.69 (q, *J* = 12.8, 8.9 Hz, 3H), 1.40 (s, 10H), 1.35 (d, *J* = 8.6 Hz, 3H), 1.12 (d, *J* = 6.7 Hz, 3H), 0.79 (d, *J* = 6.3 Hz, 3H). ^{19}F NMR (376 MHz, $CDCl_3$) δ -113.65. ^{13}C NMR (101 MHz, $CDCl_3$) δ 162.3, 161.9, 159.4, 155.6, 155.5, 143.05, 129.5, 129.4, 123.1, 123.0, 122.4, 122.1, 111.1, 110.9, 79.2, 71.5, 59.5, 40.1, 34.5, 30.0, 29.0, 28.4, 26.8, 26.7, 25.2, 21.0, 20.6. HRMS (ESI): Calculated for $C_{27}H_{41}FN_4O_3$ (M^++H): 489.32355; Found: 489.32439.

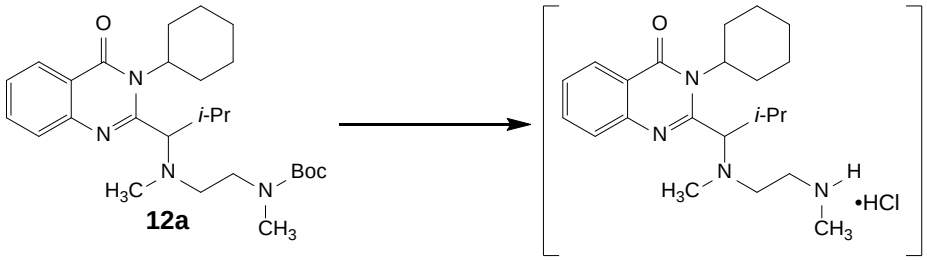


12k

tert-butyl (2-((1-(3-cyclohexyl-7-methyl-4-oxo-3,4-dihydroquinazolin-2-yl)-2-methylpropyl)(methyl)amino)ethyl)(methyl)carbamate 12k. Obtained using **general procedure C**. Purified by normal-phase chromatography (0-20% EtOAc/hexanes) to yield the quinazolinone, **12k** (84 mg, 70%) as a colorless oil, 70%. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.08 (d, *J* = 8.1 Hz, 1H), 7.38 (s, 1H), 7.21 (dd, *J* = 8.2, 1.6 Hz, 1H), 4.15 (q, *J* = 8.2, 4.8 Hz, 1H), 3.46 (d, *J* = 10.0 Hz, 1H), 3.14 (d, *J* = 25.8 Hz, 2H), 3.00 – 2.81 (m, 3H), 2.80 – 2.64 (m, 5H), 2.55 (d, *J* = 13.0 Hz, 3H), 2.45 (s, 3H), 1.96 – 1.87 (m, 2H), 1.68 (dd, *J* = 18.0, 9.6 Hz, 4H), 1.40 (s, 9H), 1.33 (d, *J* = 8.1 Hz, 3H), 1.11 (d, *J* = 6.6 Hz, 3H), 0.77 (d, *J* = 6.4 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 163.2, 156.4, 155.8, 146.6, 144.6, 128.0, 126.9, 126.3, 119.7, 79.3, 71.6, 59.4, 49.8, 47.8, 40.2, 34.6, 30.1, 29.3, 28.8, 28.53, 27.0, 26.9, 25.3, 21.9, 21.1, 20.7. HRMS (ESI): Calculated for $C_{28}H_{44}N_4O_3$ (M^++H): 485.34862; Found: 485.34772.

1.6 General procedure D: General Procedure for the synthesis of Benzamidines:

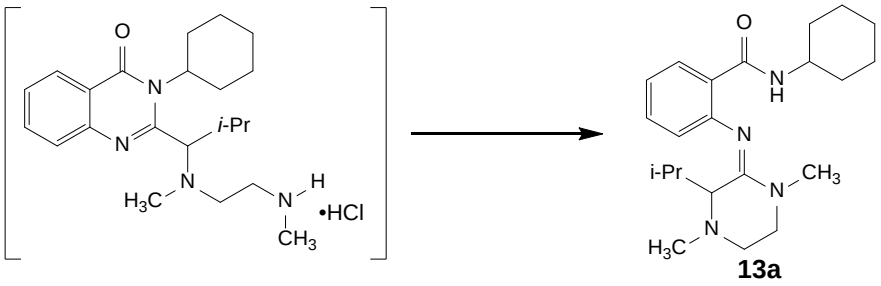
Step 1. Representative Procedure for Boc-deprotection:



12a

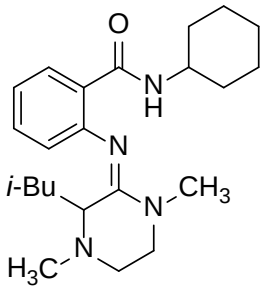
To a flame-dried, 10 mL, one-necked round bottom flask under nitrogen was dissolved quinazolinone (84 mg, 0.18 mmol, 1.0 equiv.) in dry CH_2Cl_2 (1.0 mL) and the solution cooled to 0 °C in an ice-bath with stirring. HCl in dioxane (4M, 0.22 mL, 5.0 equiv.) was added dropwise. Following the addition the ice-bath was removed and the reaction mixture allowed to warm to rt. The reaction mixture was stirred at rt for 12h after which the reaction was complete as judged by complete consumption of quinazolinone on TLC (30% EtOAC/hexanes). The solvent was removed *in vacuo* to yield the HCl salt of boc-deprotected quinazolinone which was carried forward without purification (65 mg, 89%).

Step 2. Representative Procedure for the synthesis of Benzamidines:



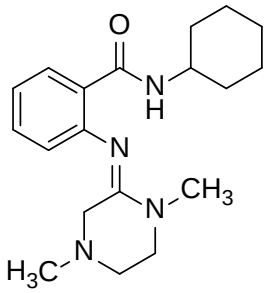
13a

(*E*)-*N*-cyclohexyl-2-((3-isopropyl-1,4-dimethylpiperazin-2-ylidene)amino)benzamide 13a. Triethylamine (100 µL, 0.7 mmol, 5 equiv.) was added to a solution of HCl salt of quinazolinone (55 mg, 0.14 mmol, 1.0 equiv.) in MeOH (1.4 mL, 0.1M) in a 4 mL microwave vial while stirring. The vial was capped and heated in a microwave reactor at 100 °C for 30 min. The reaction mixture was allowed to cool to rt and the solvent removed *in vacuo*. The crude residue was purified by normal-phase chromatography (0-5% MeOH/CH_2Cl_2) to yield the benzamidine, **13a** (37 mg, 66% over two steps) as a yellow oil. ^{1}H NMR (500 MHz, Chloroform-*d*) δ 8.93 (d, *J* = 8.0 Hz, 1H), 8.17 (dd, *J* = 7.9, 1.7 Hz, 1H), 7.28 – 7.24 (m, 1H), 7.05 – 7.01 (m, 1H), 6.59 (dd, *J* = 7.8, 1.2 Hz, 1H), 3.95 (tdt, *J* = 11.5, 7.9, 3.9 Hz, 1H), 3.47 – 3.39 (m, 2H), 3.36 (ddd, *J* = 12.3, 7.1, 4.6 Hz, 1H), 3.23 (ddd, *J* = 11.9, 7.0, 4.4 Hz, 1H), 3.11 (s, 3H), 2.63 (ddd, *J* = 12.0, 6.6, 4.7 Hz, 1H), 2.49 (s, 3H), 2.07 – 1.97 (m, 2H), 1.76 – 1.69 (m, 3H), 1.68 – 1.61 (m, 1H), 1.38 (ddt, *J* = 17.9, 12.8, 6.4 Hz, 2H), 1.21 – 1.10 (m, 3H), 0.79 (d, *J* = 6.7 Hz, 3H), 0.63 (d, *J* = 6.9 Hz, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 165.9, 160.5, 148.5, 131.2, 131.1, 125.8, 122.4, 122.0, 63.9, 48.7, 48.4, 47.8, 45.0, 37.4, 33.8, 33.7, 32.4, 25.9, 25.3, 20.1, 19.2. HRMS (ESI): Calculated for $C_{22}H_{34}N_4O$ (M^++H): 371.28054; Found: 371.28091.



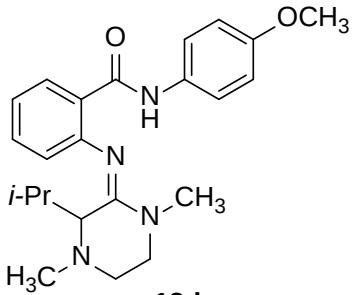
13b

(*E*)-*N*-cyclohexyl-2-((3-isobutyl-1,4-dimethylpiperazin-2-ylidene)amino)benzamide 13b. Obtained using **general procedure D**. Purified by normal-phase chromatography (0-10% MeOH/CH_2Cl_2) to yield the benzamidine, **13b** (20mg, 34%) as a pale-yellow gum. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.56 (d, *J* = 8.1 Hz, 1H), 8.18 (dd, *J* = 7.9, 1.6 Hz, 1H), 7.30 – 7.25 (m, 2H), 7.05 (td, *J* = 7.6, 1.1 Hz, 1H), 6.61 (dd, *J* = 7.8, 1.1 Hz, 1H), 3.97 (dddd, *J* = 14.6, 10.6, 7.9, 3.9 Hz, 1H), 3.61 (td, *J* = 11.8, 5.3 Hz, 1H), 3.49 (dd, *J* = 10.6, 3.7 Hz, 1H), 3.41 (ddd, *J* = 14.0, 11.7, 5.2 Hz, 1H), 3.12 (ddd, *J* = 12.1, 5.3, 1.7 Hz, 1H), 3.08 (s, 3H), 2.73 – 2.66 (m, 1H), 2.50 (s, 3H), 2.02 – 1.94 (m, 2H), 1.71 (dq, *J* = 11.7, 4.0 Hz, 2H), 1.63 (dt, *J* = 12.6, 3.8 Hz, 1H), 1.54 (ddd, *J* = 14.1, 10.6, 3.9 Hz, 1H), 1.49 – 1.34 (m, 3H), 1.16 (dddd, *J* = 26.0, 15.5, 12.6, 5.6 Hz, 3H), 0.91 (ddd, *J* = 13.6, 9.7, 3.6 Hz, 1H), 0.66 (d, *J* = 6.6 Hz, 3H), 0.27 (d, *J* = 6.5 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 165.7, 161.8, 148.6, 131.4, 131.3, 125.6, 122.9, 122.4, 56.8, 48.2, 44.9, 43.8, 42.2, 40.1, 37.3, 33.8, 33.6, 25.9, 25.1, 25.1, 24.4, 23.4, 20.3. HRMS (ESI): Calculated for $C_{23}H_{36}N_4O$ (M^++H): 385.29619; Found: 385.29719.



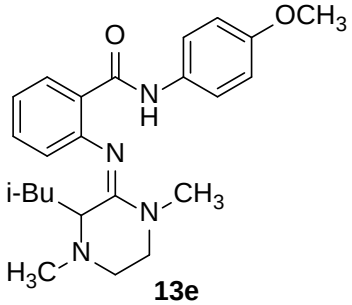
13c

(*E*)-*N*-cyclohexyl-2-((1,4-dimethylpiperazin-2-ylidene)amino)benzamide 13c. Obtained using **general procedure D**. Purified by normal-phase chromatography (0-10% MeOH/CH_2Cl_2) to yield the benzamidine, **13c** (12 mg, 58%) as an amber gum. ^{1}H NMR (500 MHz, Chloroform-*d*) δ 8.42 (d, *J* = 8.0 Hz, 1H), 8.12 (dd, *J* = 7.9, 1.7 Hz, 1H), 7.29 – 7.25 (m, 1H), 7.09 – 7.04 (m, 1H), 6.65 (d, *J* = 7.8 Hz, 1H), 3.94 (tdt, *J* = 11.0, 7.8, 3.8 Hz, 1H), 3.37 (t, *J* = 5.5 Hz, 2H), 3.12 (s, 3H), 2.95 (s, 2H), 2.61 (t, *J* = 5.6 Hz, 2H), 2.19 (s, 3H), 1.99 (dq, *J* = 12.4, 4.0 Hz, 2H), 1.71 (dp, *J* = 11.9, 3.9 Hz, 2H), 1.63 (dt, *J* = 12.2, 3.9 Hz, 1H), 1.40 (tdd, *J* = 15.5, 12.0, 3.6 Hz, 2H), 1.15 (td, *J* = 12.4, 9.1 Hz, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 166.0, 155.5, 148.3, 131.3, 131.2, 126.0, 123.2, 122.7, 55.1, 52.4, 49.7, 48.4, 45.5, 36.5, 33.6, 25.9, 25.1. HRMS (ESI): Calculated for $C_{19}H_{28}N_4O$ (M^++H): 329.23359; Found: 329.23434.



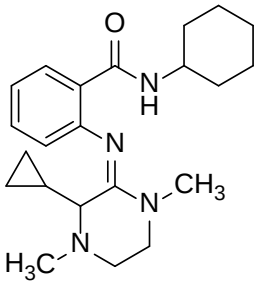
13d

(*E*)-2-((3-isopropyl-1,4-dimethylpiperazin-2-ylidene)amino)-*N*-(4-methoxyphenyl)benzamide 13d. Obtained using **general procedure D**. Purified by normal-phase chromatography (0-10% MeOH/CH_2Cl_2) to yield the benzamidine, **13d** (20mg, 23%) as a pale-yellow waxy solid. 1H NMR (400 MHz, Chloroform-d) δ 10.97 (s, 1H), 8.24 (dd, J = 8.0, 1.7 Hz, 1H), 7.53 (d, J = 8.5 Hz, 2H), 7.36 – 7.29 (m, 1H), 7.09 (t, J = 7.6 Hz, 1H), 6.88 (d, J = 8.7 Hz, 2H), 6.67 (d, J = 7.9 Hz, 1H), 3.79 (d, J = 1.3 Hz, 3H), 3.46 – 3.35 (m, 3H), 3.27 – 3.21 (m, 1H), 3.19 (s, 3H), 2.63 (dt, J = 11.9, 5.7 Hz, 1H), 2.52 (s, 3H), 1.75 (h, J = 6.8 Hz, 1H), 0.77 (d, J = 6.7 Hz, 3H), 0.65 (d, J = 6.8 Hz, 3H). ^{13}C NMR (101 MHz, CDCl3) δ 165.0, 161.0, 156.1, 148.6, 132.0, 131.7, 131.3, 125.9, 122.6, 122.3, 122.0, 114.3, 64.0, 55.6, 48.3, 47.7, 44.9, 37.6, 32.5, 20.0, 19.3. HRMS (ESI): Calculated for $C_{23}H_{30}N_4O_2$ (M^++H): 395.24415; Found: 395.24443.



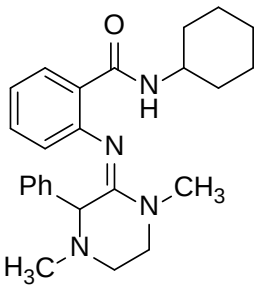
13e

(*E*)-2-((3-isobutyl-1,4-dimethylpiperazin-2-ylidene)amino)-*N*-(4-methoxyphenyl)benzamide 13e. Obtained using **general procedure D**. Purified by normal-phase chromatography (0-20% EtOAc/CH_2Cl_2) to yield the benzamidine, **13e** (26 mg, 39%) as a light-brown solid. m.p.: 109-111°C. ^{1}H NMR (400 MHz, Chloroform-d) δ 10.68 (s, 1H), 8.25 (dd, J = 8.0, 1.7 Hz, 1H), 7.62 – 7.50 (m, 2H), 7.34 (td, J = 7.6, 1.7 Hz, 1H), 7.19 – 7.06 (m, 1H), 6.94 – 6.85 (m, 2H), 6.76 – 6.64 (m, 1H), 3.80 (s, 3H), 3.61 (ddd, J = 12.4, 8.8, 4.4 Hz, 2H), 3.38 (ddd, J = 14.0, 11.6, 5.3 Hz, 1H), 3.18 (s, 3H), 3.11 (ddd, J = 12.1, 5.4, 1.7 Hz, 1H), 2.69 (dd, J = 14.3, 5.1 Hz, 1H), 2.50 (s, 3H), 1.56 (ddd, J = 14.0, 10.6, 3.7 Hz, 1H), 1.47 (dqd, J = 10.4, 6.6, 3.9 Hz, 0H), 0.92 (ddd, J = 13.5, 9.7, 3.5 Hz, 1H), 0.65 (d, J = 6.7 Hz, 3H), 0.28 (d, J = 6.5 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 164.6, 162.2, 155.9, 148.5, 132.1, 131.7, 131.3, 125.6, 123.0, 122.5, 121.6, 114.2, 77.4, 77.0, 76.7, 56.8, 55.5, 44.7, 43.4, 42.1, 39.9, 37.3, 24.4, 23.3, 20.2. HRMS (ESI): Calculated for $C_{24}H_{32}N_4O_2$ (M^++H): 409.25980; Found: 409.25931.



13f

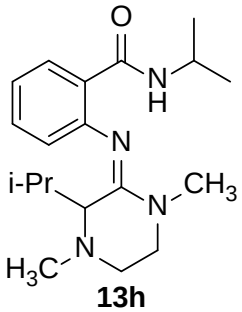
(*E*)-*N*-cyclohexyl-2-((3-cyclopropyl-1,4-dimethylpiperazin-2-ylidene)amino)benzamide 13f. Obtained using **general procedure D**. Purified by normal-phase chromatography (0-5% MeOH/CH_2Cl_2) to yield amidine **13f** (17.5 mg, 65%) as a pale-yellow gum. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.62 (d, *J* = 8.1 Hz, 1H), 8.18 (dt, *J* = 7.9, 1.5 Hz, 1H), 7.28 – 7.21 (m, 1H), 7.09 – 7.01 (m, 1H), 6.56 (d, *J* = 7.8 Hz, 1H), 3.96 (dtt, *J* = 11.2, 7.8, 4.1 Hz, 1H), 3.58 – 3.44 (m, 2H), 3.30 (dd, *J* = 10.3, 4.2 Hz, 1H), 3.09 (s, 3H), 2.92 (d, *J* = 8.6 Hz, 1H), 2.76 (dd, *J* = 11.8, 4.6 Hz, 1H), 2.46 (s, 3H), 1.98 (d, *J* = 12.9 Hz, 2H), 1.78 – 1.62 (m, 4H), 1.41 (qd, *J* = 12.5, 6.0 Hz, 3H), 1.23 – 1.06 (m, 3H), 0.90 (ddt, *J* = 13.8, 8.8, 4.3 Hz, 1H), 0.40 (td, *J* = 8.7, 4.5 Hz, 1H), 0.28 (tt, *J* = 8.6, 5.4 Hz, 1H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 165.8, 160.4, 148.9, 131.3, 131.1, 126.4, 122.6, 122.3, 62.1, 48.3, 47.4, 45.5, 42.3, 37.3, 33.8, 33.7, 25.9, 25.2, 25.2, 11.6, 4.1, 3.7, 1.2. HRMS (ESI): Calculated for $C_{22}H_{32}N_4O$ (M^++H): 369.26489; Found: 369.26570.



13g

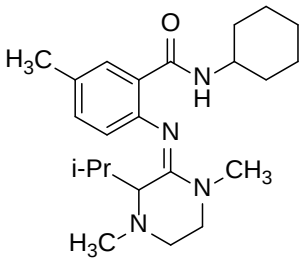
(*E*)-*N*-cyclohexyl-2-((1,4-dimethyl-3-phenylpiperazin-2-ylidene)amino)benzamide 13g. Obtained using **general procedure D**. Purified by normal-phase chromatography (0-10% MeOH/CH_2Cl_2) to yield benzamidine, **13g** (38mg, 70%) as a pale-yellow gum. ^{1}H NMR (500 MHz, CDCl3) δ 8.36 (d, J = 7.7 Hz, 1H), 7.88 (dd, J = 7.9, 1.5 Hz, 1H), 7.18 (t, J = 7.4 Hz, 1H), 7.11 (t, J = 7.5 Hz, 2H), 7.08 – 7.03 (m, 1H), 6.93 – 6.89 (m, 1H), 6.85 (d, J = 7.2 Hz, 2H), 6.37 (d, J = 7.7 Hz, 1H), 4.41 (s, 1H), 3.86-3.83 (m, 1H), 3.57 – 3.51 (m, 2H), 3.24 (s, 3H), 3.03 – 2.95 (m, 1H), 2.62-

2.58 (m, 1H), 2.16 (s, 3H), 2.05 – 1.94 (m, 2H), 1.80 – 1.69 (m, 2H), 1.68-1.584 (m, 1H), 1.46 – 1.34 (m, 2H), 1.23 – 1.11 (m, 3H). ^{13}C NMR (126 MHz, CDCl3) δ 165.3, 165.2, 157.6, 148.1, 135.7, 130.8, 130.6, 129.3, 128.0, 127.9, 125.5, 123.2, 122.1, 65.3, 49.1, 48.3, 46.6, 42.1, 37.4, 33.7, 33.7, 25.9, 25.2, 25.2. HRMS (ESI): Calculated for $C_{25}H_{32}N_4O$ (M^++H): 405.26489; Found: 405.26680.



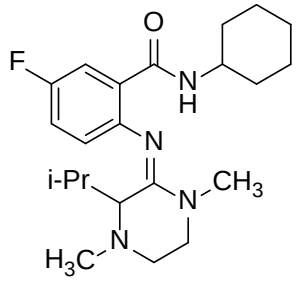
13h

(*E*)-*N*-isopropyl-2-((3-isopropyl-1,4-dimethylpiperazin-2-ylidene)amino)benzamide 13h. Obtained using **general procedure D**. Purified by normal-phase chromatography (0-5% MeOH/CH_2Cl_2) to yield the benzamidine, **13h** (11 mg, 52%) as a pale-yellow oil . ^{1}H NMR (500 MHz, Chloroform-*d*) δ 8.94 (d, *J* = 7.6 Hz, 1H), 8.18 (dd, *J* = 7.9, 1.6 Hz, 1H), 7.28 (dd, *J* = 7.5, 1.7 Hz, 1H), 7.06 – 7.02 (m, 1H), 6.60 (dd, *J* = 7.9, 1.2 Hz, 1H), 4.31 – 4.22 (m, 1H), 3.48 – 3.41 (m, 2H), 3.36 (ddd, *J* = 12.2, 7.0, 4.6 Hz, 1H), 3.24 (ddd, *J* = 11.8, 7.0, 4.4 Hz, 1H), 3.13 (s, 3H), 2.64 (ddd, *J* = 12.0, 6.7, 4.7 Hz, 1H), 2.50 (s, 3H), 1.72 (h, *J* = 6.7 Hz, 1H), 1.22 (d, *J* = 6.5 Hz, 6H), 0.80 (d, *J* = 6.7 Hz, 3H), 0.64 (d, *J* = 6.8 Hz, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 166.0, 160.5, 148.6, 131.3, 131.2, 125.8, 122.4, 122.1, 64.0, 48.8, 47.9, 45.1, 41.3, 37.3, 32.5, 23.3, 23.2, 20.2, 19.3. HRMS (ESI): Calculated for $C_{19}H_{30}N_4O$ (M^++H): 331.24924; Found: 331.24986.



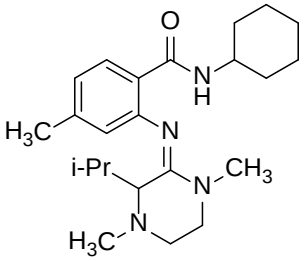
13i

(*E*)-*N*-cyclohexyl-2-((3-isopropyl-1,4-dimethylpiperazin-2-ylidene)amino)-5-methylbenzamide 13i. Obtained using **general procedure D**. Purified by normal-phase chromatography (0-5% MeOH/CH_2Cl_2) to yield the benzamidine, **13d** (41 mg, 64%) as a pale-yellow oil. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.90 (d, *J* = 8.1 Hz, 1H), 7.96 (d, *J* = 2.2 Hz, 1H), 7.06 (dd, *J* = 8.0, 2.2 Hz, 1H), 6.49 (d, *J* = 8.0 Hz, 1H), 3.93 (tdt, *J* = 11.4, 7.8, 3.9 Hz, 1H), 3.48 – 3.36 (m, 2H), 3.34 (ddd, *J* = 12.0, 7.0, 4.6 Hz, 1H), 3.22 (ddd, *J* = 11.8, 7.0, 4.5 Hz, 1H), 3.09 (s, 3H), 2.62 (ddd, *J* = 11.9, 6.6, 4.7 Hz, 1H), 2.48 (s, 3H), 2.30 (s, 3H), 2.07 – 1.94 (m, 3H), 1.73 (ddt, *J* = 11.9, 8.0, 4.5 Hz, 2H), 1.71 – 1.58 (m, 1H), 1.38 (tdt, *J* = 16.5, 12.1, 3.8 Hz, 2H), 1.13 (qdt, *J* = 10.6, 6.7, 3.2 Hz, 3H), 0.79 (d, *J* = 6.7 Hz, 3H), 0.63 (d, *J* = 6.9 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 166.1, 160.6, 146.0, 131.9, 131.5, 125.5, 122.3, 64.0, 48.7, 48.5, 47.9, 45.0, 37.4, 33.8, 33.8, 32.5, 25.9, 25.3, 20.8, 20.1, 19.3. HRMS (ESI): Calculated for $C_{23}H_{36}N_4O$ (M^++H): 385.29619; Found: 385.29605.



13j

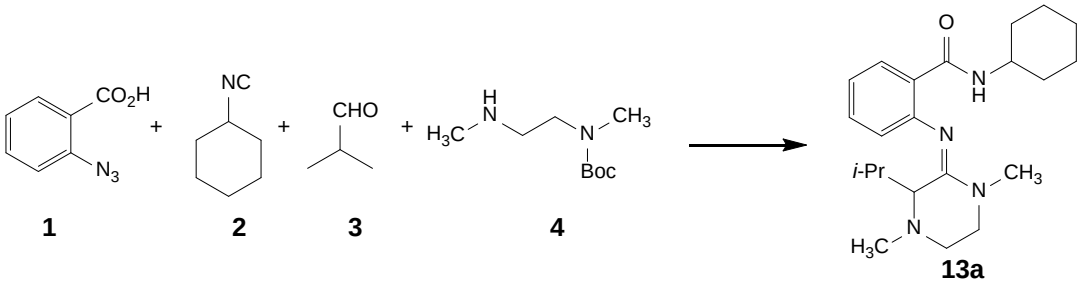
(*E*)-*N*-cyclohexyl-5-fluoro-2-((3-isopropyl-1,4-dimethylpiperazin-2-ylidene)amino)benzamide 13j. Obtained using **general procedure D**. Purified by normal-phase chromatography (0-5% MeOH/CH_2Cl_2) to yield the benzamidine, **13j** (8 mg, 40%) as a pale-yellow gum . ^{1}H NMR (400 MHz, CDCl3) δ 8.99 (d, J = 7.4 Hz, 1H), 7.90 (dd, J = 10.1, 2.9 Hz, 1H), 6.99 (td, J = 8.4, 3.1 Hz, 1H), 6.56 (dd, J = 8.6, 4.9 Hz, 1H), 4.03 – 3.87 (m, 1H), 3.48-3.46 (m, 1H), 3.41 – 3.31 (m, 2H), 3.28-3.22 (m, 1H), 3.13 (s, 3H), 2.70 – 2.60 (m, 1H), 2.50 (s, 3H), 2.10 – 1.95 (m, 2H), 1.80 – 1.59 (m, 4H), 1.51 – 1.35 (m, 2H), 1.24-1.18 (m, 3H), 0.81 (d, J = 6.7 Hz, 3H), 0.65 (d, J = 6.8 Hz, 3H). ^{19}F NMR (376 MHz, CDCl3) δ -122.09. ^{13}C NMR (101 MHz, CDCl3) δ 164.6, 164.6, 160.9, 159.6, 157.2, 149.9, 127.1, 127.1, 123.5, 123.4, 118.1, 117.8, 117.3, 117.1, 63.9, 48.7, 48.6, 47.8, 45.1, 37.3, 33.7, 33.6, 32.5, 25.8, 25.2, 20.0, 19.1. HRMS (ESI): Calculated for $C_{22}H_{33}FN_4O$ (M^++H): 389.27112; Found: 389.27059.



13k

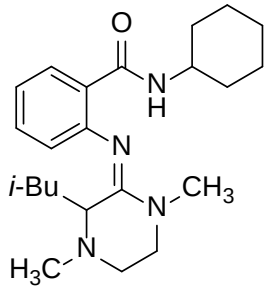
(*E*)-*N*-cyclohexyl-2-((3-isopropyl-1,4-dimethylpiperazin-2-ylidene)amino)-4-methylbenzamide 13k. Obtained using **general procedure D**. Purified by normal-phase chromatography (0-5% MeOH/CH_2Cl_2) to yield the benzamidine, **13k** (30 mg, 61%) as a pale-yellow gum. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.84 (d, *J* = 8.0 Hz, 1H), 8.06 (d, *J* = 8.0 Hz, 1H), 6.84 (d, *J* = 8.0 Hz, 1H), 6.39 (s, 1H), 3.94 (dtd, *J* = 11.3, 7.6, 4.1 Hz, 1H), 3.49 – 3.30 (m, 3H), 3.23 (ddd, *J* = 11.8, 6.9, 4.5 Hz, 1H), 3.10 (s, 3H), 2.63 (ddd, *J* = 12.0, 6.7, 4.7 Hz, 1H), 2.50 (s, 3H), 2.29 (s, 3H), 2.01 (tt, *J* = 8.4, 3.8 Hz, 2H), 1.78 – 1.59 (m, 4H), 1.46 – 1.31 (m, 2H), 1.21 – 1.06 (m, 3H), 0.80 (d, *J* = 6.7 Hz, 3H), 0.64 (d, *J* = 6.8 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 166.0, 160.5, 148.4, 141.3, 131.3, 123.3, 123.1, 123.0, 64.1, 48.8, 48.4, 47.9, 45.01, 37.4, 33.9, 33.8, 32.6, 25.9, 25.3, 21.4, 20.1, 19.4. HRMS (ESI): Calculated for $C_{23}H_{36}N_4O$ (M^++H): 385.29619; Found: 385.29585.

1.7 General procedure E: Representative Procedure for the Telescoped Synthesis of Benzamidines:



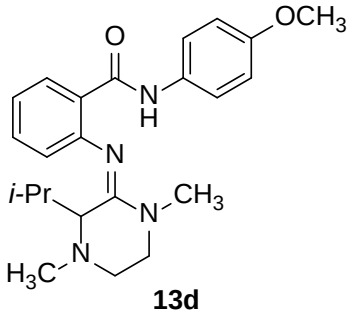
1 **2** **3** **4** **13a**

(*E*)-*N*-cyclohexyl-2-((3-isopropyl-1,4-dimethylpiperazin-2-ylidene)amino)benzamide 13a. An oven-dried, 10 mL round bottom flask under nitrogen was charged with activated, powdered, 4 Å molecular sieves (50 mg). The flask was placed under vacuum and flame-dried, then backfilled with nitrogen. Diamine **4** (115 mg, 0.61 mmol, 2.0 equiv.) was dissolved in dry CH_2Cl_2 (0.5 mL, 1.3 M) and added to the reaction vessel. Aldehyde **3** (112 µL, 1.2 mmol, 4.0 equiv.) was dissolved in dry CH_2Cl_2 (0.46 mL, 2.6 M) in a 1-dram glass vial and added dropwise over 1 min via syringe to the reaction vessel while stirring at rt. Upon completion of the addition, carboxylic acid **1** (50 mg, 0.31 mmol, 1.0 equiv.) was dissolved in a 3:1 mixture of dry CH_2Cl_2/MeOH (0.4 mL, .8 M), taken up into a syringe and the needle was placed into the septum of the reaction vessel. Isocyanide **2** (153 µL, 1.2 mmol, 4.0 equiv.) was dissolved in dry CH_2Cl_2 (0.3 mL, 3.6 M) in a 1-dram glass vial, taken up into a syringe and the needle was placed into the septum of the reaction vessel. Carboxylic acid **1** was added dropwise to the reaction vessel while stirring over 1 min followed immediately by isocyanide **2**. The reaction mixture was stirred at rt for 12 h after which the reaction was complete as judged by complete consumption of carboxylic acid **1** on TLC (10% MeOH/CH_2Cl_2). The crude reaction mixture was filtered through a 0.45 µM syringe filter and the filtrate was concentrated *in vacuo*. To a flame-dried, 10 mL round bottom flask under nitrogen was added triphenylphosphine on resin (438 mg, 0.61 mmol, 1.4 meq/g, 2.0 equiv.) followed by dry toluene (1.0 mL). A solution of the crude reaction mixture in dry toluene (2.0 mL) was added dropwise and the reaction mixture was stirred at rt for 1 h. After 1 h at rt, the reaction mixture was heated to 110 °C and stirred for 12 h at reflux after which the reaction was complete as judged by complete consumption of the intermediate by TLC (30% EtOAc/hexanes). The crude reaction mixture was filtered through a pad of celite, rinsed with CH_2Cl_2 (10 mL x 5), and the filtrate was concentrated *in vacuo*. To a flame-dried, 10 mL round bottom flask under nitrogen was dissolved the crude reaction mixture in dry CH_2Cl_2 (3.0 mL, 0.1 M) and the solution cooled to 0 °C in an ice-bath while stirring for 20 min. HCl in dioxane (4.0 M, 0.8 mL, 10.0 equiv.) was added dropwise. The reaction mixture was allowed to slowly warm to rt. The reaction mixture was stirred at rt for 12h after which the reaction was complete as judged by complete consumption of intermediate on TLC (30% EtOAC/hexanes). The reaction mixture was dried *in vacuo*. Triethylamine (0.4 mL, 3.1 mmol, 10.0 equiv.) was added to a solution of the crude reaction mixture in MeOH (3.1 mL, 0.1 M) in a 10 mL microwave vial and the reaction mixture was heated in a microwave reactor at 100 °C for 1 h. The reaction mixture was allowed to cool to rt and the solvent was removed *in vacuo*. The crude mixture was dissolved in CH_2Cl_2 (10 mL) and washed with water (10 mL), then saturated brine solution (10 mL), The organic layer was dried over $MgSO_4$, filtered and concentrated *in vacuo*. The crude residue was purified by normal-phase chromatography (0-60% EtOAc/Hex) to yield the benzamidine, **13a** (69 mg, 61%) as a pale-yellow oil.



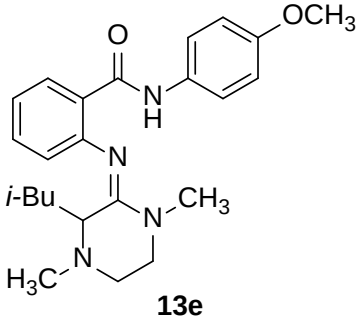
13b

(*E*)-*N*-cyclohexyl-2-((3-isobutyl-1,4-dimethylpiperazin-2-ylidene)amino)benzamide 13b. Obtained using **general procedure E**. Purified by normal-phase chromatography (0-50% EtOAc/hexane) to yield the benzamidine, **13b** (71 mg, 60%) as pale-yellow gum.



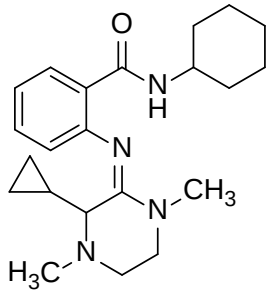
13d

(*E*)-2-((3-isopropyl-1,4-dimethylpiperazin-2-ylidene)amino)-*N*-(4-methoxyphenyl)benzamide 13d. Obtained using **general procedure E**. Purified by normal-phase chromatography (0-20% EtOAc/CH_2Cl_2) to yield the benzamidine, **13d** (120mg, 40%) as a pale-yellow waxy solid.



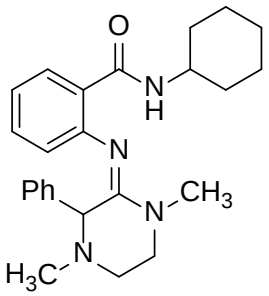
13e

(*E*)-2-((3-isobutyl-1,4-dimethylpiperazin-2-ylidene)amino)-*N*-(4-methoxyphenyl)benzamide 13e. Obtained using **general procedure E**. Purified by normal-phase chromatography (0-20% EtOAc/CH_2Cl_2) to yield the benzamidine, **13e** (56 mg, 45%) as a light-brown solid .



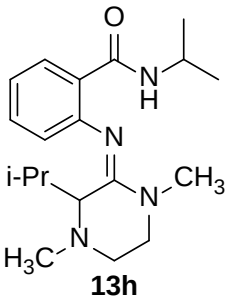
13f

(*E*)-*N*-cyclohexyl-2-((3-cyclopropyl-1,4-dimethylpiperazin-2-ylidene)amino)benzamide 13f. Obtained using **general procedure E**. Purified by normal-phase chromatography (1-5% MeOH/EtOAc) to yield the benzamidine, **13f** (80 mg, 70%) as pale-yellow gum.



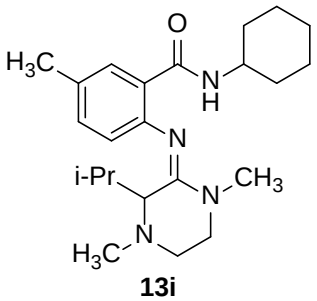
13g

(*E*)-*N*-cyclohexyl-2-((1,4-dimethyl-3-phenylpiperazin-2-ylidene)amino)benzamide 13g. Obtained using **general procedure E**. Purified by normal-phase chromatography (0-5% MeOH/EtOAc) to yield the benzamidine, **13g** (85 mg, 68%) as a pale-yellow oil.



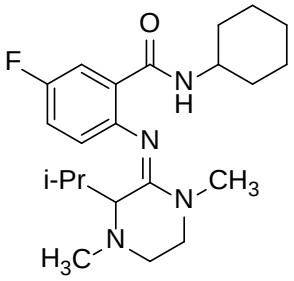
13h

(*E*)-*N*-isopropyl-2-((3-isopropyl-1,4-dimethylpiperazin-2-ylidene)amino)benzamide 13h. Obtained using **general procedure E**. Purified by normal-phase chromatography (0-20% EtOAc/CH_2Cl_2) to yield the benzamidine, **13h** (51 mg, 50%) as a pale-yellow oil.



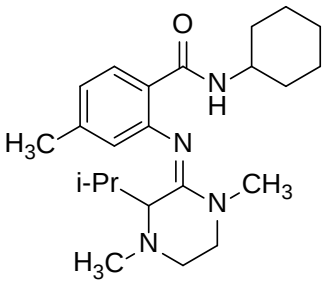
13i

(*E*)-*N*-cyclohexyl-2-((3-isopropyl-1,4-dimethylpiperazin-2-ylidene)amino)-5-methylbenzamide 13i. Obtained using **general procedure E**. Purified by normal-phase chromatography (0-40% EtOAc/hexane) to yield the benzamidine, **13i** (53 mg, 45%) as a pale-yellow gum.



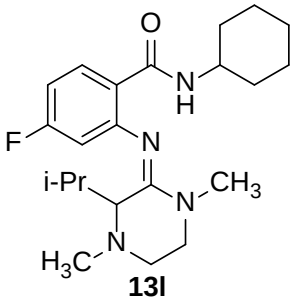
13j

***E*)-*N*-cyclohexyl-5-fluoro-2-((3-isopropyl-1,4-dimethylpiperazin-2-ylidene)amino)benzamide 13j.** Obtained using **general procedure E**. Purified by normal-phase chromatography (0-40% EtOAc/hexane) to yield the benzamidine, **13j** (67 mg, 56%) as a pale-yellow gum.



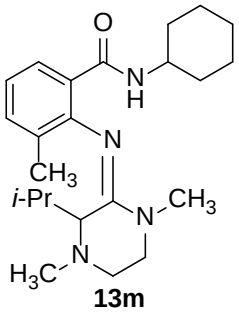
13k

(*E*)-*N*-cyclohexyl-2-((3-isopropyl-1,4-dimethylpiperazin-2-ylidene)amino)-4-methylbenzamide 13k. Obtained using **general procedure E**. Purified by normal-phase chromatography (0-50% EtOAc/hexane) to yield the benzamidine, **13k** (53 mg, 45%) as a pale-yellow gum.



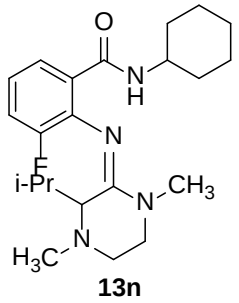
13l

(*E*)-*N*-cyclohexyl-4-fluoro-2-((3-isopropyl-1,4-dimethylpiperazin-2-ylidene)amino)benzamide 13l. Obtained using **general procedure E**. Purified by normal-phase chromatography (0-40% EtOAc/hexane) to yield the benzamidine, **13l** (25mg, 21%) as a pale-yellow gum. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.81 (d, *J* = 8.0 Hz, 1H), 8.19 (dd, *J* = 8.8, 7.1 Hz, 1H), 6.73 (td, *J* = 8.4, 2.5 Hz, 1H), 6.30 (dd, *J* = 10.3, 2.5 Hz, 1H), 3.94 (tdt, *J* = 11.5, 7.9, 3.9 Hz, 1H), 3.52 – 3.42 (m, 2H), 3.37 (ddd, *J* = 12.3, 6.8, 4.7 Hz, 1H), 3.24 (ddd, *J* = 11.7, 6.8, 4.5 Hz, 1H), 3.13 (s, 3H), 2.65 (ddd, *J* = 12.0, 6.8, 4.7 Hz, 1H), 2.52 (s, 3H), 2.02 (dt, *J* = 11.8, 4.1 Hz, 2H), 1.70 (ddt, *J* = 29.4, 14.9, 5.2 Hz, 5H), 1.40 (qd, *J* = 12.5, 3.3 Hz, 2H), 1.22 – 1.06 (m, 3H), 0.82 (d, *J* = 6.7 Hz, 3H), 0.68 (d, *J* = 6.9 Hz, 3H). ^{19}F NMR (376 MHz, $CDCl_3$) δ -109.97. ^{13}C NMR (101 MHz, CDCl3) δ 165.2, 165.0, 162.7, 160.8, 150.5, 150.4, 133.5, 133.4, 122.2, 122.2, 108.9, 108.7, 108.6, 108.4, 63.8, 48.6, 48.4, 47.7, 45.1, 37.3, 33.8, 33.7, 32.5, 29.7, 25.8, 25.2, 20.1, 19.1. HRMS (ESI): Calculated for $C_{22}H_{33}FN_4O$ (M^++H): 389.27116; Found: 389.27155.



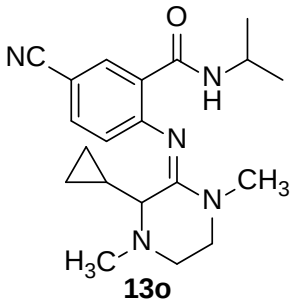
13m

(*E*)-*N*-cyclohexyl-2-((3-isopropyl-1,4-dimethylpiperazin-2-ylidene)amino)-3-methylbenzamide 13m. Obtained using **general procedure E**. Purified by normal phase chromatography (0-30% EtoAc/CH_2Cl_2) to yield the benzamidine, **13m** (41 mg, 35%) as a pale-yellow oil. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.66 (d, *J* = 7.9 Hz, 1H), 8.04 (dd, *J* = 7.9, 1.6 Hz, 1H), 7.23 (dd, *J* = 7.4, 1.5 Hz, 1H), 6.97 (t, *J* = 7.6 Hz, 1H), 3.92 (dtt, *J* = 11.6, 8.0, 4.0 Hz, 1H), 3.60 (ddd, *J* = 12.6, 9.6, 3.2 Hz, 1H), 3.20 (s, 4H), 3.13 (s, 1H), 3.05 (ddd, *J* = 15.4, 8.6, 3.7 Hz, 1H), 2.94 (d, *J* = 3.0 Hz, 1H), 2.62 (ddd, *J* = 11.4, 9.6, 3.6 Hz, 1H), 2.38 (s, 3H), 2.11 (d, *J* = 3.8 Hz, 4H), 2.07 – 1.94 (m, 1H), 1.72 (tdd, *J* = 12.8, 8.1, 5.1 Hz, 1H), 1.63 (qd, *J* = 7.0, 3.0 Hz, 1H), 1.49 – 1.30 (m, 2H), 1.14 (tddd, *J* = 11.6, 8.7, 6.3, 3.8 Hz, 3H), 0.83 (d, *J* = 6.9 Hz, 3H), 0.67 (d, *J* = 6.9 Hz, 4H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 166.1, 160.5, 147.5, 133.2, 129.1, 127.8, 126.0, 121.6, 65.8, 51.5, 49.0, 48.4, 47.4, 37.2, 33.8, 33.7, 32.8, 25.8, 25.3, 25.2, 21.2, 19.2, 17.6. HRMS (ESI): Calculated for $C_{22}H_{33}FN_4O$ (M^++H): 389.27112; Found: 389.27196.



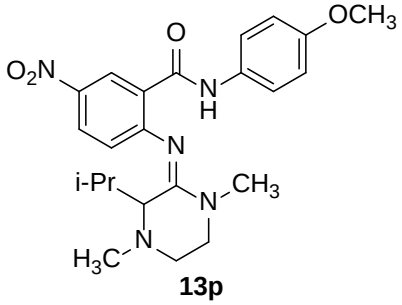
13n

(*E*)-*N*-cyclohexyl-3-fluoro-2-((3-isopropyl-1,4-dimethylpiperazin-2-ylidene)amino)benzamide 13n. Obtained using **general procedure E**. Purified by normal-phase chromatography (0-15% EtOAc/CH_2Cl_2) to yield the benzamidine, **13n** (65 mg, 42%) as a pale-yellow gum. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 9.00 (s, 1H), 7.99 (d, *J* = 7.9 Hz, 1H), 7.10 (ddd, *J* = 10.1, 8.0, 1.6 Hz, 1H), 6.94 (td, *J* = 8.0, 5.1 Hz, 1H), 3.94 (tdt, *J* = 11.4, 7.8, 3.8 Hz, 1H), 3.51 (ddd, *J* = 12.1, 8.0, 3.8 Hz, 1H), 3.33 (ddd, *J* = 12.6, 5.9, 4.1 Hz, 1H), 3.19 (s, 3H), 3.12 (t, *J* = 3.2 Hz, 2H), 2.66 (ddd, *J* = 12.1, 8.0, 4.1 Hz, 1H), 2.45 (s, 3H), 2.07 – 1.97 (m, 2H), 1.77 – 1.61 (m, 4H), 1.46 – 1.34 (m, 2H), 1.16 (dddd, *J* = 12.4, 8.6, 6.8, 2.7 Hz, 3H), 0.82 (d, *J* = 6.8 Hz, 3H), 0.70 (d, *J* = 6.9 Hz, 3H). ^{19}F NMR (376 MHz, $CDCl_3$) δ -127.01. ^{13}C NMR (101 MHz, $CDCl_3$) δ 165.1, 162.9, 153.8, 151.4, 137.2, 137.1, 127.9, 126.7, 121.4, 121.4, 118.0, 117.8, 66.0, 49.9, 48.6, 48.3, 46.0, 37.5, 33.8, 33.7, 33.6, 29.8, 25.9, 25.3, 20.6, 18.7. HRMS (ESI): Calculated for $C_{22}H_{33}FN_4O$ (M^++H): 389.27112; Found: 389.27196.



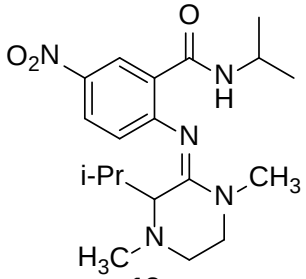
13o

(*E*)-5-cyano-2-((3-cyclopropyl-1,4-dimethylpiperazin-2-ylidene)amino)-*N*-isopropylbenzamide 13o. Obtained using **general procedure E**. Purified by normal-phase chromatography (0-15% MeOH/EtOAC) to yield the benzamidine, **13o** (48 mg, 44%) as a light-brown solid . m.p.: 147-148°C. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.61 (d, *J* = 7.7 Hz, 1H), 8.52 (d, *J* = 2.1 Hz, 1H), 7.50 (dd, *J* = 8.2, 2.1 Hz, 1H), 6.62 (d, *J* = 8.2 Hz, 1H), 4.32 – 4.18 (m, 1H), 3.58 (td, *J* = 11.0, 5.5 Hz, 1H), 3.45 (ddd, *J* = 12.8, 10.4, 5.1 Hz, 1H), 3.35 (ddd, *J* = 11.7, 5.1, 2.4 Hz, 1H), 3.13 (s, 3H), 2.93 (d, *J* = 8.4 Hz, 1H), 2.79 (ddd, *J* = 13.2, 5.7, 2.3 Hz, 1H), 2.46 (s, 3H), 1.23 (d, J = 6.6 Hz, 3H), 1.20 (d, J = 6.5 Hz, 3H), 0.89 (dtd, *J* = 13.3, 8.3, 5.0 Hz, 1H), 0.51 – 0.40 (m, 1H), 0.36 – 0.25 (m, 1H), 0.14 (dq, *J* = 10.5, 5.2 Hz, 1H), -0.48 (dq, *J* = 10.3, 5.2 Hz, 1H). ^{13}C NMR (101 MHz, CDCl3) δ 163.8, 160.6, 153.0, 135.7, 134.2, 126.8, 123.3, 119.2, 105.0, 61.9, 47.2, 45.3, 42.3, 41.4, 37.2, 23.1, 23.0, 11.5, 3.9, 3.6. HRMS (ESI): Calculated for $C_{20}H_{27}N_5O$ (M^++H): 354.22884; Found: 354.22999.



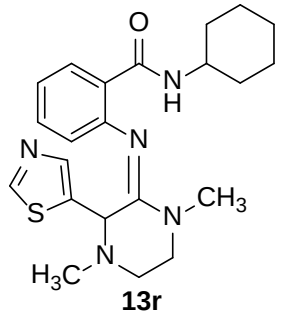
13p

(*E*)-2-((3-isopropyl-1,4-dimethylpiperazin-2-ylidene)amino)-*N*-(4-methoxyphenyl)-5-nitrobenzamide 13p. Obtained using **general procedure E**. Purified by normal-phase chromatography (0-3% EtOAc/CH_2Cl_2) to yield the benzamidine, **13p** (49 mg, 36%) as yellow solid. m.p.: 167-168°C. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 10.93 (s, 1H), 9.17 (d, *J* = 2.8 Hz, 1H), 8.16 (dd, *J* = 8.8, 2.9 Hz, 1H), 7.59 – 7.46 (m, 2H), 6.97 – 6.84 (m, 2H), 6.71 (d, *J* = 8.8 Hz, 1H), 3.81 (s, 3H), 3.57 – 3.46 (m, 2H), 3.45 (d, *J* = 6.4 Hz, 1H), 3.27 (s, 4H), 2.76 – 2.65 (m, 1H), 2.55 (s, 3H), 1.76 (h, *J* = 6.7 Hz, 1H), 0.80 (d, *J* = 6.7 Hz, 3H), 0.70 (d, *J* = 6.8 Hz, 3H). ^{13}C NMR (101 MHz, CDCl3) δ 162.5, 162.0, 156.3, 154.3, 142.3, 131.3, 127.8, 126.4, 125.7, 122.6, 121.8, 114.3, 64.3, 55.5, 48.0, 47.5, 44.9, 37.8, 32.8, 20.0, 19.1. Calculated for HRMS (ESI): $C_{23}H_{29}N_5O_4$ (M^++H): 440.22923; Found: 440.22903.



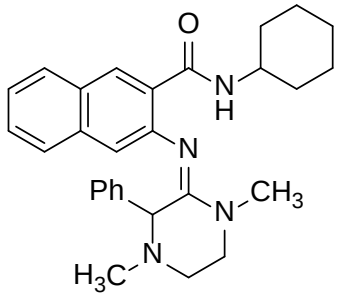
13q

(*E*)-*N*-isopropyl-2-((3-isopropyl-1,4-dimethylpiperazin-2-ylidene)amino)-5-nitrobenzamide 13q. Obtained using **general procedure E**. Purified by normal-phase chromatography (0-7% EtOAc/CH_2Cl_2) to yield the benzamidine, **13q** (36 mg, 31%) as a yellow-orange solid. m.p.: 140-141°C. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 9.10 (d, *J* = 2.8 Hz, 1H), 8.93 (d, *J* = 7.6 Hz, 1H), 8.12 (dd, *J* = 8.8, 2.9 Hz, 1H), 6.65 (d, *J* = 8.8 Hz, 1H), 4.27 (dq, *J* = 13.2, 6.5 Hz, 1H), 3.53 (ddd, *J* = 11.9, 6.9, 4.5 Hz, 1H), 3.46 (dd, *J* = 6.7, 4.9 Hz, 1H), 3.42 (d, *J* = 6.0 Hz, 1H), 3.25 (ddd, *J* = 12.6, 6.6, 4.5 Hz, 1H), 3.19 (s, 3H), 2.71 (ddd, *J* = 12.1, 6.9, 4.8 Hz, 1H), 2.53 (s, 3H), 1.71 (h, *J* = 6.7 Hz, 1H), 1.23 (dd, *J* = 6.6, 1.4 Hz, 6H), 0.83 (d, *J* = 6.7 Hz, 3H), 0.69 (d, *J* = 6.8 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 163.7, 161.3, 154.4, 142.0, 127.68, 126.0, 125.6, 122.5, 77.4, 77.0, 76.7, 64.2, 48.5, 47.7, 45.1, 41.6, 37.4, 32.9, 23.02, 23.0, 20.1, 19.0. Calculated for HRMS (ESI): $C_{19}H_{29}N_5O_3$ (M^++H): 376.23432; Found: 376.23487.



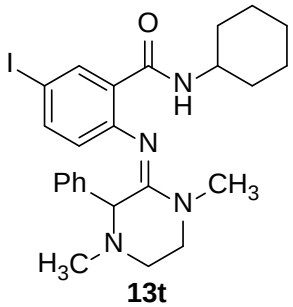
13r

(*E*)-*N*-cyclohexyl-2-((1,4-dimethyl-3-(thiazol-5-yl)piperazin-2-ylidene)amino)benzamide 13r. Obtained using **general procedure E**. Purified by normal-phase chromatography (0.5-3.5% MeOH/CH_2Cl_2) to yield the benzamidine, **13r** (76 mg, 60%) as an amber gum . ^{1}H NMR (400 MHz, Chloroform-d) δ 8.70 (s, 1H), 8.05 (d, J = 7.9 Hz, 1H), 7.99 (dd, J = 7.9, 1.7 Hz, 1H), 7.30 (s, 1H), 7.15 (td, J = 7.6, 1.7 Hz, 1H), 7.06 – 6.99 (m, 1H), 6.38 (d, J = 7.8 Hz, 1H), 4.86 (s, 1H), 3.87 (dtd, J = 10.9, 7.4, 4.0 Hz, 1H), 3.55 (td, J = 11.0, 5.1 Hz, 1H), 3.38 (ddd, J = 11.5, 4.6, 2.7 Hz, 1H), 3.18 (s, 3H), 2.97 (ddd, J = 12.6, 10.6, 4.6 Hz, 1H), 2.64 (ddd, J = 12.9, 5.3, 2.4 Hz, 1H), 2.17 (s, 3H), 2.03 – 1.94 (m, 2H), 1.79 – 1.61 (m, 5H), 1.46 – 1.32 (m, 1H), 1.16 (dq, J = 14.3, 11.5, 11.0 Hz, 3H). ^{13}C NMR (101 MHz, CDCl3) δ 165.1, 156.3, 153.5, 147.3, 143.9, 131.1, 131.0, 130.9, 125.8, 122.8, 122.6, 56.5, 49.2, 48.4, 45.8, 41.6, 37.3, 33.6, 25.8, 25.1. HRMS (ESI): Calculated for $C_{22}H_{29}N_5OS$ (M^++H): 412.21656; Found: 412.21798.



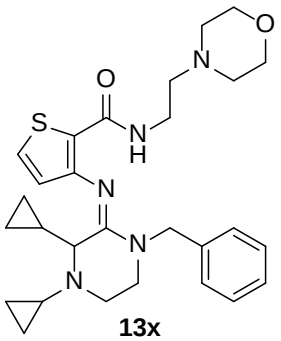
13s

(*E*)-*N*-cyclohexyl-3-((1,4-dimethyl-3-phenylpiperazin-2-ylidene)amino)-2-naphthamide 13s. Obtained using **general procedure E**. Purified by normal-phase chromatography (0-20% EtOAc/CH_2Cl_2) to yield the naphthamidine, **13s** (69 mg, 50%) as apale-yellow gum. ^{1}H NMR (400 MHz, Chloroform-d) δ 8.68 (d, J = 8.0 Hz, 1H), 8.47 (s, 1H), 7.79 (dd, J = 8.3, 1.2 Hz, 1H), 7.36 (d, J = 3.8 Hz, 2H), 7.29 (dd, J = 8.2, 3.9 Hz, 1H), 7.23 – 7.17 (m, 1H), 7.09 (t, J = 7.6 Hz, 2H), 6.81 (dd, J = 7.9, 1.4 Hz, 2H), 6.66 (s, 1H), 4.51 (s, 1H), 3.90 (tdt, J = 11.3, 7.8, 3.9 Hz, 1H), 3.63 – 3.51 (m, 2H), 3.29 (s, 3H), 3.02 (ddd, J = 12.5, 8.7, 5.0 Hz, 1H), 2.62 (dt, J = 12.5, 4.7 Hz, 1H), 2.14 (s, 3H), 2.10 – 1.98 (m, 2H), 1.84 – 1.57 (m, 2H), 1.53 – 1.35 (m, 2H), 1.28 – 1.13 (m, 4H). ^{13}C NMR (101 MHz, CDCl3) δ 164.9, 157.8, 145.04, 135.7, 134.7, 131.7, 129.3, 129.3, 128.9, 127.9, 127.8, 127.0, 126.1, 126.0, 124.0, 119.1, 64.9, 49.2, 48.4, 46.3, 42.0, 37.4, 33.6, 25.8, 25.2, 25.1. Calculated for HRMS (ESI): $C_{29}H_{34}N4O$ (M^++H): 455.28054; Found: 455.28019.



13t

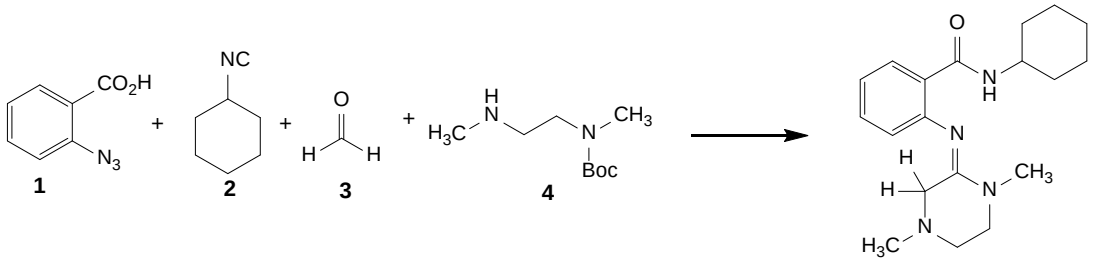
(*E*)-*N*-cyclohexyl-2-((1,4-dimethyl-3-phenylpiperazin-2-ylidene)amino)-5-iodobenzamide 13t. Obtained using **general procedure E**. Purified by normal-phase chromatography (0-15% EtOAc/CH_2Cl_2) to yield benzamidine **13t** (144mg, 88%) as pale yellow oil. Recrystallized from acetonitrile to form clear colorless crystal (See Crystallographic Experimental Section). m.p.: 85-87°C. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.27 (d, *J* = 7.9 Hz, 1H), 8.15 (d, *J* = 2.2 Hz, 1H), 7.32 (dd, *J* = 8.3, 2.3 Hz, 1H), 7.24 – 7.18 (m, 1H), 7.14 (d, *J* = 7.6 Hz, 2H), 7.13 (d, *J* = 8.6 Hz, 0H), 6.85 (d, *J* = 6.9 Hz, 1H), 6.12 (d, *J* = 8.3 Hz, 1H), 4.33 (s, 1H), 3.80 (dtd, *J* = 10.9, 7.3, 3.9 Hz, 1H), 3.64 – 3.42 (m, 2H), 3.22 (s, 3H), 2.98 (ddd, *J* = 12.7, 7.6, 5.2 Hz, 1H), 2.59 (dt, *J* = 12.5, 5.0 Hz, 1H), 2.15 (s, 3H), 2.04 – 1.88 (m, 2H), 1.80 – 1.61 (m, 2H), 1.48 – 1.32 (m, 2H), 1.15 (dtdd, *J* = 23.1, 11.8, 7.9, 3.5 Hz, 2H). ^{13}C NMR (101 MHz, CDCl3) δ 163.7, 157.7, 147.9, 139.3, 139.0, 135.4, 129.2, 128.0, 128.0, 127.4, 125.2, 84.9, 65.5, 49.2, 48.3, 46.8, 42.1, 37.3, 33.5, 33.5, 25.8, 25.1, 25.1. Calculated for HRMS (ESI): $C_{21}H_{35}IN4O$ (M^++H): 531.16154; Found: 531.16069.



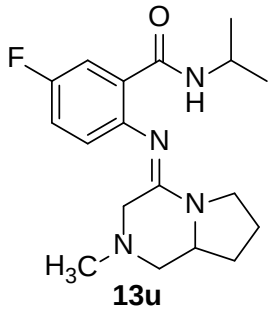
13x

(*E*)-3-((1-benzyl-3,4-dicyclopropylpiperazin-2-ylidene)amino)-*N*-(2-morpholinoethyl) thiophene-2-carboxamide 13x. Obtained using **general procedure E**. Purified by normal-phase chromatography (0-3% MeOH/CH_2Cl_2) to yield the thiophene carboxamidine, **13x** (54 mg, 40%) as a pale-yellow gum . (54 mg, 40%). ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.70 (t, *J* = 5.9 Hz, 1H), 7.43 – 7.24 (m, 7H), 6.49 (d, *J* = 5.2 Hz, 1H), 5.29 (d, *J* = 15.3 Hz, 1H), 4.40 (d, *J* = 15.3 Hz, 1H), 3.64 – 3.47 (m, 6H), 3.32 (ddd, *J* = 24.2, 11.5, 7.1 Hz, 3H), 3.19 (dq, *J* = 13.0, 6.4 Hz, 1H), 2.97 (q, *J* = 6.2, 5.1 Hz, 1H), 2.39 – 2.16 (m, 7H), 1.11 (dp, *J* = 12.5, 4.3, 3.7 Hz, 1H), 0.62 – 0.26 (m, 5H), 0.16 (dq, *J* = 10.0, 5.1 Hz, 1H), -0.30 (dq, *J* = 10.2, 5.1 Hz, 1H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 163.3, 162.3, 149.1, 137.5, 129.0, 127.8, 127.5, 127.1, 124.0, 122.9, 66.9, 62.2, 58.0, 53.5, 51.6, 45.2, 43.6, 36.1, 34.2, 12.3, 7.9, 6.7, 3.9, 3.8. HRMS (ESI): Calculated for $C_{28}H_{37}N_5O_2$ (M^++H): 508.274073; Found: 508.27490.

1.8 General procedure F: Representative Procedure for the Telescoped Synthesis of α-Unsubstituted Benzamidines:

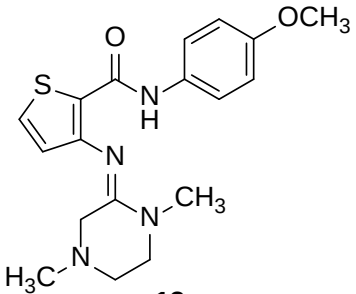


(*E*)-*N*-cyclohexyl-2-((1,4-dimethylpiperazin-2-ylidene)amino)benzamide 13c. An oven-dried, 10 mL round bottom flask under nitrogen was charged with activated, powdered 4 Å molecular sieves (150 mg). The flask was placed under vacuum and flame-dried, then backfilled with nitrogen. Diamine **4** (115 mg, 0.61 mmol, 2.0 equiv.) was dissolved in dry CH2Cl2 (0.9 ml, 0.7 M) and added to the reaction vessel. Formalin **3** (0.1 mL, 1.2 mmol, 4.0 equiv.) was measured with syringe added dropwise over a minute to the reaction vessel while stirring at rt. Upon completion of the addition, carboxylic acid **1** (50 mg, 0.31 mmol, 1.0 equiv.) was dissolved in a 3:1 mixture of dry CH_2Cl_2/HPLC grade MeOH. (0.4 mL, 0.8 M), taken up into a syringe and the needle was placed into the septum of the reaction vessel. Isocyanide **2** (153 µL, 1.2 mmol, 4.0 equiv.) was dissolved in dry CH_2Cl_2 (0.3 mL, 3.6 M) in a 1-dram glass vial, taken up into a syringe and the needle was placed into the septum of the reaction vessel. Carboxylic acid **1** was added dropwise to the reaction vessel while stirring over 1 min followed immediately by isocyanide **2**. The reaction mixture was stirred at rt for 12 h, after which the reaction was complete as judged by complete consumption of carboxylic acid **1** on TLC (10% MeOH/CH_2Cl_2). The crude reaction mixture was filtered through a 0.45 µm syringe filter and the filtrate was concentrated in vacuo. To a flame-dried, 10 mL round bottom flask under nitrogen was added triphenylphosphine on resin (438 mg, 0.61 mmol, 1,4 meq/g, 2.0 equiv.) followed by dry toluene (1.0 mL). A solution of the crude reaction mixture in dry toluene (2.0 ml) was added dropwise and the reaction mixture was stirred at rt for 1 h. Next, the reaction mixture was heated to 110 °C and stirred at reflux for 12 h after which the reaction was complete as judged by complete consumption of the intermediate by TLC (30% EtOAc/hexanes). The crude reaction mixture was filtered through a pad of celite, rinsed with CH_2Cl_2 (10 mL) x 5, and the filtrate was concentrated *in vacuo*. To a flame-dried, 10 mL round bottom flask under nitrogen was dissolved the crude reaction mixture in dry CH_2Cl_2 (3.1 mL, 0.1 M) and the solution cooled to 0 °C in an ice bath while stirring. HCl in dioxane (4.0 M, 0.75 mL, 10.0 equiv.) was added dropwise. The reaction mixture was allowed to slowly warm to rt. The reaction mixture was stirred at rt for 12 h after which the reaction was complete as judged by complete consumption of intermediate on TLC (30% EtOAC/hexanes). The solvent was removed *in vacuo*. Triethylamine (0.4 mL, 3.1 mmol, 10.0 equiv.) was added to a solution of the crude reaction mixture in MeOH (3.1 mL, 0.1 M) in a 10 mL microwave vial and the reaction mixture was heated in a microwave reactor at 100 °C for 1 h. The reaction mixture was allowed to cool to rt and the solvent removed *in vacuo*. The crude mixture was dissolved in CH_2Cl_2 (10 mL) and washed with water (10 mL), then saturated brine solution (10 mL), dried over $MgSO_4$, filtered and concentrated *in vacuo*. The crude residue was purified by normal-phase chromatography (1-5% EtOAc:MeOH) to yield the benzamidine, **13c** (48 mg, 48%) as an amber gum.



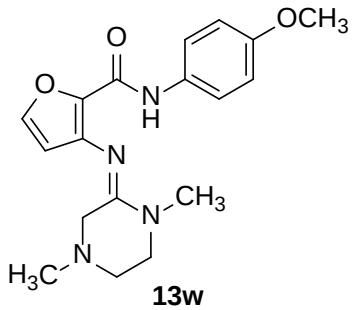
13u

(*E*)-5-fluoro-*N*-isopropyl-2-((2-methylhexahydropyrrolo[1,2-a]pyrazin-4(1H)-ylidene)amino) benzamide 13u. Obtained using **general procedure F**. Purified by normal-phase chromatography (0-3% EtOAc/CH_2Cl_2) to yield the benzamidine, **13u** (51 mg, 50%) as a pale-yellow gum. ^{1}H NMR (400 MHz, Chloroform-d) δ 8.62 (d, J = 7.7 Hz, 1H), 7.85 (dd, J = 10.0, 3.1 Hz, 1H), 7.07 – 6.94 (m, 1H), 6.69 (dd, J = 8.7, 5.0 Hz, 1H), 4.31 – 4.15 (m, 1H), 3.78 – 3.51 (m, 3H), 3.30 (d, J = 16.3 Hz, 1H), 3.08 (dd, J = 10.8, 3.8 Hz, 1H), 2.56 (d, J = 16.2 Hz, 1H), 2.24 (s, 3H), 2.09 (dq, J = 11.8, 6.2, 5.7 Hz, 2H), 1.95 (dd, J = 12.7, 8.7 Hz, 2H), 1.48 (qd, J = 11.7, 7.9 Hz, 1H), 1.19 (dd, J = 6.5, 1.6 Hz, 6H). ^{19}F NMR (376 MHz, CDCl3) δ -121.28. ^{13}C NMR (101 MHz, $CDCl_3$) δ 170.2, 164.7, 164.7, 159.9, 157.5, 154.5, 144.7, 144.7, 144.6, 127.2, 127.1, 124.7, 124.6, 118.3, 118.1, 117.2, 116.9, 57.9, 57.8, 54.2, 45.8, 45.2, 41.3, 30.4, 23.1, 23.0, 22.5. HRMS (ESI): Calculated for $C_{18}H_{25}FN_4O$ (M^++H): 333.20852; Found: 333.20814.



13v

(*E*)-3-((1,4-dimethylpiperazin-2-ylidene)amino)-*N*-(4-methoxyphenyl)thiophene-2-carboxamide 13v. Obtained using **general procedure F**. Purified by normal-phase chromatography (0-3% MeOH/EtOAc) to yield the thiophene carboxamidine, **13v** (32mg, 29%) as a light-brown solid. m.p.: 125-127°C. ^{1}H NMR (400 MHz, Chloroform-d) δ 10.21 (s, 1H), 7.55 – 7.46 (m, 2H), 7.35 (d, J = 5.2 Hz, 1H), 6.89 – 6.83 (m, 2H), 6.60 (d, J = 5.2 Hz, 1H), 3.80 (s, 3H), 3.43 (t, J = 5.6 Hz, 2H), 3.22 (s, 4H), 2.67 (t, J = 5.6 Hz, 2H), 2.30 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 160.9, 157.1, 155.7, 147.9, 132.1, 128.1, 124.5, 123.8, 121.2, 114.2, 55.5, 55.4, 52.0, 49.7, 45.5, 36.7. HRMS (ESI): Calculated for $C_{18}H_{22}N_4O_2S$ (M^++H): 359.15362; Found: 359.15459.



13w

(*E*)-3-((1,4-dimethylpiperazin-2-ylidene)amino)-*N*-(4-methoxyphenyl)furan-2-carboxamide
13w. Obtained using **general procedure F**. Purified by normal-phase chromatography (5-30% EtOAc/CH_2Cl_2) to yield furan carboxamidine, **13w** (21 mg, 20%) as a light-brown solid. m.p.: 150-151°C. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 9.82 (s, 1H), 7.51 (d, *J* = 9.0 Hz, 2H), 7.44 (d, *J* = 1.9 Hz, 1H), 6.87 (d, *J* = 9.1 Hz, 2H), 6.17 (d, *J* = 1.9 Hz, 1H), 3.79 (s, 3H), 3.43 (t, *J* = 5.6 Hz, 2H), 3.31 (s, 2H), 3.20 (s, 3H), 2.69 (t, *J* = 5.6 Hz, 2H), 2.35 (s, 3H). ^{13}C NMR (101 MHz, CDCl3) δ 157.7, 157.5, 155.7, 143.8, 137.5, 136.7, 132.0, 121.2, 114.2, 108.9, 55.5, 55.4, 51.9, 49.7, 45.5, 36.9. HRMS (ESI): Calculated for $C_{18}H_{22}N_4O_3$ (M^++H): 343.17647; Found: 343.17700.

2. Crystallographic Structural Report for 13t

2.1 Data Collection

A colorless crystal with approximate dimensions 0.14 x 0.10 x 0.06 mm^3 was selected under oil under ambient conditions and attached to the tip of a MiTeGen MicroMount©. The crystal was mounted in a stream of cold nitrogen at 100(1) K and centered in the X-ray beam by using a video camera.
The crystal evaluation and data collection were performed on a Bruker Quazar SMART APEXII diffractometer with Mo K_α (λ = 0.71073 Å) radiation and the diffractometer to crystal distance of 4.96 cm [9].
The initial cell constants were obtained from three series of ω scans at different starting angles. Each series consisted of 12 frames collected at intervals of 0.5º in a 6º range about ω with the exposure time of 10 seconds per frame. The reflections were successfully indexed by an automated indexing routine built in the APEX3 program suite. The final cell constants were calculated from a set of 9800 strong reflections from the actual data collection.

The data were collected by using the full sphere data collection routine to survey the reciprocal space to the extent of a full sphere to a resolution of 0.65 Å. A total of 33980 data were harvested by collecting 3 sets of frames with 0.5º scans in ω and ϕ with exposure times of 40 sec per frame. These highly redundant datasets were corrected for Lorentz and polarization effects. The absorption correction was based on fitting a function to the empirical transmission surface as sampled by multiple equivalent measurements.[10]

2.2 Structure Solution and Refinement

The systematic absences in the diffraction data were uniquely consistent for the space group $P2_1/c$ that yielded chemically reasonable and computationally stable results of refinement[11-16].
A successful solution by the direct methods provided most non-hydrogen atoms from the *E*-map. The remaining non-hydrogen atoms were located in an alternating series of least-squares cycles and difference Fourier maps. All non-hydrogen atoms were refined with anisotropic displacement coefficients. All hydrogen atoms except H4 (bound to N4) were included in the structure factor calculation at idealized positions and were allowed to ride on the neighboring atoms with relative isotropic displacement coefficients.
The crystal structure is that of a racemate. The shown enantiomer (*S*) was chosen arbitrarily.
The final least-squares refinement of 286 parameters against 8585 data resulted in residuals *R* (based on F^2 for $I \geq 2\sigma$) and *wR* (based on F^2 for all data) of 0.0191 and 0.0506, respectively. The final difference Fourier map was featureless.

2.3 Summary

Crystal Data for $C_{25}H_{31}IN_4O$ (*M* =530.44 g/mol): monoclinic, space group $P2_1/c$ (no. 14), *a* = 11.677(4) Å, *b* = 12.610(4) Å, *c* = 16.020(5) Å, *β* = 99.578(13)°, *V* = 2325.9(13) Å^3, *Z* = 4, *T* = 99.99 K, μ(MoKα) = 1.401 mm^{-1}, *Dcalc* = 1.515 g/cm^3, 33980 reflections measured (3.538° ≤ 2Θ ≤ 66.282°), 8585 unique (R_{int} = 0.0177, R_{sigma} = 0.0156) which were used in all calculations. The final R_1 was 0.0191 (I > 2σ(I)) and wR_2 was 0.0506 (all data).

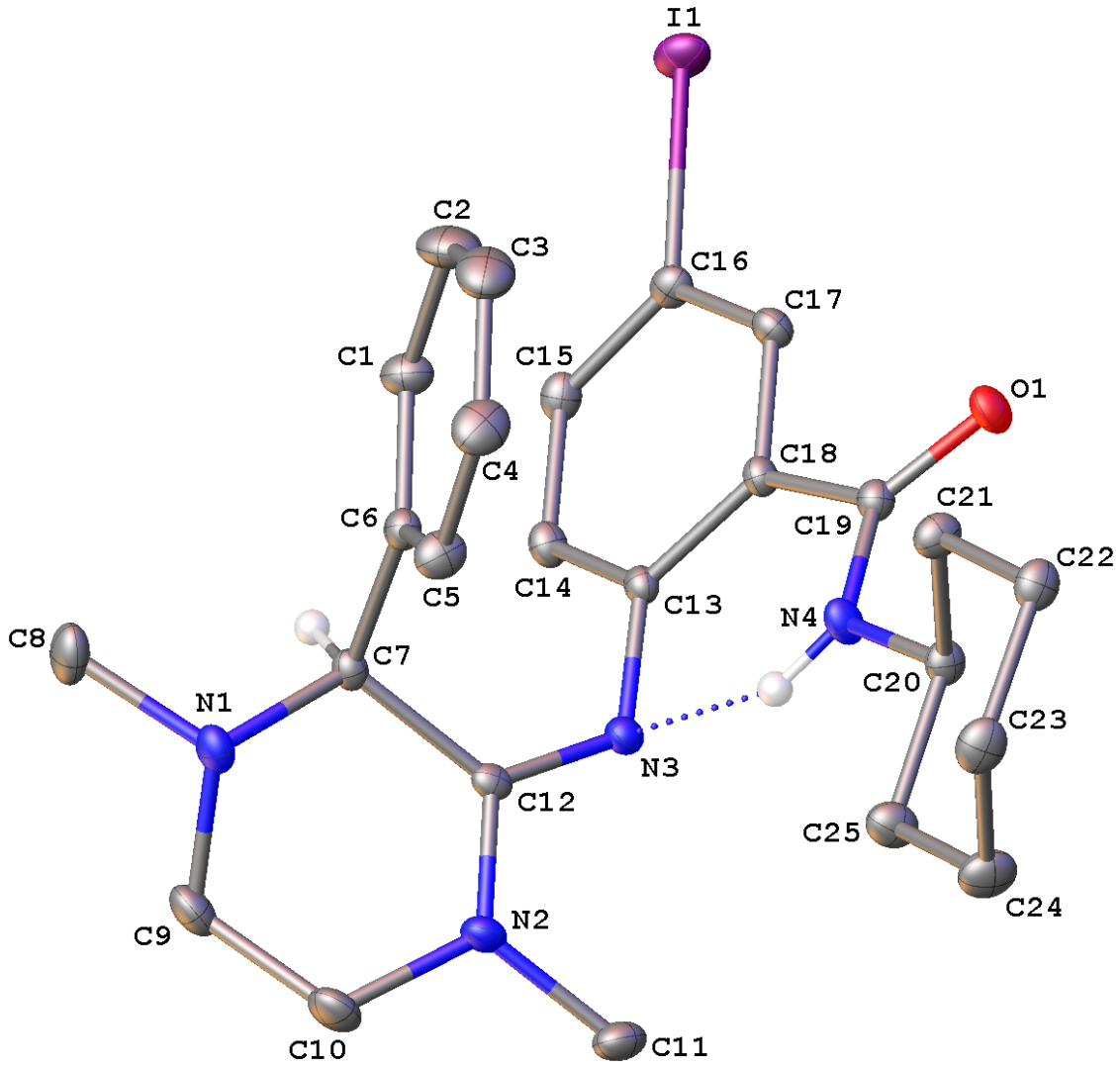


Figure 1. A molecular drawing of Golden01 shown with 50% probability ellipsoids. All H atoms are omitted, except for the amine atom H4 connected to atom N4 (the dotted line represents a hydrogen bonding interaction), and atom H7 attached to the chiral atom C7.

Table 1 Crystal data and structure refinement for golden01.

Identification code	golden01
Empirical formula	$C_{25}H_{31}IN_4O$
Formula weight	530.44
Temperature/K	99.99
Crystal system	monoclinic
Space group	$P2_1/c$
a/Å	11.677(4)
b/Å	12.610(4)
c/Å	16.020(5)
α/°	90
β/°	99.578(13)
γ/°	90
Volume/Å^3	2325.9(13)
Z	4
ρ_{calc}g/cm^3	1.515
μ/mm^{-1}	1.401
F(000)	1080.0
Crystal size/mm^3	0.138 × 0.102 × 0.06
Radiation	MoKα (λ = 0.71073)
2Θ range for data collection/°	3.538 to 66.282
Index ranges	-17 ≤ h ≤ 17, -19 ≤ k ≤ 19, -24 ≤ l ≤ 23
Reflections collected	33980
Independent reflections	8585 [R_{int} = 0.0177, R_{sigma} = 0.0156]
Data/restraints/parameters	8585/0/286
Goodness-of-fit on F^2	1.031
Final R indexes [I>=2σ (I)]	R_1 = 0.0191, wR_2 = 0.0494
Final R indexes [all data]	R_1 = 0.0219, wR_2 = 0.0506
Largest diff. peak/hole / e Å^{-3}	0.56/-0.44

Table 2 Fractional Atomic Coordinates (×10^4) and Equivalent Isotropic Displacement Parameters (Å^2×10^3) for golden01. U_{eq} is defined as 1/3 of of the trace of the orthogonalised U_{IJ} tensor.

Atom	*x*	*y*	*z*	U(eq)
I1	8640.3(2)	-1143.1(2)	3078.8(2)	20.02(2)
O1	8471.8(7)	2478.3(6)	1209.8(4)	16.19(13)
N1	6476.5(8)	4411.5(7)	4874.0(5)	14.73(14)
N2	8371.9(8)	5303.5(6)	4296.4(5)	14.62(14)
N3	8895.1(7)	3789.5(6)	3691.6(5)	11.97(14)
N4	8163.7(8)	3938.6(6)	1968.0(5)	13.45(14)
C1	6178.8(9)	2201.2(8)	3496.6(7)	17.21(17)
C2	5394.7(10)	1830.1(9)	2808.6(8)	24.5(2)
C3	4751.8(10)	2537.2(10)	2257.4(7)	24.9(2)
C4	4897.9(10)	3621.0(9)	2392.0(7)	22.6(2)
C5	5687.2(9)	3993.2(8)	3073.2(6)	17.51(18)
C6	6335.2(8)	3284.3(7)	3634.6(6)	12.20(15)
C7	7148.6(8)	3710.1(7)	4403.6(6)	11.35(15)
C8	5750.0(11)	3775.0(9)	5345.1(7)	21.4(2)
C9	7235.2(10)	5113.0(8)	5434.7(6)	19.39(19)
C10	7802.2(10)	5873.8(8)	4905.4(7)	19.77(19)
C11	9331.1(10)	5854.7(8)	4007.5(7)	18.58(18)

C12	8194.4(8)	4260.6(7)	4124.9(5)	10.82(14)
C13	8854.9(8)	2698.7(7)	3537.5(5)	10.76(14)
C14	9104.7(8)	1986.0(7)	4221.0(6)	12.93(15)
C15	9074.8(8)	898.5(8)	4098.7(6)	13.88(16)
C16	8816.0(8)	496.9(7)	3280.3(6)	13.30(15)
C17	8647.9(9)	1179.7(7)	2594.4(6)	12.82(15)
C18	8672.5(8)	2279.6(7)	2706.9(5)	10.70(14)
C19	8434.5(8)	2910.2(7)	1897.3(6)	11.96(15)
C20	7933.6(8)	4662.0(7)	1249.4(6)	13.15(15)
C21	6792.1(9)	4398.3(8)	672.2(6)	15.44(17)
C22	6530.4(9)	5200.5(8)	-51.1(6)	15.99(17)
C23	6487.0(10)	6328.0(8)	289.6(7)	18.94(18)
C24	7619.0(10)	6602.4(8)	871.6(7)	20.87(19)
C25	7911.9(10)	5793.5(8)	1587.2(6)	17.70(18)

Table 3 Anisotropic Displacement Parameters ($Å^2×10^3$) for golden01. The Anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2}U_{11}+2hka^*b^*U_{12}+\ldots]$.

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
I1	20.89(4)	10.47(3)	29.34(4)	0.14(2)	6.07(3)	1.91(2)
O1	22.1(4)	16.5(3)	10.0(3)	-1.2(2)	3.0(3)	1.4(3)
N1	16.9(4)	14.3(3)	13.9(3)	-0.5(3)	5.5(3)	4.2(3)
N2	19.3(4)	10.1(3)	14.3(3)	-2.1(3)	2.5(3)	-1.1(3)
N3	13.7(4)	11.7(3)	10.4(3)	-0.8(2)	1.9(3)	-0.4(2)
N4	18.3(4)	13.0(3)	8.9(3)	1.4(2)	2.0(3)	3.5(3)
C1	15.6(4)	14.4(4)	20.8(4)	-0.8(3)	0.7(4)	-0.3(3)
C2	22.0(5)	20.5(5)	29.1(5)	-6.6(4)	-1.0(4)	-4.8(4)
C3	20.1(5)	32.2(6)	20.2(5)	-3.8(4)	-2.7(4)	-6.1(4)
C4	19.1(5)	28.3(5)	18.0(4)	4.0(4)	-4.0(4)	-1.7(4)
C5	16.6(4)	18.0(4)	16.5(4)	3.9(3)	-1.4(3)	0.2(3)
C6	11.3(4)	13.6(4)	12.0(3)	0.2(3)	2.8(3)	0.6(3)
C7	12.7(4)	11.0(3)	10.7(3)	0.5(3)	2.8(3)	2.3(3)
C8	23.6(5)	21.7(5)	22.2(5)	2.9(4)	13.1(4)	5.4(4)
C9	26.0(5)	18.8(4)	13.5(4)	-4.4(3)	4.0(4)	3.8(4)
C10	27.0(5)	12.4(4)	20.2(4)	-4.9(3)	4.6(4)	2.4(4)
C11	21.9(5)	14.2(4)	18.9(4)	-0.1(3)	1.3(4)	-5.5(3)
C12	13.1(4)	10.2(3)	8.2(3)	0.3(3)	-1.0(3)	0.9(3)
C13	9.8(4)	12.4(3)	10.2(3)	-0.7(3)	2.2(3)	0.3(3)
C14	12.7(4)	15.7(4)	10.1(3)	0.8(3)	1.2(3)	2.4(3)
C15	12.8(4)	15.2(4)	13.7(4)	3.5(3)	2.5(3)	3.5(3)
C16	13.1(4)	10.8(3)	16.4(4)	1.2(3)	3.6(3)	2.5(3)
C17	13.8(4)	12.2(4)	12.4(4)	-1.4(3)	2.0(3)	1.6(3)
C18	10.9(4)	12.2(3)	9.1(3)	0.3(3)	1.7(3)	1.4(3)
C19	11.5(4)	13.2(4)	11.2(3)	0.9(3)	1.8(3)	0.8(3)
C20	14.7(4)	13.6(4)	11.2(3)	3.0(3)	1.9(3)	1.8(3)
C21	15.7(4)	15.1(4)	14.7(4)	3.8(3)	0.2(3)	-0.2(3)
C22	15.2(4)	18.3(4)	13.9(4)	4.9(3)	1.0(3)	2.1(3)
C23	19.1(5)	16.9(4)	20.8(4)	7.1(3)	3.1(4)	4.4(3)
C24	25.3(5)	13.7(4)	22.7(5)	4.9(3)	1.1(4)	-0.7(4)
C25	22.9(5)	13.7(4)	15.5(4)	1.7(3)	0.4(4)	0.9(3)

Table 4 Bond Lengths for golden01.

Atom	Atom	Length/Å	Atom	Atom	Length/Å
I1	C16	2.0979(11)	C5	C6	1.3979(14)
O1	C19	1.2360(11)	C6	C7	1.5228(13)
N1	C7	1.4707(12)	C7	C12	1.5343(14)
N1	C8	1.4651(14)	C9	C10	1.5048(16)
N1	C9	1.4524(14)	C13	C14	1.4090(13)
N2	C10	1.4586(13)	C13	C18	1.4146(13)
N2	C11	1.4575(14)	C14	C15	1.3848(14)
N2	C12	1.3523(12)	C15	C16	1.3910(14)
N3	C12	1.3012(12)	C16	C17	1.3839(13)
N3	C13	1.3969(12)	C17	C18	1.3983(13)
N4	C19	1.3440(12)	C18	C19	1.5071(13)
N4	C20	1.4583(12)	C20	C21	1.5271(14)
C1	C2	1.3914(15)	C20	C25	1.5277(14)
C1	C6	1.3907(14)	C21	C22	1.5302(14)
C2	C3	1.3849(18)	C22	C23	1.5268(15)
C3	C4	1.3896(17)	C23	C24	1.5252(16)
C4	C5	1.3876(15)	C24	C25	1.5300(14)

Table 5 Bond Angles for golden01.

Atom	Atom	Atom	Angle/˚	Atom	Atom	Atom	Angle/˚
C8	N1	C7	109.79(8)	N3	C13	C14	119.62(8)
C9	N1	C7	111.13(8)	N3	C13	C18	121.97(8)
C9	N1	C8	111.16(8)	C14	C13	C18	118.17(8)
C11	N2	C10	115.93(8)	C15	C14	C13	121.64(9)
C12	N2	C10	122.94(9)	C14	C15	C16	119.35(8)
C12	N2	C11	119.94(8)	C15	C16	I1	120.28(7)
C12	N3	C13	122.56(8)	C17	C16	I1	119.59(7)
C19	N4	C20	123.51(8)	C17	C16	C15	120.11(9)
C6	C1	C2	120.52(10)	C16	C17	C18	121.18(9)
C3	C2	C1	120.26(11)	C13	C18	C19	126.14(8)
C2	C3	C4	119.65(10)	C17	C18	C13	119.23(8)
C5	C4	C3	120.19(10)	C17	C18	C19	114.56(8)
C4	C5	C6	120.48(10)	O1	C19	N4	122.95(8)
C1	C6	C5	118.88(9)	O1	C19	C18	120.37(8)
C1	C6	C7	121.51(8)	N4	C19	C18	116.67(8)
C5	C6	C7	119.54(8)	N4	C20	C21	111.68(8)
N1	C7	C6	108.20(8)	N4	C20	C25	108.44(8)
N1	C7	C12	113.30(8)	C21	C20	C25	110.71(8)
C6	C7	C12	110.10(7)	C20	C21	C22	111.02(8)
N1	C9	C10	108.67(8)	C23	C22	C21	111.05(8)
N2	C10	C9	110.79(8)	C24	C23	C22	110.66(9)
N2	C12	C7	118.91(8)	C23	C24	C25	111.70(9)
N3	C12	N2	117.62(9)	C20	C25	C24	111.84(9)
N3	C12	C7	123.38(8)				

Table 6 Hydrogen Bonds for golden01.

D	H	A	d(D-H)/Å	d(H-A)/Å	d(D-A)/Å	D-H-A/°
N4	H4	N3	0.841(16)	2.067(16)	2.7576(14)	139.0(15)

Table 7 Hydrogen Atom Coordinates (Å×10^4) and Isotropic Displacement Parameters (Å^2×10^3) for golden01.

Atom	*x*	*y*	*z*	U(eq)
H4	8274(14)	4215(13)	2453(10)	24(4)
H1	6610.86	1710.86	3874.94	21
H2	5300.03	1088.71	2716.71	29
H3	4213.76	2282.74	1789.69	30
H4A	4456.14	4108.63	2016.66	27
H5	5787.9	4735.11	3158.53	21
H7	7439.5	3098.14	4776.44	14
H8A	5259.44	3299.19	4954.46	32
H8B	5257.68	4245.18	5620.18	32
H8C	6246.79	3354.73	5775.94	32
H9A	7835.37	4693.8	5802.33	23
H9B	6779.69	5509.81	5800.38	23
H10A	7208.15	6357.85	4600.12	24
H10B	8382.32	6307.72	5278.13	24
H11A	9180.67	6619.66	3992.14	28
H11B	9403.99	5607.03	3438.84	28
H11C	10053.6	5708.13	4397.17	28
H14	9298.55	2258.24	4779.56	16
H15	9229.44	431.3	4569.21	17
H17	8513.47	896.1	2037.3	15
H20	8580.74	4601.64	914.31	16
H21A	6151.69	4402.66	1006.83	19
H21B	6840.37	3677.88	434.79	19
H22A	7138.71	5155.26	-412.68	19
H22B	5775.65	5025.68	-404.69	19
H23A	6355.56	6834.55	-188.99	23
H23B	5830.92	6392.39	606.18	23
H24A	7550.78	7315.35	1117.39	25
H24B	8258.68	6624.4	536.56	25
H25A	8680.38	5965.41	1922.24	21
H25B	7327.56	5841.87	1968.05	21

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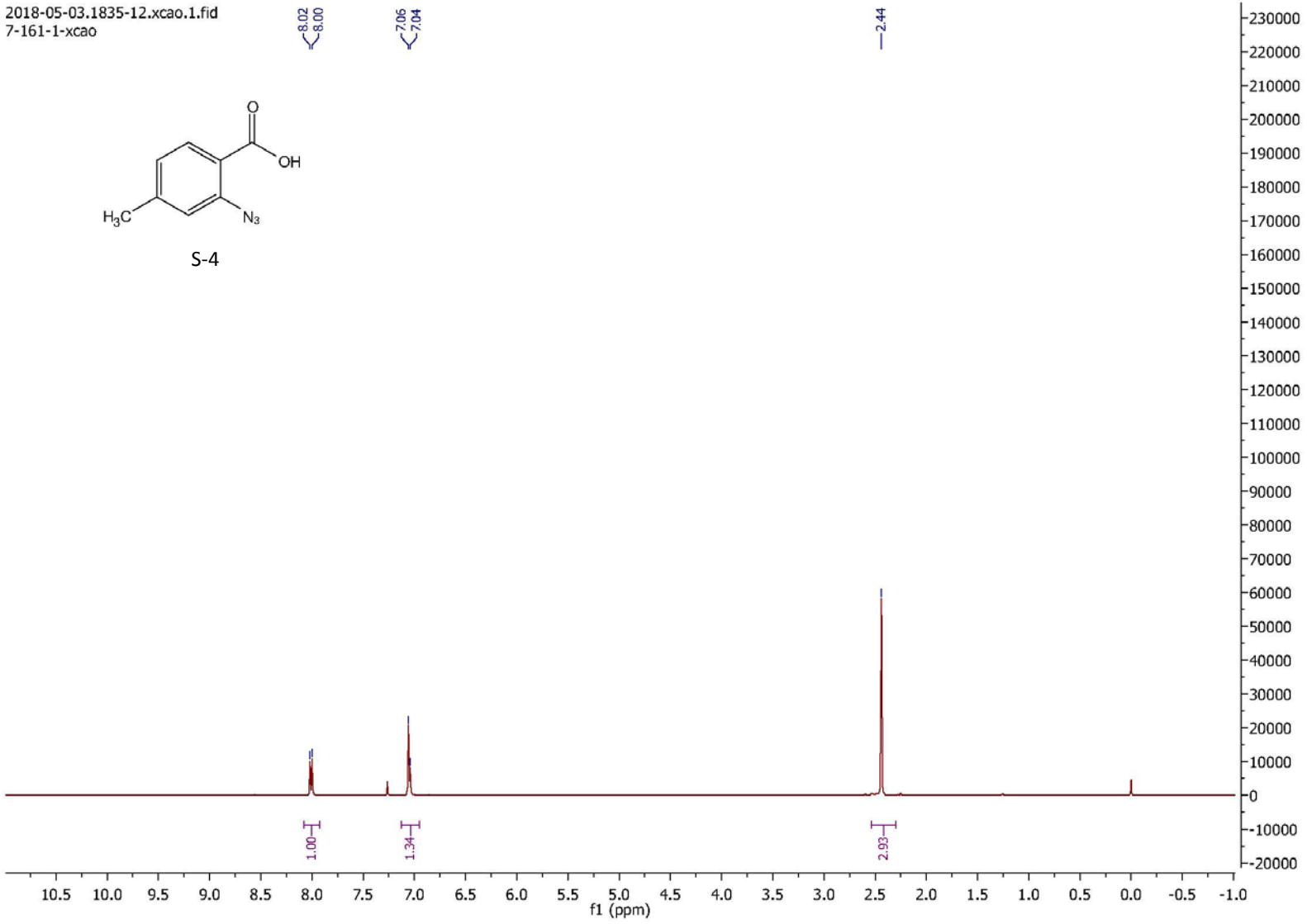
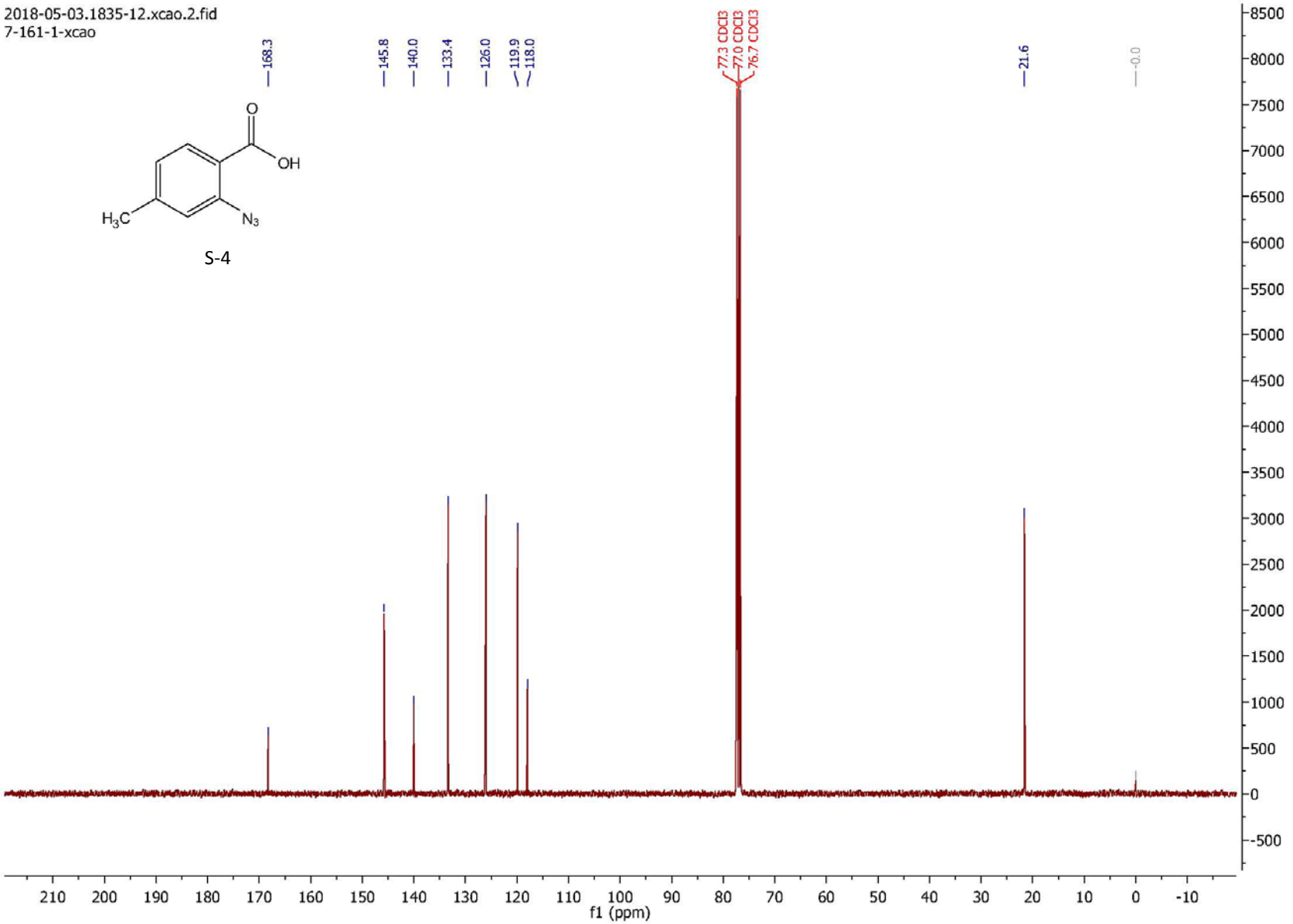
4. NMR Spectra for Compounds



S-4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118.0
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77.0 CDCl3
76.7 CDCl3
21.6
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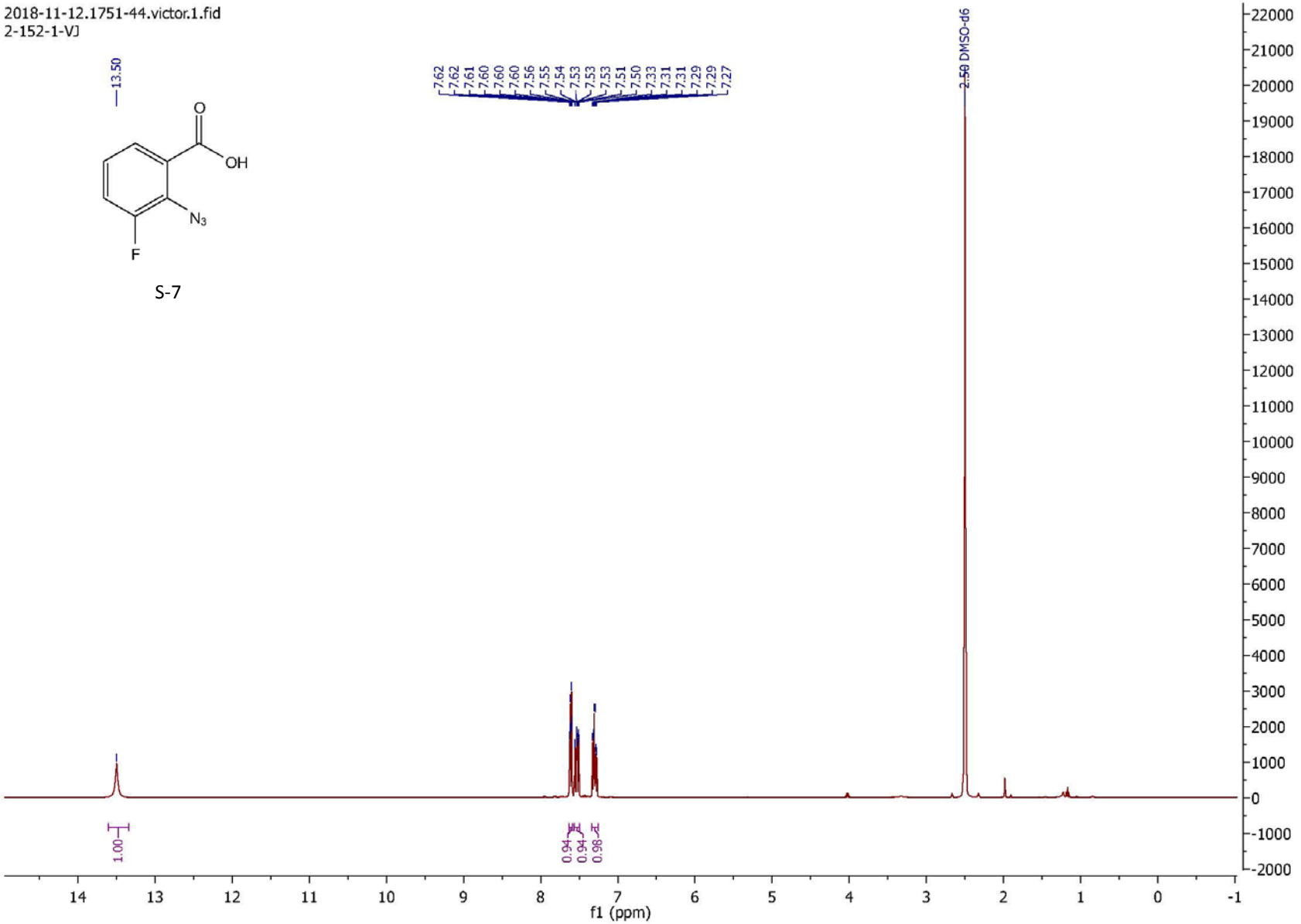
OH
H3C
N3
S-4
f1 (ppm)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7.60
7.56
7.55
7.54
7.53
7.53
7.53
7.51
7.50
7.33
7.31
7.31
7.29
7.29
7.27
2.50 DMSO-d6
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OH
N3
F
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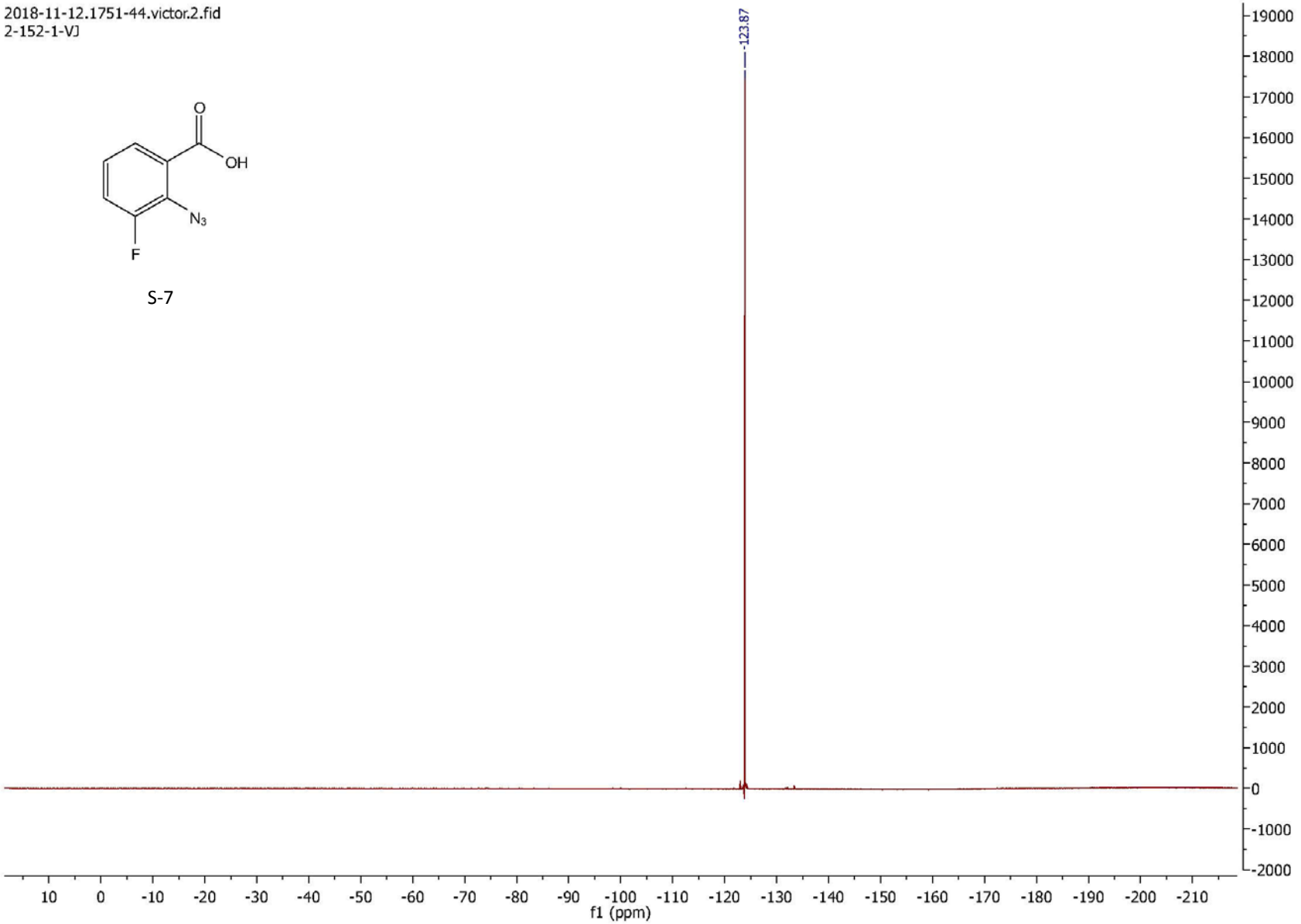
0.94
0.98
f1 (ppm)

S-7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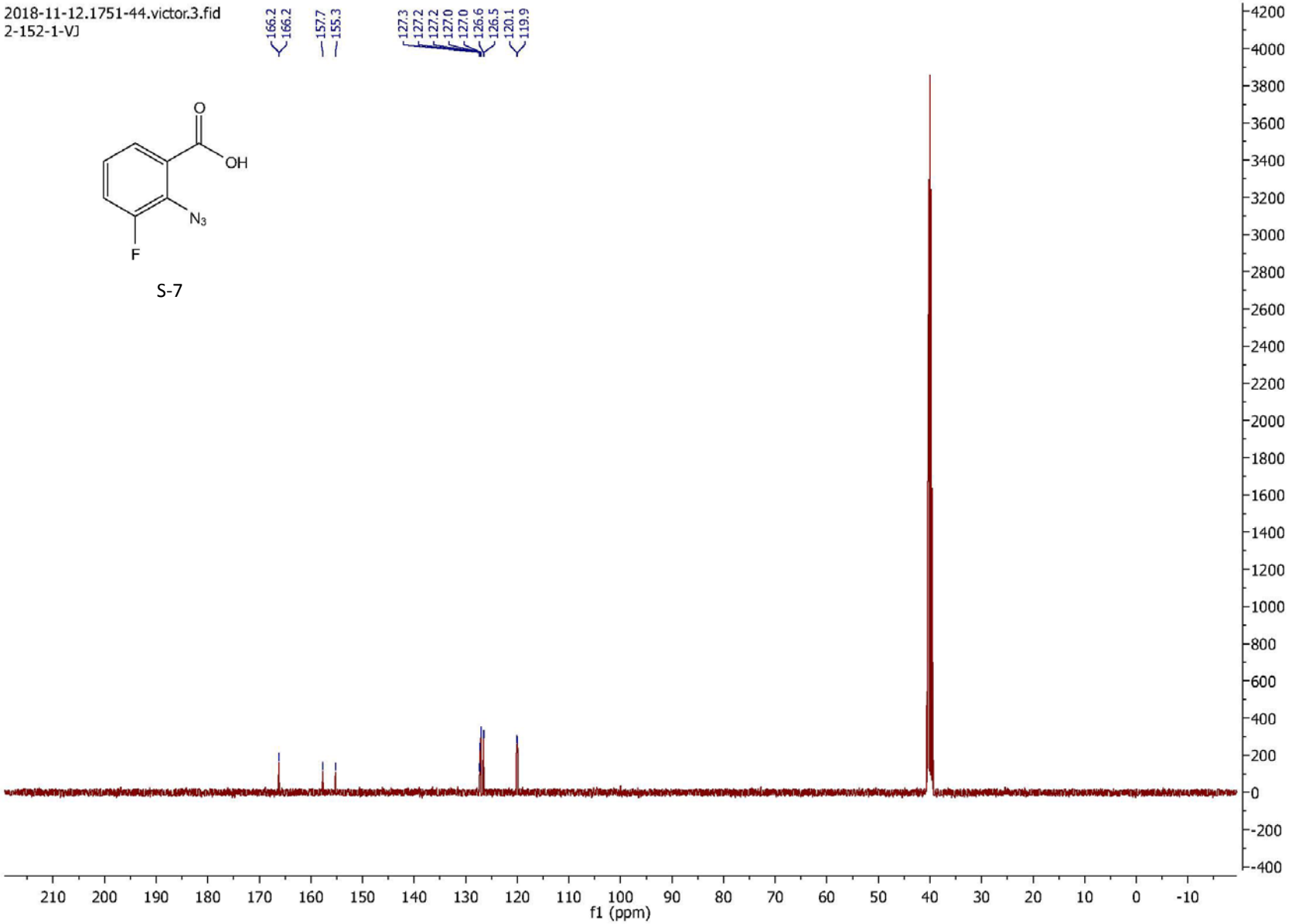
15000
14000
13000
12000
11000
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
-1000
-2000
10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 -210
f1 (ppm)

S-7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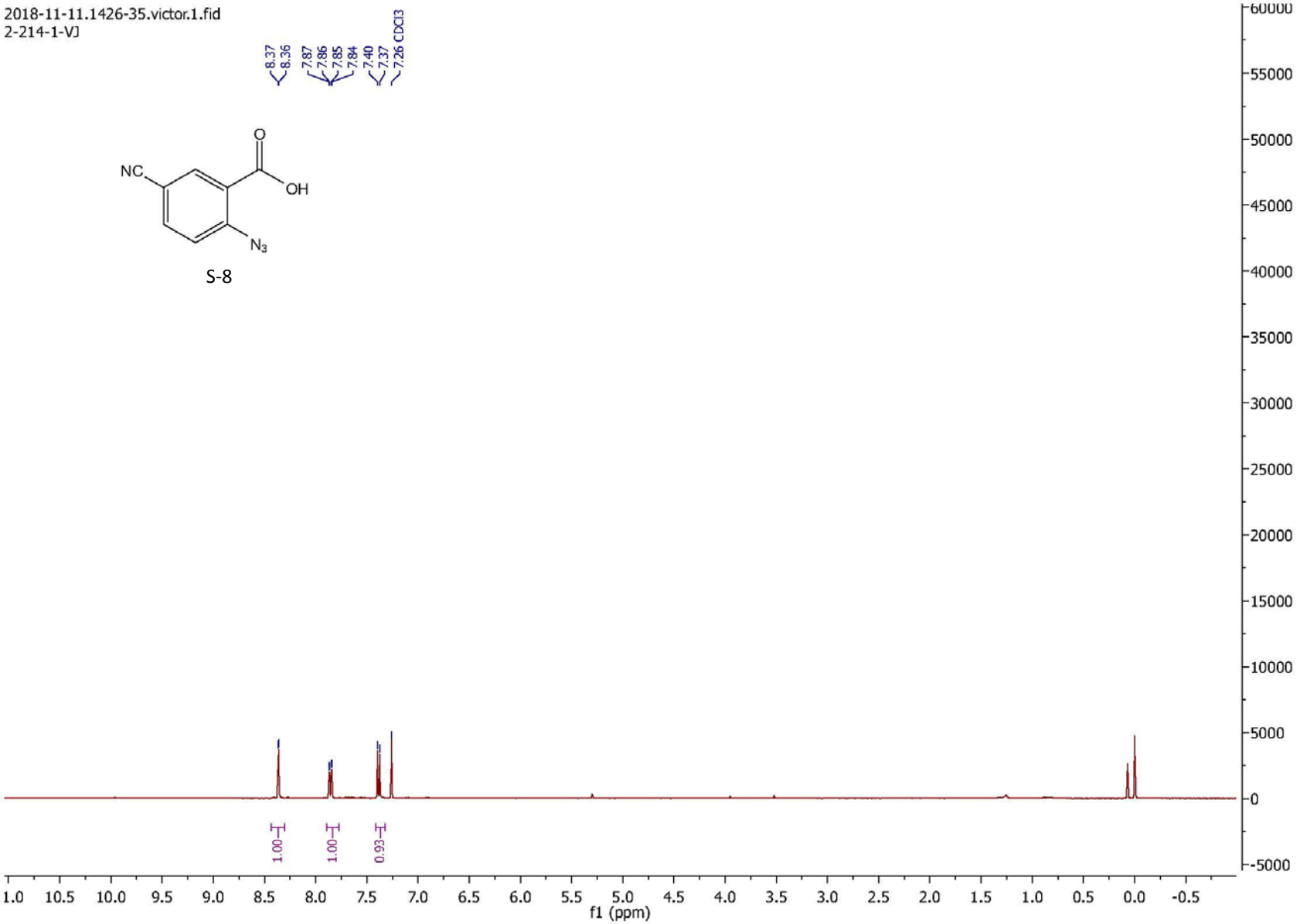
157.7
155.3
127.3
127.2
127.2
127.0
127.0
126.6
126.5
120.1
119.9
O
OH
N3
F
f1 (ppm)

S-7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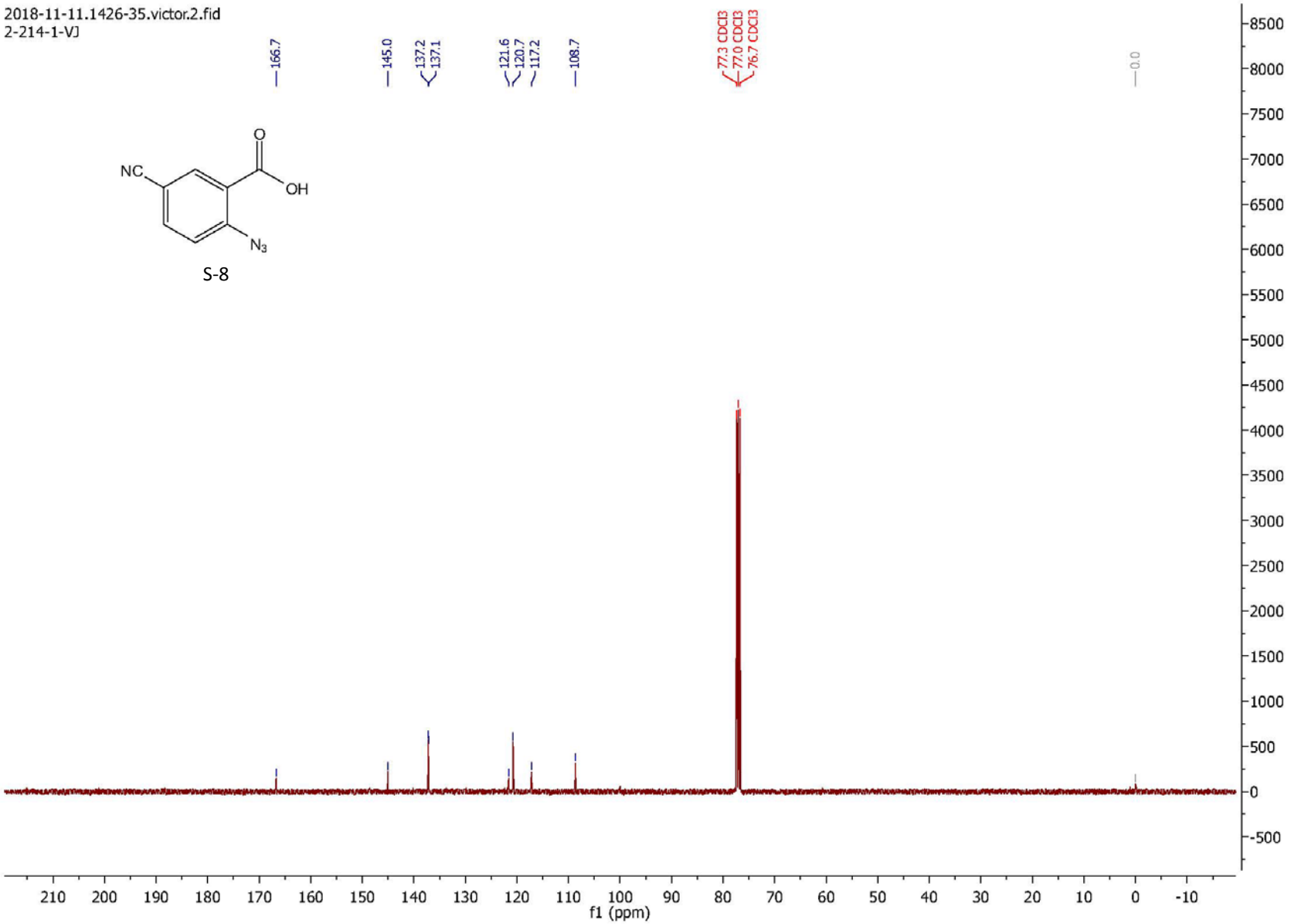
NC
O
OH
N3
1.00
1.00
0.93
f1 (ppm)

S-8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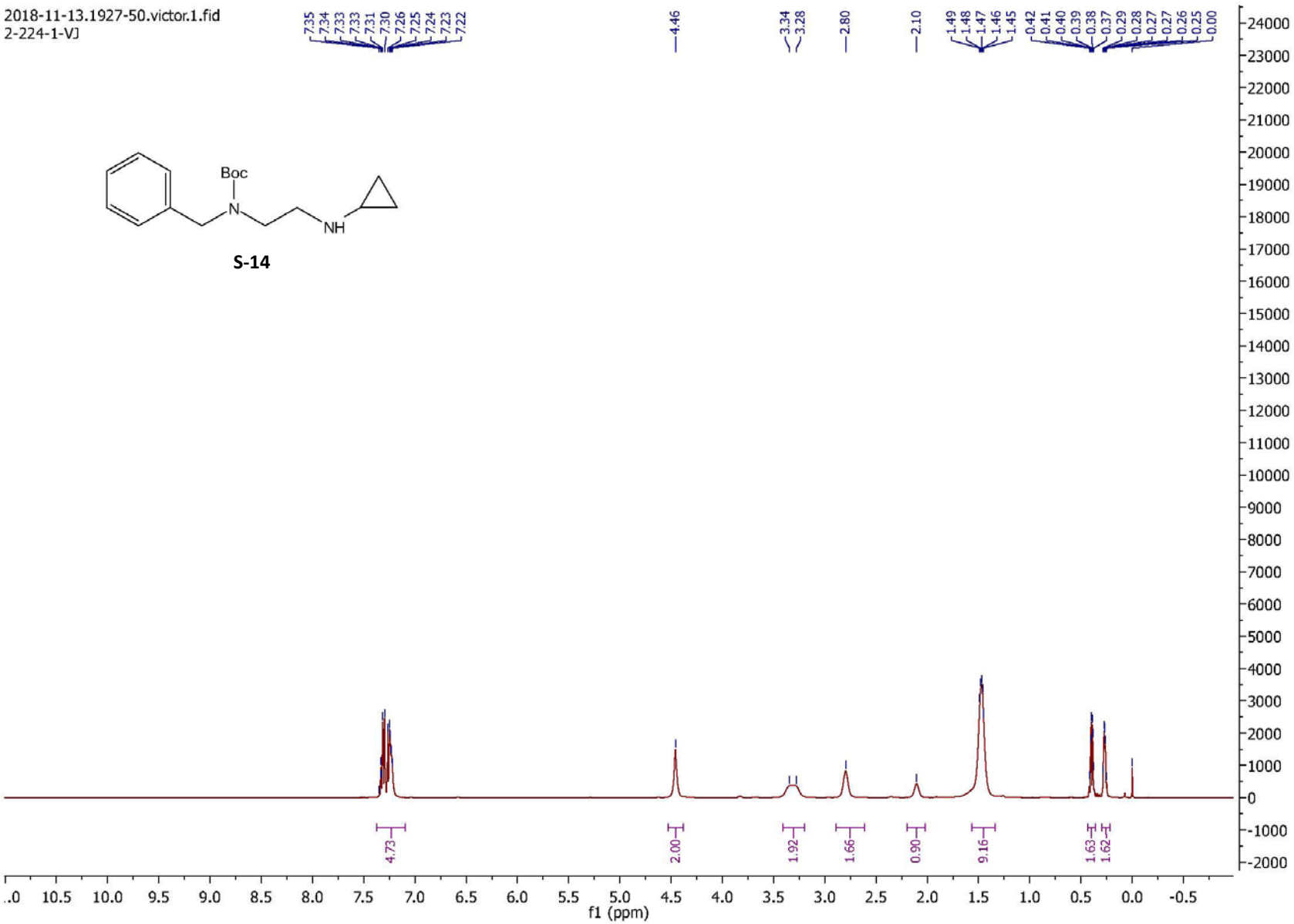
76.7 CDCl3
0.0
NC
O
OH
N3
S-8
f1 (ppm)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7.25
7.24
7.23
7.22
4.46
3.34
3.28
2.80
2.10
1.49
1.48
1.47
1.46
1.45
0.42
0.41
0.40
0.39
0.38
0.37
0.29
0.28
0.27
0.27
0.26
0.25
0.00
Boc
N
NH
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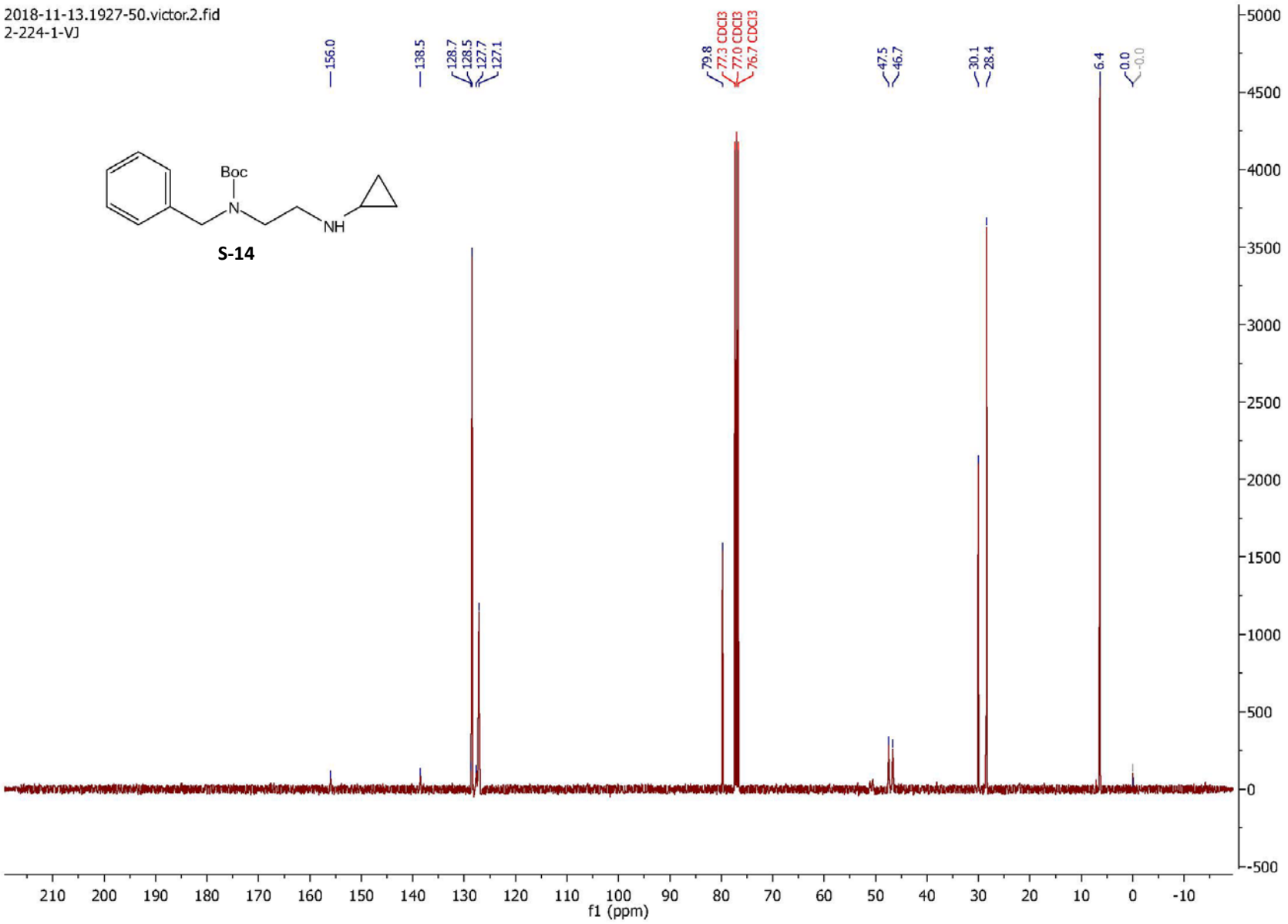
2.00
1.92
1.66
0.90
9.16
1.63
1.62
f1 (ppm)

S-14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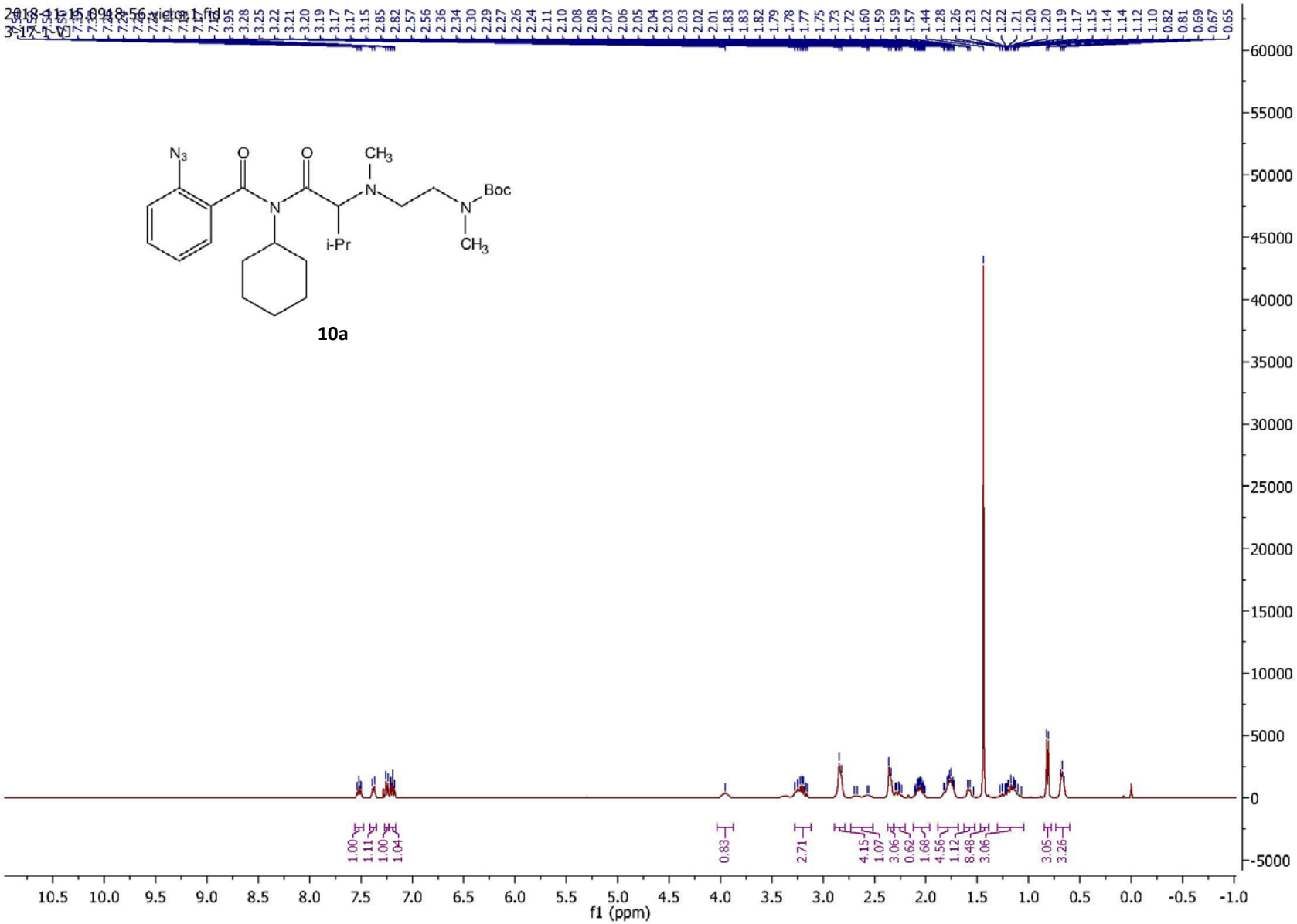
128.5
127.7
127.1
79.8
77.3 CDCl3
77.0 CDCl3
76.7 CDCl3
47.5
46.7
30.1
28.4
6.4
0.0
-0.0
Boc
N
NH
S-14
f1 (ppm)



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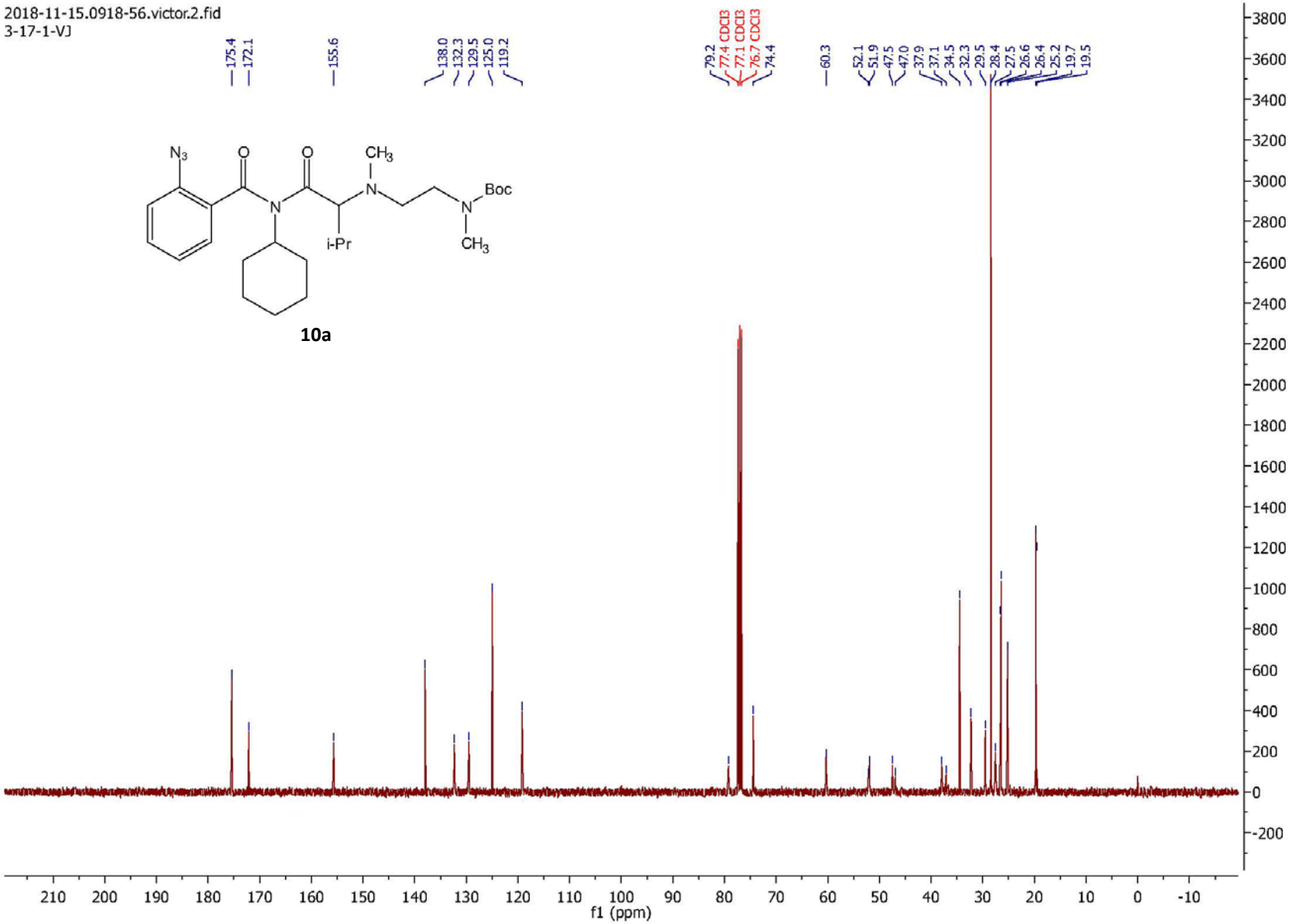
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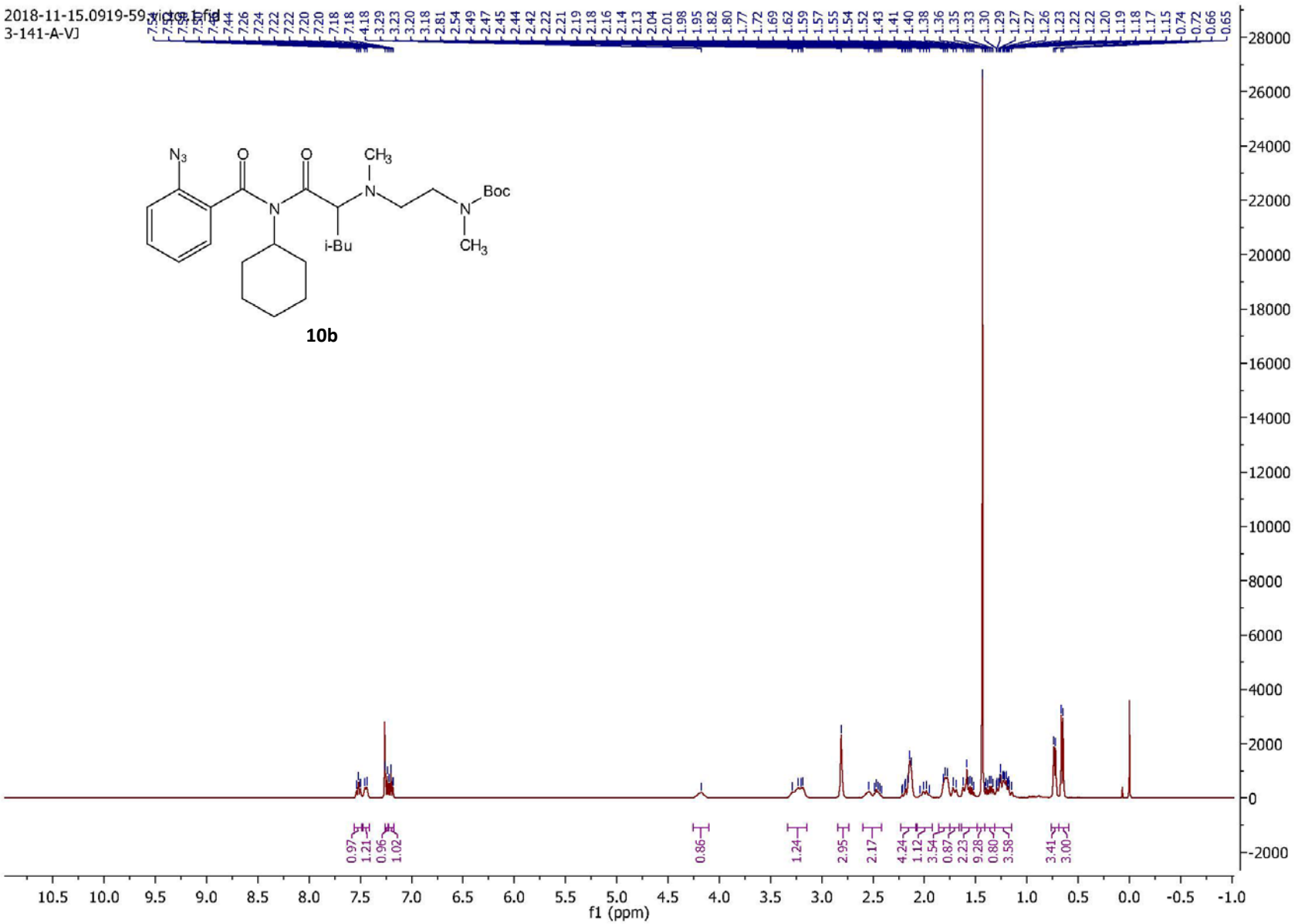
77.1 CDCl3
76.7 CDCl3
74.4
60.3
52.1
51.9
47.5
47.0
37.9
37.1
34.5
32.3
29.5
28.4
27.5
26.6
26.4
25.2
19.7
19.5
N3
O
O
CH3
N
Boc
N
i-Pr
CH3
f1 (ppm)

10a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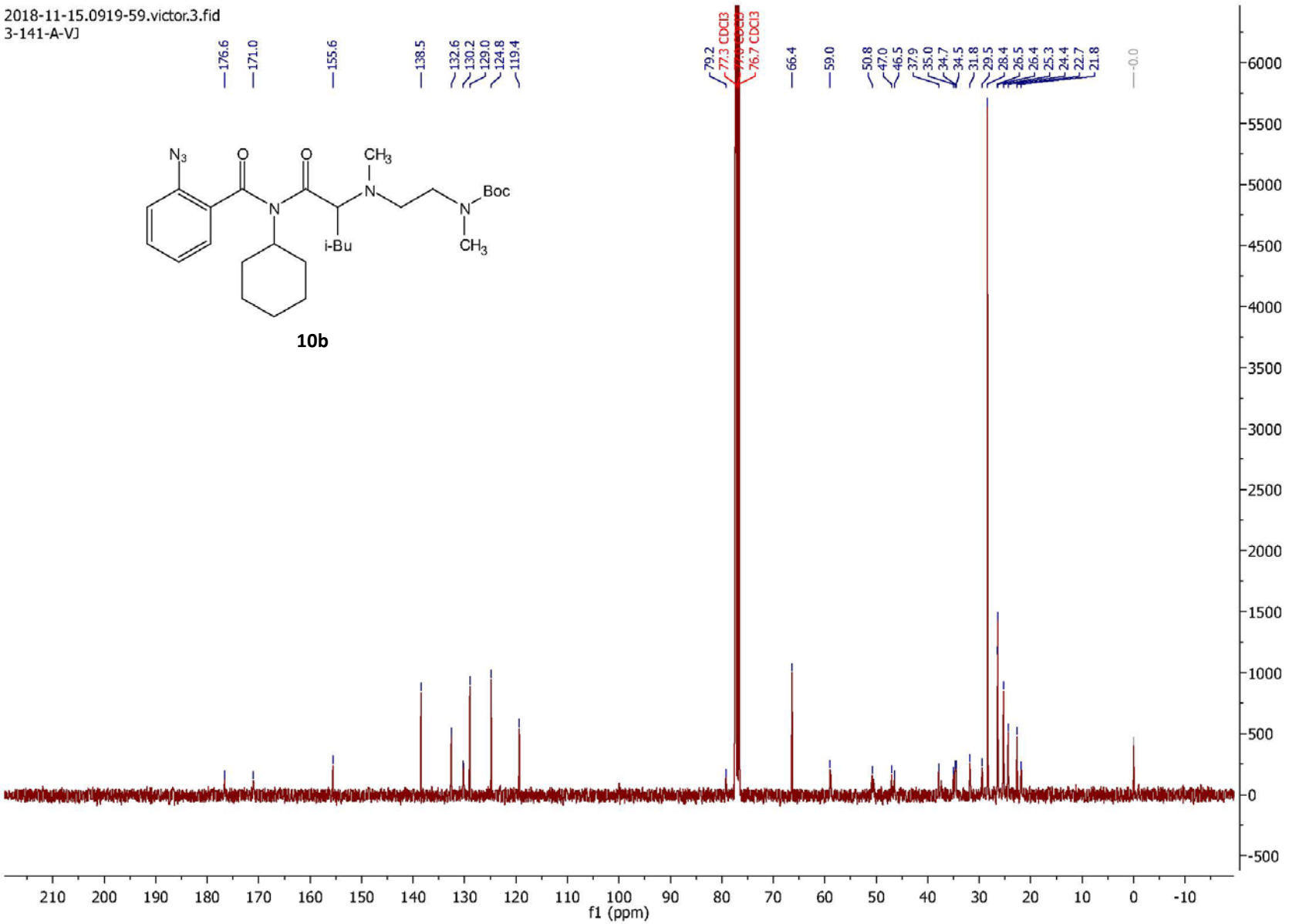
N
Boc
N
i-Bu
CH3
10b
f1 (ppm)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132.6
130.2
129.0
124.8
119.4
79.2
77.3 CDCl3
76.7 CDCl3
66.4
59.0
50.8
47.0
46.5
37.9
35.0
34.7
34.5
31.8
29.5
28.4
26.5
26.4
25.3
24.4
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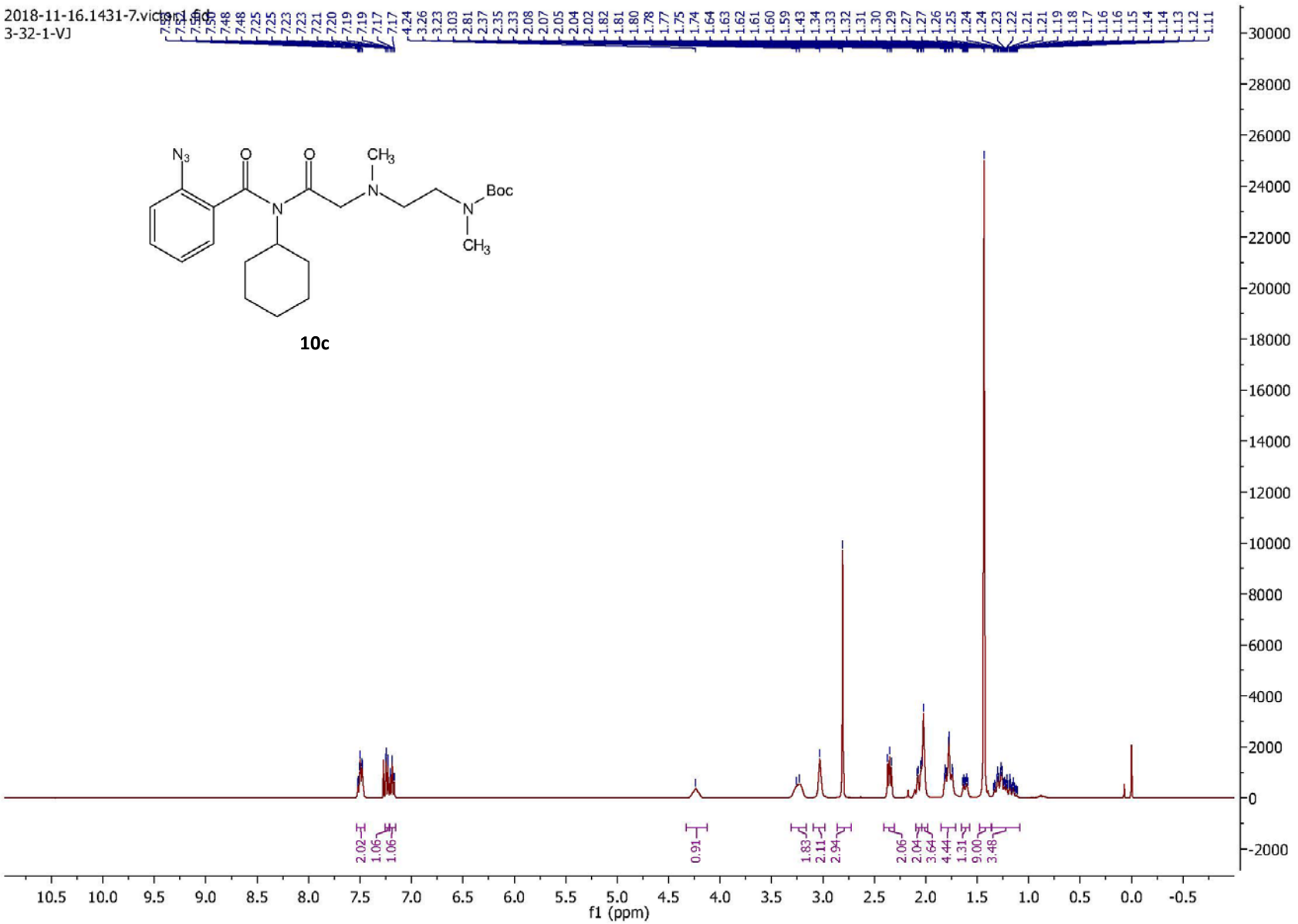
0.0
N3
O
O
CH3
N
N
Boc
i-Bu
CH3
f1 (ppm)

10b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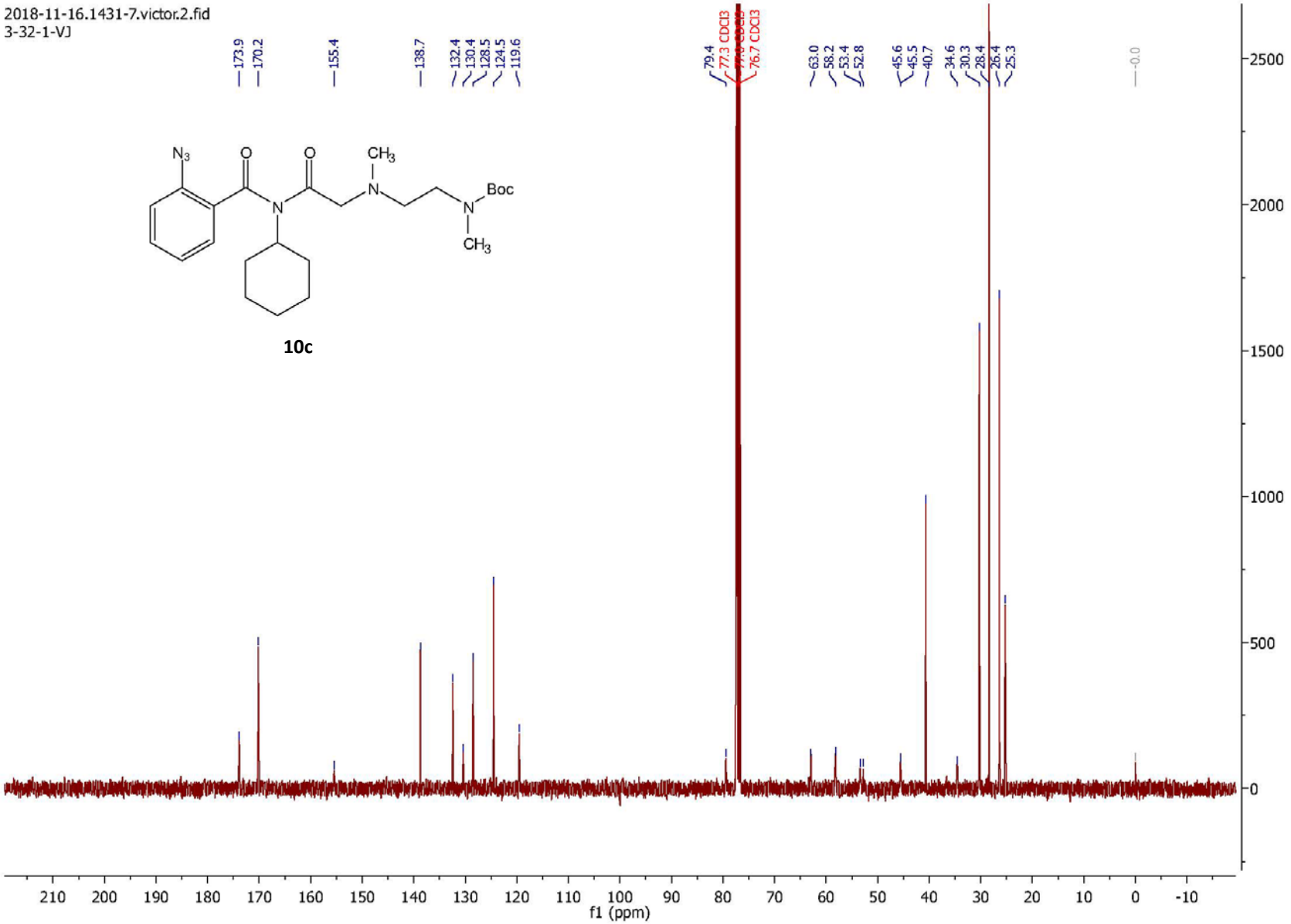
N
N
Boc
N
CH3
10c
f1 (ppm)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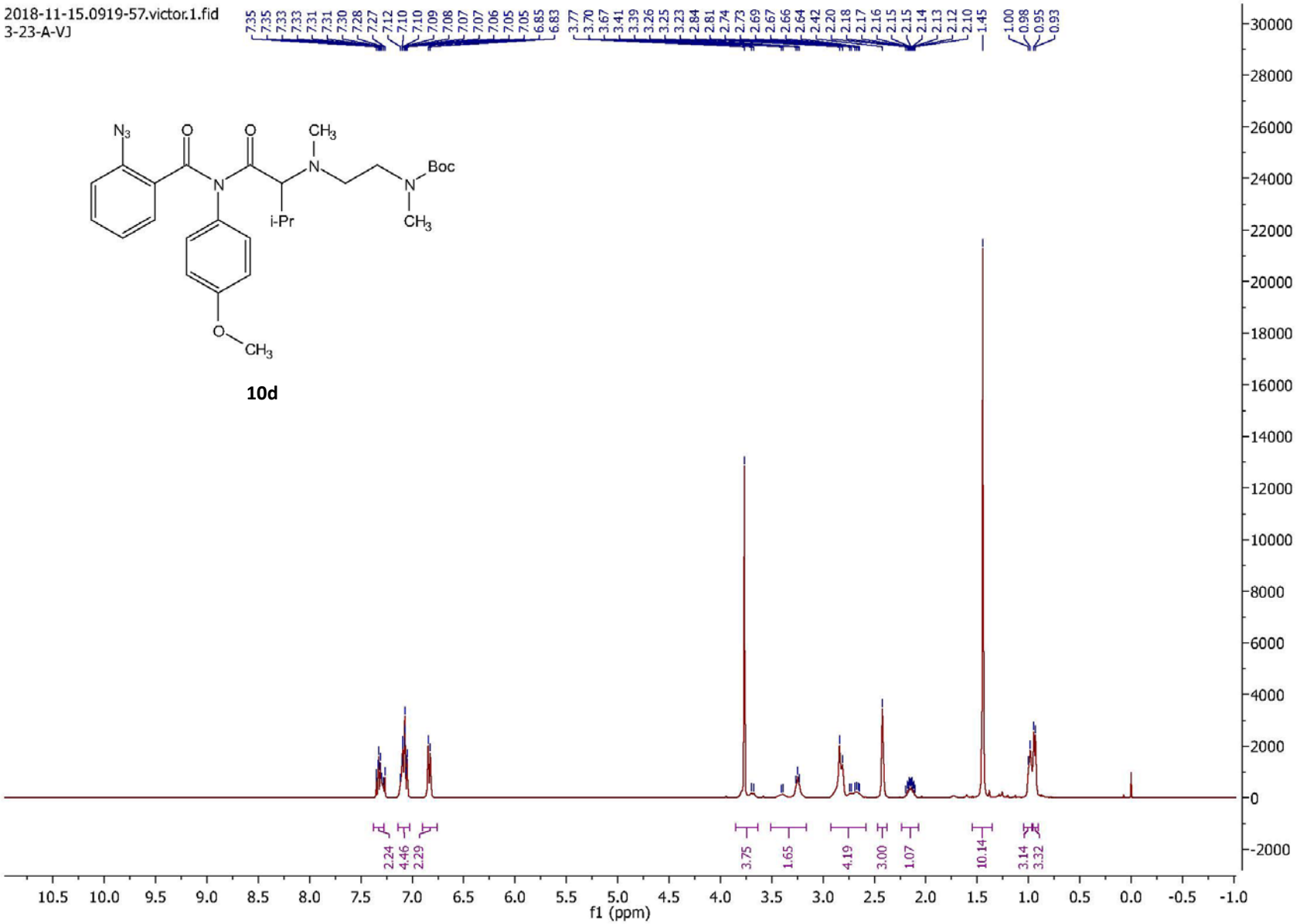
79.4
77.3 CDCl3
77.0 CDCl3
76.7 CDCl3
63.0
58.2
53.4
52.8
45.6
45.5
40.7
34.6
30.3
28.4
26.4
25.3
0.0
N3
O
O
CH3
N
N
Boc
N
CH3
2500
2000
1500
1000
500
0
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

10c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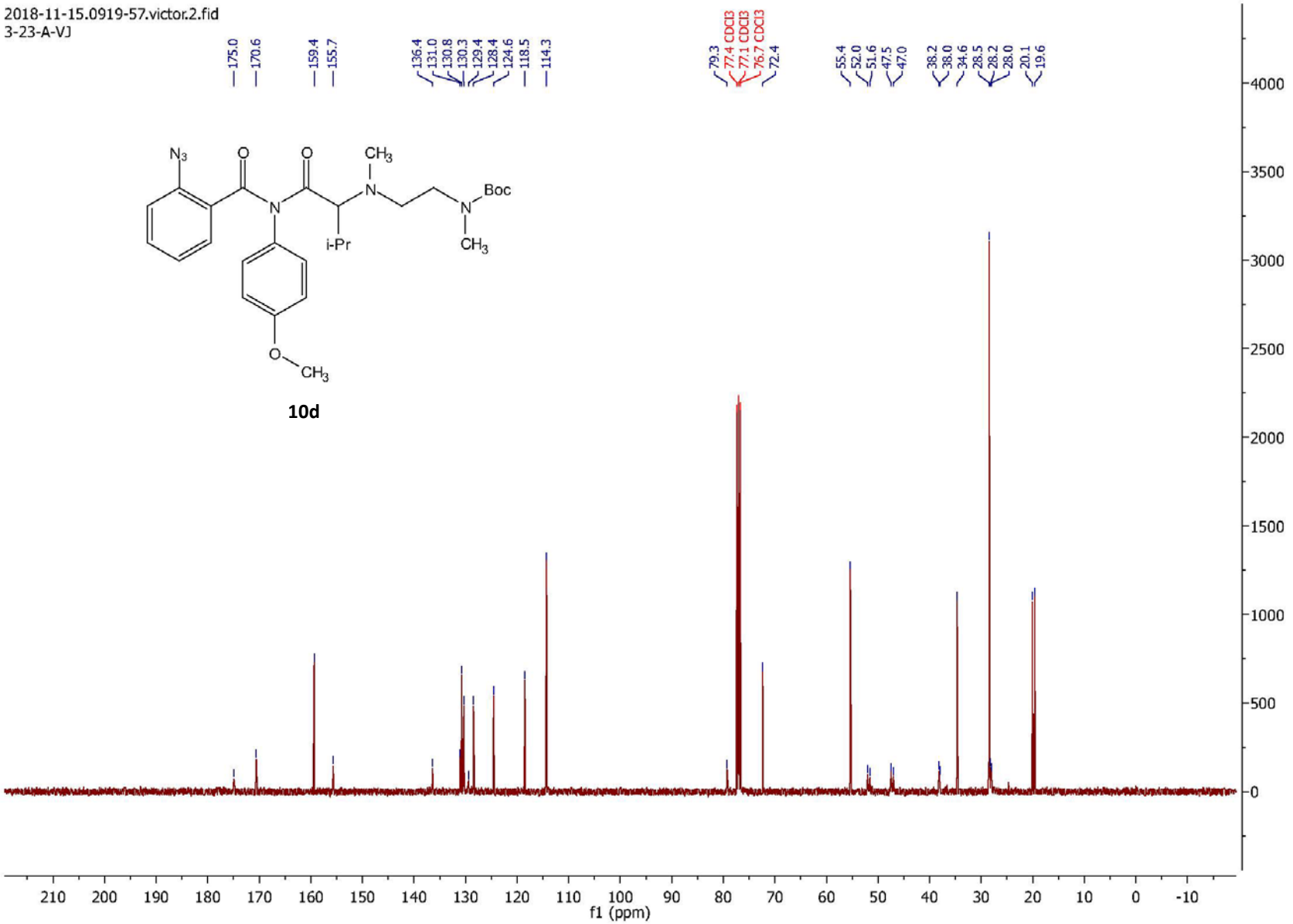
3.77 3.70 3.67 3.41 3.39 3.26 3.25 3.23 2.84 2.81 2.74 2.73 2.69 2.67 2.66 2.64 2.42 2.20 2.18 2.17 2.16 2.15 2.15 2.14 2.13 2.12 2.10 1.45
1.00 0.98 0.95 0.93
N3 O O CH3 N Boc N i-Pr CH3 O CH3
2.24 4.46 2.29 3.75 1.65 4.19 3.00 1.07 10.14 3.14 3.32
f1 (ppm)

10d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130.3
129.4
128.4
124.6
118.5
114.3
79.3
77.4 CDCl3
77.1 CDCl3
76.7 CDCl3
72.4
55.4
52.0
51.6
47.5
47.0
38.2
38.0
34.6
28.5
28.2
28.0
20.1
19.6
N3
O
O
CH3
N
N
Boc
N
i-Pr
CH3
O
CH3
10d
4000
3500
3000
2500
2000
1500
1000
500
0
210
200
190
180
170
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120
110
100
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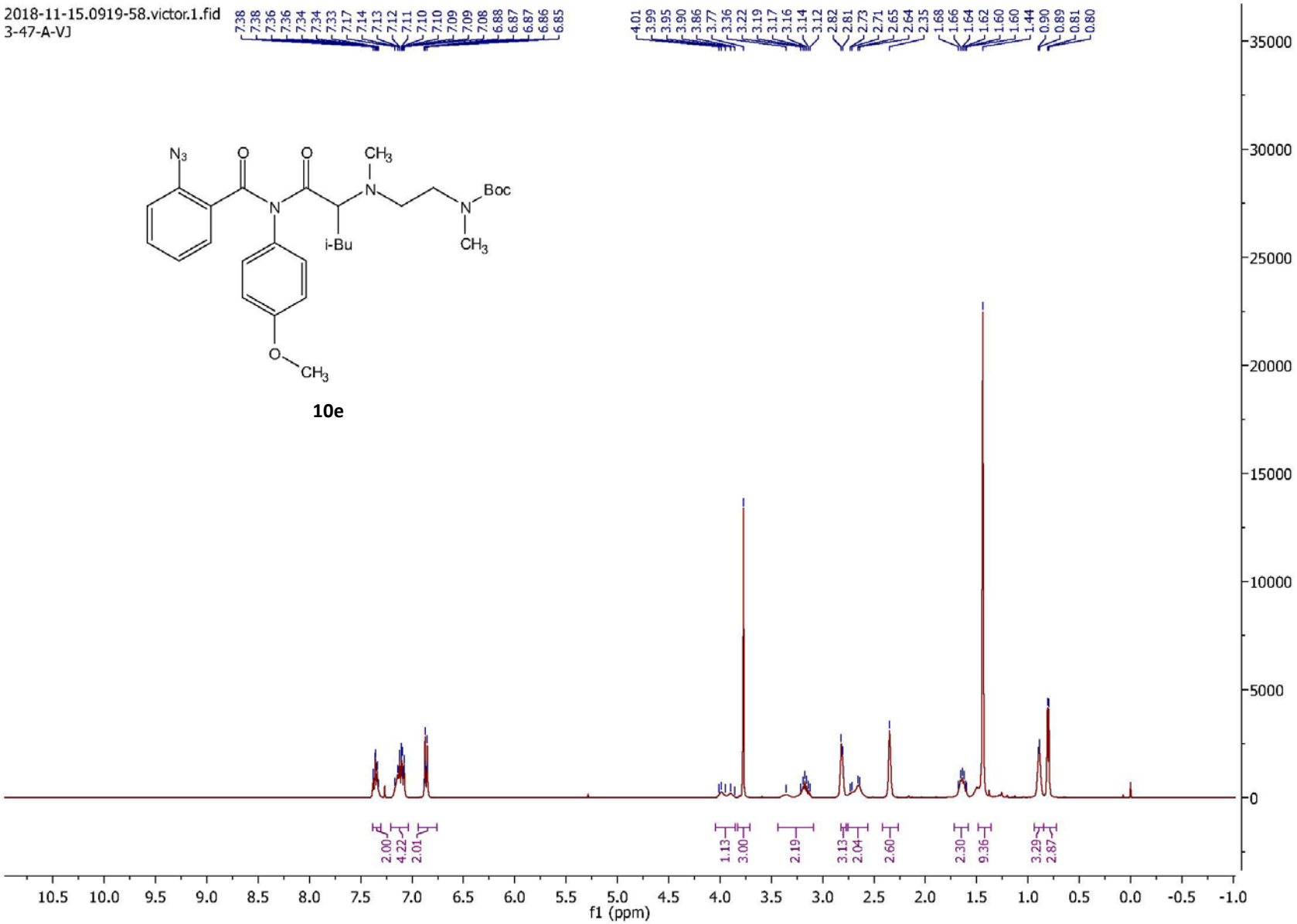
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)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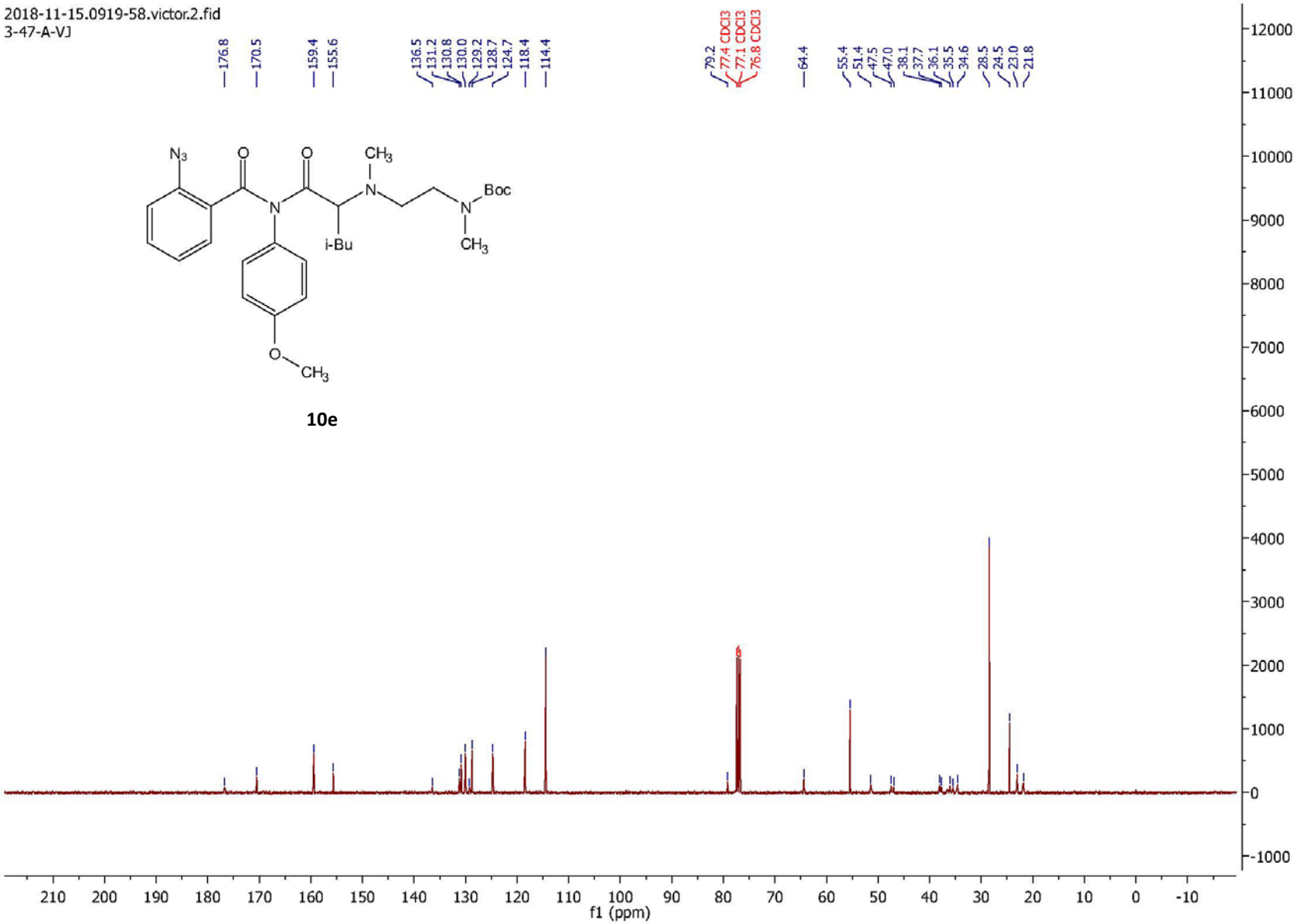
4.01 3.99 3.95 3.90 3.86 3.77 3.36 3.22 3.19 3.17 3.16 3.14 3.12 2.82 2.81 2.73 2.71 2.65 2.64 2.35 1.68 1.66 1.64 1.62 1.60 1.60 1.44 0.90 0.89 0.81 0.80
N3
O
O
CH3
N
N
Boc
i-Bu
N
CH3
O
CH3
35000
30000
25000
20000
15000
10000
5000
0
2.00 4.22 2.01 1.13 3.00 2.19 3.13 2.04 2.60 2.30 9.36 3.29 2.87
10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0
f1 (ppm)

10e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130.8
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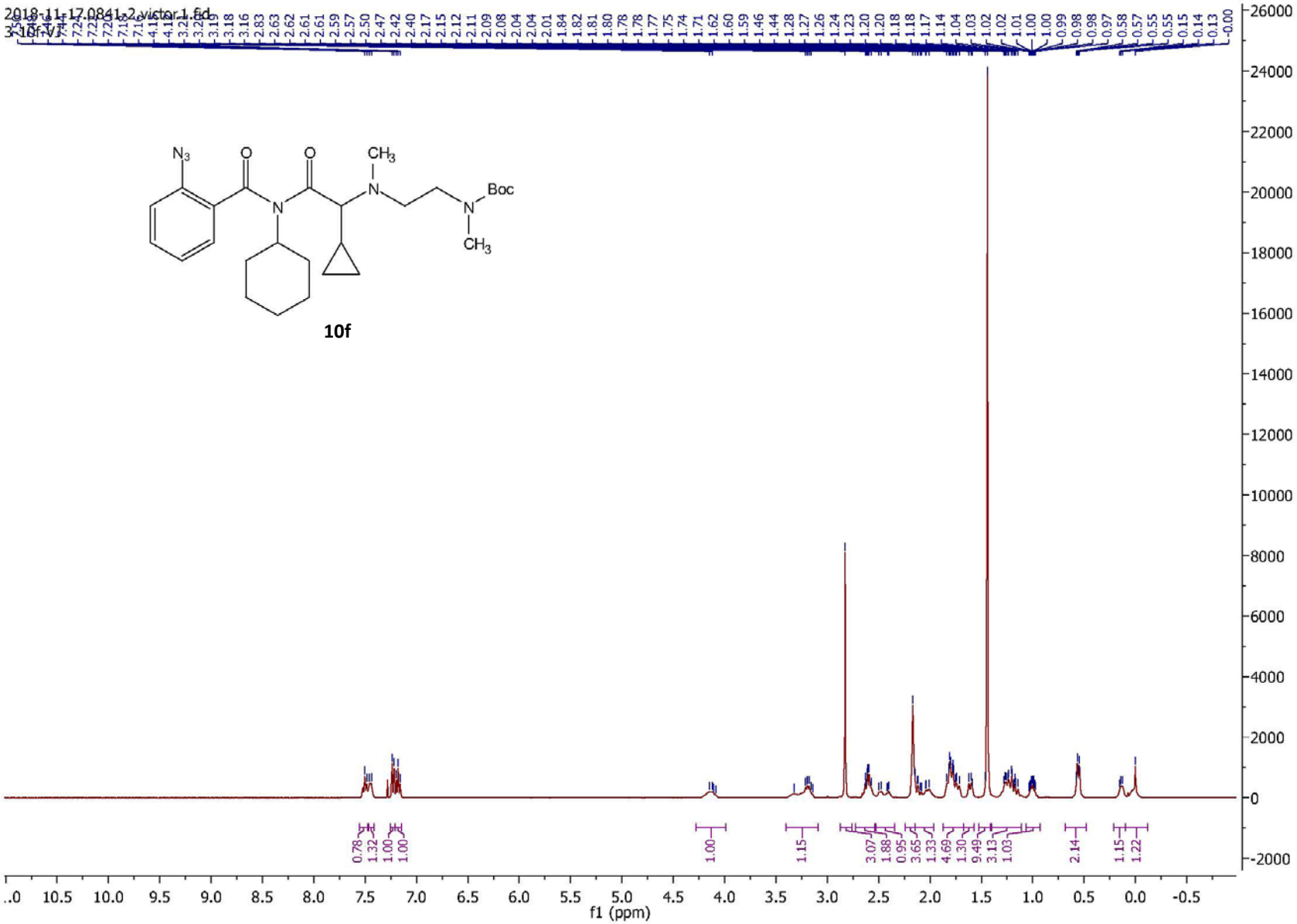
129.2
128.7
124.7
118.4
114.4
79.2
77.4 CDCl3
77.1 CDCl3
76.8 CDCl3
64.4
55.4
51.4
47.5
47.0
38.1
37.7
36.1
35.5
34.6
28.5
24.5
23.0
21.8
N3
O
O
CH3
N
N
Boc
i-Bu
N
CH3
O
CH3
f1 (ppm)

10e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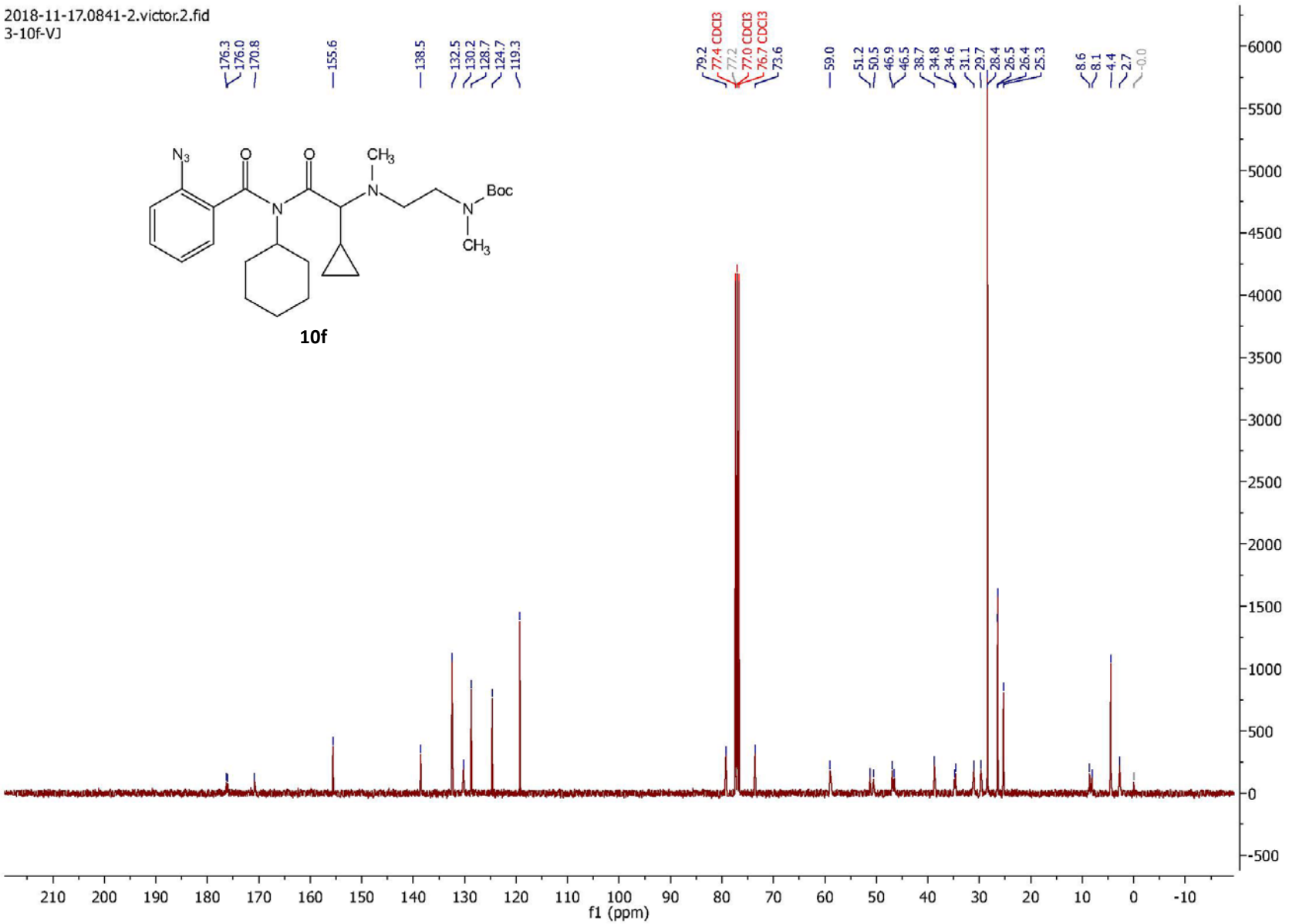
O
CH3
N
N
Boc
N
CH3
10f
f1 (ppm)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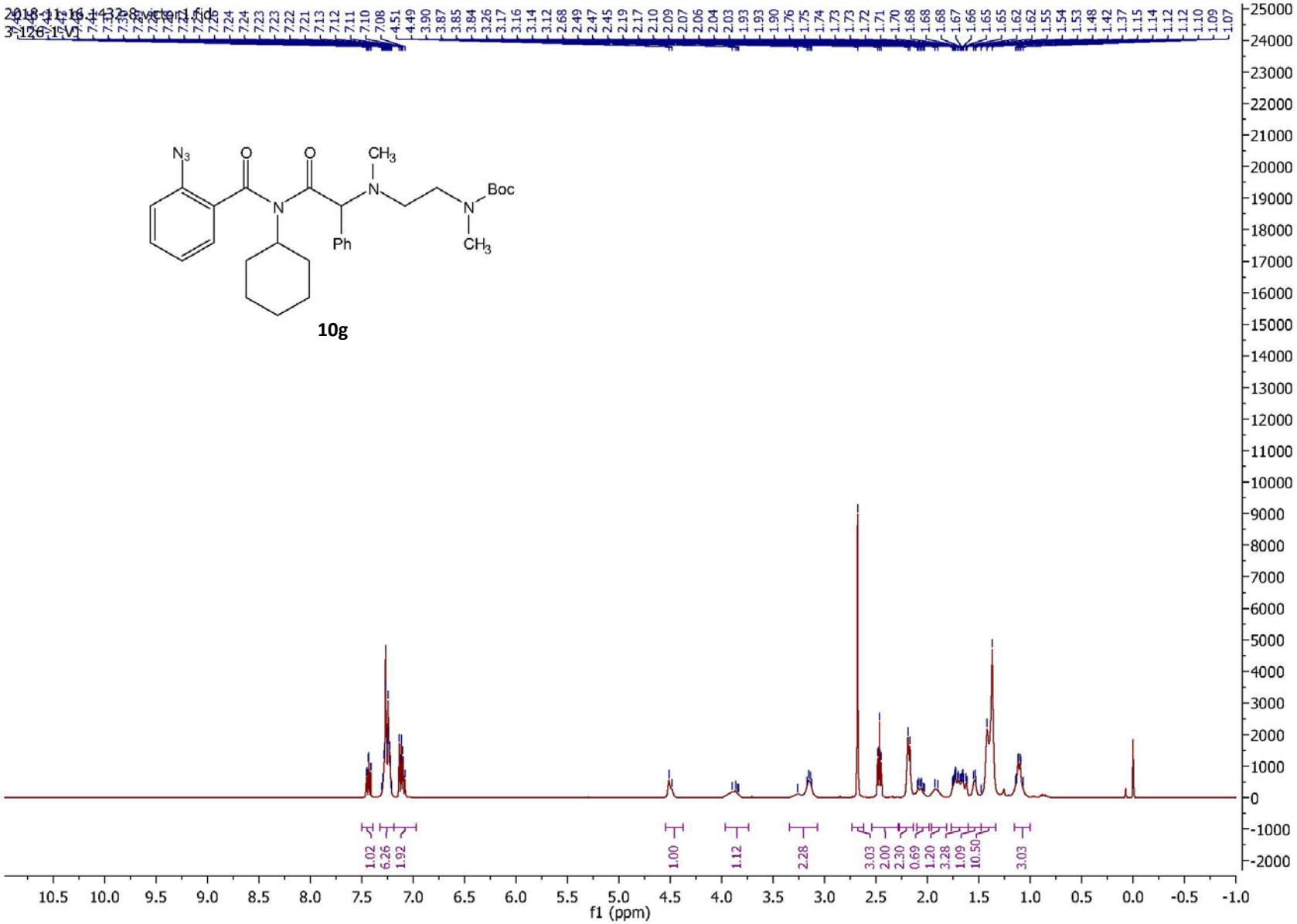
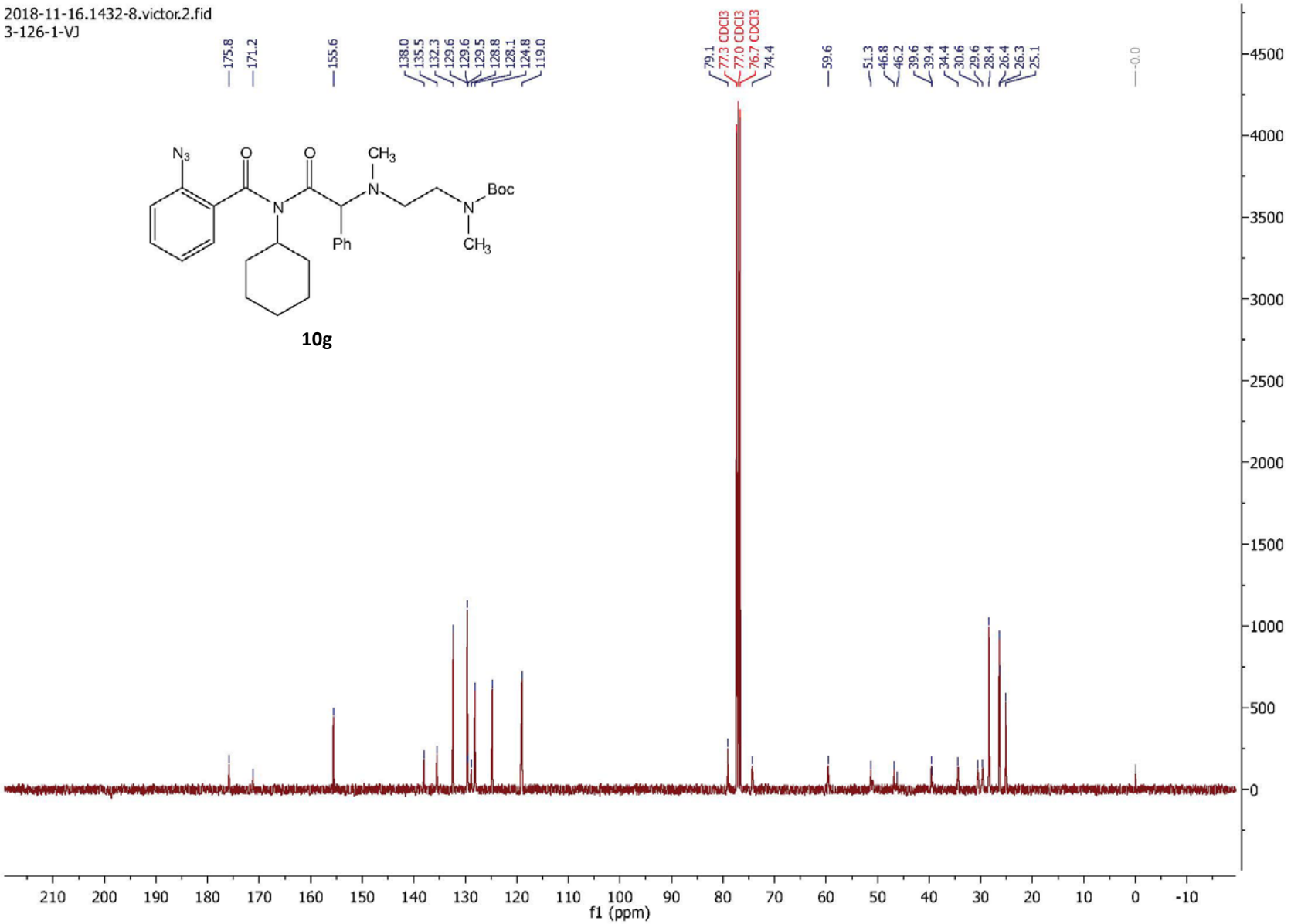
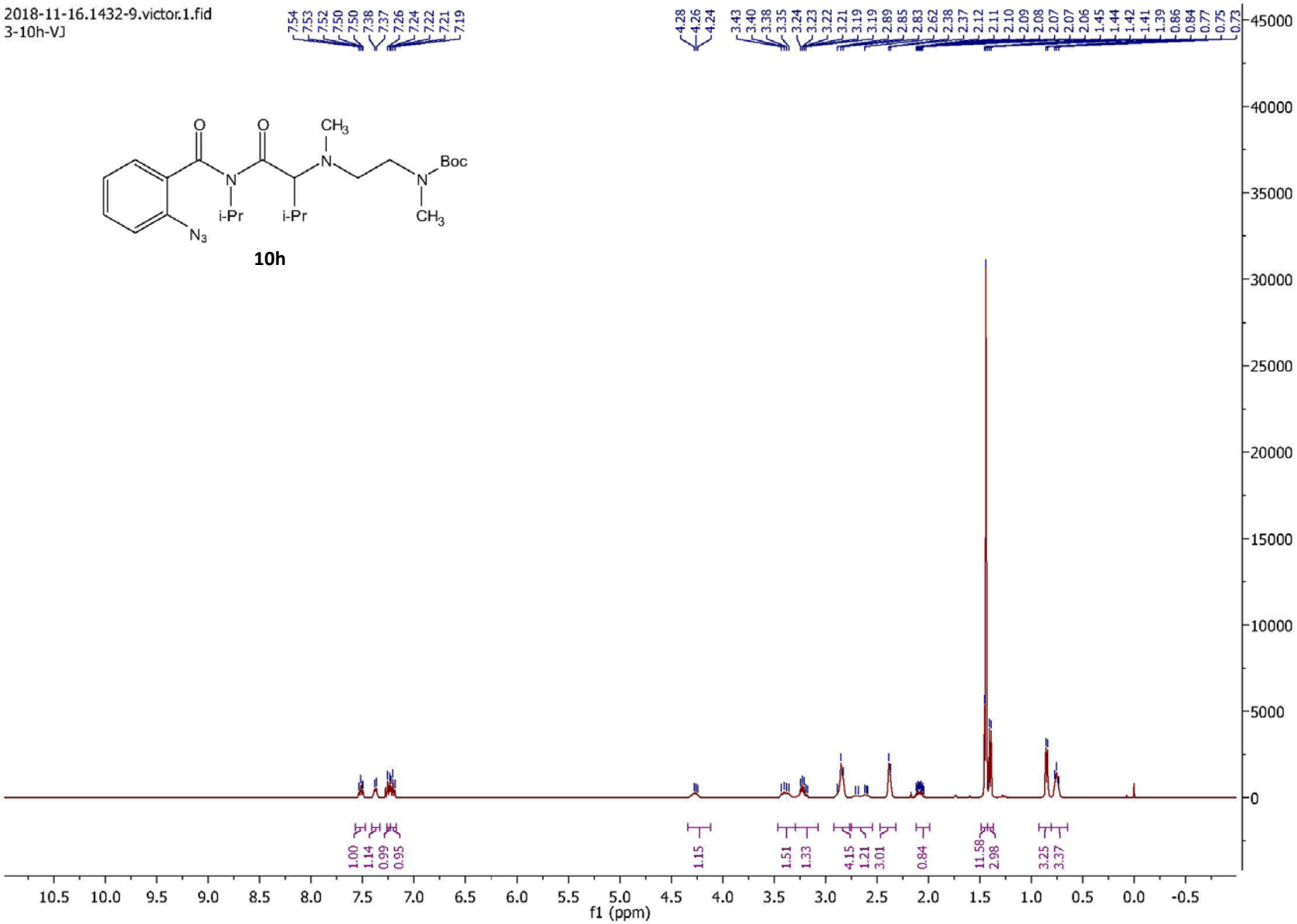
124.7
119.3
79.2
77.4 CDCl3
77.2
77.0 CDCl3
76.7 CDCl3
73.6
59.0
51.2
50.5
46.9
46.5
38.7
34.8
34.6
31.1
29.7
28.4
26.5
26.4
25.3
8.6
8.1
4.4
2.7
-0.0
N3
O
O
CH3
N
N
Boc
N
CH3
6000
5500
5000
4500
4000
3500
3000
2500
2000
1500
1000
500
0
-500
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

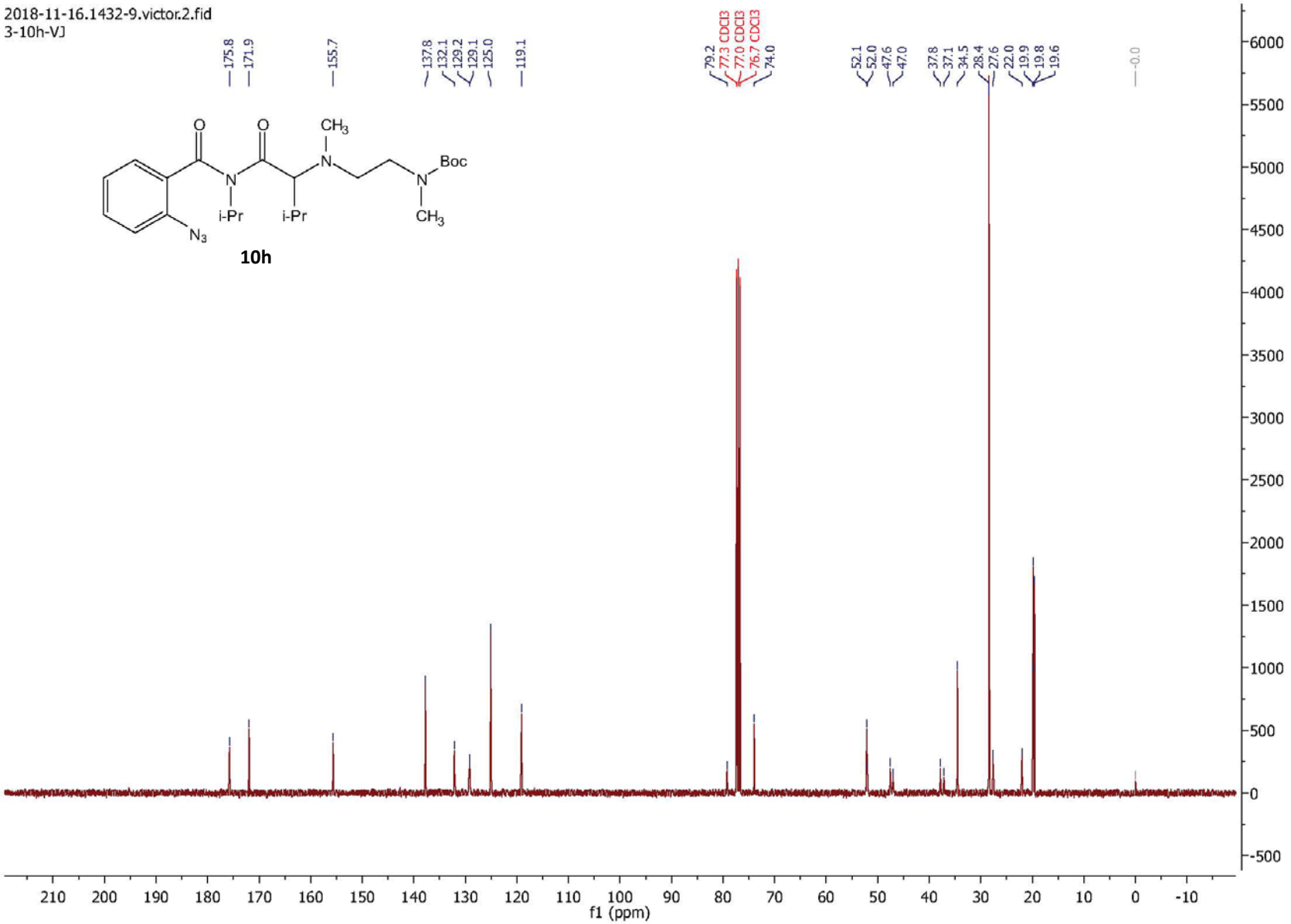
10f


2018-11-16-14328 victor1.fid
3-126-1-V1
N3
O
O
CH3
N
Boc
N
Ph
CH3
10g
f1 (ppm)

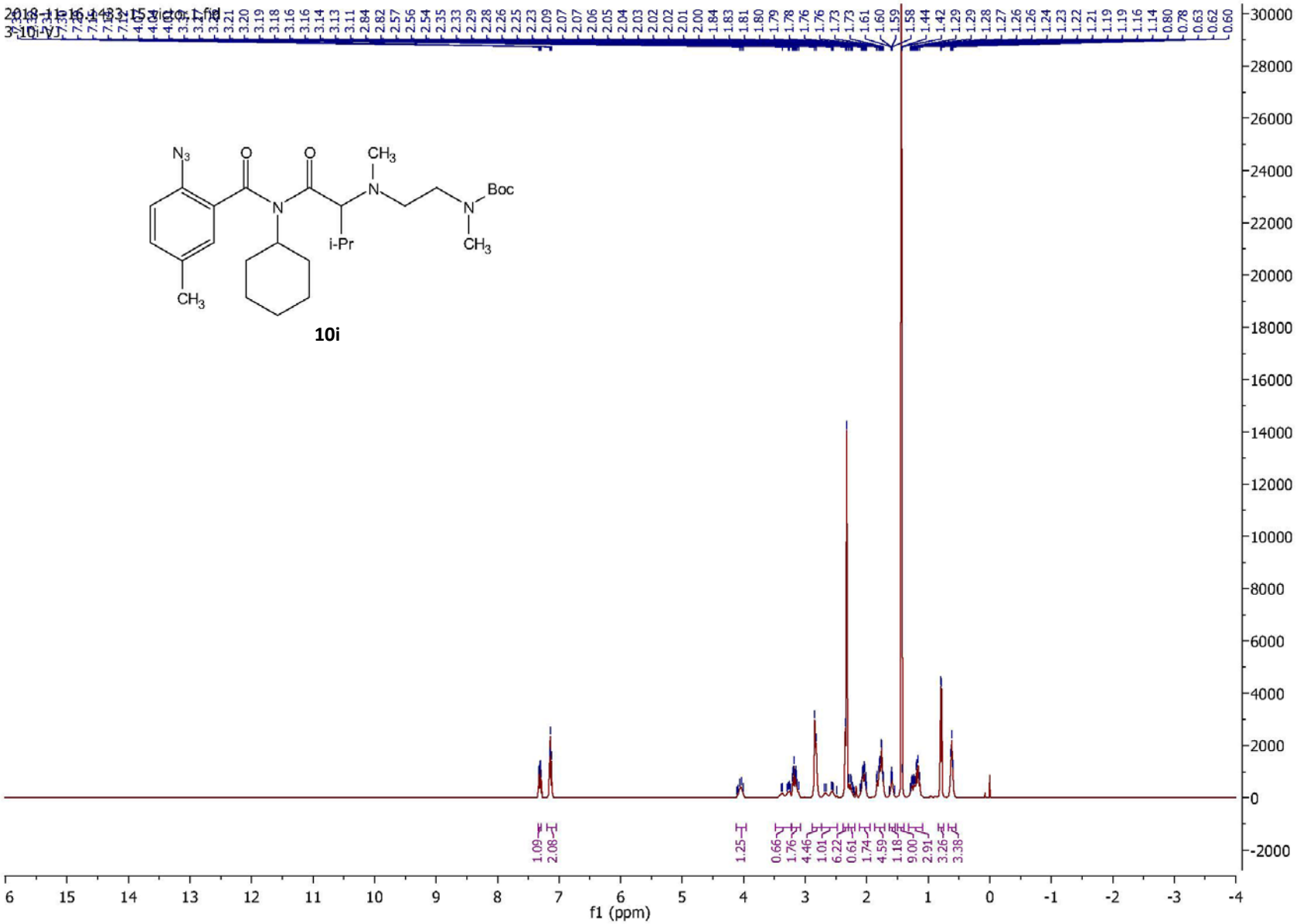

2018-11-16.1432-8.victor.2.fid
3-126-1-VJ
175.8
171.2
155.6
138.0
135.5
132.3
129.6
129.6
129.5
128.8
128.1
124.8
119.0
79.1
77.3 CDCl3
77.0 CDCl3
76.7 CDCl3
74.4
59.6
51.3
46.8
46.2
39.6
39.4
34.4
30.6
29.6
28.4
26.4
26.3
25.1
0.0
N3
O
O
CH3
N
N
Boc
Ph
N
CH3
4500
4000
3500
3000
2500
2000
1500
1000
500
0
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

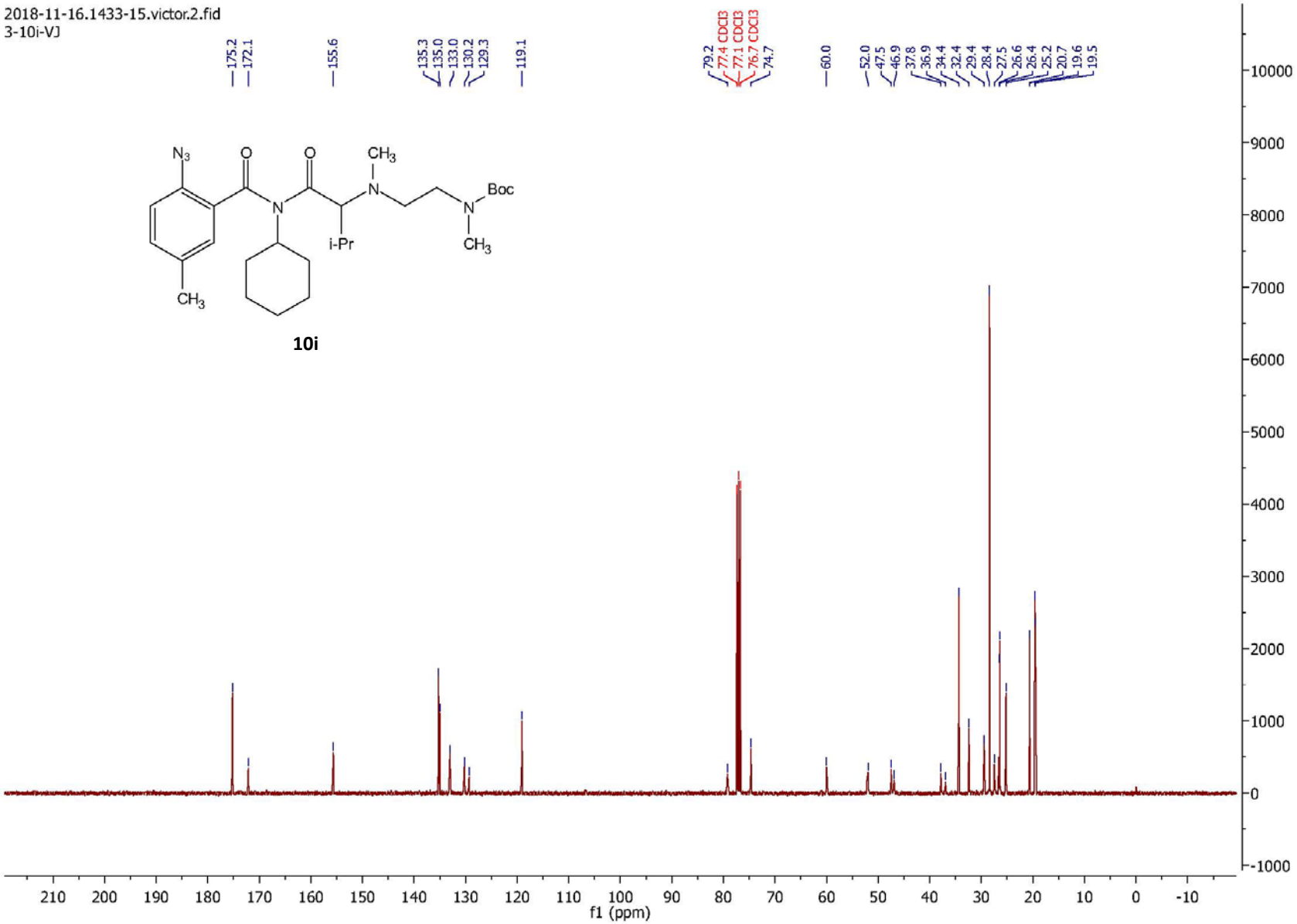
10g


2018-11-16.1432-9.victor.1.fid
3-10h-VJ
10h
f1 (ppm)

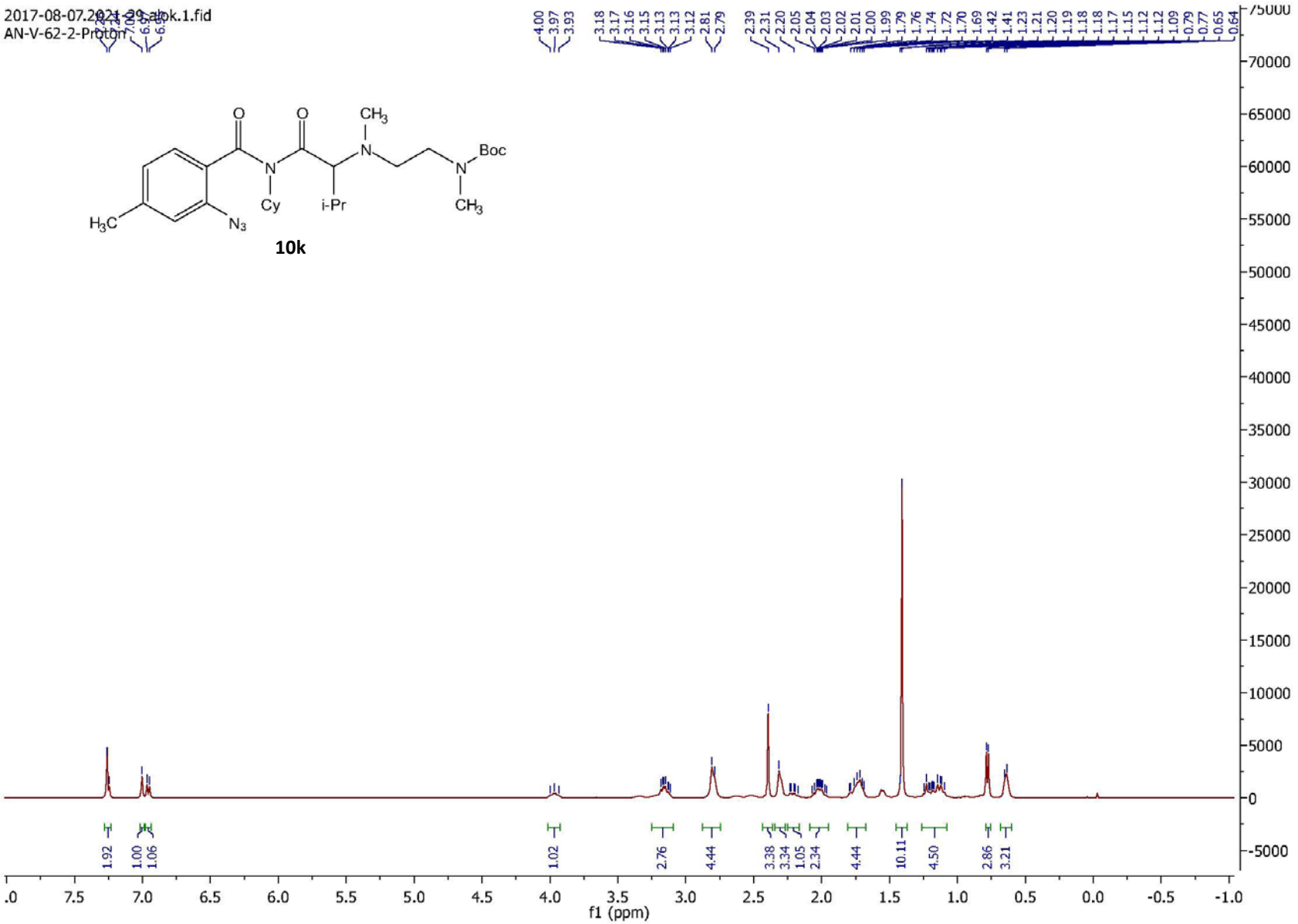

2018-11-16.1432-9.victor.2.fid
3-10h-VJ
175.8
171.9
155.7
137.8
132.1
129.2
129.1
125.0
119.1
79.2
77.3 CDCl3
77.0 CDCl3
76.7 CDCl3
74.0
52.1
52.0
47.6
47.0
37.8
37.1
34.5
28.4
27.6
22.0
19.9
19.8
19.6
0.0
O
O
CH3
N
N
Boc
N
i-Pr
i-Pr
CH3
N3
f1 (ppm)

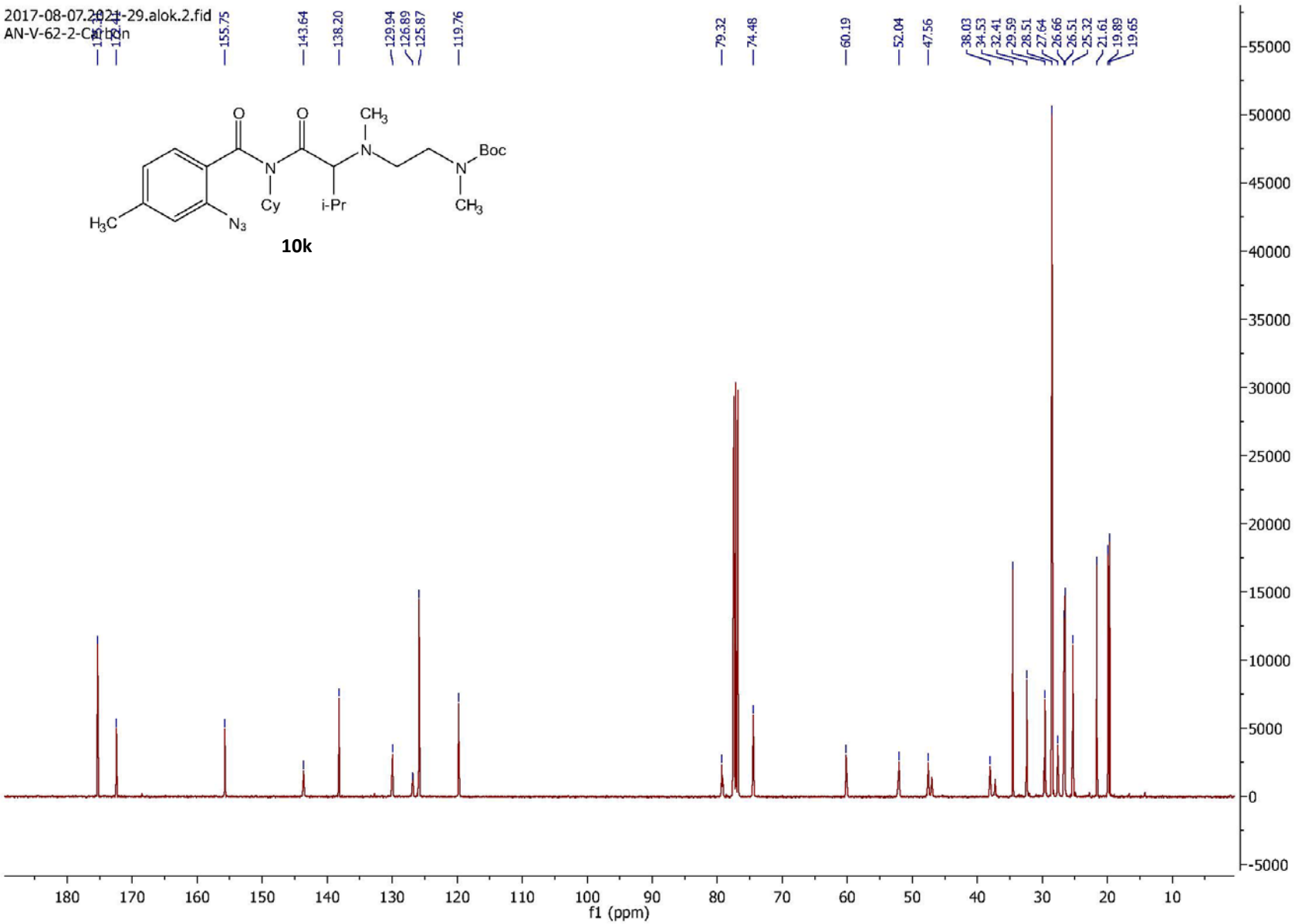
10h

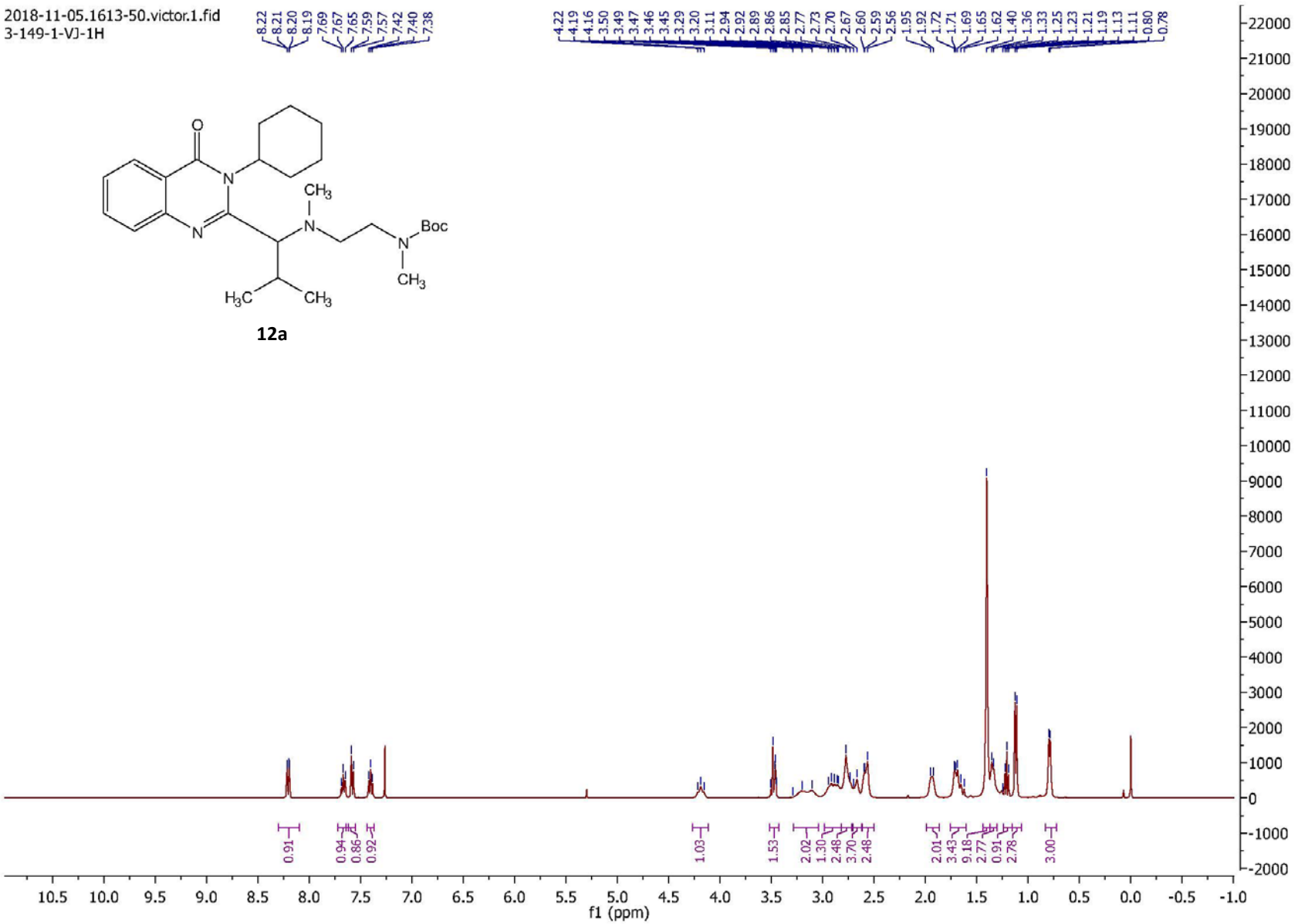

N3
O
O
CH3
N
Boc
N
i-Pr
N
CH3
CH3
10i
f1 (ppm)


2018-11-16.1433-15.victor.2.fid
3-10i-VJ
175.2
172.1
155.6
135.3
135.0
133.0
130.2
129.3
119.1
79.2
77.4 CDCl3
77.1 CDCl3
76.7 CDCl3
74.7
60.0
52.0
47.5
46.9
37.8
36.9
34.4
32.4
29.4
28.4
27.5
26.6
26.4
25.2
20.7
19.6
19.5
N3
O
O
CH3
N
N
Boc
i-Pr
N
CH3
CH3
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
-1000
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

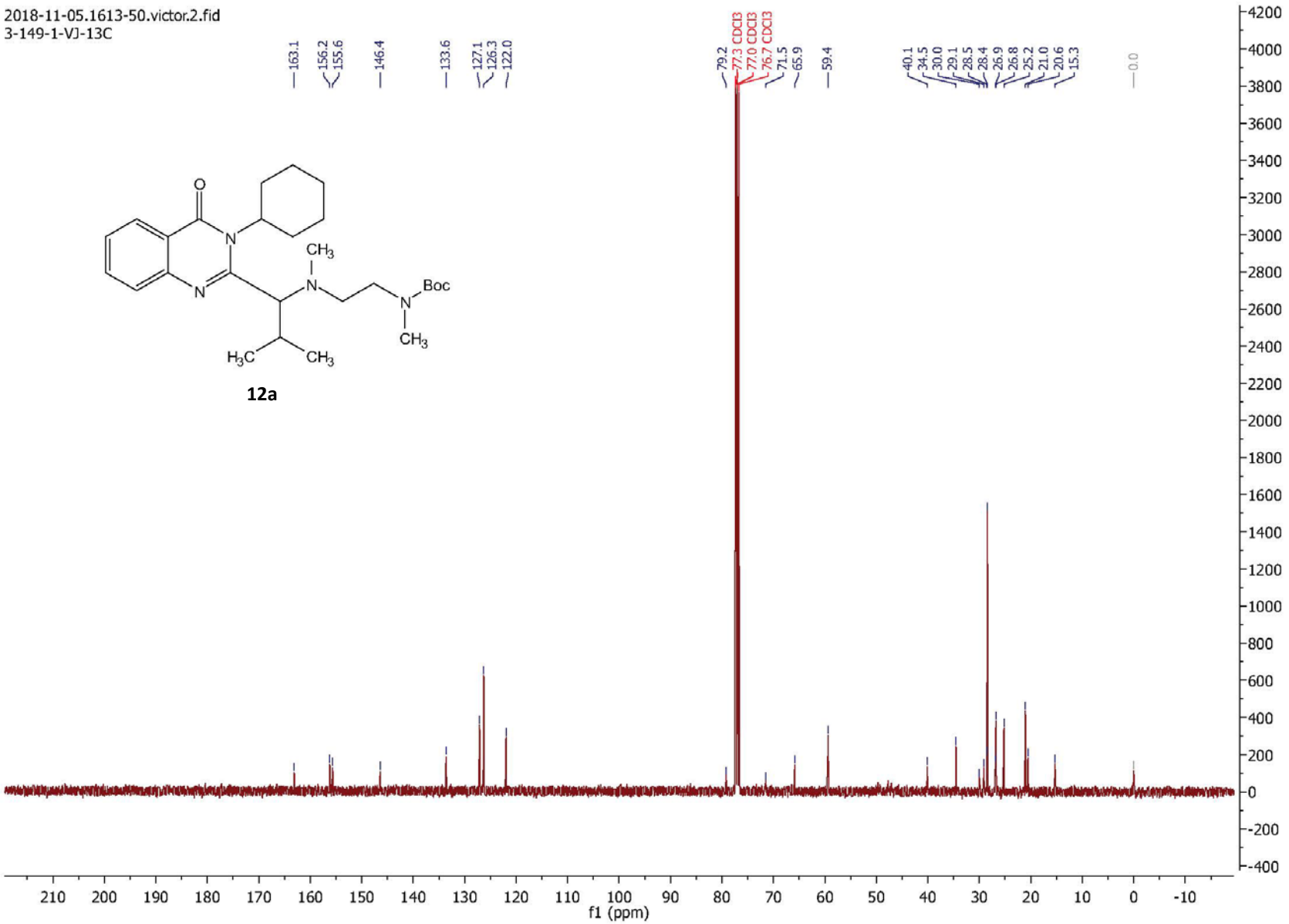
10i


2017-08-07.2021-29.alok.1.fid
AN-V-62-2-Proton
O
O
CH3
N
Boc
N
N
Cy
i-Pr
CH3
H3C
N3
10k
f1 (ppm)

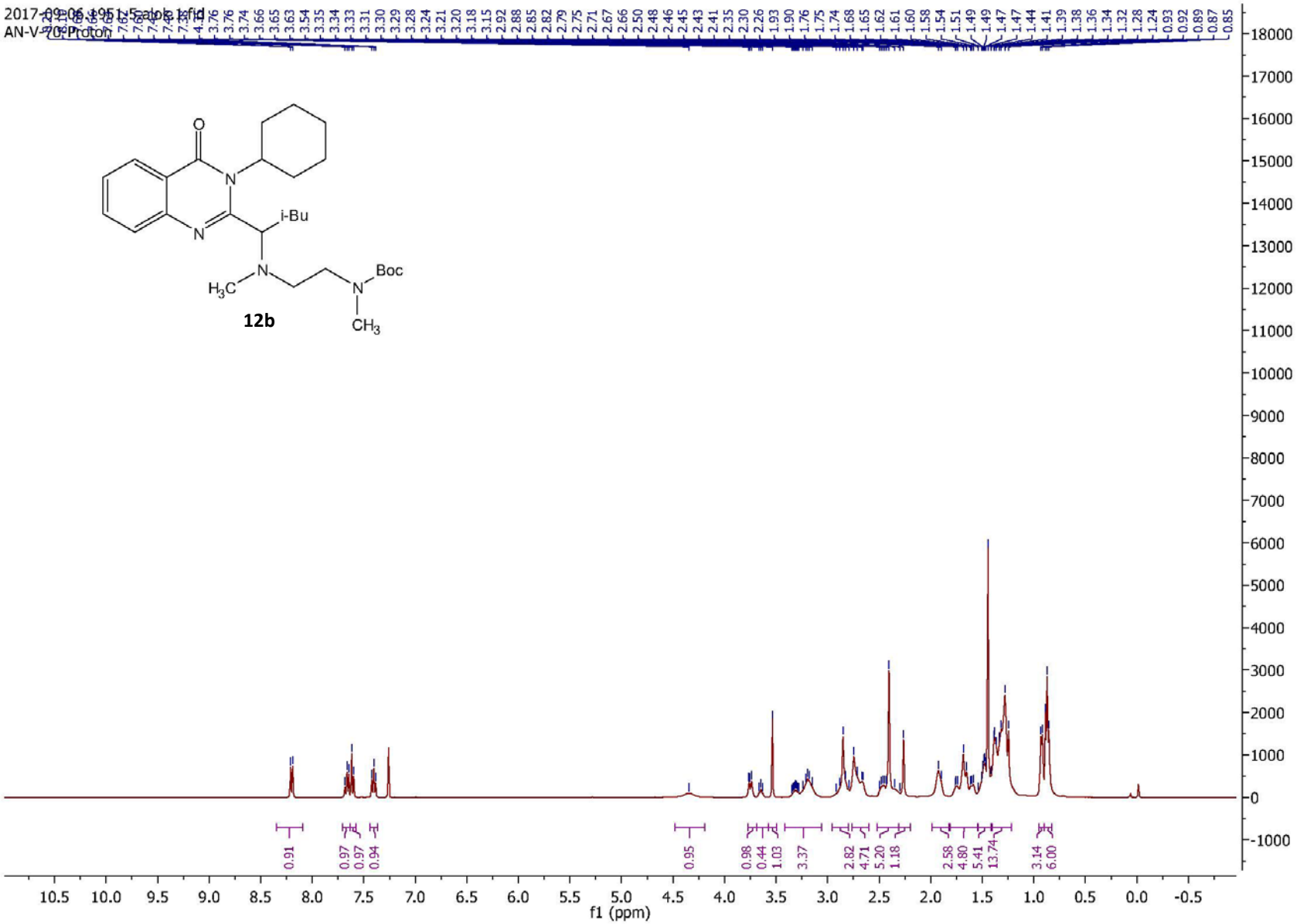

2017-08-07.2021-29.alok.2.fid
AN-V-62-2-Carbon
155.75
143.64
138.20
129.94
126.89
125.87
119.76
79.32
74.48
60.19
52.04
47.56
38.03
34.53
32.41
29.59
28.51
27.64
26.66
26.51
25.32
21.61
19.89
19.65
O
O
CH3
N
Boc
N
Cy
i-Pr
N
CH3
H3C
N3
10k
55000
50000
45000
40000
35000
30000
25000
20000
15000
10000
5000
0
-5000
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)

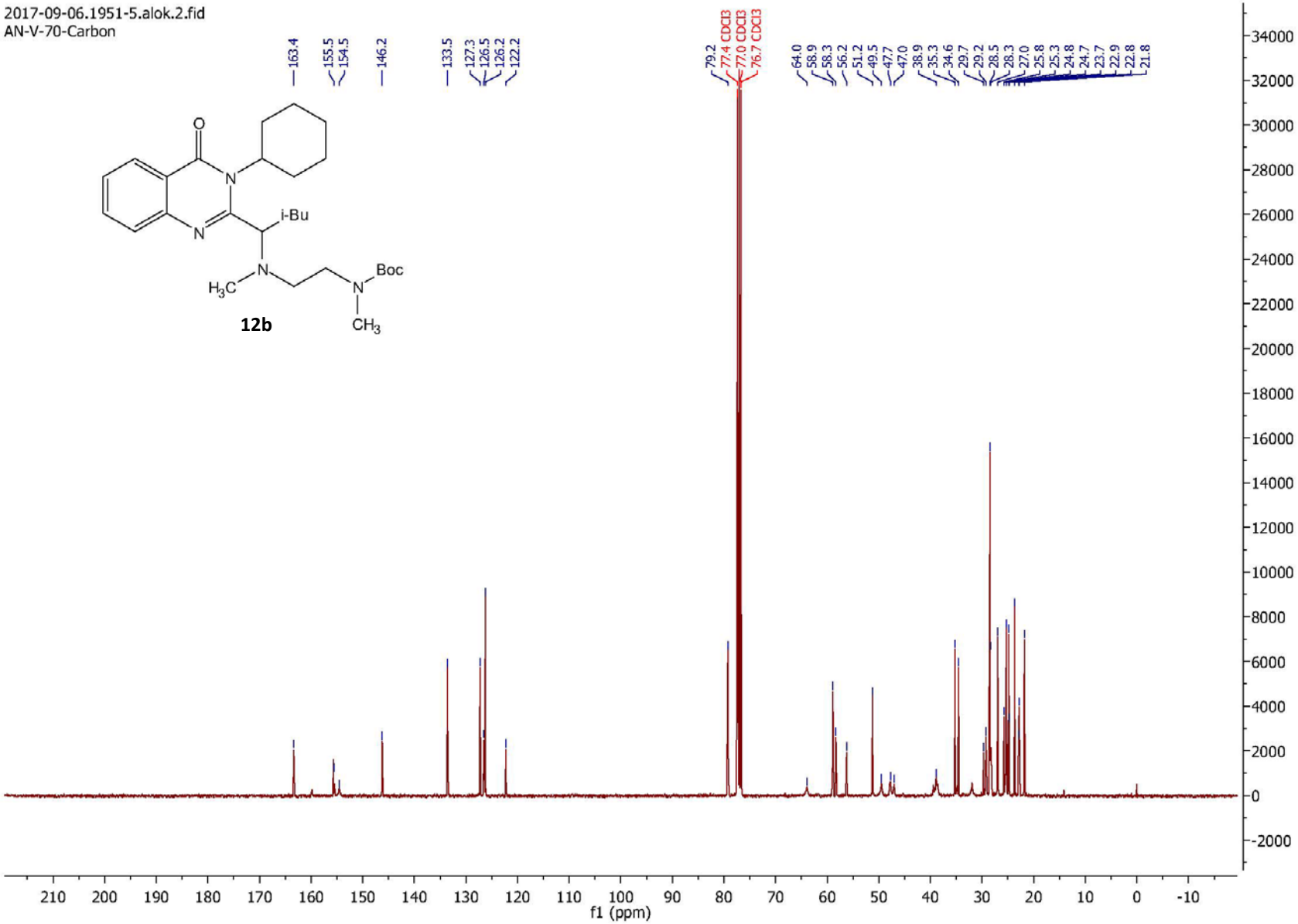

2018-11-05.1613-50.victor.1.fid
3-149-1-VJ-1H
8.22
8.21
8.20
8.19
7.69
7.67
7.65
7.59
7.57
7.42
7.40
7.38
4.22
4.19
4.16
3.50
3.49
3.47
3.46
3.45
3.29
3.20
3.11
2.94
2.92
2.89
2.86
2.85
2.77
2.73
2.70
2.67
2.60
2.59
2.56
1.95
1.92
1.72
1.71
1.69
1.65
1.62
1.40
1.36
1.33
1.25
1.23
1.21
1.19
1.13
1.11
0.80
0.78
O
N
N
CH3
N
Boc
N
CH3
H3C
CH3
0.91
0.94
0.86
0.92
1.03
1.53
2.02
1.30
2.48
3.70
2.48
2.01
3.43
9.18
2.77
0.91
2.78
3.00
f1 (ppm)

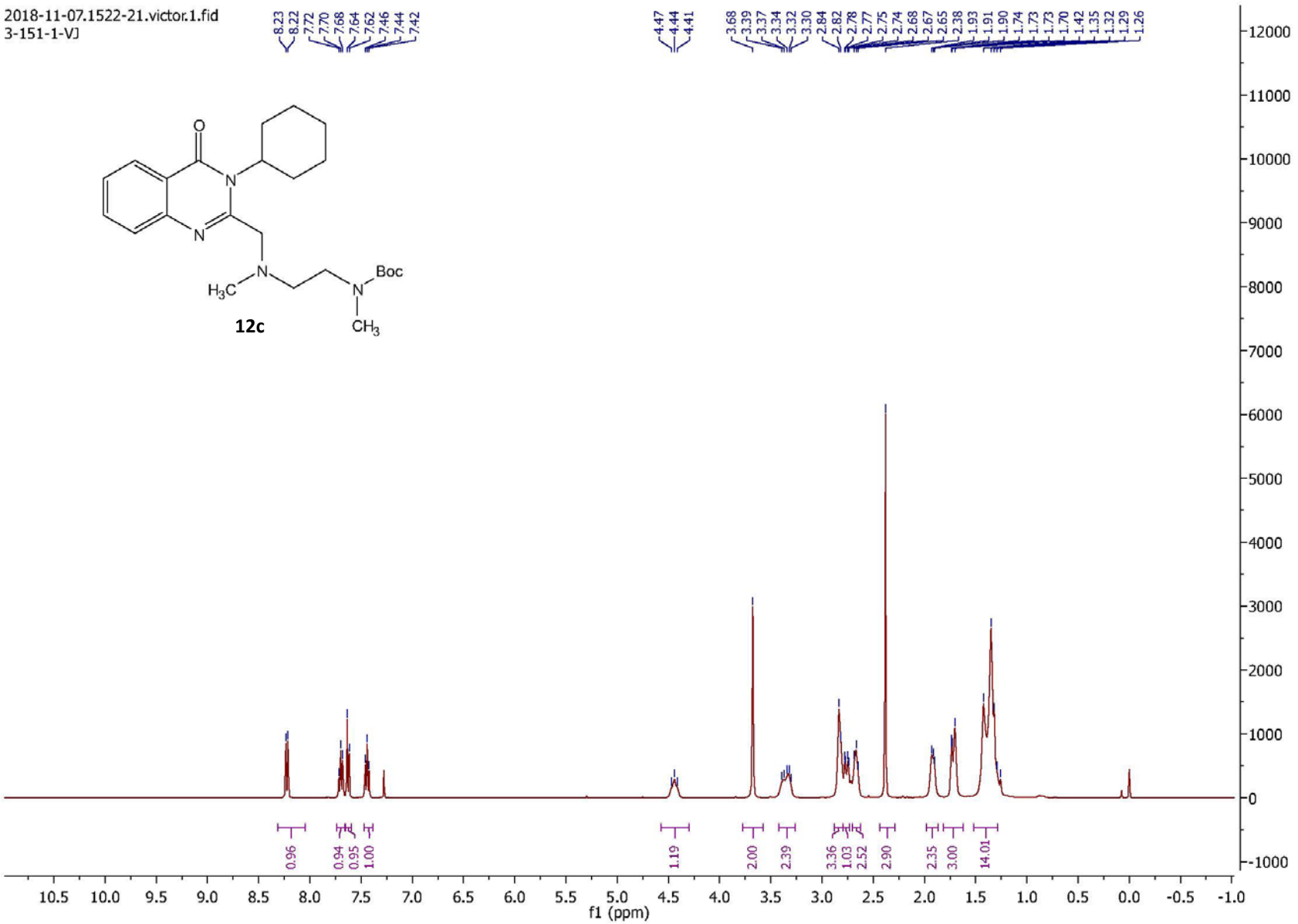
12a

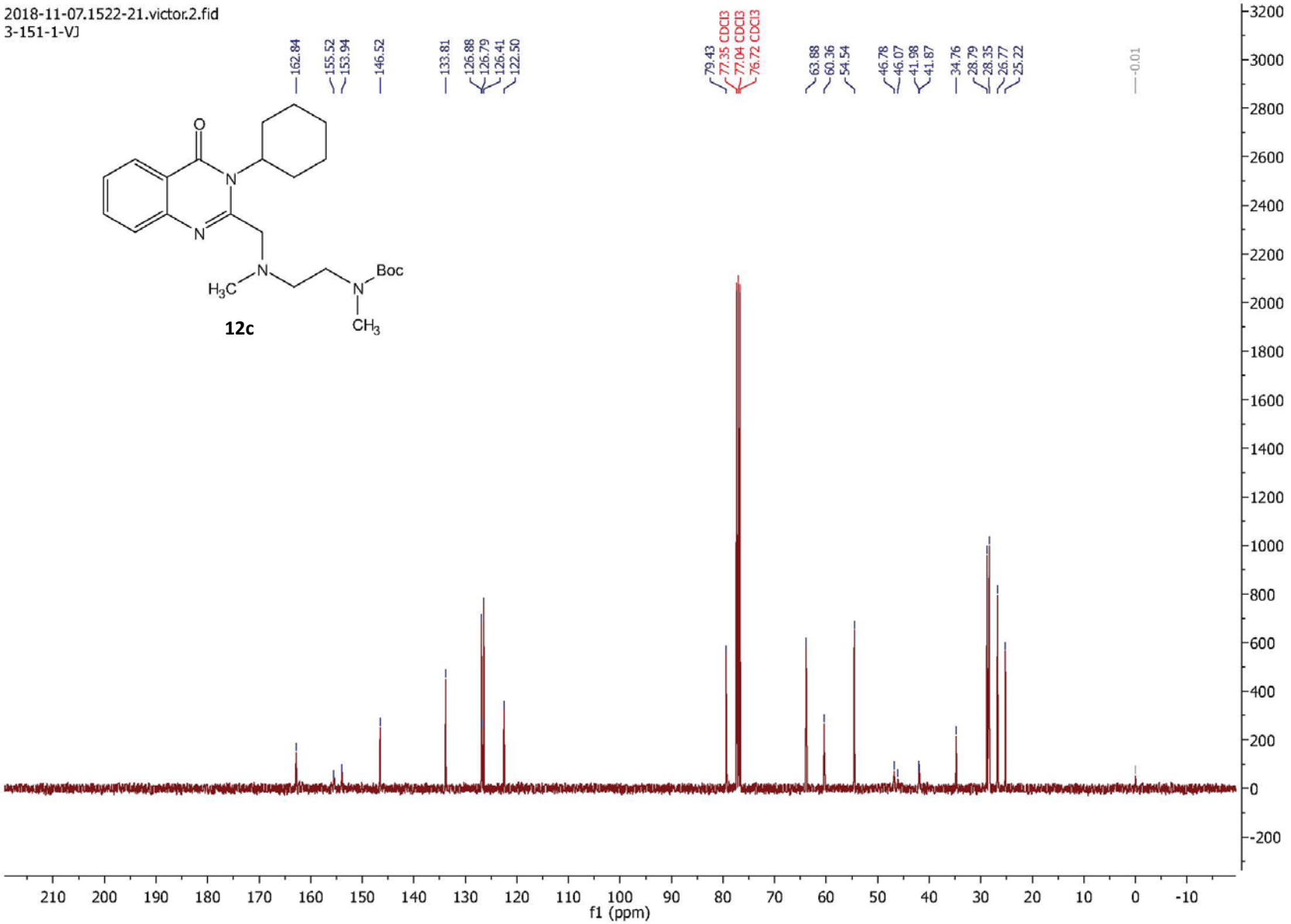

2018-11-05.1613-50.victor.2.fid
3-149-1-VJ-13C
163.1
156.2
155.6
146.4
133.6
127.1
126.3
122.0
79.2
77.3 CDCl3
77.0 CDCl3
76.7 CDCl3
71.5
65.9
59.4
40.1
34.5
30.0
29.1
28.5
28.4
26.9
26.8
25.2
21.0
20.6
15.3
0.0
O
N
N
CH3
N
Boc
N
CH3
H3C
CH3
f1 (ppm)

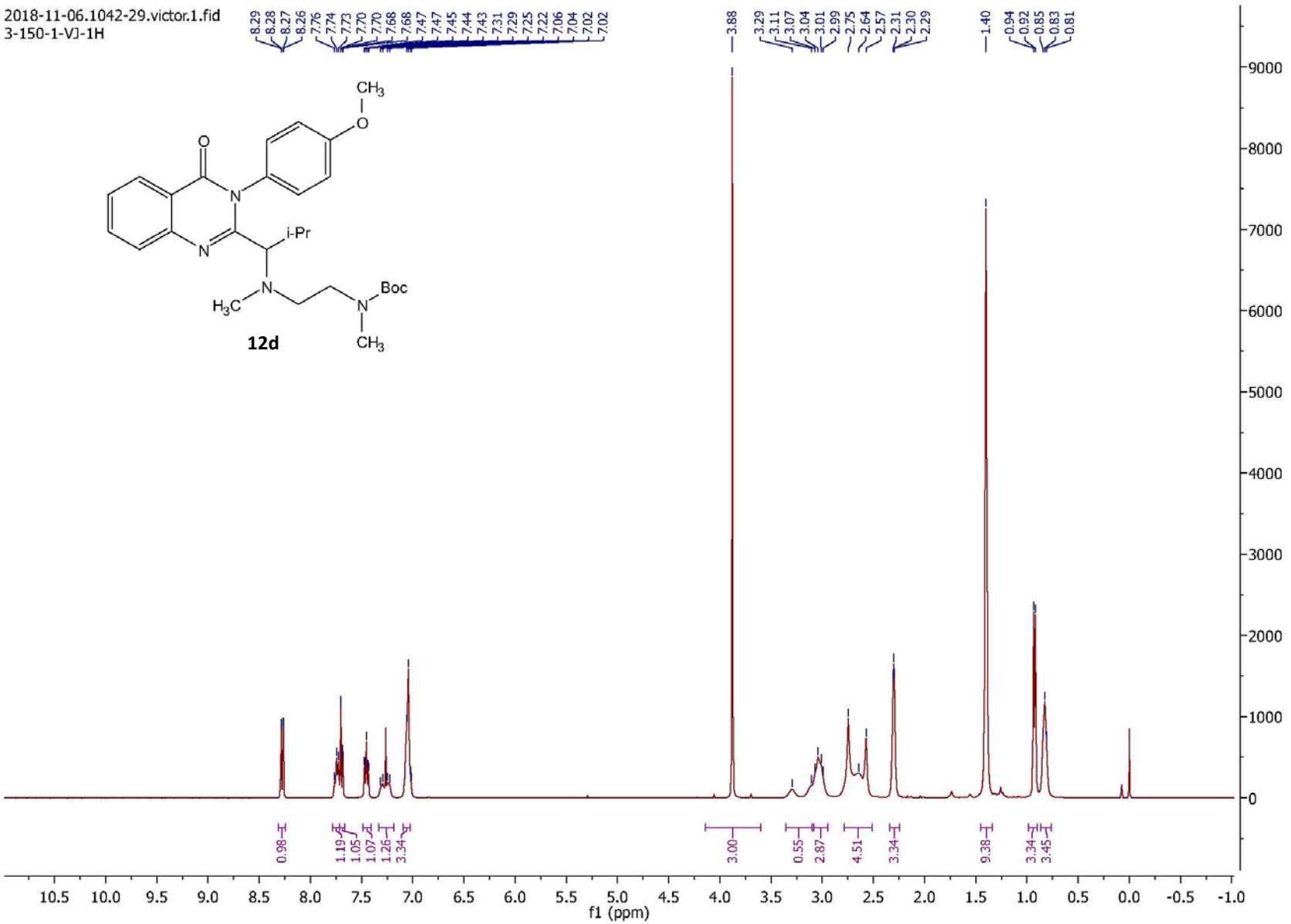
12a

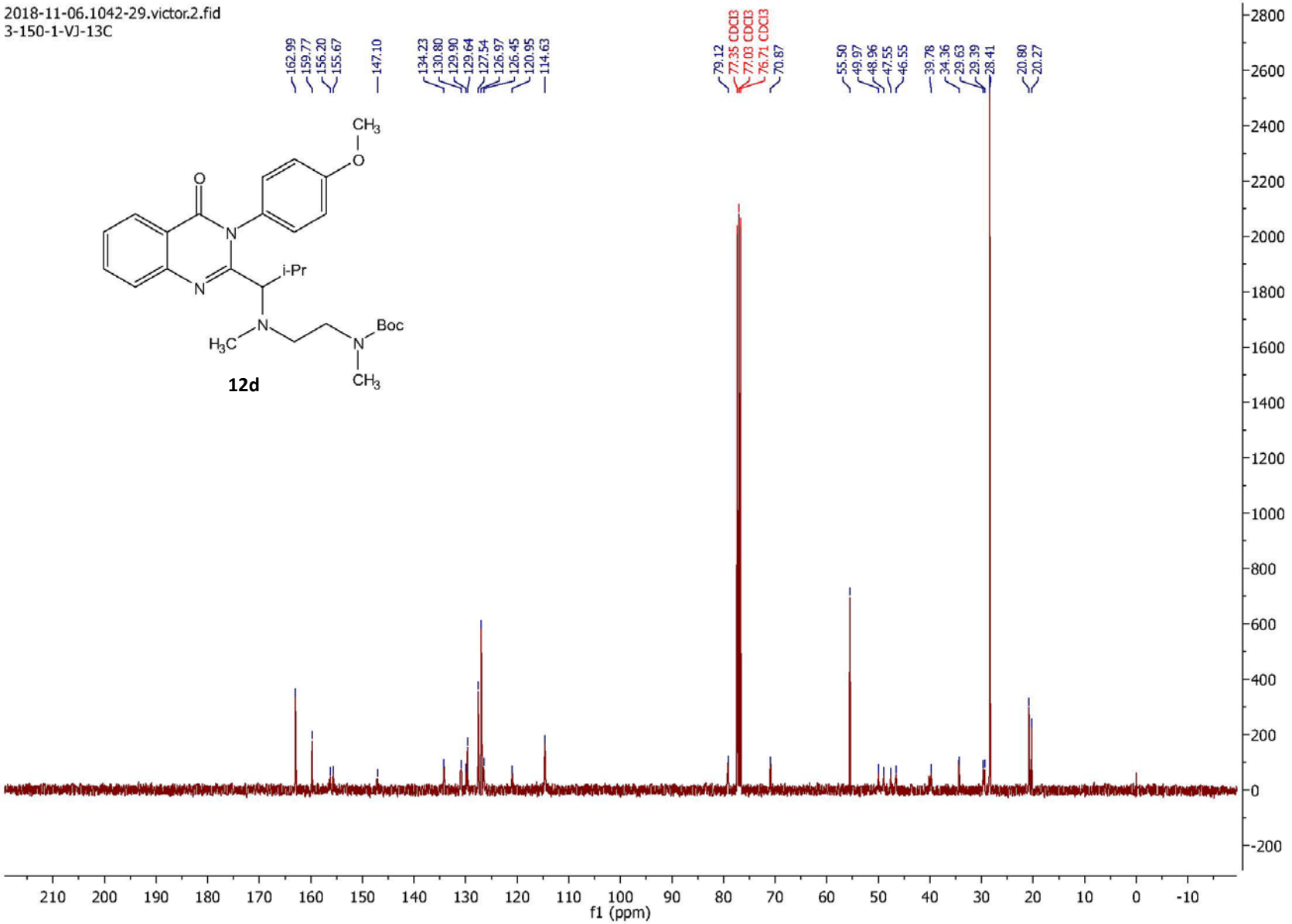

2017-09-06 +095 1-5 calok 1.fid
AN-V-70 Proton
O
N
N
i-Bu
N
H3C
N
Boc
CH3
12b
f1 (ppm)

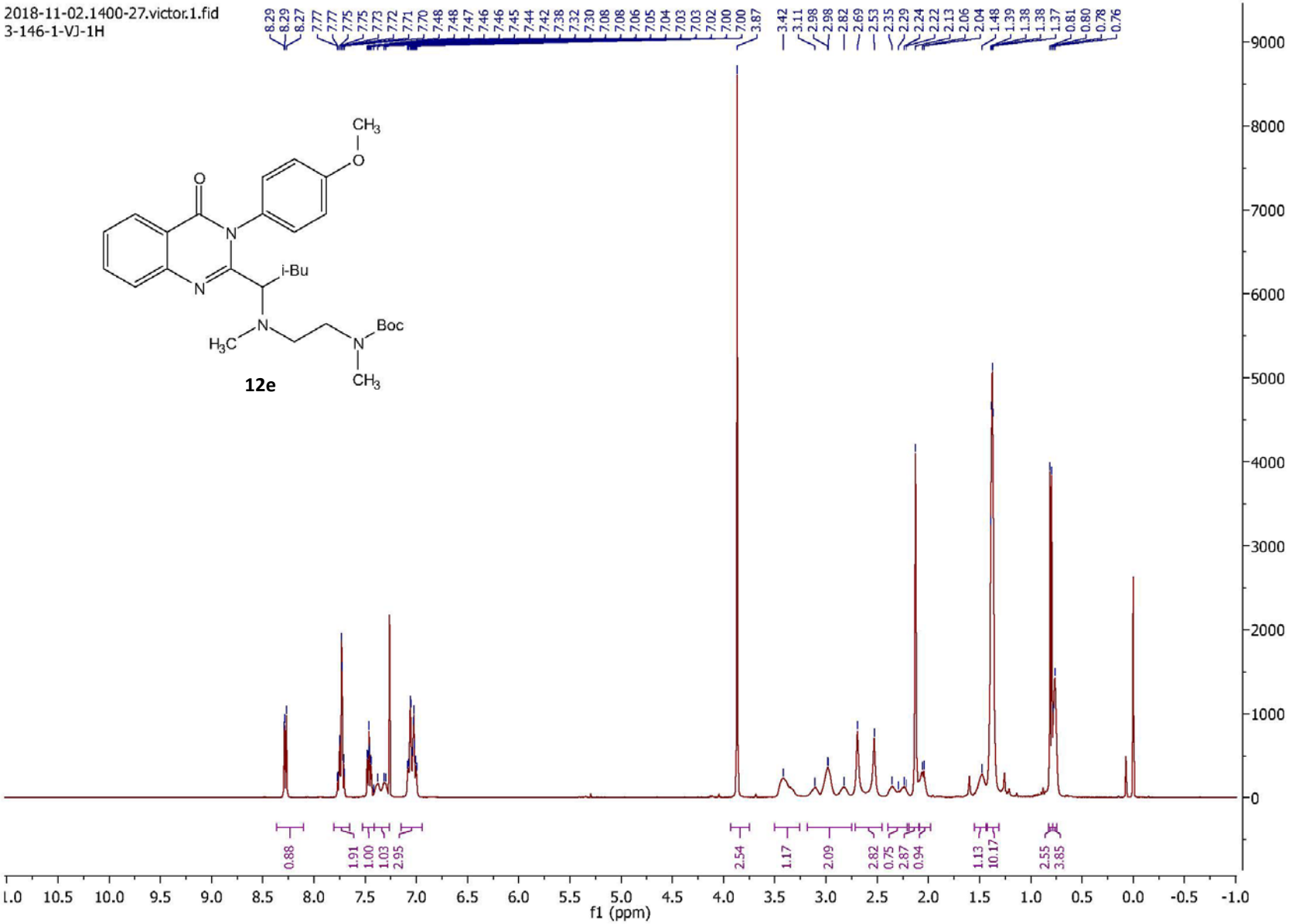

2017-09-06.1951-5.alok.2.fid
AN-V-70-Carbon
163.4
155.5
154.5
146.2
133.5
127.3
126.5
126.2
122.2
79.2
77.4 CDCl3
77.0 CDCl3
76.7 CDCl3
64.0
58.9
58.3
56.2
51.2
49.5
47.7
47.0
38.9
35.3
34.6
29.7
29.2
28.5
28.3
27.0
25.8
25.3
24.8
24.7
23.7
22.9
22.8
21.8
O
N
N
i-Bu
N
H3C
N
Boc
CH3
12b
f1 (ppm)

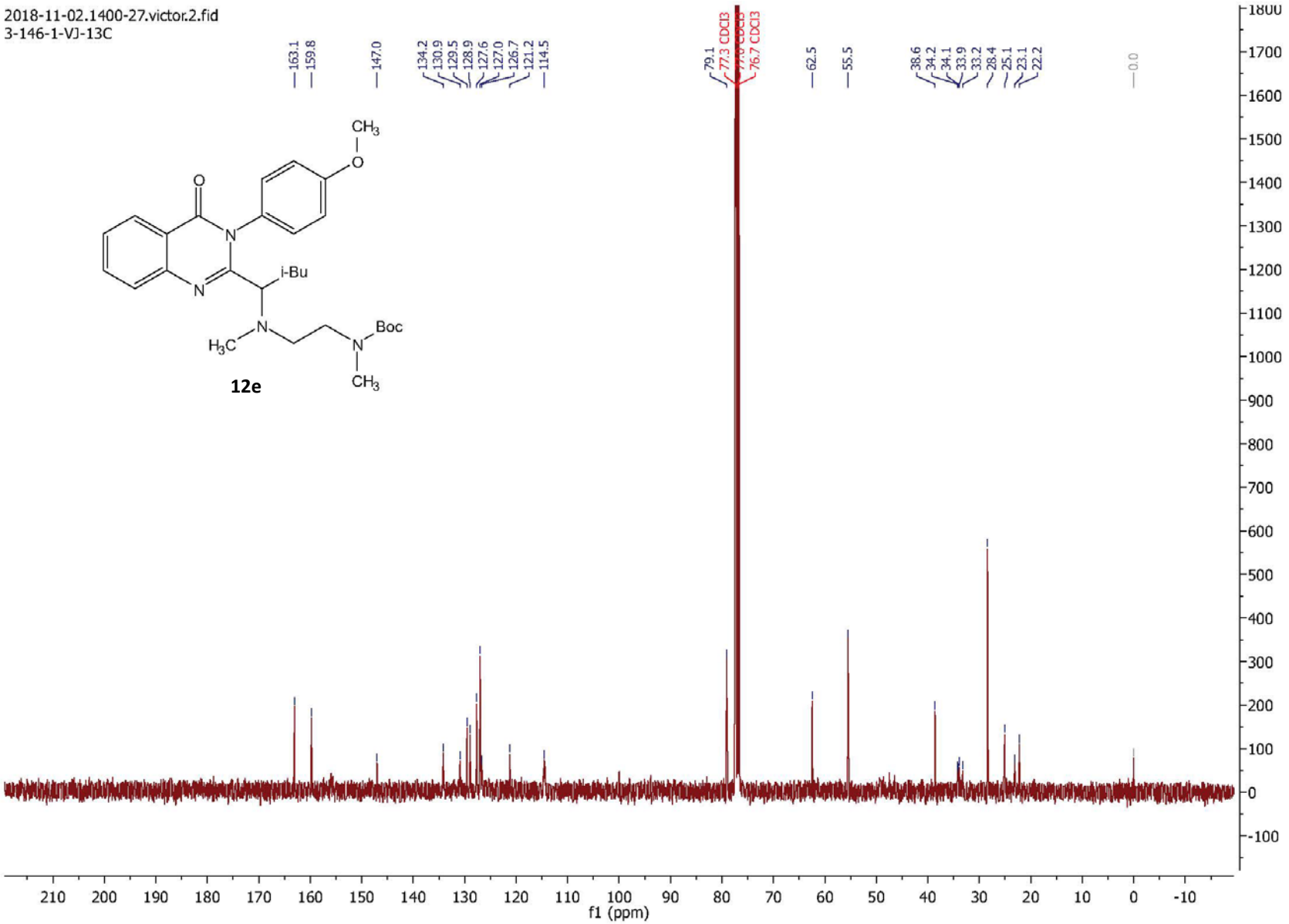

2018-11-07.1522-21.victor.1.fid
3-151-1-VJ
8.23
8.22
7.72
7.70
7.68
7.64
7.62
7.46
7.44
7.42
4.47
4.44
4.41
3.68
3.39
3.37
3.34
3.32
3.30
2.84
2.82
2.78
2.77
2.75
2.74
2.68
2.67
2.65
2.38
1.93
1.91
1.90
1.74
1.73
1.73
1.70
1.42
1.35
1.32
1.29
1.26
O
N
N
N
H3C
Boc
N
CH3
12c
0.96
0.94
0.95
1.00
1.19
2.00
2.39
3.36
1.03
2.52
2.90
2.35
3.00
14.01
f1 (ppm)

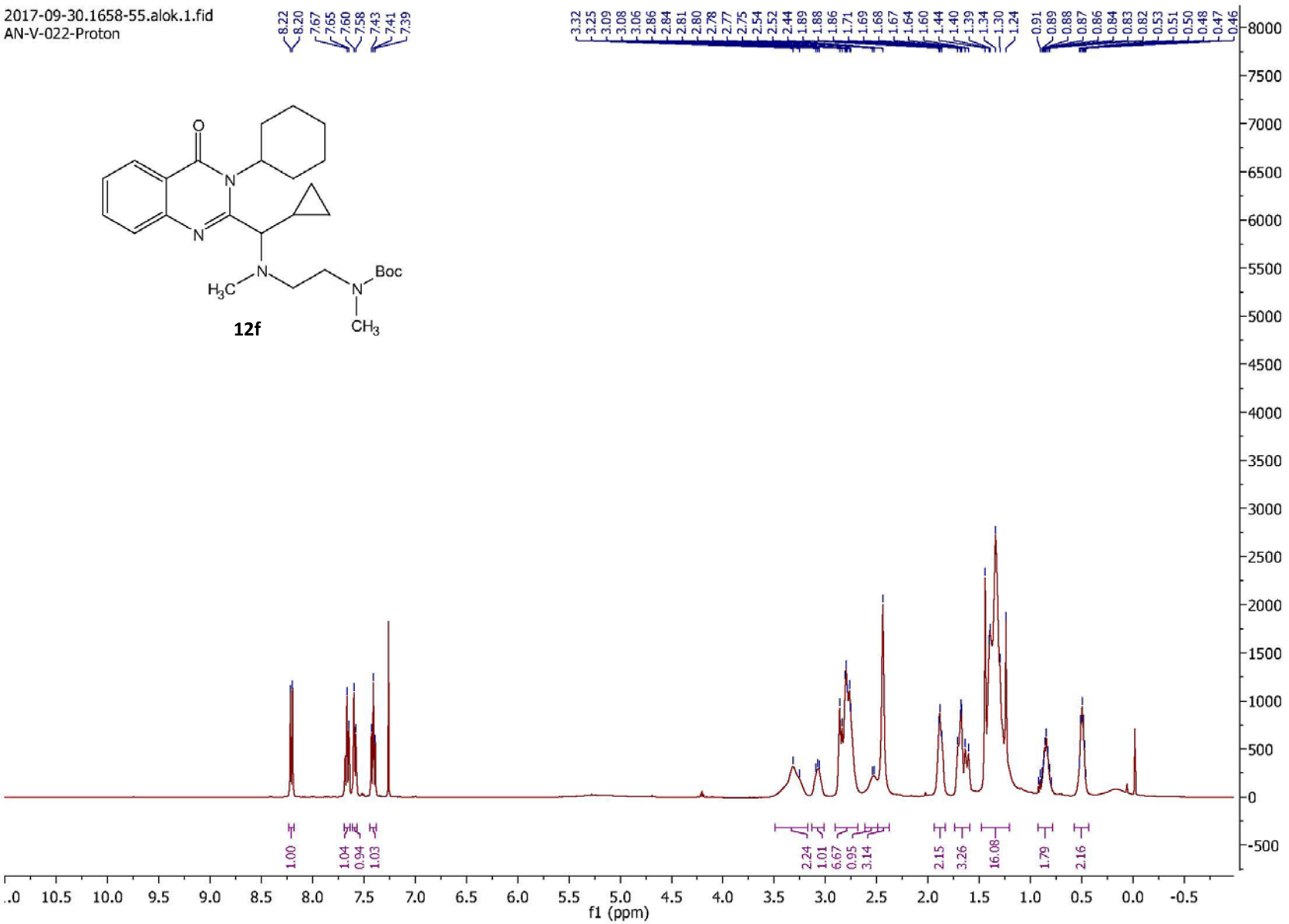

2018-11-07.1522-21.victor.2.fid
3-151-1-VJ
162.84
155.52
153.94
146.52
133.81
126.88
126.79
126.41
122.50
79.43
77.35 CDCl3
77.04 CDCl3
76.72 CDCl3
63.88
60.36
54.54
46.78
46.07
41.98
41.87
34.76
28.79
28.35
26.77
25.22
-0.01
O
N
N
N
H3C
Boc
N
CH3
12c
f1 (ppm)

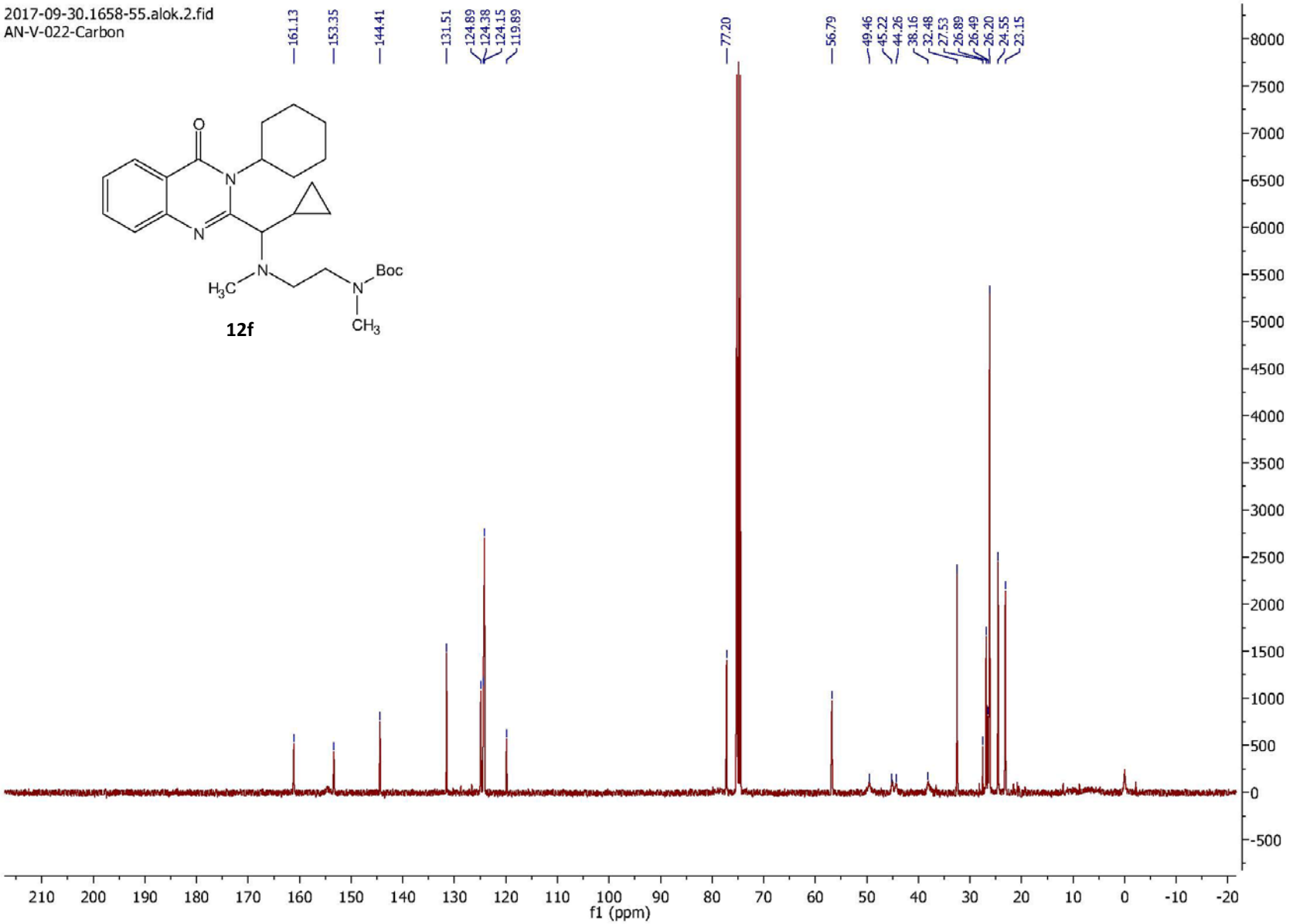

2018-11-06.1042-29.victor.1.fid
3-150-1-VJ-1H
8.29
8.28
8.27
8.26
7.76
7.74
7.73
7.70
7.70
7.68
7.68
7.47
7.47
7.45
7.44
7.43
7.31
7.29
7.25
7.22
7.06
7.04
7.02
7.02
3.88
3.29
3.11
3.07
3.04
3.01
2.99
2.75
2.64
2.57
2.31
2.30
2.29
1.40
0.94
0.92
0.85
0.83
0.81
CH3
O
O
N
N
i-Pr
N
H3C
N
Boc
CH3
12d
0.98
1.19
1.05
1.07
1.26
3.34
3.00
0.55
2.87
4.51
3.34
9.38
3.34
3.45
f1 (ppm)

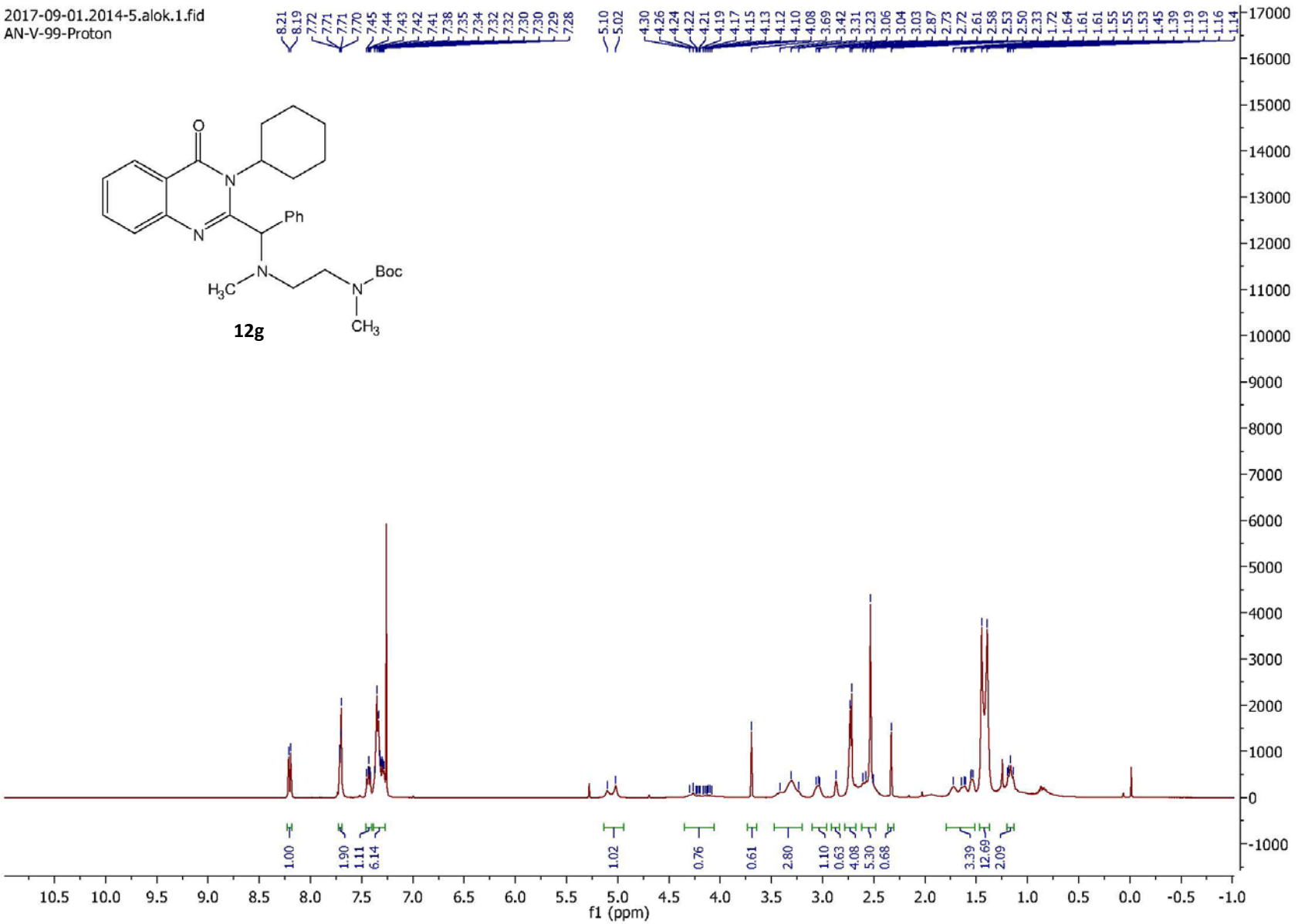

2018-11-06.1042-29.victor.2.fid
3-150-1-VJ-13C
162.99
159.77
156.20
155.67
147.10
134.23
130.80
129.90
129.64
127.54
126.97
126.45
120.95
114.63
79.12
77.35 CDCl3
77.03 CDCl3
76.71 CDCl3
70.87
55.50
49.97
48.96
47.55
46.55
39.78
34.36
29.63
29.39
28.41
20.80
20.27
CH3
O
O
N
N
i-Pr
N
H3C
N
Boc
CH3
12d
f1 (ppm)

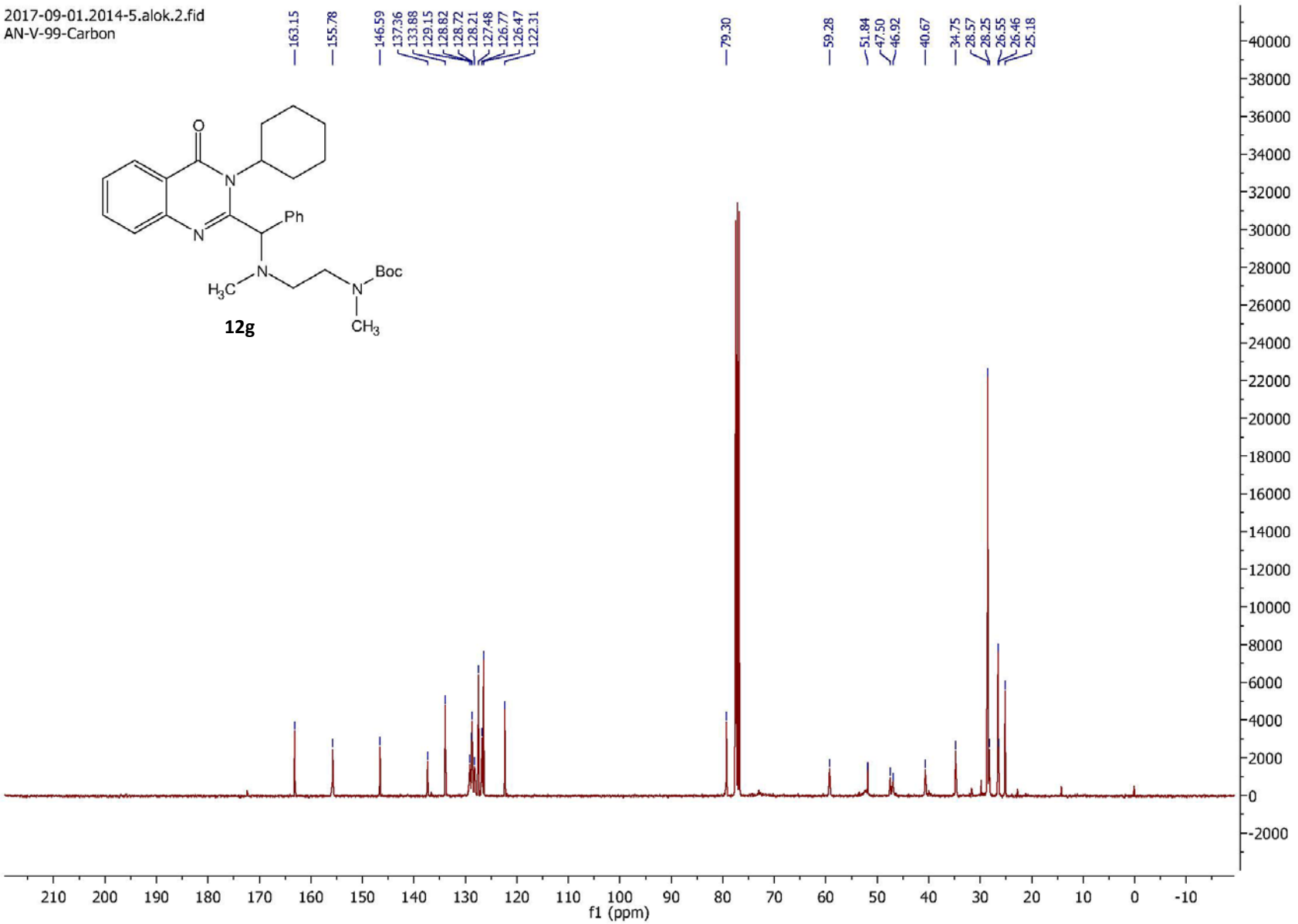

2018-11-02.1400-27.victor.1.fid
3-146-1-VJ-1H
8.29 8.29 8.27 7.77 7.77 7.75 7.75 7.73 7.72 7.71 7.70 7.48 7.48 7.47 7.46 7.46 7.45 7.44 7.42 7.38 7.32 7.30 7.08 7.08 7.06 7.05 7.04 7.03 7.03 7.02 7.00 7.00 3.87 3.42 3.11 2.98 2.98 2.82 2.69 2.53 2.35 2.29 2.24 2.22 2.13 2.06 2.04 1.48 1.39 1.38 1.38 1.37 0.81 0.80 0.78 0.76
CH3
O
O
N
N
i-Bu
N
Boc
H3C
N
CH3
12e
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
0.88 1.91 1.00 1.03 2.95 2.54 1.17 2.09 2.82 0.75 2.87 0.94 1.13 10.17 2.55 3.85
1.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0
f1 (ppm)

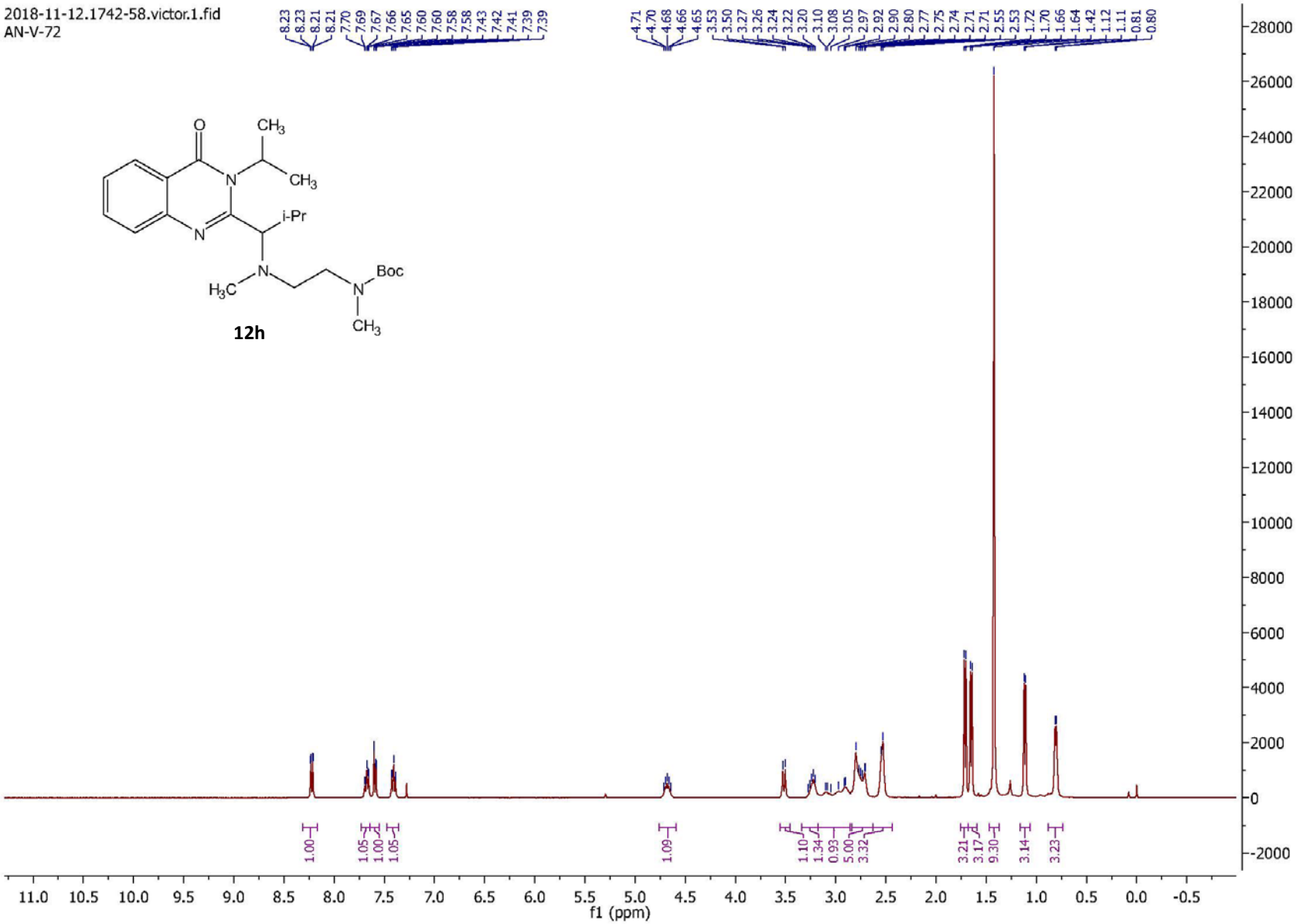

2018-11-02.1400-27.victor.2.fid
3-146-1-VJ-13C
163.1
159.8
147.0
134.2
130.9
129.5
128.9
127.6
127.0
126.7
121.2
114.5
79.1
77.3 CDCl3
76.7 CDCl3
62.5
55.5
38.6
34.2
34.1
33.9
33.2
28.4
25.1
23.1
22.2
0.0
CH3
O
O
N
N
i-Bu
N
H3C
N
Boc
CH3
12e
f1 (ppm)

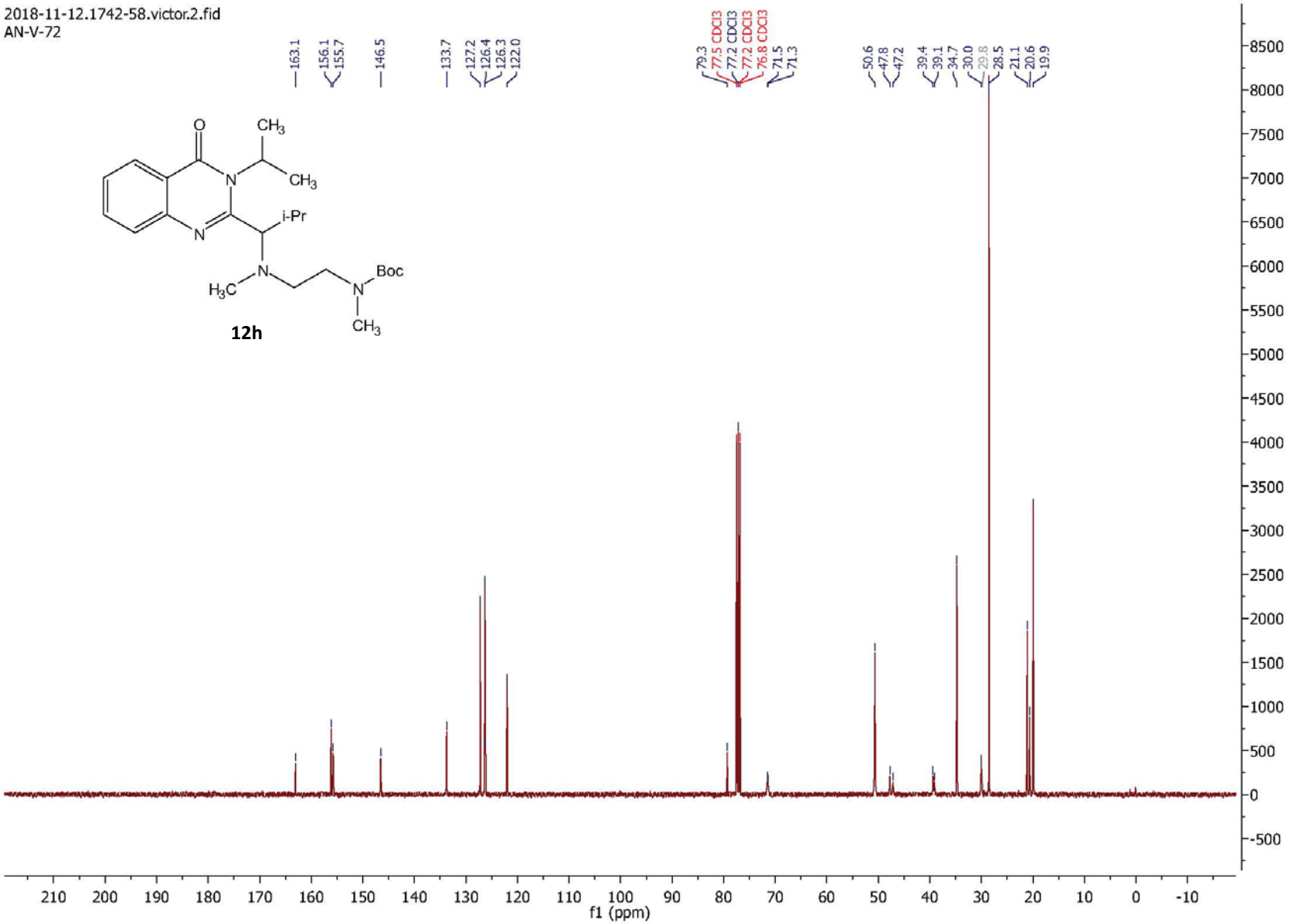

2017-09-30.1658-55.alok.1.fid
AN-V-022-Proton
12f
f1 (ppm)


2017-09-30.1658-55.alok.2.fid
AN-V-022-Carbon
161.13
153.35
144.41
131.51
124.89
124.38
124.15
119.89
77.20
56.79
49.46
45.22
44.26
38.16
32.48
27.53
26.89
26.49
26.20
24.55
23.15
O
N
N
N
H3C
Boc
N
CH3
12f
f1 (ppm)

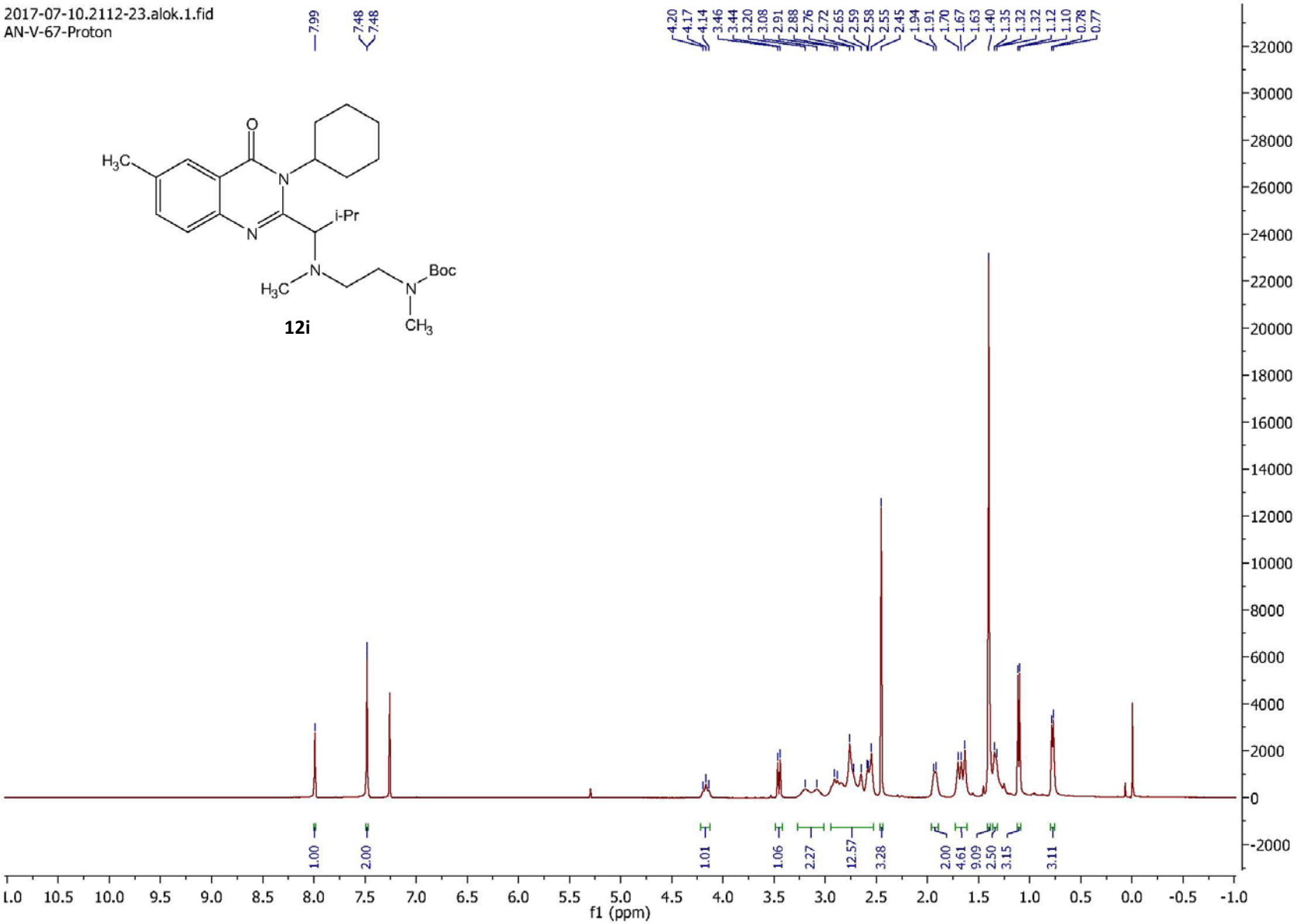

2017-09-01.2014-5.alok.1.fid
AN-V-99-Proton
12g
f1 (ppm)

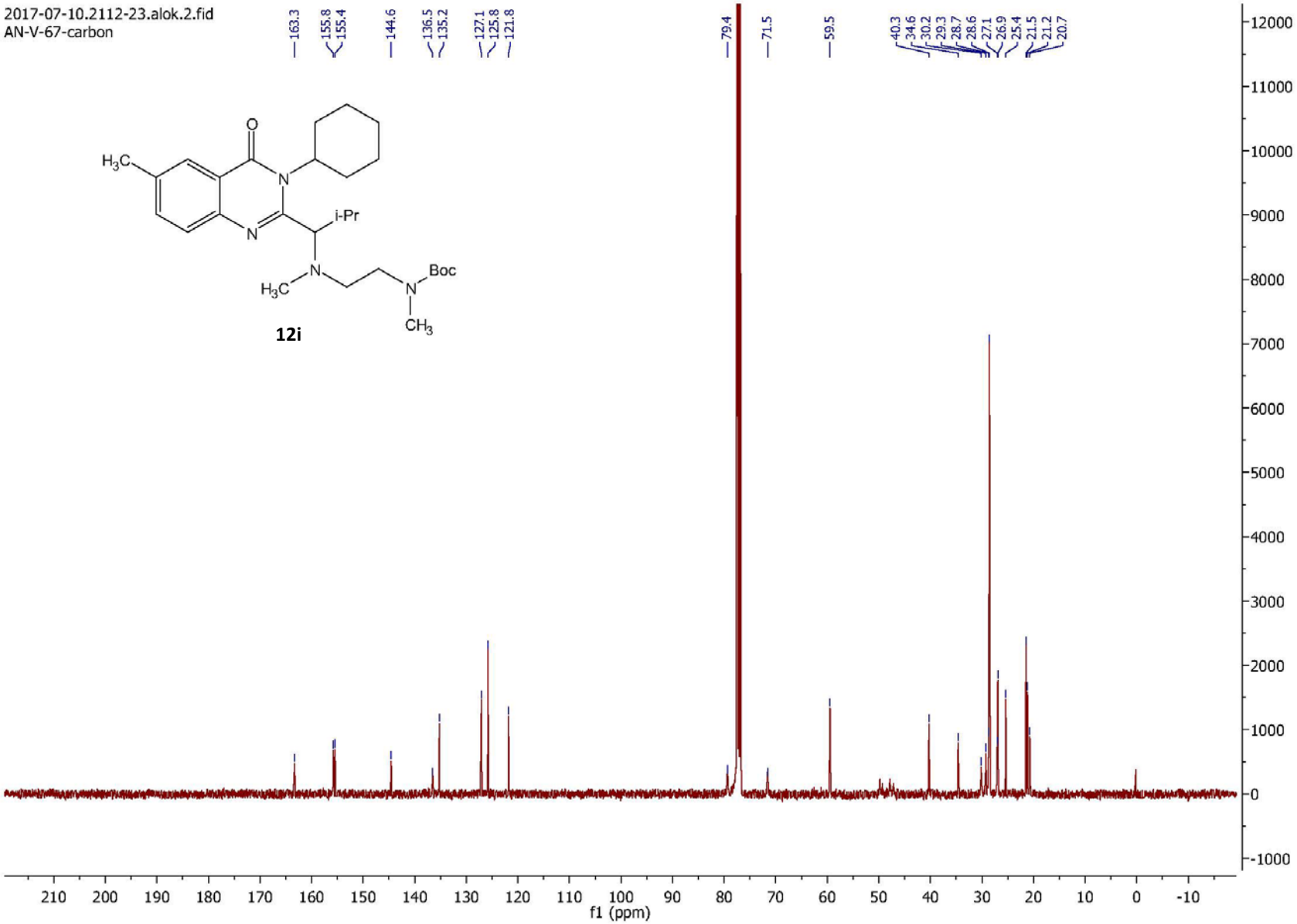

2017-09-01.2014-5.alok.2.fid
AN-V-99-Carbon
163.15
155.78
146.59
137.36
133.88
129.15
128.82
128.72
128.21
127.48
126.77
126.47
122.31
79.30
59.28
51.84
47.50
46.92
40.67
34.75
28.57
28.25
26.55
26.46
25.18
O
N
N
Ph
N
H3C
N
Boc
CH3
12g
f1 (ppm)

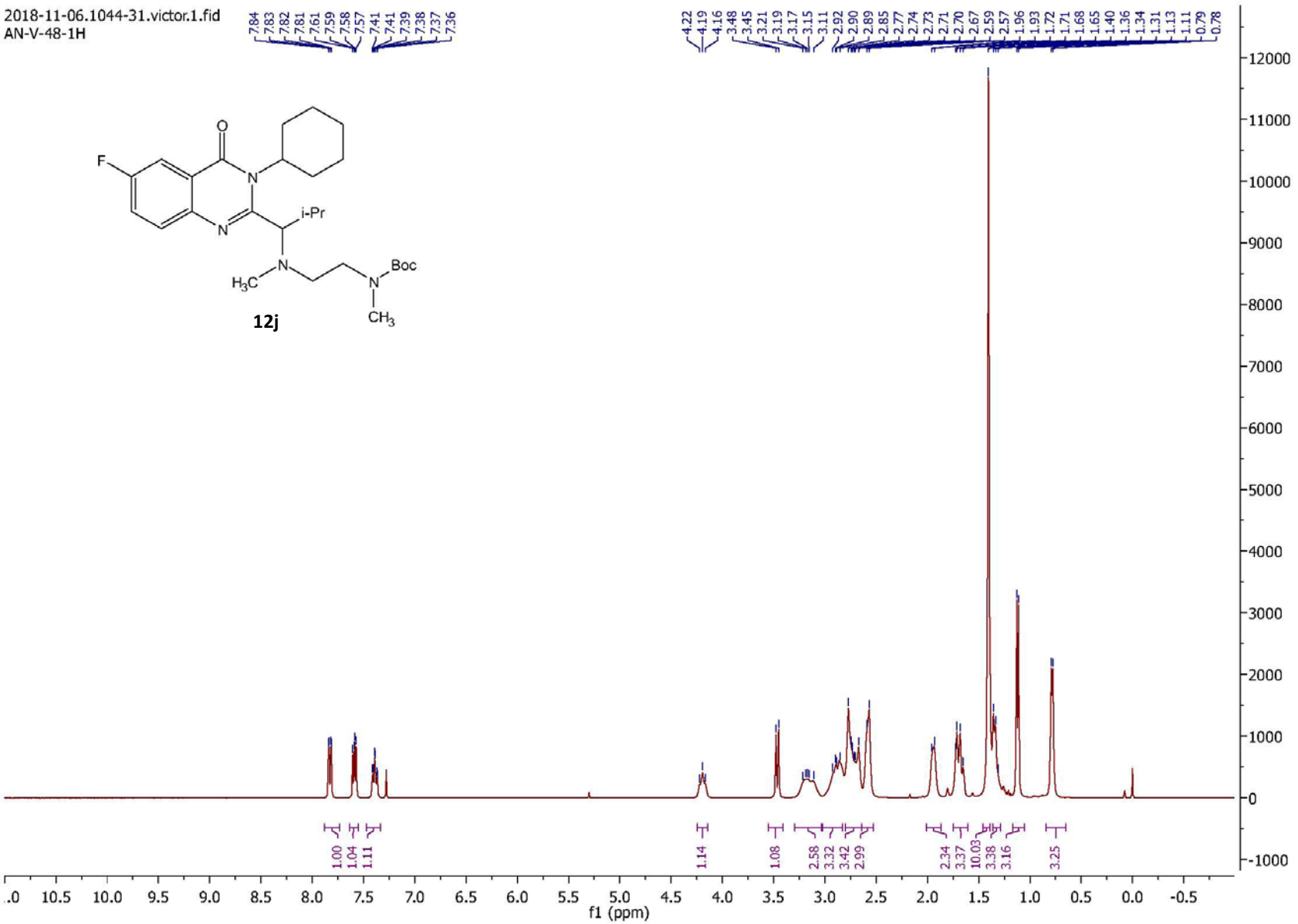

2018-11-12.1742-58.victor.1.fid
AN-V-72
8.23 8.23 8.21 8.21 7.70 7.69 7.67 7.66 7.65 7.60 7.60 7.58 7.58 7.43 7.42 7.41 7.39 7.39
4.71 4.70 4.68 4.66 4.65 3.53 3.50 3.27 3.26 3.24 3.22 3.20 3.10 3.08 3.05 2.97 2.92 2.90 2.80 2.77 2.75 2.74 2.71 2.71 2.55 2.53 1.72 1.70 1.66 1.64 1.42 1.12 1.11 0.81 0.80
O
CH3
N
CH3
i-Pr
N
H3C
N
Boc
N
CH3
12h
1.00 1.05 1.00 1.05 1.09 1.10 1.34 0.93 5.00 3.32 3.21 3.17 9.30 3.14 3.23
f1 (ppm)

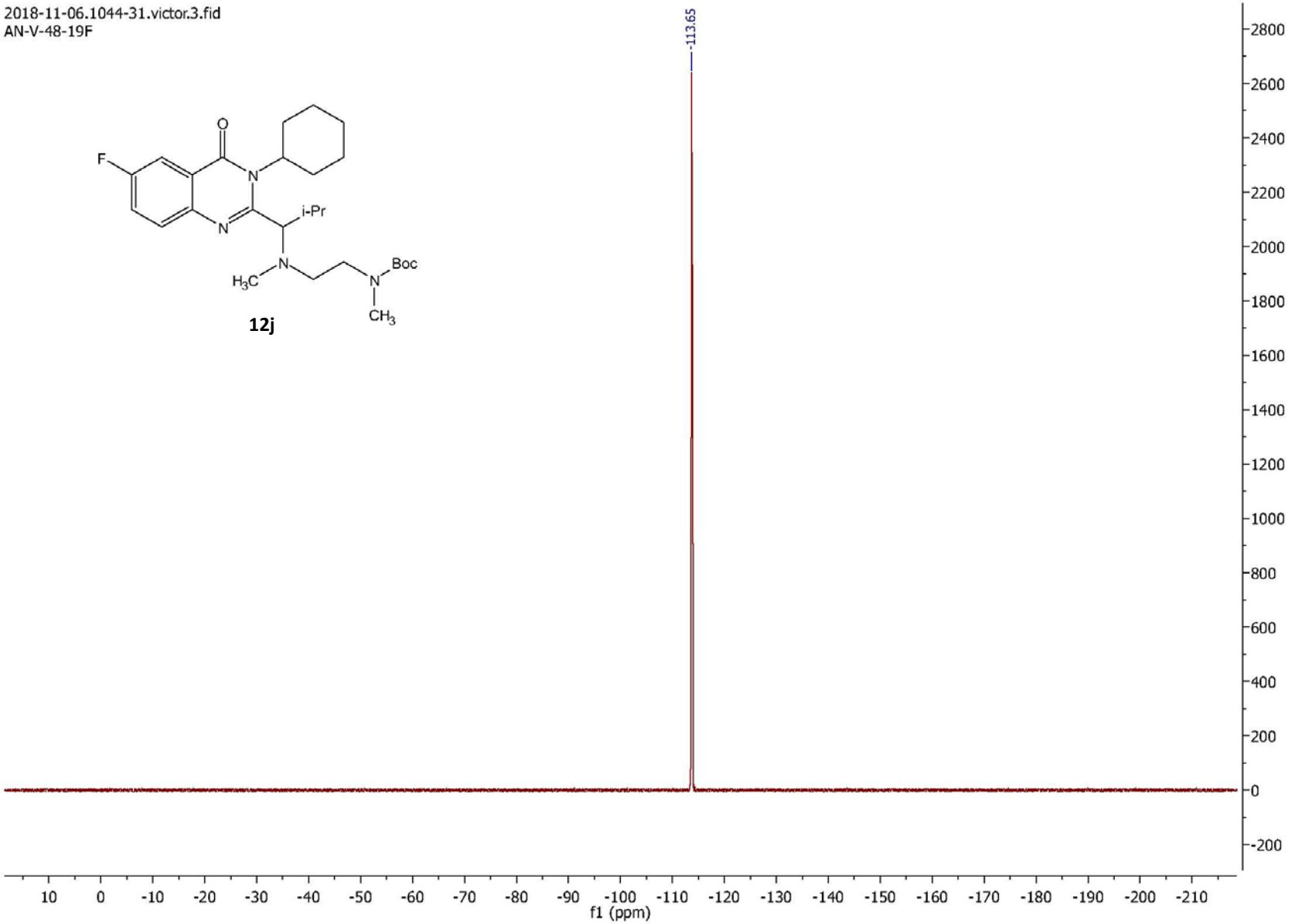

2018-11-12.1742-58.victor.2.fid
AN-V-72
163.1
156.1
155.7
146.5
133.7
127.2
126.4
126.3
122.0
79.3
77.5 CDCl3
77.2 CDCl3
77.2 CDCl3
76.8 CDCl3
71.5
71.3
50.6
47.8
47.2
39.4
39.1
34.7
30.0
29.8
28.5
21.1
20.6
19.9
O
CH3
N
CH3
i-Pr
N
H3C
N
Boc
CH3
f1 (ppm)

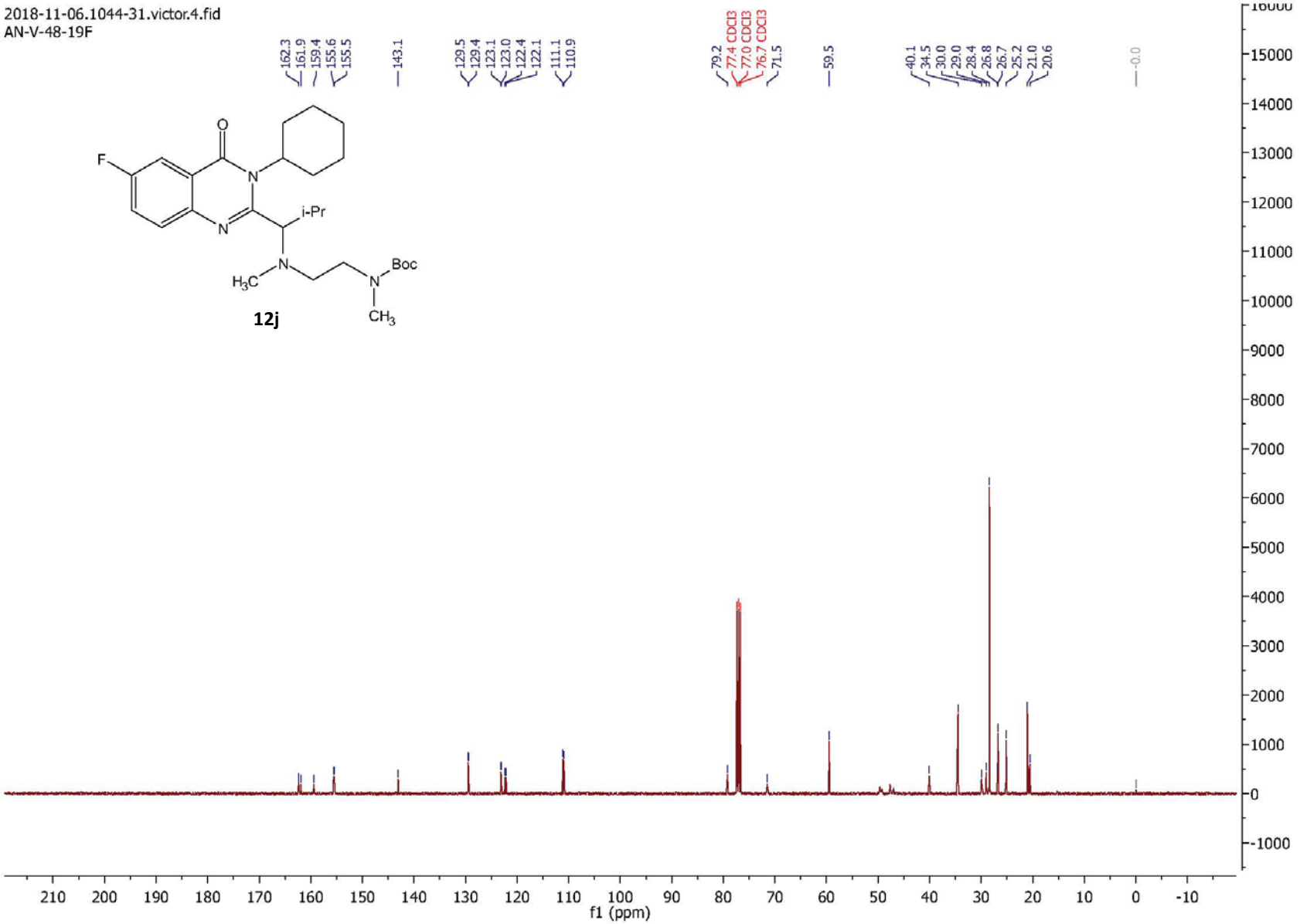
12h

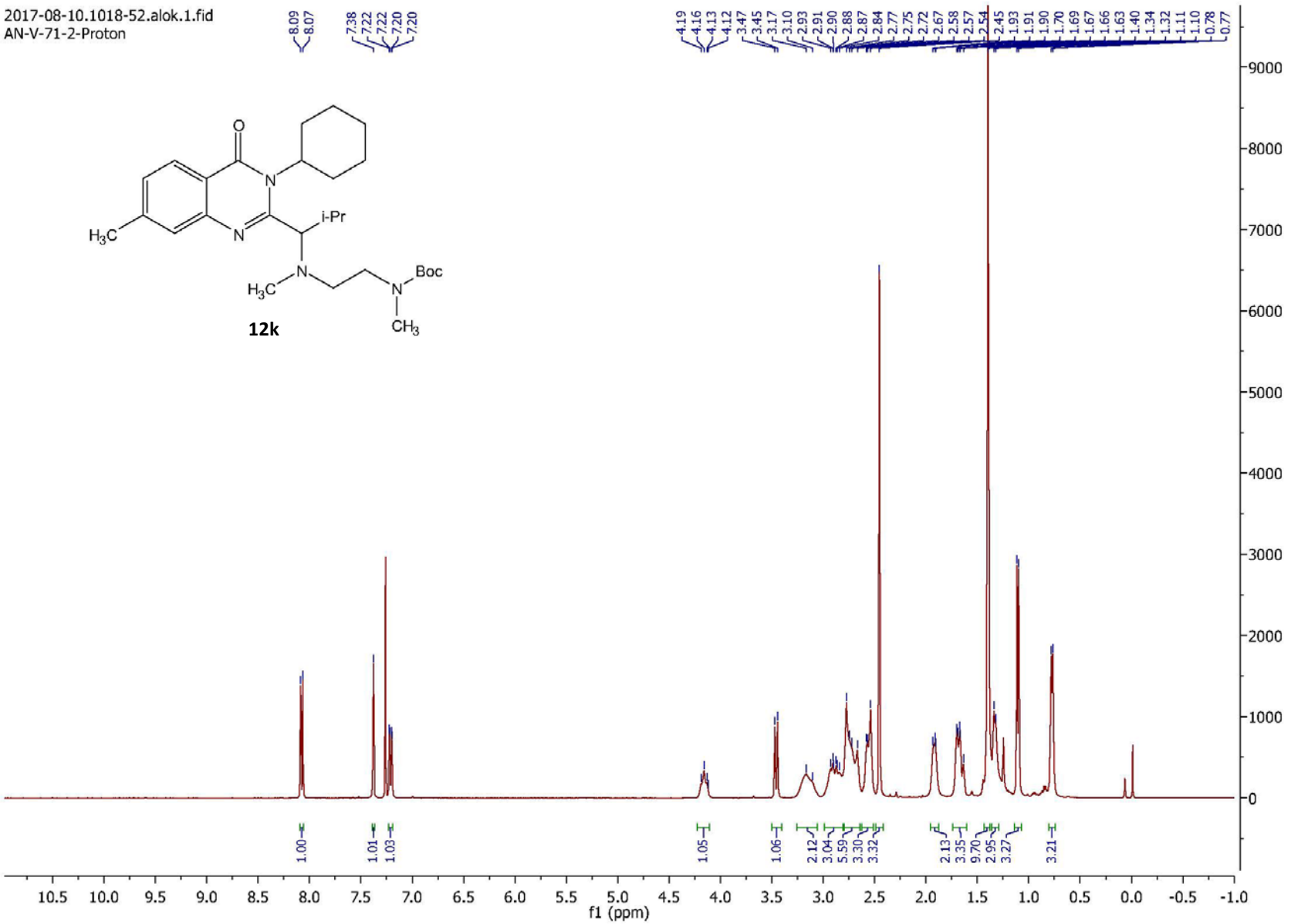

2017-07-10.2112-23.alok.1.fid
AN-V-67-Proton
7.99
7.48
7.48
4.20
4.17
4.14
3.46
3.44
3.20
3.08
2.91
2.88
2.76
2.72
2.65
2.59
2.58
2.55
2.45
1.94
1.91
1.70
1.67
1.63
1.40
1.35
1.32
1.32
1.12
1.10
0.78
0.77
O
H3C
N
i-Pr
N
H3C
N
Boc
CH3
12i
1.00
2.00
1.01
1.06
2.27
12.57
3.28
2.00
4.61
9.09
2.50
3.15
3.11
f1 (ppm)

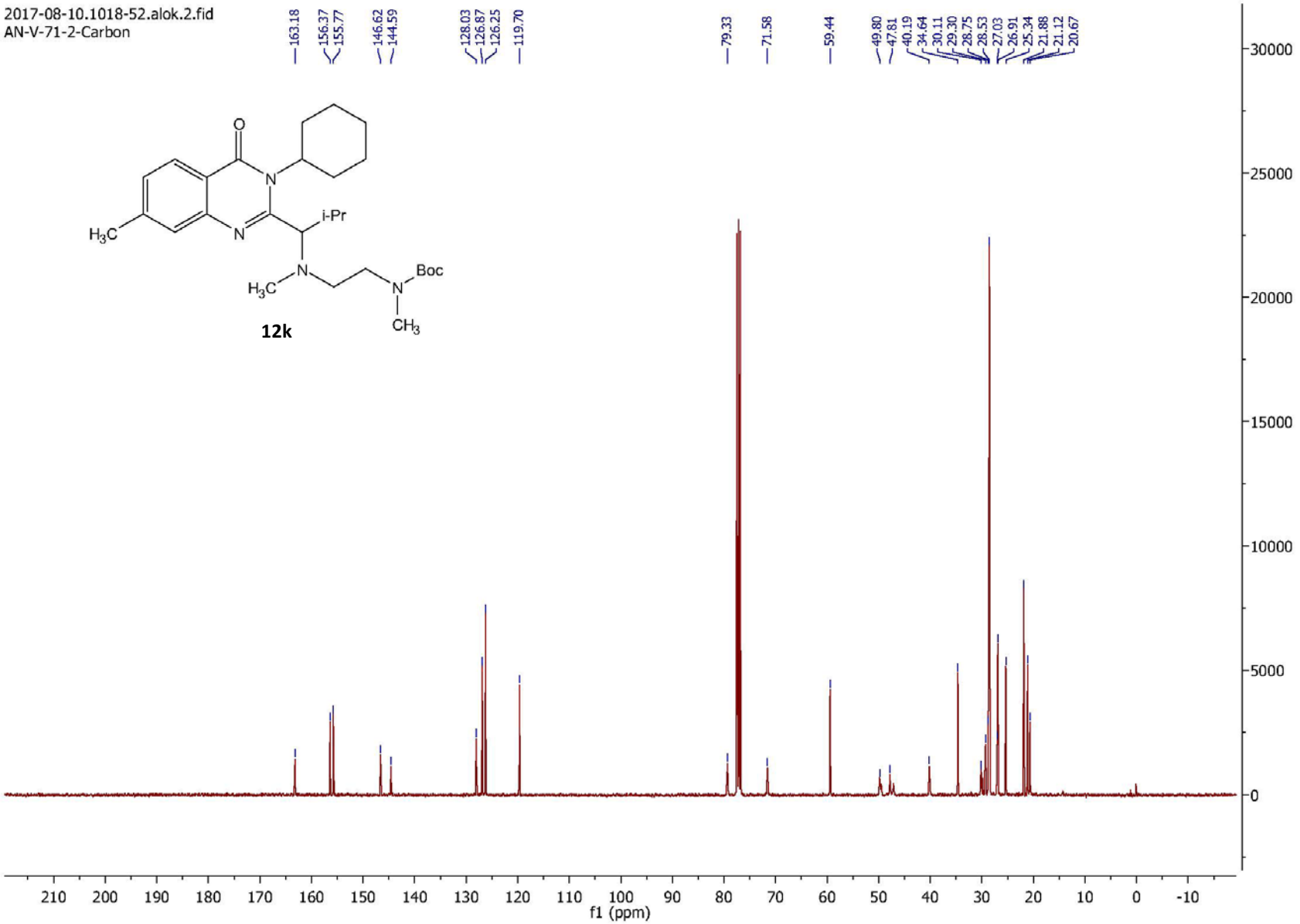

2017-07-10.2112-23.alok.2.fid
AN-V-67-carbon
163.3
155.8
155.4
144.6
136.5
135.2
127.1
125.8
121.8
79.4
71.5
59.5
40.3
34.6
30.2
29.3
28.7
28.6
27.1
26.9
25.4
21.5
21.2
20.7
O
H3C
N
i-Pr
N
H3C
N
Boc
N
CH3
12i
f1 (ppm)

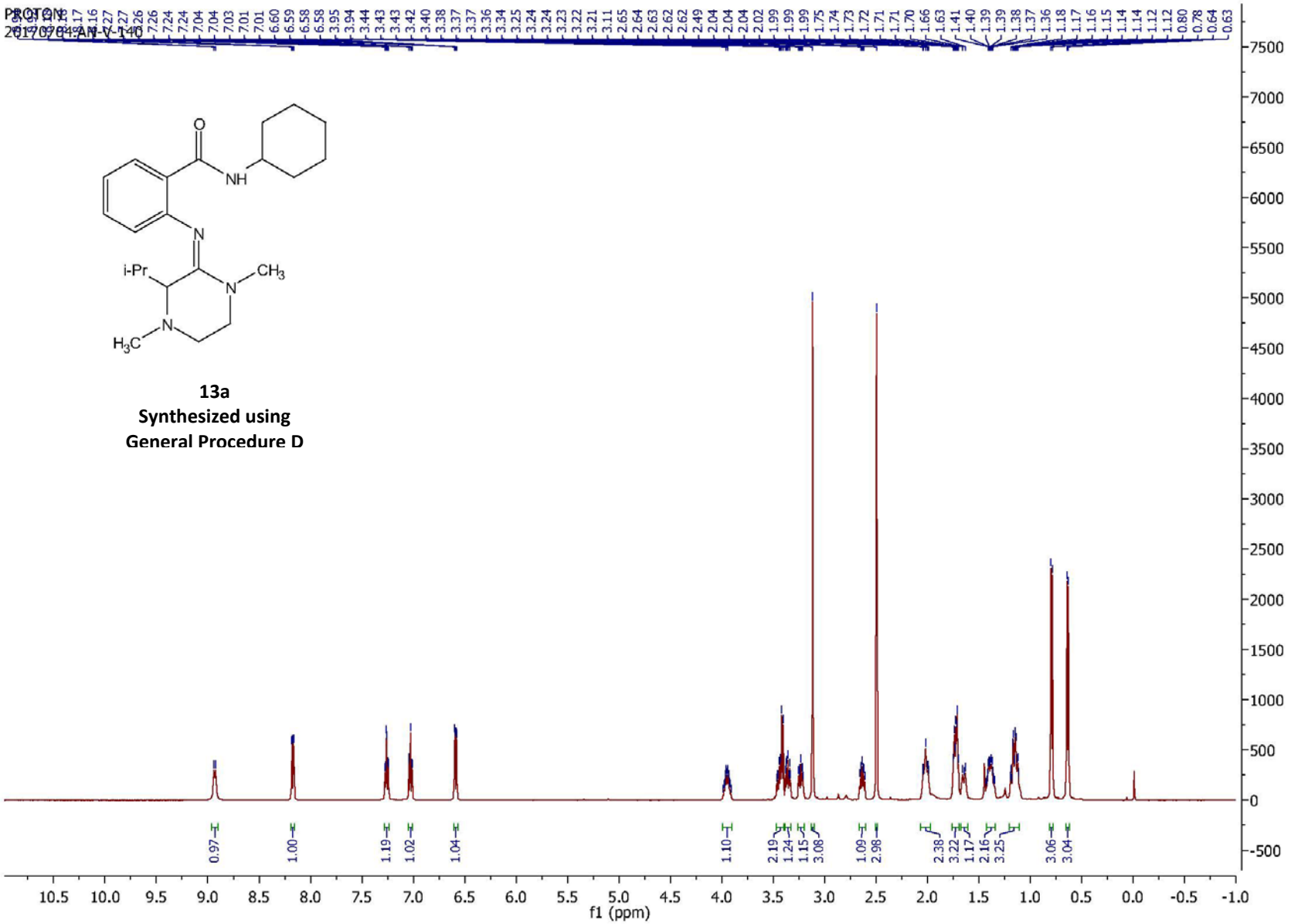

2018-11-06.1044-31.victor.1.fid
AN-V-48-1H
12j
f1 (ppm)


2018-11-06.1044-31.victor.3.fid
AN-V-48-19F
-113.65
F
O
N
N
i-Pr
N
H3C
N
Boc
CH3
12j
2800
2600
2400
2200
2000
1800
1600
1400
1200
1000
800
600
400
200
0
-200
10
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
-160
-170
-180
-190
-200
-210
f1 (ppm)

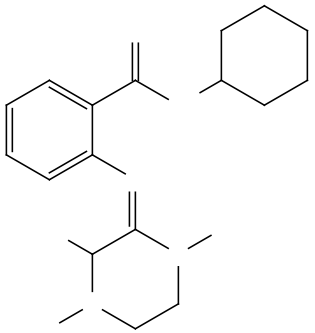

2018-11-06.1044-31.victor.4.fid
AN-V-48-19F
162.3
161.9
159.4
155.6
155.5
143.1
129.5
129.4
123.1
123.0
122.4
122.1
111.1
110.9
79.2
77.4 CDCl3
77.0 CDCl3
76.7 CDCl3
71.5
59.5
40.1
34.5
30.0
29.0
28.4
26.8
26.7
25.2
21.0
20.6
0.0
F
O
N
N
i-Pr
N
Boc
H3C
N
CH3
12j
f1 (ppm)


2017-08-10.1018-52.alok.1.fid
AN-V-71-2-Proton
12k
f1 (ppm)

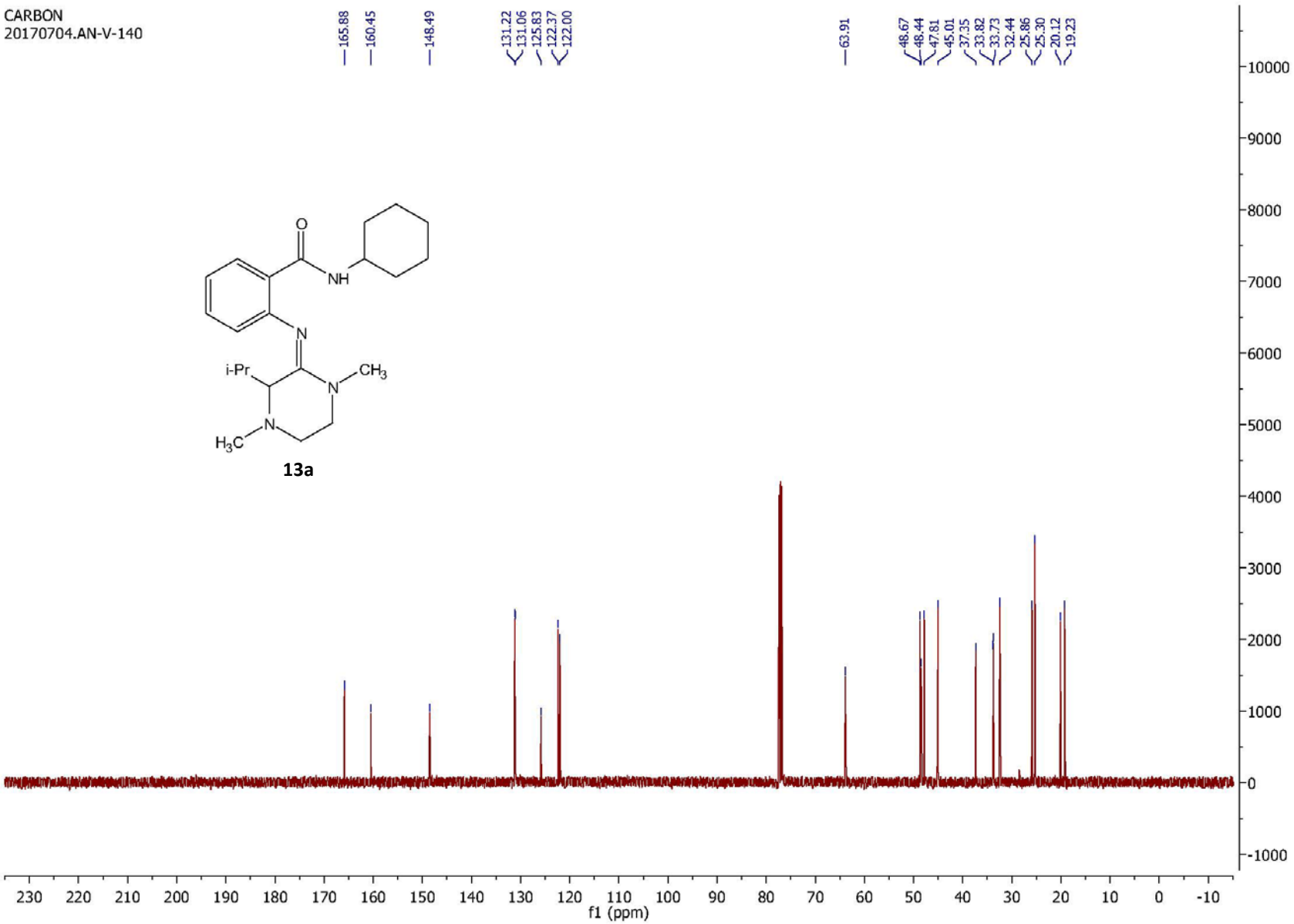

2017-08-10.1018-52.alok.2.fid
AN-V-71-2-Carbon
163.18
156.37
155.77
146.62
144.59
128.03
126.87
126.25
119.70
79.33
71.58
59.44
49.80
47.81
40.19
34.64
30.11
29.30
28.75
28.53
27.03
26.91
25.34
21.88
21.12
20.67
O
N
i-Pr
H3C
N
H3C
N
Boc
CH3
12k
30000
25000
20000
15000
10000
5000
0
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

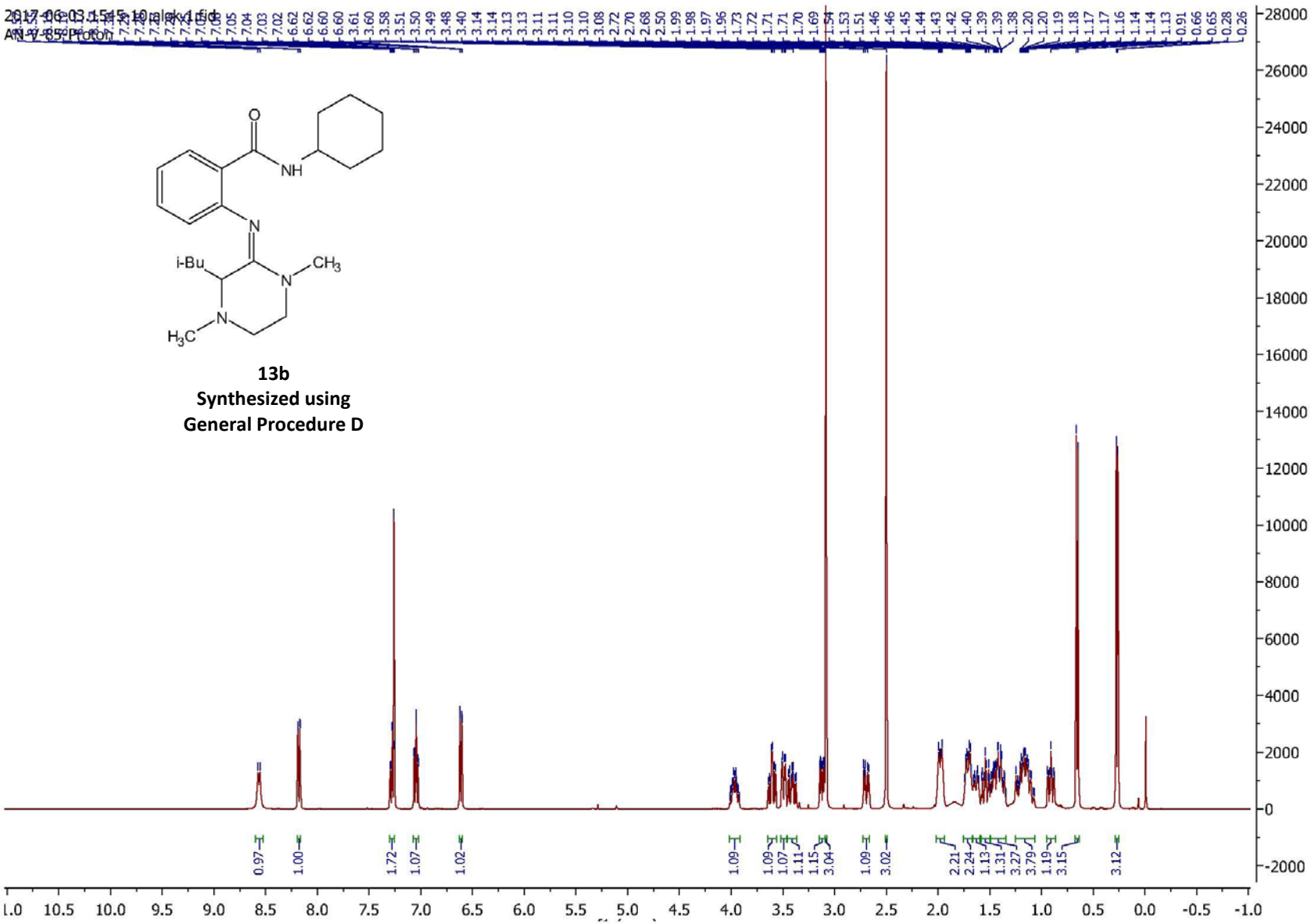


13a
Synthesized using
General Procedure D

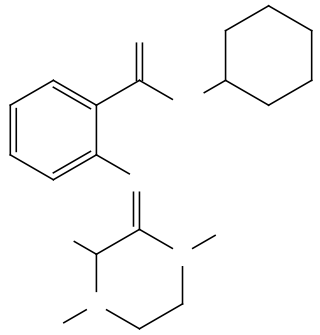


13a
Synthesized using
General Procedure E

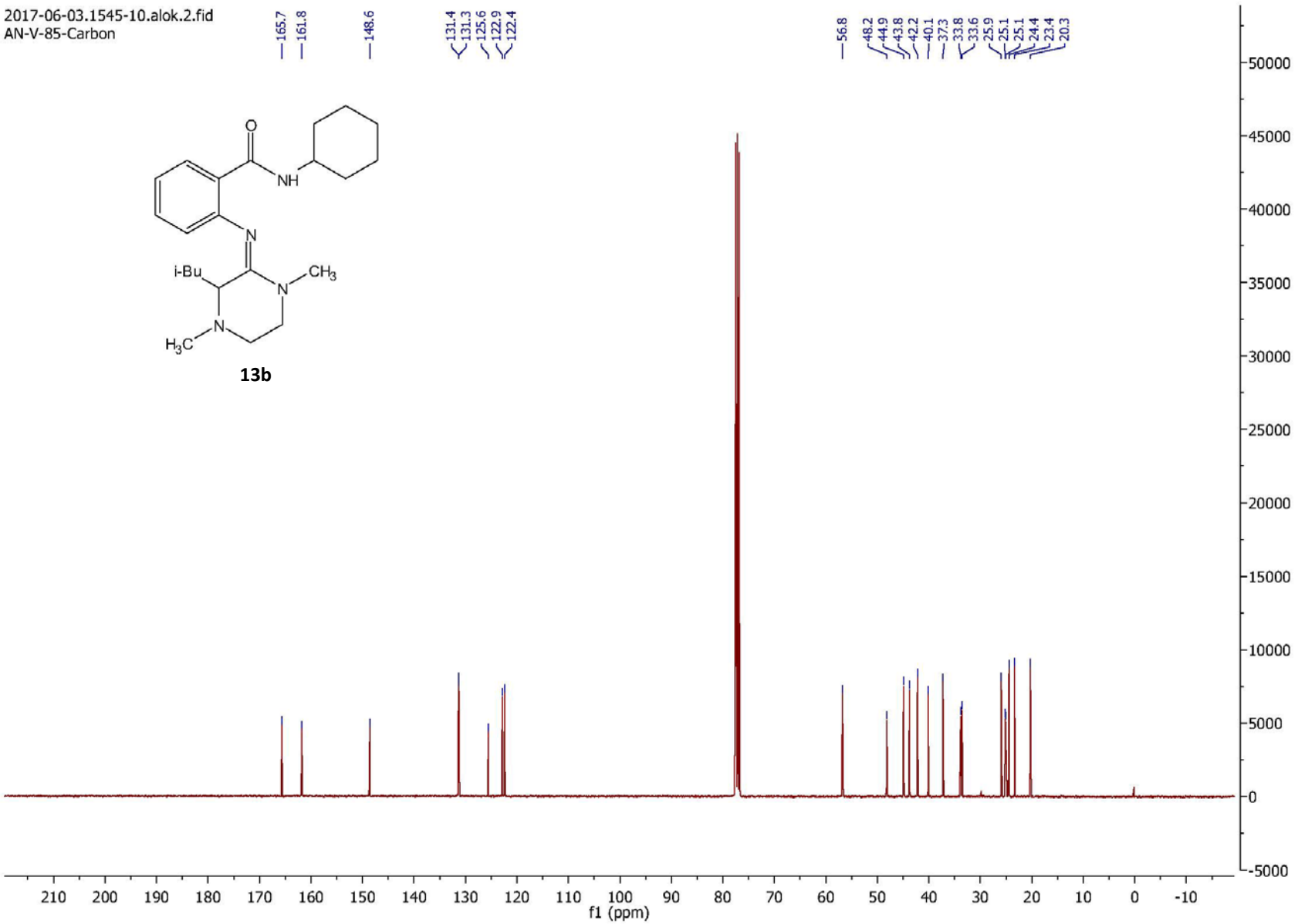

CARBON
20170704.AN-V-140
165.88
160.45
148.49
131.22
131.06
125.83
122.37
122.00
63.91
48.67
48.44
47.81
45.01
37.35
33.82
33.73
32.44
25.86
25.30
20.12
19.23
O
NH
N
i-Pr
CH3
N
N
H3C
13a
f1 (ppm)



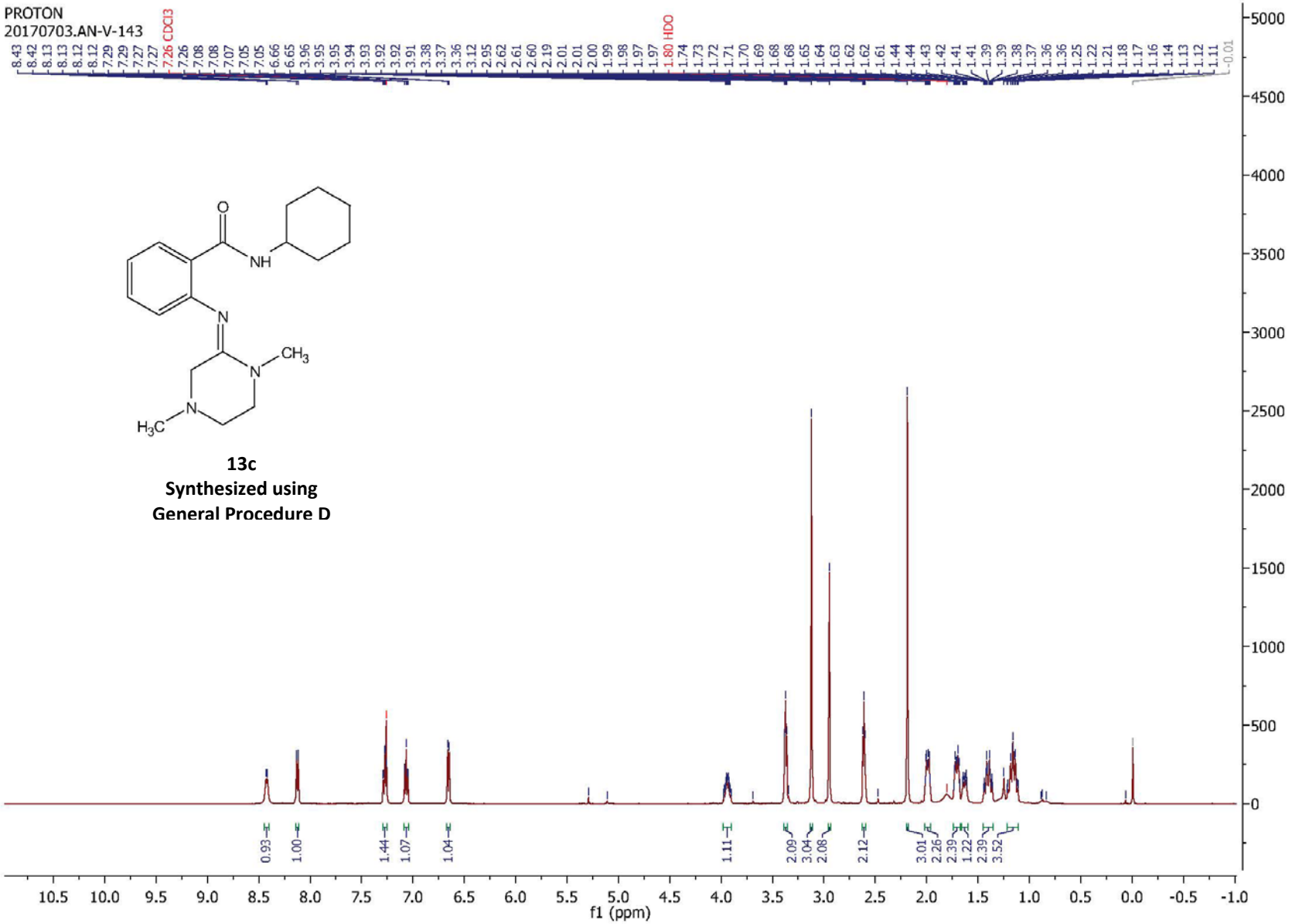
13b
Synthesized using
General Procedure D



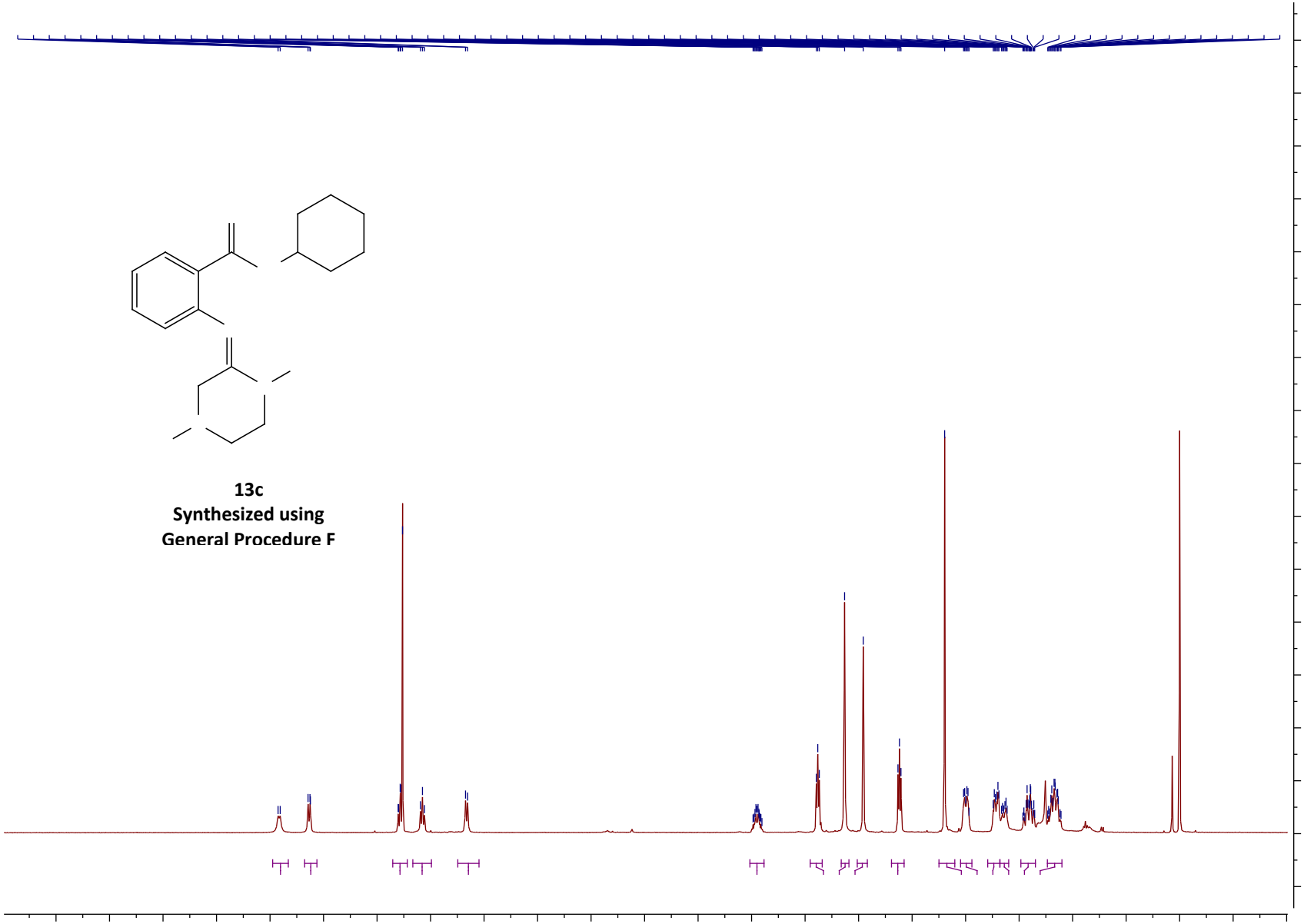
13b
Synthesized using
General Procedure E


2017-06-03.1545-10.alok.2.fid
AN-V-85-Carbon
165.7
161.8
148.6
131.4
131.3
125.6
122.9
122.4
56.8
48.2
44.9
43.8
42.2
40.1
37.3
33.8
33.6
25.9
25.1
24.4
23.4
20.3
O
NH
N
i-Bu
N
CH3
N
H3C
50000
45000
40000
35000
30000
25000
20000
15000
10000
5000
0
-5000
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

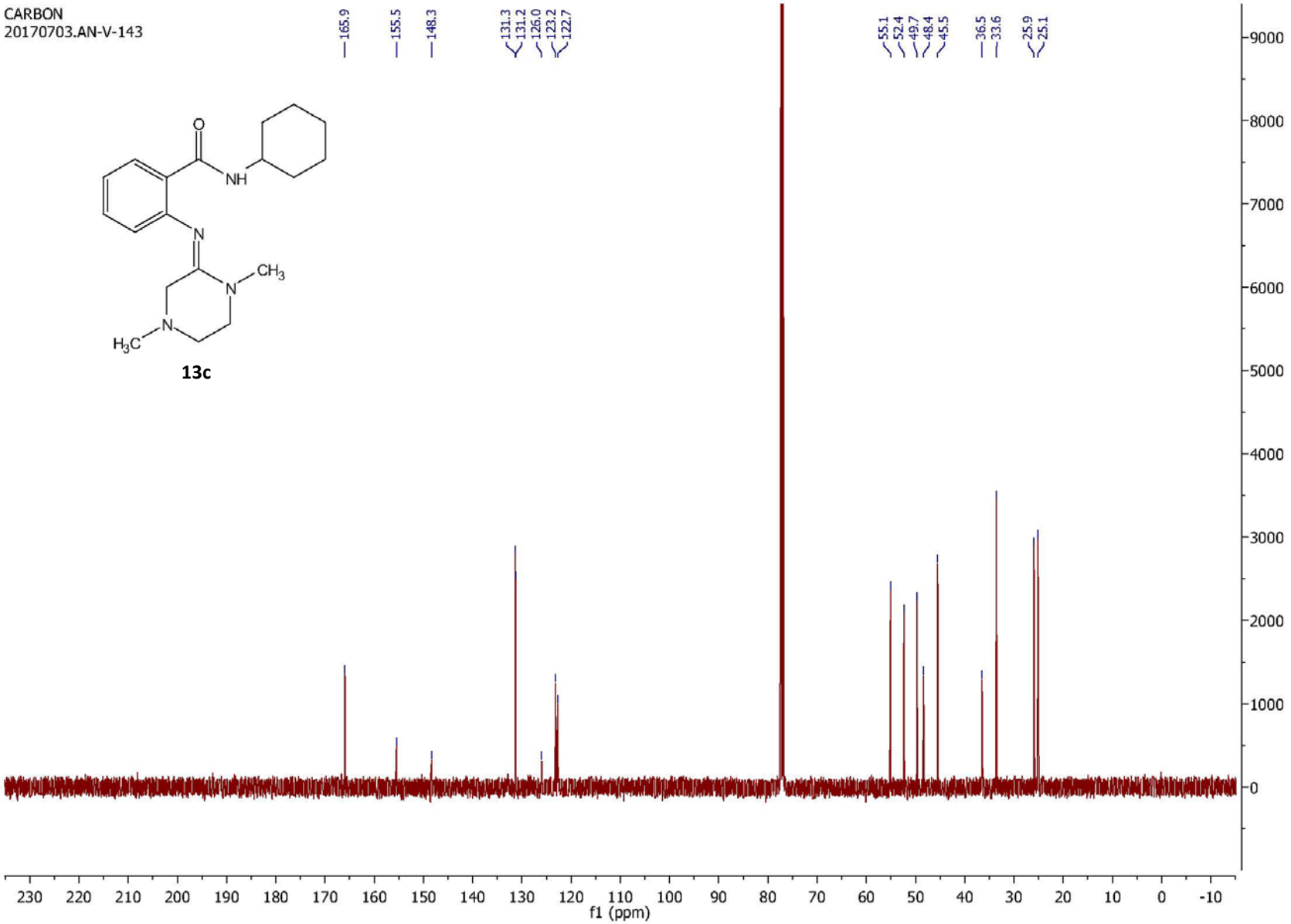
13b

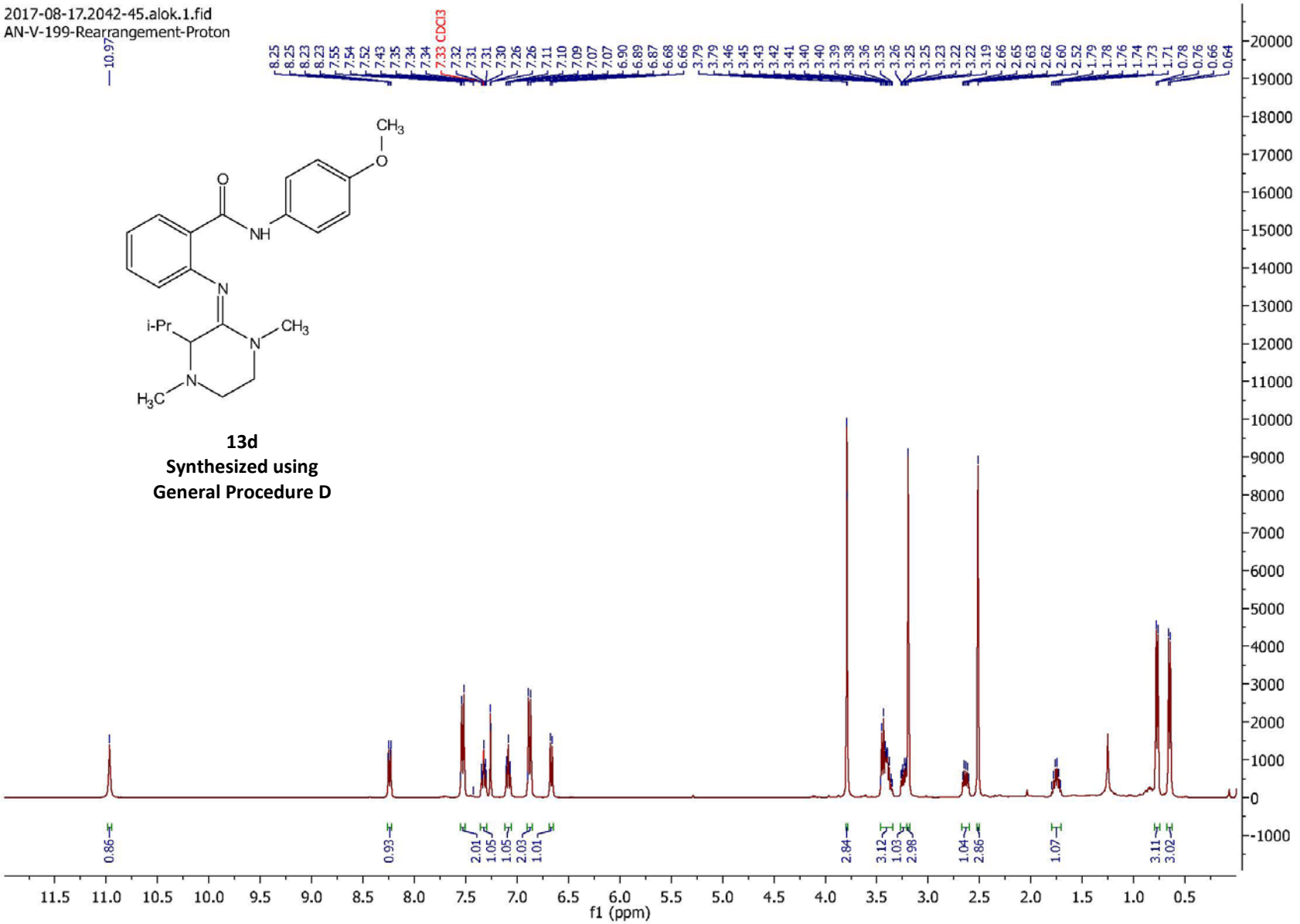


13c
Synthesized using
General Procedure D

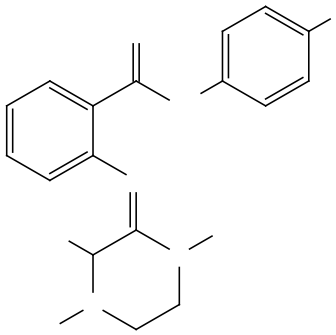


13c
Synthesized using
General Procedure F

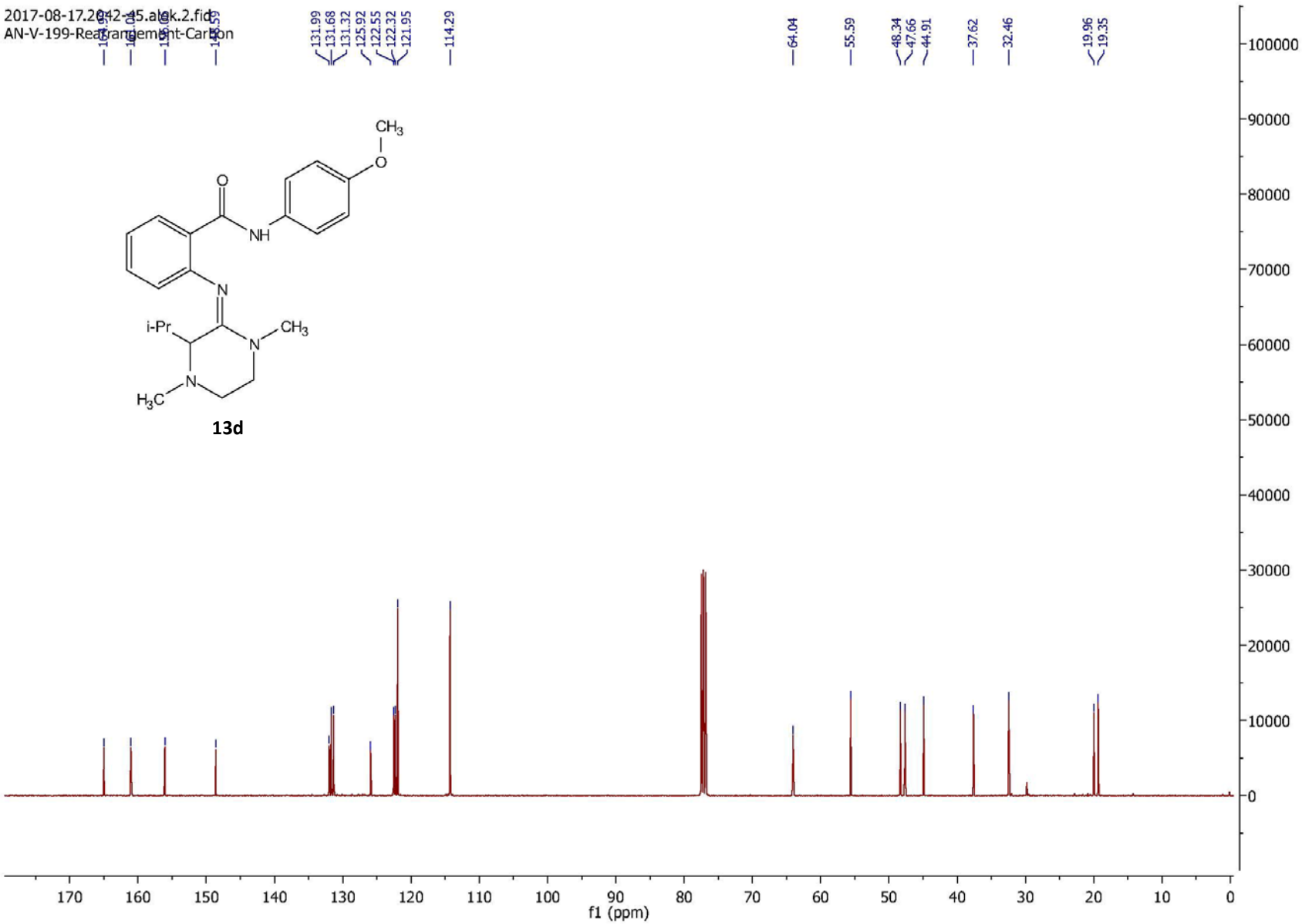

CARBON
20170703.AN-V-143
165.9
155.5
148.3
131.3
131.2
126.0
123.2
122.7
55.1
52.4
49.7
48.4
45.5
36.5
33.6
25.9
25.1
O
NH
N
N
CH3
N
H3C
13c
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

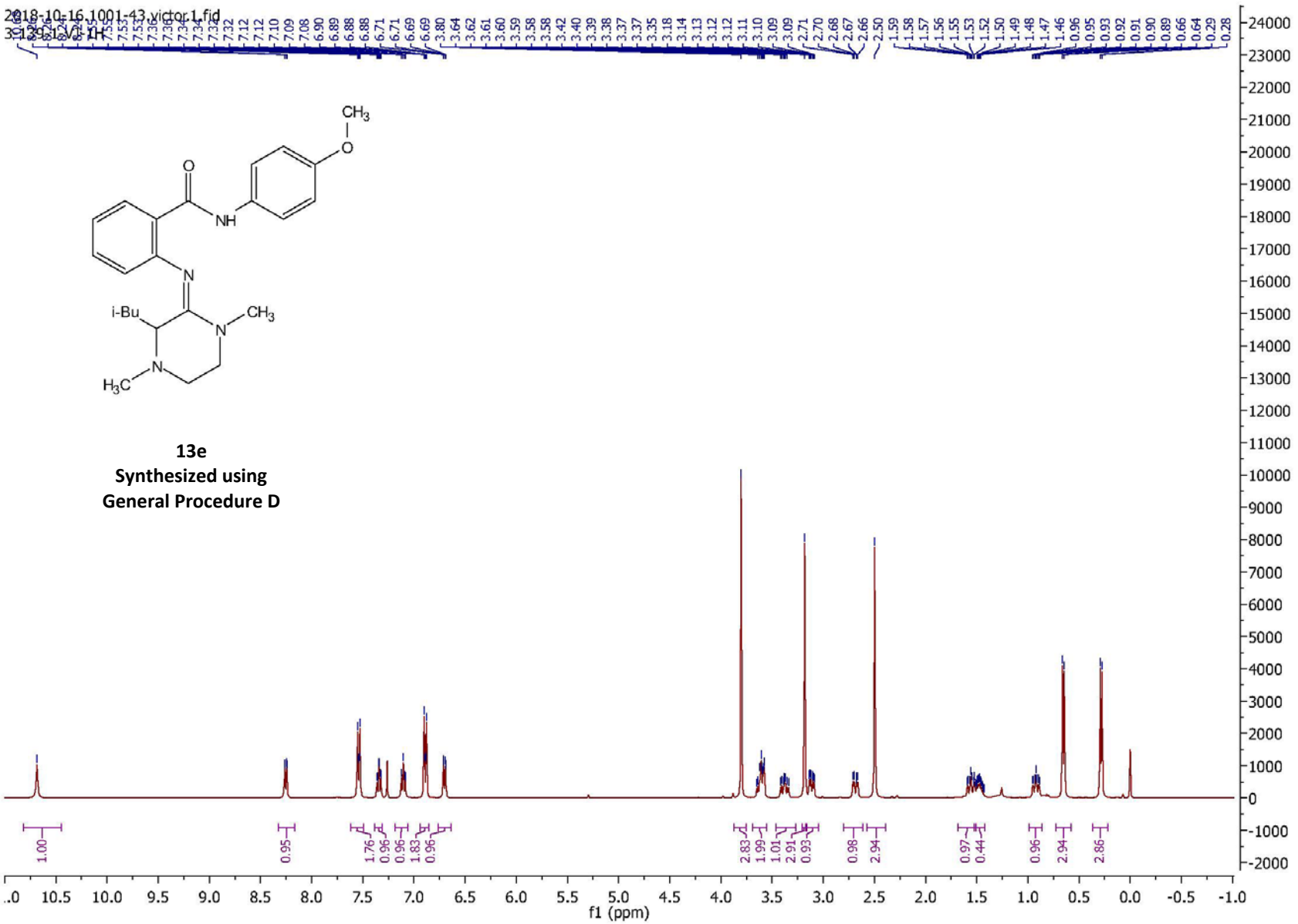


13d
Synthesized using
General Procedure D

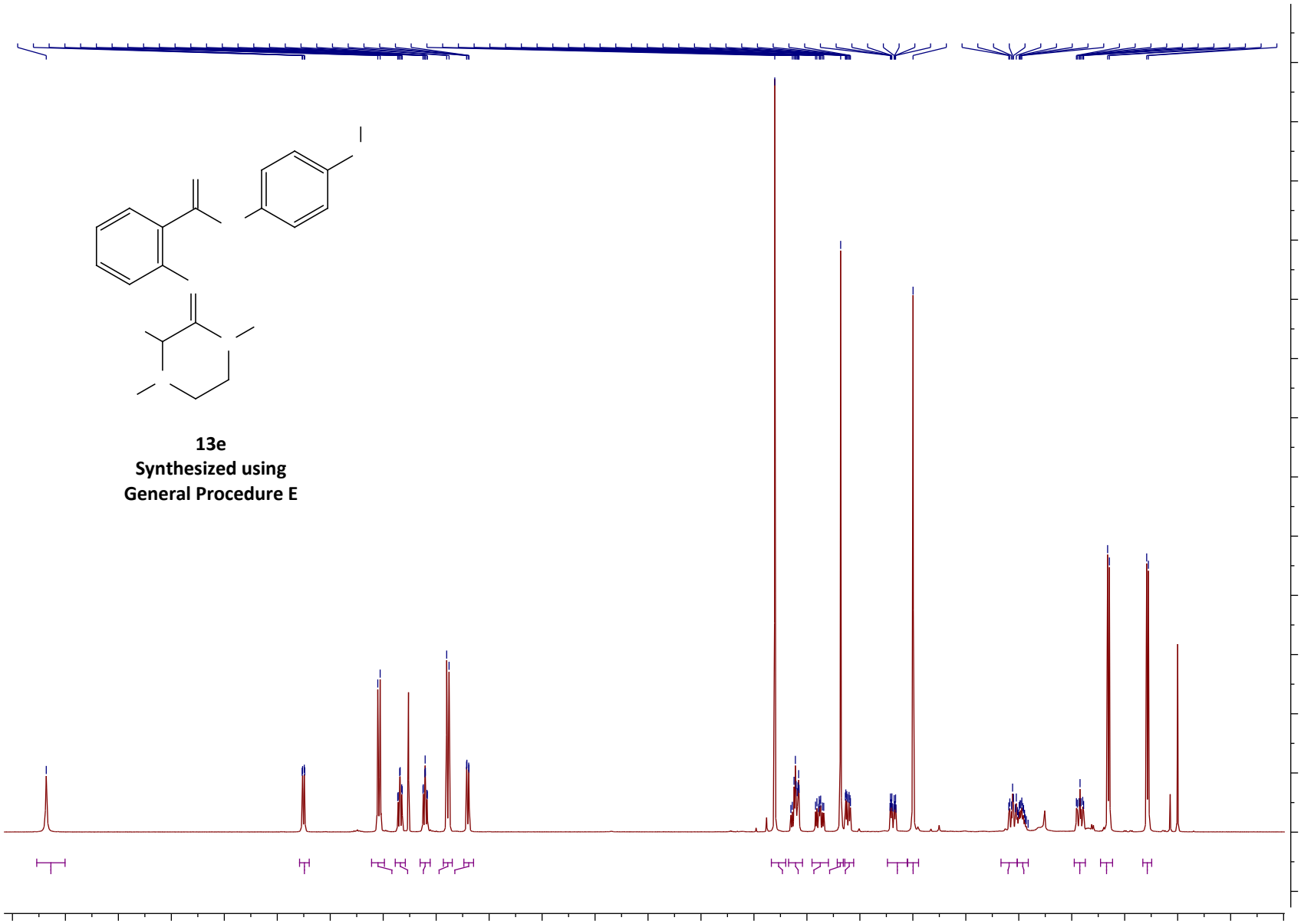


13d
Synthesized using
General Procedure E

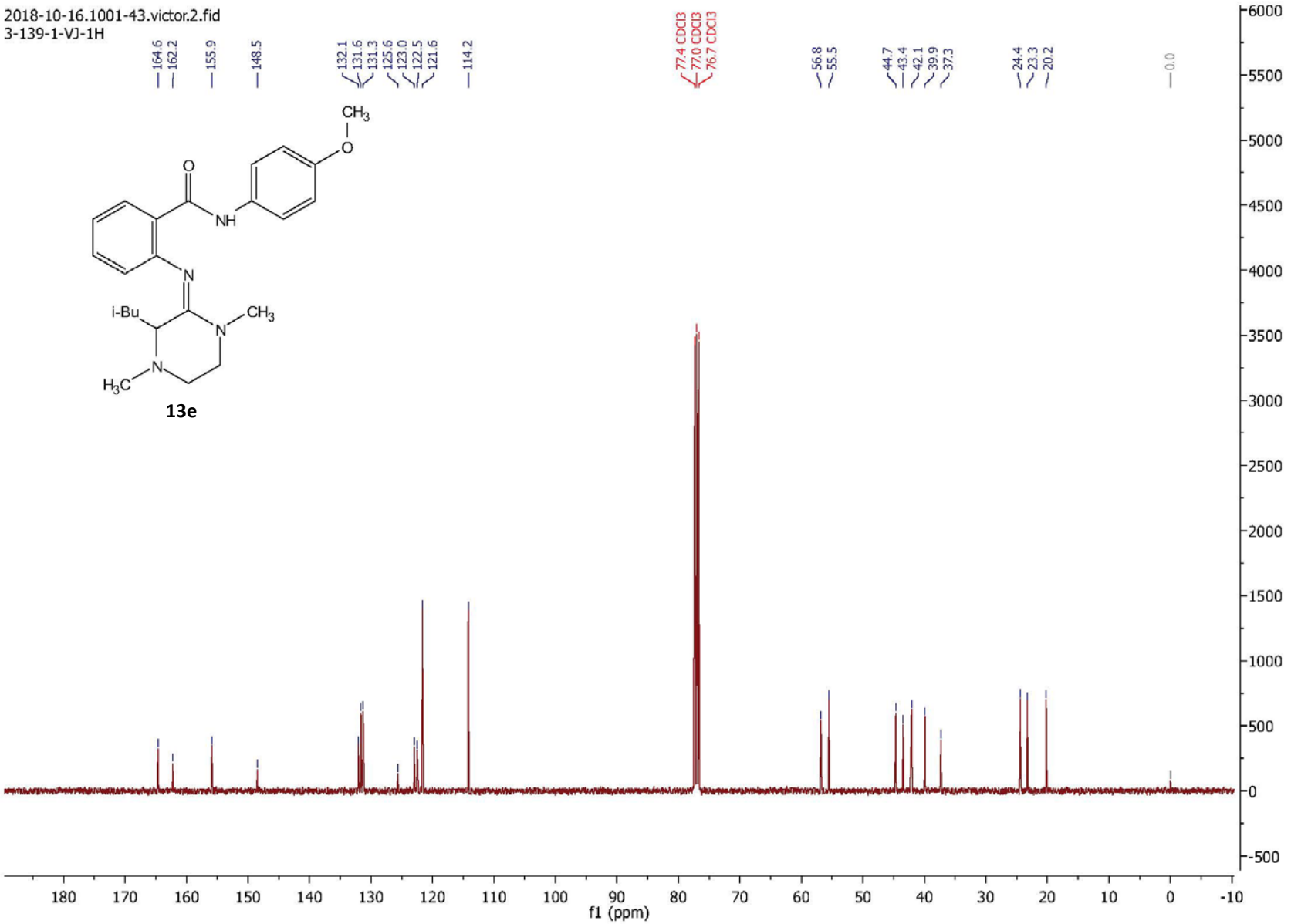

2017-08-17.2042-45.alak.2.fid
AN-V-199-Rearrangement-Carbon
131.99
131.68
131.32
125.92
122.55
122.32
121.95
114.29
64.04
55.59
48.34
47.66
44.91
37.62
32.46
19.96
19.35
CH3
O
NH
N
i-Pr
CH3
N
N
H3C
13d
f1 (ppm)



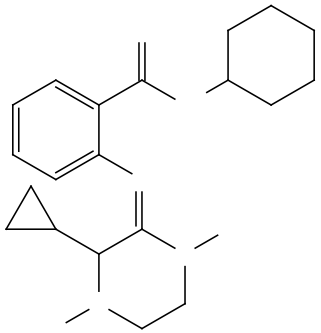
13e
Synthesized using
General Procedure D



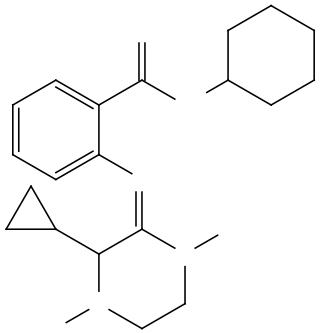
13e
Synthesized using
General Procedure E


2018-10-16.1001-43.victor.2.fid
3-139-1-VJ-1H
164.6
162.2
155.9
148.5
132.1
131.6
131.3
125.6
123.0
122.5
121.6
114.2
77.4 CDCl3
77.0 CDCl3
76.7 CDCl3
56.8
55.5
44.7
43.4
42.1
39.9
37.3
24.4
23.3
20.2
0.0
CH3
O
O
NH
N
i-Bu
CH3
N
N
H3C
f1 (ppm)

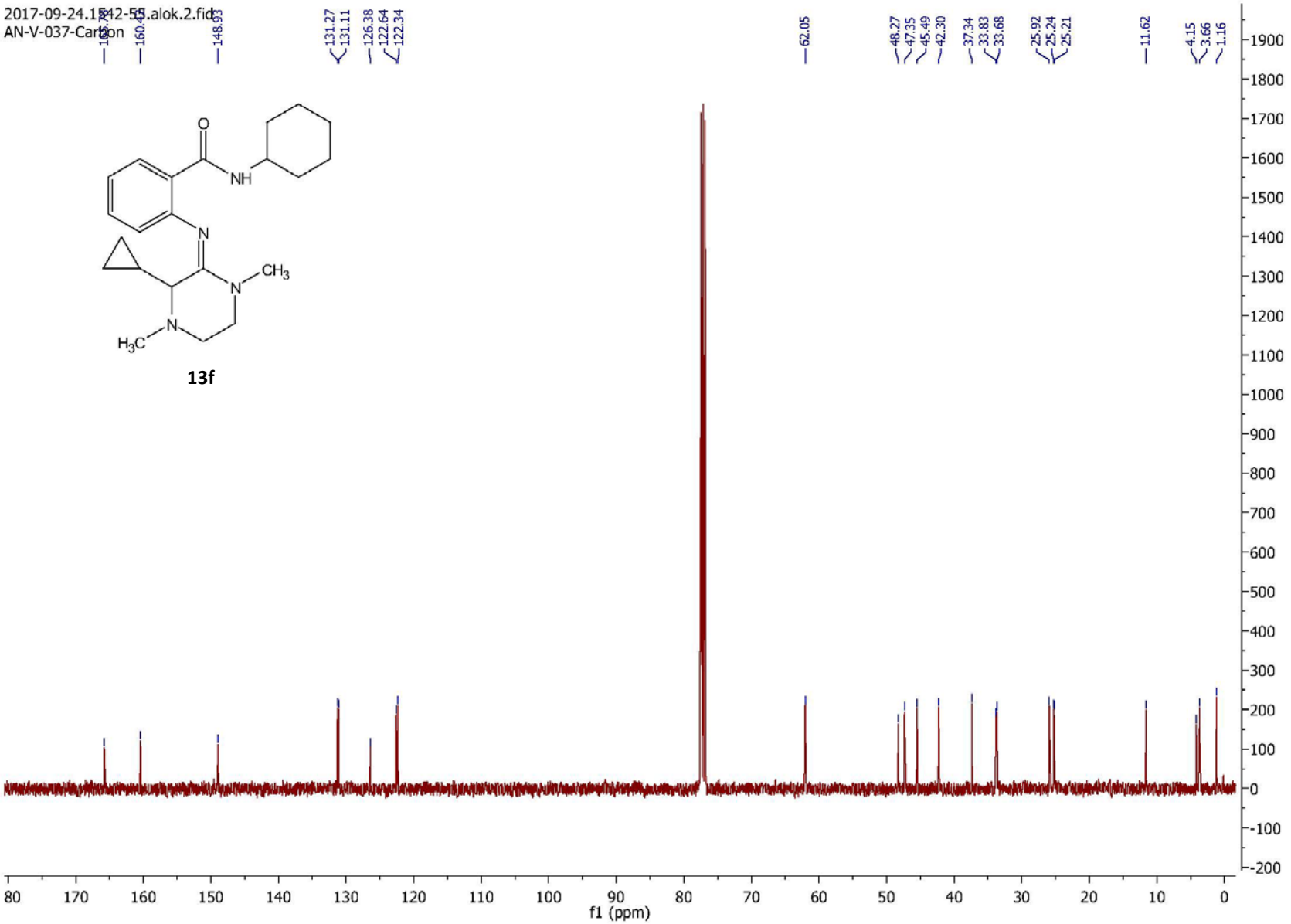
13e



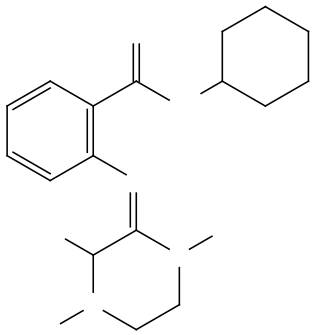
13f
Synthesized using
General Procedure D



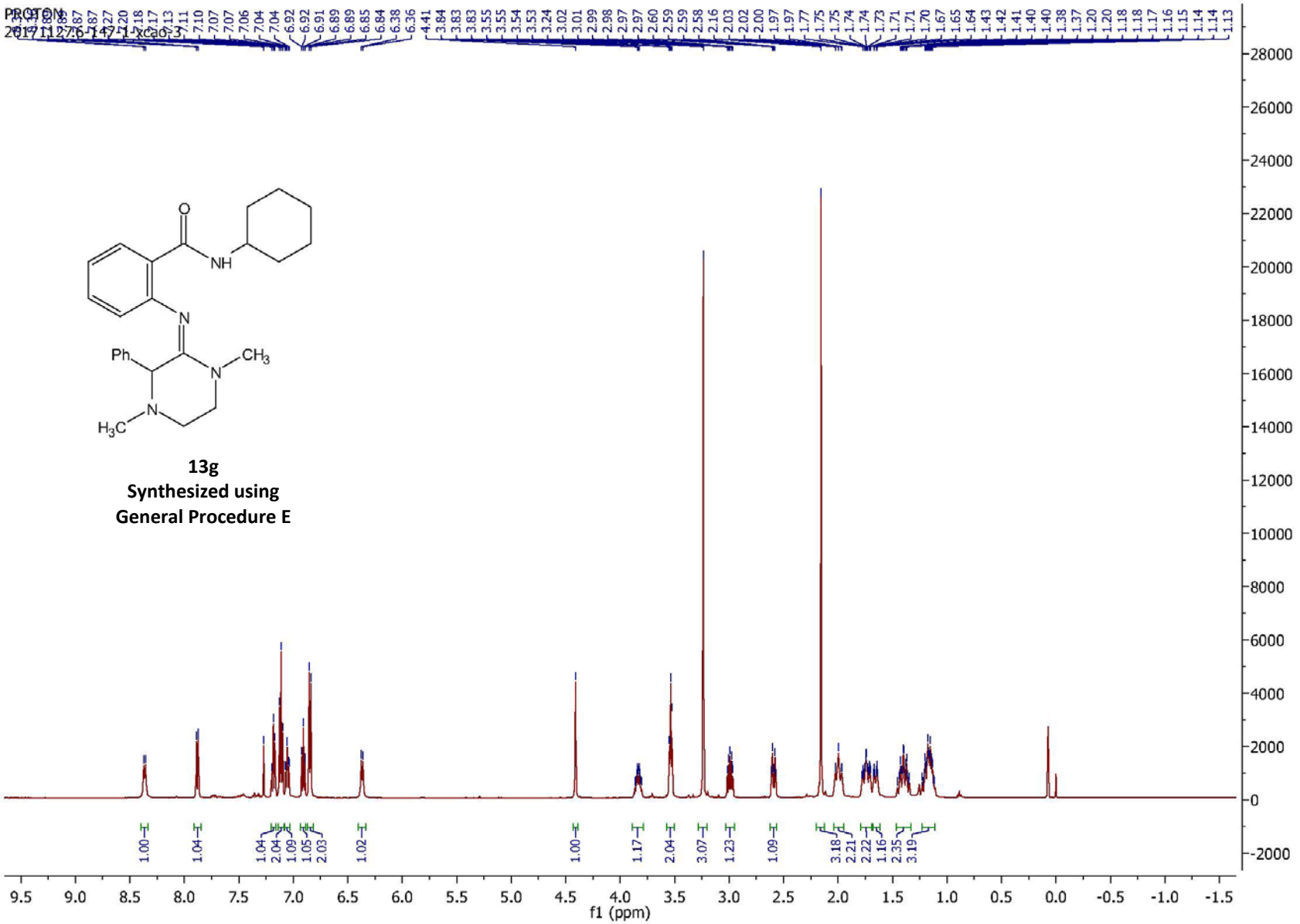
13f
Synthesized using
General Procedure E


2017-09-24.1542-55.alok.2.fid
AN-V-037-Carbon
160.45
148.93
131.27
131.11
126.38
122.64
122.34
62.05
48.27
47.35
45.49
42.30
37.34
33.83
33.68
25.92
25.24
25.21
11.62
4.15
3.66
1.16
O
NH
N
N
CH3
N
H3C
f1 (ppm)

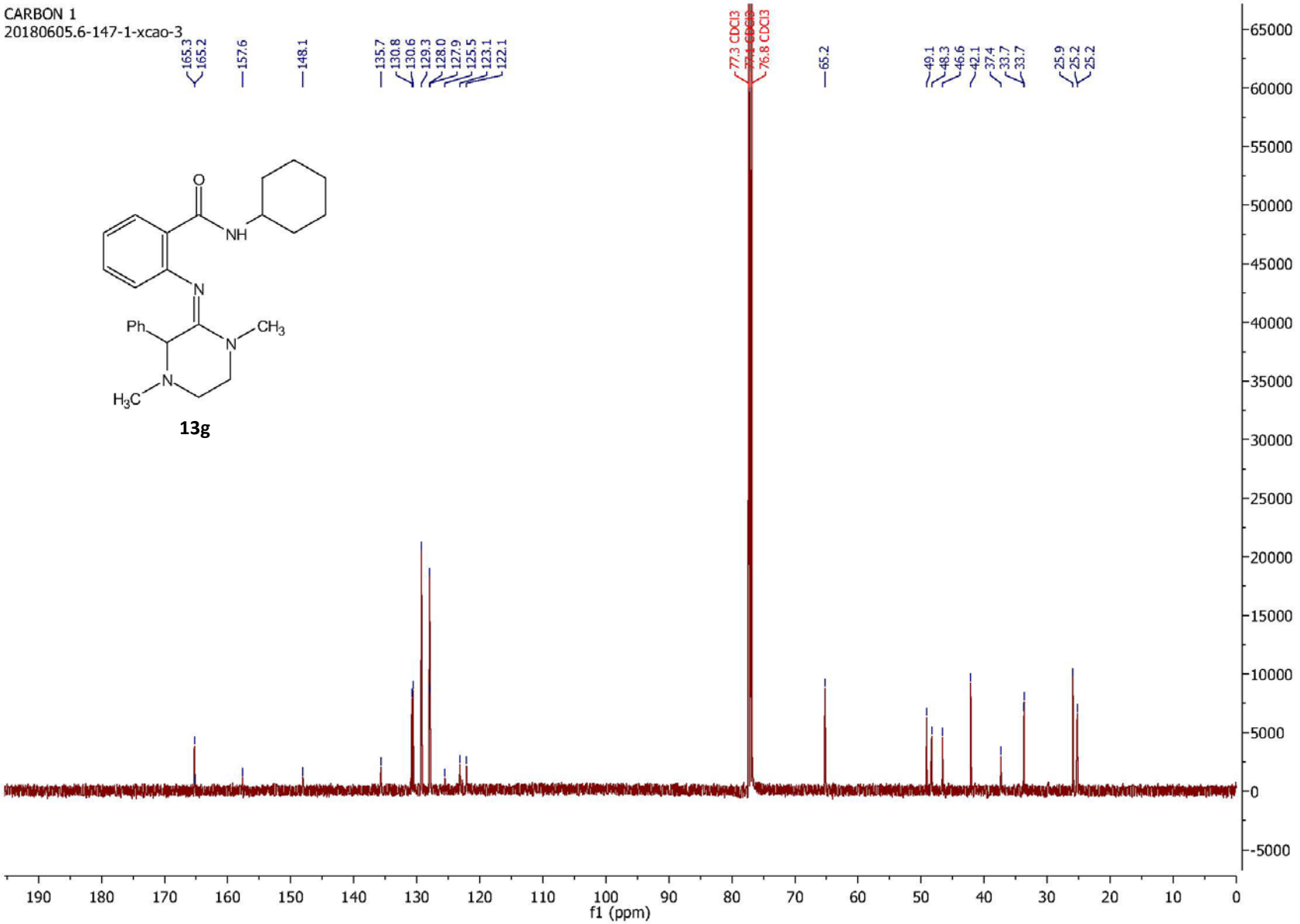
13f



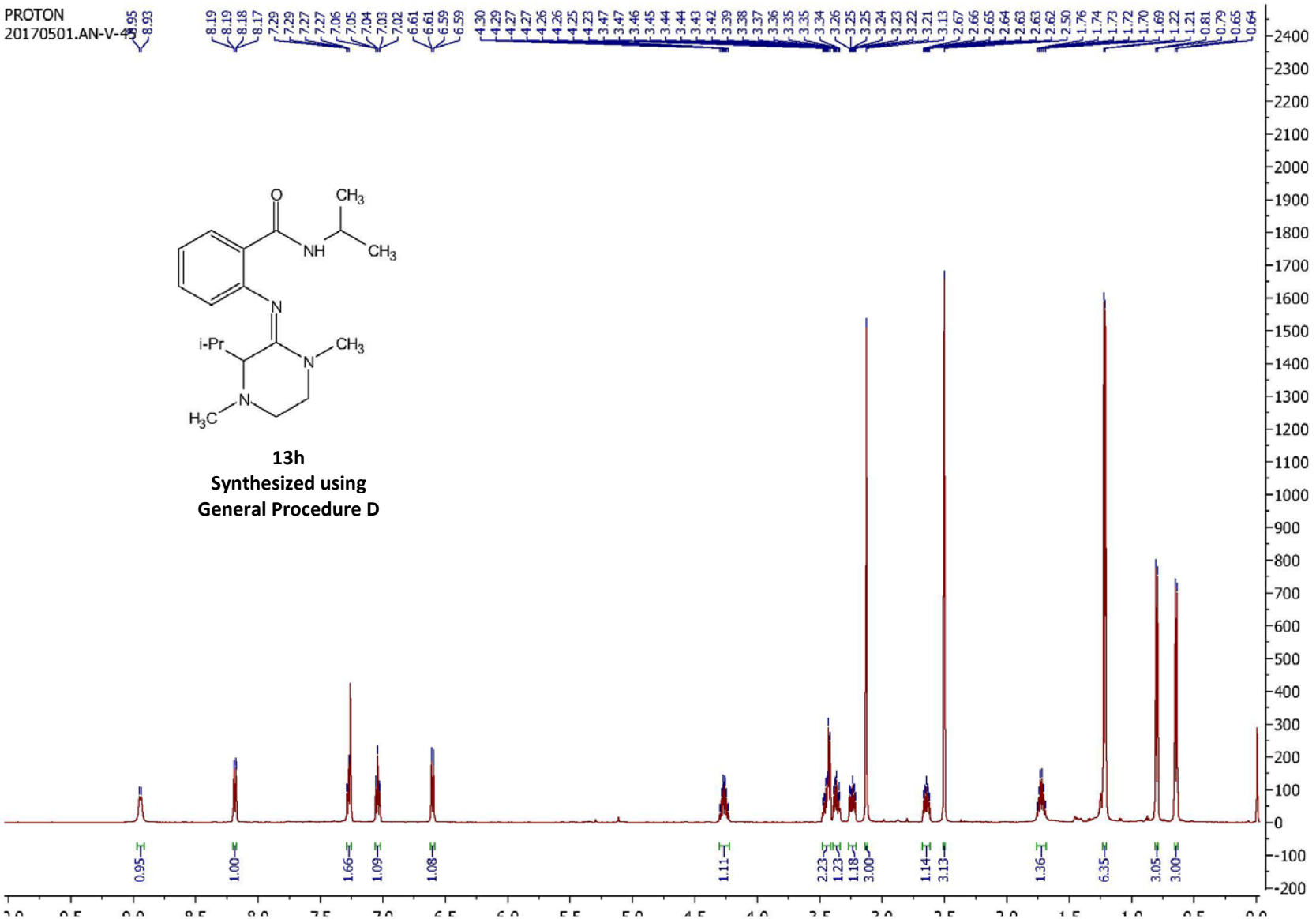
13g
Synthesized using
General Procedure D



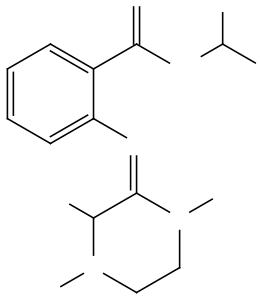
13g
Synthesized using
General Procedure E


CARBON 1
20180605.6-147-1-xcao-3
165.3
165.2
157.6
148.1
135.7
130.8
130.6
129.3
128.0
127.9
125.5
123.1
122.1
77.3 CDCl3
76.8 CDCl3
65.2
49.1
48.3
46.6
42.1
37.4
33.7
33.7
25.9
25.2
25.2
O
NH
N
Ph
N
CH3
N
H3C
65000
60000
55000
50000
45000
40000
35000
30000
25000
20000
15000
10000
5000
0
-5000
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

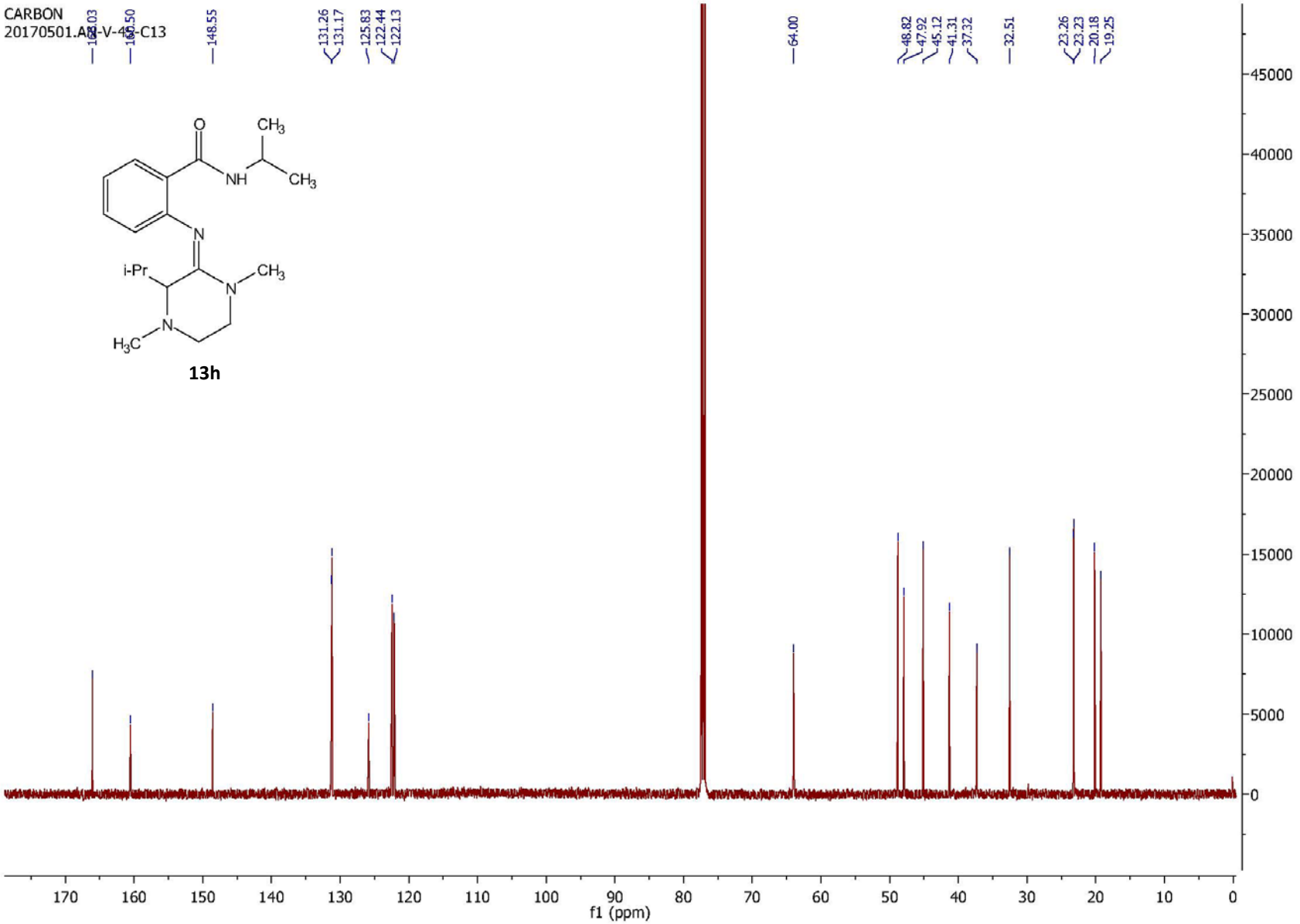
13g



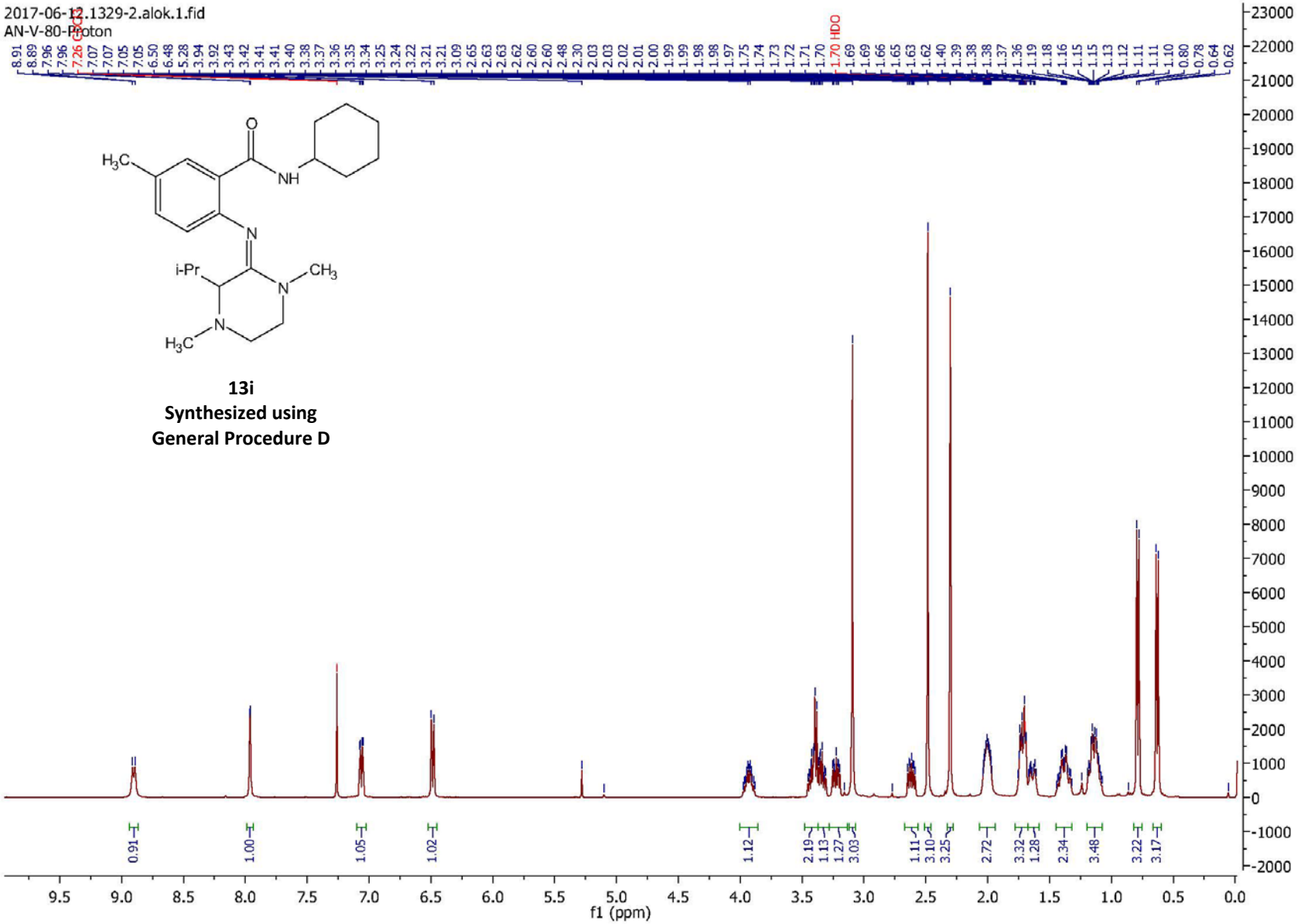
13h
Synthesized using
General Procedure D



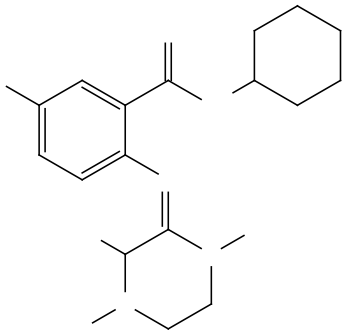
13h
Synthesized using
General Procedure E


CARBON
20170501.AM-V-49-C13
165.03
160.50
148.55
131.26
131.17
125.83
122.44
122.13
64.00
48.82
47.92
45.12
41.31
37.32
32.51
23.26
23.23
20.18
19.25
O
CH3
NH
CH3
N
i-Pr
CH3
N
N
H3C
45000
40000
35000
30000
25000
20000
15000
10000
5000
0
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

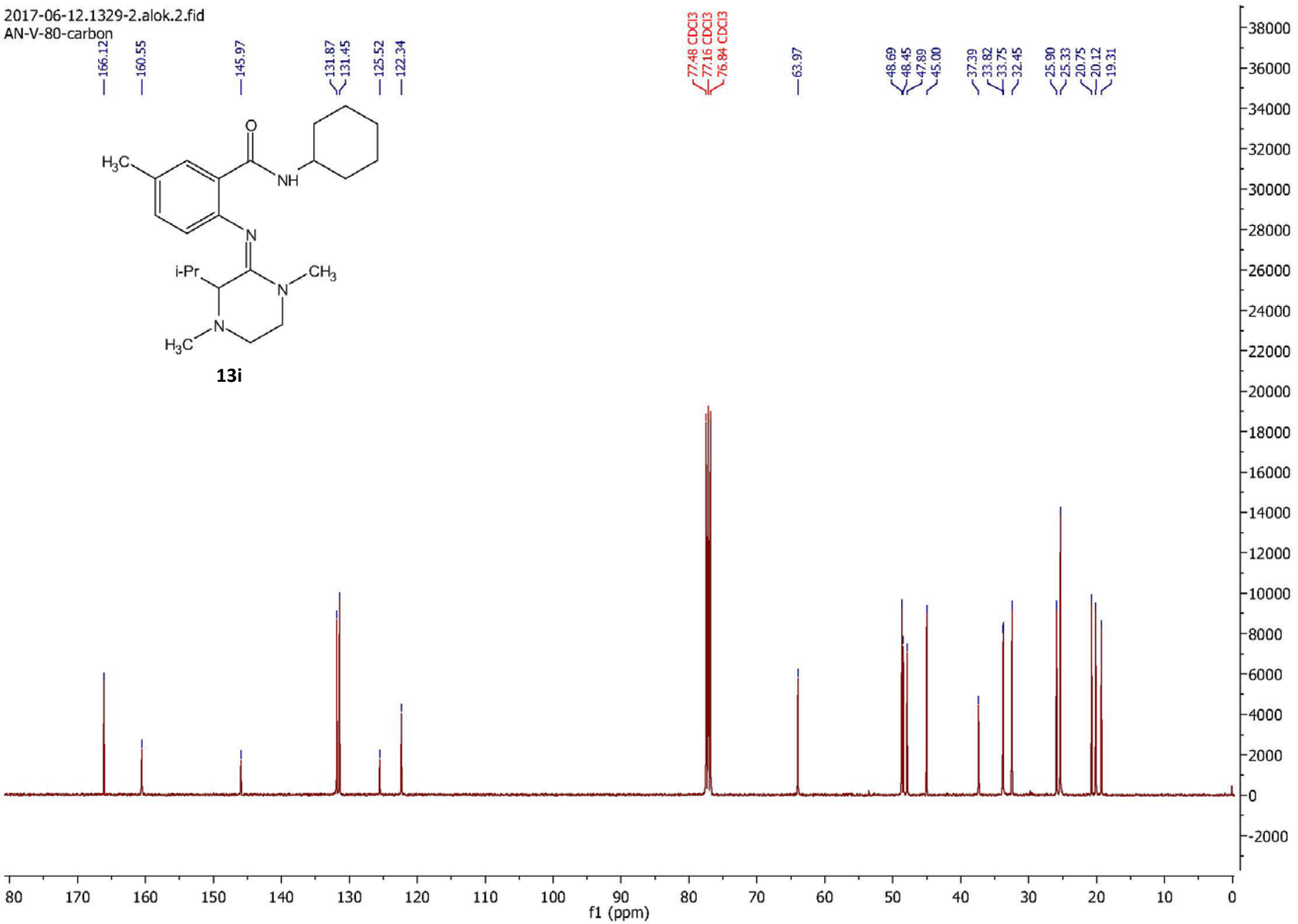
13h

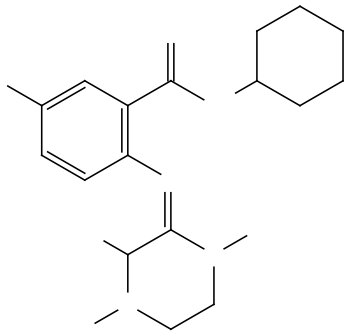


13i
Synthesized using
General Procedure D

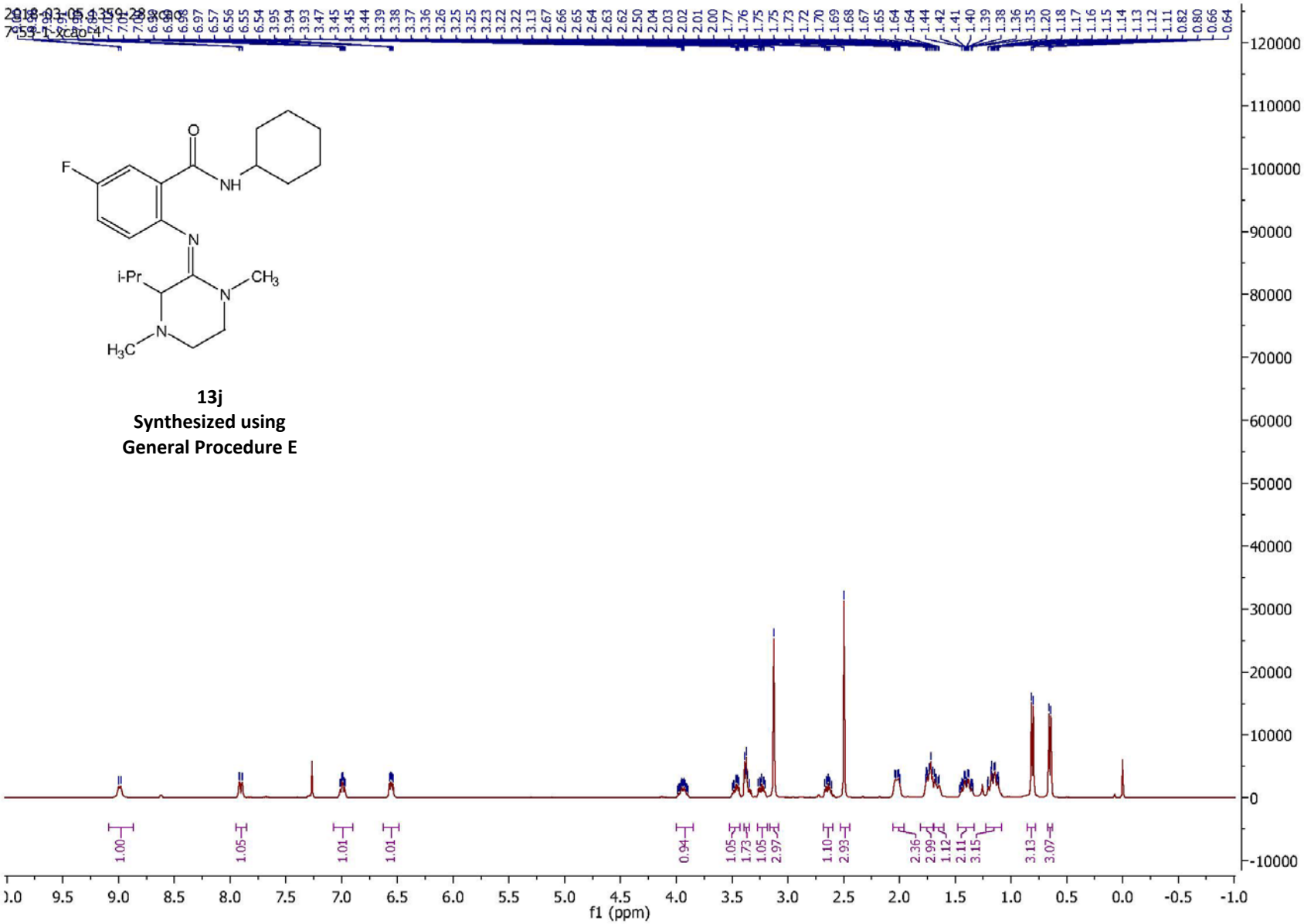


13i
Synthesized using
General Procedure E


2017-06-12.1329-2.alok.2.fid
AN-V-80-carbon
166.12
160.55
145.97
131.87
131.45
125.52
122.34
77.48 CDCl3
77.16 CDCl3
76.84 CDCl3
63.97
48.69
48.45
47.89
45.00
37.39
33.82
33.75
32.45
25.90
25.33
20.75
20.12
19.31
H3C
O
NH
N
i-Pr
CH3
N
N
H3C
13i
f1 (ppm)

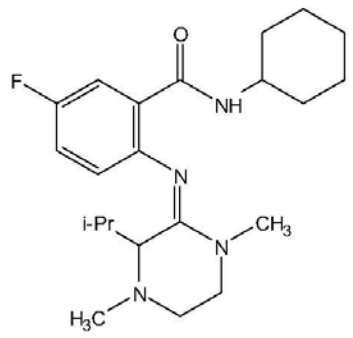


13j
Synthesized using
General Procedure D

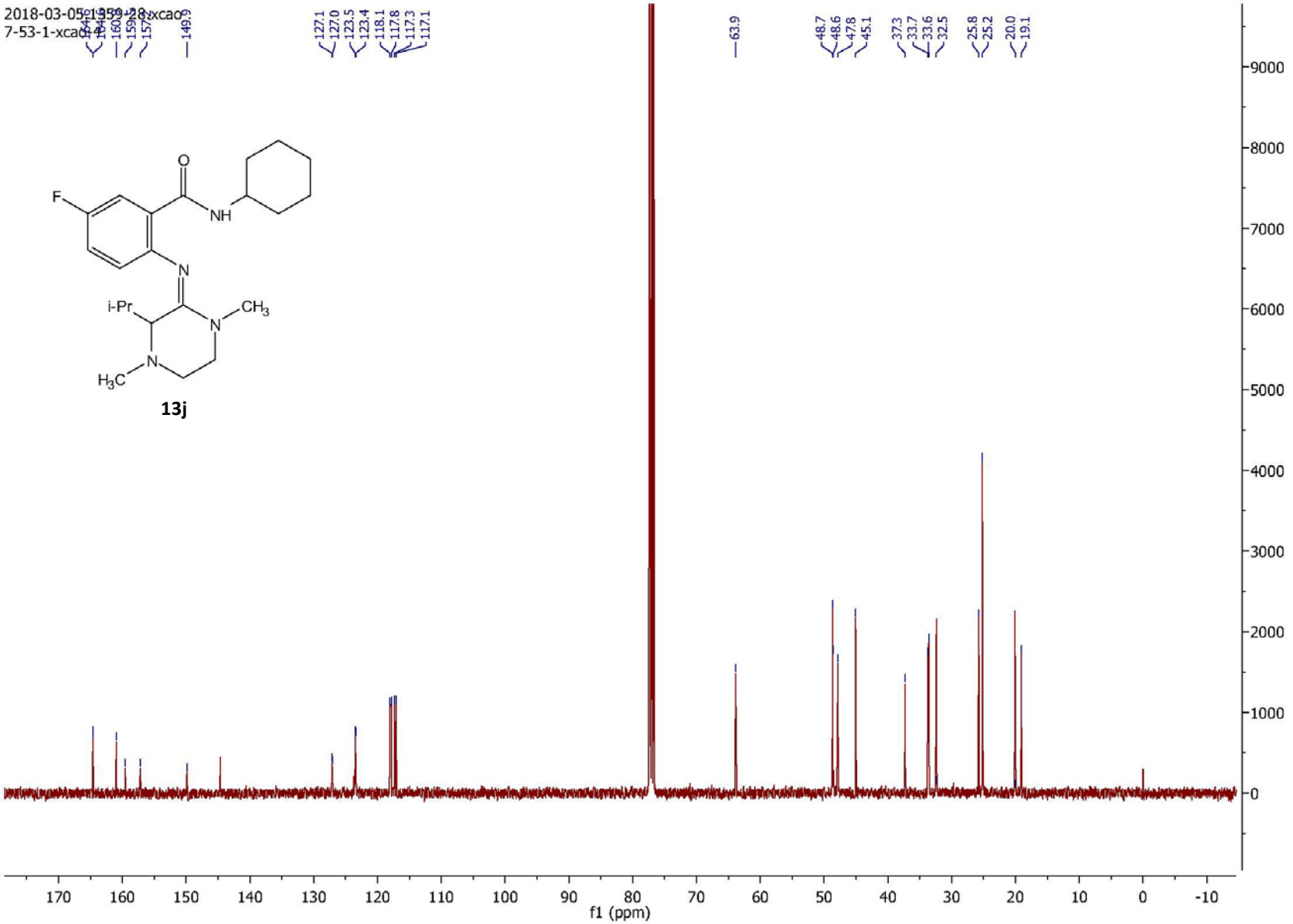


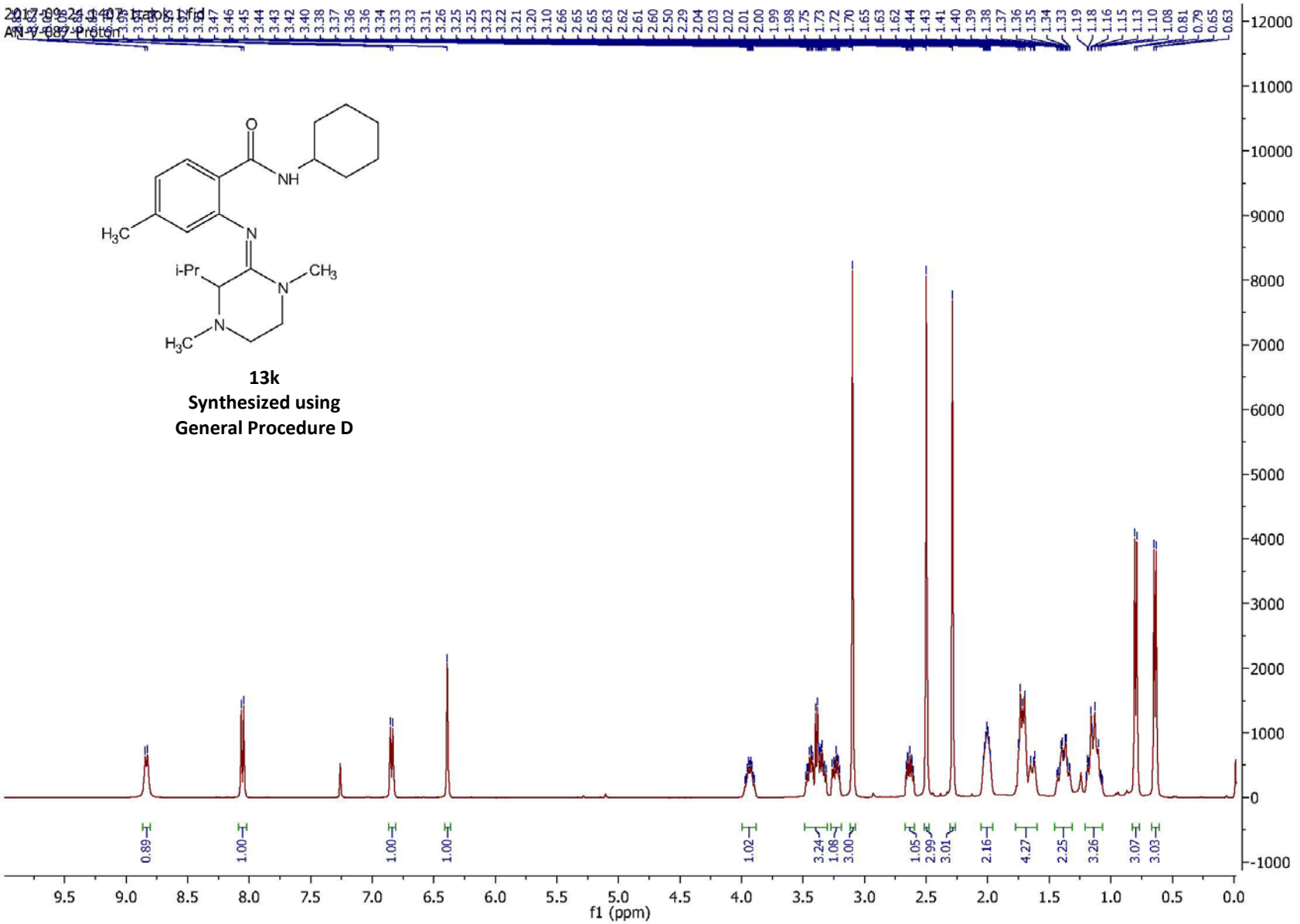
13j
Synthesized using
General Procedure E

2018-10-23.1736-1.victor.2.fid
7-53-1-xcao-4

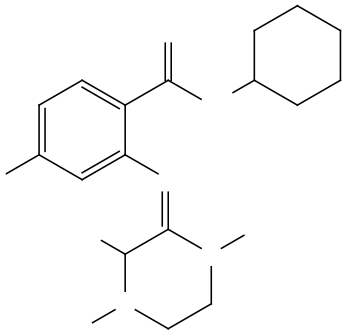


13j

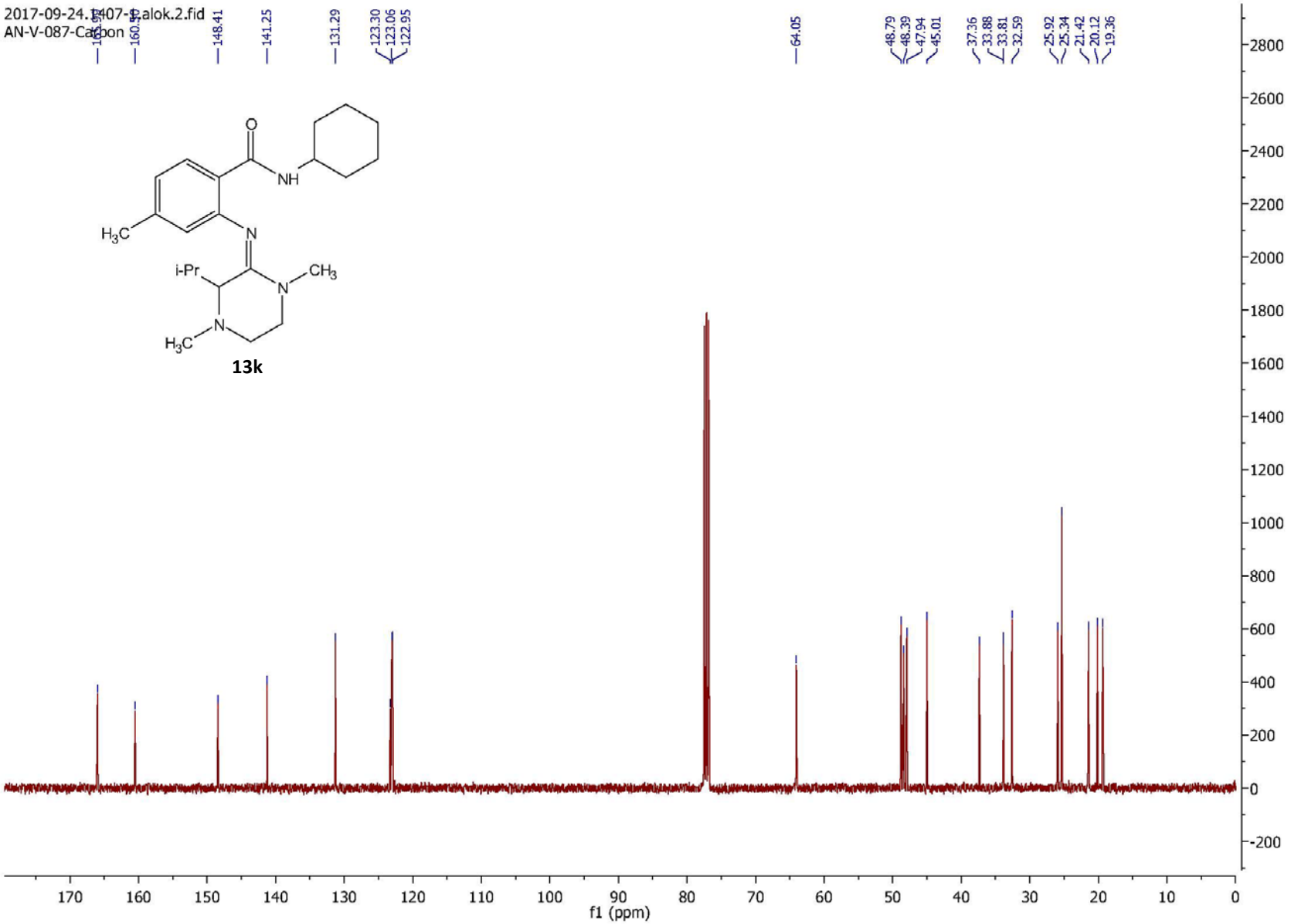

2018-03-05 1959-28.xcao
7-53-1-xcao-1-1
127.1
127.0
123.5
123.4
118.1
117.8
117.3
117.1
63.9
48.7
48.6
47.8
45.1
37.3
33.7
33.6
32.5
25.8
25.2
20.0
19.1
F
O
NH
N
i-Pr
CH3
N
N
H3C
13j
f1 (ppm)

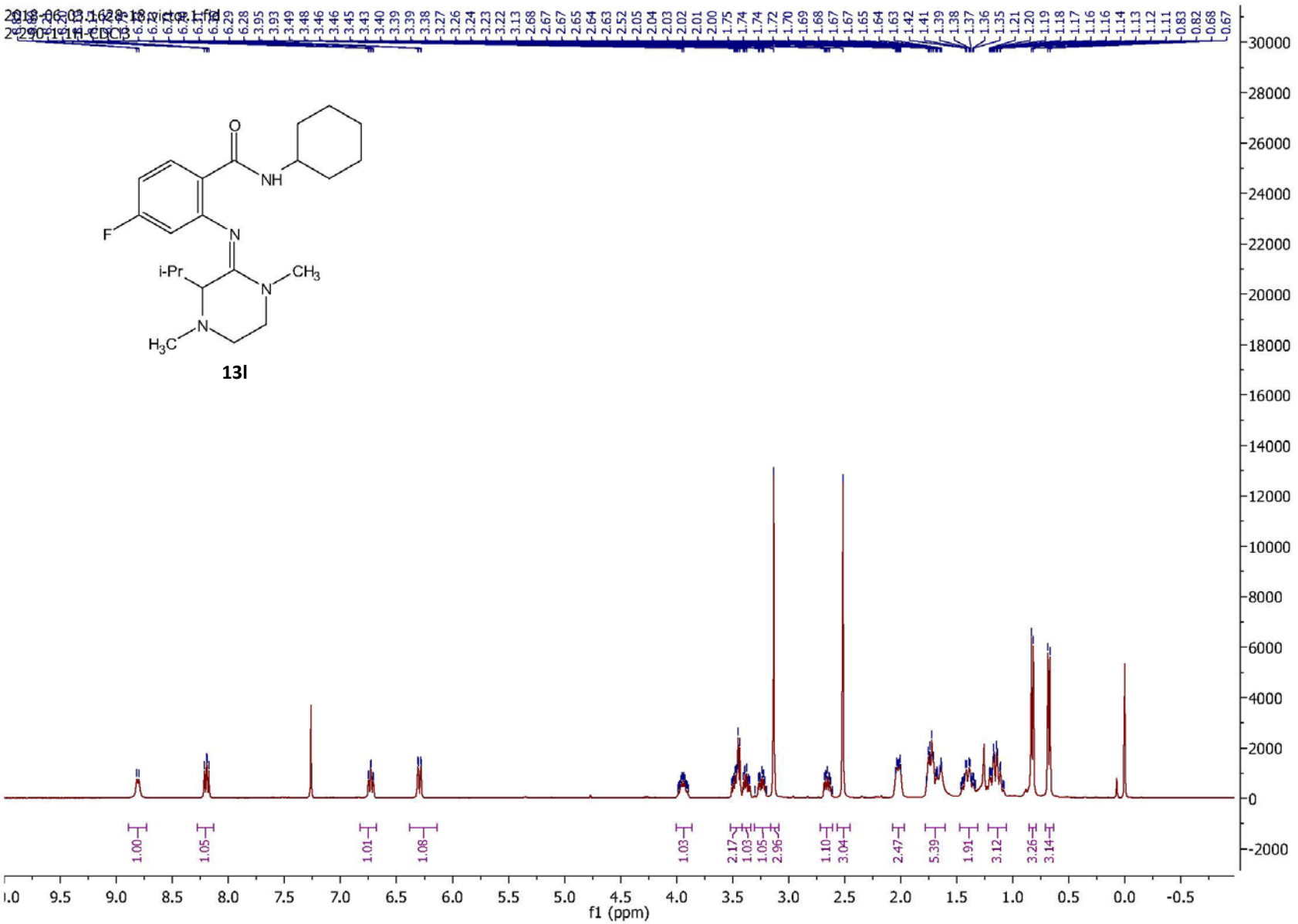


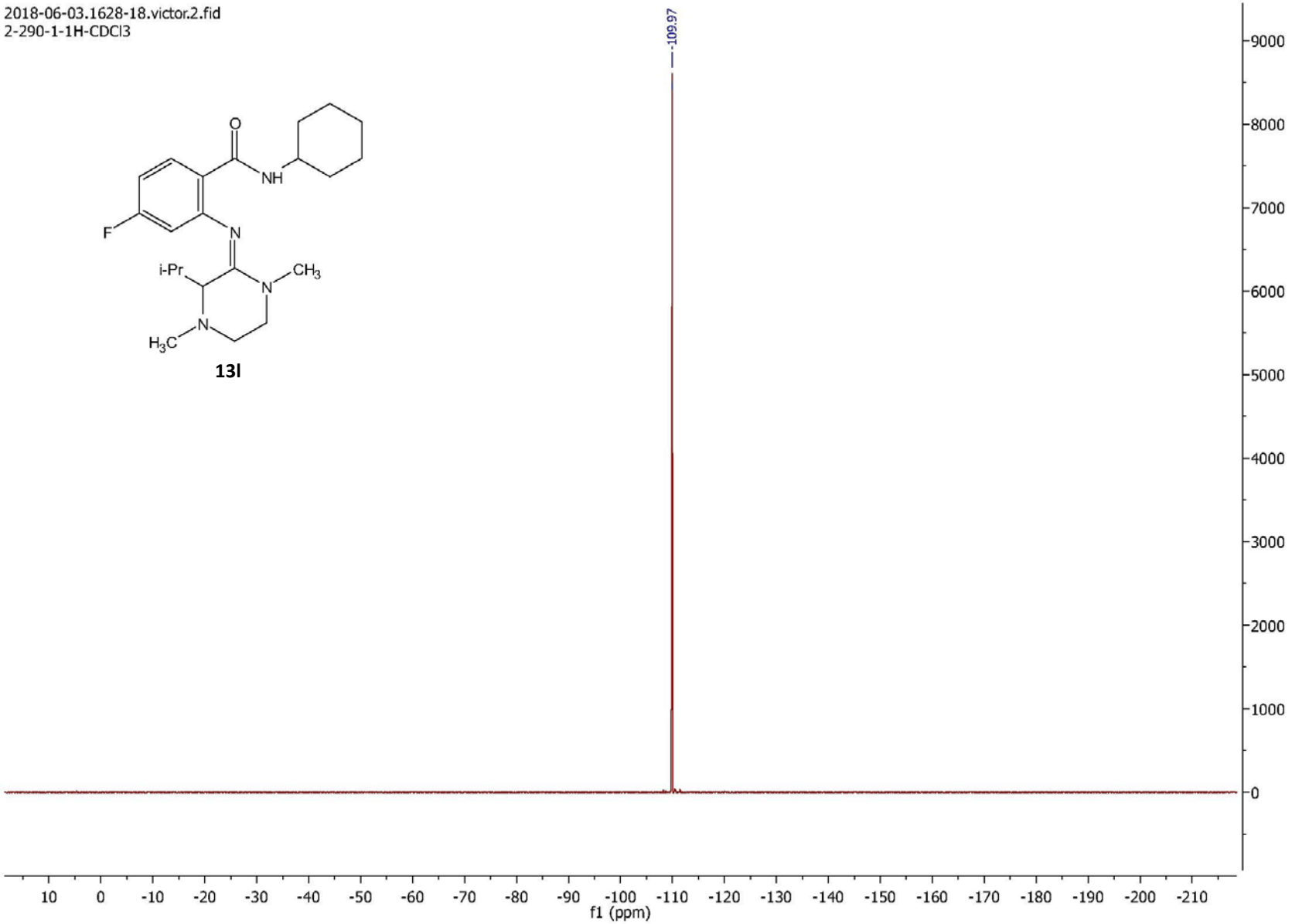
13k
Synthesized using
General Procedure D

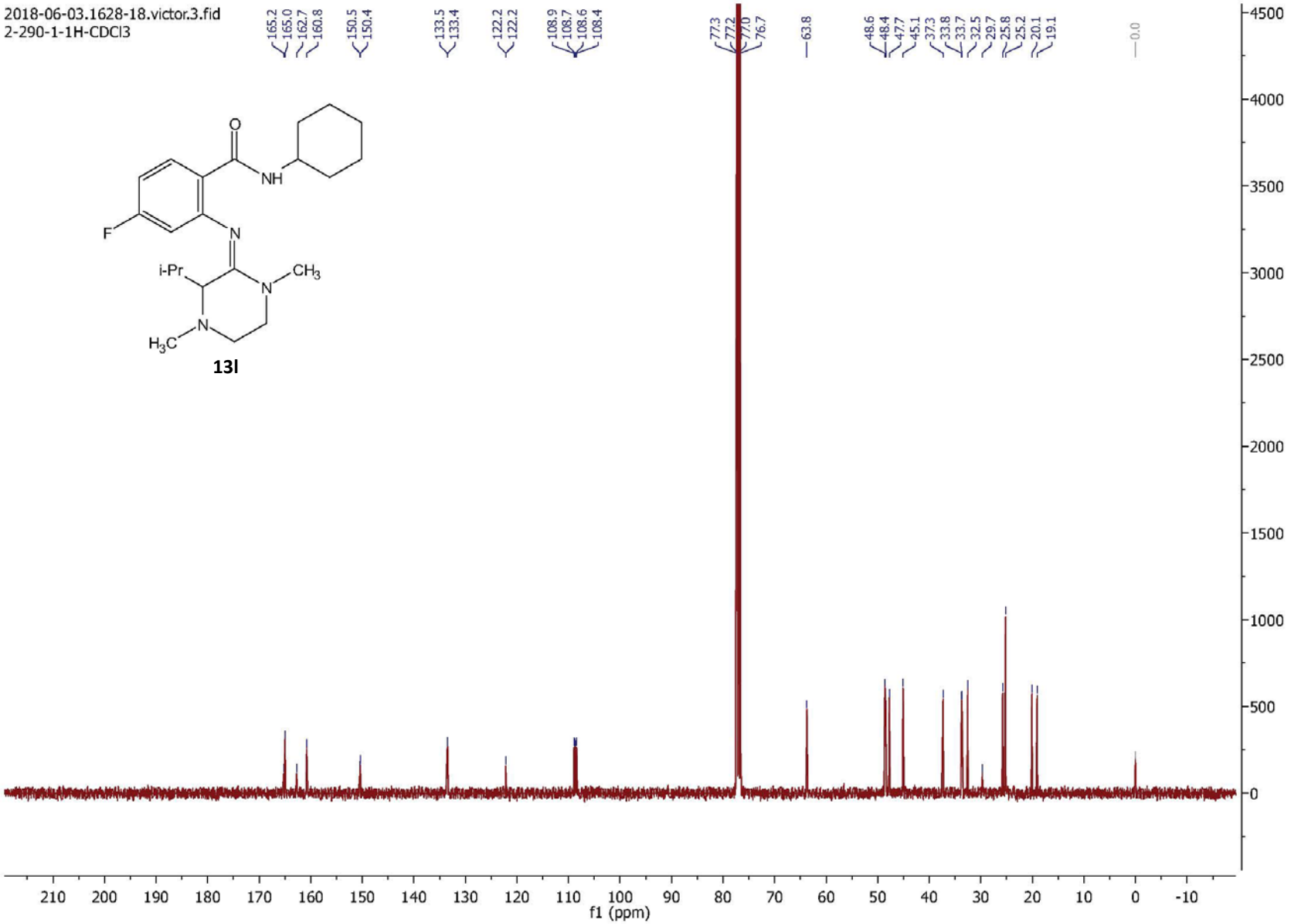


13k
Synthesized using
General Procedure E

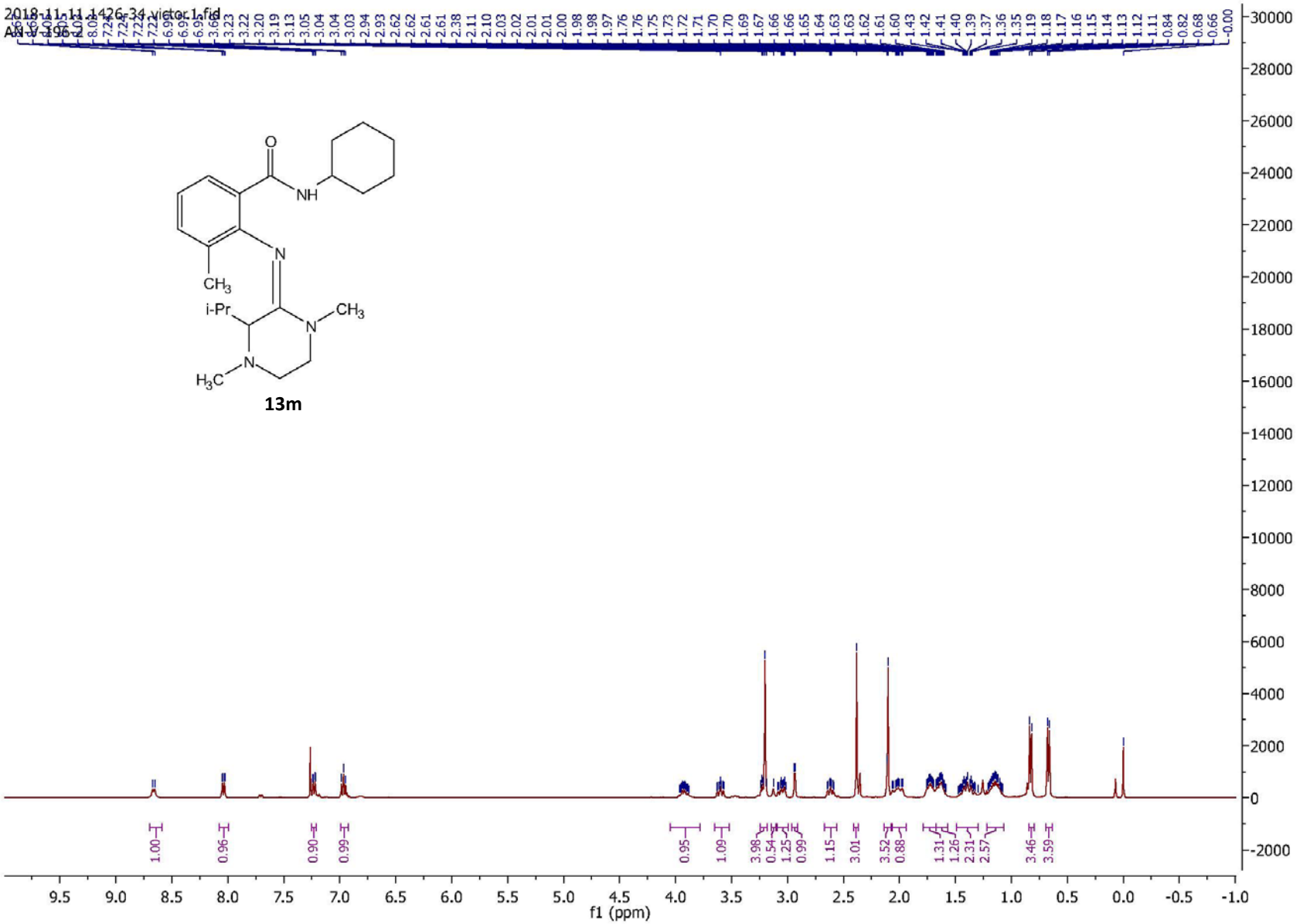

2017-09-24.1407-t.alok.2.fid
AN-V-087-Carbon
160.54
160.41
148.41
141.25
131.29
123.30
123.06
122.95
64.05
48.79
48.39
47.94
45.01
37.36
33.88
33.81
32.59
25.92
25.34
21.42
20.12
19.36
O
NH
H3C
N
i-Pr
CH3
N
N
H3C
13k
f1 (ppm)

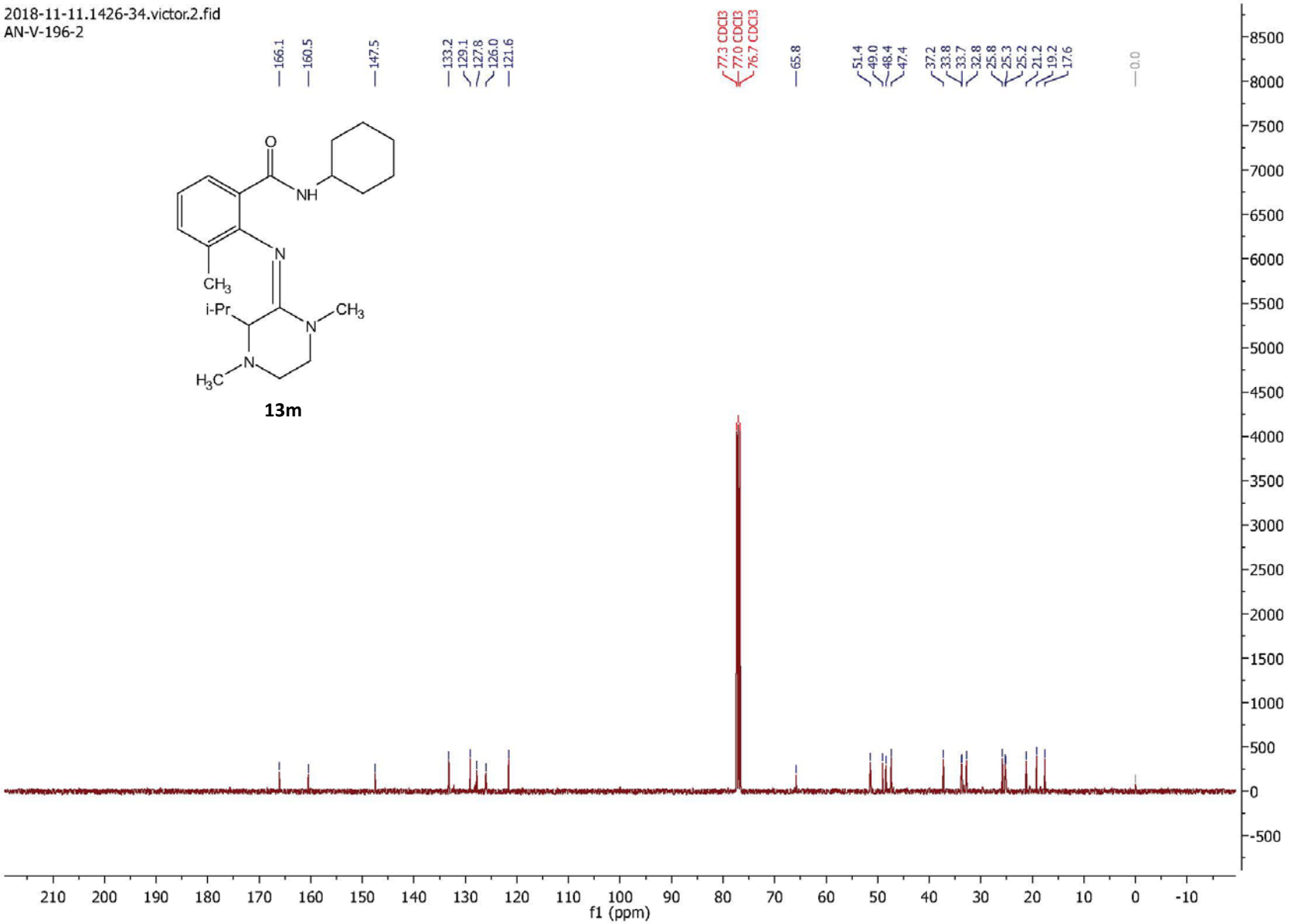

O
NH
F
N
i-Pr
N
CH3
N
H3C
13l
f1 (ppm)


2018-06-03.1628-18.victor.2.fid
2-290-1-1H-CDCl3
-109.97
O
NH
F
N
i-Pr
N
CH3
N
H3C
13l
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
10
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
-160
-170
-180
-190
-200
-210
f1 (ppm)

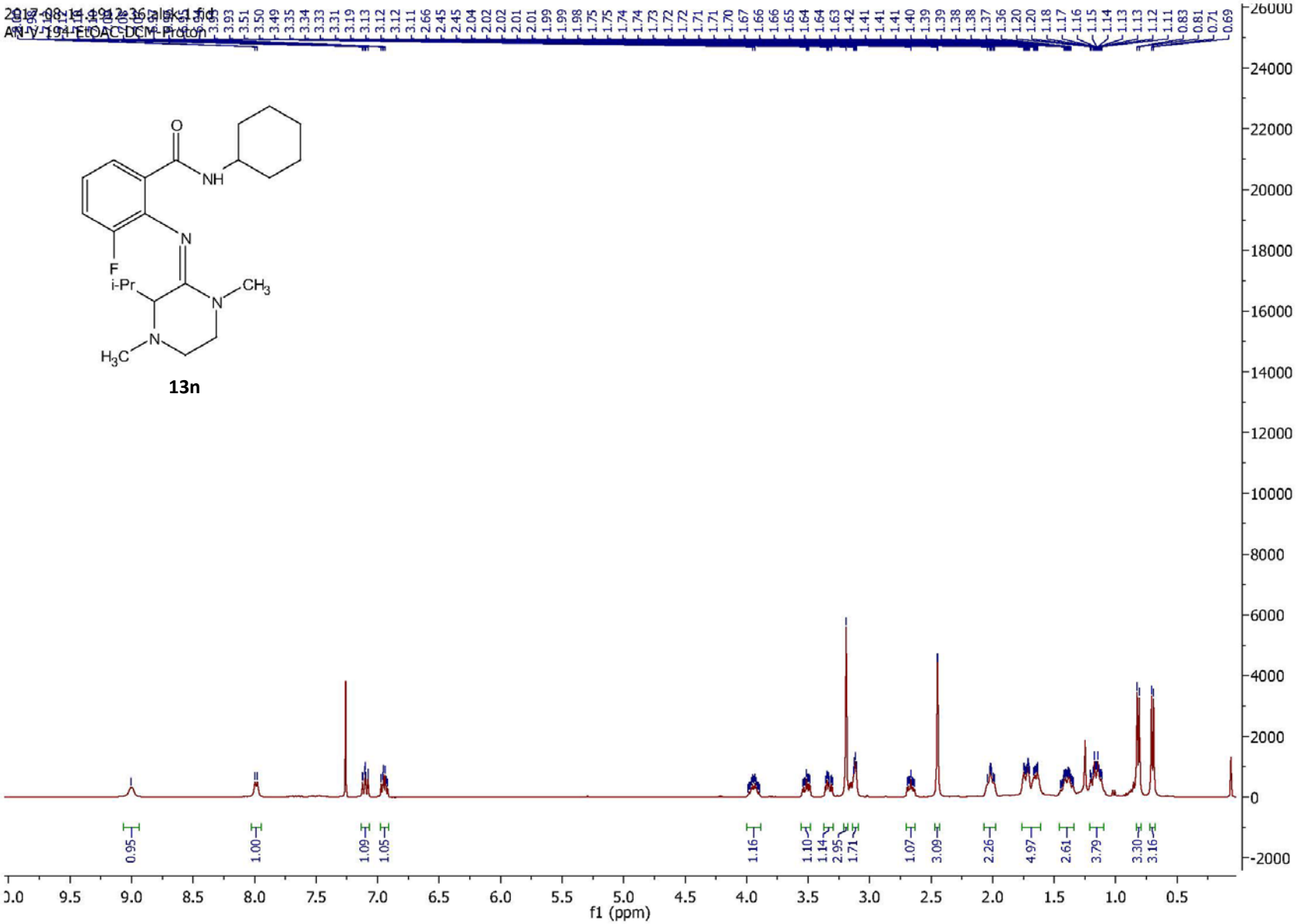

2018-06-03.1628-18.victor.3.fid
2-290-1-1H-CDCl3
165.2
165.0
162.7
160.8
150.5
150.4
133.5
133.4
122.2
122.2
108.9
108.7
108.6
108.4
77.3
77.2
77.0
76.7
63.8
48.6
48.4
47.7
45.1
37.3
33.8
33.7
32.5
29.7
25.8
25.2
20.1
19.1
0.0
O
NH
F
N
i-Pr
N
CH3
N
H3C
4500
4000
3500
3000
2500
2000
1500
1000
500
0
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

13l

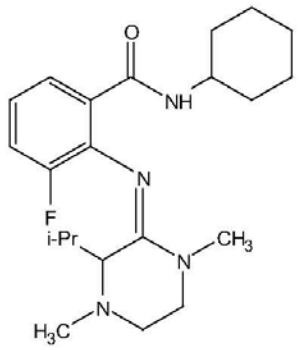

2018-11-11-1426-34 victor-1.fid
AN-V-196-2
O
NH
N
CH3
i-Pr
N
CH3
N
H3C
13m
f1 (ppm)


2018-11-11.1426-34.victor.2.fid
AN-V-196-2
166.1
160.5
147.5
133.2
129.1
127.8
126.0
121.6
77.3 CDCl3
77.0 CDCl3
76.7 CDCl3
65.8
51.4
49.0
48.4
47.4
37.2
33.8
33.7
32.8
25.8
25.3
25.2
21.2
19.2
17.6
0.0
O
NH
N
CH3
i-Pr
N
CH3
N
H3C
f1 (ppm)

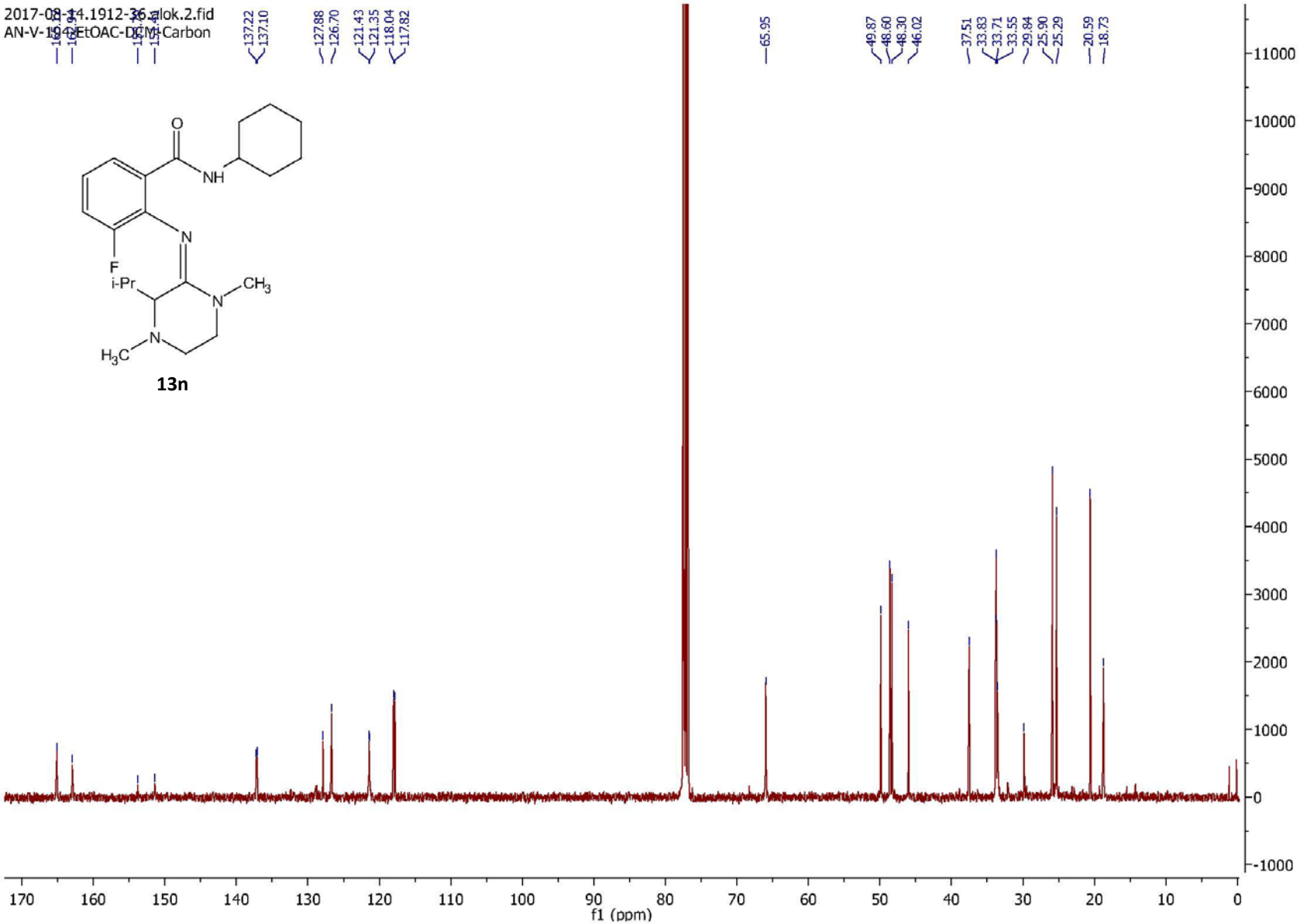
13m

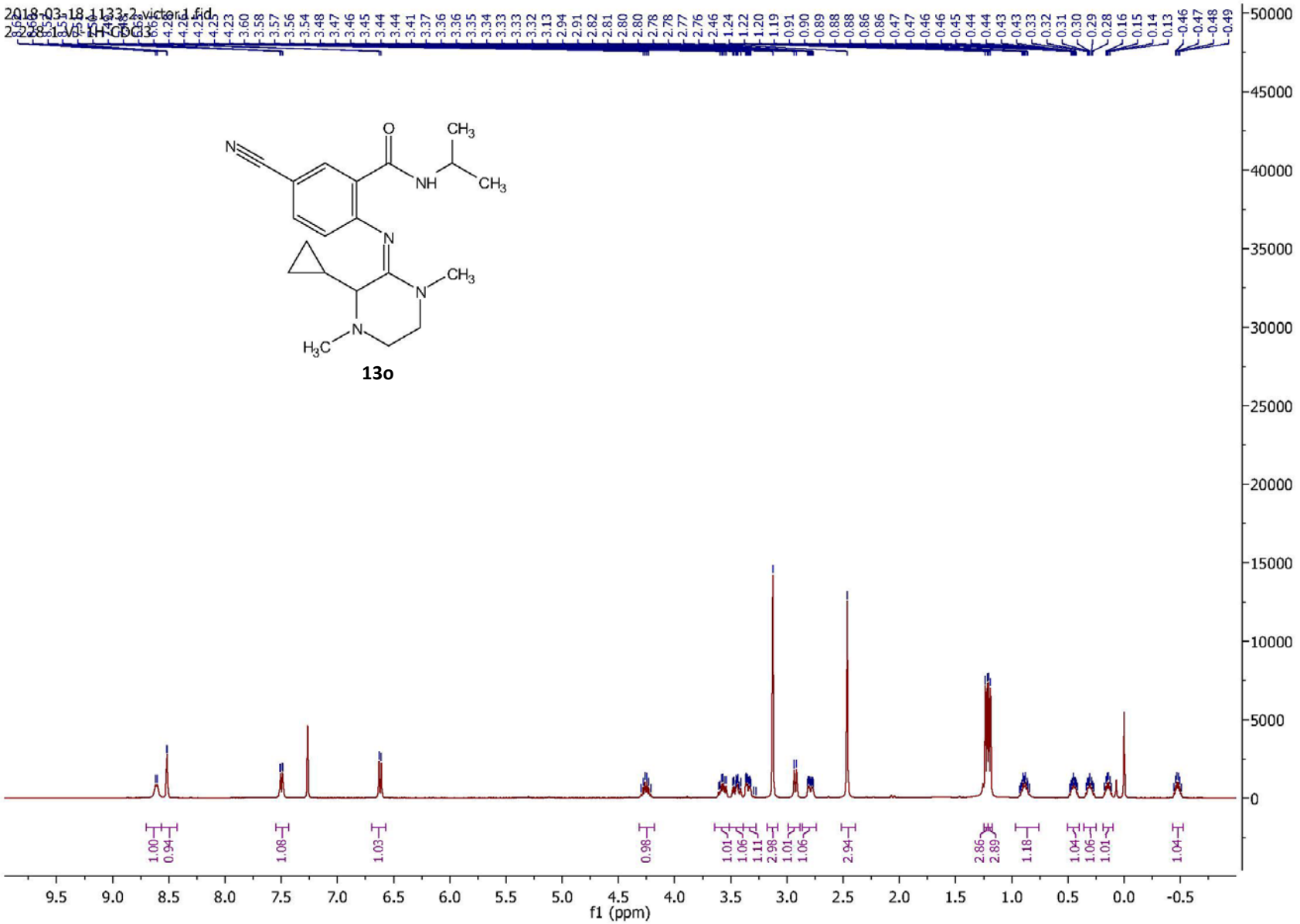

AN-V-194-EtOAC-DCM-Proton
O
NH
N
F
i-Pr
N
CH3
N
H3C
13n
f1 (ppm)

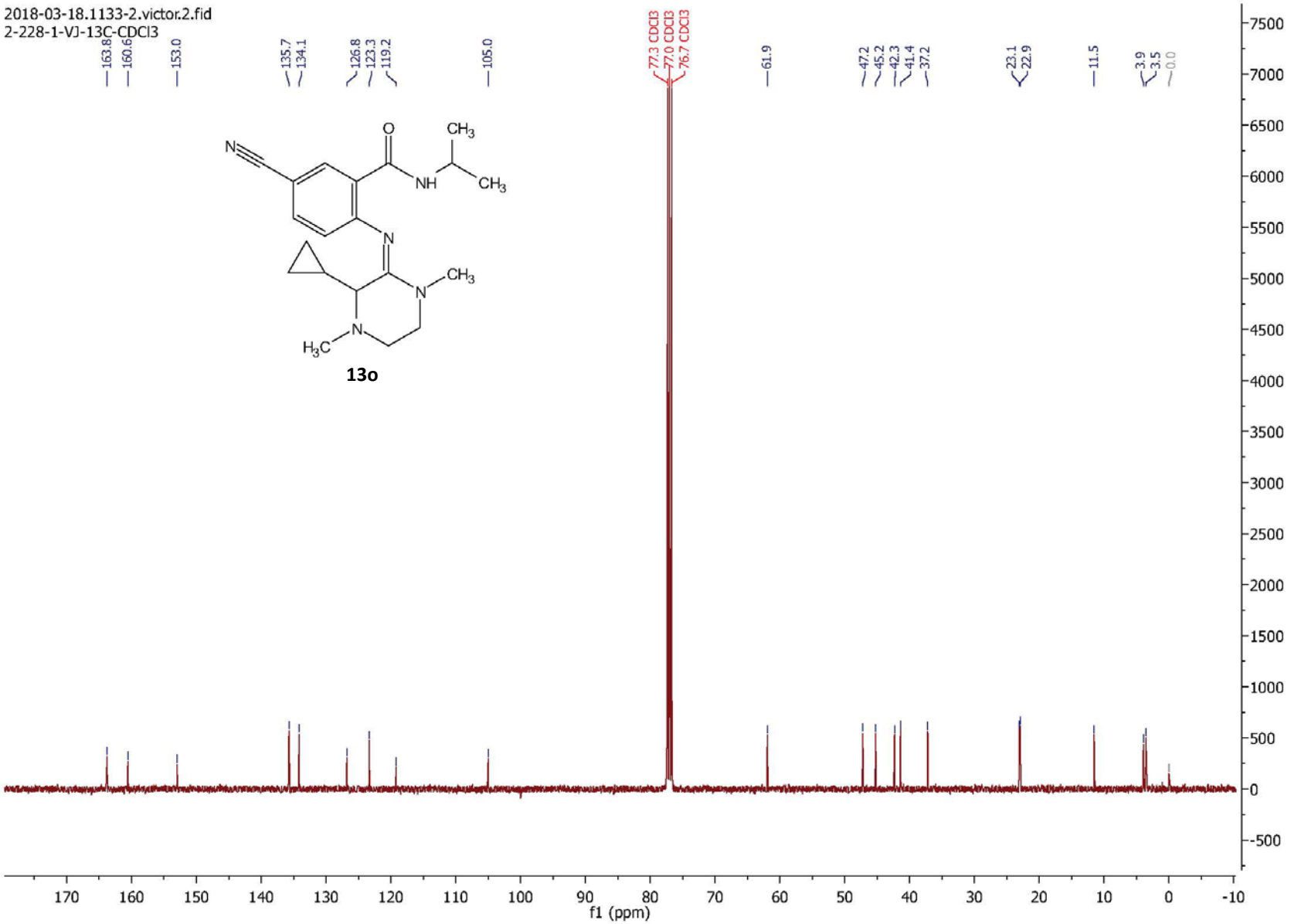
2018-10-19.1513-48.victor.2.fid
AN-V-194

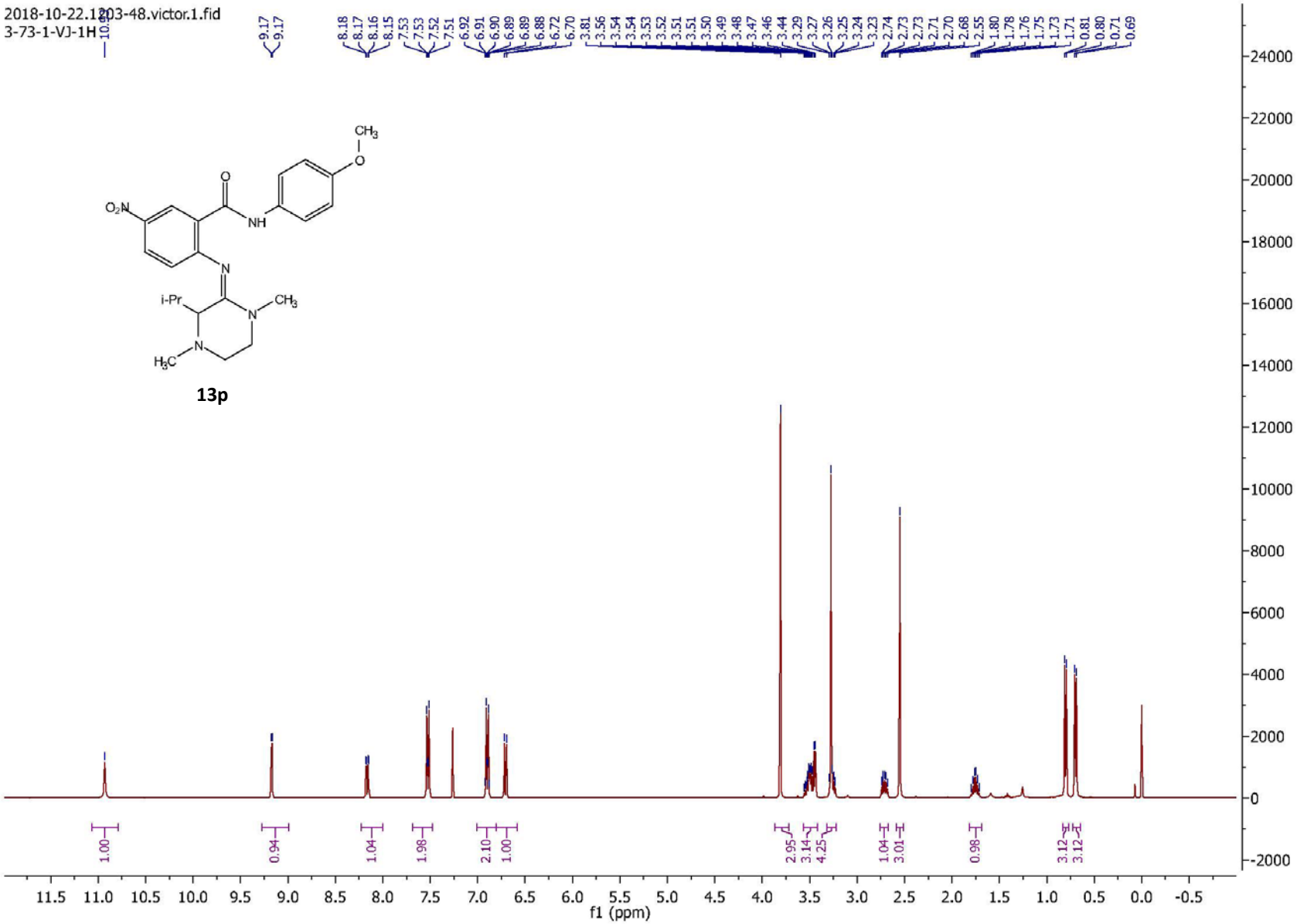


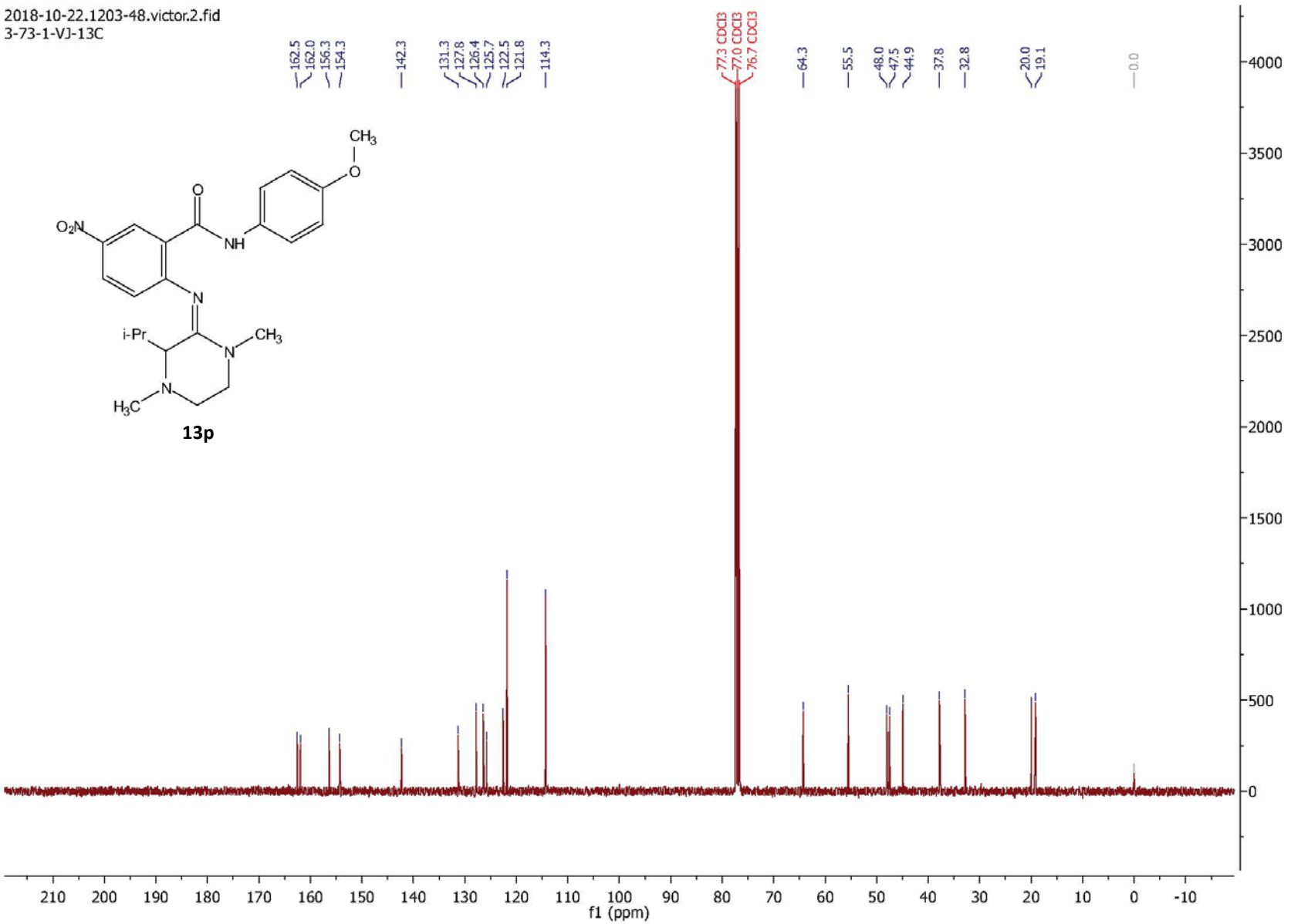
13n

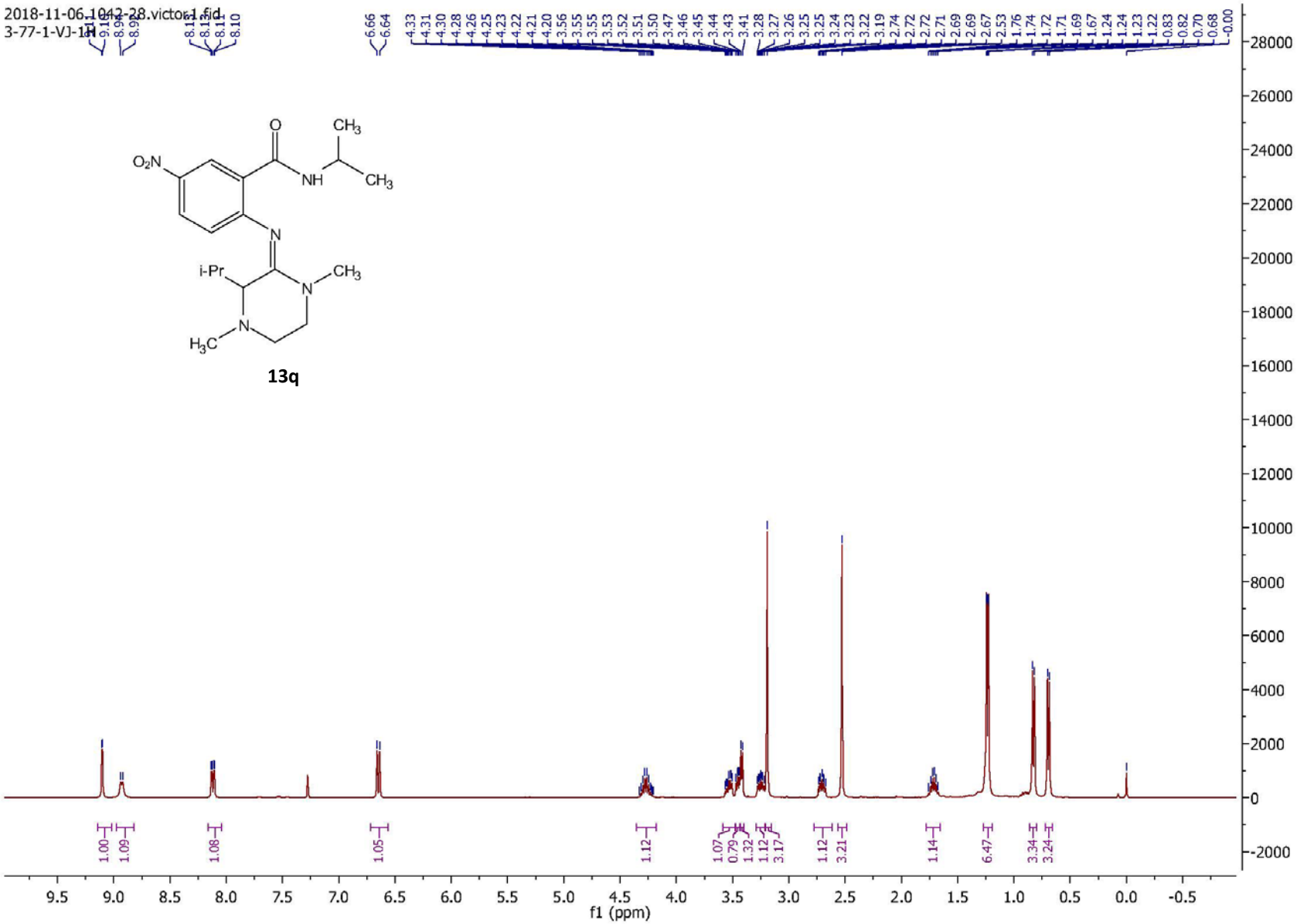

2017-08-14.1912-36.alok.2.fid
AN-V-194-EtOAC-DCM-Carbon
137.22
137.10
127.88
126.70
121.43
121.35
118.04
117.82
65.95
49.87
48.60
48.30
46.02
37.51
33.83
33.71
33.55
29.84
25.90
25.29
20.59
18.73
O
NH
N
F
i-Pr
N
CH3
N
H3C
13n
f1 (ppm)

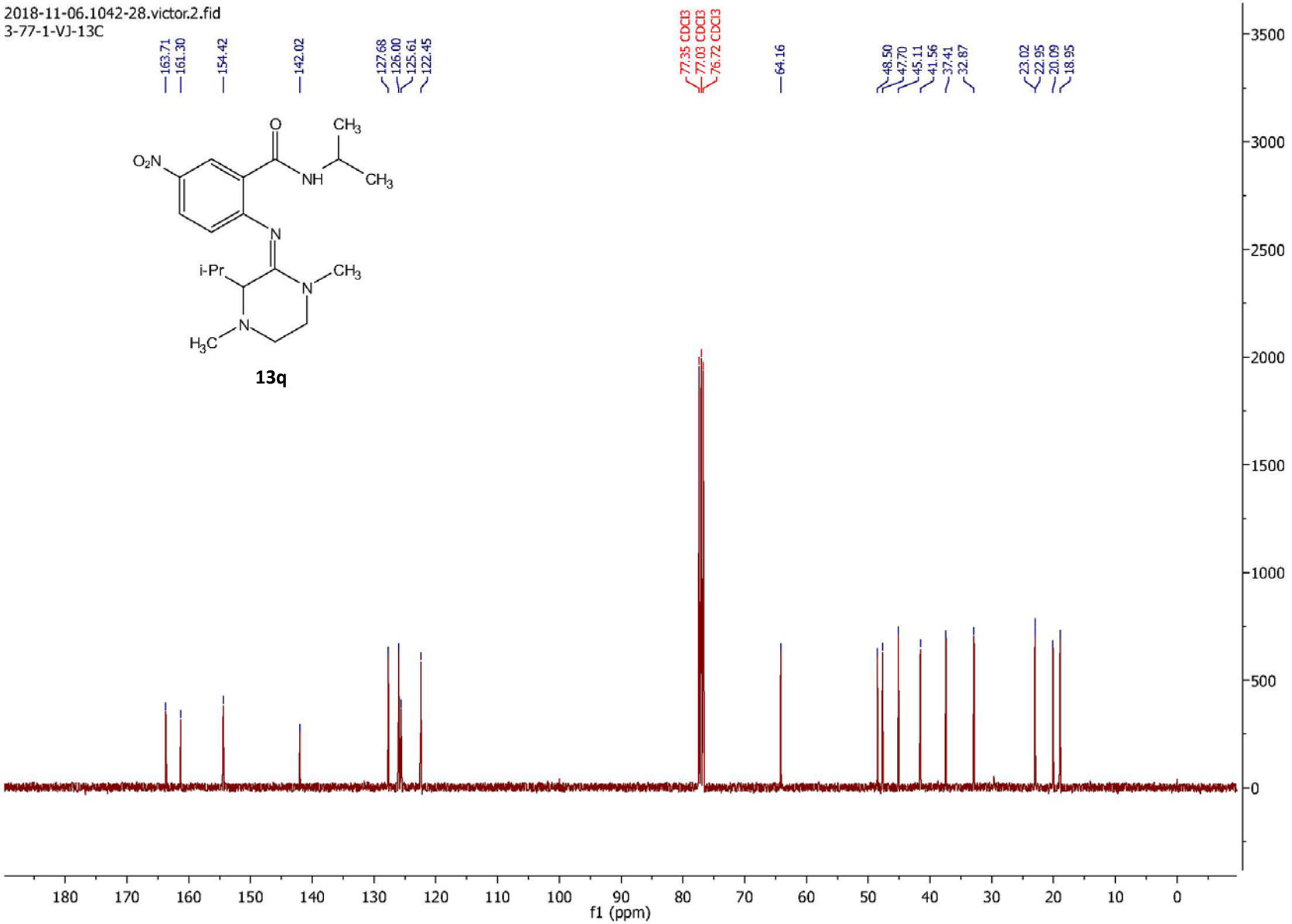

2018-03-18 1133-2 victor1.fid
2-228-1-V6-1H-CDCl3
O
CH3
N
NH
CH3
N
CH3
N
N
H3C
13o
50000
45000
40000
35000
30000
25000
20000
15000
10000
5000
0
1.00
0.94
1.08
1.03
0.98
1.01
1.06
1.11
2.98
1.01
1.06
2.94
2.86
2.89
1.18
1.04
1.06
1.01
1.04
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)

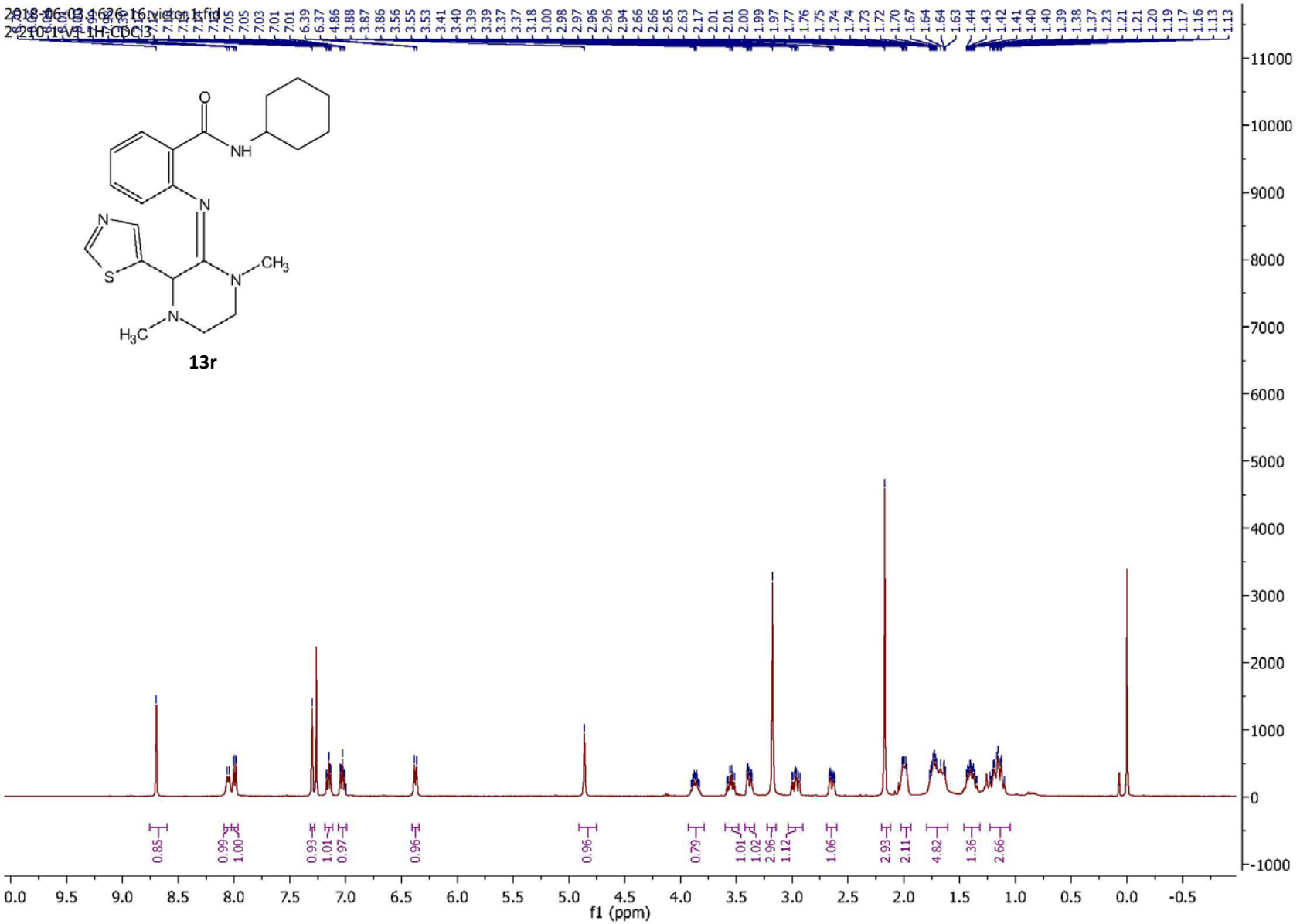

2018-03-18.1133-2.victor.2.fid
2-228-1-VJ-13C-CDCl3
163.8
160.6
153.0
135.7
134.1
126.8
123.3
119.2
105.0
77.3 CDCl3
77.0 CDCl3
76.7 CDCl3
61.9
47.2
45.2
42.3
41.4
37.2
23.1
22.9
11.5
3.9
3.5
0.0
13o
f1 (ppm)

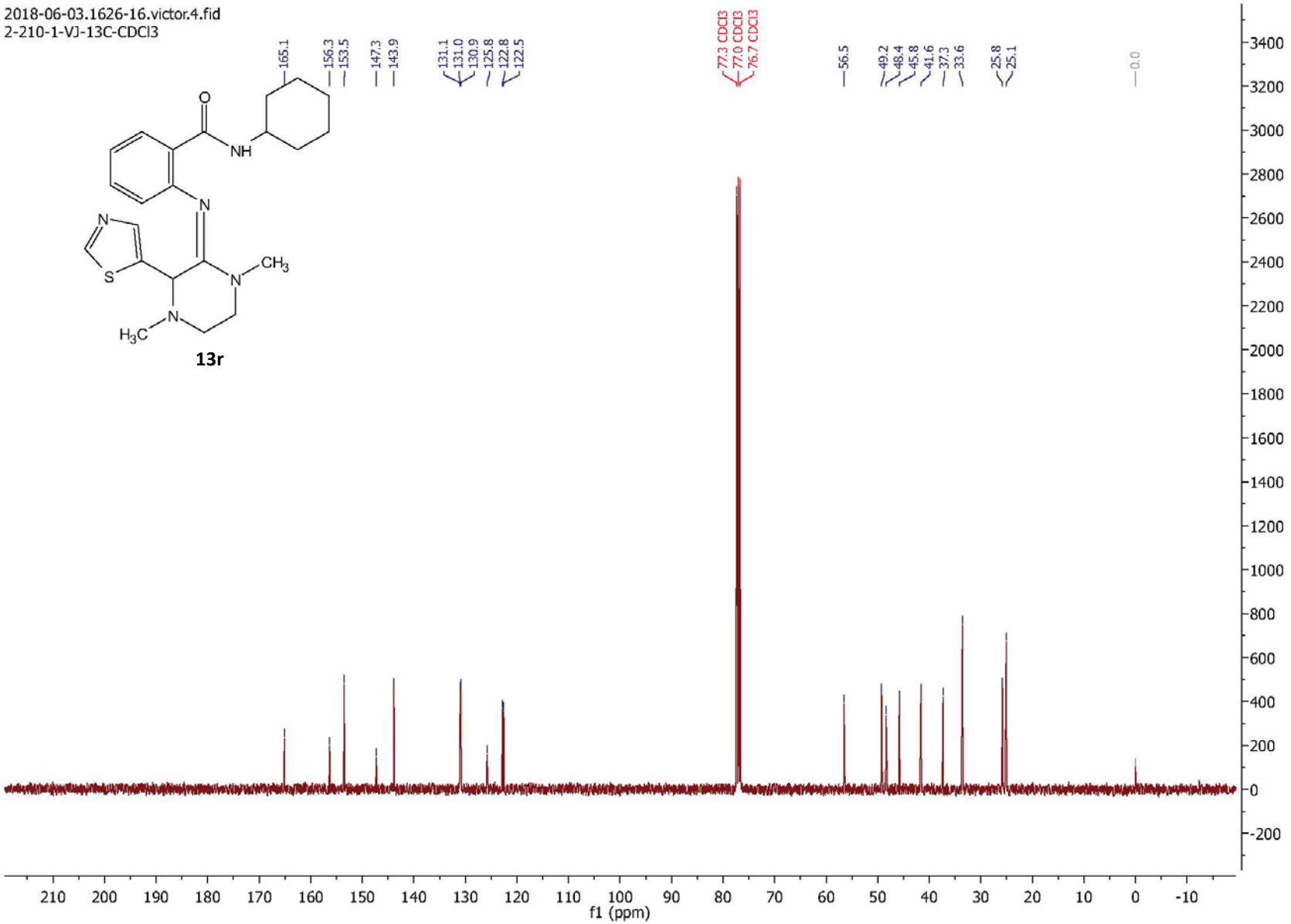

2018-10-22.1203-48.victor.1.fid
3-73-1-VJ-1H
13p
f1 (ppm)

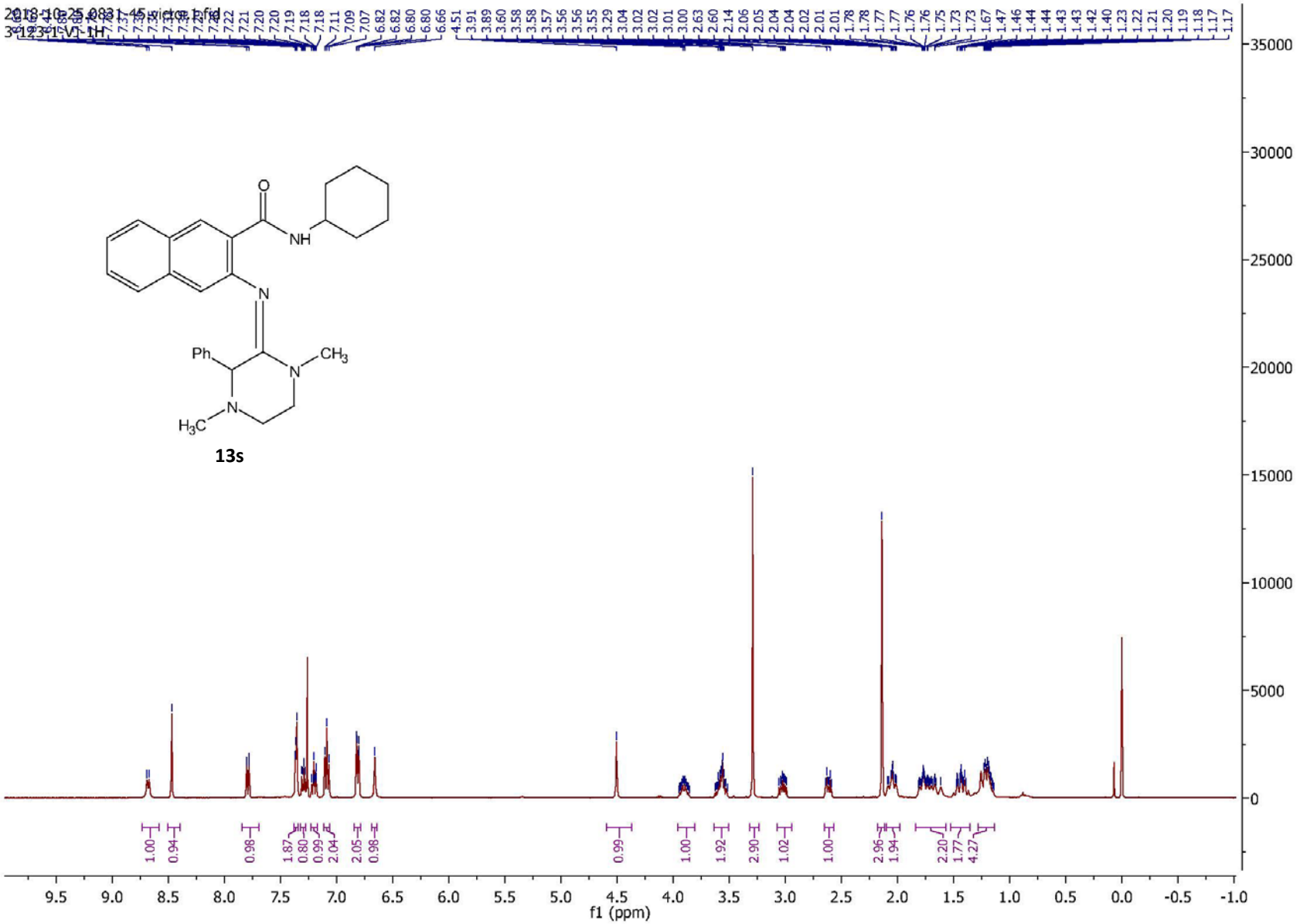

2018-10-22.1203-48.victor.2.fid
3-73-1-VJ-13C
162.5
162.0
156.3
154.3
142.3
131.3
127.8
126.4
125.7
122.5
121.8
114.3
77.3 CDCl3
77.0 CDCl3
76.7 CDCl3
64.3
55.5
48.0
47.5
44.9
37.8
32.8
20.0
19.1
0.0
O2N
NH
O
CH3
N
i-Pr
CH3
N
H3C
13p
f1 (ppm)

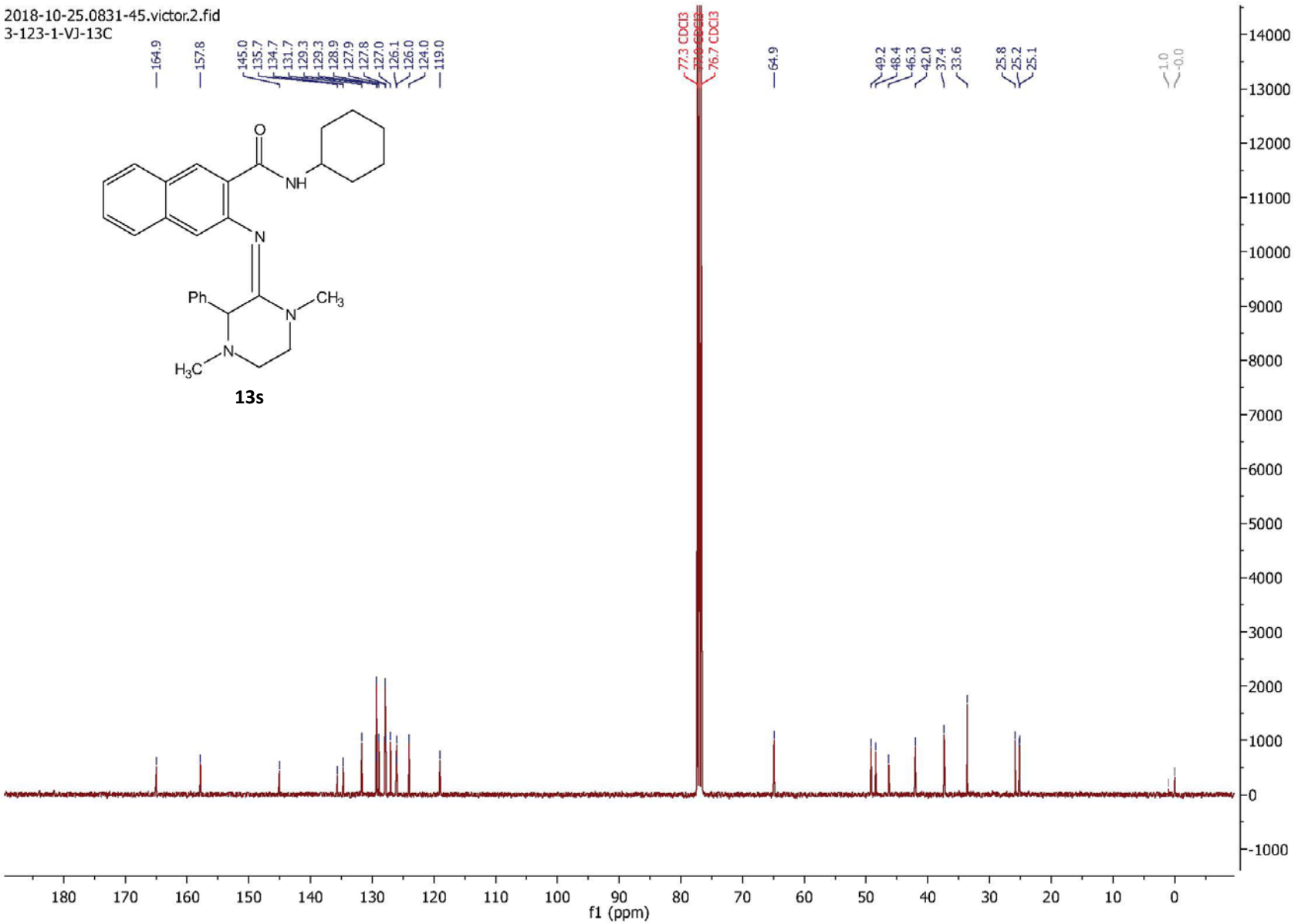

2018-11-06.1042-28.victor1.fid
3-77-1-VJ-1H
O
CH3
O2N
NH
CH3
N
i-Pr
CH3
N
N
H3C
13q
f1 (ppm)

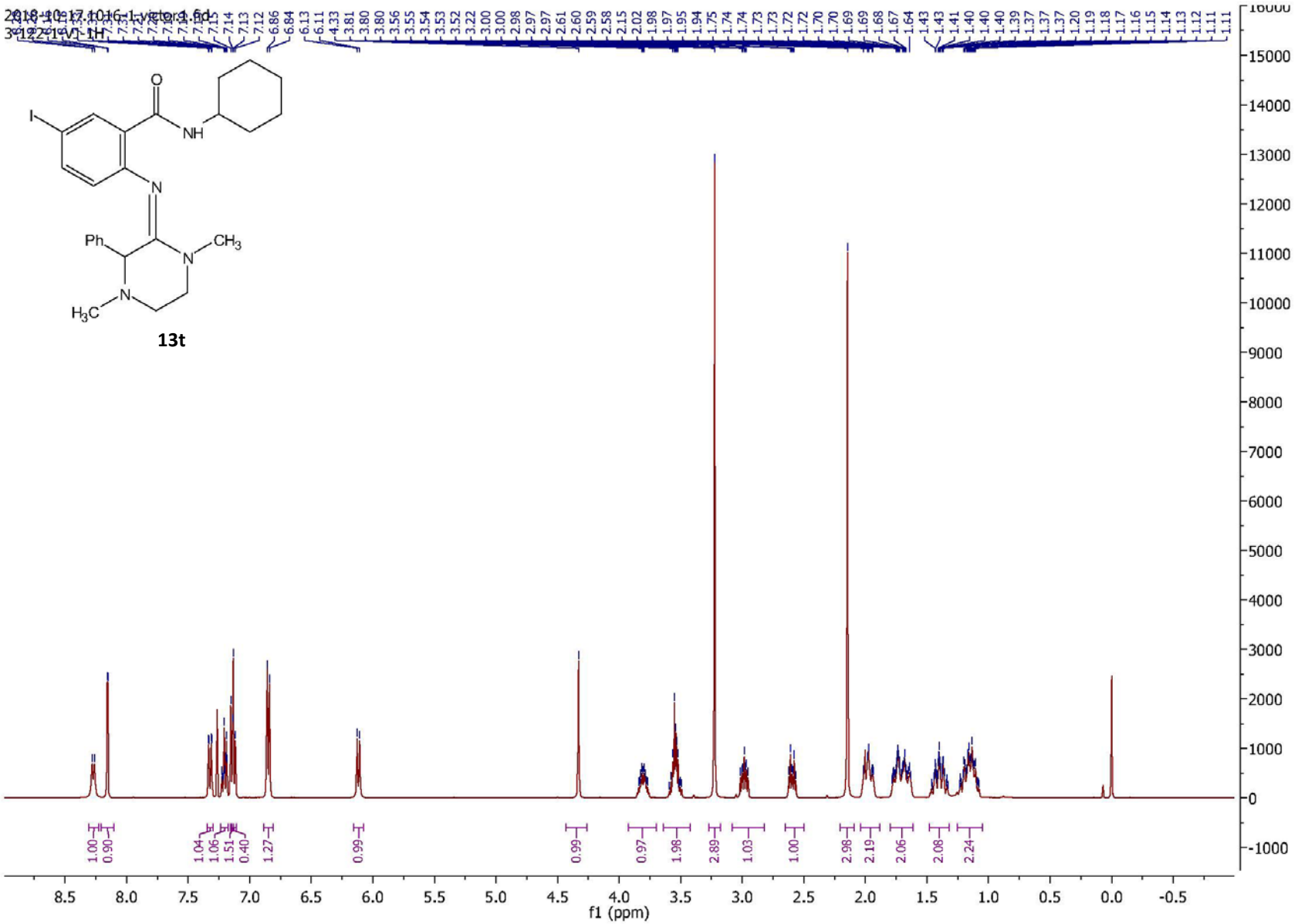

2018-11-06.1042-28.victor.2.fid
3-77-1-VJ-13C
163.71
161.30
154.42
142.02
127.68
126.00
125.61
122.45
77.35 CDCl3
77.03 CDCl3
76.72 CDCl3
64.16
48.50
47.70
45.11
41.56
37.41
32.87
23.02
22.95
20.09
18.95
O
CH3
O2N
NH
CH3
N
i-Pr
CH3
N
N
H3C
13q
3500
3000
2500
2000
1500
1000
500
0
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


O
NH
N
N
S
N
CH3
N
H3C
13r
f1 (ppm)

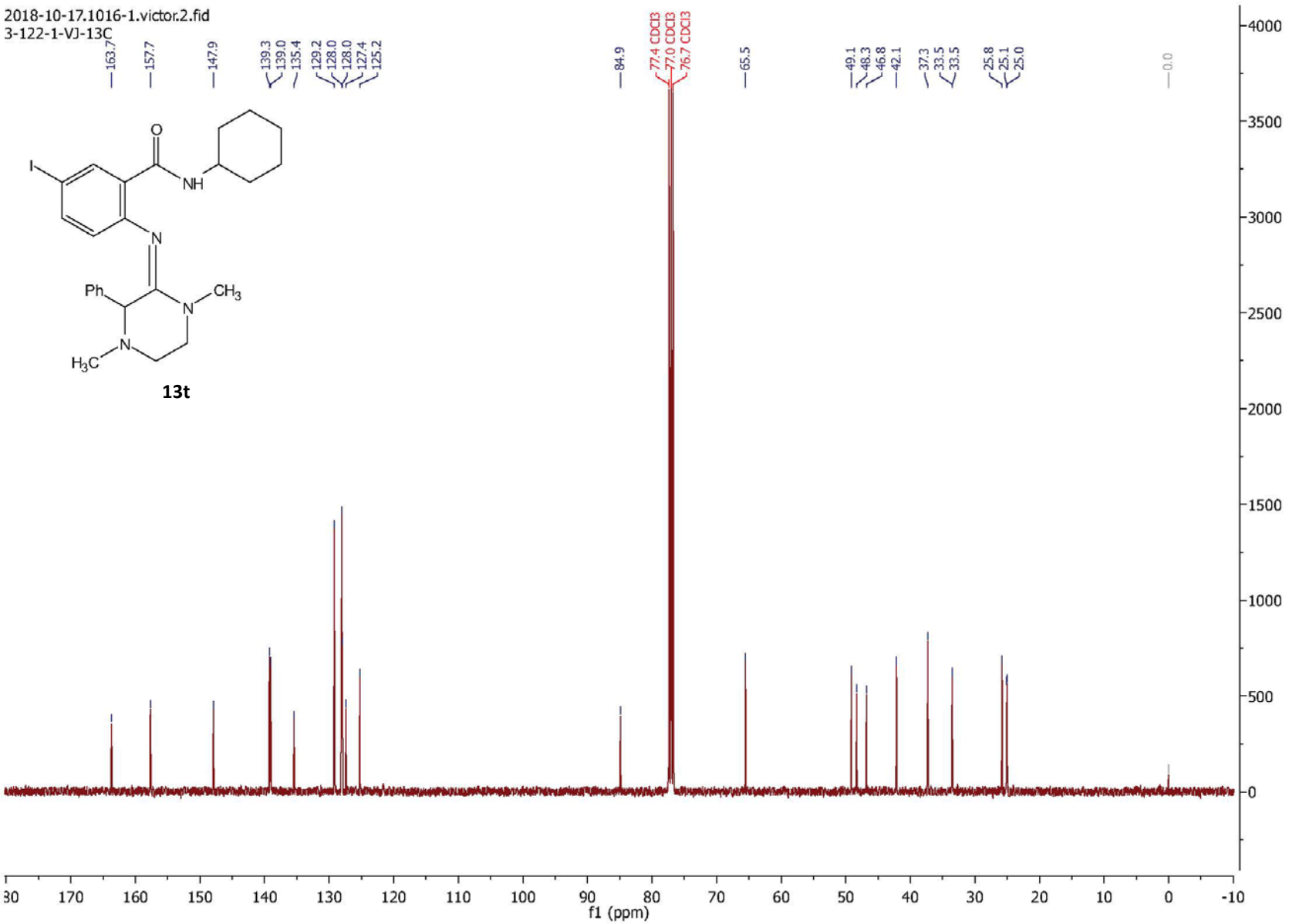

2018-06-03.1626-16.victor.4.fid
2-210-1-VJ-13C-CDCl3
165.1
156.3
153.5
147.3
143.9
131.1
131.0
130.9
125.8
122.8
122.5
77.3 CDCl3
77.0 CDCl3
76.7 CDCl3
56.5
49.2
48.4
45.8
41.6
37.3
33.6
25.8
25.1
0.0
O
NH
N
N
S
N
CH3
N
H3C
13r
f1 (ppm)

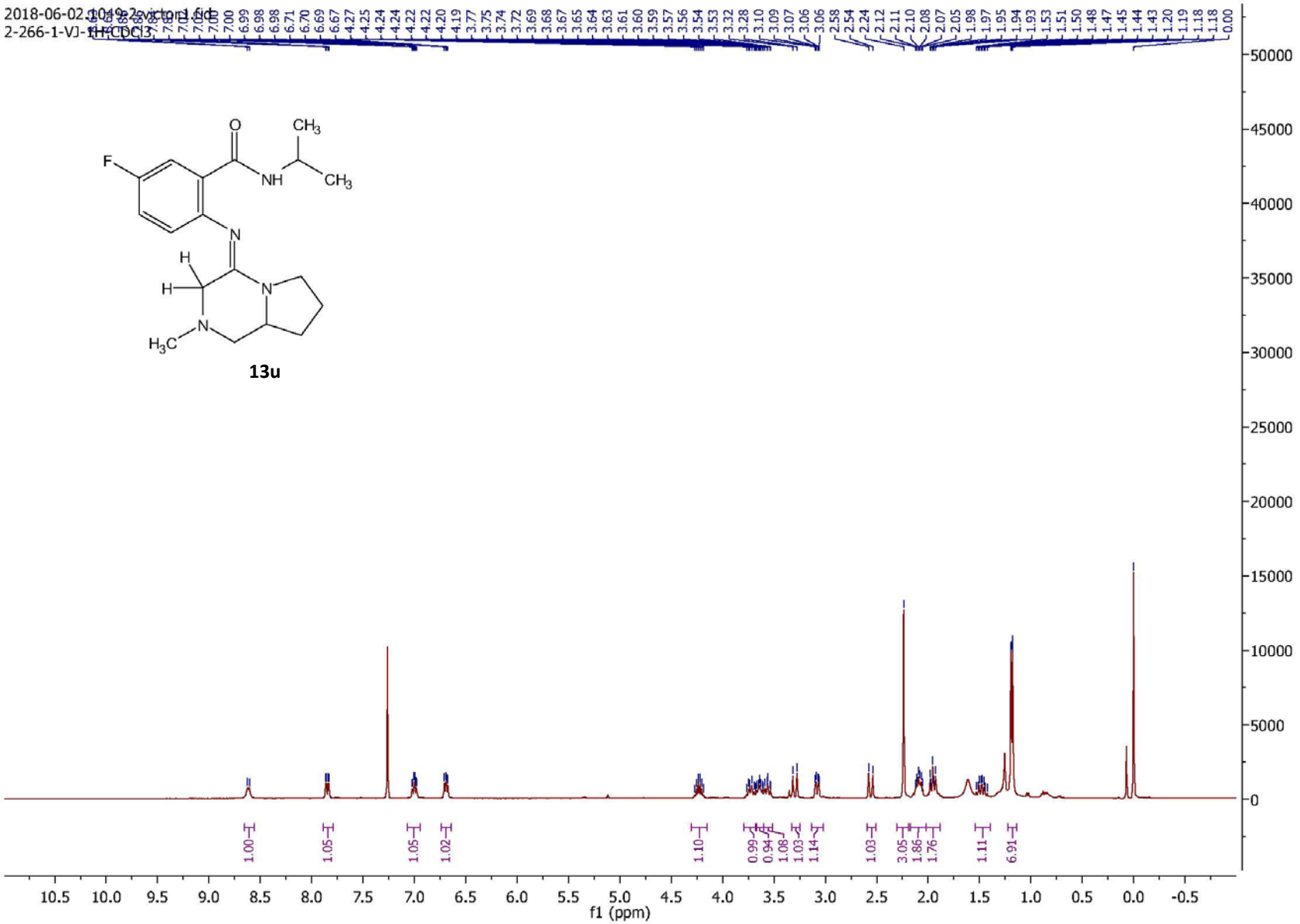

O
NH
N
Ph
CH3
N
N
H3C
13s
f1 (ppm)


2018-10-25.0831-45.victor.2.fid
3-123-1-VJ-13C
164.9
157.8
145.0
135.7
134.7
131.7
129.3
129.3
128.9
127.9
127.8
127.0
126.1
126.0
124.0
119.0
77.3 CDCl3
76.7 CDCl3
64.9
49.2
48.4
46.3
42.0
37.4
33.6
25.8
25.2
25.1
1.0
0.0
O
NH
N
Ph
CH3
N
N
H3C
13s
f1 (ppm)

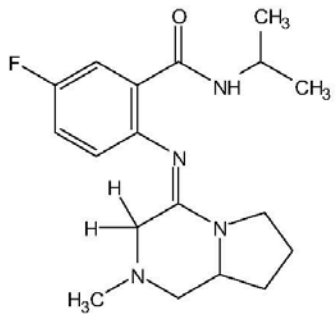


13t

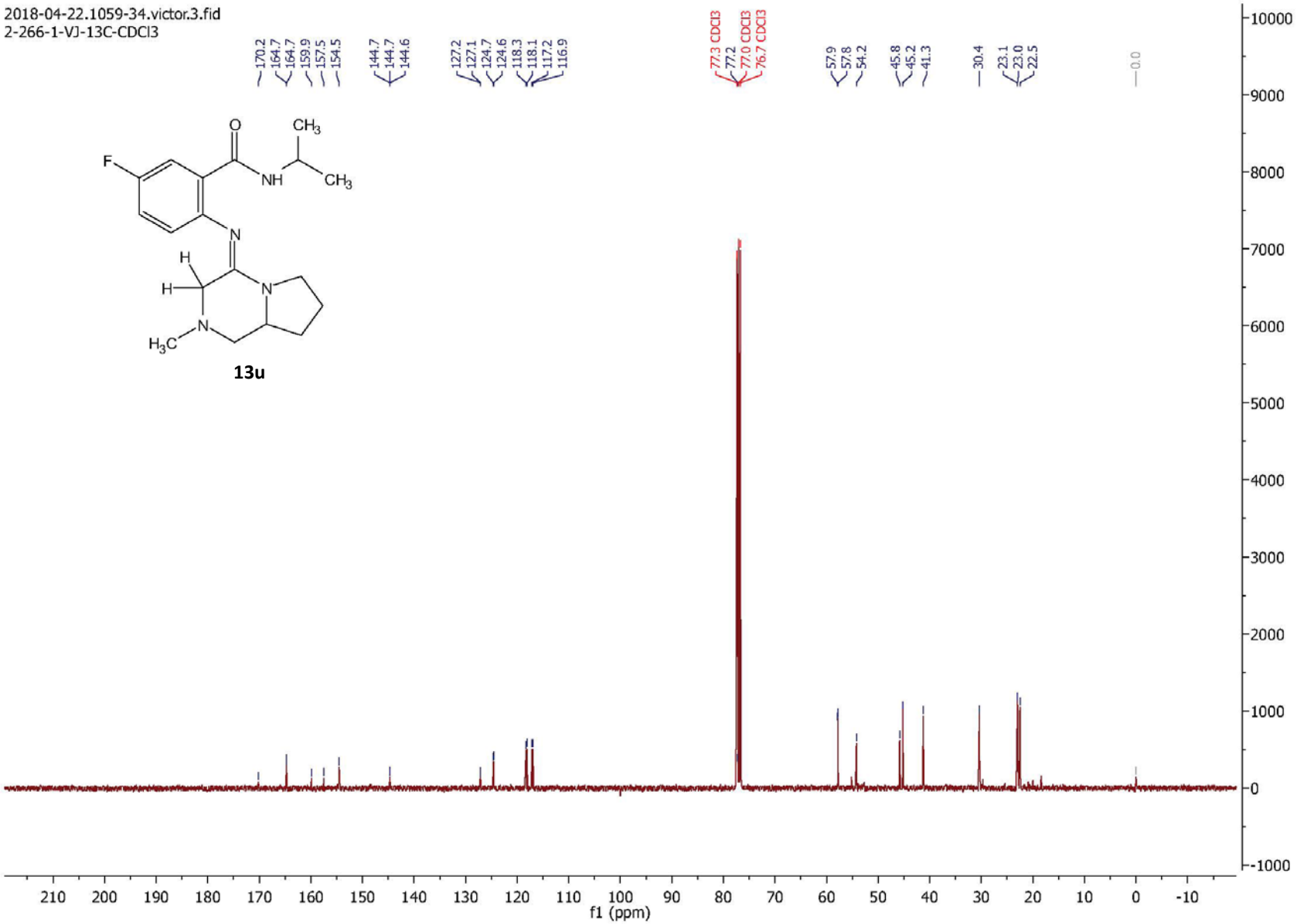

2018-10-17.1016-1.victor.2.fid
3-122-1-VJ-13C
163.7
157.7
147.9
139.3
139.0
135.4
129.2
128.0
128.0
127.4
125.2
84.9
77.4 CDCl3
77.0 CDCl3
76.7 CDCl3
65.5
49.1
48.3
46.8
42.1
37.3
33.5
33.5
25.8
25.1
25.0
0.0
O
NH
I
N
Ph
N
CH3
N
H3C
13t
f1 (ppm)

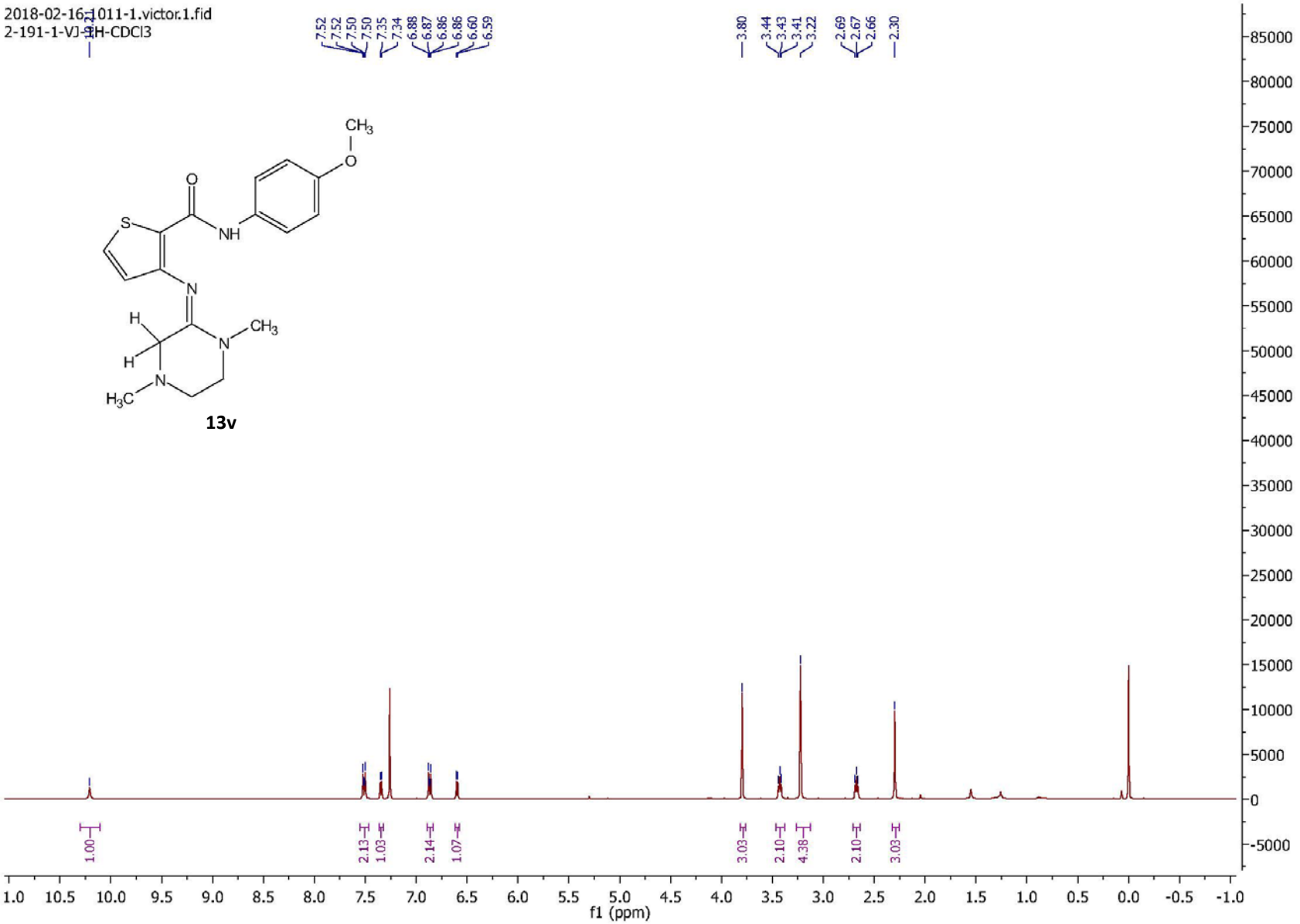

2018-06-02 1049 2 victor 1.fid
2-266-1-VJ-1H-CDCl3
O
CH3
F
NH
CH3
N
H
H
N
N
H3C
13u
50000
45000
40000
35000
30000
25000
20000
15000
10000
5000
0
1.00
1.05
1.05
1.02
1.10
0.99
0.94
1.08
1.03
1.14
1.03
3.05
1.86
1.76
1.11
6.91
10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)

2018-04-22.1059-34.victor.2.fid
2-266-1-VJ-19F-CDCl3

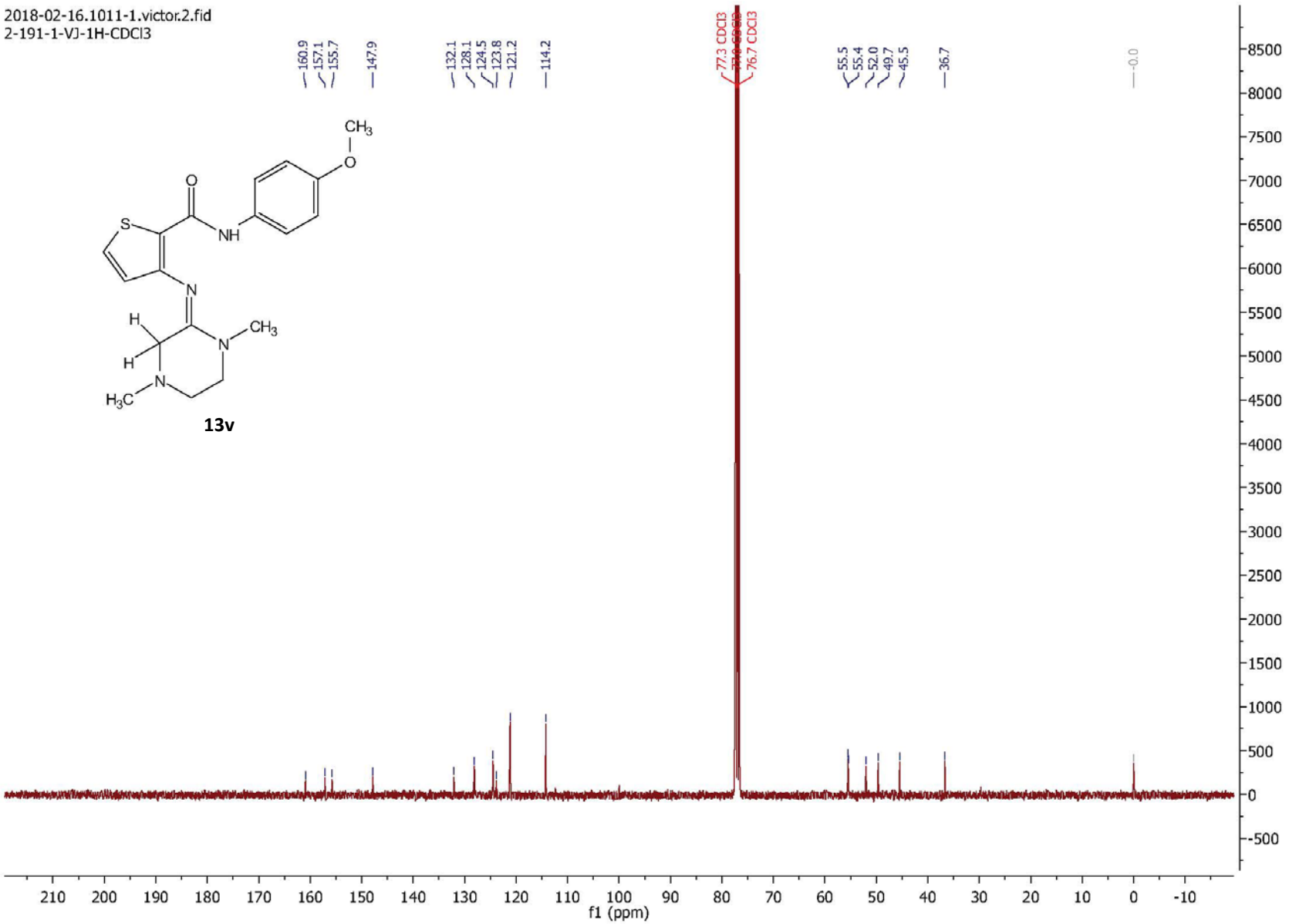


13u

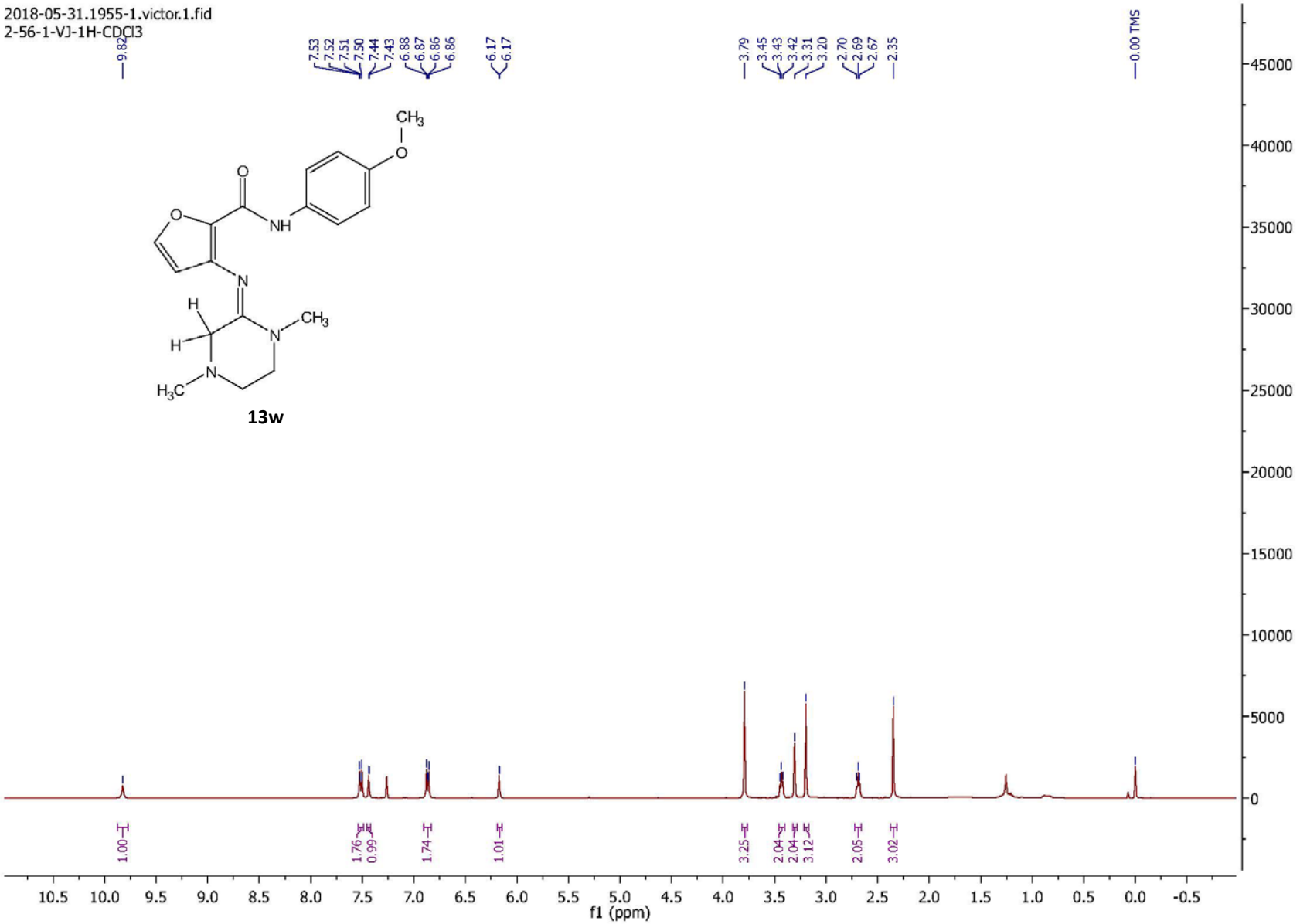

2018-04-22.1059-34.victor.3.fid
2-266-1-VJ-13C-CDCl3
170.2
164.7
164.7
159.9
157.5
154.5
144.7
144.7
144.6
127.2
127.1
124.7
124.6
118.3
118.1
117.2
116.9
77.3 CDCl3
77.2
77.0 CDCl3
76.7 CDCl3
57.9
57.8
54.2
45.8
45.2
41.3
30.4
23.1
23.0
22.5
0.0
F
O
NH
CH3
CH3
N
H
H
N
N
H3C
13u
f1 (ppm)


2018-02-16-1011-1.victor.1.fid
2-191-1-VJ-1H-CDCl3
7.52
7.52
7.50
7.50
7.35
7.34
6.88
6.87
6.86
6.86
6.60
6.59
3.80
3.44
3.43
3.41
3.22
2.69
2.67
2.66
2.30
O
CH3
O
S
NH
N
H
H
N
CH3
N
H3C
1.00
2.13
1.03
2.14
1.07
3.03
2.10
4.38
2.10
3.03
f1 (ppm)

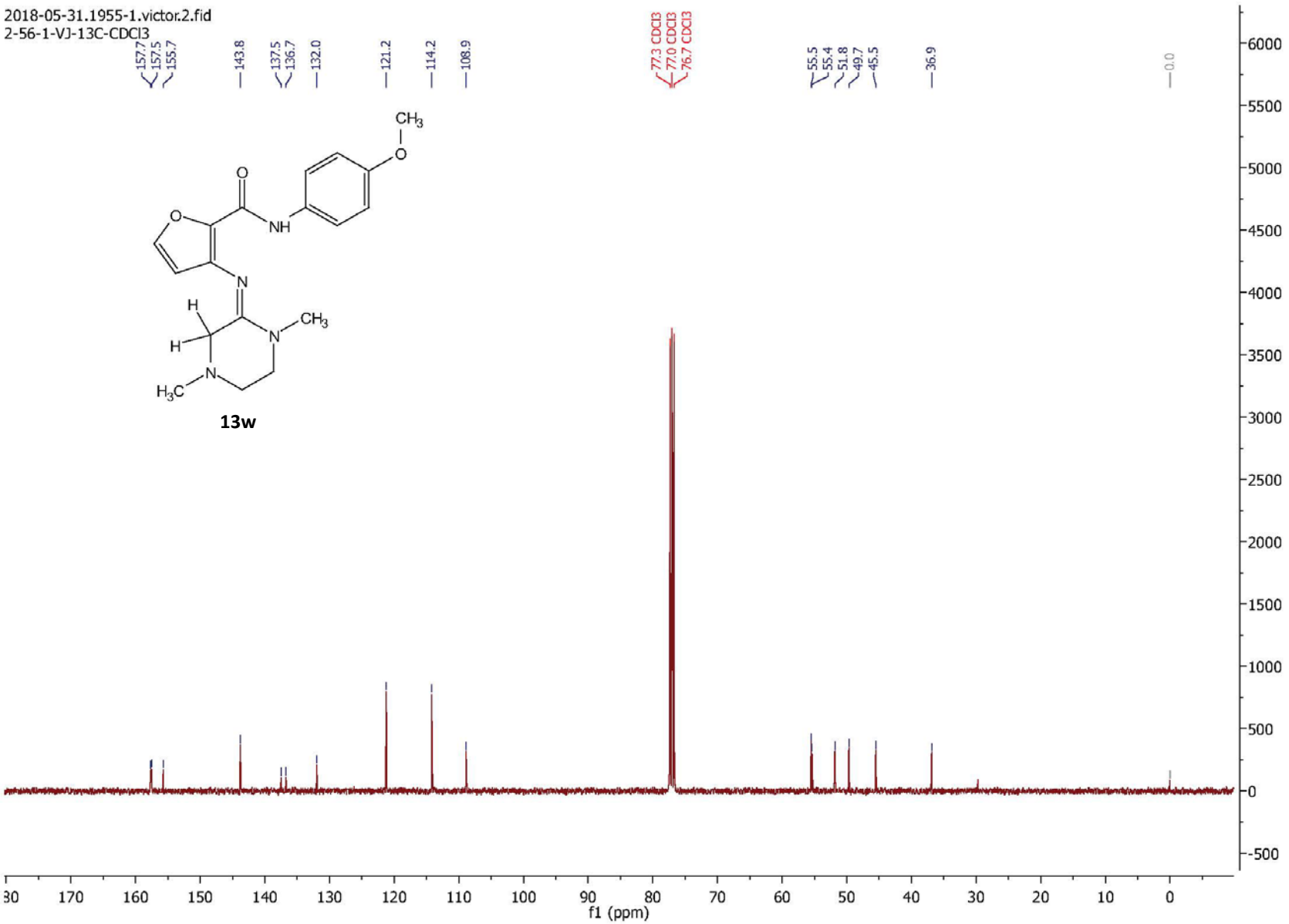
13v


2018-02-16.1011-1.victor.2.fid
2-191-1-VJ-1H-CDCl3
160.9
157.1
155.7
147.9
132.1
128.1
124.5
123.8
121.2
114.2
77.3 CDCl3
76.7 CDCl3
55.5
55.4
52.0
49.7
45.5
36.7
0.0
f1 (ppm)

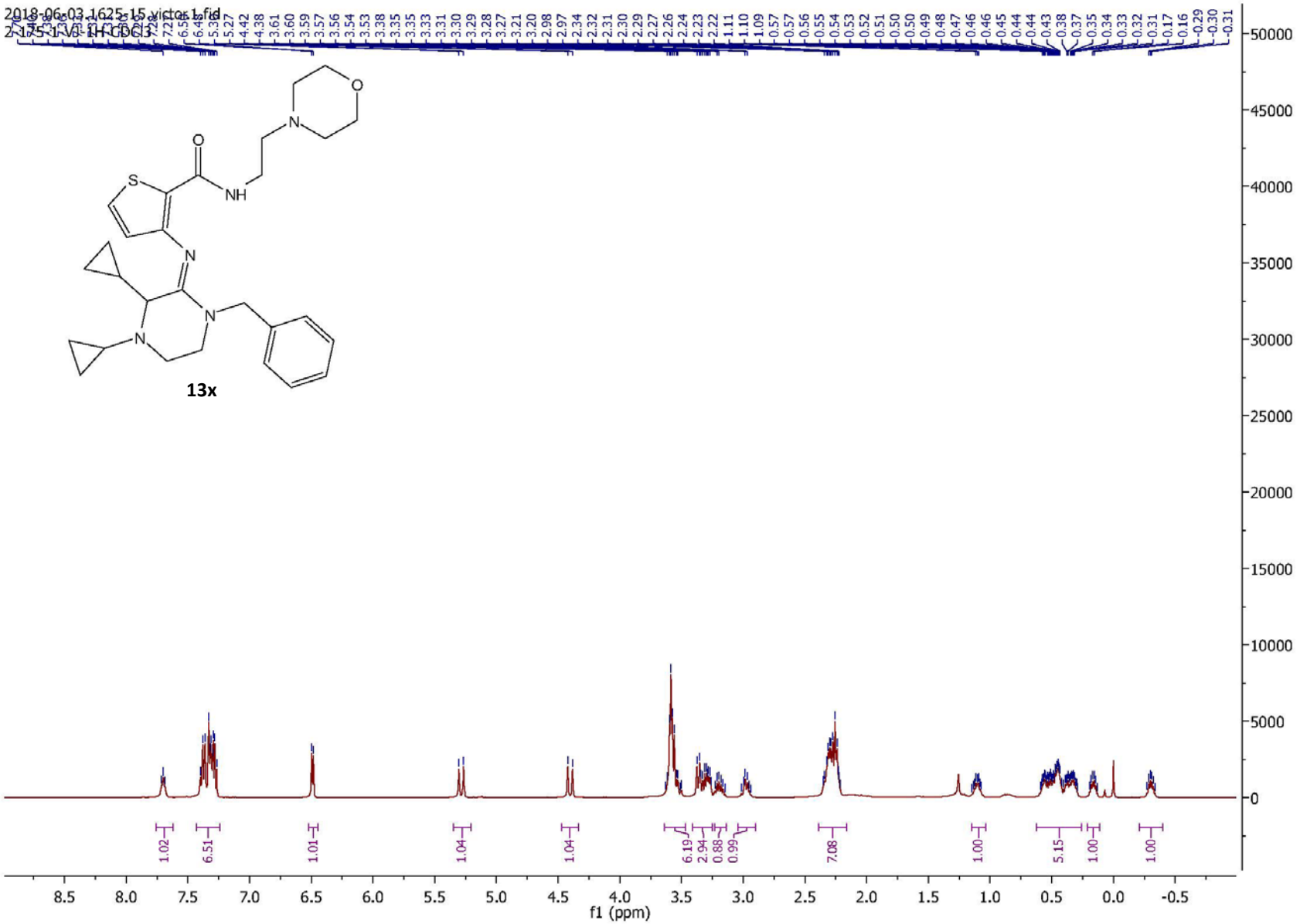
13v

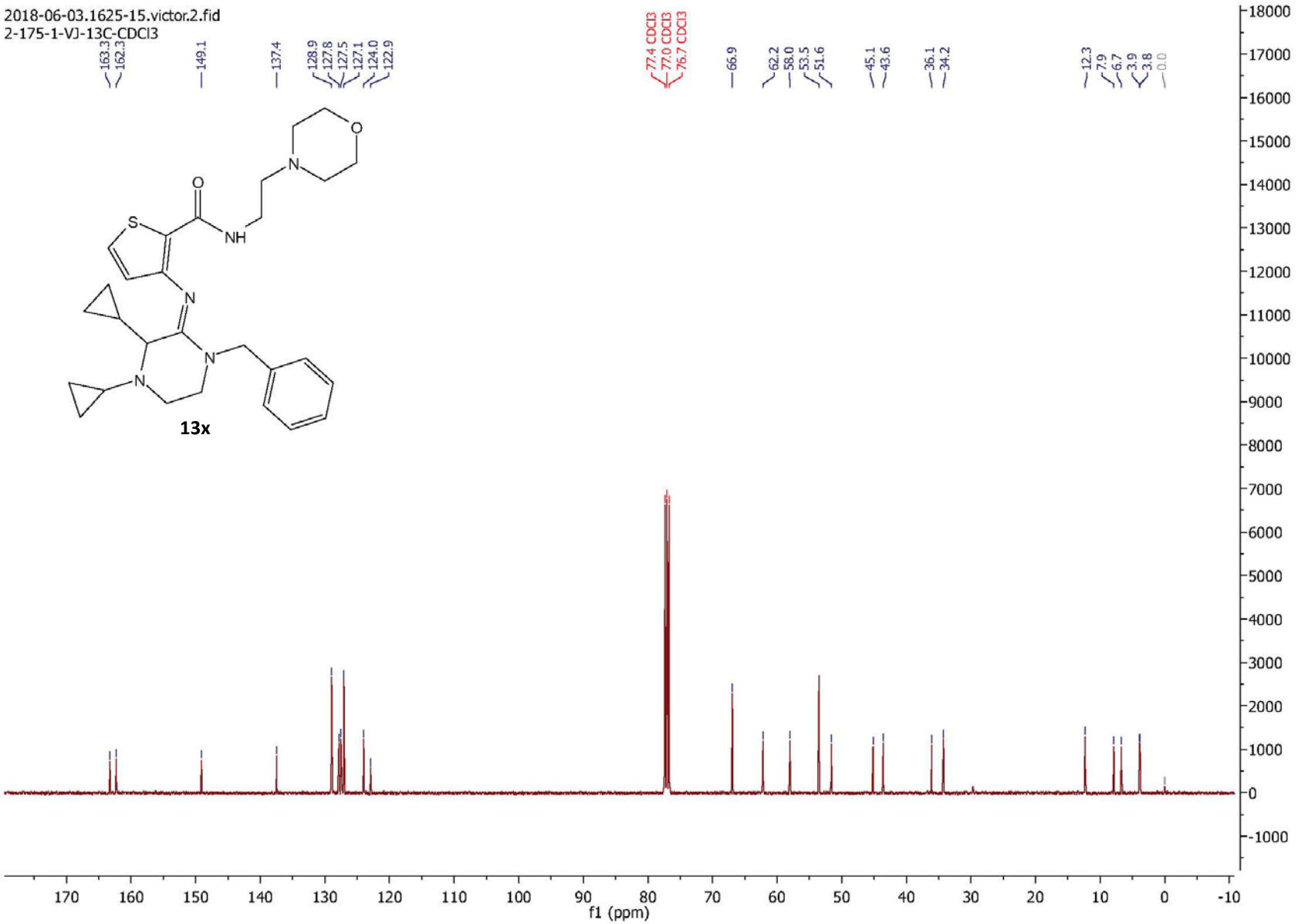

2018-05-31.1955-1.victor.1.fid
2-56-1-VJ-1H-CDCl3
9.82
7.53
7.52
7.51
7.50
7.44
7.43
6.88
6.87
6.86
6.86
6.17
6.17
3.79
3.45
3.43
3.42
3.31
3.20
2.70
2.69
2.67
2.35
0.00 TMS
CH3
O
O
NH
O
N
H
H
N
CH3
N
H3C
1.00
1.76
0.99
1.74
1.01
3.25
2.04
2.04
3.12
2.05
3.02
f1 (ppm)

13w


2018-05-31.1955-1.victor.2.fid
2-56-1-VJ-13C-CDCl3
157.7
157.5
155.7
143.8
137.5
136.7
132.0
121.2
114.2
108.9
77.3 CDCl3
77.0 CDCl3
76.7 CDCl3
55.5
55.4
51.8
49.7
45.5
36.9
0.0
CH3
O
O
NH
O
N
H
H
N
CH3
N
H3C
f1 (ppm)

13w


2018-06-03 1625-15 victor 1.fid
2-175-1-V6-1H-CDCl3
O
N
O
S
NH
N
N
N
13x
f1 (ppm)


2018-06-03.1625-15.victor.2.fid
2-175-1-VJ-13C-CDCl3
163.3
162.3
149.1
137.4
128.9
127.8
127.5
127.1
124.0
122.9
77.4 CDCl3
77.0 CDCl3
76.7 CDCl3
66.9
62.2
58.0
53.5
51.6
45.1
43.6
36.1
34.2
12.3
7.9
6.7
3.9
3.8
0.0
O
N
S
NH
N
N
N
O
13x
f1 (ppm)