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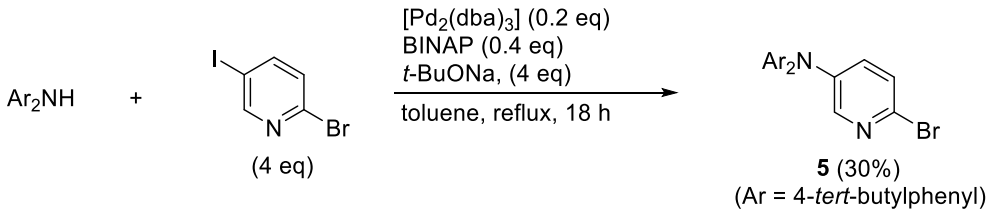
General remarks

All the manipulations were carried out under dry argon atmosphere using the standard Schlenk technique. Flash column chromatography (SiO_2) was carried out using a Biotage Isolera One system. Preparative gel-permeation chromatography (GPC) was carried out by a JAI LC-9204 recycling preparative HPLC equipped with a JAI JAIGEL-2HR Plus GPC column (solvent: $CHCl_3$). ^1H, ^{19}F, ^{13}C NMR spectra were recorded on a JEOL JNM-ECZ600R (^1H: 600 MHz, ^{19}F: 564 MHz, ^{13}C: 150 MHz) spectrometer equipped with a JEOL ROYAL probe and referenced to appropriate internal or external standards (^1H and ^{13}C: $SiMe_4$ (0 ppm), ^{19}F: C_6F_6 in $CDCl_3$ (–161.64 ppm)[1]). $^{13}C\{^1H,^{19}F\}$ NMR spectra were recorded using the ^1H and ^{19}F simultaneous decoupling method. Chemical shifts were reported as the delta scale in ppm. High-resolution mass spectra (HRMS) were recorded on a Bruker micrOTOF mass spectrometer (APCI or ESI). Electrochemical measurements were performed under dry argon atmosphere using a BAS Electrochemical Analyzer Model 1210C, a glassy carbon working electrode (diameter: 3 mm), a Pt wire counter electrode, and a Ag wire/$AgNO_3$ in MeCN reference electrode. UV-vis-NIR spectra were recorded on a JASCO V-670 spectrometer. ESR spectra were recorded at the X-band frequency using a JEOL JES-X320 spectrometer equipped with a 100 kHz magnetic-field modulation. ESR microwave power low enough to prevent the saturation and distortion of the spectrum. ESR spectra were recorded at 25 °C. Elemental analysis was carried out using a YANACO MT-6 microanalyzer. Tris(4-bromophenyl)aminium hexachloroantimonate ([(*p*-$BrC_6H_4)_3N$]$SbCl_6$, Magic Blue) was purchased from Merck and purified by re-precipitation from CH_2Cl_2 and Et_2O before use. $(C_6F_5)_2BOEt$ and *p*-$(Ar_2N)C_6H_4B(OH)_2$ (Ar = 4-*tert*-butylphenyl) were prepared according to literatures.[2,3]

Synthesis of the new compounds

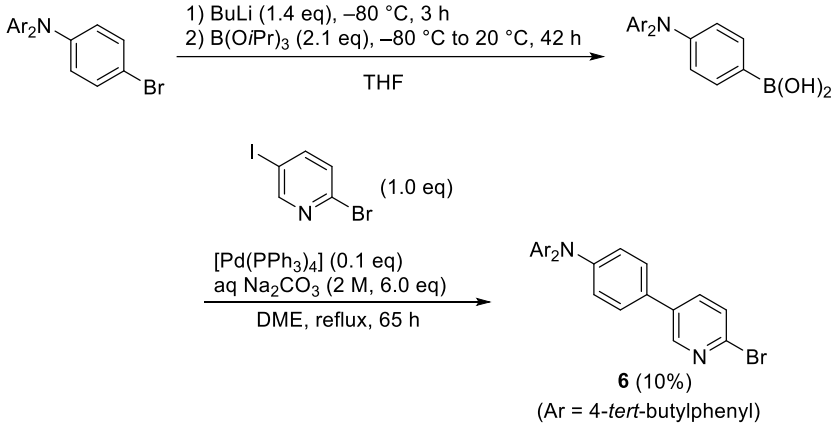
4-(6-Bromopyridin-3-yl)-*N*,*N*-bis(4-(*tert*-butyl)phenyl)aniline (5).



A mixture of 2-bromo-5-iodopyridine (8.51 g, 30.0 mmol), Ar_2NH (2.19 g, 7.77 mmol), [$Pd_2(dba)_3$] (0.19 g, 0.21 mmol), BINAP (0.26 g, 0.41 mmol) and *t*-BuONa (2.91 g, 30.00 mmol) and toluene (40 mL) was refluxed for 18 h. The reaction was quenched with aq. NH_4Cl and extracted with AcOEt three times. The organic layer was dried over $MgSO_4$ and concentrated under reduced pressure. The residue was subjected to GPC to give **5** as an orange solid (1.00 g, 2.30 mmol, 30%).

^{1}H NMR (600 MHz, $CDCl_3$) δ 8.08 (d, *J* = 2.8 Hz, 1H), 7.32 (d, *J* = 8.3 Hz, 4H), 7.28 (d, *J* = 9.6 Hz, 1H), 7.23 (dd, *J* = 8.3, 2.8 Hz, 1H), 7.04 (d, *J* = 9.0 Hz, 4H), 1.34 (s, 18H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 147.1, 144.3, 143.6, 143.6, 131.9, 130.9, 127.6, 126.5, 124.2, 34.4, 31.3; HRMS (APCI$^+$) *m/z* 437.1584 ([M+H]$^+$, calcd 437.1587).

4-(6-Bromopyridin-3-yl)-*N*,*N*-bis(4-(*tert*-butyl)phenyl)aniline 6.

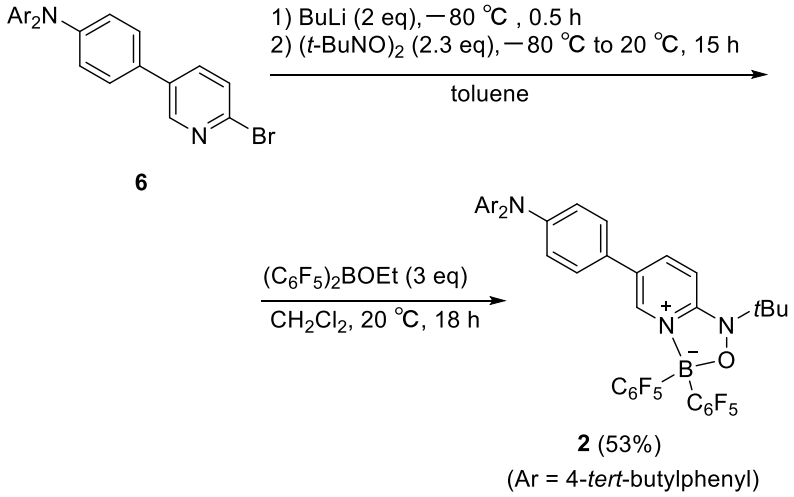


To a THF (50 mL) solution of 4-bromo-*N*,*N*-bis(4-*tert*-butylphenyl)aniline (3.76 g, 8.60 mmol) was added BuLi (1.58 M in hexane, 8.0 mL, 13 mmol) at –80 °C, and the mixture was stirred for 3 h. B(O*i*Pr)$_3$ (4.1 mL, 18 mmol) was added, and the mixture was gradually warmed to 20 °C for 42 h and then quenched with H_2O (50 mL). The mixture was extracted with AcOEt and the organic layer was dried over $MgSO_4$ and concentrated in vacuo to afford the boronic acid as a colorless solid. The crude boronic acid was mixed with 2-bromo-5-iodopyridine (2.48

g, 8.73 mmol), [Pd(PPh_3)$_4$] (0.98 g, 0.85 mmol), aq. Na_2CO_3 (2 M, 26 mL, 52 mmol), and DME (80 mL). The mixture was was refluxed for 65 h and then quenched with aq. NH_4Cl and extracted with AcOEt three times. The organic layer was dried over $MgSO_4$ and concentrated under reduced pressure. The residue was subjected to GPC to give **6** as an orange solid (0.44 g, 0.85 mmol, 10% from 4-bromo-*N*,*N*-bis(4-*tert*-butylphenyl)aniline).

^{1}H NMR (600 MHz, $CDCl_3$) δ 8.55 (d, *J* = 2.8 Hz, 1H), 7.69 (dd, *J* = 8.3, 2.8 Hz, 1H), 7.50 (d, *J* = 8.3 Hz, 1H), 7.38 (d, *J* = 8.3 Hz, 2H), 7.29 (d, *J* = 8.3 Hz, 4H), 7.11 (d, *J* = 8.3 Hz, 2H), 7.05 (d, *J* = 2.8 Hz, 4H), 1.32 (s, 18H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 148.7, 147.9, 146.4, 144.5, 139.9, 136.2, 135.7, 128.6, 127.9, 127.4, 126.2, 124.5, 122.3, 34.3, 31.4; HRMS (APCI$^+$) *m/z* 513.1899 ([M+H]$^+$, calcd 513.1900).

Aminoxide-boron complex 2.



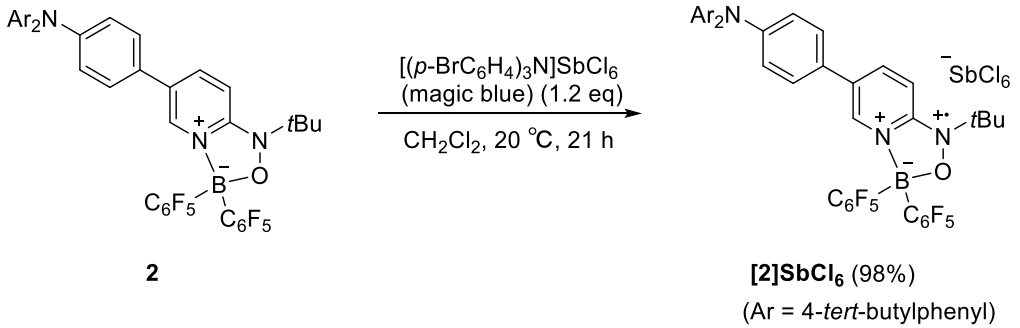
2 (53%)
(Ar = 4-*tert*-butylphenyl)

To a toluene (10 mL) solution of **6** (0.40 g, 0.78 mmol) was added BuLi (1.58 M in hexane, 1.0 mL, 1.6 mmol) at –80 °C, and the mixture was stirred for 0.5 h before the addition of (*t*-BuNO)$_2$ (0.16 g, 1.8 mmol as the monomer). The mixture was gradually warmed to 20 °C for 15 h. The reaction was quenched with aq. NH_4Cl and extracted with EtOAc three times. The organic layer was dried over $MgSO_4$ and concentrated under reduced pressure. The residue was dissolved in CH_2Cl_2 (10 mL), and (C_6F_5)$_2$BOEt (830 mg, 2.13 mmol) was added. The mixture was stirred at 20 °C for 18 h and concentrated under reduced pressure. The residue was subjected to a flash chromatography (SiO_2, DCM/hexane = 1:20 to 2:3) to give **2** as a yellow solid (360 mg, 0.42 mmol, 53%).

^{1}H NMR (600 MHz, $CDCl_3$) δ 7.98 (s, 1H), 7.82 (dd, *J* = 9.6, 2.1 Hz, 1H), 7.28 (d, *J* = 9.0 Hz, 4H), 7.25 (d, *J* = 7.6 Hz, 2H), 7.12 (d, *J* = 9.0 Hz, 1H), 7.09 (d, *J* = 9.0 Hz, 2H), 7.05 (d, *J* = 8.3

Hz, 4H), 1.54 (s, 9H), 1.32 (s, 18H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 152.4, 148.6, 148.2, 146.5, 144.4, 139.5, 137.9, 137.0, 136.3, 135.8, 128.0, 127.4, 126.7, 126.2, 124.5, 122.3, 108.2, 61.6, 34.3, 31.4, 27.9; ^{19}F NMR (565 MHz, $CDCl_3$) δ -134.6 (d, *J* = 25.4 Hz, 4F), -156.6 (t, *J* = 19.1 Hz, 2F), -163.4 (t, *J* = 22.3 Hz, 4F); HRMS (APCI$^+$) *m/z* 866.3340 ([M+H]$^+$, calcd 866.3342).

Aminoxyl-boron complex [2]$SbCl_6$.



2 **[2]$SbCl_6$** (98%)

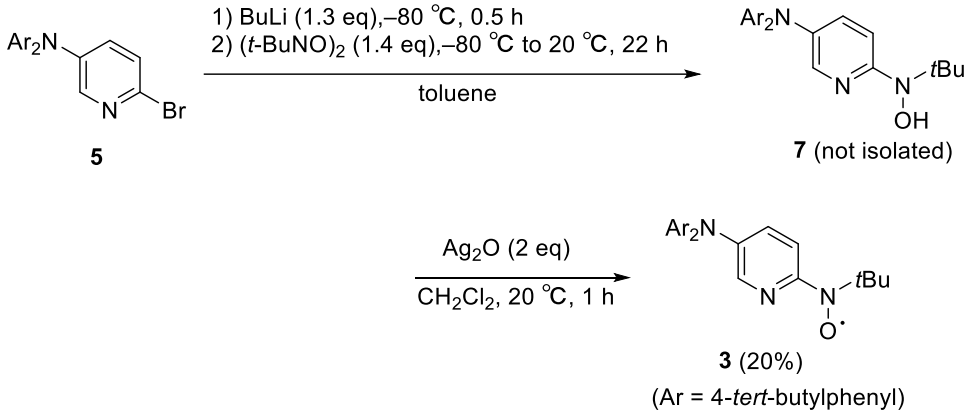
(Ar = 4-*tert*-butylphenyl)

To a CH_2Cl_2 (3 mL) solution of **2** (129 mg, 0.15 mmol) was added Magic Blue (150 mg, 0.18 mmol), and the solution was stirred at 20 °C for 21 h. The reaction mixture was concentrated under reduced pressure. The residue was washed with anhydrous Et_2O and dried to afford **[2]$SbCl_6$** as green crystals (176 mg, 0.15 mmol, 98%).

HRMS (APCI$^+$) *m/z* 866.3550 ([M+H]$^+$, calcd 866.3342).

Anal. calcd for $C_{47}H_{42}BCl_6F_{10}N_3OSb$ + 2 CH_2Cl_2: C, 42.96; H, 3.38; N, 3.07. Found: C, 42.78; H, 3.14; N. 3.00.

Aminoxyl radical 3.



5 **7** (not isolated)

3 (20%)

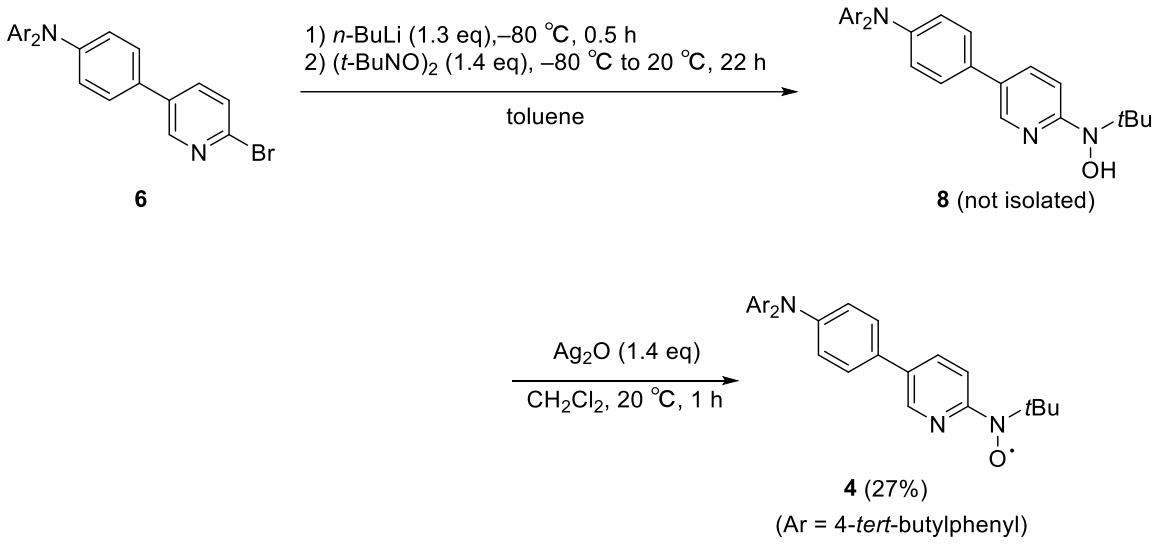
(Ar = 4-*tert*-butylphenyl)

To a toluene (10 mL) solution of **5** (546 mg, 1.25 mmol) was added BuLi (1.58 M in hexane, 1.0 mL, 1.8 mmol as the monomer) at –80 °C, and the mixture was stirred for 0.5 h before the addition of (*t*-BuNO)$_2$ (155 mg, 1.78 mmol as the monomer). The mixture was gradually warmed to 20 °C for 22 h. The reaction was quenched with aq. NH_4Cl and extracted with

EtOAc three times. The organic layer was dried over $MgSO_4$ and concentrated under reduced pressure to give hydroxylamine **7**. Crude **7** was dissolved in DCM (60 mL), and Ag_2O (1.08 g, 2.50 mmol) was added. The mixture was stirred at rt in the dark for 1 h and then was filtered over a Celite pad. The filtrate was concentrated under reduced pressure. The residue was subjected to a flash chromatography (SiO_2, DCM) to give **3** as a dark blue solid (108 mg, 0.24 mmol, 20% from **5**).

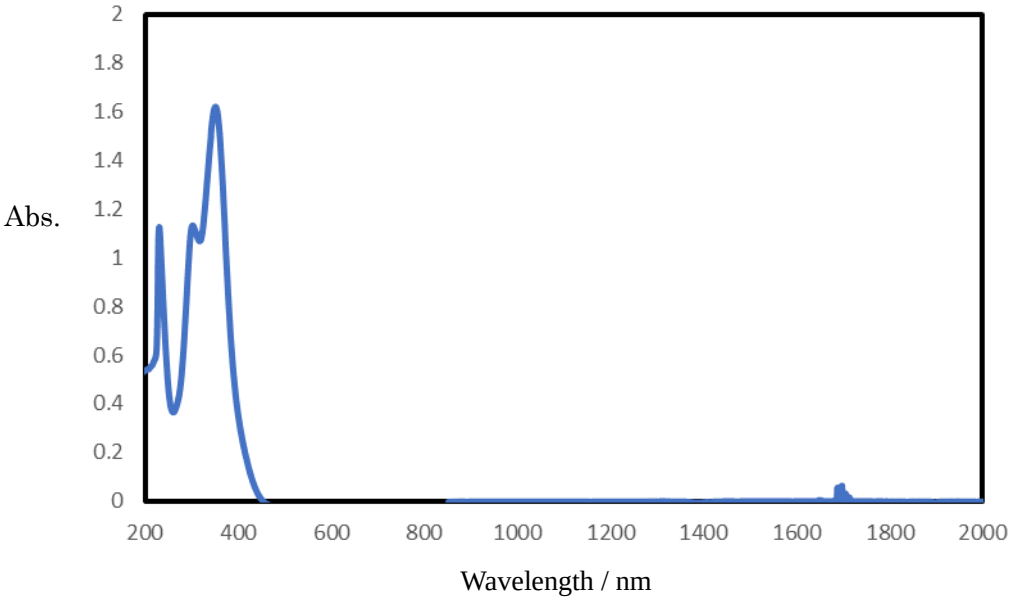
HRMS (APCI$^+$) *m/z* 445.3107 ([M+H]$^+$, calcd 445.3213). Anal. calcd for $C_{29}H_{38}N_3O_1$: C, 78.34; H, 8.61; N, 9.45; found: C, 72.61; H, 8.05; N, 7.80.

Aminoxyl radical 4.



To a toluene (10 mL) solution of **6** (546 mg, 1.25 mmol) was added BuLi (1.58 M in hexane, 1.0 mL, 1.6 mmol) at –80 °C, and the mixture was stirred for 0.5 h before the addition of (*t*-BuNO)$_2$ (152 mg, 1.74 mmol as the monomer). The mixture was gradually warmed to 20 °C for 22 h. The reaction was quenched with aq. NH_4Cl and extracted with EtOAc three times. The organic layer was dried over $MgSO_4$ and concentrated under reduced pressure to give hydroxylamine **8**. Crude **8** was dissolved in DCM (50 mL), and Ag_2O (745 mg, 1.82 mmol) was added. The mixture was stirred for 1 h and then was filtered over a Celite pad. The filtrate was concentrated under reduced pressure. The residue was subjected to a flash chromatography (SiO_2, DCM/hexane = 1:10 to 2:1) to give **4** as a dark purple solid (171 mg, 0.33 mmol, 27% from **6**).
HRMS (APCI$^+$) *m/z* 521.3247 ([M+H]$^+$, calcd 521.3401).

UV-vis spectrum of aminoxide complex 2



In DCM at 20 °C (5.0×10^{-5} M).

Single crystal X-ray diffraction analysis

X-Ray diffraction measurements were carried out on a Rigaku MicroMax-007HF diffractometer equipped with a VariMax light source or on a Bruker D8 VENTURE diffractometer (Mo Kα, λ = 0.71073 Å). The crystals were kept at 90 or 213 K while the data collection. The collected data were processed using the CrysAlisPro (ver. 1.171.41.117a) (Rigaku Oxford Diffraction, 2021) or the SAINT (V8.40A) (Bruker Nano, Inc., 2019) program packages. Using the Olex2 program,[4] the structures were solved with the SHELXT and refined with the SHELXL program packages.[5] The full-matrix least-squares refinements were performed on F^2. All non-hydrogen atoms were refined anisotropically. Hydrogen atoms were refined using the riding model.

Complete citation for the reference 15

M. J. Frisch, G. W. Trucks, H. B. Schlegel, G. E. Scuseria, M. A. Robb, J. R. Cheeseman, G. Scalmani, V. Barone, G. A. Petersson, H. Nakatsuji, X. Li, M. Caricato, A. V. Marenich, J. Bloino, B. G. Janesko, R. Gomperts, B. Mennucci, H. P. Hratchian, J. V. Ortiz, A. F. Izmaylov, J. L. Sonnenberg, D. Williams-Young, F. Ding, F. Lipparini, F. Egidi, J. Goings, B. Peng, A. Petrone, T. Henderson, D. Ranasinghe, V. G. Zakrzewski, J. Gao, N. Rega, G. Zheng, W. Liang, M. Hada, M. Ehara, K. Toyota, R. Fukuda, J. Hasegawa, M. Ishida, T. Nakajima, Y. Honda, O. Kitao, H. Nakai, T. Vreven, K. Throssell, J. A. Montgomery, Jr., J. E. Peralta, F. Ogliaro, M. J. Bearpark, J. J. Heyd, E. N. Brothers, K. N. Kudin, V. N. Staroverov, T. A. Keith, R. Kobayashi, J. Normand, K. Raghavachari, A. P. Rendell, J. C. Burant, S. S. Iyengar, J. Tomasi, M. Cossi, J. M. Millam, M. Klene, C. Adamo, R. Cammi, J. W. Ochterski, R. L. Martin, K. Morokuma, O. Farkas, J. B. Foresman and D. J. Fox, Gaussian 16, Revision C.02, Gaussian, Inc., Wallingford CT, 2019.

DFT-optimized coordinates

Aminoxide complex **1**

F	-5.09942	-1.12678	-0.32373
F	-0.67467	-2.11641	-1.65635
F	-3.42459	0.896237	-2.04822
F	-6.02454	-2.76792	-2.22347
F	-4.29563	-4.0839	-3.85203
F	-1.61699	-3.73267	-3.54141
O	-3.08265	-1.07338	1.612046
F	-2.47264	5.110435	-0.30464
F	-1.65859	1.348533	2.326276
F	-3.38621	3.530337	-2.31267
N	-0.92363	-0.92362	0.851043
F	-1.63135	3.988506	2.018695
N	2.58748	0.000452	0.560412
N	-2.25366	-2.02352	2.24372
C	-0.96465	-1.73799	1.92115
C	1.42625	-0.68422	0.964738
C	4.897724	-2.69402	-0.53219
H	4.809187	-3.68749	-0.95217
C	0.219482	-0.41844	0.37118
H	0.130595	0.24772	-0.47502
C	3.772341	-0.71301	0.265816
C	-2.85052	-1.57366	-0.87592
C	3.725984	-1.99344	-0.273
H	2.768974	-2.44636	-0.50582
C	-2.55958	0.980659	0.16128
C	6.151547	-2.14479	-0.28649
C	2.967393	1.950323	-0.88648
H	3.496965	1.324603	-1.59541
C	0.250638	-2.15477	2.494139
H	0.269691	-2.85062	3.314967
C	2.467714	1.392818	0.287814
C	1.419379	-1.60895	2.02607

H	2.358659	-1.87675	2.495836
C	5.021937	-0.14807	0.524807
H	5.081169	0.847643	0.948195
C	-2.80669	-2.38622	3.572547
C	2.774924	3.294946	-1.15634
H	3.168942	3.688915	-2.0864
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C	6.177641	-0.85112	0.243898
H	7.128023	-0.37538	0.458676
C	-2.99289	1.606289	-0.9988
C	-2.47381	-3.09735	-2.7395
C	-4.20763	-1.77779	-1.08365
C	-2.01497	-2.25359	-1.74086
C	1.791567	2.220322	1.177898
H	1.395002	1.809667	2.099499
C	-4.71038	-2.60814	-2.06541
C	-2.13101	1.840855	1.167021
C	1.58936	3.56128	0.885451
H	1.038473	4.157527	1.600414
C	1.84038	5.604004	-0.65255
C	-3.83223	-3.27757	-2.89993
C	-2.06327	-3.59426	4.14696
H	-2.67819	-4.0447	4.926767
H	-1.11587	-3.32972	4.614772
H	-1.88806	-4.3542	3.383324
C	-2.10322	3.215058	1.037038
C	-2.53022	3.789855	-0.14445
C	-2.98376	2.982635	-1.16719
C	3.191796	6.29419	-0.89655
H	3.041292	7.3474	-1.14689
H	3.744249	5.838088	-1.71989
H	3.823251	6.246843	-0.00643
C	7.223773	-4.2821	-1.14918
H	8.181574	-4.77256	-1.33538

H	6.68665	-4.24574	-2.09955
H	6.660062	-4.91866	-0.4634
C	7.461652	-2.88455	-0.57059
C	1.1079	6.365645	0.456399
H	0.974676	7.408165	0.159831
H	1.668881	6.361623	1.393897
H	0.114712	5.956172	0.650532
B	-2.40855	-0.62462	0.378474
C	-2.72138	-1.18444	4.514804
H	-3.16646	-1.42796	5.481723
H	-3.25169	-0.33173	4.093375
H	-1.68347	-0.88937	4.686512
C	-4.26483	-2.78715	3.336885
H	-4.72317	-3.05981	4.289055
H	-4.32219	-3.64727	2.667116
H	-4.83302	-1.97405	2.89332
C	0.988019	5.685365	-1.93007
H	0.846034	6.727656	-2.22749
H	-0.00083	5.249232	-1.772
H	1.454865	5.163036	-2.76722
C	8.294549	-2.08279	-1.5839
H	9.235007	-2.59714	-1.7985
H	8.541824	-1.0868	-1.21237
H	7.754933	-1.96047	-2.52563
C	8.259169	-3.0364	0.734867
H	9.199794	-3.56179	0.54981
H	7.694533	-3.60753	1.475306
H	8.504371	-2.07	1.178871

Aminoxyl complex **1**$^{+}$

F	-1.679	0.329048	2.060904
F	-3.94244	-3.34213	0.115007
F	-1.21499	0.998661	-2.40798
O	-2.95329	-1.58663	-1.61631

F	-1.61178	3.619334	-2.62112
F	-4.52534	0.807731	0.968858
F	-4.46963	-4.27861	2.529896
F	-2.21604	-0.65369	4.491013
N	2.524327	-0.03388	-0.20848
N	-0.88992	-1.04515	-0.71745
F	-3.62674	-2.96347	4.749072
F	-3.48908	4.854633	-1.1007
F	-4.95772	3.408393	0.669109
N	-1.98603	-2.42396	-2.04652
C	3.829186	-0.58383	-0.0489
C	-2.81505	-1.43395	0.980086
C	-0.78309	-2.09127	-1.58741
C	0.163314	-0.42032	-0.23861
H	-0.02711	0.400318	0.43802
C	4.932634	0.071035	-0.59192
H	4.797724	0.991333	-1.1473
C	2.324792	1.369017	-0.02698
C	1.464955	-0.80806	-0.59022
C	-2.81872	0.783134	-0.66899
C	-2.39015	-0.81773	2.147349
C	6.19338	-0.46581	-0.42531
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C	6.405863	-1.65591	0.28147
C	1.846205	4.112427	0.342896
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C	1.613894	2.093955	-0.98512
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H	2.965356	3.845565	2.161857
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H	0.819452	3.974459	-1.54929
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H	2.583617	-2.35282	-1.62691
C	0.499261	-2.6191	-1.8779
H	0.611681	-3.48079	-2.51616
C	-3.5135	-2.61673	1.163018
C	2.823121	2.016379	1.095842
H	3.380809	1.461031	1.840593
C	-3.368	-2.47406	3.547002
C	-3.79802	-3.13911	2.414933
C	-2.64948	-1.29755	3.413695
C	7.824215	-2.20478	0.441249
C	4.010861	-1.76226	0.664662
H	3.162598	-2.25679	1.124817
C	-3.78545	1.456787	0.066755
C	-2.3967	-3.49932	-3.01431
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C	5.284976	-2.28635	0.820582
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H	-1.91412	-2.25982	-4.7314
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H	8.475346	-1.58184	-1.56303
H	7.824367	-3.22089	-1.49435
C	7.85527	-3.50595	1.247507
H	7.476772	-3.37131	2.263271
H	8.884077	-3.85868	1.332805
H	7.281297	-4.3024	0.76822
C	-3.90687	-3.40345	-3.21248
H	-4.19852	-2.44859	-3.64623

H	-4.44509	-3.5355	-2.27713
H	-4.20363	-4.1964	-3.89937
C	-2.04214	-4.85278	-2.39521
H	-2.37245	-5.6475	-3.06462
H	-2.55193	-4.9801	-1.4403
H	-0.97119	-4.98539	-2.23834
C	8.69047	-1.16324	1.169516
H	8.292486	-0.94063	2.161856
H	8.75893	-0.22394	0.618236
H	9.706729	-1.54236	1.294735
C	0.012542	5.797482	0.536995
H	-0.46171	5.466684	-0.38908
H	-0.44686	5.252328	1.364745
H	-0.2264	6.855335	0.662753
C	2.143039	6.383835	-0.65784
H	1.730182	6.070697	-1.61822
H	1.933341	7.449355	-0.5446
H	3.227201	6.259727	-0.7021
C	2.114034	6.160739	1.821821
H	3.202936	6.082534	1.859086
H	1.865423	7.219703	1.905235
H	1.702351	5.663305	2.70292

Aminoxide complex **2**

F	5.186851	2.566319	0.04278
F	3.581785	-0.76673	-2.91475
O	5.302822	-1.39358	-0.62151
F	2.647891	1.480629	1.260214
F	6.616754	-1.06278	1.701434
F	3.611887	1.006891	-4.8976
N	3.045765	-1.24026	-0.21339
F	2.900288	2.277906	3.794622
N	4.780314	-2.63625	-0.21943
F	5.196539	4.306552	-1.95316

F	6.840286	-0.25092	4.202803
F	4.414603	3.5619	-4.44313
N	-4.72217	-0.13633	0.045225
F	5.004604	1.422428	5.296121
C	3.426481	-2.53428	-0.18925
C	-0.67842	-1.36778	-0.06351
C	1.755345	-0.87501	-0.19438
H	1.573554	0.18721	-0.25817
C	0.735166	-1.7921	-0.10663
C	-5.05279	1.244269	0.100584
C	-7.87467	-2.986	0.03372
C	-5.77064	-1.09393	0.039956
C	-1.67365	-2.09605	-0.71559
H	-1.40812	-2.97467	-1.29425
C	4.598756	0.151632	1.357195
C	5.596118	-3.74969	-0.77229
C	4.3603	0.813439	-1.32774
C	-5.72696	-2.21169	0.864056
H	-4.87878	-2.35659	1.523158
C	-3.37915	-0.54656	0.011293
C	-2.99649	-1.6958	-0.68649
H	-3.74359	-2.26941	-1.22042
C	-6.75765	-3.14205	0.84977
H	-6.67834	-3.99667	1.509082
C	1.124035	-3.14396	-0.04254
H	0.36133	-3.9082	0.055794
C	2.439465	-3.52857	-0.08628
H	2.704214	-4.56987	-0.02618
C	-1.06571	-0.21999	0.629586
H	-0.32568	0.35413	1.177396
C	-6.01643	1.70865	0.986236
H	-6.51224	1.009731	1.649789
C	-5.73232	3.988706	0.197467
C	-6.35203	3.055686	1.023689

H	-7.11002	3.369782	1.729395
C	-2.38721	0.183686	0.672095
H	-2.65957	1.068031	1.234104
C	3.983327	0.482807	-2.6244
C	-4.43069	2.163034	-0.74345
H	-3.68276	1.819808	-1.44882
C	3.699761	1.010006	1.968601
C	-7.90682	-1.85023	-0.77978
H	-8.75164	-1.68127	-1.43808
C	4.771045	2.126425	-1.15191
C	-9.03371	-3.98627	0.000775
C	-4.76143	3.504151	-0.68353
H	-4.25367	4.186422	-1.35599
C	5.657512	-0.24717	2.158506
C	-6.87912	-0.92515	-0.7879
H	-6.93238	-0.05887	-1.43692
C	3.990122	1.38041	-3.67489
C	7.05537	-3.42872	-0.44298
H	7.196121	-3.33748	0.634198
H	7.369939	-2.49562	-0.90193
H	7.69029	-4.23526	-0.81371
C	5.230349	-5.06837	-0.08594
H	6.03943	-5.78174	-0.24575
H	4.329971	-5.52762	-0.4918
H	5.113229	-4.9361	0.990967
C	3.805403	1.4503	3.271268
C	5.389641	-3.83468	-2.28541
H	5.641078	-2.8858	-2.75782
H	4.352423	-4.07368	-2.53234
H	6.024878	-4.61344	-2.71235
C	4.793043	3.056788	-2.17835
C	4.874917	1.01931	4.034905
C	4.39889	2.681438	-3.44713
C	-6.07079	5.481931	0.219219

C	5.804794	0.167885	3.474148
B	4.376268	-0.34278	-0.19211
C	-8.81693	-5.15692	0.963657
H	-7.91884	-5.72812	0.717888
H	-9.66462	-5.84316	0.907404
H	-8.73577	-4.82404	2.00083
C	-10.3372	-3.27503	0.398777
H	-10.2657	-2.86029	1.406729
H	-11.1762	-3.97566	0.38104
H	-10.5787	-2.45519	-0.27982
C	-9.17946	-4.55578	-1.4197
H	-9.38332	-3.77459	-2.15401
H	-10.0046	-5.27162	-1.46232
H	-8.26869	-5.07265	-1.7304
C	-7.15149	5.816313	1.251156
H	-8.09054	5.298519	1.043136
H	-7.3599	6.888165	1.233075
H	-6.83996	5.562285	2.266808
C	-6.5819	5.91197	-1.16544
H	-5.8375	5.744746	-1.94581
H	-6.82845	6.977006	-1.16831
H	-7.48144	5.357047	-1.44126
C	-4.80989	6.289053	0.568399
H	-4.42291	6.007333	1.550193
H	-5.03395	7.358837	0.588677
H	-4.01103	6.13424	-0.15882

Aminoxyl complex **2**$^{+}$

B	-4.35412	-0.33966	-0.01316
N	-3.05365	-1.27626	-0.06811
C	-3.44822	-2.57803	-0.20725
C	-2.44344	-3.57556	-0.34303
H	-2.70497	-4.61116	-0.4653
C	-1.1391	-3.19903	-0.30111

H	-0.38586	-3.97345	-0.3777
C	-0.74255	-1.84435	-0.1218
C	-1.77278	-0.92645	-0.01558
H	-1.59883	0.135819	0.076527
N	-4.76713	-2.65957	-0.20342
O	-5.35961	-1.43925	0.01372
C	-4.45668	0.461687	1.394717
C	-4.86674	1.77726	1.557372
C	-4.99683	2.380385	2.798239
C	-4.7242	1.656032	3.942673
C	-4.3295	0.334509	3.833276
C	-4.21414	-0.22657	2.577215
F	-5.17252	2.539218	0.501293
F	-5.38677	3.647228	2.895401
F	-4.84314	2.219689	5.13684
F	-4.07191	-0.37873	4.927313
F	-3.84844	-1.52534	2.533155
C	-4.39298	0.554191	-1.37625
C	-5.25173	0.330108	-2.44171
C	-5.24044	1.101033	-3.59534
C	-4.33944	2.139665	-3.71829
C	-3.45621	2.395944	-2.68406
C	-3.50746	1.605465	-1.55623
F	-6.14924	-0.66376	-2.41816
F	-6.08676	0.839204	-4.58657
F	-4.31216	2.879762	-4.81858
F	-2.57477	3.388657	-2.78658
F	-2.62634	1.905533	-0.57085
C	-5.76183	-3.76992	-0.13175
C	-5.12087	-5.1243	-0.40783
H	-4.62882	-5.1617	-1.38105
H	-5.9178	-5.86728	-0.42587
H	-4.42545	-5.42812	0.375072
C	-6.35941	-3.74826	1.277258

H	-5.58748	-3.89843	2.034045
H	-7.09454	-4.54872	1.372107
H	-6.85531	-2.80044	1.474958
C	-6.83015	-3.48421	-1.18782
H	-7.29534	-2.5155	-1.02705
H	-7.60009	-4.25507	-1.13472
H	-6.40234	-3.49292	-2.19136
C	0.645955	-1.42124	-0.07822
C	1.669912	-2.19626	-0.66147
H	1.421545	-3.10646	-1.1922
C	2.980437	-1.79825	-0.63681
H	3.734663	-2.39329	-1.13415
C	3.346529	-0.57914	-0.02576
C	2.332237	0.204585	0.567343
H	2.596699	1.116267	1.086081
C	1.029475	-0.21332	0.544374
H	0.291681	0.385673	1.062581
N	4.659449	-0.1627	-0.00986
C	4.984406	1.219163	0.034021
C	4.318467	2.127317	-0.78489
H	3.559255	1.779783	-1.47536
C	4.65241	3.470558	-0.74089
H	4.126728	4.148388	-1.399
C	5.648101	3.950029	0.110901
C	6.306492	3.013845	0.920148
H	7.081842	3.342166	1.600915
C	5.993747	1.671583	0.886077
H	6.512997	0.971598	1.52921
C	6.037403	5.426126	0.183334
C	5.213949	6.291529	-0.77426
H	5.356081	6.002393	-1.81798
H	5.52558	7.333147	-0.68473
H	4.145789	6.25529	-0.54867
C	7.523603	5.575214	-0.18432

H	7.719486	5.217316	-1.19739
H	8.175708	5.026045	0.496926
H	7.816315	6.626126	-0.13932
C	5.809738	5.937438	1.616312
H	6.082215	6.992522	1.684275
H	6.411677	5.397704	2.349296
H	4.761774	5.844064	1.909243
C	5.723377	-1.10407	-0.03873
C	5.69369	-2.22734	0.782754
H	4.863654	-2.37976	1.462398
C	6.744347	-3.12959	0.756705
H	6.699566	-3.98374	1.418081
C	7.845822	-2.94684	-0.07982
C	7.851837	-1.80571	-0.89342
H	8.68379	-1.62651	-1.5629
C	6.819376	-0.89195	-0.87661
H	6.846905	-0.0219	-1.52122
C	9.019733	-3.92434	-0.13136
C	9.149318	-4.4811	-1.55953
H	9.983844	-5.18311	-1.61317
H	9.336013	-3.69632	-2.29443
H	8.244973	-5.01409	-1.86119
C	8.838116	-5.10137	0.830702
H	8.772236	-4.77722	1.871729
H	9.69713	-5.76973	0.756528
H	7.949825	-5.6925	0.596544
C	10.31245	-3.18254	0.249077
H	10.25118	-2.77126	1.258935
H	10.53473	-2.36107	-0.43403
H	11.16064	-3.86945	0.219404

Aminoxyl radical **3**

N	-0.44598	-0.02338	-0.1021
C	0.877387	-0.49357	-0.19491

C	1.915497	0.124431	0.50012
H	1.709921	0.985585	1.127647
N	3.181157	-0.26706	0.437515
C	3.485779	-1.31802	-0.31236
C	2.523924	-2.02212	-1.04577
H	2.825864	-2.86652	-1.64631
C	1.216961	-1.59626	-0.98515
H	0.448935	-2.11383	-1.54738
N	4.830281	-1.74332	-0.36834
O	5.087834	-2.75472	-1.09112
C	5.964583	-1.07719	0.368097
C	7.235709	-1.84842	0.026483
H	7.168442	-2.8907	0.334113
H	8.072567	-1.38265	0.550395
H	7.440956	-1.83167	-1.04272
C	5.712945	-1.15343	1.876233
H	4.834153	-0.58296	2.16463
H	6.57816	-0.75173	2.408329
H	5.579289	-2.19106	2.189843
C	6.10595	0.371605	-0.10591
H	6.246757	0.408085	-1.18839
H	6.985149	0.820862	0.361246
H	5.234174	0.965299	0.155576
C	-1.52366	-0.94311	-0.0554
C	-1.44306	-2.09959	0.720149
H	-0.54874	-2.29396	1.300666
C	-2.49535	-2.99601	0.753962
H	-2.3906	-3.88253	1.369486
C	-3.67668	-2.78007	0.039759
C	-3.74464	-1.6157	-0.72009
H	-4.63132	-1.3931	-1.29941
C	-2.68912	-0.71575	-0.77798
H	-2.77348	0.175002	-1.38925
C	-4.81797	-3.79796	0.11785

C	-6.02055	-3.38678	-0.73618
H	-6.44521	-2.43365	-0.41312
H	-6.80772	-4.13913	-0.65211
H	-5.76149	-3.30314	-1.79403
C	-5.28897	-3.93007	1.57526
H	-4.48724	-4.26739	2.234479
H	-6.1041	-4.65478	1.650114
H	-5.65198	-2.97363	1.958117
C	-4.32229	-5.1649	-0.38122
H	-3.98256	-5.10437	-1.41758
H	-5.1257	-5.90474	-0.3323
H	-3.49134	-5.54156	0.217709
C	-0.70547	1.371428	-0.0808
C	-1.63983	1.906762	0.804948
H	-2.16878	1.248767	1.484343
C	-1.89421	3.265614	0.817732
H	-2.62821	3.640877	1.522129
C	-1.22586	4.153314	-0.02985
C	-0.29149	3.603034	-0.90241
H	0.254732	4.237142	-1.5886
C	-0.03916	2.238089	-0.93867
H	0.685543	1.843759	-1.64157
C	-1.53475	5.651864	0.030554
C	-0.70569	6.455539	-0.97515
H	-0.95744	7.515442	-0.89786
H	-0.90154	6.148488	-2.00497
H	0.36659	6.361178	-0.78967
C	-3.02191	5.88296	-0.28287
H	-3.67331	5.374329	0.429923
H	-3.27372	5.51726	-1.28087
H	-3.26009	6.949269	-0.24434
C	-1.22423	6.183197	1.439329
H	-1.81952	5.682648	2.20496
H	-1.44002	7.253177	1.50115

H	-0.17134	6.03663	1.690327

Aminoxyl radical **4**

O	-8.26572	-1.90411	-0.07448
N	-5.75794	0.512717	0.045343
N	-7.74355	-0.74892	-0.02153
N	2.091198	-0.00419	-0.00213
C	-6.33378	-0.68043	-0.0174
C	-2.12378	-0.40042	-0.01806
C	-4.4316	0.571305	0.047209
H	-4.00744	1.56815	0.125457
C	-3.59358	-0.53895	-0.01929
C	2.689747	1.281616	-0.01489
C	4.584102	-3.44581	-0.00815
C	2.919825	-1.15568	-0.00469
C	-1.31388	-1.29578	0.6826
H	-1.77061	-2.09013	1.263192
C	-8.6554	0.451417	0.033142
C	2.647534	-2.23501	-0.83668
H	1.788978	-2.19706	-1.49698
C	0.688567	-0.13637	-0.00888
C	0.062724	-1.16689	0.696218
H	0.663779	-1.86451	1.266297
C	3.461869	-3.35964	-0.8272
H	3.207367	-4.17476	-1.49222
C	-4.22765	-1.78439	-0.08067
H	-3.63472	-2.68953	-0.1528
C	-5.60126	-1.87117	-0.0783
H	-6.1223	-2.81463	-0.13297
C	-1.49051	0.629145	-0.71573
H	-2.08301	1.323052	-1.30196
C	3.773963	1.555561	-0.83962
H	4.160298	0.778305	-1.48849
C	3.89772	3.842222	-0.02788

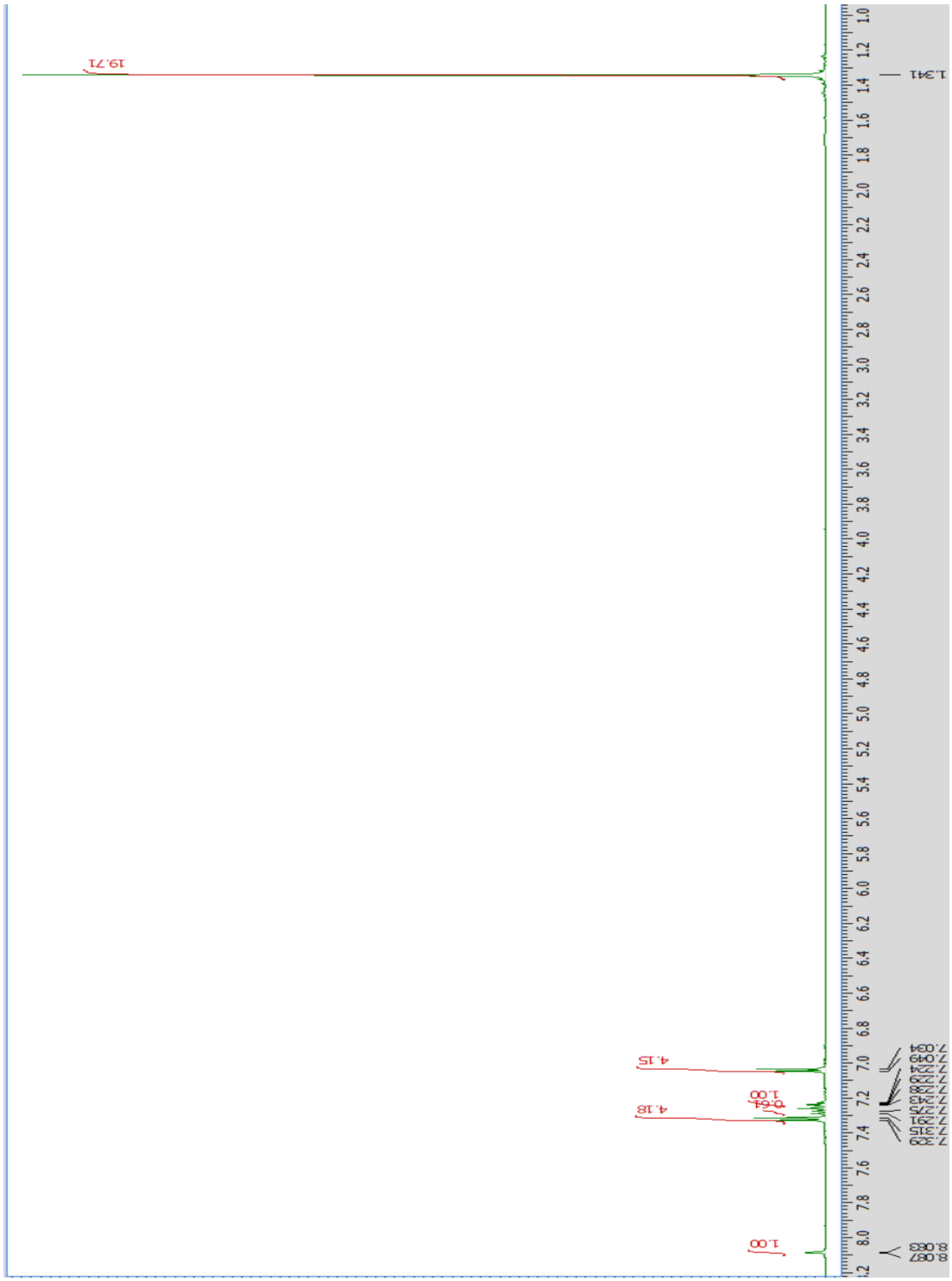
C	4.367848	2.810785	-0.83546
H	5.210786	2.97417	-1.49437
C	-0.11338	0.758868	-0.71962
H	0.350765	1.555404	-1.28797
C	2.213202	2.300795	0.809377
H	1.373981	2.106985	1.467125
C	4.848765	-2.34726	0.814034
H	5.707144	-2.36294	1.47643
C	5.508658	-4.66617	0.020517
C	2.802796	3.55116	0.790528
H	2.400958	4.315088	1.446878
C	4.03629	-1.22859	0.827317
H	4.266017	-0.39832	1.484814
C	-8.41077	1.339058	-1.18977
H	-8.54517	0.767842	-2.11091
H	-7.41082	1.764236	-1.18244
H	-9.13568	2.156039	-1.19392
C	-10.0879	-0.07216	0.001005
H	-10.7672	0.781487	0.039404
H	-10.2977	-0.72258	0.848648
H	-10.2895	-0.63706	-0.90781
C	-8.42161	1.214863	1.339215
H	-7.421	1.636304	1.382896
H	-8.56549	0.556449	2.198641
H	-9.14566	2.028987	1.416438
C	4.522072	5.240265	-0.00573
C	5.055716	-5.7597	-0.95093
H	4.057257	-6.13217	-0.71119
H	5.741282	-6.60819	-0.89763
H	5.049409	-5.41045	-1.98588
C	6.933425	-4.24064	-0.36971
H	6.953616	-3.81418	-1.37512
H	7.607783	-5.10105	-0.35455
H	7.338096	-3.49312	0.314767

C	5.527315	-5.26274	1.437222
H	5.887212	-4.5459	2.177291
H	6.184525	-6.13556	1.476715
H	4.527486	-5.57957	1.742301
C	5.705109	5.362114	-0.9702
H	6.510685	4.668452	-0.7192
H	6.118145	6.372025	-0.92242
H	5.409103	5.179778	-2.00571
C	5.026474	5.555165	1.411932
H	4.220036	5.531694	2.146929
H	5.474979	6.551646	1.446369
H	5.783348	4.833739	1.727856
C	3.465707	6.280913	-0.41126
H	3.089172	6.085469	-1.41783
H	3.894798	7.286433	-0.40088
H	2.611032	6.280738	0.267432

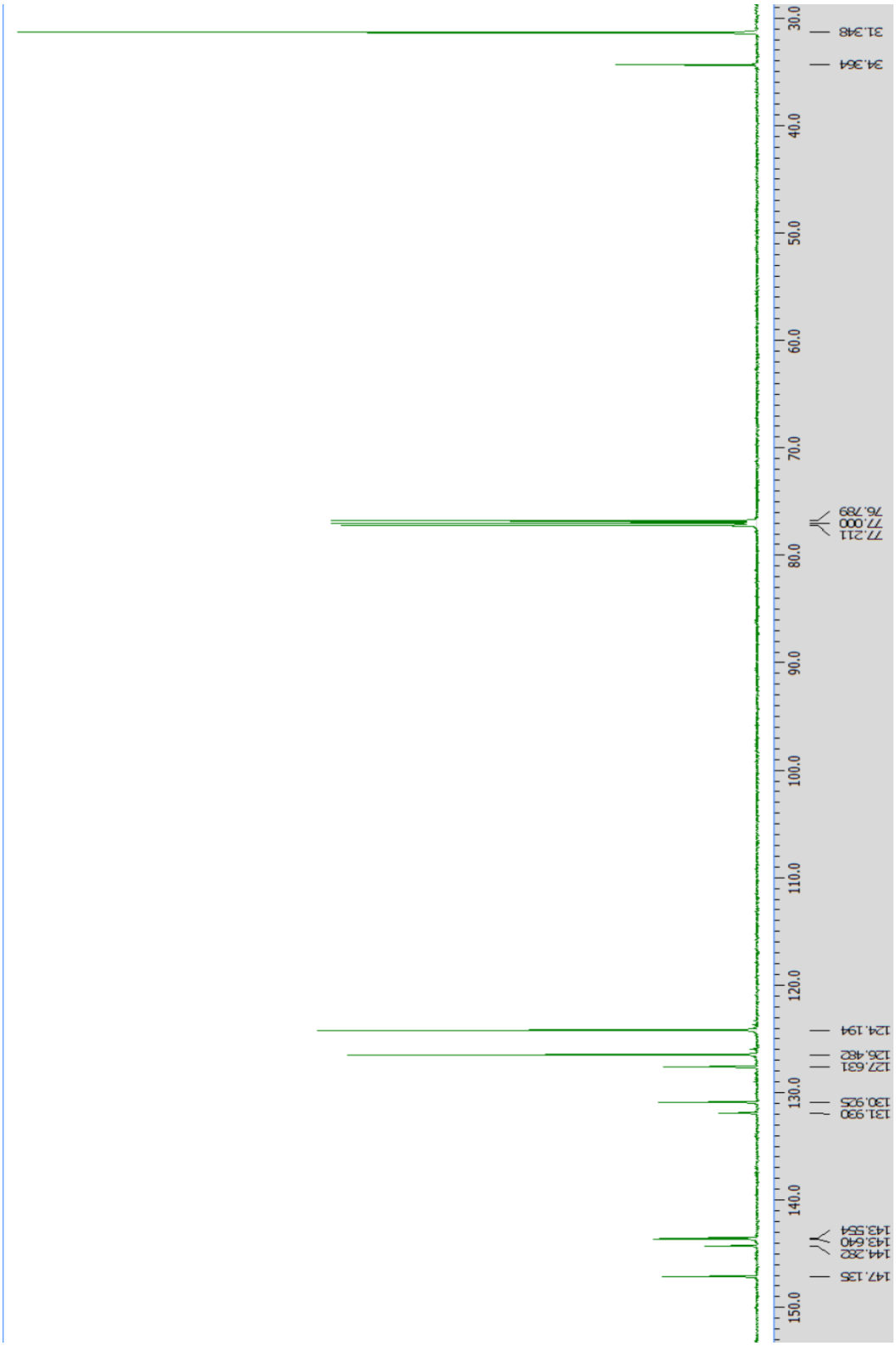
Copies of the ^{1}H and ^{13}C NMR and HRMS spectra

Compound **5**

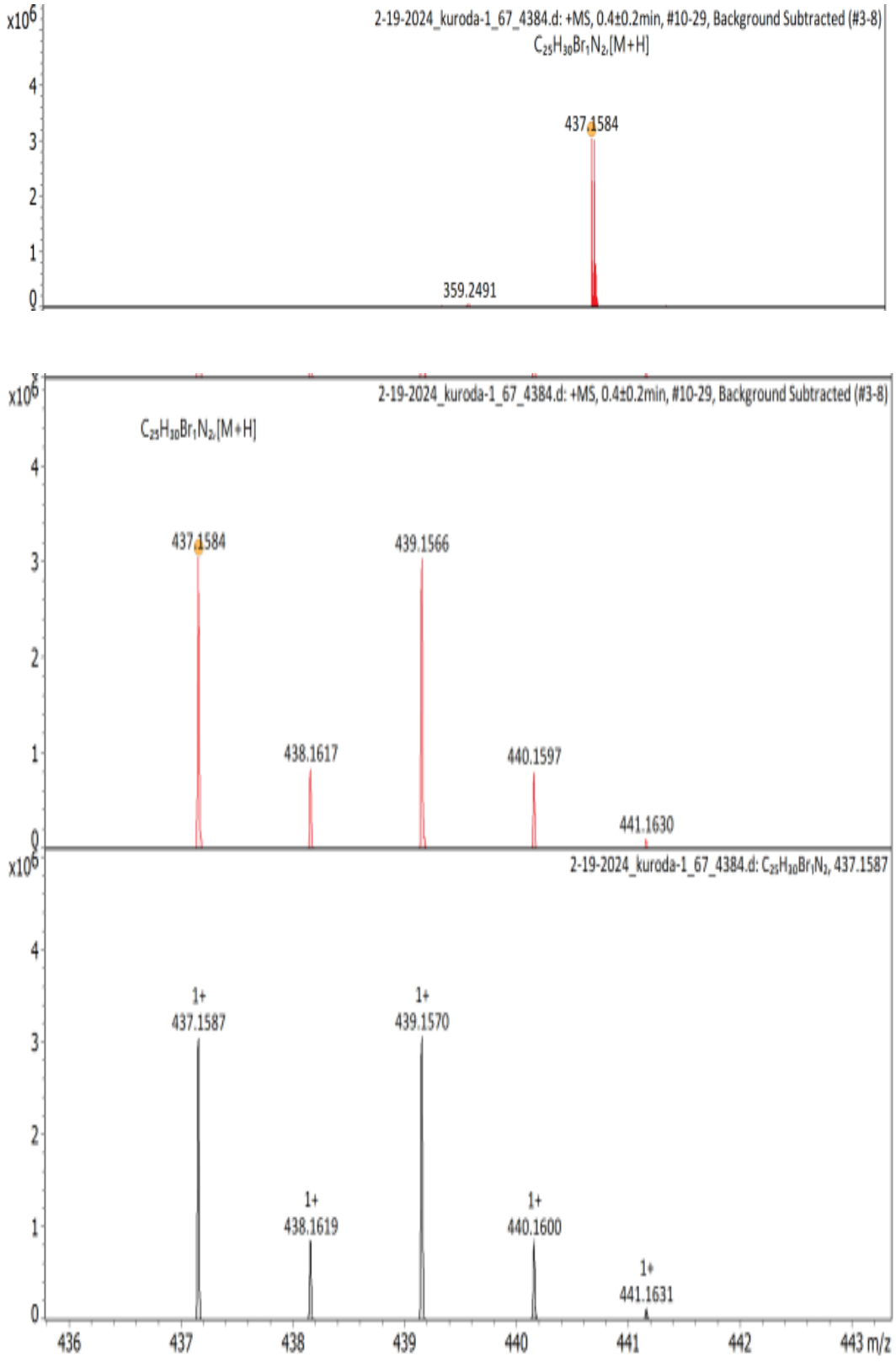
^{1}H NMR (600 MHz, $CDCl_3$)



^{13}C NMR (150 MHz, $CDCl_3$)

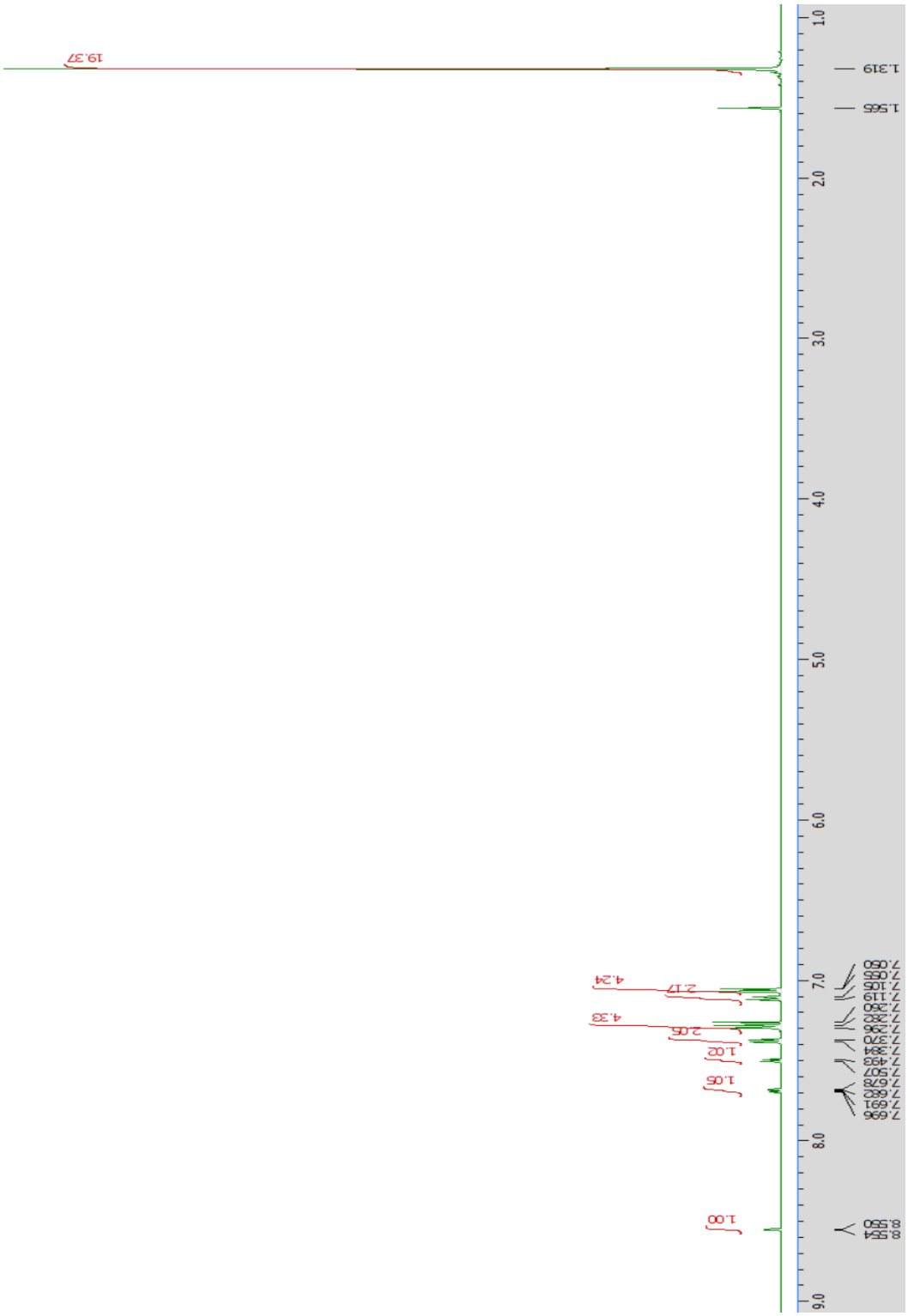


HRMS (APCI$^+$)

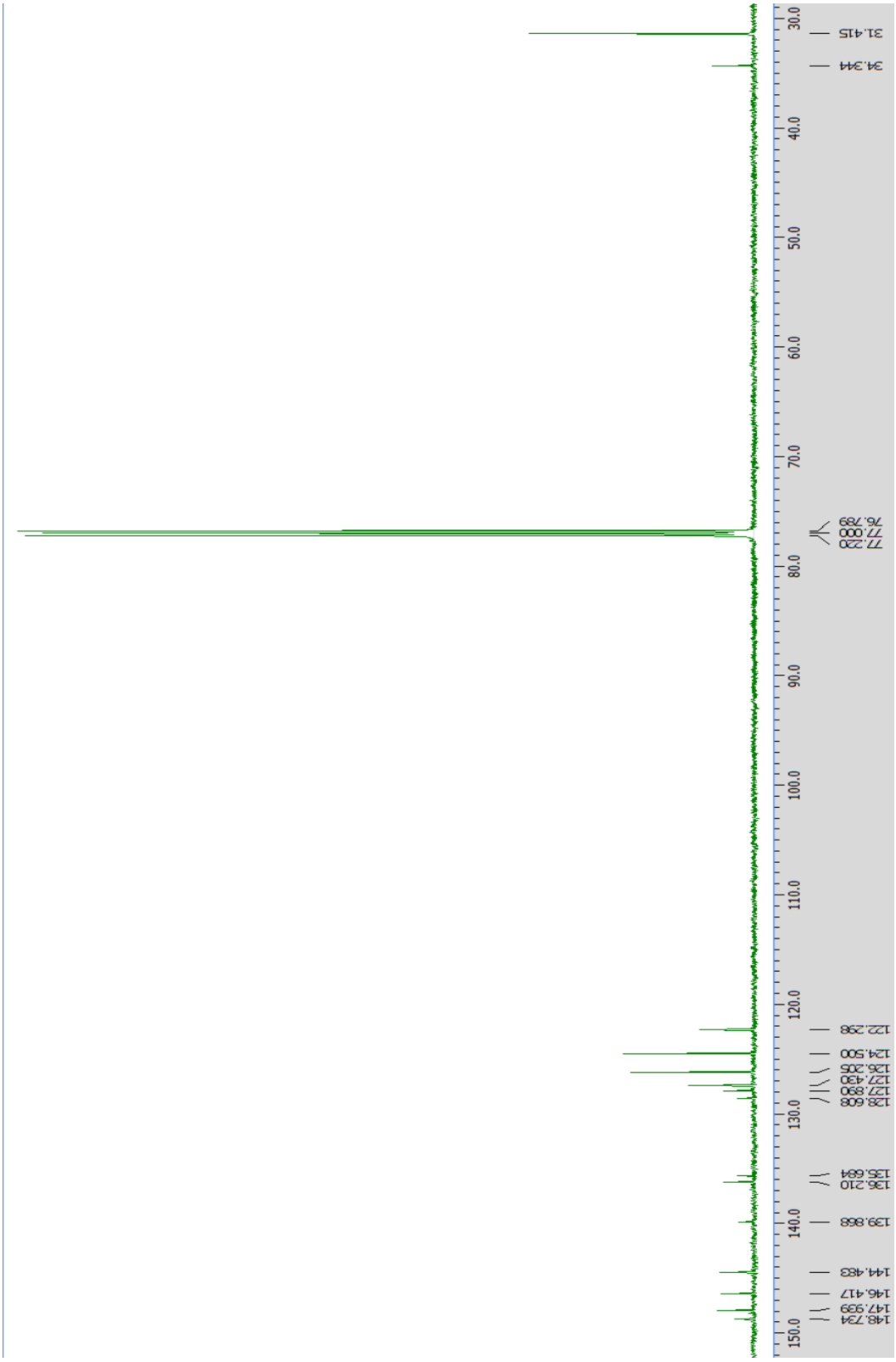

2-19-2024_kuroda-1_67_4384.d: +MS, 0.4±0.2min, #10-29, Background Subtracted (#3-8)
C25H30Br1N2,[M+H]
437.1584
359.2491
2-19-2024_kuroda-1_67_4384.d: +MS, 0.4±0.2min, #10-29, Background Subtracted (#3-8)
C25H30Br1N2,[M+H]
437.1584
439.1566
438.1617
440.1597
441.1630
2-19-2024_kuroda-1_67_4384.d: C25H30Br1N2, 437.1587
1+
437.1587
1+
439.1570
1+
438.1619
1+
440.1600
1+
441.1631
436
437
438
439
440
441
442
443 m/z

Compound **6**

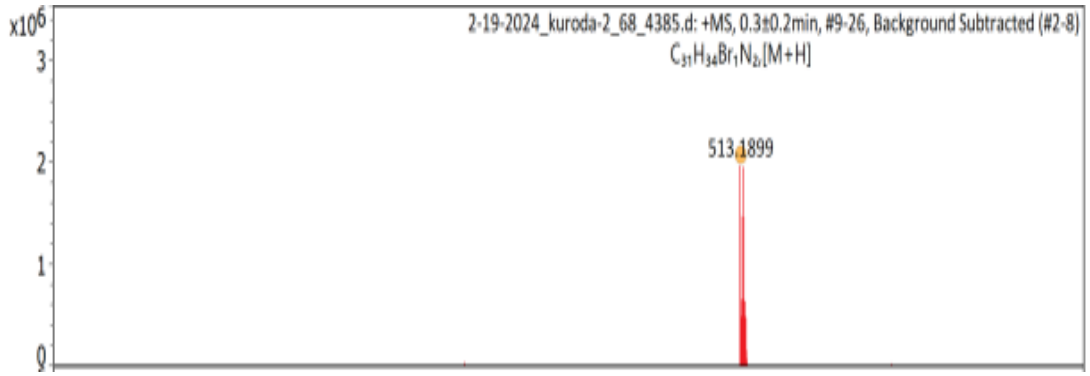
^{1}H NMR (600 MHz, $CDCl_3$)

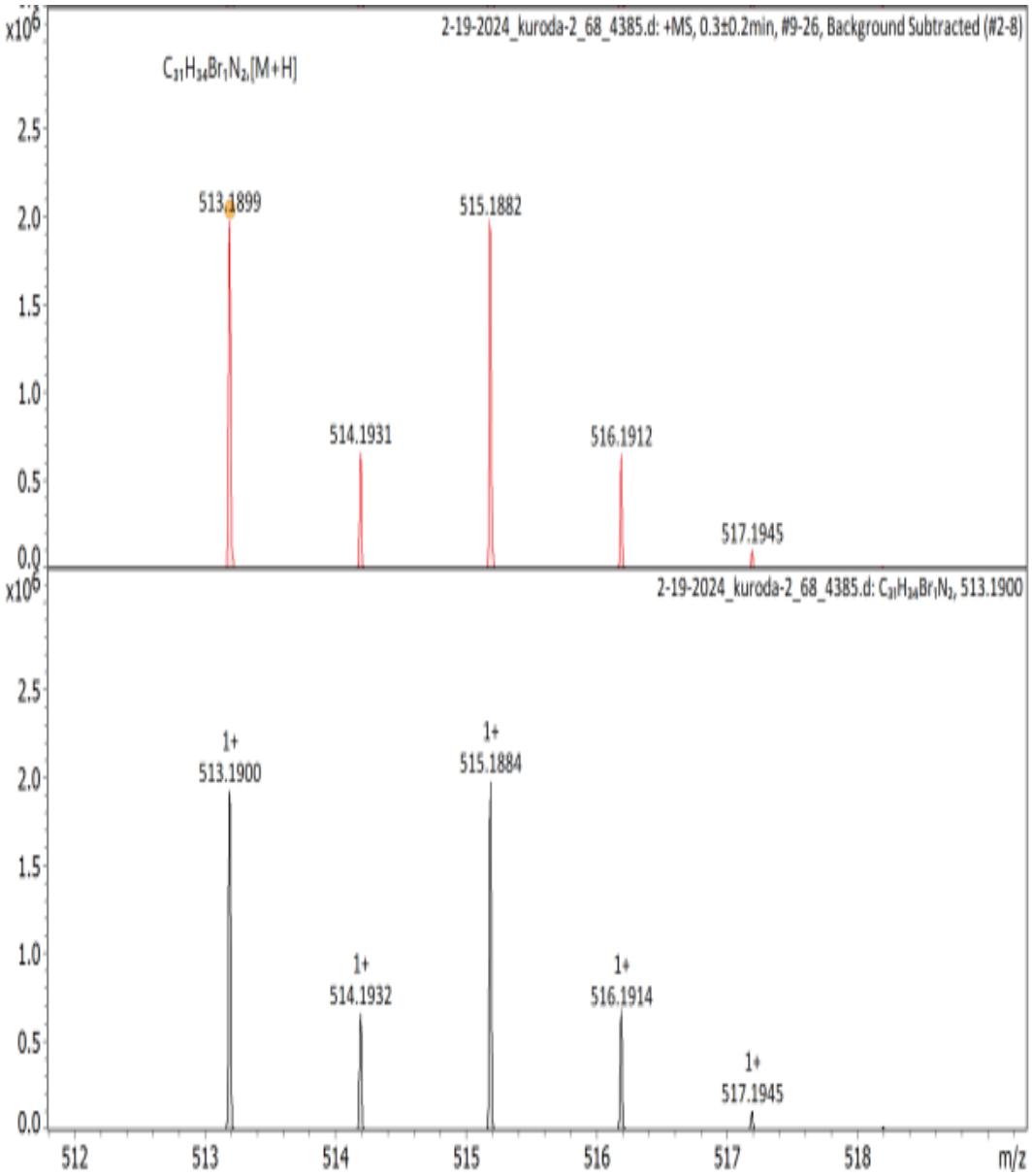


^{13}C NMR (151 MHz, $CDCl_3$)



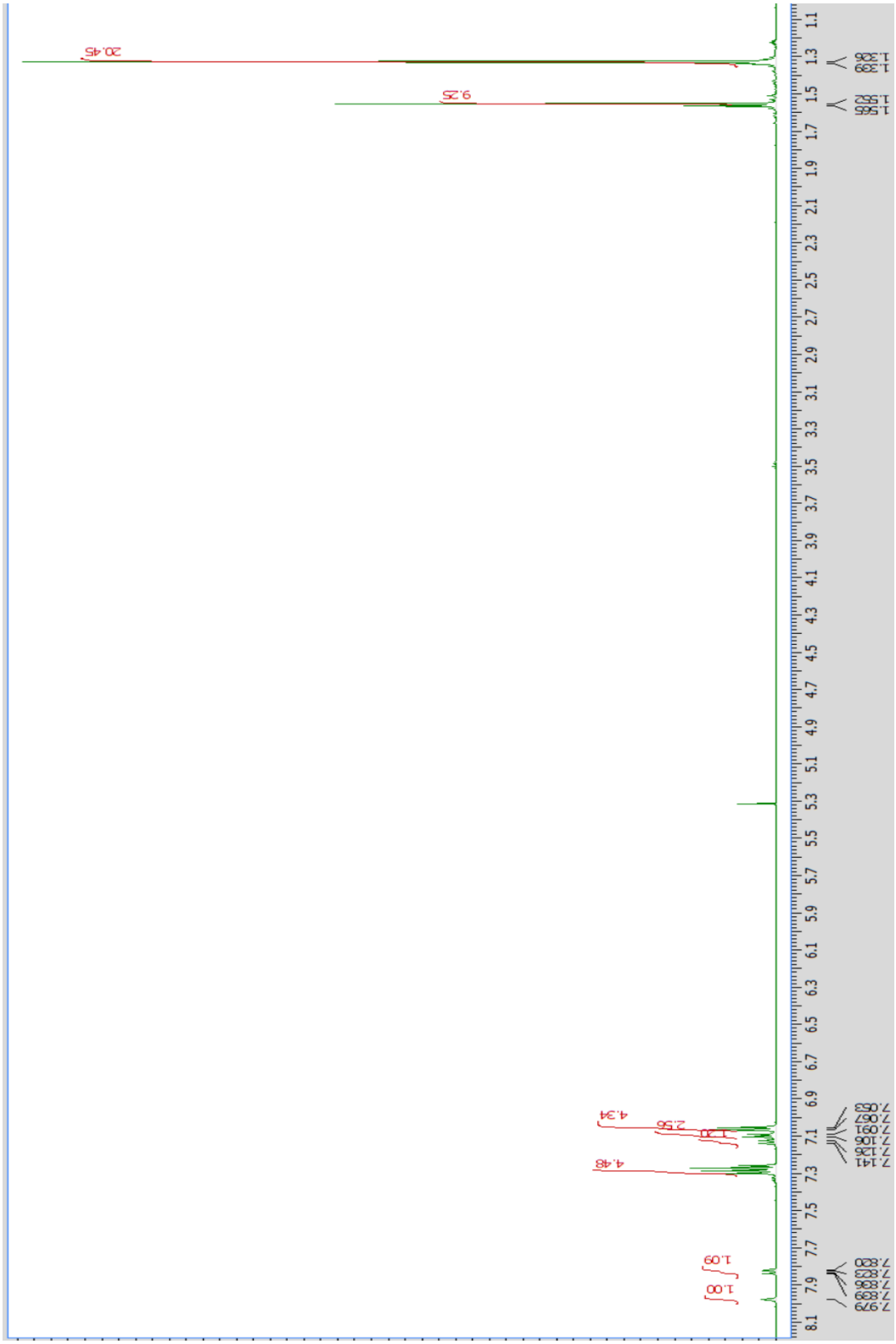
HRMS (APCI$^+$)


x10^6
2-19-2024_kuroda-2_68_4385.d: +MS, 0.3±0.2min, #9-26, Background Subtracted (#2-8)
C31H34Br1N2[M+H]
513.1899

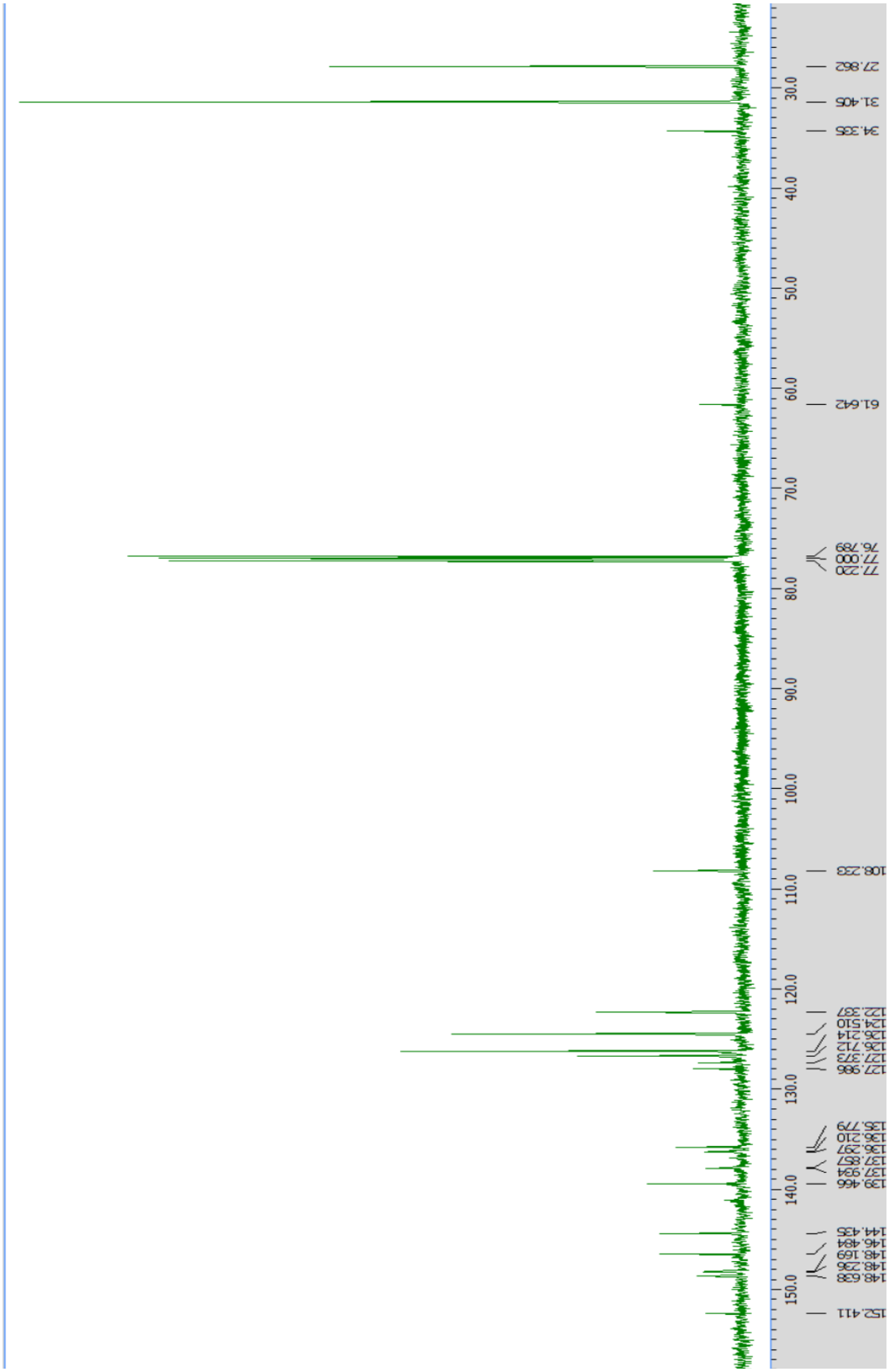

x10^6
2-19-2024_kuroda-2_68_4385.d: +MS, 0.3±0.2min, #9-26, Background Subtracted (#2-8)
C31H34Br1N2[M+H]
513.1899
514.1931
515.1882
516.1912
517.1945
x10^6
2-19-2024_kuroda-2_68_4385.d: C31H34Br1N2, 513.1900
1+
513.1900
1+
514.1932
1+
515.1884
1+
516.1914
1+
517.1945
m/z

Aminoxide complex **2**

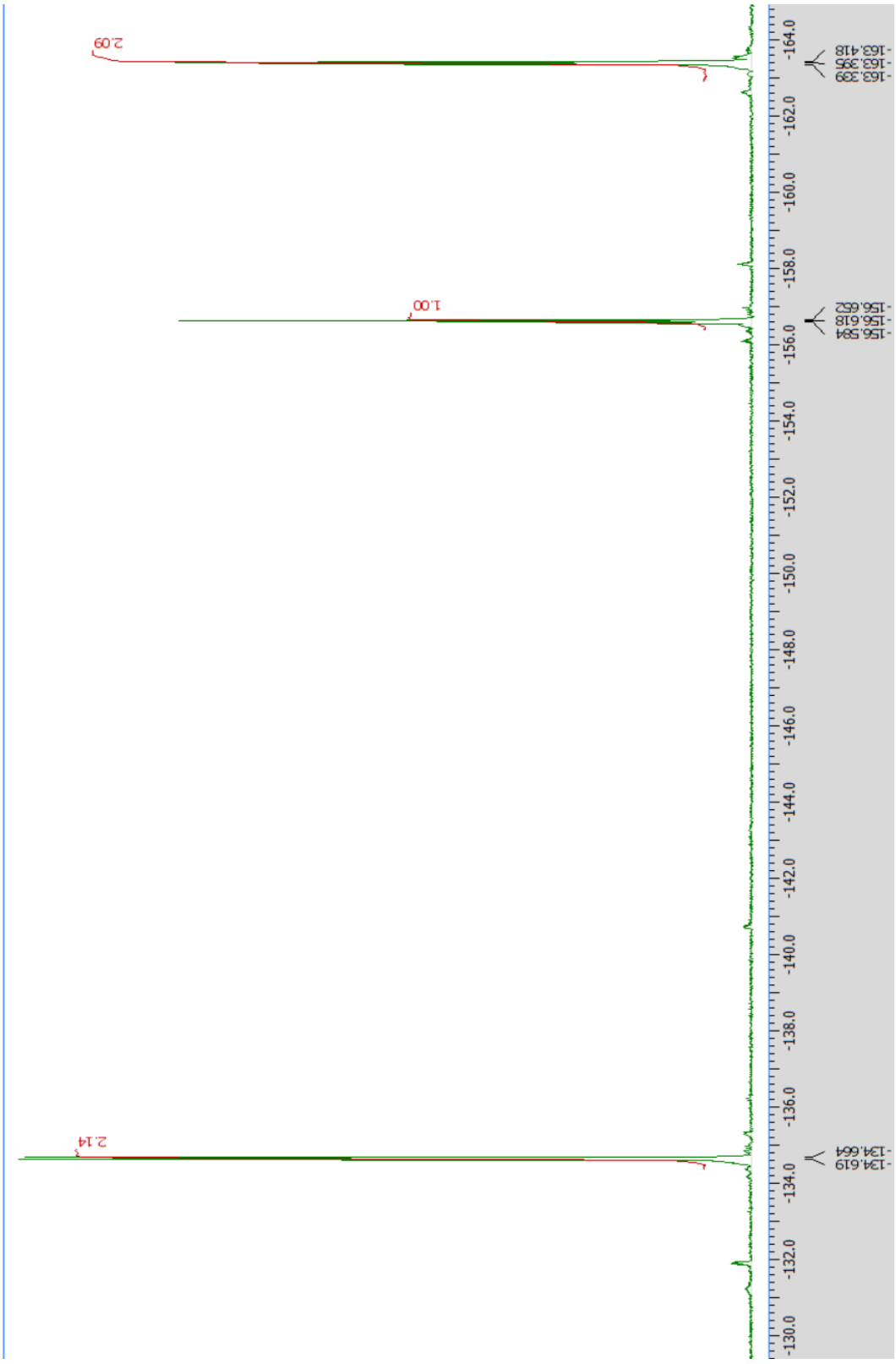
^{1}H NMR (600 MHz, $CDCl_3$)



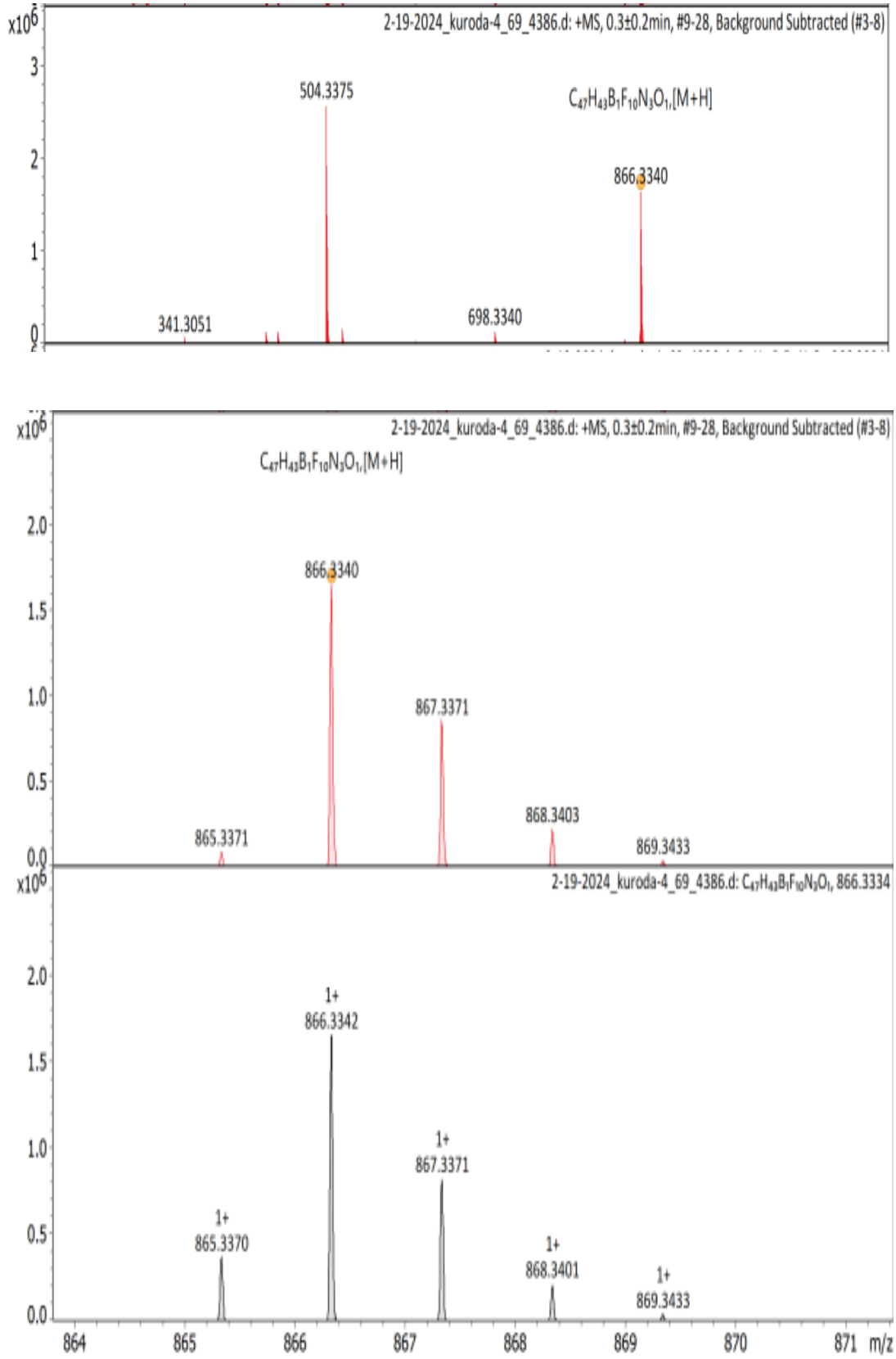
^{13}C NMR (151 MHz, $CDCl_3$)



^{19}F NMR (575 MHz, $CDCl_3$)

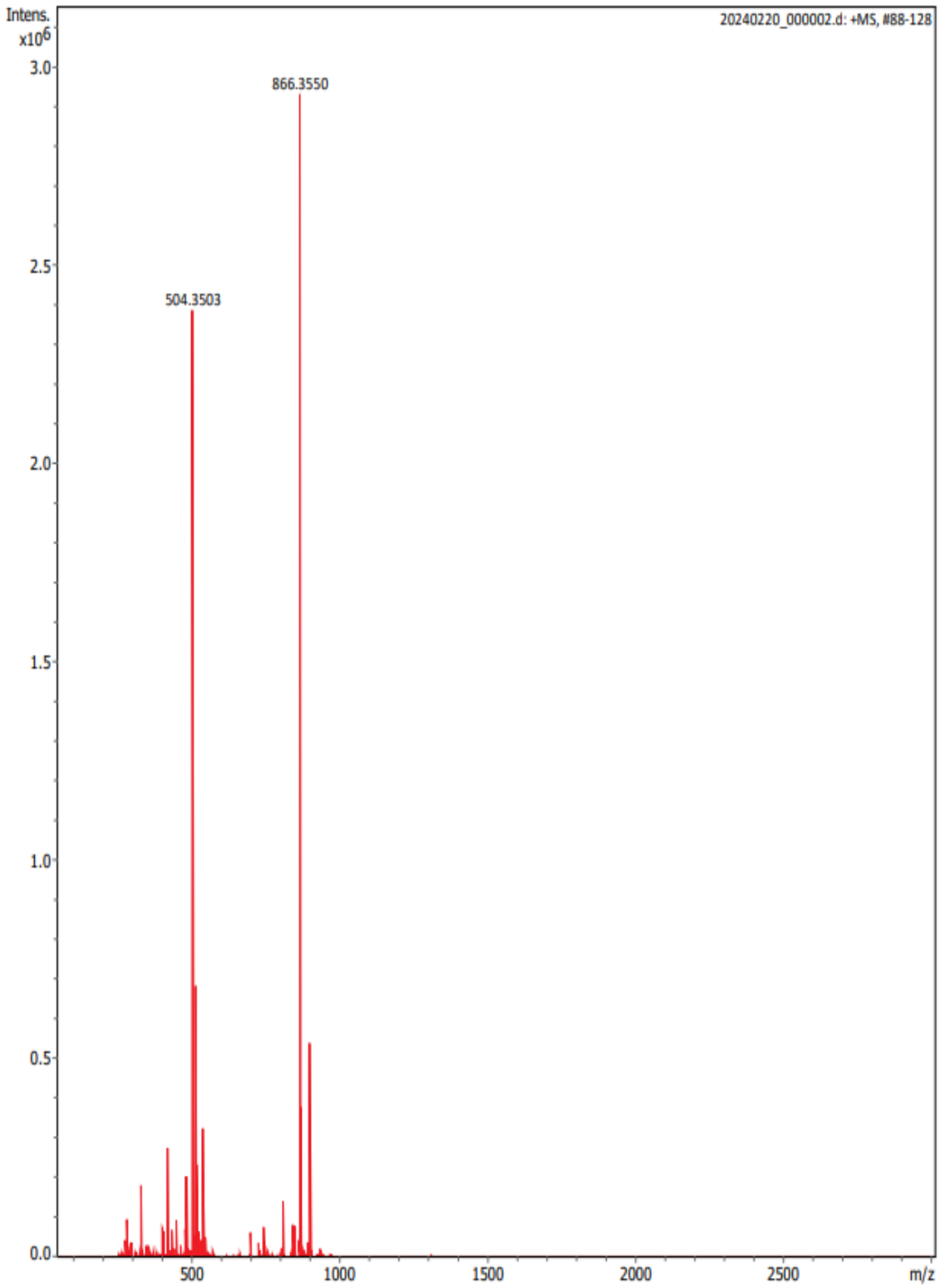


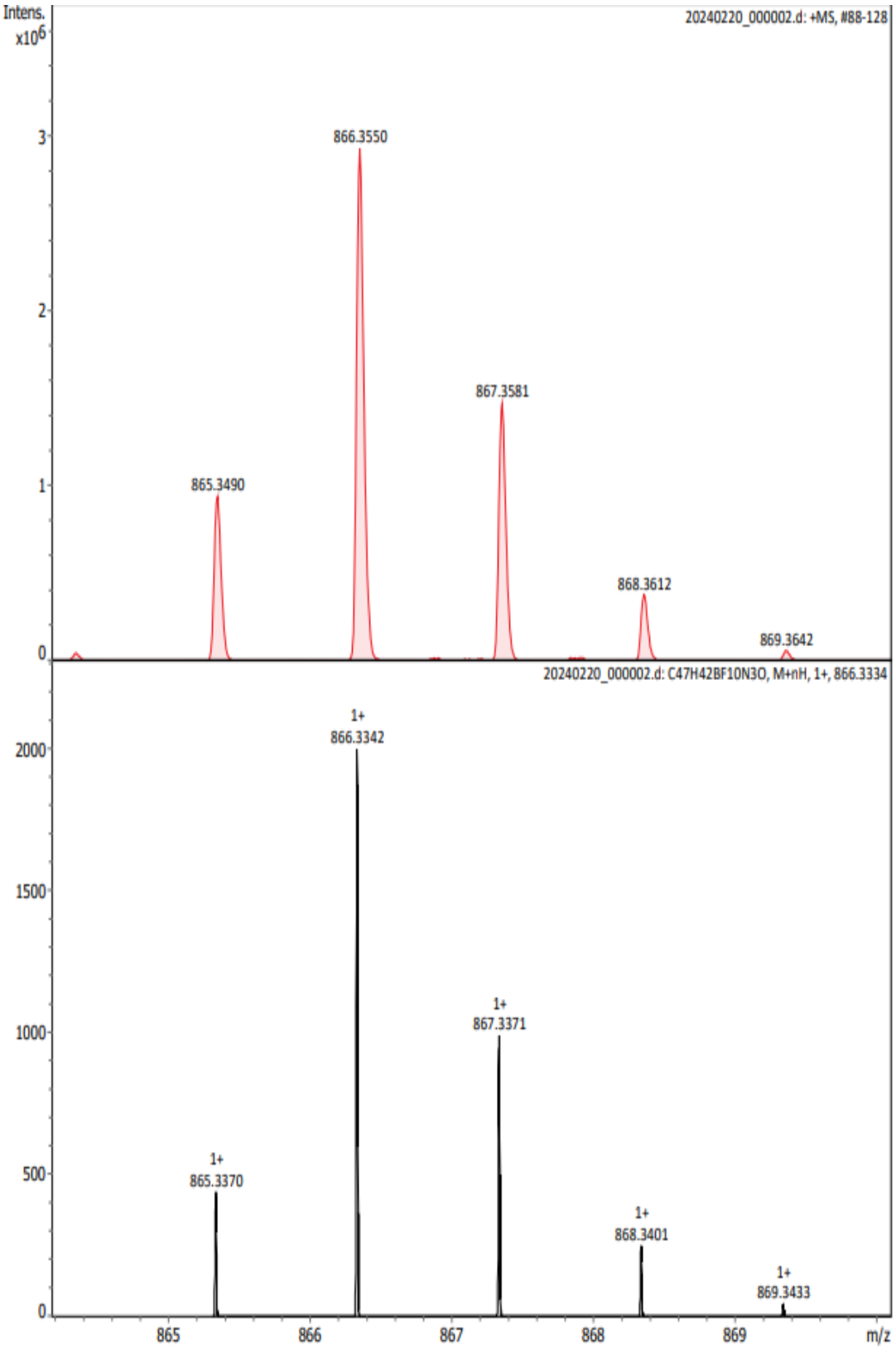
HRMS (APCI$^+$)


2-19-2024_kuroda-4_69_4386.d: +MS, 0.3±0.2min, #9-28, Background Subtracted (#3-8)
x10^6
504.3375
C47H43B1F10N3O1,[M+H]
866.3340
341.3051
698.3340
2-19-2024_kuroda-4_69_4386.d: +MS, 0.3±0.2min, #9-28, Background Subtracted (#3-8)
C47H43B1F10N3O1,[M+H]
866.3340
867.3371
865.3371
868.3403
869.3433
2-19-2024_kuroda-4_69_4386.d: C47H43B1F10N3O1, 866.3334
1+
866.3342
1+
867.3371
1+
865.3370
1+
868.3401
1+
869.3433
m/z

Aminoxyl complex **[2]SbCl$_6$**

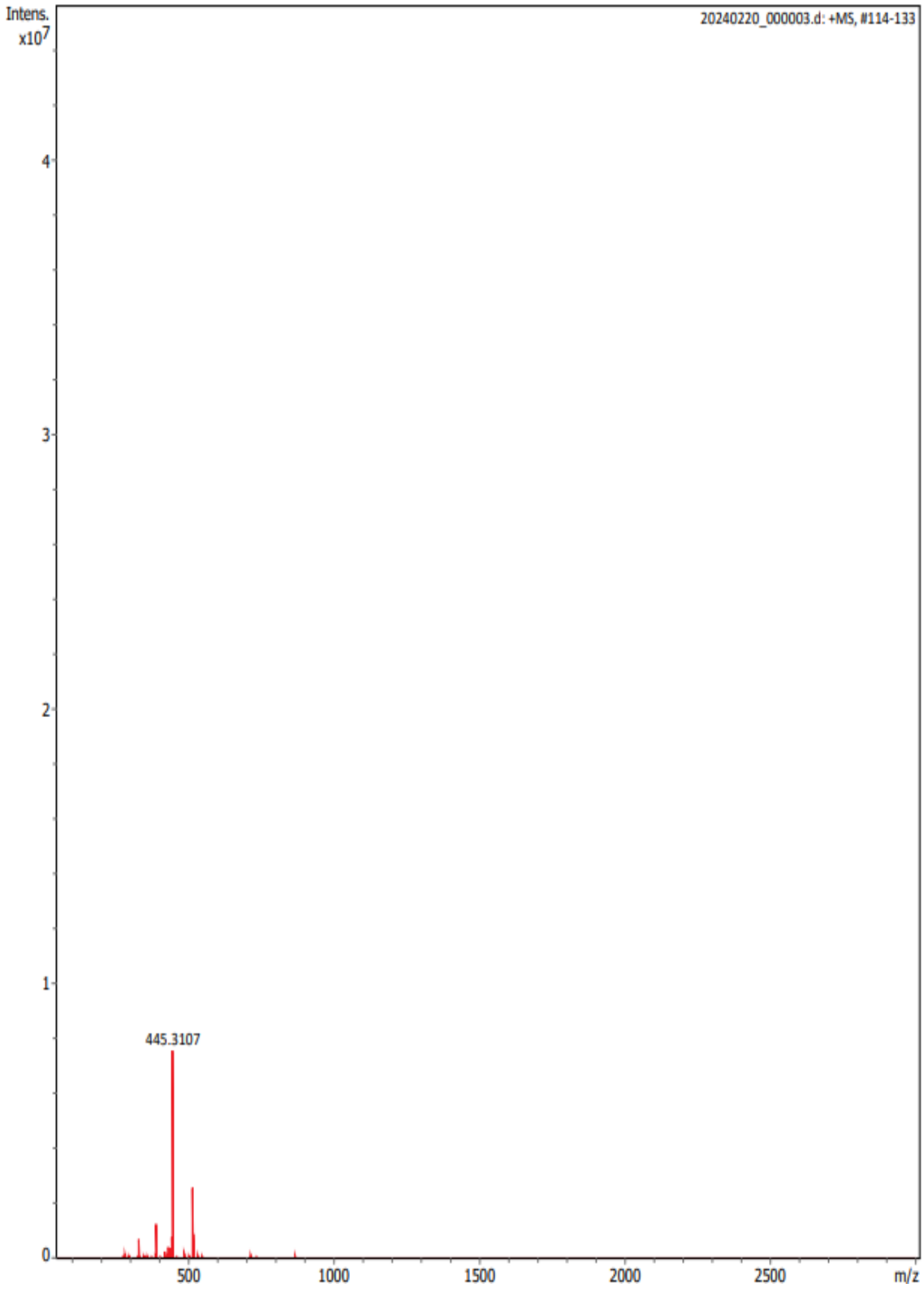
HRMS (APCI$^+$)

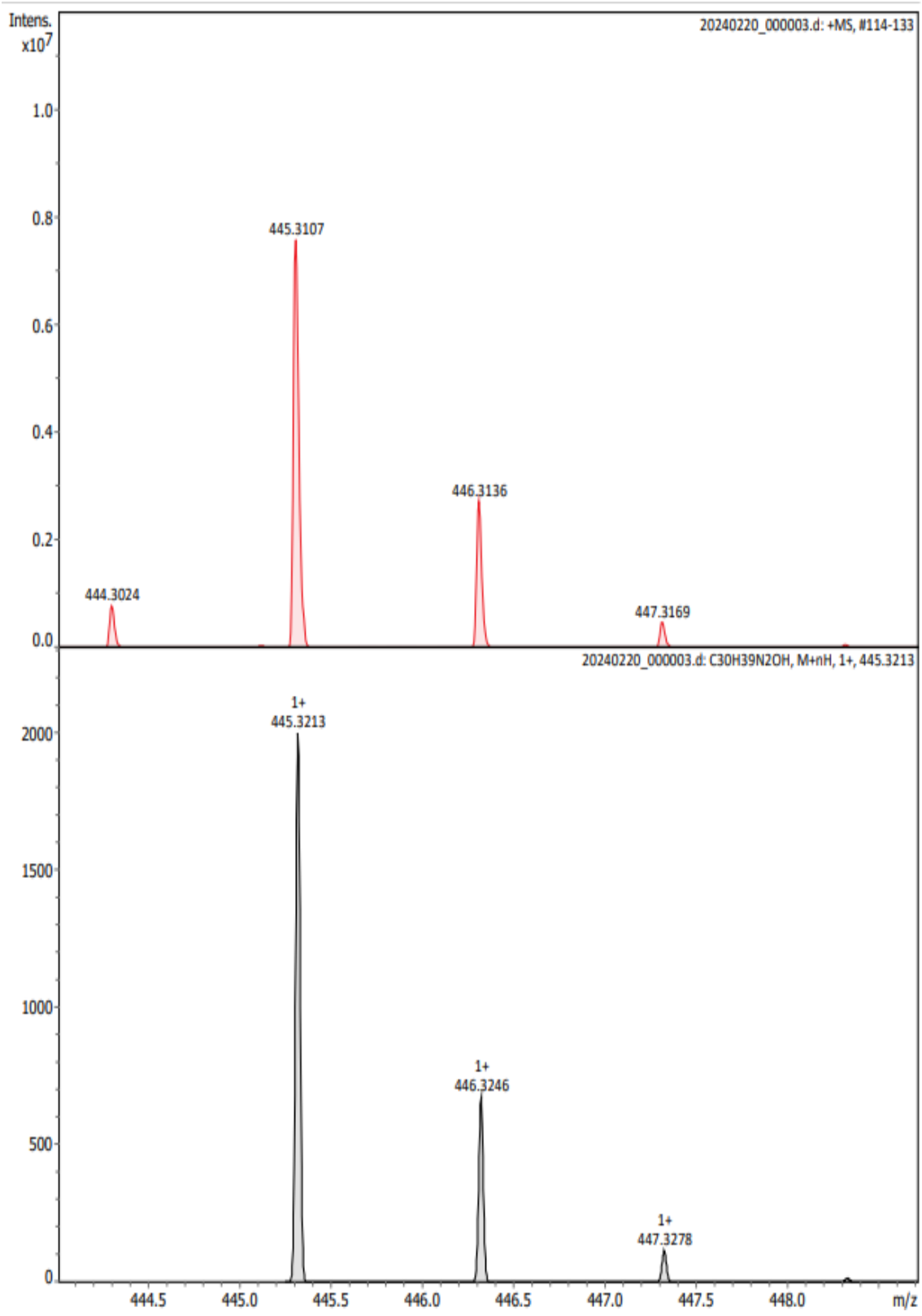



Intens.
x10^6
20240220_000002.d: +MS, #88-128
866.3550
867.3581
865.3490
868.3612
869.3642
20240220_000002.d: C47H42BF10N3O, M+nH, 1+, 866.3334
1+
866.3342
1+
867.3371
1+
865.3370
1+
868.3401
1+
869.3433
m/z

Aminoxyl radical **3**

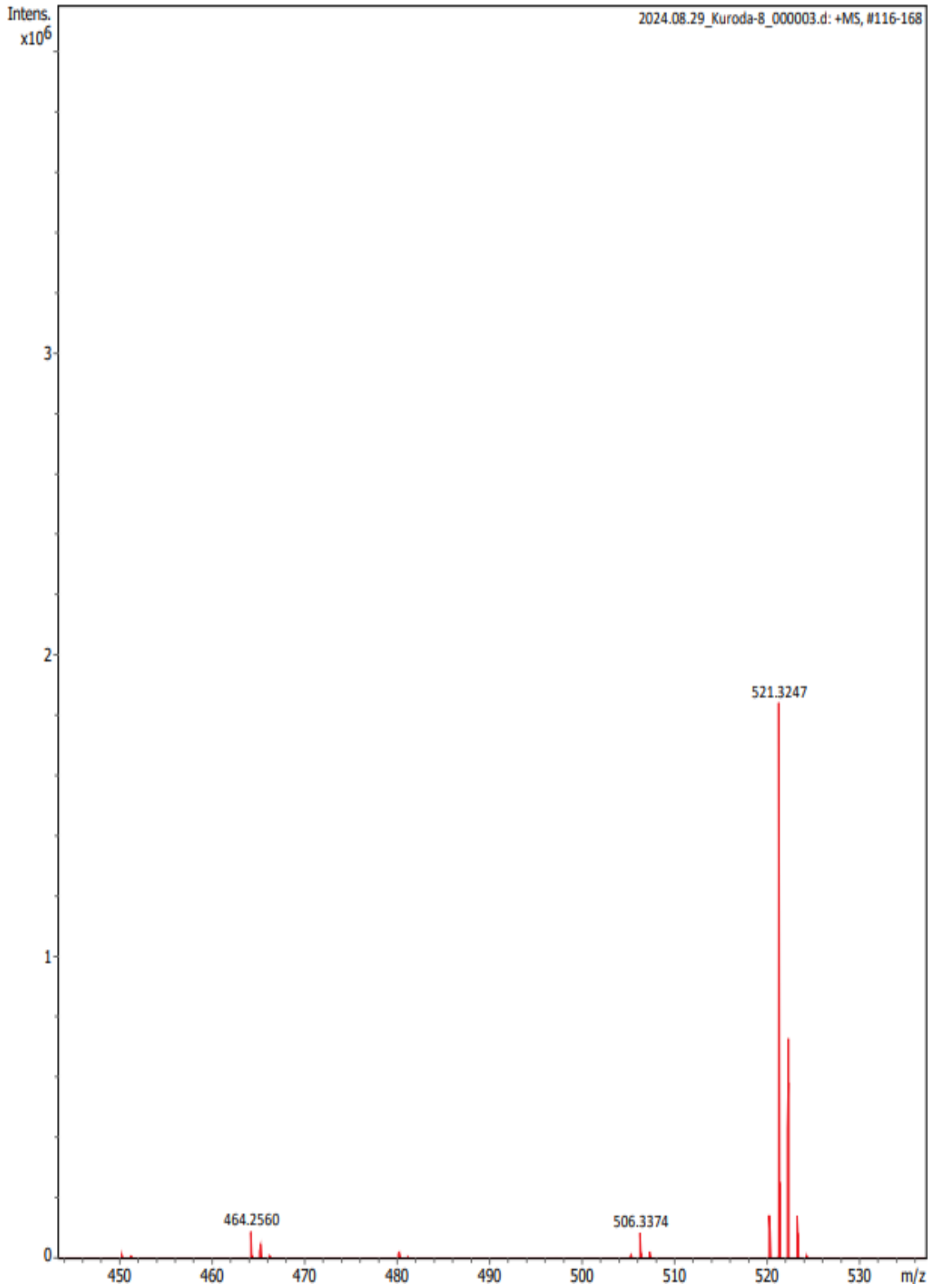
HRMS (APCI$^+$)

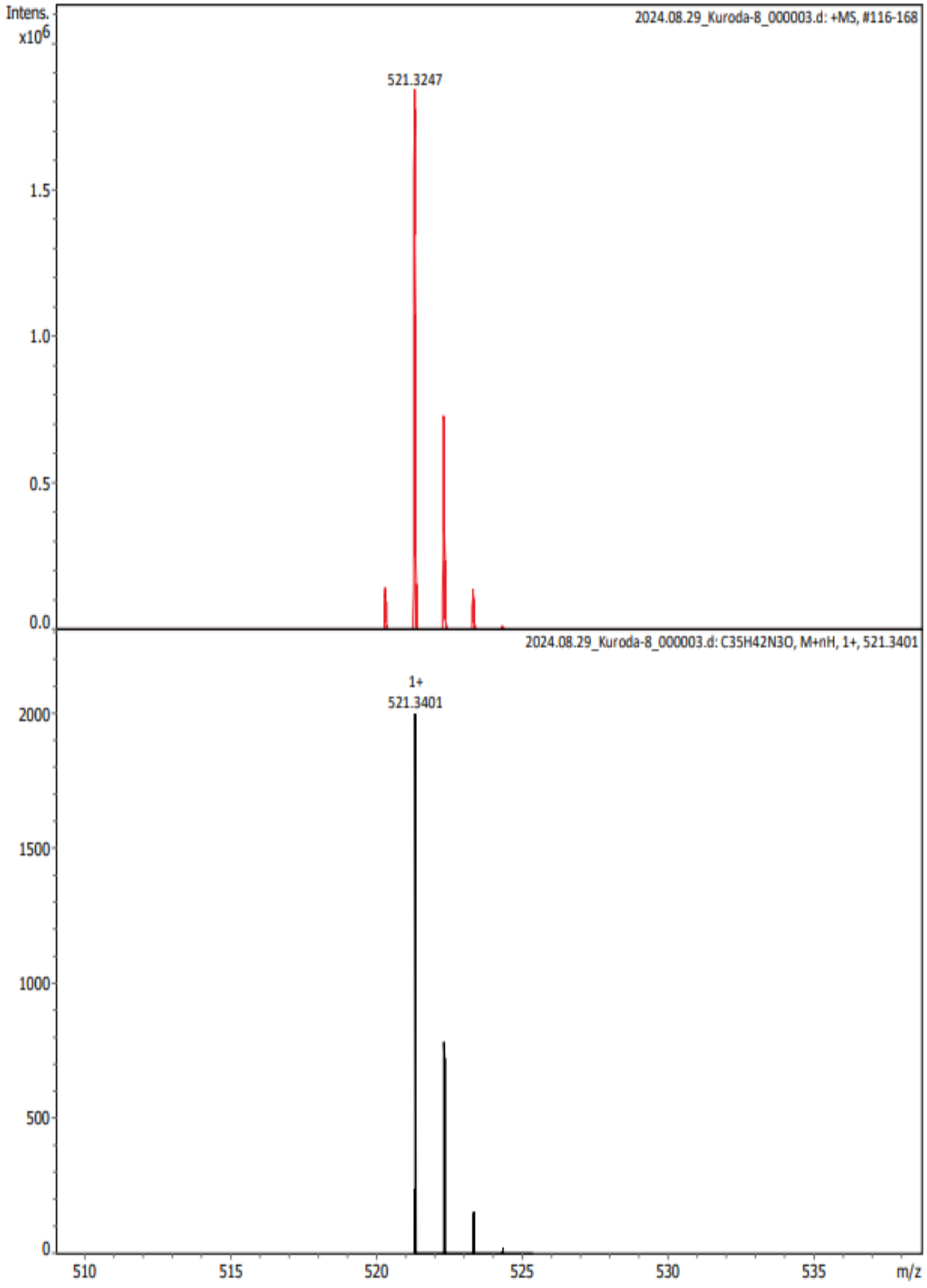



Intens.
x10^7
1.0
0.8
0.6
0.4
0.2
0.0
20240220_000003.d: +MS, #114-133
445.3107
446.3136
444.3024
447.3169
20240220_000003.d: C30H39N2OH, M+nH, 1+, 445.3213
2000
1500
1000
500
0
1+
445.3213
1+
446.3246
1+
447.3278
444.5
445.0
445.5
446.0
446.5
447.0
447.5
448.0
m/z

Aminoxyl radical **4**

HRMS (APCI$^+$)




Intens.
x10^6
2024.08.29_Kuroda-8_000003.d: +MS, #116-168
521.3247
1.5
1.0
0.5
0.0
2024.08.29_Kuroda-8_000003.d: C35H42N3O, M+nH, 1+, 521.3401
1+
521.3401
2000
1500
1000
500
0
510
515
520
525
530
535
m/z

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