Supporting Information

Unified and Green Oxidation of Amides and Aldehydes for Hofmann and Curtius Rearrangements

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Table of Contents

General Information: Reactions were carried out in oven or flame-dried glassware under a nitrogen atmosphere, unless otherwise noted. Tetrahydrofuran (THF) was freshly distilled before use from sodium using benzophenone as indicator. Dichloromethane was freshly distilled before use from calcium hydride (CaH_2). All other anhydrous solvents were dried over 3Å or 4Å molecular sieves. Solvents used in workup, extraction and column chromatography were used as received from commercial suppliers without prior purification. Reactions were magnetically stirred and monitored by thin layer chromatography (TLC, 0.25 mm) on Liangchen pre-coated silica gel plates. Flash chromatography was performed with silica gel 60 (particle size 0.040 – 0.062 mm) supplied by Liangchen. Infrared spectra were collected on a Bruker model TENSOR27 spectrophotometer. ^{1}H and ^{13}C NMR spectra were recorded on a Bruker AVIII-400 spectrometer (400 MHz for ^{1}H, 100 MHz for ^{13}C, 376 MHz for ^{19}F). Chemical shifts are reported in parts per million (ppm) as values relative to the internal chloroform (7.26 ppm for ^{1}H and 77.16 ppm for ^{13}C) or DMSO (2.50 ppm for ^{1}H and 39.52 ppm for ^{13}C). Abbreviations for signal coupling are as follows: s, singlet; d, doublet; t, triplet; q, quartet; m, multiplet; br, broad peak. Optical rotations were measured on a JASCO Perkin-Elmer model P-2000 polarimeter. High resolution mass spectra were measured at Keecloud Mass Spectrometry Service Company on either an Thermo Scientific LTQ Orbitrap XL system or a Bruker solariX System. Melting point was recorded on a Laboratory Devices model MEL-TEMP II melting point apparatus. The aldehyde substrates and (chiral) amines were purchased from Aldrich or J&K Scientific.

The References for Known Compounds

Compound	References
Urethylane anthranilate	Moriyama, K.; Ishida, K.; Togo, H. *Org. Lett*. **2012**, *14*, 946-949.
1ad	Flynn, D. L.; Caldwell, T. M.; Kaufman, M. D.; Patt, W. C.; Samarakoon, T.; Vogeti, L.; Yates, K. M. *N*-Acyl-*N*'-(Pyridin-2-yl) Ureas and Analogs Exhibiting Anti-cancer and Anti-proliferative Activities. U.S. Patent 2014/0275080, Sep. 18. 2014.
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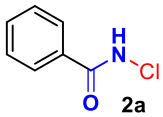
Preparation of Primary Amide Substrates

The amide substrates are known compounds. Most of them are purchased from Aldrich or J&K Scientific directly. **1ac**-**1ad** were prepared following the related procedures. Analytical data (^{1}H NMR and ^{13}C NMR) matches with the literature.

Preparation of *N*-Chlorobenzamide (2a)

To a stirred solution of benzamide (36 mg, 0.3 mmol) in the MeCN/H_2O (10/1, 1.1 mL) at 0 ℃ were added KCl (34 mg, 0.45 mmol) and Oxone (138 mg, 0.45 mmol). After completion of the addition, the resulting mixture was stirred for 10 min before warmed to room temperature and stirred for an additional 4 h. When the aromatic amide substrate was fully consumed as determined by TLC analysis, the mixture was quenched by addition of water (5 mL). The aqueous mixture was extracted with EA (3 X 10 mL). The combined organic fractions were washed with brine, dried over Na_2SO_4, and concentrated under reduced pressure. The residue was purified by flash column chromatography on silica gel using eluents (petroleum ether/ethyl acetate = 10/1) to provide the desired compound **2a** (38 mg, 81% yield) as an off-white solid.

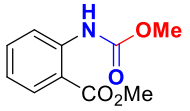


2a

^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.19 (s, br), 7.80-7.77 (m, 2H), 7.54-7.50 (m, 1H), 7.42-7.39 (m, 2H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ: 167.5, 132.6, 131.9, 128.9 (2×C), 127.8 (2×C).

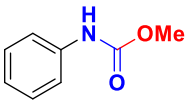
Oxone-KCl Halogenation of Primary Amides for Hofmann Rearrangement

***General Procedure A*:** To a stirred solution of aromatic amide substrate (0.3 mmol) in the MeCN/H_2O (10/1, 1.1 mL) at 0 ℃ were added KCl (34 mg, 0.45 mmol) and Oxone (138 mg, 0.45 mmol). After completion of the addition, the resulting mixture was stirred for 10 min before warmed to room temperature and stirred for an additional 2 h. When the aromatic amide substrate was fully consumed as determined by TLC analysis, methanol (0.6 mL) and NaOH (18 mg, 0.45 mmol) was added sequentially. The mixture was allowed to be stirred for 5 hrs and quenched by addition of sat. aq. Na_2SO_3 (5 mL). The volatiles (mainly methanol and MeCN) was removed under reduced pressure and the aqueous mixture was extracted with EA (3 X 10 mL). The combined organic fractions were washed with brine, dried over Na_2SO_4, and concentrated under reduced pressure. The residue was purified by flash column chromatography on silica gel using eluents (petroleum ether/ethyl acetate = 10/1) to provide the desired compound **3**.



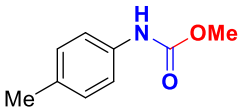
Urethylane anthranilate

Urethylane anthranilate. 24 mg, 38% yield; white solid. **^{1}H NMR** (400 MHz, $CDCl_3$) δ: 10.5 (br, 1H), 8.42 (dd, *J* = 8.4, 0.4 Hz, 1H), 8.00 (dd, *J* = 8.4, 1.6 Hz, 1H), 7.55-7.51 (m, 1H), 7.04-7.00 (m, 1H), 3.91 (s, 3H), 3.78 (s, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ: 168.6, 154.2, 141.9, 134.7, 131.0, 121.7, 118.9, 114.6, 52.4.



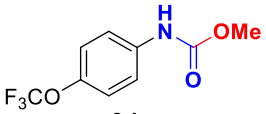
3a

3a. 40 mg, 89% yield; white solid. **^{1}H NMR** (400 MHz, $CDCl_3$) δ: 7.40-7.37 (m, 2H), 7.33-7.28 (m, 2H), 7.09-7.04 (m, 1H), 6.70 (br, s), 3.78 (s, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ: 154.2, 137.9, 129.2 (2×C), 123.6 (2×C), 118.8, 52.5.



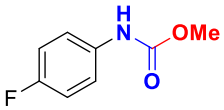
3c

3c. 41 mg, 83% yield; white solid. **^{1}H NMR** (400 MHz, $CDCl_3$) δ: 7.26 (d, *J* = 8.4 Hz, 2H), 7.11 (d, *J* = 8.4 Hz, 2H), 6.62 (br, 1H), 3.76 (s, 3H), 2.30 (s, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ: 154.3, 135.3, 133.2, 129.7 (2×C), 118.9 (2×C), 52.4, 20.9.



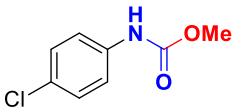
3d

3d. 59 mg, 84% yield; white solid. **^{1}H NMR** (400 MHz, $CDCl_3$) δ: 7.40 (d, *J* = 8.8 Hz, 2H), 7.15 (d, *J* = 8.8 Hz, 2H), 6.89 (br, 1H), 3.77 (s, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ: 154.2, 144.9, 136.7, 122.0 (2×C), 120.6 (q, *J* = 255 Hz), 119.9 (2×C), 52.6.



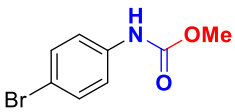
3e

3e. 44 mg, 87% yield; white solid. **^{1}H NMR** (400 MHz, $CDCl_3$) δ: 7.36-7.30 (m, 2H), 7.02-6.96 (m, 2H), 6.78 (br, 1H), 3.76 (s, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ: 159.1 (d, *J* = 241 Hz), 154.4, 133.9, 120.6 (2×C), 115.8 (d, *J* = 23 Hz, 2×C), 52.5.



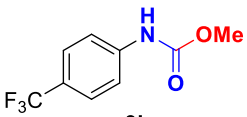
3f

3f. 46 mg, 83% yield; white solid. **^{1}H NMR** (400 MHz, $CDCl_3$) δ: 7.36-7.33 (m, 2H), 7.28-7.25 (m, 2H), 6.77 (br, 1H), 3.78 (s, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ: 154.1, 136.6, 129.1 (2×C), 128.5, 120.0 (2×C), 52.6.



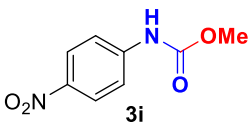
3g

3g. 58 mg, 84% yield; white solid. **^{1}H NMR** (400 MHz, $CDCl_3$) δ: 7.40 (d, *J* = 8.8 Hz, 2H), 7.28 (d, *J* = 8.8 Hz, 2H), 6.79 (br, 1H), 3.76 (s, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ: 154.0, 137.1, 132.1 (2×C), 120.3 (2×C), 116.0, 52.6.



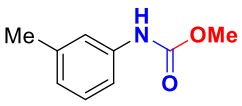
3h

3h. 49 mg, 75% yield; white solid. **^{1}H NMR** (400 MHz, $CDCl_3$) δ: 7.55 (d, *J* = 8.8 Hz, 2H), 7.50 (d, *J* = 8.8 Hz, 2H), 6.93 (br, 1H), 3.79 (s, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ: 153.8, 141.1, 126.5 (q, *J* = 3.4 Hz, 2×C), 125.3 (q, *J* = 33 Hz), 124.3 (q, *J* = 270 Hz), 118.2 (2×C), 52.8.



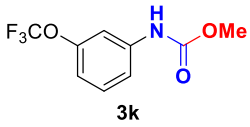
3i

3i. 34 mg, 58% yield; yellowish solid. **^{1}H NMR** (400 MHz, $CDCl_3$) δ: 8.20 (d, *J* = 9.2 Hz, 2H), 7.56 (d, *J* = 9.2 Hz, 2H), 7.04 (br, 1H), 3.82 (s, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ: 153.4, 144.0, 143.1, 125.4 (2×C), 117.8 (2×C), 53.1.

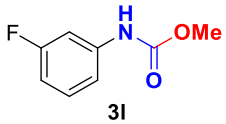


3j

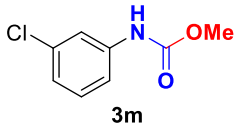
3j. 40 mg, 81% yield; white solid. **^{1}H NMR** (400 MHz, $CDCl_3$) δ: 7.23 (s, 1H), 7.21-7.17 (m, 2H), 6.89-6.87 (m, 1H), 6.70 (br, 1H), 3.77 (s, 3H), 2.33 (s, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ: 154.2, 139.1, 137.8, 129.0, 124.4, 119.4, 115.9, 52.4, 21.6.



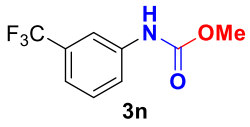
3k. 56.5 mg, 80% yield; white solid. m.p. = 79-81 ℃. **^{1}H NMR** (400 MHz, $CDCl_3$) δ: 7.43 (s, 1H), 7.31-7.22 (m, 2H), 6.97 (br, 1H), 6.92-6.89 (m, 1H), 3.78 (s, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ: 154.0, 149.8 (q, *J* = 1.9 Hz), 139.5, 130.2, 120.5 (q, *J* = 256 Hz), 116.8, 115.6, 111.5, 52.7. **^{19}F NMR** (376 MHz, $CDCl_3$) δ: -57.8 (3×F). **HRMS** (ESI) *m*/*z* calculated for $C_9H_9O_3NF_3^+$ $[M+H]^+$ 236.0529, found 236.0530.



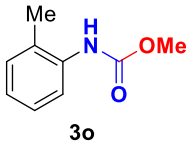
3l. 42 mg, 83% yield; white solid. **^{1}H NMR** (400 MHz, $CDCl_3$) δ: 7.35 (d, *J* = 10.8 Hz, 1H), 7.25 (td, *J* = 8.4, 6.8 Hz, 1H), 7.05 (d, *J* = 8.4 Hz, 1H), 6.86 (br, 1H), 6.78 (tdd, *J* = 8.4, 2.4, 0.8 Hz, 1H), 3.80 (s, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ: 163.3 (d, *J* = 243 Hz), 153.9, 139.6 (d, *J* = 11 Hz), 130.3 (d, *J* = 9.5 Hz), 114.0, 110.2 (d, *J* = 21 Hz), 106.2 (d, *J* = 26 Hz), 52.6.



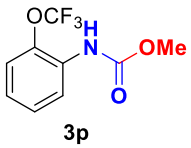
3m. 42 mg, 76% yield; white solid. **^{1}H NMR** (400 MHz, $CDCl_3$) δ: 7.50 (s, 1H), 7.22-7.18 (m, 2H), 7.04-7.01 (m, 1H), 6.81 (br, 1H), 3.77 (s, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ: 153.9, 139.2, 134.8, 130.1, 123.6, 118.8, 116.7, 52.6.



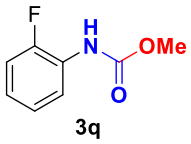
3n. 54.5 mg, 83% yield; white solid. **^{1}H NMR** (400 MHz, $CDCl_3$) δ: 7.71 (s, 1H), 7.56 (d, *J* = 8.0 Hz, 1H), 7.40 (t, *J* = 8.0 Hz, 1H), 7.31 (d, *J* = 8.0 Hz, 1H), 6.96 (br, 1H), 3.79 (s, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ: 154.1, 138.6, 131.6 (q, *J* = 32 Hz), 129.7, 124.0 (q, *J* = 271 Hz), 121.7, 120.1 (q, *J* = 3.7 Hz), 115.4, 52.7.



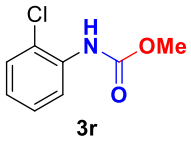
3o. 41 mg, 82% yield; white solid. **^{1}H NMR** (400 MHz, $CDCl_3$) δ: 7.76 (br, 1H), 7.21 (t, *J* = 8.0 Hz, 1H), 7.16 (d, *J* = 7.2 Hz, 1H), 7.04 (t, *J* = 7.2 Hz, 1H), 6.46 (s, 1H), 3.78 (s, 3H), 2.25 (s, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ: 154.5, 135.9, 130.5 (2×C), 127.0, 124.3, 121.3, 52.5, 17.7.



3p. 54 mg, 76% yield; white solid. **^{1}H NMR** (400 MHz, $CDCl_3$) δ: 8.19 (d, *J* = 7.6 Hz, 1H), 7.30-7.22 (m, 2H), 7.07-7.03 (m, 1H), 6.96 (br, 1H), 3.80 (s, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ: 153.7, 137.8, 130.8, 127.7, 123.4, 120.7 (q, *J* = 258 Hz), 120.5 (q, *J* = 1.0 Hz), 120.4, 52.7.

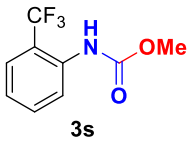


3q. 39.5 mg, 78% yield; colorless oil. **^{1}H NMR** (400 MHz, $CDCl_3$) δ: 8.13-8.03 (m, 1H), 7.14-6.96 (m, 3H), 6.89 (br, 1H), 3.79(s, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ: 153.8, 152.2 (d, *J* = 241 Hz), 126.5 (d, *J* = 10 Hz), 124.7 (d, *J* = 3.7 Hz), 123.5 (d, *J* = 7.2 Hz), 120.3, 114.9 (d, *J* = 18.9 Hz), 52.7. **^{19}F NMR** (376 MHz, $CDCl_3$) δ: -129.9.



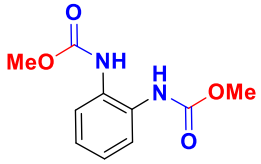
3r. 44.5 mg, 80% yield; colorless oil. **^{1}H NMR** (400 MHz, $CDCl_3$) δ: 8.18 (d, *J* = 8.0 Hz, 1H), 7.36 (dd, *J* = 8.0, 1.6 Hz, 1H), 7.29 (td, *J* = 8.0, 1.2 Hz, 1H), 7.18 (br, 1H), 7.01 (td, *J* = 8.0, 1.6 Hz, 1H), 3.82 (s,

3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ: 153.8, 134.8, 129.2, 127.9, 123.8, 122.2, 120.0, 52.7.



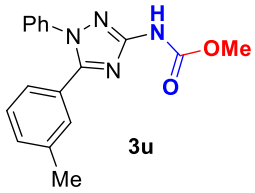
3s

3s. 51 mg, 77% yield; white solid. **^{1}H NMR** (400 MHz, $CDCl_3$) δ: 8.12 (d, *J* = 8.0 Hz, 1H), 7.58-7.82 (m, 2H), 7.17 (t, *J* = 8.0 Hz, 1H), 6.95 (br, 1H), 3.80 (s, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ: 154.0, 135.7, 133.1, 126.2 (q, *J* = 5.3 Hz), 124.2 (q, *J* = 271 Hz), 123.6, 122.6, 119.3 (q, *J* = 28.5 Hz), 52.8. **^{19}F NMR** (376 MHz, $CDCl_3$) δ: -60.8 (3×F).



3t

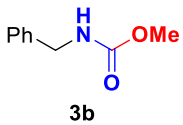
3t. 42 mg, 63% yield; white solid. **^{1}H NMR** (400 MHz, $CDCl_3$) δ: 10.51 (br, 1H), 8.42 (dd, *J* = 8.4, 0.8 Hz, 1H), 8.00 (dd, *J* = 8.4, 1.6 Hz, 1H), 7.53 (ddd, *J* = 8.4, 7.6, 1.6 Hz, 1H), 7.04-7.00 (m, 1H), 3.91 (s, 3H), 3.78 (s, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ: 168.6, 154.2, 141.9, 134.7, 131.0, 121.7, 118.9, 114.6, 52.4 (2×C).



3u

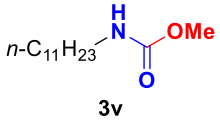
3u. 49 mg, 53% yield; colorless oil. **^{1}H NMR** (400 MHz, $CDCl_3$) δ: 8.69 (br, 1H), 7.40-7.33(m, 6H), 7.21-7.12 (m, 3H), 3.68 (s, 3H), 2.29 (s, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ: 156.2, 153.4, 153.2, 138.5, 138.0, 131.1, 129.8, 129.3 (2×C), 128.9, 128.3, 127.2, 126.1, 128.8 (2×C), 52.8, 21.4. **HRMS** (ESI) *m/z* calculated for $C_{17}H_{17}O_2N_4^+$ $[M+H]^+$ 309.1346, found 309.1344.

***General Procedure B*:** To a stirred solution of aliphatic amide substrate (0.3 mmol) in the MeCN/H_2O (10/1, 1.1 mL) at 0 ℃ were added KCl (34 mg, 0.45 mmol) and Oxone (138 mg, 0.45 mmol). After completion of the addition, the resulting mixture was stirred for 10 min before warmed to room temperature and stirred for an additional 2 h. When the aromatic amide substrate was fully consumed as determined by TLC analysis, methanol (1.5 mL) and Cs_2CO_3 (147 mg, 0.45 mmol) was added sequentially. The mixture was allowed to be stirred for 5 hrs and quenched by addition of sat. aq. Na_2SO_3 (5 mL). Voletiles (mainly methanol and MeCN) was removed under reduced pressure and the aqueous mixture was extracted with EA (3 X 10 mL). The combined organic fractions were washed with brine, dried over Na_2SO_4, and concentrated under reduced pressure. The residue was purified by flash column chromatography on silica gel using eluents (petroleum ether/ethyl acetate = 10/1) to provide the desired compound **3**.



3b

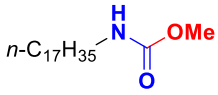
3b. 44 mg, 89% yield; white solid. **^{1}H NMR** (400 MHz, $CDCl_3$) δ: 7.37-7.27 (m, 5H), 5.18 (br, 1H), 4.38 (d, *J* = 6.0 Hz, 2H), 3.71 (s, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ: 157.2, 138.6, 128.7 (2×C), 127.6, 127.5 (2×C), 52.3, 45.1.



3v

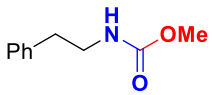
3v. 60 mg, 87% yield; white solid. m.p. = 49-51 ℃. **^{1}H NMR** (400 MHz, $CDCl_3$) δ: 4.71 (br, 1H), 3.64 (s, 3H), 3.15 (q, *J* = 6.8 Hz, 2H), 1.50-1.43 (m, 2H), 1.30-1.23 (m, 16H), 0.86 (t, *J* = 6.8 Hz, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ: 157.2, 52.1, 41.2, 32.0, 30.1, 29.7 (2×C),

29.6, 29.5, 29.4, 26.8, 22.8, 14.2. **HRMS** (ESI) *m/z* calculated for $C_{13}H_{28}O_2N^+$ $[M+H]^+$ 230.2115, found 230.2116.



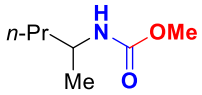
3w

3w. 83 mg, 88% yield; white solid. m.p. = 51-53 ℃. **^{1}H NMR** (400 MHz, $CDCl_3$) δ: 4.71 (br, 1H), 3.64 (s, 3H), 3.14 (q, *J* = 6.8 Hz, 2H), 1.48-1.23 (m, 30H), 0.86 (t, *J* = 6.8 Hz, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ: 157.2, 52.1, 41.2, 32.0, 30.1, 29.81 (2×C), 29.79 (2×C), 29.78 (2×C), 29.76, 29.7, 29.6, 29.5, 29.4, 26.8, 22.8, 14.2. **HRMS** (ESI) *m/z* calculated for $C_{19}H_{40}O_2N^+$ $[M+H]^+$ 314.3054, found 314.3049.



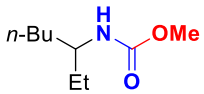
3x

3x. 46 mg, 86% yield; colorless oil. **^{1}H NMR** (400 MHz, $CDCl_3$) δ: 7.33-7.29 (m, 2H), 7.25-7.18 (m, 3H), 4.71 (br, 1H), 3.65 (s, 3H), 3.42 (q, *J* = 6.8 Hz, 2H), 2.81 (t, *J* = 6.8 Hz, 2H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ: 157.1, 138.9, 128.9 (2×C), 128.8 (2×C), 126.6, 52.2, 42.3, 36.3.



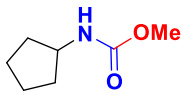
3y

3y. 34.5 mg, 79% yield; colorless oil. **^{1}H NMR** (400 MHz, $CDCl_3$) δ: 4.49 (br, 1H), 3.70-3.64 (m, 1H), 3.63 (s, 3H), 1.41-1.27 (m, 4H), 1.11 (d, *J* = 6.4 Hz, 3H), 0.89 (t, *J* = 7.2 Hz, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ: 156.6, 51.9, 46.9, 39.5, 21.4, 19.3, 14.0. **HRMS** (ESI) *m/z* calculated for $C_7H_{16}O_2N^+$ $[M+H]^+$ 146.1176, found 146.1177.



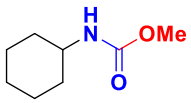
3z

3z. 46 mg, 88% yield; colorless oil. **^{1}H NMR** (400 MHz, $CDCl_3$) δ: 4.45 (br, 1H), 3.63 (s, 3H), 3.54-3.47 (m, 1H), 1.54-1.24 (m, 8H), 0.89-0.85 (m, 6H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ: 157.0, 52.7, 52.0, 34.7, 28.2, 28.1, 22.7, 14.1, 10.2.



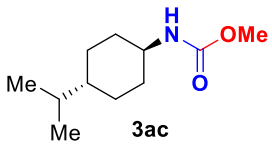
3aa

3aa. 33 mg, 76% yield; colorless oil. **^{1}H NMR** (400 MHz, $CDCl_3$) δ: 4.66 (br, 1H), 3.97-3.95 (m, 1H), 3.63 (s, 3H), 1.97-1.89 (m, 2H), 1.69-1.52 (m, 4H), 1.41-1.33 (m, 2H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ: 156.7, 52.9, 52.0, 33.3 (2×C), 23.6 (2×C).



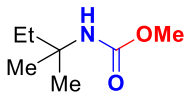
3ab

3ab. 37 mg, 78% yield; white solid. **^{1}H NMR** (400 MHz, $CDCl_3$) δ: 4.64 (br, 1H), 3.62 (s, 3H), 3.49-3.41 (m, 1H), 1.92-1.88 (m, 2H), 1.70-1.64 (m, 2H), 1.59-1.54 (m, 1H), 1.36-1.23 (m, 2H), 1.18-1.05 (m, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ: 156.3, 51.9, 49.9, 33.5, 25.6 (2×C), 24.9 (2×C).



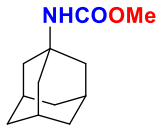
3ac

3ac. 50 mg, 84% yield; colorless oil. **^{1}H NMR** (400 MHz, $CDCl_3$) δ: 4.57 (br, 1H), 3.62 (s, 3H), 3.43-3.23 (m, 1H), 2.03-1.94 (m, 2H), 1.74-1.67 (m, 2H), 1.46-1.34 (m, 1H), 1.10-0.95 (m, 5H), 0.83 (d, *J* = 6.8 Hz, 6H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ: 156.4, 51.9, 50.5, 43.3, 33.7, 32.6 (2×C), 28.5 (2×C), 20.0 (2×C).



3ad

3ad. 24.5 mg, 56% yield; colorless oil. **^{1}H NMR** (400 MHz, $CDCl_3$) δ: 4.53 (br, 1H), 3.60 (s, 3H), 1.65 (q, *J* = 7.6 Hz, 2H), 1.26 (s, 6H), 0.85 (t, *J* = 7.6 Hz, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ: 155.5, 53.0, 51.5, 33.2, 29.8, 26.7, 8.5. **HRMS** (ESI) *m/z* calculated for $C_7H_{16}O_2N^+$ $[M+H]^+$ 146.1176, found 146.1176.

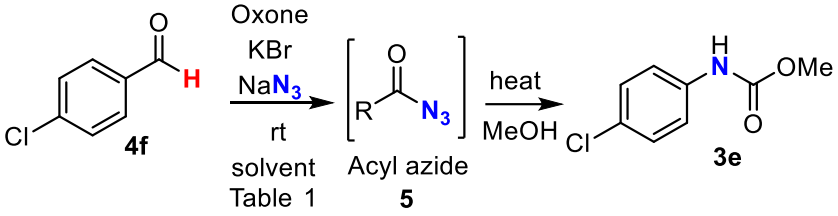


3ae

3ae. 45 mg, 72% yield; white solid. **^{1}H NMR** (400 MHz, $CDCl_3$) δ: 4.55 (br, 1H), 3.58 (s, 3H), 2.05 (s, 3H), 1.90 (d, *J* = 1.6 Hz, 6H), 1.64 (t, *J* = 3.2 Hz, 6H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ: 155.1, 51.4, 50.7, 41.9 (3×C), 36.4 (3×C), 29.5 (3×C).

The Screening of Solvents

Many solvents were applied to the oxidation of aldehydes for Curtius rearrangements. The results were summarized in Table 1. We found that $PhCF_3$ outperformed other solvents (except CCl_4) to secure the best yield. Octafluorotoluene is much more expensive than $PhCF_3$.



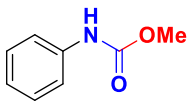
Entry	Solvent	Yield (%)
1	$CHCl_3$	68
2	CH_2Cl_2	57
3	CCl_4	76
4	pentane	50
5	hexane	46
6	heptane	33
7	octane	27
8	nonane	26
9	decane	20
10	petroleum ether (60-90)	26
11	naphthane	38
12	cyclohexane	45
13	Methylcyclohexane	46
14	Ethylcyclohexane	45
15	toluene	53
16	cumene	21
17	DMF	0
18	THF	0

19	MeCN	0
20	ethyl acetate	0
21	ether	0
22	1,4-dioxane	0
23	anisole	0
24	isopropanol	0
25	*t*BuOH	0
26	methanol	0
27	ethanol	**0**
28	CF_3CH_2OH	**38**
29	perfluorohexane	58
30	perfluorobenzene	65
31	$PhCF_3$	71
32	octafluorotoluene	71

[a]Condition: to a solution of **4f** (0.3 mmol) in the solvent (3 mL, ACS grade and used as received) at rt were added KBr (0.45 mmol), Oxone (0.45 mmol) and NaN_3 (0.6 mmol). After stirring for 24 hrs, the reaction mixture was filtered and the filtrate was concentrated. The residue was dissolved in dry toluene/MeOH [10:1 (v/v), 3.3 mL] and stirred at 100 ℃ for 2 hrs. The reaction progress was monitored by TLC.

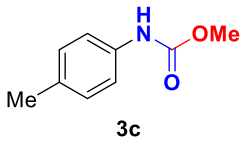
Oxone-KBr Mediated Azidation of Aldehydes for Curtius Rearrangement

***General Procedure C*:** To a stirred solution of aromatic aldehyde substrate (0.3 mmol) in the $PhCF_3$ (analytical grade, 3.0 mL) or CCl_4 (analytical grade, 3.0 mL) at 0 ℃ were added KBr (54 mg, 0.45 mmol) and Oxone (166 mg, 0.54 mmol) and stirred for 5 min. Then sodium azide (49 mg, 0.75 mmol or 43 mg, 0.66 mmol) was added. After the completion of the addition, the resulting mixture was stirred for 5 min before warmed to room temperature and vigorously stirred for an additional 24-36 h. When the aromatic aldehyde substrate was fully consumed as determined by TLC analysis, the mixture was filtered through a pad of celite. The filtrate was concentrated under reduced pressure and the residue was used for the next step without further purification. An oven-dried pressure tube under nitrogen atmosphere was charged with the residue and dry toluene (3 mL) and MeOH (0.3 mL). The reaction mixture was heated to 100°C for 2 hours and then cooled to room temperature. The solvent (toluene and methanol) was removed under reduced pressure and the residue was purified by flash chromatography on silica gel using eluents (petroleum ether/ethyl acetate = 10/1) to provide the desired compound **3**.



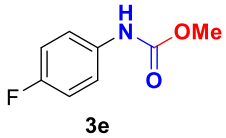
3a

3a. 38.5 mg, 85% yield ($PhCF_3$); 40 mg, 89% yield (CCl_4); white solid. **^{1}H NMR** (400 MHz, $CDCl_3$) δ: 7.40-7.37 (m, 2H), 7.33-7.28 (m, 2H), 7.09-7.04 (m, 1H), 6.70 (br, s), 3.78 (s, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ: 154.2, 137.9, 129.2 (2×C), 123.6 (2×C), 118.8, 52.5.



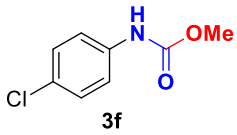
3c

3c. 41 mg, 83% yield ($PhCF_3$); 37.5 mg, 76% yield (CCl_4); white solid. **^{1}H NMR** (400 MHz, $CDCl_3$) δ: 7.26 (d, *J* = 8.4 Hz, 2H), 7.11 (d, *J* = 8.4 Hz, 2H), 6.62 (br, 1H), 3.76 (s, 3H), 2.30 (s, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ: 154.3, 135.3, 133.2, 129.7 (2×C), 118.9 (2×C), 52.4, 20.9.



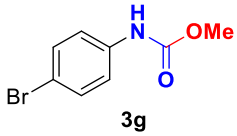
3e

3e. 36 mg, 71% yield ($PhCF_3$); 39.5 mg, 78% yield (CCl_4); white solid. **^{1}H NMR** (400 MHz, $CDCl_3$) δ: 7.36-7.30 (m, 2H), 7.02-6.96 (m, 2H), 6.78 (br, 1H), 3.76 (s, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ: 159.1 (d, *J* = 241 Hz), 154.4, 133.9, 120.6 (2×C), 115.8 (d, *J* = 23 Hz, 2×C), 52.5.



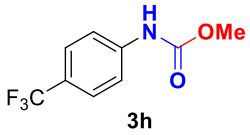
3f

3f. 45.5 mg, 82% yield ($PhCF_3$); 48 mg, 86% yield (CCl_4); white solid. **^{1}H NMR** (400 MHz, $CDCl_3$) δ: 7.36-7.33 (m, 2H), 7.28-7.25 (m, 2H), 6.77 (br, 1H), 3.78 (s, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ: 154.1, 136.6, 129.1 (2×C), 128.5, 120.0 (2×C), 52.6.



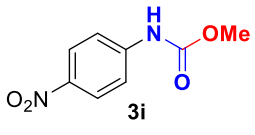
3g

3g. 50.5 mg, 73% yield ($PhCF_3$); 57.5 mg, 83% yield (CCl_4); white solid. **^{1}H NMR** (400 MHz, $CDCl_3$) δ: 7.40 (d, *J* = 8.8 Hz, 2H), 7.28 (d, *J* = 8.8 Hz, 2H), 6.79 (br, 1H), 3.76 (s, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ: 154.0, 137.1, 132.1 (2×C), 120.3 (2×C), 116.0, 52.6.



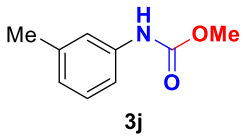
3h

3h. 45 mg, 68% yield ($PhCF_3$); 50 mg, 76% yield (CCl_4); white solid. **^{1}H NMR** (400 MHz, $CDCl_3$) δ: 7.55 (d, *J* = 8.8 Hz, 2H), 7.50 (d, *J* = 8.8 Hz, 2H), 6.93 (br, 1H), 3.79 (s, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ: 153.8, 141.1, 126.5 (q, *J* = 3.4 Hz, 2×C), 125.3 (q, *J* = 33 Hz), 124.3 (q, *J* = 270 Hz), 118.2 (2×C), 52.8.



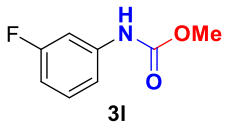
3i

3i. 39 mg, 66% yield ($PhCF_3$); 34 mg, 58% yield (CCl_4); yellowish solid. **^{1}H NMR** (400 MHz, $CDCl_3$) δ: 8.20 (d, *J* = 9.2 Hz, 2H), 7.56 (d, *J* = 9.2 Hz, 2H), 7.04 (br, 1H), 3.82 (s, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ: 153.4, 144.0, 143.1, 125.4 (2×C), 117.8 (2×C), 53.1.



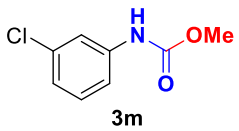
3j

3j. 39 mg, 79% yield ($PhCF_3$); 38 mg, 77% yield (CCl_4); white solid. **^{1}H NMR** (400 MHz, $CDCl_3$) δ: 7.23 (s, 1H), 7.21-7.17 (m, 2H), 6.89-6.87 (m, 1H), 6.70 (br, 1H), 3.77 (s, 3H), 2.33 (s, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ: 154.2, 139.1, 137.8, 129.0, 124.4, 119.4, 115.9, 52.4, 21.6.



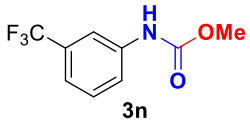
3l

3l. 40 mg, 79% yield ($PhCF_3$); 39 mg, 77% yield (CCl_4); white solid. **^{1}H NMR** (400 MHz, $CDCl_3$) δ: 7.35 (d, *J* = 10.8 Hz, 1H), 7.25 (td, *J* = 8.4, 6.8 Hz, 1H), 7.05 (d, *J* = 8.4 Hz, 1H), 6.86 (br, 1H), 6.78 (tdd, *J* = 8.4, 2.4, 0.8 Hz, 1H), 3.80 (s, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ: 163.3 (d, *J* = 243 Hz), 153.9, 139.6 (d, *J* = 11 Hz), 130.3 (d, *J* = 9.5 Hz), 114.0, 110.2 (d, *J* = 21 Hz), 106.2 (d, *J* = 26 Hz), 52.6.



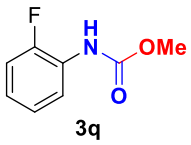
3m

3m. 43.5 mg, 78% yield ($PhCF_3$); 48 mg, 86% yield (CCl_4); white solid. **^{1}H NMR** (400 MHz, $CDCl_3$) δ: 7.50 (s, 1H), 7.22-7.18 (m, 2H), 7.04-7.01 (m, 1H), 6.81 (br, 1H), 3.77 (s, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ: 153.9, 139.2, 134.8, 130.1, 123.6, 118.8, 116.7, 52.6.



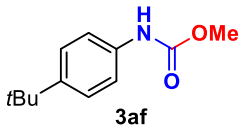
3n

3n. 58 mg, 88% yield ($PhCF_3$); 50.5 mg, 77% yield (CCl_4); white solid. **^{1}H NMR** (400 MHz, $CDCl_3$) δ: 7.71 (s, 1H), 7.56 (d, *J* = 8.0 Hz, 1H), 7.40 (t, *J* = 8.0 Hz, 1H), 7.31 (d, *J* = 8.0 Hz, 1H), 6.96 (br, 1H), 3.79 (s, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ: 154.1, 138.6, 131.6 (q, *J* = 32 Hz), 129.7, 124.0 (q, *J* = 271 Hz), 121.7, 120.1 (q, *J* = 3.7 Hz), 115.4, 52.7.



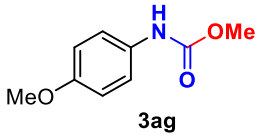
3q

3q. 0 mg, 0% yield ($PhCF_3$); 27 mg, 53% yield (CCl_4); colorless oil. **^{1}H NMR** (400 MHz, $CDCl_3$) δ: 8.13-8.03 (m, 1H), 7.14-6.96 (m, 3H), 6.89 (br, 1H), 3.79(s, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ: 153.8, 152.2 (d, *J* = 241 Hz), 126.5 (d, *J* = 10 Hz), 124.7 (d, *J* = 3.7 Hz), 123.5 (d, *J* = 7.2 Hz), 120.3, 114.9 (d, *J* = 18.9 Hz), 52.7. **^{19}F NMR** (376 MHz, $CDCl_3$) δ: -129.9.



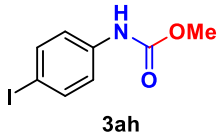
3af

3af. 49 mg, 79% yield ($PhCF_3$); 48.5 mg, 78% yield (CCl_4); white solid. **^{1}H NMR** (400 MHz, $CDCl_3$) δ: 7.32 (s, 4H), 6.74 (br, 1H), 3.77 (s, 3H), 1.31 (s, 9H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ: 154.3, 146.5, 135.3, 126.0 (2×C), 118.6 (2×C), 52.4, 34.4, 31.5 (3×C).



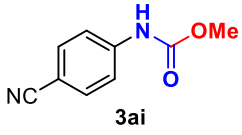
3ag

3ag. 33.5 mg, 62% yield ($PhCF_3$); 44.5 mg, 82% yield (CCl_4); white solid. **^{1}H NMR** (400 MHz, $CDCl_3$) δ: 7.28 (d, *J* = 8.8 Hz, 2H), 6.84 (d, *J* = 8.8 Hz, 2H), 6.66 (br, 1H), 3.78 (s, 3H), 3.75 (s, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ: 156.0, 155.0, 131.0, 120.8 (2×C), 114.3 (2×C), 55.6, 52.4.



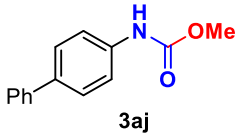
3ah

3ah. 60 mg, 72% yield ($PhCF_3$); 68 mg, 82% yield (CCl_4); white solid. **^{1}H NMR** (400 MHz, $CDCl_3$) δ: 7.57 (d, *J* = 8.8 Hz, 2H), 7.16 (d, *J* = 8.8 Hz, 2H), 6.84 (br, 1H), 3.76 (s, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ: 154.0, 138.0 (2×C), 137.8, 120.6 (2×C), 86.4, 52.6.



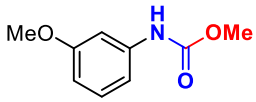
3ai

3ai. 35.5 mg, 67% yield ($PhCF_3$); 40 mg, 75% yield (CCl_4); white solid. **^{1}H NMR** (400 MHz, $CDCl_3$) δ: 7.58 (d, *J* = 8.8 Hz, 2H), 7.52 (d, *J* = 8.8 Hz, 2H), 7.19 (br, 1H), 3.79 (s, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ: 153.6, 142.3, 133.4 (2×C), 119.1, 118.4 (2×C), 106.1, 52.9.



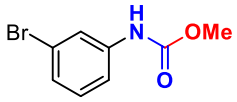
3aj

3aj. 54.5 mg, 80% yield ($PhCF_3$); 54 mg, 79% yield (CCl_4); yellowish solid. **^{1}H NMR** (400 MHz, $CDCl_3$) δ: 7.58-7.54 (m, 4H), 7.47-7.41 (m, 4H), 7.35-7.30 (m, 1H), 6.74 (br, 1H), 3.80 (s, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ: 154.2, 140.6, 137.2, 136.5, 128.9 (2×C), 127.8 (2×C), 127.2 (2×C), 126.9 (2×C), 119.1, 52.6.



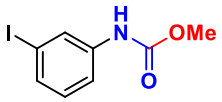
3ak

3ak. 35.5 mg, 65% yield ($PhCF_3$); 46 mg, 85% yield (CCl_4); yellowish solid. **^{1}H NMR** (400 MHz, $CDCl_3$) δ: 7.18 (t, *J* = 8.4 Hz, 1H), 7.12 (s, 1H), 6.86 (dd, *J* = 8.4, 1.2 Hz, 1H), 6.78 (br, 1H), 6.61 (ddd, *J* = 8.4, 2.4, 0.8 Hz, 1H), 3.79 (s, 3H), 3.76 (s, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ: 160.3, 154.1, 139.2, 129.8, 110.9, 109.3, 104.4, 55.4, 52.4.



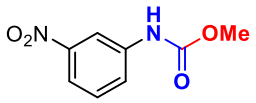
3al

3al. 59 mg, 85% yield ($PhCF_3$); 57 mg, 82% yield (CCl_4); white solid. **^{1}H NMR** (400 MHz, $CDCl_3$) δ: 7.64 (s, 1H), 7.28 (d, *J* = 7.6 Hz, 1H), 7.20-7.13 (m, 2H), 6.80 (br, 1H), 3.77 (s, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ: 153.9, 139.3, 130.4, 126.5, 122.8, 121.6, 117.2, 52.7.



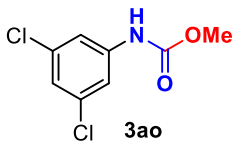
3am

3am. 64 mg, 77% yield ($PhCF_3$); 67 mg, 81% yield (CCl_4); white solid. m.p. = 66-68 ℃. **^{1}H NMR** (400 MHz, $CDCl_3$) δ: 7.79 (s, 1H), 7.39 (ddd, *J* = 8.0, 1.6, 0.8 Hz, 1H), 7.34 (d, *J* = 8.0 Hz, 1H), 7.01 (t, *J* = 8.0 Hz, 1H), 670 (br, 1H), 3.77 (s, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ: 153.8, 139.1, 132.6, 130.6, 127.4, 117.9, 94.4, 53.7. **HRMS** (ESI) *m/z* calculated for $C_8H_9O_2NI^+$ $[M+H]^+$ 277.9673, found 277.9673.



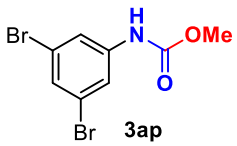
3an

3an. 34 mg, 58% yield ($PhCF_3$); 35 mg, 59% yield (CCl_4); yellowish solid. **^{1}H NMR** (400 MHz, $CDCl_3$) δ: 8.29 (t, *J* = 2.0 Hz, 1H), 7.91 (ddd, *J* = 8.4, 2.0, 0.8 Hz, 1H), 7.76 (d, *J* = 8.4 Hz, 1H), 7.47 (t, *J* = 8.4 Hz, 1H), 6.96 (br, 1H), 3.82 (s, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ: 153.8, 148.8, 139.2, 130.0, 124.2, 118.2, 113.4, 52.9.



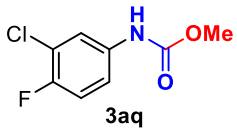
3ao

3ao. 56 mg, 85% yield ($PhCF_3$); 55 mg, 83% yield (CCl_4); white solid. **^{1}H NMR** (400 MHz, $CDCl_3$) δ: 7.34 (d, *J* = 1.2 Hz, 2H), 7.05 (t, *J* = 2.0 Hz, 1H), 6.67 (br, 1H), 3.79 (s, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ: 153.6, 139.8, 135.5 (2×C), 123.5 (2×C), 116.9, 52.9.



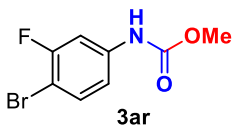
3ap

3ap. 67 mg, 72% yield ($PhCF_3$); 75 mg, 81% yield (CCl_4); white solid. m.p. = 41-43 ℃. **^{1}H NMR** (400 MHz, $CDCl_3$) δ: 7.52 (s, 2H), 7.33 (t, *J* = 1.6 Hz, 1H), 6.89 (br, 1H), 3.78 (s, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ: 153.7, 140.2, 128.9 (2×C), 123.2 (2×C), 120.2, 52.9. **HRMS** (ESI) *m/z* calculated for $C_8H_8O_2NBr_2$ $[M+H]^+$ 307.8916, found 307.8917.



3aq

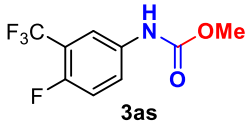
3aq. 51 mg, 84% yield ($PhCF_3$); 50 mg, 82% yield (CCl_4); white solid. m.p. = 88-90 ℃. **^{1}H NMR** (400 MHz, $CDCl_3$) δ: 7.56-7.52 (m, 1H), 7.20-7.17 (m, 1H), 7.05 (t, *J* = 8.8 Hz, 1H), 6.87 (br, 1H), 3.77 (s, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ: 154.4 (d, *J* = 243 Hz), 154.2, 134.6, 121.2 (d, *J* = 19 Hz), 121.0, 118.4, 116.7 (d, *J* = 22 Hz), 52.7. **^{19}F NMR** (376 MHz, $CDCl_3$) δ: -122.1. **HRMS** (ESI) *m/z* calculated for $C_8H_8O_2NClF$ $[M+H]^+$ 204.0222, found 204.0223.9



3ar

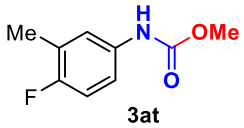
3ar. 52 mg, 70% yield ($PhCF_3$); 59 mg, 79% yield (CCl_4); white solid. **^{1}H NMR** (400 MHz, $CDCl_3$) δ: 7.41 (dd, *J* = 8.8, 7.6 Hz, 2H), 6.96 (dd, *J* = 8.8, 1.6 Hz, 1H), 6.90 (br, 1H), 3.77 (s, 3H). **^{13}C NMR**

(100 MHz, $CDCl_3$) δ: 159.3 (d, *J* = 244 Hz), 153.8, 138.8 (d, *J* = 10 Hz), 133.4 (d, *J* = 1.3 Hz), 115.2, 107.2 (d, *J* = 28 Hz), 102.3 (d, *J* = 21 Hz), 52.8. **^{19}F NMR** (376 MHz, $CDCl_3$) δ: -105.4.



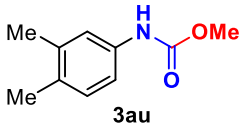
3as

3as. 53 mg, 74% yield ($PhCF_3$); 56 mg, 79% yield (CCl_4); white solid. m.p. = 68-70 ℃. **^{1}H NMR** (400 MHz, $CDCl_3$) δ: 7.66-7.64 (m, 1H), 7.58-7.55 (m, 1H), 7.13 (t, *J* = 9.2 Hz, 1H), 6.86 (br, 1H), 3.78 (s, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ: 155.7 (d, *J* = 253 Hz), 154.4, 134.2 (d, *J* = 2.0 Hz), 128.7 (d, *J* = 82 Hz), 124.0, 122.4 (q, *J* = 270 Hz), 118.6 (qd, *J* = 33, 14 Hz), 117.5 (d, *J* = 22 Hz), 52.7. **^{19}F NMR** (376 MHz, $CDCl_3$) δ: -61.7 (d, *J* = 12 Hz, 3×F), -121.5. **HRMS** (ESI) *m/z* calculated for $C_9H_8O_2NF_4$ $[M+H]^+$ 238.0486, found 238.0486.



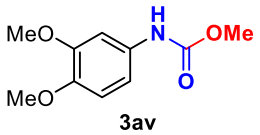
3at

3at. 43.5 mg, 79% yield ($PhCF_3$); 41 mg, 75% yield (CCl_4); white solid. m.p. = 41-43 ℃. **^{1}H NMR** (400 MHz, $CDCl_3$) δ: 7.23-7.19 (m, 1H), 7.13-7.10 (m, 1H), 6.92 (t, *J* = 9.2 Hz, 1H), 6.63 (br, 1H), 3.76 (s, 3H), 2.24 (d, *J* = 2.0 Hz, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ: 157.7 (d, *J* = 240 Hz), 154.4, 133.5, 125.5 (d, *J* = 18 Hz), 122.1, 117.8, 115.3 (d, *J* = 23 Hz), 52.5, 14.8 (d, *J* = 3.1 Hz). **^{19}F NMR** (376 MHz, $CDCl_3$) δ: -123.8. **HRMS** (ESI) *m/z* calculated for $C_9H_{11}O_2NF$ $[M+H]^+$ 184.0768, found 184.0769.



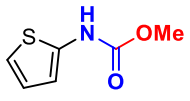
3au

3au. 44.5 mg, 83% yield ($PhCF_3$); 43 mg, 80% yield (CCl_4); yellowish solid. **^{1}H NMR** (400 MHz, $CDCl_3$) δ: 7.16-7.04 (m, 3H), 6.58 (br, 1H), 3.76 (s, 3H), 2.23 (s, 3H), 2.21 (s, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ: 154.3, 137.4, 135.6, 131.9, 130.1, 120.2, 116.3, 52.4, 30.0, 19.2.



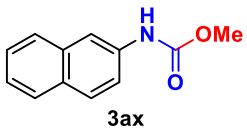
3av

3av. 39 mg, 62% yield ($PhCF_3$); 46 mg, 73% yield (CCl_4); white solid. **^{1}H NMR** (400 MHz, $CDCl_3$) δ: 7.17 (br, 1H), 6.78-6.73 (m, 3H), 3.84 (s, 3H), 3.82 (s, 3H), 3.74 (s, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ: 154.5, 149.2, 145.3, 131.5, 111.5, 110.7, 103.9, 56.2, 55.9, 52.4.



3aw

3aw. 22 mg, 47% yield ($PhCF_3$); 31.5 mg, 67% yield (CCl_4); white solid. **^{1}H NMR** (400 MHz, $CDCl_3$) δ: 7.34 (br, 1H), 6.86 (d, *J* = 5.2 Hz, 1H), 6.81 (dd, *J* = 5.2, 3.6 Hz, 1H), 6.60 (d, *J* = 2.4 Hz, 1H), 3.80 (s, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ: 154.2, 139.9, 124.7, 117.7, 112.5, 53.0.



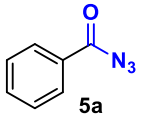
3ax

3ax. 48 mg, 79% yield ($PhCF_3$); 48 mg, 79% yield (CCl_4); white solid. **^{1}H NMR** (400 MHz, $CDCl_3$) δ: 7.99 (s, 1H), 7.77 (d, *J* = 8.8 Hz, 2H), 7.76 (d, *J* = 8.0 Hz, 1H), 7.47-7.37 (m, 3H), 6.91 (br, 1H), 3.82 (s, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ: 154.3, 135.4, 134.0, 130.3, 129.0, 127.7, 127.5, 126.6, 124.8, 119.3, 115.0, 52.6.

Oxone-KBr Mediated Azidation of Aldehydes to Afford Acyl Azides

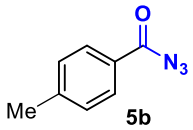
***General Procedure D*:** To a stirred solution of aromatic aldehyde substrate (0.3 mmol) in the $PhCF_3$ (analytical grade, 3.0 mL) or CCl_4 (analytical grade, 3.0 mL) at 0 ℃ were added KBr (54 mg, 0.45 mmol) and Oxone (166 mg, 0.54 mmol) and stirred for 5 min.

Then sodium azide (49 mg, 0.75 mmol or 43 mg, 0.66 mmol) was added. After the completion of the addition, the resulting mixture was vigorously stirred for 5 min before warmed to room temperature and stirred for an additional 24-36 h. When the aromatic aldehyde substrate was fully consumed as determined by TLC analysis, the mixture was filtered through a pad of celite. The filtrate was concentrated under reduced pressure and the residue was purified by flash chromatography on silica gel using eluents (petroleum ether/ethyl acetate = 50/1) to provide the desired compound **5**.



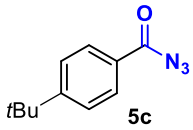
5a

5a. 37 mg, 84% yield ($PhCF_3$); 38 mg, 86% yield (CCl_4); colorless oil. **^{1}H NMR** (400 MHz, $CDCl_3$) δ: 8.04-8.01 (m, 2H), 7.64-7.60 (m, 1H), 7.48-7.44 (m, 2H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ: 172.7, 134.5, 130.8, 129.6 (2×C), 128.8 (2×C).



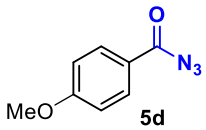
5b

5b. 32 mg, 66% yield ($PhCF_3$); 34.5 mg, 71% yield (CCl_4); colorless oil. **^{1}H NMR** (400 MHz, $CDCl_3$) δ: 7.92 (d, *J* = 8.4 Hz, 2H), 7.25 (d, *J* = 8.4 Hz, 2H), 2.42 (s, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ: 172.5, 145.6, 129.7 (2×C), 129.5 (2×C), 128.1, 21.9.



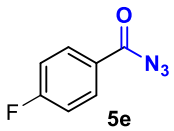
5c

5c. 49 mg, 80% yield ($PhCF_3$); 44.5 mg, 73% yield (CCl_4); white solid. **^{1}H NMR** (400 MHz, $CDCl_3$) δ: 7.96 (d, *J* = 8.8 Hz, 2H), 7.47 (d, *J* = 8.8 Hz, 2H), 1.34 (s, 9H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ: 172.5, 158.5, 129.5 (2×C), 128.1, 125.8 (2×C), 35.4, 31.2 (3×C).



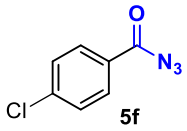
5d

5d. 31 mg, 58% yield ($PhCF_3$); 41 mg, 77% yield (CCl_4); white solid. **^{1}H NMR** (400 MHz, $CDCl_3$) δ: 7.97 (d, *J* = 8.8 Hz, 2H), 6.91 (d, *J* = 8.8 Hz, 2H), 3.86 (s, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ: 171.8, 164.7, 131.8 (2×C), 123.3, 114.0 (2×C), 55.6.



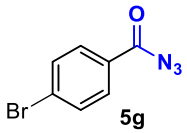
5e

5e. 35.5 mg, 72% yield ($PhCF_3$); 36 mg, 73% yield (CCl_4); white solid. **^{1}H NMR** (400 MHz, $CDCl_3$) δ: 8.08-8.03 (m, 2H), 7.16-7.10 (m, 2H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ: 171.5, 166.8 (d, *J* = 255 Hz), 132.3 (d, *J* = 9.5 Hz, 2×C), 127.1 (d, *J* = 2.8 Hz), 116.1 (d, *J* = 22 Hz, 2×C). **^{19}F NMR** (376 MHz, $CDCl_3$) δ: -103.0.



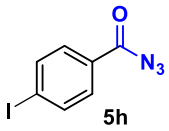
5f

5f. 42.5 mg, 78% yield ($PhCF_3$); 45 mg, 83% yield (CCl_4); white solid. **^{1}H NMR** (400 MHz, $CDCl_3$) δ: 7.95 (d, *J* = 8.8 Hz, 2H), 7.42 (d, *J* = 8.8 Hz, 2H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ: 171.7, 141.1, 130.9 (2×C), 129.2, 129.1 (2×C).

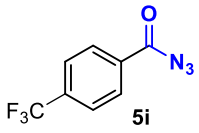


5g

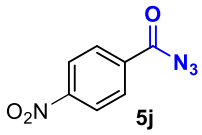
5g. 47 mg, 69% yield ($PhCF_3$); 53 mg, 78% yield (CCl_4); white solid. **^{1}H NMR** (400 MHz, $CDCl_3$) δ: 7.88 (d, *J* = 8.8 Hz, 2H), 7.60 (d, *J* = 8.8 Hz, 2H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ: 171.9, 132.2 (2×C), 131.0 (2×C), 129.9, 129.6.



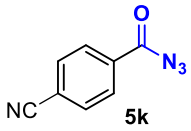
5h. 61.5 mg, 75% yield ($PhCF_3$); 63 mg, 77% yield (CCl_4); white solid. **^{1}H NMR** (400 MHz, $CDCl_3$) δ: 7.82 (d, *J* = 8.4 Hz, 2H), 7.72 (d, *J* = 8.4 Hz, 2H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ: 172.2, 138.2 (2×C), 130.8 (2×C), 130.2, 102.8.



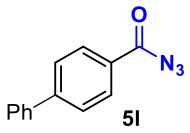
5i. 53 mg, 82% yield ($PhCF_3$); 44.5 mg, 69% yield (CCl_4); colorless oil. **^{1}H NMR** (400 MHz, $CDCl_3$) δ: 8.14 (d, *J* = 8.0 Hz, 2H), 7.72 (d, *J* = 8.0 Hz, 2H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ: 171.6, 135.7 (q, *J* = 32.5 Hz), 133.8, 130.0 (2×C), 125.9 (q, *J* = 3.6 Hz, 2×C), 123.6 (q, *J* = 271 Hz). **^{19}F NMR** (376 MHz, $CDCl_3$) δ: -63.3 (3×F).



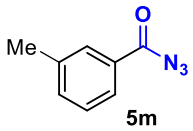
5j. 34.5 mg, 60% yield ($PhCF_3$); 33 mg, 57% yield (CCl_4); yellowish solid. **^{1}H NMR** (400 MHz, $CDCl_3$) δ: 8.30 (d, *J* = 9.2 Hz, 2H), 8.20 (d, *J* = 9.2 Hz, 2H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ: 171.0, 151.3, 135.8, 130.7 (2×C), 123.9 (2×C).



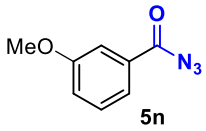
5k. 36 mg, 70% yield ($PhCF_3$); 34.5 mg, 67% yield (CCl_4); white solid. **^{1}H NMR** (400 MHz, $CDCl_3$) δ: 8.13 (d, *J* = 8.8 Hz, 2H), 7.76 (d, *J* = 8.8 Hz, 2H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ: 171.3, 134.3, 132.6 (2×C), 130.0 (2×C), 117.8, 117.7.



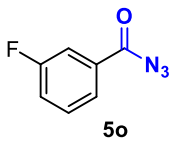
5l. 51 mg, 76% yield ($PhCF_3$); 47.5 mg, 71% yield (CCl_4); white solid. **^{1}H NMR** (400 MHz, $CDCl_3$) δ: 8.10 (d, *J* = 8.8 Hz, 2H), 7.68 (d, *J* = 8.8 Hz, 2H), 7.64-7.61 (m, 2H), 7.51-7.46 (m, 2H), 7.44-7.40 (m, 1H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ: 172.4, 147.2, 139.7, 130.1 (2×C), 129.5, 129.1 (2×C), 128.6, 127.4 (4×C).



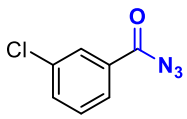
5m. 39 mg, 81% yield ($PhCF_3$); 35 mg, 72% yield (CCl_4); colorless oil. **^{1}H NMR** (400 MHz, $CDCl_3$) δ: 7.85-7.81 (m, 2H), 7.43 (d, *J* = 8.0 Hz, 1H), 7.34 (t, *J* = 8.0 Hz, 1H), 2.41 (s, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ: 172.8, 138.7, 135.3, 130.7, 130.1, 128.7, 126.8, 21.4.



5n. 32.5 mg, 61% yield ($PhCF_3$); 43 mg, 81% yield (CCl_4); colorless oil. **^{1}H NMR** (400 MHz, $CDCl_3$) δ: 7.62 (ddd, *J* = 8.0, 1.6, 1.2 Hz, 1H), 7.54 (dd, *J* = 2.4, 1.6 Hz, 1H), 7.36 (t, *J* = 8.0 Hz, 1H), 7.16 (ddd, *J* = 8.0, 2.8, 0.8 Hz, 1H), 3.86 (s, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ: 172.6, 159.9, 132.0, 129.8, 122.1, 121.2, 113.6, 55.6.

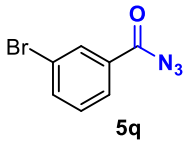


5o. 38.5 mg, 78% yield ($PhCF_3$); 36 mg, 72% yield (CCl_4); colorless oil. **^{1}H NMR** (400 MHz, $CDCl_3$) δ: 7.83 (ddd, *J* = 7.6, 1.6, 1.2 Hz, 1H), 7.71 (ddd, *J* = 9.2, 2.4, 1.6 Hz, 1H), 7.45 (td, *J* = 8.0, 5.6 Hz, 1H), 7.32 (tdd, *J* = 8.4, 2.8, 1.2 Hz, 1H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ: 171.6 (d, *J* = 3.0 Hz), 162.7 (d, *J* = 247 Hz), 132.8 (d, *J* = 7.4 Hz), 130.5 (d, *J* = 7.6 Hz), 125.3 (d, *J* = 3.2 Hz), 121.5 (d, *J* = 21 Hz), 116.4 (d, *J* = 23 Hz). **^{19}F NMR** (376 MHz, $CDCl_3$) δ: -111.6.



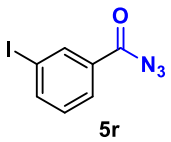
5p

5p. 41 mg, 75% yield ($PhCF_3$); 43.5 mg, 80% yield (CCl_4); colorless oil. **^{1}H NMR** (400 MHz, $CDCl_3$) δ: 8.01 (t, *J* = 1.6 Hz, 1H), 7.91 (ddd, *J* = 8.0, 1.6, 1.2 Hz, 1H), 7.59 (ddd, *J* = 8.0, 2.0, 1.2 Hz, 1H), 7.41 (t, *J* = 8.0 Hz, 1H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ: 171.6, 135.1, 134.4, 132.4, 130.1, 129.6, 127.7.



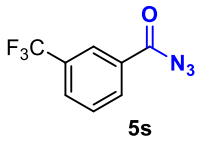
5q

5q. 47.5 mg, 70% yield ($PhCF_3$); 52 mg, 77% yield (CCl_4); colorless oil. **^{1}H NMR** (400 MHz, $CDCl_3$) δ: 8.16 (t, *J* = 1.6 Hz, 1H), 7.95 (dt, *J* = 8.0, 1.2 Hz, 1H), 7.74 (ddd, *J* = 8.0, 2.0, 1.2 Hz, 1H), 7.34 (t, *J* = 8.0 Hz, 1H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ: 171.4, 137.3, 132.6, 132.5, 130.4, 128.1, 122.9.



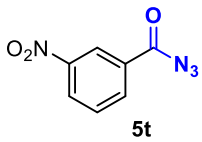
5r

5r. 66 mg, 81% yield ($PhCF_3$); 62 mg, 76% yield (CCl_4); colorless oil. **^{1}H NMR** (400 MHz, $CDCl_3$) δ: 8.35 (t, *J* = 1.6 Hz, 1H), 7.98 (ddd, *J* = 8.0, 1.6, 1.2 Hz, 1H), 7.93 (ddd, *J* = 8.0, 1.6, 1.2 Hz, 1H), 7.20 (t, *J* = 8.0 Hz, 1H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ: 171.2, 143.2, 138.3, 132.5, 130.4, 128.7, 94.1.



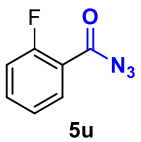
5s

5s. 50.5 mg, 78% yield ($PhCF_3$); 44 mg, 68% yield (CCl_4); colorless oil. **^{1}H NMR** (400 MHz, $CDCl_3$) δ: 8.29 (s, 1H), 8.22 (d, *J* = 8.0 Hz, 1H), 7.88 (dd, *J* = 8.0, 0.8 Hz, 1H), 7.62 (t, *J* = 8.0 Hz, 1H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ: 171.5, 132.7, 131.6, 131.5 (q, *J* = 33 Hz), 130.9 (q, *J* = 3.6 Hz), 129.6, 126.5 (q, *J* = 3.8 Hz), 123.6 (q, *J* = 271 Hz). **^{19}F NMR** (376 MHz, $CDCl_3$) δ: -62.9 (3×F).



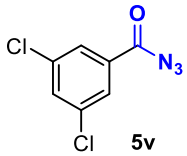
5t

5t. 33.5 mg, 58% yield ($PhCF_3$); 32 mg, 56% yield (CCl_4); yellowish solid. **^{1}H NMR** (400 MHz, $CDCl_3$) δ: 8.86 (t, *J* = 2.0 Hz, 1H), 8.47 (ddd, *J* = 8.0, 2.4, 1.2 Hz, 1H), 8.36 (dt, *J* = 8.0, 1.2 Hz, 1H), 7.69 (t, *J* = 8.0 Hz, 1H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ: 170.7, 148.5, 135.0, 132.4, 130.1, 128.7, 124.5.



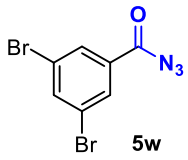
5u

5u. 0 mg, 0% yield ($PhCF_3$); 26 mg, 53% yield (CCl_4); colorless oil. **^{1}H NMR** (400 MHz, $CDCl_3$) δ: 7.93 (td, *J* = 7.6, 2.0 Hz, 1H), 7.61-7.55 (m, 1H), 7.22 (td, *J* = 8.0, 1.2 Hz, 1H), 7.16 (ddd, *J* = 10.8, 8.4, 1.2 Hz, 1H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ: 170.0 (d, *J* = 4.4 Hz), 162.3 (d, *J* = 261 Hz), 136.0 (d, *J* = 9.2 Hz), 132.0, 124.3 (d, *J* = 3.8 Hz), 119.2 (d, *J* = 8.6 Hz), 117.4 (d, *J* = 22 Hz). **^{19}F NMR** (376 MHz, $CDCl_3$) δ: -131.0.



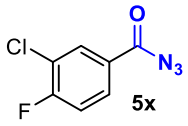
5v

5v. 52.5 mg, 81% yield ($PhCF_3$); 50.5 mg, 78% yield (CCl_4); white solid. **^{1}H NMR** (400 MHz, $CDCl_3$) δ: 7.89 (d, *J* = 2.0 Hz, 2H), 7.59 (t, *J* = 2.0 Hz). **^{13}C NMR** (100 MHz, $CDCl_3$) δ: 170.5, 135.8, 134.1 (2×C), 133.4, 127.9 (2×C).



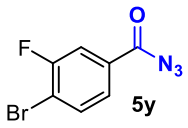
5w

5w. 61 mg, 67% yield ($PhCF_3$); 68 mg, 74% yield (CCl_4); yellowish solid. **^{1}H NMR** (400 MHz, $CDCl_3$) δ: 8.08 (d, *J* = 1.6 Hz, 2H), 7.89 (t, *J* = 1.6 Hz, 1H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ: 170.2, 139.6 (2×C), 133.8, 131.2 (2×C), 123.5.



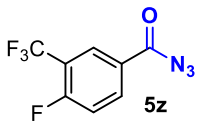
5x

5x. 50 mg, 83% yield ($PhCF_3$); 47 mg, 78% yield (CCl_4); colorless oil. **^{1}H NMR** (400 MHz, $CDCl_3$) δ: 8.11 (dd, *J* = 7.2, 2.0 Hz, 1H), 7.94 (ddd, *J* = 8.8, 4.8, 2.0 Hz, 1H), 7.22 (t, *J* = 8.8 Hz, 1H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ: 170.6, 162.1 (d, *J* = 257 Hz), 132.4, 130.0 (d, *J* = 8.7 Hz), 128.0 (d, *J* = 3.5 Hz), 122.1 (d, *J* = 18 Hz), 117.2 (d, *J* = 22 Hz). **^{19}F NMR** (376 MHz, $CDCl_3$) δ: -105.3.



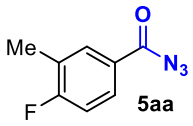
5y

5y. 49 mg, 67% yield ($PhCF_3$); 53 mg, 73% yield (CCl_4); colorless oil. **^{1}H NMR** (400 MHz, $CDCl_3$) δ: 7.74 (dd, *J* = 8.8, 1.6 Hz, 1H), 7.70-7.63 (m, 2H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ: 170.9 (d, *J* = 2.7 Hz), 159.2 (d, *J* = 248 Hz), 134.2, 131.8 (d, *J* = 6.7 Hz), 126.1 (d, *J* = 3.7 Hz), 117.2 (d, *J* = 24 Hz), 116.7 (d, *J* = 21 Hz). **^{19}F NMR** (376 MHz, $CDCl_3$) δ: -105.1.



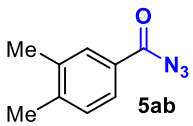
5z

5z. 45 mg, 64% yield ($PhCF_3$); 50 mg, 71% yield (CCl_4); colorless oil. **^{1}H NMR** (400 MHz, $CDCl_3$) δ: 8.32 (dd, *J* = 6.8, 2.0 Hz, 1H), 8.25 (ddd, *J* = 8.4, 4.8, 2.0 Hz, 1H), 7.30 (t, *J* = 9.2 Hz, 1H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ: 170.5, 163.5 (dq, *J* = 264, 1.6 Hz), 135.6 (d, *J* = 10 Hz), 129.3 (dq, *J* = 4.4, 3.0 Hz), 127.3 (d, *J* = 3.5 Hz), 122.0 (q, *J* = 271 Hz), 119.4 (qd, *J* = 33.8, 13.3 Hz), 117.8 (d, *J* = 21 Hz). **^{19}F NMR** (376 MHz, $CDCl_3$) δ: -61.8 (d, *J* = 12.7 Hz, 3×F), -104.6 (q, *J* = 12.7 Hz).



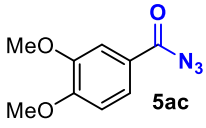
5aa

5aa. 42 mg, 78% yield ($PhCF_3$); 39 mg, 72% yield (CCl_4); colorless oil. **^{1}H NMR** (400 MHz, $CDCl_3$) δ: 7.90-7.83 (m, 2H), 7.05 (t, *J* = 8.8 Hz, 1H), 2.31 (d, *J* = 2.0 Hz, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ: 171.7, 165.4 (d, *J* = 254 Hz), 133.3 (d, *J* = 7.0 Hz), 129.6 (d, *J* = 9.6 Hz), 126.7 (d, *J* = 3.0 Hz), 125.8 (d, 18 Hz), 115.6 (d, *J* = 23 Hz), 14.6 (d, *J* = 3.5 Hz). **^{19}F NMR** (376 MHz, $CDCl_3$) δ: -107.1.



5ab

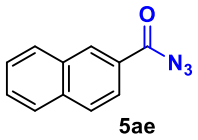
5ab. 42.5 mg, 81% yield ($PhCF_3$); 40 mg, 76% yield (CCl_4); yellowish solid. **^{1}H NMR** (400 MHz, $CDCl_3$) δ: 7.79 (s, 1H), 7.75 (dd, *J* = 8.0, 1.6 Hz, 1H), 7.20 (d, *J* = 8.0 Hz, 1H), 2.32 (s, 3H), 2.30 (s, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ: 172.7, 144.3, 137.3, 130.6, 130.1, 128.4, 127.3, 20.3. 19.8.



5ac

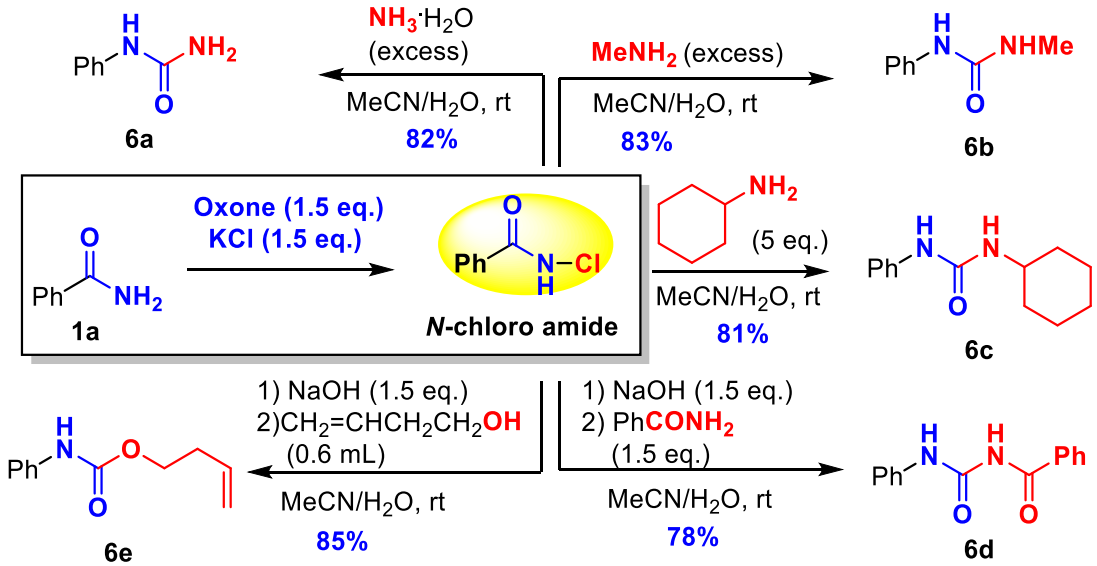
5ac. 36 mg, 58% yield ($PhCF_3$); 42 mg, 68% yield (CCl_4); white solid. **^{1}H NMR** (400 MHz, $CDCl_3$) δ: 7.66 (dd, *J* = 8.4, 2.0 Hz, 1H), 7.50 (d, *J* = 2.0 Hz, 1H), 6.87 (d, *J* = 8.4 Hz, 1H), 3.94 (s, 3H), 3.92 (s, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ: 171.8, 154.4, 149.0, 124.1, 123.4, 111.5, 110.4, 56.2, 56.1.

5ad. 19 mg, 41% yield ($PhCF_3$); 28 mg, 61% yield (CCl_4); colorless oil. **^{1}H NMR** (400 MHz, $CDCl_3$) δ: 7.85 (dd, *J* = 4.0, 1.2 Hz, 1H), 7.67 (dd, *J* = 4.8, 1.2 Hz, 1H), 7.14 (dd, *J* = 4.8, 4.0 Hz, 1H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ: 166.8, 135.0, 134.9, 134.6, 128.6.

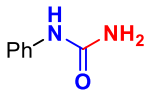


5ae. 40 mg, 68% yield ($PhCF_3$); 44 mg, 74% yield (CCl_4); colorless oil. **^{1}H NMR** (400 MHz, $CDCl_3$) δ: 8.60 (s, 1H), 8.03 (dd, *J* = 8.8, 2.0 Hz, 1H), 7.96 (dd, *J* = 8.0, 0.4 Hz, 1H), 7.89 (d, *J* = 8.8 Hz, 2H), 7.65-7.54 (m, 2H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ: 172.7, 136.3, 132.5, 131.6, 129.8, 129.1, 128.7, 128.0, 127.9, 127.1, 124.7.

Hofmann Rearrangement with Alcohol/Amine/Amide

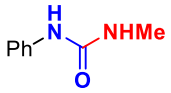


***Hofmann rearrangement with amine*:** To a stirred solution of benzamide **1a** (37 mg, 0.3 mmol) in the $MeCN/H_2O$ (10/1, 1.1 mL) at 0 ℃ were added KCl (34 mg, 0.45 mmol) and Oxone (138 mg, 0.45 mmol). After completion of the addition, the resulting mixture was stirred for 10 min before warmed to room temperature and stirred for an additional 2 h. When the benzamide **1a** was fully consumed as determined by TLC analysis, ammonia solution (25%, 0.2 mL) or methylamine solution (40%, 0.2 mL) or cyclohexyl amine (0.173 mL, 1.5 mmol) was added. The mixture was allowed to be stirred for 5 hrs and quenched by addition of sat. aq. Na_2SO_3 (5 mL). Volatiles (mainly MeCN) was removed under reduced pressure and the aqueous mixture was extracted with EA (3 X 10 mL). The combined organic fractions were washed with brine, dried over Na_2SO_4, and concentrated under reduced pressure. The residue was purified by flash column chromatography on silica gel using eluents (petroleum ether/ethyl acetate = 5/1) to provide the desired urea **6a**-**6c**.



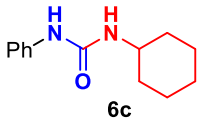
6a

6a. 33.5 mg, 82% yield, white solid. **^{1}H NMR** (400 MHz, DMSO-d_6) δ: 8.50 (br, 1H), 7.40-7.37 (m, 2H), 7.23-7.18 (m, 2H), 6.90-6.86 (m, 1H), 5.84 (br, 2H). **^{13}C NMR** (100 MHz, DMSO-d_6) δ: 156.0, 140.6, 128.6 (2×C), 121.1, 117.7 (2×C).



6b

6b. 37 mg, 83% yield, white solid. **^{1}H NMR** (400 MHz, DMSO-d_6) δ: 8.48 (br, 1H), 7.38 (d, *J* = 7.6 Hz, 2H), 7.20 (t, *J* = 7.6 Hz, 2H), 6.87 (t, *J* = 7.6 Hz, 1H), 5.98 (q, *J* = 4.4 Hz, 1H), 2.63 (d, *J* = 4.4 Hz, 3H). **^{13}C NMR** (100 MHz, DMSO-d_6) δ: 155.9, 140.7, 128.7 (2×C), 121.0, 117.7 (2×C), 26.3.



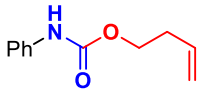
6c

6c. 53 mg, 81% yield, white solid. **^{1}H NMR** (400 MHz, $CDCl_3$) δ: 7.32-7.27 (m, 4H), 7.09-7.03 (m, 1H), 6.94 (br, 1H), 5.11 (br, 1H), 3.71-3.62 (m, 1H), 1.97-1.93 (m, 2H), 1.71-1.66 (m, 2H), 1.62-1.57 (m, 1H), 1.38-1.27 (m, 2H), 1.19-1.06 (m, 3H), **^{13}C NMR** (100 MHz, $CDCl_3$) δ: 155.5, 139.0, 129.3 (2×C), 123.5, 120.8 (2×C), 49.0, 33.8 (2×C), 25.7, 25.0 (2×C).

***Hofmann rearrangement with amide or alcohol*:** To a stirred solution of benzamide **1a** (37 mg, 0.3 mmol) in the MeCN/H_2O (10/1, 1.1 mL) at 0 ℃ were added KCl (34 mg, 0.45 mmol) and Oxone (138 mg, 0.45 mmol). After completion of the addition, the resulting mixture was stirred for 10 min before warmed to room temperature and stirred for an additional 2 h. When the benzamide **1a** was fully consumed as determined by TLC analysis, benzamide (55 mg, 0.45 mmol, to afford **6d**) or homoallyl alcohol (0.6 mL, to afford **6e**) and NaOH (18 mg, 0.45 mmol) was added sequentially. The mixture was allowed to be stirred for 5 hrs and quenched by addition of sat. aq. Na_2SO_3 (5 mL). Volatiles (mainly MeCN and homoallyl alcohol) was removed under reduced pressure and the aqueous mixture was extracted with EA (3 X 10 mL). The combined organic fractions were washed with brine, dried over Na_2SO_4, and concentrated under reduced pressure. The residue was purified by flash column chromatography on silica gel using eluents (petroleum ether/ethyl acetate = 5/1) to provide the desired compounds **6d**-**6e**.

6d

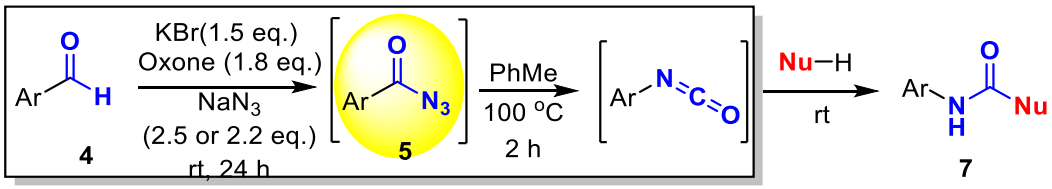
6d. 51 mg, 78% yield, brownish solid. **^{1}H NMR** (400 MHz, $CDCl_3$) δ: 10.97 (br, 1H), 9.80 (br, 1H), 8.08-8.04 (m, 2H), 7.67-7.59 (m, 3H), 7.53 (t, *J* = 8.0 Hz, 2H), 7.39-7.34 (m, 2H), 7.18-7.14 (m, 1H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ: 168.7, 152.0, 137.3, 133.5, 132.2, 129.1 (2×C), 129.0 (2×C), 128.1 (2×C), 124.6, 120.6 (2×C).



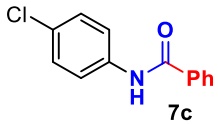
6e

6e. 49 mg, 85% yield, yellowish oil. **^{1}H NMR** (400 MHz, $CDCl_3$) δ: 7.40-7.37 (m, 2H), 7.32-7.28 (m, 2H), 7.08-7.04 (m, 1H), 6.67 (br, 1H), 5.83 (ddt, *J* = 17.2, 10.4, 6.8 Hz, 1H), 5.18-5.08 (m, 2H), 4.23 (t, *J* = 6.8 Hz, 2H), 2.44 (qt, *J* = 6.8, 1.2 Hz, 2H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ: 153.6, 138.0, 134.2 (2×C), 129.2 (2×C), 123.5, 118.7, 117.5, 64.4, 33.5.

Curtius Rearrangement with Amines to Generate (Chiral) Ureas/Amides



***Curtius rearrangement with benzoic acid to generate amide*:** To a stirred solution of aromatic aldehyde substrate (0.2 mmol) in the $PhCF_3$ (analytical grade, 2.0 mL) or CCl_4 (analytical grade, 2.0 mL) at 0 ℃ were added KBr (36 mg, 0.3 mmol) and Oxone (111 mg, 0.36 mmol) and stirred for 5 min. Then sodium azide (29 mg, 0.44 mmol) was added. After the completion of the addition, the resulting mixture was stirred for 5 min before warmed to room temperature and stirred for an additional 24-36 h. When the aromatic aldehyde substrate was fully consumed as determined by TLC analysis, the mixture was filtered through a pad of celite. The filtrate was concentrated under reduced pressure and the residue was used for the next step without further purification. Following Mahajan's protocol[1], an oven-dried pressure tube under nitrogen atmosphere was charged with the residue, benzoic acid (27 mg, 0.22 mmol), DBU (31 mg, 0.2 mmol) and dry toluene (1 mL). The reaction mixture was heated to 100°C for 2 hours and then cooled to room temperature. The solvent (toluene) was removed under reduced pressure and the residue was purified by flash chromatography on silica gel using eluents (petroleum ether/ethyl acetate = 10/1) to provide the desired amide **7c** (34 mg, 73% yield or 37.5 mg, 81% yield) as a white solid.

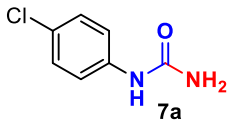


7c

^{1}H NMR (400 MHz, DMSO-d_6) δ: 10.39 (br, 1H), 7.96-7.93 (m, 2H), 7.82 (d, J = 8.8 Hz, 2H), 7.62-7.58 (m, 1H), 7.56-7.51 (m, 2H), 7.41 (d, J = 8.8 Hz, 2H). **^{13}C NMR** (100 MHz, DMSO-d_6) δ: 165.7, 138.2, 134.7, 131.8, 128.6 (2×C), 128.5 (2×C), 127.7 (2×C), 127.3, 121.8 (2×C).

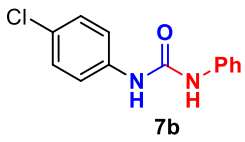
***Curtius rearrangement with amines to generate (chiral) ureas*:** To a stirred solution of aromatic aldehyde substrate (0.2 mmol) in the $PhCF_3$ (analytical grade, 2.0 mL) or CCl_4 (analytical grade, 2.0 mL) at 0 ℃ were added KBr (36 mg, 0.3 mmol) and Oxone (111 mg, 0.36 mmol) and stirred for 5 min. Then sodium azide (29 mg, 0.44 mmol) was added. After the completion of the addition, the resulting mixture was stirred for 5 min before warmed to room temperature and stirred for an additional 24-36 h. When the aromatic aldehyde substrate was fully consumed as determined by TLC analysis, the mixture was filtered through a pad of celite. The filtrate was concentrated under reduced pressure and the residue was used for the next step without further purification. An oven-dried pressure tube under nitrogen atmosphere was charged with the residue and dry toluene (1 mL). The reaction mixture was heated to 100°C for 2 hours and

then cooled to room temperature. The corresponding amine (0.22 mmol) was dissolved in dry toluene (1 mL) and added in one portion into the pressure tube under a positive pressure of nitrogen flow [in the case of **7a**, ammonia solution (25%, 0.1 mL) was used instead]. The resulting mixture was stirred at rt for 24 hours. The volatiles were removed under reduced pressure and the residue was purified by flash chromatography on silica gel using eluents (petroleum ether/ethyl acetate = 10/1) to provide the desired urea **7**.



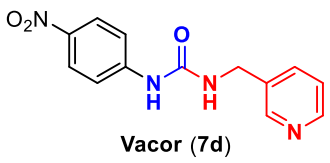
7a

7a. 26 mg, 76% yield ($PhCF_3$); 28 mg, 83% yield (CCl_4); white solid. **^{1}H NMR** (400 MHz, DMSO-d_6) δ: 8.67 (br, 1H), 7.42 (d, J = 8.8 Hz, 2H), 7.24 (d, J = 8.8 Hz, 2H), 5.91 (br, 2H). **^{13}C NMR** (100 MHz, DMSO-d_6) δ: 155.9, 139.6, 128.5 (2×C), 124.5, 119.2 (2×C).



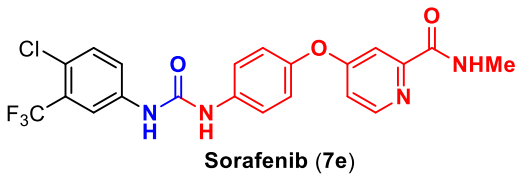
7b

7b. 38.5 mg, 78% yield ($PhCF_3$); 42 mg, 85% yield (CCl_4); white solid. **^{1}H NMR** (400 MHz, DMSO-d_6) δ: 8.81 (s, 1H), 8.70 (s, 1H), 7.48 (d, J = 8.8 Hz, 2H), 7.46-7.43 (m, 2H), 7.32 (d, J = 8.8 Hz, 2H), 7.30-7.25 (m, 2H), 6.97 (t, J = 7.2 Hz, 1H). **^{13}C NMR** (100 MHz, DMSO-d_6) δ: 152.5, 139.6, 138.8, 128.9 (2×C), 128.7 (2×C), 125.3, 122.0, 119.7 (2×C), 118.3 (2×C).



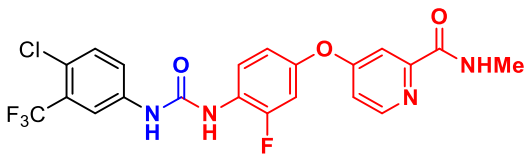
Vacor (**7d**)

7d. 46 mg, 84% yield ($PhCF_3$); 45 mg, 82% yield (CCl_4); white solid. **^{1}H NMR** (400 MHz, DMSO-d_6) δ: 9.47 (s, 1H), 8.54 (d, J = 1.6 Hz, 1H), 8.46 (dd, J = 4.8, 1.6 Hz, 1H), 8.14 (d, J = 9.2 Hz, 2H), 7.72 (dt, J = 8.0, 2.0 Hz,1H), 7.64 (d, J = 9.2 Hz, 2H), 7.36 (ddd, J = 7.6, 4.8, 0.4 Hz, 1H), 7.04 (t, J = 6.0 Hz, 1H), 4.35 (d, J = 6.0 Hz, 2H). **^{13}C NMR** (100 MHz, DMSO-d_6) δ: 154.6, 148.8, 148.2, 147.1, 140.5, 135.5, 135.1, 125.2 (2×C), 123.5, 117.0 (2×C), 40.6. **HRMS** (ESI) *m/z* calculated for $C_{13}H_{13}O_3N_4^+$ $[M+H]^+$ 273.0982, found 273.0976.



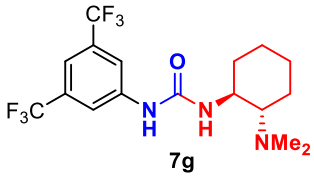
Sorafenib (**7e**)

7e. 78 mg, 84% yield ($PhCF_3$); 80 mg, 86% yield (CCl_4); yellowish solid. **^{1}H NMR** (400 MHz, DMSO-d_6) δ: 9.22 (s, 1H), 9.01 (s, 1H), 8.79 (q, J = 4.8 Hz, 1H), 8.49 (d, J = 5.6 Hz, 1H), 8.12 (d, J = 2.4 Hz, 1H), 7.67-7.59 (m, 2H), 7.60 (d, J = 9.2 Hz, 2H), 7.38 (d, J = 2.4 Hz, 1H), 7.17 (d, J = 9.2 Hz, 2H), 7.14 (dd, J = 5.6, 2.8 Hz, 1H), 2.78 (d, J = 4.8 Hz, 3H). **^{13}C NMR** (100 MHz, DMSO-d_6) δ: 166.0, 163.9, 125.5, 125.4, 150.4, 147.9, 139.4, 137.1, 132.0, 126.8 (q, J = 30 Hz), 123.1, 122.9 (q, J = 273 Hz), 122.4, 121.5 (2×C), 120.6 (2×C), 116.8 (q, J = 5.7 Hz), 114.1, 108.7, 26.1. **^{19}F NMR** (376 MHz, DMSO-d_6) δ: -61.5 (3×F). **HRMS** (ESI) *m/z* calculated for $C_{21}H_{17}O_3N_4F_3Cl^+$ $[M+H]^+$ 465.0936, found 465.0929.



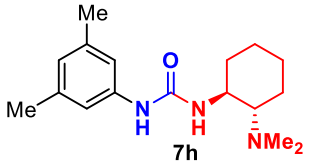
Regorafenib (7f)

7f. 82 mg, 85% yield ($PhCF_3$); 85 mg, 88% yield (CCl_4); yellowish solid. **^{1}H NMR** (400 MHz, DMSO-d_6) δ: 9.53 (s, 1H), 8.81 (q, *J* = 4.8 Hz, 1H), 8.75 (d, *J* = 2.0 Hz, 1H), 8.52 (d, *J* = 5.6 Hz, 1H), 8.16 (t, *J* = 9.2 Hz, 1H), 8.12 (s, 1H), 7.62 (s, 2H), 7.41 (d, *J* = 2.4 Hz, 1H), 7.34 (dd, *J* = 11.6, 2.8 Hz, 1H), 7.18 (dd, *J* = 5.6, 2.8 Hz, 1H), 7.07 (ddd, *J* = 9.2, 2.8, 1.2 Hz, 1H), 2.79 (d, *J* = 4.8 Hz, 3H). **^{13}C NMR** (100 MHz, DMSO-d_6) δ: 165.5, 163.8, 152.8 (d, *J* = 244 Hz), 152.6, 152.2, 150.5, 148.1 (d, *J* = 10.3 Hz), 139.0, 132.2, 126.8 (q, *J* = 30 Hz), 125.0 (d, *J* = 10.8 Hz), 123.0, 122.8 (q, *J* = 271 Hz), 122.6 (d, *J* = 2.3 Hz), 122.5 (d, *J* = 2.2 Hz), 117.2 (d, *J* = 2.9 Hz), 116.6 (q, *J* = 5.6 Hz), 114.2, 109.2 (d, *J* = 22 Hz), 108.9, 26.1. **^{19}F NMR** (376 MHz, DMSO-d_6) δ: -61.6 (3×F), -124.6. **HRMS** (ESI) *m/z* calculated for $C_{21}H_{16}O_3N_4F_4Cl^+$ $[M+H]^+$ 483.0842, found 483.0843.



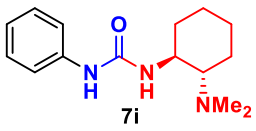
7g

7g. 64 mg, 80% yield ($PhCF_3$); 62 mg, 78% yield (CCl_4); white solid. $[\alpha]_D^{25}$= +33.5 (*c* 0.93, $CHCl_3$). **^{1}H NMR** (400 MHz, $CDCl_3$) δ: 8.48 (br, 1H), 7.84 (s, 2H), 7.37 (s, 1H), 5.95 (br, 1H), 3.69-3.61 (m, 1H), 3.56-3.51 (m, 1H), 2.50 (td, *J* = 10.8, 2.8 Hz, 1H), 2.40 (s, 6H), 2.30 (d, *J* = 10.8 Hz, 1H), 1.92 (d, *J* = 12.4 Hz, 1H), 1.86 (d, *J* = 12.4 Hz, 1H), 1.72 (d, *J* = 12.4 Hz, 1H), 1.27-1.16 (m, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ: 155.5, 141.3, 132.0 (q, *J* = 33 Hz, 2×C), 123.4 (q, *J* = 271 Hz, 2×C), 118.2 (q, *J* = 3.3 Hz, 2×C), 115.3 (quint, *J* = 3.8 Hz), 67.4, 51.2, 40.0 (2×C), 33.9, 24.9, 24.8, 21.9. **^{19}F NMR** (376 MHz, $CDCl_3$) δ: -63.1 (6×F). **HRMS** (ESI) *m/z* calculated for $C_{17}H_{22}ON_3F_6^+$ $[M+H]^+$ 398.1662, found 398.1666.



7h

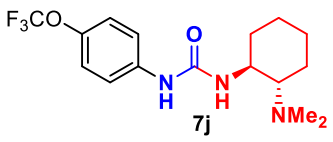
7h. 48 mg, 83% yield ($PhCF_3$); 49 mg, 85% yield (CCl_4); yellowish oil. $[\alpha]_D^{25}$= +51.9 (*c* 1.02, $CHCl_3$)[3]. **^{1}H NMR** (400 MHz, $CDCl_3$) δ: 7.52 (s, 1H), 6.98 (s, 2H), 6.64 (s, 1H), 6.01 (d, *J* = 5.6 Hz, 1H), 3.60 (s, 1H), 3.56-3.48 (m, 1H), 2.39-2.30 (m, 2H), 2.27 (s, 6H), 2.24 (s, 6H), 1.83-1.77 (m, 2H), 1.67-1.63 (m, 1H), 1.20-1.06 (m, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ: 156.6, 139.3, 138.7 (2×C), 124.7, 118.0 (2×C), 66.8, 51.4, 39.9 (2×C), 34.0, 25.1, 24.8, 21.6, 21.5 (2×C). **HRMS** (ESI) *m/z* calculated for $C_{17}H_{28}ON_3^+$ $[M+H]^+$ 290.2227, found 290.2232.



7i

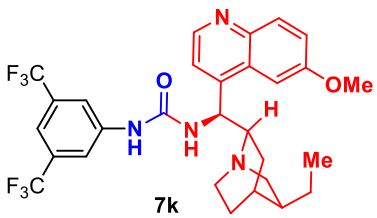
7i. 42 mg, 81% yield ($PhCF_3$); 42 mg, 81% yield (CCl_4); white solid. $[\alpha]_D^{25}$= +58.3 (*c* 0.65, $CHCl_3$). **^{1}H NMR** (400 MHz, $CDCl_3$) δ: 7.54 (br, 1H), 7.35-7.32 (m, 2H), 7.29-7.24 (m, 2H), 7.04-6.99 (m, 1H), 5.79 (br, 1H), 3.54-3.46 (m, 1H), 3.73-3.68 (m, 1H), 2.45-2.41 (m, 1H), 2.29 (dd,

J = 10.8, 3.2 Hz, 1H), 2.25 (s, 6H), 1.87-1.79 (m, 2H), 1.70-1.66 (m, 1H), 1.36-1.25 (m, 1H), 1.23-1.07 (m, 2H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ: 156.7, 139.5, 129.1 (2×C), 123.0, 120.3 (2×C), 66.9, 51.8, 40.1 (2×C), 34.0, 25.3, 24.8, 21.6. **HRMS** (ESI) *m*/*z* calculated for $C_{15}H_{24}ON_3^+$ $[M+H]^+$ 262.1914, found 262.1914.



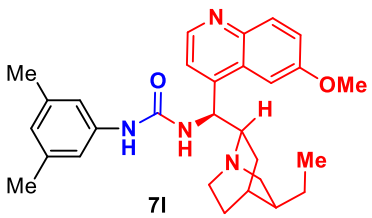
7j

7j. 59.5 mg, 86% yield ($PhCF_3$); 61 mg, 88% yield (CCl_4); white solid. m.p. = 90-92 ℃. $[\alpha]_D^{25}$= +29.8 (*c* 0.65, $CHCl_3$). **^{1}H NMR** (400 MHz, $CDCl_3$) δ: 7.68 (br, 1H), 7.34 (d, *J* = 8.8 Hz, 2H), 7.07 (d, *J* = 8.8 Hz, 2H), 5.68 (br, 1H), 3.54-3.46 (m, 1H), 2.78-2.75 (m, 1H), 2.38-2.34 (m, 1H), 2.30 (dd, *J* = 11.2, 3.2 Hz, 1H), 2.27 (s, 6H), 1.88-1.79 (m, 2H), 1.70-1.66 (m, 1H), 1.32-1.07 (m, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ: 156.3, 144.2 (q, *J* = 2.0 Hz), 138.3, 121.8 (2×C), 120.6 (q, *J* = 255 Hz), 120.5 (2×C), 67.3, 51.7, 40.2 (2×C), 34.0, 25.2, 24.8, 21.6. **^{19}F NMR** (376 MHz, $CDCl_3$) δ: -58.2 (3×F). **HRMS** (ESI) *m*/*z* calculated for $C_{16}H_{23}O_2N_3F_3^+$ $[M+H]^+$ 346.1737, found 346.1731.



7k

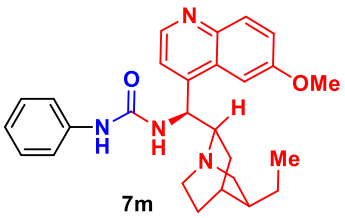
7k. 93 mg, 80% yield ($PhCF_3$); 89 mg, 77% yield (CCl_4); white solid. $[\alpha]_D^{25}$= -21.6 (*c* 0.36, $CHCl_3$). **^{1}H NMR** (400 MHz, $CDCl_3$) δ: 9.43 (br, 1H), 8.79 (d, *J* = 4.8 Hz, 1H), 8.01 (d, *J* = 9.2 Hz, 1H), 7.84 (s, 2H), 7.72 (s, 1H), 7.49 (d, *J* = 4.8 Hz, 1H), 7.38 (dd, *J* = 9.2, 2.8 Hz, 1H), 7.29 (s, 1H), 5.91 (br, 1H), 4.18-4.08 (m, 2H), 3.98 (s, 3H), 3.65 (dd, *J* = 13.2, 10.4 Hz, 1H), 3.18-3.10 (m, 1H), 2.83 (dd, *J* = 12.0, 7.2 Hz, 1H), 2.14-2.04 (m, 1H), 1.96 (s, 2H), 1.92-1.85 (m, 2H), 1.38-1.31 (m, 2H), 1.26 (d, *J* = 12.4 Hz, 1H), 1.19-1.15 (m, 1H), 0.85 (t, *J* = 7.2 Hz, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ: 158.8, 154.8, 147.7, 145.0, 142.4, 141.1, 131.9, 131.7 (q, *J* = 33 Hz, 2×C), 128.2, 123.3 (q, *J* = 271 Hz, 2×C), 122.7, 117.7 (2×C), 116.0, 115.2, 101.7, 59.6, 56.7, 56.0, 41.5, 35.4, 29.7, 26.5, 25.8, 25.0, 24.7, 11.6. **^{19}F NMR** (376 MHz, $CDCl_3$) δ: -58.3 (6×F). **HRMS** (ESI) *m*/*z* calculated for $C_{29}H_{31}O_2N_4F_6^+$ $[M+H]^+$ 581.2346, found 581.2351.



7l

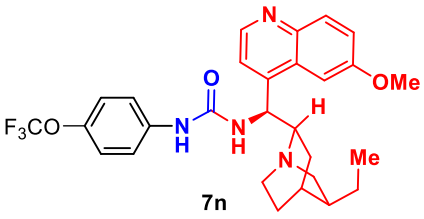
7l. 76 mg, 80% yield ($PhCF_3$); 74 mg, 78% yield (CCl_4); white solid. $[\alpha]_D^{25}$= +8.4 (*c* 0.5, $CHCl_3$). **^{1}H NMR** (400 MHz, $CDCl_3$) δ: 8.76 (d, *J* = 4.8 Hz, 1H), 8.54 (br, 1H), 7.97 (d, *J* = 9.2 Hz, 1H), 7.74 (d, *J* = 2.4 Hz, 1H), 7.59 (d, *J* = 4.8 Hz, 1H), 7.34 (dd, *J* = 9.2, 2.4 Hz, 1H), 7.08 (s, 2H), 6.55 (s, 1H), 5.87 (br, 1H), 4.22-4.16 (m, 1H), 4.11-4.05 (m, 1H), 4.00 (s, 3H), 3.59 (dd, *J* = 13.2, 10.4 Hz, 1H), 3.19-3.11 (m, 1H), 2.75 (dd, *J* = 11.6, 6.8 Hz, 1H), 2.18 (s, 6H), 2.02-1.95 (m, 1H), 1.84 (s, 2H), 1.81-1.64 (m, 2H), 1.27-1.19 (m, 3H), 1.11-1.07 (m, 1H), 0.77 (t, *J* = 7.2 Hz, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ: 158.7, 155.4,

147.8, 144.8, 142.2, 139.4, 138.4 (2×C), 131.7, 128.1, 124.3, 122.5, 119.7, 116.5 (2×C), 101.7, 59.8, 56.1, 56.0, 50.5, 41.7, 34.8, 29.7, 25.9, 25.0, 24.4, 21.5 (2×C), 11.5. **HRMS** (ESI) *m/z* calculated for $C_{29}H_{37}O_2N_4^+$ $[M+H]^+$ 473.2911, found 473.2911.



7m

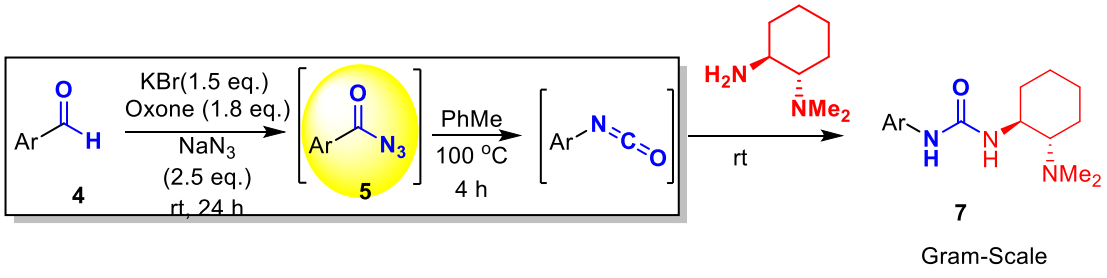
7m. 73 mg, 82% yield ($PhCF_3$); 70 mg, 79% yield (CCl_4); white solid. $[\alpha]_D^{25}$= +12.4 (*c* 0.5, $CHCl_3$). **^{1}H NMR** (400 MHz, $CDCl_3$) δ: 8.75 (d, *J* = 4.8 Hz, 1H), 8.52 (br, 1H), 7.98 (d, *J* = 9.2 Hz, 1H), 7.75 (d, *J* = 2.0 Hz, 1H), 7.54 (d, *J* = 4.8 Hz, 1H), 7.41 (d, *J* = 8.0 Hz, 2H), 7.35 (dd, *J* = 9.2, 2.4 Hz, 1H), 7.16 (t, *J* = 7.6 Hz, 2H), 6.91 (t, *J* = 7.6 Hz, 1H), 5.88 (br, 1H), 4.25-4.19 (m, 1H), 4.13-4.07 (m, 1H), 3.98 (s, 3H), 3.57 (d, *J* = 12.4, 10.8 Hz, 1H), 3.19-3.11 (m, 1H), 2.82 (dd, *J* = 11.6, 6.8 Hz, 1H), 2.03-1.96 (m, 1H), 1.86 (s, 2H), 1.81-1.70 (m, 2H), 1.24-1.18 (m, 3H), 1.12-1.08 (m, 1H), 0.79 (t, *J* = 7.2 Hz, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ: 158.8, 155.4, 147.8, 144.9, 142.0, 139.5, 131.8, 128.9 (2×C), 128.2, 122.6, 122.5, 119.7, 119.0 (2×C), 101.7, 59.7, 56.1 (2×C), 41.8, 34.8, 29.8, 26.0, 24.9, 24.5, 24.4, 11.6. **HRMS** (ESI) *m/z* calculated for $C_{27}H_{33}O_2N_4^+$ $[M+H]^+$ 445.2598, found 445.2592.



7n

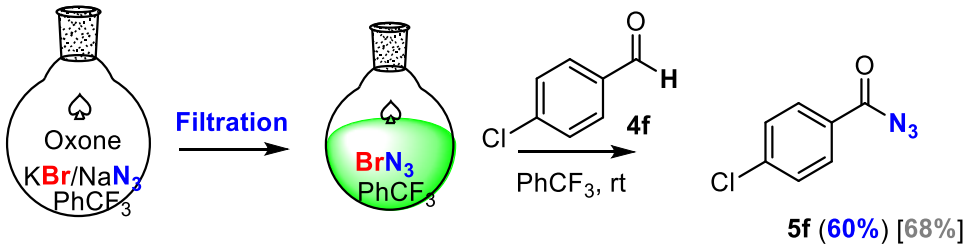
7n. 82.5 mg, 78% yield ($PhCF_3$); 84.5 mg, 80% yield (CCl_4); white solid. m.p. = 168-170 °C. $[\alpha]_D^{25}$= +3.8 (*c* 0.5, $CHCl_3$). **^{1}H NMR** (400 MHz, $CDCl_3$) δ: 8.82 (d, *J* = 4.8 Hz, 1H), 8.77 (br, 1H), 7.89 (d, *J* = 9.2 Hz, 1H), 7.70 (s, 1H), 7.69 (s, 1H), 7.35 (d, *J* = 8.8 Hz, 2H), 7.28 (dd, *J* = 9.2, 2.4 Hz, 1H), 6.89 (d, *J* = 8.8 Hz, 2H), 5.85 (br, 1H), 4.54-4.48 (m, 1H), 4.21-4.15 (m, 1H), 3.94 (s, 3H), 3.56 (dd, *J* = 12.8, 10.4 Hz, 1H), 3.17-3.09 (m, 1H), 3.01 (dd, *J* = 12.0, 7.6 Hz, 1H), 2.00-1.93 (m, 1H), 1.86-1.68 (m, 4H), 1.32-1.15 (m, 3H), 1.01-0.97 (m, 1H), 0.70 (t, *J* = 7.2 Hz, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ: 158.8, 155.3, 148.0, 144.5, 143.9, 141.9, 138.2, 131.4, 128.2, 122.8, 121.5 (2×C), 120.5 (q, *J* = 255 Hz), 119.8, 119.7 (2×C), 101.6, 59.5, 56.1, 56.0, 49.6, 41.8, 35.0, 25.9, 25.0, 24.7, 24.6, 11.5. **^{19}F NMR** (376 MHz, $CDCl_3$) δ: -63.0 (3×F). **HRMS** (ESI) *m/z* calculated for $C_{28}H_{32}O_3N_4F_3^+$ $[M+H]^+$ 529.2421, found 529.2426.

Gram Scale Synthesis of Chiral Ligands 7i and 7j



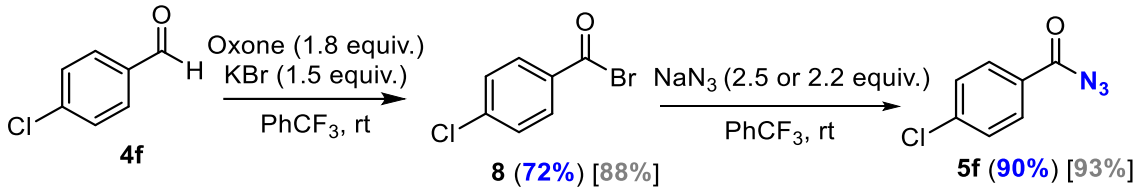
To a stirred solution of aromatic aldehyde substrate (6 mmol) in the $PhCF_3$ (analytical grade, 60 mL) at 0 ℃ were added KBr (1.06 g, 9 mmol) and Oxone (3.32 g, 10.8 mmol) and stirred for 5 min. Then sodium azide (975 mg, 15 mmol) was added. After the completion of the addition, the resulting mixture was stirred for 5 min before warmed to room temperature and stirred for an additional 36 h. When the aromatic aldehyde substrate was fully consumed as determined by TLC analysis, the mixture was filtered through a pad of celite. The filtrate was concentrated under reduced pressure and the residue was used for the next step without further purification. An oven-dried pressure tube under nitrogen atmosphere was charged with the residue and dry toluene (25 mL). The reaction mixture was heated to 100°C for 4 hours and then cooled to room temperature. The chiral amine (852 mg, 6 mmol) was dissolved in dry toluene (5 mL) and added dropwise into the pressure tube under a positive pressure of nitrogen flow. The resulting mixture was stirred at rt for 24 hours. The volatiles were removed under reduced pressure and the residue was purified by flash chromatography on silica gel using eluents (petroleum ether/ethyl acetate = 10/1) to provide the desired chiral ligand **7i** (1.17 g, 75% yield) and **7j** (1.66 g, 80%).

Control Experiments



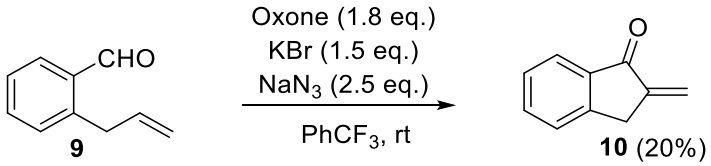
***In situ generation of BrN_3 from oxone-KBr-NaN_3*:** Following our previous procedure[2], a round bottom flask equipped with a stir bar was immersed in ice-water bath and charged with Oxone (666 mg, 2.16 mmol, 7.2 equiv.) and KBr (213 mg, 1.8 mmol, 6 equiv.). $PhCF_3$ (10 mL) or CCl_4 (10 mL) was added to the round bottom in one portion. The reaction mixture was stirred and kept at 0 ℃ and protected from light. After 5 minutes, sodium azide (195 mg, 3.0 mmol, 10 equiv. or 172 mg, 2.64 mmol, 8.8 equiv.) was added in one portion. After the completion of the addition, the resulting mixture was stirred for 5 min before warmed to room temperature and stirred for an additional 2 h. The mixture was filtered through a pad of celite and the filtrate was added to a round bottom flask. To the resulting solution *p*-chlorobenzaldehyde **4f** (43 mg, 0.3 mmol, 1 equiv.) was added. The solution was allowed to stir at rt until the starting material had been totally consumed (as monitored by TLC). Saturated aqueous

Na_2SO_3 solution (10 mL) was added. The resulting mixture was extracted with CH_2Cl_2 (3 × 10 mL). Organic layer was collected, then washed with brine, dried by anhydrous Na_2SO_4 and filtered. The filtrate was concentrated under reduced pressure and the residue was purified by flash chromatography on silica gel using eluents (petroleum ether/ethyl acetate = 50/1) to provide the desired compound **5f** (32.5 mg, 60% yield or 37 mg, 68% yield) as a white solid.



***Oxidation of aldehyde with Oxone-KBr*:** To a stirred solution of aromatic aldehyde **4f** (42 mg, 0.3 mmol) in the $PhCF_3$ (analytical grade, 3.0 mL) or CCl_4 (analytical grade, 3.0 mL) at 0 ℃ were added KBr (54 mg, 0.45 mmol) and Oxone (166 mg, 0.54 mmol). After the completion of the addition, the resulting mixture was stirred for 5 min before warmed to room temperature and stirred for an additional 2 h. When the aromatic aldehyde **4f** was fully consumed as determined by TLC analysis, the mixture was filtered through a pad of celite. The filtrate was concentrated under reduced pressure to afford the benzoyl bromide **8** (47 mg, 72% yield or 58 mg, 88% yield) as a colorless oil. **^{1}H NMR** (400 MHz, $CDCl_3$) δ: 8.01 (d, *J* = 8.8 Hz, 2H), 7.51 (d, *J* = 8.8 Hz, 2H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ: 162.3, 141.3, 131.3 (2×C), 129.5 (2×C), 123.9.

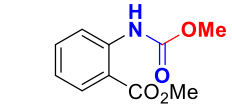
***Preparation of acyl azide from benzoyl bromide*:** To a stirred solution of self-prepared benzoyl bromide **8** (55 mg, 0.25 mmol) in the $PhCF_3$ (analytical grade, 3.0 mL) or CCl_4 (analytical grade, 2.5 mL) at 0 ℃ were added sodium azide (41 mg, 0.625 mmol or 36 mg, 0.55 mmol). After the completion of the addition, the resulting mixture was stirred for 5 min before warmed to room temperature and stirred for an additional 24 h. The mixture was filtered through a pad of celite. The filtrate was concentrated under reduced pressure and the residue was purified by flash chromatography on silica gel using eluents (petroleum ether/ethyl acetate = 50/1) to provide the desired acyl azide **5f** (41 mg, 90% yield or 42 mg, 93% yield) as a white solid.



***Radical cyclization reaction*:** To a stirred solution of aromatic aldehyde **9** (44 mg, 0.3 mmol) in the $PhCF_3$ (analytical grade, 3.0 mL) or CCl_4 (analytical grade, 3.0 mL) at 0 ℃ were added KBr (54 mg, 0.45 mmol) and Oxone (166 mg, 0.54 mmol) and stirred for 5 min. Then sodium azide (49 mg, 0.75 mmol or 43 mg, 0.66 mmol) was added. After the completion of the addition, the resulting mixture was stirred for 5 min before warmed to room temperature and stirred for an additional 24 h. When the substrate was fully consumed as determined by TLC analysis, the mixture was filtered through a pad of celite. The filtrate was concentrated under reduced pressure and the residue was purified by flash chromatography on silica gel using eluents (petroleum ether/ethyl acetate = 40/1) to provide the desired compound **10** (9 mg, 20% yield or 10 mg, 23% yield) as a colorless oil. **^{1}H NMR** (400 MHz, $CDCl_3$) δ: 7.86 (dd, *J* = 7.6, 0.4 Hz, 1H), 7.60 (td, *J* = 7.6, 1.2 Hz, 1H), 7.49 (dt, *J* = 7.6, 0.8 Hz, 1H), 7.42-7.38 (m, 1H), 6.36 (td, *J* = 2.4, 0.8 Hz, 1H), 5.64 (td, *J* = 2.0, 0.8 Hz, 1H), 3.75 (s, 2H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ: 193.6, 150.0, 143.4, 138.3, 135.0, 127.7, 126.5, 124.7, 119.5, 31.9.

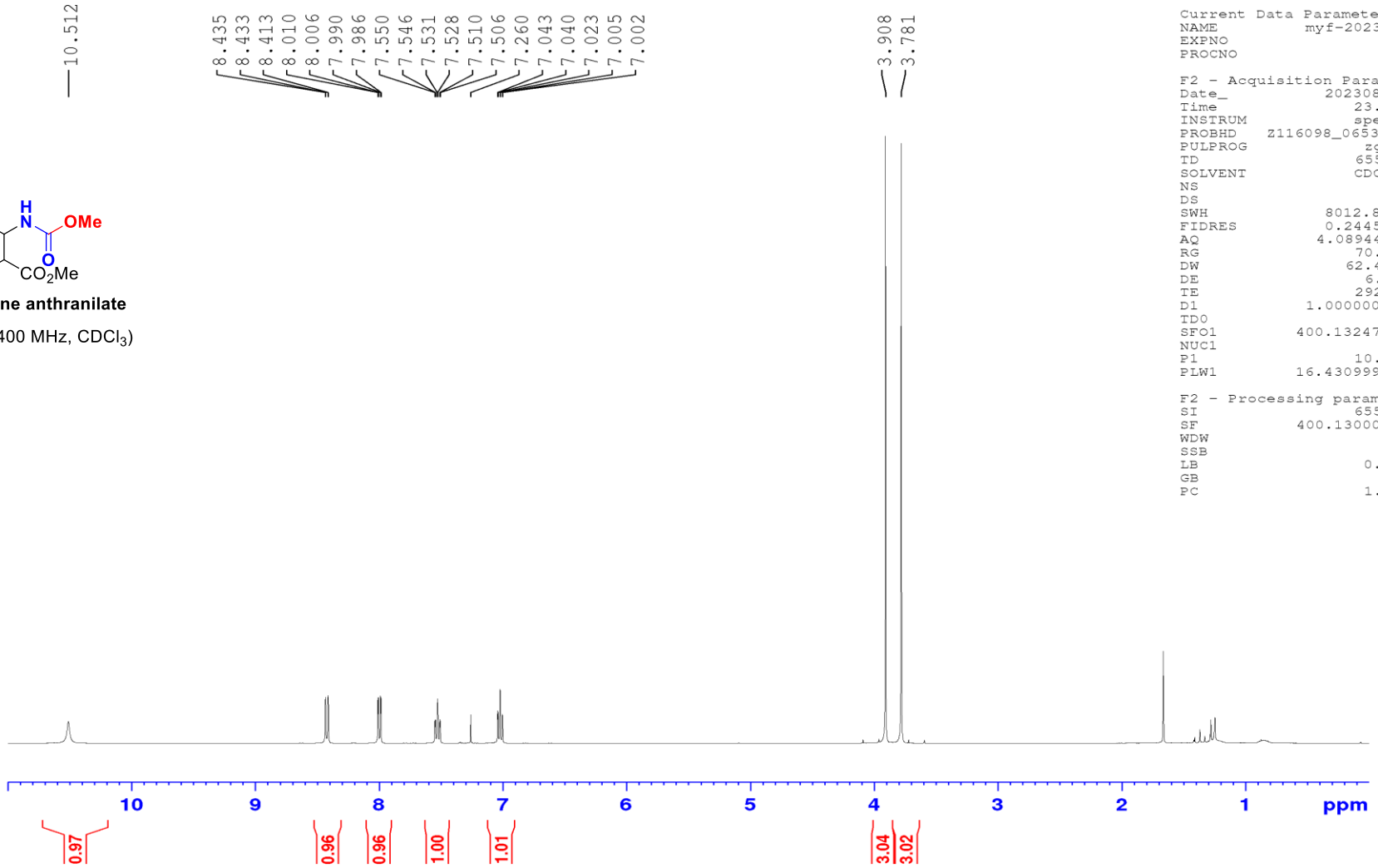
References

1. Kumar, A.; Kumar, N.; Sharma, R.; Bhargava, G.; Mahajan, D. *J. Org. Chem*. **2019**, *84*, 11323-11334.
2. Ren, J.; Tong, R. *Org. Biomol. Chem*. **2013**, *11*, 4312.
3. Berkessel, A.; Mukherjee, S.; Müller, T. N.; Cleemann, F.; Roland, K.; Brandenberg, M.; Neudörfl, J.-M.; Lex, J. *Org. Biomol. Chem*. **2006**, *4*, 4319-4330.



Urethylane anthranilate

^{1}H NMR (400 MHz, $CDCl_3$)

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EXPNO                          1
PROCNO                         1

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Time                       23.03 h
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TD                        65536
SOLVENT                   CDCl3
NS                           16
DS                            2
SWH                    8012.820 Hz
FIDRES                 0.244532 Hz
AQ                     4.0894465 sec
RG                         70.89
DW                        62.400 usec
DE                         6.50 usec
TE                        292.4 K
D1                     1.00000000 sec
TD0                            1
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NUC1                         1H
P1                        10.00 usec
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F2 - Processing parameters
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WDW                          EM
SSB                           0
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PC                         1.00
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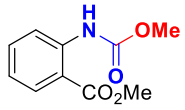
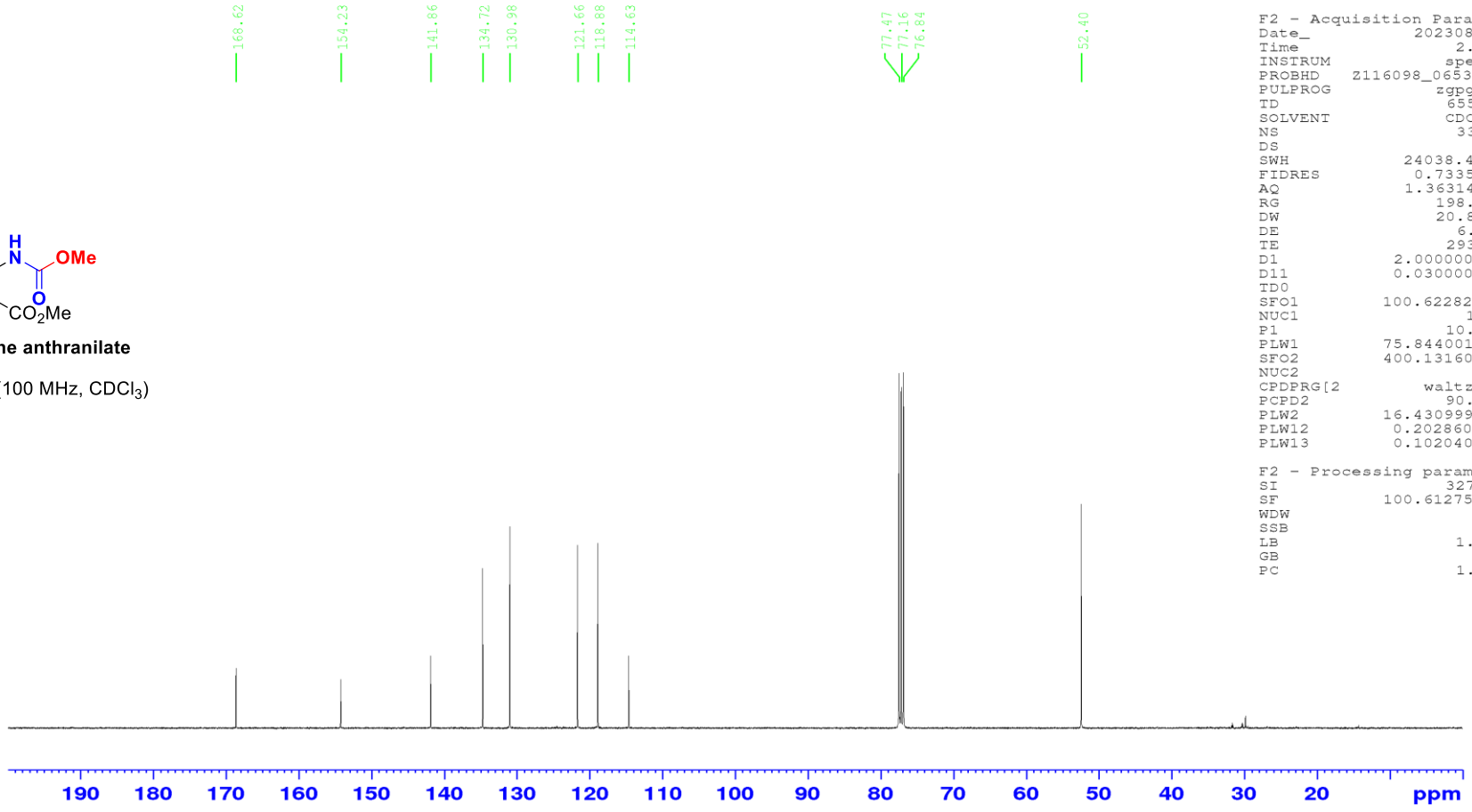


Urethylane anthranilate

^{13}C NMR (100 MHz, $CDCl_3$)

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EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
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Time               2.14 h
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PULPROG         zgpg30
TD              65536
SOLVENT           CDCl3
NS                 3333
DS                    4
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FIDRES         0.733596 Hz
AQ            1.3631488 sec
RG               198.36
DW               20.800 usec
DE                6.50 usec
TE                293.2 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1
SFO1        100.6228298 MHz
NUC1               13C
P1               10.00 usec
PLW1        75.84400177 W
SFO2        400.1316005 MHz
NUC2               1H
CPDPRG[2       waltz16
PCPD2            90.00 usec
PLW2        16.43099976 W
PLW12        0.20286000 W
PLW13        0.10204000 W

F2 - Processing parameters
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WDW                EM
SSB                 0
LB               1.00 Hz
GB                  0
PC               1.40
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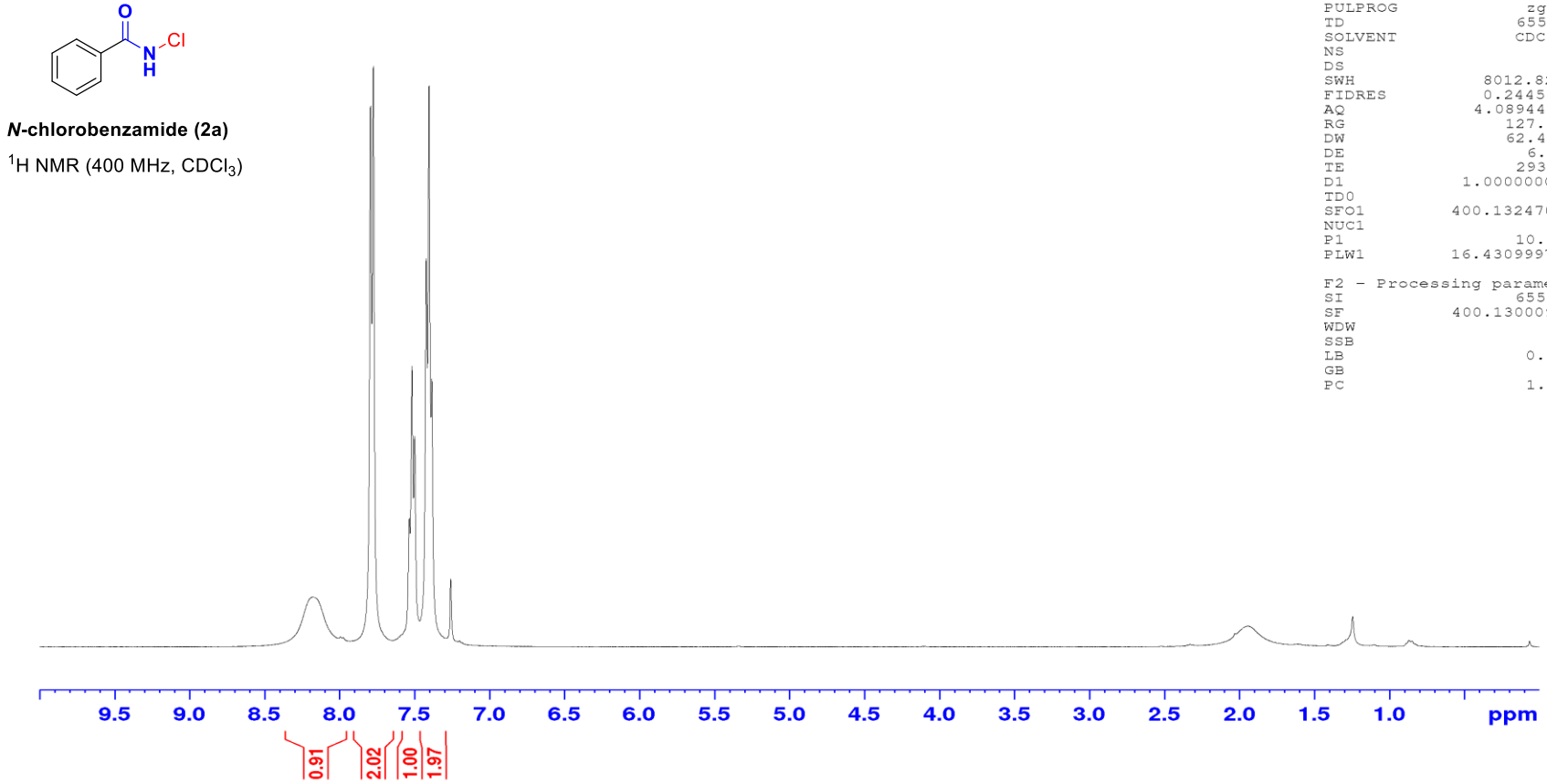

***N*-chlorobenzamide (2a)**

^{1}H NMR (400 MHz, $CDCl_3$)

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Current Data Parameters
NAME                      NHCl
EXPNO                        1
PROCNO                       1

F2 - Acquisition Parameters
Date_                 20230926
Time                     23.32 h
INSTRUM                  spect
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PULPROG                  zg30
TD                      65536
SOLVENT                 CDCl3
NS                          16
DS                           2
SWH                   8012.820 Hz
FIDRES                0.244532 Hz
AQ                    4.0894465 sec
RG                       127.66
DW                       62.400 usec
DE                        6.50 usec
TE                       293.6 K
D1                 1.00000000 sec
TD0                          1
SFO1           400.1324708 MHz
NUC1                        1H
P1                       10.00 usec
PLW1           16.43099976 W

F2 - Processing parameters
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WDW                        EM
SSB                         0
LB                       0.30 Hz
GB                          0
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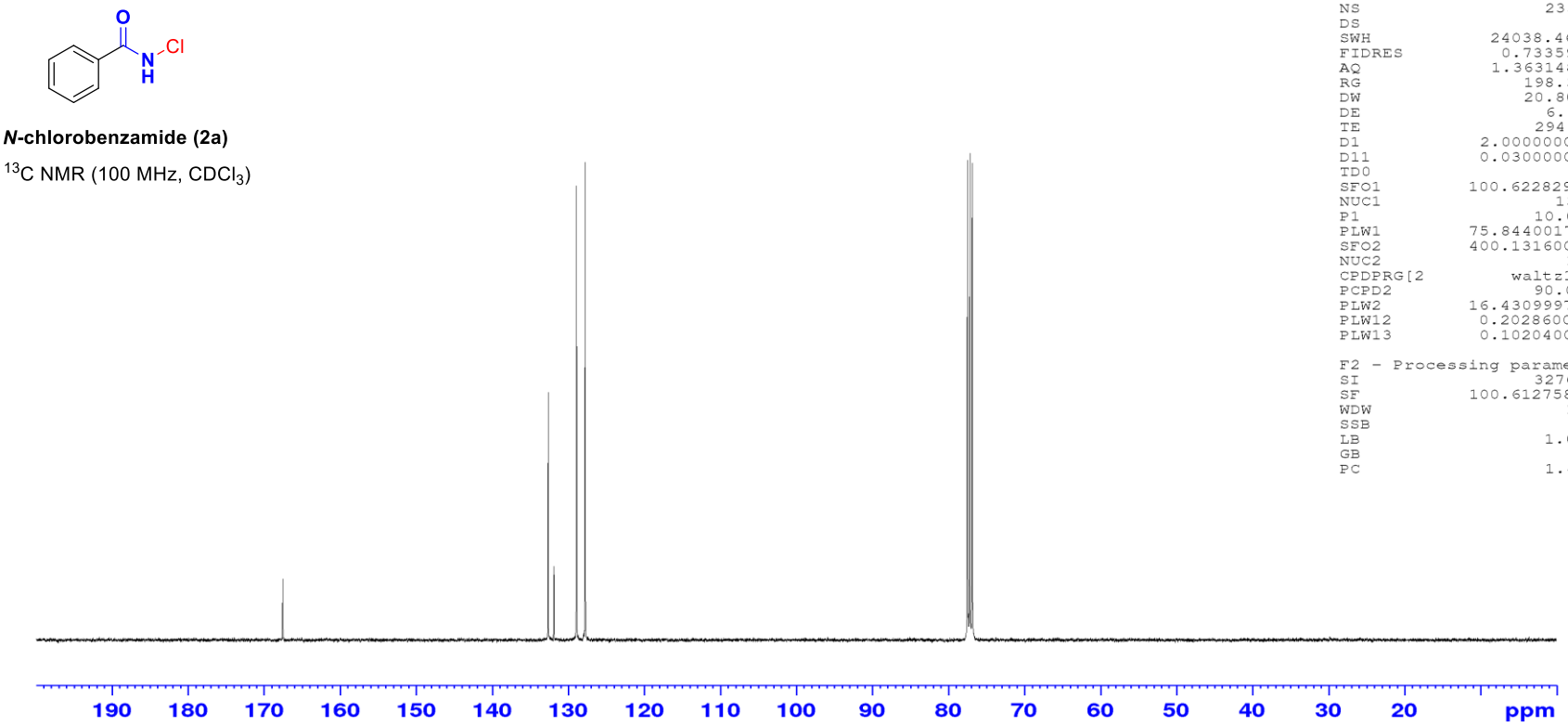

***N*-chlorobenzamide (2a)**

^{13}C NMR (100 MHz, $CDCl_3$)

```
Current Data Parameters
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EXPNO                     2
PROCNO                    1

F2 - Acquisition Parameters
Date_               20230927
Time                    1.47 h
INSTRUM                 spect
PROBHD    Z116098_0653 (
PULPROG                 zgpg30
TD                      65536
SOLVENT                 CDCl3
NS                      2333
DS                         4
SWH                24038.461 Hz
FIDRES             0.733596 Hz
AQ                 1.3631488 sec
RG                    198.36
DW                    20.800 usec
DE                     6.50 usec
TE                    294.4 K
D1                 2.00000000 sec
D11                0.03000000 sec
TD0                        1
SFO1             100.6228298 MHz
NUC1                     13C
P1                    10.00 usec
PLW1              75.84400177 W
SFO2              400.1316005 MHz
NUC2                     1H
CPDPRG[2              waltz16
PCPD2                 90.00 usec
PLW2              16.43099976 W
PLW12              0.20286000 W
PLW13              0.10204000 W

F2 - Processing parameters
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WDW                        EM
SSB                         0
LB                     1.00 Hz
GB                          0
PC                     1.40
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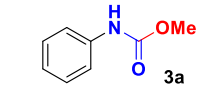
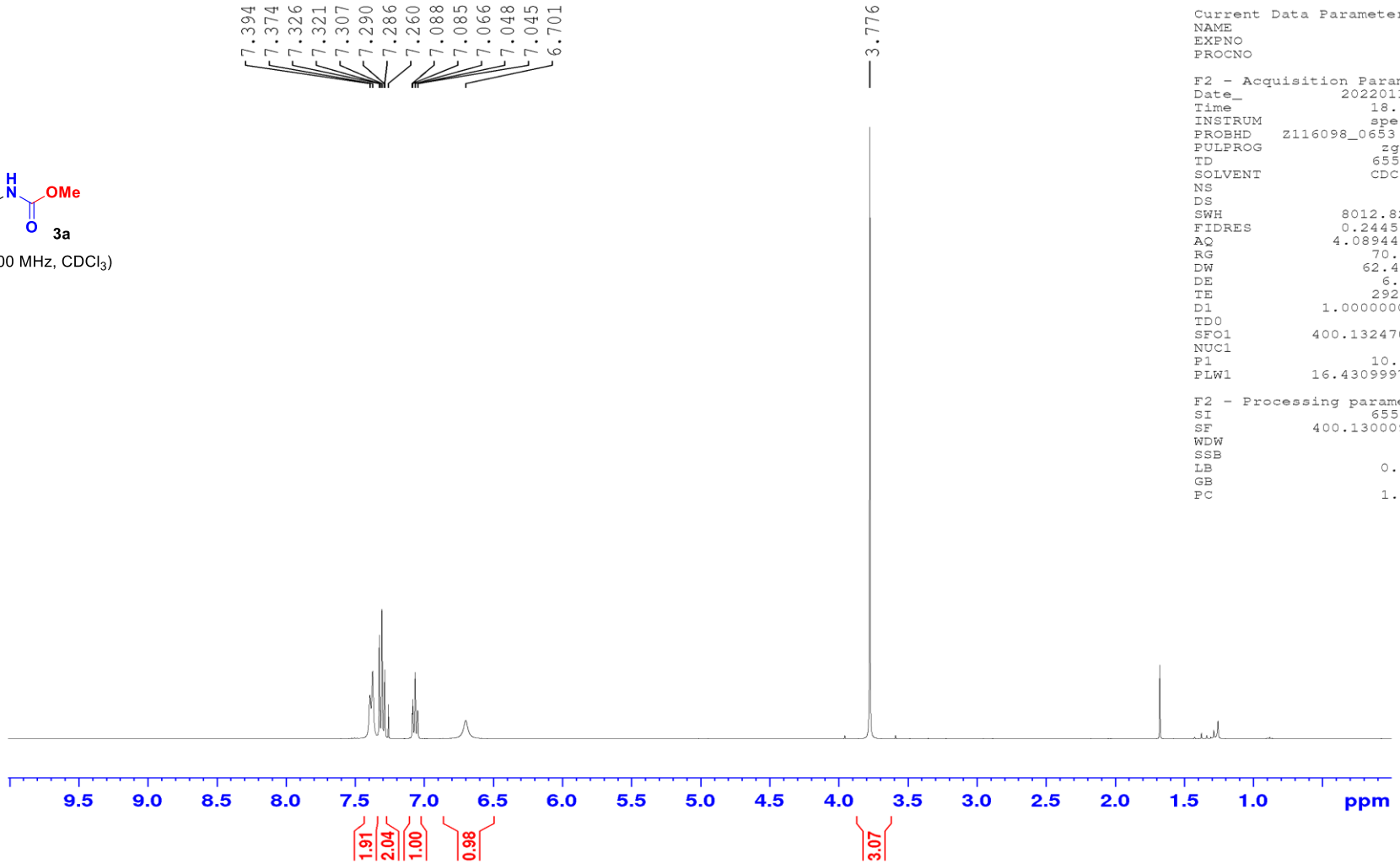


3a

^{1}H NMR (400 MHz, $CDCl_3$)

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EXPNO                      1
PROCNO                     1

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Time                   18.04 h
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PULPROG                zg30
TD                    65536
SOLVENT               CDCl3
NS                       16
DS                        2
SWH                8012.820 Hz
FIDRES            0.244532 Hz
AQ                4.0894465 sec
RG                    70.89
DW                   62.400 usec
DE                    6.50 usec
TE                   292.9 K
D1              1.00000000 sec
TD0                       1
SFO1           400.1324708 MHz
NUC1                    1H
P1                    10.00 usec
PLW1           16.43099976 W

F2 - Processing parameters
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WDW                     EM
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GB                       0
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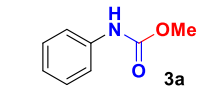
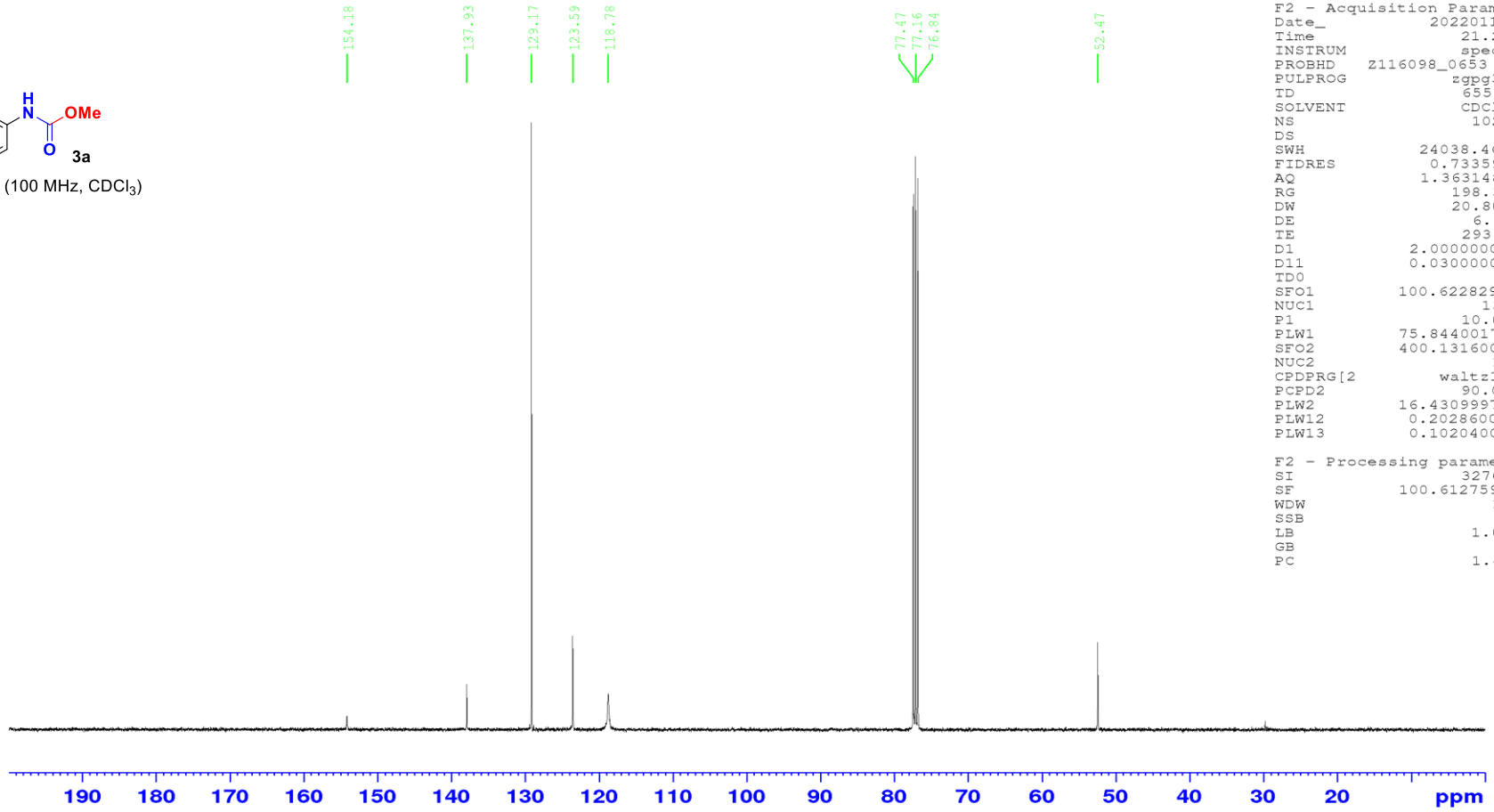


3a

^{13}C NMR (100 MHz, $CDCl_3$)

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EXPNO                      2
PROCNO                     1

F2 - Acquisition Parameters
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SOLVENT              CDCl3
NS                    1024
DS                       4
SWH             24038.461 Hz
FIDRES           0.733596 Hz
AQ               1.3631488 sec
RG                  198.36
DW                20.800 usec
DE                 6.50 usec
TE                293.6 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                      1
SFO1          100.6228298 MHz
NUC1                  13C
P1                 10.00 usec
PLW1          75.84400177 W
SFO2          400.1316005 MHz
NUC2                  1H
CPDPRG[2          waltz16
PCPD2              90.00 usec
PLW2          16.43099976 W
PLW12          0.20286000 W
PLW13          0.10204000 W

F2 - Processing parameters
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LB                    1.00 Hz
GB                       0
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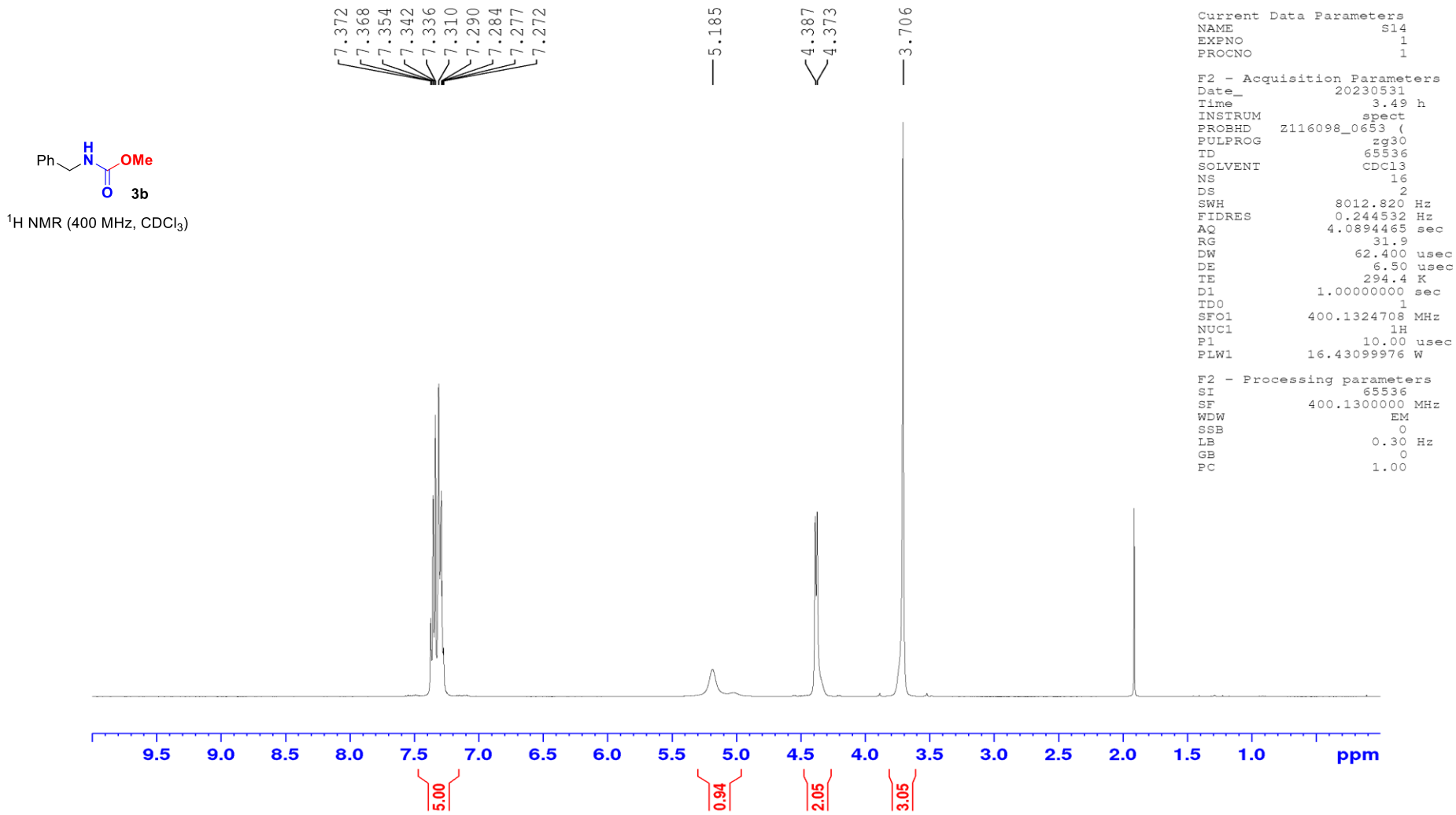
7.372
7.368
7.354
7.342
7.336
7.310
7.290
7.284
7.277
7.272
5.185
4.387
4.373
3.706
Ph
N
H
OMe
O
3b
1H NMR (400 MHz, CDCl3)
Current Data Parameters
NAME S14
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20230531
Time 3.49 h
INSTRUM spect
PROBHD Z116098_0653 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.244532 Hz
AQ 4.0894465 sec
RG 31.9
DW 62.400 usec
DE 6.50 usec
TE 294.4 K
D1 1.00000000 sec
TD0 1
SFO1 400.1324708 MHz
NUC1 1H
P1 10.00 usec
PLW1 16.43099976 W
F2 - Processing parameters
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SSB 0
LB 0.30 Hz
GB 0
PC 1.00
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
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ppm
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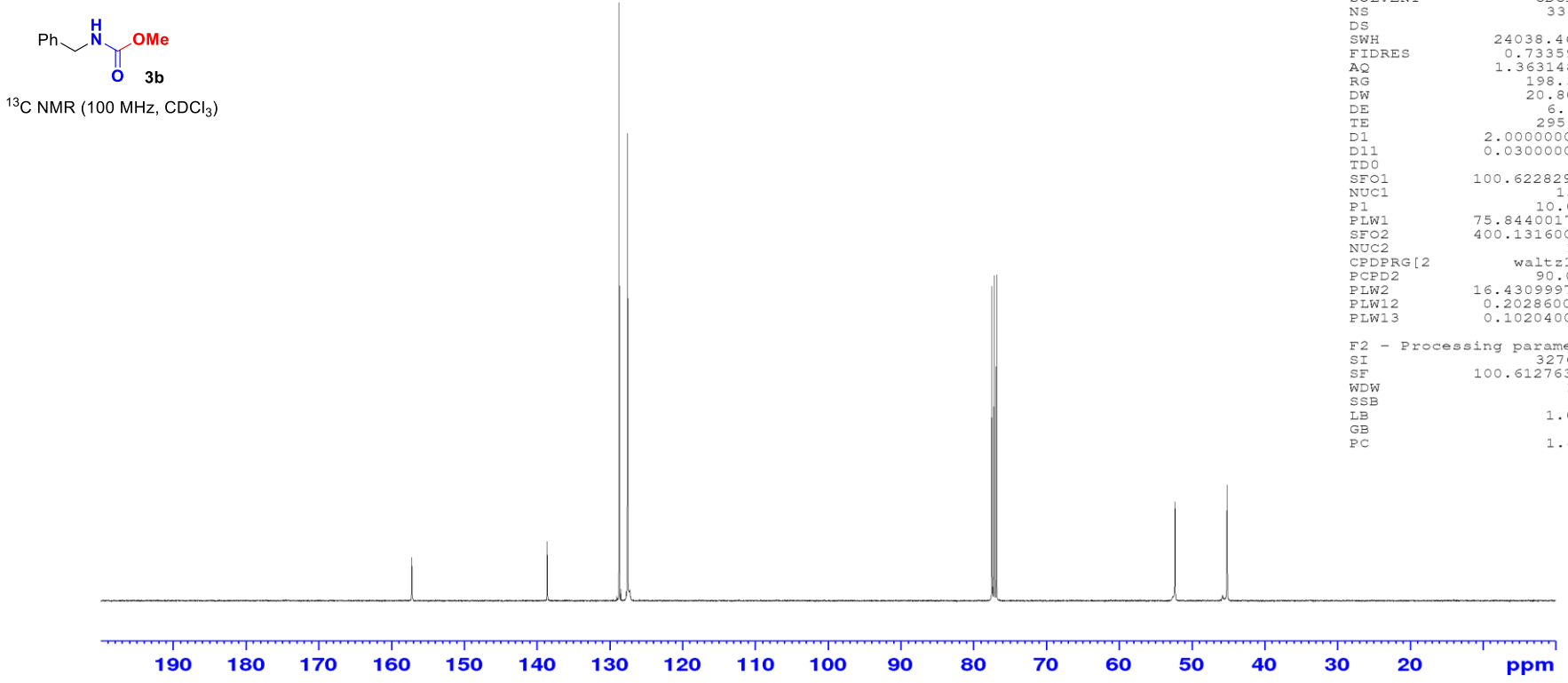
3.05



^{13}C NMR (100 MHz, $CDCl_3$)

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Current Data Parameters
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EXPNO                       2
PROCNO                      1

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Time                    7.00 h
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TD                     65536
SOLVENT                CDCl3
NS                      3333
DS                         4
SWH               24038.461 Hz
FIDRES             0.733596 Hz
AQ                1.3631488 sec
RG                   198.36
DW                   20.800 usec
DE                    6.50 usec
TE                   295.1 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                        1
SFO1          100.6228298 MHz
NUC1                    13C
P1                    10.00 usec
PLW1          75.84400177 W
SFO2          400.1316005 MHz
NUC2                    1H
CPDPRG[2             waltz16
PCPD2                 90.00 usec
PLW2          16.43099976 W
PLW12          0.20286000 W
PLW13          0.10204000 W

F2 - Processing parameters
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SF            100.6127638 MHz
WDW                       EM
SSB                        0
LB                     1.00 Hz
GB                         0
PC                     1.40
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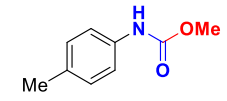
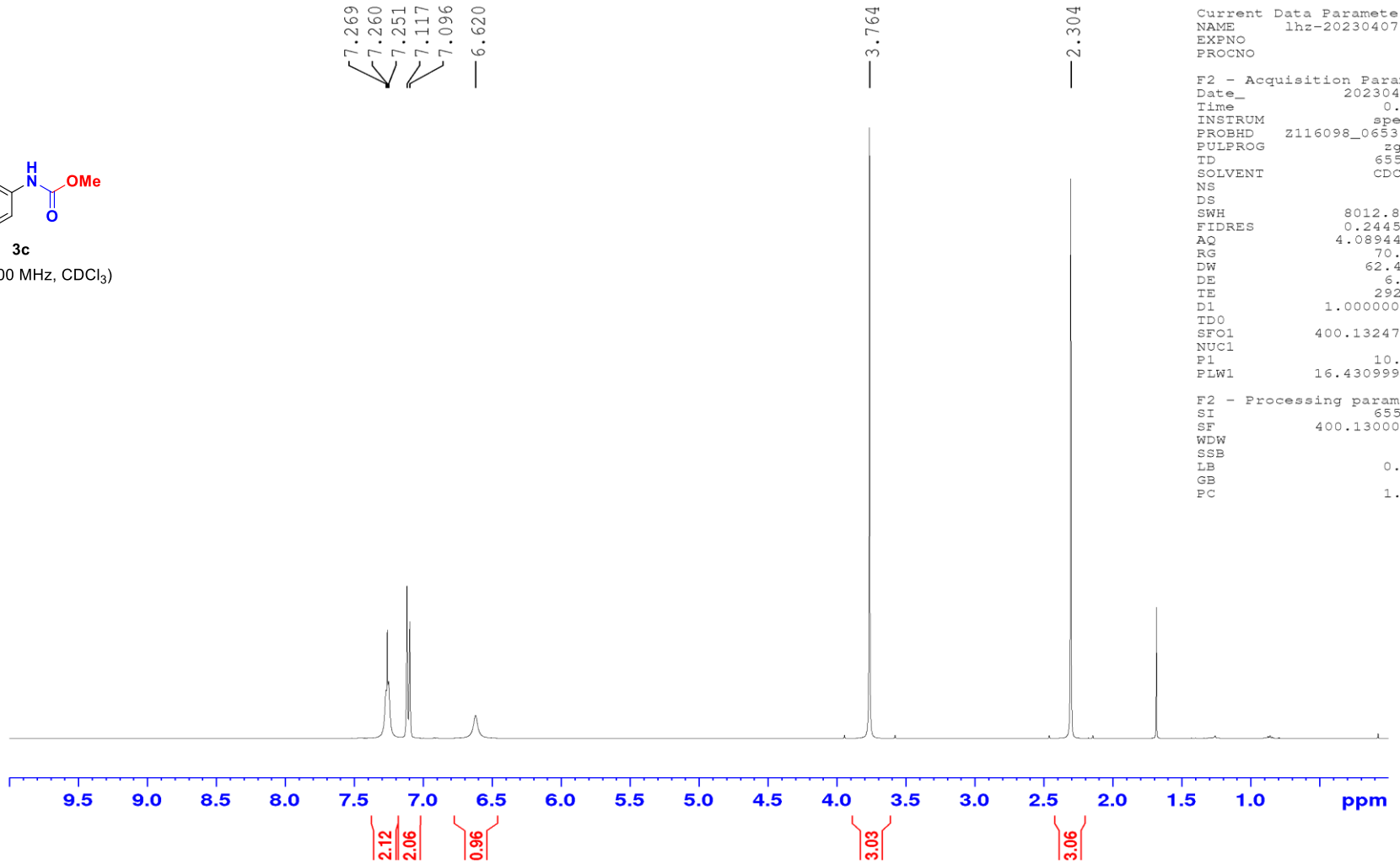


3c

^{1}H NMR (400 MHz, $CDCl_3$)

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PROCNO                 1

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PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH              8012.820 Hz
FIDRES           0.244532 Hz
AQ               4.0894465 sec
RG                  70.89
DW                 62.400 usec
DE                  6.50 usec
TE                 292.6 K
D1              1.00000000 sec
TD0                    1
SFO1          400.1324708 MHz
NUC1                  1H
P1                 10.00 usec
PLW1          16.43099976 W

F2 - Processing parameters
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SF            400.1300099 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
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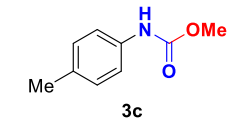
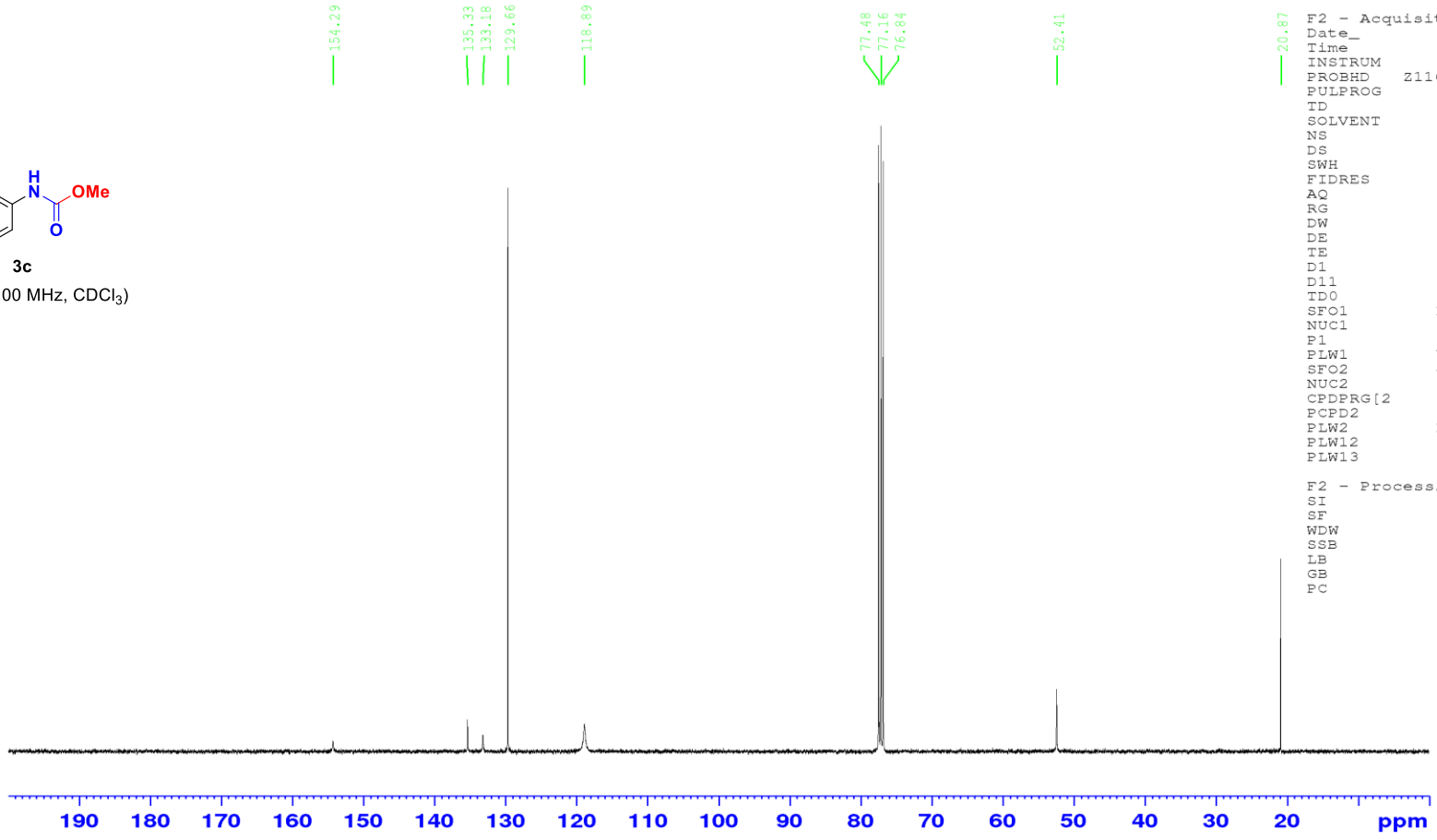


3c

^{13}C NMR (100 MHz, $CDCl_3$)

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PROCNO                 1

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Time              1.42 h
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PULPROG         zgpg30
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SOLVENT          CDCl3
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DS                   4
SWH          24038.461 Hz
FIDRES        0.733596 Hz
AQ            1.3631488 sec
RG               198.36
DW               20.800 usec
DE                6.50 usec
TE               292.9 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                  1
SFO1       100.6228298 MHz
NUC1              13C
P1               10.00 usec
PLW1       75.84400177 W
SFO2       400.1316005 MHz
NUC2              1H
CPDPRG[2       waltz16
PCPD2            90.00 usec
PLW2       16.43099976 W
PLW12       0.20286000 W
PLW13       0.10204000 W

F2 - Processing parameters
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SF         100.6127594 MHz
WDW                EM
SSB                 0
LB              1.00 Hz
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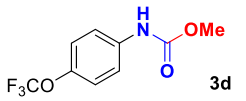
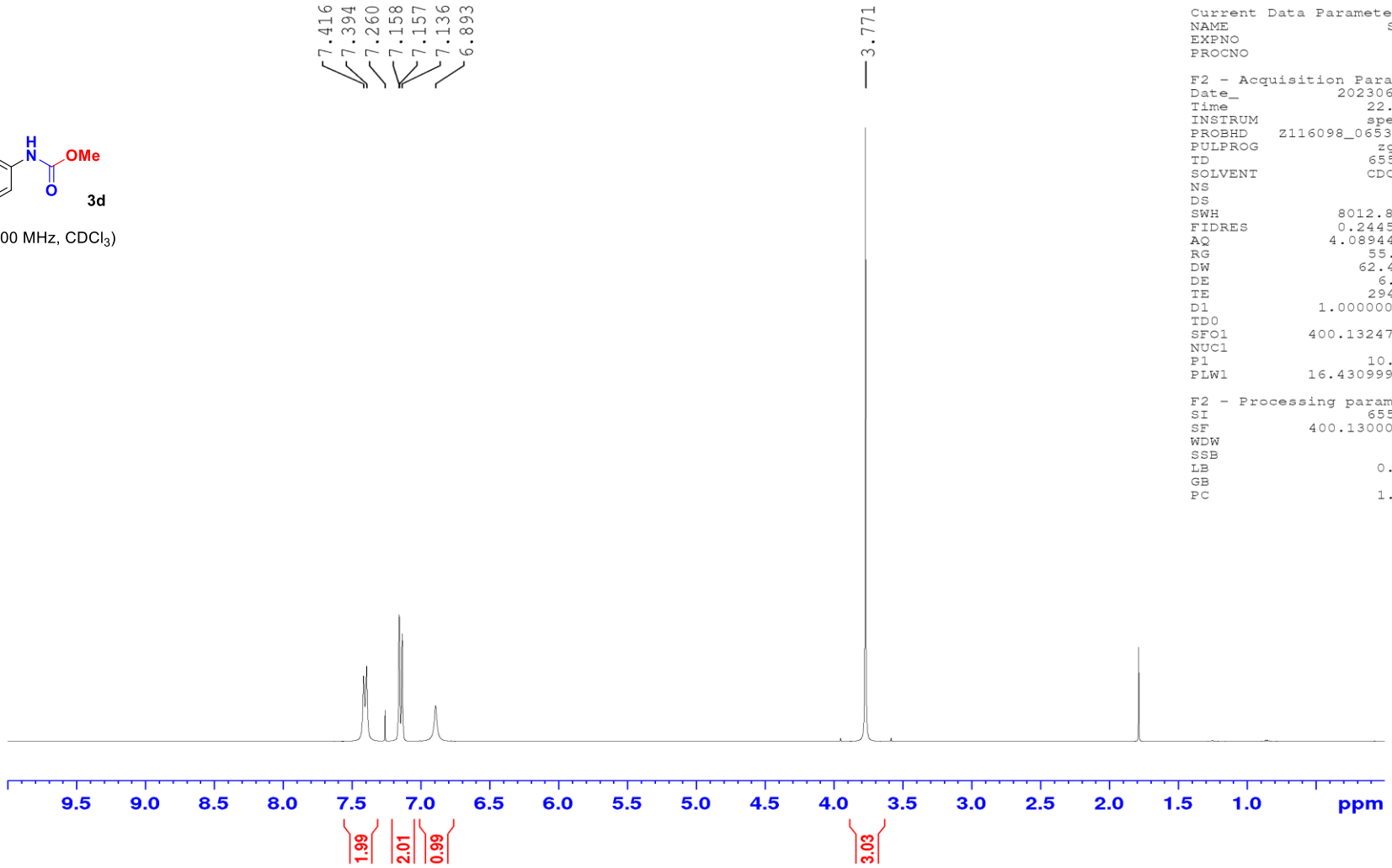


3d

^{1}H NMR (400 MHz, $CDCl_3$)

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Current Data Parameters
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EXPNO                       1
PROCNO                      1

F2 - Acquisition Parameters
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Time                 22.57 h
INSTRUM                spect
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PULPROG                zg30
TD                    65536
SOLVENT               CDCl3
NS                       16
DS                        2
SWH                8012.820 Hz
FIDRES            0.244532 Hz
AQ                4.0894465 sec
RG                     55.99
DW                   62.400 usec
DE                    6.50 usec
TE                   294.6 K
D1              1.00000000 sec
TD0                       1
SFO1           400.1324708 MHz
NUC1                     1H
P1                   10.00 usec
PLW1           16.43099976 W

F2 - Processing parameters
SI                    65536
SF             400.1300097 MHz
WDW                      EM
SSB                       0
LB                     0.30 Hz
GB                        0
PC                     1.00
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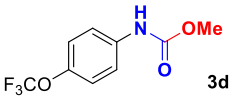
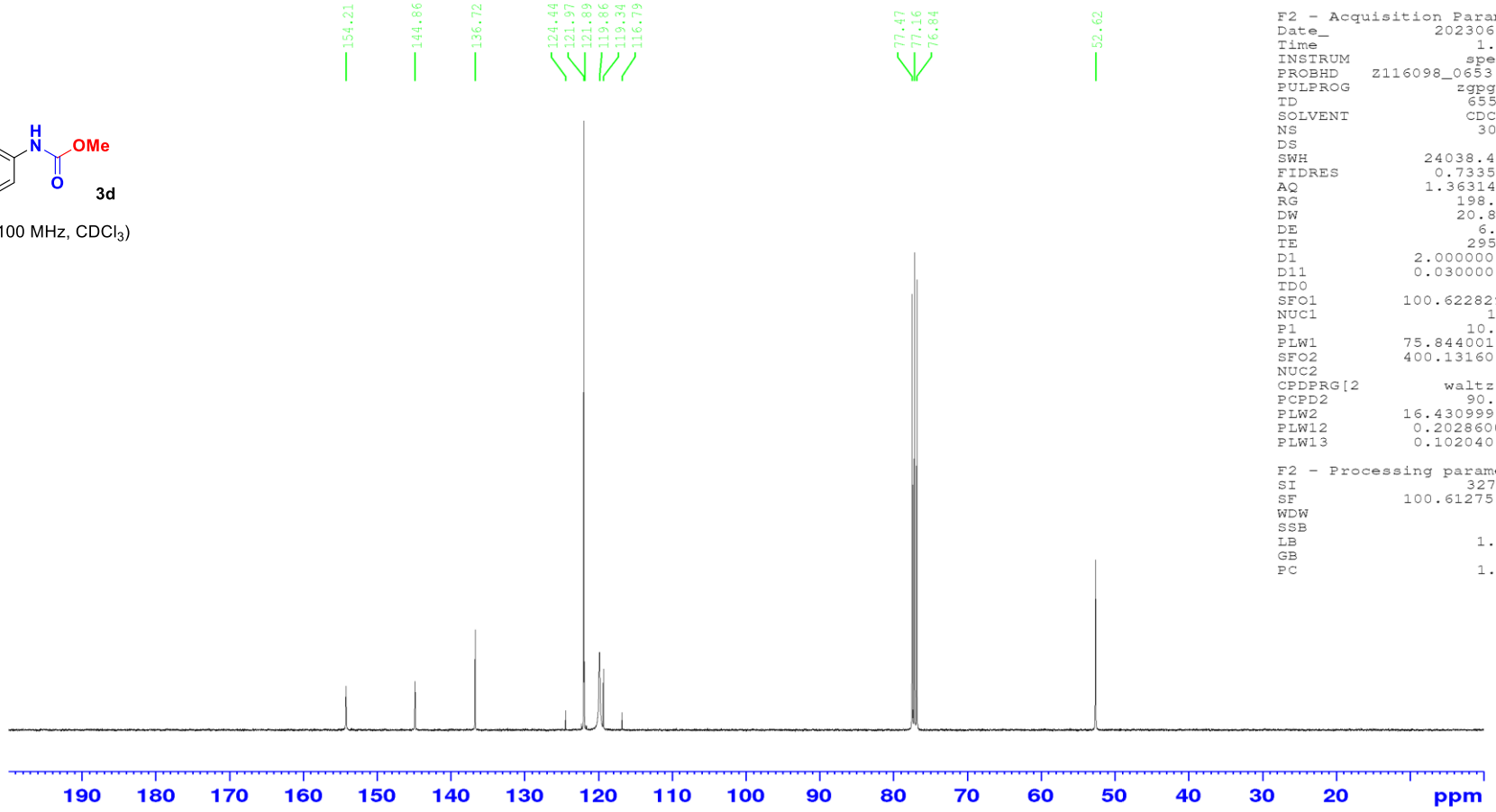


3d

^{13}C NMR (100 MHz, $CDCl_3$)

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Current Data Parameters
NAME                      S29
EXPNO                       2
PROCNO                      1

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Time                    1.50 h
INSTRUM                spect
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PULPROG              zgpg30
TD                    65536
SOLVENT               CDCl3
NS                     3000
DS                        4
SWH               24038.461 Hz
FIDRES             0.733596 Hz
AQ                 1.3631488 sec
RG                    198.36
DW                    20.800 usec
DE                     6.50 usec
TE                    295.4 K
D1              2.00000000 sec
D11             0.03000000 sec
TD0                       1
SFO1           100.6228298 MHz
NUC1                    13C
P1                    10.00 usec
PLW1           75.84400177 W
SFO2           400.1316005 MHz
NUC2                    1H
CPDPRG[2             waltz16
PCPD2                 90.00 usec
PLW2           16.43099976 W
PLW12           0.20286000 W
PLW13           0.10204000 W

F2 - Processing parameters
SI                    32768
SF             100.6127572 MHz
WDW                     EM
SSB                      0
LB                    1.00 Hz
GB                       0
PC                    1.40
```
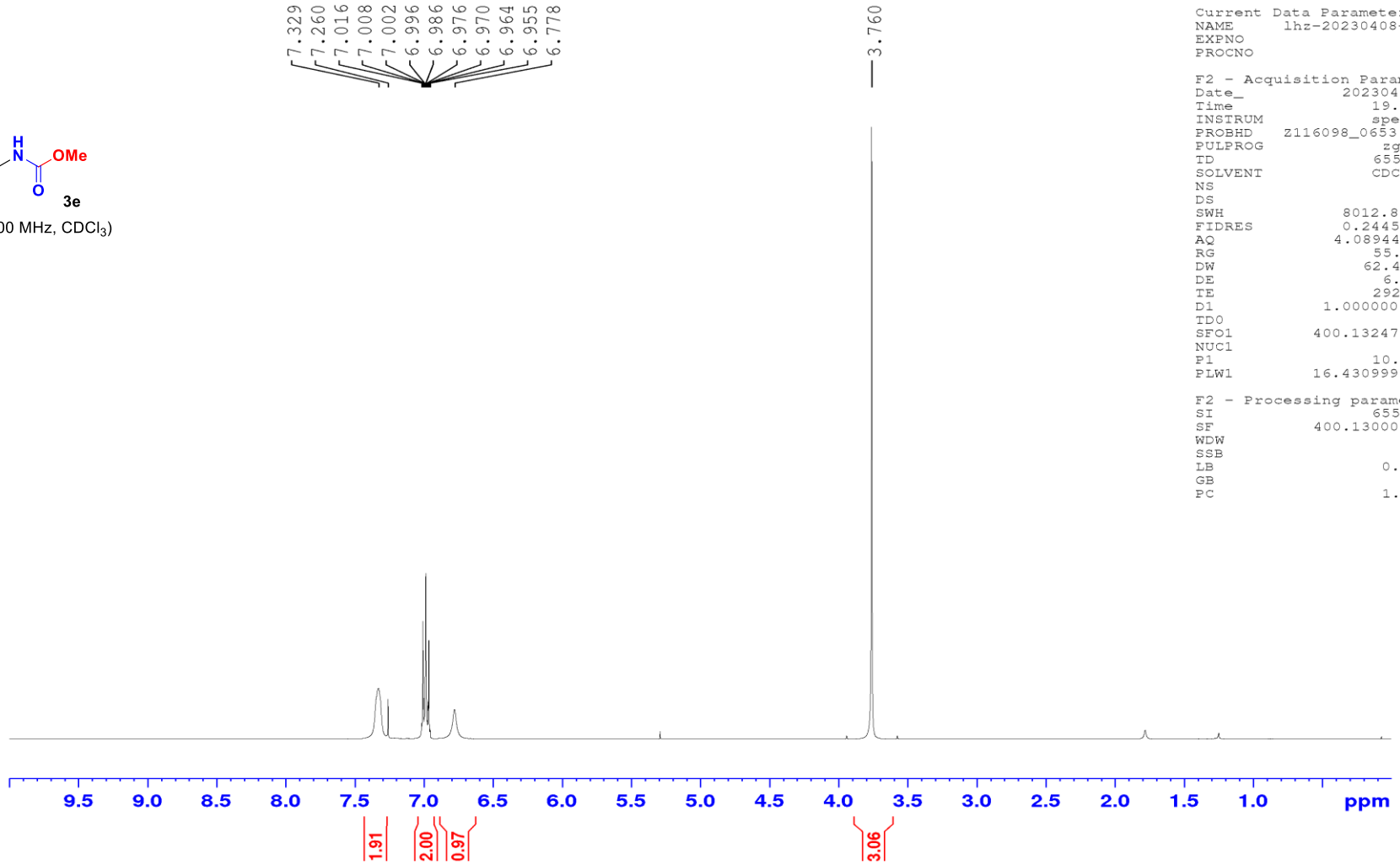
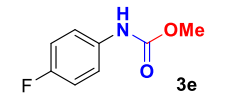

3e

^{1}H NMR (400 MHz, $CDCl_3$)

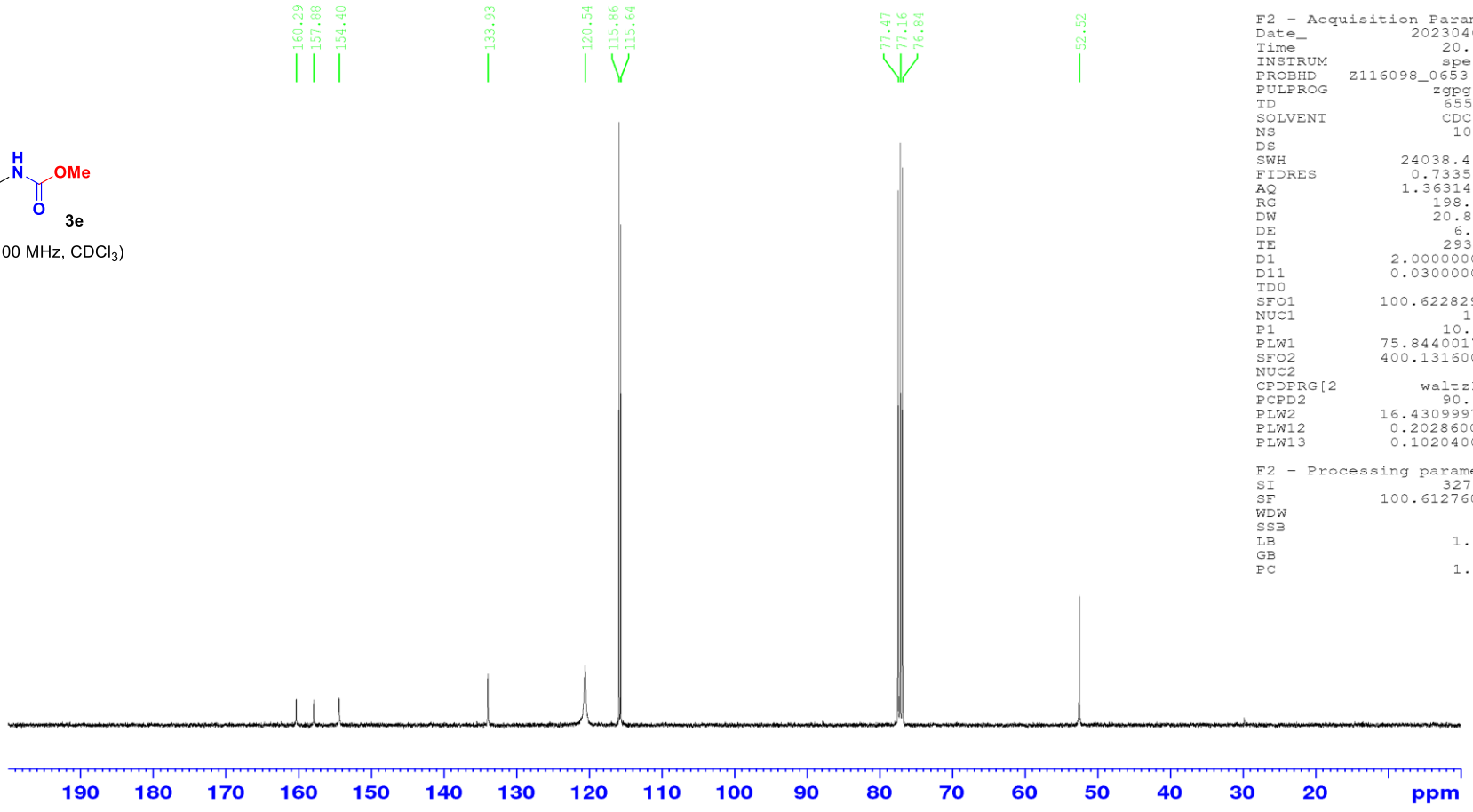



```
Current Data Parameters
NAME      1hz-20230408-3
EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
Date_            20230408
Time                19.45 h
INSTRUM              spect
PROBHD    Z116098_0653 (
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                     16
DS                      2
SWH              8012.820 Hz
FIDRES           0.244532 Hz
AQ               4.0894465 sec
RG                  55.99
DW                 62.400 usec
DE                  6.50 usec
TE                 292.4 K
D1              1.00000000 sec
TD0                     1
SFO1         400.1324708 MHz
NUC1                   1H
P1                 10.00 usec
PLW1         16.43099976 W

F2 - Processing parameters
SI                  65536
SF           400.1300098 MHz
WDW                    EM
SSB                     0
LB                   0.30 Hz
GB                      0
PC                   1.00
```

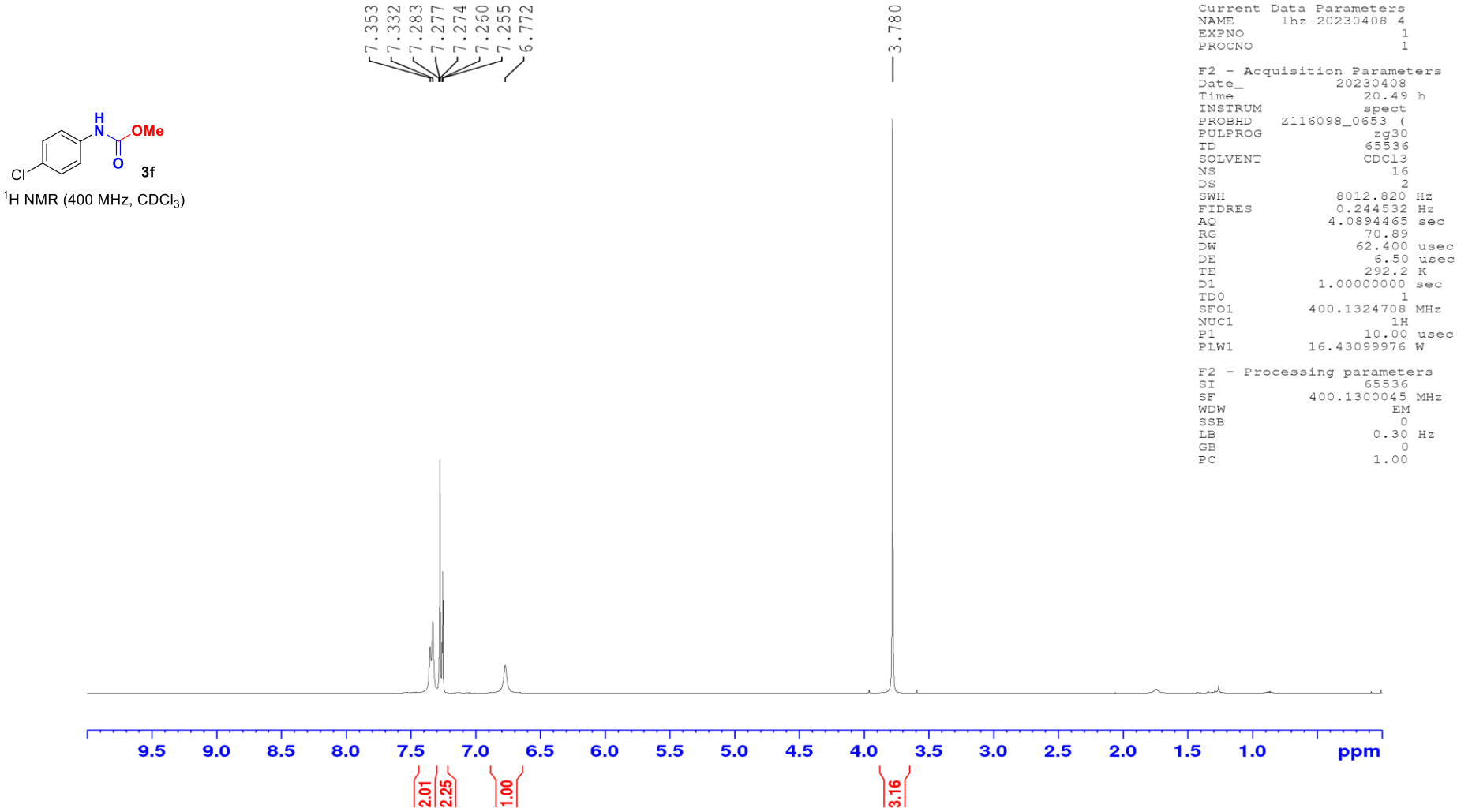


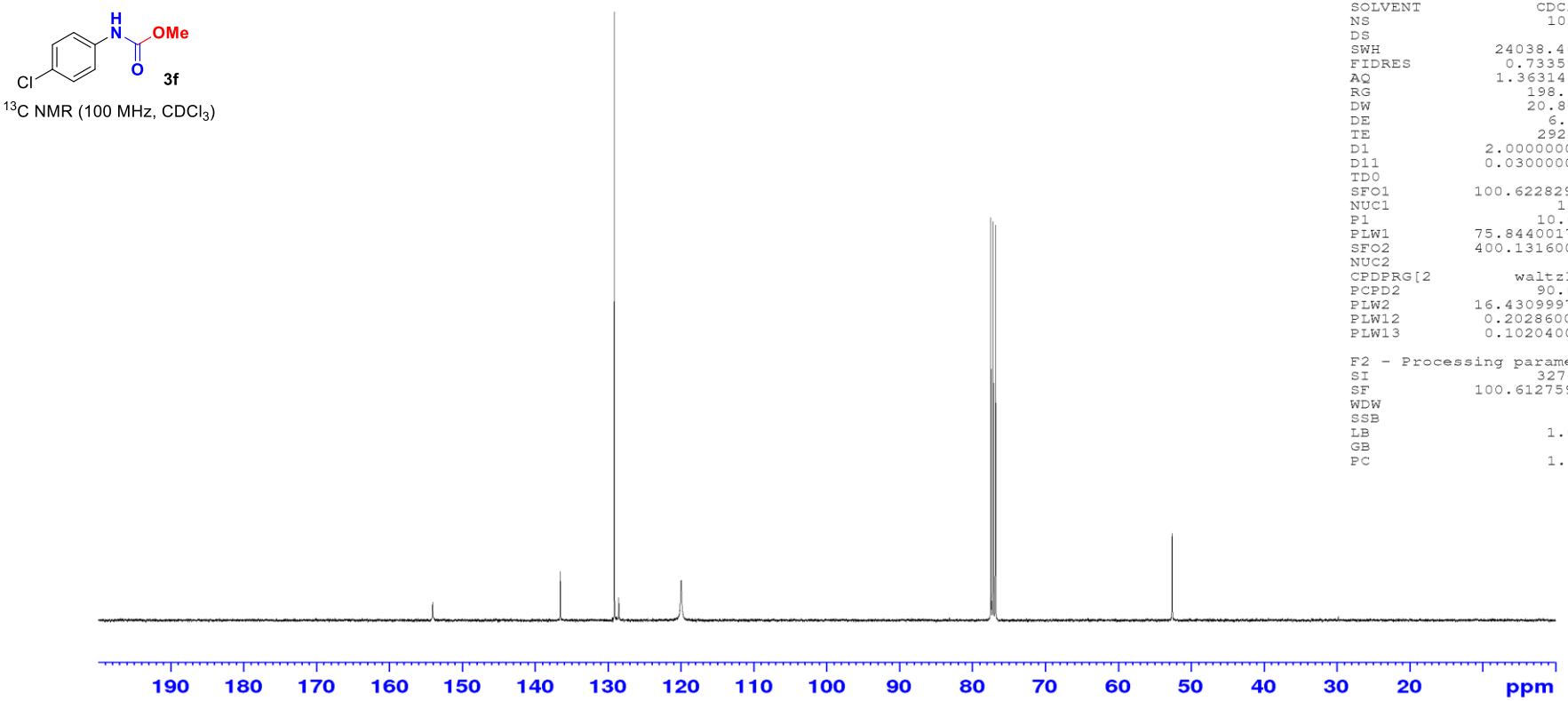
3e

^{13}C NMR (100 MHz, $CDCl_3$)




```
Current Data Parameters
NAME      lhz-20230408-3
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20230408
Time              20.44 h
INSTRUM             spect
PROBHD   Z116098_0653 (
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                 1024
DS                    4
SWH           24038.461 Hz
FIDRES         0.733596 Hz
AQ             1.3631488 sec
RG               198.36
DW               20.800 usec
DE                6.50 usec
TE                293.0 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1
SFO1        100.6228298 MHz
NUC1                13C
P1                10.00 usec
PLW1        75.84400177 W
SFO2        400.1316005 MHz
NUC2                1H
CPDPRG[2        waltz16
PCPD2             90.00 usec
PLW2        16.43099976 W
PLW12        0.20286000 W
PLW13        0.10204000 W

F2 - Processing parameters
SI                32768
SF          100.6127601 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```

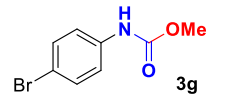

7.353
7.332
7.283
7.277
7.274
7.260
7.255
6.772
3.780
Current Data Parameters
NAME lhz-20230408-4
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20230408
Time 20.49 h
INSTRUM spect
PROBHD Z116098_0653 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.244532 Hz
AQ 4.0894465 sec
RG 70.89
DW 62.400 usec
DE 6.50 usec
TE 292.2 K
D1 1.00000000 sec
TD0 1
SFO1 400.1324708 MHz
NUC1 1H
P1 10.00 usec
PLW1 16.43099976 W
F2 - Processing parameters
SI 65536
SF 400.1300045 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
H
N
OMe
O
Cl
3f
1H NMR (400 MHz, CDCl3)
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
ppm
2.01
2.25
1.00
3.16



3f

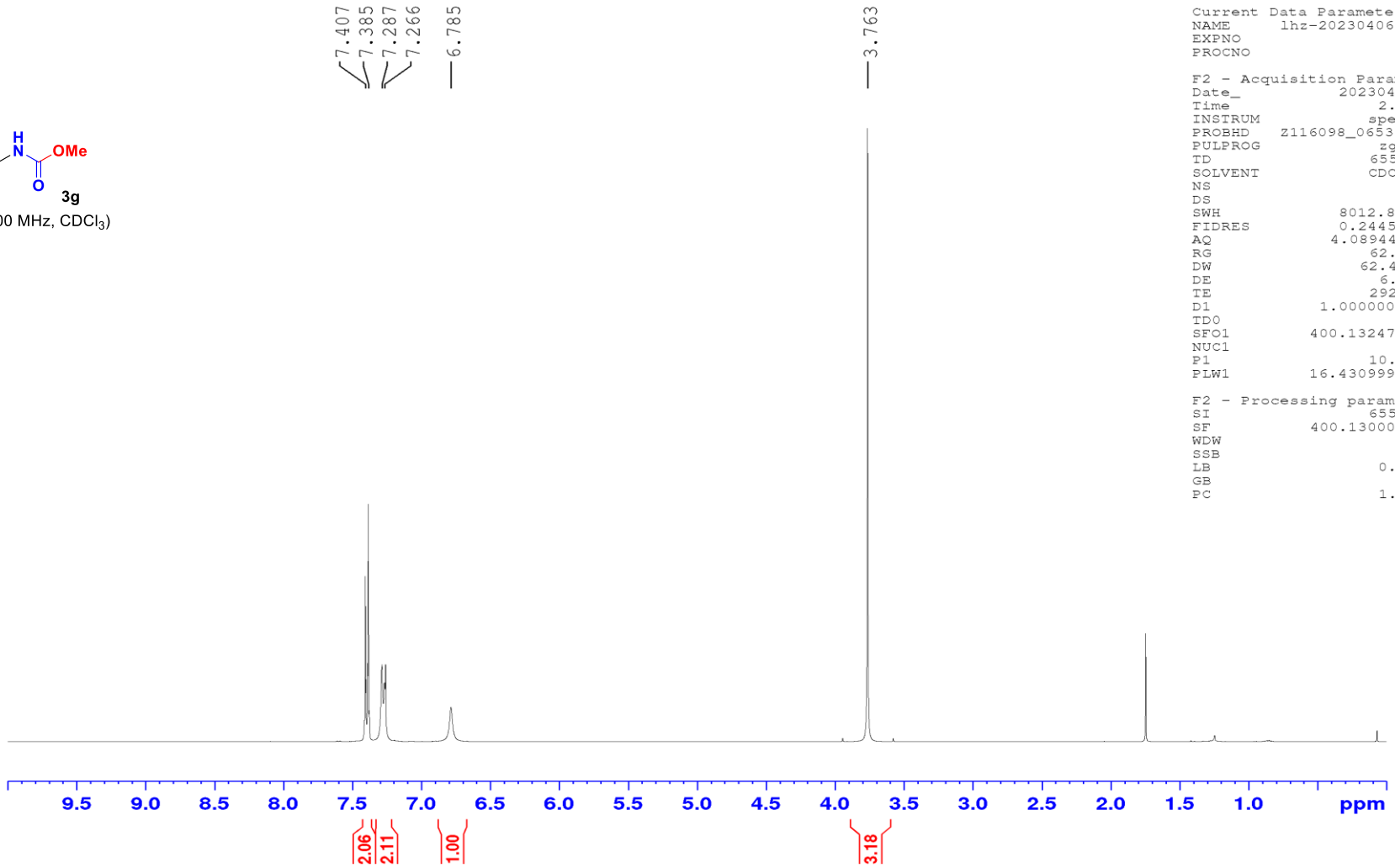
^{13}C NMR (100 MHz, $CDCl_3$)

```
Current Data Parameters
NAME      lhz-20230408-4
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20230408
Time              21.48 h
INSTRUM             spect
PROBHD   Z116098_0653 (
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                 1024
DS                    4
SWH           24038.461 Hz
FIDRES         0.733596 Hz
AQ            1.3631488 sec
RG               198.36
DW               20.800 usec
DE                6.50 usec
TE               292.6 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                   1
SFO1        100.6228298 MHz
NUC1                13C
P1                10.00 usec
PLW1         75.84400177 W
SFO2        400.1316005 MHz
NUC2                1H
CPDPRG[2        waltz16
PCPD2             90.00 usec
PLW2         16.43099976 W
PLW12         0.20286000 W
PLW13         0.10204000 W

F2 - Processing parameters
SI                32768
SF          100.6127594 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```

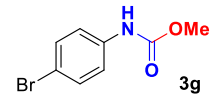


3g

^{1}H NMR (400 MHz, $CDCl_3$)

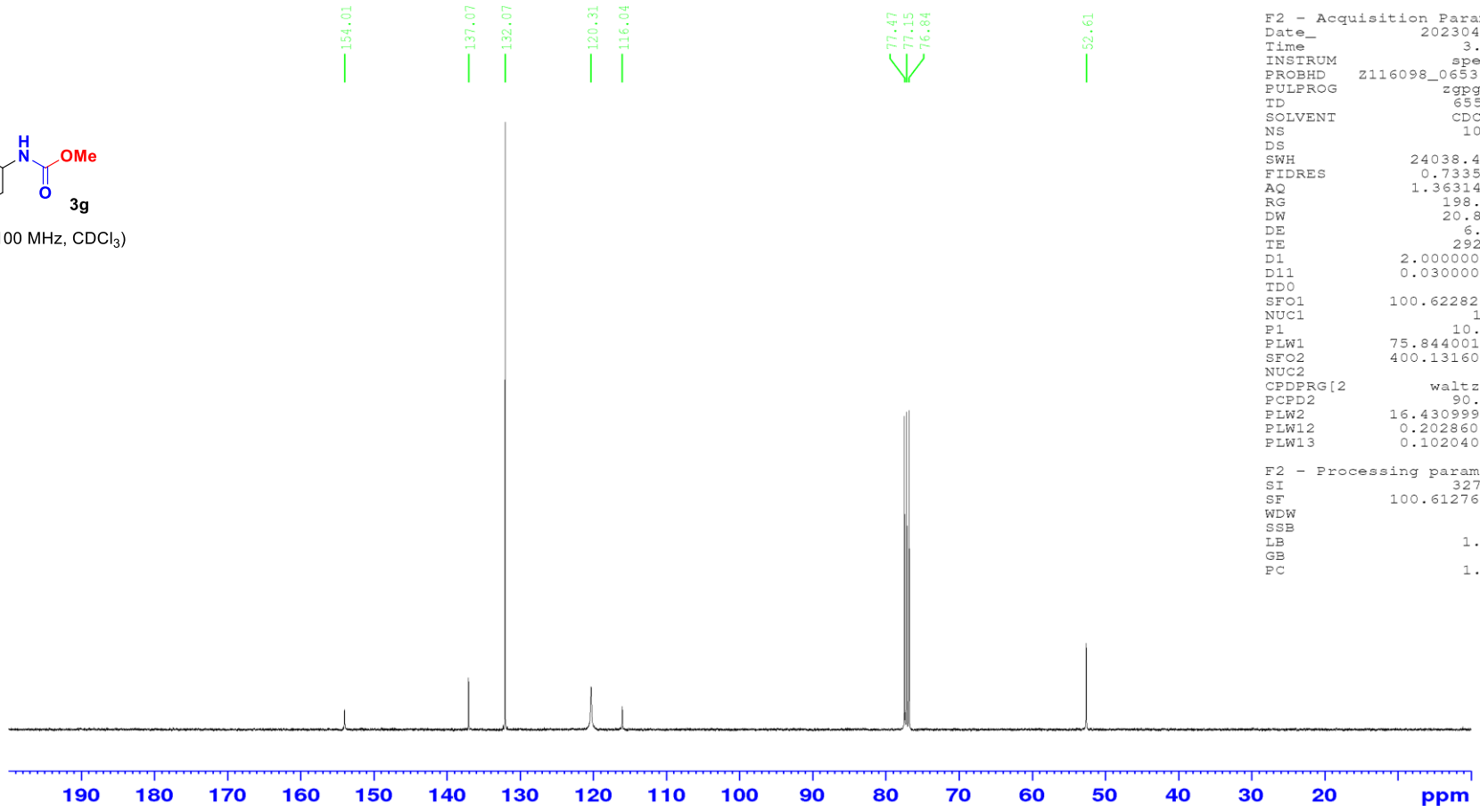



```
Current Data Parameters
NAME      lhz-20230406-6
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20230407
Time              2.54 h
INSTRUM           spect
PROBHD   Z116098_0653 (
PULPROG           zg30
TD               65536
SOLVENT          CDCl3
NS                  16
DS                   2
SWH           8012.820 Hz
FIDRES        0.244532 Hz
AQ            4.0894465 sec
RG               62.98
DW              62.400 usec
DE               6.50 usec
TE              292.0 K
D1            1.00000000 sec
TD0                  1
SFO1        400.1324708 MHz
NUC1               1H
P1               10.00 usec
PLW1        16.43099976 W

F2 - Processing parameters
SI               65536
SF          400.1300099 MHz
WDW                EM
SSB                 0
LB               0.30 Hz
GB                  0
PC               1.00
```

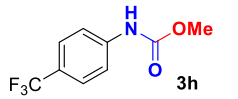


3g

^{13}C NMR (100 MHz, $CDCl_3$)

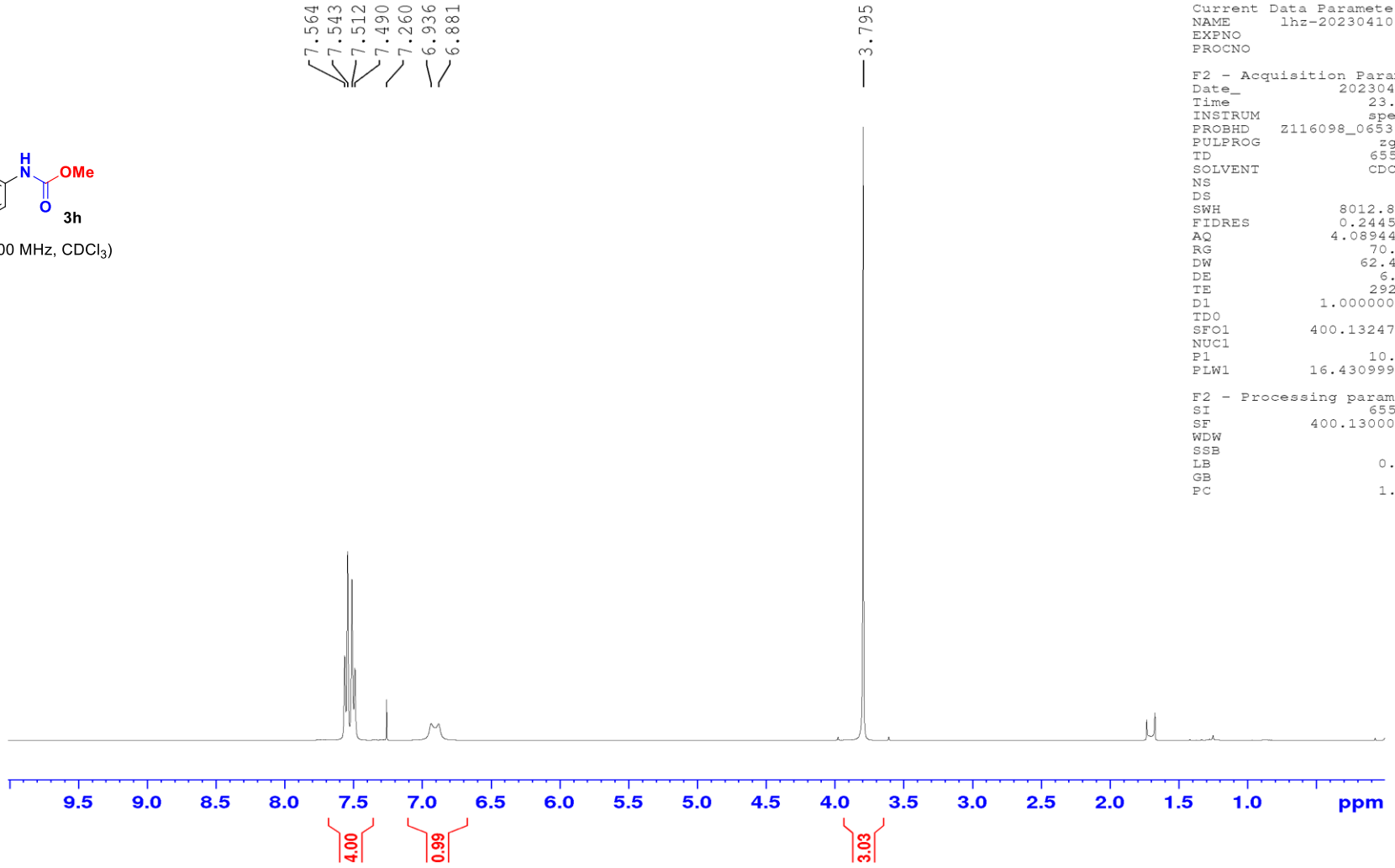



```
Current Data Parameters
NAME      lhz-20230406-6
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20230407
Time               3.53 h
INSTRUM           spect
PROBHD    Z116098_0653 (
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                 1024
DS                    4
SWH           24038.461 Hz
FIDRES         0.733596 Hz
AQ            1.3631488 sec
RG               198.36
DW               20.800 usec
DE                6.50 usec
TE                292.8 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1
SFO1        100.6228298 MHz
NUC1               13C
P1               10.00 usec
PLW1        75.84400177 W
SFO2        400.1316005 MHz
NUC2               1H
CPDPRG[2        waltz16
PCPD2            90.00 usec
PLW2        16.43099976 W
PLW12        0.20286000 W
PLW13        0.10204000 W

F2 - Processing parameters
SI                32768
SF          100.6127608 MHz
WDW                EM
SSB                 0
LB               1.00 Hz
GB                  0
PC               1.40
```

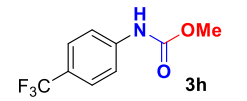


3h

^{1}H NMR (400 MHz, $CDCl_3$)

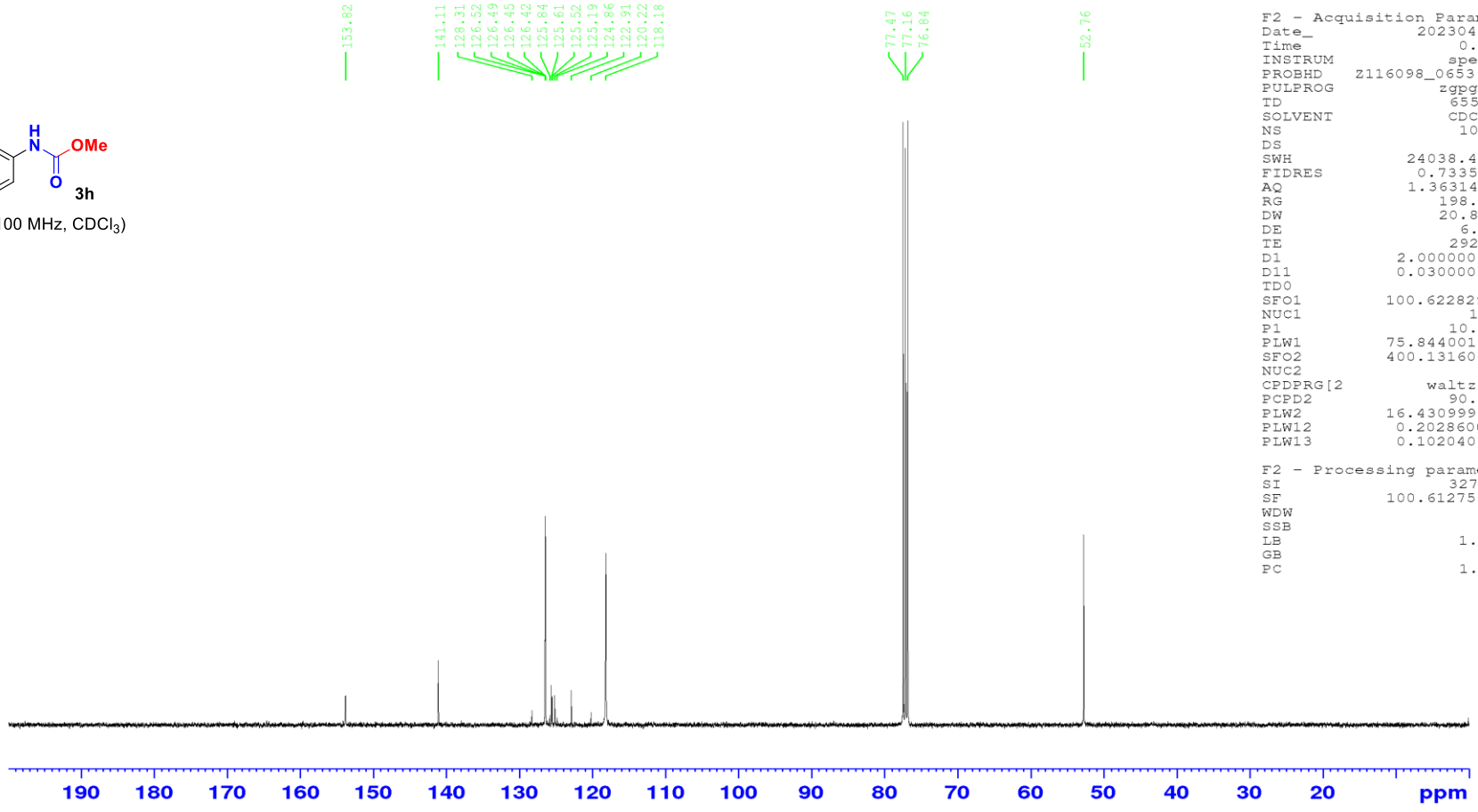



```
Current Data Parameters
NAME      lhz-20230410-3
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20230410
Time              23.37 h
INSTRUM            spect
PROBHD    Z116098_0653 (
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                   16
DS                    2
SWH            8012.820 Hz
FIDRES         0.244532 Hz
AQ             4.0894465 sec
RG                70.89
DW               62.400 usec
DE                6.50 usec
TE               292.1 K
D1            1.00000000 sec
TD0                   1
SFO1         400.1324708 MHz
NUC1                 1H
P1                10.00 usec
PLW1         16.43099976 W

F2 - Processing parameters
SI                65536
SF           400.1300099 MHz
WDW                  EM
SSB                   0
LB                 0.30 Hz
GB                    0
PC                 1.00
```

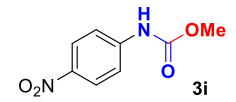


3h

^{13}C NMR (100 MHz, $CDCl_3$)

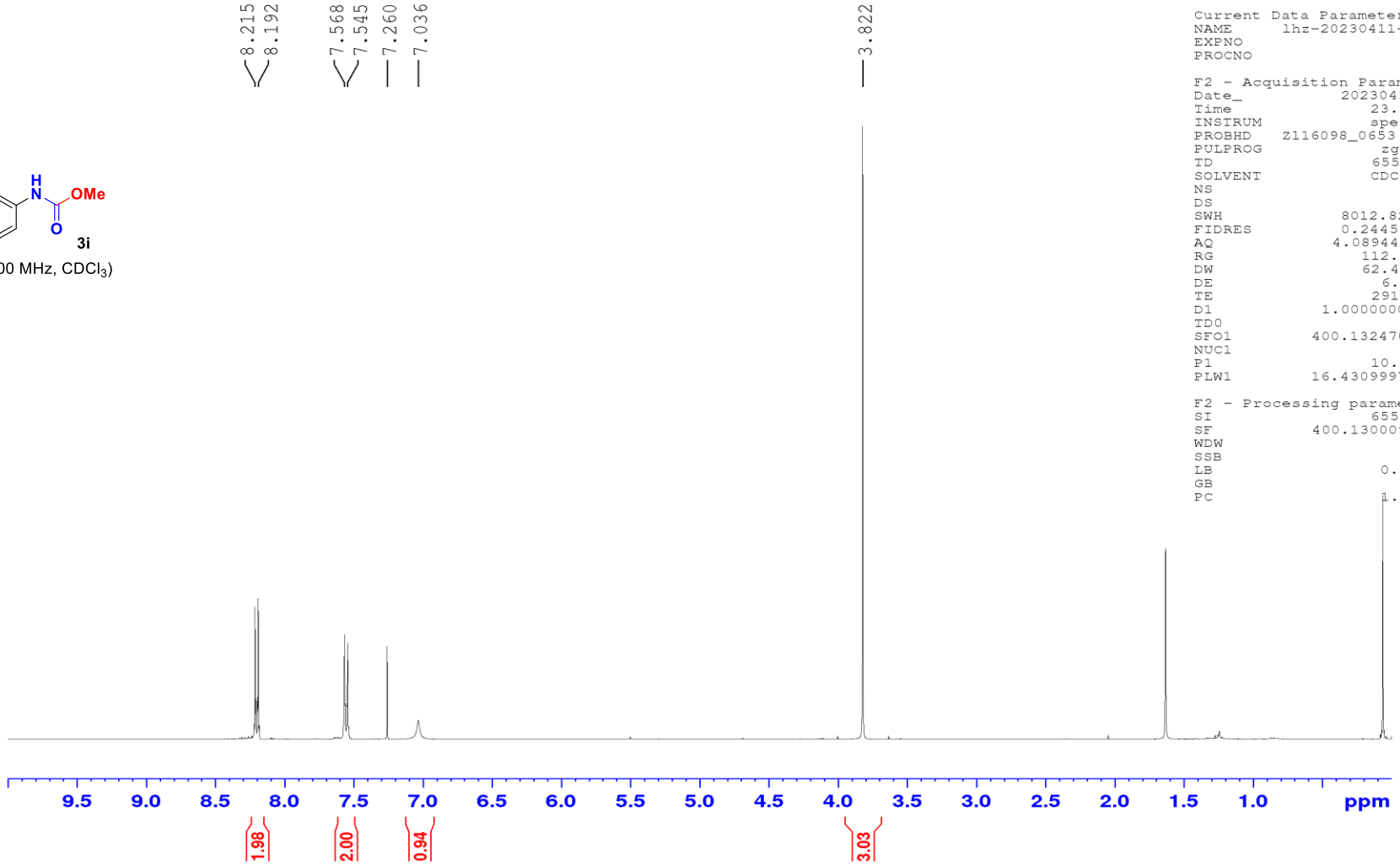



```
Current Data Parameters
NAME      1hz-20230410-3
EXPNO                   2
PROCNO                  1

F2 - Acquisition Parameters
Date_            20230411
Time                0.36 h
INSTRUM            spect
PROBHD    Z116098_0653 (
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                 1024
DS                    4
SWH           24038.461 Hz
FIDRES         0.733596 Hz
AQ             1.3631488 sec
RG                198.36
DW                20.800 usec
DE                 6.50 usec
TE                292.4 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1
SFO1        100.6228298 MHz
NUC1               13C
P1               10.00 usec
PLW1        75.84400177 W
SFO2        400.1316005 MHz
NUC2               1H
CPDPRG[2       waltz16
PCPD2            90.00 usec
PLW2        16.43099976 W
PLW12        0.20286000 W
PLW13        0.10204000 W

F2 - Processing parameters
SI                32768
SF          100.6127575 MHz
WDW                EM
SSB                 0
LB               1.00 Hz
GB                  0
PC               1.40
```

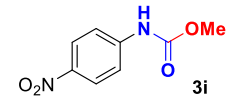


3i

^{1}H NMR (400 MHz, $CDCl_3$)

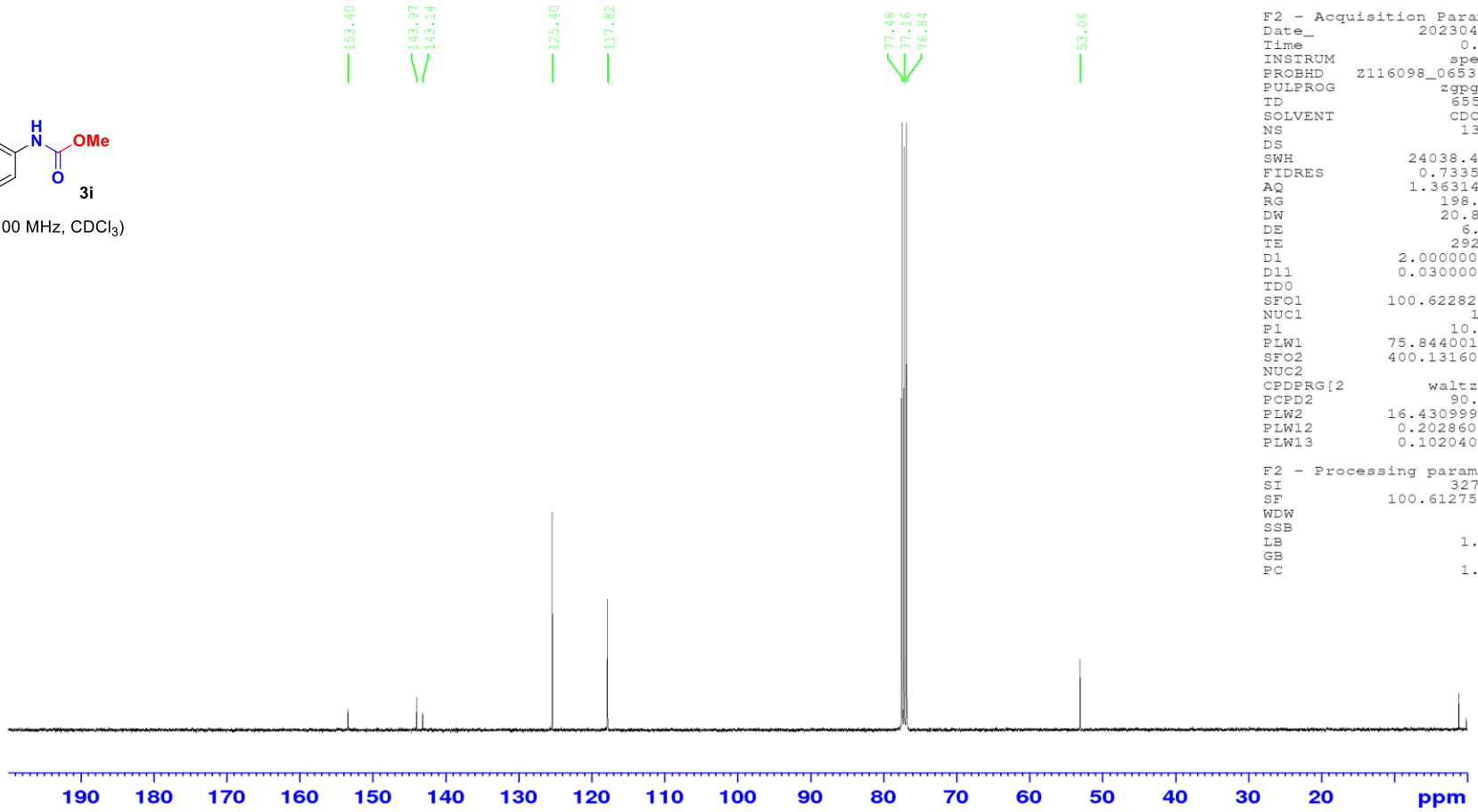



```
Current Data Parameters
NAME      lhz-20230411-1
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20230411
Time              23.33 h
INSTRUM             spect
PROBHD   Z116098_0653 (
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             8012.820 Hz
FIDRES          0.244532 Hz
AQ              4.0894465 sec
RG                 112.15
DW                62.400 usec
DE                 6.50 usec
TE                291.8 K
D1             1.00000000 sec
TD0                    1
SFO1          400.1324708 MHz
NUC1                  1H
P1                10.00 usec
PLW1          16.43099976 W

F2 - Processing parameters
SI                 65536
SF            400.1300099 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```

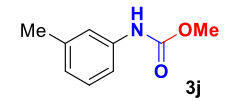


3i

^{13}C NMR (100 MHz, $CDCl_3$)

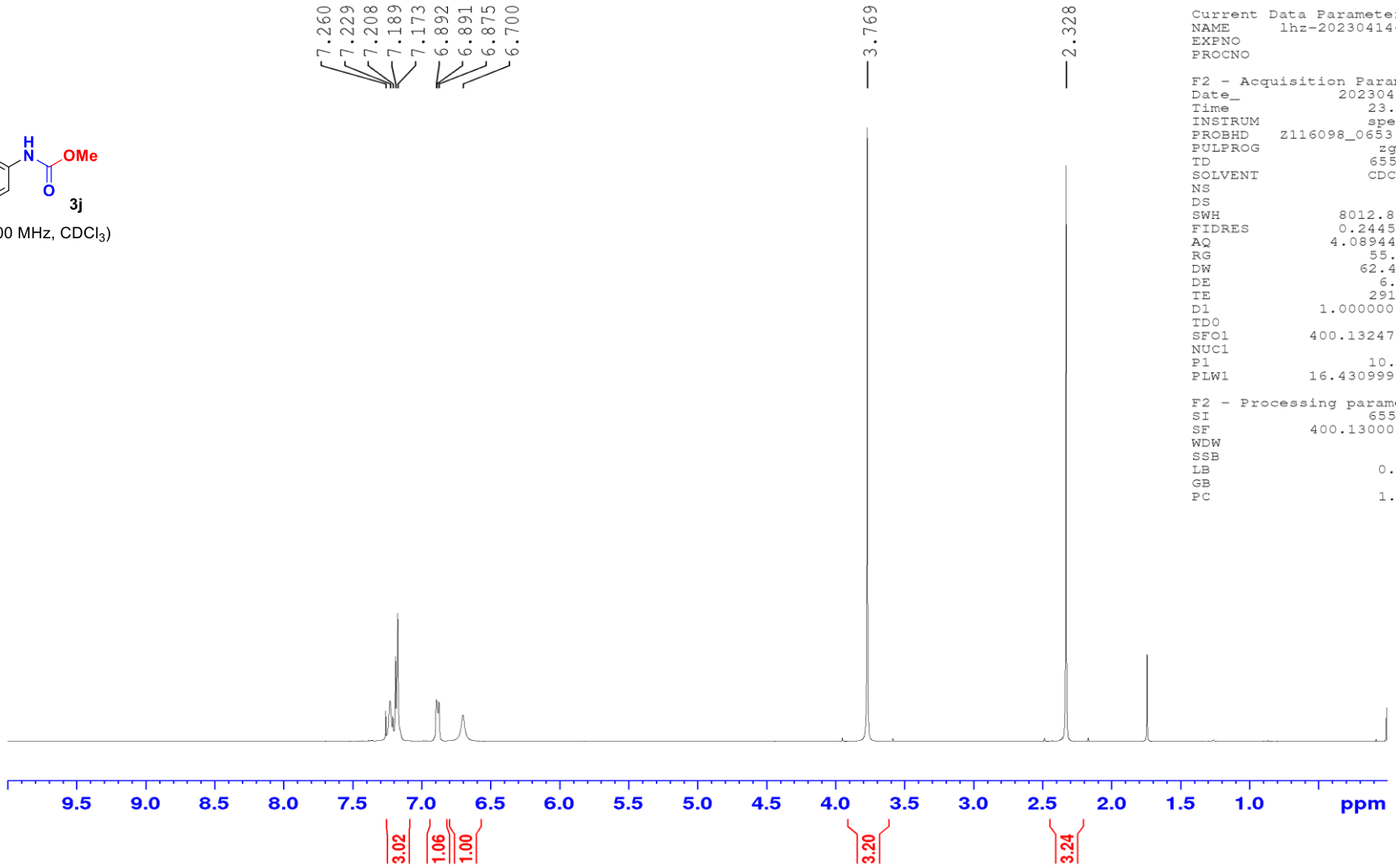



```
Current Data Parameters
NAME      lhz-20230411-1
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20230412
Time              0.51 h
INSTRUM           spect
PROBHD   Z116098_0653 (
PULPROG         zgpg30
TD               65536
SOLVENT          CDCl3
NS                1333
DS                   4
SWH          24038.461 Hz
FIDRES        0.733596 Hz
AQ           1.3631488 sec
RG              198.36
DW              20.800 usec
DE               6.50 usec
TE              292.4 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                  1
SFO1        100.6228298 MHz
NUC1                13C
P1                10.00 usec
PLW1        75.84400177 W
SFO2        400.1316005 MHz
NUC2                1H
CPDPRG[2        waltz16
PCPD2             90.00 usec
PLW2        16.43099976 W
PLW12        0.20286000 W
PLW13        0.10204000 W

F2 - Processing parameters
SI                32768
SF          100.6127572 MHz
WDW                 EM
SSB                  0
LB                1.00 Hz
GB                   0
PC                1.40
```

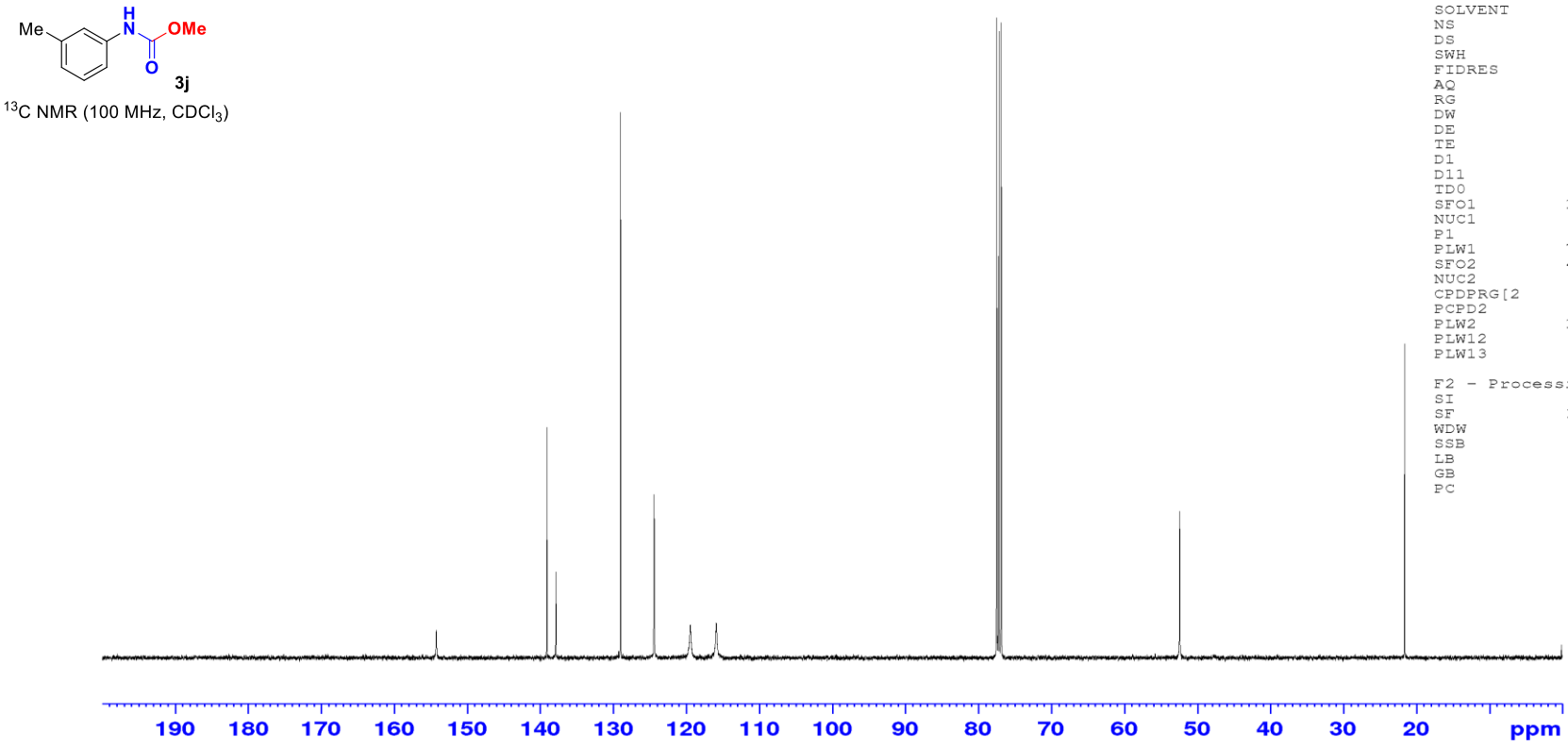


3j

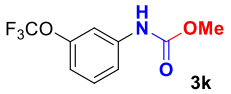
^{1}H NMR (400 MHz, $CDCl_3$)




```
Current Data Parameters
NAME      1hz-20230414-2
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20230414
Time              23.29 h
INSTRUM             spect
PROBHD    Z116098_0653 (
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             8012.820 Hz
FIDRES          0.244532 Hz
AQ              4.0894465 sec
RG                 55.99
DW                62.400 usec
DE                 6.50 usec
TE                291.8 K
D1             1.00000000 sec
TD0                    1
SFO1          400.1324708 MHz
NUC1                  1H
P1                 10.00 usec
PLW1          16.43099976 W

F2 - Processing parameters
SI                 65536
SF            400.1300098 MHz
WDW                   EM
SSB                    0
LB                0.30 Hz
GB                     0
PC                1.00
```

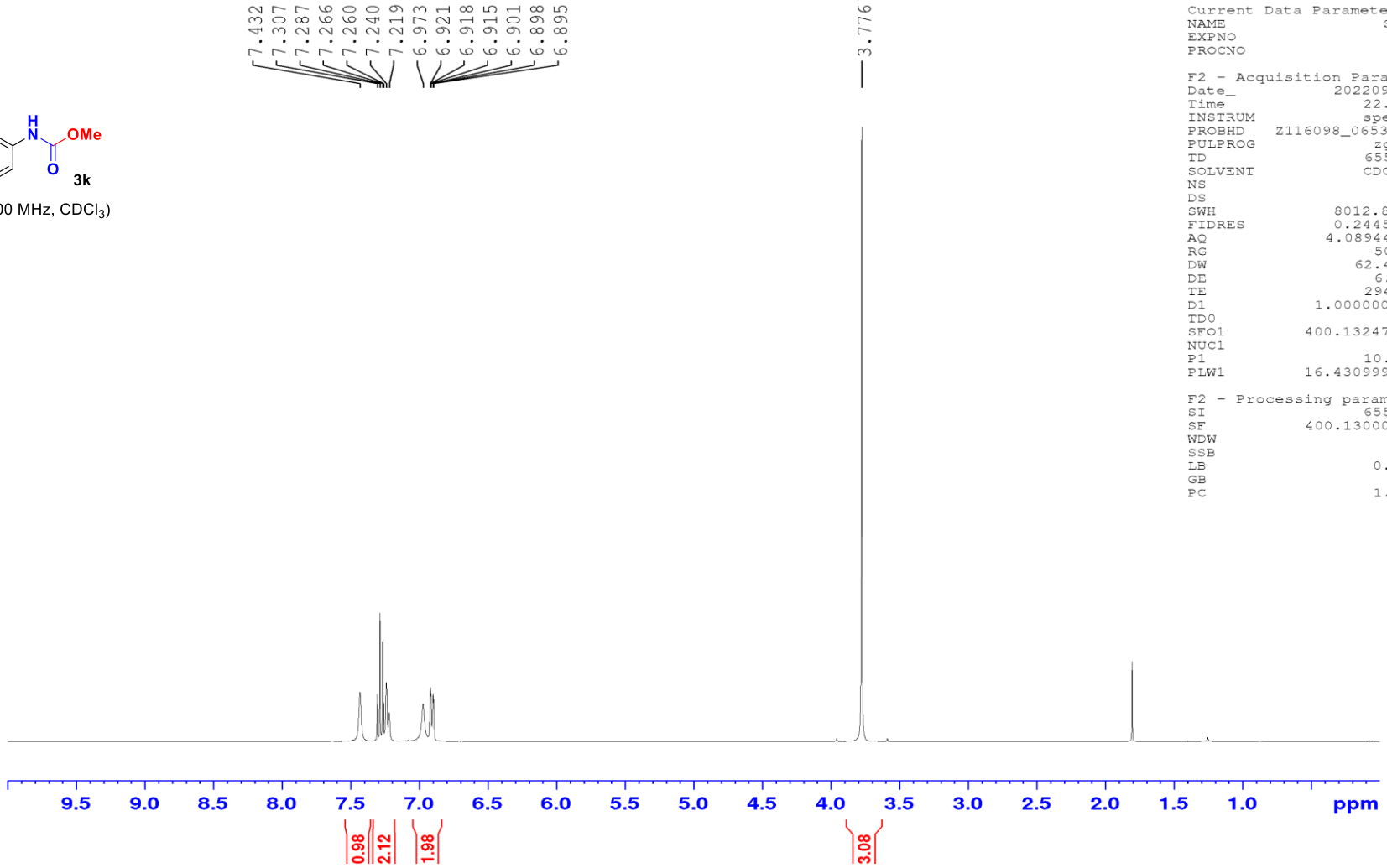



```
Current Data Parameters
NAME      1hz-20230414-2
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20230415
Time               0.29 h
INSTRUM           spect
PROBHD    Z116098_0653 (
PULPROG         zgpg30
TD               65536
SOLVENT          CDCl3
NS                1024
DS                   4
SWH          24038.461 Hz
FIDRES        0.733596 Hz
AQ            1.3631488 sec
RG               198.36
DW               20.800 usec
DE                6.50 usec
TE               292.5 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                  1
SFO1       100.6228298 MHz
NUC1              13C
P1               10.00 usec
PLW1       75.84400177 W
SFO2       400.1316005 MHz
NUC2              1H
CPDPRG[2       waltz16
PCPD2            90.00 usec
PLW2       16.43099976 W
PLW12       0.20286000 W
PLW13       0.10204000 W

F2 - Processing parameters
SI               32768
SF         100.6127608 MHz
WDW                EM
SSB                 0
LB              1.00 Hz
GB                  0
PC              1.40
```

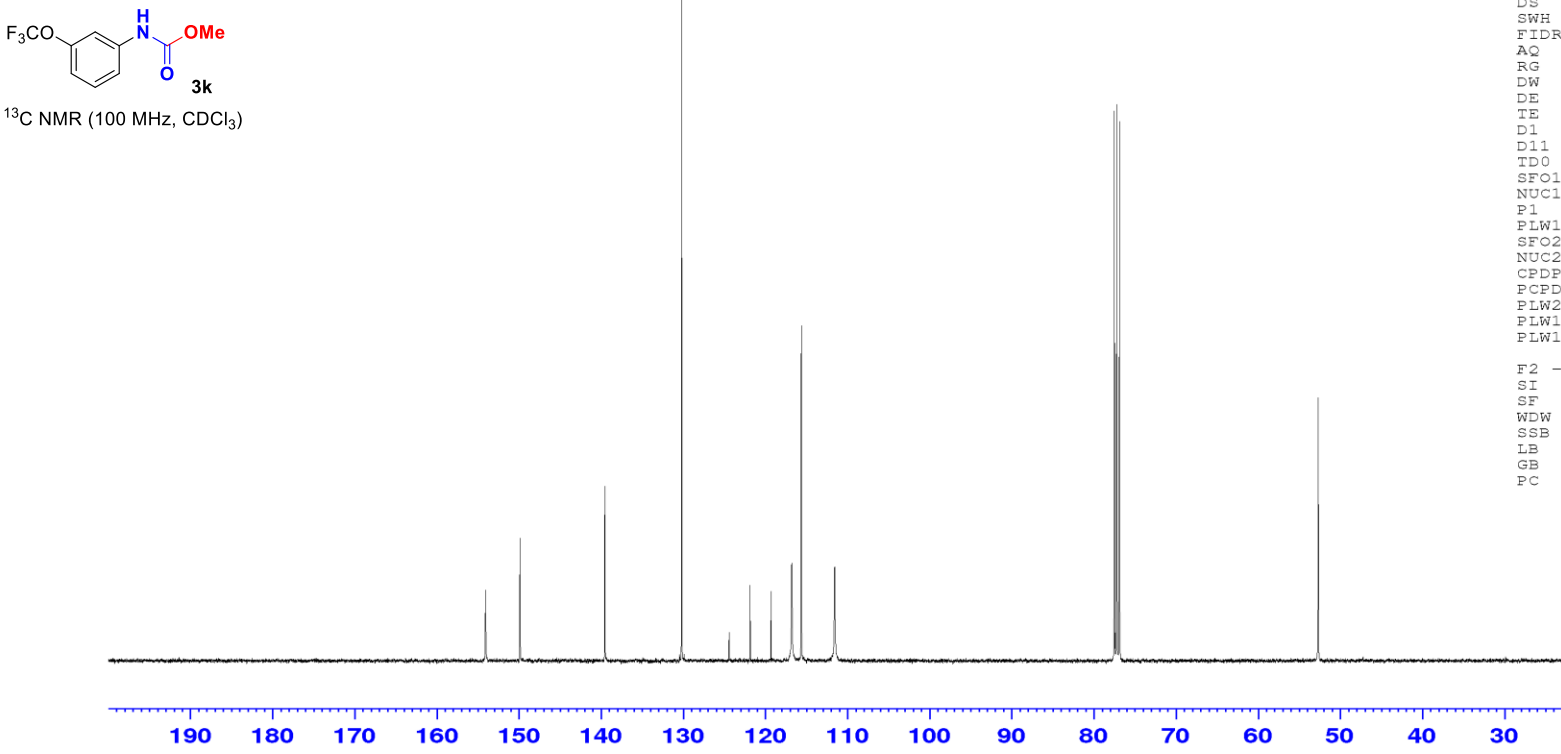


3k

^{1}H NMR (400 MHz, $CDCl_3$)



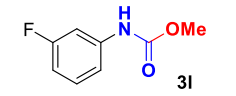

```
Current Data Parameters
NAME                      S12
EXPNO                       1
PROCNO                      1

F2 - Acquisition Parameters
Date_              20220922
Time                  22.29 h
INSTRUM               spect
PROBHD    Z116098_0653 (
PULPROG                zg30
TD                    65536
SOLVENT               CDCl3
NS                        8
DS                        2
SWH                8012.820 Hz
FIDRES            0.244532 Hz
AQ                4.0894465 sec
RG                     50.3
DW                   62.400 usec
DE                    6.50 usec
TE                   294.4 K
D1                1.00000000 sec
TD0                       1
SFO1           400.1324708 MHz
NUC1                    1H
P1                   10.00 usec
PLW1           16.43099976 W

F2 - Processing parameters
SI                    65536
SF             400.1300098 MHz
WDW                     EM
SSB                      0
LB                    0.30 Hz
GB                       0
PC                    1.00
```



3k

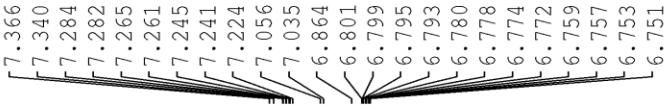
^{13}C NMR (100 MHz, $CDCl_3$)

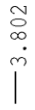
```
Current Data Parameters
NAME                   S12
EXPNO                    2
PROCNO                   1

F2 - Acquisition Parameters
Date_               20220922
Time                  23.47 h
INSTRUM                spect
PROBHD    Z116098_0653 (
PULPROG             zgpg30
TD                   65536
SOLVENT              CDCl3
NS                    1333
DS                       4
SWH             24038.461 Hz
FIDRES          0.733596 Hz
AQ              1.3631488 sec
RG                  198.36
DW                  20.800 usec
DE                    6.50 usec
TE                   294.9 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                      1
SFO1          100.6228298 MHz
NUC1                   13C
P1                   10.00 usec
PLW1          75.84400177 W
SFO2          400.1316005 MHz
NUC2                   1H
CPDPRG[2          waltz16
PCPD2                90.00 usec
PLW2          16.43099976 W
PLW12          0.20286000 W
PLW13          0.10204000 W

F2 - Processing parameters
SI                   32768
SF            100.6127571 MHz
WDW                     EM
SSB                      0
LB                    1.00 Hz
GB                       0
PC                    1.40
```



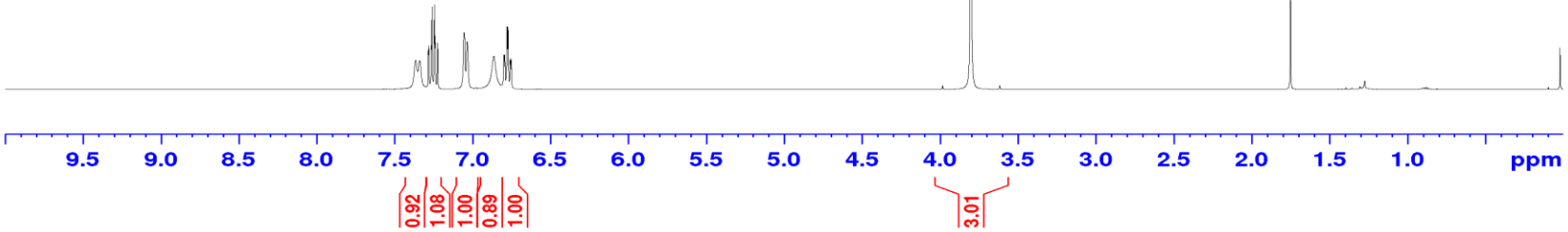
3l

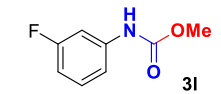
^{1}H NMR (400 MHz, $CDCl_3$)





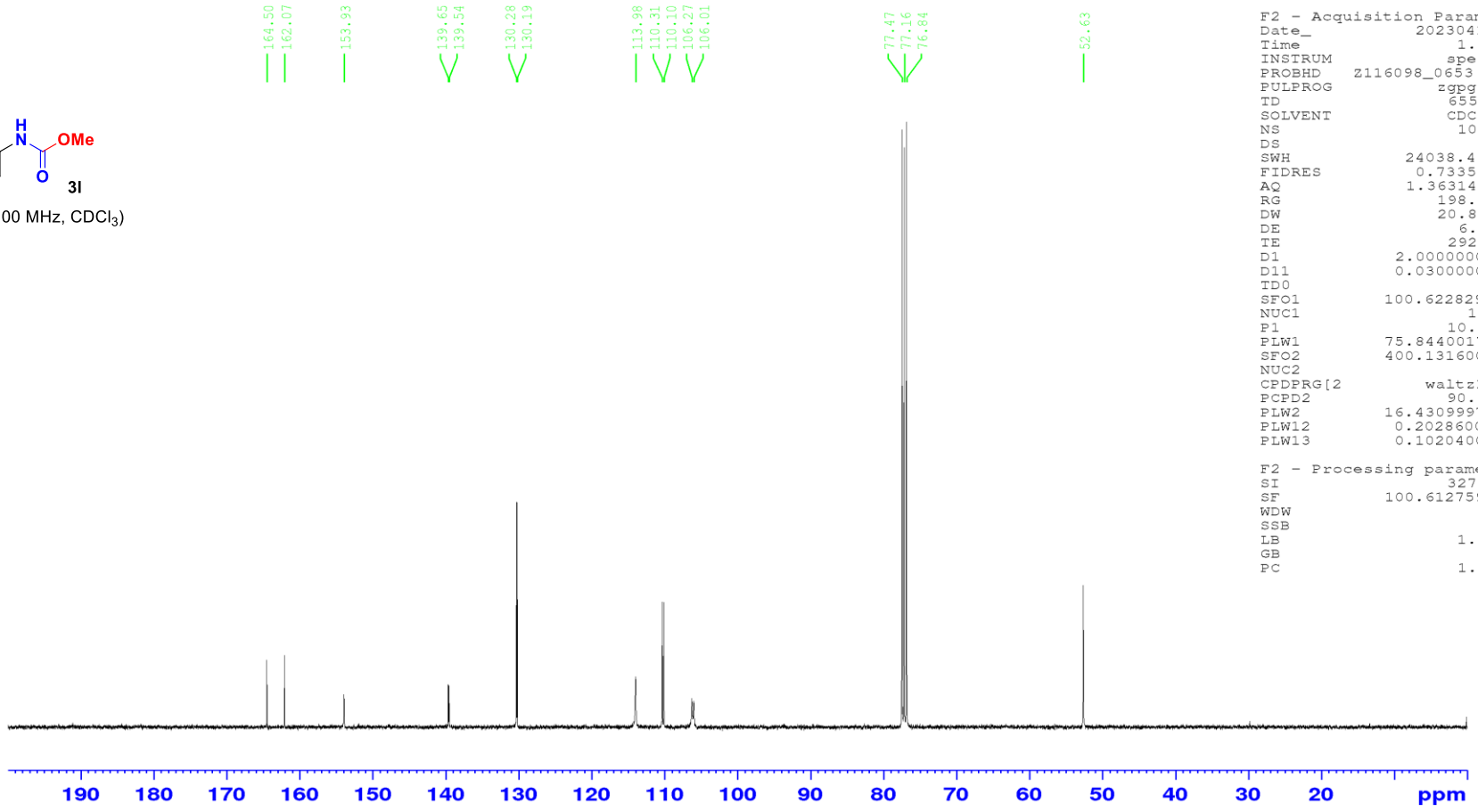

```
Current Data Parameters
NAME      1hz-20230410-4
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20230411
Time              0.40 h
INSTRUM           spect
PROBHD    Z116098_0653 (
PULPROG           zg30
TD               65536
SOLVENT          CDCl3
NS                  16
DS                   2
SWH           8012.820 Hz
FIDRES        0.244532 Hz
AQ            4.0894465 sec
RG               62.98
DW              62.400 usec
DE               6.50 usec
TE              292.3 K
D1            1.00000000 sec
TD0                  1
SFO1        400.1324708 MHz
NUC1                1H
P1                10.00 usec
PLW1        16.43099976 W

F2 - Processing parameters
SI               65536
SF          400.1300000 MHz
WDW                 EM
SSB                  0
LB                0.30 Hz
GB                   0
PC                1.00
```





3l

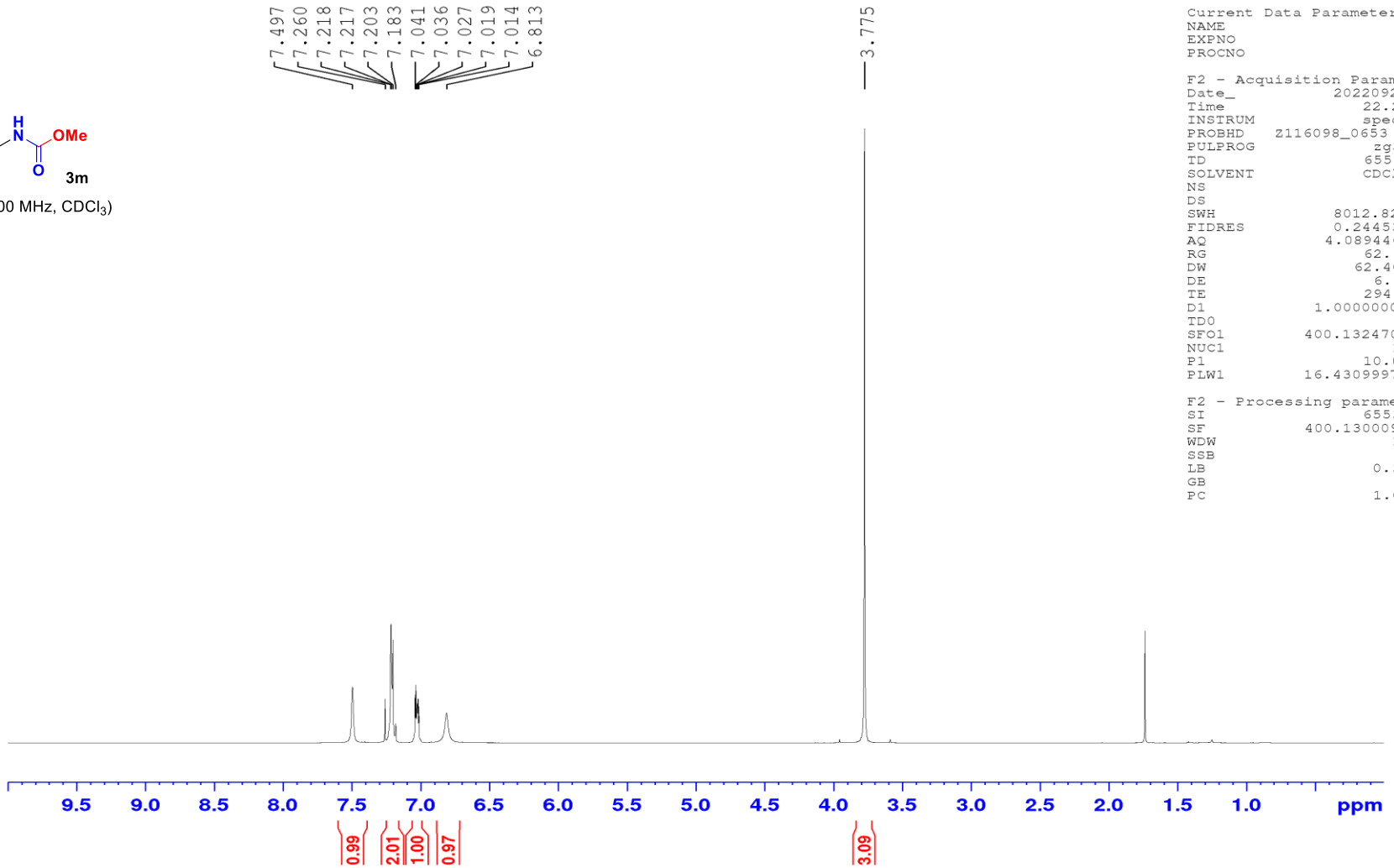
^{13}C NMR (100 MHz, $CDCl_3$)



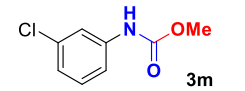

```
Current Data Parameters
NAME      lhz-20230410-4
EXPNO                   2
PROCNO                  1

F2 - Acquisition Parameters
Date_              20230411
Time                  1.40 h
INSTRUM               spect
PROBHD    Z116098_0653 (
PULPROG             zgpg30
TD                   65536
SOLVENT              CDCl3
NS                    1024
DS                       4
SWH              24038.461 Hz
FIDRES           0.733596 Hz
AQ               1.3631488 sec
RG                  198.36
DW                  20.800 usec
DE                    6.50 usec
TE                   292.6 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                      1
SFO1          100.6228298 MHz
NUC1                   13C
P1                   10.00 usec
PLW1          75.84400177 W
SFO2          400.1316005 MHz
NUC2                   1H
CPDPRG[2           waltz16
PCPD2                90.00 usec
PLW2          16.43099976 W
PLW12          0.20286000 W
PLW13          0.10204000 W

F2 - Processing parameters
SI                   32768
SF          100.6127592 MHz
WDW                     EM
SSB                      0
LB                    1.00 Hz
GB                       0
PC                    1.40
```

3m

^{1}H NMR (400 MHz, $CDCl_3$)

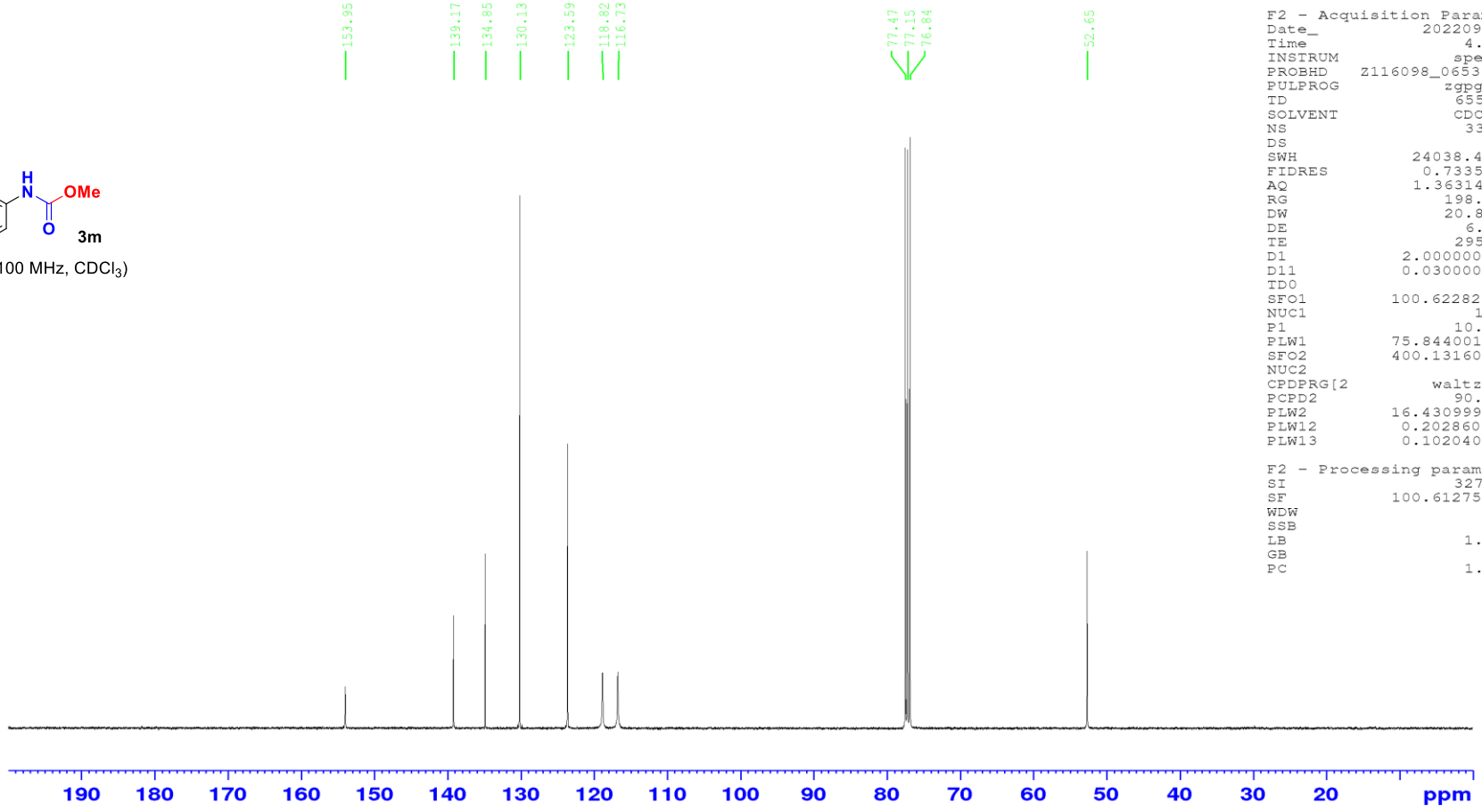



```
Current Data Parameters
NAME                      S3
EXPNO                      1
PROCNO                     1

F2 - Acquisition Parameters
Date_               20220923
Time                   22.22 h
INSTRUM                spect
PROBHD    Z116098_0653 (
PULPROG                zg30
TD                    65536
SOLVENT               CDCl3
NS                        8
DS                        2
SWH                8012.820 Hz
FIDRES            0.244532 Hz
AQ                4.0894465 sec
RG                    62.98
DW                  62.400 usec
DE                   6.50 usec
TE                  294.1 K
D1                1.00000000 sec
TD0                       1
SFO1           400.1324708 MHz
NUC1                      1H
P1                  10.00 usec
PLW1           16.43099976 W

F2 - Processing parameters
SI                    65536
SF             400.1300098 MHz
WDW                      EM
SSB                       0
LB                   0.30 Hz
GB                        0
PC                     1.00
```

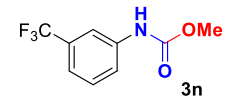


3m

^{13}C NMR (100 MHz, $CDCl_3$)

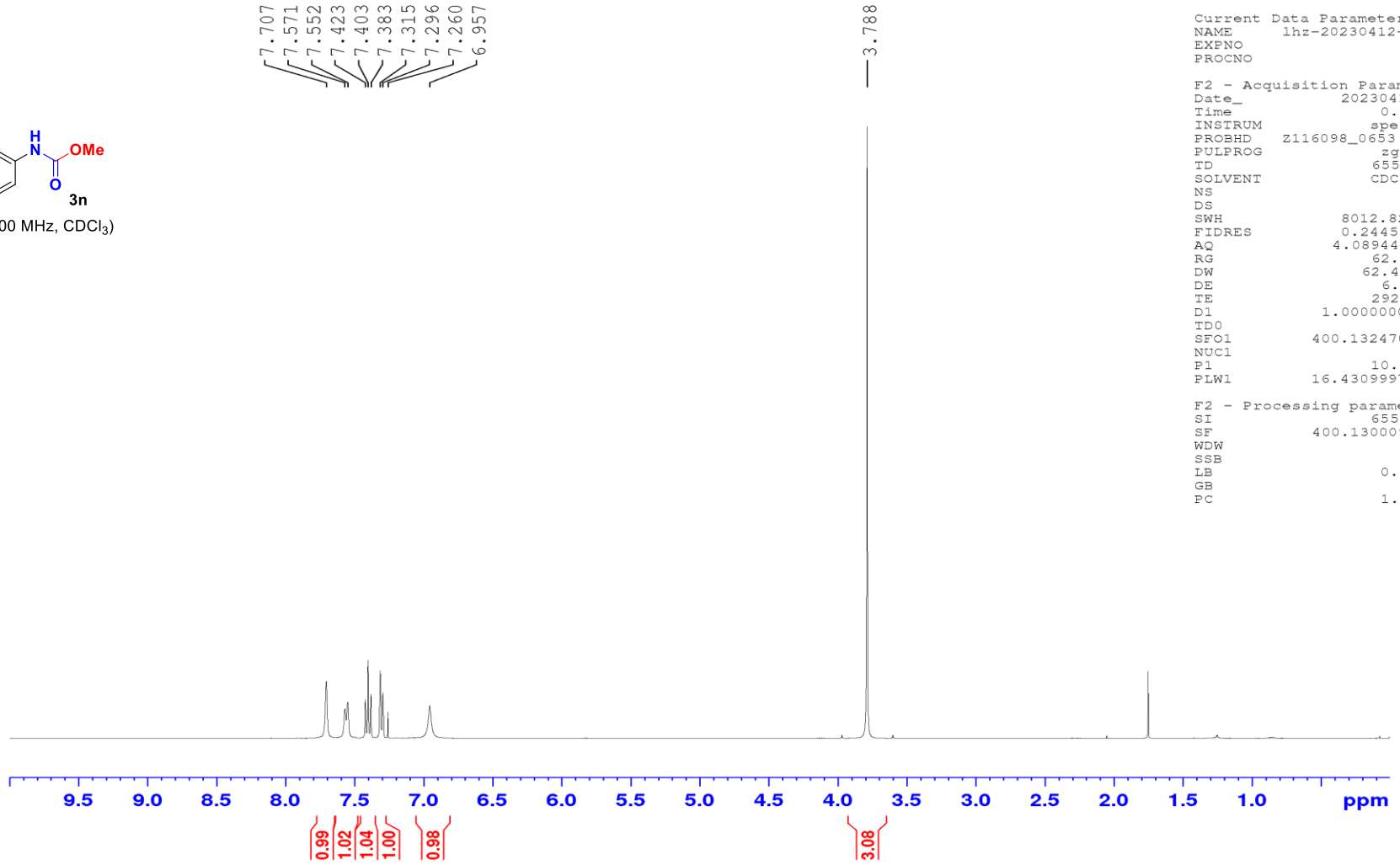



```
Current Data Parameters
NAME                      S3
EXPNO                      2
PROCNO                     1

F2 - Acquisition Parameters
Date_              20220924
Time                 4.51 h
INSTRUM              spect
PROBHD    Z116098_0653 (
PULPROG            zgpg30
TD                  65536
SOLVENT             CDCl3
NS                   3333
DS                      4
SWH            24038.461 Hz
FIDRES          0.733596 Hz
AQ             1.3631488 sec
RG                 198.36
DW                 20.800 usec
DE                  6.50 usec
TE                 295.2 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                     1
SFO1         100.6228298 MHz
NUC1                 13C
P1                 10.00 usec
PLW1         75.84400177 W
SFO2         400.1316005 MHz
NUC2                 1H
CPDPRG[2         waltz16
PCPD2              90.00 usec
PLW2         16.43099976 W
PLW12         0.20286000 W
PLW13         0.10204000 W

F2 - Processing parameters
SI                  32768
SF           100.6127594 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```

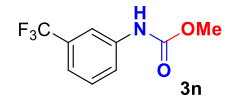


3n

^{1}H NMR (400 MHz, $CDCl_3$)

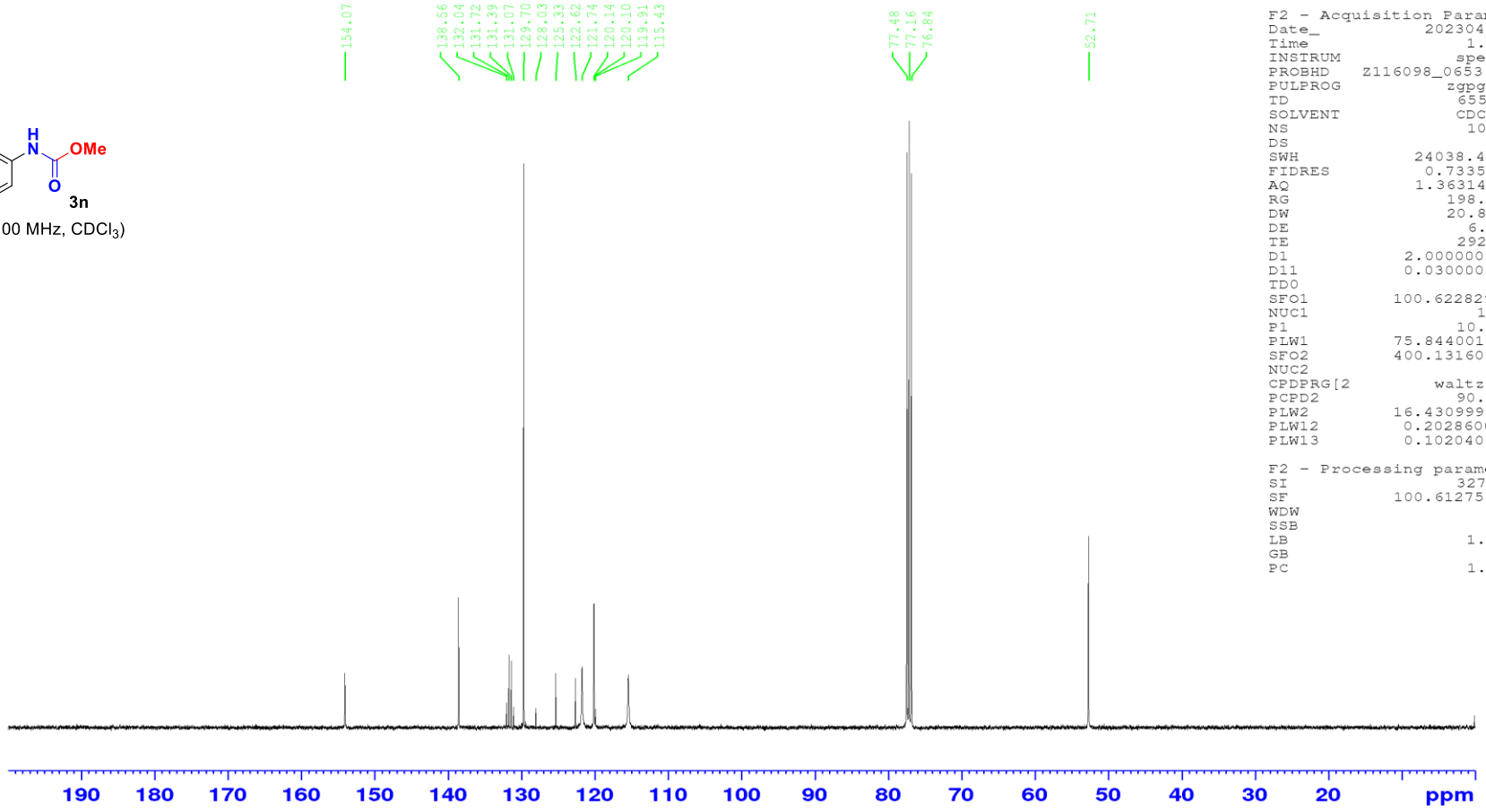



```
Current Data Parameters
NAME      lhz-20230412-3
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20230413
Time               0.38 h
INSTRUM           spect
PROBHD    Z116098_0653 (
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                   16
DS                    2
SWH            8012.820 Hz
FIDRES            0.244532 Hz
AQ             4.0894465 sec
RG                 62.98
DW                62.400 usec
DE                 6.50 usec
TE                292.2 K
D1             1.00000000 sec
TD0                   1
SFO1         400.1324708 MHz
NUC1                 1H
P1                10.00 usec
PLW1         16.43099976 W

F2 - Processing parameters
SI                65536
SF           400.1300098 MHz
WDW                  EM
SSB                   0
LB                 0.30 Hz
GB                    0
PC                 1.00
```

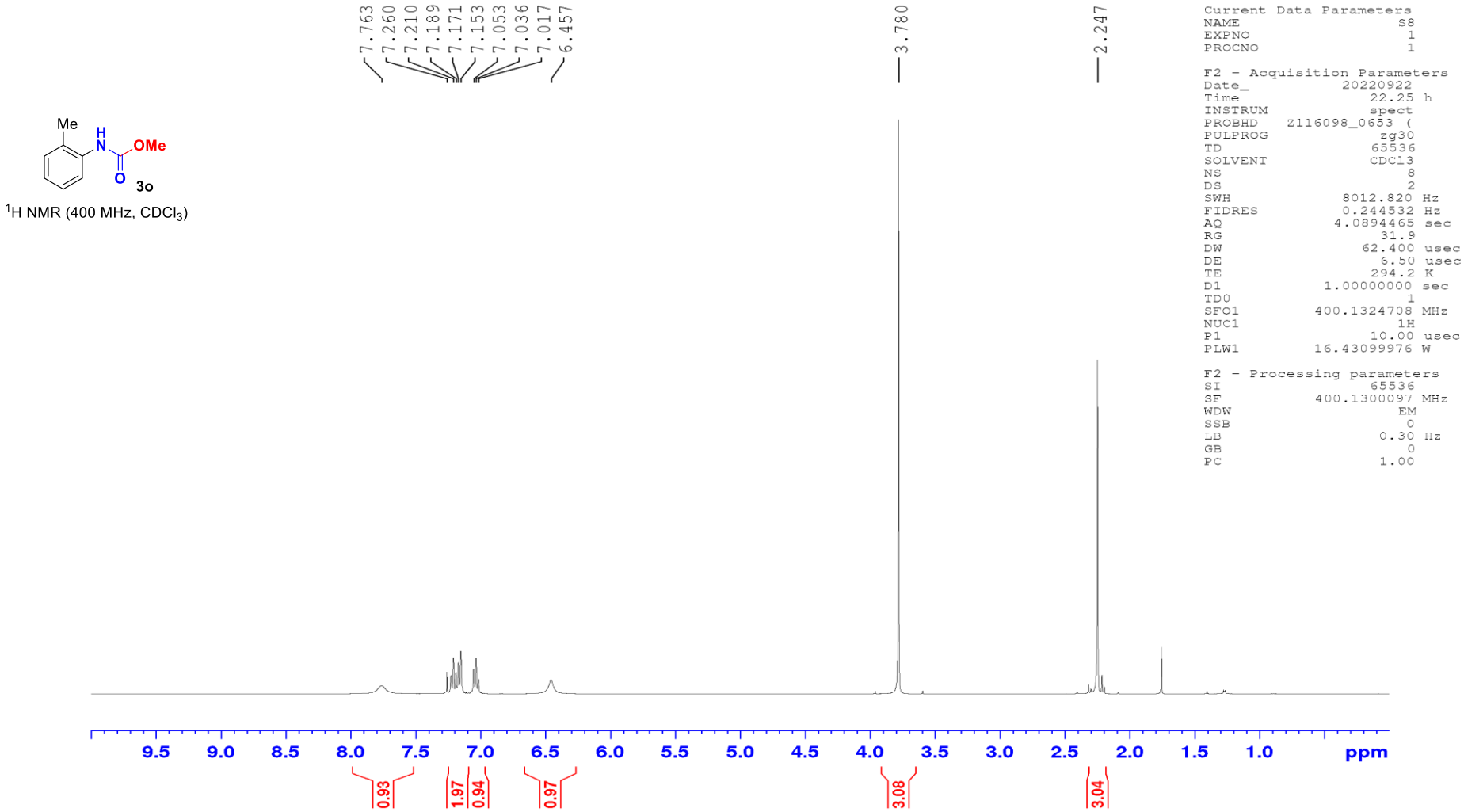


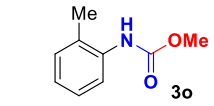
3n

^{13}C NMR (100 MHz, $CDCl_3$)



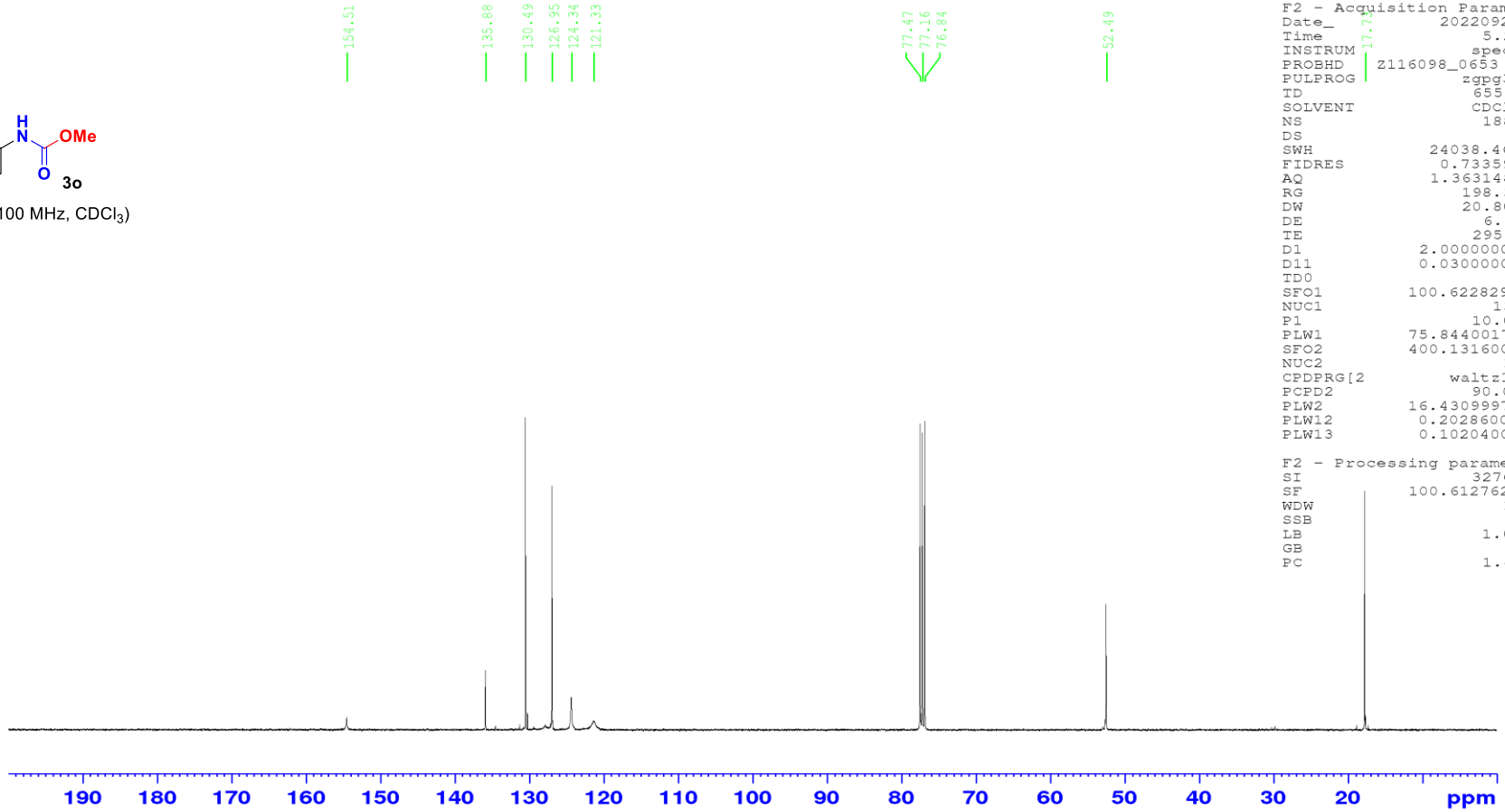

```
Current Data Parameters
NAME      1hz-20230412-3
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20230413
Time                1.38 h
INSTRUM            spect
PROBHD    Z116098_0653 (
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                 1024
DS                    4
SWH           24038.461 Hz
FIDRES         0.733596 Hz
AQ             1.3631488 sec
RG                198.36
DW                20.800 usec
DE                 6.50 usec
TE                292.7 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1
SFO1        100.6228298 MHz
NUC1               13C
P1                10.00 usec
PLW1        75.84400177 W
SFO2        400.1316005 MHz
NUC2               1H
CPDPRG[2       waltz16
PCPD2             90.00 usec
PLW2        16.43099976 W
PLW12        0.20286000 W
PLW13        0.10204000 W

F2 - Processing parameters
SI                32768
SF          100.6127579 MHz
WDW                EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```


Me
H
N
OMe
O
3o
^{1}H NMR (400 MHz, $CDCl_3$)
7.763
7.260
7.210
7.189
7.171
7.153
7.053
7.036
7.017
6.457
3.780
2.247
Current Data Parameters
NAME S8
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20220922
Time 22.25 h
INSTRUM spect
PROBHD Z116098_0653 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 8012.820 Hz
FIDRES 0.244532 Hz
AQ 4.0894465 sec
RG 31.9
DW 62.400 usec
DE 6.50 usec
TE 294.2 K
D1 1.00000000 sec
TD0 1
SFO1 400.1324708 MHz
NUC1 1H
P1 10.00 usec
PLW1 16.43099976 W
F2 - Processing parameters
SI 65536
SF 400.1300097 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
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 ppm
0.93
1.97
0.94
0.97
3.08
3.04

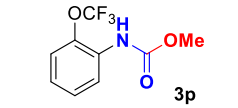


3o

^{13}C NMR (100 MHz, $CDCl_3$)

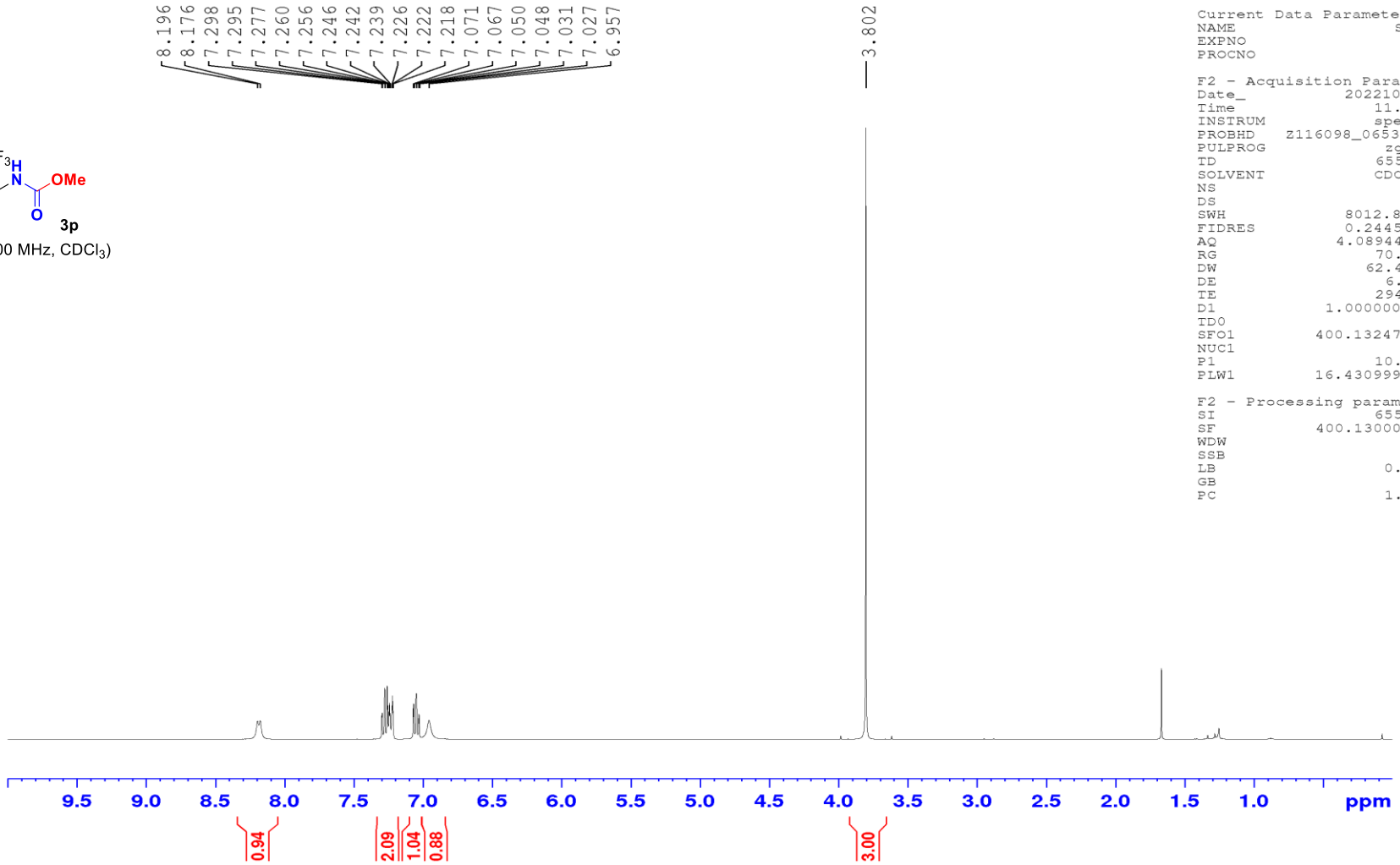



```
Current Data Parameters
NAME                      S8
EXPNO                      2
PROCNO                     1

F2 - Acquisition Parameters
Date_               20220923
Time                    5.20 h
INSTRUM                spect
PROBHD   Z116098_0653 (
PULPROG                zgpg30
TD                      65536
SOLVENT                 CDCl3
NS                       1888
DS                          4
SWH                24038.461 Hz
FIDRES              0.733596 Hz
AQ                  1.3631488 sec
RG                     198.36
DW                     20.800 usec
DE                      6.50 usec
TE                     295.2 K
D1                 2.00000000 sec
D11                0.03000000 sec
TD0                         1
SFO1             100.6228298 MHz
NUC1                      13C
P1                     10.00 usec
PLW1            75.84400177 W
SFO2            400.1316005 MHz
NUC2                      1H
CPDPRG[2              waltz16
PCPD2                  90.00 usec
PLW2            16.43099976 W
PLW12            0.20286000 W
PLW13            0.10204000 W

F2 - Processing parameters
SI                      32768
SF               100.6127620 MHz
WDW                        EM
SSB                         0
LB                      1.00 Hz
GB                          0
PC                      1.40
```

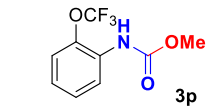


3p

^{1}H NMR (400 MHz, $CDCl_3$)

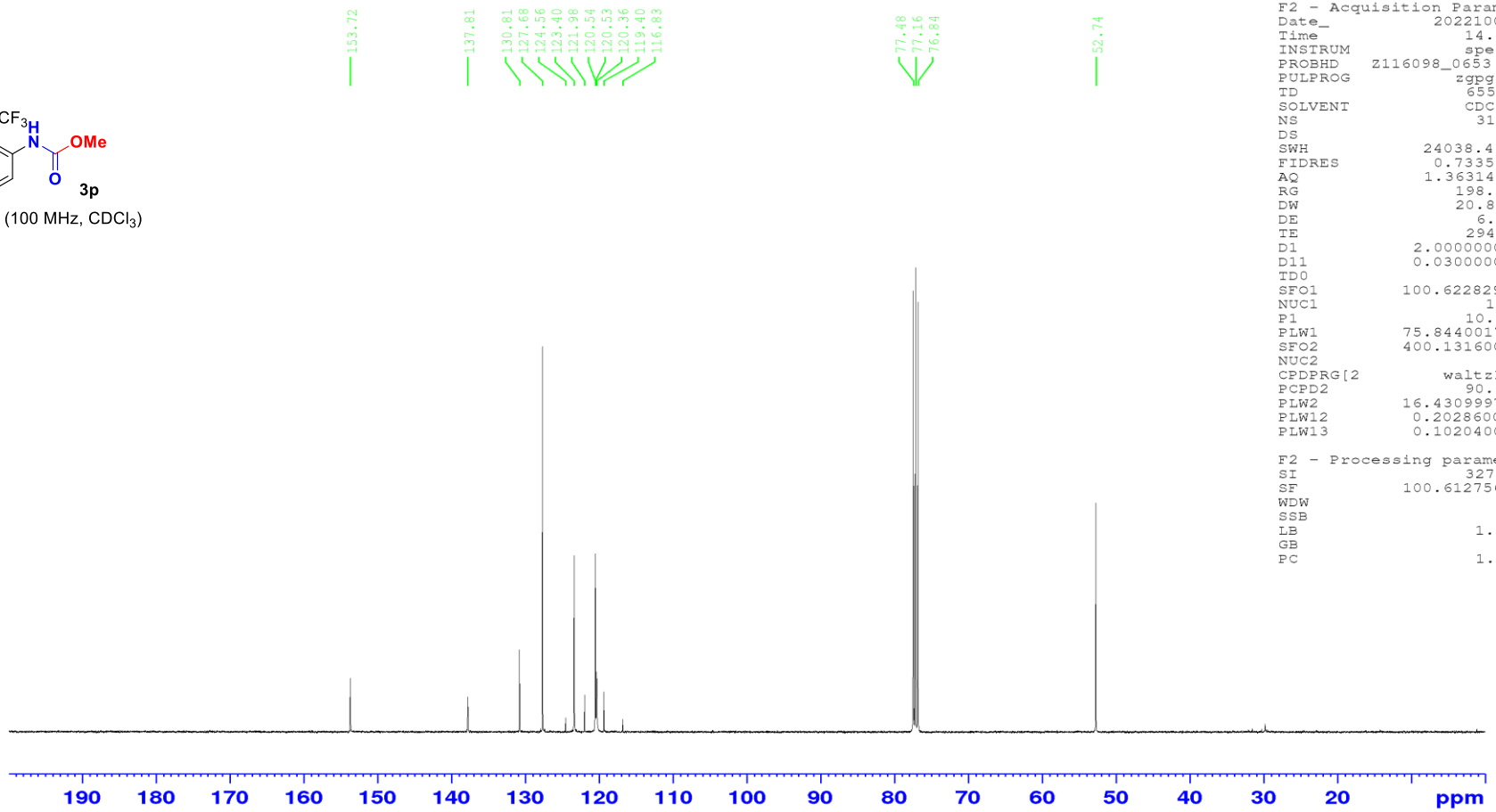



```
Current Data Parameters
NAME                      S11
EXPNO                       1
PROCNO                      1

F2 - Acquisition Parameters
Date_              20221001
Time                 11.35 h
INSTRUM              spect
PROBHD    Z116098_0653 (
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                     16
DS                      2
SWH               8012.820 Hz
FIDRES               0.244532 Hz
AQ                4.0894465 sec
RG                    70.89
DW                  62.400 usec
DE                    6.50 usec
TE                   294.4 K
D1              1.00000000 sec
TD0                     1
SFO1           400.1324708 MHz
NUC1                   1H
P1                   10.00 usec
PLW1           16.43099976 W

F2 - Processing parameters
SI                  65536
SF             400.1300098 MHz
WDW                    EM
SSB                     0
LB                   0.30 Hz
GB                      0
PC                   1.00
```

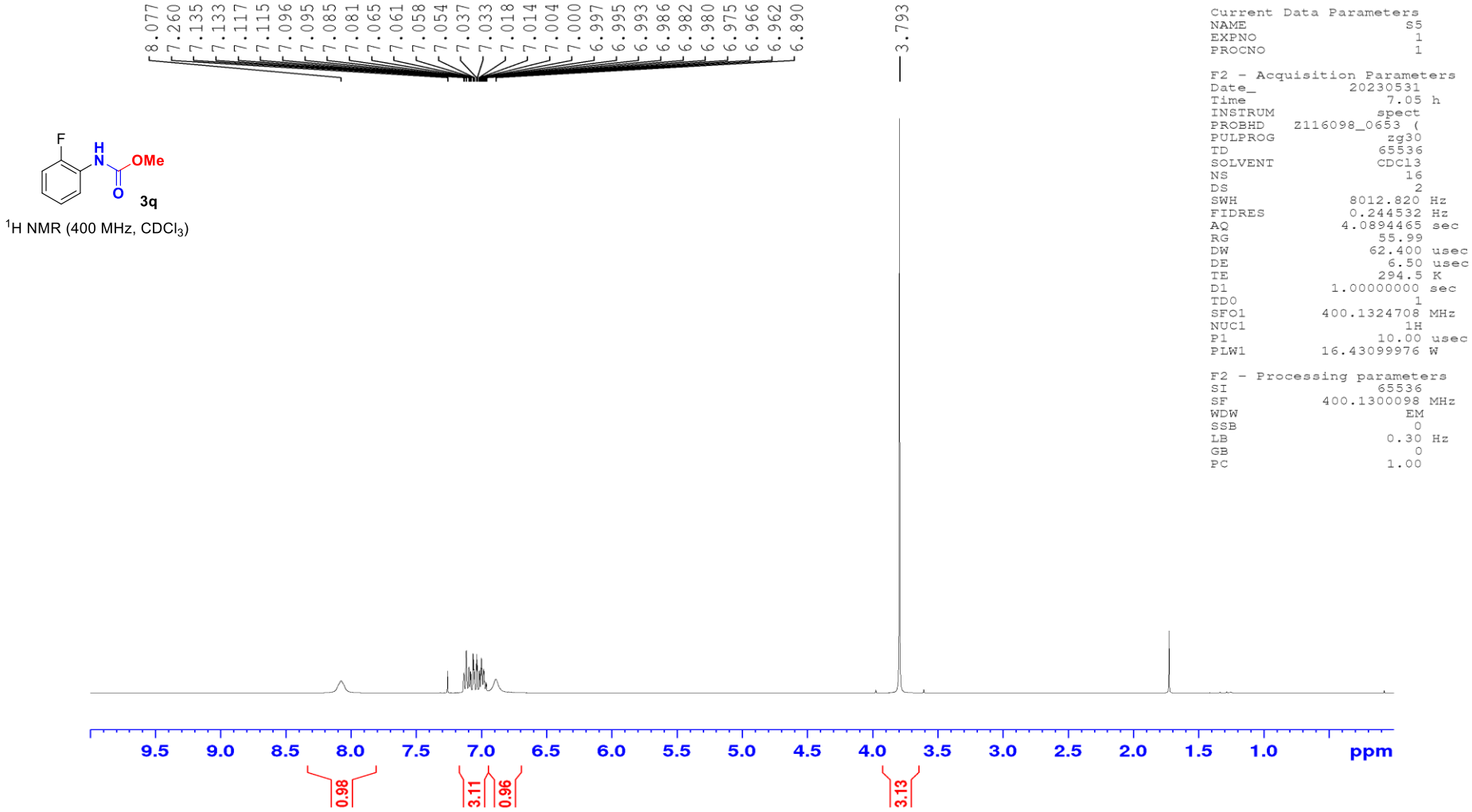


3p

^{13}C NMR (100 MHz, $CDCl_3$)



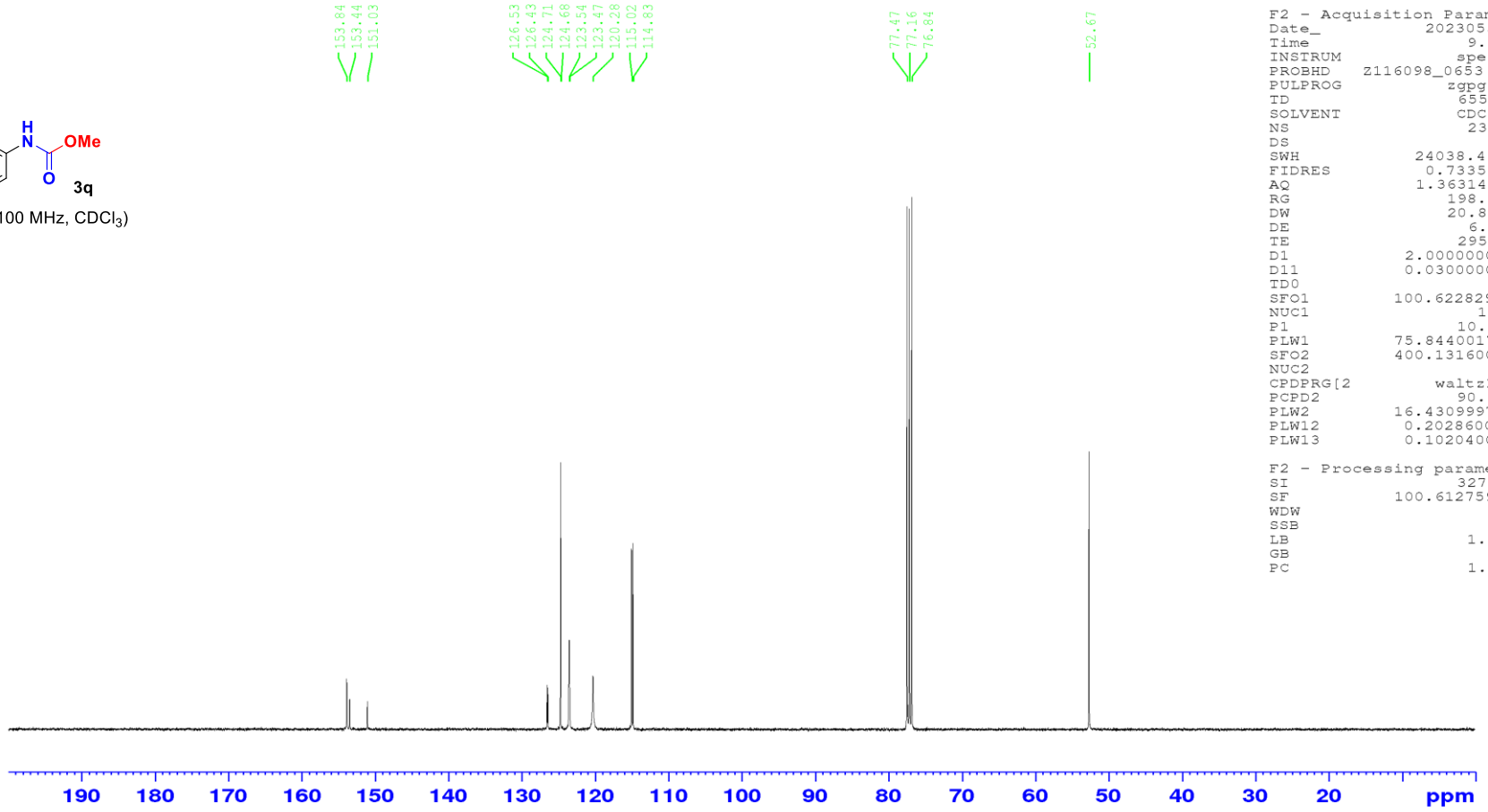

```
Current Data Parameters
NAME                  S11
EXPNO                   2
PROCNO                  1

F2 - Acquisition Parameters
Date_              20221001
Time                 14.37 h
INSTRUM              spect
PROBHD    Z116098_0653 (
PULPROG            zgpg30
TD                  65536
SOLVENT             CDCl3
NS                   3168
DS                      4
SWH             24038.461 Hz
FIDRES          0.733596 Hz
AQ              1.3631488 sec
RG                 198.36
DW                 20.800 usec
DE                   6.50 usec
TE                 294.8 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                     1
SFO1          100.6228298 MHz
NUC1                  13C
P1                  10.00 usec
PLW1          75.84400177 W
SFO2          400.1316005 MHz
NUC2                  1H
CPDPRG[2          waltz16
PCPD2               90.00 usec
PLW2          16.43099976 W
PLW12          0.20286000 W
PLW13          0.10204000 W

F2 - Processing parameters
SI                  32768
SF            100.6127564 MHz
WDW                    EM
SSB                     0
LB                   1.00 Hz
GB                      0
PC                   1.40
```



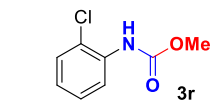

```
Current Data Parameters
NAME                      S5
EXPNO                      1
PROCNO                     1

F2 - Acquisition Parameters
Date_              20230531
Time                   7.05 h
INSTRUM               spect
PROBHD    Z116098_0653 (
PULPROG                zg30
TD                    65536
SOLVENT               CDCl3
NS                       16
DS                        2
SWH                8012.820 Hz
FIDRES            0.244532 Hz
AQ                4.0894465 sec
RG                    55.99
DW                   62.400 usec
DE                    6.50 usec
TE                   294.5 K
D1              1.00000000 sec
TD0                       1
SFO1           400.1324708 MHz
NUC1                    1H
P1                   10.00 usec
PLW1           16.43099976 W

F2 - Processing parameters
SI                    65536
SF             400.1300098 MHz
WDW                     EM
SSB                      0
LB                    0.30 Hz
GB                       0
PC                    1.00
```

3q

^{13}C NMR (100 MHz, $CDCl_3$)

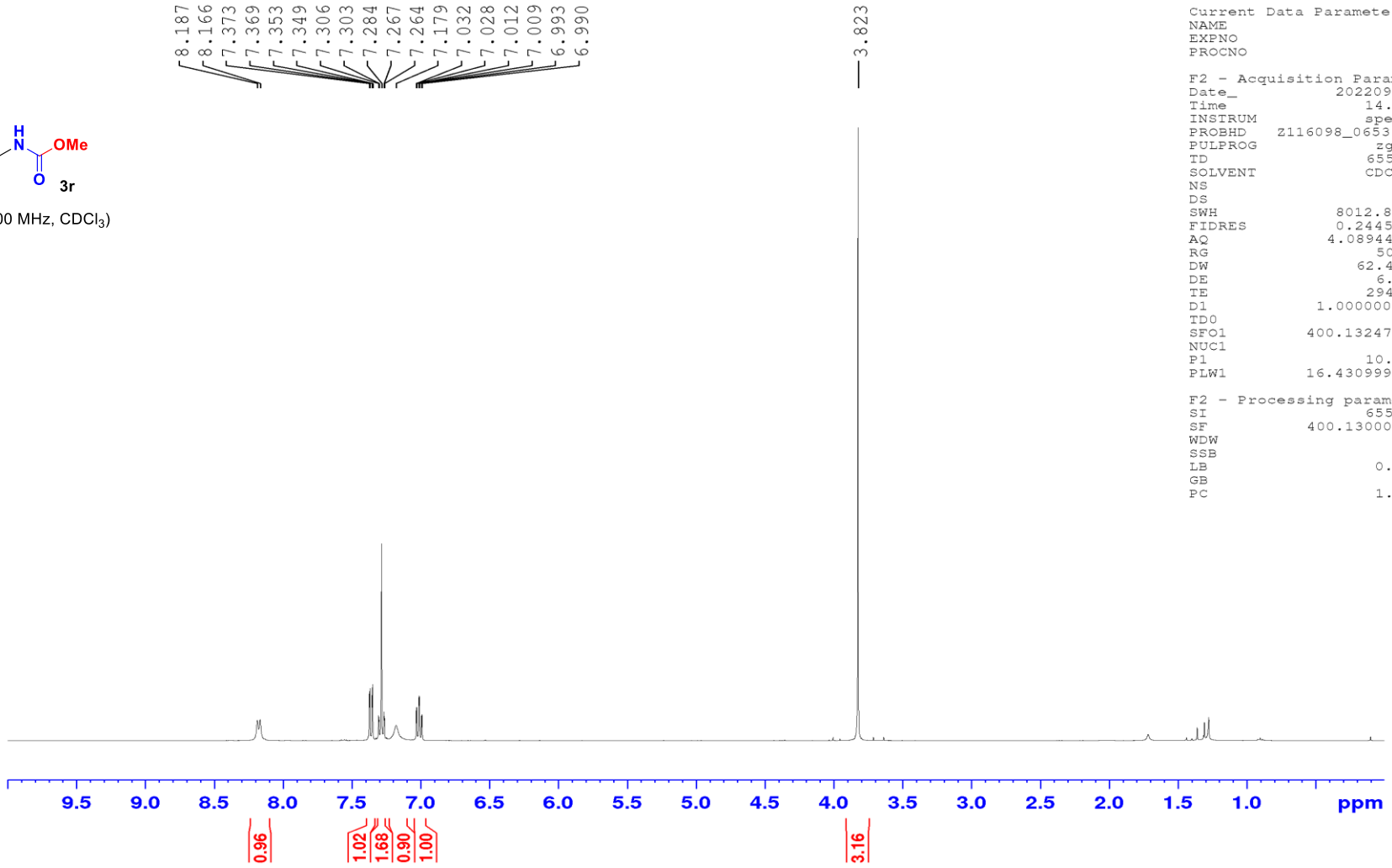



```
Current Data Parameters
NAME                      S5
EXPNO                      2
PROCNO                     1

F2 - Acquisition Parameters
Date_               20230531
Time                   9.18 h
INSTRUM                spect
PROBHD    Z116098_0653 (
PULPROG              zgpg30
TD                    65536
SOLVENT               CDCl3
NS                     2313
DS                        4
SWH             24038.461 Hz
FIDRES           0.733596 Hz
AQ              1.3631488 sec
RG                   198.36
DW                 20.800 usec
DE                   6.50 usec
TE                   295.1 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                       1
SFO1          100.6228298 MHz
NUC1                   13C
P1                   10.00 usec
PLW1          75.84400177 W
SFO2          400.1316005 MHz
NUC2                   1H
CPDPRG[2          waltz16
PCPD2                90.00 usec
PLW2          16.43099976 W
PLW12          0.20286000 W
PLW13          0.10204000 W

F2 - Processing parameters
SI                    32768
SF            100.6127591 MHz
WDW                      EM
SSB                       0
LB                     1.00 Hz
GB                        0
PC                     1.40
```

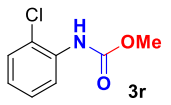


3r

^{1}H NMR (400 MHz, $CDCl_3$)

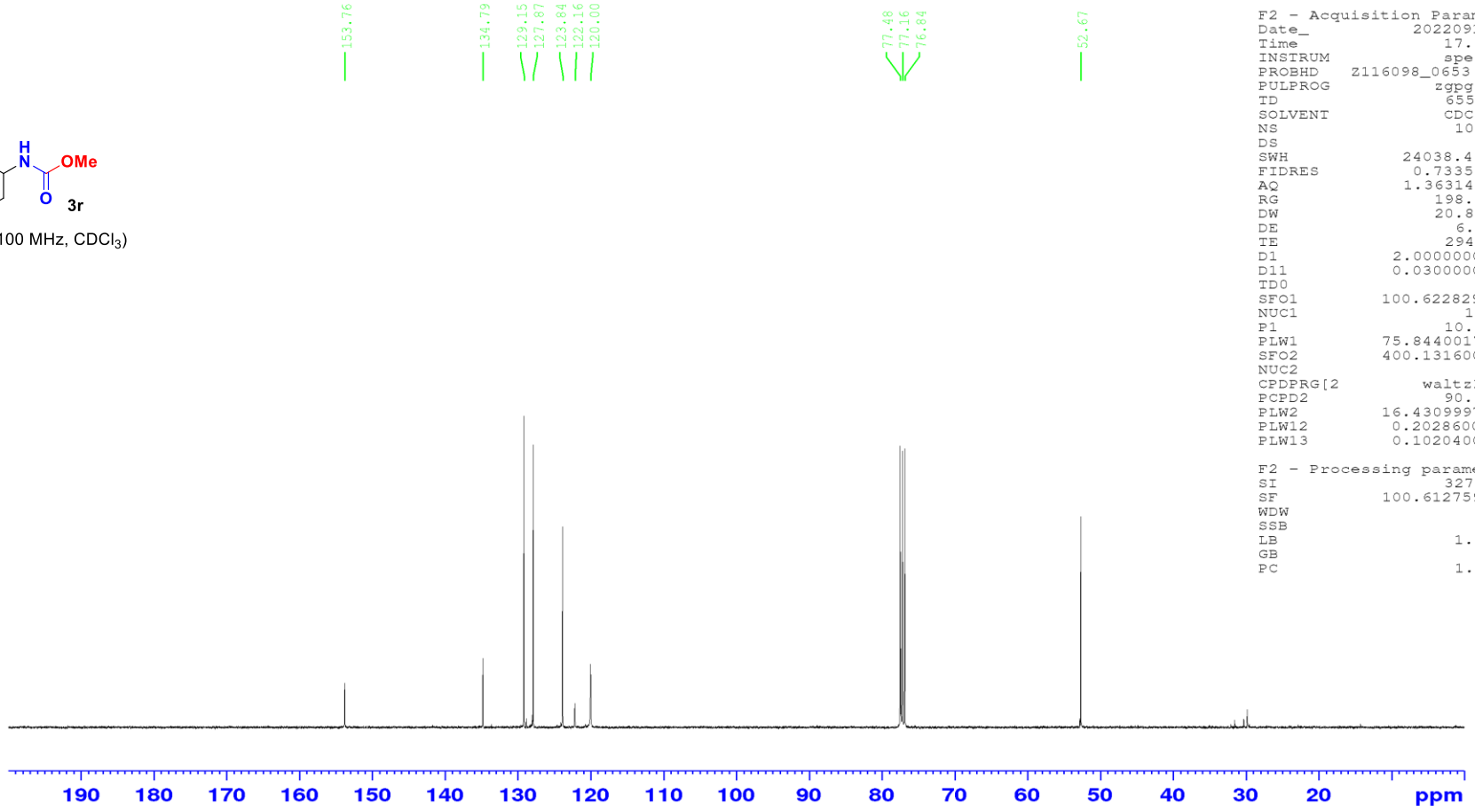



```
Current Data Parameters
NAME                      S2
EXPNO                      1
PROCNO                     1

F2 - Acquisition Parameters
Date_               20220914
Time                  14.50 h
INSTRUM                spect
PROBHD    Z116098_0653 (
PULPROG                zg30
TD                    65536
SOLVENT               CDCl3
NS                        8
DS                        2
SWH                8012.820 Hz
FIDRES            0.244532 Hz
AQ                4.0894465 sec
RG                     50.3
DW                   62.400 usec
DE                    6.50 usec
TE                   294.4 K
D1              1.00000000 sec
TD0                       1
SFO1           400.1324708 MHz
NUC1                    1H
P1                   10.00 usec
PLW1           16.43099976 W

F2 - Processing parameters
SI                    65536
SF             400.1300000 MHz
WDW                     EM
SSB                       0
LB                    0.30 Hz
GB                        0
PC                     1.00
```

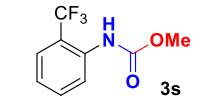


3r

^{13}C NMR (100 MHz, $CDCl_3$)

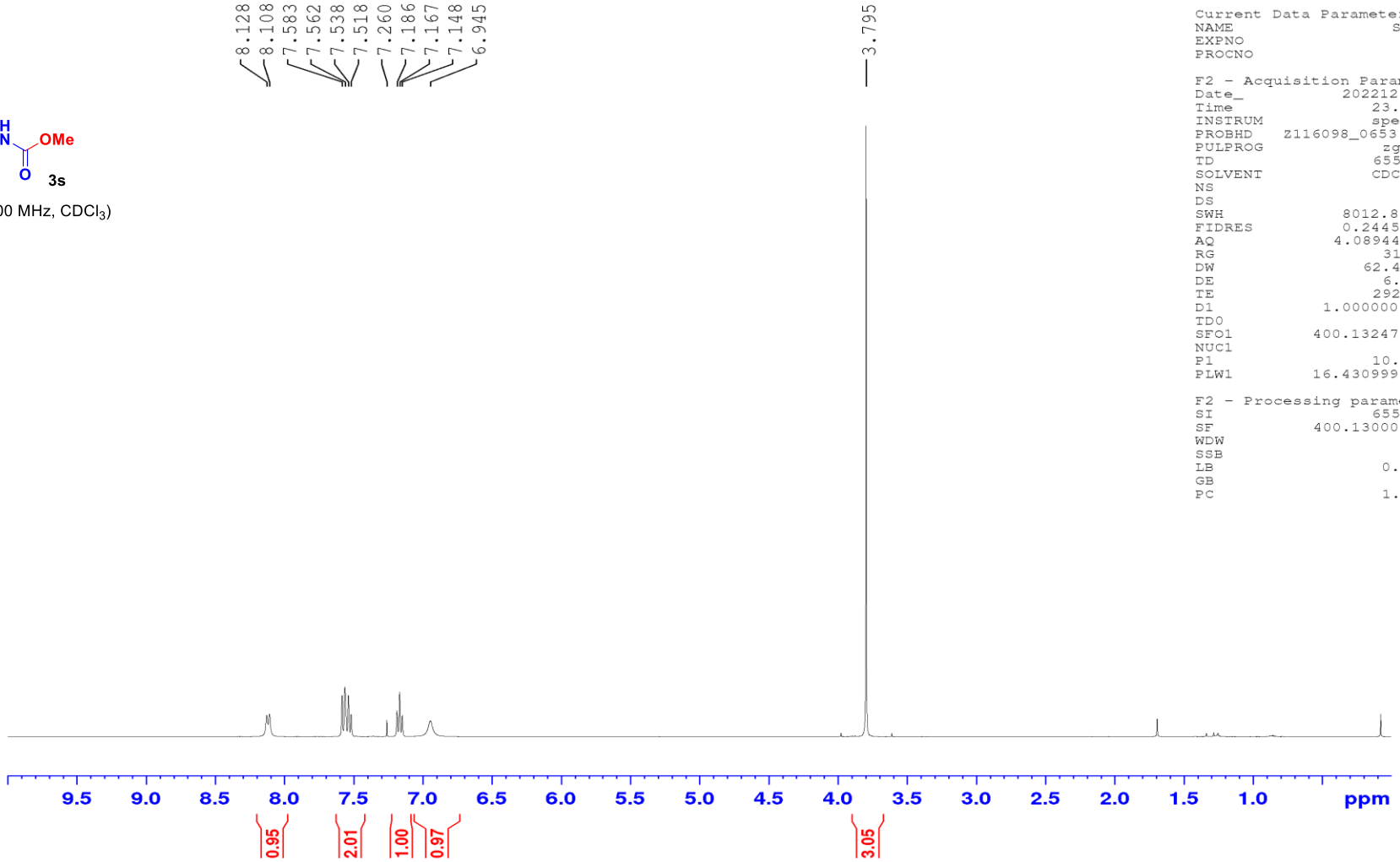



```
Current Data Parameters
NAME                      S2
EXPNO                      2
PROCNO                     1

F2 - Acquisition Parameters
Date_               20220914
Time                   17.46 h
INSTRUM                spect
PROBHD    Z116098_0653 (
PULPROG              zgpg30
TD                    65536
SOLVENT               CDCl3
NS                     1024
DS                        4
SWH               24038.461 Hz
FIDRES             0.733596 Hz
AQ                1.3631488 sec
RG                   198.36
DW                   20.800 usec
DE                    6.50 usec
TE                   294.7 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                       1
SFO1          100.6228298 MHz
NUC1                   13C
P1                    10.00 usec
PLW1          75.84400177 W
SFO2          400.1316005 MHz
NUC2                   1H
CPDPRG[2           waltz16
PCPD2                 90.00 usec
PLW2          16.43099976 W
PLW12          0.20286000 W
PLW13          0.10204000 W

F2 - Processing parameters
SI                    32768
SF            100.6127594 MHz
WDW                     EM
SSB                      0
LB                    1.00 Hz
GB                       0
PC                    1.40
```

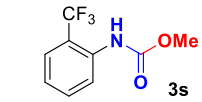


3s

^{1}H NMR (400 MHz, $CDCl_3$)

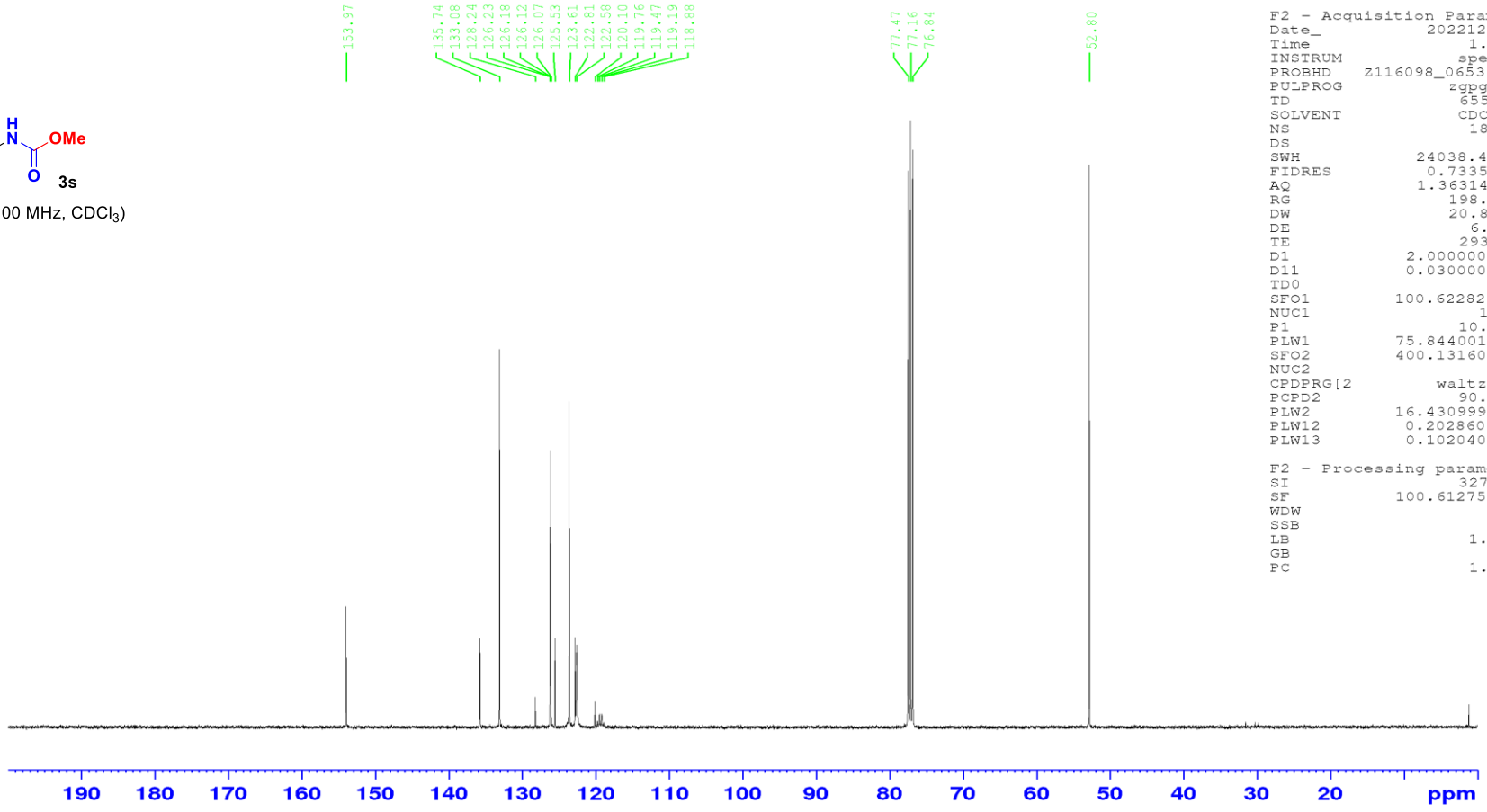



```
Current Data Parameters
NAME                      S27
EXPNO                       1
PROCNO                      1

F2 - Acquisition Parameters
Date_              20221201
Time                 23.55 h
INSTRUM              spect
PROBHD    Z116098_0653 (
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                      8
DS                      2
SWH               8012.820 Hz
FIDRES            0.244532 Hz
AQ                4.0894465 sec
RG                   31.9
DW                  62.400 usec
DE                   6.50 usec
TE                  292.9 K
D1                1.00000000 sec
TD0                     1
SFO1           400.1324708 MHz
NUC1                   1H
P1                  10.00 usec
PLW1           16.43099976 W

F2 - Processing parameters
SI                  65536
SF             400.1300098 MHz
WDW                    EM
SSB                     0
LB                   0.30 Hz
GB                      0
PC                   1.00
```

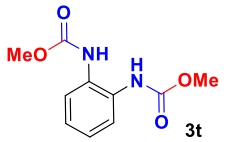


3s

^{13}C NMR (100 MHz, $CDCl_3$)

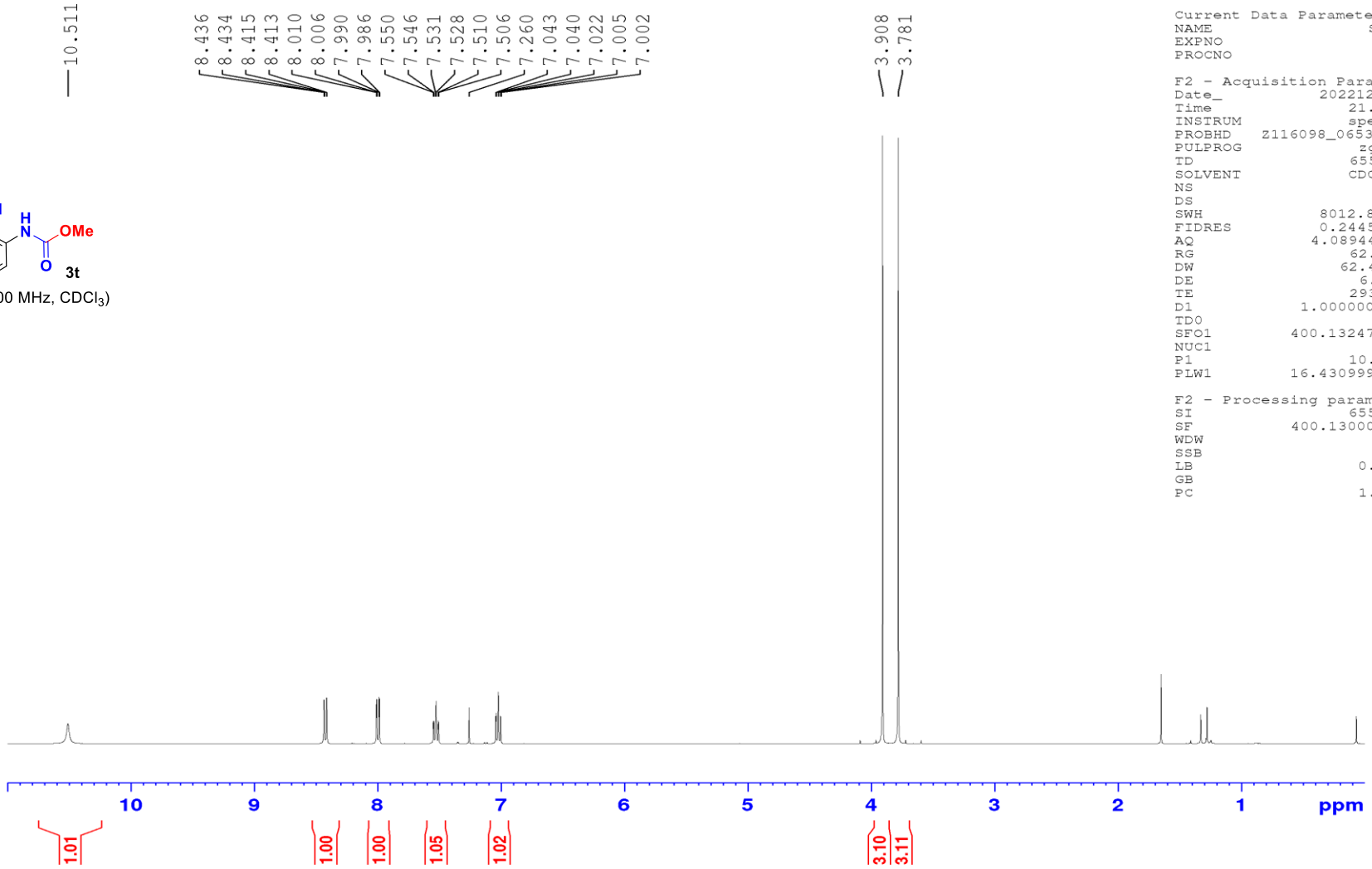



```
Current Data Parameters
NAME                      S27
EXPNO                       2
PROCNO                      1

F2 - Acquisition Parameters
Date_              20221202
Time                    1.44 h
INSTRUM                spect
PROBHD    Z116098_0653 (
PULPROG                zgpg30
TD                      65536
SOLVENT                 CDCl3
NS                       1888
DS                          4
SWH               24038.461 Hz
FIDRES             0.733596 Hz
AQ                1.3631488 sec
RG                    198.36
DW                   20.800 usec
DE                    6.50 usec
TE                   293.6 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                         1
SFO1          100.6228298 MHz
NUC1                     13C
P1                    10.00 usec
PLW1          75.84400177 W
SFO2          400.1316005 MHz
NUC2                     1H
CPDPRG[2              waltz16
PCPD2                 90.00 usec
PLW2          16.43099976 W
PLW12          0.20286000 W
PLW13          0.10204000 W

F2 - Processing parameters
SI                      32768
SF            100.6127579 MHz
WDW                        EM
SSB                         0
LB                    1.00 Hz
GB                          0
PC                    1.40
```

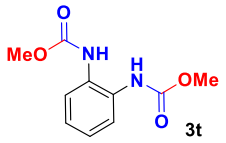


3t

^{1}H NMR (400 MHz, $CDCl_3$)

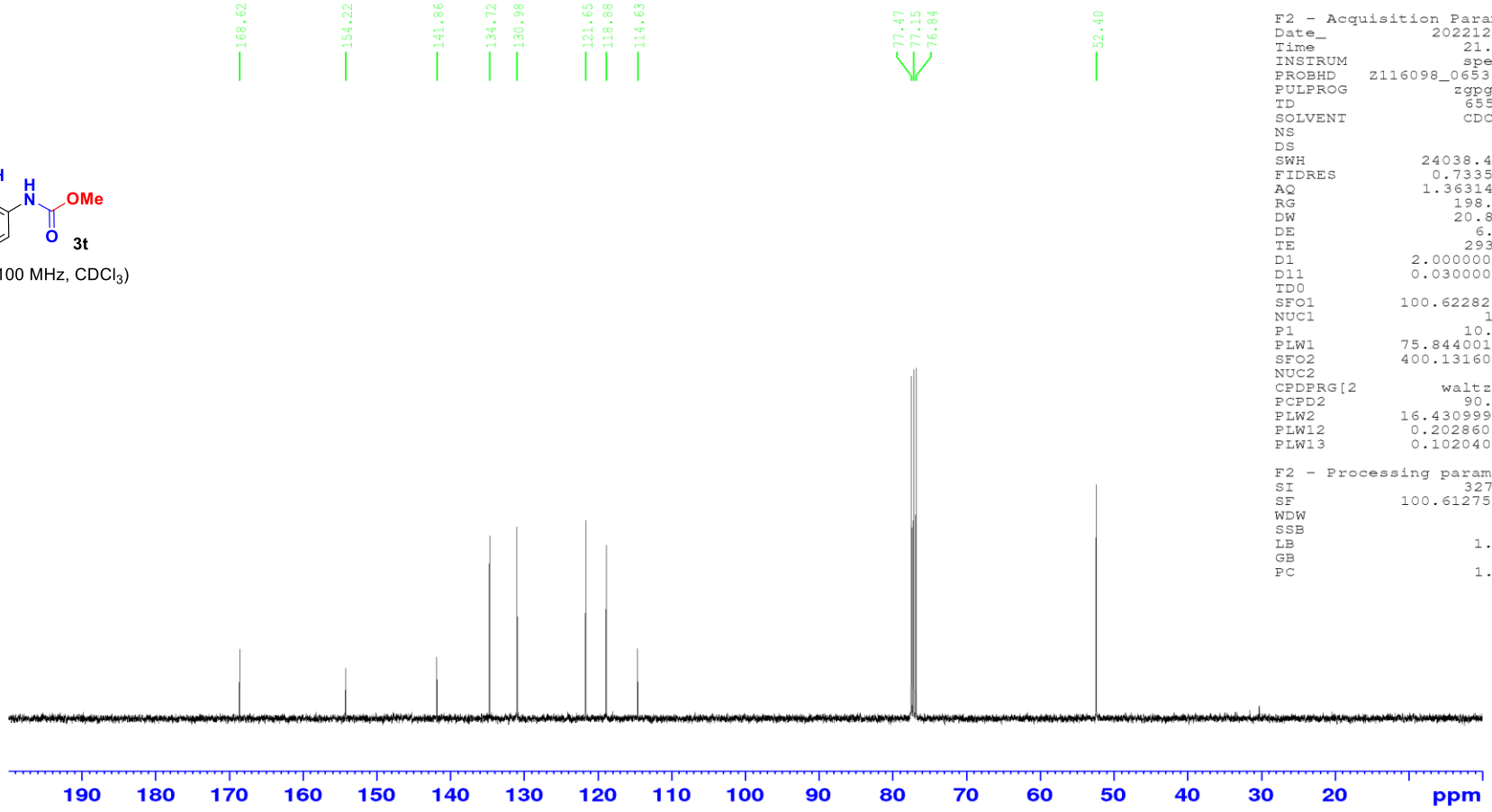



```
Current Data Parameters
NAME                      S30
EXPNO                       1
PROCNO                      1

F2 - Acquisition Parameters
Date_              20221206
Time                 21.50 h
INSTRUM                spect
PROBHD    Z116098_0653 (
PULPROG                zg30
TD                    65536
SOLVENT               CDCl3
NS                       16
DS                        2
SWH                8012.820 Hz
FIDRES            0.244532 Hz
AQ                4.0894465 sec
RG                    62.98
DW                   62.400 usec
DE                    6.50 usec
TE                   293.1 K
D1                1.00000000 sec
TD0                       1
SFO1           400.1324708 MHz
NUC1                     1H
P1                   10.00 usec
PLW1           16.43099976 W

F2 - Processing parameters
SI                    65536
SF             400.1300098 MHz
WDW                      EM
SSB                       0
LB                    0.30 Hz
GB                        0
PC                    1.00
```

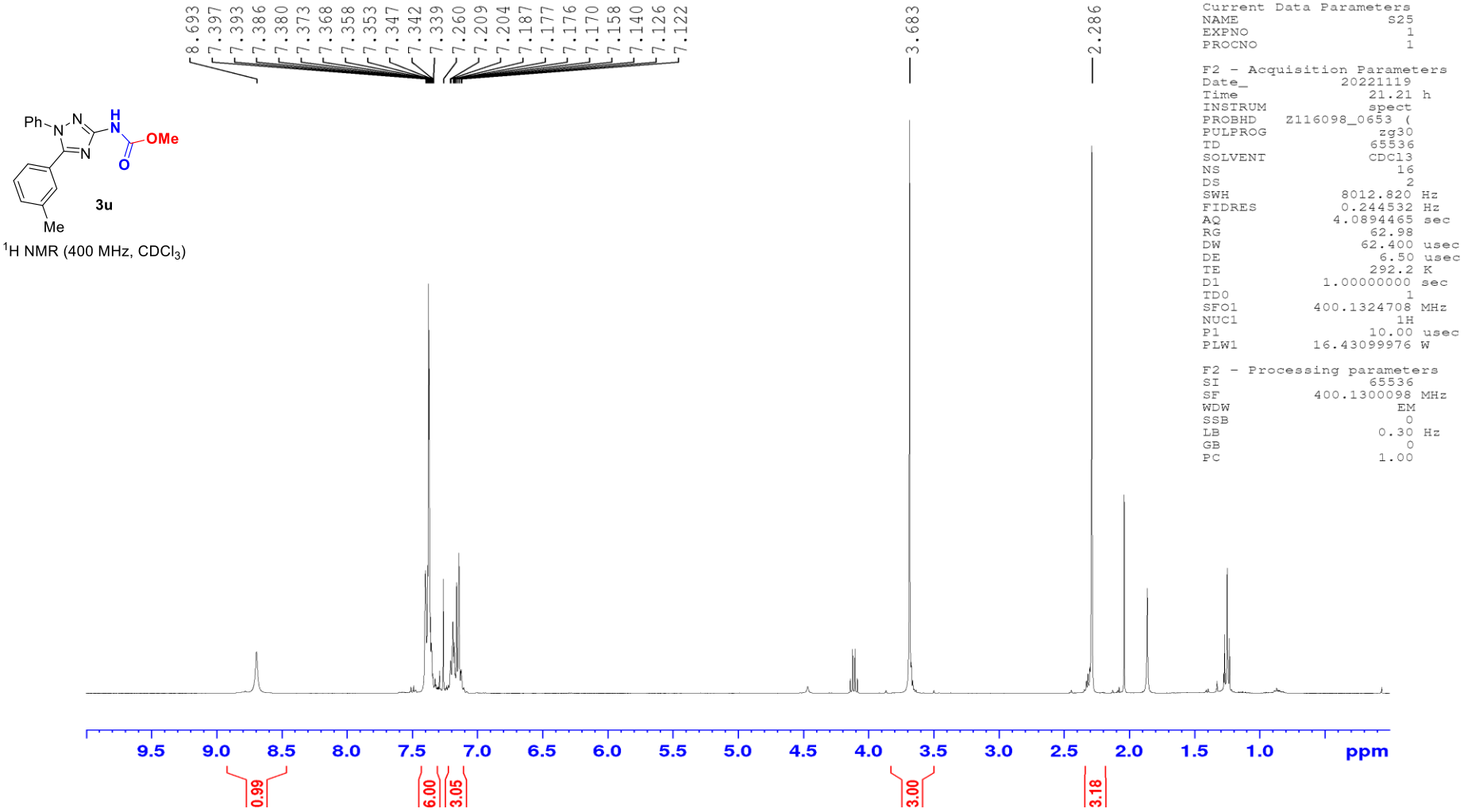


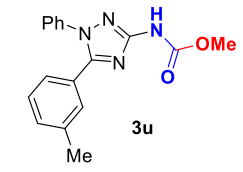
3t

^{13}C NMR (100 MHz, $CDCl_3$)



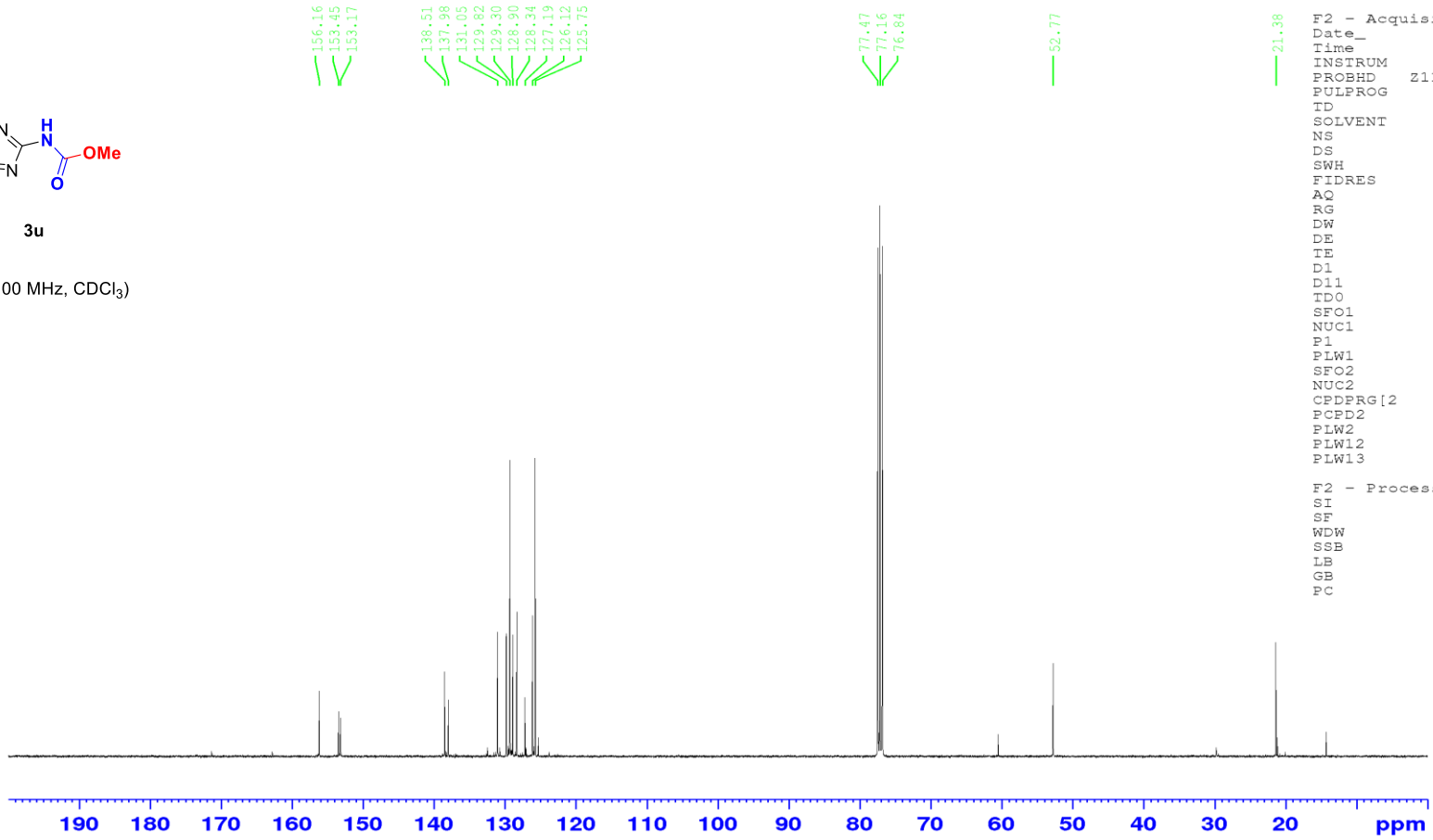

```
Current Data Parameters
NAME                      S30
EXPNO                       2
PROCNO                      1

F2 - Acquisition Parameters
Date_              20221206
Time                  21.56 h
INSTRUM                spect
PROBHD    Z116098_0653 (
PULPROG              zgpg30
TD                    65536
SOLVENT               CDCl3
NS                       88
DS                        4
SWH               24038.461 Hz
FIDRES             0.733596 Hz
AQ                1.3631488 sec
RG                   198.36
DW                   20.800 usec
DE                    6.50 usec
TE                   293.4 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                       1
SFO1          100.6228298 MHz
NUC1                    13C
P1                   10.00 usec
PLW1          75.84400177 W
SFO2          400.1316005 MHz
NUC2                    1H
CPDPRG[2          waltz16
PCPD2                90.00 usec
PLW2          16.43099976 W
PLW12          0.20286000 W
PLW13          0.10204000 W

F2 - Processing parameters
SI                    32768
SF            100.6127586 MHz
WDW                     EM
SSB                      0
LB                    1.00 Hz
GB                       0
PC                    1.40
```


8.693
7.397
7.393
7.386
7.380
7.373
7.368
7.358
7.353
7.347
7.342
7.339
7.260
7.209
7.204
7.187
7.177
7.176
7.170
7.158
7.140
7.126
7.122
3.683
2.286
Ph
N
N
N
H
N
OMe
O
Me
3u
1H NMR (400 MHz, CDCl3)
Current Data Parameters
NAME S25
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20221119
Time 21.21 h
INSTRUM spect
PROBHD Z116098_0653 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.244532 Hz
AQ 4.0894465 sec
RG 62.98
DW 62.400 usec
DE 6.50 usec
TE 292.2 K
D1 1.00000000 sec
TD0 1
SFO1 400.1324708 MHz
NUC1 1H
P1 10.00 usec
PLW1 16.43099976 W
F2 - Processing parameters
SI 65536
SF 400.1300098 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
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
ppm
0.99
6.00
3.05
3.00
3.18

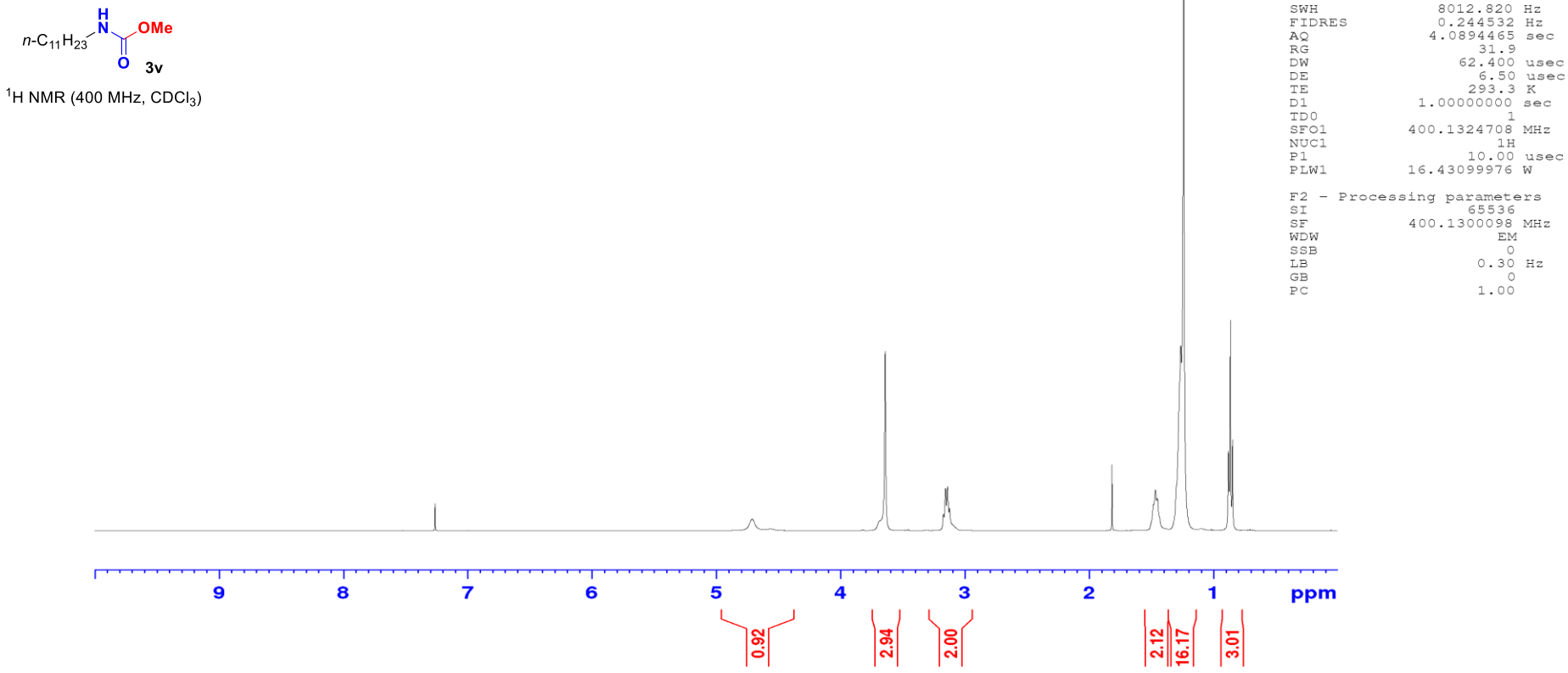


3u

^{13}C NMR (100 MHz, $CDCl_3$)

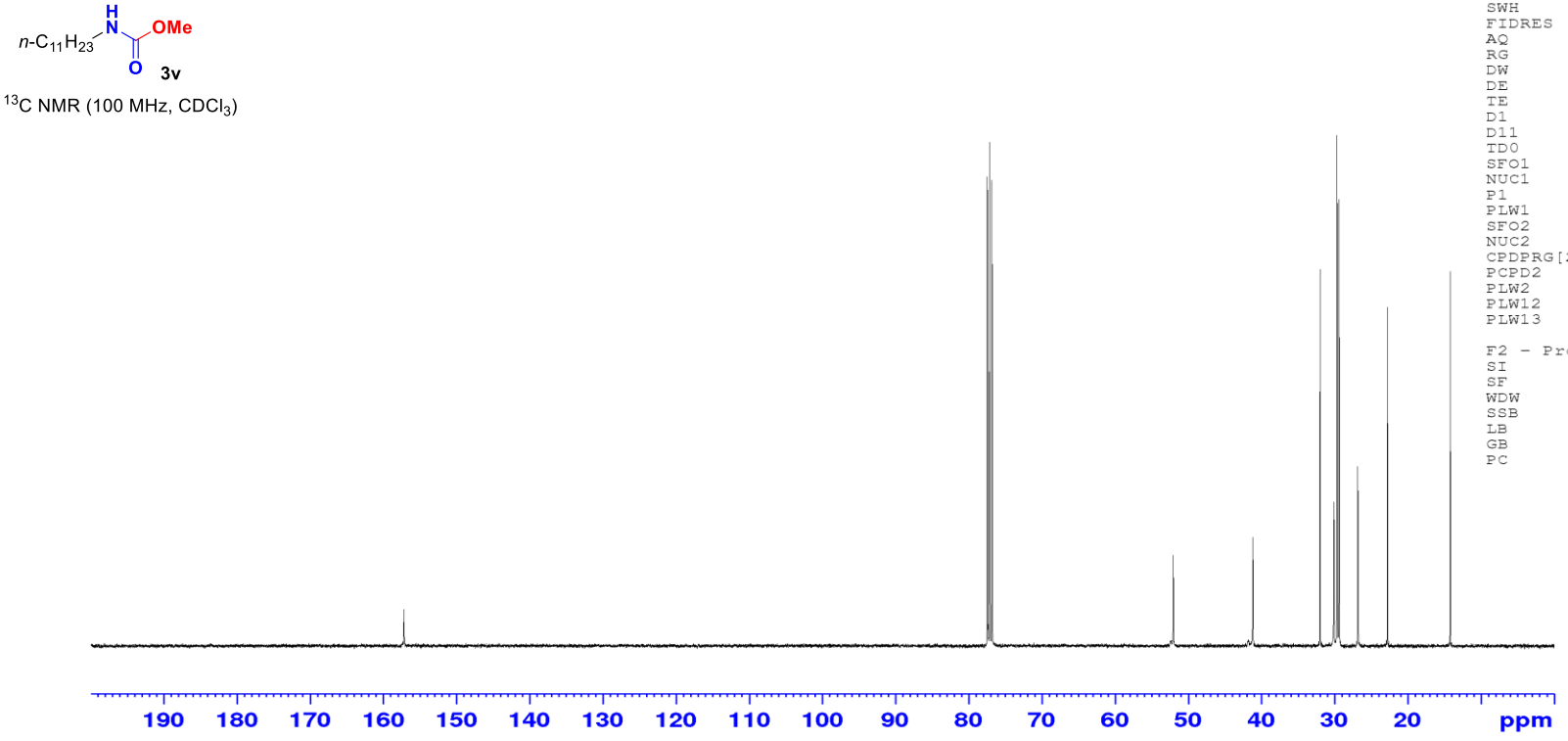



```
Current Data Parameters
NAME                      S25
EXPNO                       2
PROCNO                      1

F2 - Acquisition Parameters
Date_                20221120
Time                     0.32 h
INSTRUM                 spect
PROBHD    Z116098_0653 (
PULPROG                zgpg30
TD                      65536
SOLVENT                 CDCl3
NS                       3333
DS                          4
SWH                24038.461 Hz
FIDRES              0.733596 Hz
AQ                  1.3631488 sec
RG                     198.36
DW                     20.800 usec
DE                       6.50 usec
TE                      293.0 K
D1                 2.00000000 sec
D11                0.03000000 sec
TD0                         1
SFO1           100.6228298 MHz
NUC1                     13C
P1                     10.00 usec
PLW1           75.84400177 W
SFO2           400.1316005 MHz
NUC2                     1H
CPDPRG[2             waltz16
PCPD2                  90.00 usec
PLW2           16.43099976 W
PLW12           0.20286000 W
PLW13           0.10204000 W

F2 - Processing parameters
SI                      32768
SF             100.6127601 MHz
WDW                        EM
SSB                         0
LB                      1.00 Hz
GB                          0
PC                      1.40
```



3v

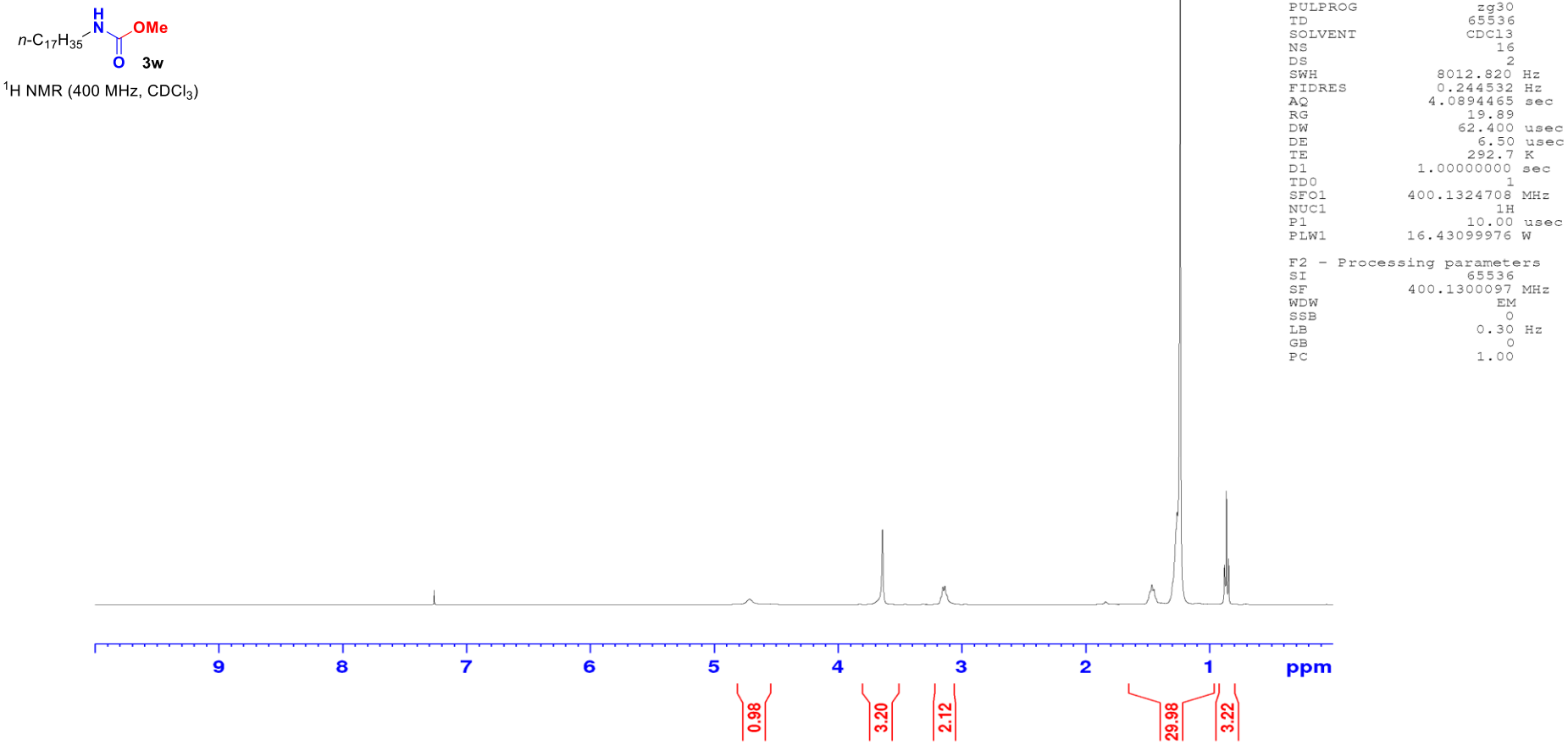
^{1}H NMR (400 MHz, $CDCl_3$)

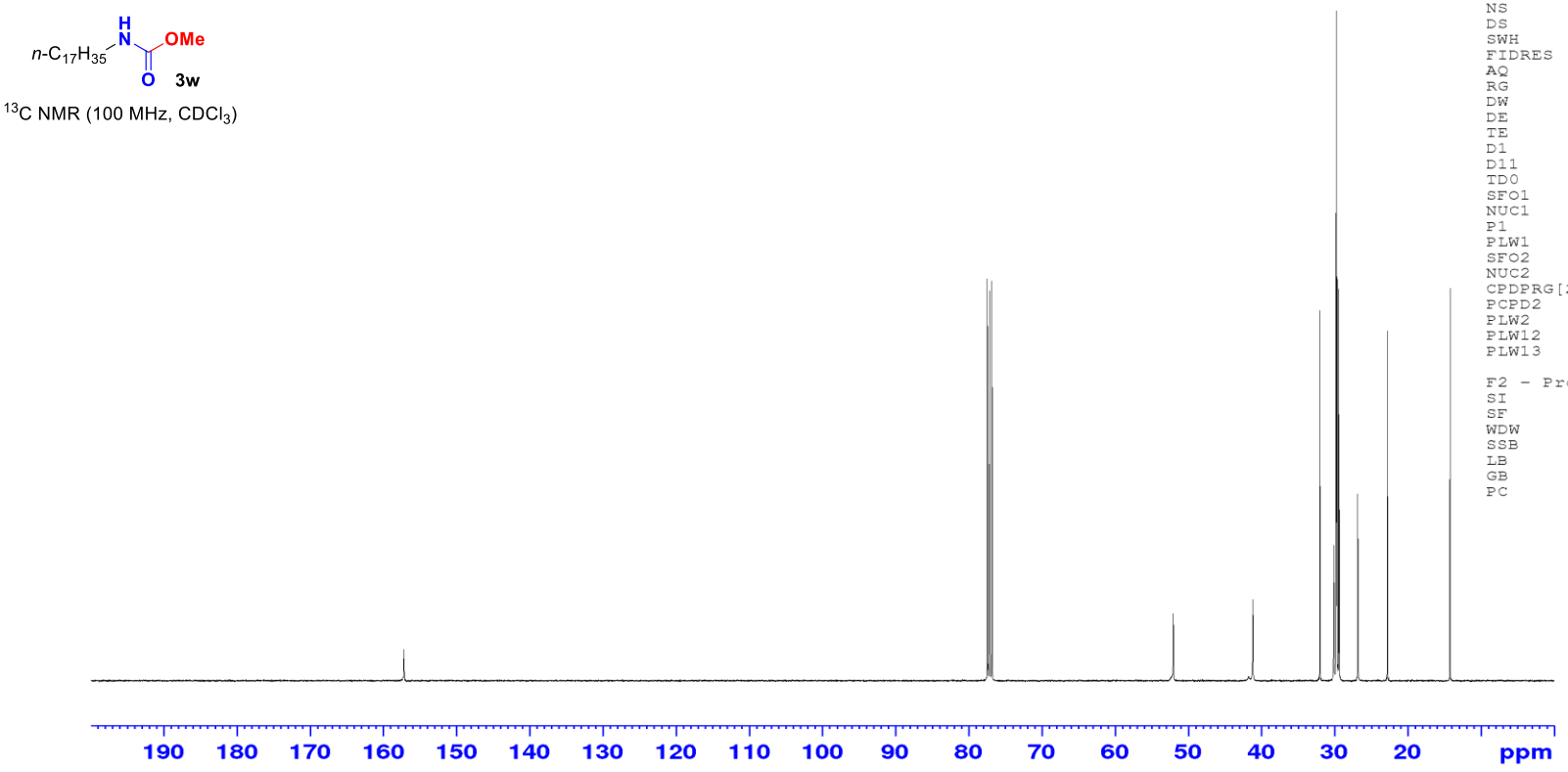


3v

^{13}C NMR (100 MHz, $CDCl_3$)

```
Current Data Parameters
NAME                   S19
EXPNO                    2
PROCNO                   1

F2 - Acquisition Parameters
Date_               20221005
Time                  22.43 h
INSTRUM                spect
PROBHD    Z116098_0653 (
PULPROG             zgpg30
TD                   65536
SOLVENT              CDCl3
NS                    1033
DS                       4
SWH              24038.461 Hz
FIDRES           0.733596 Hz
AQ               1.3631488 sec
RG                  198.36
DW                  20.800 usec
DE                    6.50 usec
TE                   293.7 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                      1
SFO1          100.6228298 MHz
NUC1                  13C
P1                  10.00 usec
PLW1          75.84400177 W
SFO2          400.1316005 MHz
NUC2                  1H
CPDPRG[2          waltz16
PCPD2               90.00 usec
PLW2          16.43099976 W
PLW12          0.20286000 W
PLW13          0.10204000 W

F2 - Processing parameters
SI                   32768
SF            100.6127579 MHz
WDW                    EM
SSB                     0
LB                   1.00 Hz
GB                      0
PC                   1.40
```

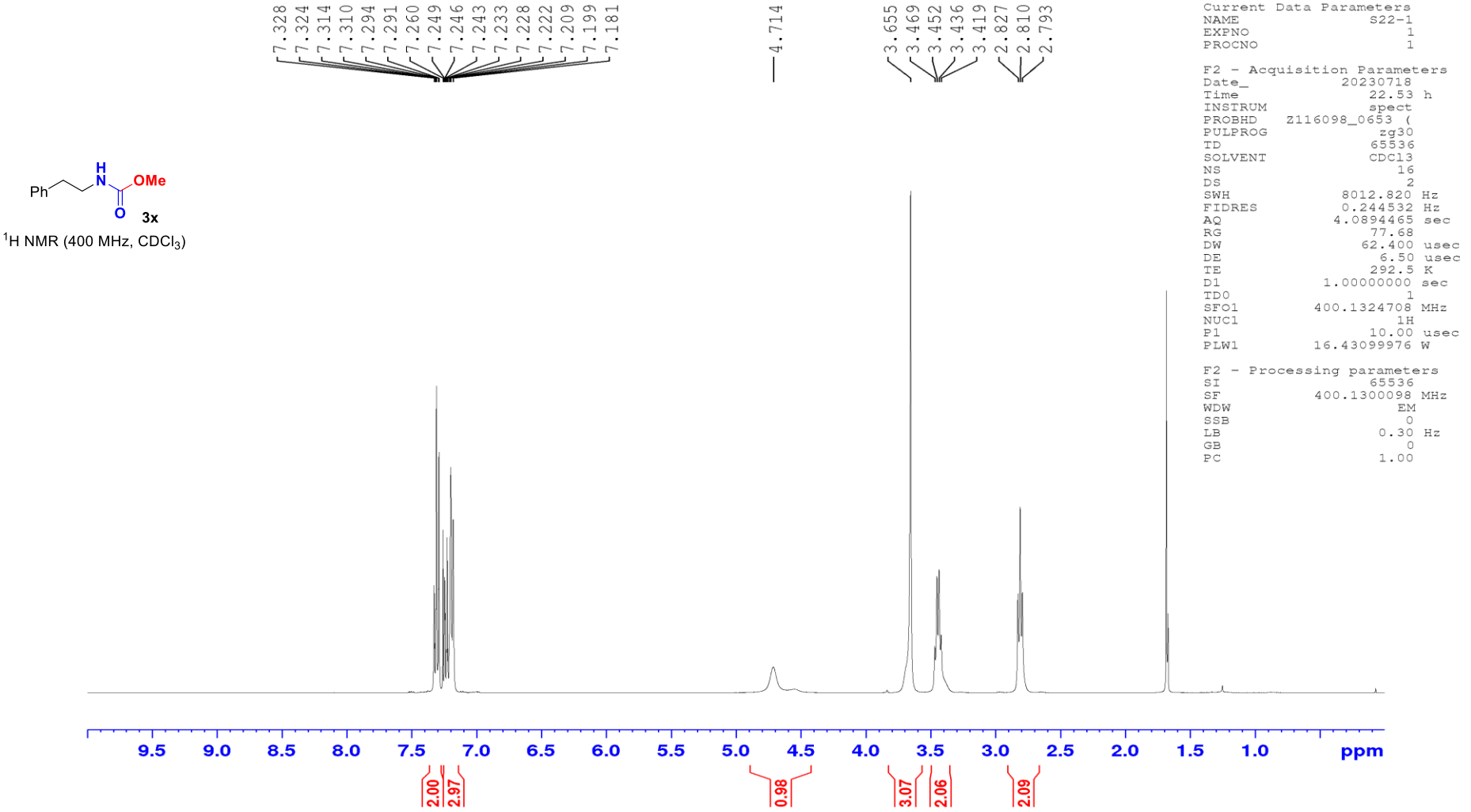

n-C17H35
H
N
OMe
O
3w
1H NMR (400 MHz, CDCl3)
7.260
4.713
3.639
3.167
3.152
3.136
3.121
1.480
1.464
1.447
1.260
1.235
0.878
0.861
0.844
Current Data Parameters
NAME S26
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20221124
Time 0.54 h
INSTRUM spect
PROBHD Z116098_0653 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.244532 Hz
AQ 4.0894465 sec
RG 19.89
DW 62.400 usec
DE 6.50 usec
TE 292.7 K
D1 1.00000000 sec
TD0 1
SFO1 400.1324708 MHz
NUC1 1H
P1 10.00 usec
PLW1 16.43099976 W
F2 - Processing parameters
SI 65536
SF 400.1300097 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
9
8
7
6
5
4
3
2
1
ppm
0.98
3.20
2.12
29.98
3.22

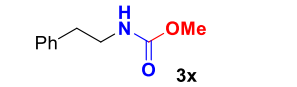


3w

^{13}C NMR (100 MHz, $CDCl_3$)

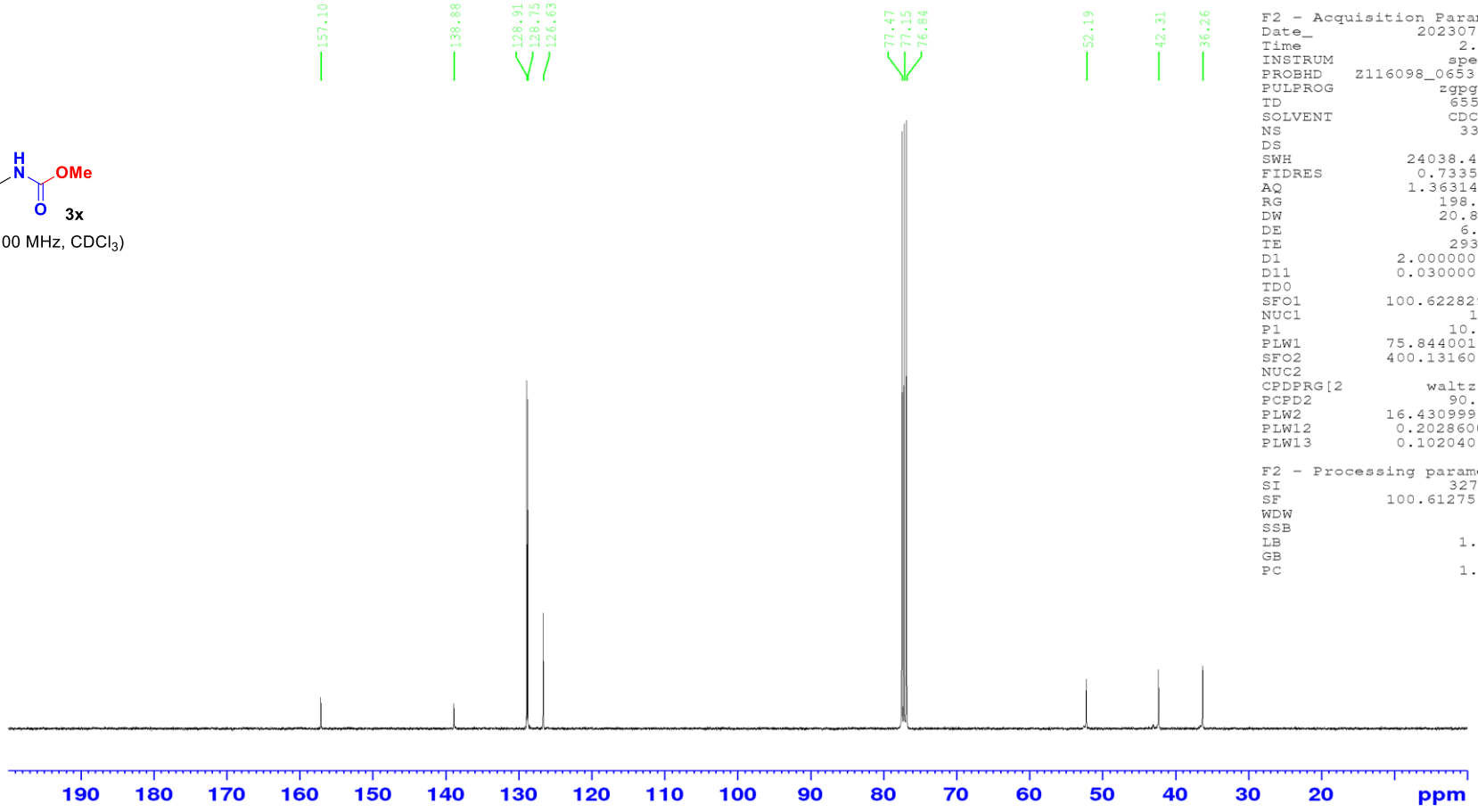
```
Current Data Parameters
NAME                  S26
EXPNO                   2
PROCNO                  1

F2 - Acquisition Parameters
Date_            20221124
Time                4.06 h
INSTRUM             spect
PROBHD    Z116098_0653 (
PULPROG          zgpg30
TD                 65536
SOLVENT            CDCl3
NS                  3333
DS                     4
SWH           24038.461 Hz
FIDRES         0.733596 Hz
AQ             1.3631488 sec
RG                198.36
DW                20.800 usec
DE                 6.50 usec
TE                293.1 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                    1
SFO1         100.6228298 MHz
NUC1                13C
P1                 10.00 usec
PLW1         75.84400177 W
SFO2         400.1316005 MHz
NUC2                1H
CPDPRG[2        waltz16
PCPD2              90.00 usec
PLW2         16.43099976 W
PLW12         0.20286000 W
PLW13         0.10204000 W

F2 - Processing parameters
SI                 32768
SF           100.6127579 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```


7.328
7.324
7.314
7.310
7.294
7.291
7.260
7.249
7.246
7.243
7.233
7.228
7.222
7.209
7.199
7.181
4.714
3.655
3.469
3.452
3.436
3.419
2.827
2.810
2.793
Ph
N
H
OMe
O
3x
1H NMR (400 MHz, CDCl3)
Current Data Parameters
NAME S22-1
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20230718
Time 22.53 h
INSTRUM spect
PROBHD Z116098_0653 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.244532 Hz
AQ 4.0894465 sec
RG 77.68
DW 62.400 usec
DE 6.50 usec
TE 292.5 K
D1 1.00000000 sec
TD0 1
SFO1 400.1324708 MHz
NUC1 1H
P1 10.00 usec
PLW1 16.43099976 W
F2 - Processing parameters
SI 65536
SF 400.1300098 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
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
ppm
2.00
2.97
0.98
3.07
2.06
2.09

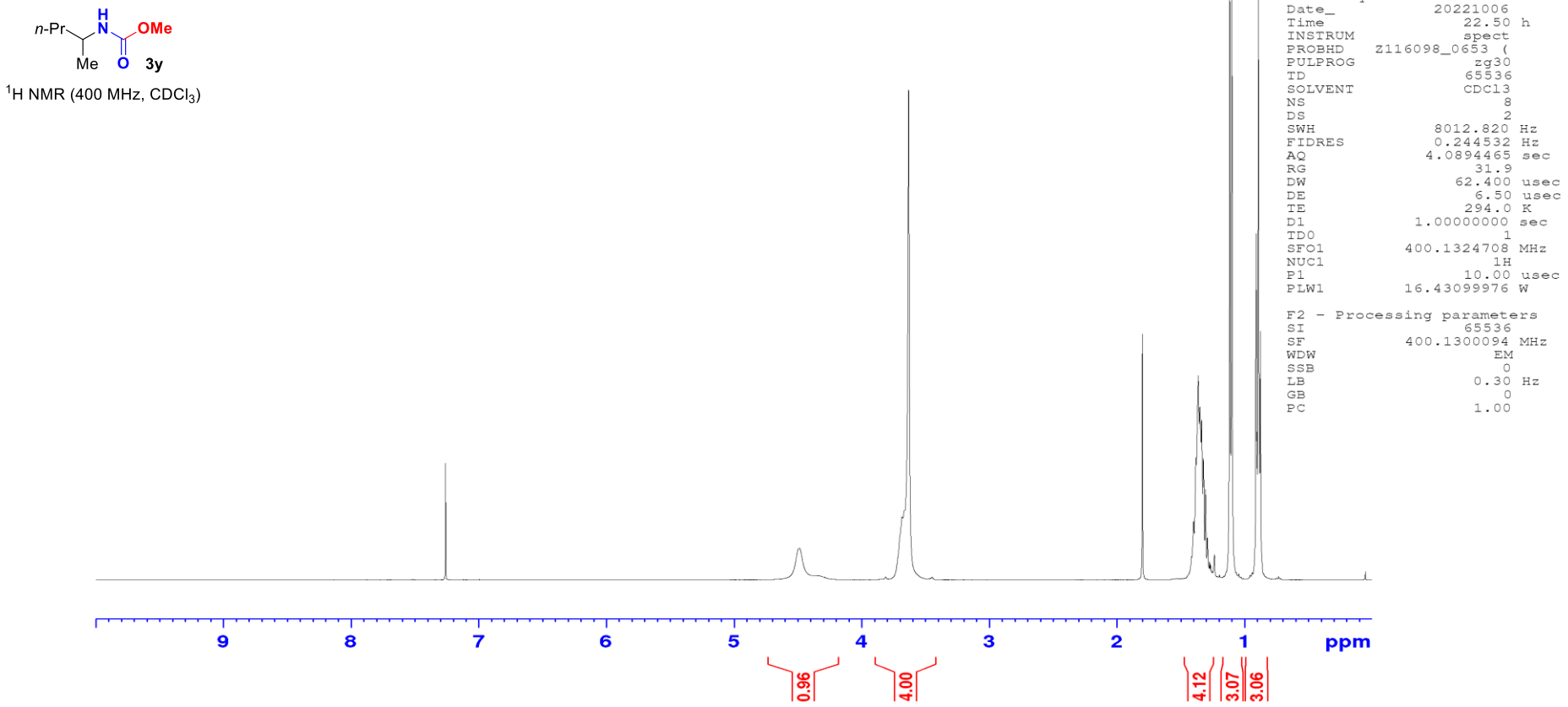


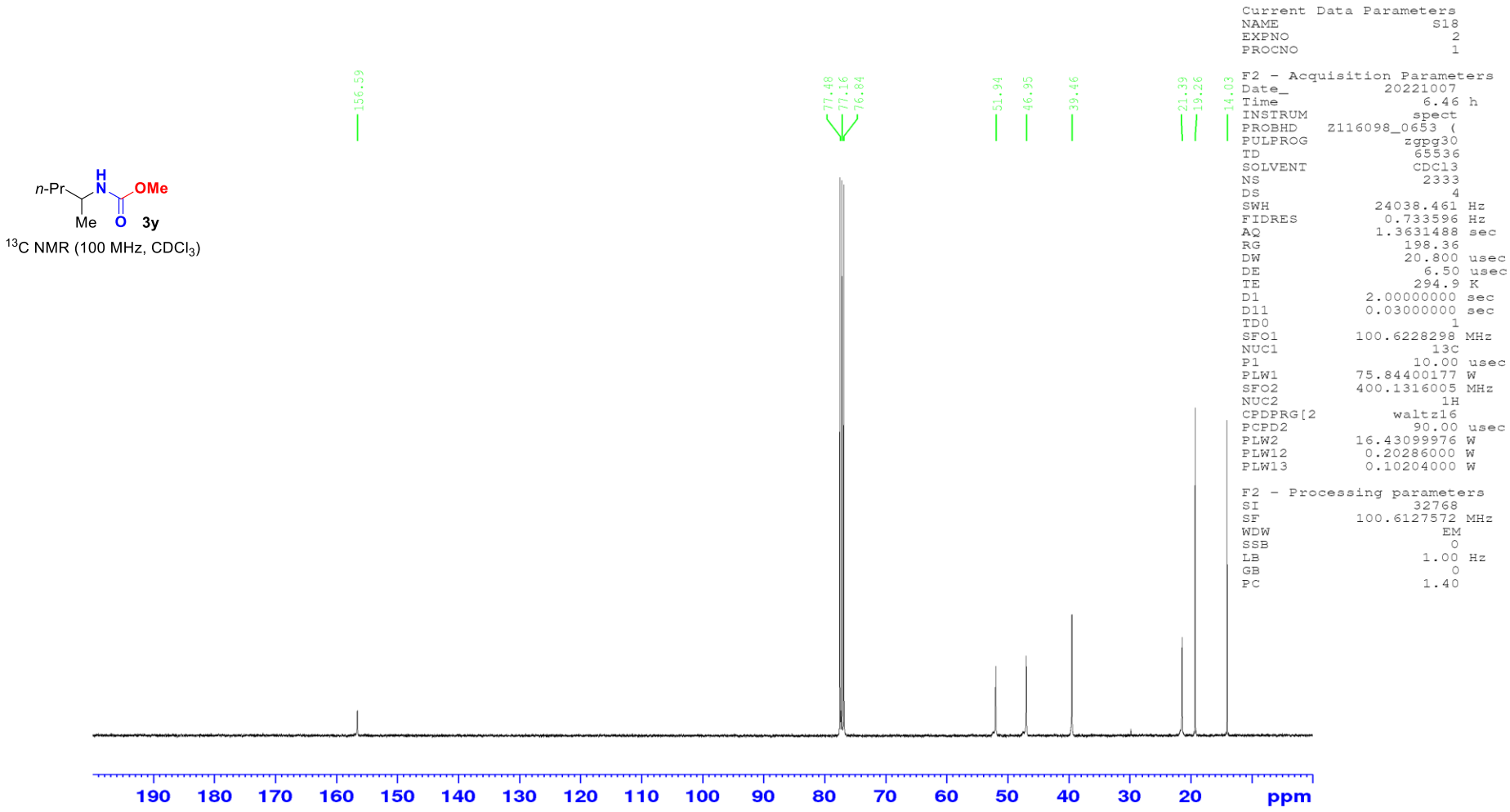
3x

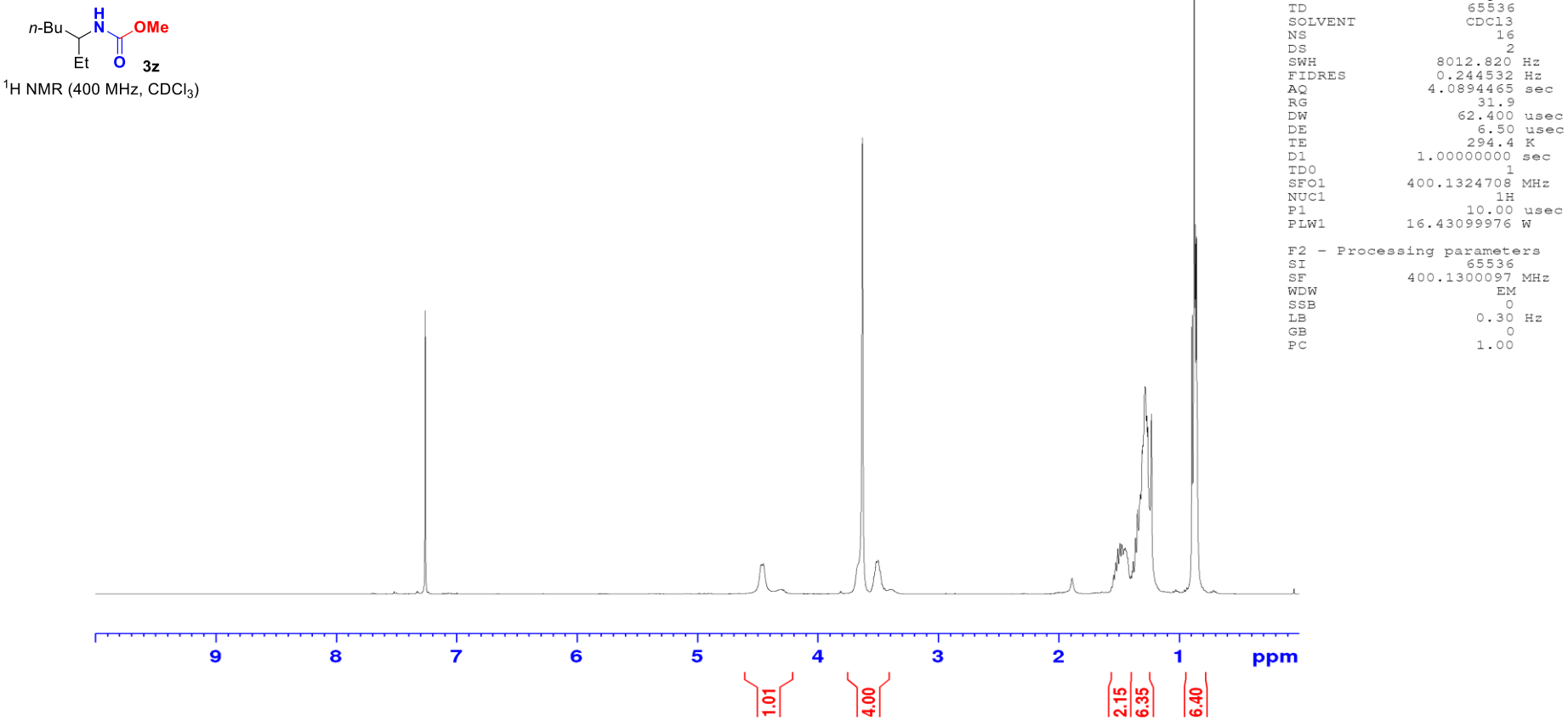
^{13}C NMR (100 MHz, $CDCl_3$)



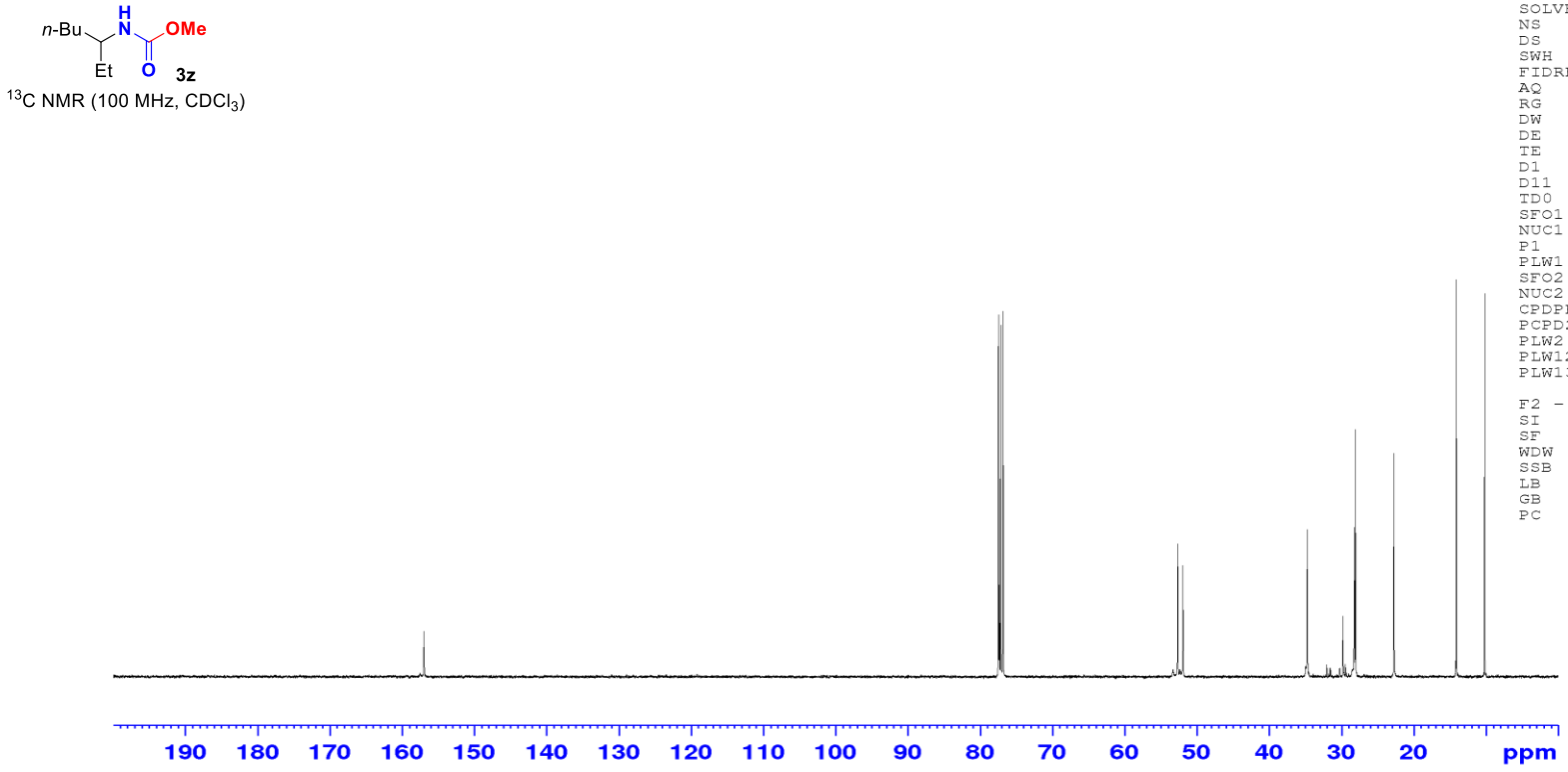

```
Current Data Parameters
NAME                  S22-1
EXPNO                     2
PROCNO                    1

F2 - Acquisition Parameters
Date_              20230719
Time                  2.05 h
INSTRUM               spect
PROBHD    Z116098_0653 (
PULPROG             zgpg30
TD                   65536
SOLVENT              CDCl3
NS                    3333
DS                       4
SWH              24038.461 Hz
FIDRES            0.733596 Hz
AQ                1.3631488 sec
RG                   198.36
DW                   20.800 usec
DE                    6.50 usec
TE                   293.2 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                      1
SFO1          100.6228298 MHz
NUC1                  13C
P1                   10.00 usec
PLW1          75.84400177 W
SFO2          400.1316005 MHz
NUC2                  1H
CPDPRG[2          waltz16
PCPD2                90.00 usec
PLW2          16.43099976 W
PLW12          0.20286000 W
PLW13          0.10204000 W

F2 - Processing parameters
SI                   32768
SF            100.6127586 MHz
WDW                     EM
SSB                      0
LB                    1.00 Hz
GB                       0
PC                    1.40
```


n-Pr
H
N
OMe
Me
O
3y
1H NMR (400 MHz, CDCl3)
7.260
4.487
3.682
3.631
1.414
1.399
1.379
1.362
1.351
1.348
1.335
1.323
1.320
1.305
1.290
1.273
1.266
1.114
Current Data Parameters
NAME S18
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20221006
Time 22.50 h
INSTRUM spect
PROBHD Z116098_0653 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 8012.820 Hz
FIDRES 0.244532 Hz
AQ 4.0894465 sec
RG 31.9
DW 62.400 usec
DE 6.50 usec
TE 294.0 K
D1 1.00000000 sec
TD0 1
SFO1 400.1324708 MHz
NUC1 1H
P1 10.00 usec
PLW1 16.43099976 W
F2 - Processing parameters
SI 65536
SF 400.1300094 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
9
8
7
6
5
4
3
2
1
ppm
0.96
4.00
4.12
3.07
3.06

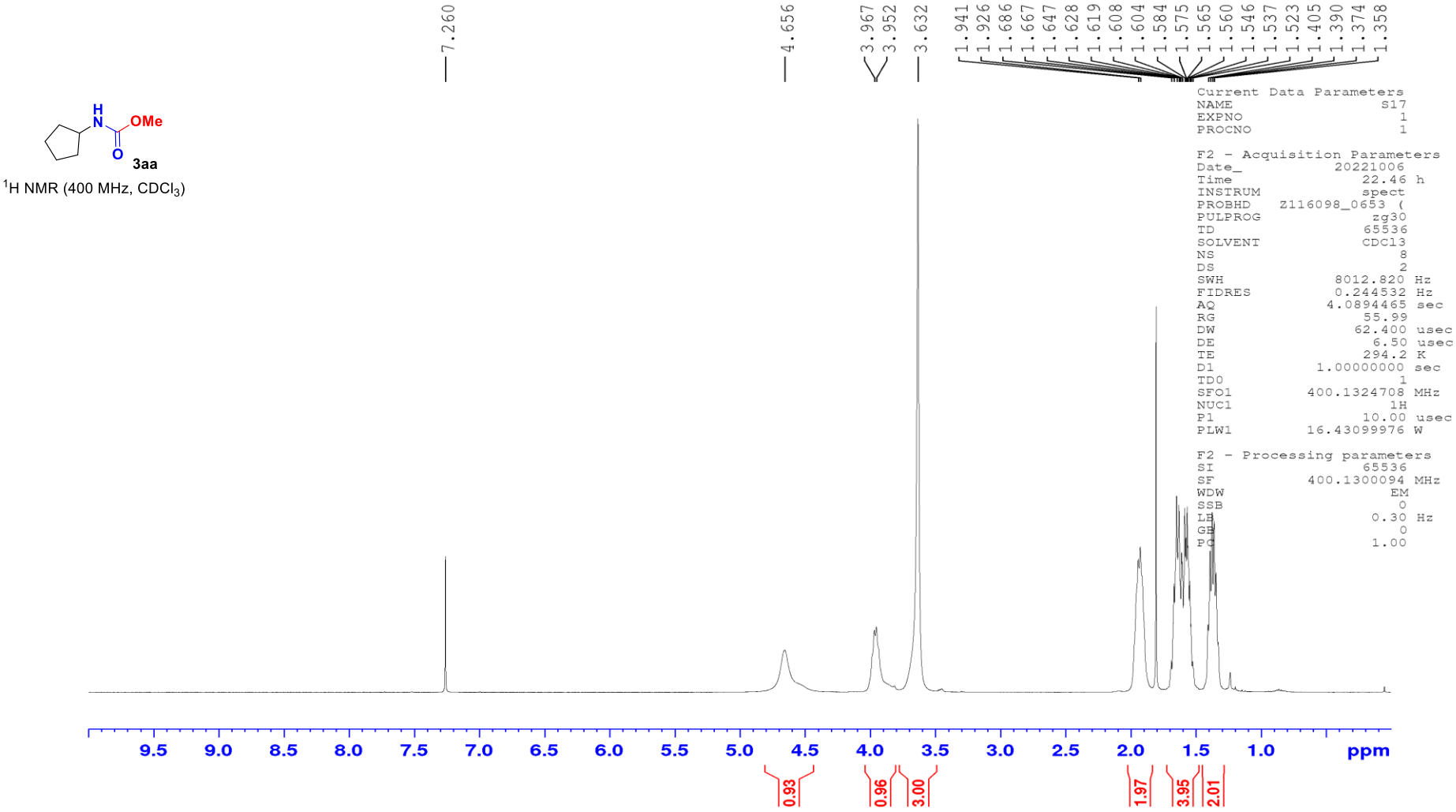

n-Pr
H
N
OMe
Me
O
3y
13C NMR (100 MHz, CDCl3)
156.59
77.48
77.16
76.84
51.94
46.95
39.46
21.39
19.26
14.03
Current Data Parameters
NAME S18
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20221007
Time 6.46 h
INSTRUM spect
PROBHD Z116098_0653 (
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 2333
DS 4
SWH 24038.461 Hz
FIDRES 0.733596 Hz
AQ 1.3631488 sec
RG 198.36
DW 20.800 usec
DE 6.50 usec
TE 294.9 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 100.6228298 MHz
NUC1 13C
P1 10.00 usec
PLW1 75.84400177 W
SFO2 400.1316005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 90.00 usec
PLW2 16.43099976 W
PLW12 0.20286000 W
PLW13 0.10204000 W
F2 - Processing parameters
SI 32768
SF 100.6127572 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm




```
Current Data Parameters
NAME                      S15
EXPNO                       1
PROCNO                      1

F2 - Acquisition Parameters
Date_              20220902
Time                 21.55 h
INSTRUM                spect
PROBHD    Z116098_0653 (
PULPROG                zg30
TD                    65536
SOLVENT               CDCl3
NS                       16
DS                        2
SWH                8012.820 Hz
FIDRES            0.244532 Hz
AQ                4.0894465 sec
RG                     31.9
DW                   62.400 usec
DE                    6.50 usec
TE                   294.4 K
D1              1.00000000 sec
TD0                       1
SFO1           400.1324708 MHz
NUC1                     1H
P1                   10.00 usec
PLW1           16.43099976 W

F2 - Processing parameters
SI                    65536
SF             400.1300097 MHz
WDW                      EM
SSB                       0
LB                     0.30 Hz
GB                        0
PC                     1.00
```

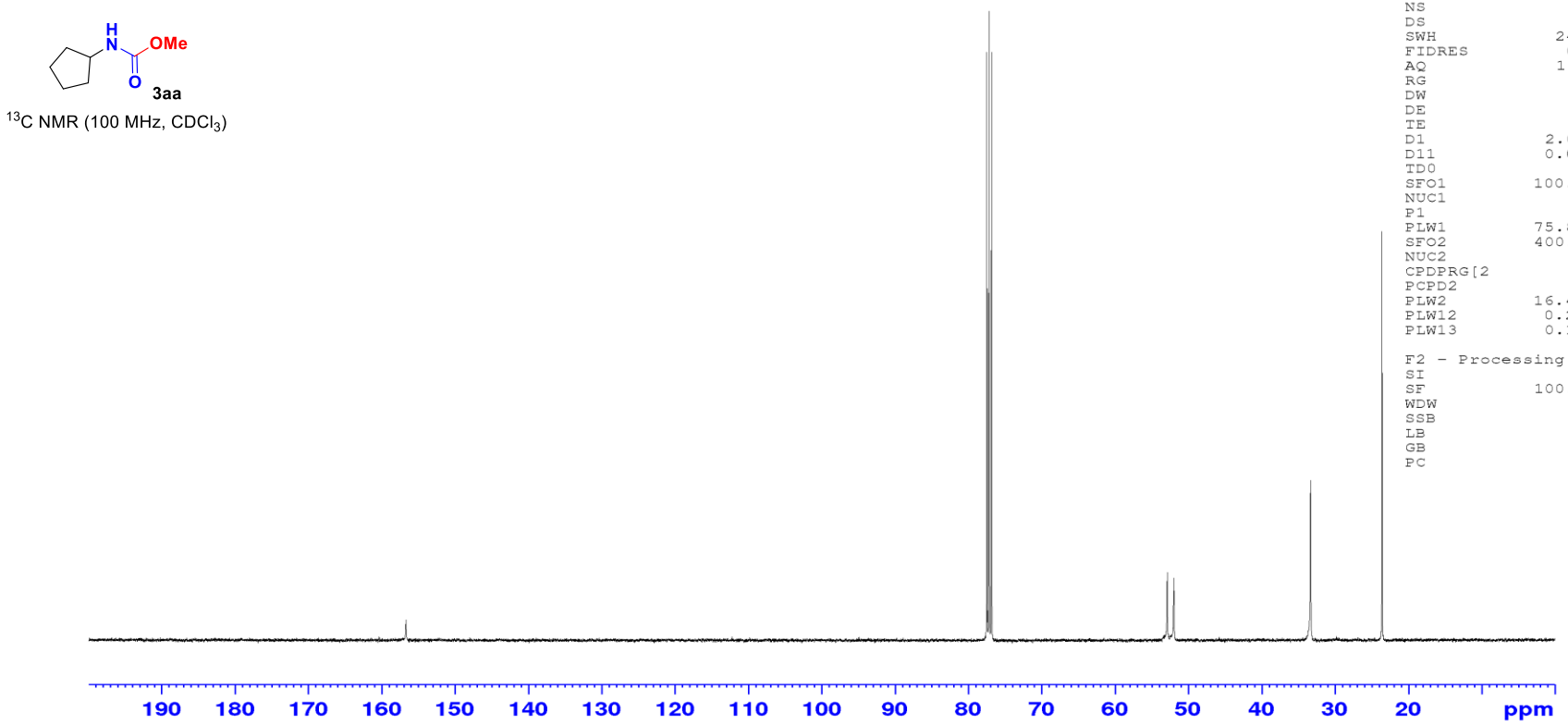



```
Current Data Parameters
NAME                      S15
EXPNO                       2
PROCNO                      1

F2 - Acquisition Parameters
Date_              20220902
Time                 22.55 h
INSTRUM                spect
PROBHD    Z116098_0653 (
PULPROG              zgpg30
TD                    65536
SOLVENT               CDCl3
NS                     1024
DS                        4
SWH              24038.461 Hz
FIDRES            0.733596 Hz
AQ                1.3631488 sec
RG                   198.36
DW                   20.800 usec
DE                    6.50 usec
TE                   294.9 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                       1
SFO1          100.6228298 MHz
NUC1                    13C
P1                   10.00 usec
PLW1          75.84400177 W
SFO2          400.1316005 MHz
NUC2                    1H
CPDPRG[2           waltz16
PCPD2                90.00 usec
PLW2          16.43099976 W
PLW12          0.20286000 W
PLW13          0.10204000 W

F2 - Processing parameters
SI                    32768
SF            100.6127583 MHz
WDW                      EM
SSB                       0
LB                    1.00 Hz
GB                        0
PC                    1.40
```



3aa

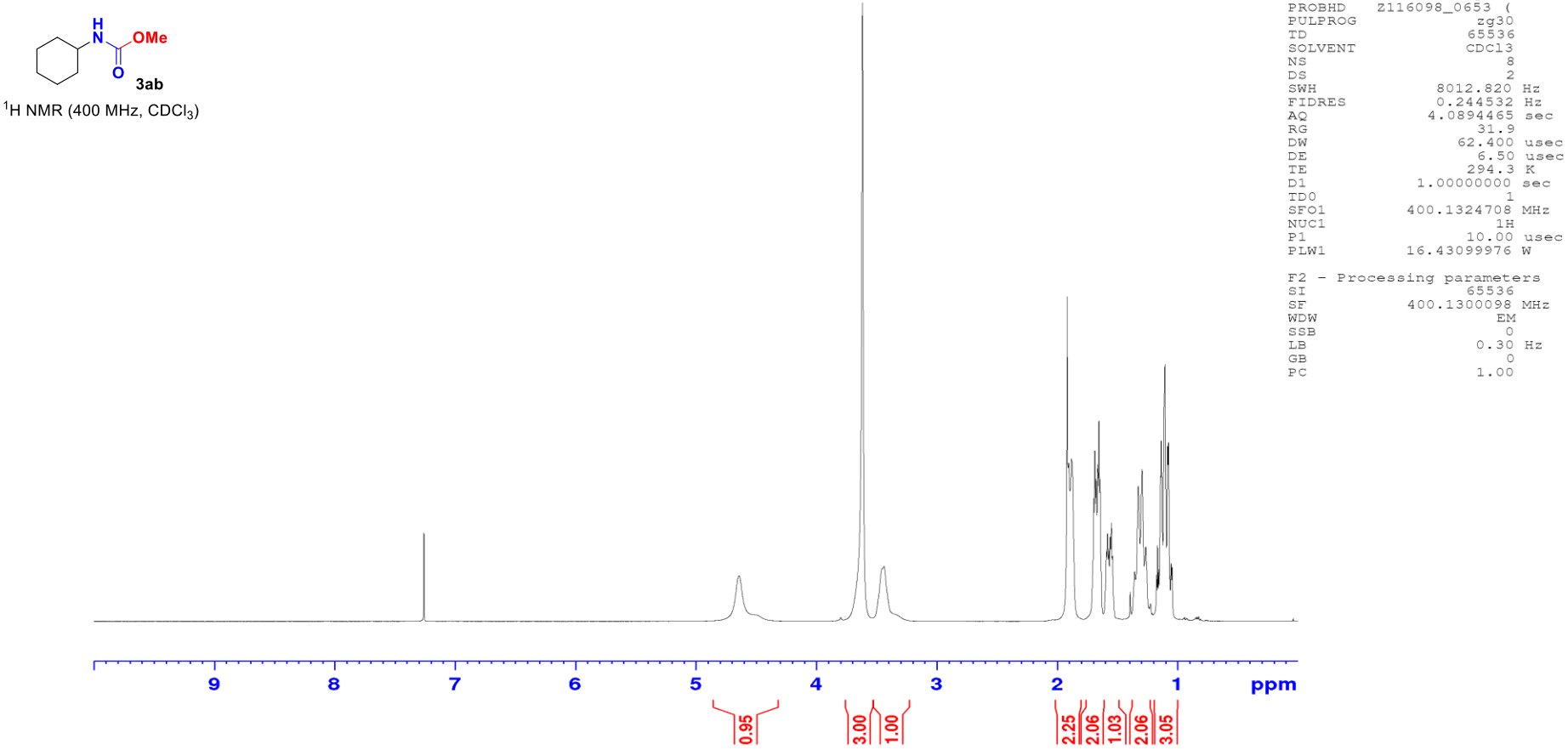
^{1}H NMR (400 MHz, $CDCl_3$)



3aa

^{13}C NMR (100 MHz, $CDCl_3$)

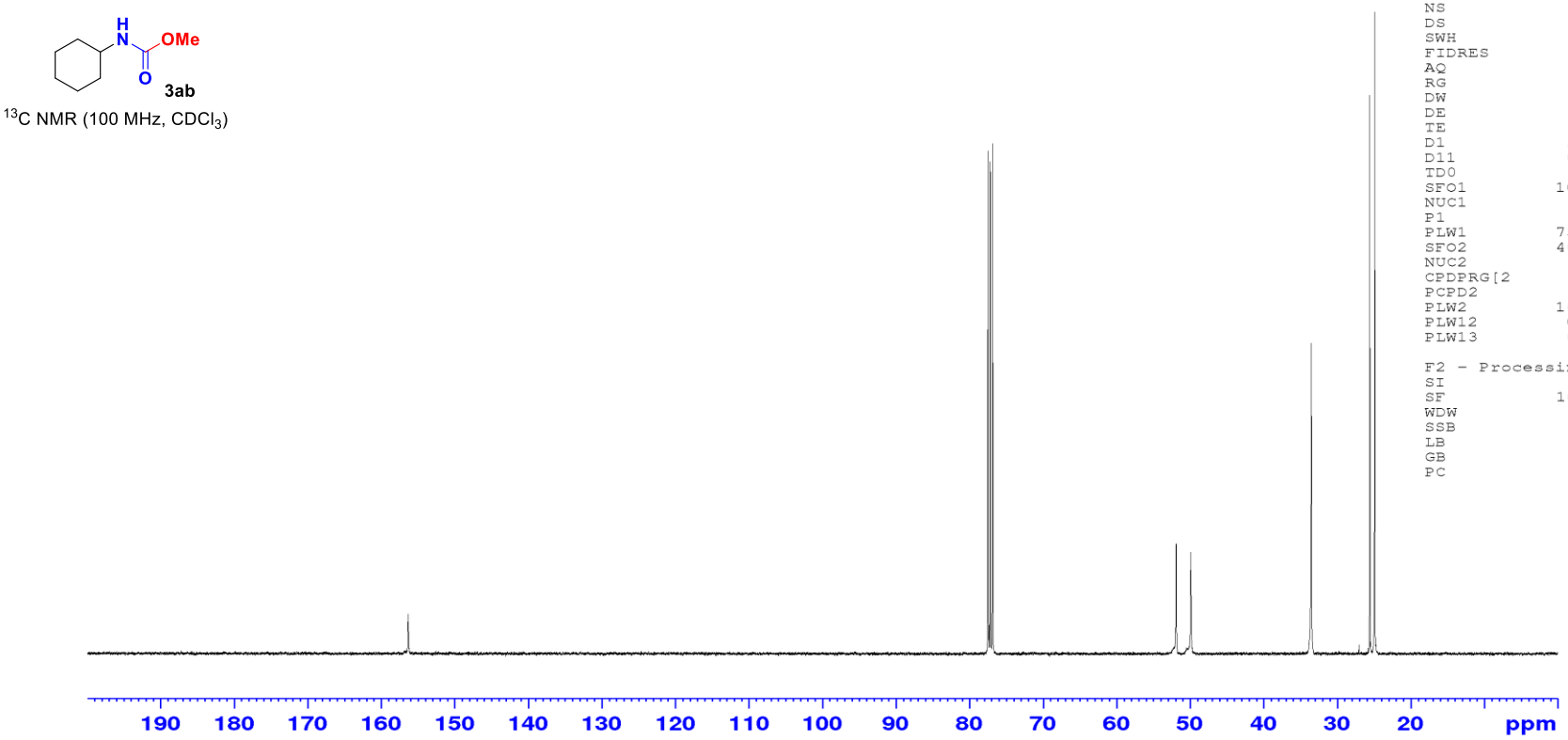
```
Current Data Parameters
NAME                   S17
EXPNO                    2
PROCNO                   1

F2 - Acquisition Parameters
Date_               20221007
Time                   4.29 h
INSTRUM                spect
PROBHD    Z116098_0653 (
PULPROG              zgpg30
TD                    65536
SOLVENT               CDCl3
NS                     2333
DS                        4
SWH             24038.461 Hz
FIDRES          0.733596 Hz
AQ              1.3631488 sec
RG                   198.36
DW                 20.800 usec
DE                   6.50 usec
TE                   295.0 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                       1
SFO1          100.6228298 MHz
NUC1                    13C
P1                   10.00 usec
PLW1          75.84400177 W
SFO2          400.1316005 MHz
NUC2                    1H
CPDPRG[2           waltz16
PCPD2                90.00 usec
PLW2          16.43099976 W
PLW12          0.20286000 W
PLW13          0.10204000 W

F2 - Processing parameters
SI                    32768
SF            100.6127579 MHz
WDW                      EM
SSB                       0
LB                     1.00 Hz
GB                        0
PC                     1.40
```



3ab

^{1}H NMR (400 MHz, $CDCl_3$)

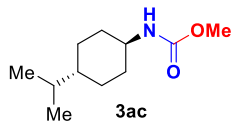
```
Current Data Parameters
NAME                  S16
EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
Date_            20220922
Time                22.18 h
INSTRUM             spect
PROBHD    Z116098_0653 (
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                     8
DS                     2
SWH             8012.820 Hz
FIDRES             0.244532 Hz
AQ              4.0894465 sec
RG                  31.9
DW                 62.400 usec
DE                  6.50 usec
TE                294.3 K
D1             1.00000000 sec
TD0                    1
SFO1          400.1324708 MHz
NUC1                  1H
P1                 10.00 usec
PLW1          16.43099976 W

F2 - Processing parameters
SI                 65536
SF            400.1300098 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```



3ab

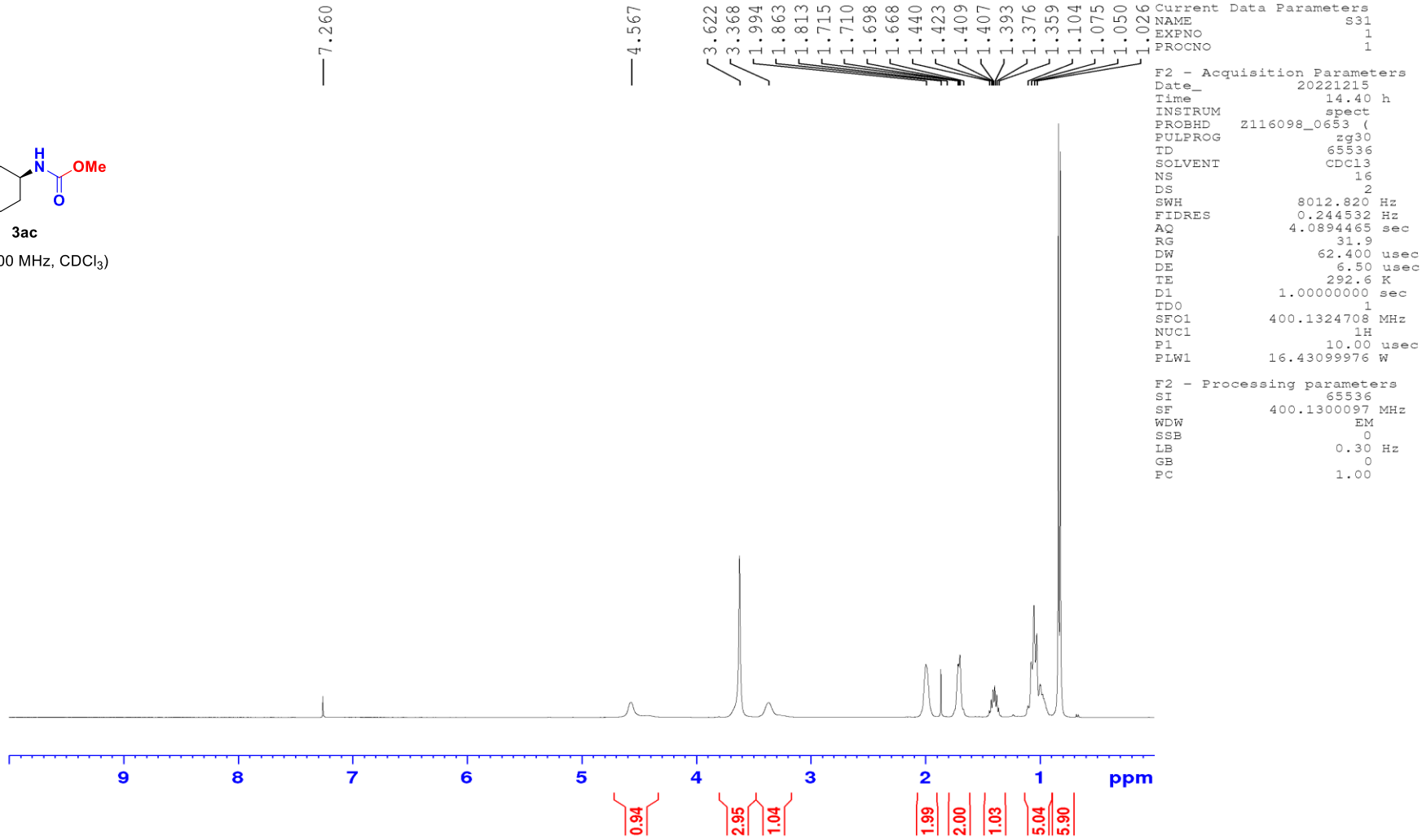
^{13}C NMR (100 MHz, $CDCl_3$)

```
Current Data Parameters
NAME                   S16
EXPNO                    2
PROCNO                   1

F2 - Acquisition Parameters
Date_               20220923
Time                   1.38 h
INSTRUM                spect
PROBHD    Z116098_0653 (
PULPROG              zgpg30
TD                    65536
SOLVENT               CDCl3
NS                     1888
DS                        4
SWH               24038.461 Hz
FIDRES             0.733596 Hz
AQ                 1.3631488 sec
RG                    198.36
DW                    20.800 usec
DE                     6.50 usec
TE                    295.2 K
D1              2.00000000 sec
D11             0.03000000 sec
TD0                       1
SFO1           100.6228298 MHz
NUC1                    13C
P1                    10.00 usec
PLW1           75.84400177 W
SFO2           400.1316005 MHz
NUC2                    1H
CPDPRG[2           waltz16
PCPD2                 90.00 usec
PLW2           16.43099976 W
PLW12           0.20286000 W
PLW13           0.10204000 W

F2 - Processing parameters
SI                    32768
SF             100.6127597 MHz
WDW                     EM
SSB                      0
LB                    1.00 Hz
GB                       0
PC                    1.40
```

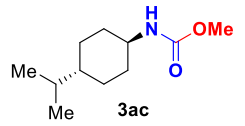


3ac

^{1}H NMR (400 MHz, $CDCl_3$)

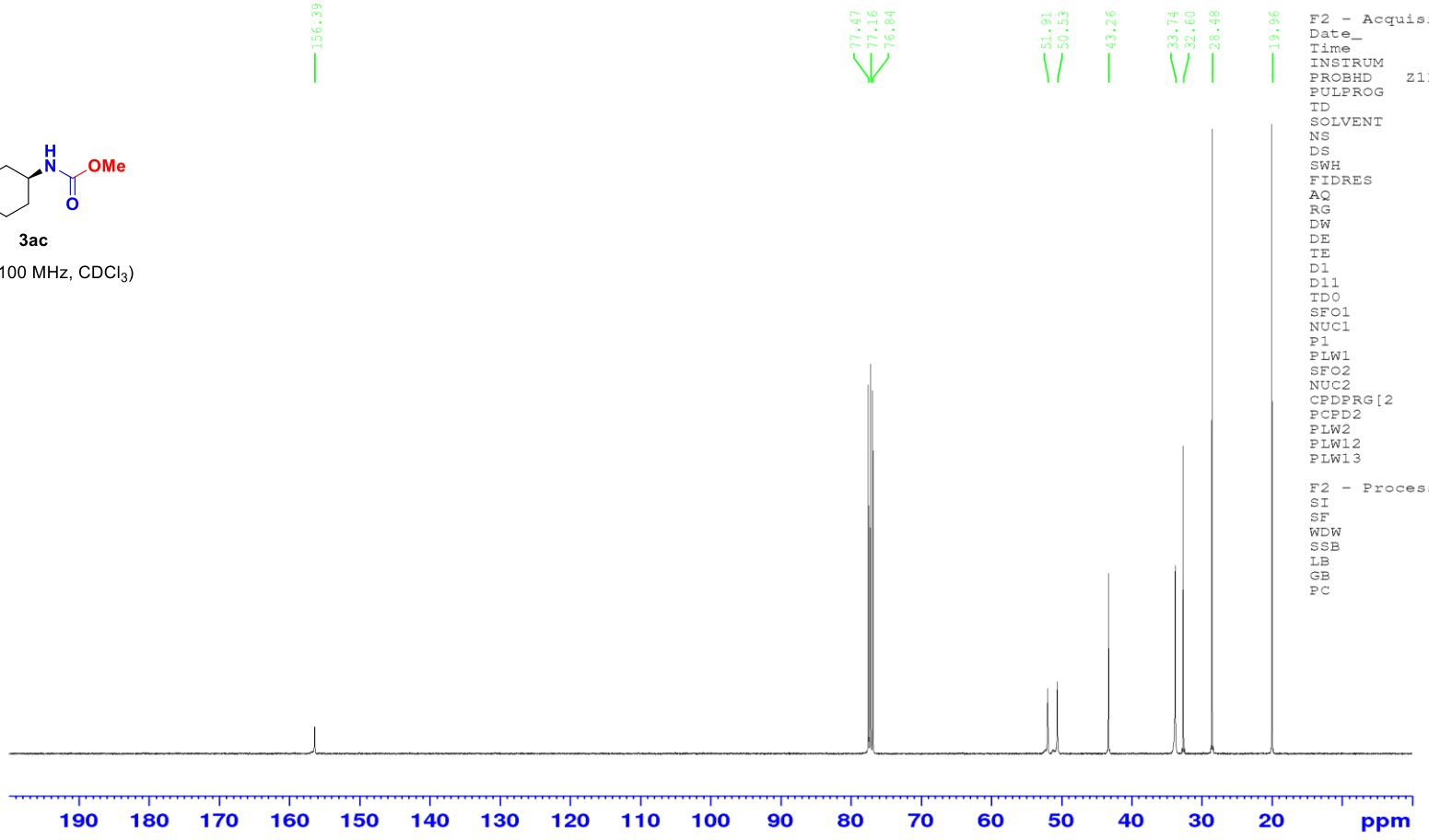



```
Current Data Parameters
NAME                  S31
EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
Date_              20221215
Time                 14.40 h
INSTRUM              spect
PROBHD   Z116098_0653 (
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                     16
DS                      2
SWH               8012.820 Hz
FIDRES               0.244532 Hz
AQ                4.0894465 sec
RG                     31.9
DW                   62.400 usec
DE                    6.50 usec
TE                   292.6 K
D1                1.00000000 sec
TD0                     1
SFO1           400.1324708 MHz
NUC1                   1H
P1                   10.00 usec
PLW1           16.43099976 W

F2 - Processing parameters
SI                  65536
SF             400.1300097 MHz
WDW                    EM
SSB                     0
LB                   0.30 Hz
GB                      0
PC                   1.00
```

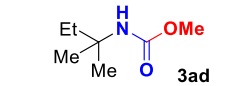


3ac

^{13}C NMR (100 MHz, $CDCl_3$)

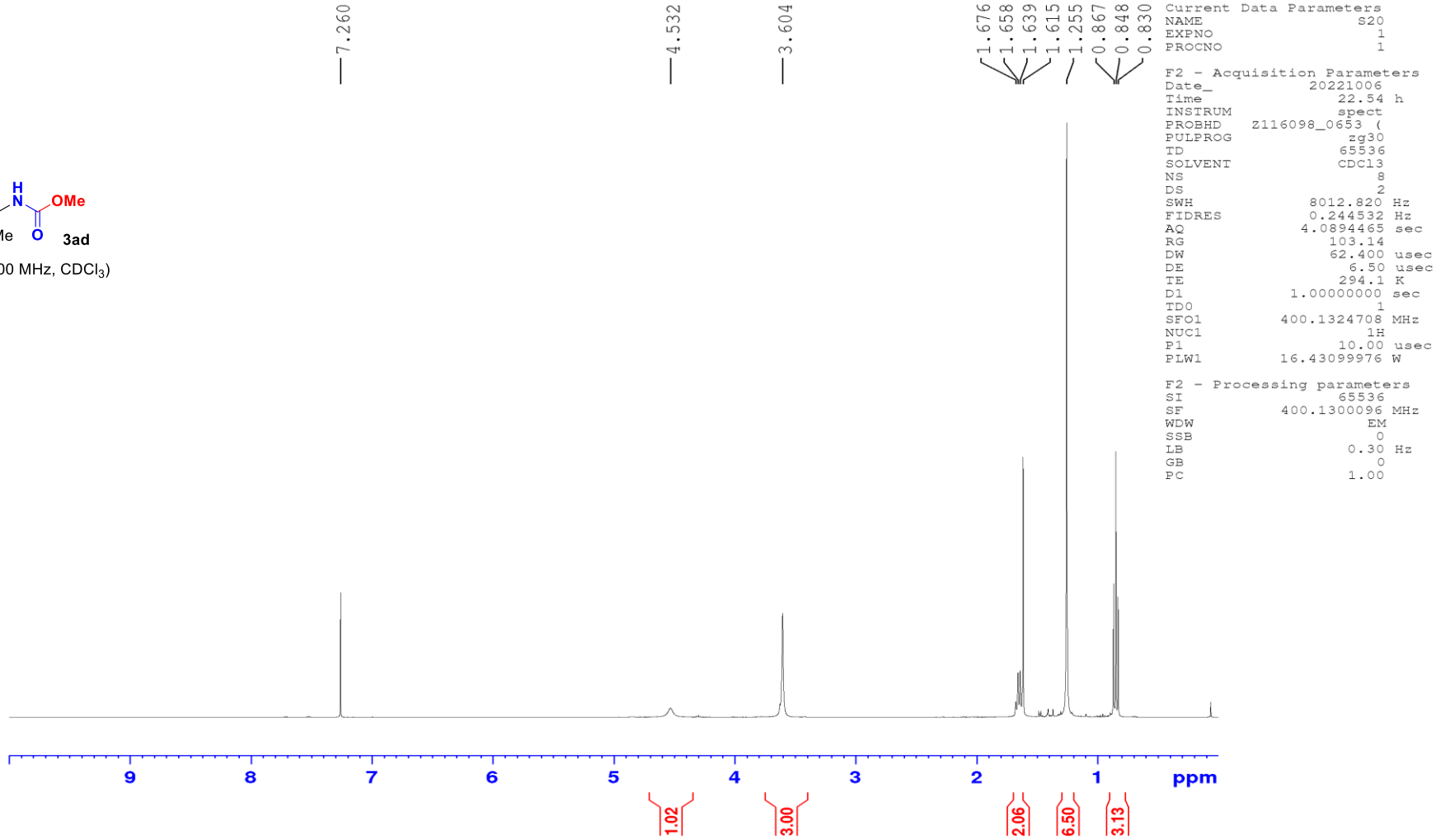



```
Current Data Parameters
NAME                  S31
EXPNO                   2
PROCNO                  1

F2 - Acquisition Parameters
Date_            20221215
Time                17.52 h
INSTRUM             spect
PROBHD   Z116098_0653 (
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                  3333
DS                     4
SWH           24038.461 Hz
FIDRES         0.733596 Hz
AQ             1.3631488 sec
RG                198.36
DW                20.800 usec
DE                 6.50 usec
TE                293.5 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                    1
SFO1        100.6228298 MHz
NUC1                13C
P1                10.00 usec
PLW1        75.84400177 W
SFO2        400.1316005 MHz
NUC2                1H
CPDPRG[2        waltz16
PCPD2             90.00 usec
PLW2        16.43099976 W
PLW12        0.20286000 W
PLW13        0.10204000 W

F2 - Processing parameters
SI                 32768
SF          100.6127594 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```

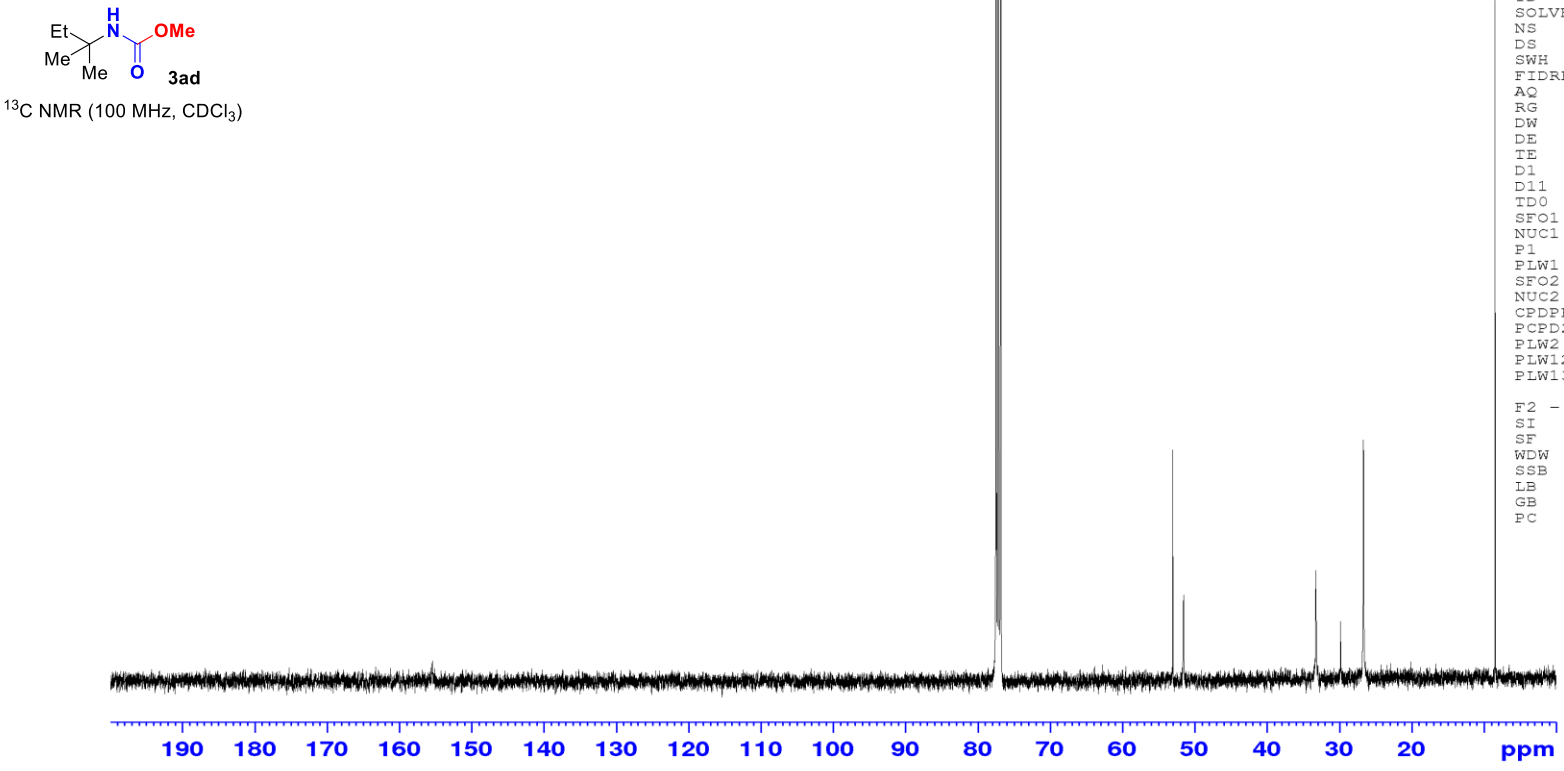


3ad

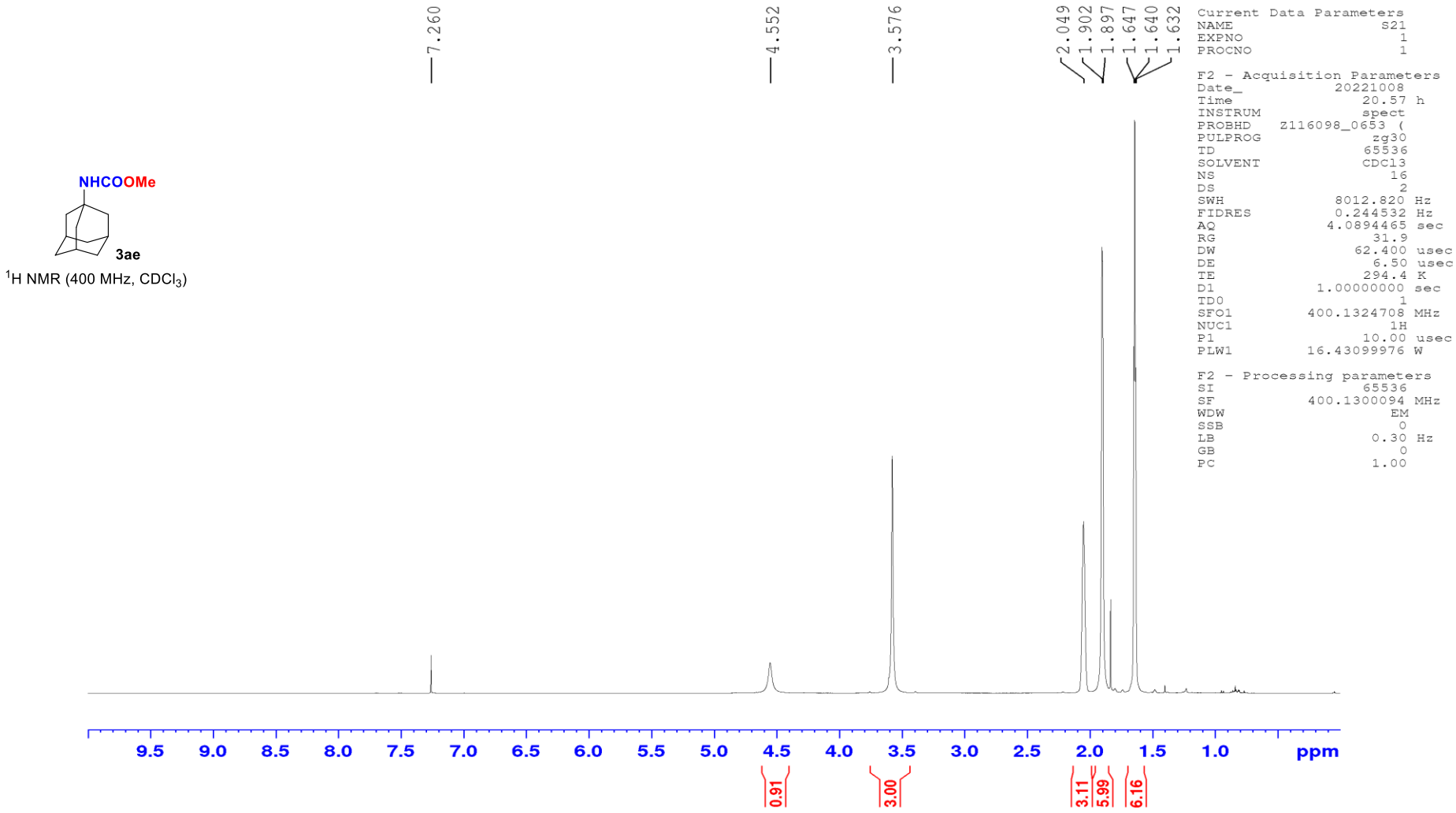
^{1}H NMR (400 MHz, $CDCl_3$)

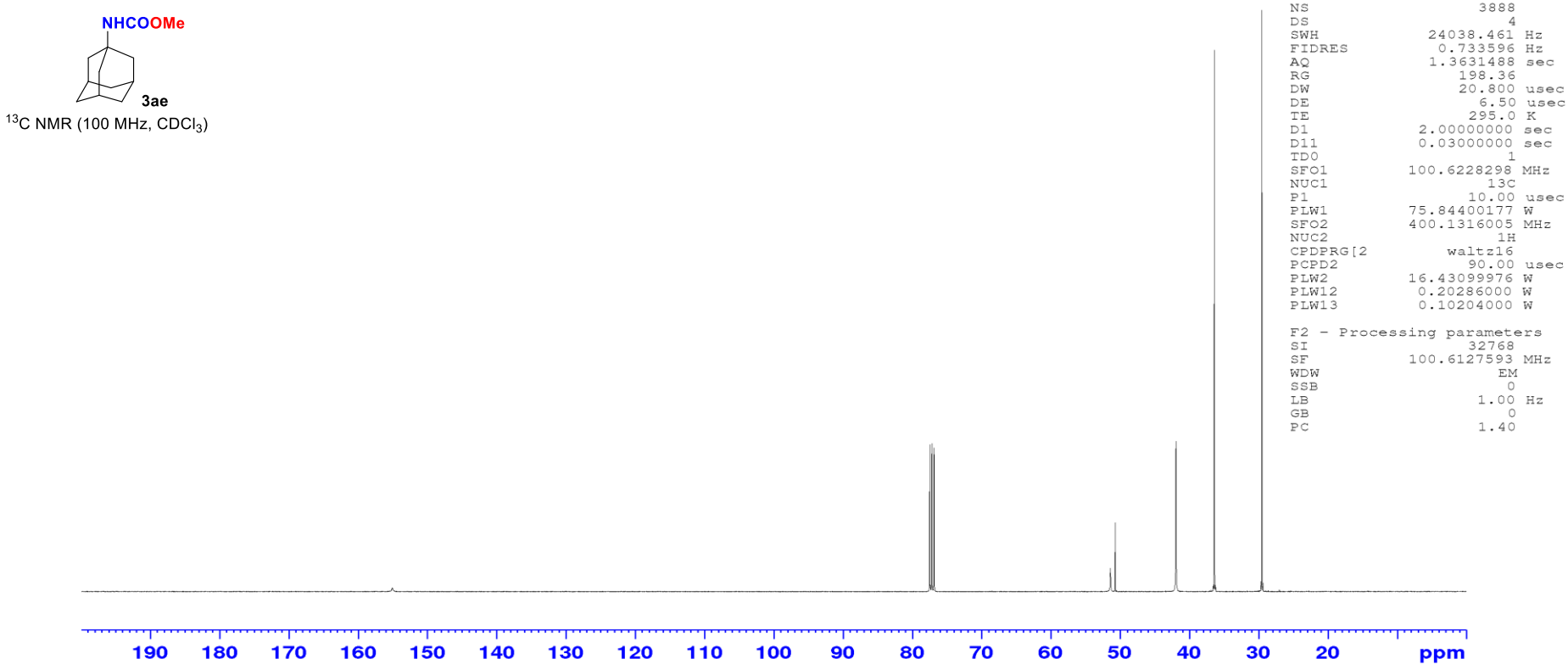



```
Current Data Parameters
NAME                      S20
EXPNO                       1
PROCNO                      1

F2 - Acquisition Parameters
Date_              20221006
Time                  22.54 h
INSTRUM                spect
PROBHD    Z116098_0653 (
PULPROG                zg30
TD                    65536
SOLVENT               CDCl3
NS                        8
DS                        2
SWH                8012.820 Hz
FIDRES            0.244532 Hz
AQ                4.0894465 sec
RG                   103.14
DW                   62.400 usec
DE                    6.50 usec
TE                   294.1 K
D1              1.00000000 sec
TD0                       1
SFO1           400.1324708 MHz
NUC1                    1H
P1                   10.00 usec
PLW1           16.43099976 W

F2 - Processing parameters
SI                    65536
SF             400.1300096 MHz
WDW                     EM
SSB                       0
LB                     0.30 Hz
GB                        0
PC                     1.00
```

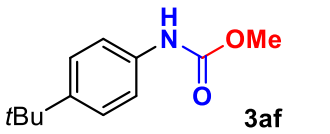



```
Current Data Parameters
NAME                      S20
EXPNO                       3
PROCNO                      1

F2 - Acquisition Parameters
Date_              20221007
Time                   2.12 h
INSTRUM                spect
PROBHD    Z116098_0653 (
PULPROG              zgpg30
TD                    65536
SOLVENT               CDCl3
NS                     3333
DS                        4
SWH              24038.461 Hz
FIDRES            0.733596 Hz
AQ                1.3631488 sec
RG                   198.36
DW                  20.800 usec
DE                    6.50 usec
TE                   294.7 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                       1
SFO1          100.6228298 MHz
NUC1                    13C
P1                    10.00 usec
PLW1          75.84400177 W
SFO2          400.1316005 MHz
NUC2                    1H
CPDPRG[2           waltz16
PCPD2                 90.00 usec
PLW2          16.43099976 W
PLW12          0.20286000 W
PLW13          0.10204000 W

F2 - Processing parameters
SI                    32768
SF            100.6127559 MHz
WDW                     EM
SSB                      0
LB                    1.00 Hz
GB                       0
PC                    1.40
```


NHCOOMe
3ae
1H NMR (400 MHz, CDCl3)
7.260
4.552
3.576
2.049
1.902
1.897
1.647
1.640
1.632
Current Data Parameters
NAME S21
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20221008
Time 20.57 h
INSTRUM spect
PROBHD Z116098_0653 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.244532 Hz
AQ 4.0894465 sec
RG 31.9
DW 62.400 usec
DE 6.50 usec
TE 294.4 K
D1 1.00000000 sec
TD0 1
SFO1 400.1324708 MHz
NUC1 1H
P1 10.00 usec
PLW1 16.43099976 W
F2 - Processing parameters
SI 65536
SF 400.1300094 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
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 ppm
0.91
3.00
3.11
5.99
6.16

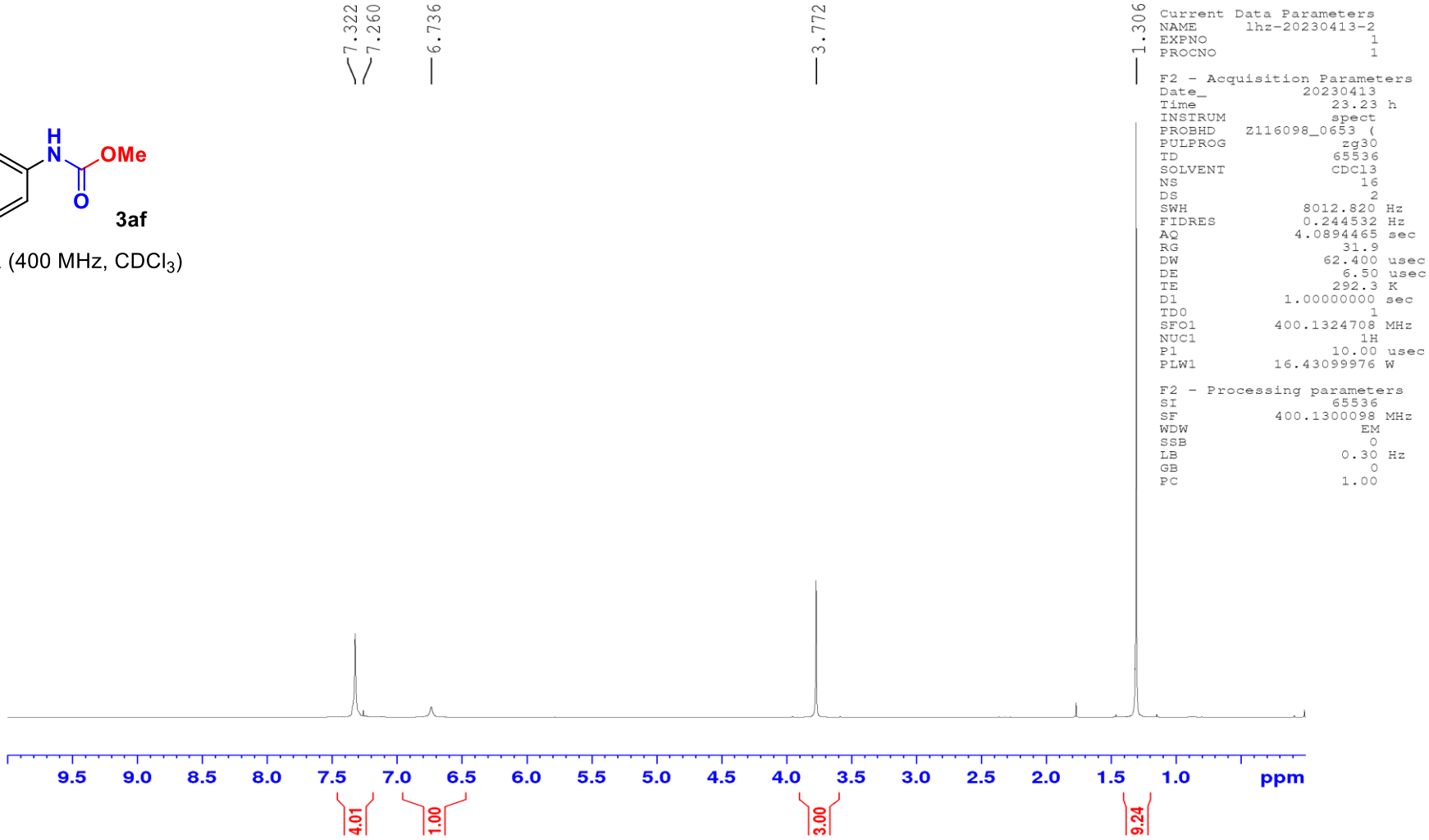



```
Current Data Parameters
NAME                  S21
EXPNO                   2
PROCNO                  1

F2 - Acquisition Parameters
Date_            20221009
Time                0.40 h
INSTRUM            spect
PROBHD    Z116098_0653 (
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                 3888
DS                    4
SWH           24038.461 Hz
FIDRES         0.733596 Hz
AQ             1.3631488 sec
RG                198.36
DW                20.800 usec
DE                 6.50 usec
TE                295.0 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                   1
SFO1        100.6228298 MHz
NUC1               13C
P1                10.00 usec
PLW1        75.84400177 W
SFO2        400.1316005 MHz
NUC2               1H
CPDPRG[2       waltz16
PCPD2             90.00 usec
PLW2        16.43099976 W
PLW12        0.20286000 W
PLW13        0.10204000 W

F2 - Processing parameters
SI                32768
SF          100.6127593 MHz
WDW                EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```

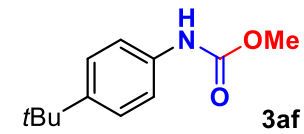


3af

^{1}H NMR (400 MHz, $CDCl_3$)

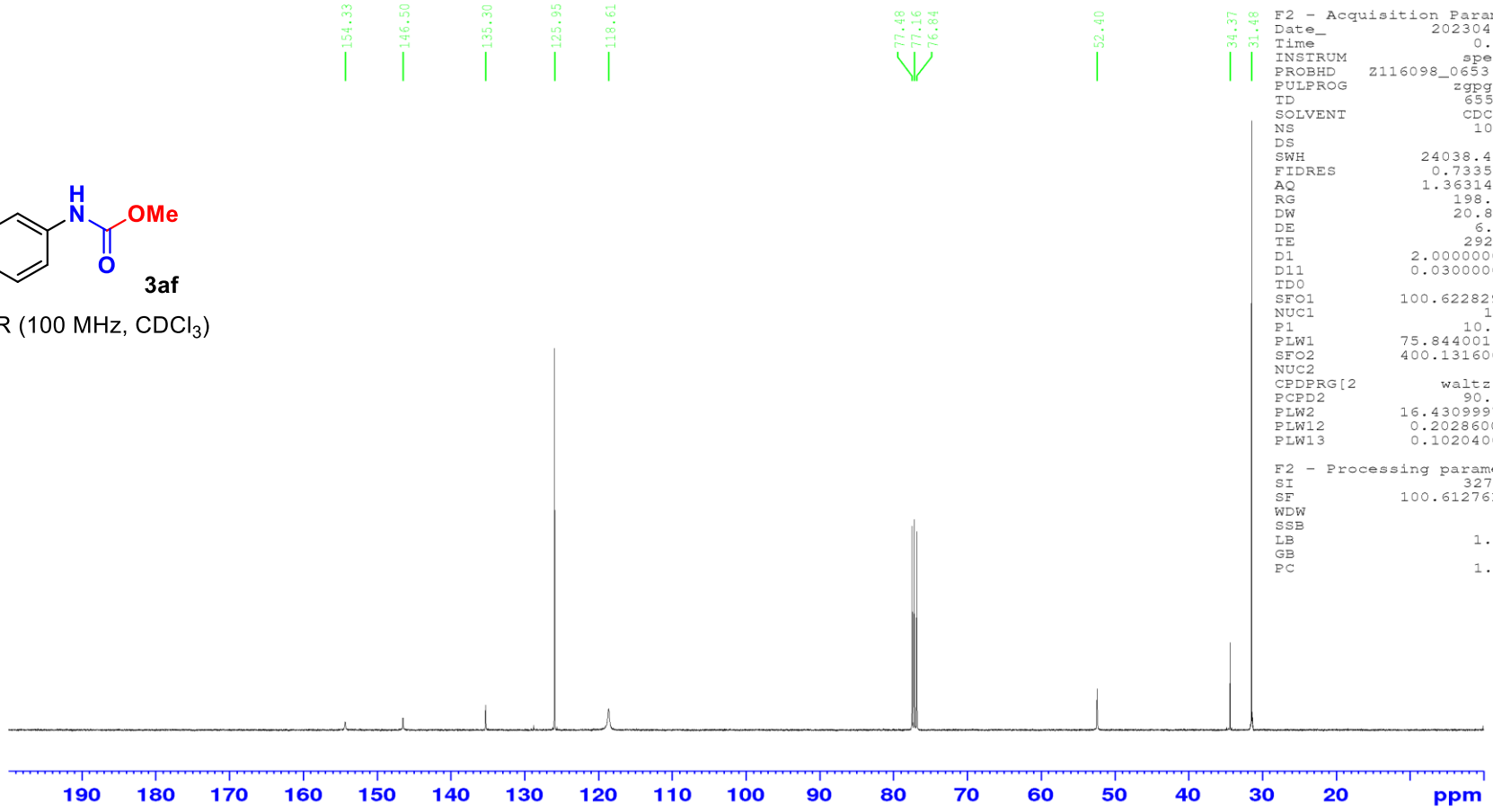



```
Current Data Parameters
NAME      lhz-20230413-2
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20230413
Time              23.23 h
INSTRUM             spect
PROBHD    Z116098_0653 (
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             8012.820 Hz
FIDRES          0.244532 Hz
AQ              4.0894465 sec
RG                  31.9
DW                62.400 usec
DE                 6.50 usec
TE                292.3 K
D1             1.00000000 sec
TD0                    1
SFO1          400.1324708 MHz
NUC1                  1H
P1                 10.00 usec
PLW1          16.43099976 W

F2 - Processing parameters
SI                 65536
SF            400.1300098 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```

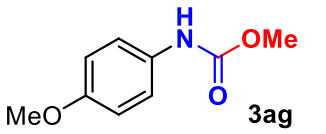


3af

^{13}C NMR (100 MHz, $CDCl_3$)

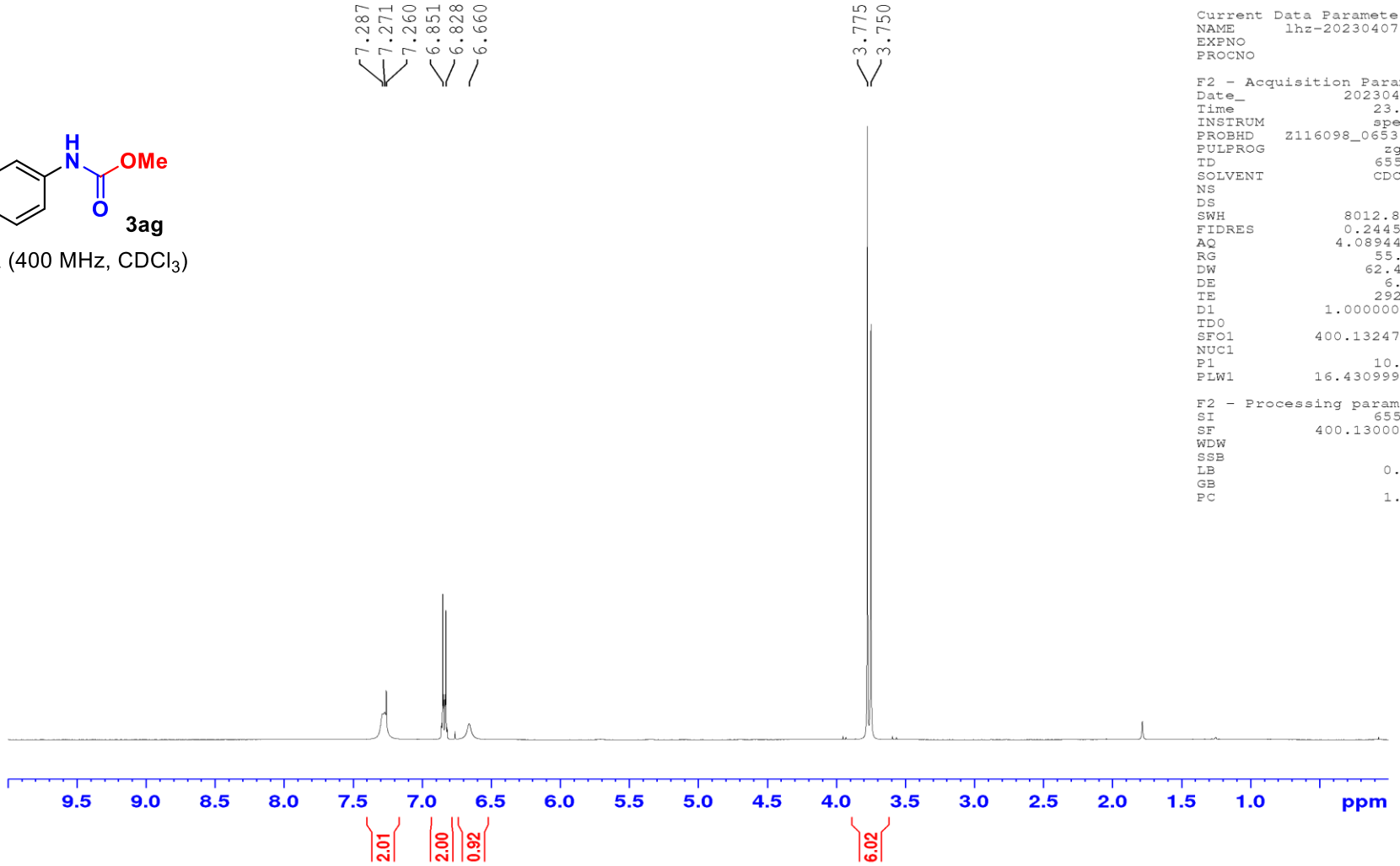



```
Current Data Parameters
NAME      lhz-20230413-2
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20230414
Time              0.23 h
INSTRUM           spect
PROBHD    Z116098_0653 (
PULPROG         zgpg30
TD               65536
SOLVENT          CDCl3
NS                1024
DS                   4
SWH          24038.461 Hz
FIDRES        0.733596 Hz
AQ            1.3631488 sec
RG               198.36
DW               20.800 usec
DE                6.50 usec
TE               292.5 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                  1
SFO1       100.6228298 MHz
NUC1               13C
P1               10.00 usec
PLW1       75.84400177 W
SFO2       400.1316005 MHz
NUC2               1H
CPDPRG[2       waltz16
PCPD2            90.00 usec
PLW2       16.43099976 W
PLW12       0.20286000 W
PLW13       0.10204000 W

F2 - Processing parameters
SI               32768
SF         100.6127616 MHz
WDW                EM
SSB                 0
LB               1.00 Hz
GB                  0
PC               1.40
```

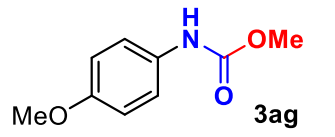


3ag

^{1}H NMR (400 MHz, $CDCl_3$)

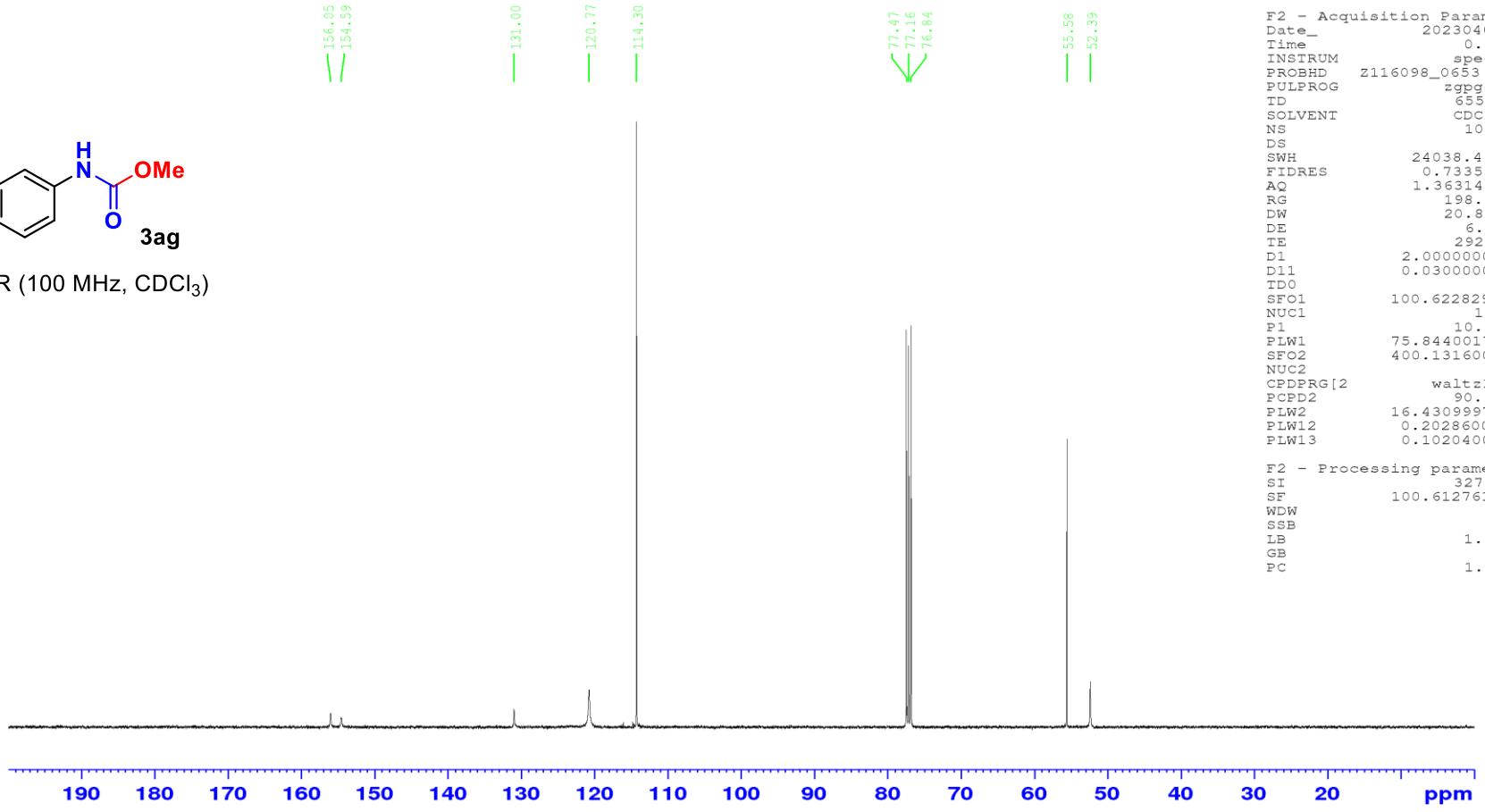



```
Current Data Parameters
NAME      1hz-20230407-2
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20230407
Time              23.39 h
INSTRUM            spect
PROBHD    Z116098_0653 (
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                   16
DS                    2
SWH            8012.820 Hz
FIDRES         0.244532 Hz
AQ             4.0894465 sec
RG                55.99
DW               62.400 usec
DE                6.50 usec
TE               292.2 K
D1            1.00000000 sec
TD0                   1
SFO1         400.1324708 MHz
NUC1                 1H
P1                10.00 usec
PLW1         16.43099976 W

F2 - Processing parameters
SI                65536
SF           400.1300098 MHz
WDW                  EM
SSB                   0
LB                 0.30 Hz
GB                    0
PC                 1.00
```



3ag

^{13}C NMR (100 MHz, $CDCl_3$)




```
Current Data Parameters
NAME      1hz-20230407-2
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20230408
Time                0.38 h
INSTRUM            spect
PROBHD    Z116098_0653 (
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                 1024
DS                    4
SWH           24038.461 Hz
FIDRES         0.733596 Hz
AQ             1.3631488 sec
RG                198.36
DW                20.800 usec
DE                 6.50 usec
TE                292.8 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1
SFO1        100.6228298 MHz
NUC1               13C
P1               10.00 usec
PLW1        75.84400177 W
SFO2        400.1316005 MHz
NUC2               1H
CPDPRG[2       waltz16
PCPD2            90.00 usec
PLW2        16.43099976 W
PLW12        0.20286000 W
PLW13        0.10204000 W

F2 - Processing parameters
SI                32768
SF          100.6127613 MHz
WDW                EM
SSB                 0
LB               1.00 Hz
GB                  0
PC               1.40
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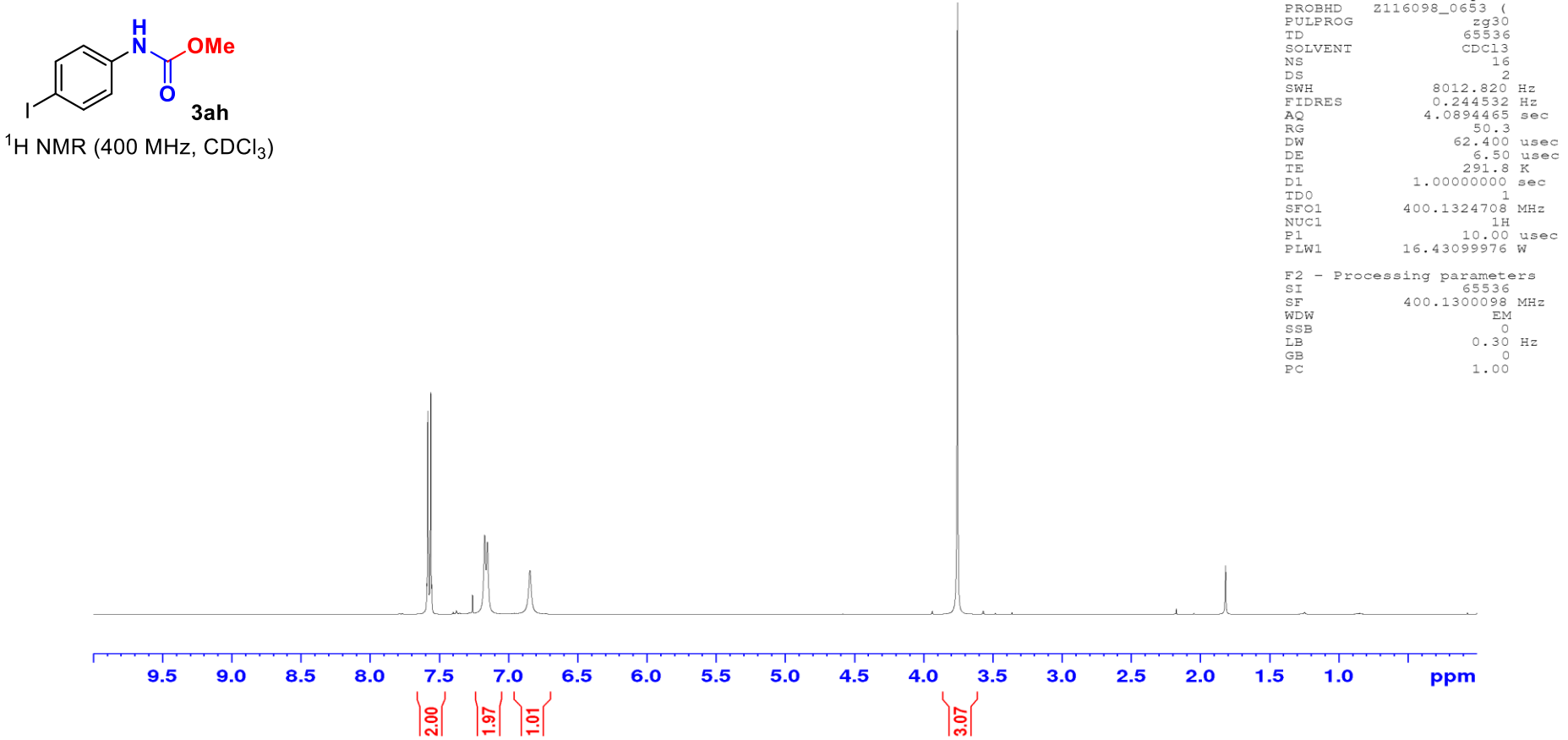
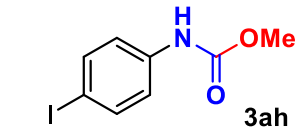
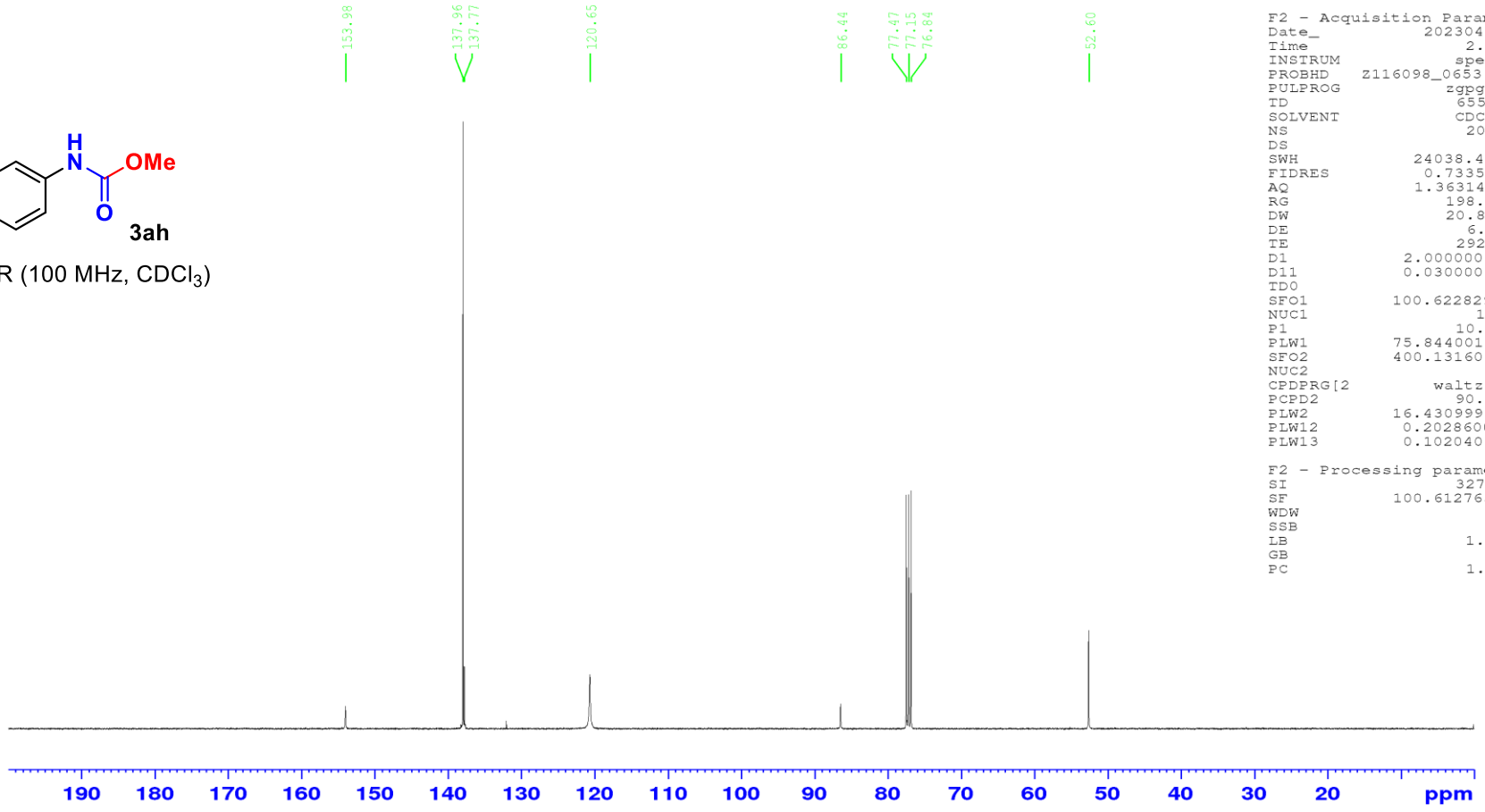

7.583
7.561
7.260
7.173
7.152
6.845
3.755
H
N
OMe
O
I
3ah
^{1}H NMR (400 MHz, $CDCl_3$)
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 ppm
2.00
1.97
1.01
3.07
Current Data Parameters
NAME lhz-20230419-3
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20230420
Time 0.39 h
INSTRUM spect
PROBHD Z116098_0653 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.244532 Hz
AQ 4.0894465 sec
RG 50.3
DW 62.400 usec
DE 6.50 usec
TE 291.8 K
D1 1.00000000 sec
TD0 1
SFO1 400.1324708 MHz
NUC1 1H
P1 10.00 usec
PLW1 16.43099976 W
F2 - Processing parameters
SI 65536
SF 400.1300098 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



3ah

^{13}C NMR (100 MHz, $CDCl_3$)




```
Current Data Parameters
NAME      lhz-20230419-3
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20230420
Time                2.34 h
INSTRUM            spect
PROBHD    Z116098_0653 (
PULPROG         zgpg30
TD                65536
SOLVENT           CDCl3
NS                 2000
DS                    4
SWH           24038.461 Hz
FIDRES         0.733596 Hz
AQ            1.3631488 sec
RG               198.36
DW               20.800 usec
DE                6.50 usec
TE               292.7 K
D1          2.00000000 sec
D11         0.03000000 sec
TD0                   1
SFO1       100.6228298 MHz
NUC1               13C
P1               10.00 usec
PLW1       75.84400177 W
SFO2       400.1316005 MHz
NUC2               1H
CPDPRG[2        waltz16
PCPD2            90.00 usec
PLW2       16.43099976 W
PLW12       0.20286000 W
PLW13       0.10204000 W

F2 - Processing parameters
SI                32768
SF         100.6127638 MHz
WDW                EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
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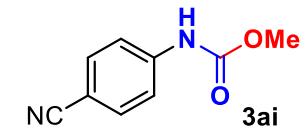
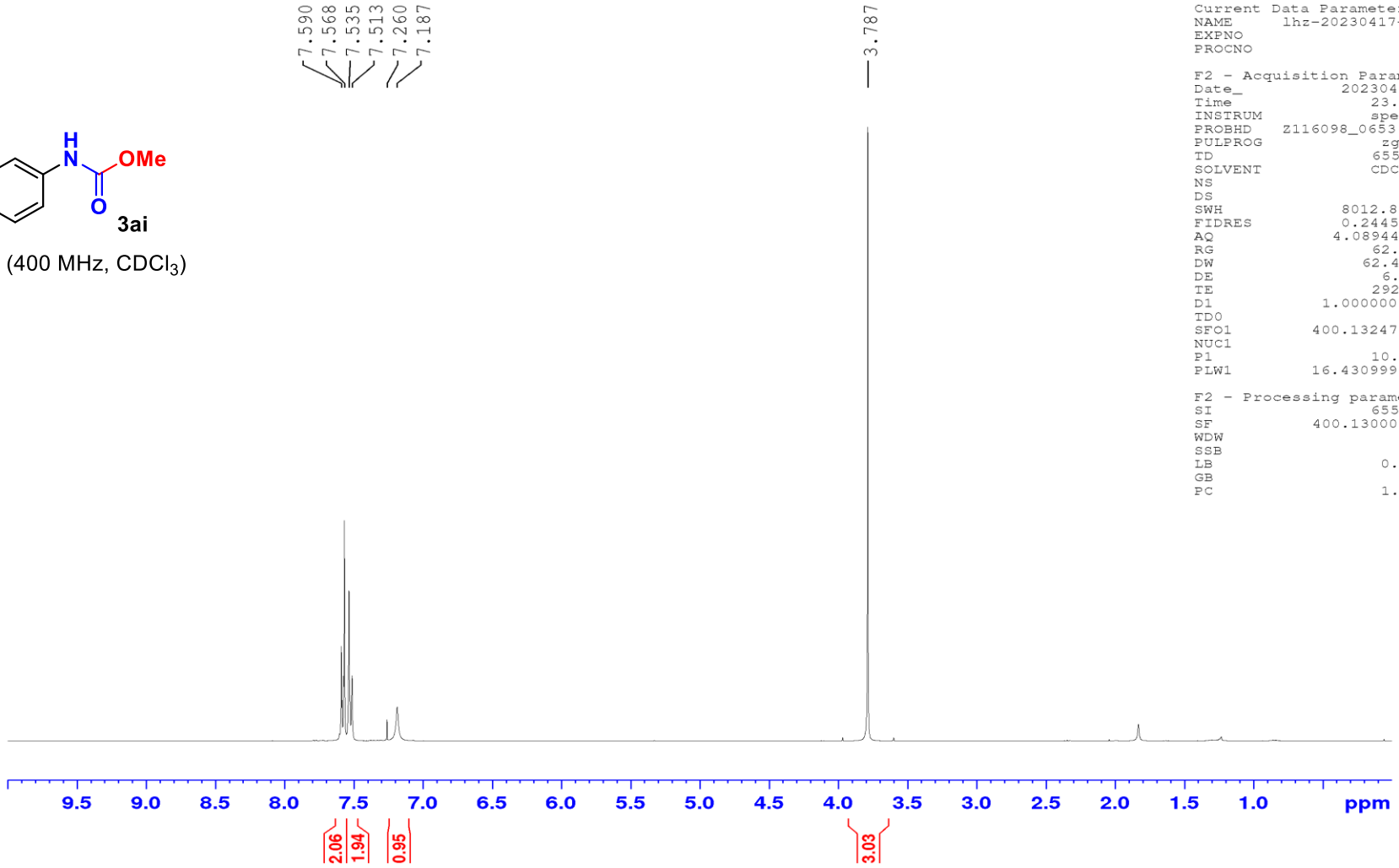


3ai

^{1}H NMR (400 MHz, $CDCl_3$)

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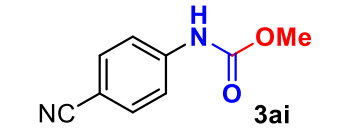
Current Data Parameters
NAME      1hz-20230417-2
EXPNO                  1
PROCNO                 1

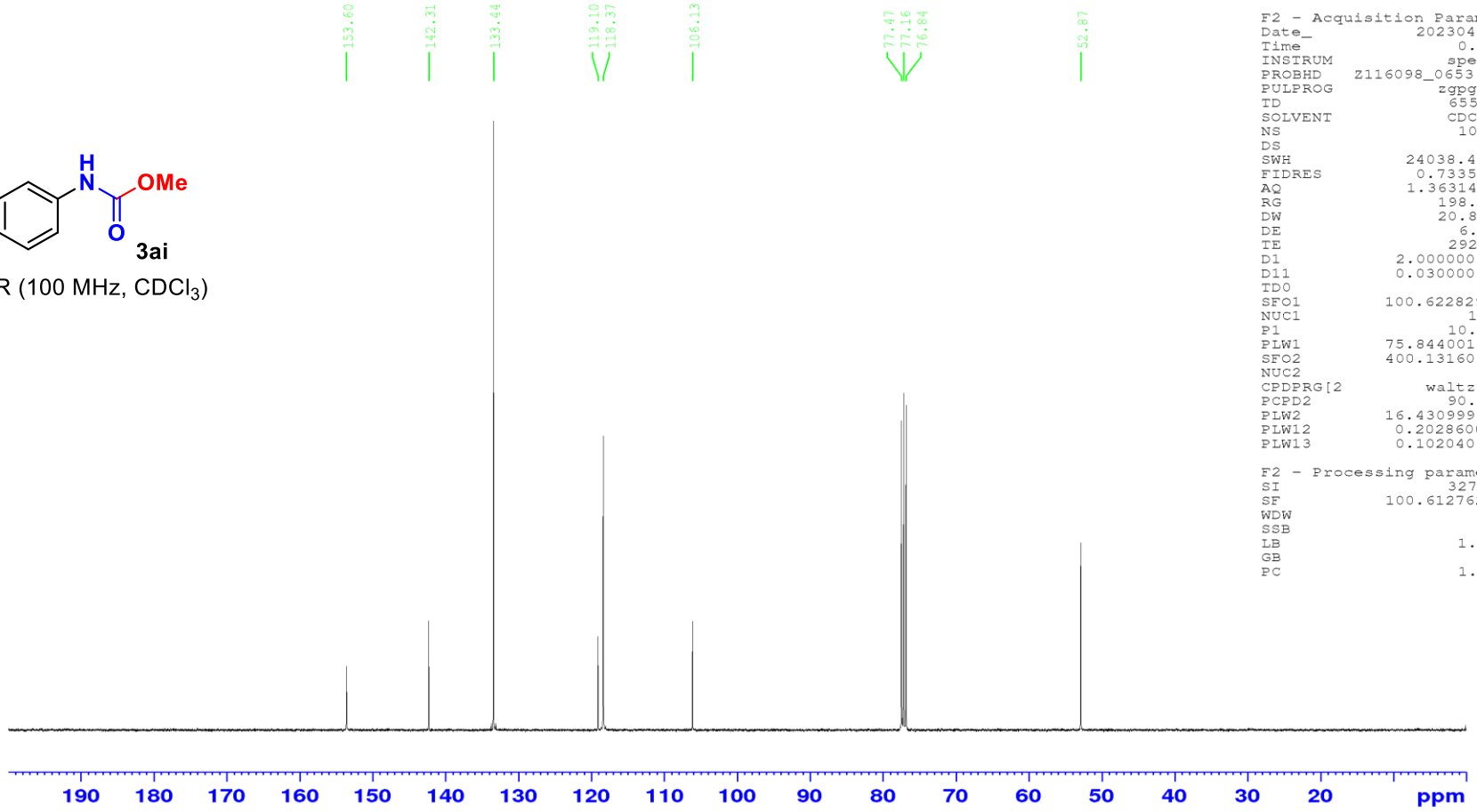
F2 - Acquisition Parameters
Date_           20230417
Time              23.27 h
INSTRUM             spect
PROBHD    Z116098_0653 (
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             8012.820 Hz
FIDRES          0.244532 Hz
AQ              4.0894465 sec
RG                 62.98
DW                62.400 usec
DE                 6.50 usec
TE                292.1 K
D1             1.00000000 sec
TD0                    1
SFO1          400.1324708 MHz
NUC1                  1H
P1                 10.00 usec
PLW1          16.43099976 W

F2 - Processing parameters
SI                 65536
SF            400.1300098 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```



3ai

^{13}C NMR (100 MHz, $CDCl_3$)

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Current Data Parameters
NAME      lhz-20230417-2
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20230418
Time                0.27 h
INSTRUM            spect
PROBHD   Z116098_0653 (
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                 1024
DS                    4
SWH           24038.461 Hz
FIDRES         0.733596 Hz
AQ            1.3631488 sec
RG               198.36
DW               20.800 usec
DE                6.50 usec
TE               292.5 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                   1
SFO1        100.6228298 MHz
NUC1               13C
P1                10.00 usec
PLW1         75.84400177 W
SFO2        400.1316005 MHz
NUC2               1H
CPDPRG[2       waltz16
PCPD2             90.00 usec
PLW2         16.43099976 W
PLW12         0.20286000 W
PLW13         0.10204000 W

F2 - Processing parameters
SI                32768
SF          100.6127623 MHz
WDW                EM
SSB                0
LB              1.00 Hz
GB                 0
PC              1.40
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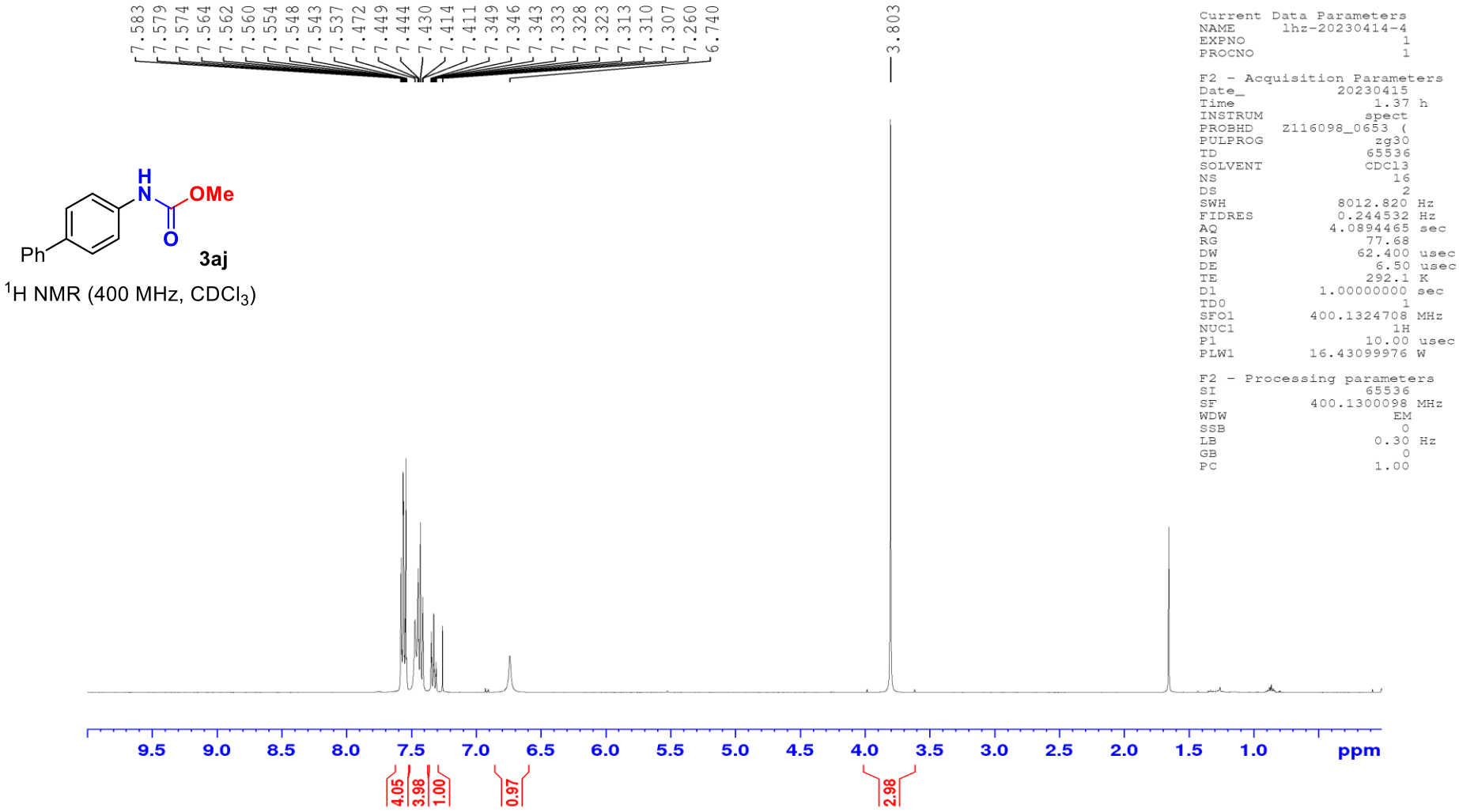
7.583
7.579
7.574
7.564
7.562
7.560
7.554
7.548
7.543
7.537
7.472
7.449
7.444
7.430
7.414
7.411
7.349
7.346
7.343
7.333
7.328
7.323
7.313
7.310
7.307
7.260
6.740
3.803
H
N
OMe
O
Ph
3aj
1H NMR (400 MHz, CDCl3)
Current Data Parameters
NAME lhz-20230414-4
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20230415
Time 1.37 h
INSTRUM spect
PROBHD Z116098_0653 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.244532 Hz
AQ 4.0894465 sec
RG 77.68
DW 62.400 usec
DE 6.50 usec
TE 292.1 K
D1 1.00000000 sec
TD0 1
SFO1 400.1324708 MHz
NUC1 1H
P1 10.00 usec
PLW1 16.43099976 W
F2 - Processing parameters
SI 65536
SF 400.1300098 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
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2.5
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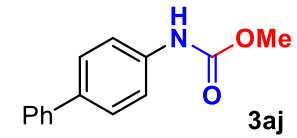
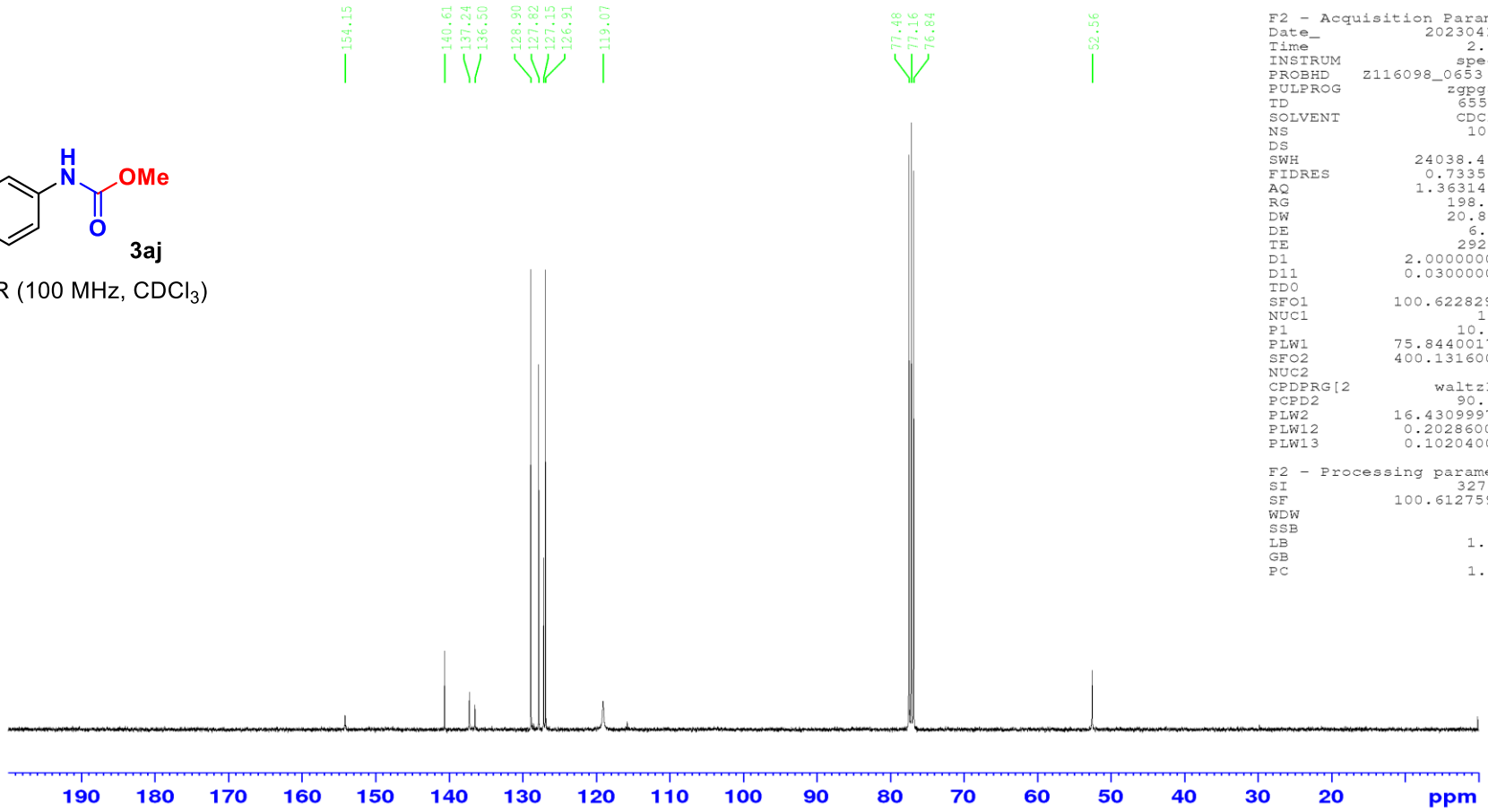
ppm
4.05
3.98
1.00
0.97
2.98



3aj

^{13}C NMR (100 MHz, $CDCl_3$)




```
Current Data Parameters
NAME      1hz-20230414-4
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20230415
Time              2.37 h
INSTRUM           spect
PROBHD    Z116098_0653 (
PULPROG         zgpg30
TD               65536
SOLVENT          CDCl3
NS                1024
DS                   4
SWH          24038.461 Hz
FIDRES        0.733596 Hz
AQ            1.3631488 sec
RG               198.36
DW               20.800 usec
DE                6.50 usec
TE               292.4 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                  1
SFO1       100.6228298 MHz
NUC1              13C
P1               10.00 usec
PLW1       75.84400177 W
SFO2       400.1316005 MHz
NUC2              1H
CPDPRG[2       waltz16
PCPD2            90.00 usec
PLW2       16.43099976 W
PLW12       0.20286000 W
PLW13       0.10204000 W

F2 - Processing parameters
SI               32768
SF         100.6127594 MHz
WDW                EM
SSB                 0
LB              1.00 Hz
GB                  0
PC              1.40
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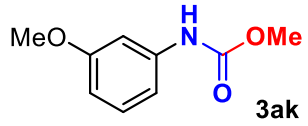
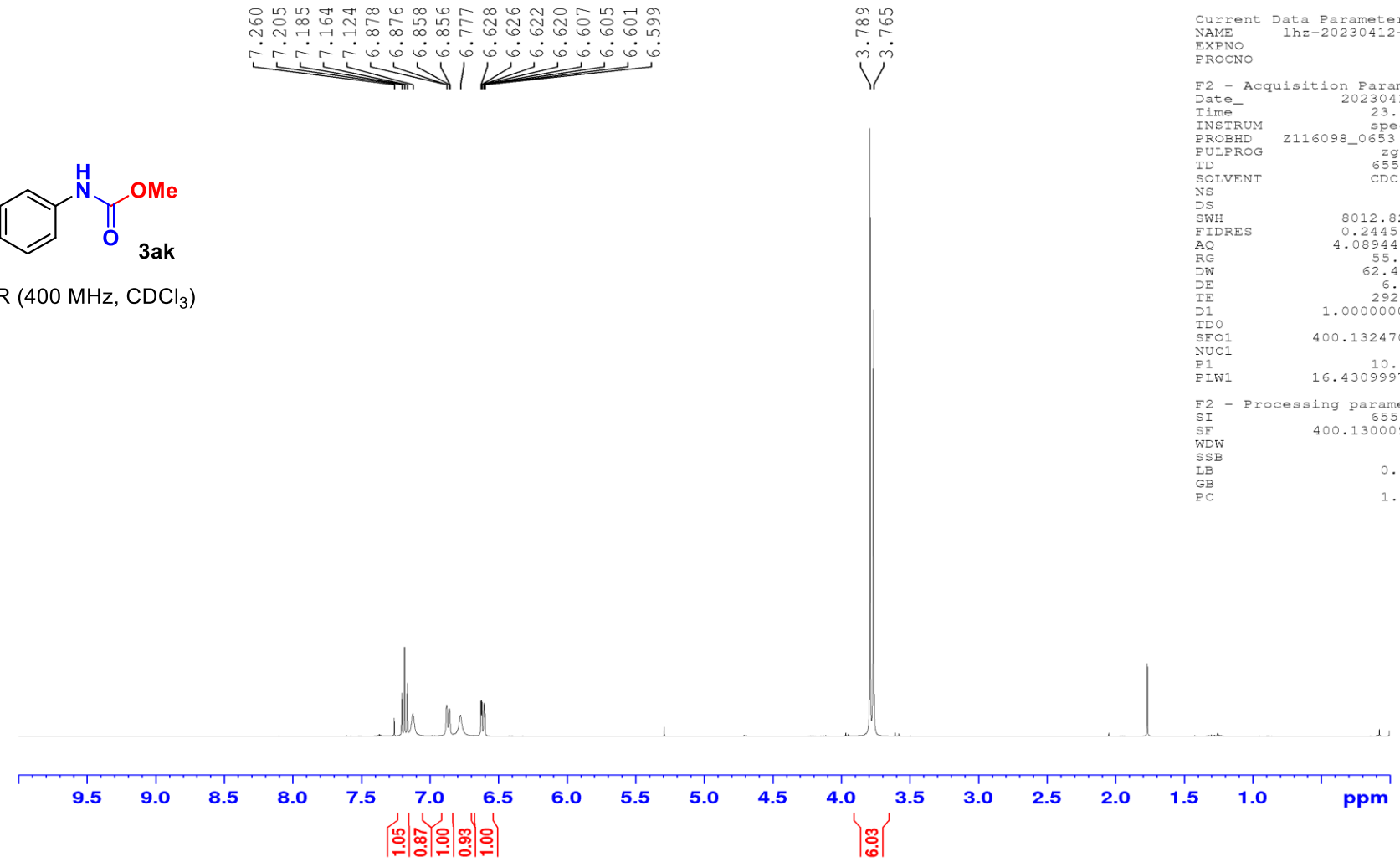
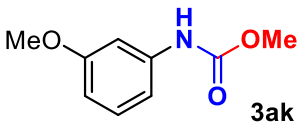


3ak

^{1}H NMR (400 MHz, $CDCl_3$)

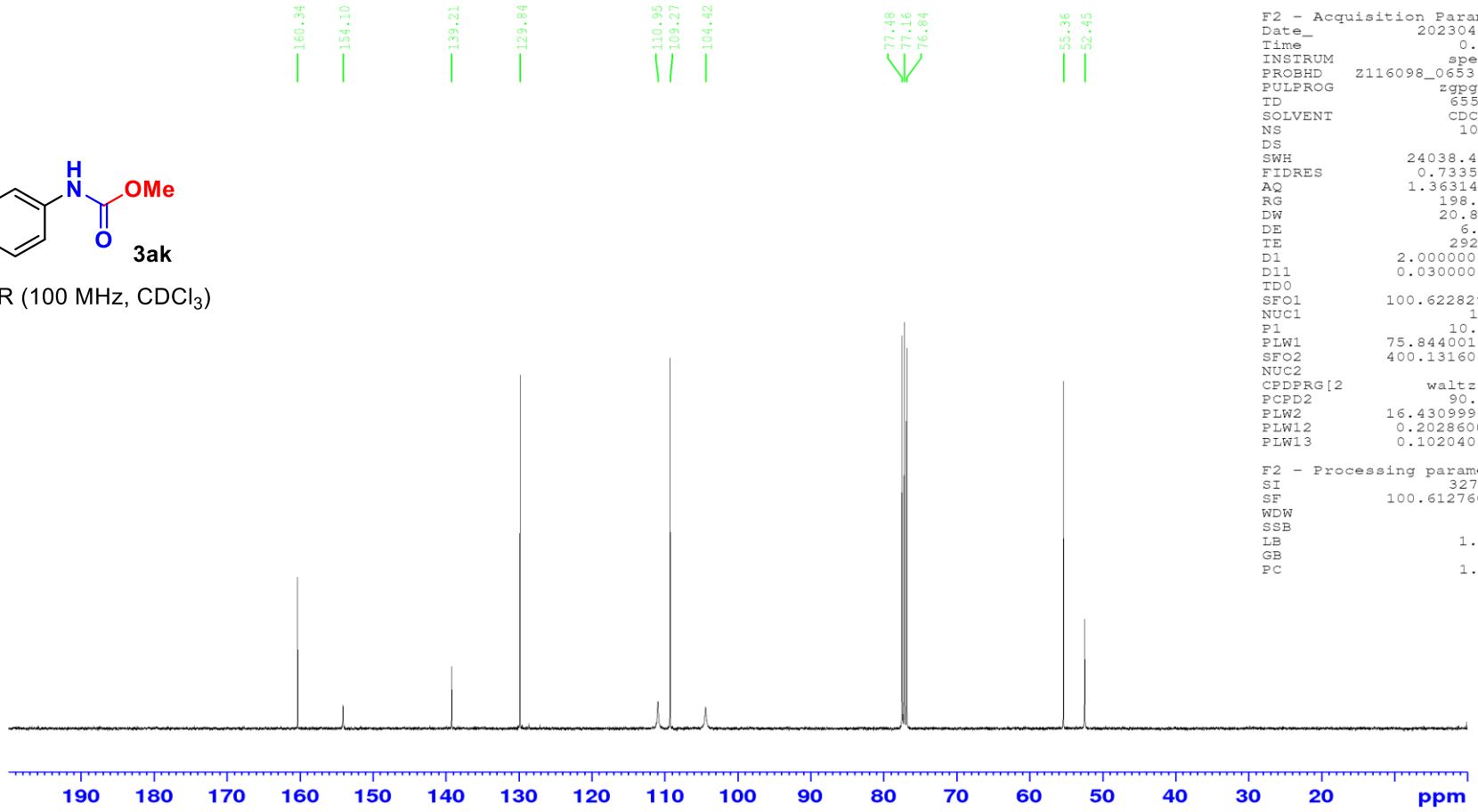



```
Current Data Parameters
NAME      1hz-20230412-2
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20230412
Time              23.34 h
INSTRUM             spect
PROBHD    Z116098_0653 (
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             8012.820 Hz
FIDRES          0.244532 Hz
AQ              4.0894465 sec
RG                 55.99
DW                62.400 usec
DE                 6.50 usec
TE                292.3 K
D1             1.00000000 sec
TD0                    1
SFO1          400.1324708 MHz
NUC1                  1H
P1                 10.00 usec
PLW1          16.43099976 W

F2 - Processing parameters
SI                 65536
SF            400.1300098 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```



3ak

^{13}C NMR (100 MHz, $CDCl_3$)




```
Current Data Parameters
NAME      1hz-20230412-2
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20230413
Time              0.34 h
INSTRUM           spect
PROBHD   Z116098_0653 (
PULPROG         zgpg30
TD               65536
SOLVENT          CDCl3
NS                1024
DS                   4
SWH          24038.461 Hz
FIDRES        0.733596 Hz
AQ            1.3631488 sec
RG              198.36
DW              20.800 usec
DE               6.50 usec
TE              292.6 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                  1
SFO1       100.6228298 MHz
NUC1              13C
P1              10.00 usec
PLW1       75.84400177 W
SFO2       400.1316005 MHz
NUC2              1H
CPDPRG[2       waltz16
PCPD2             90.00 usec
PLW2       16.43099976 W
PLW12       0.20286000 W
PLW13       0.10204000 W

F2 - Processing parameters
SI               32768
SF         100.6127608 MHz
WDW                EM
SSB                 0
LB               1.00 Hz
GB                  0
PC               1.40
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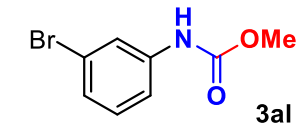
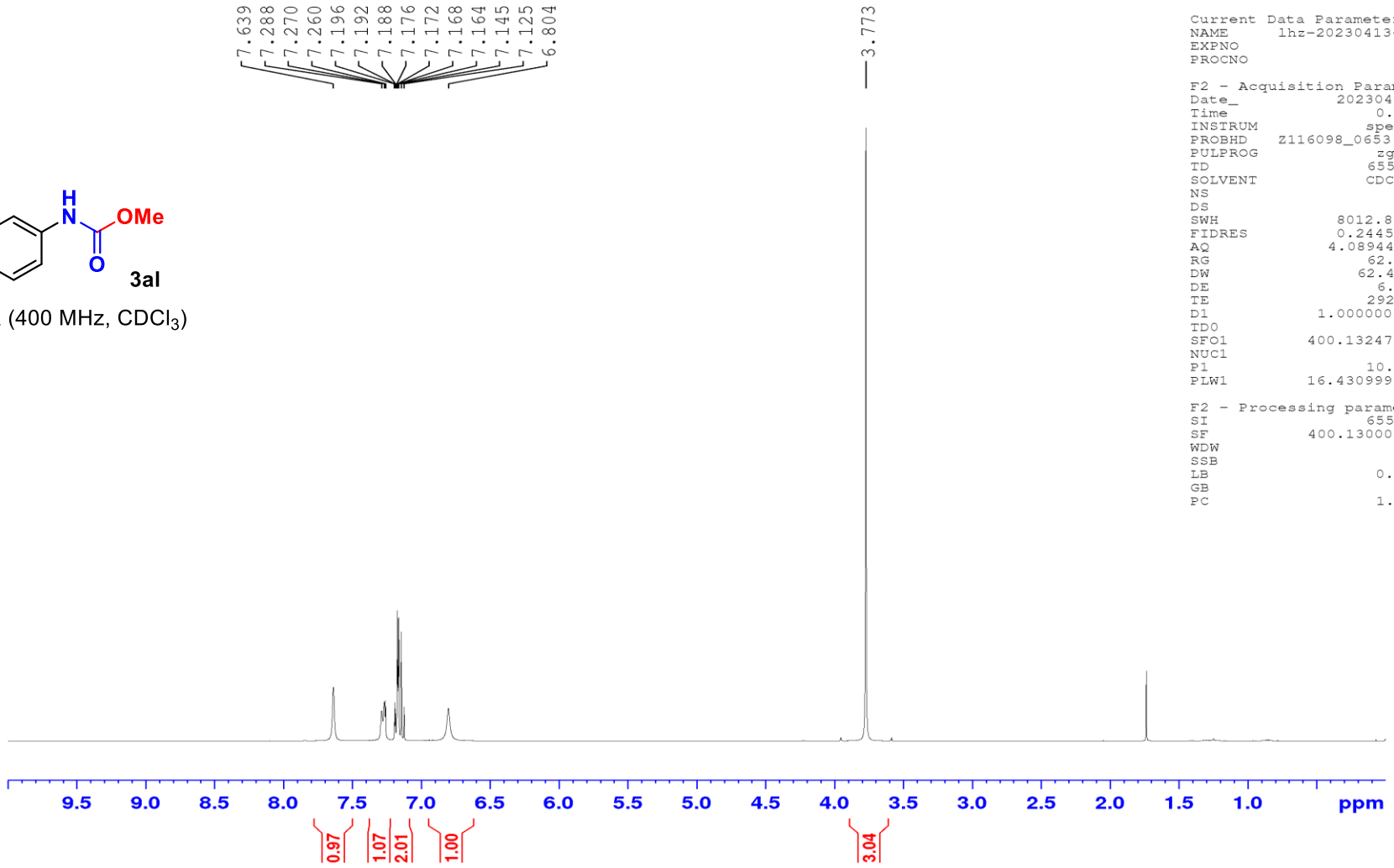


3al

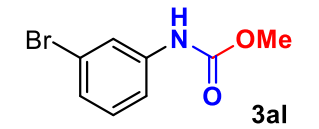
^{1}H NMR (400 MHz, $CDCl_3$)




```
Current Data Parameters
NAME      1hz-20230413-3
EXPNO                  1
PROCNO                 1

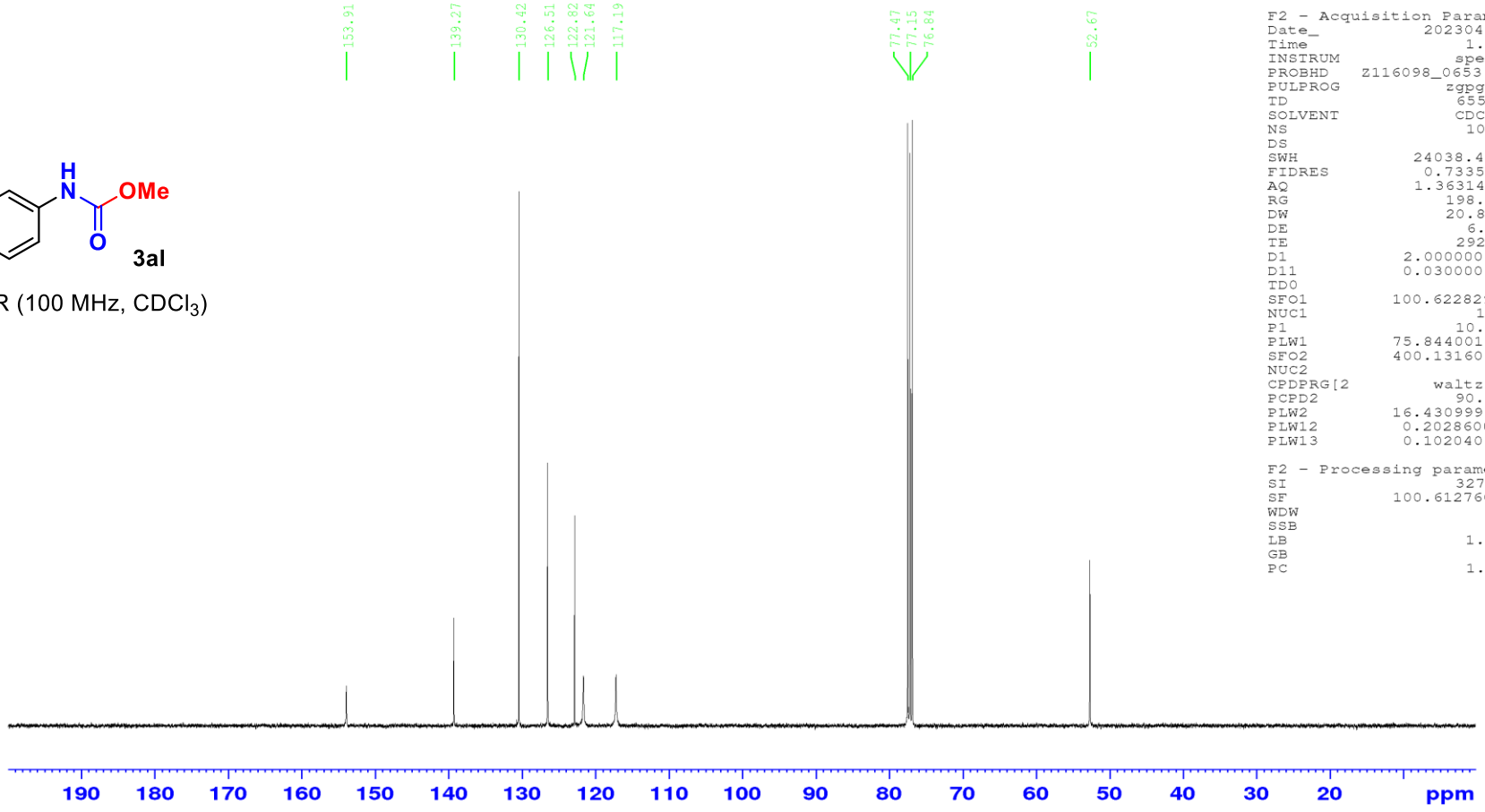
F2 - Acquisition Parameters
Date_           20230414
Time              0.27 h
INSTRUM           spect
PROBHD    Z116098_0653 (
PULPROG           zg30
TD               65536
SOLVENT          CDCl3
NS                  16
DS                   2
SWH           8012.820 Hz
FIDRES        0.244532 Hz
AQ          4.0894465 sec
RG               62.98
DW             62.400 usec
DE               6.50 usec
TE              292.1 K
D1          1.00000000 sec
TD0                  1
SFO1       400.1324708 MHz
NUC1               1H
P1              10.00 usec
PLW1       16.43099976 W

F2 - Processing parameters
SI               65536
SF         400.1300098 MHz
WDW                EM
SSB                 0
LB               0.30 Hz
GB                  0
PC               1.00
```



3al

^{13}C NMR (100 MHz, $CDCl_3$)




```
Current Data Parameters
NAME      1hz-20230413-3
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20230414
Time                1.27 h
INSTRUM            spect
PROBHD   Z116098_0653 (
PULPROG         zgpg30
TD               65536
SOLVENT          CDCl3
NS                1024
DS                   4
SWH          24038.461 Hz
FIDRES        0.733596 Hz
AQ            1.3631488 sec
RG               198.36
DW               20.800 usec
DE                6.50 usec
TE               292.9 K
D1          2.00000000 sec
D11         0.03000000 sec
TD0                  1
SFO1       100.6228298 MHz
NUC1               13C
P1               10.00 usec
PLW1       75.84400177 W
SFO2       400.1316005 MHz
NUC2               1H
CPDPRG[2       waltz16
PCPD2            90.00 usec
PLW2       16.43099976 W
PLW12       0.20286000 W
PLW13       0.10204000 W

F2 - Processing parameters
SI               32768
SF         100.6127606 MHz
WDW                EM
SSB                 0
LB               1.00 Hz
GB                  0
PC               1.40
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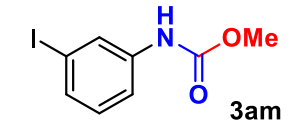
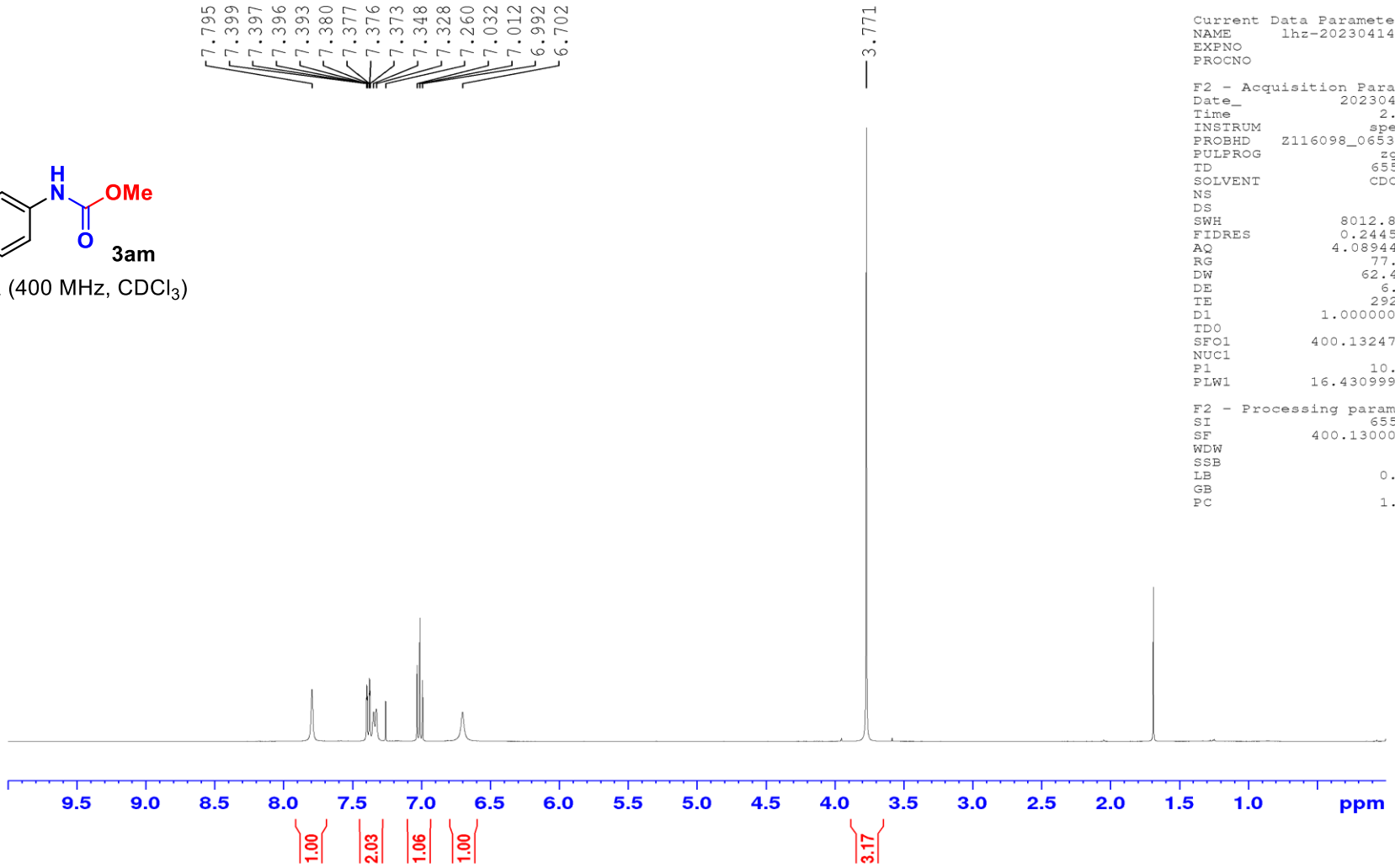


3am

^{1}H NMR (400 MHz, $CDCl_3$)

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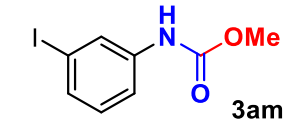
Current Data Parameters
NAME      1hz-20230414-5
EXPNO                  1
PROCNO                 1

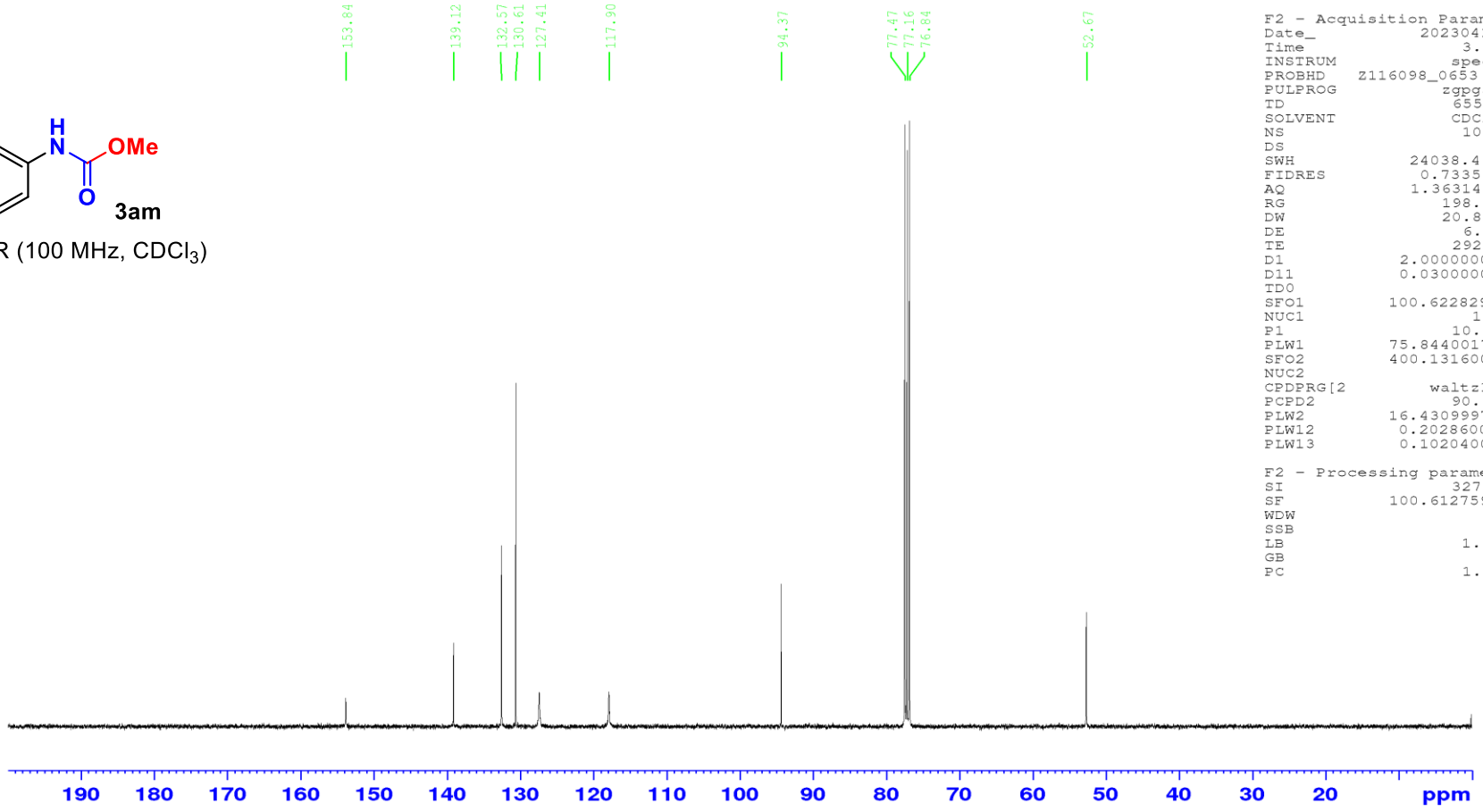
F2 - Acquisition Parameters
Date_           20230415
Time              2.41 h
INSTRUM           spect
PROBHD    Z116098_0653 (
PULPROG           zg30
TD               65536
SOLVENT          CDCl3
NS                  16
DS                   2
SWH           8012.820 Hz
FIDRES           0.244532 Hz
AQ            4.0894465 sec
RG                77.68
DW               62.400 usec
DE                6.50 usec
TE               292.1 K
D1            1.00000000 sec
TD0                  1
SFO1        400.1324708 MHz
NUC1               1H
P1               10.00 usec
PLW1         16.43099976 W

F2 - Processing parameters
SI               65536
SF          400.1300099 MHz
WDW                EM
SSB                 0
LB               0.30 Hz
GB                  0
PC               1.00
```



3am

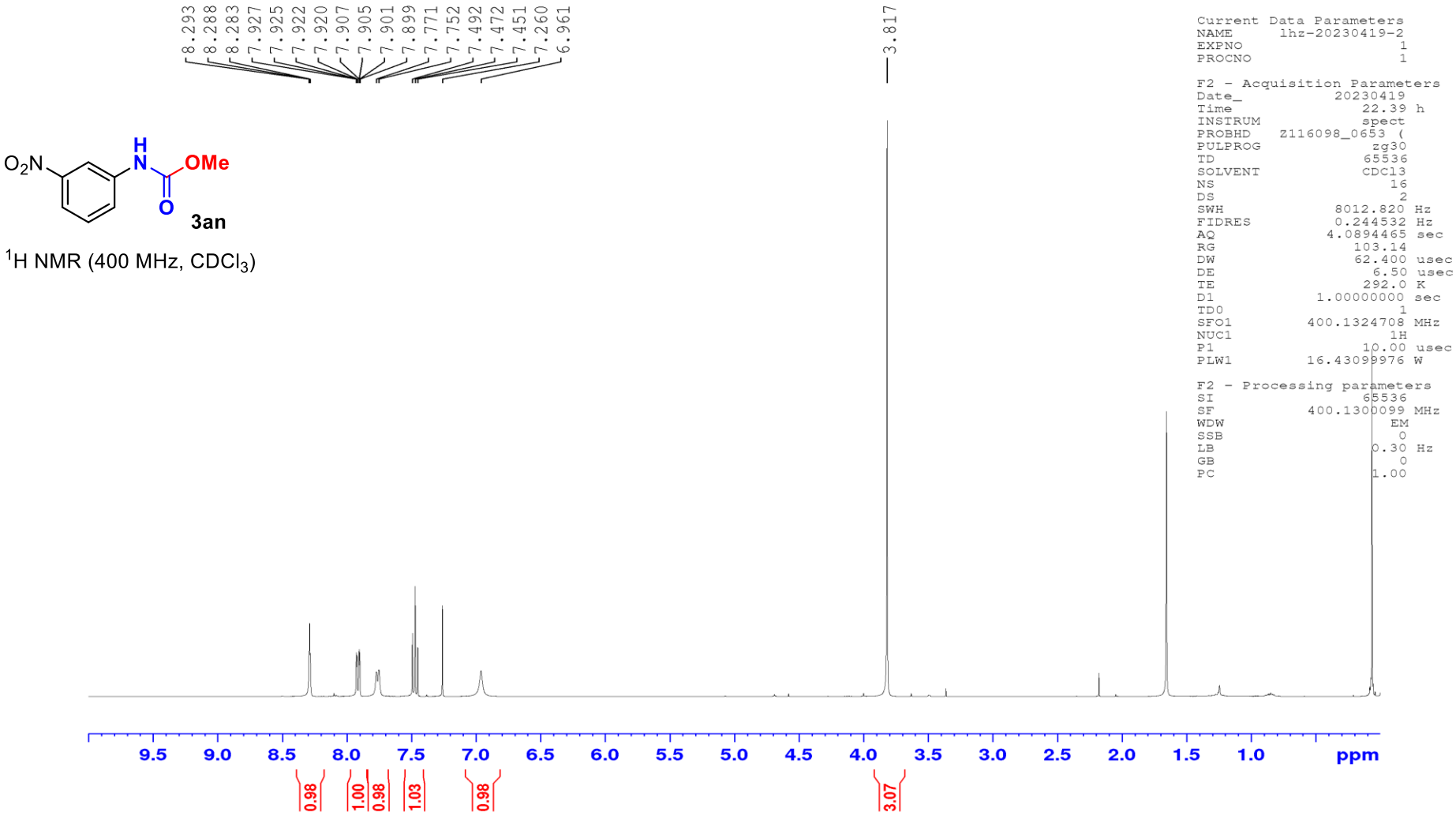
^{13}C NMR (100 MHz, $CDCl_3$)




```
Current Data Parameters
NAME      1hz-20230414-5
EXPNO                  2
PROCNO                 1

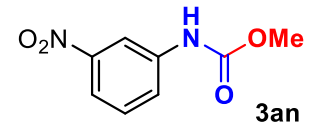
F2 - Acquisition Parameters
Date_           20230415
Time               3.41 h
INSTRUM           spect
PROBHD    Z116098_0653 (
PULPROG         zgpg30
TD               65536
SOLVENT          CDCl3
NS                1024
DS                   4
SWH          24038.461 Hz
FIDRES        0.733596 Hz
AQ            1.3631488 sec
RG               198.36
DW               20.800 usec
DE                6.50 usec
TE               292.4 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                  1
SFO1       100.6228298 MHz
NUC1              13C
P1               10.00 usec
PLW1       75.84400177 W
SFO2       400.1316005 MHz
NUC2              1H
CPDPRG[2       waltz16
PCPD2            90.00 usec
PLW2       16.43099976 W
PLW12       0.20286000 W
PLW13       0.10204000 W

F2 - Processing parameters
SI               32768
SF         100.6127599 MHz
WDW                EM
SSB                 0
LB              1.00 Hz
GB                  0
PC              1.40
```



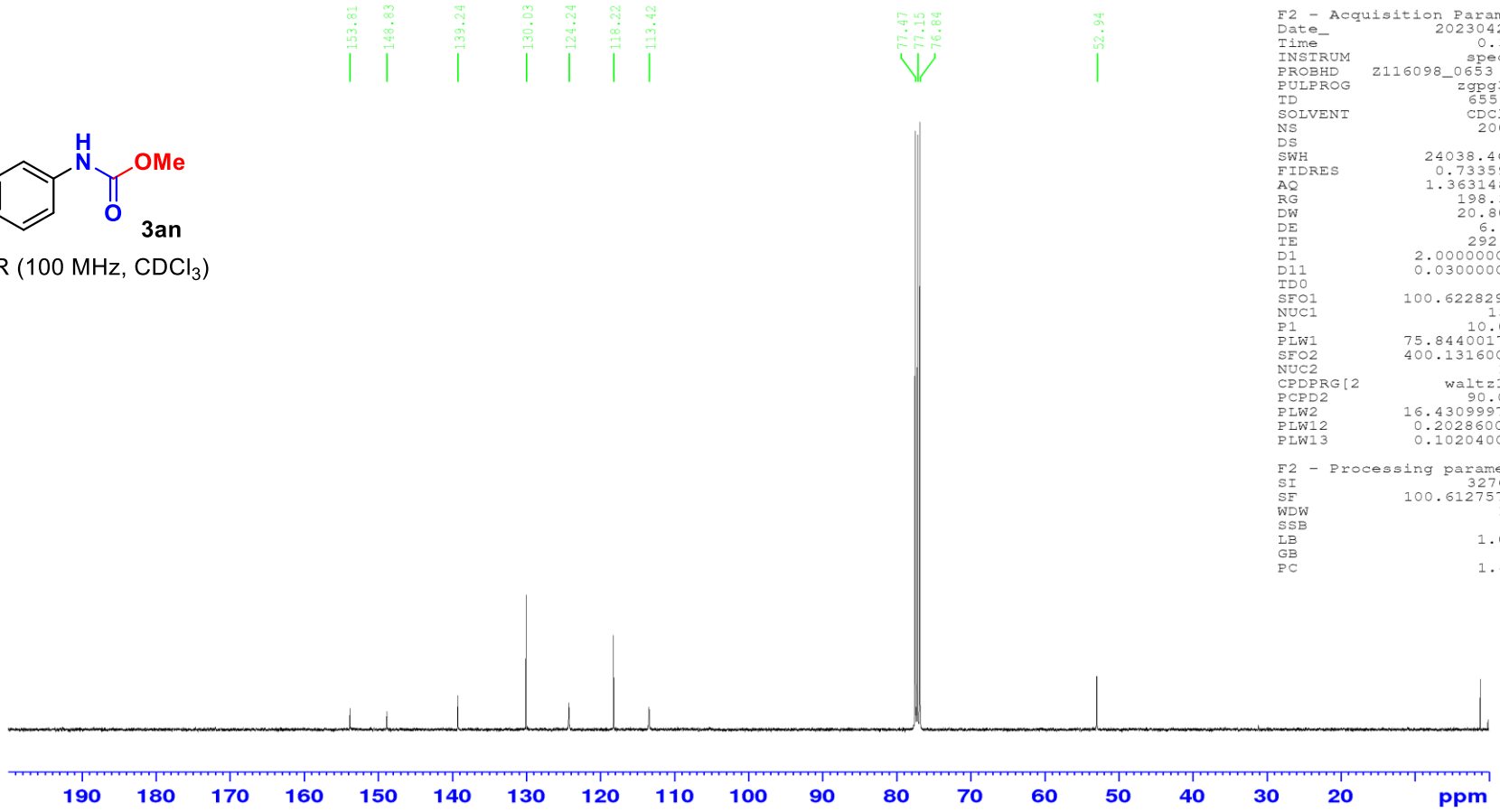
3an

^{1}H NMR (400 MHz, $CDCl_3$)



3an

^{13}C NMR (100 MHz, $CDCl_3$)




```
Current Data Parameters
NAME      lhz-20230419-2
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20230420
Time                0.35 h
INSTRUM            spect
PROBHD    Z116098_0653 (
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                 2000
DS                    4
SWH           24038.461 Hz
FIDRES         0.733596 Hz
AQ            1.3631488 sec
RG               198.36
DW               20.800 usec
DE                6.50 usec
TE               292.6 K
D1          2.00000000 sec
D11         0.03000000 sec
TD0                   1
SFO1        100.6228298 MHz
NUC1                13C
P1                10.00 usec
PLW1        75.84400177 W
SFO2        400.1316005 MHz
NUC2                1H
CPDPRG[2        waltz16
PCPD2             90.00 usec
PLW2        16.43099976 W
PLW12        0.20286000 W
PLW13        0.10204000 W

F2 - Processing parameters
SI                32768
SF          100.6127579 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
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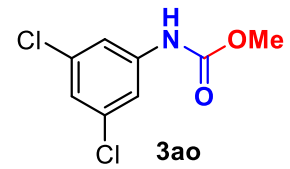
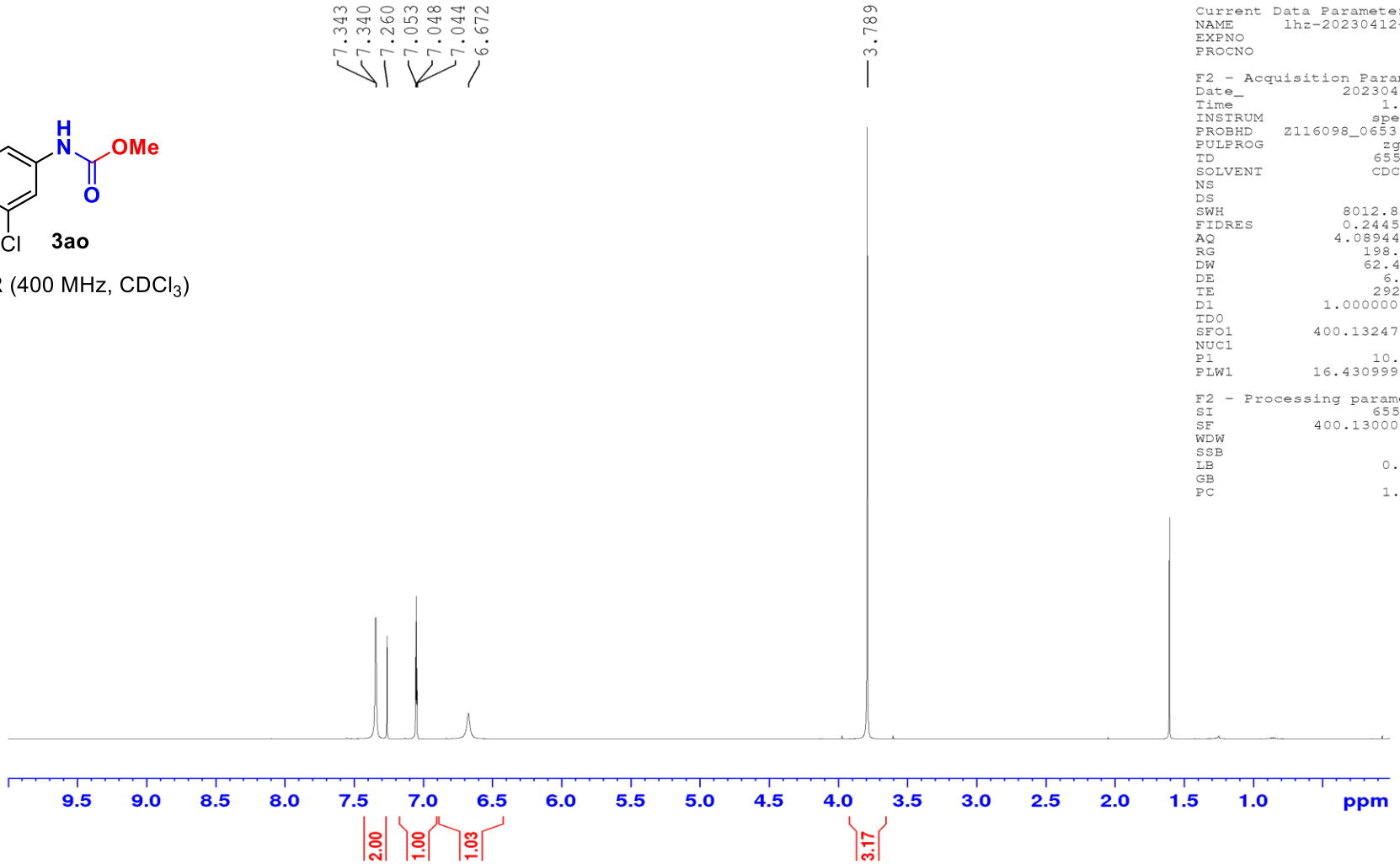
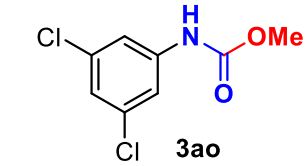


3ao

^{1}H NMR (400 MHz, $CDCl_3$)

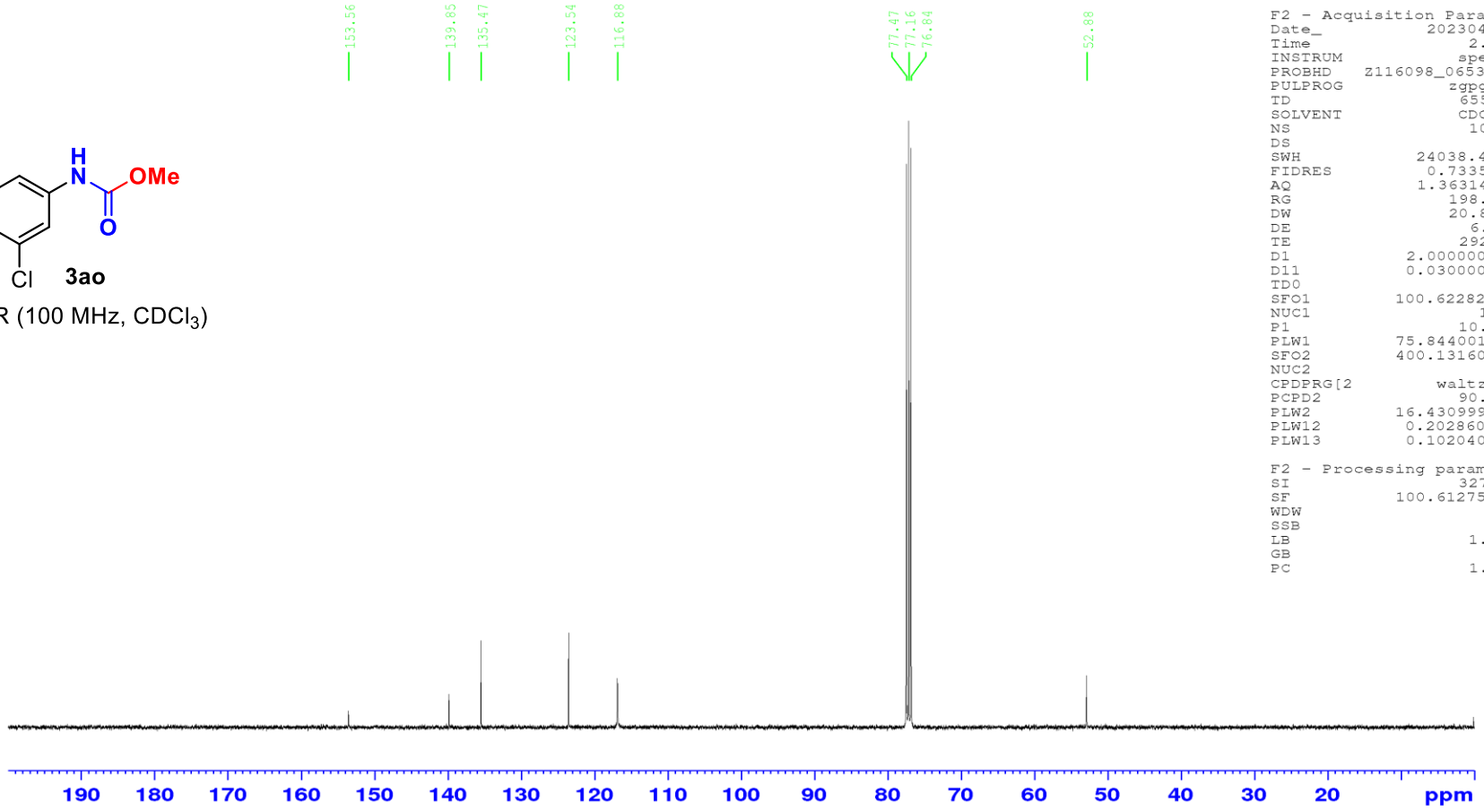



```
Current Data Parameters
NAME      1hz-20230412-4
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20230413
Time              1.43 h
INSTRUM           spect
PROBHD   Z116098_0653 (
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                   16
DS                    2
SWH            8012.820 Hz
FIDRES         0.244532 Hz
AQ             4.0894465 sec
RG                198.36
DW                62.400 usec
DE                 6.50 usec
TE                292.0 K
D1             1.00000000 sec
TD0                   1
SFO1       400.1324708 MHz
NUC1                  1H
P1                10.00 usec
PLW1       16.43099976 W

F2 - Processing parameters
SI                65536
SF         400.1300099 MHz
WDW                   EM
SSB                   0
LB                 0.30 Hz
GB                    0
PC                 1.00
```

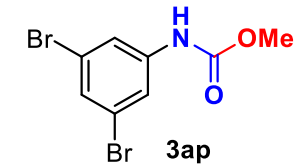


3ao

^{13}C NMR (100 MHz, $CDCl_3$)

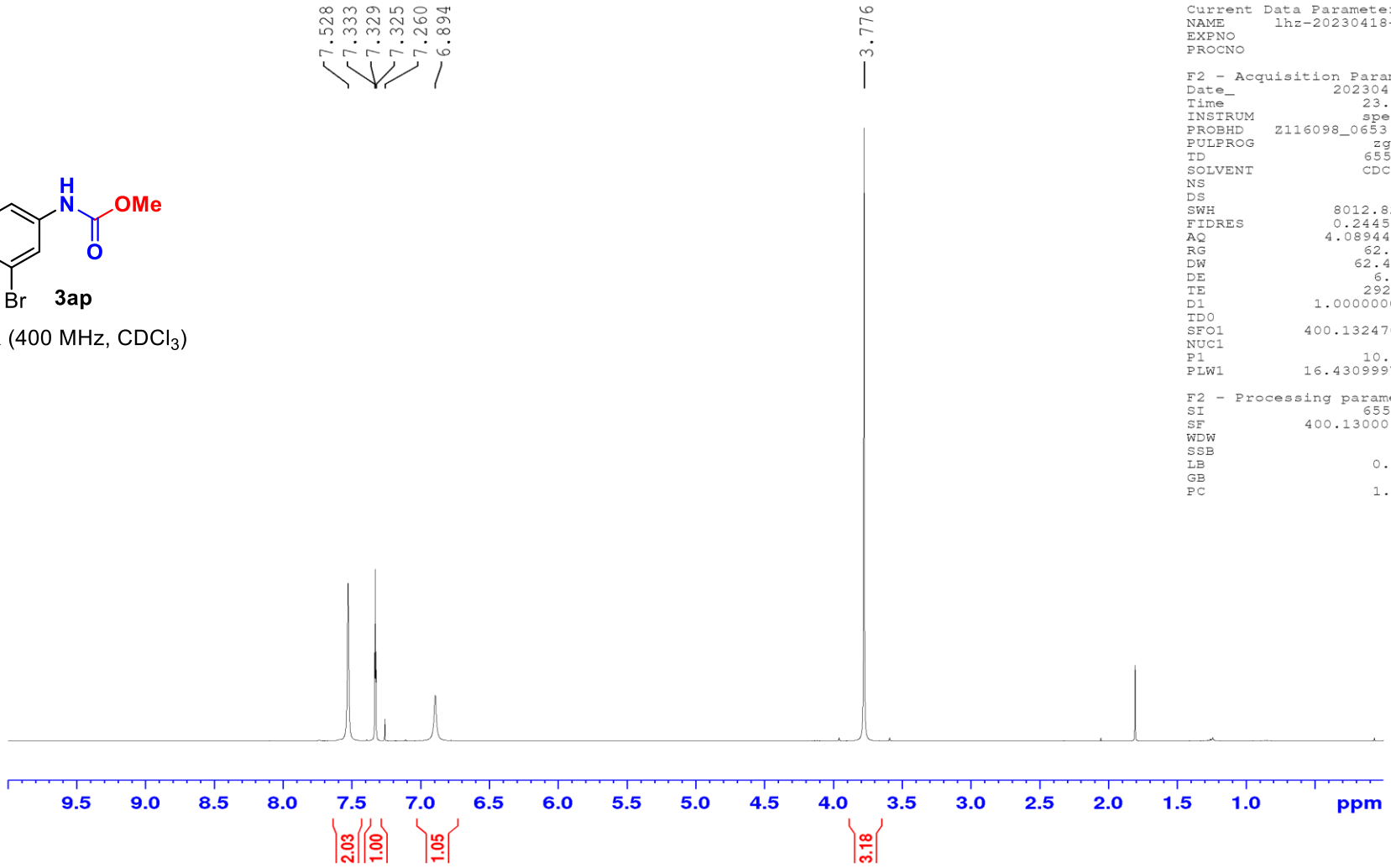



```
Current Data Parameters
NAME      lhz-20230412-4
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20230413
Time                2.42 h
INSTRUM            spect
PROBHD    Z116098_0653 (
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                 1024
DS                    4
SWH           24038.461 Hz
FIDRES         0.733596 Hz
AQ             1.3631488 sec
RG                198.36
DW                20.800 usec
DE                 6.50 usec
TE                292.8 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1
SFO1        100.6228298 MHz
NUC1               13C
P1               10.00 usec
PLW1        75.84400177 W
SFO2        400.1316005 MHz
NUC2               1H
CPDPRG[2       waltz16
PCPD2            90.00 usec
PLW2        16.43099976 W
PLW12        0.20286000 W
PLW13        0.10204000 W

F2 - Processing parameters
SI                32768
SF          100.6127571 MHz
WDW                EM
SSB                 0
LB               1.00 Hz
GB                  0
PC               1.40
```

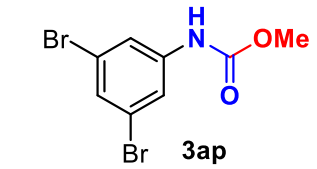


3ap

^{1}H NMR (400 MHz, $CDCl_3$)

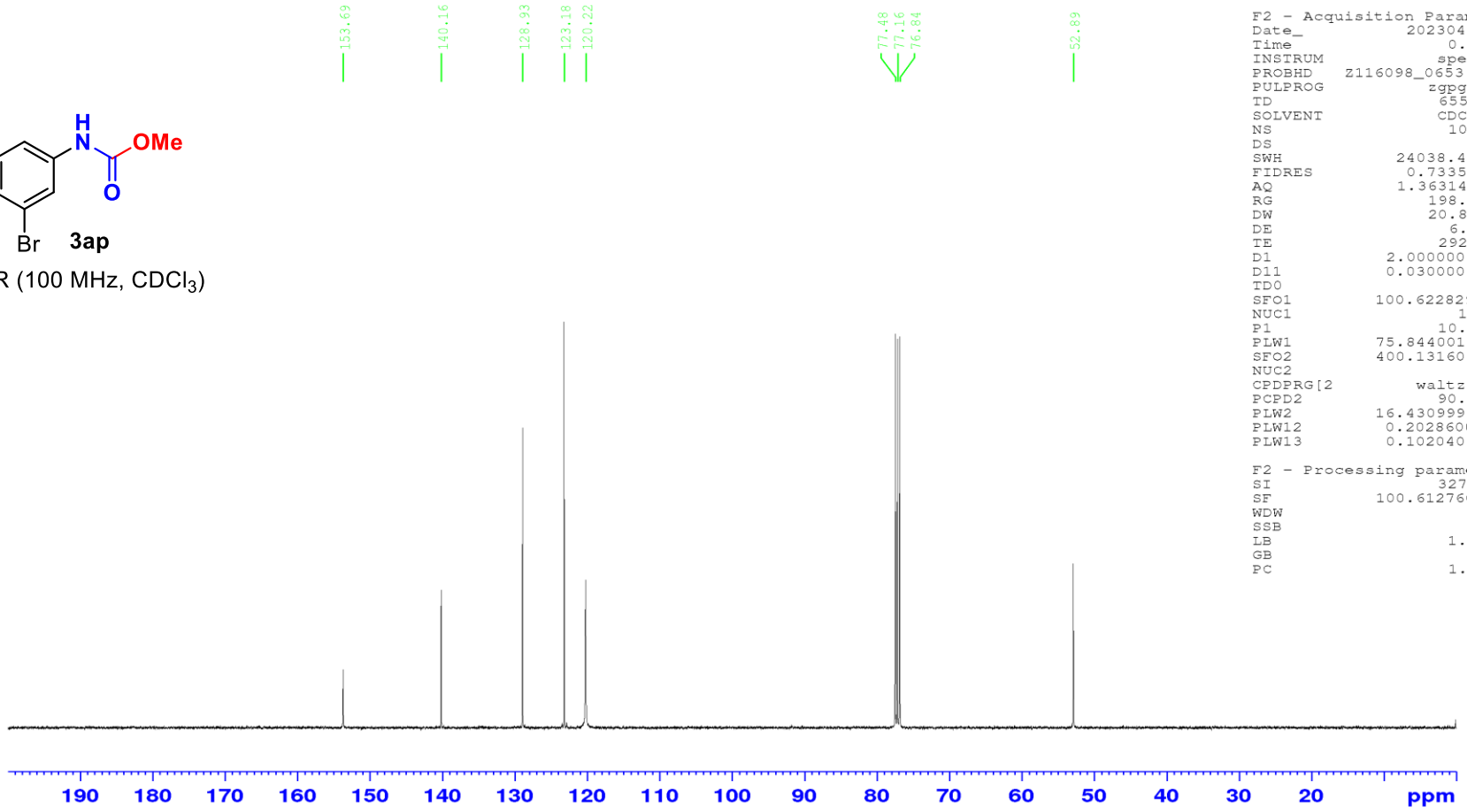



```
Current Data Parameters
NAME      lhz-20230418-2
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20230418
Time              23.34 h
INSTRUM             spect
PROBHD   Z116098_0653 (
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             8012.820 Hz
FIDRES          0.244532 Hz
AQ              4.0894465 sec
RG                 62.98
DW                62.400 usec
DE                 6.50 usec
TE                292.3 K
D1             1.00000000 sec
TD0                    1
SFO1         400.1324708 MHz
NUC1                 1H
P1                10.00 usec
PLW1         16.43099976 W

F2 - Processing parameters
SI                 65536
SF           400.1300098 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```

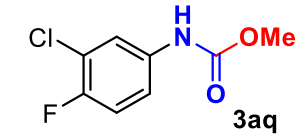


3ap

^{13}C NMR (100 MHz, $CDCl_3$)

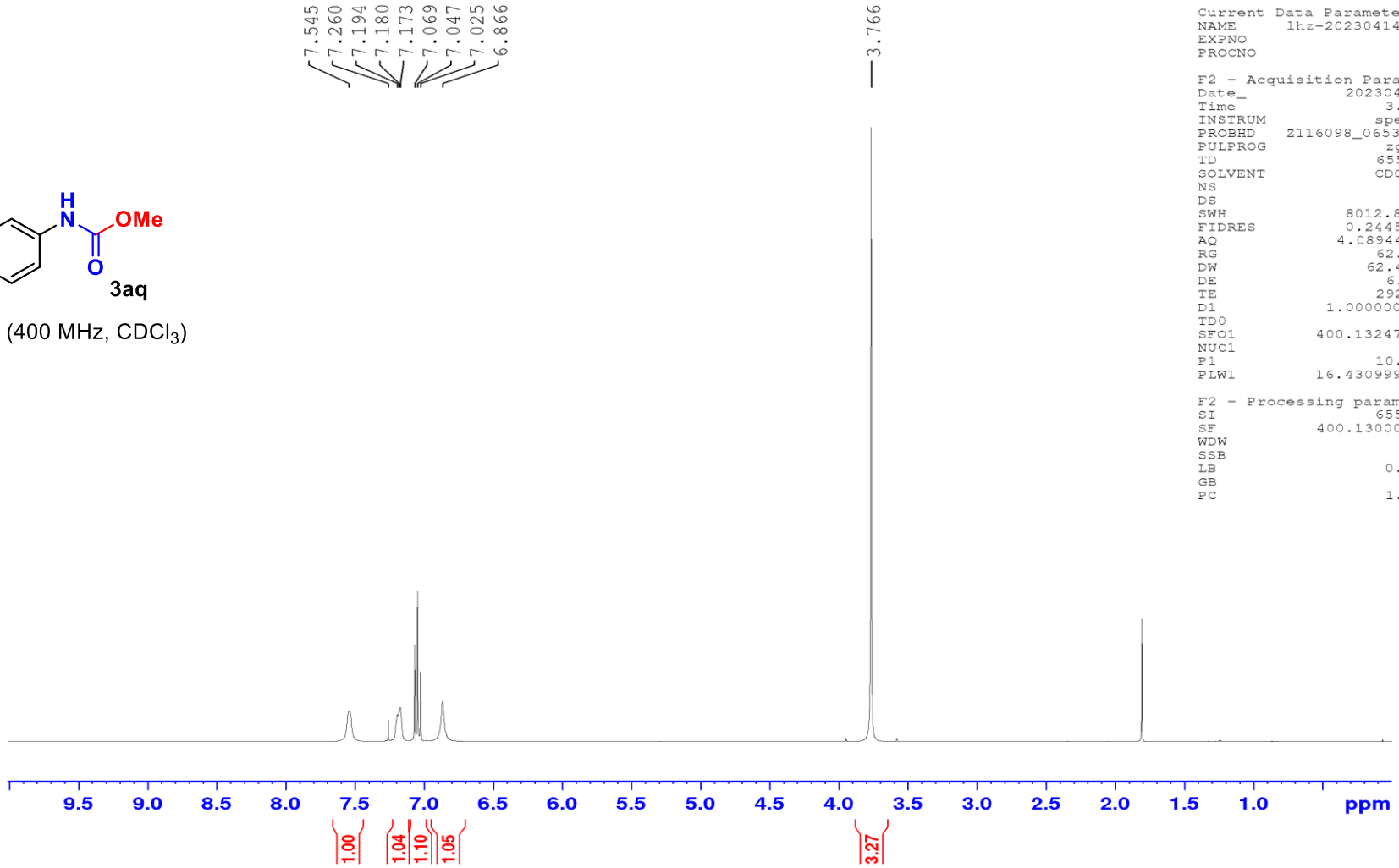



```
Current Data Parameters
NAME      lhz-20230418-2
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20230419
Time                0.34 h
INSTRUM            spect
PROBHD   Z116098_0653 (
PULPROG           zgpg30
TD                65536
SOLVENT           CDCl3
NS                 1024
DS                    4
SWH           24038.461 Hz
FIDRES         0.733596 Hz
AQ             1.3631488 sec
RG                198.36
DW                20.800 usec
DE                 6.50 usec
TE                292.6 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                   1
SFO1         100.6228298 MHz
NUC1                13C
P1                10.00 usec
PLW1         75.84400177 W
SFO2         400.1316005 MHz
NUC2                1H
CPDPRG[2        waltz16
PCPD2             90.00 usec
PLW2         16.43099976 W
PLW12         0.20286000 W
PLW13         0.10204000 W

F2 - Processing parameters
SI                32768
SF           100.6127609 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```

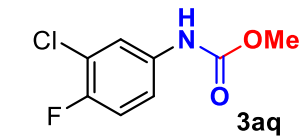


3aq

^{1}H NMR (400 MHz, $CDCl_3$)

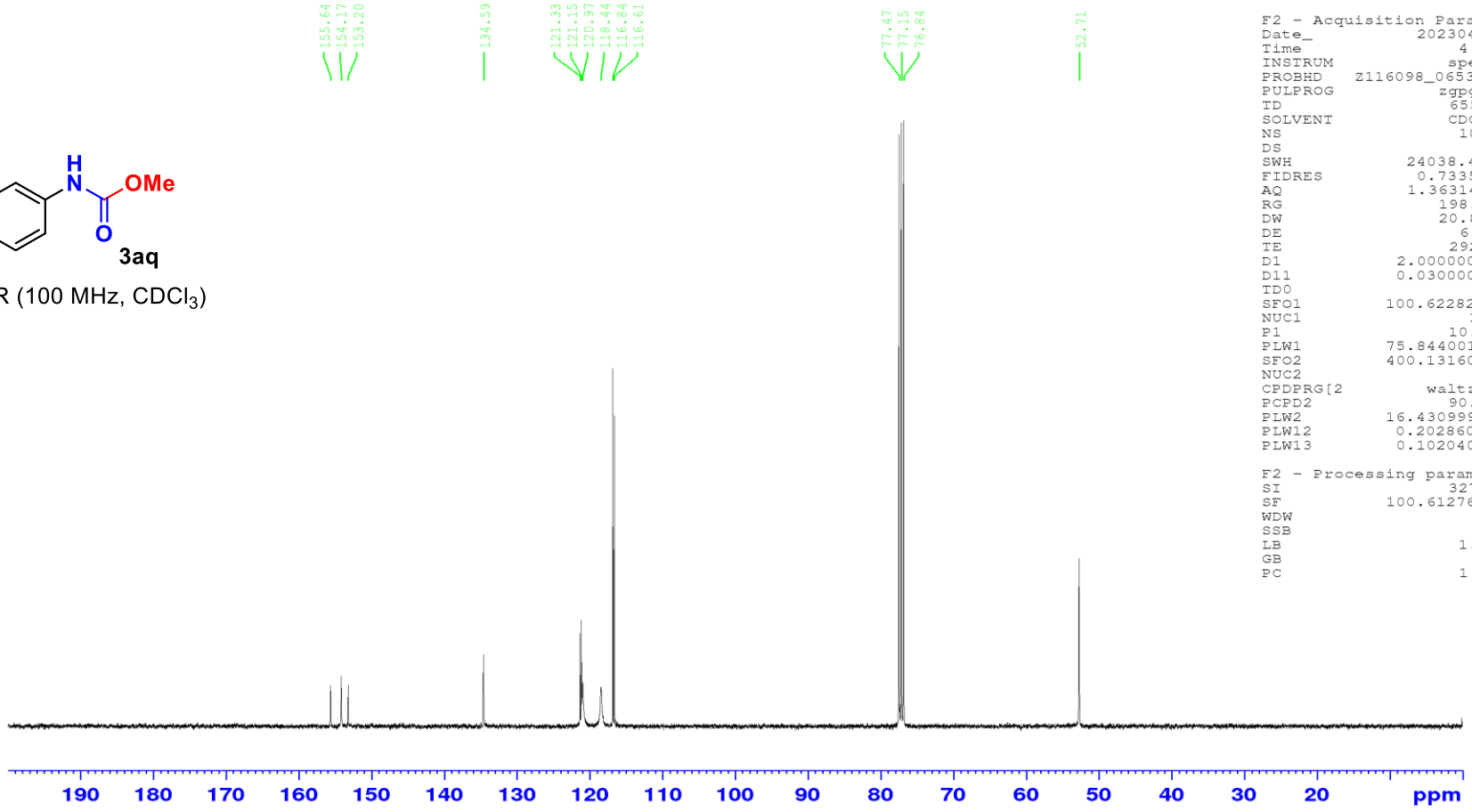



```
Current Data Parameters
NAME      1hz-20230414-6
EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
Date_            20230415
Time                3.45 h
INSTRUM             spect
PROBHD    Z116098_0653 (
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             8012.820 Hz
FIDRES          0.244532 Hz
AQ             4.0894465 sec
RG                 62.98
DW                62.400 usec
DE                 6.50 usec
TE                292.0 K
D1             1.00000000 sec
TD0                    1
SFO1          400.1324708 MHz
NUC1                  1H
P1                 10.00 usec
PLW1          16.43099976 W

F2 - Processing parameters
SI                 65536
SF            400.1300098 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```

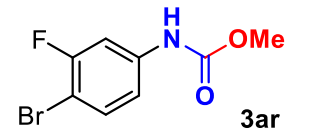


3aq

^{13}C NMR (100 MHz, $CDCl_3$)

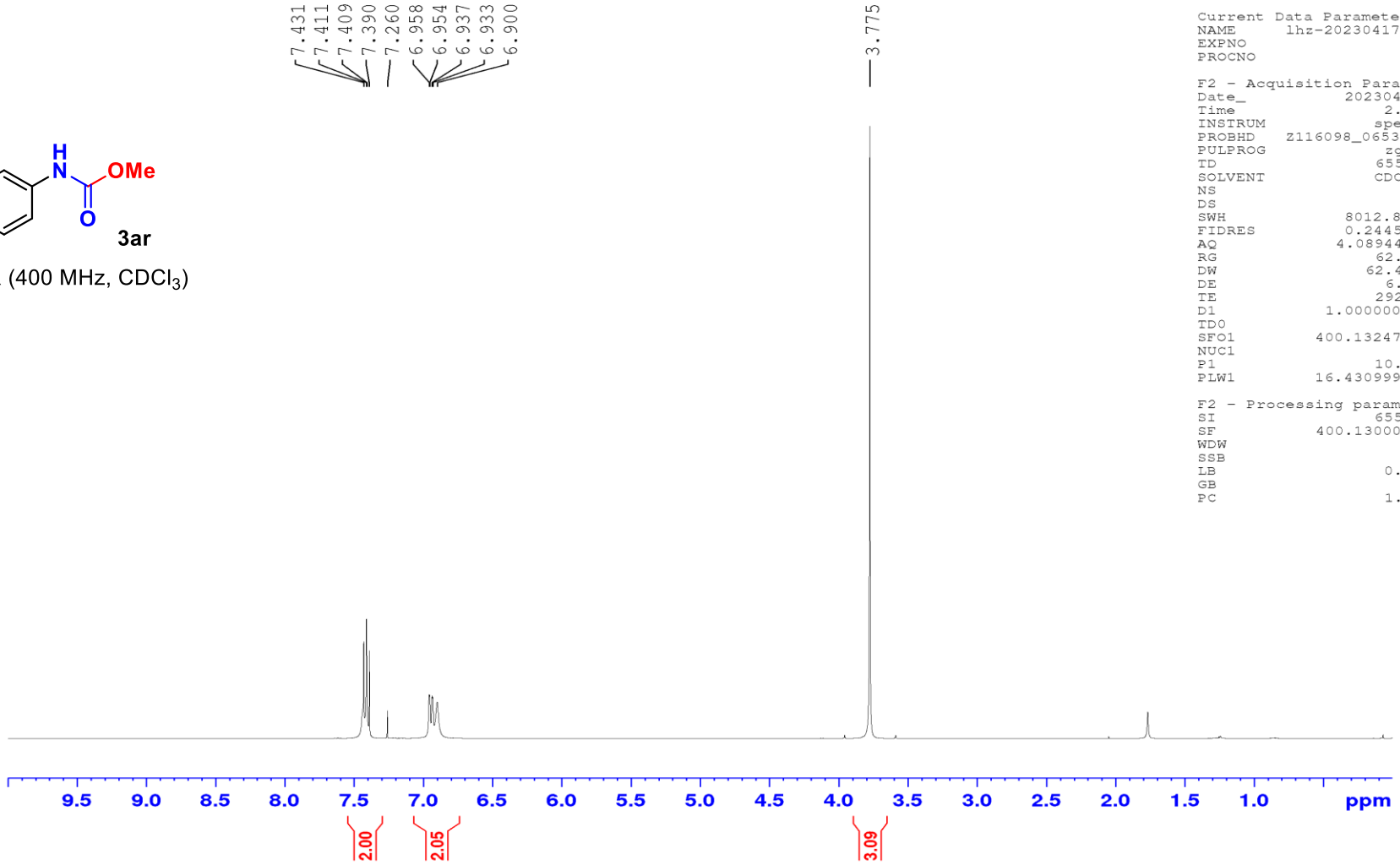



```
Current Data Parameters
NAME      lhz-20230414-6
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20230415
Time              4.45 h
INSTRUM           spect
PROBHD   Z116098_0653 (
PULPROG         zgpg30
TD               65536
SOLVENT          CDCl3
NS                1024
DS                   4
SWH          24038.461 Hz
FIDRES        0.733596 Hz
AQ           1.3631488 sec
RG              198.36
DW              20.800 usec
DE               6.50 usec
TE              292.7 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                  1
SFO1       100.6228298 MHz
NUC1              13C
P1              10.00 usec
PLW1       75.84400177 W
SFO2       400.1316005 MHz
NUC2              1H
CPDPRG[2       waltz16
PCPD2             90.00 usec
PLW2       16.43099976 W
PLW12       0.20286000 W
PLW13       0.10204000 W

F2 - Processing parameters
SI               32768
SF         100.6127601 MHz
WDW                EM
SSB                 0
LB               1.00 Hz
GB                  0
PC               1.40
```

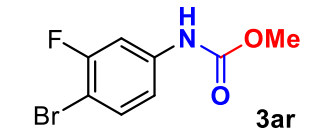


3ar

^{1}H NMR (400 MHz, $CDCl_3$)

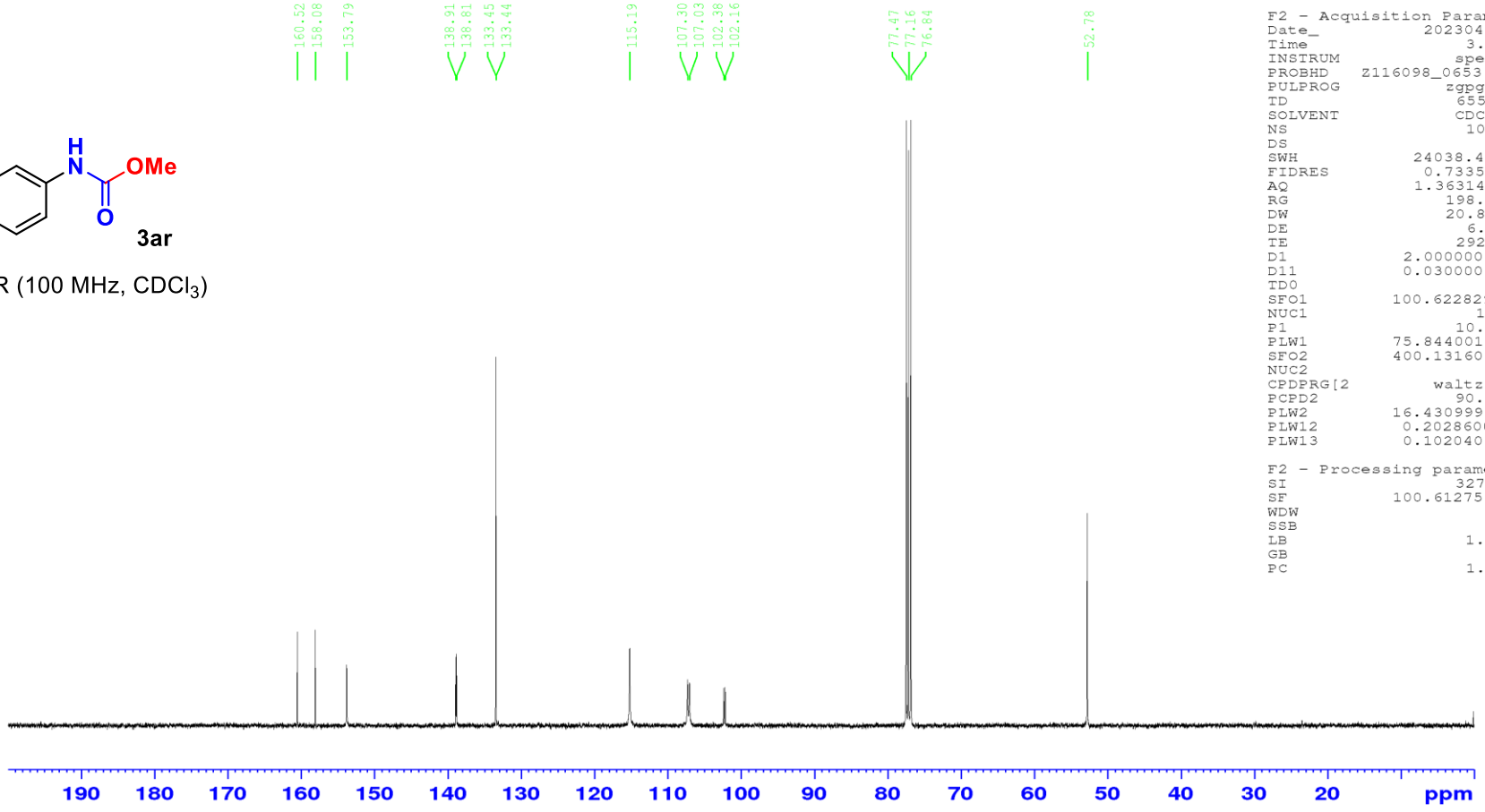



```
Current Data Parameters
NAME      1hz-20230417-5
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20230418
Time                2.39 h
INSTRUM            spect
PROBHD    Z116098_0653 (
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH              8012.820 Hz
FIDRES           0.244532 Hz
AQ               4.0894465 sec
RG                 62.98
DW                62.400 usec
DE                 6.50 usec
TE                292.3 K
D1             1.00000000 sec
TD0                    1
SFO1          400.1324708 MHz
NUC1                  1H
P1                 10.00 usec
PLW1          16.43099976 W

F2 - Processing parameters
SI                 65536
SF            400.1300098 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```

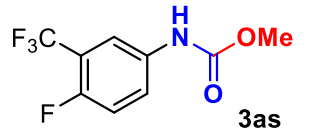


3ar

^{13}C NMR (100 MHz, $CDCl_3$)

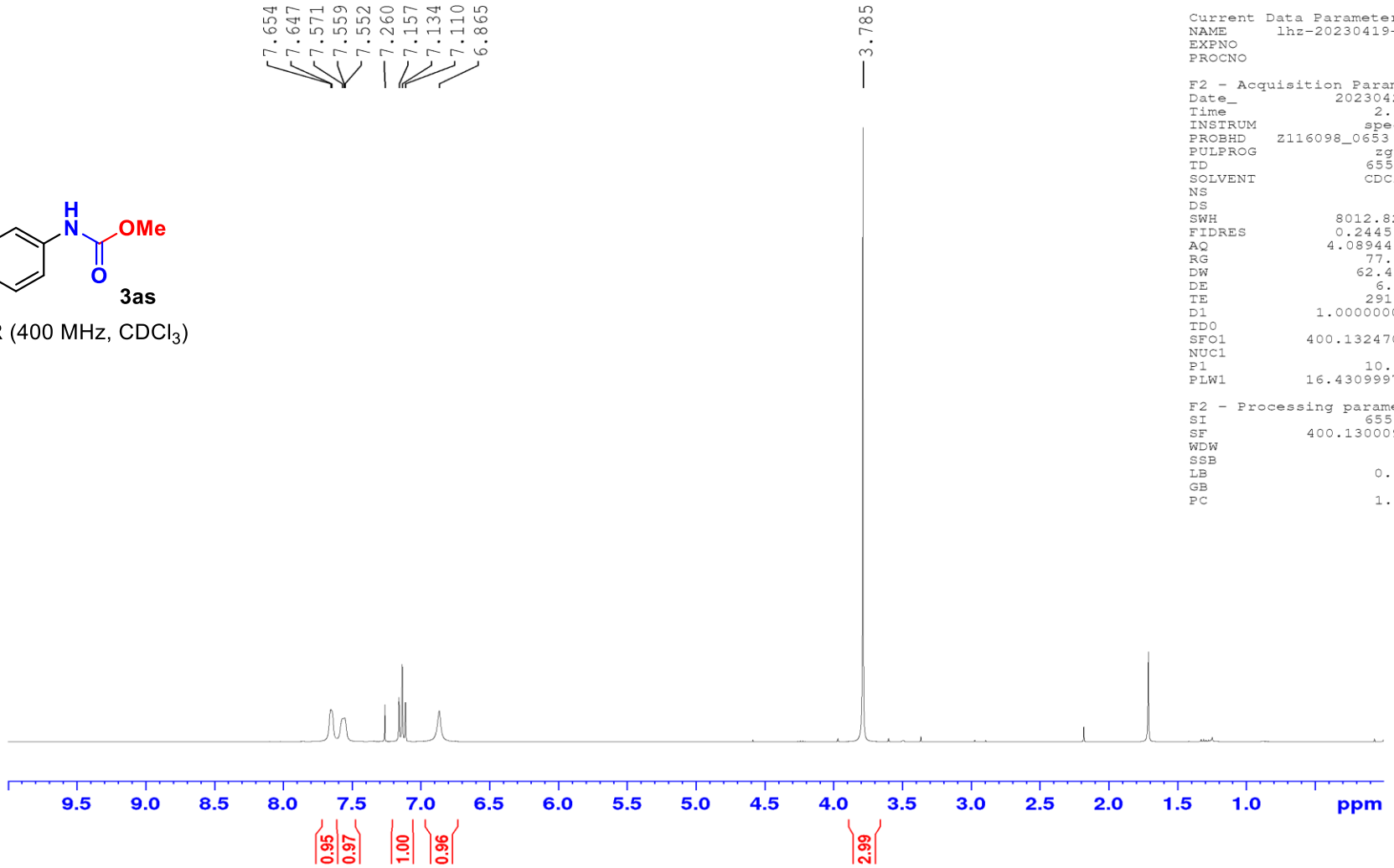



```
Current Data Parameters
NAME      1hz-20230417-5
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20230418
Time                3.39 h
INSTRUM            spect
PROBHD    Z116098_0653 (
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                 1024
DS                    4
SWH           24038.461 Hz
FIDRES         0.733596 Hz
AQ            1.3631488 sec
RG               198.36
DW               20.800 usec
DE                6.50 usec
TE               292.6 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                   1
SFO1        100.6228298 MHz
NUC1               13C
P1               10.00 usec
PLW1        75.84400177 W
SFO2        400.1316005 MHz
NUC2               1H
CPDPRG[2        waltz16
PCPD2            90.00 usec
PLW2        16.43099976 W
PLW12        0.20286000 W
PLW13        0.10204000 W

F2 - Processing parameters
SI                32768
SF          100.6127598 MHz
WDW                EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```

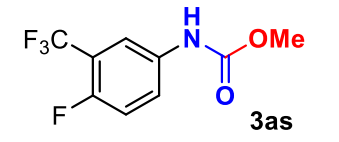


3as

^{1}H NMR (400 MHz, $CDCl_3$)

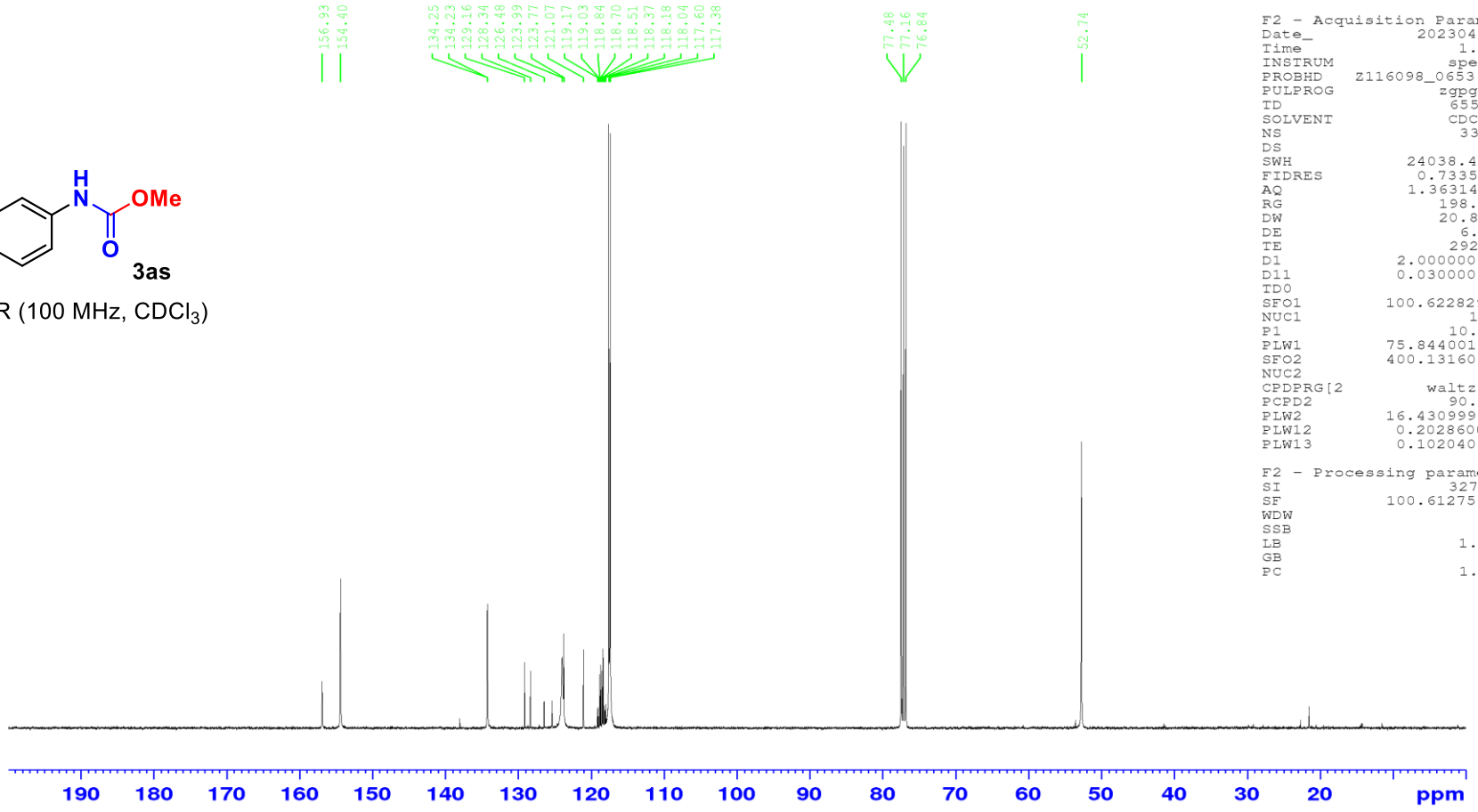



```
Current Data Parameters
NAME      lhz-20230419-4
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20230420
Time                2.39 h
INSTRUM            spect
PROBHD   Z116098_0653 (
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             8012.820 Hz
FIDRES          0.244532 Hz
AQ              4.0894465 sec
RG                 77.68
DW                62.400 usec
DE                 6.50 usec
TE                291.9 K
D1             1.00000000 sec
TD0                    1
SFO1          400.1324708 MHz
NUC1                  1H
P1                 10.00 usec
PLW1          16.43099976 W

F2 - Processing parameters
SI                 65536
SF            400.1300098 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```

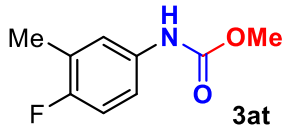


3as

^{13}C NMR (100 MHz, $CDCl_3$)

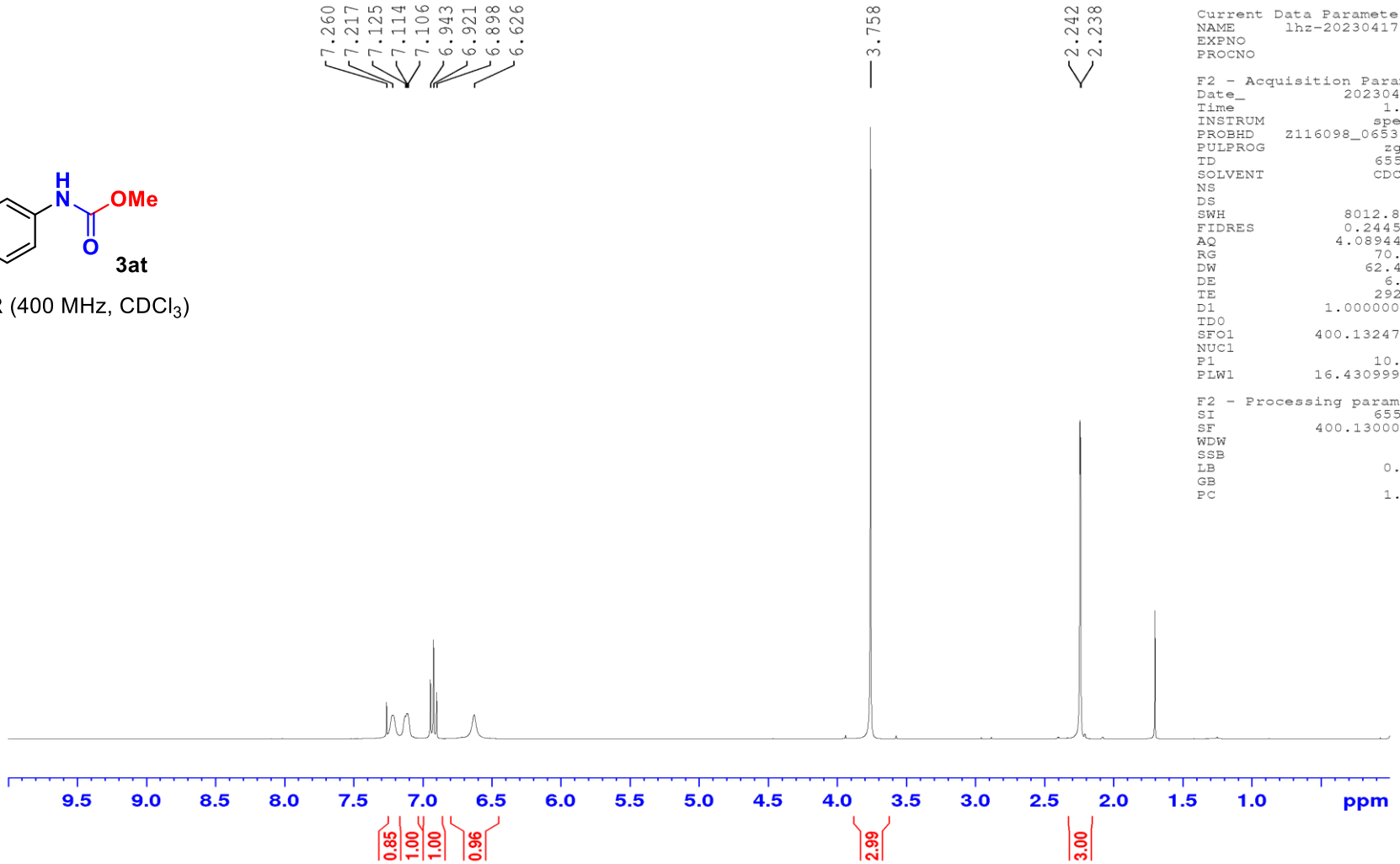



```
Current Data Parameters
NAME      1hz-20230428-1
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20230429
Time              1.31 h
INSTRUM           spect
PROBHD   Z116098_0653 (
PULPROG         zgpg30
TD               65536
SOLVENT          CDCl3
NS                3333
DS                   4
SWH          24038.461 Hz
FIDRES        0.733596 Hz
AQ           1.3631488 sec
RG              198.36
DW              20.800 usec
DE               6.50 usec
TE              292.3 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                  1
SFO1       100.6228298 MHz
NUC1              13C
P1              10.00 usec
PLW1       75.84400177 W
SFO2       400.1316005 MHz
NUC2              1H
CPDPRG[2       waltz16
PCPD2             90.00 usec
PLW2       16.43099976 W
PLW12       0.20286000 W
PLW13       0.10204000 W

F2 - Processing parameters
SI               32768
SF         100.6127580 MHz
WDW                EM
SSB                 0
LB              1.00 Hz
GB                  0
PC              1.40
```

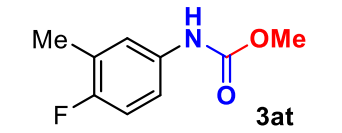


3at

^{1}H NMR (400 MHz, $CDCl_3$)

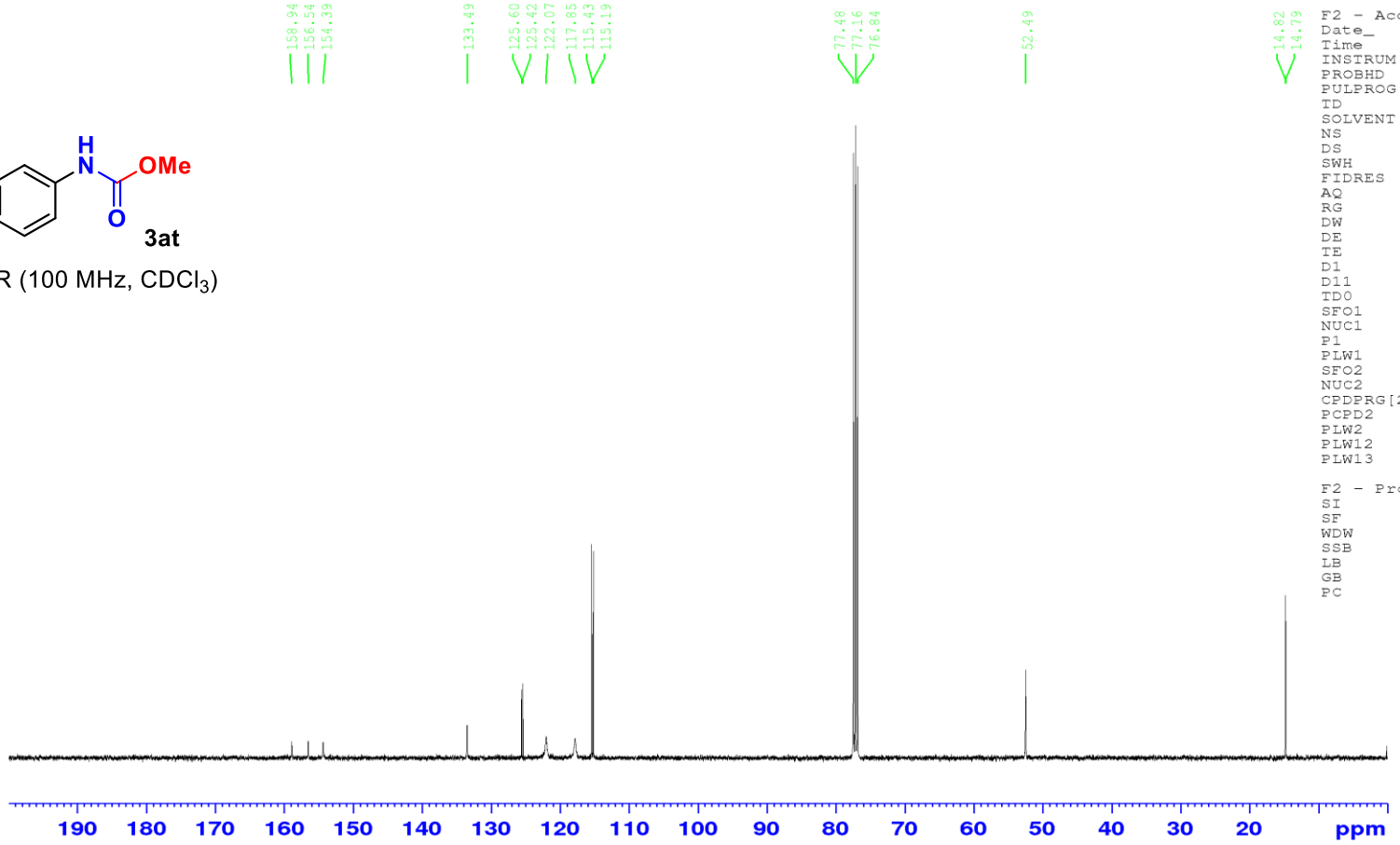



```
Current Data Parameters
NAME      1hz-20230417-4
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20230418
Time               1.35 h
INSTRUM           spect
PROBHD    Z116098_0653 (
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                   16
DS                    2
SWH            8012.820 Hz
FIDRES         0.244532 Hz
AQ             4.0894465 sec
RG                70.89
DW               62.400 usec
DE                6.50 usec
TE               292.4 K
D1            1.00000000 sec
TD0                   1
SFO1       400.1324708 MHz
NUC1               1H
P1                10.00 usec
PLW1       16.43099976 W

F2 - Processing parameters
SI                65536
SF         400.1300098 MHz
WDW                EM
SSB                   0
LB                0.30 Hz
GB                    0
PC                1.00
```

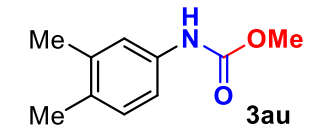


3at

^{13}C NMR (100 MHz, $CDCl_3$)

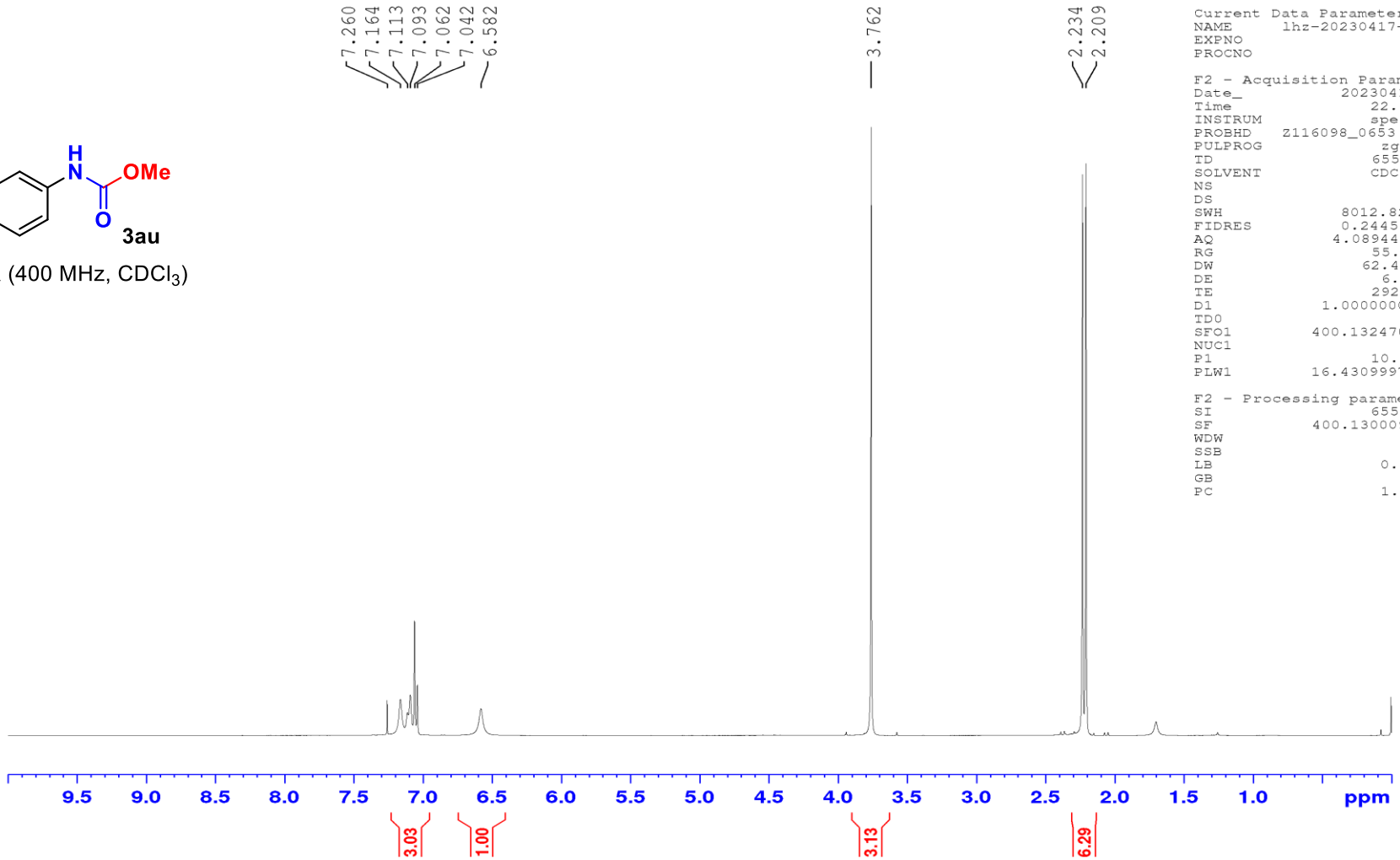



```
Current Data Parameters
NAME      1hz-20230417-4
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20230418
Time              2.35 h
INSTRUM           spect
PROBHD    Z116098_0653 (
PULPROG         zgpg30
TD               65536
SOLVENT          CDCl3
NS                1024
DS                   4
SWH          24038.461 Hz
FIDRES        0.733596 Hz
AQ            1.3631488 sec
RG               198.36
DW               20.800 usec
DE                6.50 usec
TE               292.6 K
D1          2.00000000 sec
D11         0.03000000 sec
TD0                  1
SFO1       100.6228298 MHz
NUC1              13C
P1               10.00 usec
PLW1       75.84400177 W
SFO2       400.1316005 MHz
NUC2              1H
CPDPRG[2       waltz16
PCPD2            90.00 usec
PLW2       16.43099976 W
PLW12       0.20286000 W
PLW13       0.10204000 W

F2 - Processing parameters
SI               32768
SF         100.6127586 MHz
WDW                EM
SSB                 0
LB               1.00 Hz
GB                  0
PC               1.40
```

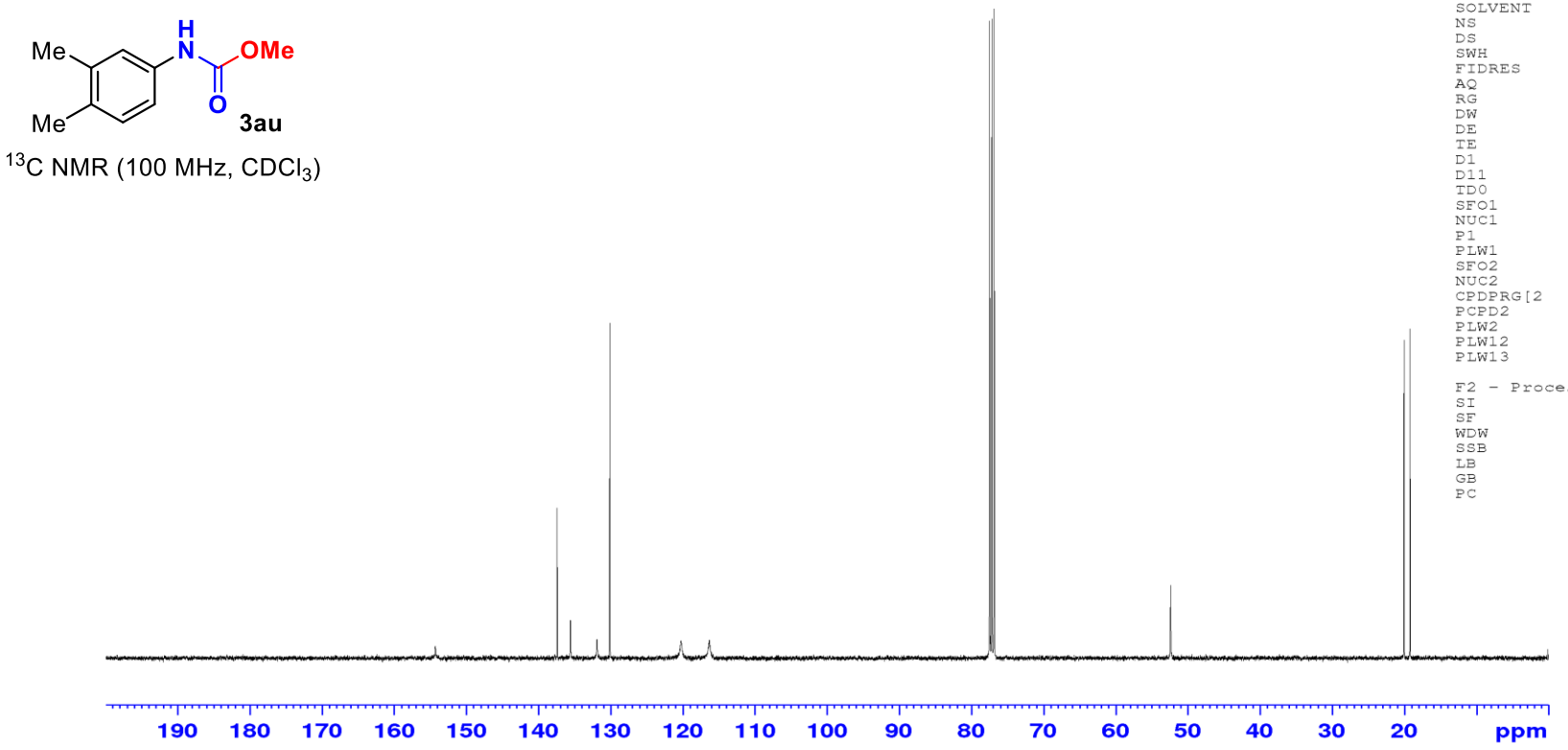


3au

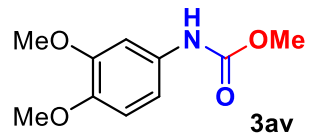
^{1}H NMR (400 MHz, $CDCl_3$)




```
Current Data Parameters
NAME      lhz-20230417-1
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20230417
Time              22.23 h
INSTRUM             spect
PROBHD    Z116098_0653 (
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             8012.820 Hz
FIDRES          0.244532 Hz
AQ              4.0894465 sec
RG                 55.99
DW                62.400 usec
DE                 6.50 usec
TE                292.0 K
D1             1.00000000 sec
TD0                    1
SFO1          400.1324708 MHz
NUC1                  1H
P1                10.00 usec
PLW1          16.43099976 W

F2 - Processing parameters
SI                 65536
SF            400.1300099 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```

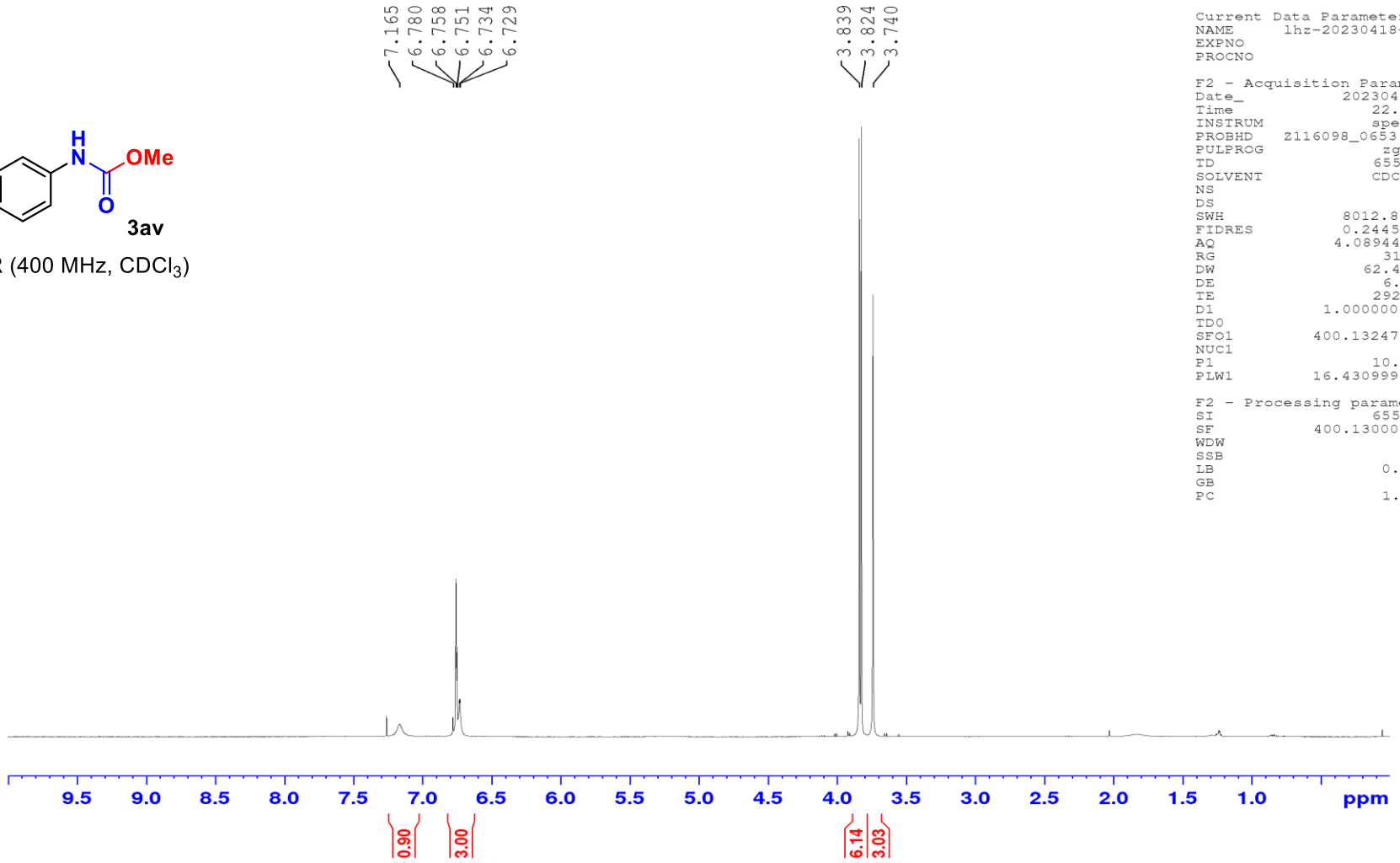



```
Current Data Parameters
NAME      1hz-20230417-1
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20230417
Time              23.22 h
INSTRUM             spect
PROBHD    Z116098_0653 (
PULPROG           zgpg30
TD                65536
SOLVENT           CDCl3
NS                 1024
DS                    4
SWH           24038.461 Hz
FIDRES         0.733596 Hz
AQ             1.3631488 sec
RG                198.36
DW                20.800 usec
DE                 6.50 usec
TE                292.4 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                   1
SFO1        100.6228298 MHz
NUC1                13C
P1                10.00 usec
PLW1        75.84400177 W
SFO2        400.1316005 MHz
NUC2                1H
CPDPRG[2        waltz16
PCPD2             90.00 usec
PLW2        16.43099976 W
PLW12        0.20286000 W
PLW13        0.10204000 W

F2 - Processing parameters
SI                32768
SF          100.6127601 MHz
WDW                EM
SSB                 0
LB                1.00 Hz
GB                  0
PC                1.40
```

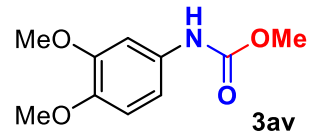


3av

^{1}H NMR (400 MHz, $CDCl_3$)

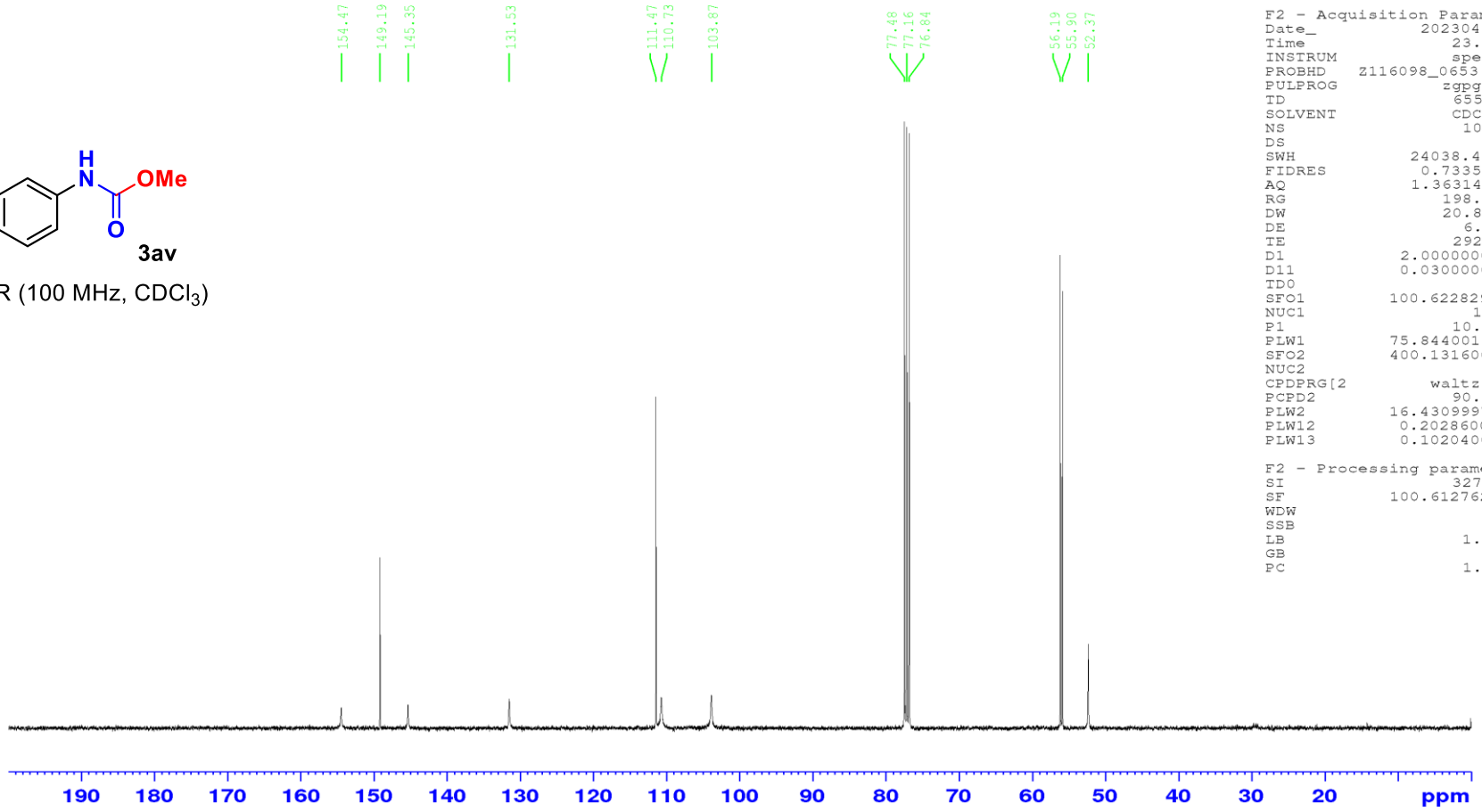



```
Current Data Parameters
NAME      1hz-20230418-1
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20230418
Time               22.30 h
INSTRUM            spect
PROBHD    Z116098_0653 (
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                   16
DS                    2
SWH            8012.820 Hz
FIDRES         0.244532 Hz
AQ             4.0894465 sec
RG                31.9
DW               62.400 usec
DE                6.50 usec
TE               292.1 K
D1            1.00000000 sec
TD0                   1
SFO1         400.1324708 MHz
NUC1                 1H
P1                10.00 usec
PLW1         16.43099976 W

F2 - Processing parameters
SI                65536
SF           400.1300097 MHz
WDW                  EM
SSB                   0
LB                 0.30 Hz
GB                    0
PC                 1.00
```

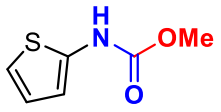


3av

^{13}C NMR (100 MHz, $CDCl_3$)

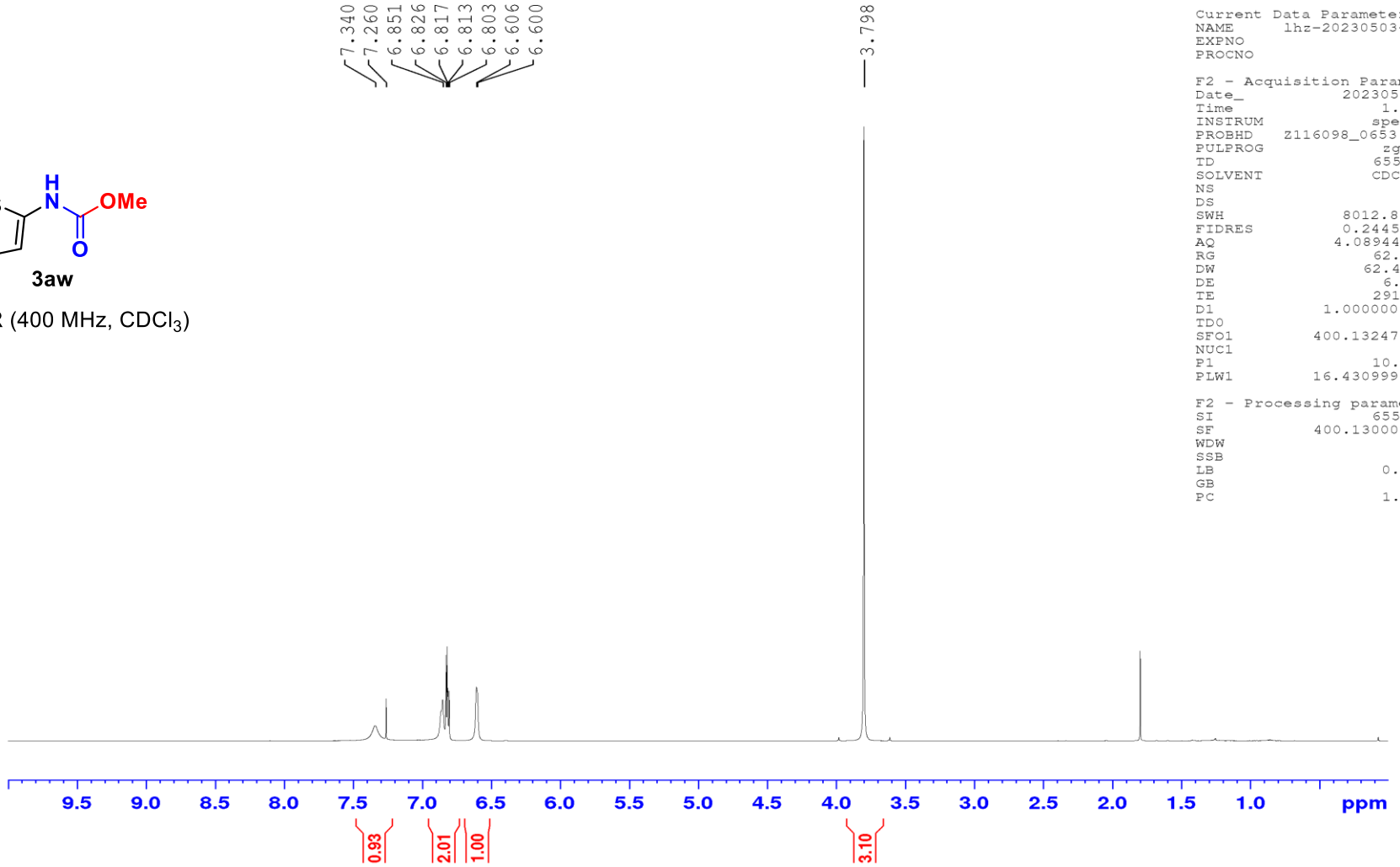



```
Current Data Parameters
NAME      lhz-20230418-1
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20230418
Time              23.30 h
INSTRUM            spect
PROBHD   Z116098_0653 (
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                 1024
DS                    4
SWH           24038.461 Hz
FIDRES         0.733596 Hz
AQ            1.3631488 sec
RG               198.36
DW               20.800 usec
DE                6.50 usec
TE               292.5 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                   1
SFO1        100.6228298 MHz
NUC1               13C
P1               10.00 usec
PLW1        75.84400177 W
SFO2        400.1316005 MHz
NUC2               1H
CPDPRG[2        waltz16
PCPD2            90.00 usec
PLW2        16.43099976 W
PLW12        0.20286000 W
PLW13        0.10204000 W

F2 - Processing parameters
SI                32768
SF          100.6127623 MHz
WDW                EM
SSB                0
LB              1.00 Hz
GB                 0
PC              1.40
```

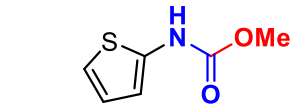


3aw

^{1}H NMR (400 MHz, $CDCl_3$)

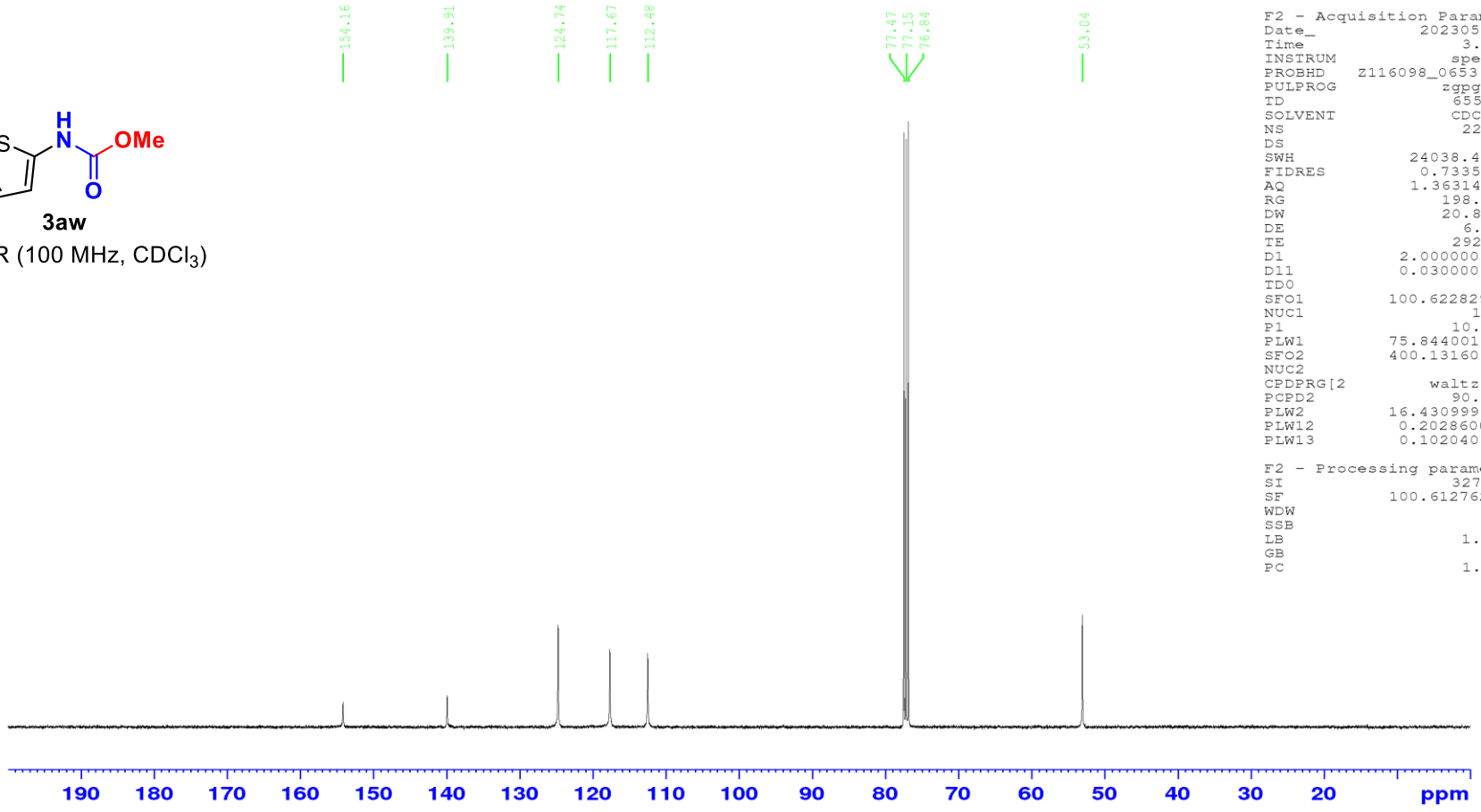



```
Current Data Parameters
NAME      1hz-20230503-3
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20230504
Time              1.27 h
INSTRUM           spect
PROBHD    Z116098_0653 (
PULPROG           zg30
TD               65536
SOLVENT          CDCl3
NS                  16
DS                   2
SWH           8012.820 Hz
FIDRES        0.244532 Hz
AQ            4.0894465 sec
RG               62.98
DW              62.400 usec
DE               6.50 usec
TE              291.5 K
D1           1.00000000 sec
TD0                  1
SFO1       400.1324708 MHz
NUC1               1H
P1              10.00 usec
PLW1       16.43099976 W

F2 - Processing parameters
SI               65536
SF         400.1300098 MHz
WDW                EM
SSB                 0
LB               0.30 Hz
GB                  0
PC               1.00
```

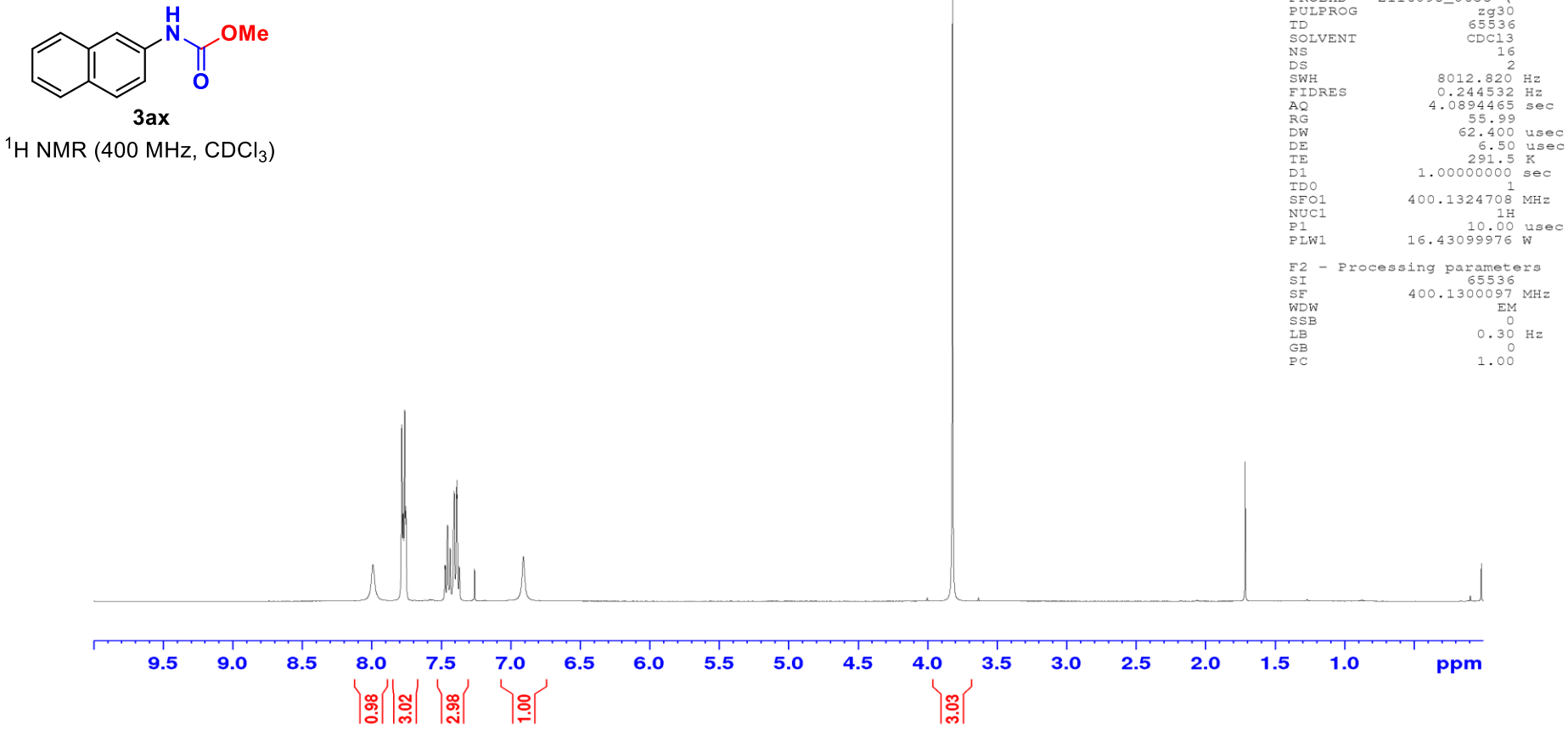


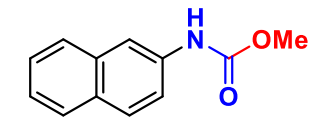
3aw

^{13}C NMR (100 MHz, $CDCl_3$)



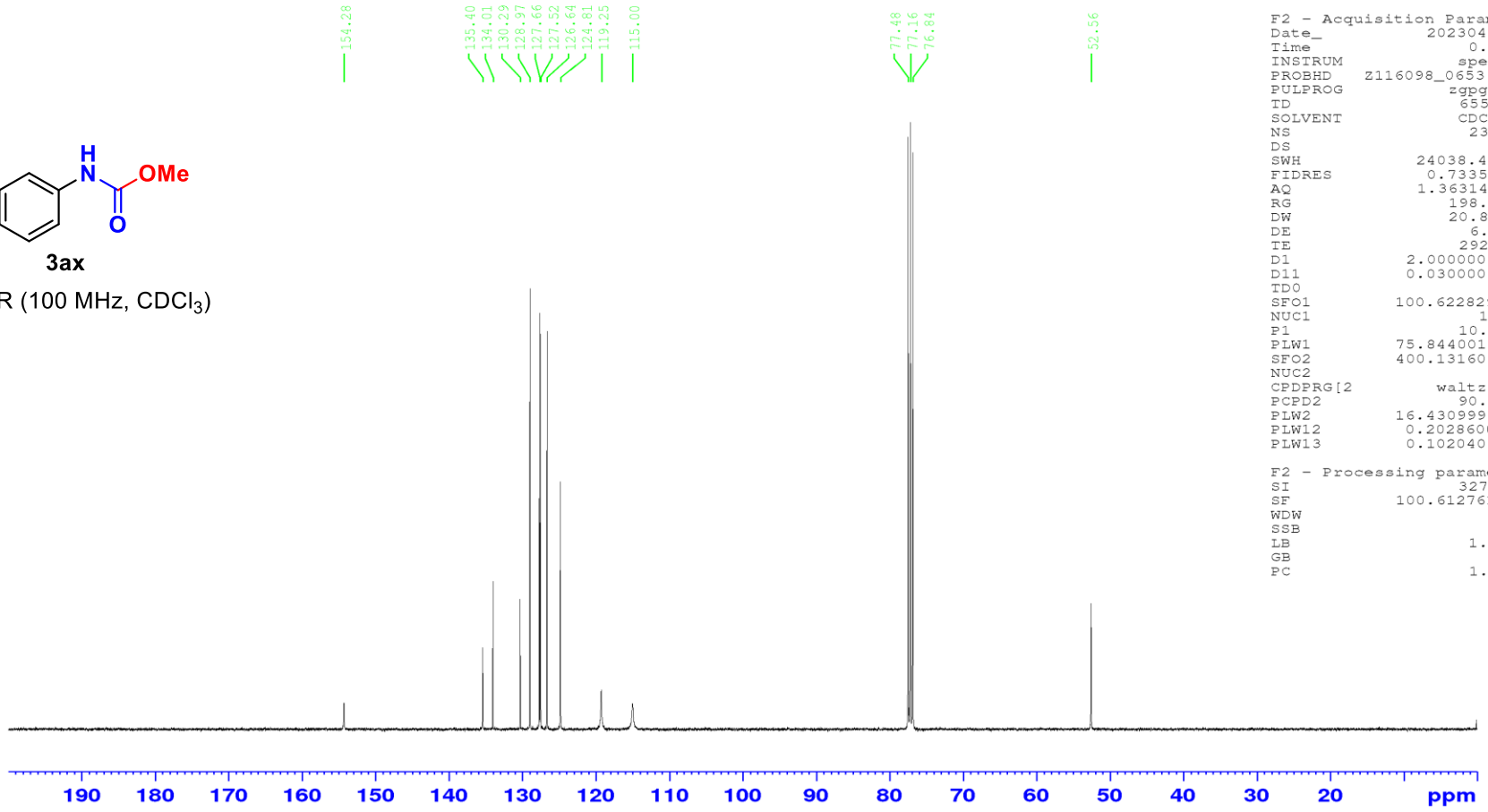

```
Current Data Parameters
NAME      1hz-20230503-3
EXPNO                   2
PROCNO                  1

F2 - Acquisition Parameters
Date_           20230504
Time              3.35 h
INSTRUM            spect
PROBHD    Z116098_0653 (
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                 2222
DS                    4
SWH           24038.461 Hz
FIDRES         0.733596 Hz
AQ             1.3631488 sec
RG                198.36
DW                20.800 usec
DE                 6.50 usec
TE                292.3 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1
SFO1        100.6228298 MHz
NUC1               13C
P1               10.00 usec
PLW1        75.84400177 W
SFO2        400.1316005 MHz
NUC2               1H
CPDPRG[2       waltz16
PCPD2            90.00 usec
PLW2        16.43099976 W
PLW12        0.20286000 W
PLW13        0.10204000 W

F2 - Processing parameters
SI                32768
SF          100.6127623 MHz
WDW                EM
SSB                 0
LB              1.00 Hz
GB                  0
PC              1.40
```


7.992
7.784
7.774
7.763
7.754
7.474
7.471
7.457
7.454
7.436
7.434
7.408
7.406
7.388
7.385
7.371
7.368
7.260
6.909
3.820
H
N
OMe
O
3ax
1H NMR (400 MHz, CDCl3)
Current Data Parameters
NAME 1hz-20230420-2
EXPNO 4
PROCNO 1
F2 - Acquisition Parameters
Date_ 20230420
Time 22.21 h
INSTRUM spect
PROBHD Z116098_0653 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.244532 Hz
AQ 4.0894465 sec
RG 55.99
DW 62.400 usec
DE 6.50 usec
TE 291.5 K
D1 1.00000000 sec
TD0 1
SFO1 400.1324708 MHz
NUC1 1H
P1 10.00 usec
PLW1 16.43099976 W
F2 - Processing parameters
SI 65536
SF 400.1300097 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
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
ppm
0.98
3.02
2.98
1.00
3.03

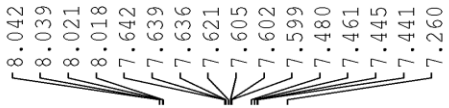


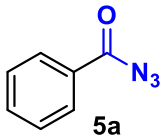
3ax

^{13}C NMR (100 MHz, $CDCl_3$)




```
Current Data Parameters
NAME      1hz-20230420-2
EXPNO                  3
PROCNO                 1

F2 - Acquisition Parameters
Date_           20230421
Time               0.35 h
INSTRUM           spect
PROBHD    Z116098_0653 (
PULPROG         zgpg30
TD               65536
SOLVENT          CDCl3
NS                2333
DS                   4
SWH          24038.461 Hz
FIDRES        0.733596 Hz
AQ            1.3631488 sec
RG               198.36
DW               20.800 usec
DE                6.50 usec
TE               292.5 K
D1          2.00000000 sec
D11         0.03000000 sec
TD0                  1
SFO1       100.6228298 MHz
NUC1              13C
P1               10.00 usec
PLW1       75.84400177 W
SFO2       400.1316005 MHz
NUC2              1H
CPDPRG[2       waltz16
PCPD2            90.00 usec
PLW2       16.43099976 W
PLW12       0.20286000 W
PLW13       0.10204000 W

F2 - Processing parameters
SI               32768
SF         100.6127616 MHz
WDW                EM
SSB                 0
LB              1.00 Hz
GB                  0
PC              1.40
```

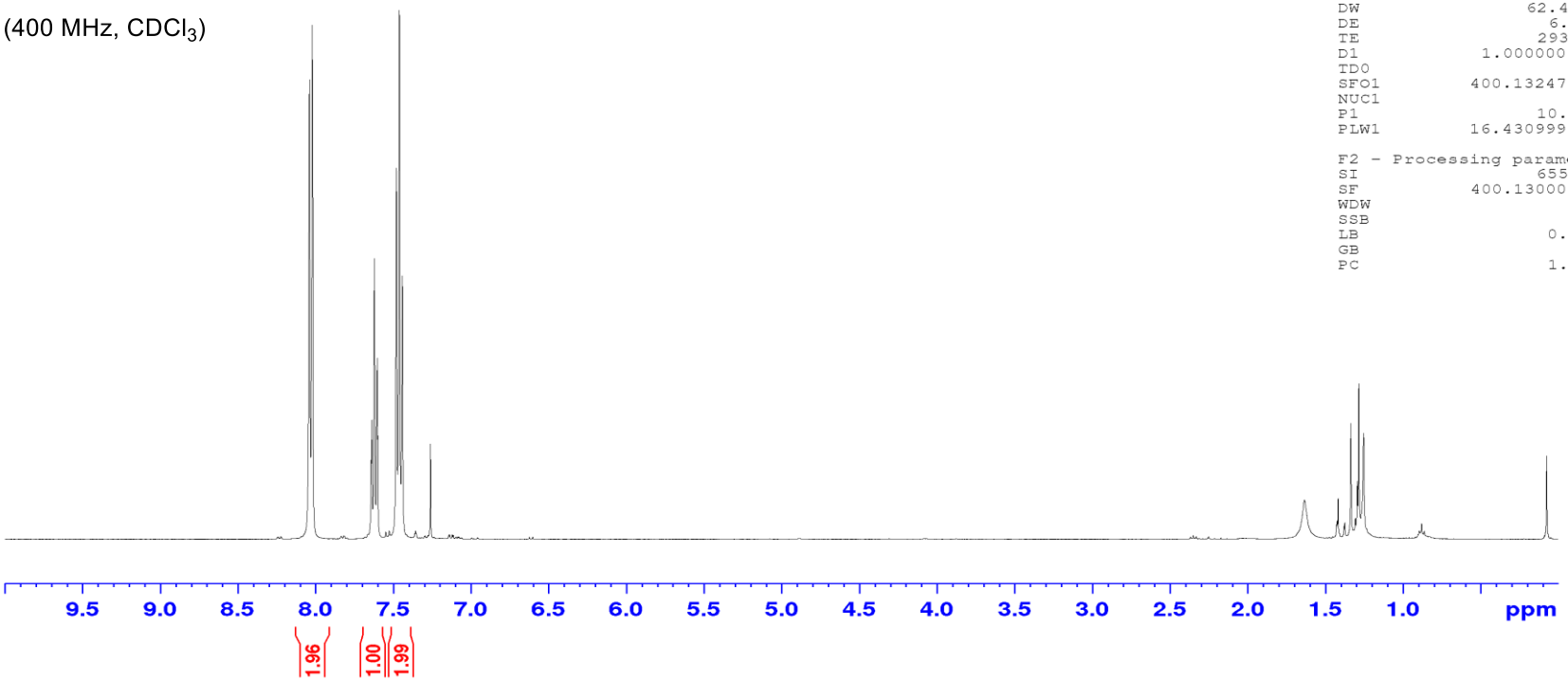


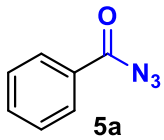


5a

^{1}H NMR (400 MHz, $CDCl_3$)

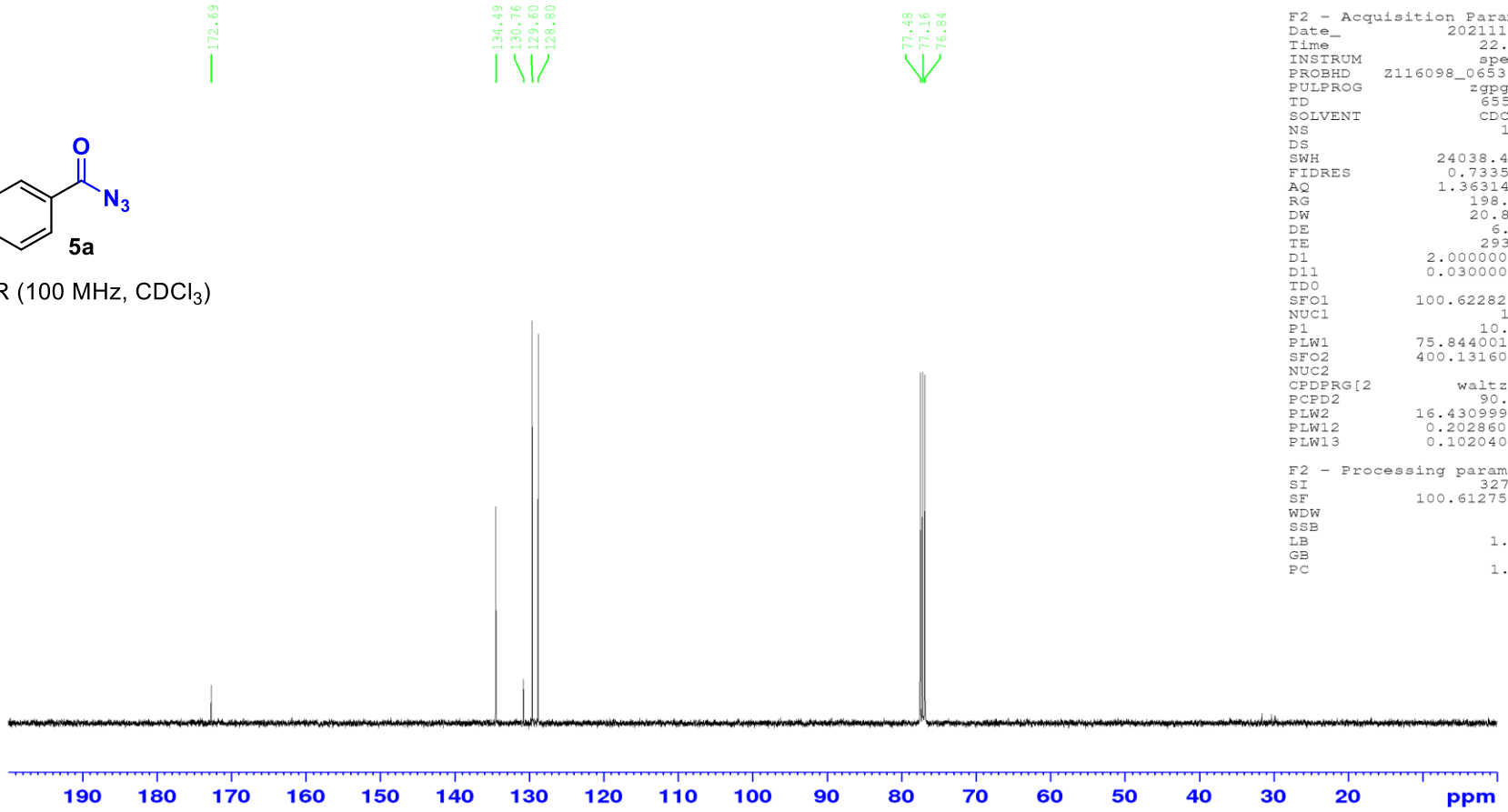
```
Current Data Parameters
NAME      lhz-20211105-4
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20211105
Time               22.29 h
INSTRUM            spect
PROBHD    Z116098_0653 (
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                    8
DS                    2
SWH            8012.820 Hz
FIDRES         0.244532 Hz
AQ             4.0894465 sec
RG                77.68
DW               62.400 usec
DE                6.50 usec
TE                293.4 K
D1           1.00000000 sec
TD0                   1
SFO1         400.1324708 MHz
NUC1                  1H
P1                10.00 usec
PLW1         16.43099976 W

F2 - Processing parameters
SI                65536
SF           400.1300097 MHz
WDW                  EM
SSB                   0
LB                 0.30 Hz
GB                    0
PC                 1.00
```



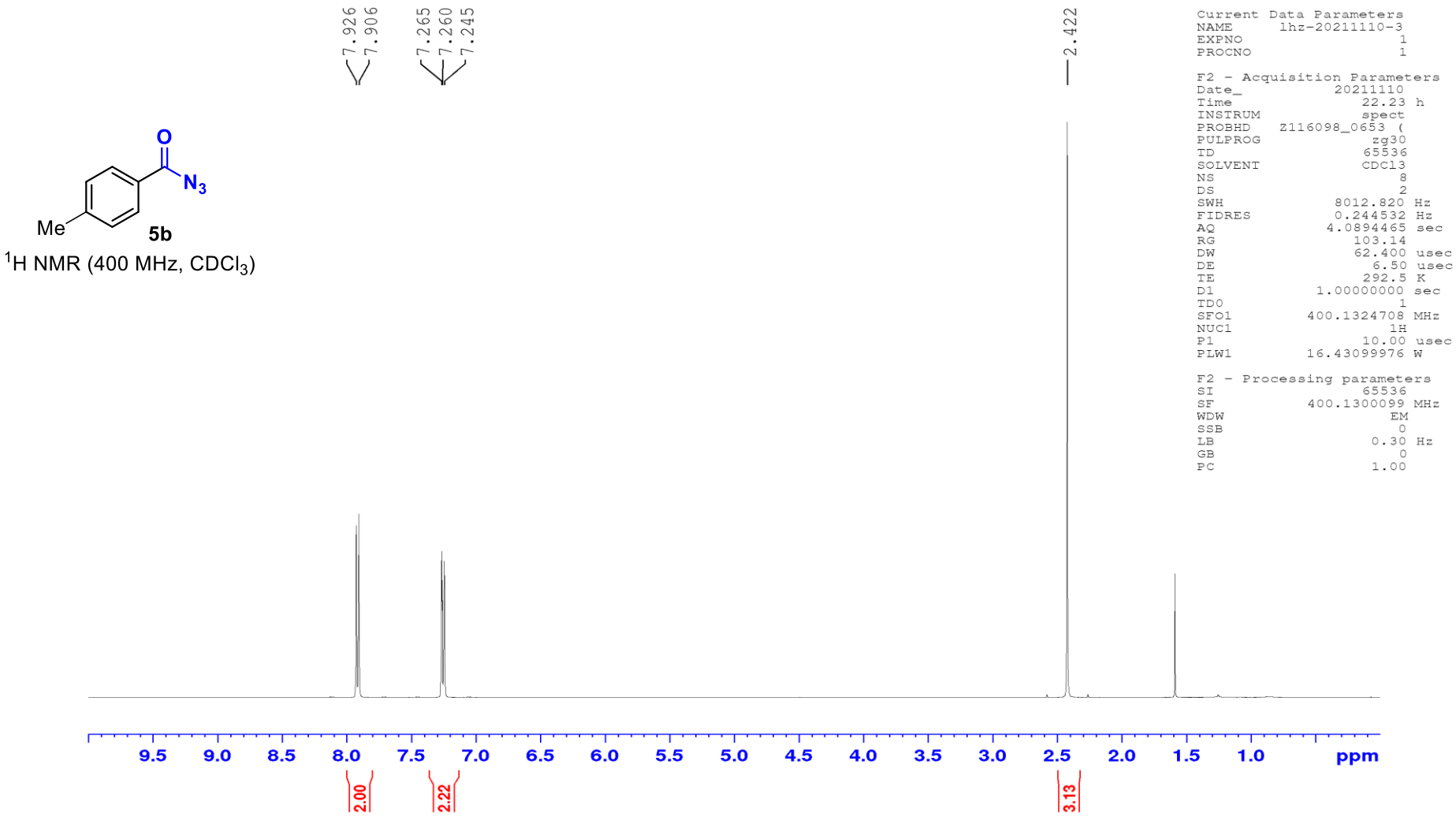


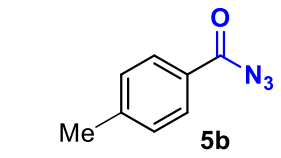
5a

^{13}C NMR (100 MHz, $CDCl_3$)



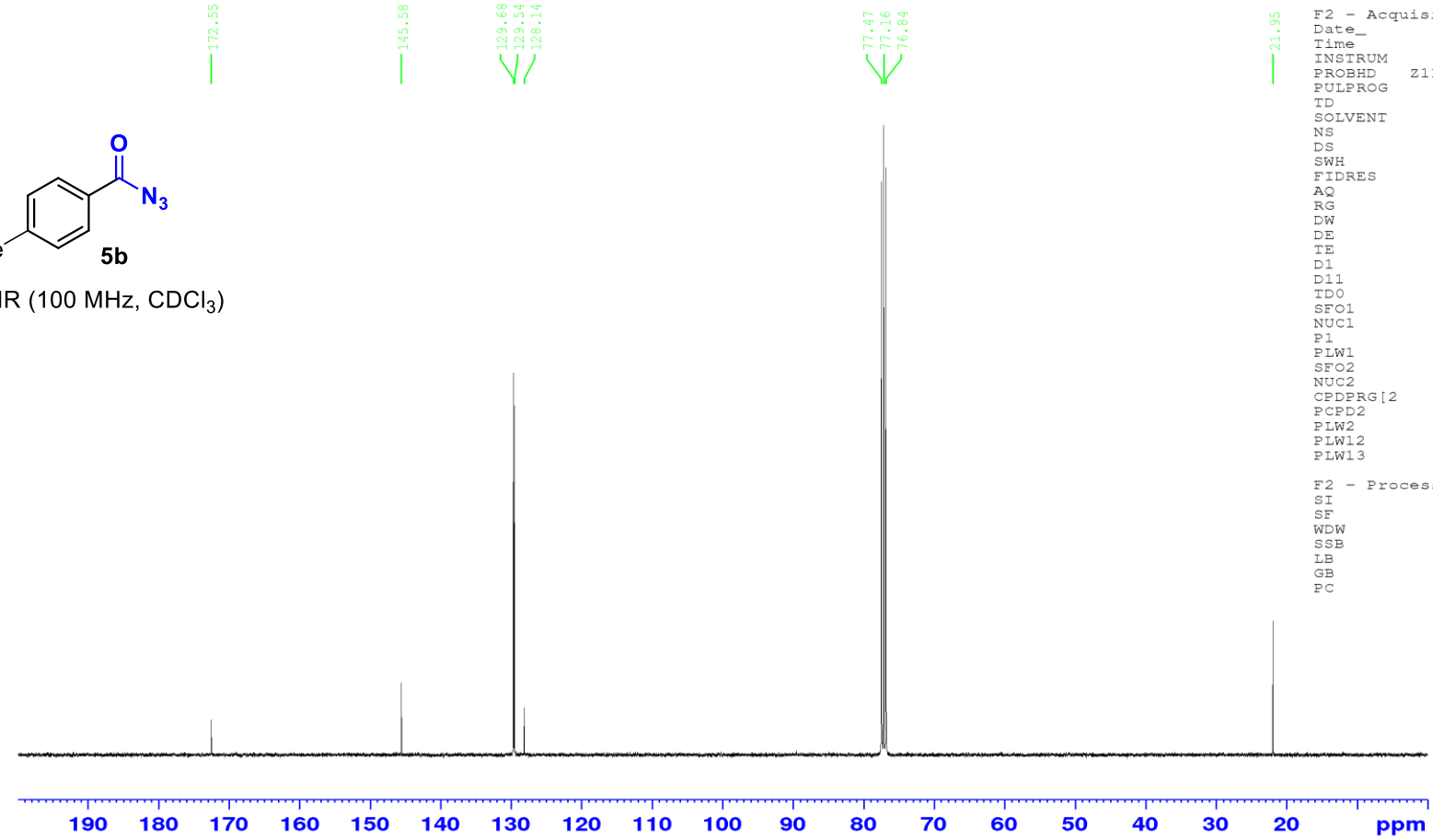

```
Current Data Parameters
NAME      lhz-20211105-4
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20211105
Time               22.37 h
INSTRUM            spect
PROBHD    Z116098_0653 (
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  133
DS                    4
SWH           24038.461 Hz
FIDRES         0.733596 Hz
AQ            1.3631488 sec
RG               198.36
DW               20.800 usec
DE                6.50 usec
TE               293.9 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1
SFO1        100.6228298 MHz
NUC1               13C
P1               10.00 usec
PLW1        75.84400177 W
SFO2        400.1316005 MHz
NUC2               1H
CPDPRG[2        waltz16
PCPD2            90.00 usec
PLW2        16.43099976 W
PLW12        0.20286000 W
PLW13        0.10204000 W

F2 - Processing parameters
SI                32768
SF          100.6127579 MHz
WDW                EM
SSB                0
LB              1.00 Hz
GB                 0
PC              1.40
```


7.926
7.906
7.265
7.260
7.245
2.422
O
N3
Me
5b
1H NMR (400 MHz, CDCl3)
Current Data Parameters
NAME 1hz-20211110-3
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20211110
Time 22.23 h
INSTRUM spect
PROBHD Z116098_0653 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 8012.820 Hz
FIDRES 0.244532 Hz
AQ 4.0894465 sec
RG 103.14
DW 62.400 usec
DE 6.50 usec
TE 292.5 K
D1 1.00000000 sec
TD0 1
SFO1 400.1324708 MHz
NUC1 1H
P1 10.00 usec
PLW1 16.43099976 W
F2 - Processing parameters
SI 65536
SF 400.1300099 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
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 ppm
2.00
2.22
3.13

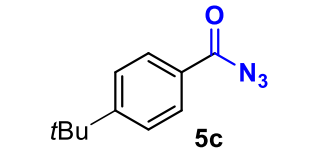


5b

^{13}C NMR (100 MHz, $CDCl_3$)

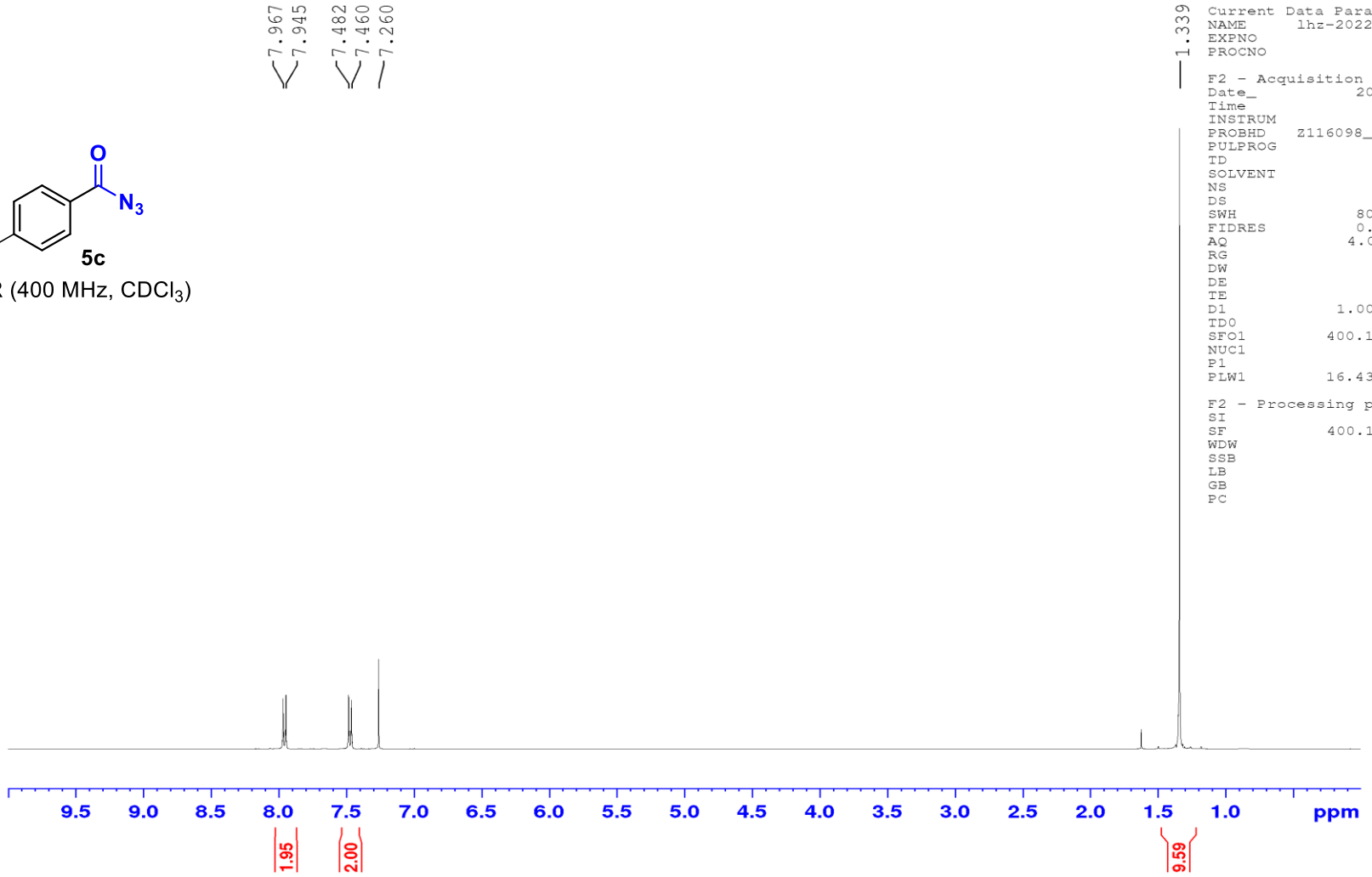



```
Current Data Parameters
NAME      1hz-20211110-3
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20211110
Time              23.22 h
INSTRUM             spect
PROBHD    Z116098_0653 (
PULPROG           zgpg30
TD                65536
SOLVENT           CDCl3
NS                 1024
DS                    4
SWH           24038.461 Hz
FIDRES         0.733596 Hz
AQ             1.3631488 sec
RG               198.36
DW               20.800 usec
DE                6.50 usec
TE               293.5 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                   1
SFO1        100.6228298 MHz
NUC1                13C
P1                10.00 usec
PLW1         75.84400177 W
SFO2        400.1316005 MHz
NUC2                1H
CPDPRG[2        waltz16
PCPD2             90.00 usec
PLW2         16.43099976 W
PLW12         0.20286000 W
PLW13         0.10204000 W

F2 - Processing parameters
SI                32768
SF          100.6127571 MHz
WDW                EM
SSB                 0
LB                1.00 Hz
GB                  0
PC                1.40
```

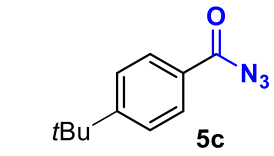


5c

^{1}H NMR (400 MHz, $CDCl_3$)

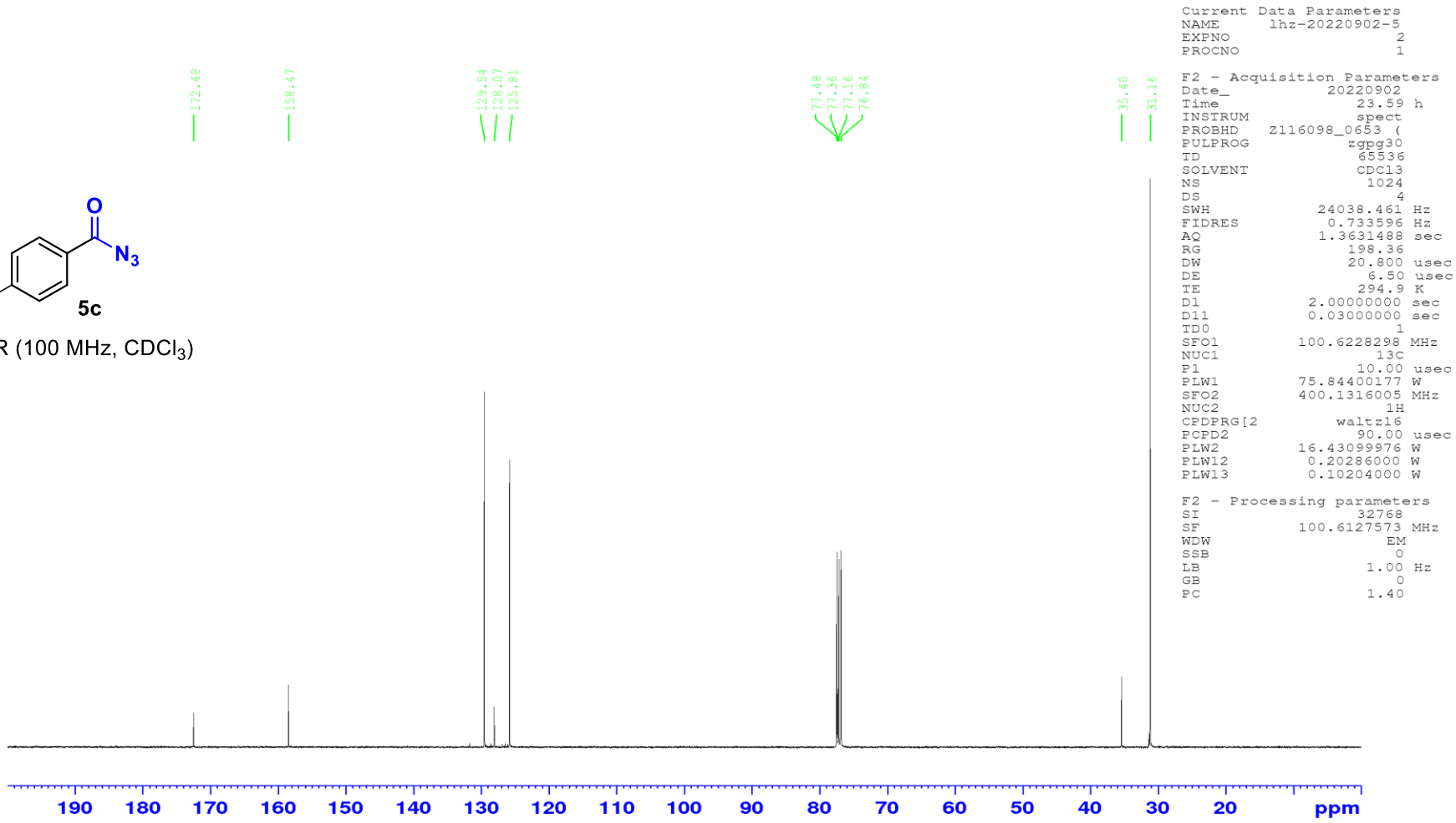



```
Current Data Parameters
NAME      lhz-20220902-5
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20220902
Time              23.00 h
INSTRUM             spect
PROBHD    Z116098_0653 (
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             8012.820 Hz
FIDRES          0.244532 Hz
AQ              4.0894465 sec
RG                  50.3
DW                62.400 usec
DE                 6.50 usec
TE                294.6 K
D1             1.00000000 sec
TD0                    1
SFO1          400.1324708 MHz
NUC1                  1H
P1                10.00 usec
PLW1          16.43099976 W

F2 - Processing parameters
SI                 65536
SF            400.1300098 MHz
WDW                   EM
SSB                    0
LB                0.30 Hz
GB                     0
PC                1.00
```

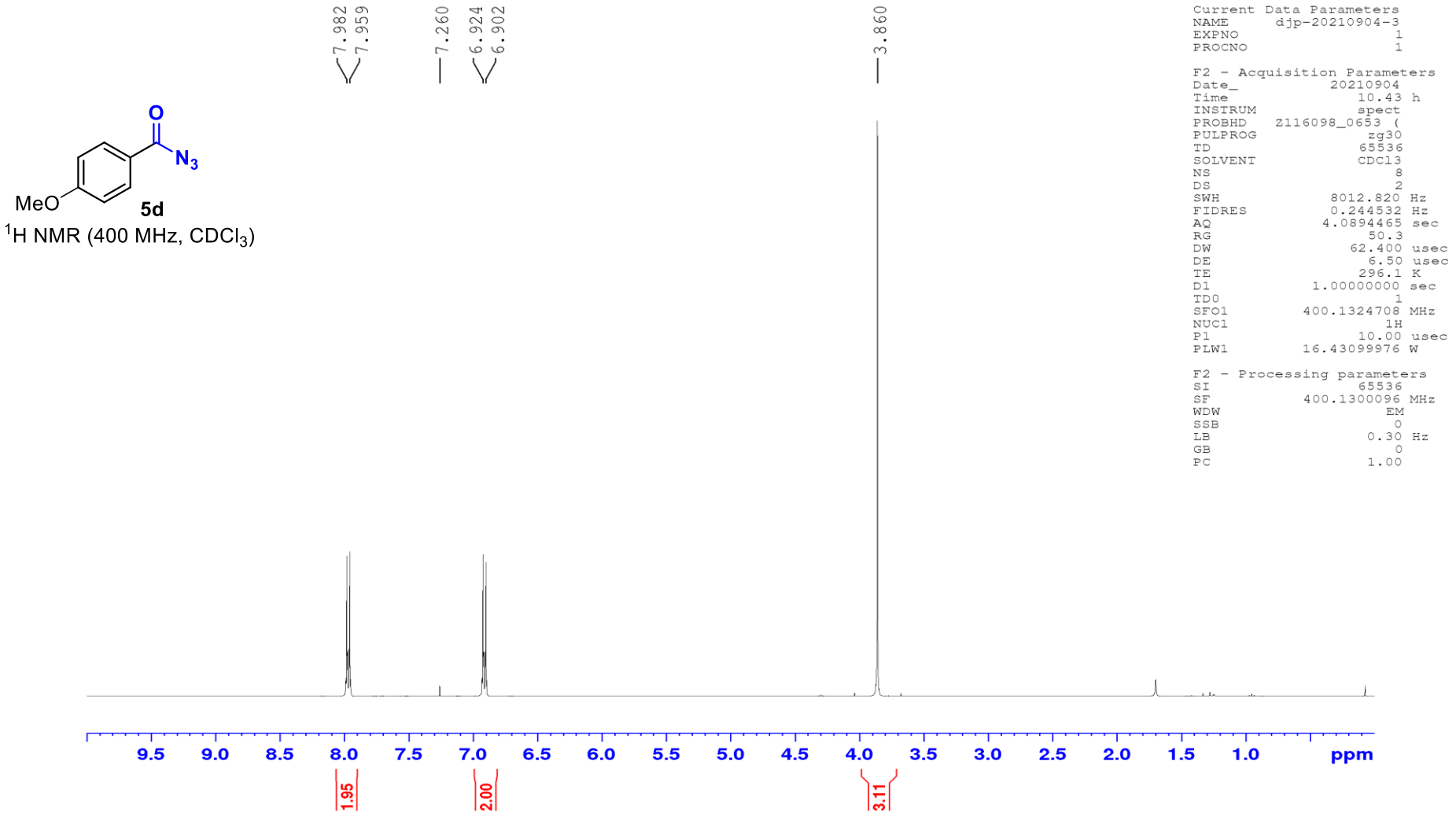


5c

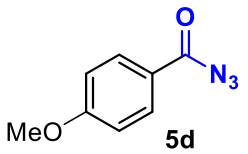
^{13}C NMR (100 MHz, $CDCl_3$)




```
Current Data Parameters
NAME      lhz-20220902-5
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20220902
Time              23.59 h
INSTRUM             spect
PROBHD   Z116098_0653 (
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                 1024
DS                    4
SWH           24038.461 Hz
FIDRES        0.733596 Hz
AQ            1.3631488 sec
RG               198.36
DW               20.800 usec
DE                6.50 usec
TE                294.9 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1
SFO1        100.6228298 MHz
NUC1                13C
P1                10.00 usec
PLW1        75.84400177 W
SFO2        400.1316005 MHz
NUC2                1H
CPDPRG[2        waltz16
PCPD2             90.00 usec
PLW2        16.43099976 W
PLW12        0.20286000 W
PLW13        0.10204000 W

F2 - Processing parameters
SI                32768
SF          100.6127573 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```

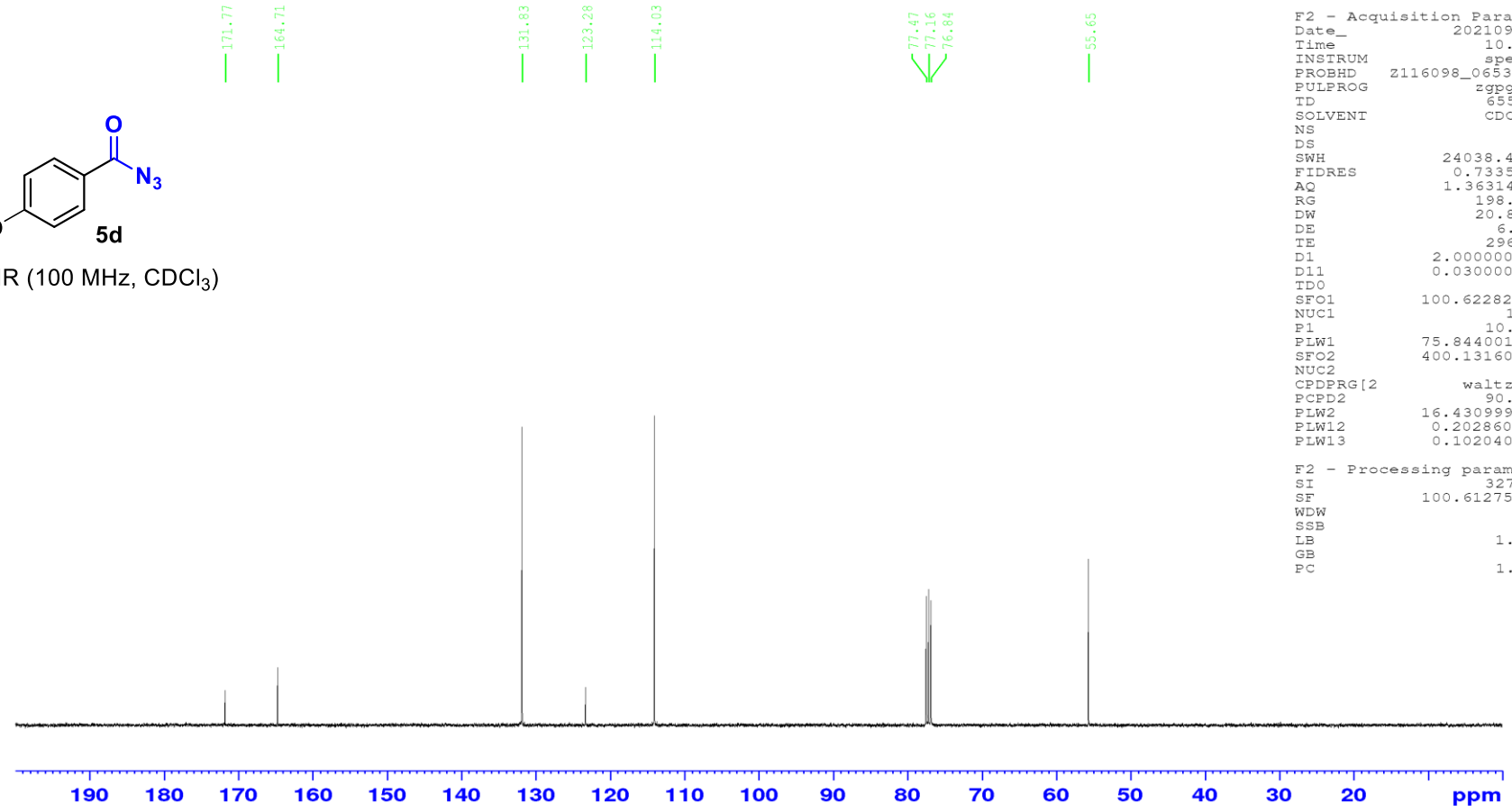



```
Current Data Parameters
NAME      djp-20210904-3
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20210904
Time              10.43 h
INSTRUM             spect
PROBHD   Z116098_0653 (
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                     8
DS                     2
SWH             8012.820 Hz
FIDRES          0.244532 Hz
AQ              4.0894465 sec
RG                  50.3
DW                62.400 usec
DE                 6.50 usec
TE                296.1 K
D1             1.00000000 sec
TD0                    1
SFO1          400.1324708 MHz
NUC1                  1H
P1                 10.00 usec
PLW1          16.43099976 W

F2 - Processing parameters
SI                 65536
SF            400.1300096 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```

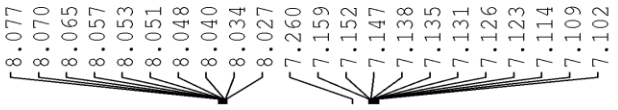


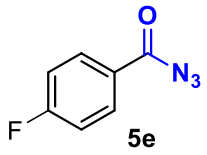
5d

^{13}C NMR (100 MHz, $CDCl_3$)




```
Current Data Parameters
NAME      djp-20210904-3
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20210904
Time               10.48 h
INSTRUM            spect
PROBHD   Z116098_0653 (
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                   68
DS                    4
SWH           24038.461 Hz
FIDRES         0.733596 Hz
AQ            1.3631488 sec
RG               198.36
DW               20.800 usec
DE                6.50 usec
TE               296.2 K
D1          2.00000000 sec
D11         0.03000000 sec
TD0                   1
SFO1        100.6228298 MHz
NUC1                13C
P1               10.00 usec
PLW1        75.84400177 W
SFO2        400.1316005 MHz
NUC2                1H
CPDPRG[2         waltz16
PCPD2            90.00 usec
PLW2        16.43099976 W
PLW12        0.20286000 W
PLW13        0.10204000 W

F2 - Processing parameters
SI                32768
SF          100.6127594 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```

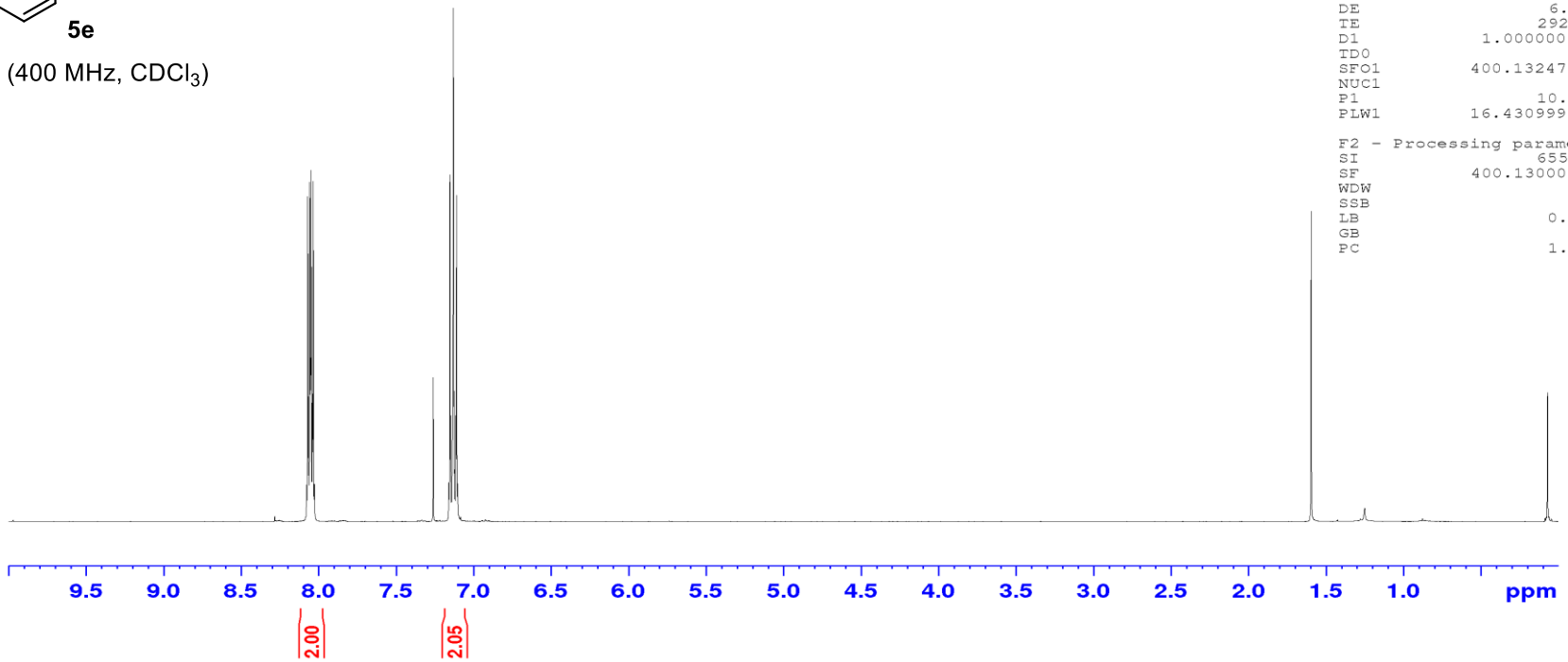


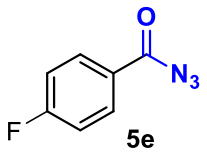


5e

^{1}H NMR (400 MHz, $CDCl_3$)

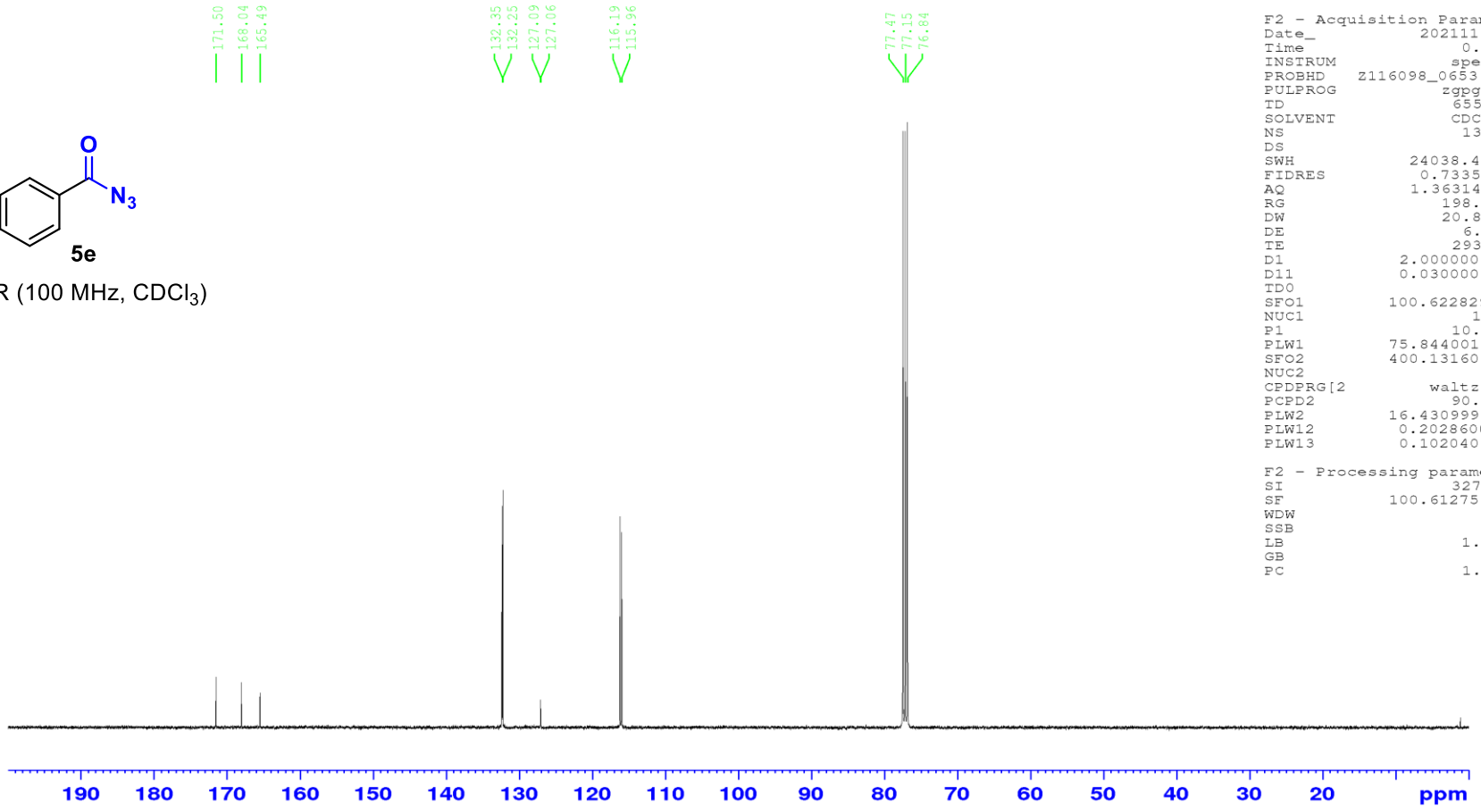
```
Current Data Parameters
NAME      1hz-20211110-4
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20211110
Time              23.26 h
INSTRUM             spect
PROBHD    Z116098_0653 (
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                     8
DS                     2
SWH           8012.820 Hz
FIDRES        0.244532 Hz
AQ            4.0894465 sec
RG               103.14
DW               62.400 usec
DE                 6.50 usec
TE                292.9 K
D1           1.00000000 sec
TD0                    1
SFO1        400.1324708 MHz
NUC1                  1H
P1                10.00 usec
PLW1        16.43099976 W

F2 - Processing parameters
SI                 65536
SF          400.1300098 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```



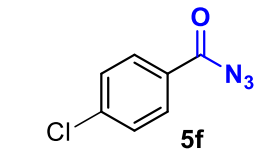


5e

^{13}C NMR (100 MHz, $CDCl_3$)

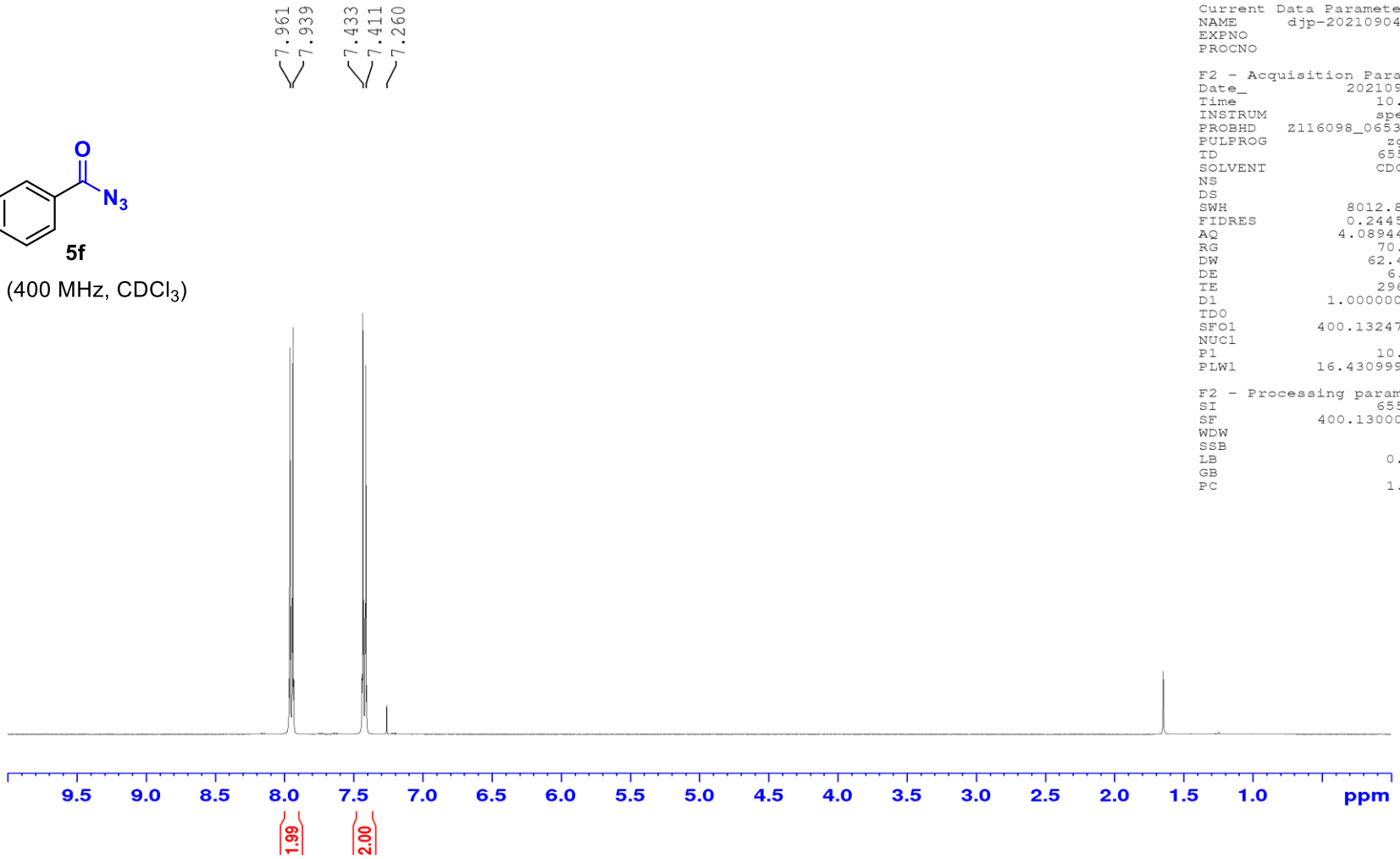



```
Current Data Parameters
NAME      lhz-20211110-4
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20211111
Time                0.43 h
INSTRUM            spect
PROBHD    Z116098_0653 (
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                 1333
DS                    4
SWH           24038.461 Hz
FIDRES         0.733596 Hz
AQ             1.3631488 sec
RG                198.36
DW                20.800 usec
DE                 6.50 usec
TE                293.5 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1
SFO1        100.6228298 MHz
NUC1               13C
P1                10.00 usec
PLW1        75.84400177 W
SFO2        400.1316005 MHz
NUC2               1H
CPDPRG[2       waltz16
PCPD2             90.00 usec
PLW2        16.43099976 W
PLW12        0.20286000 W
PLW13        0.10204000 W

F2 - Processing parameters
SI                32768
SF          100.6127571 MHz
WDW                EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```

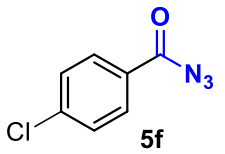


5f

^{1}H NMR (400 MHz, $CDCl_3$)

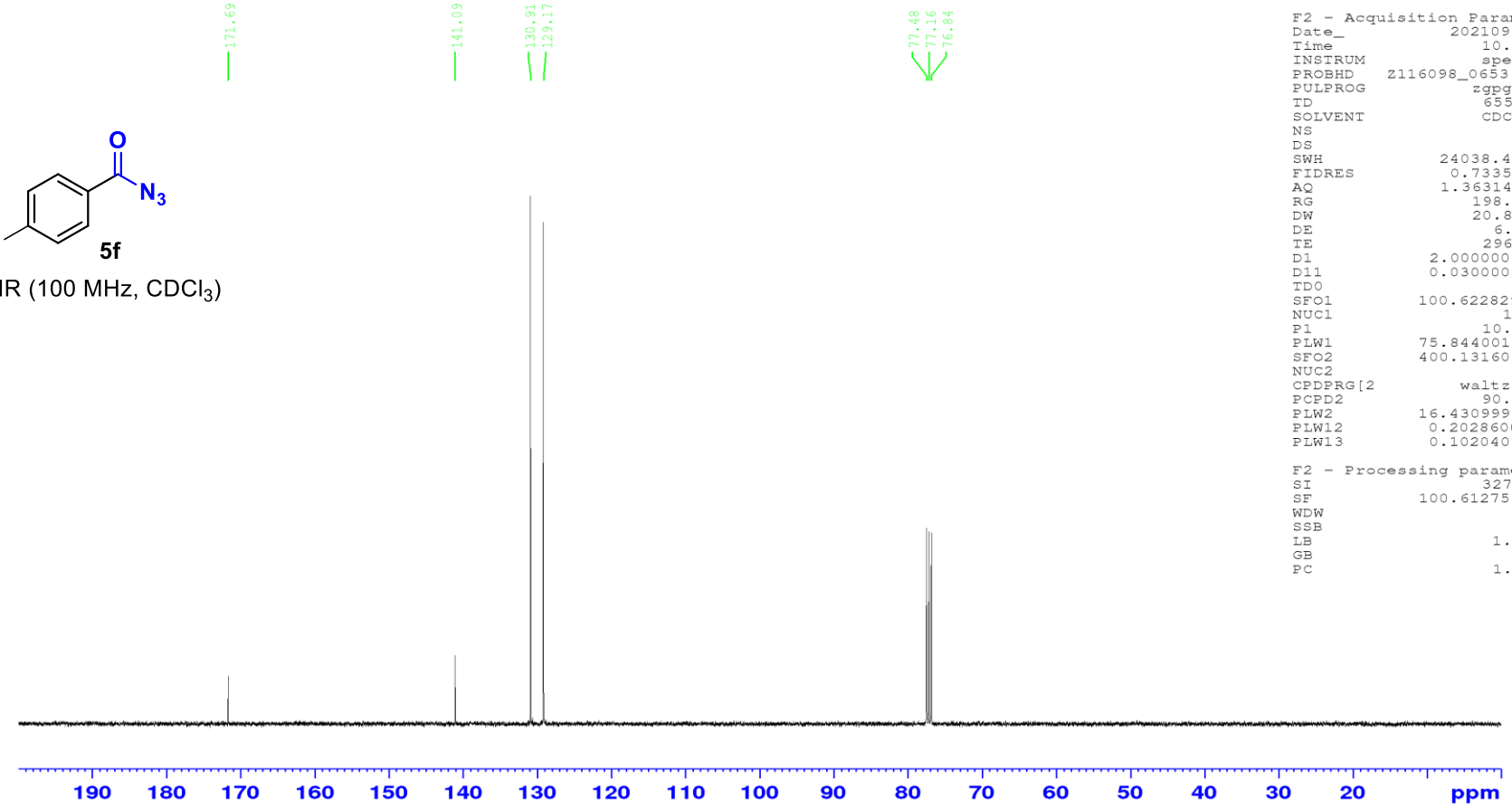



```
Current Data Parameters
NAME      djp-20210904-2
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20210904
Time              10.33 h
INSTRUM           spect
PROBHD    Z116098_0653 (
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                     8
DS                     2
SWH             8012.820 Hz
FIDRES          0.244532 Hz
AQ              4.0894465 sec
RG                 70.89
DW                62.400 usec
DE                 6.50 usec
TE                296.0 K
D1             1.00000000 sec
TD0                    1
SFO1          400.1324708 MHz
NUC1                  1H
P1                10.00 usec
PLW1          16.43099976 W

F2 - Processing parameters
SI                 65536
SF            400.1300096 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```

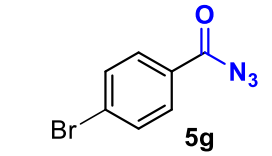


5f

^{13}C NMR (100 MHz, $CDCl_3$)

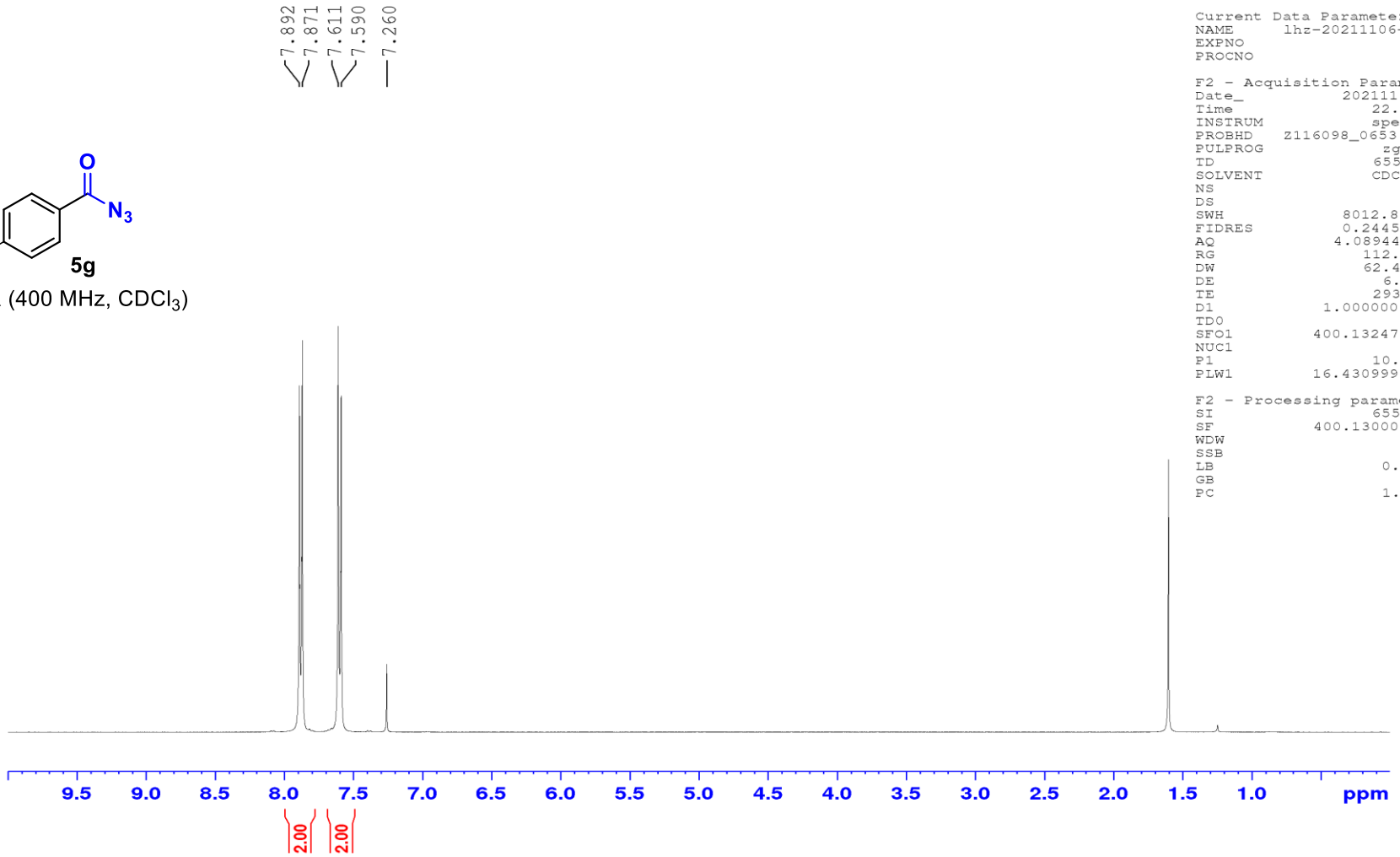



```
Current Data Parameters
NAME      djp-20210904-2
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20210904
Time               10.39 h
INSTRUM            spect
PROBHD    Z116098_0653 (
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                   90
DS                    4
SWH           24038.461 Hz
FIDRES         0.733596 Hz
AQ             1.3631488 sec
RG                198.36
DW                20.800 usec
DE                 6.50 usec
TE                296.2 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1
SFO1        100.6228298 MHz
NUC1               13C
P1                10.00 usec
PLW1        75.84400177 W
SFO2        400.1316005 MHz
NUC2               1H
CPDPRG[2       waltz16
PCPD2             90.00 usec
PLW2        16.43099976 W
PLW12        0.20286000 W
PLW13        0.10204000 W

F2 - Processing parameters
SI                32768
SF          100.6127579 MHz
WDW                EM
SSB                0
LB              1.00 Hz
GB                 0
PC              1.40
```

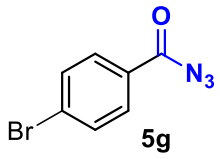


5g

^{1}H NMR (400 MHz, $CDCl_3$)

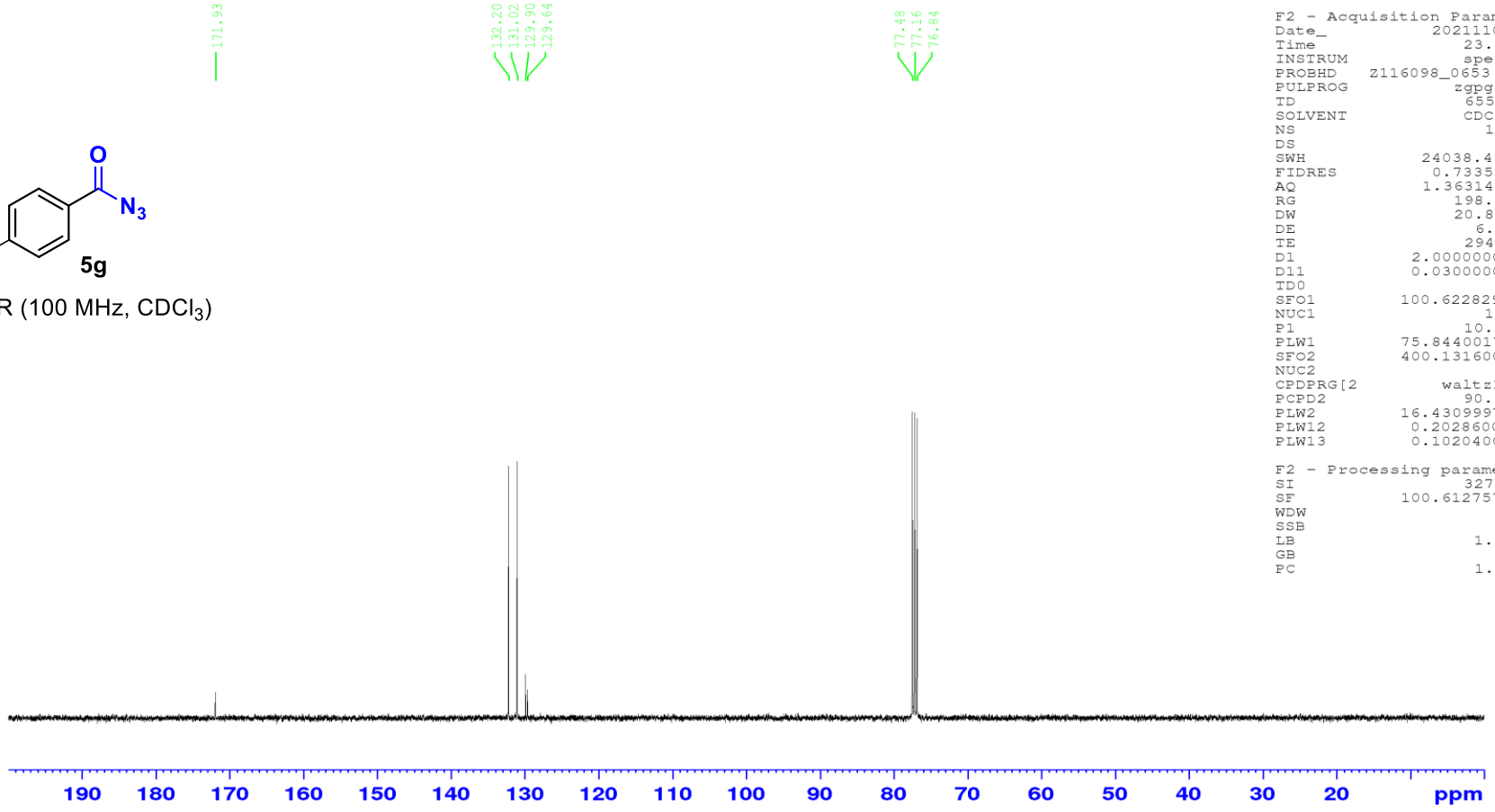



```
Current Data Parameters
NAME      lhz-20211106-1
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20211106
Time               22.36 h
INSTRUM            spect
PROBHD    Z116098_0653 (
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                     8
DS                     2
SWH             8012.820 Hz
FIDRES          0.244532 Hz
AQ              4.0894465 sec
RG                112.15
DW                62.400 usec
DE                 6.50 usec
TE                293.0 K
D1             1.00000000 sec
TD0                    1
SFO1          400.1324708 MHz
NUC1                  1H
P1                 10.00 usec
PLW1          16.43099976 W

F2 - Processing parameters
SI                 65536
SF            400.1300098 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```

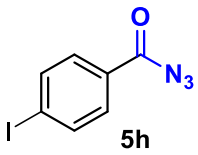


5g

^{13}C NMR (100 MHz, $CDCl_3$)

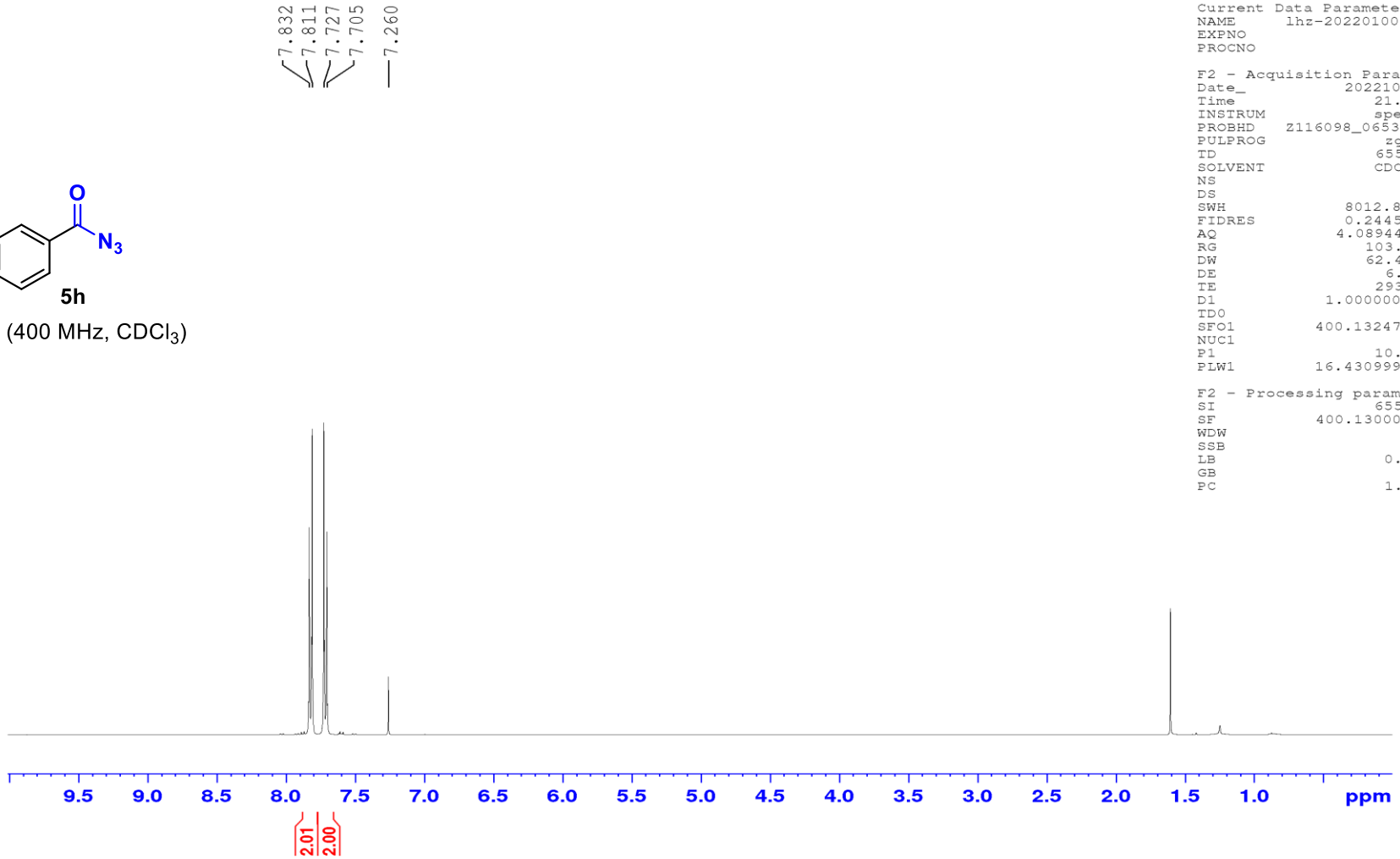



```
Current Data Parameters
NAME      lhz-20211106-1
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20211106
Time              23.15 h
INSTRUM           spect
PROBHD    Z116098_0653 (
PULPROG         zgpg30
TD               65536
SOLVENT          CDCl3
NS                 133
DS                   4
SWH          24038.461 Hz
FIDRES        0.733596 Hz
AQ            1.3631488 sec
RG               198.36
DW               20.800 usec
DE                6.50 usec
TE                294.1 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                  1
SFO1       100.6228298 MHz
NUC1               13C
P1               10.00 usec
PLW1       75.84400177 W
SFO2       400.1316005 MHz
NUC2               1H
CPDPRG[2       waltz16
PCPD2            90.00 usec
PLW2       16.43099976 W
PLW12       0.20286000 W
PLW13       0.10204000 W

F2 - Processing parameters
SI               32768
SF         100.6127572 MHz
WDW                EM
SSB                 0
LB               1.00 Hz
GB                  0
PC               1.40
```

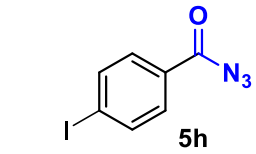


5h

^{1}H NMR (400 MHz, $CDCl_3$)

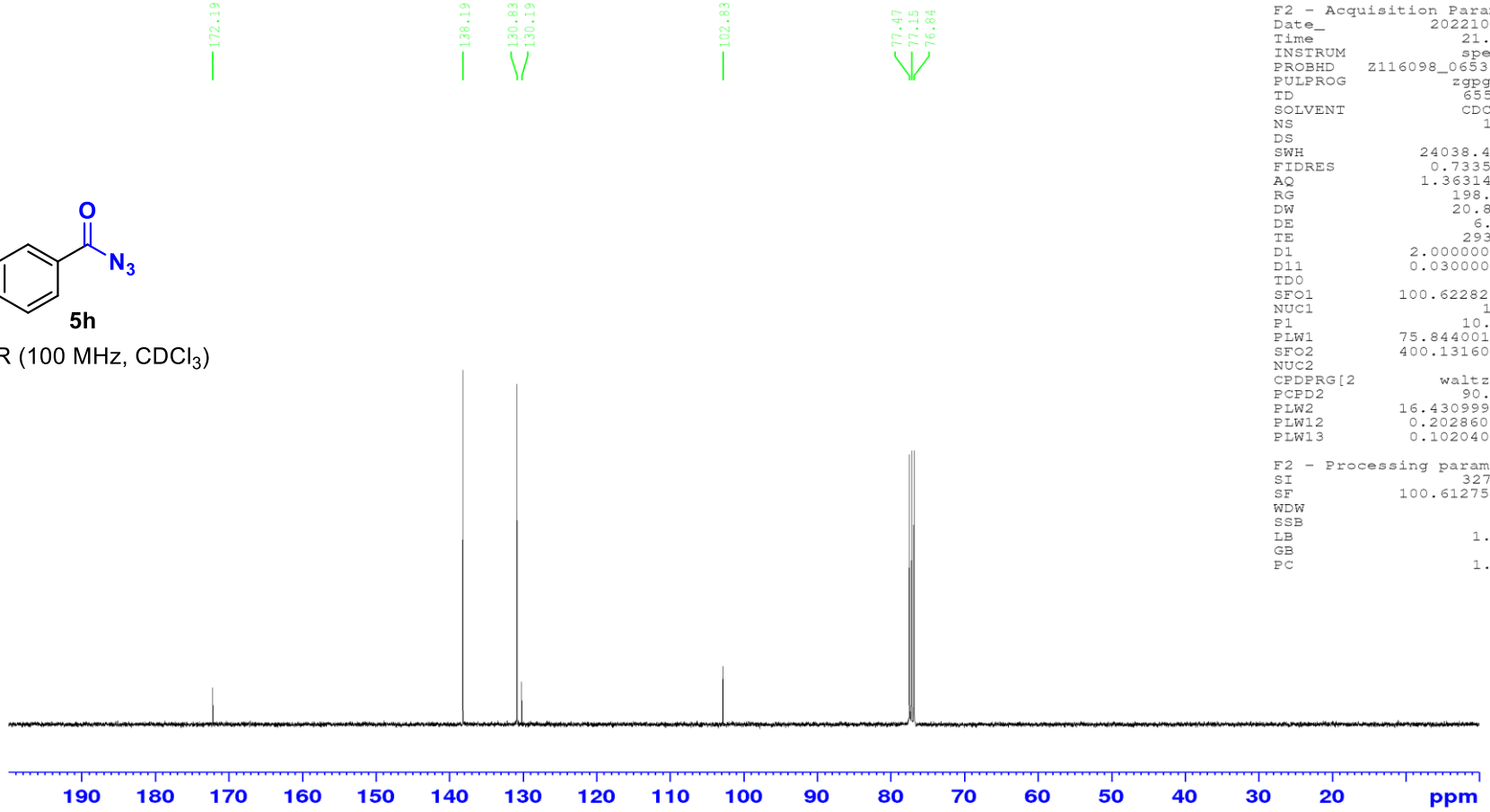



```
Current Data Parameters
NAME      lhz-202201005-1
EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
Date_            20221005
Time                21.28 h
INSTRUM             spect
PROBHD    Z116098_0653 (
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             8012.820 Hz
FIDRES          0.244532 Hz
AQ              4.0894465 sec
RG                 103.14
DW                 62.400 usec
DE                   6.50 usec
TE                  293.4 K
D1            1.00000000 sec
TD0                    1
SFO1          400.1324708 MHz
NUC1                  1H
P1                 10.00 usec
PLW1          16.43099976 W

F2 - Processing parameters
SI                 65536
SF            400.1300099 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```

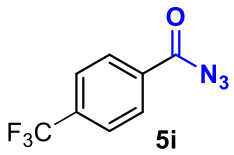


5h

^{13}C NMR (100 MHz, $CDCl_3$)




```
Current Data Parameters
NAME      lhz-202201005-1
EXPNO                   2
PROCNO                  1

F2 - Acquisition Parameters
Date_             20221005
Time                21.40 h
INSTRUM             spect
PROBHD    Z116098_0653 (
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                   188
DS                     4
SWH            24038.461 Hz
FIDRES          0.733596 Hz
AQ             1.3631488 sec
RG                198.36
DW                20.800 usec
DE                 6.50 usec
TE                293.7 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                    1
SFO1          100.6228298 MHz
NUC1                 13C
P1                 10.00 usec
PLW1          75.84400177 W
SFO2          400.1316005 MHz
NUC2                 1H
CPDPRG[2         waltz16
PCPD2              90.00 usec
PLW2          16.43099976 W
PLW12          0.20286000 W
PLW13          0.10204000 W

F2 - Processing parameters
SI                 32768
SF            100.6127586 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```

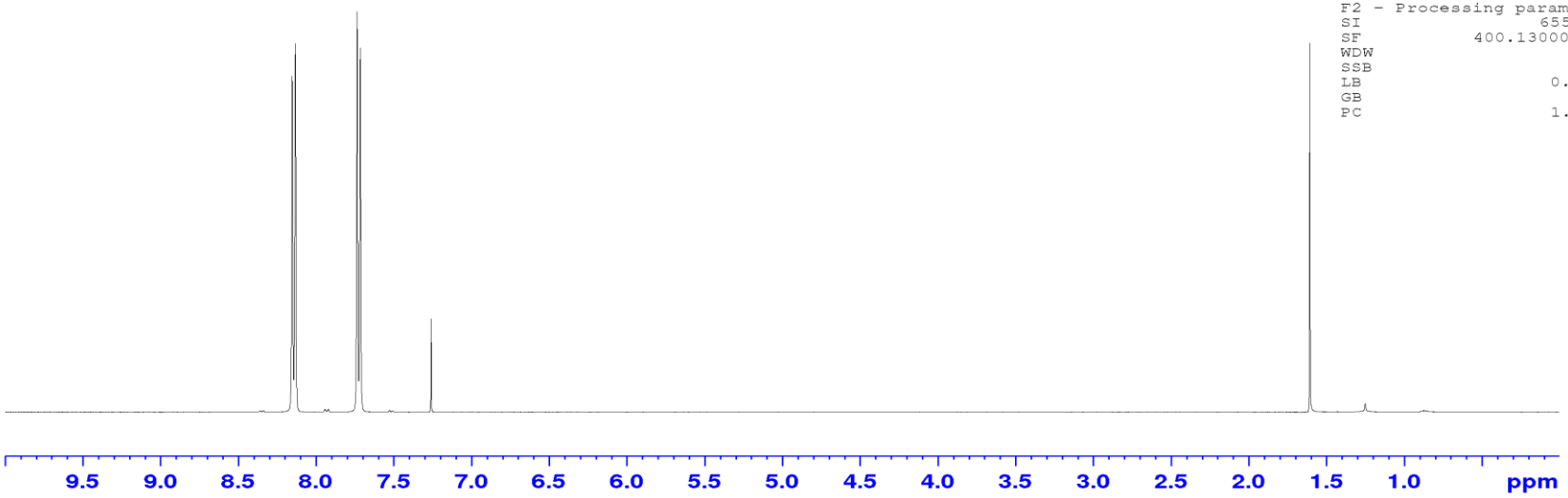


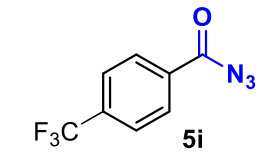
5i

^{1}H NMR (400 MHz, $CDCl_3$)

8.154
8.133
7.736
7.715
7.260

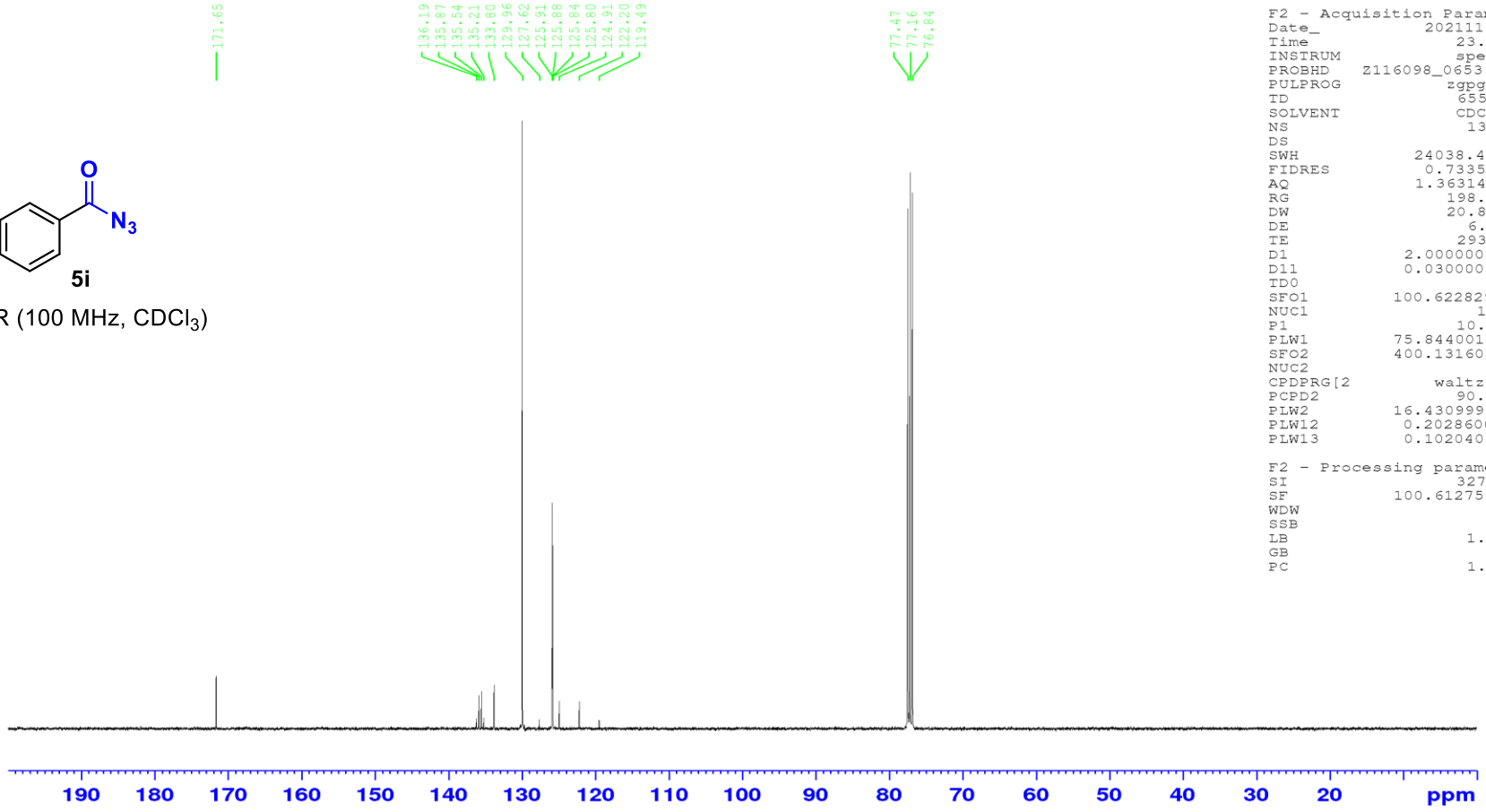
```
Current Data Parameters
NAME      1hz-20211117-3
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20211117
Time              22.11 h
INSTRUM            spect
PROBHD    Z116098_0653 (
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                     8
DS                     2
SWH             8012.820 Hz
FIDRES          0.244532 Hz
AQ              4.0894465 sec
RG                 143.4
DW                62.400 usec
DE                 6.50 usec
TE                292.4 K
D1             1.00000000 sec
TD0                    1
SFO1          400.1324708 MHz
NUC1                  1H
P1                10.00 usec
PLW1          16.43099976 W

F2 - Processing parameters
SI                 65536
SF            400.1300098 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```



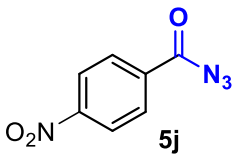


5i

^{13}C NMR (100 MHz, $CDCl_3$)




```
Current Data Parameters
NAME      1hz-20211117-3
EXPNO                   2
PROCNO                  1

F2 - Acquisition Parameters
Date_           20211117
Time              23.28 h
INSTRUM             spect
PROBHD   Z116098_0653 (
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                 1333
DS                    4
SWH           24038.461 Hz
FIDRES         0.733596 Hz
AQ             1.3631488 sec
RG                198.36
DW                20.800 usec
DE                 6.50 usec
TE                293.0 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1
SFO1        100.6228298 MHz
NUC1               13C
P1               10.00 usec
PLW1        75.84400177 W
SFO2        400.1316005 MHz
NUC2               1H
CPDPRG[2       waltz16
PCPD2            90.00 usec
PLW2        16.43099976 W
PLW12        0.20286000 W
PLW13        0.10204000 W

F2 - Processing parameters
SI                32768
SF          100.6127564 MHz
WDW                EM
SSB                 0
LB               1.00 Hz
GB                  0
PC               1.40
```

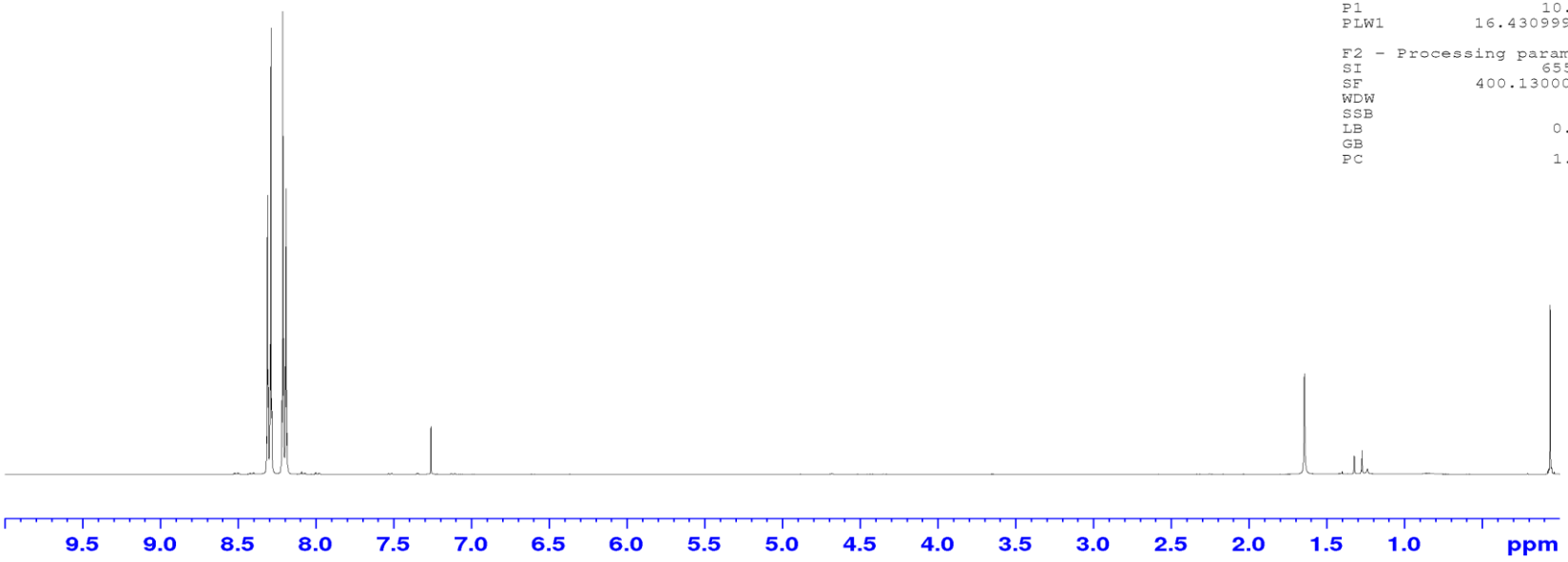


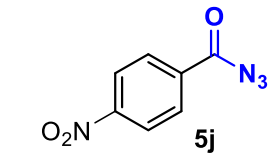
5j

^{1}H NMR (400 MHz, $CDCl_3$)

8.311
8.289
8.213
8.191
7.260

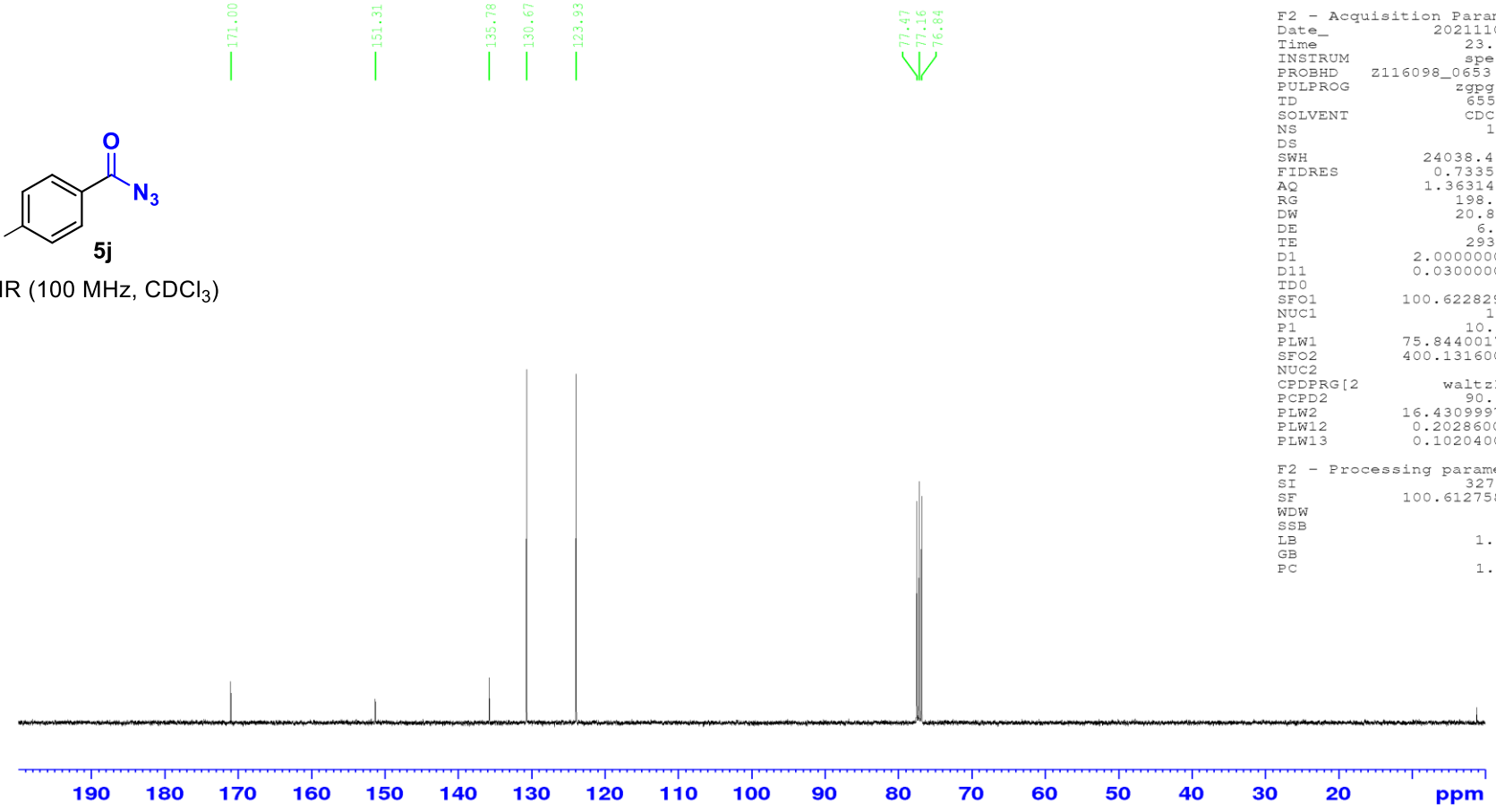
```
Current Data Parameters
NAME      1hz-20211106-2
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20211106
Time               22.40 h
INSTRUM            spect
PROBHD    Z116098_0653 (
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                    8
DS                    2
SWH            8012.820 Hz
FIDRES            0.244532 Hz
AQ             4.0894465 sec
RG                127.66
DW                62.400 usec
DE                 6.50 usec
TE                293.2 K
D1             1.00000000 sec
TD0                   1
SFO1         400.1324708 MHz
NUC1                  1H
P1                10.00 usec
PLW1         16.43099976 W

F2 - Processing parameters
SI                65536
SF           400.1300098 MHz
WDW                   EM
SSB                   0
LB                 0.30 Hz
GB                    0
PC                 1.00
```





5j

^{13}C NMR (100 MHz, $CDCl_3$)




```
Current Data Parameters
NAME      lhz-20211106-2
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20211106
Time              23.26 h
INSTRUM             spect
PROBHD    Z116098_0653 (
PULPROG           zgpg30
TD                65536
SOLVENT             CDCl3
NS                    133
DS                      4
SWH           24038.461 Hz
FIDRES         0.733596 Hz
AQ            1.3631488 sec
RG                198.36
DW                20.800 usec
DE                 6.50 usec
TE                293.9 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                    1
SFO1        100.6228298 MHz
NUC1                13C
P1                10.00 usec
PLW1        75.84400177 W
SFO2        400.1316005 MHz
NUC2                1H
CPDPRG[2         waltz16
PCPD2             90.00 usec
PLW2        16.43099976 W
PLW12        0.20286000 W
PLW13        0.10204000 W

F2 - Processing parameters
SI                 32768
SF          100.6127586 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
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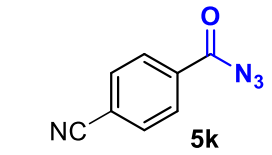
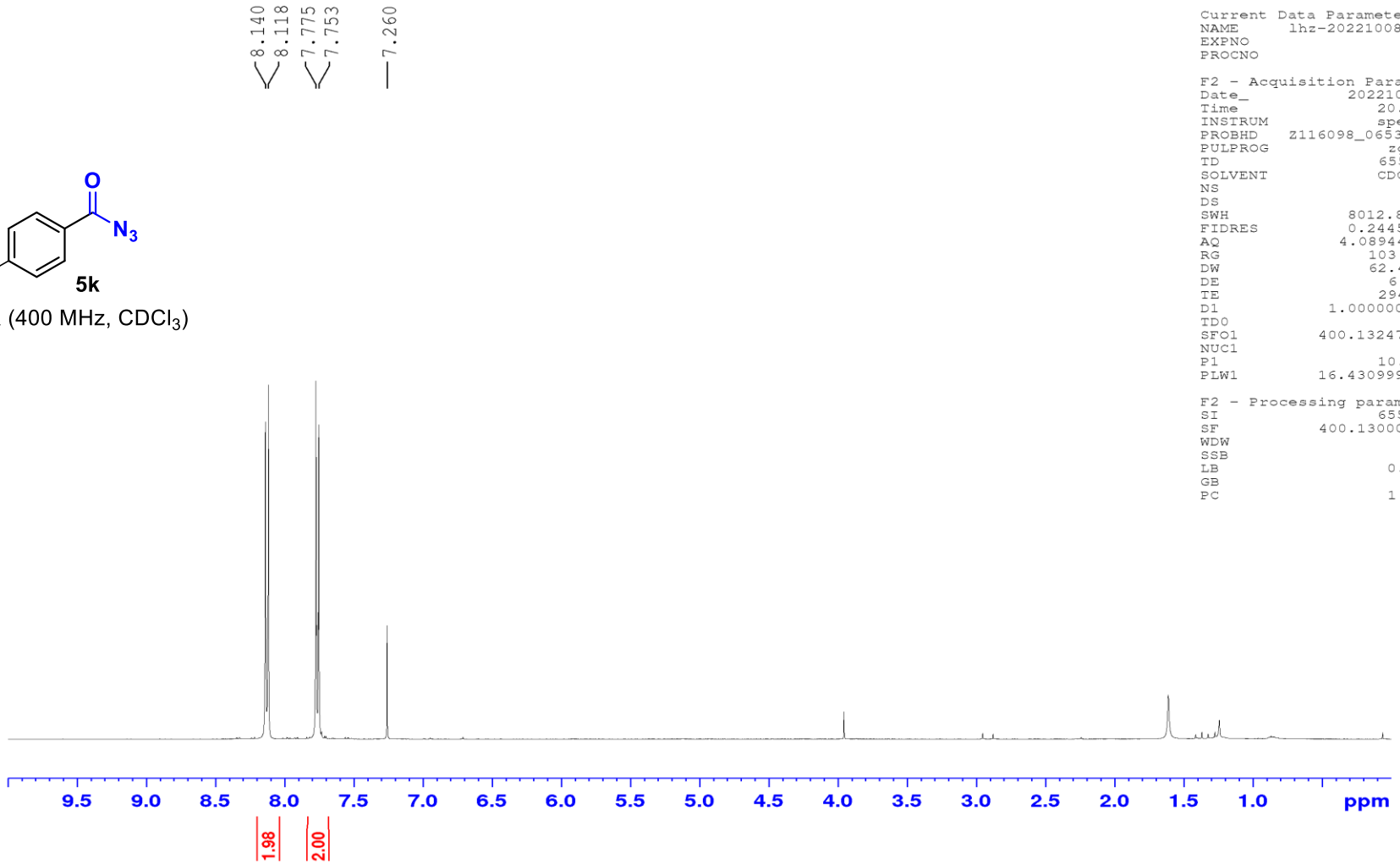


5k

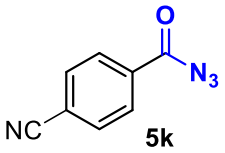
^{1}H NMR (400 MHz, $CDCl_3$)




```
Current Data Parameters
NAME      lhz-20221008-1
EXPNO                  1
PROCNO                 1

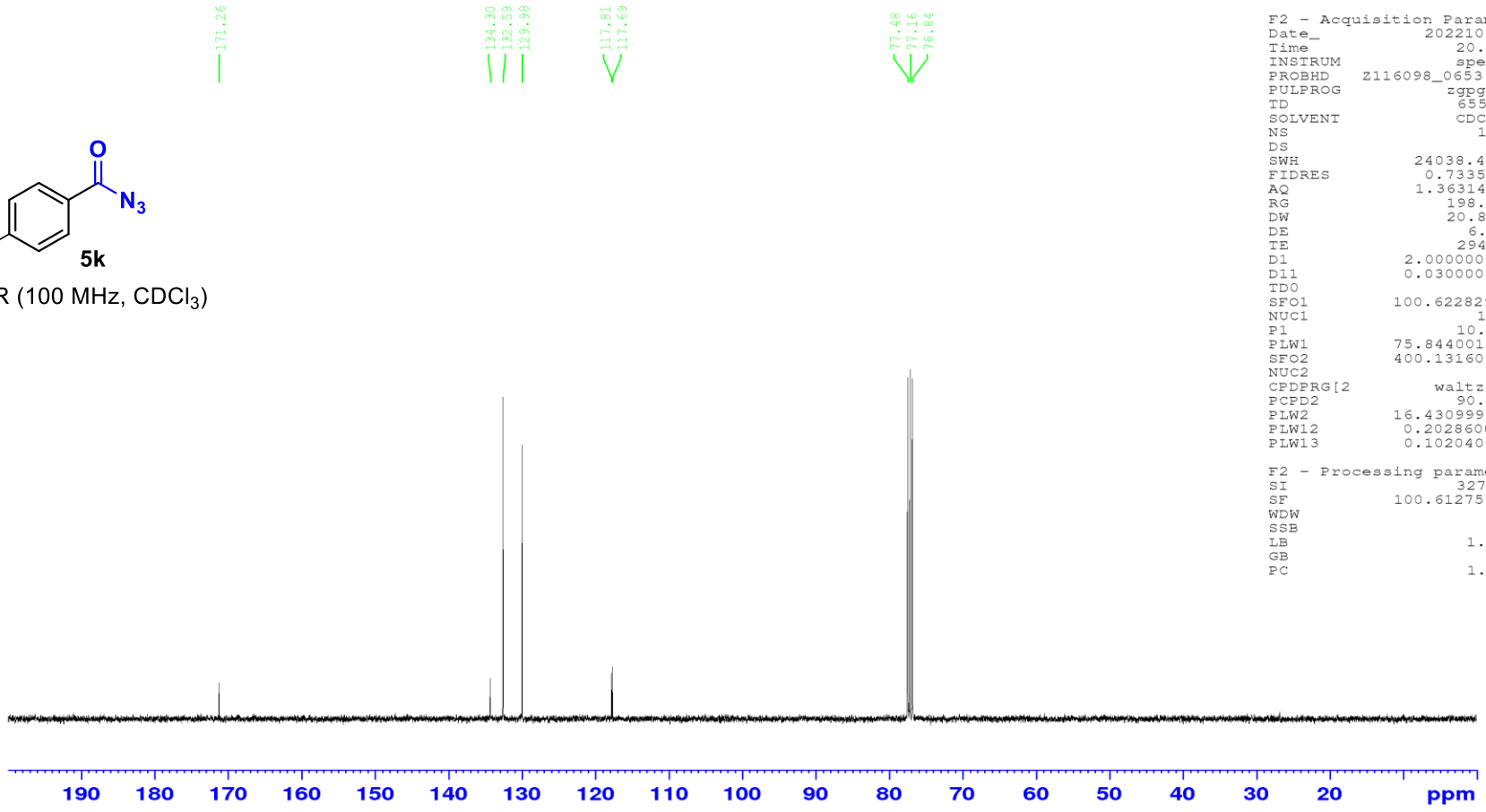
F2 - Acquisition Parameters
Date_           20221008
Time               20.34 h
INSTRUM            spect
PROBHD    Z116098_0653 (
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                   16
DS                    2
SWH             8012.820 Hz
FIDRES          0.244532 Hz
AQ              4.0894465 sec
RG                103.14
DW                62.400 usec
DE                 6.50 usec
TE                294.3 K
D1             1.00000000 sec
TD0                   1
SFO1          400.1324708 MHz
NUC1                 1H
P1                10.00 usec
PLW1          16.43099976 W

F2 - Processing parameters
SI                65536
SF          400.1300096 MHz
WDW                  EM
SSB                   0
LB                 0.30 Hz
GB                    0
PC                 1.00
```



5k

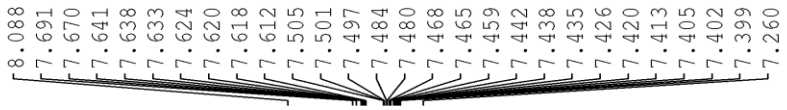
^{13}C NMR (100 MHz, $CDCl_3$)

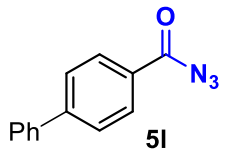



```
Current Data Parameters
NAME      1hz-20221008-1
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20221008
Time               20.52 h
INSTRUM            spect
PROBHD    Z116098_0653 (
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  139
DS                    4
SWH           24038.461 Hz
FIDRES         0.733596 Hz
AQ             1.3631488 sec
RG                198.36
DW                20.800 usec
DE                 6.50 usec
TE                294.8 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                   1
SFO1        100.6228298 MHz
NUC1               13C
P1                10.00 usec
PLW1        75.84400177 W
SFO2        400.1316005 MHz
NUC2               1H
CPDPRG[2        waltz16
PCPD2             90.00 usec
PLW2        16.43099976 W
PLW12        0.20286000 W
PLW13        0.10204000 W

F2 - Processing parameters
SI                32768
SF          100.6127579 MHz
WDW                EM
SSB                 0
LB              1.00 Hz
GB                  0
PC              1.40
```





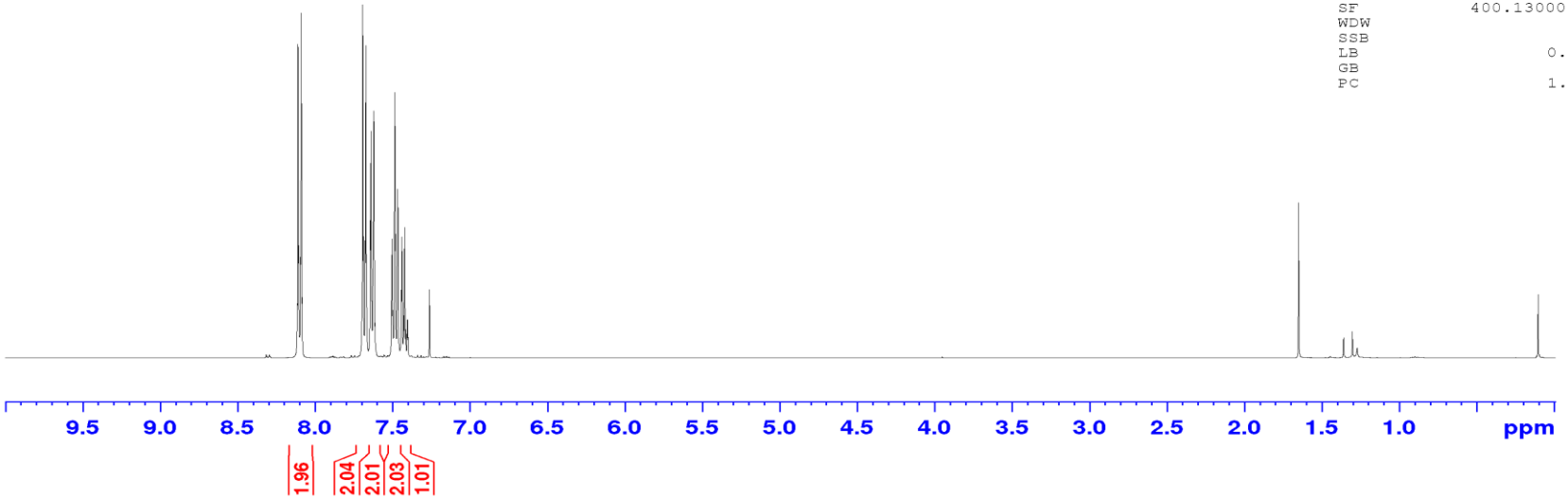
5l

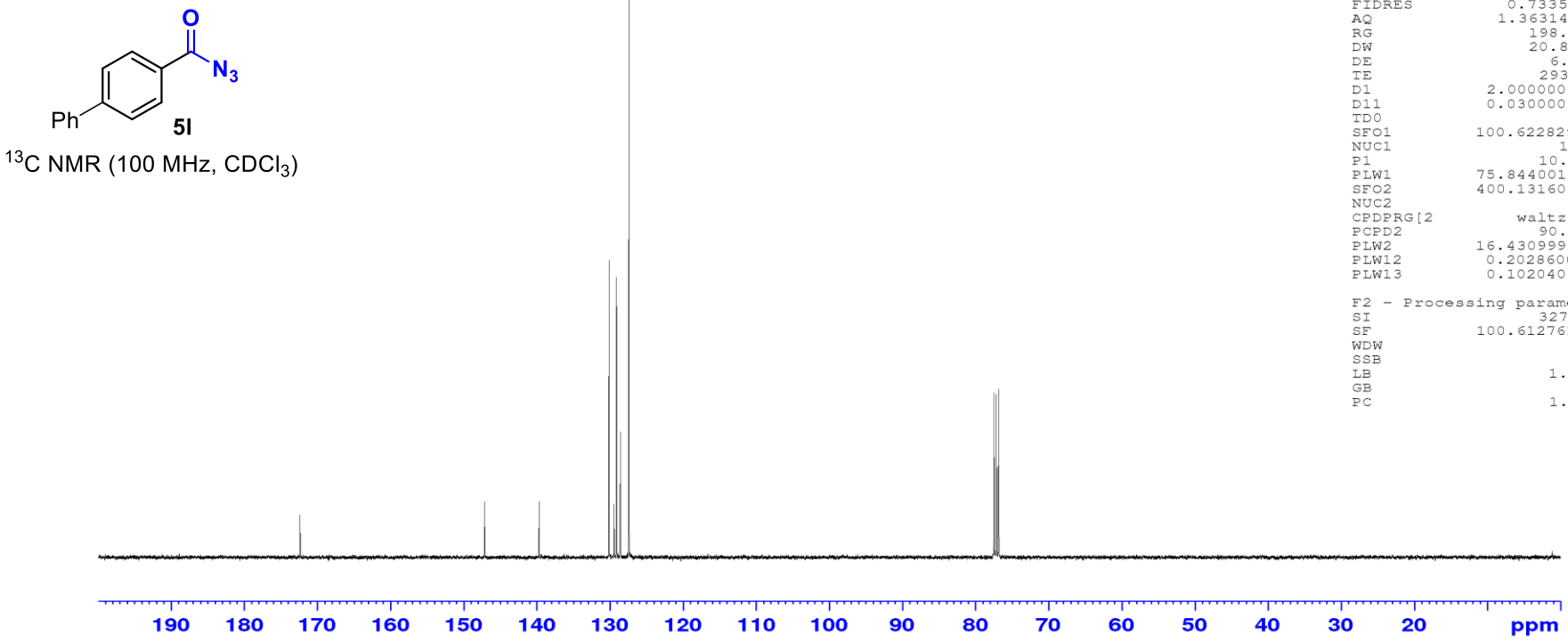
^{1}H NMR (400 MHz, $CDCl_3$)

```
Current Data Parameters
NAME      lhz-20221004-5
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20221004
Time              23.16 h
INSTRUM             spect
PROBHD    Z116098_0653 (
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                     8
DS                     2
SWH             8012.820 Hz
FIDRES          0.244532 Hz
AQ              4.0894465 sec
RG                 55.99
DW                62.400 usec
DE                 6.50 usec
TE                293.1 K
D1             1.00000000 sec
TD0                    1
SFO1          400.1324708 MHz
NUC1                  1H
P1                10.00 usec
PLW1          16.43099976 W

F2 - Processing parameters
SI                 65536
SF            400.1300098 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```

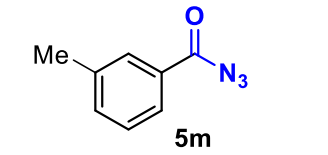





```
Current Data Parameters
NAME      1hz-20221004-5
EXPNO                  2
PROCNO                 1

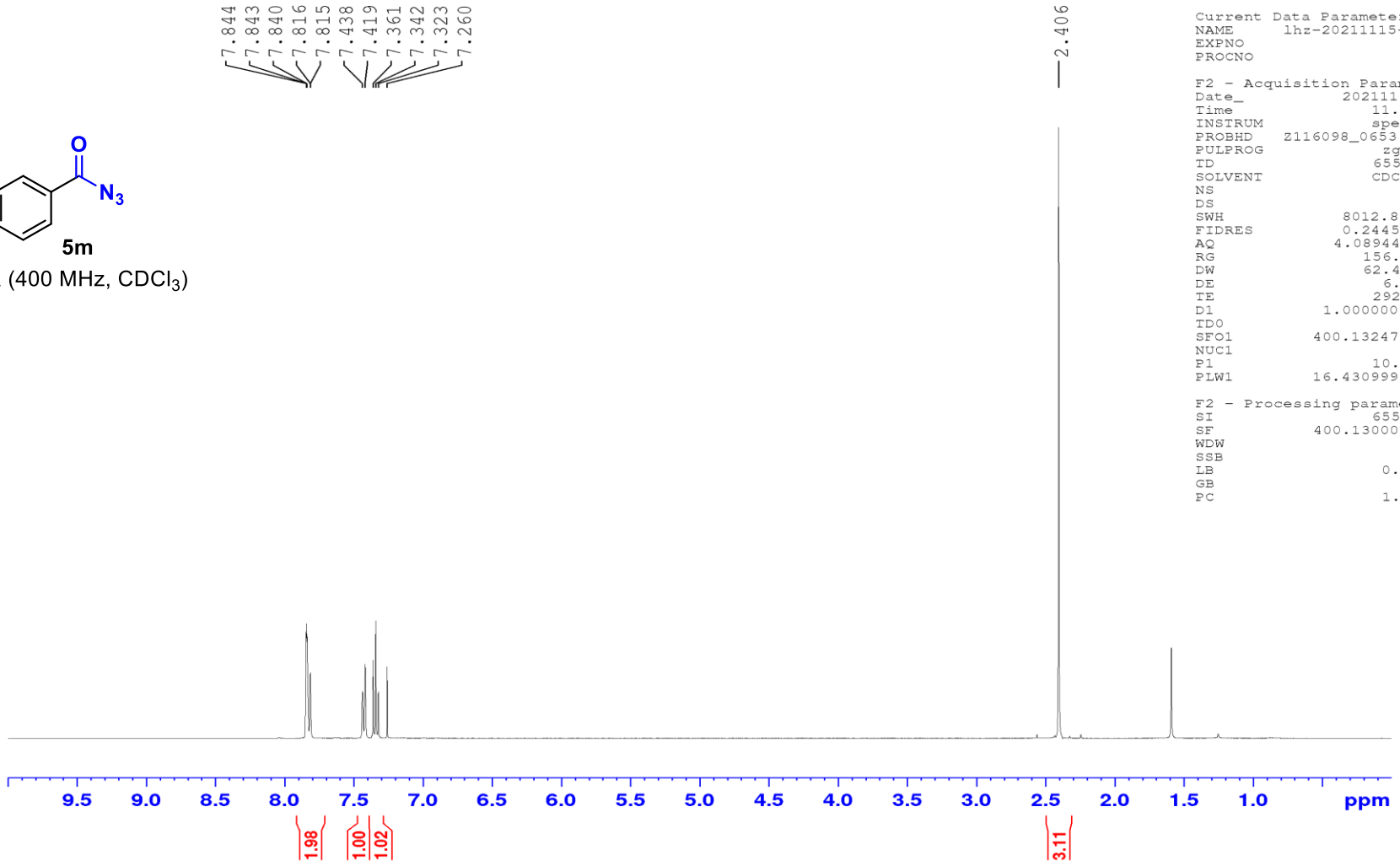
F2 - Acquisition Parameters
Date_           20221004
Time              23.22 h
INSTRUM             spect
PROBHD    Z116098_0653 (
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                   88
DS                    4
SWH           24038.461 Hz
FIDRES         0.733596 Hz
AQ             1.3631488 sec
RG                198.36
DW                20.800 usec
DE                 6.50 usec
TE                293.5 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1
SFO1        100.6228298 MHz
NUC1                13C
P1                10.00 usec
PLW1        75.84400177 W
SFO2        400.1316005 MHz
NUC2                1H
CPDPRG[2        waltz16
PCPD2             90.00 usec
PLW2        16.43099976 W
PLW12        0.20286000 W
PLW13        0.10204000 W

F2 - Processing parameters
SI                32768
SF          100.6127615 MHz
WDW                EM
SSB                 0
LB               1.00 Hz
GB                  0
PC                1.40
```



5m

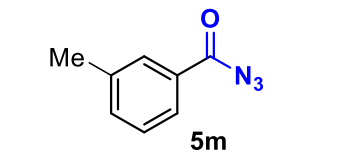
^{1}H NMR (400 MHz, $CDCl_3$)




```
Current Data Parameters
NAME      1hz-20211115-4
EXPNO                  1
PROCNO                 1

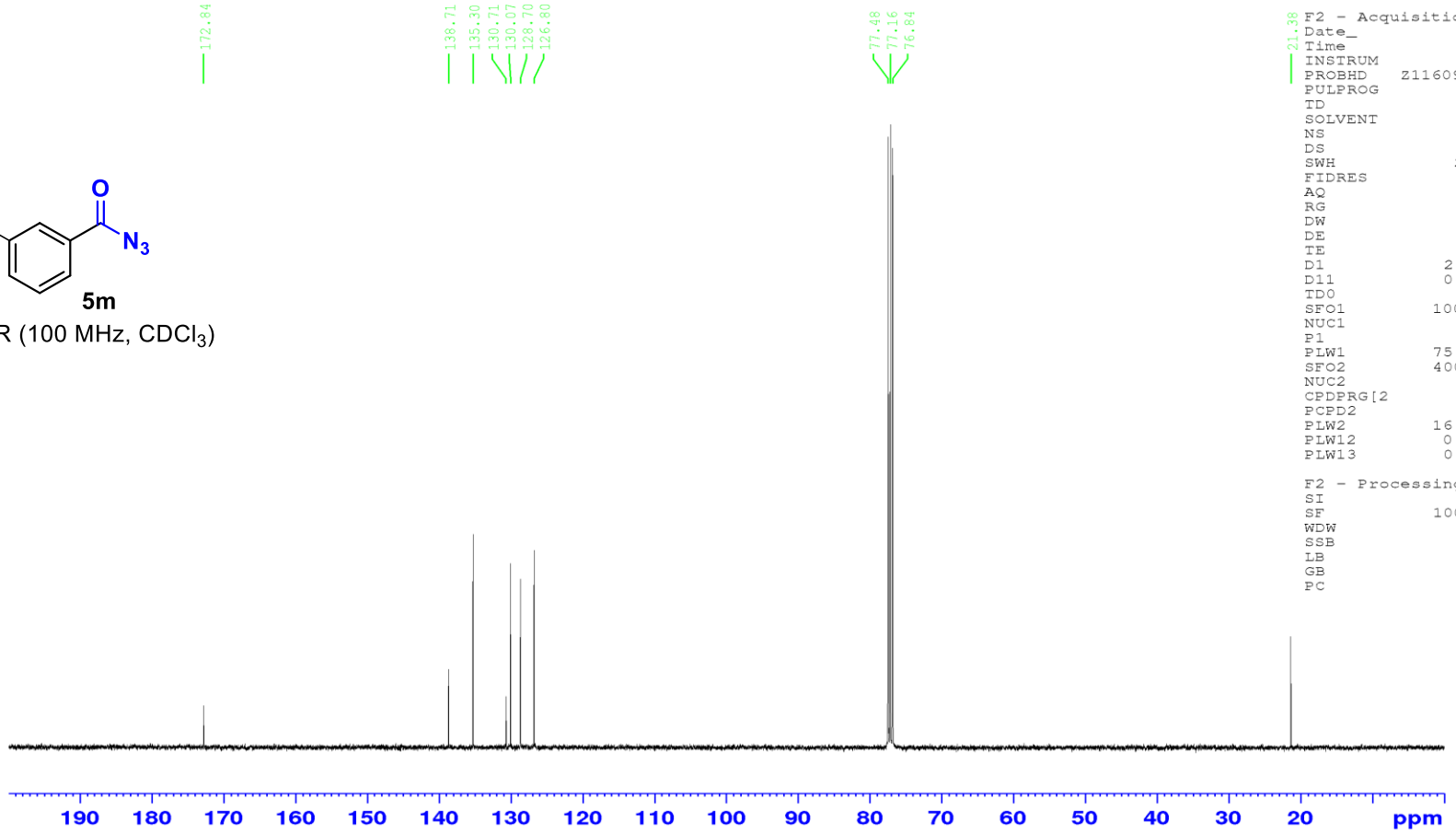
F2 - Acquisition Parameters
Date_           20211115
Time              11.22 h
INSTRUM           spect
PROBHD    Z116098_0653 (
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                     8
DS                     2
SWH            8012.820 Hz
FIDRES         0.244532 Hz
AQ             4.0894465 sec
RG                156.98
DW                62.400 usec
DE                 6.50 usec
TE                292.7 K
D1             1.00000000 sec
TD0                    1
SFO1         400.1324708 MHz
NUC1                1H
P1                10.00 usec
PLW1         16.43099976 W

F2 - Processing parameters
SI                 65536
SF           400.1300098 MHz
WDW                   EM
SSB                    0
LB                0.30 Hz
GB                     0
PC                1.00
```



5m

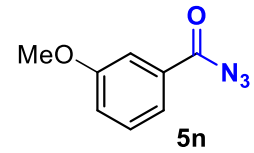
^{13}C NMR (100 MHz, $CDCl_3$)




```
Current Data Parameters
NAME      1hz-20211115-4
EXPNO                   2
PROCNO                  1

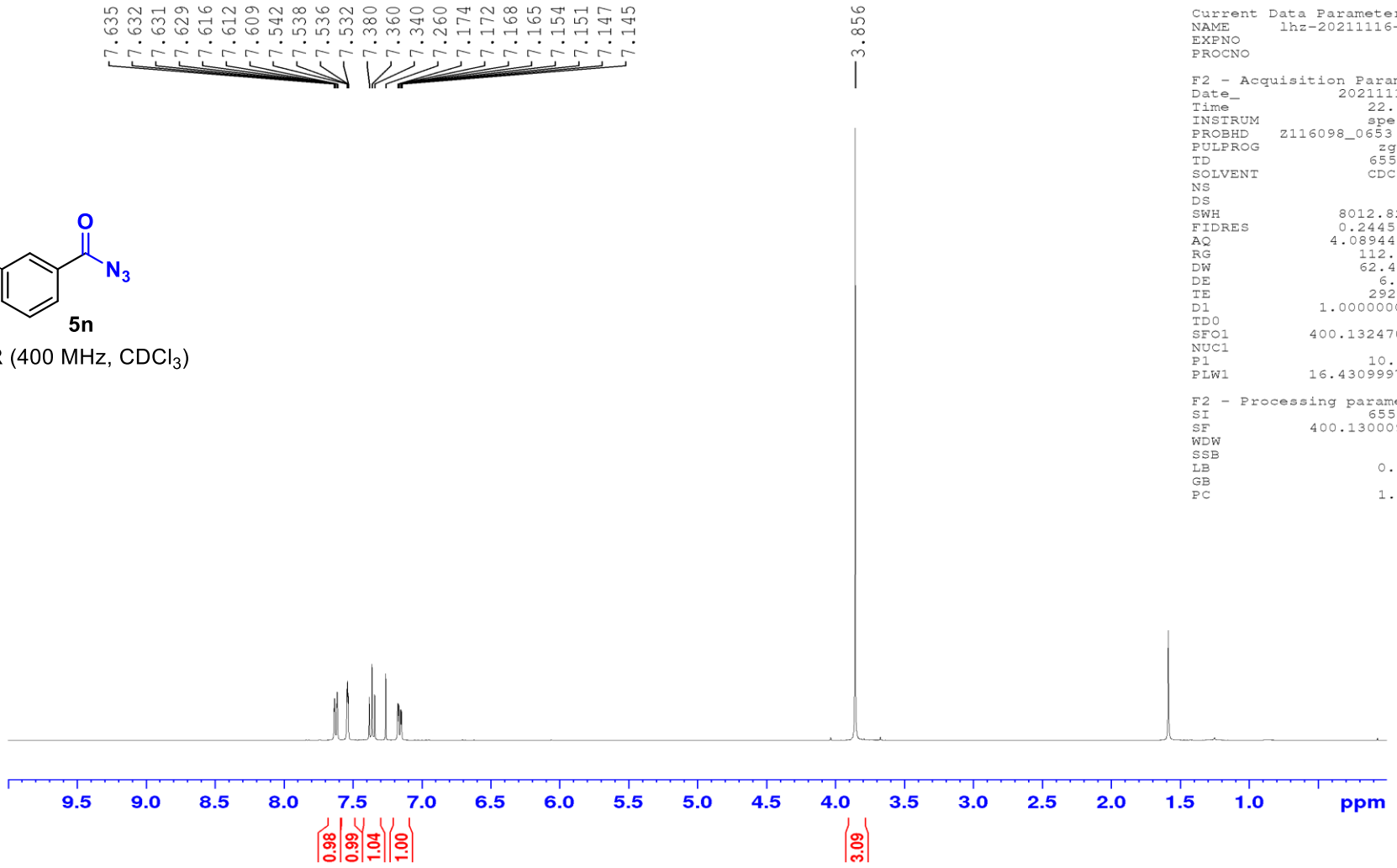
F2 - Acquisition Parameters
Date_            20211115
Time                15.32 h
INSTRUM             spect
PROBHD    Z116098_0653 (
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  588
DS                    4
SWH           24038.461 Hz
FIDRES         0.733596 Hz
AQ             1.3631488 sec
RG                198.36
DW                20.800 usec
DE                 6.50 usec
TE                293.2 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                   1
SFO1         100.6228298 MHz
NUC1                13C
P1                10.00 usec
PLW1         75.84400177 W
SFO2         400.1316005 MHz
NUC2                1H
CPDPRG[2         waltz16
PCPD2             90.00 usec
PLW2         16.43099976 W
PLW12         0.20286000 W
PLW13         0.10204000 W

F2 - Processing parameters
SI                32768
SF           100.6127572 MHz
WDW                 EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```



5n

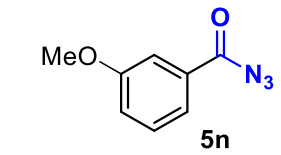
^{1}H NMR (400 MHz, $CDCl_3$)




```
Current Data Parameters
NAME      lhz-20211116-1
EXPNO                  1
PROCNO                 1

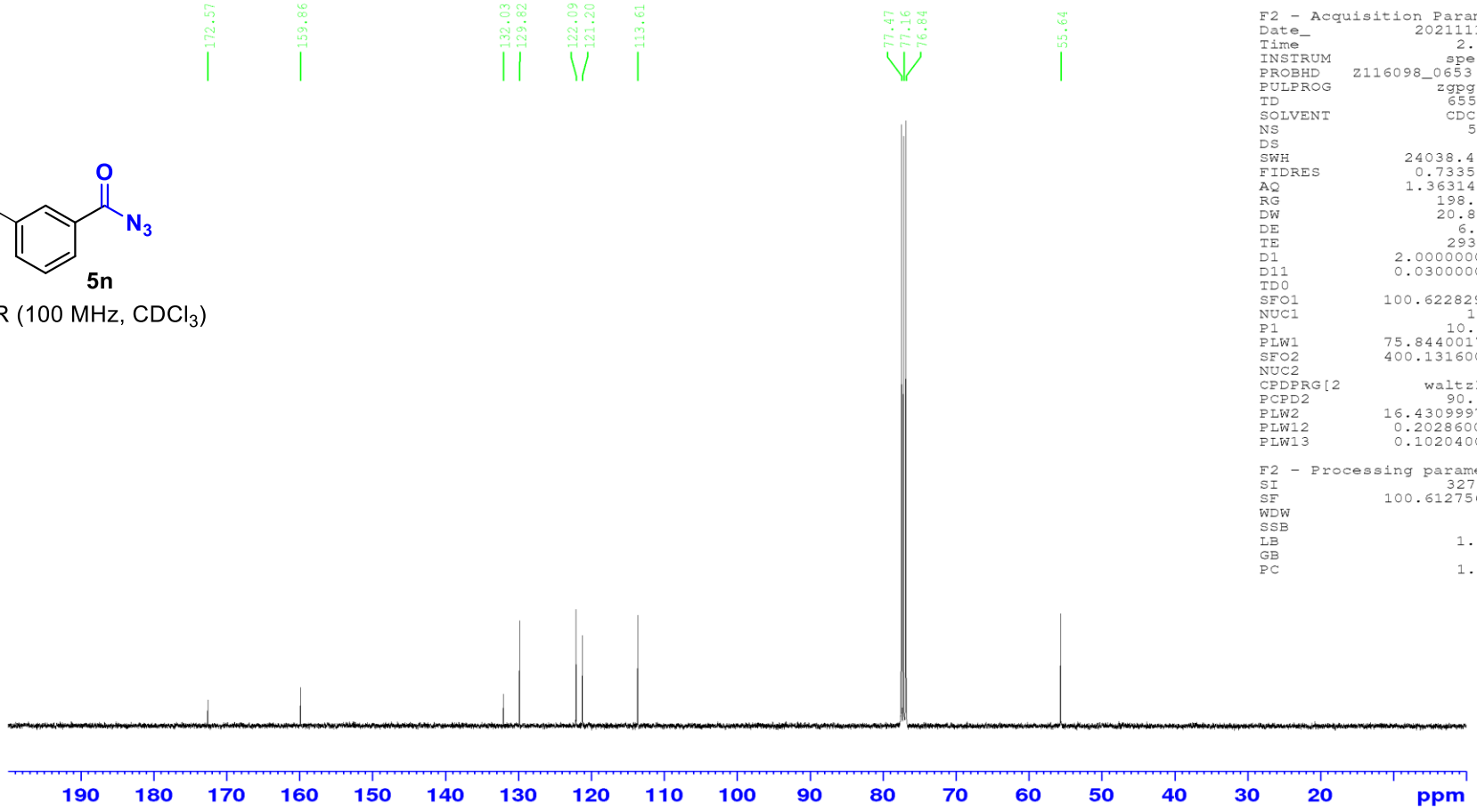
F2 - Acquisition Parameters
Date_           20211116
Time              22.30 h
INSTRUM             spect
PROBHD   Z116098_0653 (
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                     8
DS                     2
SWH             8012.820 Hz
FIDRES          0.244532 Hz
AQ              4.0894465 sec
RG                 112.15
DW                 62.400 usec
DE                   6.50 usec
TE                 292.5 K
D1              1.00000000 sec
TD0                    1
SFO1          400.1324708 MHz
NUC1                  1H
P1                 10.00 usec
PLW1          16.43099976 W

F2 - Processing parameters
SI                 65536
SF            400.1300099 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```



5n

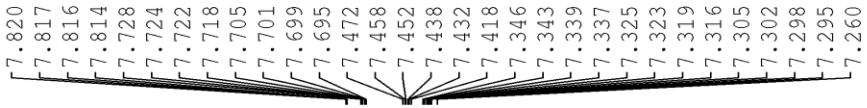
^{13}C NMR (100 MHz, $CDCl_3$)

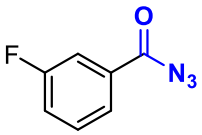



```
Current Data Parameters
NAME      lhz-20211116-1
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20211117
Time              2.17 h
INSTRUM           spect
PROBHD   Z116098_0653 (
PULPROG         zgpg30
TD               65536
SOLVENT          CDCl3
NS                 512
DS                   4
SWH          24038.461 Hz
FIDRES        0.733596 Hz
AQ           1.3631488 sec
RG              198.36
DW              20.800 usec
DE                6.50 usec
TE               293.2 K
D1          2.00000000 sec
D11         0.03000000 sec
TD0                  1
SFO1       100.6228298 MHz
NUC1               13C
P1               10.00 usec
PLW1       75.84400177 W
SFO2       400.1316005 MHz
NUC2               1H
CPDPRG[2       waltz16
PCPD2            90.00 usec
PLW2       16.43099976 W
PLW12       0.20286000 W
PLW13       0.10204000 W

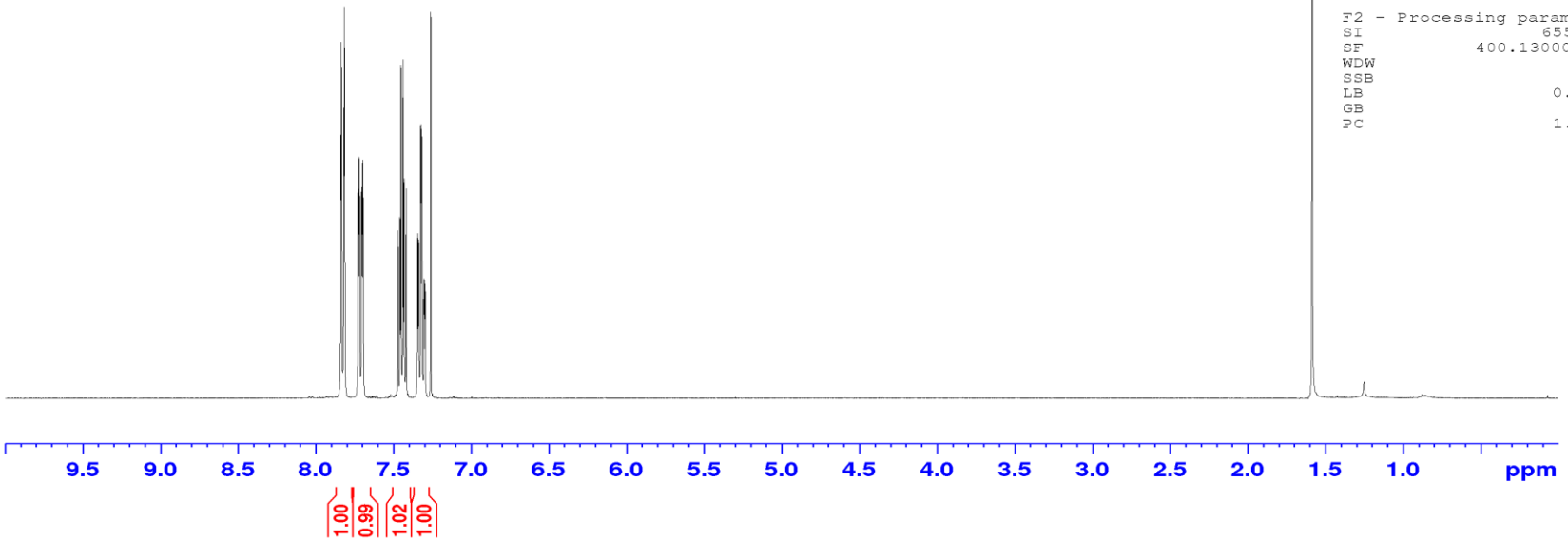
F2 - Processing parameters
SI               32768
SF         100.6127569 MHz
WDW                EM
SSB                 0
LB               1.00 Hz
GB                  0
PC               1.40
```





5o

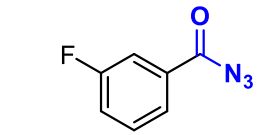
^{1}H NMR (400 MHz, $CDCl_3$)




```
Current Data Parameters
NAME      lhz-20211115-1
EXPNO                  1
PROCNO                 1

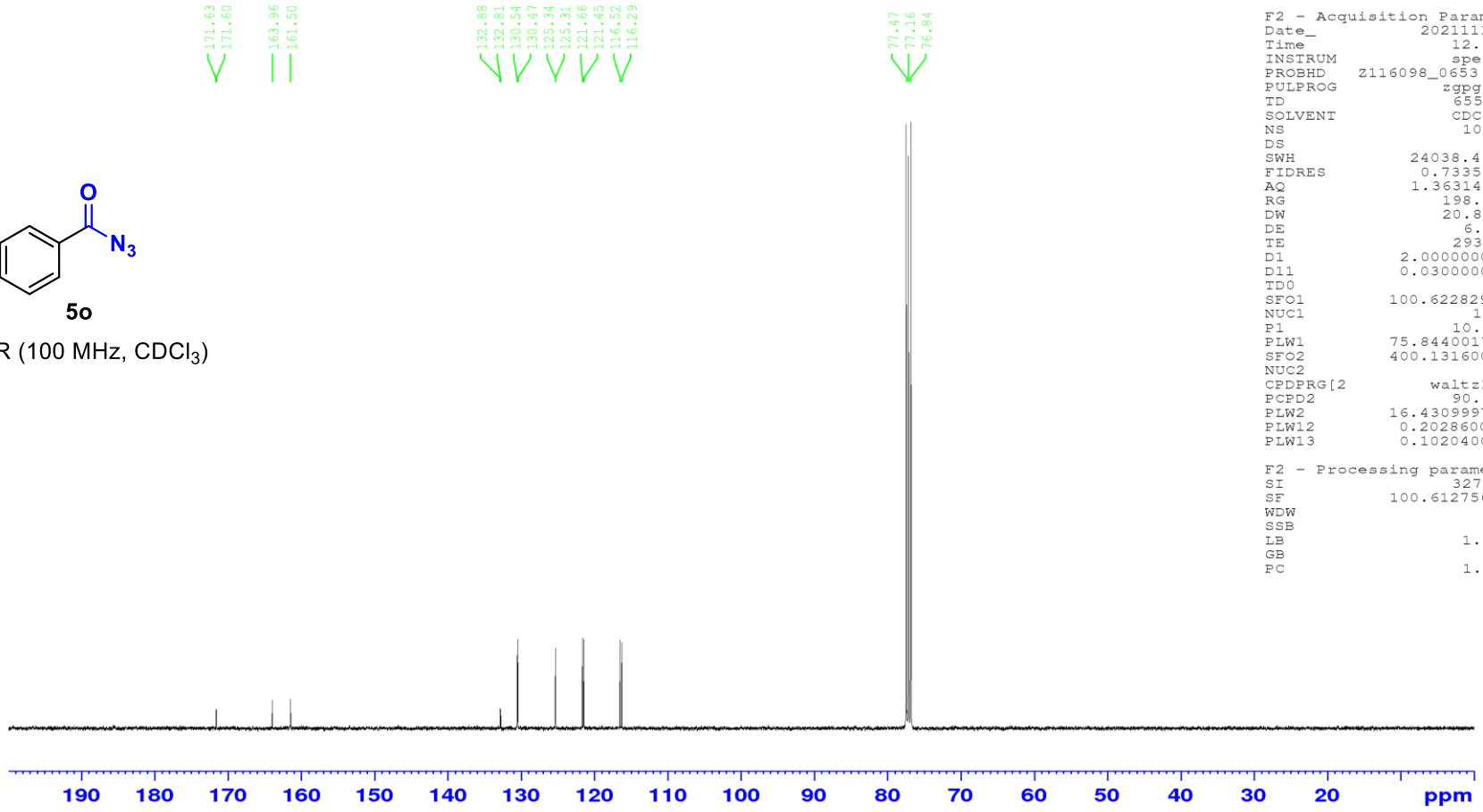
F2 - Acquisition Parameters
Date_           20211115
Time              11.10 h
INSTRUM             spect
PROBHD   Z116098_0653 (
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                     8
DS                     2
SWH             8012.820 Hz
FIDRES          0.244532 Hz
AQ              4.0894465 sec
RG                112.15
DW                62.400 usec
DE                 6.50 usec
TE                292.6 K
D1             1.00000000 sec
TD0                    1
SFO1          400.1324708 MHz
NUC1                  1H
P1                10.00 usec
PLW1          16.43099976 W

F2 - Processing parameters
SI                 65536
SF            400.1300099 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```



5o

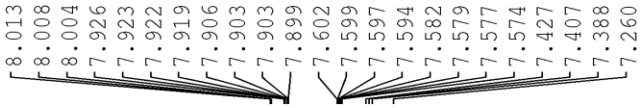
^{13}C NMR (100 MHz, $CDCl_3$)

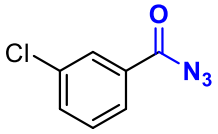



```
Current Data Parameters
NAME      lhz-20211115-1
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20211115
Time              12.57 h
INSTRUM             spect
PROBHD   Z116098_0653 (
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                 1024
DS                    4
SWH           24038.461 Hz
FIDRES         0.733596 Hz
AQ            1.3631488 sec
RG               198.36
DW               20.800 usec
DE                 6.50 usec
TE                293.3 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1
SFO1        100.6228298 MHz
NUC1                13C
P1                10.00 usec
PLW1        75.84400177 W
SFO2        400.1316005 MHz
NUC2                1H
CPDPRG[2        waltz16
PCPD2             90.00 usec
PLW2        16.43099976 W
PLW12        0.20286000 W
PLW13        0.10204000 W

F2 - Processing parameters
SI                32768
SF          100.6127569 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```





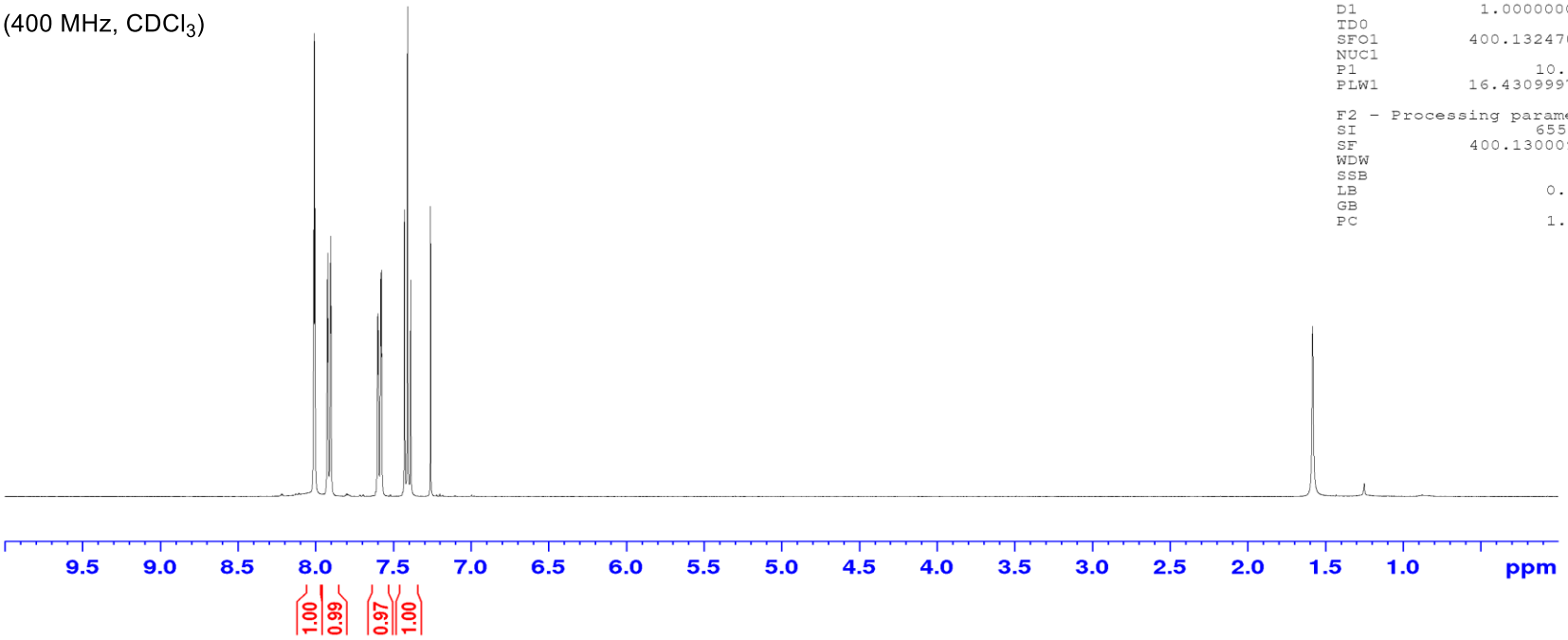
5p

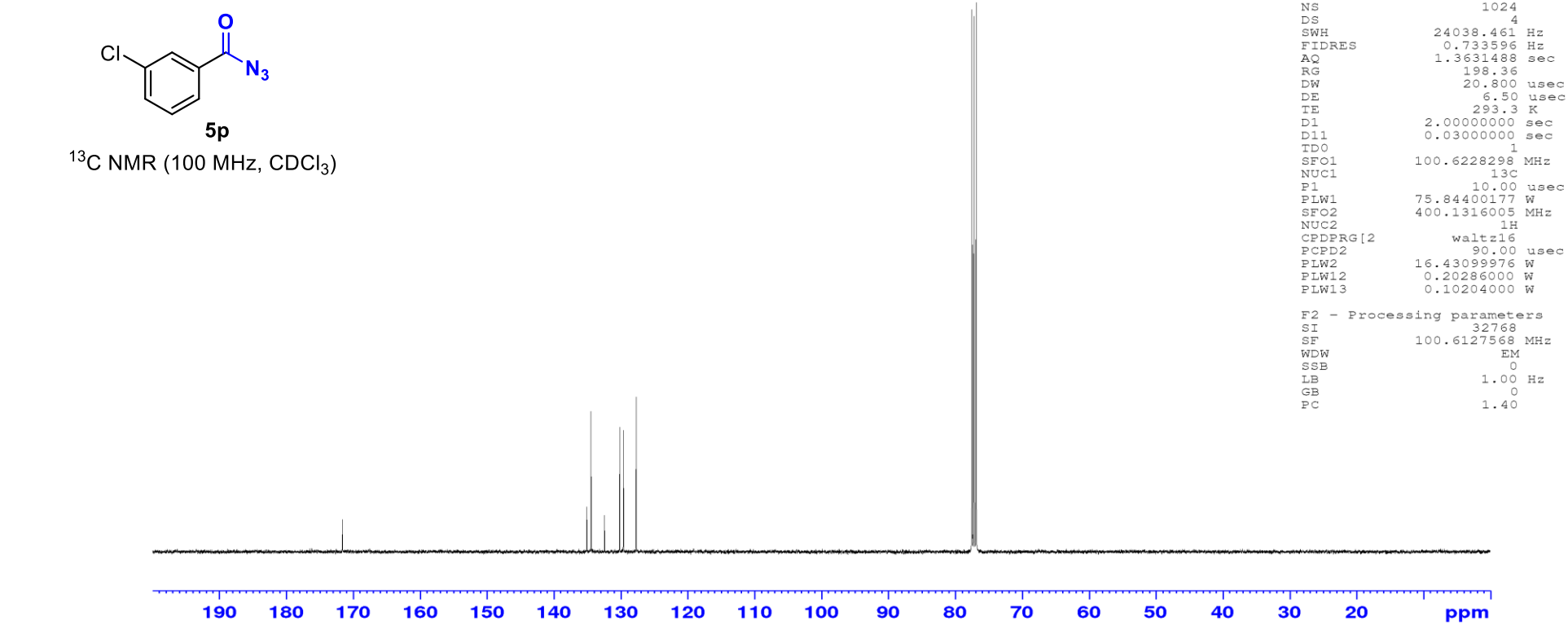
^{1}H NMR (400 MHz, $CDCl_3$)

```
Current Data Parameters
NAME      lhz-20211115-2
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20211115
Time              11.14 h
INSTRUM             spect
PROBHD   Z116098_0653 (
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                     8
DS                     2
SWH             8012.820 Hz
FIDRES          0.244532 Hz
AQ              4.0894465 sec
RG                175.57
DW                62.400 usec
DE                 6.50 usec
TE                292.5 K
D1             1.00000000 sec
TD0                    1
SFO1          400.1324708 MHz
NUC1                  1H
P1                10.00 usec
PLW1          16.43099976 W

F2 - Processing parameters
SI                 65536
SF            400.1300099 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```





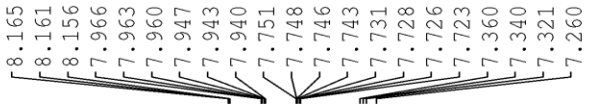
5p

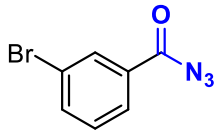
^{13}C NMR (100 MHz, $CDCl_3$)

```
Current Data Parameters
NAME      1hz-20211115-2
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20211115
Time              13.59 h
INSTRUM            spect
PROBHD    Z116098_0653 (
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                 1024
DS                    4
SWH           24038.461 Hz
FIDRES         0.733596 Hz
AQ            1.3631488 sec
RG               198.36
DW               20.800 usec
DE                6.50 usec
TE                293.3 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1
SFO1        100.6228298 MHz
NUC1               13C
P1               10.00 usec
PLW1        75.84400177 W
SFO2        400.1316005 MHz
NUC2               1H
CPDPRG[2        waltz16
PCPD2            90.00 usec
PLW2        16.43099976 W
PLW12        0.20286000 W
PLW13        0.10204000 W

F2 - Processing parameters
SI                32768
SF          100.6127568 MHz
WDW                EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```





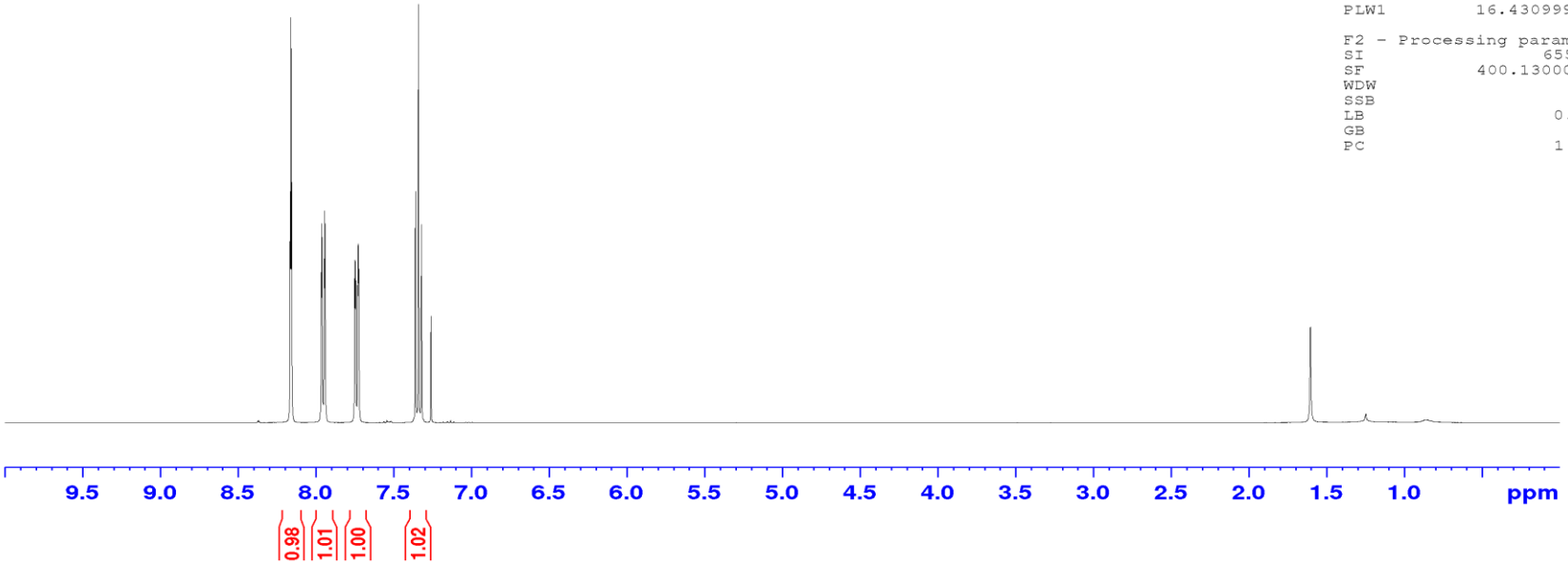
5q

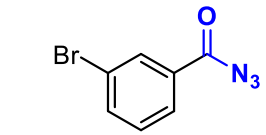
^{1}H NMR (400 MHz, $CDCl_3$)

```
Current Data Parameters
NAME      1hz-20211115-3
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20211115
Time              11.18 h
INSTRUM             spect
PROBHD   Z116098_0653 (
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                     8
DS                     2
SWH             8012.820 Hz
FIDRES          0.244532 Hz
AQ              4.0894465 sec
RG                156.98
DW                62.400 usec
DE                 6.50 usec
TE                292.5 K
D1             1.00000000 sec
TD0                    1
SFO1          400.1324708 MHz
NUC1                  1H
P1                10.00 usec
PLW1          16.43099976 W

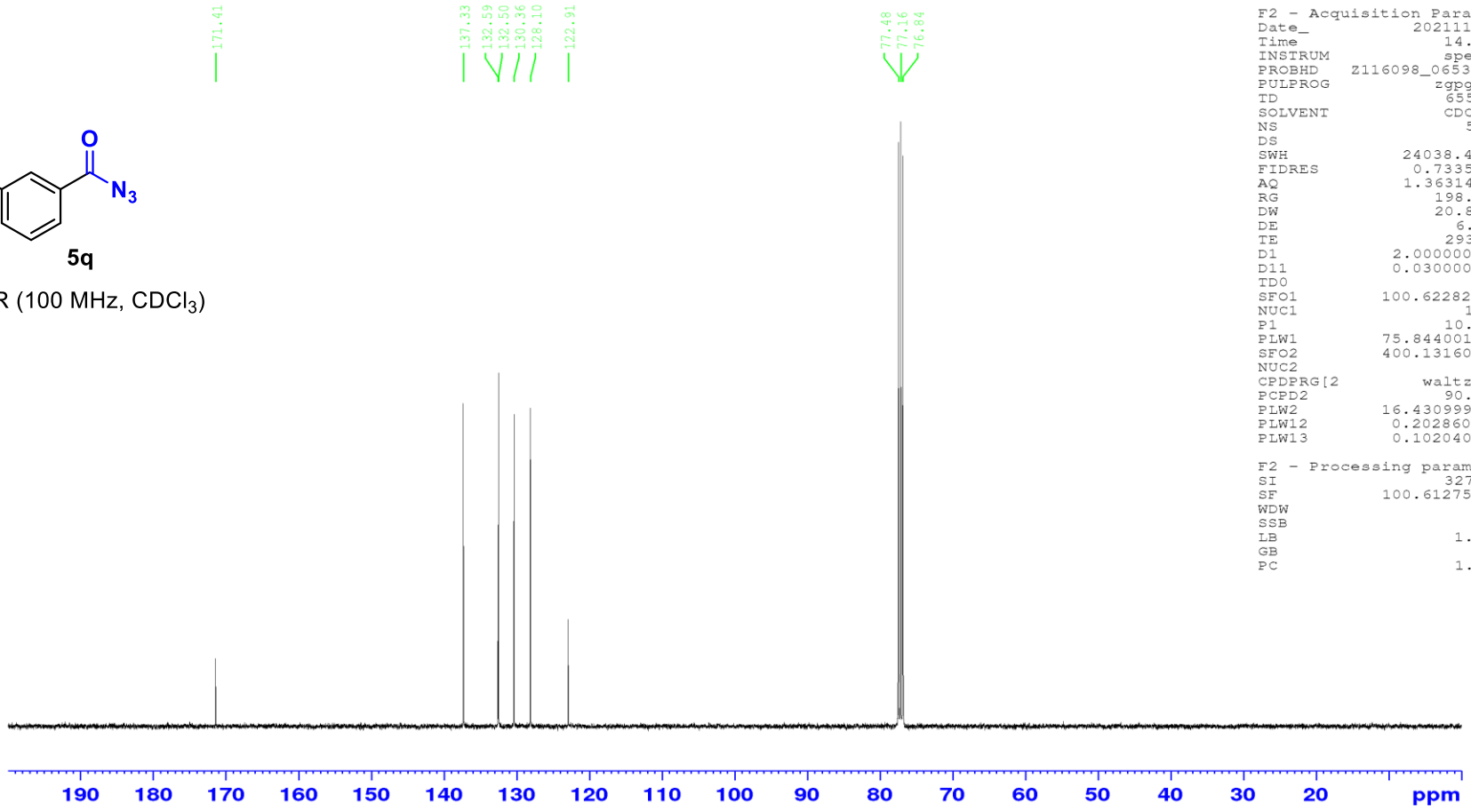
F2 - Processing parameters
SI                 65536
SF            400.1300099 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```





5q

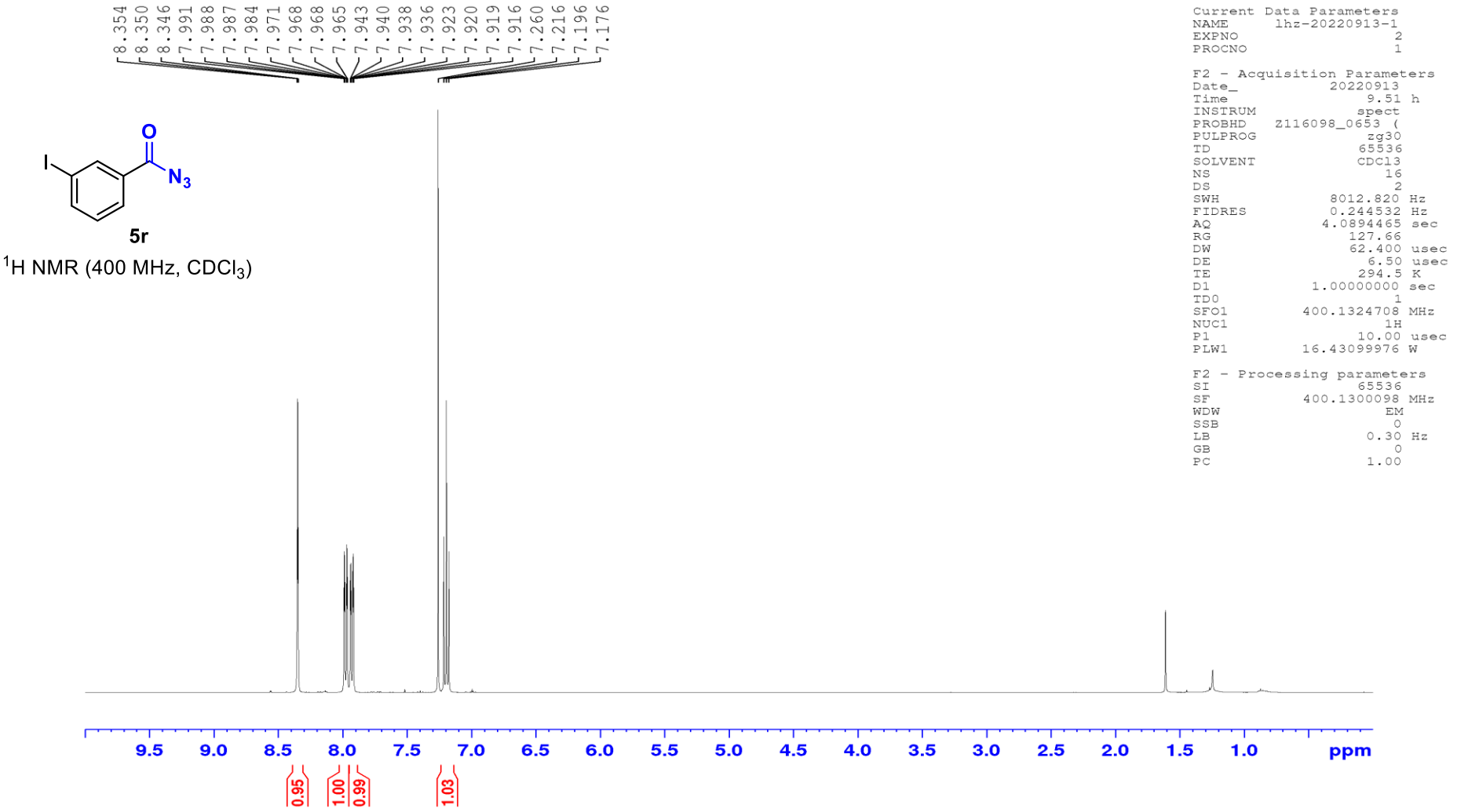
^{13}C NMR (100 MHz, $CDCl_3$)

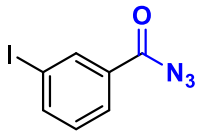



```
Current Data Parameters
NAME      lhz-20211115-3
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20211115
Time              14.36 h
INSTRUM             spect
PROBHD   Z116098_0653 (
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  588
DS                    4
SWH           24038.461 Hz
FIDRES         0.733596 Hz
AQ             1.3631488 sec
RG                198.36
DW                20.800 usec
DE                 6.50 usec
TE                293.1 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1
SFO1        100.6228298 MHz
NUC1                13C
P1                10.00 usec
PLW1        75.84400177 W
SFO2        400.1316005 MHz
NUC2                1H
CPDPRG[2        waltz16
PCPD2             90.00 usec
PLW2        16.43099976 W
PLW12        0.20286000 W
PLW13        0.10204000 W

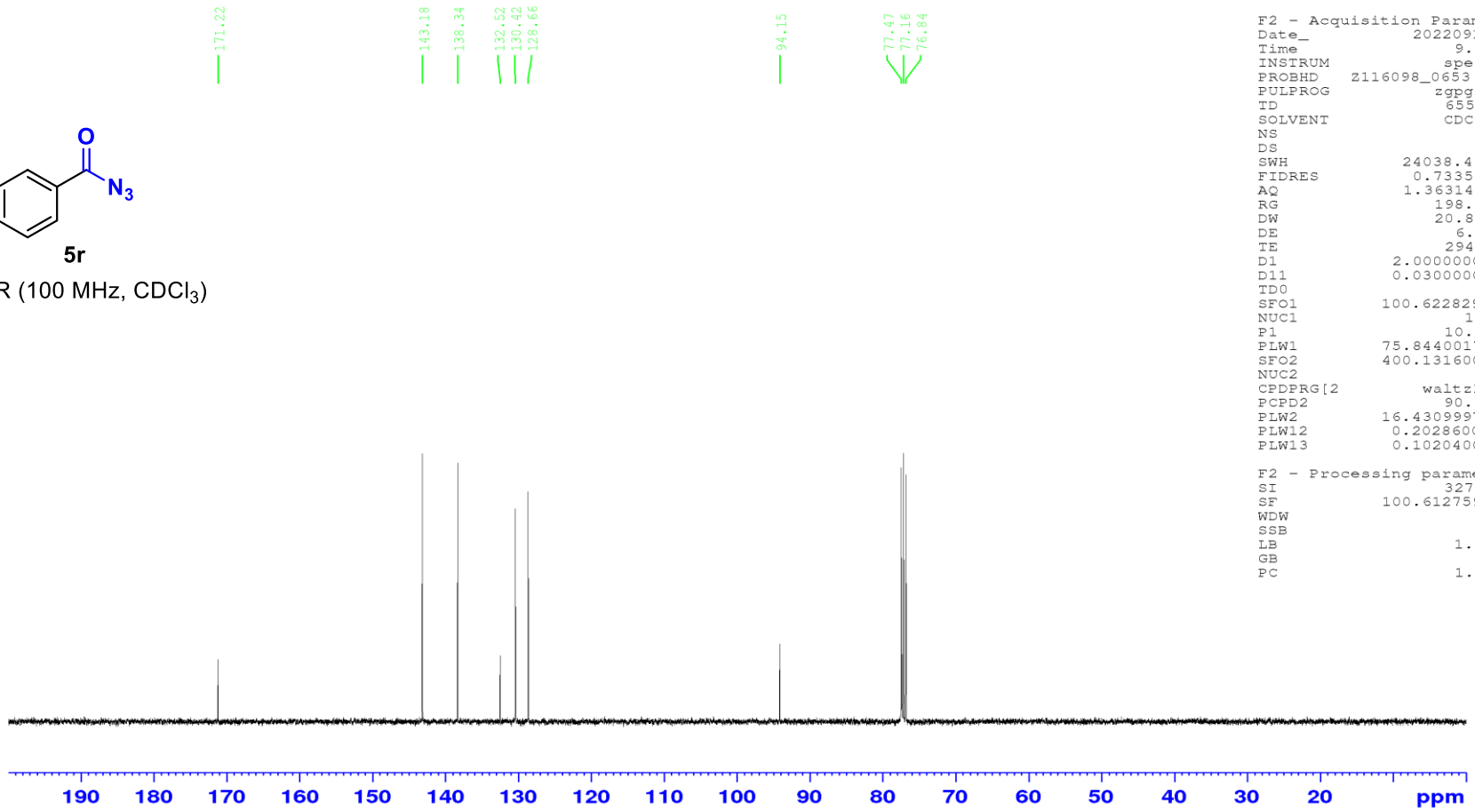
F2 - Processing parameters
SI                32768
SF          100.6127579 MHz
WDW                EM
SSB                 0
LB               1.00 Hz
GB                  0
PC               1.40
```


8.354
8.350
8.346
7.991
7.988
7.987
7.984
7.971
7.968
7.968
7.965
7.943
7.940
7.938
7.936
7.923
7.920
7.919
7.916
7.260
7.216
7.196
7.176
O
I
N3
5r
1H NMR (400 MHz, CDCl3)
Current Data Parameters
NAME lhz-20220913-1
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20220913
Time 9.51 h
INSTRUM spect
PROBHD Z116098_0653 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.244532 Hz
AQ 4.0894465 sec
RG 127.66
DW 62.400 usec
DE 6.50 usec
TE 294.5 K
D1 1.00000000 sec
TD0 1
SFO1 400.1324708 MHz
NUC1 1H
P1 10.00 usec
PLW1 16.43099976 W
F2 - Processing parameters
SI 65536
SF 400.1300098 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
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
ppm
0.95
1.00
0.99
1.03



5r

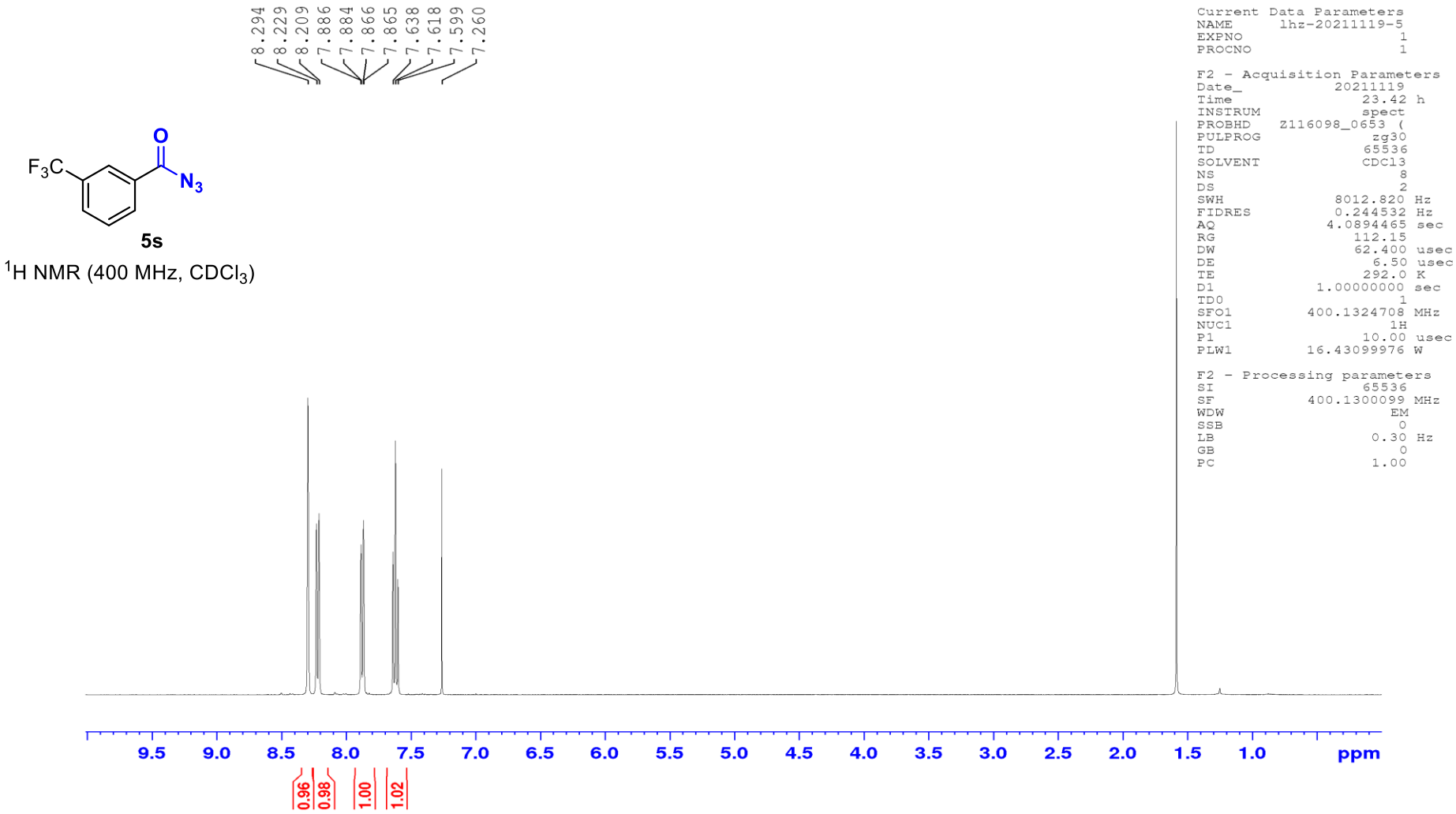
^{13}C NMR (100 MHz, $CDCl_3$)

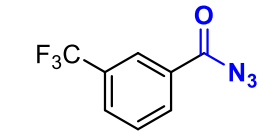



```
Current Data Parameters
NAME      lhz-20220913-1
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20220913
Time               9.49 h
INSTRUM           spect
PROBHD    Z116098_0653 (
PULPROG         zgpg30
TD               65536
SOLVENT          CDCl3
NS                  88
DS                   4
SWH          24038.461 Hz
FIDRES        0.733596 Hz
AQ            1.3631488 sec
RG               198.36
DW               20.800 usec
DE                6.50 usec
TE               294.6 K
D1          2.00000000 sec
D11         0.03000000 sec
TD0                  1
SFO1       100.6228298 MHz
NUC1              13C
P1               10.00 usec
PLW1       75.84400177 W
SFO2       400.1316005 MHz
NUC2              1H
CPDPRG[2       waltz16
PCPD2            90.00 usec
PLW2       16.43099976 W
PLW12       0.20286000 W
PLW13       0.10204000 W

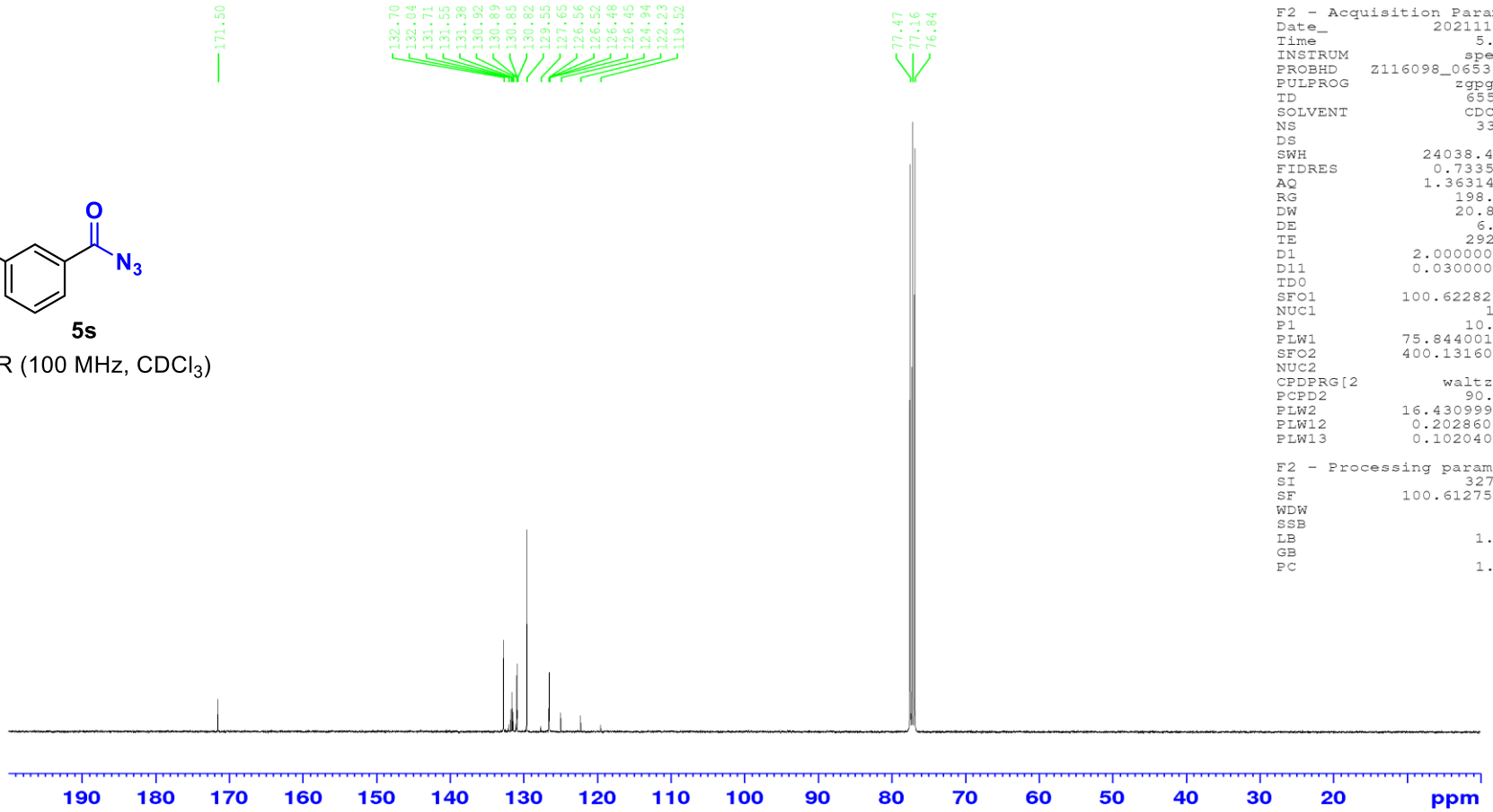
F2 - Processing parameters
SI                32768
SF         100.6127594 MHz
WDW               EM
SSB                  0
LB                1.00 Hz
GB                   0
PC                1.40
```


8.294
8.229
8.209
7.886
7.884
7.866
7.865
7.638
7.618
7.599
7.260
O
F3C
N3
5s
1H NMR (400 MHz, CDCl3)
Current Data Parameters
NAME lhz-20211119-5
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20211119
Time 23.42 h
INSTRUM spect
PROBHD Z116098_0653 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 8012.820 Hz
FIDRES 0.244532 Hz
AQ 4.0894465 sec
RG 112.15
DW 62.400 usec
DE 6.50 usec
TE 292.0 K
D1 1.00000000 sec
TD0 1
SFO1 400.1324708 MHz
NUC1 1H
P1 10.00 usec
PLW1 16.43099976 W
F2 - Processing parameters
SI 65536
SF 400.1300099 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
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 ppm
0.96
0.98
1.00
1.02



5s

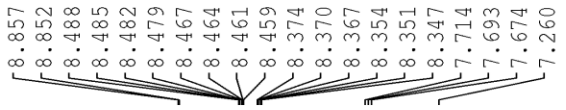
^{13}C NMR (100 MHz, $CDCl_3$)

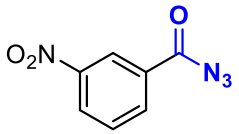



```
Current Data Parameters
NAME      1hz-20211119-5
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20211120
Time                5.05 h
INSTRUM            spect
PROBHD    Z116098_0653 (
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                 3333
DS                    4
SWH           24038.461 Hz
FIDRES         0.733596 Hz
AQ            1.3631488 sec
RG               198.36
DW               20.800 usec
DE                6.50 usec
TE               292.9 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                   1
SFO1         100.6228298 MHz
NUC1                13C
P1                10.00 usec
PLW1         75.84400177 W
SFO2         400.1316005 MHz
NUC2                1H
CPDPRG[2        waltz16
PCPD2             90.00 usec
PLW2         16.43099976 W
PLW12         0.20286000 W
PLW13         0.10204000 W

F2 - Processing parameters
SI                32768
SF           100.6127564 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```





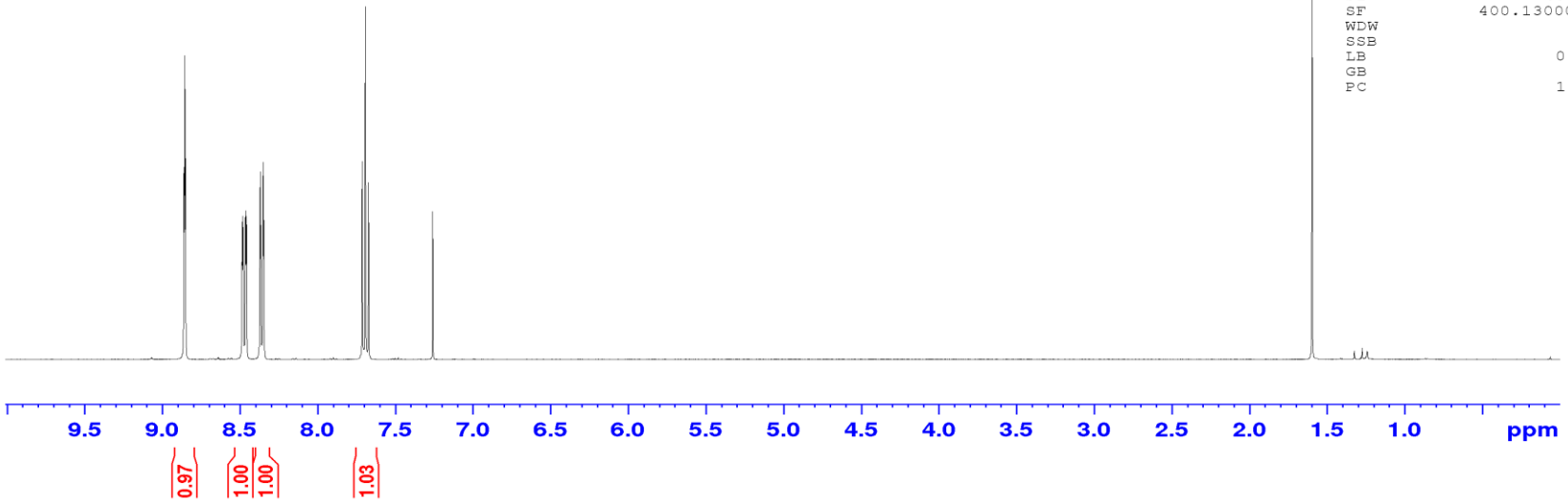
5t

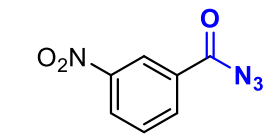
^{1}H NMR (400 MHz, $CDCl_3$)

```
Current Data Parameters
NAME      1hz-20211120-1
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20211120
Time              23.57 h
INSTRUM             spect
PROBHD    Z116098_0653 (
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                     8
DS                     2
SWH             8012.820 Hz
FIDRES          0.244532 Hz
AQ              4.0894465 sec
RG                112.15
DW                62.400 usec
DE                 6.50 usec
TE                292.2 K
D1             1.00000000 sec
TD0                    1
SFO1          400.1324708 MHz
NUC1                  1H
P1                10.00 usec
PLW1          16.43099976 W

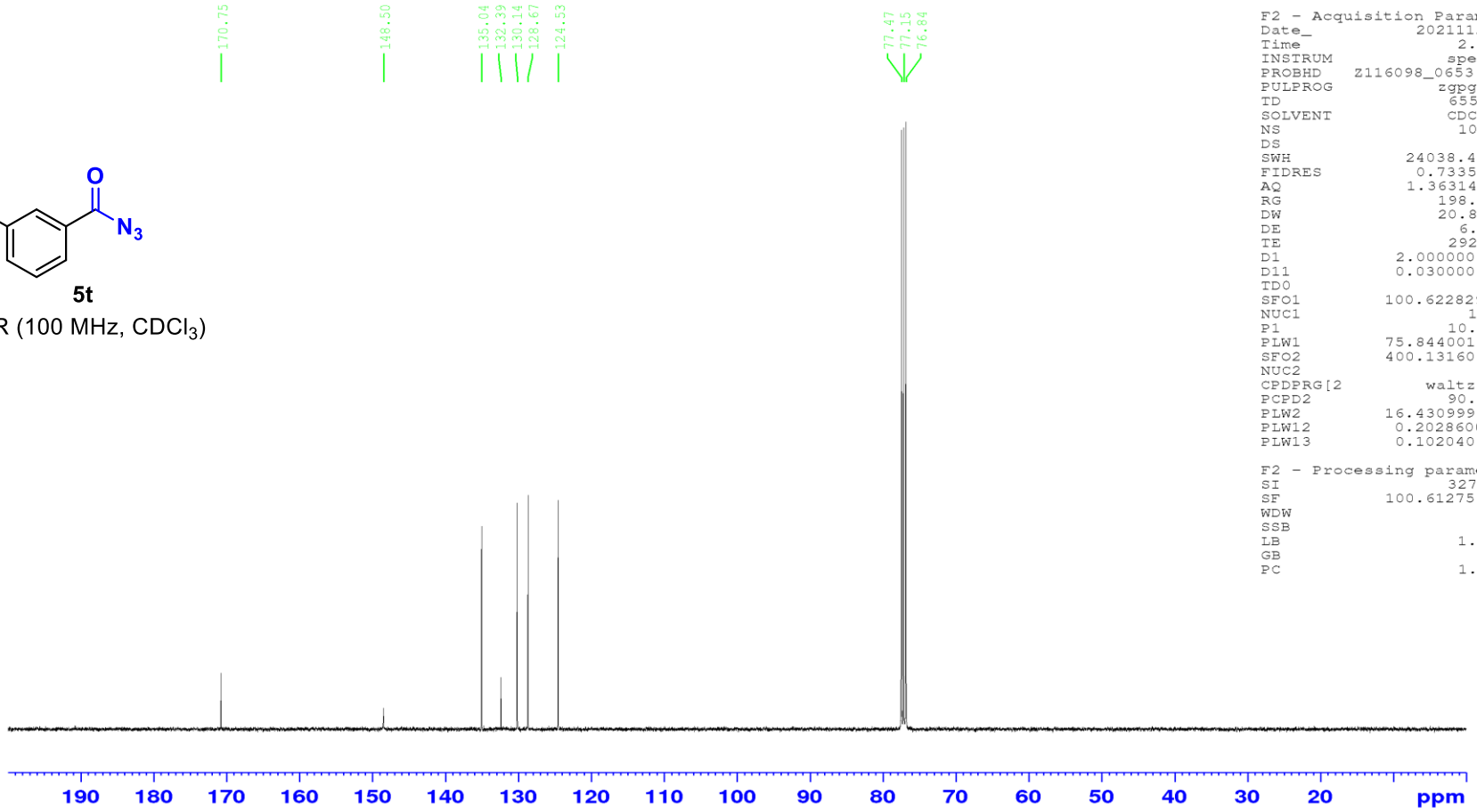
F2 - Processing parameters
SI                 65536
SF            400.1300098 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```





5t

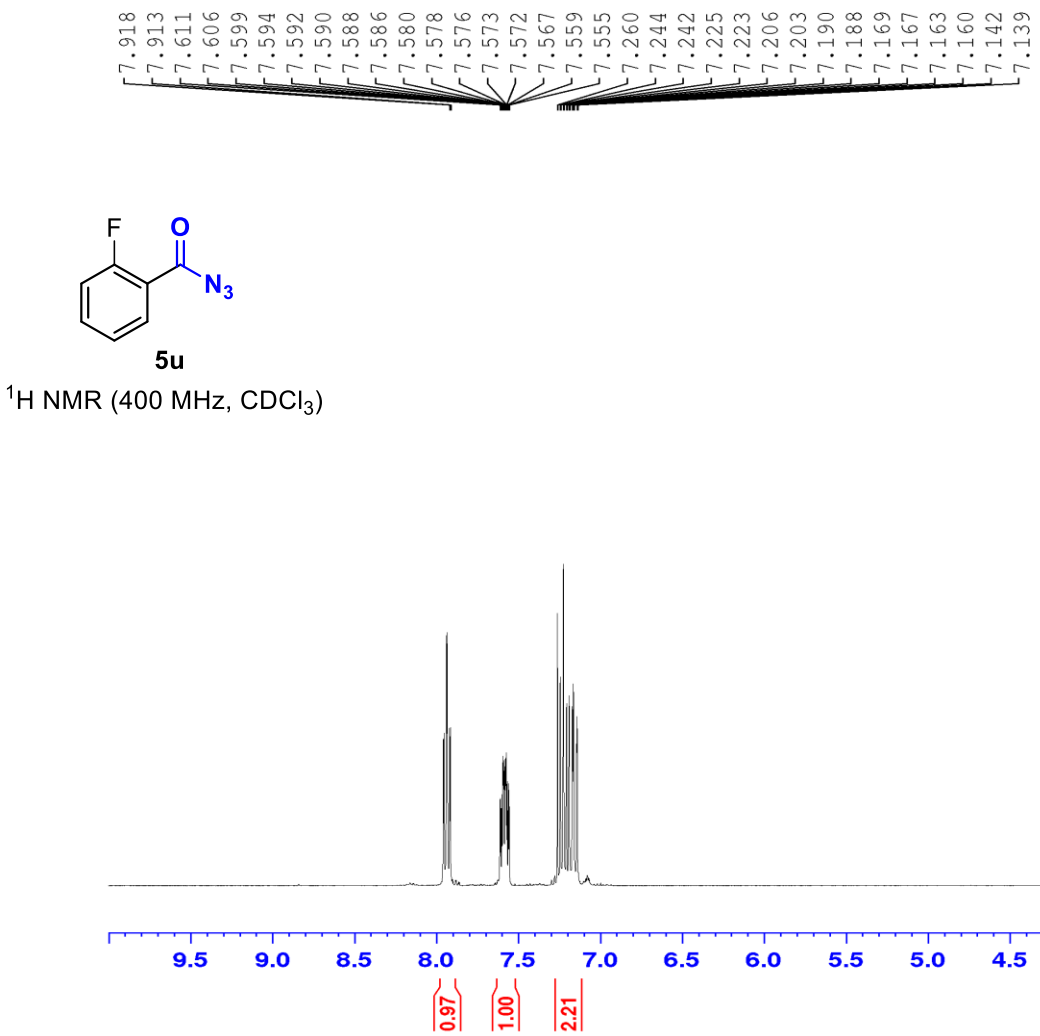
^{13}C NMR (100 MHz, $CDCl_3$)

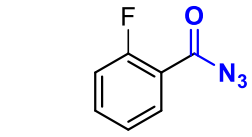



```
Current Data Parameters
NAME      lhz-20211120-1
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20211121
Time              2.18 h
INSTRUM           spect
PROBHD   Z116098_0653 (
PULPROG         zgpg30
TD               65536
SOLVENT          CDCl3
NS                1024
DS                   4
SWH          24038.461 Hz
FIDRES        0.733596 Hz
AQ           1.3631488 sec
RG              198.36
DW              20.800 usec
DE                6.50 usec
TE               292.9 K
D1          2.00000000 sec
D11         0.03000000 sec
TD0                  1
SFO1       100.6228298 MHz
NUC1               13C
P1               10.00 usec
PLW1       75.84400177 W
SFO2       400.1316005 MHz
NUC2               1H
CPDPRG[2       waltz16
PCPD2            90.00 usec
PLW2       16.43099976 W
PLW12       0.20286000 W
PLW13       0.10204000 W

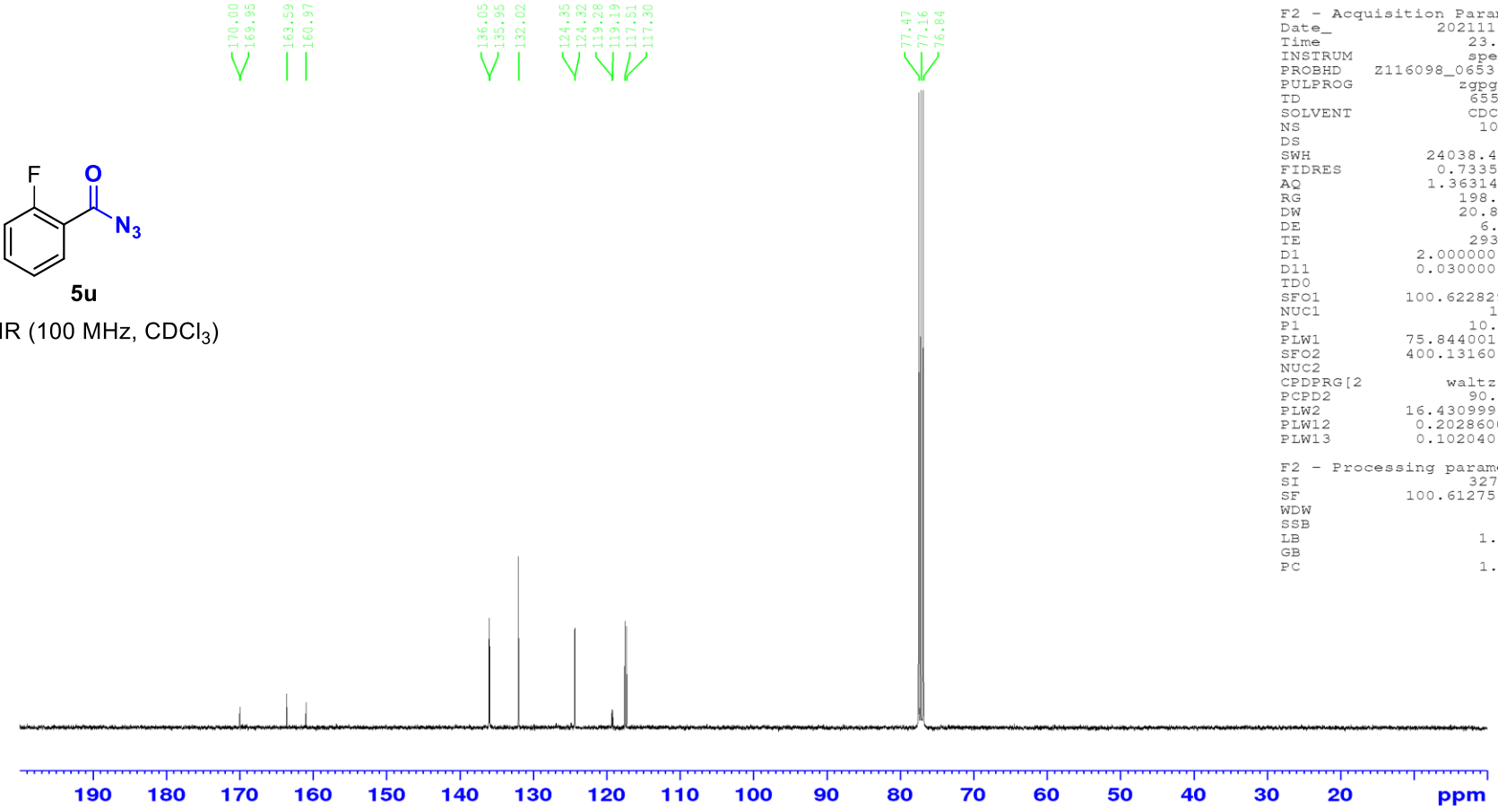
F2 - Processing parameters
SI               32768
SF         100.6127579 MHz
WDW                EM
SSB                 0
LB               1.00 Hz
GB                  0
PC               1.40
```


7.918
7.913
7.611
7.606
7.599
7.594
7.592
7.590
7.588
7.586
7.580
7.578
7.576
7.573
7.572
7.567
7.559
7.555
7.260
7.244
7.242
7.225
7.223
7.206
7.203
7.190
7.188
7.169
7.167
7.163
7.160
7.142
7.139
F
O
N3
5u
1H NMR (400 MHz, CDCl3)
Current Data Parameters
NAME lhz-20211118-2
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20211118
Time 21.57 h
INSTRUM spect
PROBHD Z116098_0653 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 8012.820 Hz
FIDRES 0.244532 Hz
AQ 4.0894465 sec
RG 112.15
DW 62.400 usec
DE 6.50 usec
TE 292.5 K
D1 1.00000000 sec
TD0 1
SFO1 400.1324708 MHz
NUC1 1H
P1 10.00 usec
PLW1 16.43099976 W
F2 - Processing parameters
SI 65536
SF 400.1300098 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
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
ppm
0.97
1.00
2.21



5u

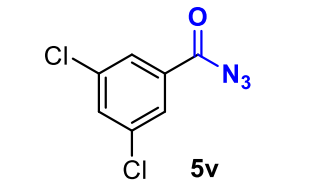
^{13}C NMR (100 MHz, $CDCl_3$)




```
Current Data Parameters
NAME      1hz-20211118-2
EXPNO                  2
PROCNO                 1

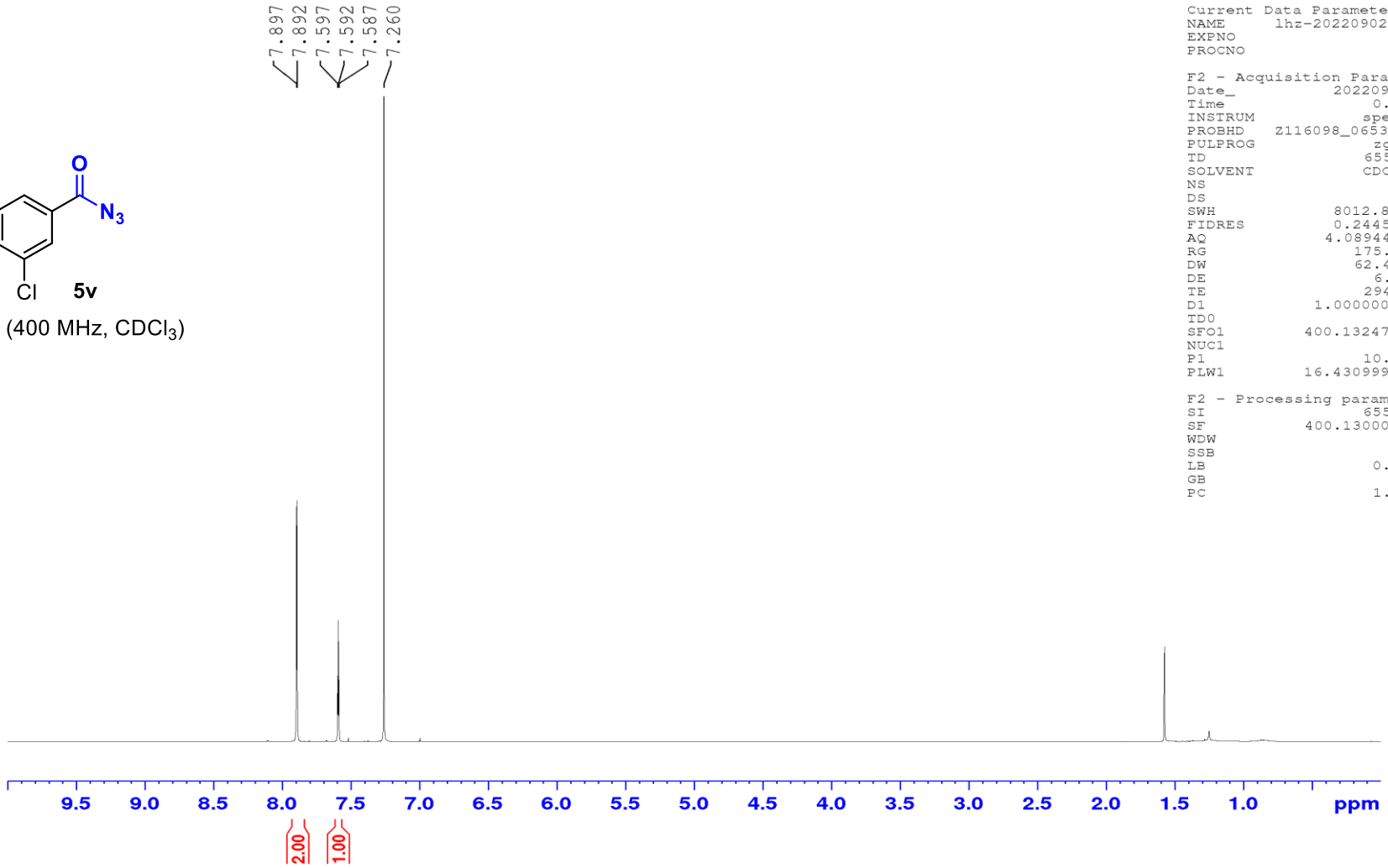
F2 - Acquisition Parameters
Date_           20211118
Time              23.29 h
INSTRUM            spect
PROBHD   Z116098_0653 (
PULPROG         zgpg30
TD               65536
SOLVENT          CDCl3
NS                1024
DS                   4
SWH          24038.461 Hz
FIDRES        0.733596 Hz
AQ            1.3631488 sec
RG               198.36
DW               20.800 usec
DE                6.50 usec
TE               293.0 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                  1
SFO1        100.6228298 MHz
NUC1               13C
P1               10.00 usec
PLW1        75.84400177 W
SFO2        400.1316005 MHz
NUC2               1H
CPDPRG[2       waltz16
PCPD2            90.00 usec
PLW2        16.43099976 W
PLW12        0.20286000 W
PLW13        0.10204000 W

F2 - Processing parameters
SI               32768
SF          100.6127572 MHz
WDW                EM
SSB                 0
LB              1.00 Hz
GB                  0
PC              1.40
```



5v

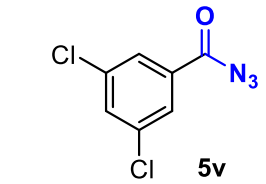
^{1}H NMR (400 MHz, $CDCl_3$)




```
Current Data Parameters
NAME      lhz-20220902-6
EXPNO                  1
PROCNO                 1

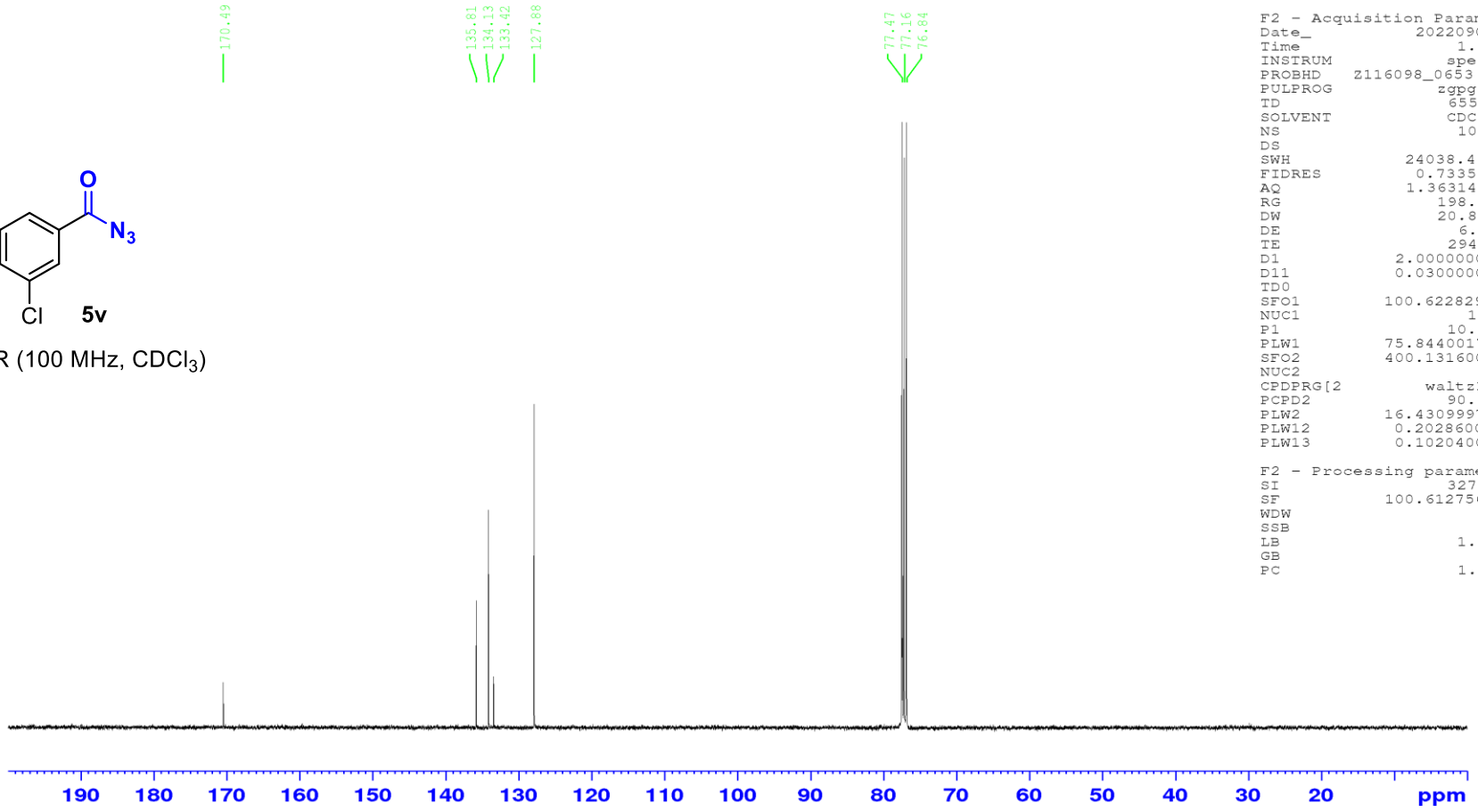
F2 - Acquisition Parameters
Date_           20220903
Time                0.04 h
INSTRUM            spect
PROBHD   Z116098_0653 (
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH              8012.820 Hz
FIDRES           0.244532 Hz
AQ               4.0894465 sec
RG                 175.57
DW                 62.400 usec
DE                  6.50 usec
TE                 294.8 K
D1            1.00000000 sec
TD0                    1
SFO1         400.1324708 MHz
NUC1                  1H
P1                 10.00 usec
PLW1         16.43099976 W

F2 - Processing parameters
SI                 65536
SF           400.1300098 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```



5v

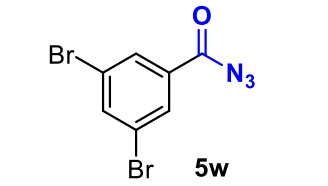
^{13}C NMR (100 MHz, $CDCl_3$)




```
Current Data Parameters
NAME      lhz-20220902-6
EXPNO                  2
PROCNO                 1

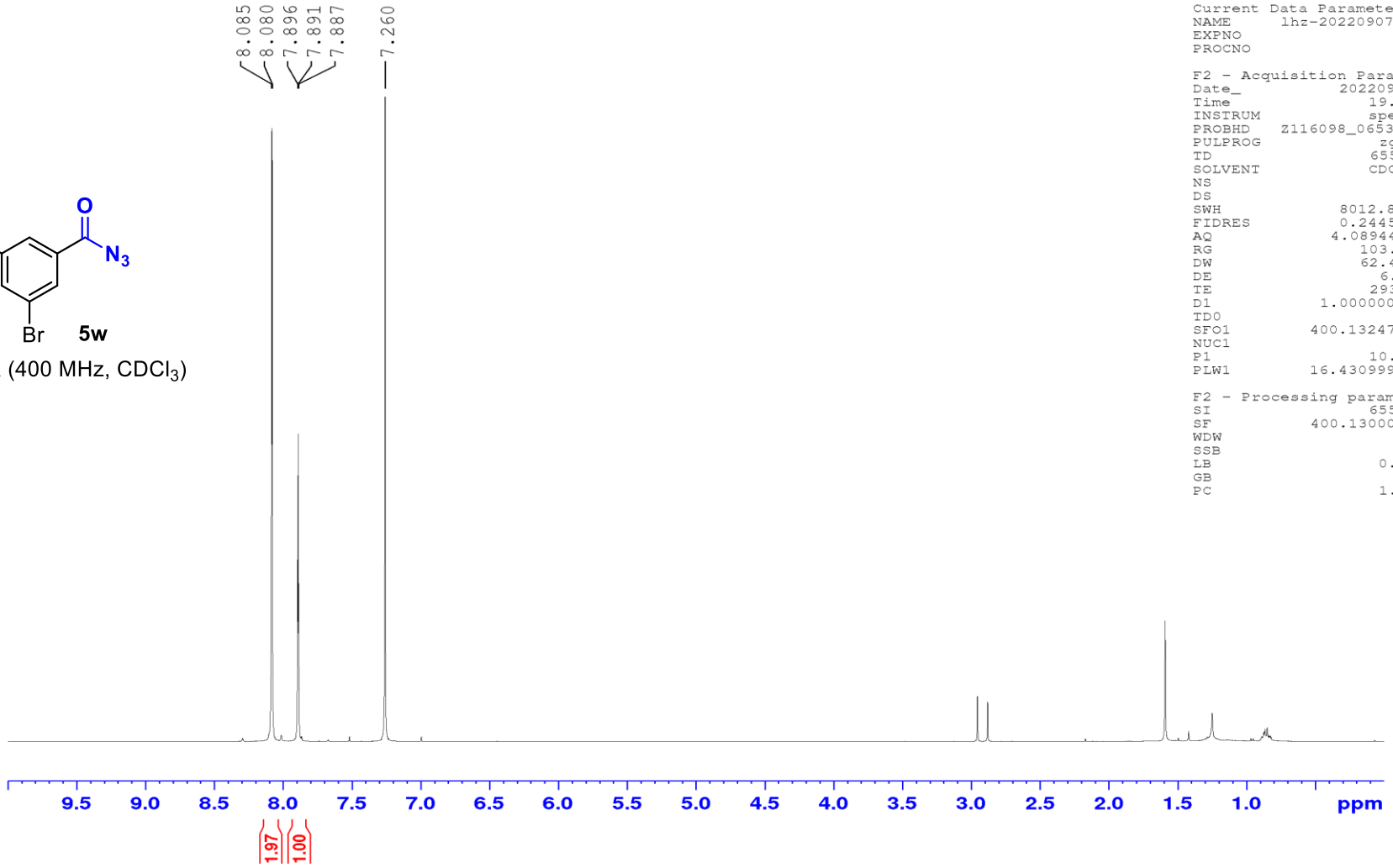
F2 - Acquisition Parameters
Date_           20220903
Time                1.03 h
INSTRUM             spect
PROBHD    Z116098_0653 (
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                 1024
DS                    4
SWH           24038.461 Hz
FIDRES         0.733596 Hz
AQ            1.3631488 sec
RG               198.36
DW               20.800 usec
DE                 6.50 usec
TE                294.9 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1
SFO1        100.6228298 MHz
NUC1                13C
P1                10.00 usec
PLW1        75.84400177 W
SFO2        400.1316005 MHz
NUC2                1H
CPDPRG[2        waltz16
PCPD2             90.00 usec
PLW2        16.43099976 W
PLW12        0.20286000 W
PLW13        0.10204000 W

F2 - Processing parameters
SI                32768
SF          100.6127561 MHz
WDW                EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```



5w

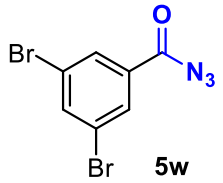
^{1}H NMR (400 MHz, $CDCl_3$)




```
Current Data Parameters
NAME      lhz-20220907-1
EXPNO                  1
PROCNO                 1

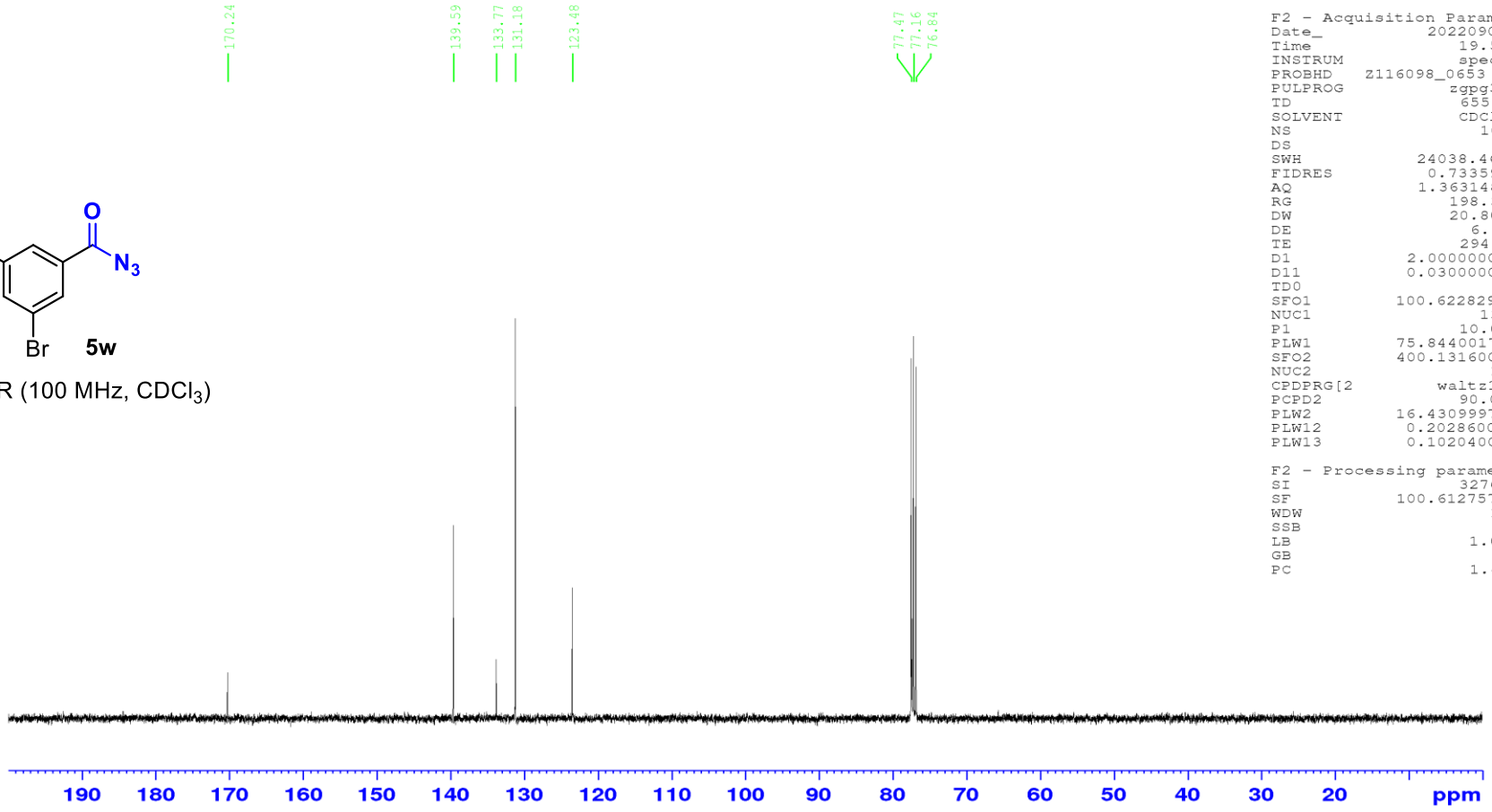
F2 - Acquisition Parameters
Date_           20220907
Time              19.45 h
INSTRUM            spect
PROBHD    Z116098_0653 (
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                    8
DS                    2
SWH            8012.820 Hz
FIDRES         0.244532 Hz
AQ           4.0894465 sec
RG                103.14
DW                62.400 usec
DE                 6.50 usec
TE                293.9 K
D1           1.00000000 sec
TD0                   1
SFO1       400.1324708 MHz
NUC1                 1H
P1                10.00 usec
PLW1       16.43099976 W

F2 - Processing parameters
SI                65536
SF         400.1300098 MHz
WDW                  EM
SSB                   0
LB                 0.30 Hz
GB                    0
PC                 1.00
```



5w

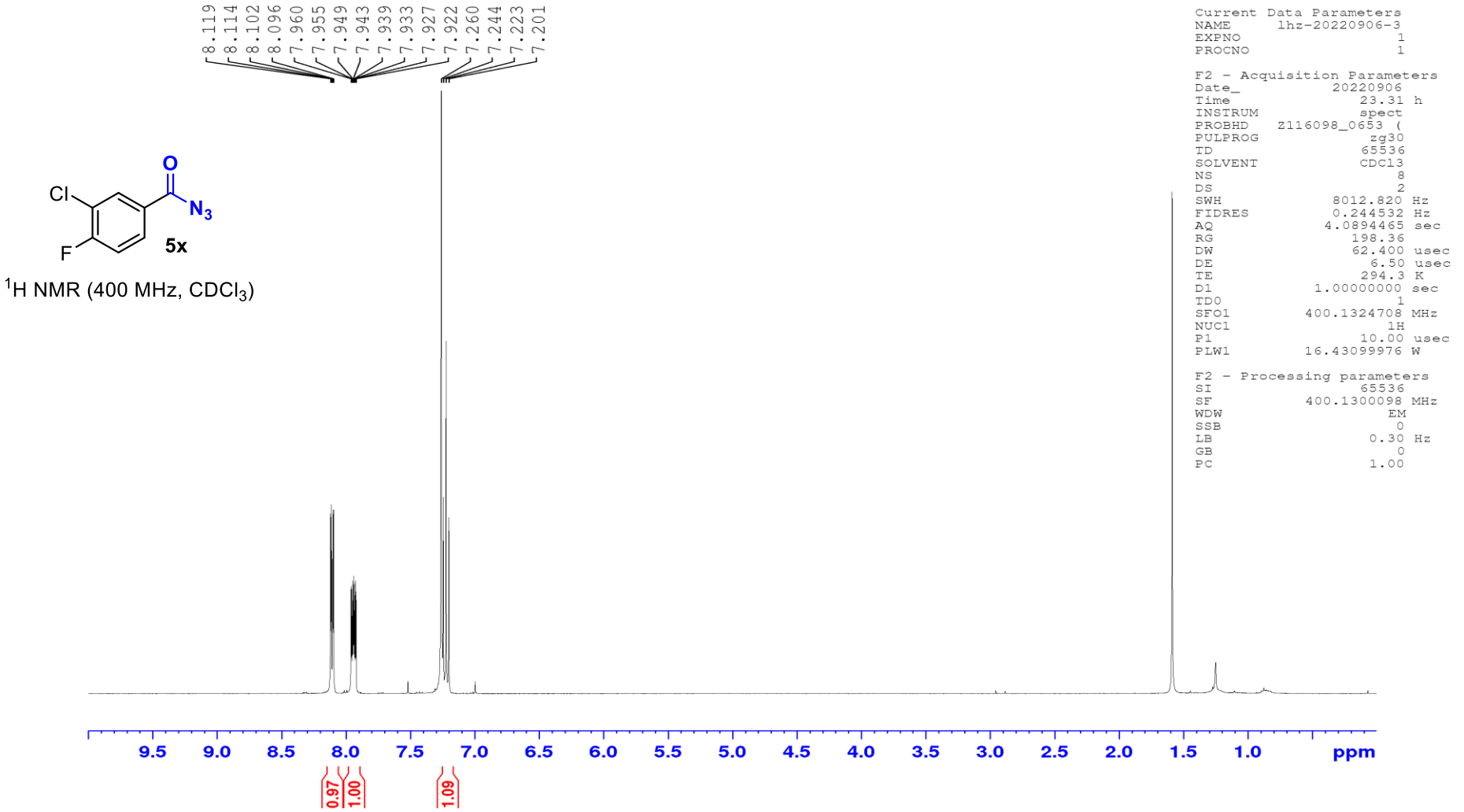
^{13}C NMR (100 MHz, $CDCl_3$)




```
Current Data Parameters
NAME      lhz-20220907-1
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20220907
Time               19.52 h
INSTRUM             spect
PROBHD    Z116098_0653 (
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  108
DS                    4
SWH           24038.461 Hz
FIDRES         0.733596 Hz
AQ             1.3631488 sec
RG                198.36
DW                20.800 usec
DE                 6.50 usec
TE                294.5 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1
SFO1        100.6228298 MHz
NUC1                13C
P1                10.00 usec
PLW1         75.84400177 W
SFO2        400.1316005 MHz
NUC2                1H
CPDPRG[2        waltz16
PCPD2             90.00 usec
PLW2         16.43099976 W
PLW12         0.20286000 W
PLW13         0.10204000 W

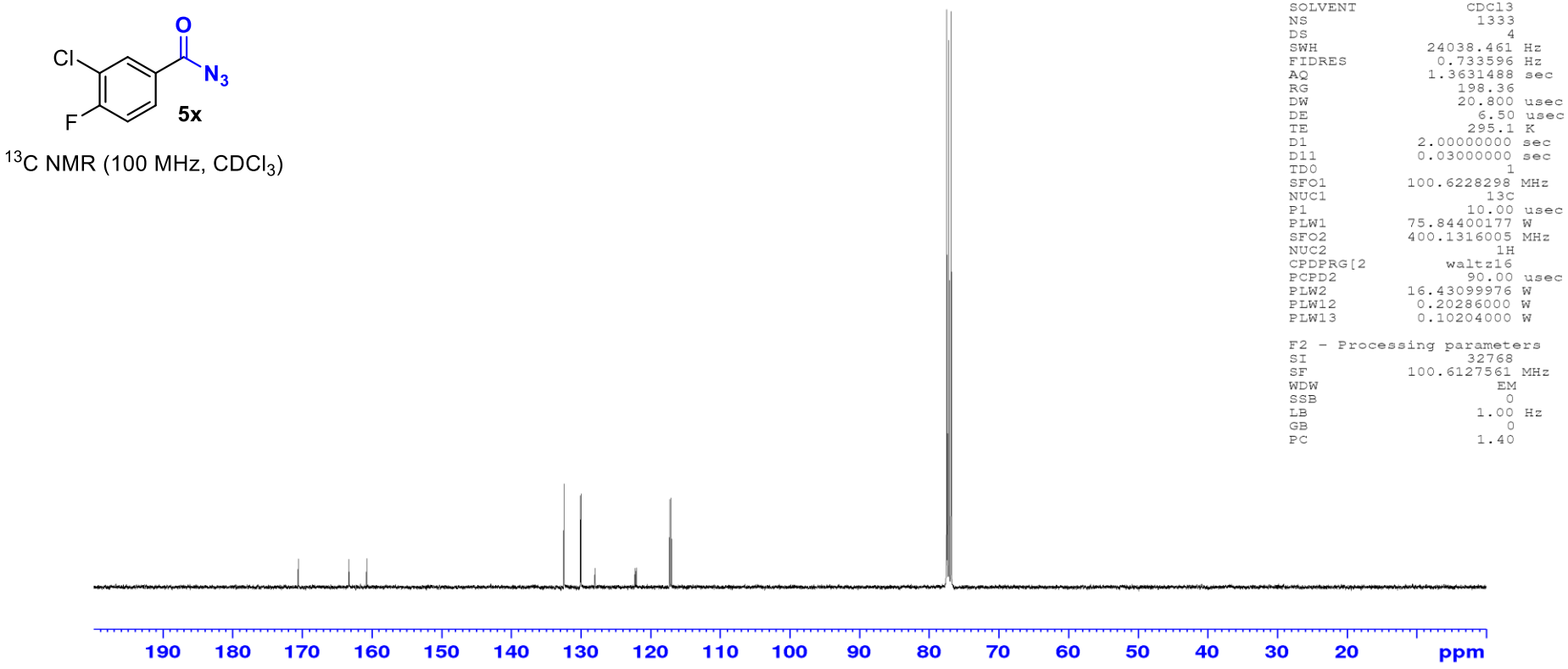
F2 - Processing parameters
SI                32768
SF          100.6127571 MHz
WDW                 EM
SSB                  0
LB                1.00 Hz
GB                   0
PC                1.40
```




```
Current Data Parameters
NAME      1hz-20220906-3
EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
Date_            20220906
Time                23.31 h
INSTRUM             spect
PROBHD    Z116098_0653 (
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                     8
DS                     2
SWH             8012.820 Hz
FIDRES          0.244532 Hz
AQ              4.0894465 sec
RG                198.36
DW                62.400 usec
DE                 6.50 usec
TE                294.3 K
D1             1.00000000 sec
TD0                    1
SFO1          400.1324708 MHz
NUC1                  1H
P1                 10.00 usec
PLW1          16.43099976 W

F2 - Processing parameters
SI                 65536
SF            400.1300098 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```

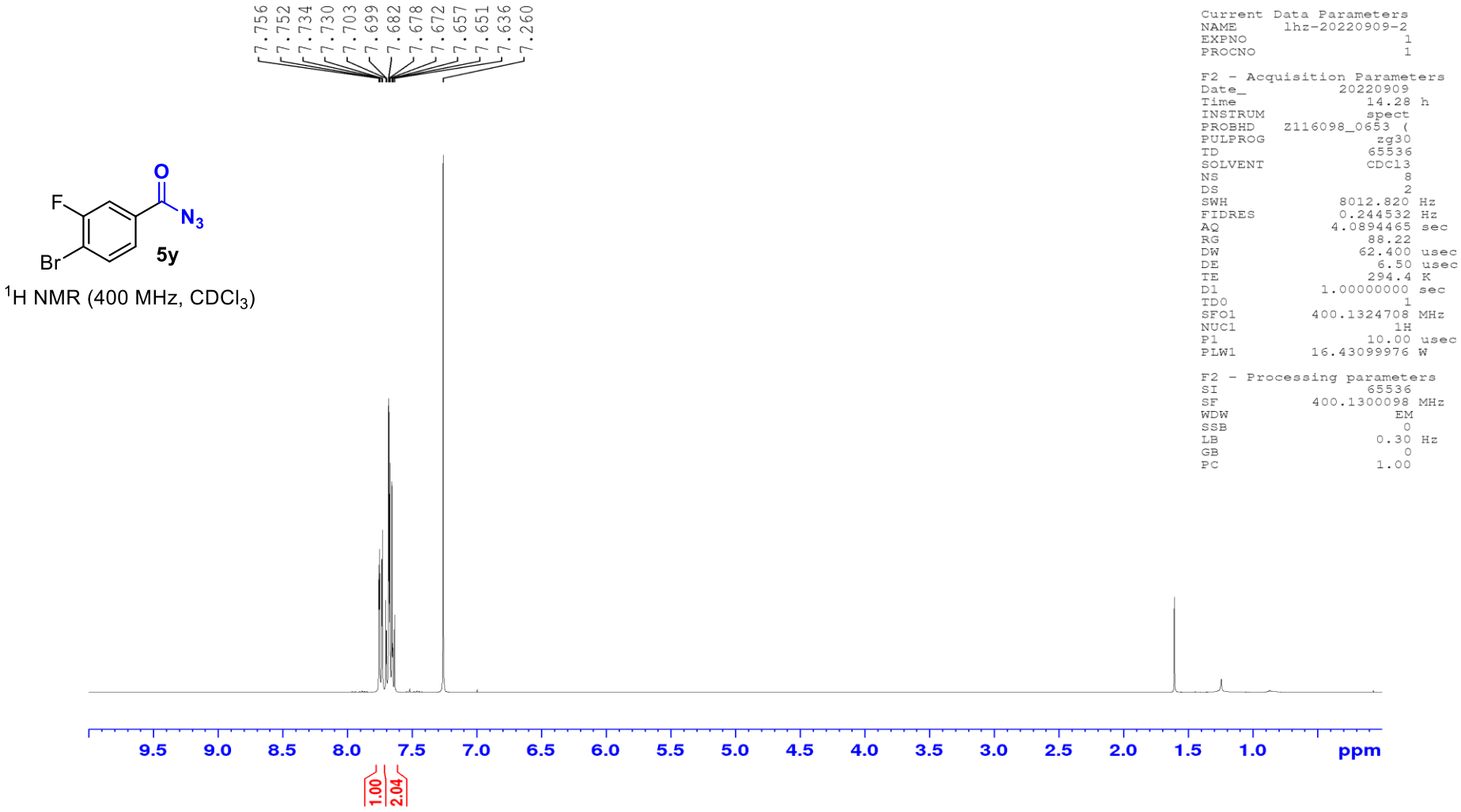


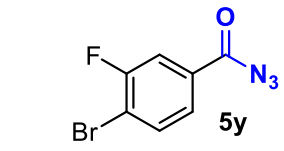
^{13}C NMR (100 MHz, $CDCl_3$)

```
Current Data Parameters
NAME      lhz-20220906-3
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20220907
Time              0.48 h
INSTRUM           spect
PROBHD    Z116098_0653 (
PULPROG         zgpg30
TD               65536
SOLVENT          CDCl3
NS                1333
DS                   4
SWH          24038.461 Hz
FIDRES        0.733596 Hz
AQ            1.3631488 sec
RG               198.36
DW               20.800 usec
DE                6.50 usec
TE               295.1 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                  1
SFO1       100.6228298 MHz
NUC1               13C
P1               10.00 usec
PLW1       75.84400177 W
SFO2       400.1316005 MHz
NUC2               1H
CPDPRG[2        waltz16
PCPD2            90.00 usec
PLW2       16.43099976 W
PLW12       0.20286000 W
PLW13       0.10204000 W

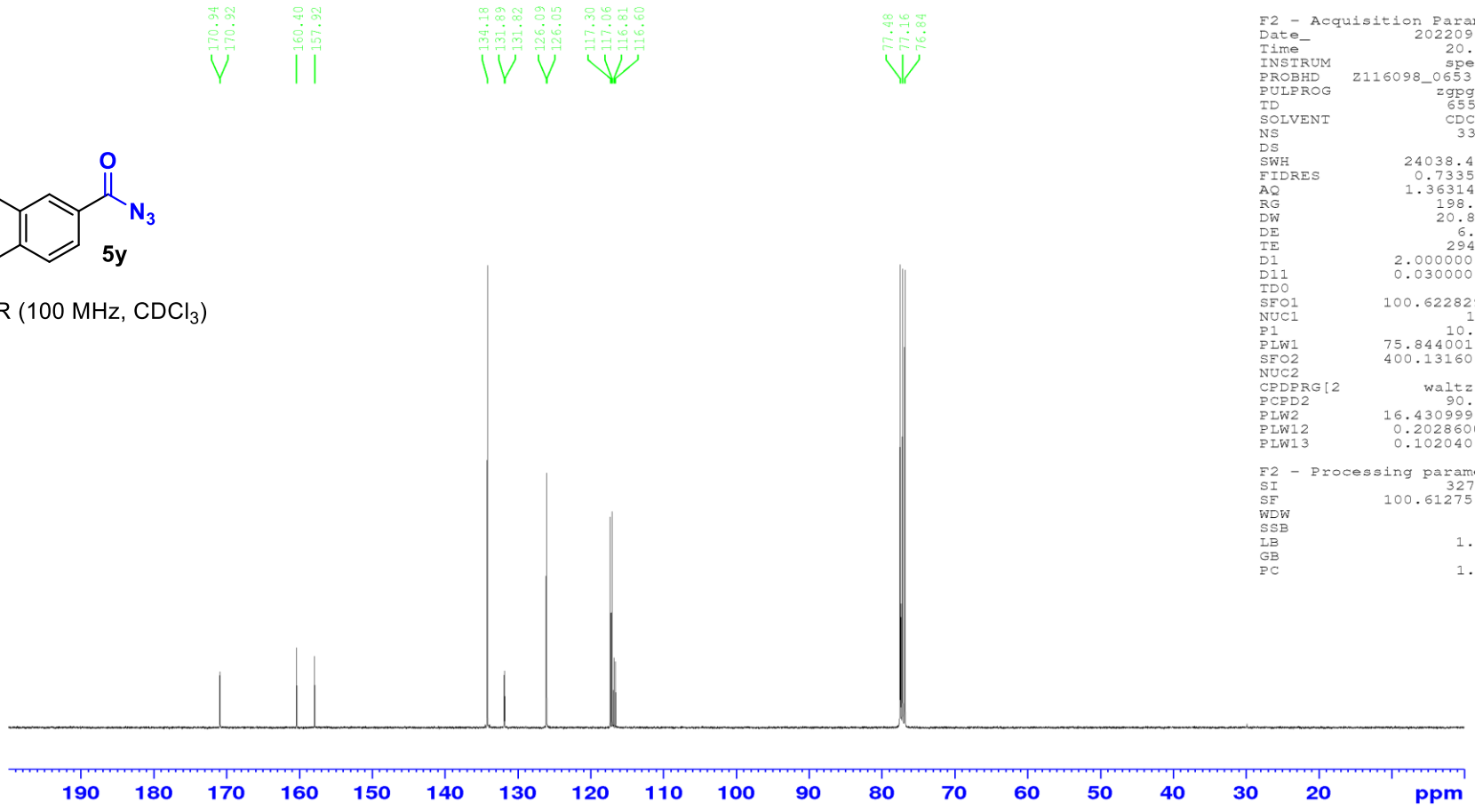
F2 - Processing parameters
SI               32768
SF         100.6127561 MHz
WDW                EM
SSB                 0
LB               1.00 Hz
GB                  0
PC               1.40
```


7.756
7.752
7.734
7.730
7.703
7.699
7.682
7.678
7.672
7.657
7.651
7.636
7.260
O
F
N3
Br
5y
1H NMR (400 MHz, CDCl3)
Current Data Parameters
NAME lhz-20220909-2
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20220909
Time 14.28 h
INSTRUM spect
PROBHD Z116098_0653 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 8012.820 Hz
FIDRES 0.244532 Hz
AQ 4.0894465 sec
RG 88.22
DW 62.400 usec
DE 6.50 usec
TE 294.4 K
D1 1.00000000 sec
TD0 1
SFO1 400.1324708 MHz
NUC1 1H
P1 10.00 usec
PLW1 16.43099976 W
F2 - Processing parameters
SI 65536
SF 400.1300098 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
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 ppm
1.00
2.04



5y

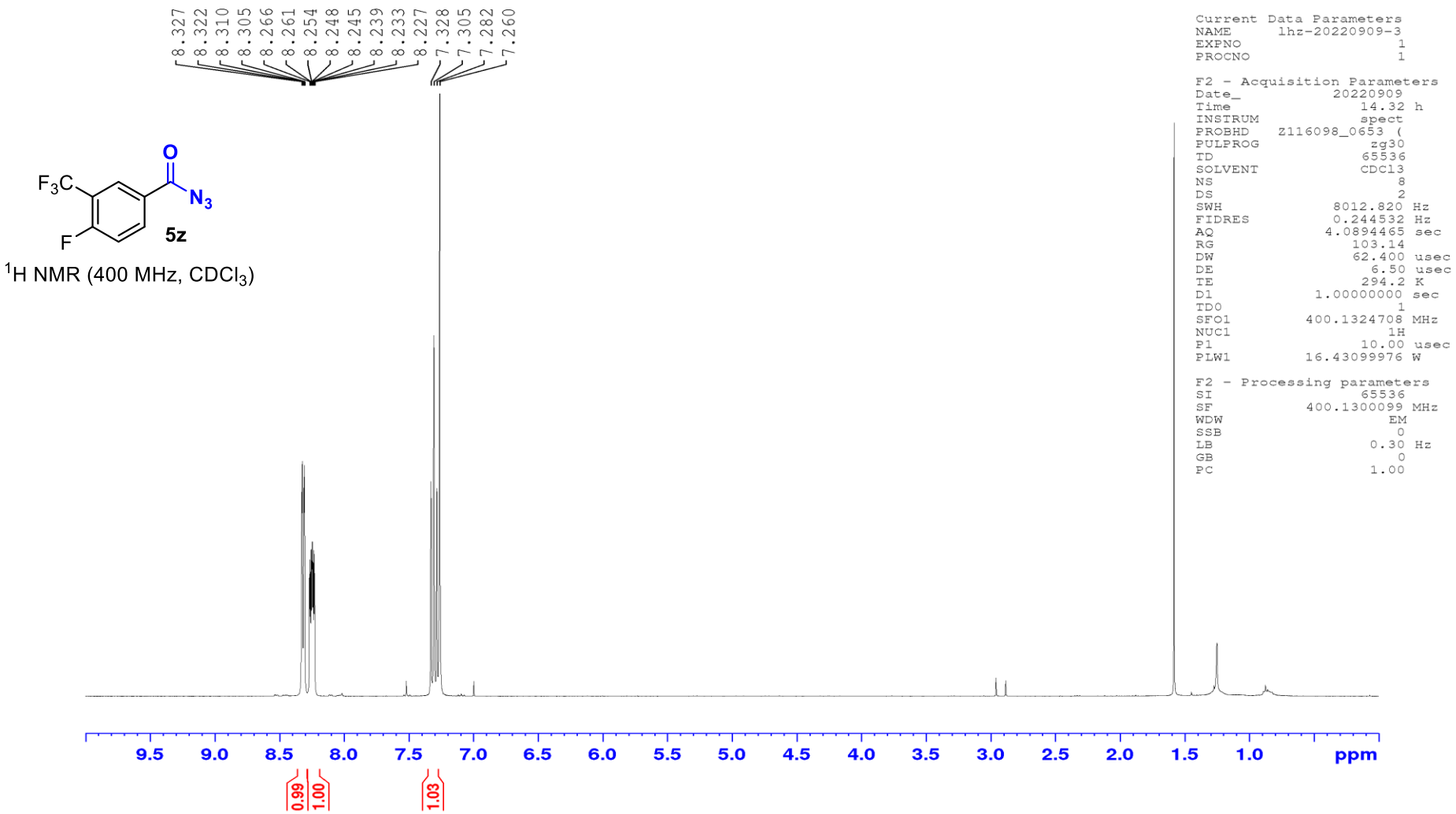
^{13}C NMR (100 MHz, $CDCl_3$)




```
Current Data Parameters
NAME      1hz-20220909-2
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20220909
Time               20.58 h
INSTRUM            spect
PROBHD   Z116098_0653 (
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                 3333
DS                    4
SWH           24038.461 Hz
FIDRES         0.733596 Hz
AQ             1.3631488 sec
RG                198.36
DW                20.800 usec
DE                 6.50 usec
TE                294.7 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                    1
SFO1        100.6228298 MHz
NUC1                13C
P1                10.00 usec
PLW1        75.84400177 W
SFO2        400.1316005 MHz
NUC2                1H
CPDPRG[2        waltz16
PCPD2             90.00 usec
PLW2        16.43099976 W
PLW12        0.20286000 W
PLW13        0.10204000 W

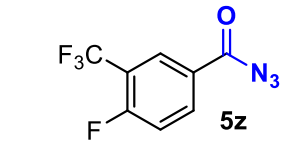
F2 - Processing parameters
SI                32768
SF          100.6127572 MHz
WDW                EM
SSB                 0
LB                1.00 Hz
GB                  0
PC                1.40
```



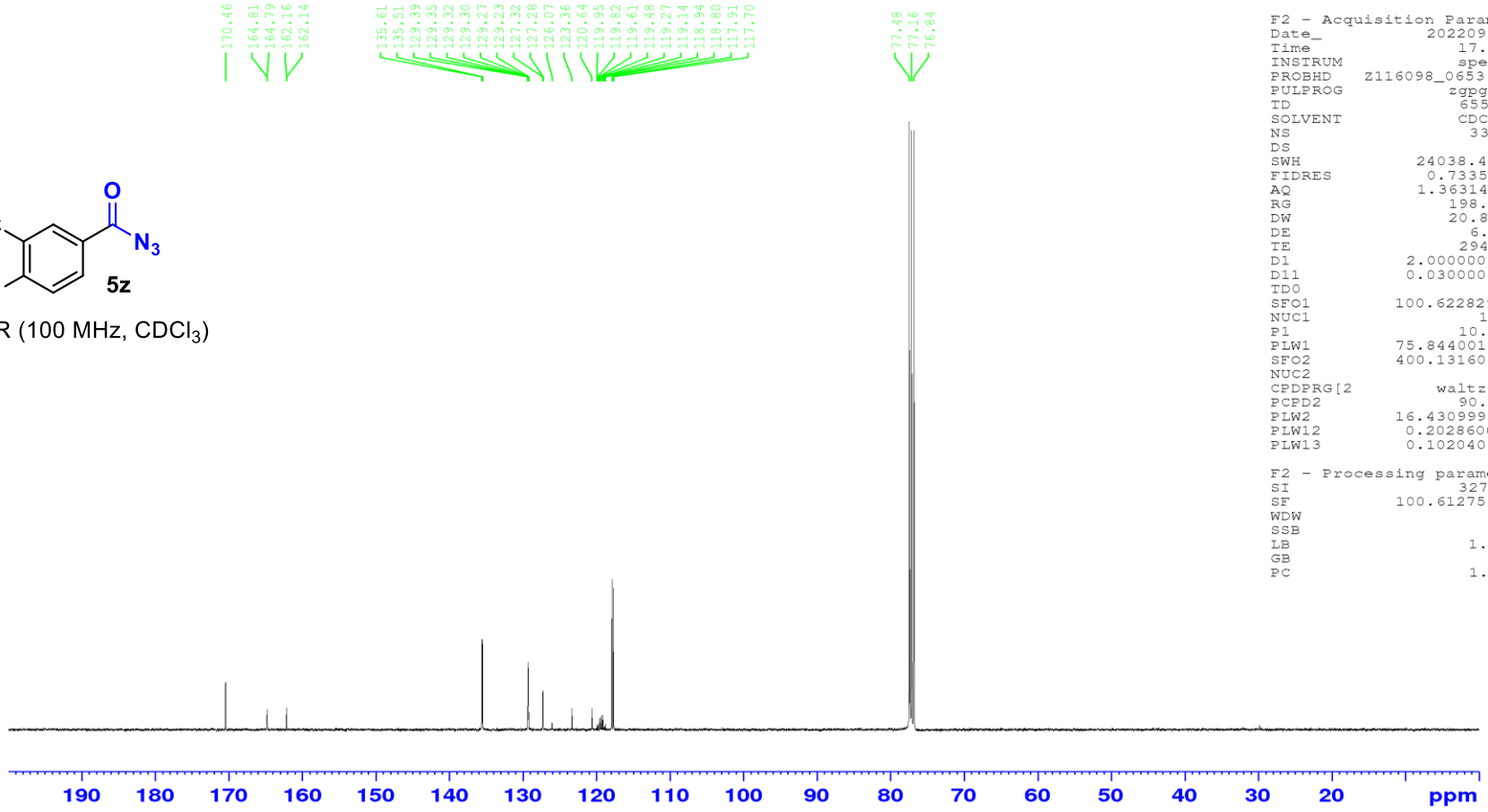

```
Current Data Parameters
NAME      1hz-20220909-3
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20220909
Time              14.32 h
INSTRUM             spect
PROBHD    Z116098_0653 (
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                     8
DS                     2
SWH             8012.820 Hz
FIDRES          0.244532 Hz
AQ              4.0894465 sec
RG                 103.14
DW                 62.400 usec
DE                   6.50 usec
TE                  294.2 K
D1             1.00000000 sec
TD0                    1
SFO1          400.1324708 MHz
NUC1                  1H
P1                 10.00 usec
PLW1          16.43099976 W

F2 - Processing parameters
SI                 65536
SF            400.1300099 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```



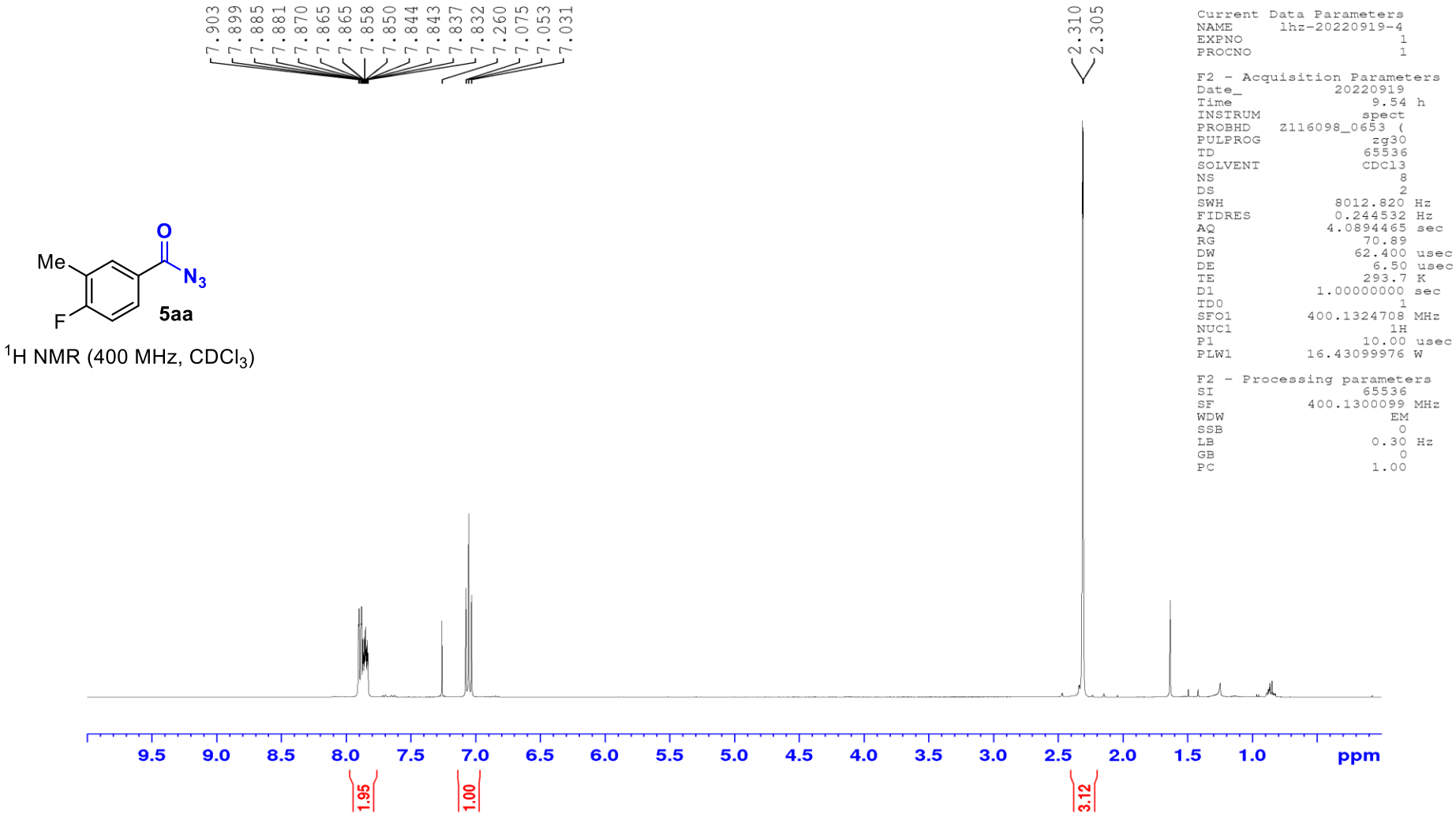
^{13}C NMR (100 MHz, $CDCl_3$)

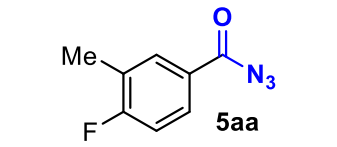



```
Current Data Parameters
NAME      lhz-20220909-3
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20220909
Time              17.43 h
INSTRUM             spect
PROBHD    Z116098_0653 (
PULPROG           zgpg30
TD                65536
SOLVENT           CDCl3
NS                 3333
DS                    4
SWH           24038.461 Hz
FIDRES         0.733596 Hz
AQ             1.3631488 sec
RG               198.36
DW               20.800 usec
DE                6.50 usec
TE                294.8 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1
SFO1        100.6228298 MHz
NUC1                13C
P1                10.00 usec
PLW1        75.84400177 W
SFO2        400.1316005 MHz
NUC2                1H
CPDPRG[2        waltz16
PCPD2             90.00 usec
PLW2        16.43099976 W
PLW12        0.20286000 W
PLW13        0.10204000 W

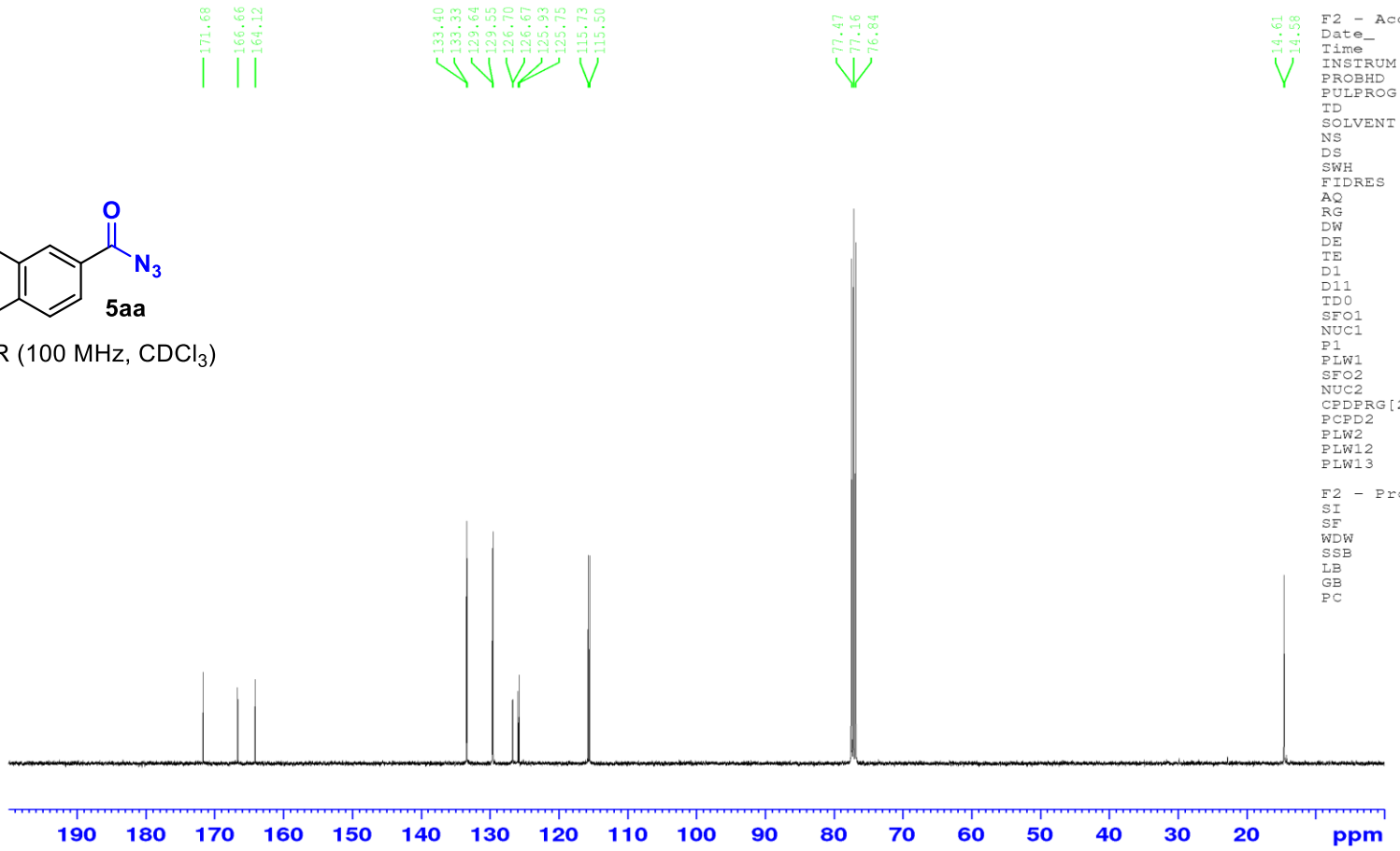
F2 - Processing parameters
SI                32768
SF          100.6127550 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```


7.903
7.899
7.885
7.881
7.870
7.865
7.865
7.858
7.850
7.844
7.843
7.837
7.832
7.260
7.075
7.053
7.031
2.310
2.305
Me
O
N3
F
5aa
1H NMR (400 MHz, CDCl3)
Current Data Parameters
NAME lhz-20220919-4
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20220919
Time 9.54 h
INSTRUM spect
PROBHD Z116098_0653 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 8012.820 Hz
FIDRES 0.244532 Hz
AQ 4.0894465 sec
RG 70.89
DW 62.400 usec
DE 6.50 usec
TE 293.7 K
D1 1.00000000 sec
TD0 1
SFO1 400.1324708 MHz
NUC1 1H
P1 10.00 usec
PLW1 16.43099976 W
F2 - Processing parameters
SI 65536
SF 400.1300099 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
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
ppm
1.95
1.00
3.12



5aa

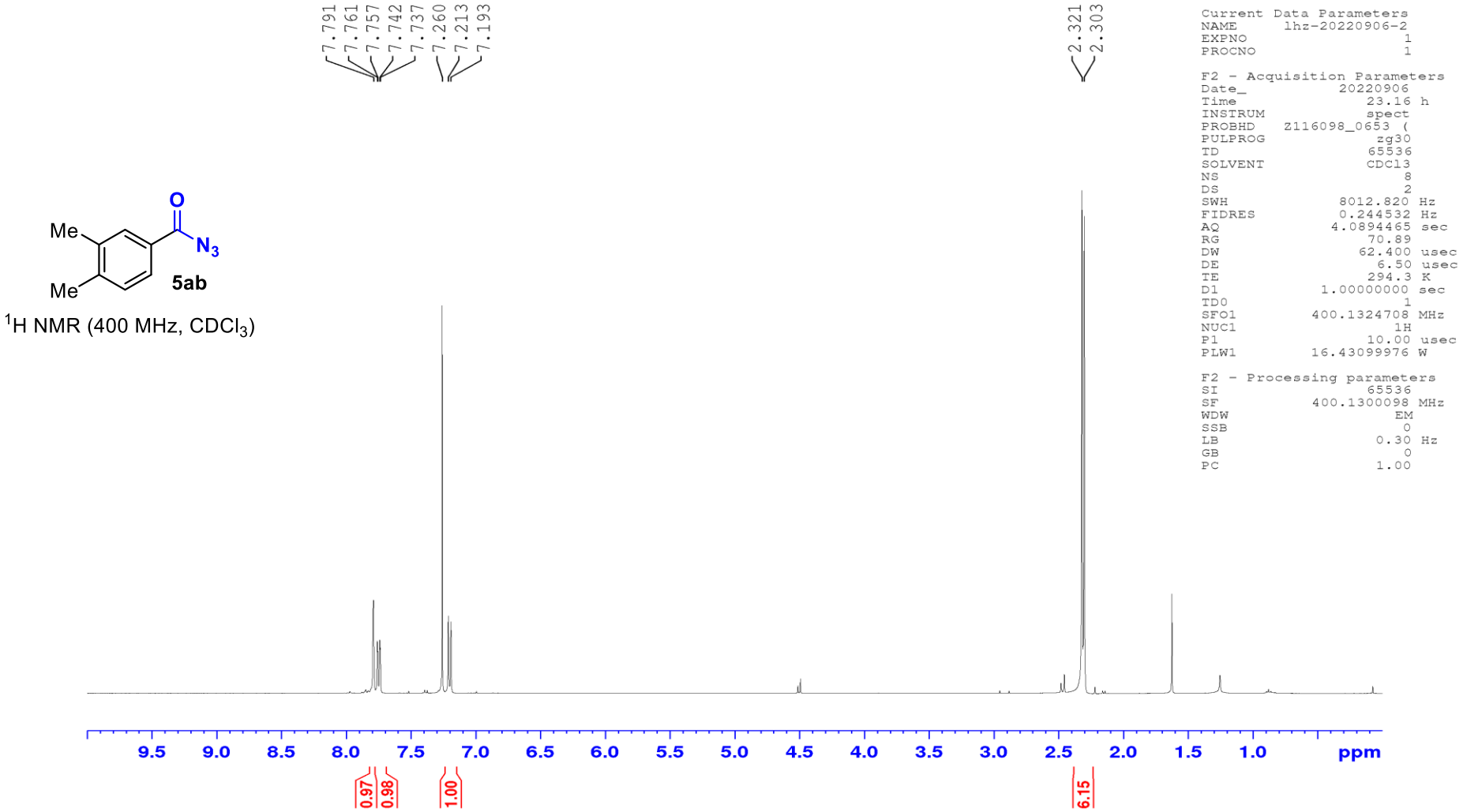
^{13}C NMR (100 MHz, $CDCl_3$)

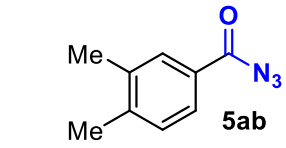



```
Current Data Parameters
NAME      lhz-20220919-4
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20220919
Time              11.13 h
INSTRUM             spect
PROBHD    Z116098_0653 (
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  888
DS                    4
SWH           24038.461 Hz
FIDRES         0.733596 Hz
AQ             1.3631488 sec
RG                198.36
DW                20.800 usec
DE                 6.50 usec
TE                294.5 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1
SFO1        100.6228298 MHz
NUC1                13C
P1                10.00 usec
PLW1        75.84400177 W
SFO2        400.1316005 MHz
NUC2                1H
CPDPRG[2        waltz16
PCPD2             90.00 usec
PLW2        16.43099976 W
PLW12        0.20286000 W
PLW13        0.10204000 W

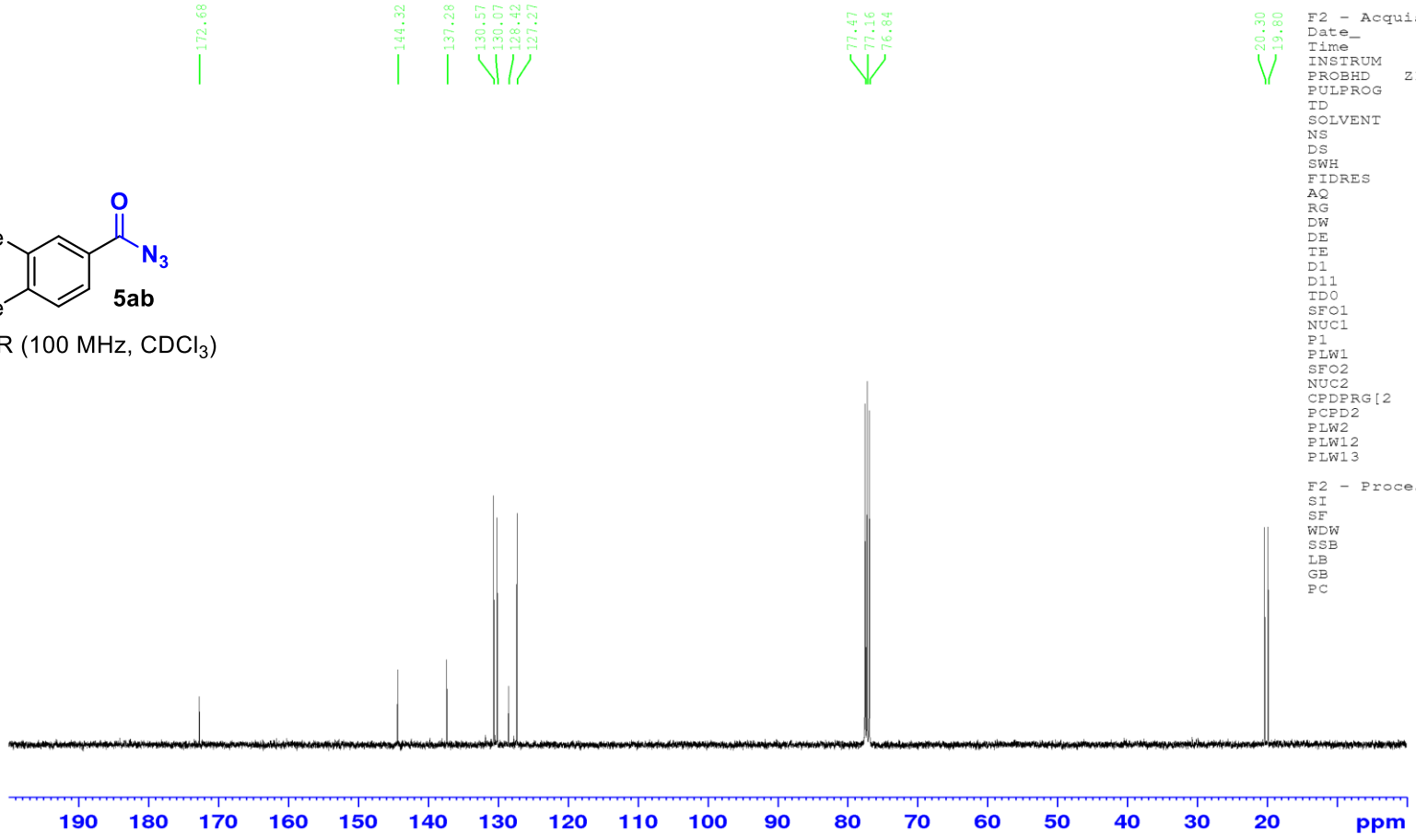
F2 - Processing parameters
SI                32768
SF          100.6127571 MHz
WDW                EM
SSB                 0
LB                1.00 Hz
GB                  0
PC                1.40
```


7.791
7.761
7.757
7.742
7.737
7.260
7.213
7.193
2.321
2.303
Current Data Parameters
NAME 1hz-20220906-2
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20220906
Time 23.16 h
INSTRUM spect
PROBHD Z116098_0653 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 8012.820 Hz
FIDRES 0.244532 Hz
AQ 4.0894465 sec
RG 70.89
DW 62.400 usec
DE 6.50 usec
TE 294.3 K
D1 1.00000000 sec
TD0 1
SFO1 400.1324708 MHz
NUC1 1H
P1 10.00 usec
PLW1 16.43099976 W
F2 - Processing parameters
SI 65536
SF 400.1300098 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
O
N3
Me
Me
5ab
1H NMR (400 MHz, CDCl3)
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 ppm
0.97
0.98
1.00
6.15



5ab

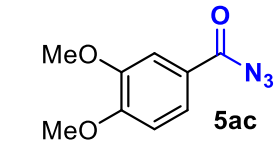
^{13}C NMR (100 MHz, $CDCl_3$)




```
Current Data Parameters
NAME      lhz-20220906-2
EXPNO                  2
PROCNO                 1

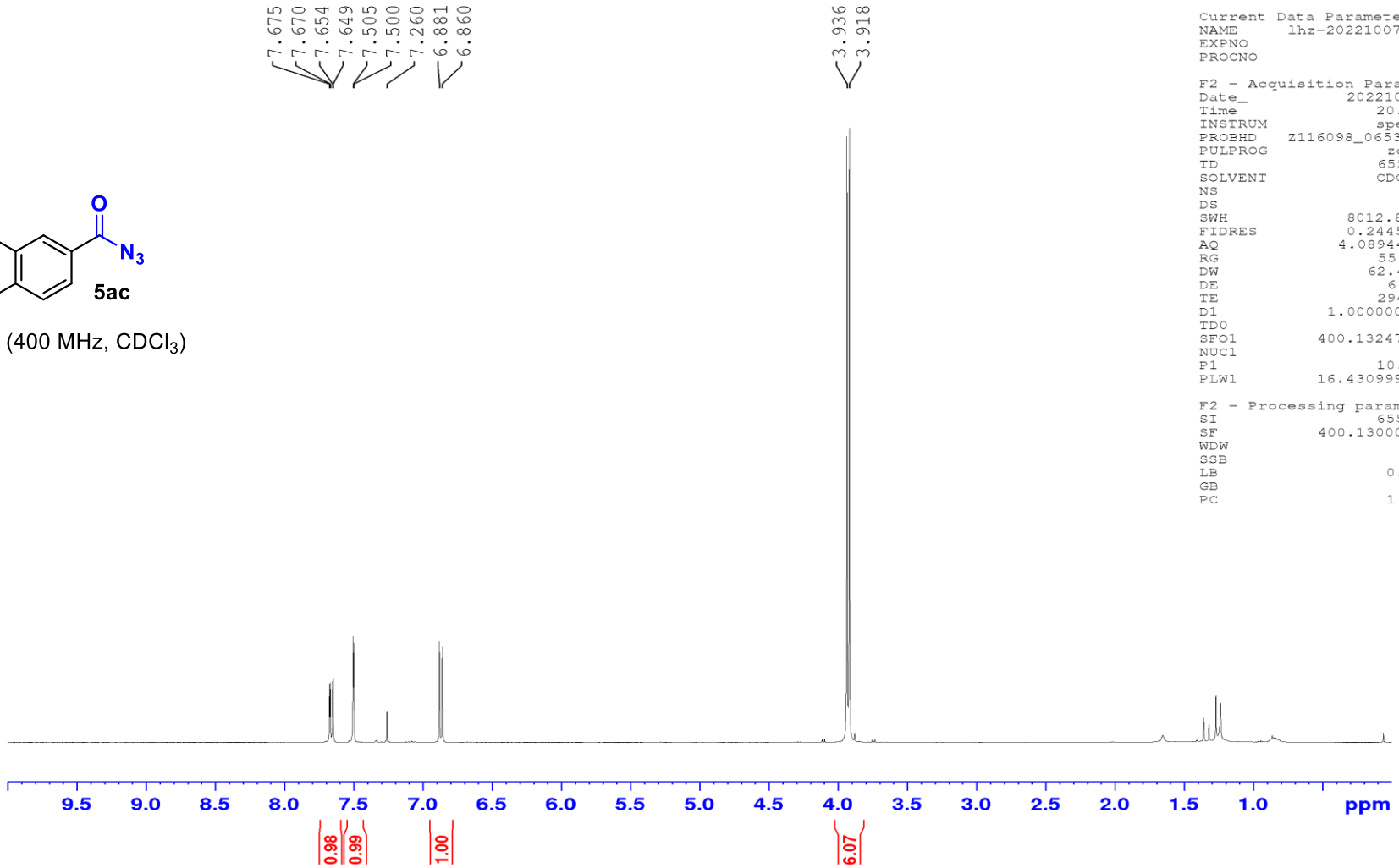
F2 - Acquisition Parameters
Date_           20220906
Time              23.27 h
INSTRUM             spect
PROBHD    Z116098_0653 (
PULPROG           zgpg30
TD                65536
SOLVENT           CDCl3
NS                  169
DS                    4
SWH           24038.461 Hz
FIDRES         0.733596 Hz
AQ             1.3631488 sec
RG                198.36
DW                20.800 usec
DE                 6.50 usec
TE                295.0 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1
SFO1        100.6228298 MHz
NUC1                13C
P1                10.00 usec
PLW1        75.84400177 W
SFO2        400.1316005 MHz
NUC2                1H
CPDPRG[2         waltz16
PCPD2             90.00 usec
PLW2        16.43099976 W
PLW12        0.20286000 W
PLW13        0.10204000 W

F2 - Processing parameters
SI                32768
SF          100.6127579 MHz
WDW                 EM
SSB                  0
LB                1.00 Hz
GB                   0
PC                1.40
```



5ac

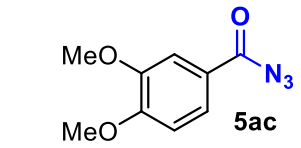
^{1}H NMR (400 MHz, $CDCl_3$)




```
Current Data Parameters
NAME      1hz-20221007-2
EXPNO                  1
PROCNO                 1

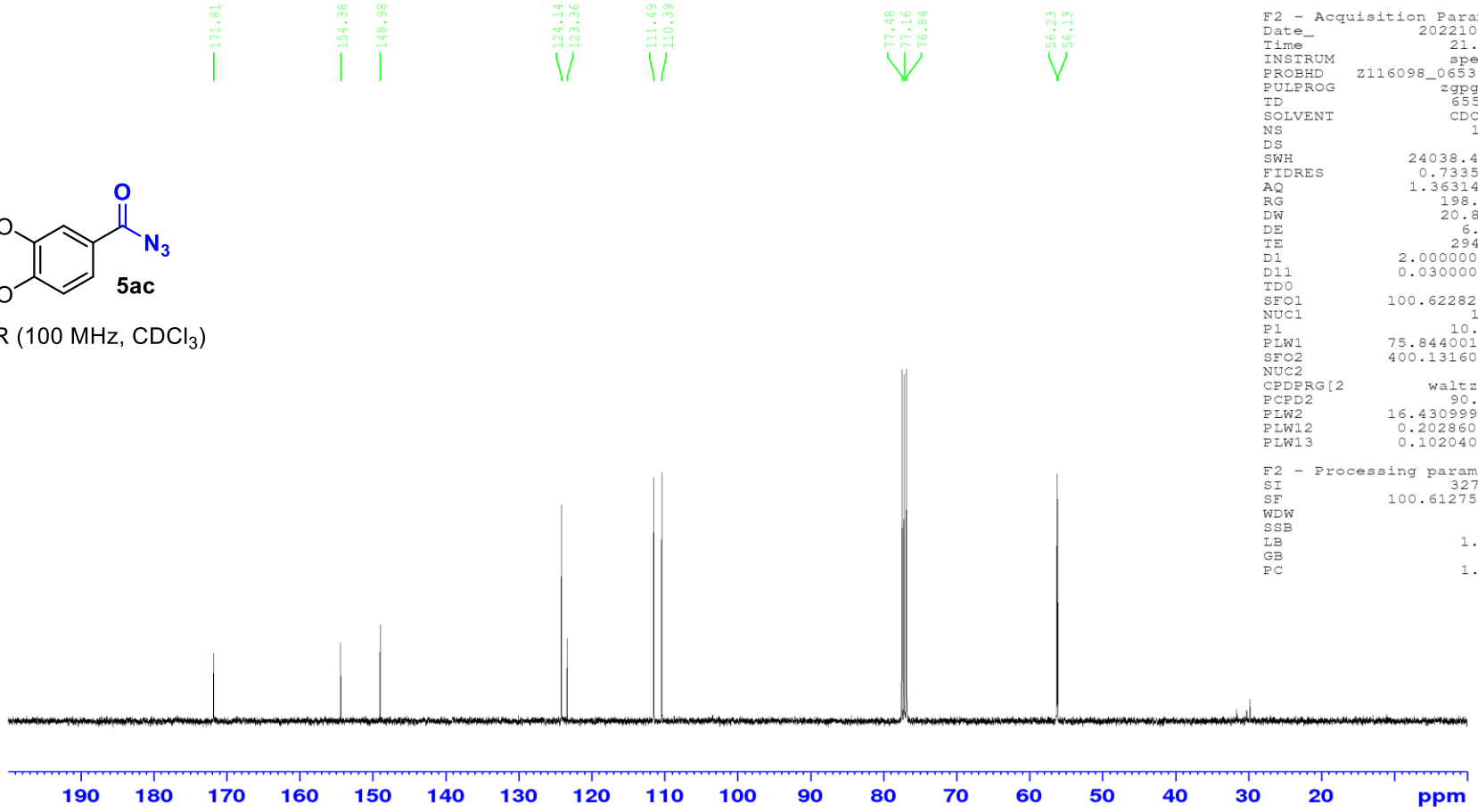
F2 - Acquisition Parameters
Date_           20221007
Time              20.54 h
INSTRUM             spect
PROBHD   Z116098_0653 (
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             8012.820 Hz
FIDRES          0.244532 Hz
AQ              4.0894465 sec
RG                 55.99
DW                62.400 usec
DE                 6.50 usec
TE                294.1 K
D1             1.00000000 sec
TD0                    1
SFO1          400.1324708 MHz
NUC1                  1H
P1                10.00 usec
PLW1          16.43099976 W

F2 - Processing parameters
SI                 65536
SF            400.1300093 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```



5ac

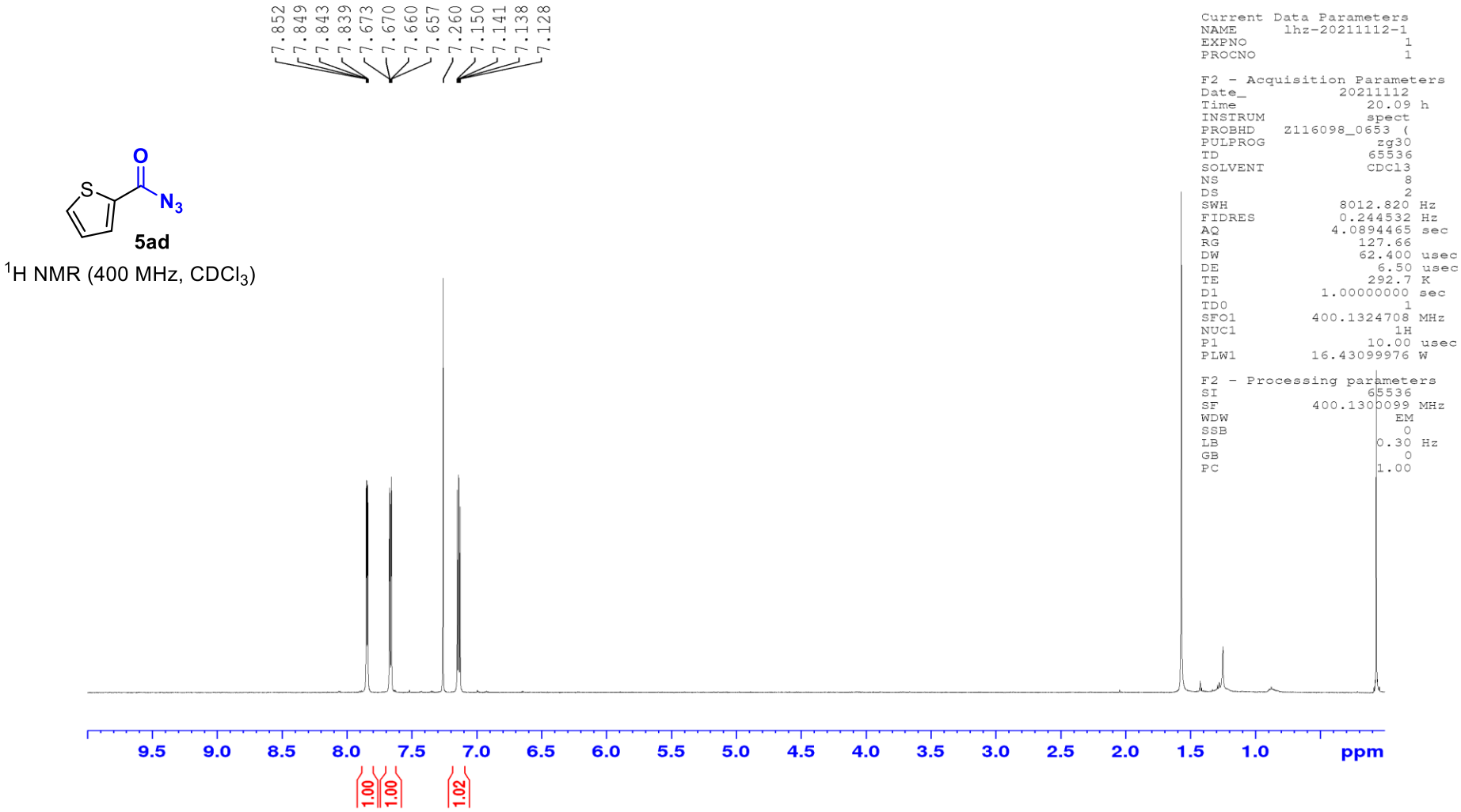
^{13}C NMR (100 MHz, $CDCl_3$)

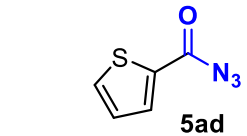



```
Current Data Parameters
NAME      lhz-20221007-2
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20221007
Time              21.01 h
INSTRUM             spect
PROBHD    Z116098_0653 (
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  108
DS                    4
SWH           24038.461 Hz
FIDRES         0.733596 Hz
AQ             1.3631488 sec
RG                198.36
DW                20.800 usec
DE                 6.50 usec
TE                294.5 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                    1
SFO1          100.6228298 MHz
NUC1                 13C
P1                 10.00 usec
PLW1          75.84400177 W
SFO2          400.1316005 MHz
NUC2                 1H
CPDPRG[2         waltz16
PCPD2              90.00 usec
PLW2          16.43099976 W
PLW12          0.20286000 W
PLW13          0.10204000 W

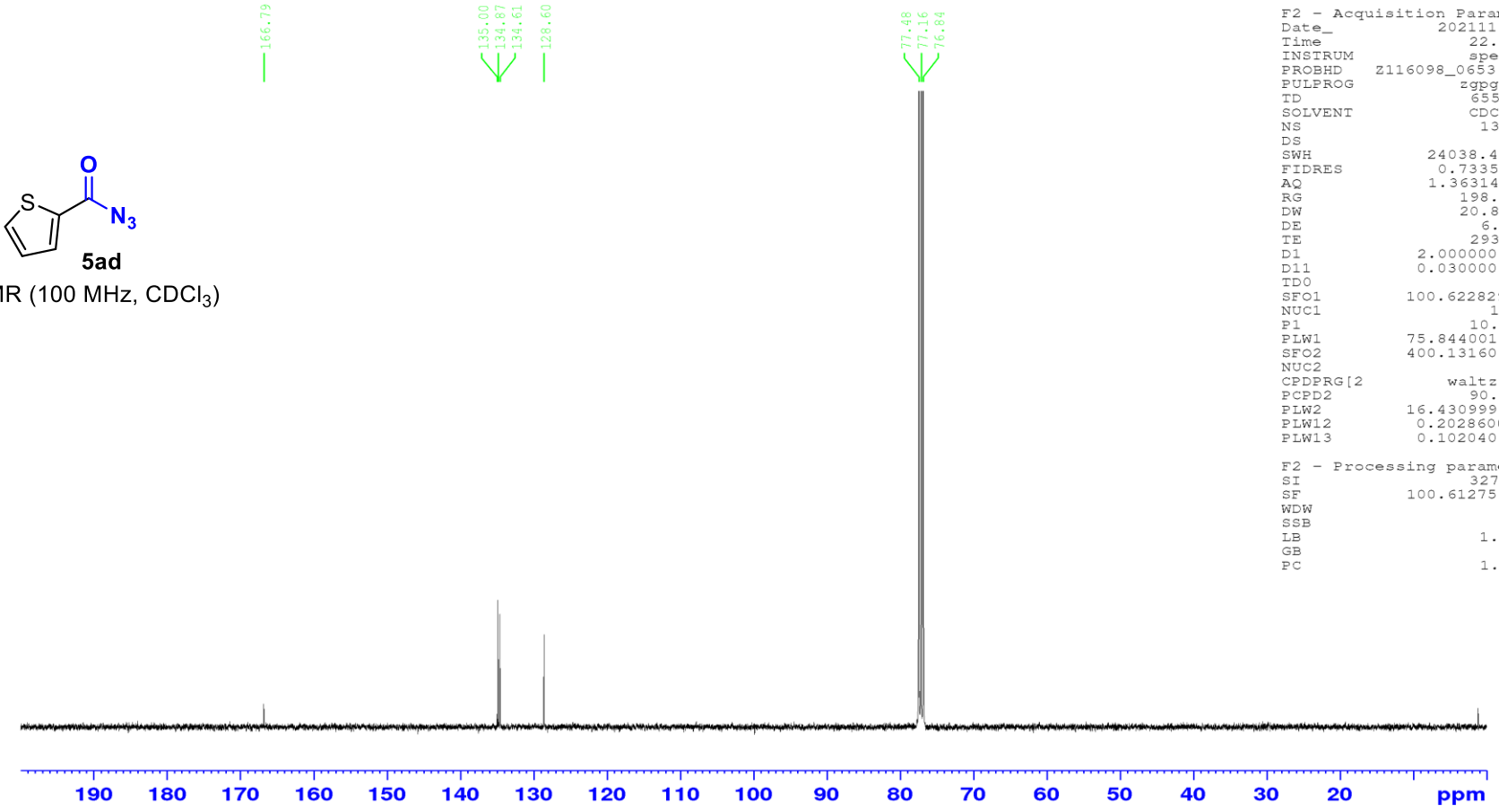
F2 - Processing parameters
SI                 32768
SF            100.6127594 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```


7.852
7.849
7.843
7.839
7.673
7.670
7.660
7.657
7.260
7.150
7.141
7.138
7.128
O
S
N3
5ad
1H NMR (400 MHz, CDCl3)
Current Data Parameters
NAME lhz-20211112-1
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20211112
Time 20.09 h
INSTRUM spect
PROBHD Z116098_0653 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 8012.820 Hz
FIDRES 0.244532 Hz
AQ 4.0894465 sec
RG 127.66
DW 62.400 usec
DE 6.50 usec
TE 292.7 K
D1 1.00000000 sec
TD0 1
SFO1 400.1324708 MHz
NUC1 1H
P1 10.00 usec
PLW1 16.43099976 W
F2 - Processing parameters
SI 65536
SF 400.1300099 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
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 ppm
1.00
1.00
1.02



5ad

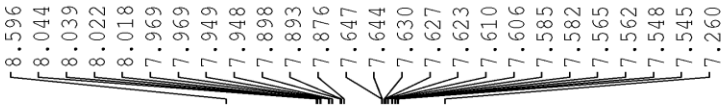
^{13}C NMR (100 MHz, $CDCl_3$)

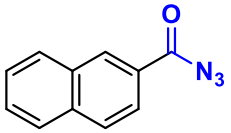



```
Current Data Parameters
NAME      1hz-20211112-1
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20211112
Time              22.00 h
INSTRUM             spect
PROBHD   Z116098_0653 (
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                 1333
DS                    4
SWH           24038.461 Hz
FIDRES         0.733596 Hz
AQ             1.3631488 sec
RG                198.36
DW                20.800 usec
DE                 6.50 usec
TE                293.0 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1
SFO1        100.6228298 MHz
NUC1                13C
P1                10.00 usec
PLW1        75.84400177 W
SFO2        400.1316005 MHz
NUC2                1H
CPDPRG[2        waltz16
PCPD2             90.00 usec
PLW2        16.43099976 W
PLW12        0.20286000 W
PLW13        0.10204000 W

F2 - Processing parameters
SI                32768
SF          100.6127564 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```





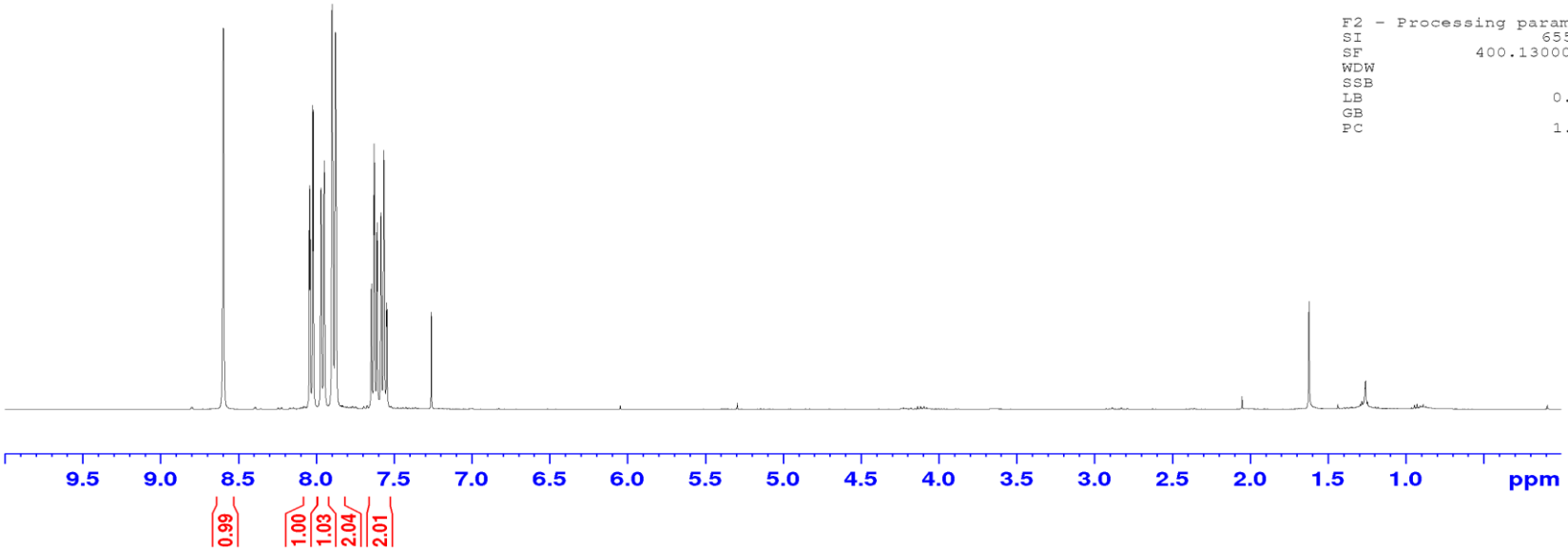
5ae

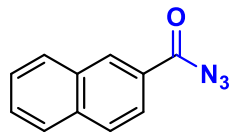
^{1}H NMR (400 MHz, $CDCl_3$)

```
Current Data Parameters
NAME      1hz-20211112-2
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20211112
Time               20.13 h
INSTRUM            spect
PROBHD    Z116098_0653 (
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                    8
DS                    2
SWH            8012.820 Hz
FIDRES         0.244532 Hz
AQ             4.0894465 sec
RG                70.89
DW                62.400 usec
DE                 6.50 usec
TE                292.5 K
D1             1.00000000 sec
TD0                   1
SFO1         400.1324708 MHz
NUC1                 1H
P1                10.00 usec
PLW1         16.43099976 W

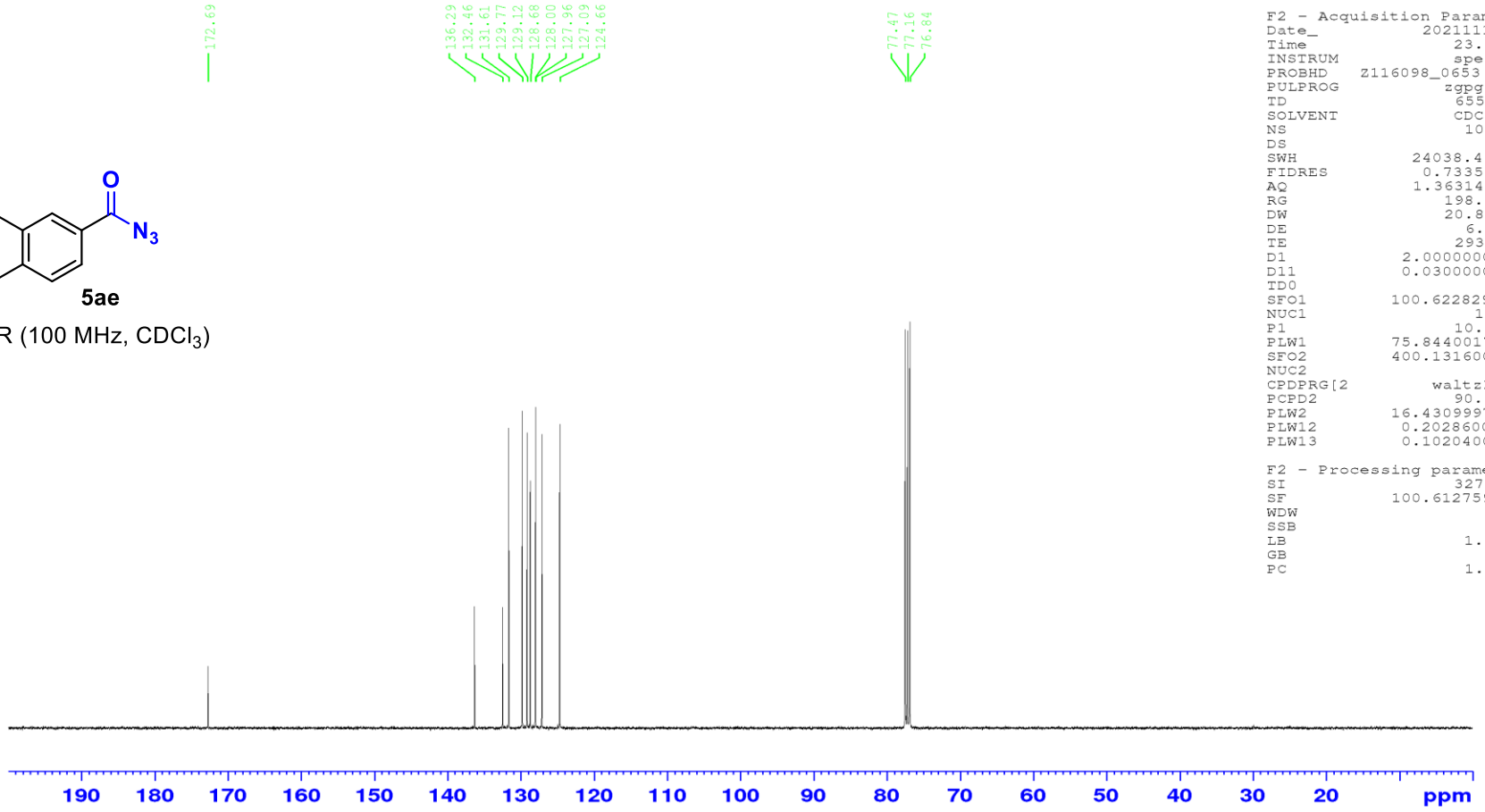
F2 - Processing parameters
SI                65536
SF           400.1300098 MHz
WDW                  EM
SSB                   0
LB                 0.30 Hz
GB                    0
PC                 1.00
```





5ae

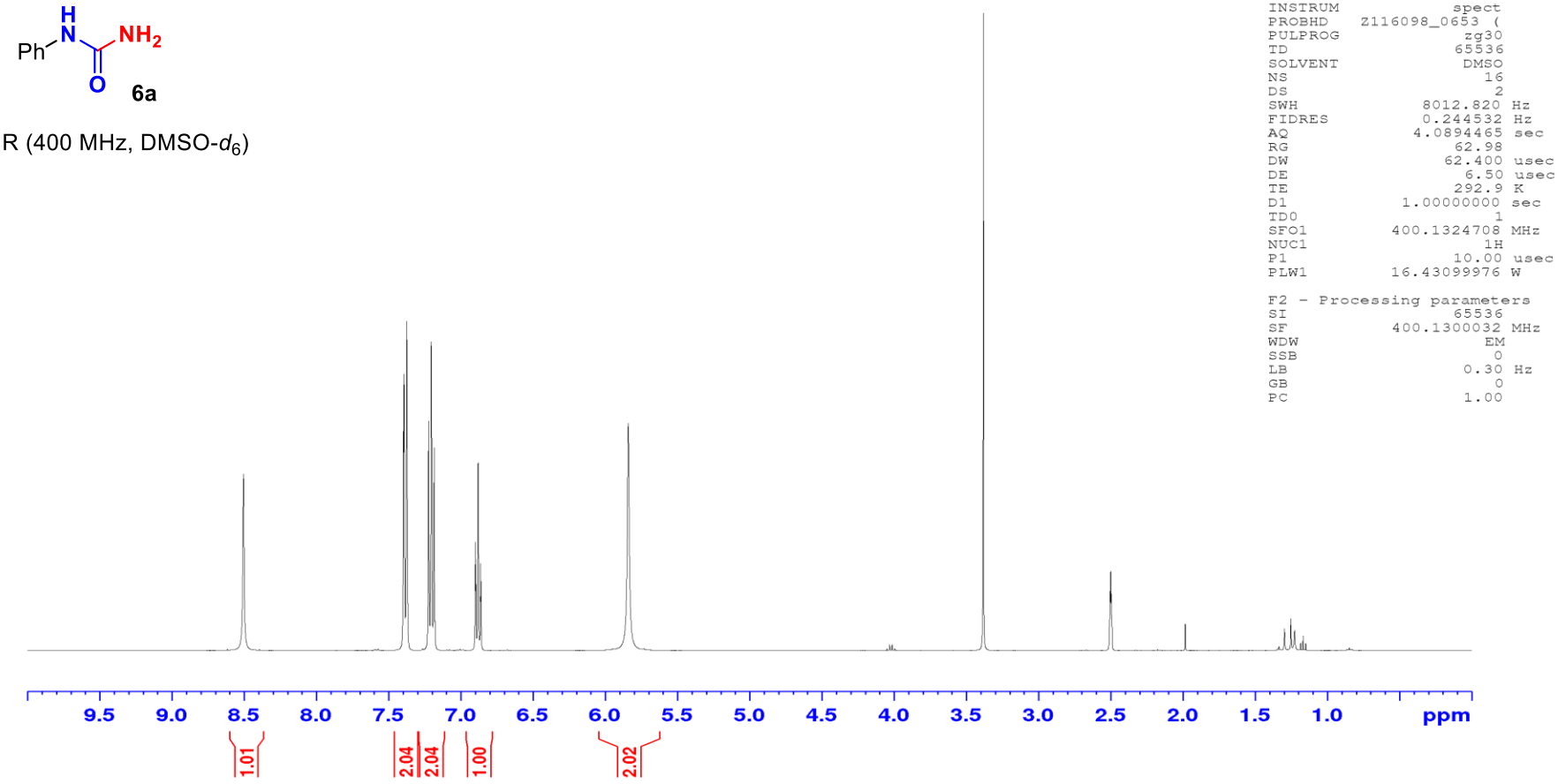
^{13}C NMR (100 MHz, $CDCl_3$)



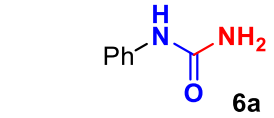

```
Current Data Parameters
NAME      lhz-20211112-2
EXPNO                   2
PROCNO                  1

F2 - Acquisition Parameters
Date_            20211112
Time                23.02 h
INSTRUM              spect
PROBHD    Z116098_0653 (
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                  1024
DS                     4
SWH            24038.461 Hz
FIDRES          0.733596 Hz
AQ             1.3631488 sec
RG                198.36
DW                20.800 usec
DE                 6.50 usec
TE                293.1 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                    1
SFO1         100.6228298 MHz
NUC1                13C
P1                10.00 usec
PLW1         75.84400177 W
SFO2         400.1316005 MHz
NUC2                1H
CPDPRG[2         waltz16
PCPD2             90.00 usec
PLW2         16.43099976 W
PLW12         0.20286000 W
PLW13         0.10204000 W

F2 - Processing parameters
SI                 32768
SF           100.6127597 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```

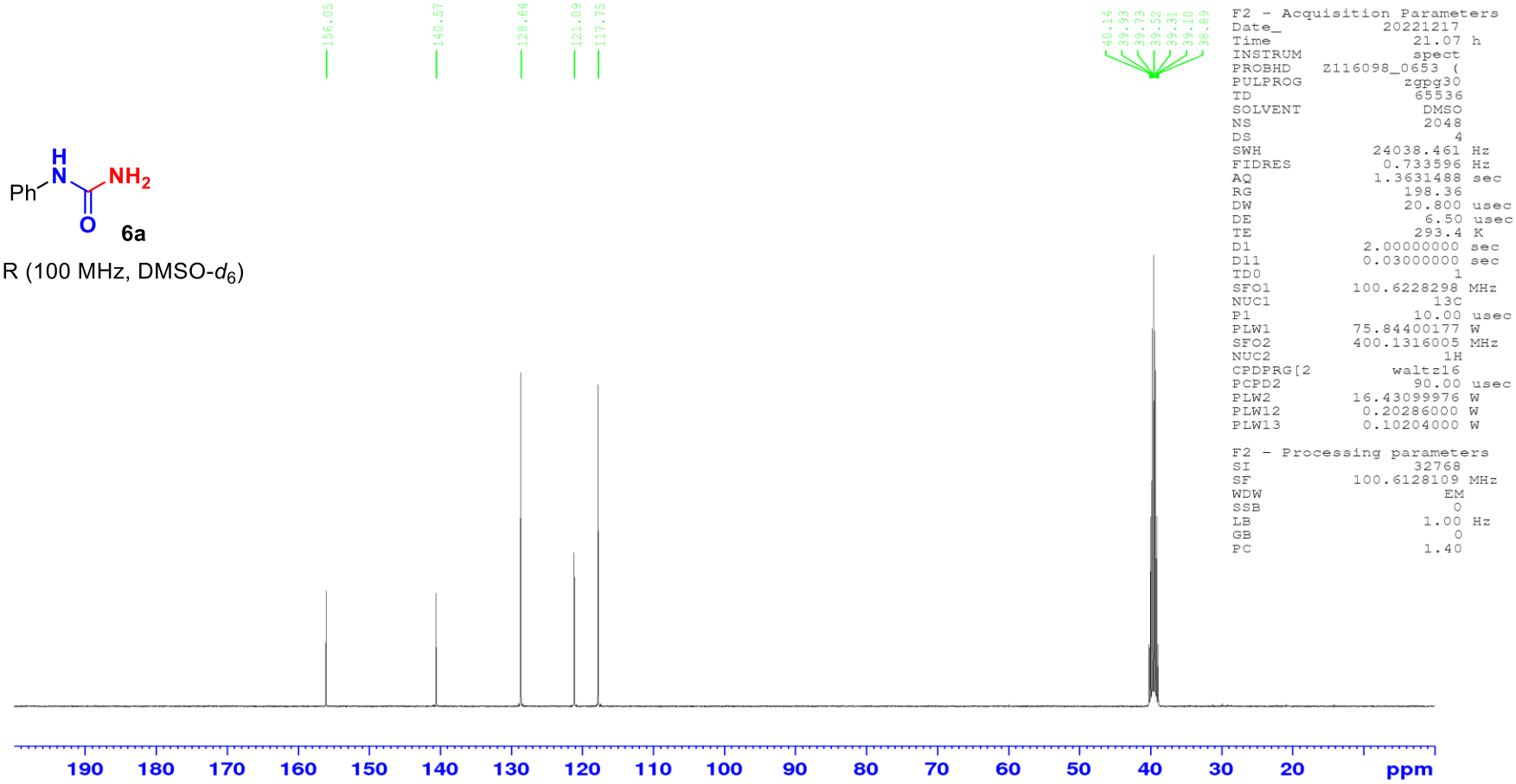


^{1}H NMR (400 MHz, DMSO-d_6)



6a

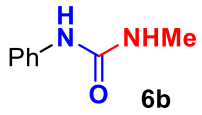
^{13}C NMR (100 MHz, DMSO-d_6)




```
Current Data Parameters
NAME      lyr-20221217-1
EXPNO                  2
PROCNO                 1

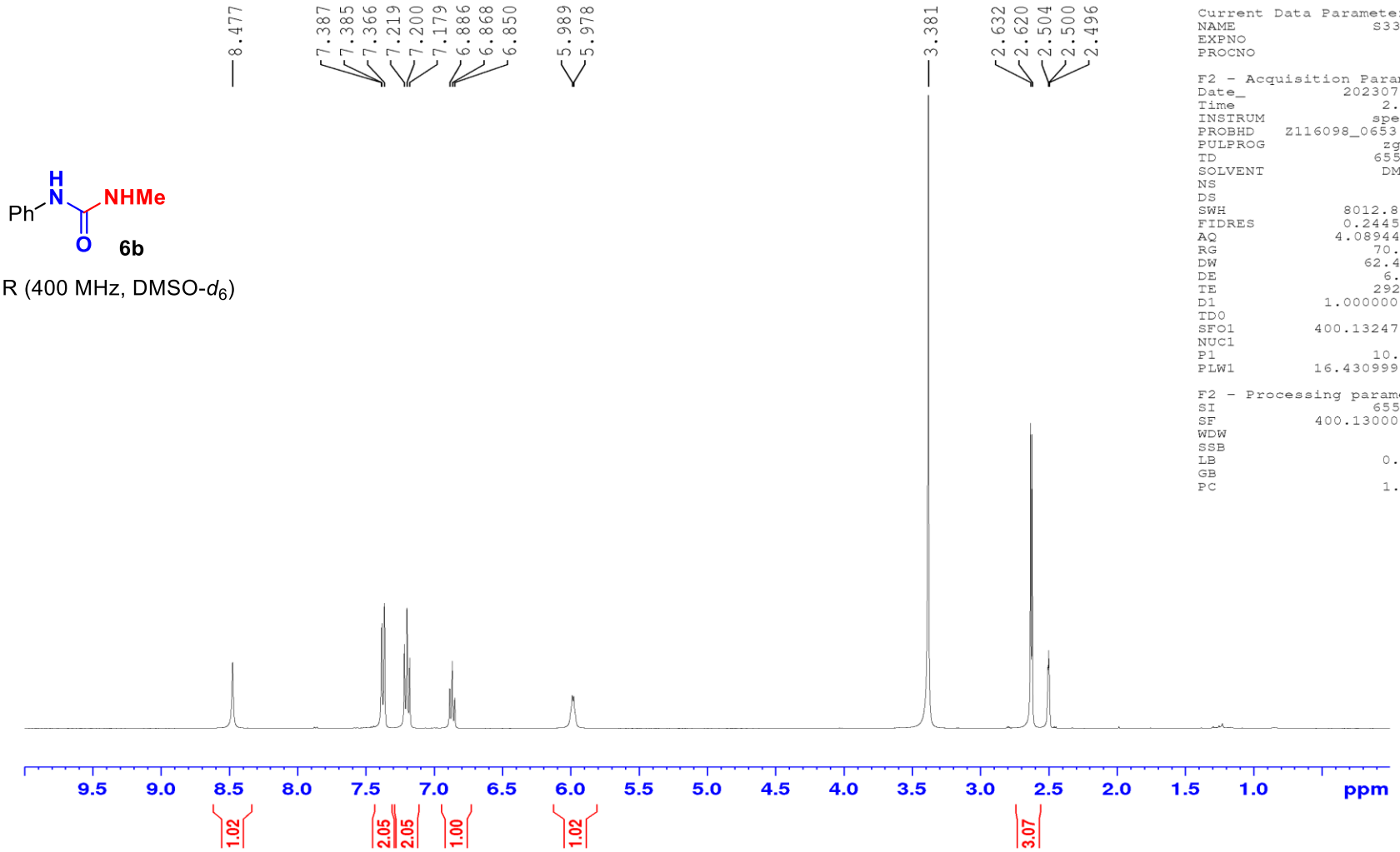
F2 - Acquisition Parameters
Date_           20221217
Time              21.07 h
INSTRUM             spect
PROBHD    Z116098_0653 (
PULPROG          zgpg30
TD                65536
SOLVENT            DMSO
NS                 2048
DS                    4
SWH           24038.461 Hz
FIDRES         0.733596 Hz
AQ            1.3631488 sec
RG               198.36
DW               20.800 usec
DE                6.50 usec
TE                293.4 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1
SFO1        100.6228298 MHz
NUC1                13C
P1                10.00 usec
PLW1        75.84400177 W
SFO2        400.1316005 MHz
NUC2                1H
CPDPRG[2        waltz16
PCPD2             90.00 usec
PLW2        16.43099976 W
PLW12        0.20286000 W
PLW13        0.10204000 W

F2 - Processing parameters
SI                32768
SF          100.6128109 MHz
WDW                EM
SSB                 0
LB               1.00 Hz
GB                  0
PC               1.40
```



6b

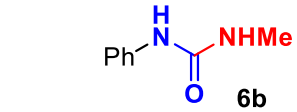
^{1}H NMR (400 MHz, DMSO-d_6)




```
Current Data Parameters
NAME                  S33-1
EXPNO                     1
PROCNO                    1

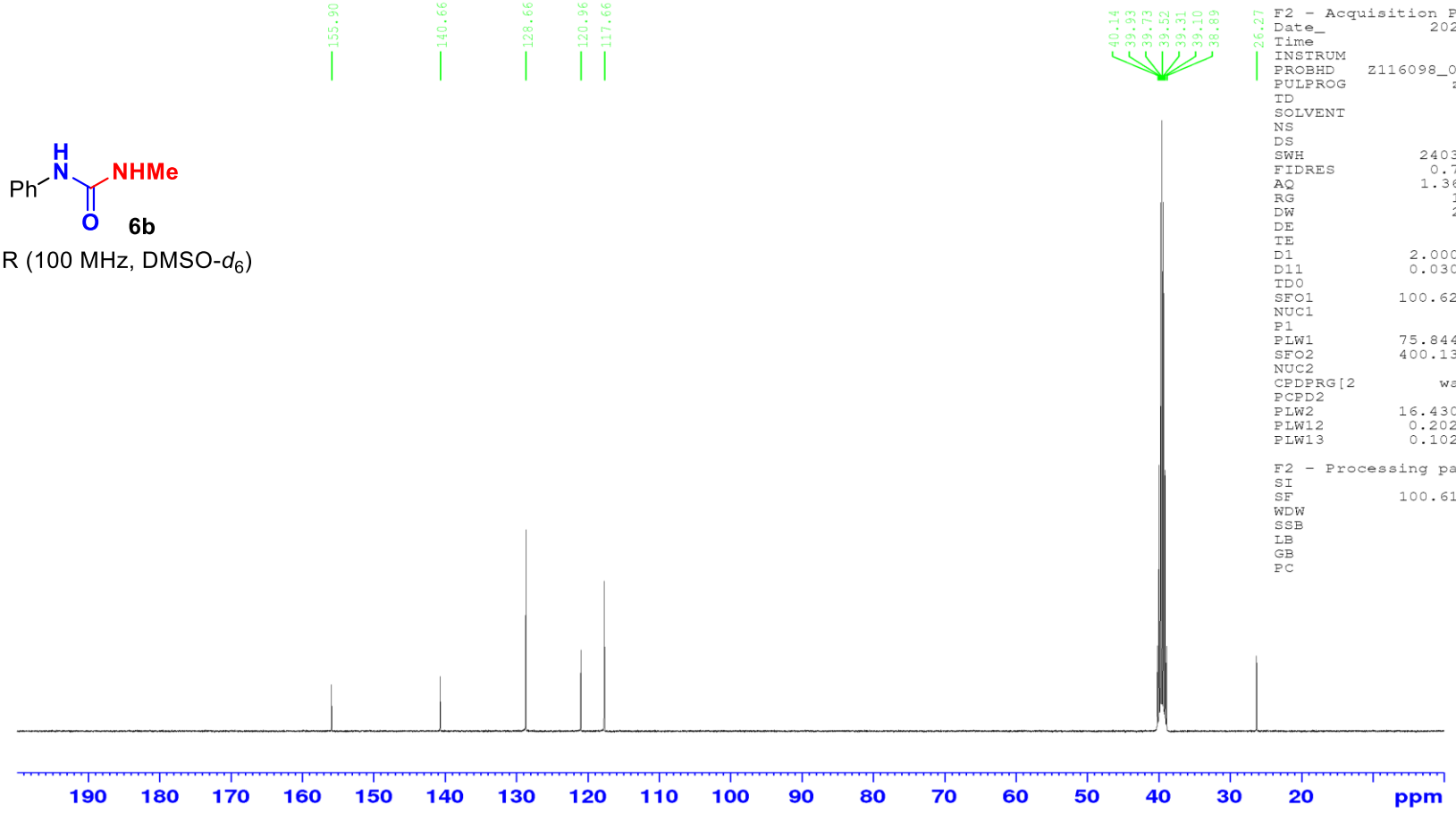
F2 - Acquisition Parameters
Date_              20230708
Time                   2.04 h
INSTRUM               spect
PROBHD    Z116098_0653 (
PULPROG                zg30
TD                    65536
SOLVENT                DMSO
NS                       16
DS                        2
SWH                8012.820 Hz
FIDRES                0.244532 Hz
AQ                 4.0894465 sec
RG                    70.89
DW                    62.400 usec
DE                     6.50 usec
TE                    292.4 K
D1                 1.00000000 sec
TD0                       1
SFO1           400.1324708 MHz
NUC1                     1H
P1                    10.00 usec
PLW1           16.43099976 W

F2 - Processing parameters
SI                    65536
SF             400.1300031 MHz
WDW                      EM
SSB                       0
LB                     0.30 Hz
GB                        0
PC                     1.00
```



6b

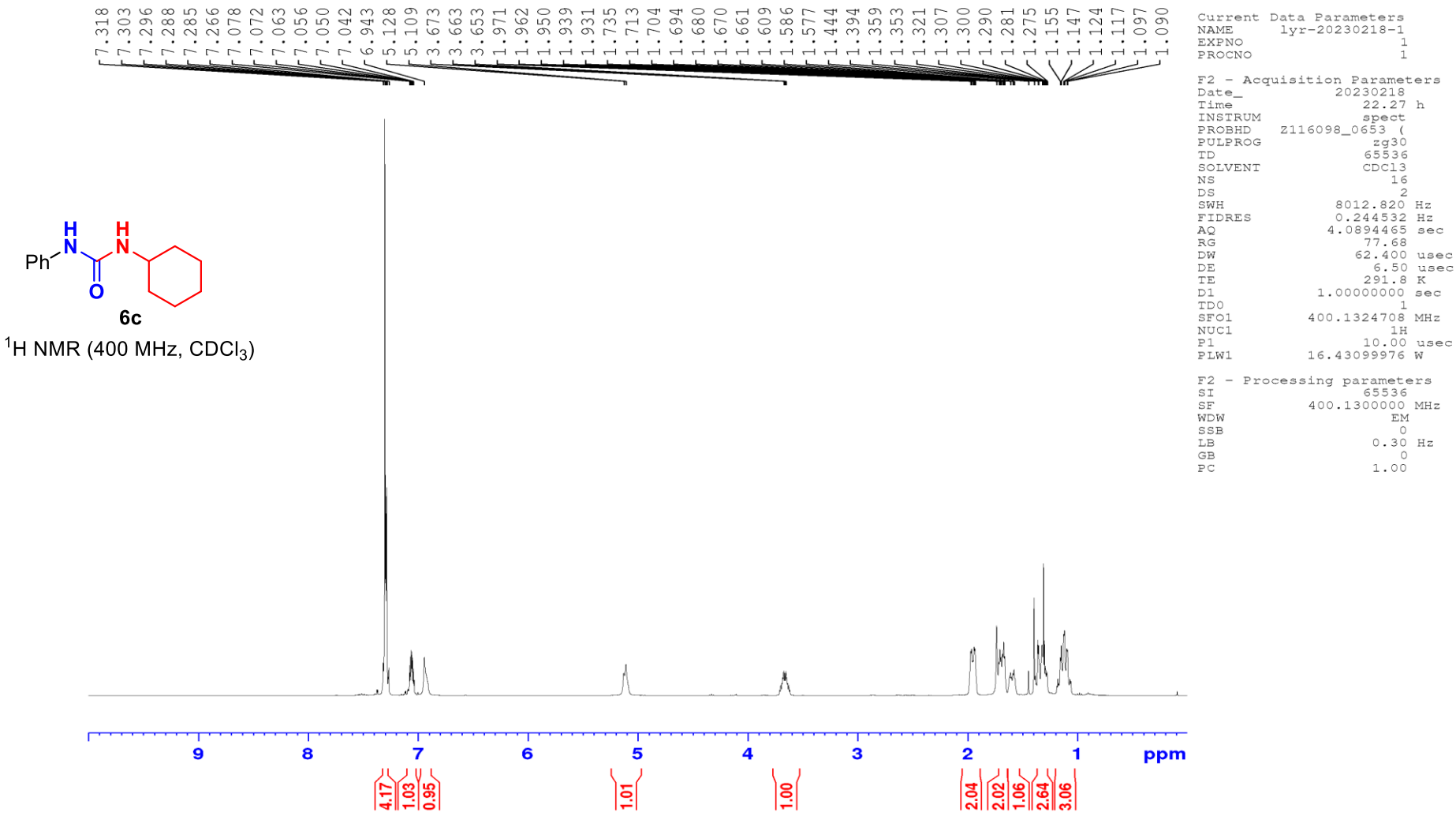
^{13}C NMR (100 MHz, DMSO-d_6)




```
Current Data Parameters
NAME                  S33-1
EXPNO                     2
PROCNO                    1

F2 - Acquisition Parameters
Date_              20230708
Time                 4.19 h
INSTRUM              spect
PROBHD    Z116098_0653 (
PULPROG            zgpg30
TD                  65536
SOLVENT              DMSO
NS                    2333
DS                       4
SWH            24038.461 Hz
FIDRES          0.733596 Hz
AQ             1.3631488 sec
RG                  198.36
DW                20.800 usec
DE                 6.50 usec
TE                293.1 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                      1
SFO1         100.6228298 MHz
NUC1                  13C
P1                 10.00 usec
PLW1         75.84400177 W
SFO2         400.1316005 MHz
NUC2                  1H
CPDPRG[2          waltz16
PCPD2              90.00 usec
PLW2         16.43099976 W
PLW12         0.20286000 W
PLW13         0.10204000 W

F2 - Processing parameters
SI                   32768
SF           100.6128098 MHz
WDW                    EM
SSB                     0
LB                   1.00 Hz
GB                      0
PC                   1.40
```



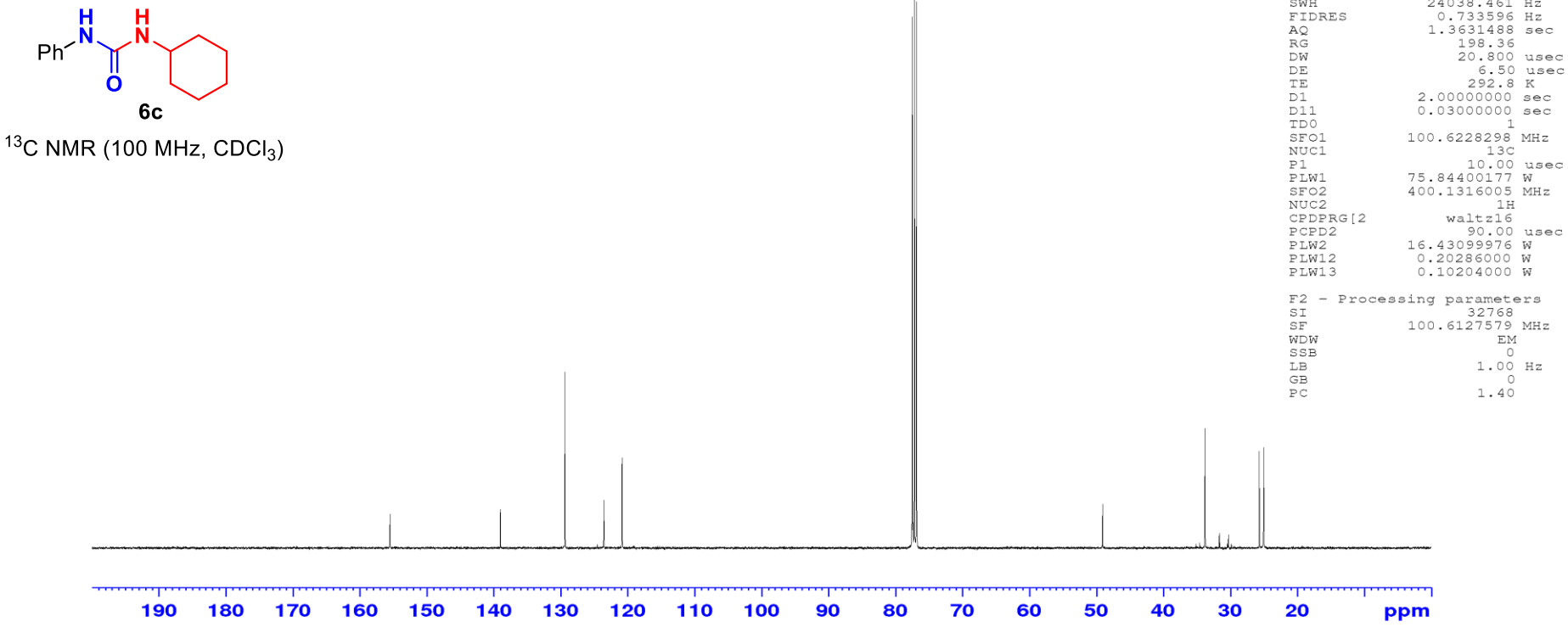
6c

^{1}H NMR (400 MHz, $CDCl_3$)

```
Current Data Parameters
NAME      lyr-20230218-1
EXPNO                  1
PROCNO                 1

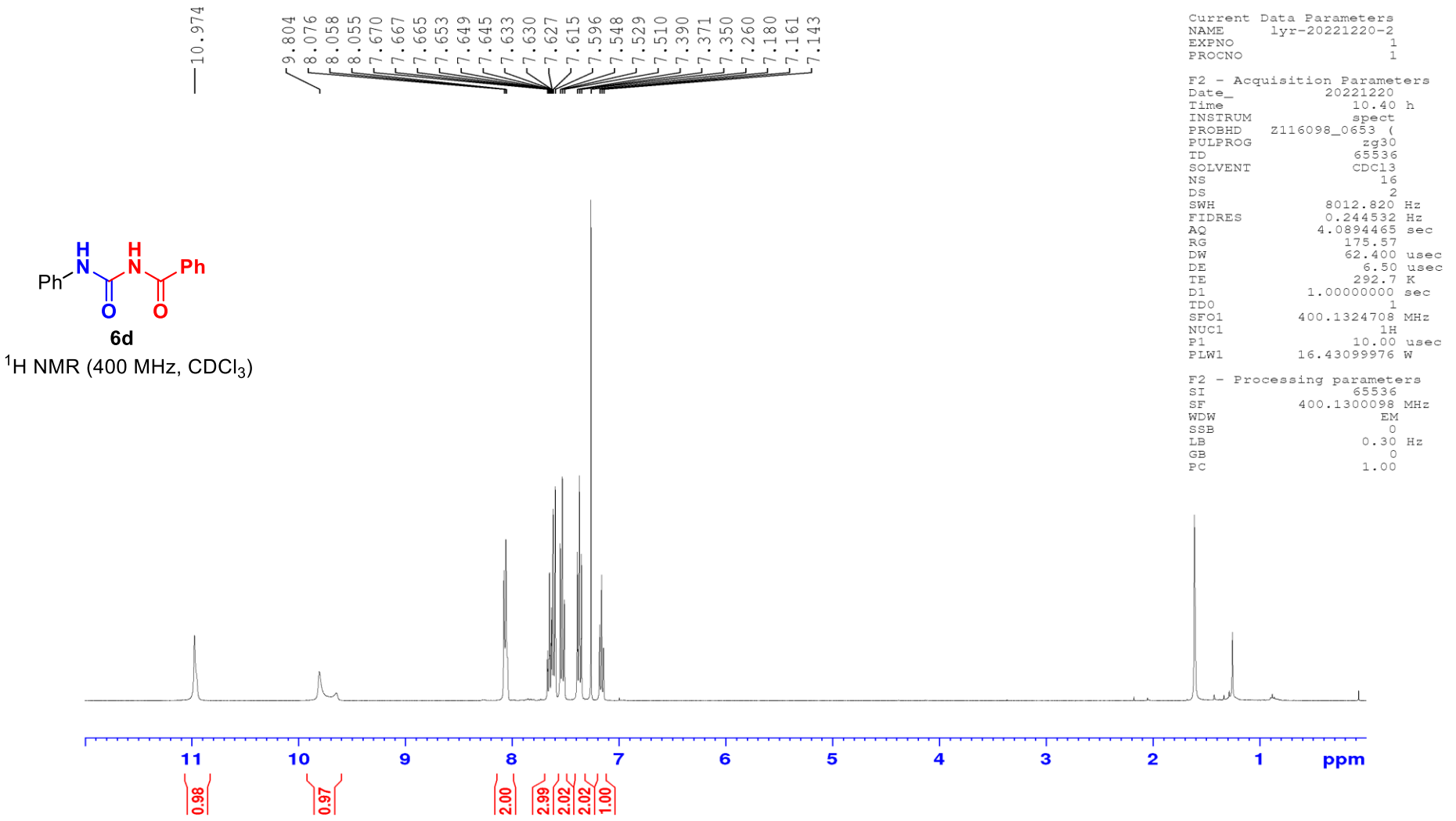
F2 - Acquisition Parameters
Date_           20230218
Time               22.27 h
INSTRUM            spect
PROBHD   Z116098_0653 (
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                   16
DS                    2
SWH            8012.820 Hz
FIDRES            0.244532 Hz
AQ             4.0894465 sec
RG                 77.68
DW                62.400 usec
DE                 6.50 usec
TE                291.8 K
D1             1.00000000 sec
TD0                   1
SFO1         400.1324708 MHz
NUC1                 1H
P1                10.00 usec
PLW1         16.43099976 W

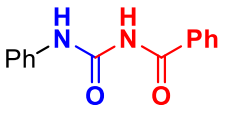
F2 - Processing parameters
SI                65536
SF           400.1300000 MHz
WDW                  EM
SSB                   0
LB                 0.30 Hz
GB                    0
PC                 1.00
```



6c

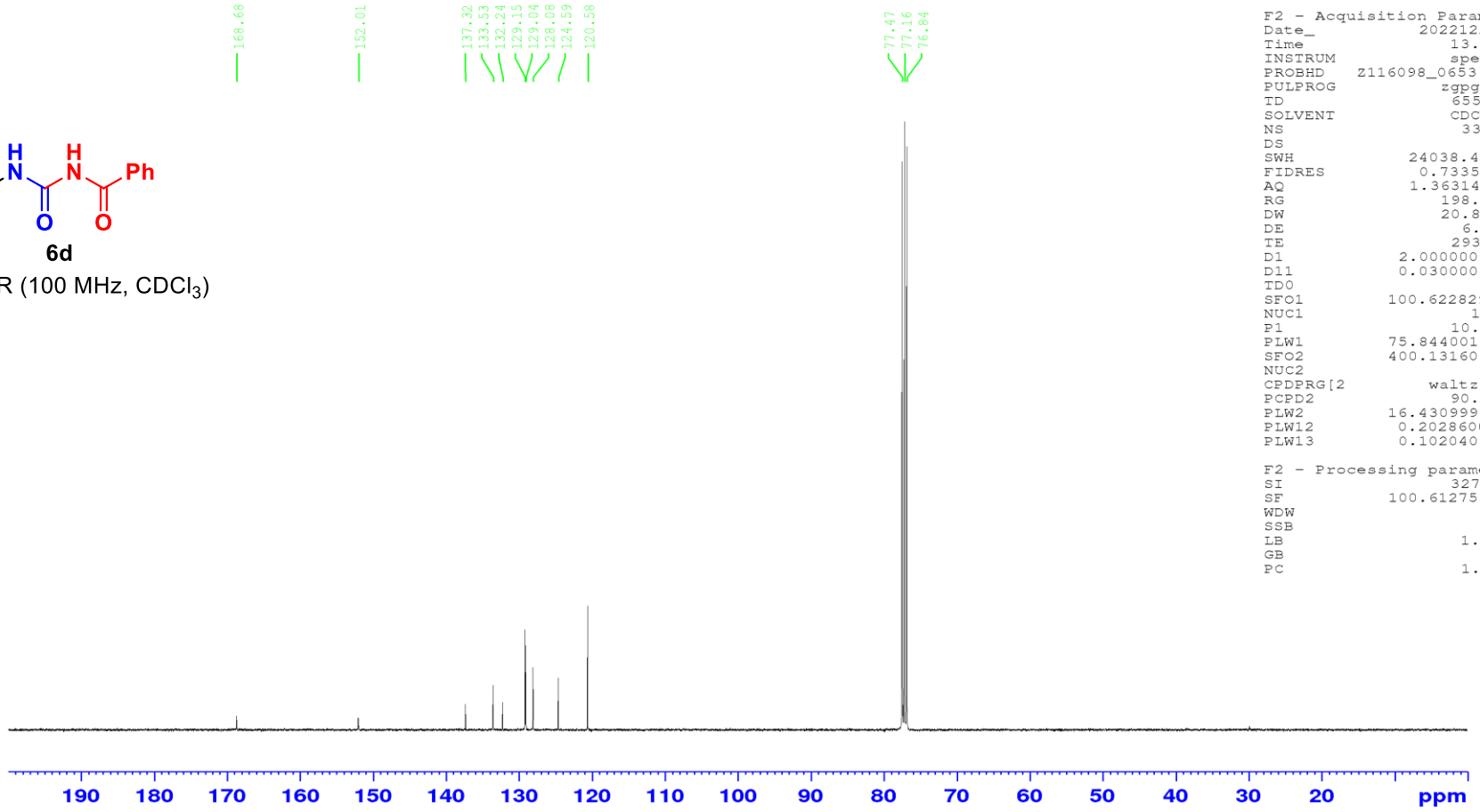
^{13}C NMR (100 MHz, $CDCl_3$)


10.974
9.804
8.076
8.058
8.055
7.670
7.667
7.665
7.653
7.649
7.645
7.633
7.630
7.627
7.615
7.596
7.548
7.529
7.510
7.390
7.371
7.350
7.260
7.180
7.161
7.143
Current Data Parameters
NAME lyr-20221220-2
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20221220
Time 10.40 h
INSTRUM spect
PROBHD Z116098_0653 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.244532 Hz
AQ 4.0894465 sec
RG 175.57
DW 62.400 usec
DE 6.50 usec
TE 292.7 K
D1 1.00000000 sec
TD0 1
SFO1 400.1324708 MHz
NUC1 1H
P1 10.00 usec
PLW1 16.43099976 W
F2 - Processing parameters
SI 65536
SF 400.1300098 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
Ph
H
N
O
Ph
6d
1H NMR (400 MHz, CDCl3)
11
10
9
8
7
6
5
4
3
2
1
ppm
0.98
0.97
2.00
2.99
2.02
2.02
1.00



6d

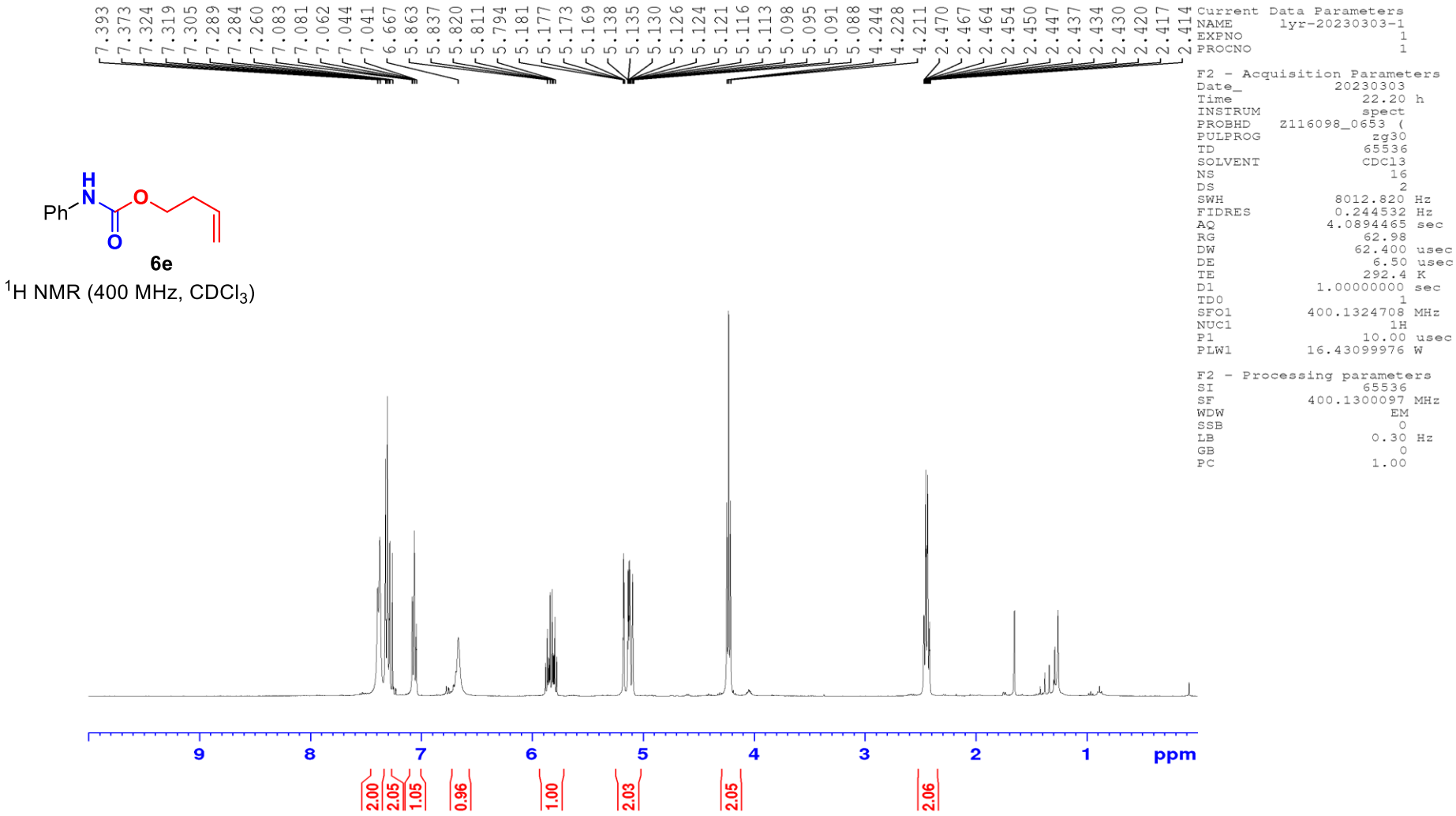
^{13}C NMR (100 MHz, $CDCl_3$)




```
Current Data Parameters
NAME      lyr-20221220-2
EXPNO                  2
PROCNO                 1

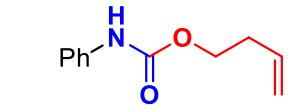
F2 - Acquisition Parameters
Date_           20221220
Time               13.51 h
INSTRUM            spect
PROBHD   Z116098_0653 (
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                 3333
DS                    4
SWH           24038.461 Hz
FIDRES         0.733596 Hz
AQ            1.3631488 sec
RG               198.36
DW               20.800 usec
DE                6.50 usec
TE               293.6 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                   1
SFO1        100.6228298 MHz
NUC1               13C
P1                10.00 usec
PLW1        75.84400177 W
SFO2        400.1316005 MHz
NUC2               1H
CPDPRG[2        waltz16
PCPD2             90.00 usec
PLW2        16.43099976 W
PLW12        0.20286000 W
PLW13        0.10204000 W

F2 - Processing parameters
SI                32768
SF          100.6127571 MHz
WDW                EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```



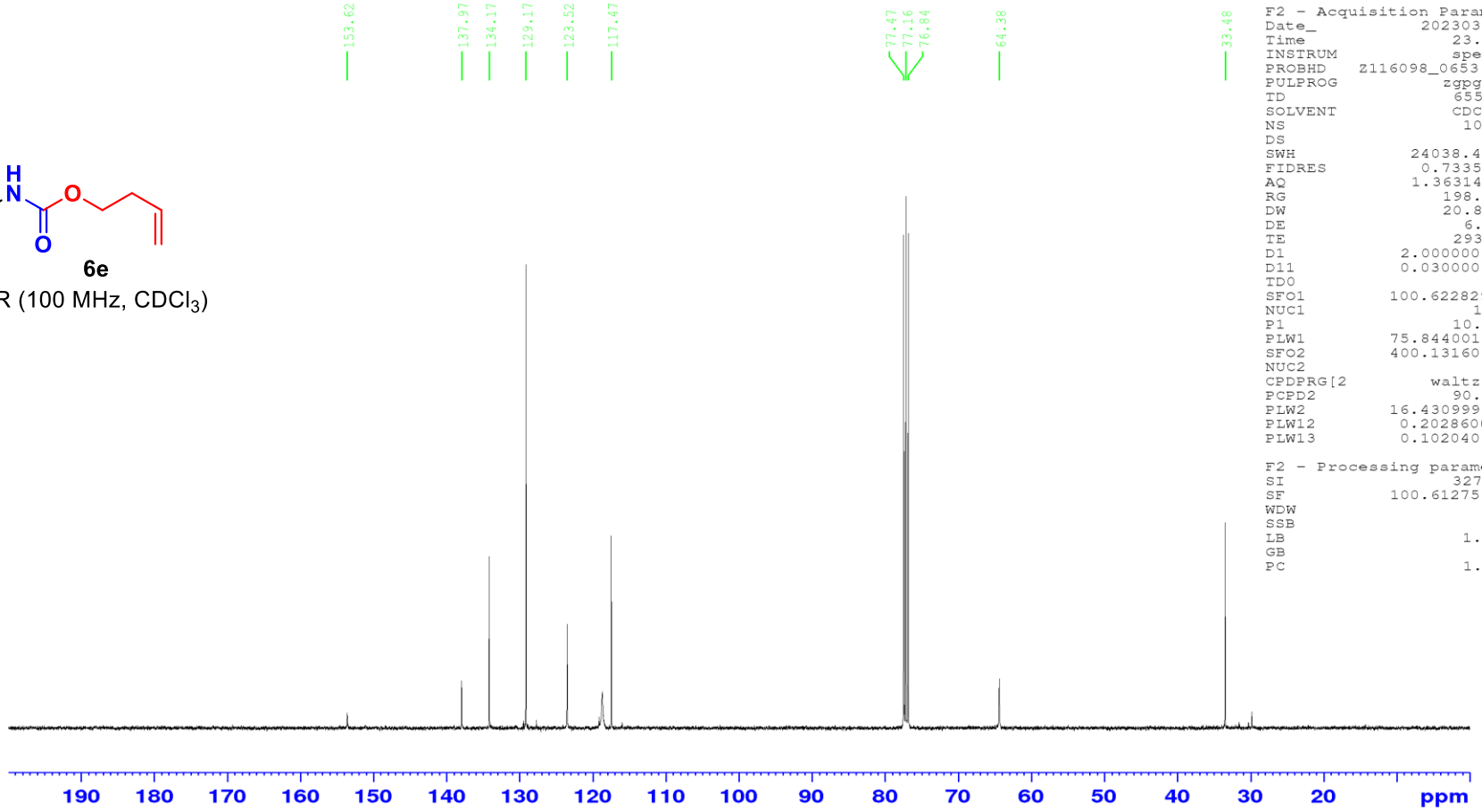
6e

^{1}H NMR (400 MHz, $CDCl_3$)



6e

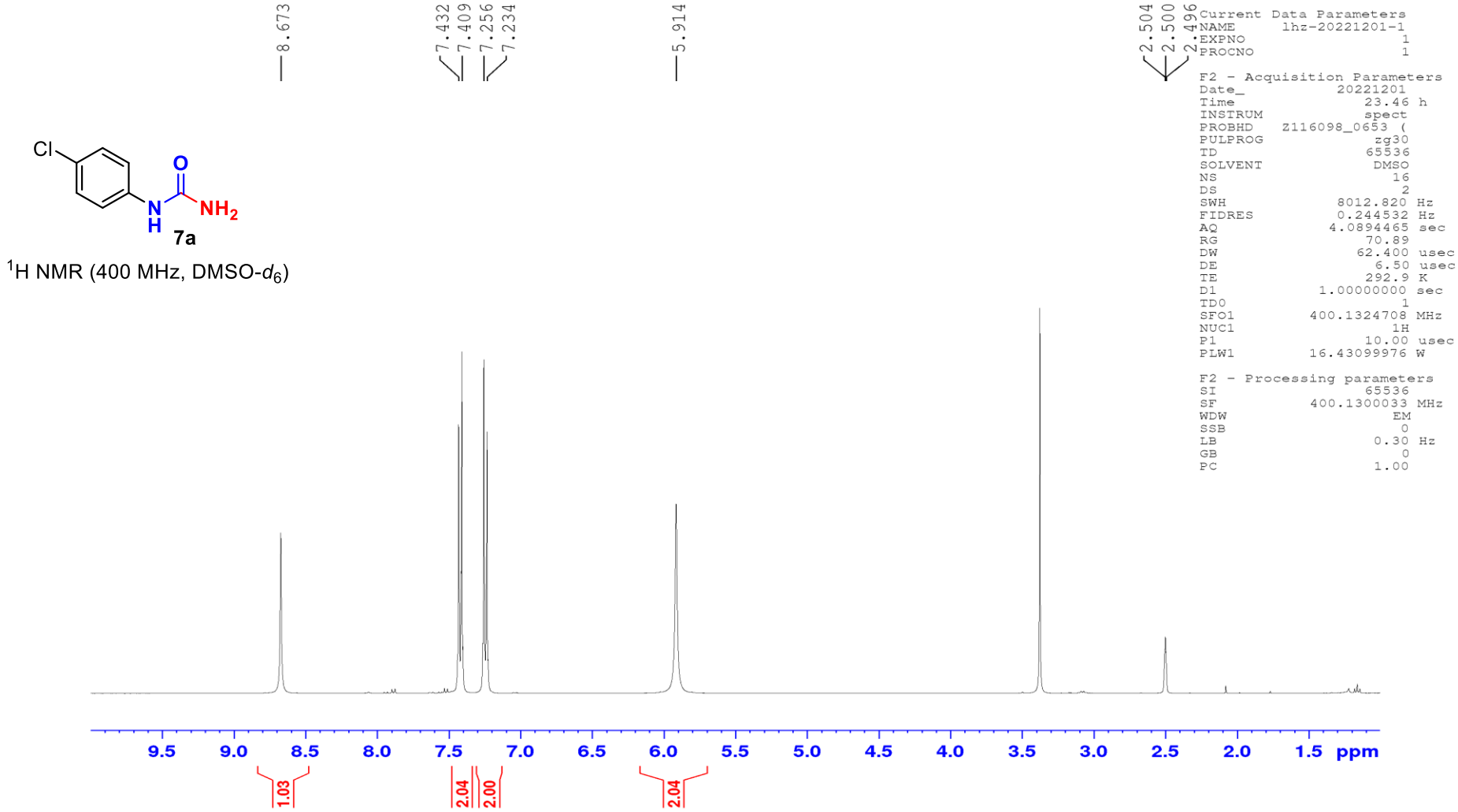
^{13}C NMR (100 MHz, $CDCl_3$)

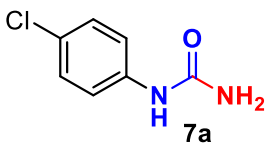



```
Current Data Parameters
NAME      lyr-20230303-1
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20230303
Time              23.20 h
INSTRUM             spect
PROBHD   Z116098_0653 (
PULPROG           zgpg30
TD                65536
SOLVENT           CDCl3
NS                 1024
DS                    4
SWH           24038.461 Hz
FIDRES         0.733596 Hz
AQ             1.3631488 sec
RG                198.36
DW                20.800 usec
DE                 6.50 usec
TE                293.0 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1
SFO1        100.6228298 MHz
NUC1               13C
P1                10.00 usec
PLW1        75.84400177 W
SFO2        400.1316005 MHz
NUC2               1H
CPDPRG[2        waltz16
PCPD2             90.00 usec
PLW2        16.43099976 W
PLW12        0.20286000 W
PLW13        0.10204000 W

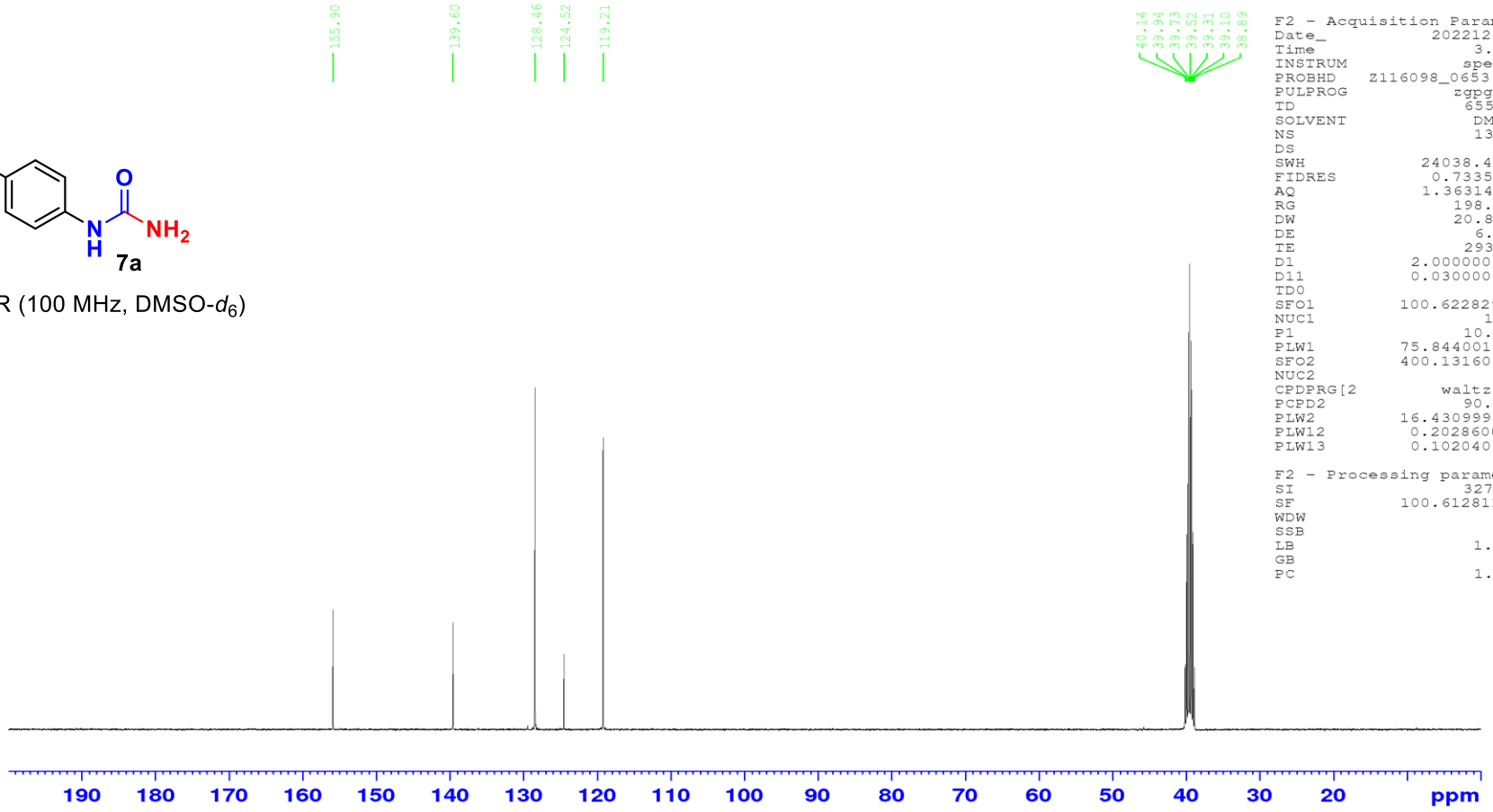
F2 - Processing parameters
SI                32768
SF          100.6127594 MHz
WDW                EM
SSB                 0
LB              1.00 Hz
GB                  0
PC              1.40
```


8.673
7.432
7.409
7.256
7.234
5.914
2.504
2.500
2.496
Cl
O
N
H
NH2
7a
1H NMR (400 MHz, DMSO-d6)
Current Data Parameters
NAME lhz-20221201-1
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20221201
Time 23.46 h
INSTRUM spect
PROBHD Z116098_0653 (
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.244532 Hz
AQ 4.0894465 sec
RG 70.89
DW 62.400 usec
DE 6.50 usec
TE 292.9 K
D1 1.00000000 sec
TD0 1
SFO1 400.1324708 MHz
NUC1 1H
P1 10.00 usec
PLW1 16.43099976 W
F2 - Processing parameters
SI 65536
SF 400.1300033 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
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 ppm
1.03
2.04
2.00
2.04



7a

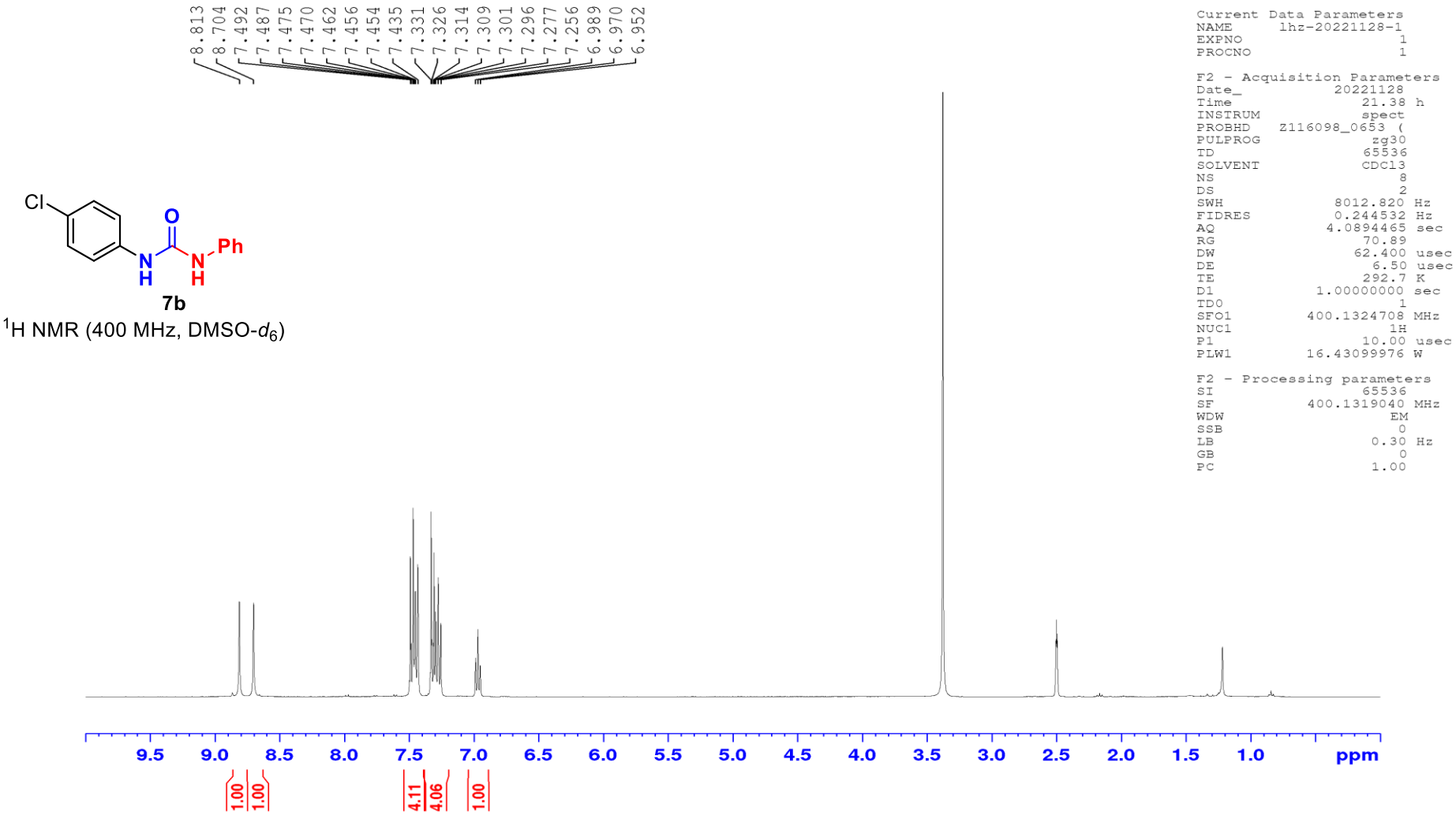
^{13}C NMR (100 MHz, DMSO-d_6)

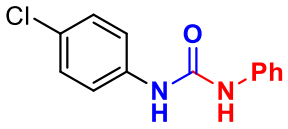



```
Current Data Parameters
NAME      lhz-20221201-1
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20221202
Time                3.03 h
INSTRUM           spect
PROBHD    Z116098_0653 (
PULPROG          zgpg30
TD                65536
SOLVENT            DMSO
NS                 1333
DS                    4
SWH           24038.461 Hz
FIDRES         0.733596 Hz
AQ             1.3631488 sec
RG                198.36
DW                20.800 usec
DE                 6.50 usec
TE                293.6 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1
SFO1        100.6228298 MHz
NUC1               13C
P1                10.00 usec
PLW1        75.84400177 W
SFO2        400.1316005 MHz
NUC2               1H
CPDPRG[2       waltz16
PCPD2             90.00 usec
PLW2        16.43099976 W
PLW12        0.20286000 W
PLW13        0.10204000 W

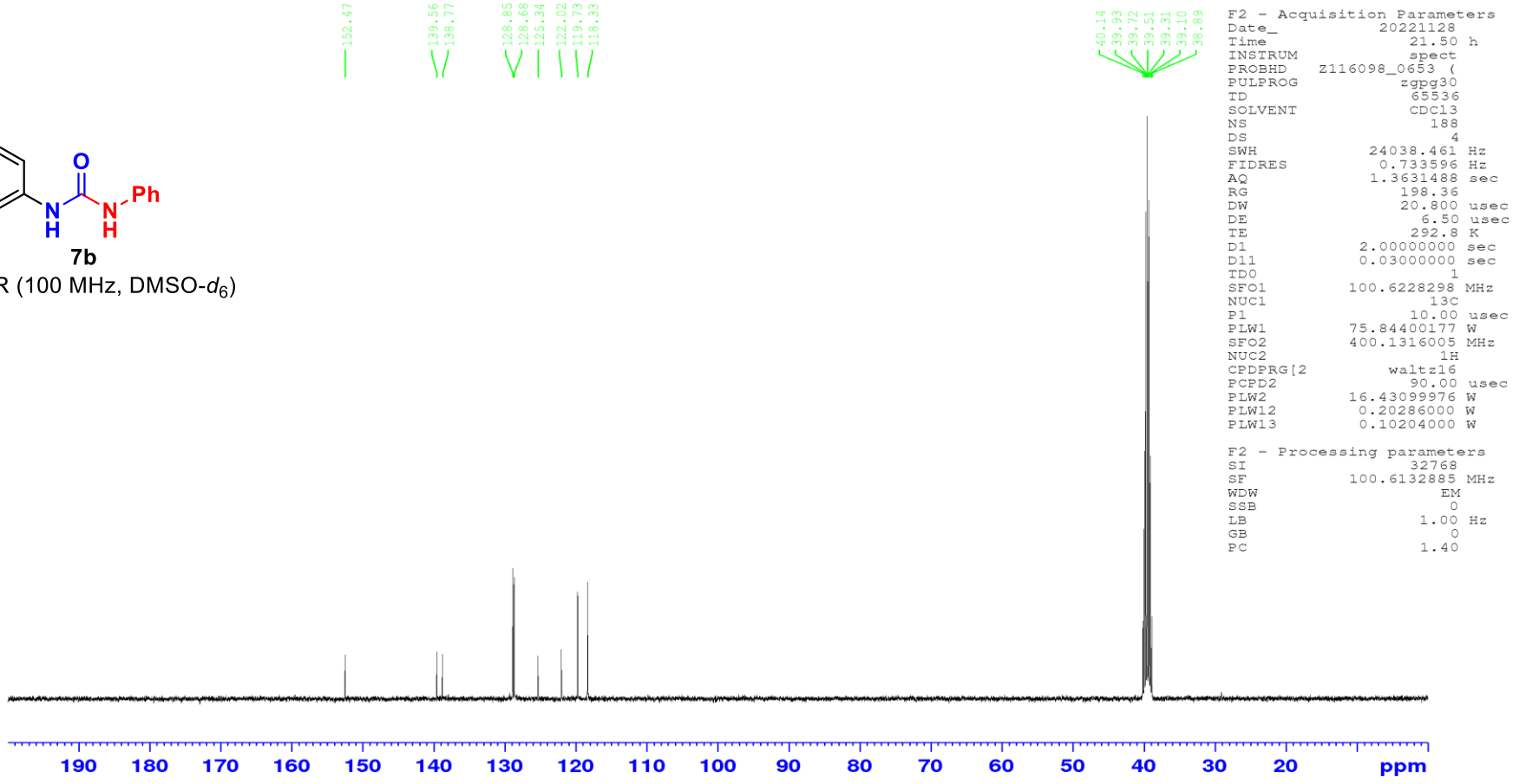
F2 - Processing parameters
SI                32768
SF          100.6128110 MHz
WDW                EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```


8.813
8.704
7.492
7.487
7.475
7.470
7.462
7.456
7.454
7.435
7.331
7.326
7.314
7.309
7.301
7.296
7.277
7.256
6.989
6.970
6.952
Cl
O
N
H
N
H
Ph
7b
1H NMR (400 MHz, DMSO-d6)
Current Data Parameters
NAME lhz-20221128-1
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20221128
Time 21.38 h
INSTRUM spect
PROBHD Z116098_0653 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 8012.820 Hz
FIDRES 0.244532 Hz
AQ 4.0894465 sec
RG 70.89
DW 62.400 usec
DE 6.50 usec
TE 292.7 K
D1 1.00000000 sec
TD0 1
SFO1 400.1324708 MHz
NUC1 1H
P1 10.00 usec
PLW1 16.43099976 W
F2 - Processing parameters
SI 65536
SF 400.1319040 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
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
ppm
1.00
1.00
4.11
4.06
1.00



7b

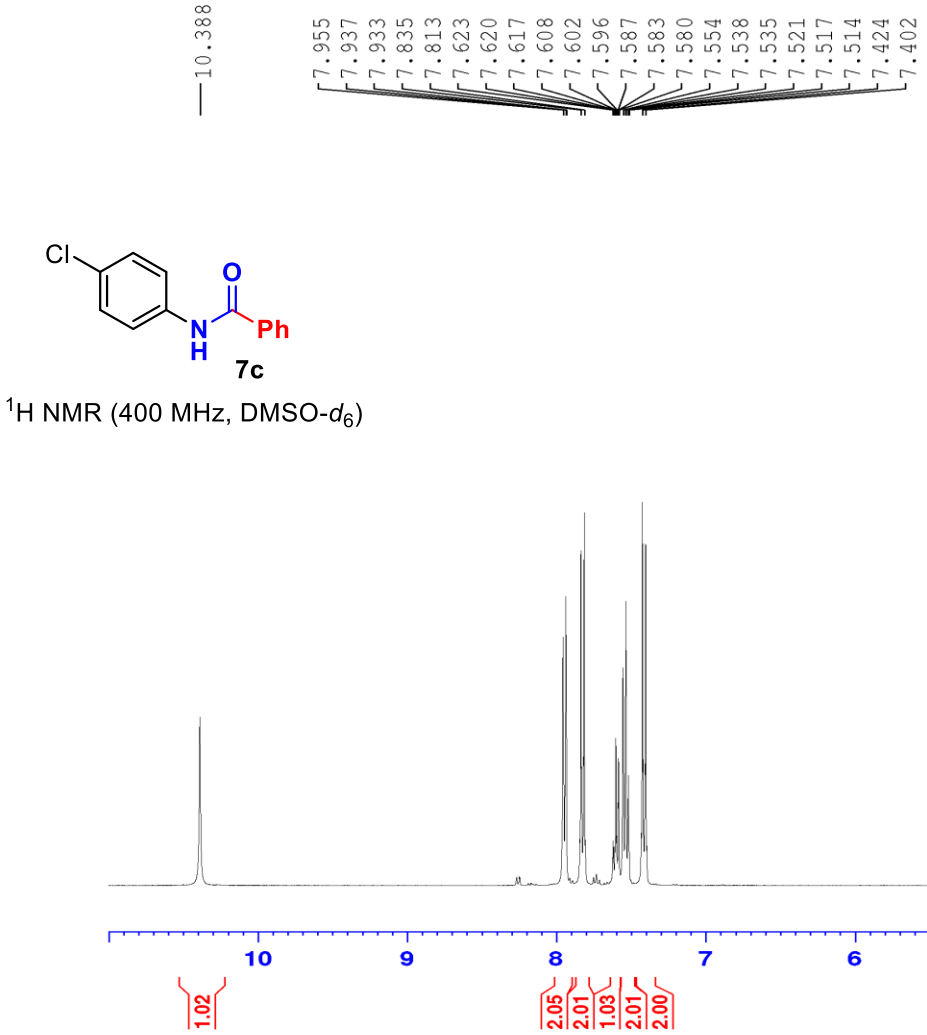
^{13}C NMR (100 MHz, DMSO-d_6)




```
Current Data Parameters
NAME      lhz-20221128-1
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20221128
Time               21.50 h
INSTRUM            spect
PROBHD    Z116098_0653 (
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  188
DS                    4
SWH           24038.461 Hz
FIDRES         0.733596 Hz
AQ             1.3631488 sec
RG                198.36
DW                20.800 usec
DE                 6.50 usec
TE                292.8 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1
SFO1        100.6228298 MHz
NUC1                13C
P1                10.00 usec
PLW1        75.84400177 W
SFO2        400.1316005 MHz
NUC2                1H
CPDPRG[2        waltz16
PCPD2             90.00 usec
PLW2        16.43099976 W
PLW12        0.20286000 W
PLW13        0.10204000 W

F2 - Processing parameters
SI                32768
SF          100.6132885 MHz
WDW                EM
SSB                 0
LB                1.00 Hz
GB                  0
PC                1.40
```



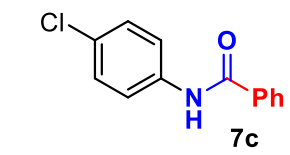
7c

^{1}H NMR (400 MHz, DMSO-d_6)

```
Current Data Parameters
NAME      1hz-20221220-8
EXPNO                  1
PROCNO                 1

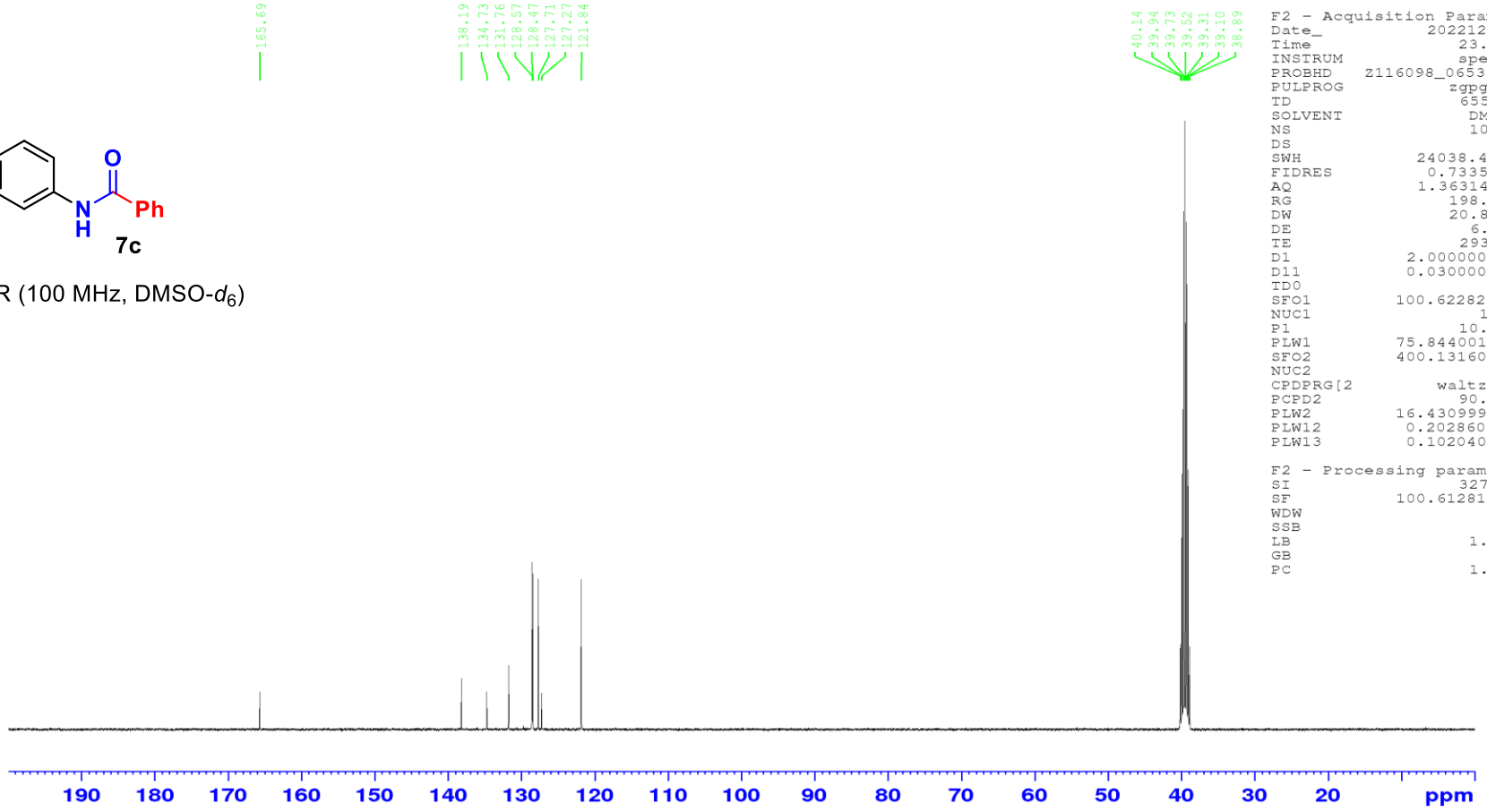
F2 - Acquisition Parameters
Date_           20221220
Time              22.18 h
INSTRUM            spect
PROBHD   Z116098_0653 (
PULPROG            zg30
TD                65536
SOLVENT            DMSO
NS                   16
DS                    2
SWH            8012.820 Hz
FIDRES        0.244532 Hz
AQ            4.0894465 sec
RG                 88.22
DW               62.400 usec
DE                6.50 usec
TE               293.0 K
D1            1.00000000 sec
TD0                   1
SFO1        400.1324708 MHz
NUC1                1H
P1                10.00 usec
PLW1        16.43099976 W

F2 - Processing parameters
SI                65536
SF          400.1300032 MHz
WDW                 EM
SSB                  0
LB                0.30 Hz
GB                   0
PC                1.00
```



7c

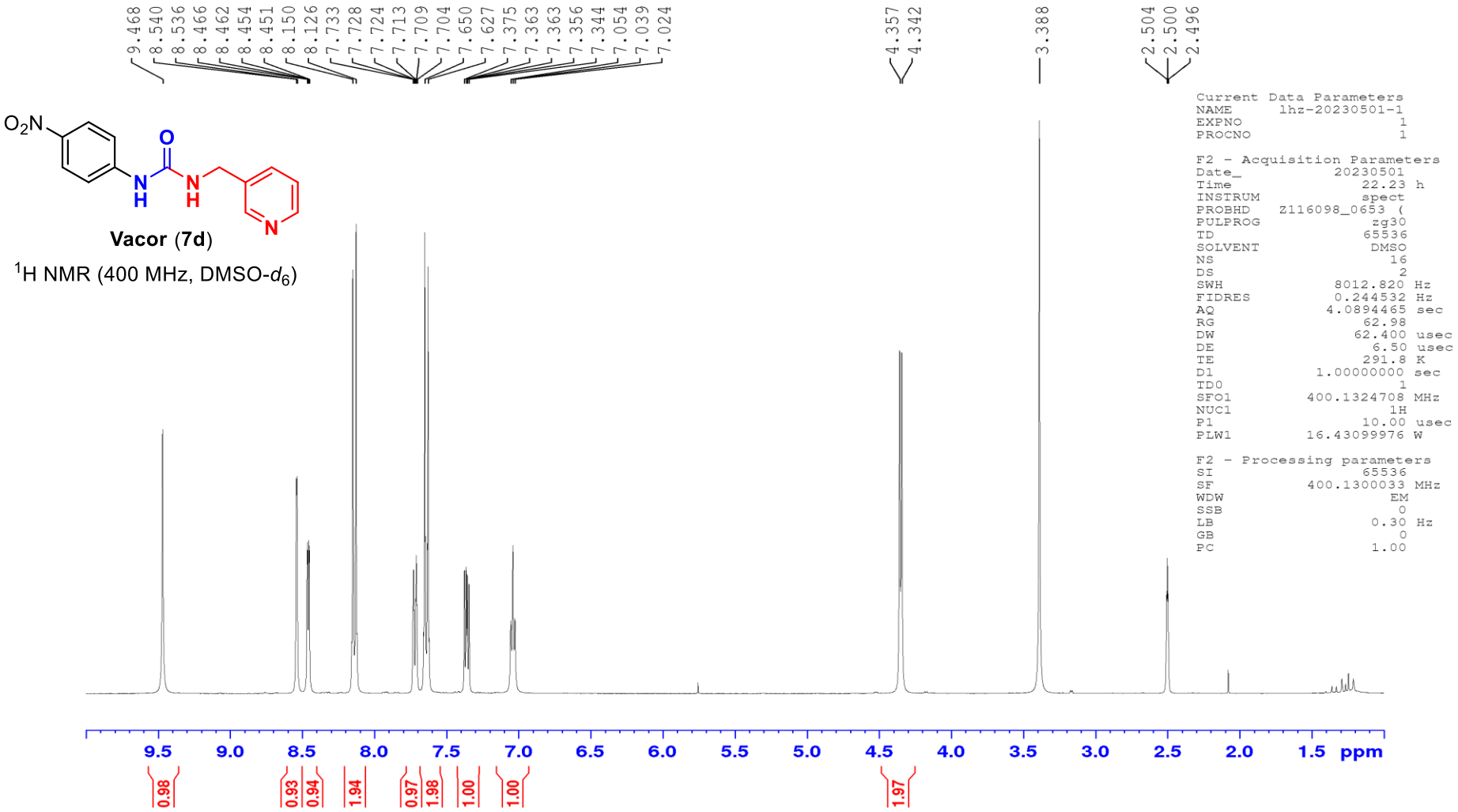
^{13}C NMR (100 MHz, DMSO-d_6)

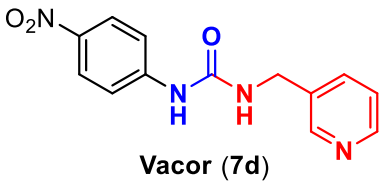



```
Current Data Parameters
NAME      lhz-20221220-8
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20221220
Time              23.17 h
INSTRUM            spect
PROBHD    Z116098_0653 (
PULPROG         zgpg30
TD               65536
SOLVENT           DMSO
NS                1024
DS                   4
SWH           24038.461 Hz
FIDRES         0.733596 Hz
AQ             1.3631488 sec
RG               198.36
DW               20.800 usec
DE                6.50 usec
TE               293.6 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                  1
SFO1        100.6228298 MHz
NUC1               13C
P1               10.00 usec
PLW1         75.84400177 W
SFO2        400.1316005 MHz
NUC2               1H
CPDPRG[2       waltz16
PCPD2            90.00 usec
PLW2         16.43099976 W
PLW12         0.20286000 W
PLW13         0.10204000 W

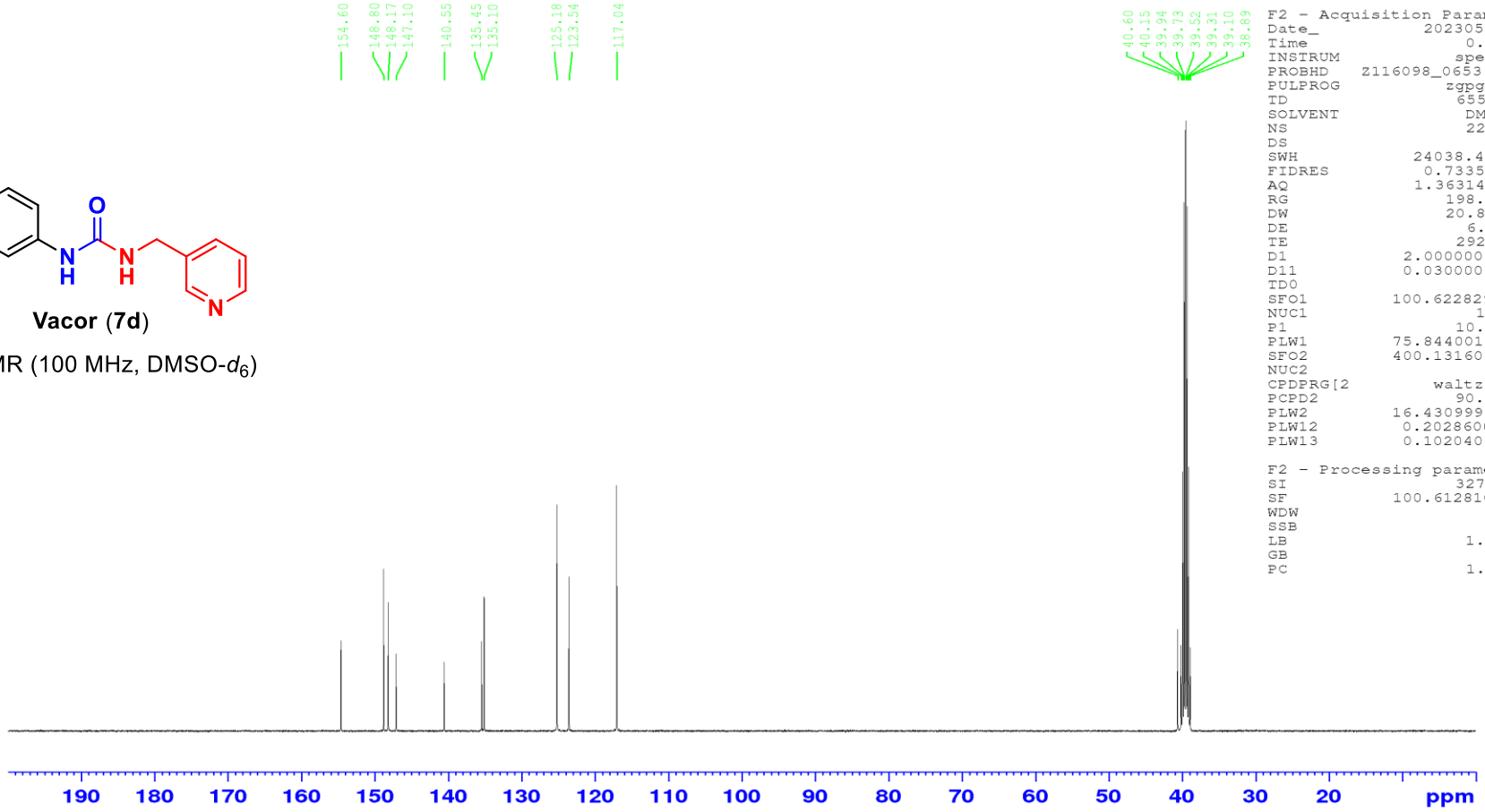
F2 - Processing parameters
SI                32768
SF          100.6128124 MHz
WDW                EM
SSB                 0
LB               1.00 Hz
GB                  0
PC               1.40
```


9.468
8.540
8.536
8.466
8.462
8.454
8.451
8.150
8.126
7.733
7.728
7.724
7.713
7.709
7.704
7.650
7.627
7.375
7.363
7.363
7.356
7.344
7.054
7.039
7.024
4.357
4.342
3.388
2.504
2.500
2.496
O2N
O
N
H
N
H
N
Vacor (7d)
1H NMR (400 MHz, DMSO-d6)
Current Data Parameters
NAME lhz-20230501-1
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20230501
Time 22.23 h
INSTRUM spect
PROBHD Z116098_0653 (
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.244532 Hz
AQ 4.0894465 sec
RG 62.98
DW 62.400 usec
DE 6.50 usec
TE 291.8 K
D1 1.00000000 sec
TD0 1
SFO1 400.1324708 MHz
NUC1 1H
P1 10.00 usec
PLW1 16.43099976 W
F2 - Processing parameters
SI 65536
SF 400.1300033 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
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
ppm
0.98
0.93
0.94
1.94
0.97
1.98
1.00
1.00
1.97



Vacor (**7d**)

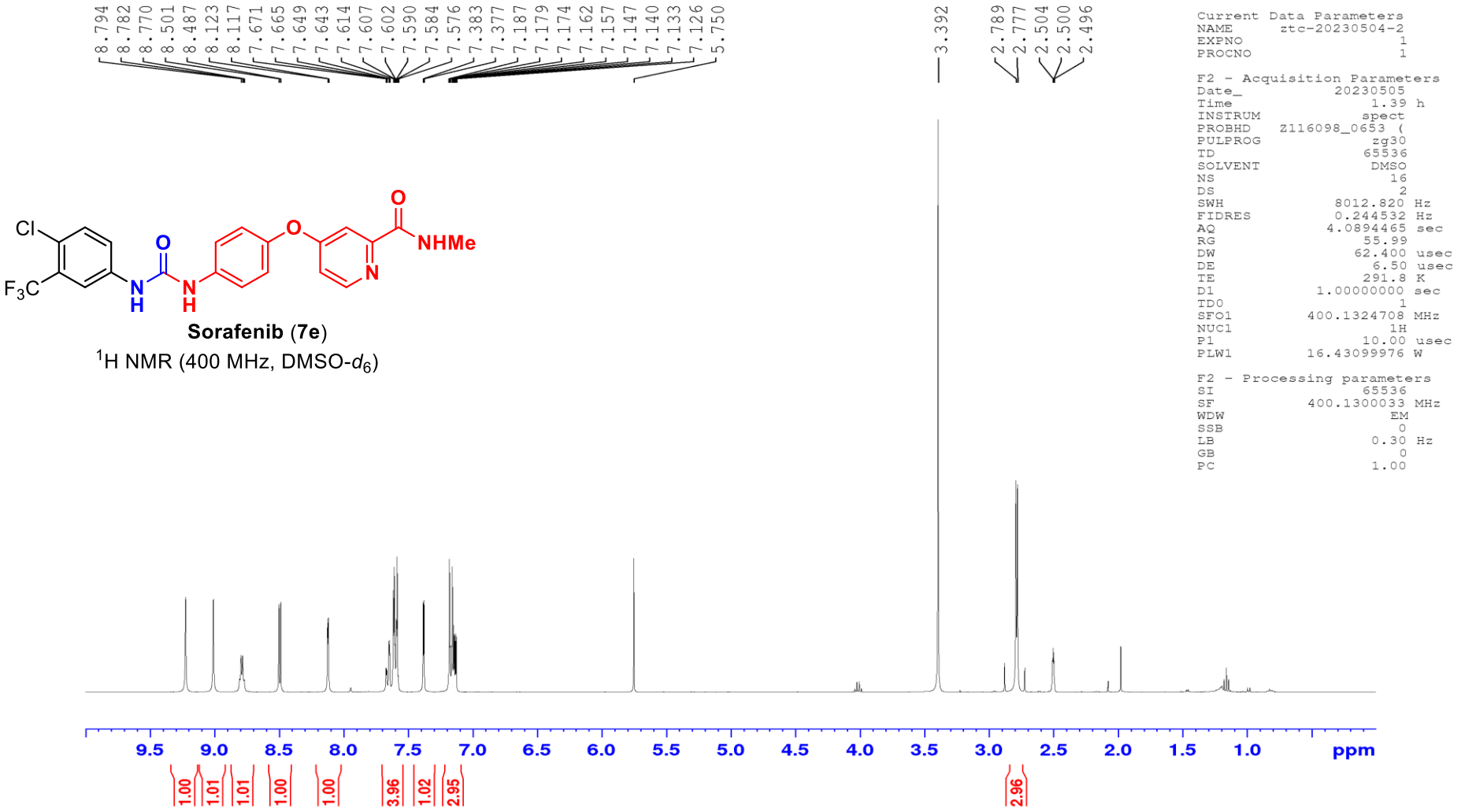
^{13}C NMR (100 MHz, DMSO-d_6)




```
Current Data Parameters
NAME      1hz-20230501-1
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20230502
Time               0.31 h
INSTRUM           spect
PROBHD    Z116098_0653 (
PULPROG         zgpg30
TD               65536
SOLVENT           DMSO
NS                2222
DS                   4
SWH          24038.461 Hz
FIDRES        0.733596 Hz
AQ            1.3631488 sec
RG               198.36
DW               20.800 usec
DE                6.50 usec
TE               292.7 K
D1          2.00000000 sec
D11         0.03000000 sec
TD0                  1
SFO1       100.6228298 MHz
NUC1              13C
P1               10.00 usec
PLW1       75.84400177 W
SFO2       400.1316005 MHz
NUC2              1H
CPDPRG[2       waltz16
PCPD2            90.00 usec
PLW2       16.43099976 W
PLW12       0.20286000 W
PLW13       0.10204000 W

F2 - Processing parameters
SI               32768
SF         100.6128102 MHz
WDW                EM
SSB                 0
LB               1.00 Hz
GB                  0
PC               1.40
```



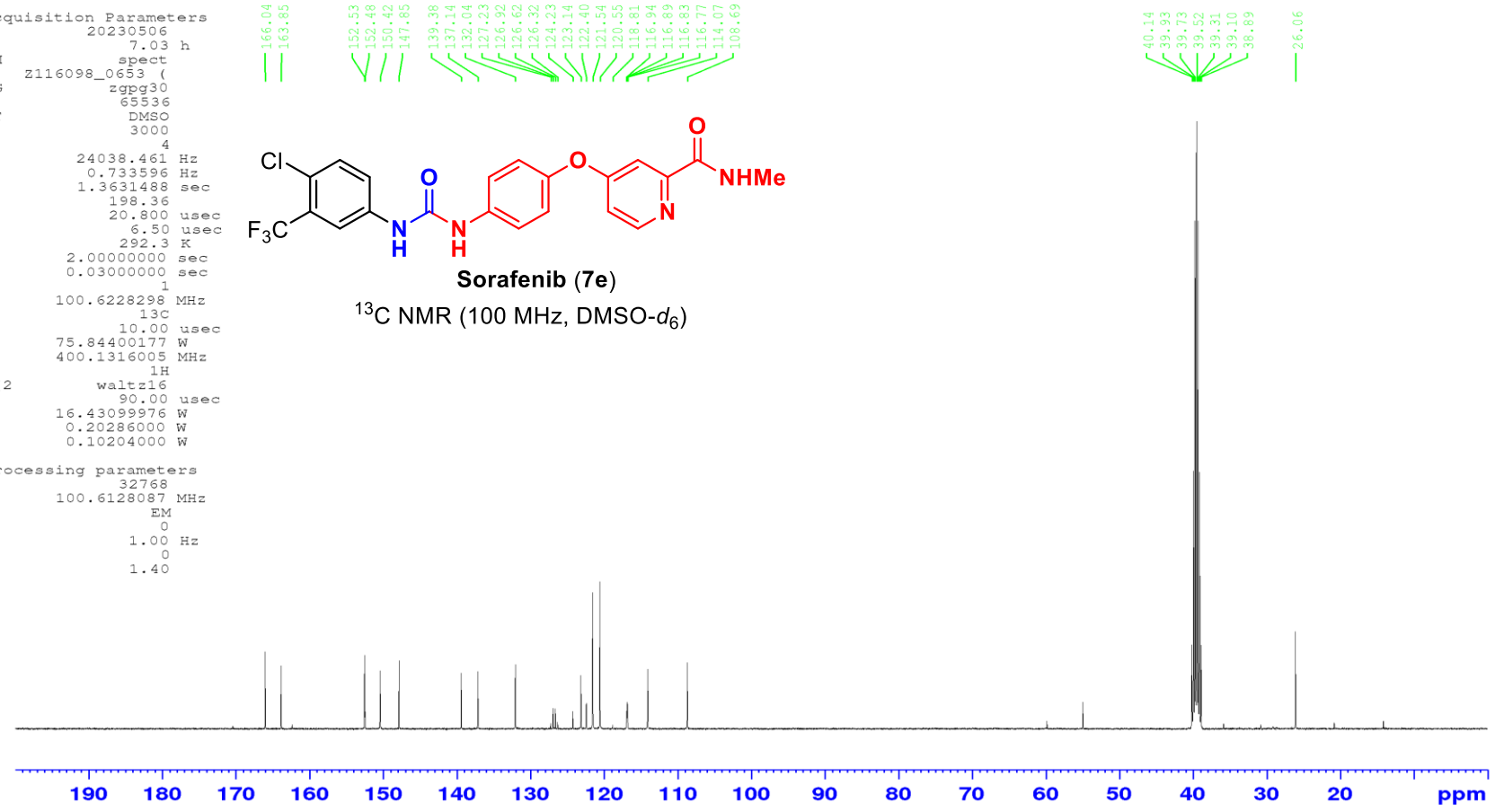
Sorafenib (7e)

^{1}H NMR (400 MHz, DMSO-d_6)

```
Current Data Parameters
NAME      ztc-20230504-2
EXPNO                  3
PROCNO                 1

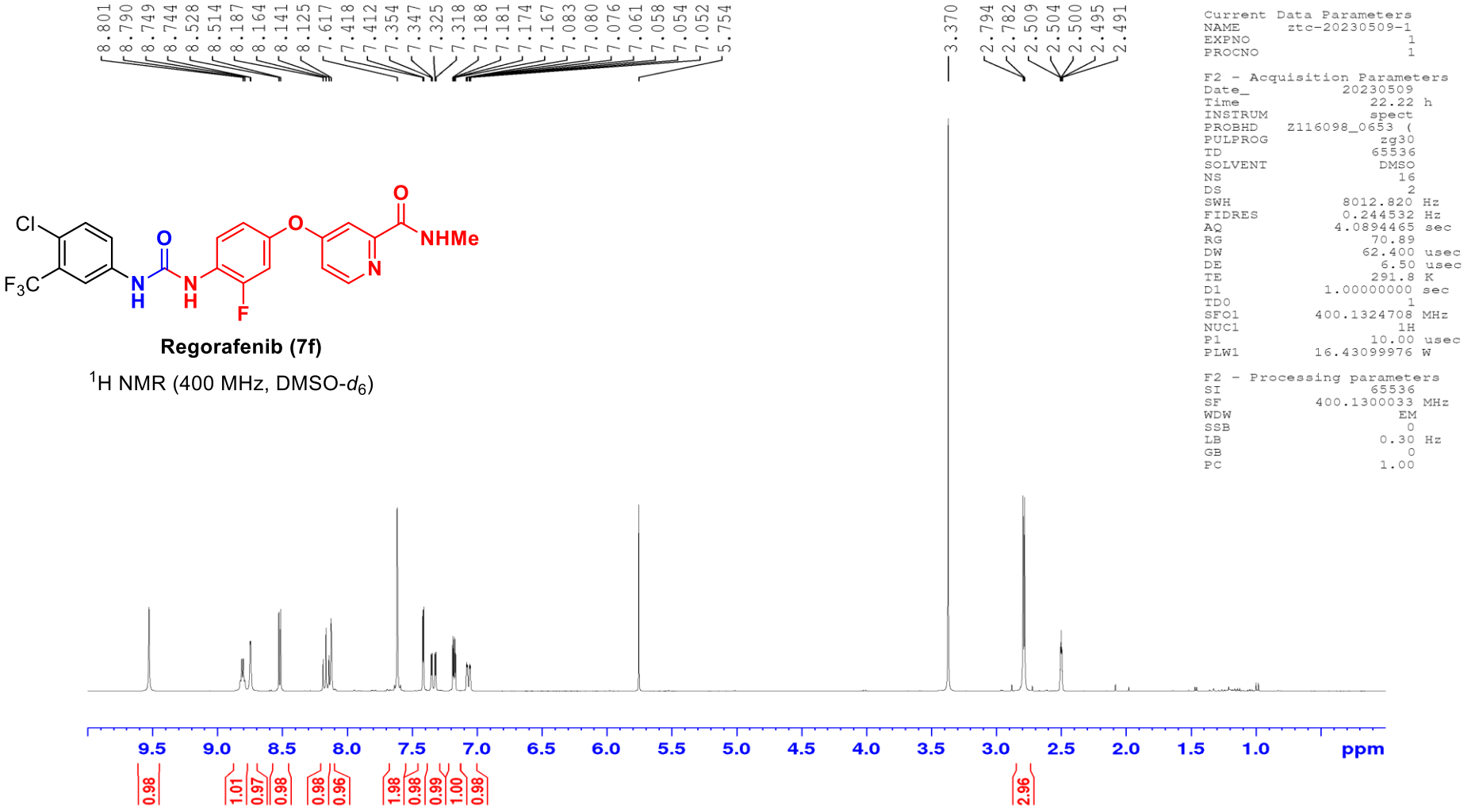
F2 - Acquisition Parameters
Date_           20230506
Time                7.03 h
INSTRUM            spect
PROBHD    Z116098_0653 (
PULPROG          zgpg30
TD                65536
SOLVENT            DMSO
NS                 3000
DS                    4
SWH           24038.461 Hz
FIDRES         0.733596 Hz
AQ            1.3631488 sec
RG               198.36
DW               20.800 usec
DE                6.50 usec
TE               292.3 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1
SFO1        100.6228298 MHz
NUC1                13C
P1                10.00 usec
PLW1        75.84400177 W
SFO2        400.1316005 MHz
NUC2                1H
CPDPRG[2        waltz16
PCPD2             90.00 usec
PLW2        16.43099976 W
PLW12        0.20286000 W
PLW13        0.10204000 W

F2 - Processing parameters
SI                32768
SF          100.6128087 MHz
WDW                 EM
SSB                  0
LB                1.00 Hz
GB                   0
PC                1.40
```



Sorafenib (7e)

^{13}C NMR (100 MHz, DMSO-d_6)



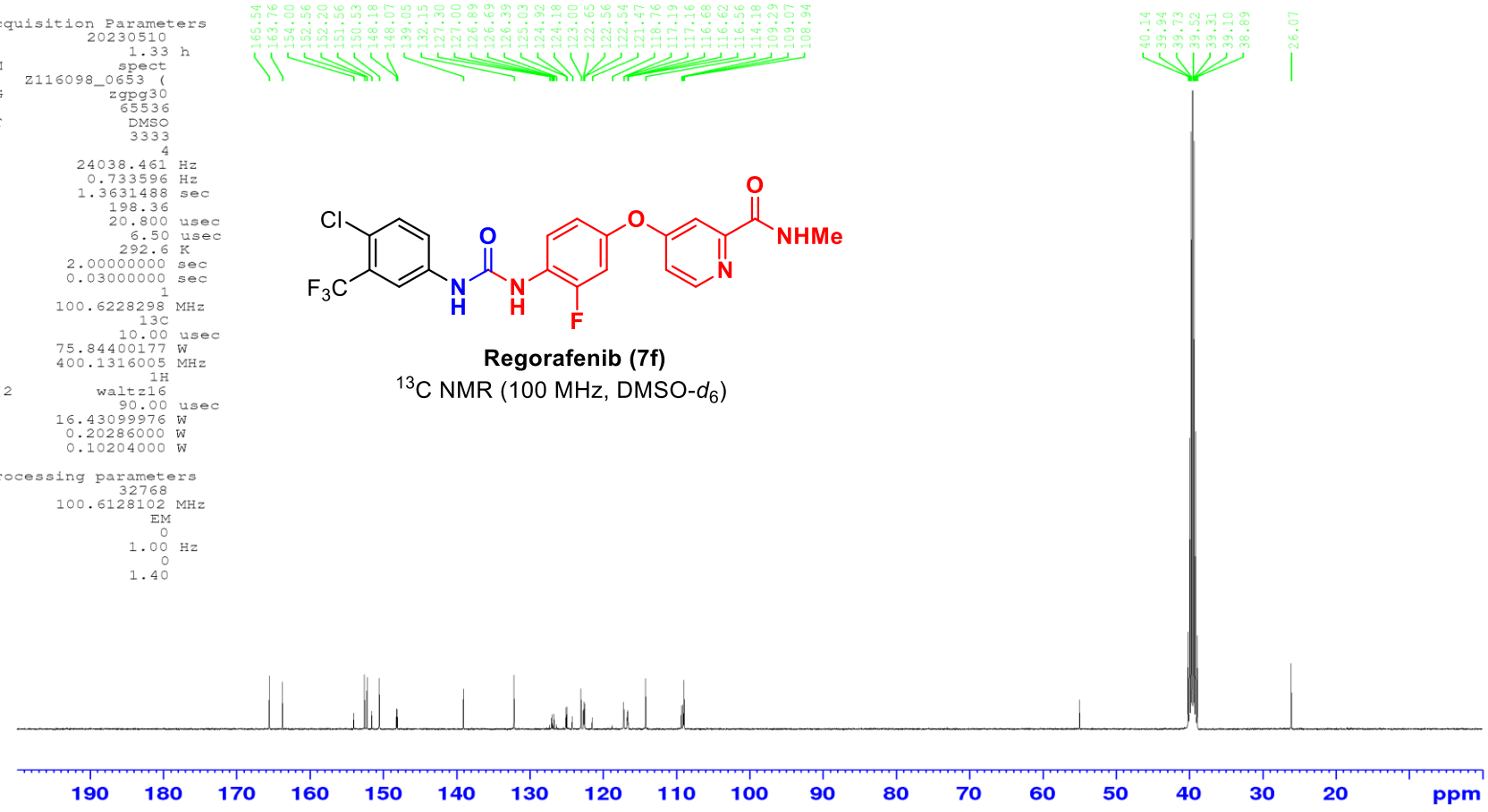
Regorafenib (7f)

^{1}H NMR (400 MHz, DMSO-d_6)

```
Current Data Parameters
NAME      ztc-20230509-1
EXPNO                  2
PROCNO                 1

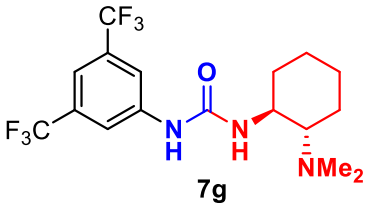
F2 - Acquisition Parameters
Date_           20230510
Time                1.33 h
INSTRUM            spect
PROBHD    Z116098_0653 (
PULPROG           zgpg30
TD                65536
SOLVENT            DMSO
NS                 3333
DS                    4
SWH           24038.461 Hz
FIDRES         0.733596 Hz
AQ            1.3631488 sec
RG               198.36
DW               20.800 usec
DE                6.50 usec
TE               292.6 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1
SFO1        100.6228298 MHz
NUC1                13C
P1                10.00 usec
PLW1        75.84400177 W
SFO2        400.1316005 MHz
NUC2                1H
CPDPRG[2         waltz16
PCPD2             90.00 usec
PLW2        16.43099976 W
PLW12        0.20286000 W
PLW13        0.10204000 W

F2 - Processing parameters
SI                32768
SF          100.6128102 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```



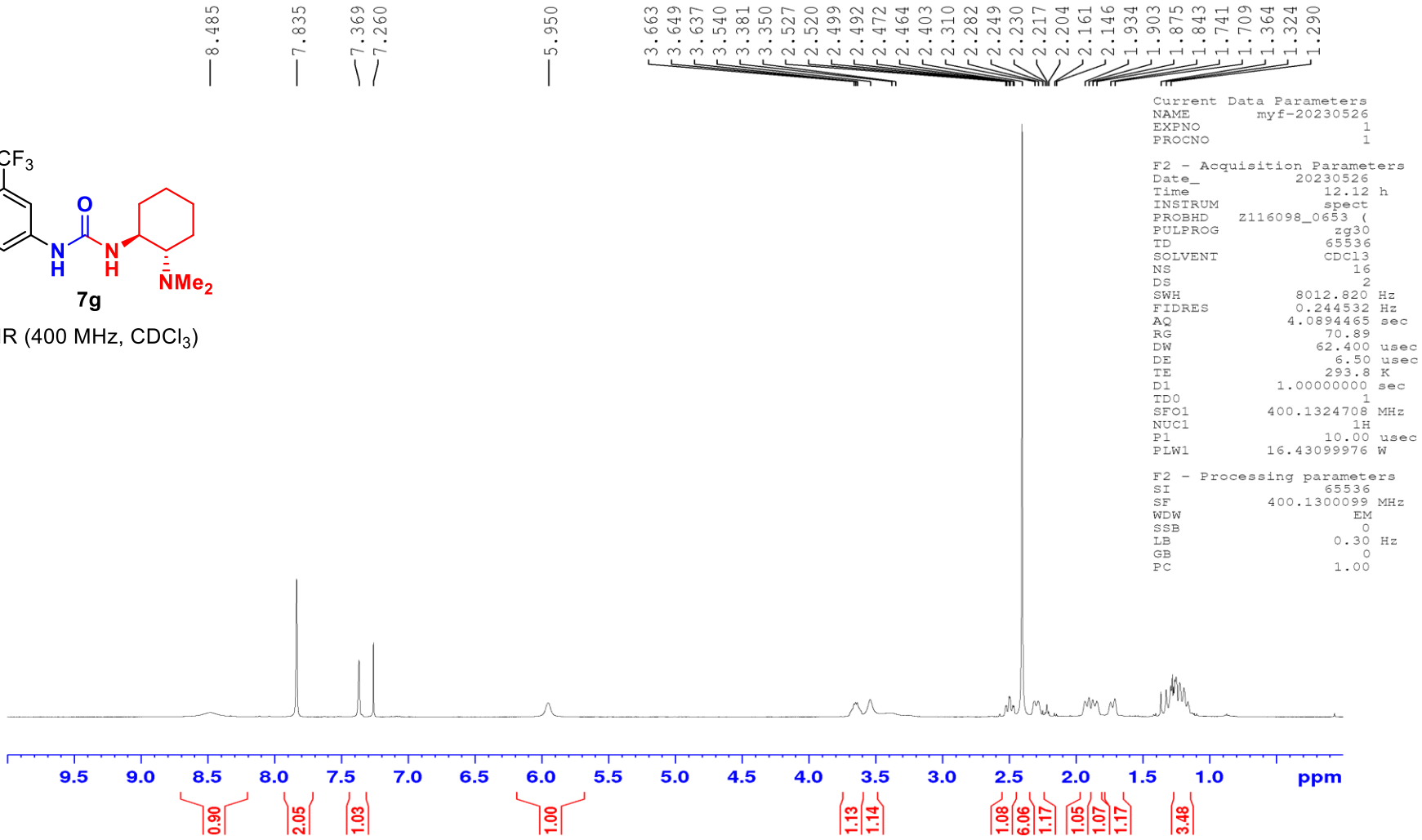
Regorafenib (7f)

^{13}C NMR (100 MHz, DMSO-d_6)



7g

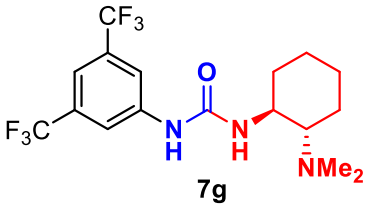
^{1}H NMR (400 MHz, $CDCl_3$)




```
Current Data Parameters
NAME                  myf-20230526
EXPNO                            1
PROCNO                           1

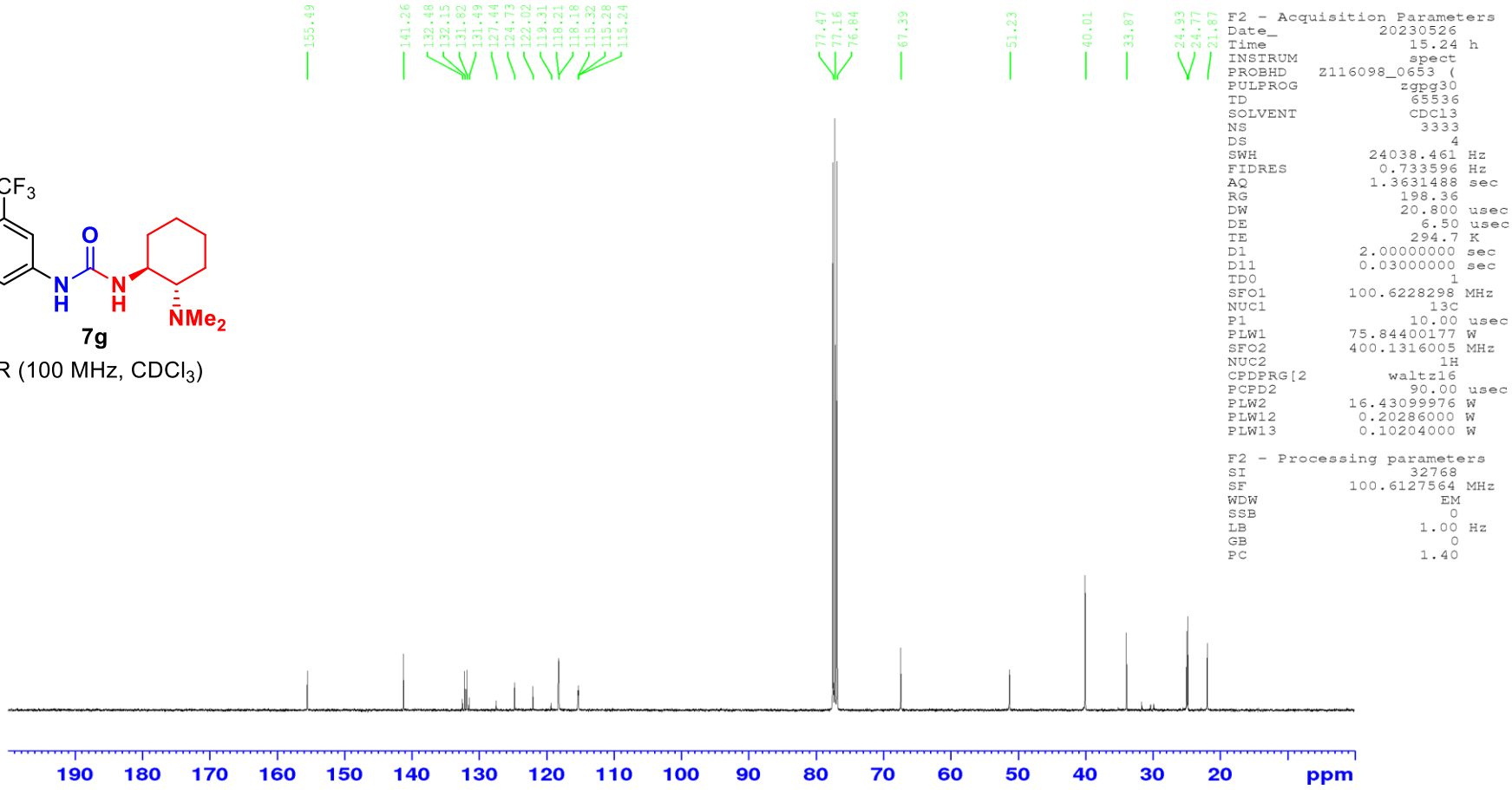
F2 - Acquisition Parameters
Date_                     20230526
Time                         12.12 h
INSTRUM                      spect
PROBHD    Z116098_0653 (
PULPROG                       zg30
TD                           65536
SOLVENT                      CDCl3
NS                              16
DS                               2
SWH                       8012.820 Hz
FIDRES                    0.244532 Hz
AQ                        4.0894465 sec
RG                           70.89
DW                           62.400 usec
DE                            6.50 usec
TE                           293.8 K
D1                        1.00000000 sec
TD0                              1
SFO1                  400.1324708 MHz
NUC1                            1H
P1                           10.00 usec
PLW1                 16.43099976 W

F2 - Processing parameters
SI                           65536
SF                    400.1300099 MHz
WDW                             EM
SSB                              0
LB                            0.30 Hz
GB                               0
PC                            1.00
```



7g

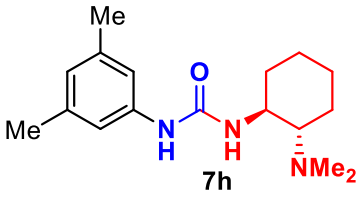
^{13}C NMR (100 MHz, $CDCl_3$)




```
Current Data Parameters
NAME                  myf-20230526
EXPNO                            2
PROCNO                           1

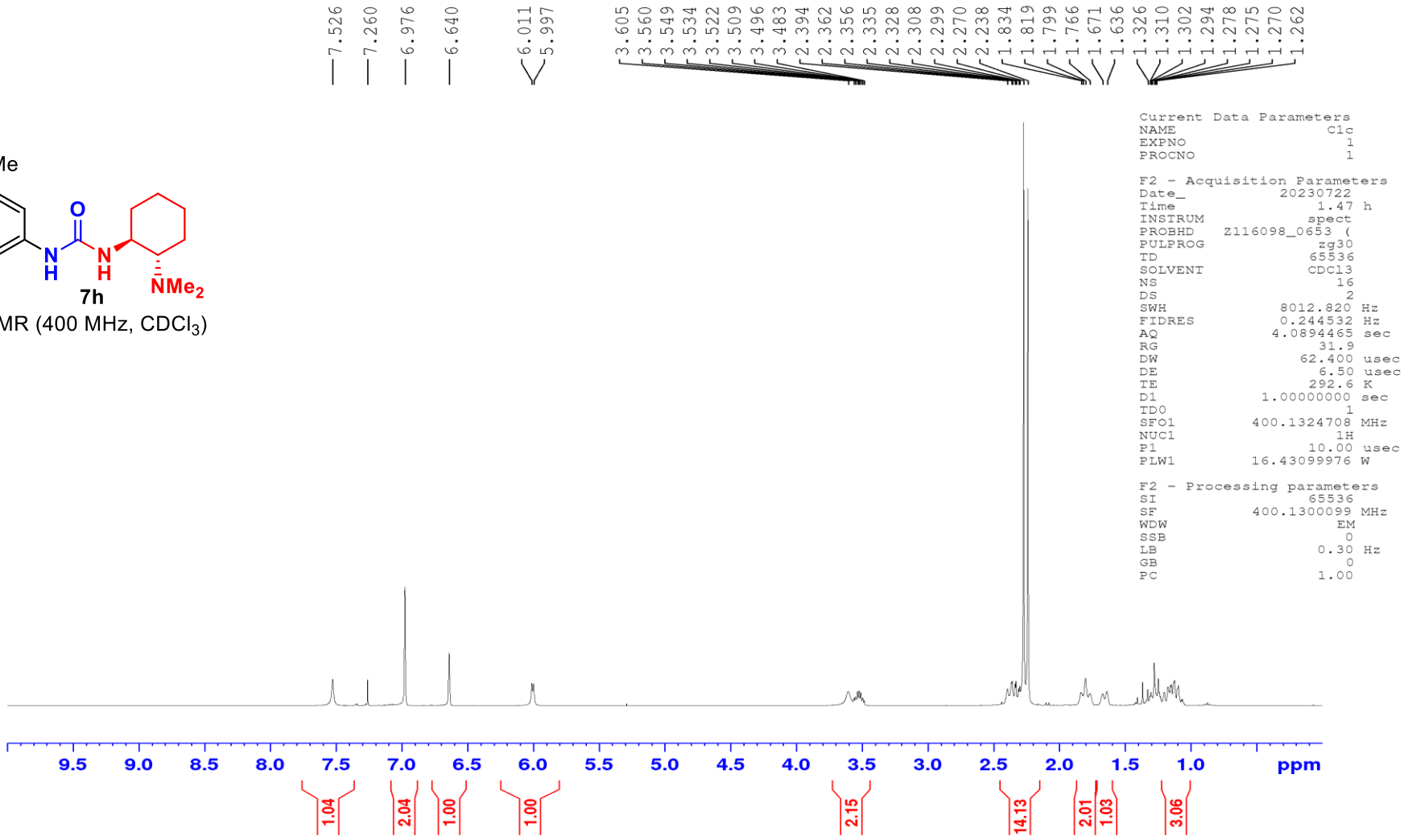
F2 - Acquisition Parameters
Date_                     20230526
Time                         15.24 h
INSTRUM                      spect
PROBHD    Z116098_0653 (
PULPROG                   zgpg30
TD                            65536
SOLVENT                       CDCl3
NS                             3333
DS                                4
SWH                   24038.461 Hz
FIDRES                 0.733596 Hz
AQ                    1.3631488 sec
RG                           198.36
DW                           20.800 usec
DE                             6.50 usec
TE                            294.7 K
D1                    2.00000000 sec
D11                   0.03000000 sec
TD0                               1
SFO1                100.6228298 MHz
NUC1                         13C
P1                           10.00 usec
PLW1                75.84400177 W
SFO2                400.1316005 MHz
NUC2                         1H
CPDPRG[2                 waltz16
PCPD2                        90.00 usec
PLW2                16.43099976 W
PLW12                0.20286000 W
PLW13                0.10204000 W

F2 - Processing parameters
SI                            32768
SF                  100.6127564 MHz
WDW                             EM
SSB                              0
LB                            1.00 Hz
GB                               0
PC                            1.40
```



7h

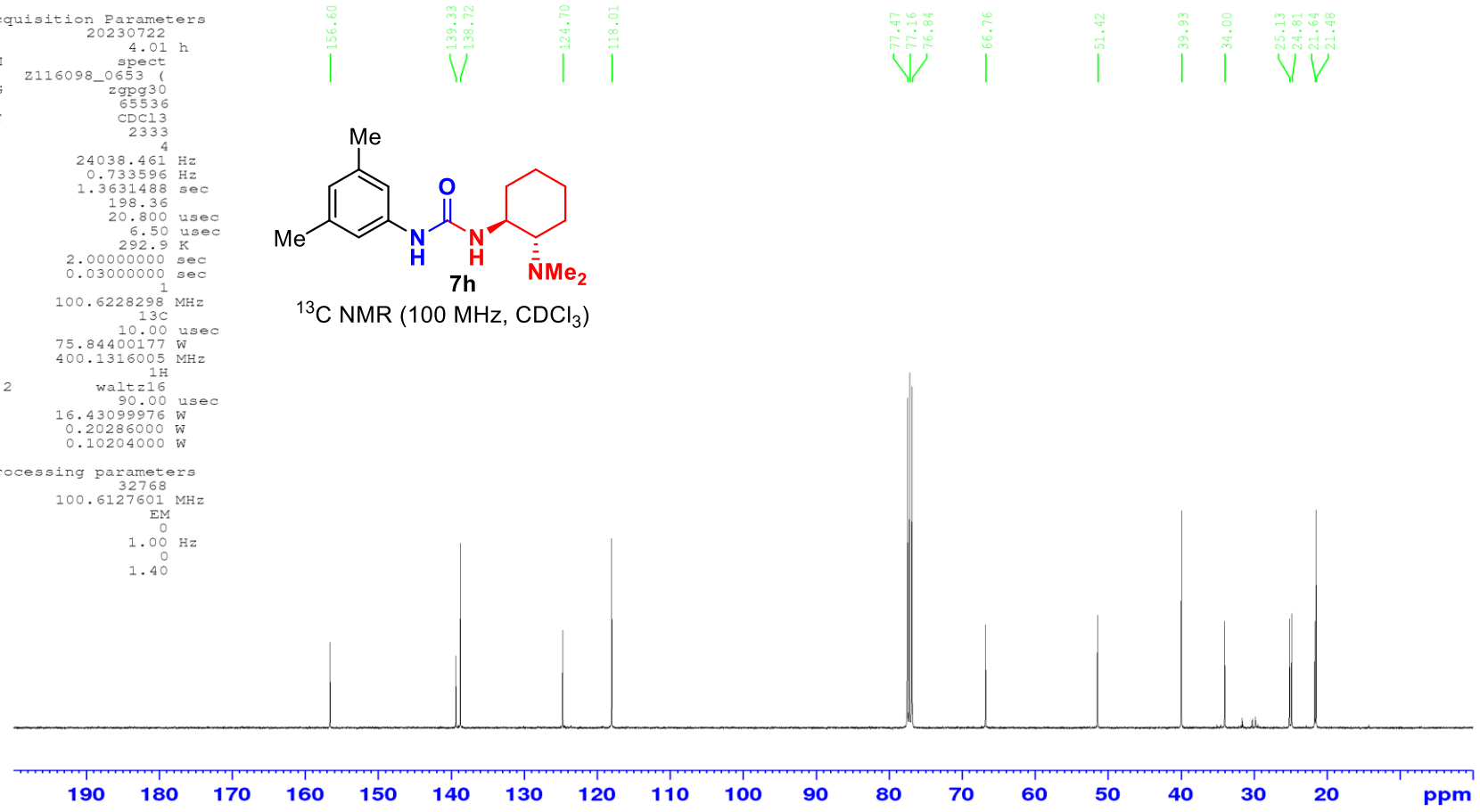
^{1}H NMR (400 MHz, $CDCl_3$)



```
Current Data Parameters
NAME                   C1c
EXPNO                    2
PROCNO                   1

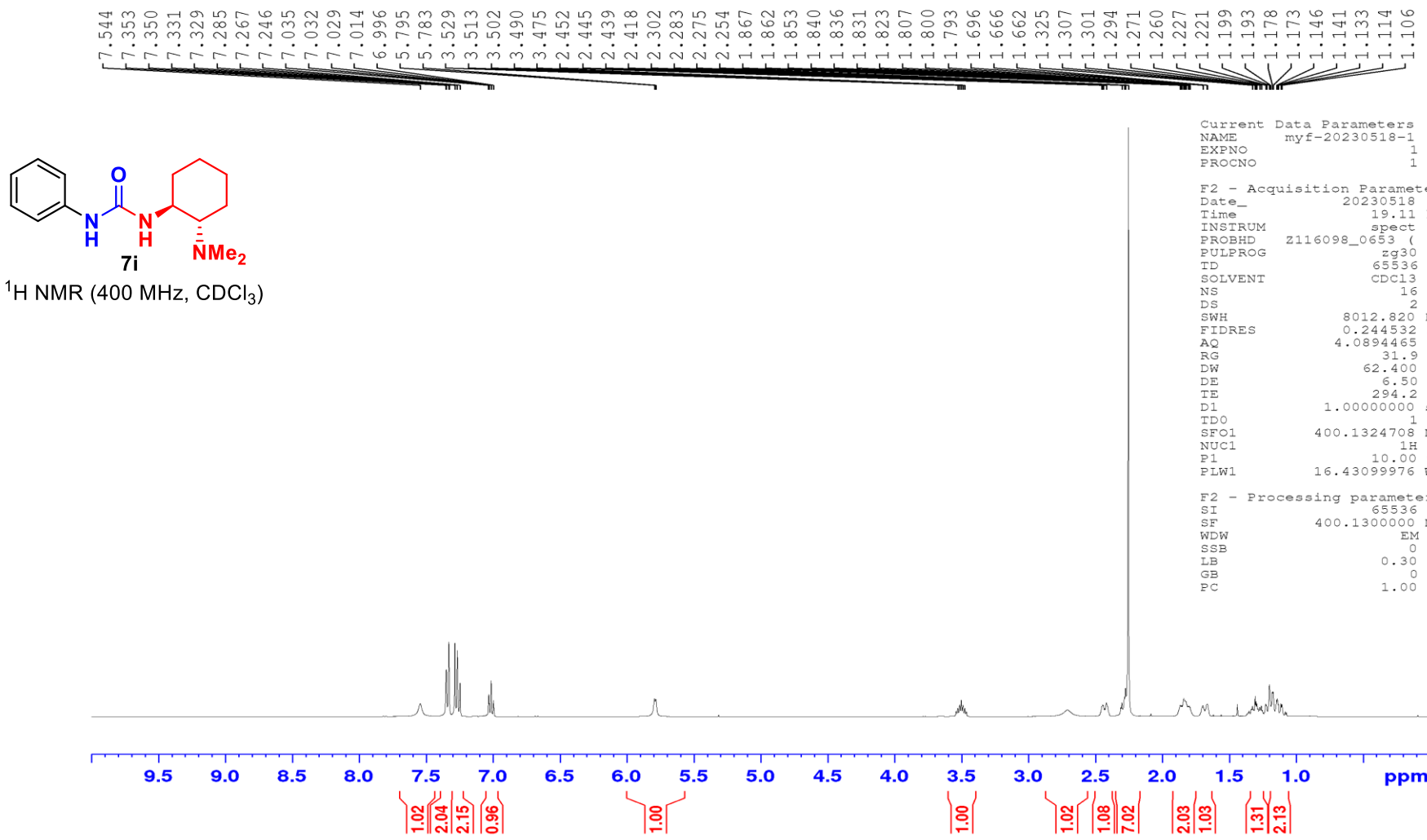
F2 - Acquisition Parameters
Date_           20230722
Time                4.01 h
INSTRUM            spect
PROBHD    Z116098_0653 (
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                 2333
DS                    4
SWH           24038.461 Hz
FIDRES         0.733596 Hz
AQ            1.3631488 sec
RG               198.36
DW               20.800 usec
DE                6.50 usec
TE               292.9 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                   1
SFO1        100.6228298 MHz
NUC1                13C
P1                10.00 usec
PLW1        75.84400177 W
SFO2        400.1316005 MHz
NUC2                1H
CPDPRG[2        waltz16
PCPD2             90.00 usec
PLW2        16.43099976 W
PLW12        0.20286000 W
PLW13        0.10204000 W

F2 - Processing parameters
SI                32768
SF          100.6127601 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```



7h

^{13}C NMR (100 MHz, $CDCl_3$)


7.544
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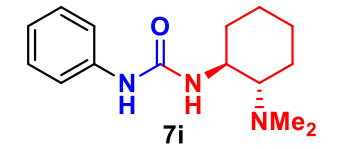
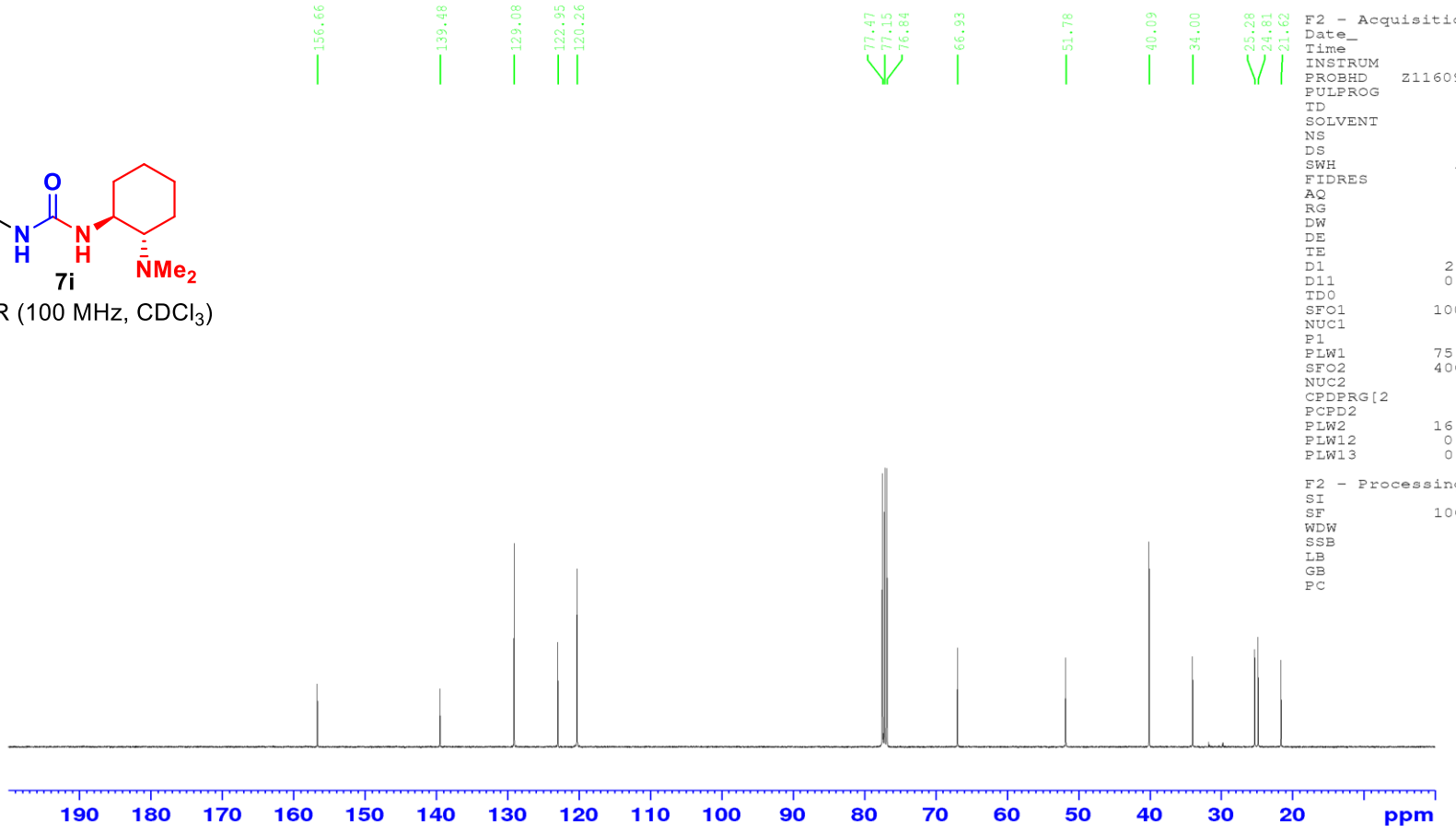
7.329
7.285
7.267
7.246
7.035
7.032
7.029
7.014
6.996
5.795
5.783
3.529
3.513
3.502
3.490
3.475
2.452
2.445
2.439
2.418
2.302
2.283
2.275
2.254
1.867
1.862
1.853
1.840
1.836
1.831
1.823
1.807
1.800
1.793
1.696
1.666
1.662
1.325
1.307
1.301
1.294
1.271
1.260
1.227
1.221
1.199
1.193
1.178
1.173
1.146
1.141
1.133
1.114
1.106
O
N
H
N
H
NMe2
7i
1H NMR (400 MHz, CDCl3)
Current Data Parameters
NAME myf-20230518-1
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20230518
Time 19.11 h
INSTRUM spect
PROBHD Z116098_0653 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.244532 Hz
AQ 4.0894465 sec
RG 31.9
DW 62.400 usec
DE 6.50 usec
TE 294.2 K
D1 1.00000000 sec
TD0 1
SFO1 400.1324708 MHz
NUC1 1H
P1 10.00 usec
PLW1 16.43099976 W
F2 - Processing parameters
SI 65536
SF 400.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
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
ppm
1.02
2.04
2.15
0.96
1.00
1.00
1.02
1.08
7.02
2.03
1.03
1.31
2.13



7i

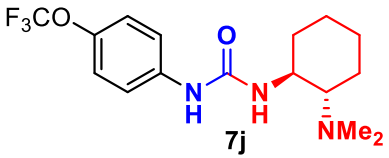
^{13}C NMR (100 MHz, $CDCl_3$)




```
Current Data Parameters
NAME      myf-20230518-1
EXPNO                  2
PROCNO                 1

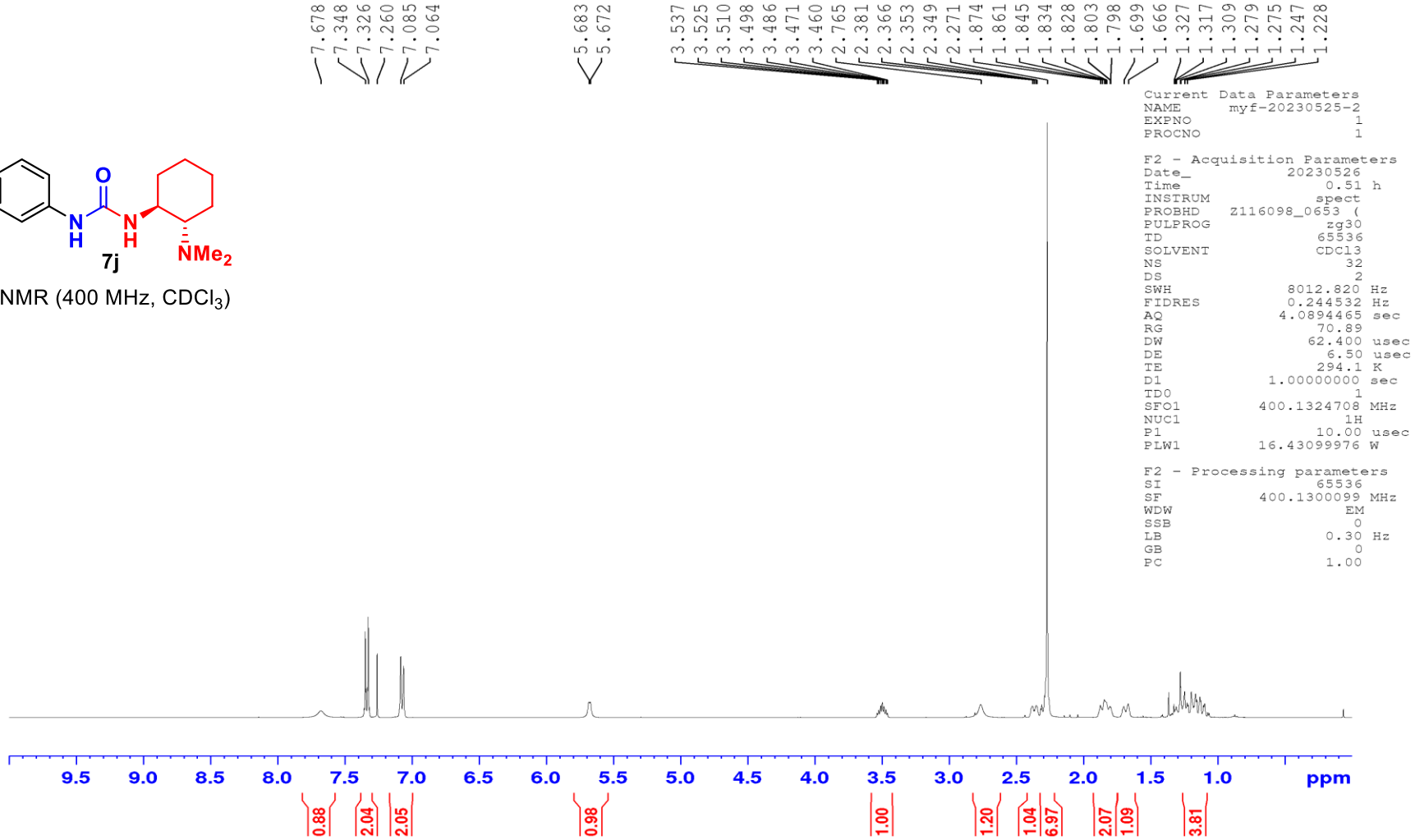
F2 - Acquisition Parameters
Date_           20230518
Time              21.45 h
INSTRUM            spect
PROBHD   Z116098_0653 (
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                 2666
DS                    4
SWH           24038.461 Hz
FIDRES         0.733596 Hz
AQ            1.3631488 sec
RG               198.36
DW               20.800 usec
DE                6.50 usec
TE                295.1 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1
SFO1        100.6228298 MHz
NUC1               13C
P1               10.00 usec
PLW1        75.84400177 W
SFO2        400.1316005 MHz
NUC2               1H
CPDPRG[2        waltz16
PCPD2            90.00 usec
PLW2        16.43099976 W
PLW12        0.20286000 W
PLW13        0.10204000 W

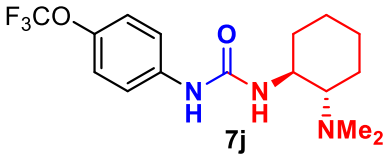
F2 - Processing parameters
SI                32768
SF          100.6127594 MHz
WDW                EM
SSB                 0
LB               1.00 Hz
GB                  0
PC               1.40
```



7j

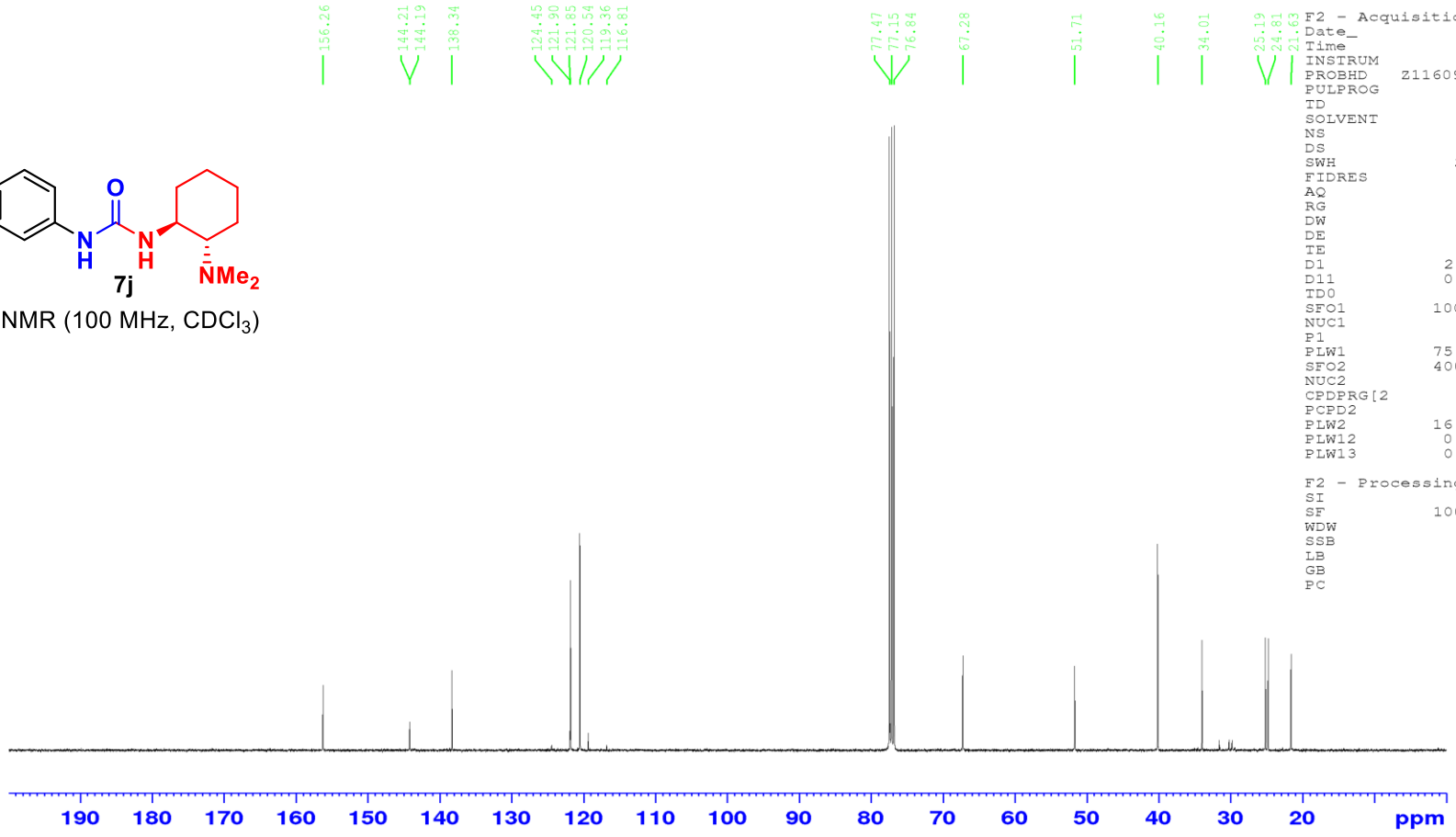
^{1}H NMR (400 MHz, $CDCl_3$)





7j

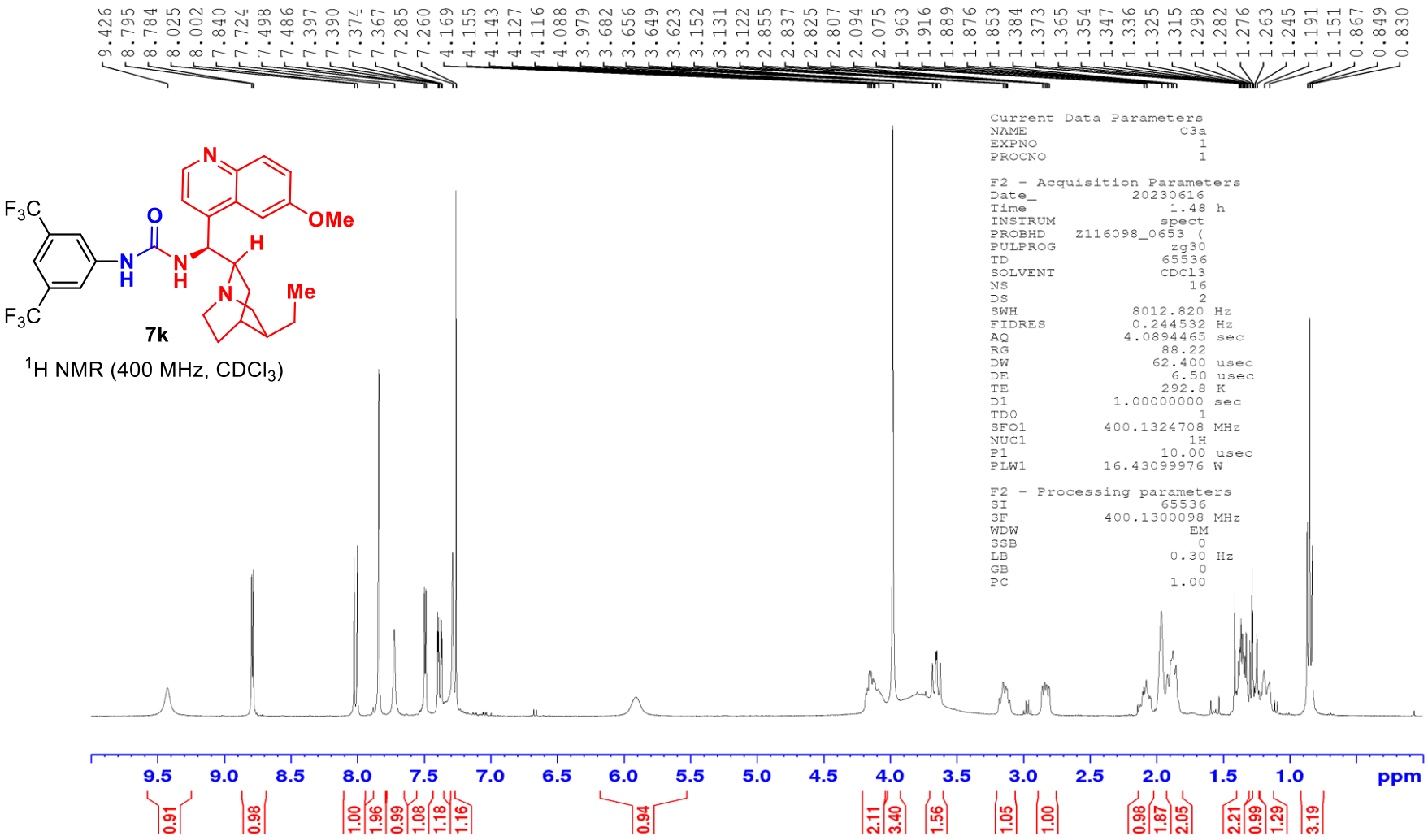
^{13}C NMR (100 MHz, $CDCl_3$)

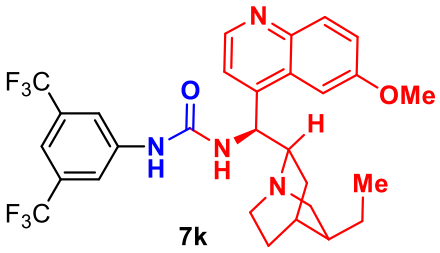



```
Current Data Parameters
NAME      myf-20230525-2
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20230526
Time              4.03 h
INSTRUM           spect
PROBHD    Z116098_0653 (
PULPROG         zgpg30
TD                65536
SOLVENT           CDCl3
NS                 3333
DS                    4
SWH           24038.461 Hz
FIDRES         0.733596 Hz
AQ             1.3631488 sec
RG                198.36
DW                20.800 usec
DE                 6.50 usec
TE                294.6 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1
SFO1        100.6228298 MHz
NUC1               13C
P1                10.00 usec
PLW1        75.84400177 W
SFO2        400.1316005 MHz
NUC2               1H
CPDPRG[2       waltz16
PCPD2             90.00 usec
PLW2        16.43099976 W
PLW12        0.20286000 W
PLW13        0.10204000 W

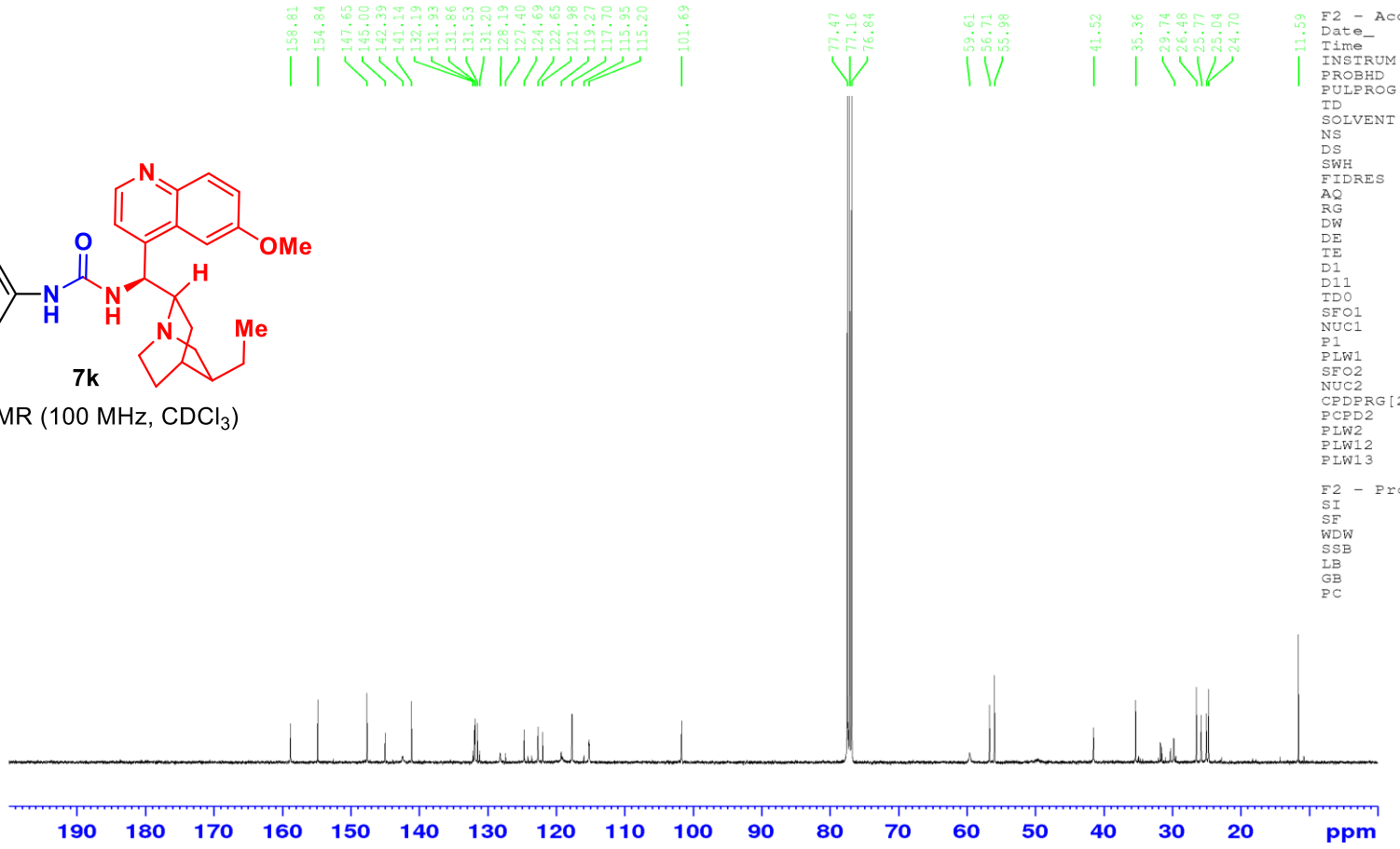
F2 - Processing parameters
SI                32768
SF          100.6127571 MHz
WDW                EM
SSB                 0
LB                1.00 Hz
GB                  0
PC                1.40
```


9.426
8.795
8.784
8.025
8.002
7.840
7.724
7.498
7.486
7.397
7.390
7.374
7.367
7.285
7.260
4.169
4.155
4.143
4.127
4.116
4.088
3.979
3.682
3.656
3.649
3.623
3.152
3.131
3.122
2.855
2.837
2.825
2.807
2.094
2.075
1.963
1.916
1.889
1.876
1.853
1.384
1.373
1.365
1.354
1.347
1.336
1.325
1.315
1.298
1.282
1.276
1.263
1.245
1.191
1.151
0.867
0.849
0.830
F3C
F3C
O
N
H
N
H
N
OMe
H
N
Me
7k
1H NMR (400 MHz, CDCl3)
Current Data Parameters
NAME C3a
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20230616
Time 1.48 h
INSTRUM spect
PROBHD Z116098_0653 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.244532 Hz
AQ 4.0894465 sec
RG 88.22
DW 62.400 usec
DE 6.50 usec
TE 292.8 K
D1 1.00000000 sec
TD0 1
SFO1 400.1324708 MHz
NUC1 1H
P1 10.00 usec
PLW1 16.43099976 W
F2 - Processing parameters
SI 65536
SF 400.1300098 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
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
ppm
0.91
0.98
1.00
1.96
0.99
1.08
1.18
1.16
0.94
2.11
3.40
1.56
1.05
1.00
0.98
1.87
2.05
2.21
0.99
1.29
3.19



7k

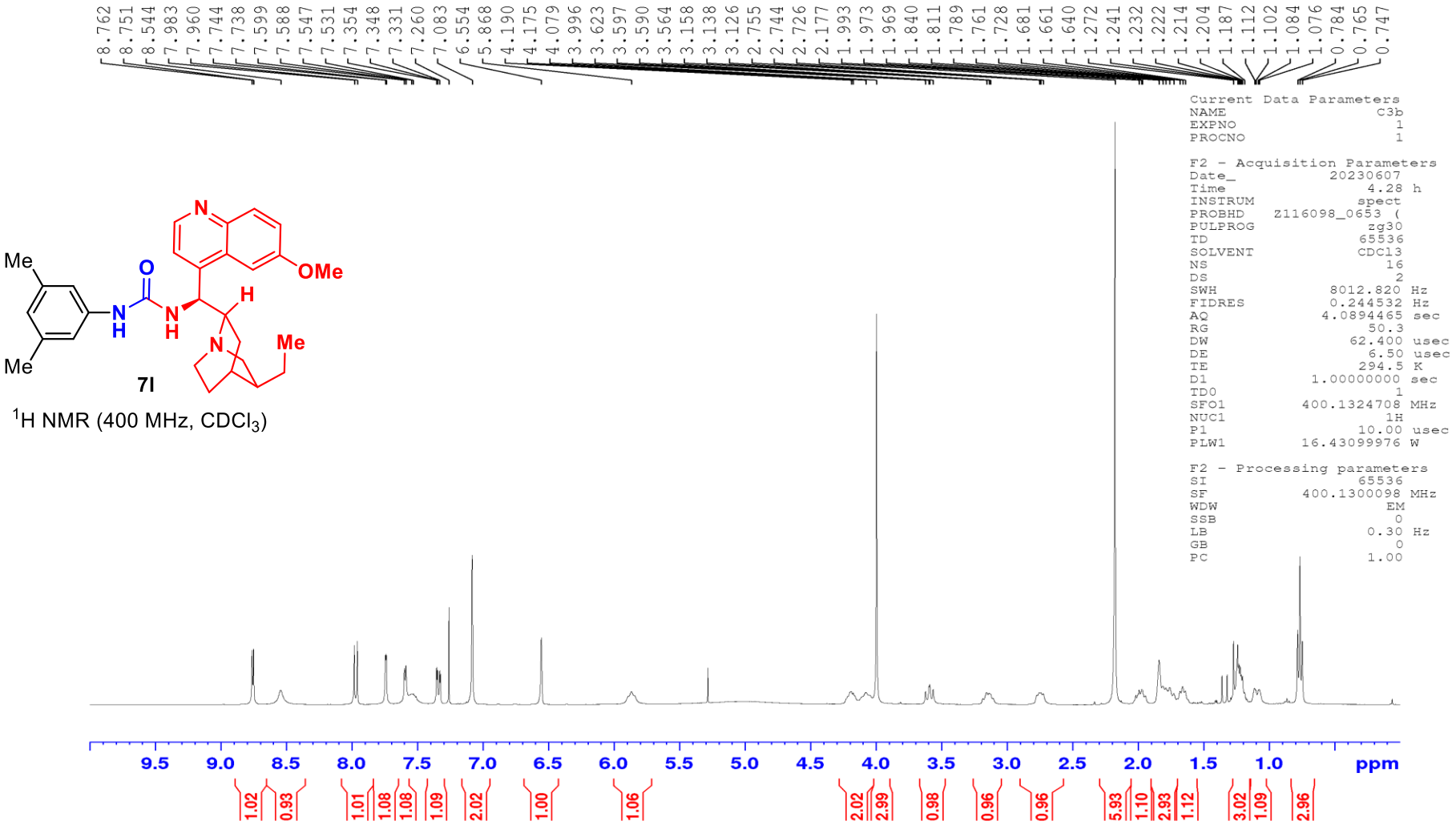
^{13}C NMR (100 MHz, $CDCl_3$)




```
Current Data Parameters
NAME                   C3a
EXPNO                    2
PROCNO                   1

F2 - Acquisition Parameters
Date_               20230617
Time                   2.52 h
INSTRUM               spect
PROBHD    Z116098_0653 (
PULPROG             zgpg30
TD                   65536
SOLVENT              CDCl3
NS                    4333
DS                       4
SWH             24038.461 Hz
FIDRES          0.733596 Hz
AQ              1.3631488 sec
RG                  198.36
DW                  20.800 usec
DE                    6.50 usec
TE                   293.2 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                      1
SFO1          100.6228298 MHz
NUC1                  13C
P1                   10.00 usec
PLW1          75.84400177 W
SFO2          400.1316005 MHz
NUC2                  1H
CPDPRG[2          waltz16
PCPD2                90.00 usec
PLW2          16.43099976 W
PLW12          0.20286000 W
PLW13          0.10204000 W

F2 - Processing parameters
SI                   32768
SF            100.6127579 MHz
WDW                     EM
SSB                      0
LB                    1.00 Hz
GB                       0
PC                    1.40
```


8.762
8.751
8.544
7.983
7.960
7.744
7.738
7.599
7.588
7.547
7.531
7.354
7.348
7.331
7.260
7.083
6.554
5.868
4.190
4.175
4.079
3.996
3.623
3.597
3.590
3.564
3.158
3.138
3.126
2.755
2.744
2.726
2.177
1.993
1.973
1.969
1.840
1.811
1.789
1.761
1.728
1.681
1.661
1.640
1.272
1.241
1.232
1.222
1.214
1.204
1.187
1.112
1.102
1.084
1.076
0.784
0.765
0.747
Me
Me
O
N
H
N
H
N
H
OMe
N
Me
7l
1H NMR (400 MHz, CDCl3)
Current Data Parameters
NAME C3b
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20230607
Time 4.28 h
INSTRUM spect
PROBHD Z116098_0653 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.244532 Hz
AQ 4.0894465 sec
RG 50.3
DW 62.400 usec
DE 6.50 usec
TE 294.5 K
D1 1.00000000 sec
TD0 1
SFO1 400.1324708 MHz
NUC1 1H
P1 10.00 usec
PLW1 16.43099976 W
F2 - Processing parameters
SI 65536
SF 400.1300098 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
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
ppm
1.02
0.93
1.01
1.08
1.08
1.09
2.02
1.00
1.06
2.02
2.99
0.98
0.96
0.96
5.93
1.10
2.93
1.12
3.02
1.09
2.96

```
Current Data Parameters
NAME                   C3b
EXPNO                    2
PROCNO                   1

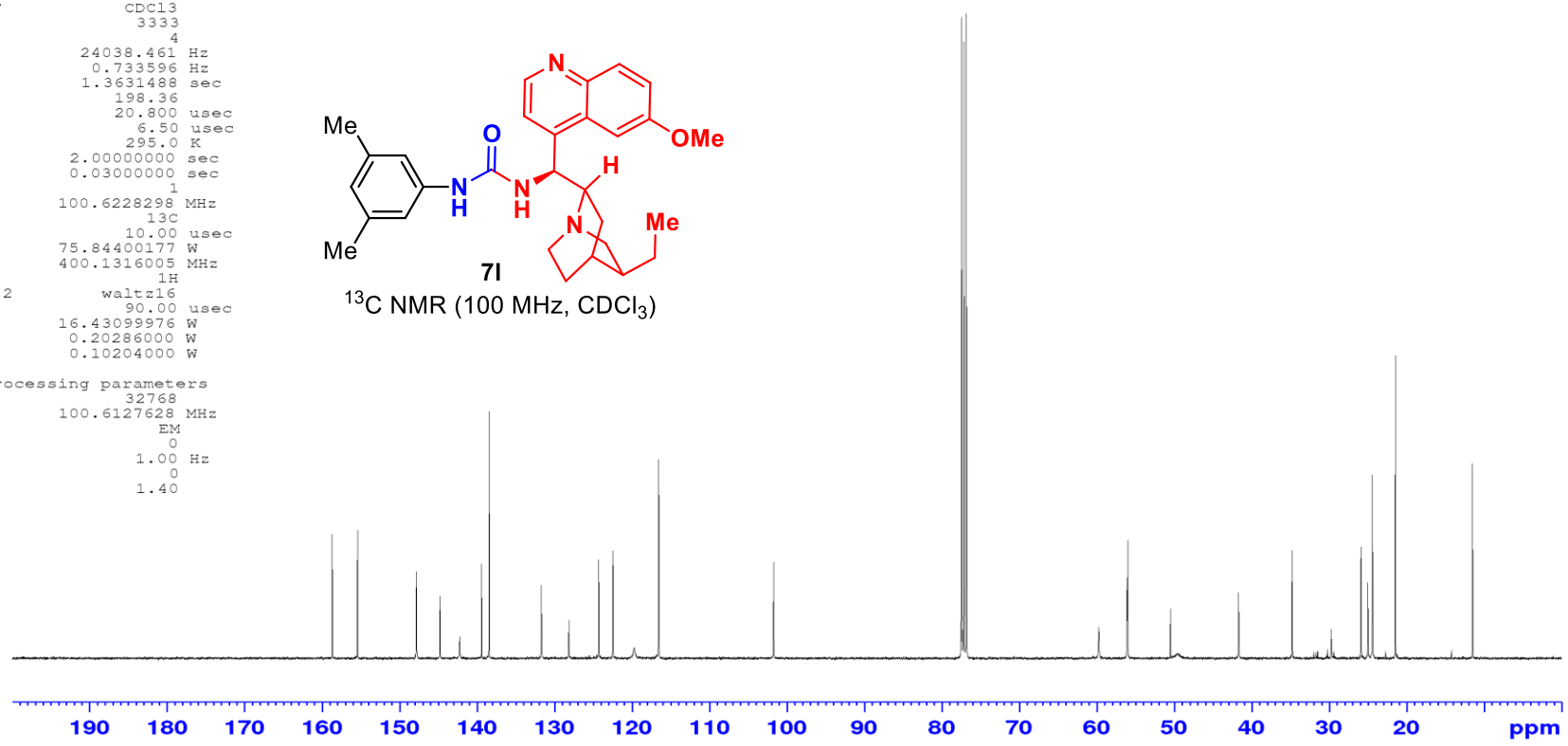
F2 - Acquisition Parameters
Date_           20230606
Time                6.26 h
INSTRUM            spect
PROBHD    Z116098_0653 (
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                 3333
DS                    4
SWH           24038.461 Hz
FIDRES         0.733596 Hz
AQ            1.3631488 sec
RG               198.36
DW               20.800 usec
DE                6.50 usec
TE                295.0 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1
SFO1        100.6228298 MHz
NUC1               13C
P1               10.00 usec
PLW1        75.84400177 W
SFO2        400.1316005 MHz
NUC2               1H
CPDPRG[2       waltz16
PCPD2            90.00 usec
PLW2        16.43099976 W
PLW12        0.20286000 W
PLW13        0.10204000 W

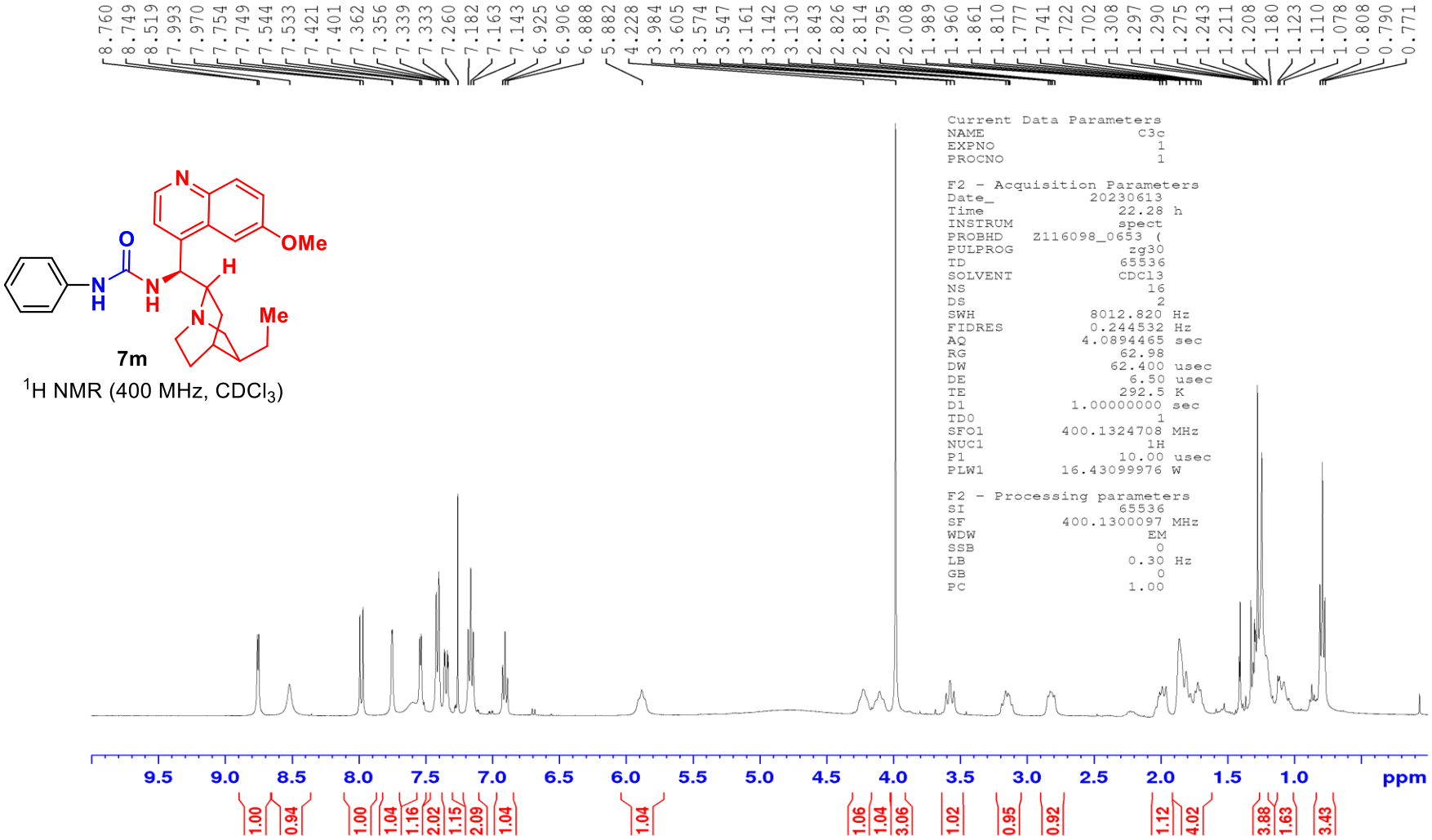
F2 - Processing parameters
SI                32768
SF          100.6127628 MHz
WDW                EM
SSB                 0
LB               1.00 Hz
GB                  0
PC               1.40
```

158.68 155.42 147.83 144.78 142.22 139.41 138.43 131.70 128.14 124.27 122.45 119.70 116.55 101.71 77.47 77.16 76.84 59.75 56.09 56.02 50.51 41.70 34.79 29.75 25.90 25.02 24.44 21.47 11.51

7l

^{13}C NMR (100 MHz, $CDCl_3$)

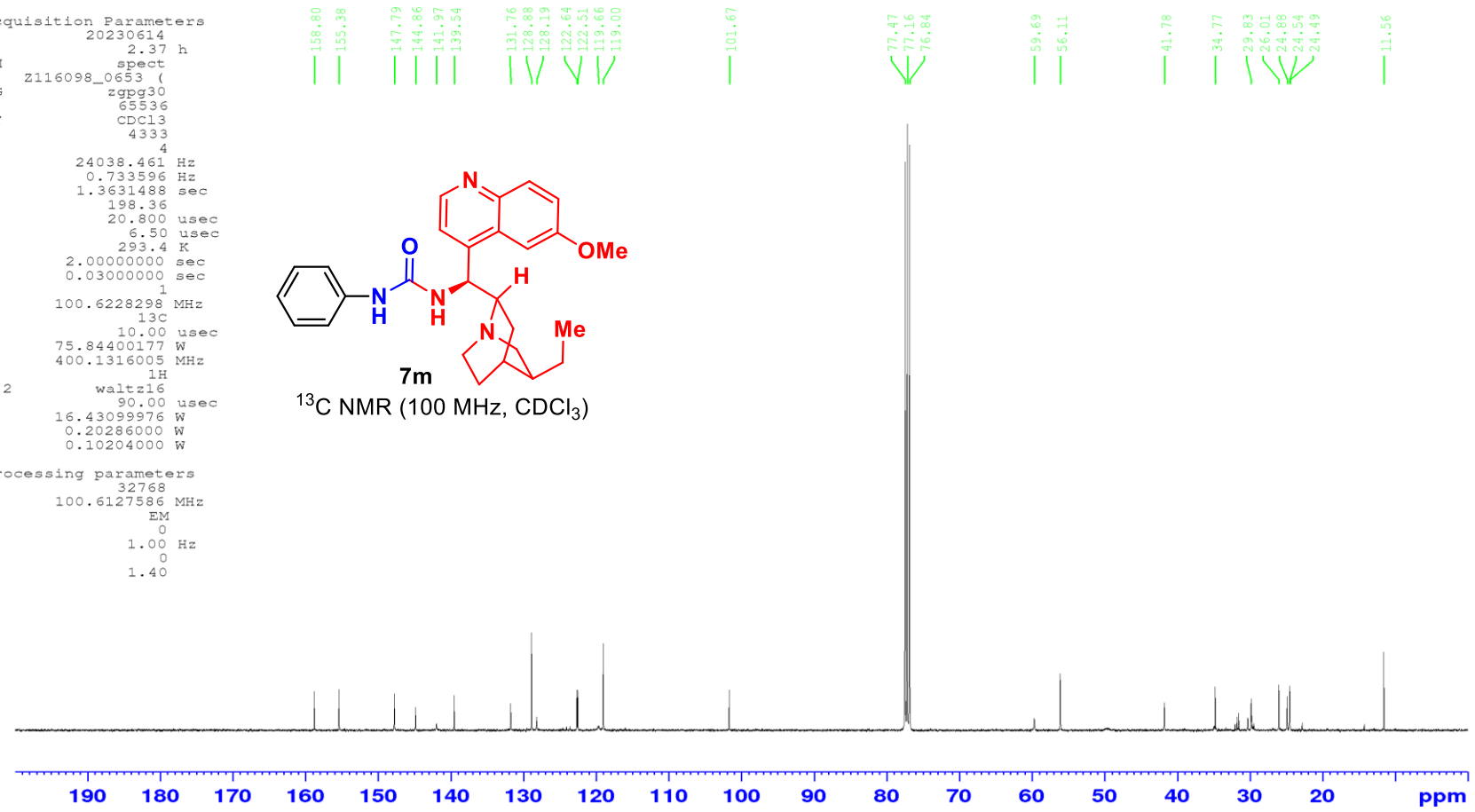


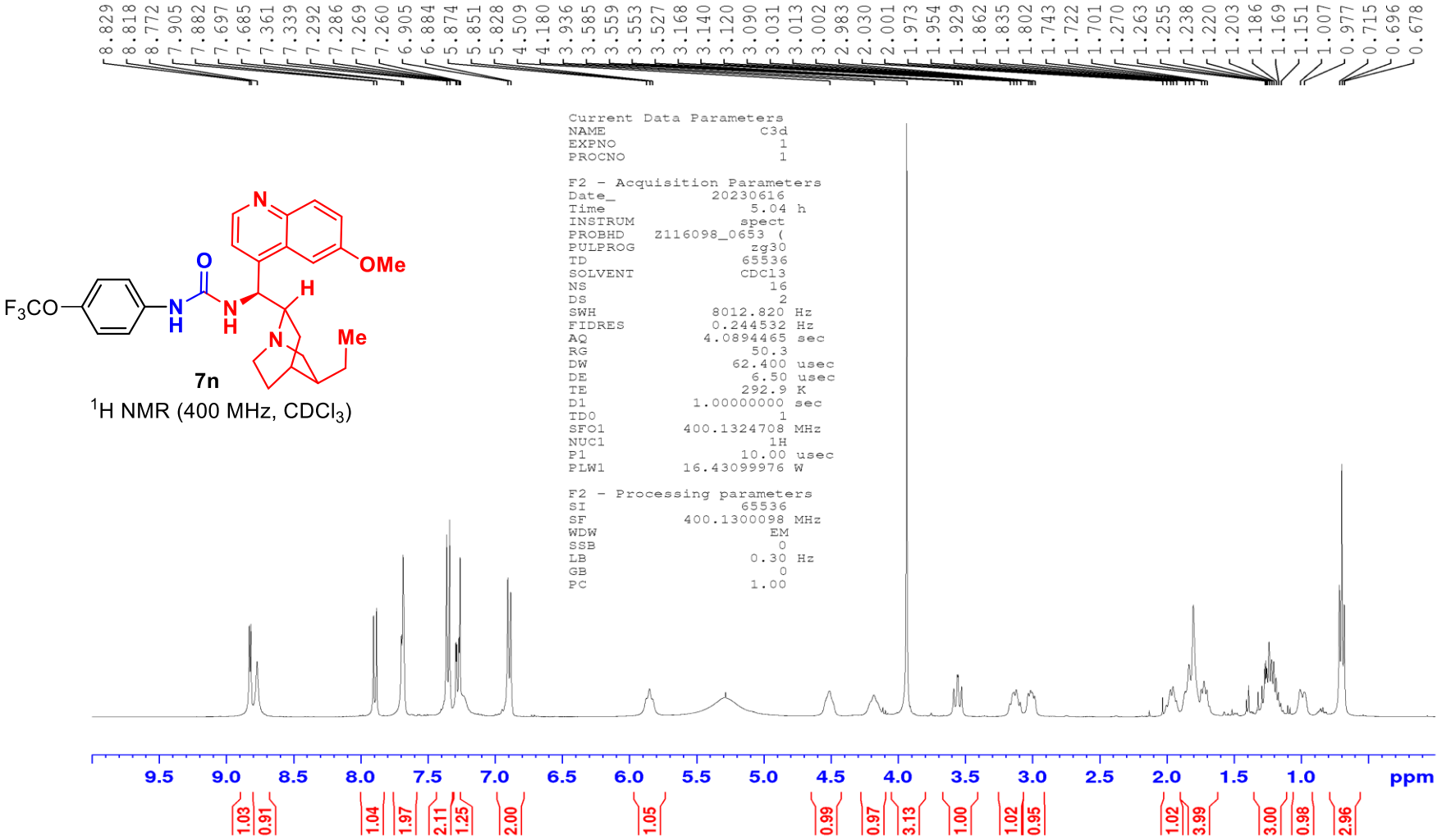

8.760
8.749
8.519
7.993
7.970
7.754
7.749
7.544
7.533
7.421
7.401
7.362
7.356
7.339
7.333
7.260
7.182
7.163
7.143
6.925
6.906
6.888
5.882
4.228
3.984
3.605
3.574
3.547
3.161
3.142
3.130
2.843
2.826
2.814
2.795
2.008
1.989
1.960
1.861
1.810
1.777
1.741
1.722
1.702
1.308
1.297
1.290
1.275
1.243
1.211
1.208
1.180
1.123
1.110
1.078
0.808
0.790
0.771
N
O
OMe
N
H
N
H
H
N
Me
7m
1H NMR (400 MHz, CDCl3)
Current Data Parameters
NAME C3c
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20230613
Time 22.28 h
INSTRUM spect
PROBHD Z116098_0653 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.244532 Hz
AQ 4.0894465 sec
RG 62.98
DW 62.400 usec
DE 6.50 usec
TE 292.5 K
D1 1.00000000 sec
TD0 1
SFO1 400.1324708 MHz
NUC1 1H
P1 10.00 usec
PLW1 16.43099976 W
F2 - Processing parameters
SI 65536
SF 400.1300097 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
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
ppm
1.00
0.94
1.00
1.04
1.16
2.02
1.15
2.09
1.04
1.04
1.06
1.04
3.06
1.02
0.95
0.92
1.12
4.02
3.88
1.63
3.43

```
Current Data Parameters
NAME                   C3c
EXPNO                    2
PROCNO                   1

F2 - Acquisition Parameters
Date_             20230614
Time                 2.37 h
INSTRUM              spect
PROBHD    Z116098_0653 (
PULPROG            zgpg30
TD                  65536
SOLVENT             CDCl3
NS                   4333
DS                      4
SWH             24038.461 Hz
FIDRES           0.733596 Hz
AQ               1.3631488 sec
RG                  198.36
DW                  20.800 usec
DE                   6.50 usec
TE                  293.4 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                     1
SFO1          100.6228298 MHz
NUC1                  13C
P1                  10.00 usec
PLW1          75.84400177 W
SFO2          400.1316005 MHz
NUC2                  1H
CPDPRG[2          waltz16
PCPD2               90.00 usec
PLW2          16.43099976 W
PLW12          0.20286000 W
PLW13          0.10204000 W

F2 - Processing parameters
SI                  32768
SF            100.6127586 MHz
WDW                    EM
SSB                     0
LB                   1.00 Hz
GB                      0
PC                   1.40
```



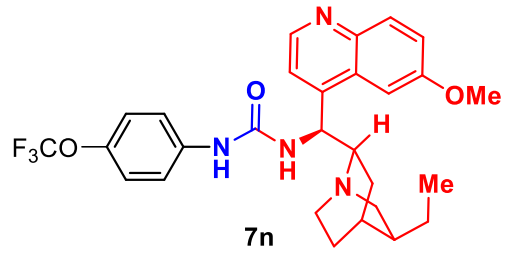

8.829
8.818
8.772
7.905
7.882
7.697
7.685
7.361
7.339
7.292
7.286
7.269
7.260
6.905
6.884
5.874
5.851
5.828
4.509
4.180
3.936
3.585
3.559
3.553
3.527
3.168
3.140
3.120
3.090
3.031
3.013
3.002
2.983
2.030
2.001
1.973
1.954
1.929
1.862
1.835
1.802
1.743
1.722
1.701
1.270
1.263
1.255
1.238
1.220
1.203
1.186
1.169
1.151
1.007
0.977
0.715
0.696
0.678
F3CO
N
H
O
N
H
N
OMe
H
N
Me
7n
1H NMR (400 MHz, CDCl3)
Current Data Parameters
NAME C3d
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20230616
Time 5.04 h
INSTRUM spect
PROBHD Z116098_0653 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.244532 Hz
AQ 4.0894465 sec
RG 50.3
DW 62.400 usec
DE 6.50 usec
TE 292.9 K
D1 1.00000000 sec
TD0 1
SFO1 400.1324708 MHz
NUC1 1H
P1 10.00 usec
PLW1 16.43099976 W
F2 - Processing parameters
SI 65536
SF 400.1300098 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
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
ppm
1.03
0.91
1.04
1.97
2.11
1.25
2.00
1.05
0.99
0.97
3.13
1.00
1.02
0.95
1.02
3.99
3.00
0.98
2.96

```
Current Data Parameters
NAME                   C3d
EXPNO                    2
PROCNO                   1

F2 - Acquisition Parameters
Date_             20230616
Time                 8.16 h
INSTRUM              spect
PROBHD    Z116098_0653 (
PULPROG            zgpg30
TD                  65536
SOLVENT             CDCl3
NS                   3333
DS                      4
SWH             24038.461 Hz
FIDRES           0.733596 Hz
AQ               1.3631488 sec
RG                 198.36
DW                 20.800 usec
DE                  6.50 usec
TE                 293.5 K
D1              2.00000000 sec
D11             0.03000000 sec
TD0                     1
SFO1           100.6228298 MHz
NUC1                  13C
P1                 10.00 usec
PLW1           75.84400177 W
SFO2           400.1316005 MHz
NUC2                  1H
CPDPRG[2          waltz16
PCPD2              90.00 usec
PLW2           16.43099976 W
PLW12           0.20286000 W
PLW13           0.10204000 W

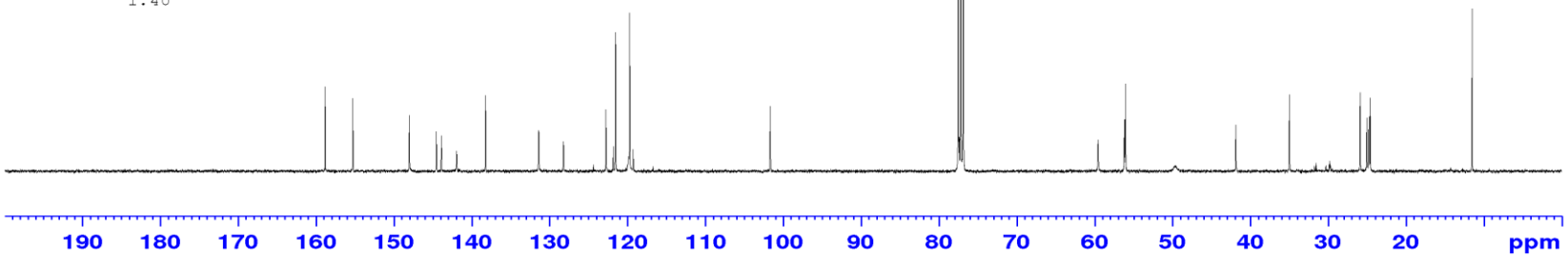
F2 - Processing parameters
SI                  32768
SF             100.6127594 MHz
WDW                    EM
SSB                     0
LB                   1.00 Hz
GB                      0
PC                   1.40
```

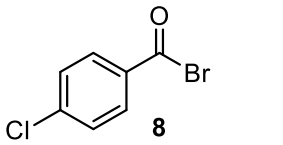
158.82 155.26 148.00 144.53 143.90 141.93 138.21 131.38 128.21 124.34 122.75 121.79 121.50 119.70 119.25 116.70 101.64 77.47 77.36 77.16 76.84 59.53 56.14 56.01 49.60 41.84 34.95 25.86 25.01 24.75 24.58 11.48



7n

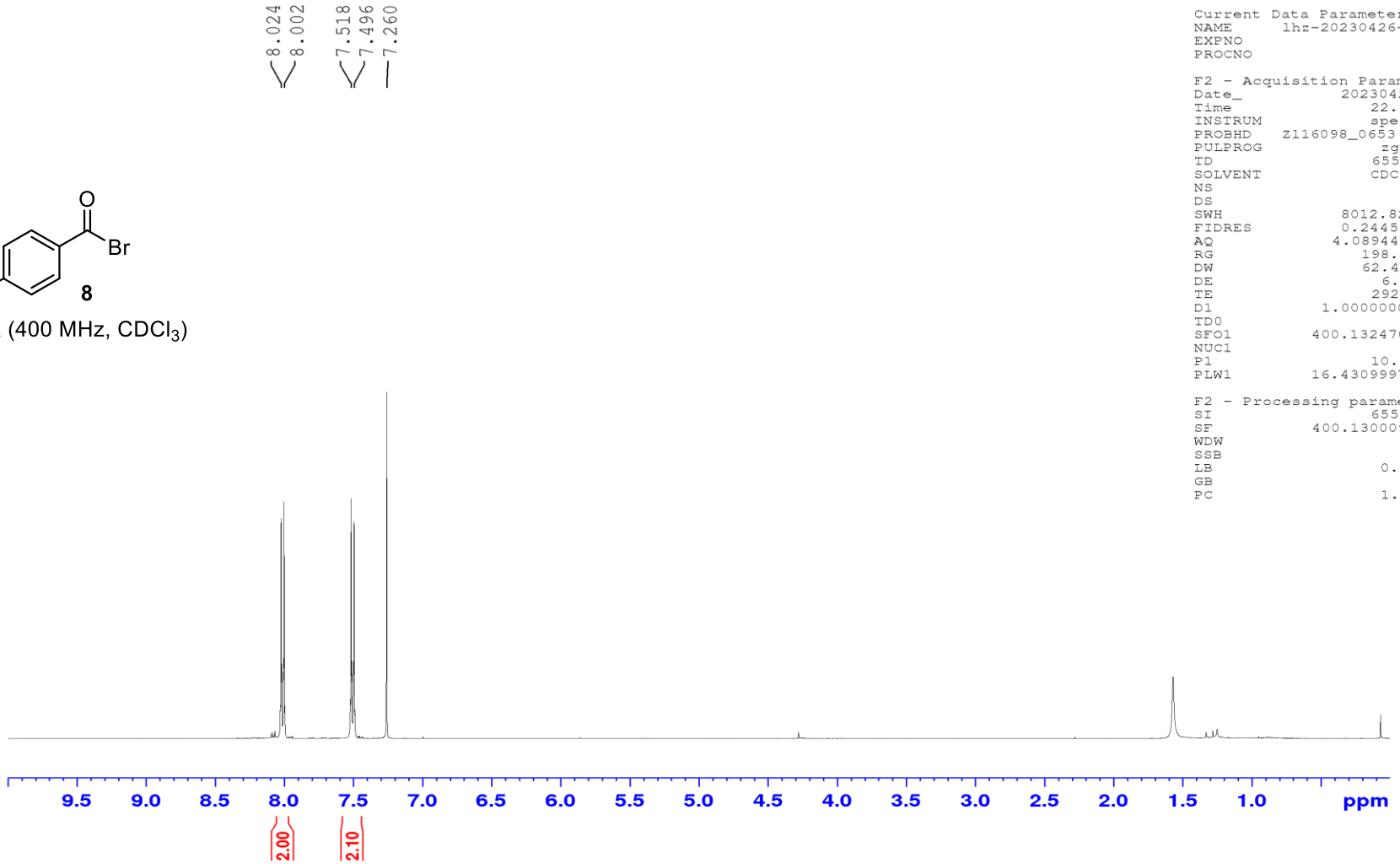
^{13}C NMR (100 MHz, $CDCl_3$)





8

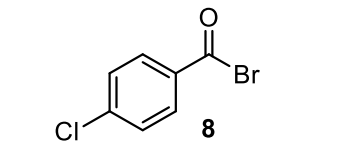
^{1}H NMR (400 MHz, $CDCl_3$)




```
Current Data Parameters
NAME      lhz-20230426-4
EXPNO                  1
PROCNO                 1

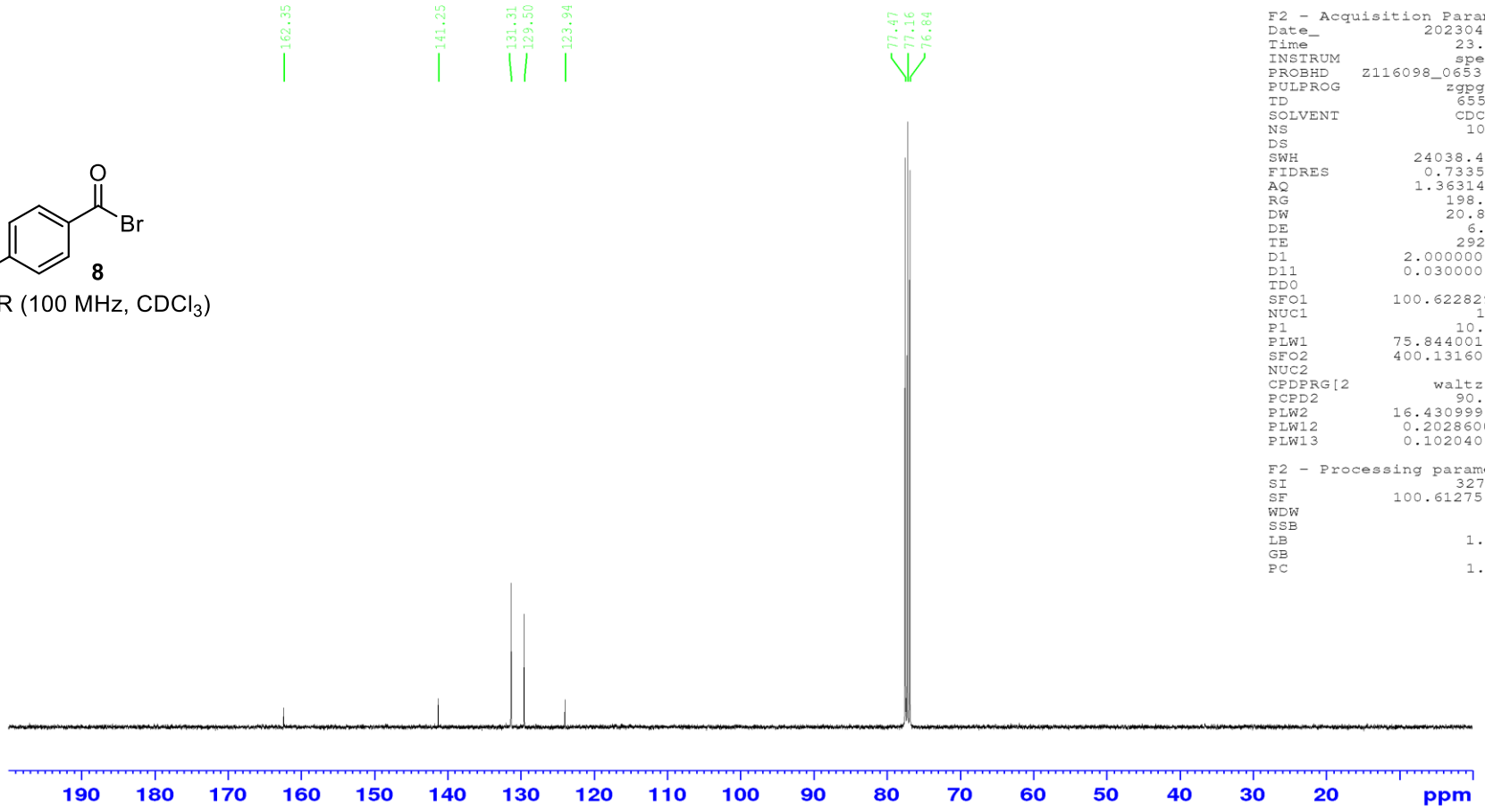
F2 - Acquisition Parameters
Date_           20230426
Time               22.30 h
INSTRUM            spect
PROBHD    Z116098_0653 (
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                   16
DS                    2
SWH            8012.820 Hz
FIDRES         0.244532 Hz
AQ             4.0894465 sec
RG                198.36
DW                62.400 usec
DE                 6.50 usec
TE                292.0 K
D1           1.00000000 sec
TD0                   1
SFO1         400.1324708 MHz
NUC1                 1H
P1                10.00 usec
PLW1         16.43099976 W

F2 - Processing parameters
SI                65536
SF           400.1300099 MHz
WDW                  EM
SSB                   0
LB                 0.30 Hz
GB                    0
PC                 1.00
```



8

^{13}C NMR (100 MHz, $CDCl_3$)




```
Current Data Parameters
NAME      1hz-20230426-4
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20230426
Time              23.30 h
INSTRUM             spect
PROBHD   Z116098_0653 (
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                 1024
DS                    4
SWH           24038.461 Hz
FIDRES         0.733596 Hz
AQ            1.3631488 sec
RG               198.36
DW               20.800 usec
DE                6.50 usec
TE               292.7 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1
SFO1        100.6228298 MHz
NUC1                13C
P1                10.00 usec
PLW1        75.84400177 W
SFO2        400.1316005 MHz
NUC2                1H
CPDPRG[2        waltz16
PCPD2             90.00 usec
PLW2        16.43099976 W
PLW12        0.20286000 W
PLW13        0.10204000 W

F2 - Processing parameters
SI                32768
SF          100.6127564 MHz
WDW                EM
SSB                 0
LB               1.00 Hz
GB                  0
PC               1.40
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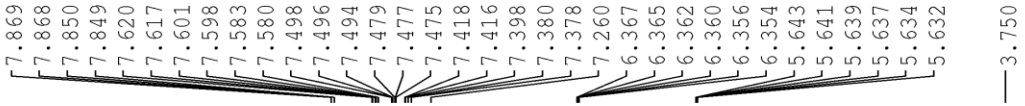

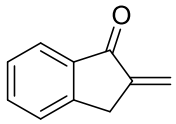

10

^{1}H NMR (400 MHz, $CDCl_3$)

```
Current Data Parameters
NAME      ztc-20230820-1
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20230820
Time              22.43 h
INSTRUM           spect
PROBHD   Z116098_0653 (
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH            8012.820 Hz
FIDRES         0.244532 Hz
AQ             4.0894465 sec
RG                 62.98
DW                62.400 usec
DE                 6.50 usec
TE                292.1 K
D1             1.00000000 sec
TD0                    1
SFO1         400.1324708 MHz
NUC1                  1H
P1                10.00 usec
PLW1         16.43099976 W

F2 - Processing parameters
SI                 65536
SF           400.1300097 MHz
WDW                   EM
SSB                    0
LB                0.30 Hz
GB                     0
PC                 1.00
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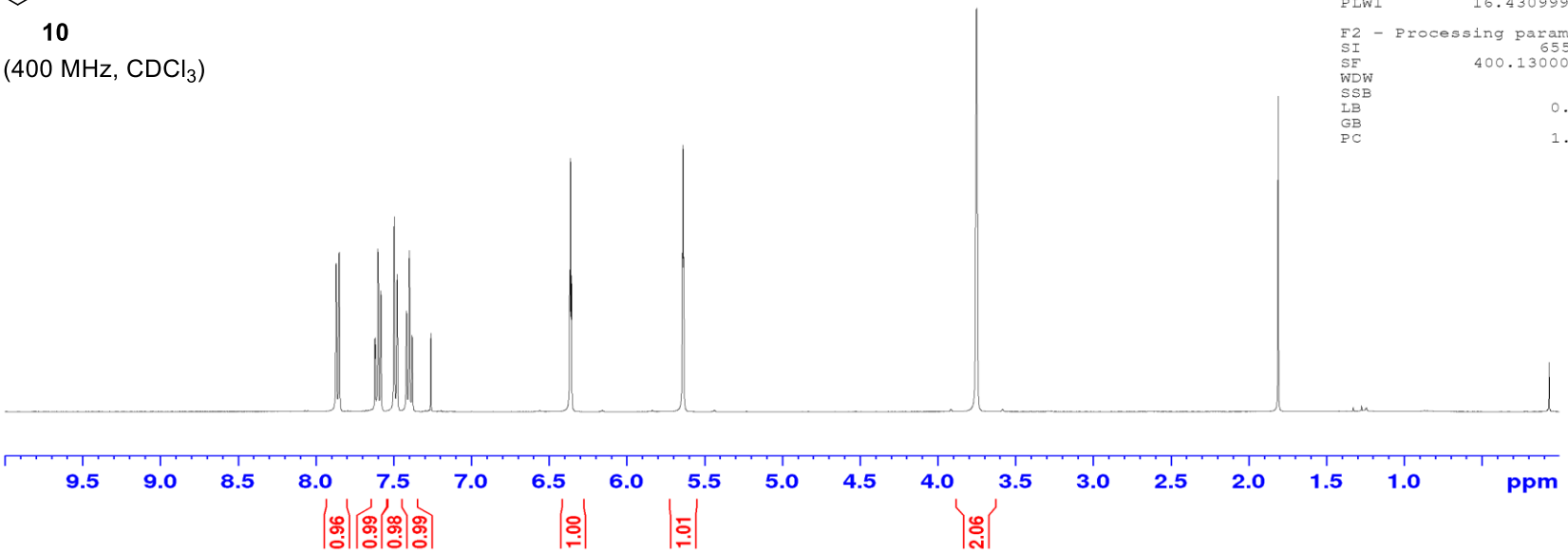
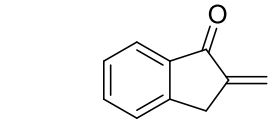
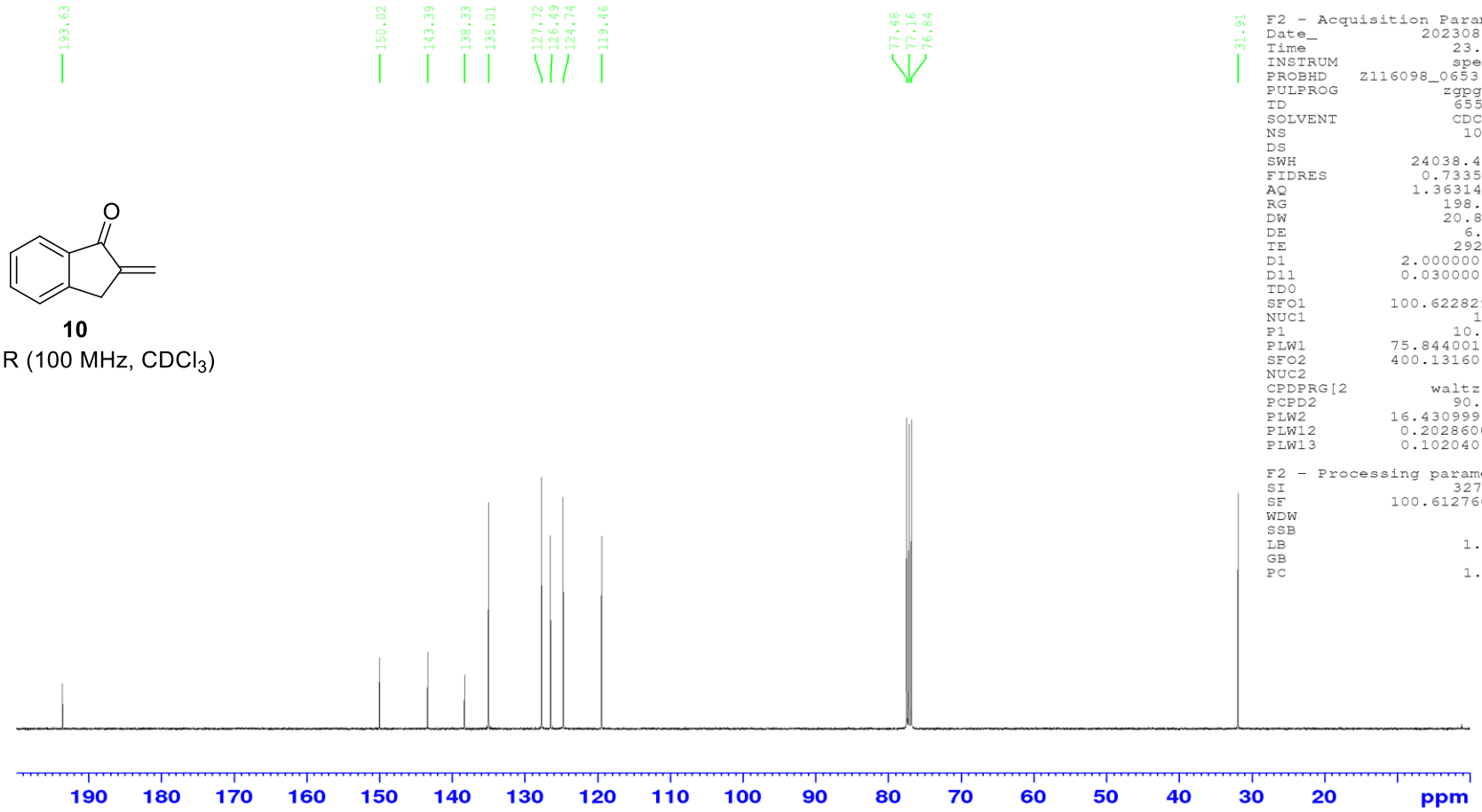




10

^{13}C NMR (100 MHz, $CDCl_3$)

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Current Data Parameters
NAME      ztc-20230820-1
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20230820
Time              23.43 h
INSTRUM             spect
PROBHD   Z116098_0653 (
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                 1024
DS                    4
SWH           24038.461 Hz
FIDRES         0.733596 Hz
AQ             1.3631488 sec
RG                198.36
DW                20.800 usec
DE                 6.50 usec
TE                292.9 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                   1
SFO1         100.6228298 MHz
NUC1                13C
P1                 10.00 usec
PLW1          75.84400177 W
SFO2         400.1316005 MHz
NUC2                1H
CPDPRG[2        waltz16
PCPD2              90.00 usec
PLW2          16.43099976 W
PLW12          0.20286000 W
PLW13          0.10204000 W

F2 - Processing parameters
SI                 32768
SF           100.6127601 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```