Supplementary Materials for

Electrochemical Oxidative Rearrangement of Tetrahydro-β-carbolines in Zero-gap Flow Cell

Yiting Zheng[#[a][c]], Yuen Tsz Cheung[#[b]], Lixin Liang[[b]], Huiying Qiu[[b]], Lei Zhang[[c]], Anson Tsang[[a]], Qing Chen[*[a][b]] & Rongbiao Tong[*[b]]

[*]Corresponding author. Email: chenqing@ust.hk; rtong@ust.hk;

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General Information

All the electrochemical experiments and synthesis were carried on electrochemical workstation VMP-300 (as Figure S1A present). Reactions in a flow cell were carried on the two PEEK housing zero-gap membrane reactor with PTFE gasket and celgard 3501, the detail information could be found in section Flow cell set-up in this file. The inlet and outlet injectors were connected to PEEK housing with SMC stainless steel air-tight connector and PTFE tube. The reaction solutions were sealed in bottle with holes, as Figure S1B present, and bumped through the PTFE tube connected with PharMed BPT tube by LongerPump BT-600-2J with YZ1515x in 5-20 mL/min.

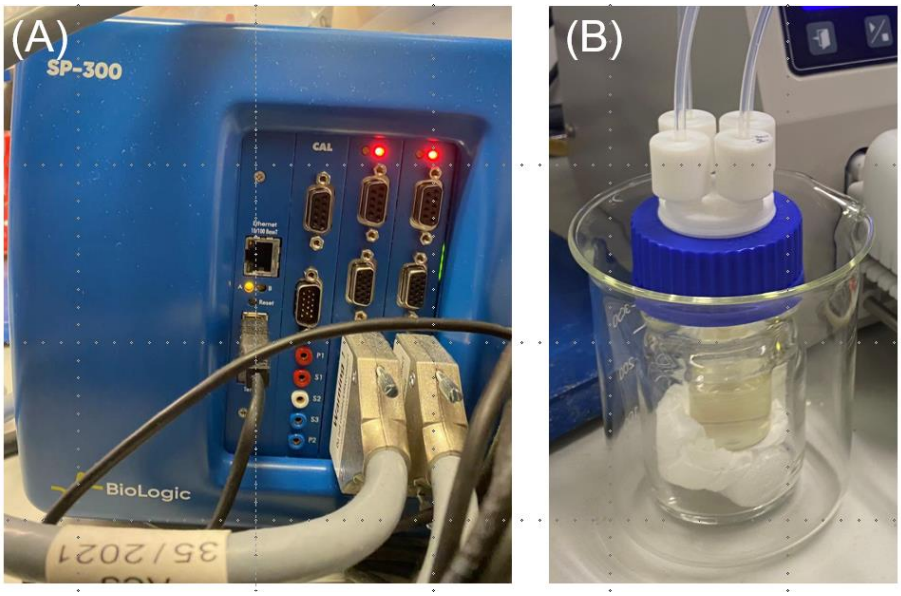
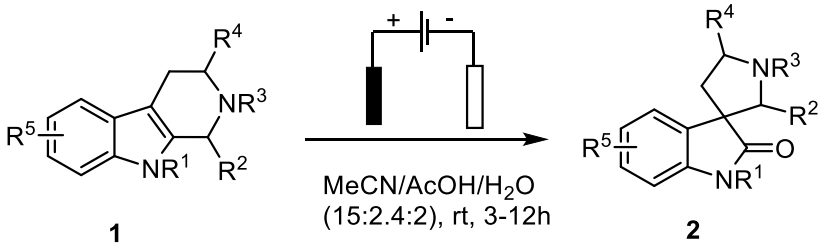


Figure S1. (A) Electrochemical workstation VMP-300; **(B)** Sealed bottle with holes connect with PTFE tubes connect with zero-gap flow cell.

Solvents used in workup, extraction and column chromatography were used as received from commercial suppliers without prior purification. Reactions for substrate preparation were magnetically stirred and monitored by thin layer chromatography (TLC, 0.25 mm) on pre-coated silica gel plates. Flash chromatography was performed with silica gel 60 (particle size 0.040 – 0.062 mm). ^{1}H and ^{13}C NMR spectra were recorded on a Bruker AV-400 spectrometer (400 MHz for ^{1}H, 100 MHz for ^{13}C). Chemical shifts are reported in parts per million (ppm) as values relative to the internal chloroform (7.26 ppm for **^{1}H NMR** and 77.16 ppm for **^{13}C NMR**). Abbreviations for signal coupling are as follows: s, singlet; d, doublet; t, triplet; q, quartet; m, multiplet; br, broad. High resolution mass spectra were measured at the Hong Kong University of Science and Technology Mass Spectrometry Service

Center on an Agilent GC/MS. Carbon paper (CP, SGL-39AA) and organic deposition on CP after the synthesis mediated by KBr were characterized on the scanning electron microscopy (SEM) (JEOL-7100, 10 kV).

General Procedure A (using flow cell, Figure S2A) of Electrochemical oxidative rearrangement of indoles to 2-oxindoles: Reactions were conducted in two 20 mL vials with anolyte and catholyte. The assemble and detail information were discussed in **Flow cell setup.** The reaction was performed in the two PEEK housing flow cell at constant voltage 1.2 V using potentiostatic method in VMP-300. Anolyte solution of **1a** (0.2 mmol) in MeCN/AcOH/H_2O (15:2.4:2, 10 mL) was added bromide salt (0.4 mmol, 2.0 equiv.). The anolyte was separated from the cathode electrolyte in H_2SO_4 (0.25M, 10 mL) by a proton-exchange membrane Nafion 117. The 20 mL/min was set for synthsis, and the cut-off charge was set 2 F/mol. Reaction was monitored by TLC. The reaction mixture was diluted with EtOAc (10 mL). Saturated aqueous Na_2CO_3 solution (10 mL) was added. The organic fraction was collected, and the aqueous fraction was extracted with EtOAc (2 x 10 mL). The combined organic fraction was washed with brine, dried over Na_2SO_4, filtered and concentrated under reduced pressure. The resulting residue was purified by flash column chromatography on silica gel using hexane/ethyl acetate eluents.



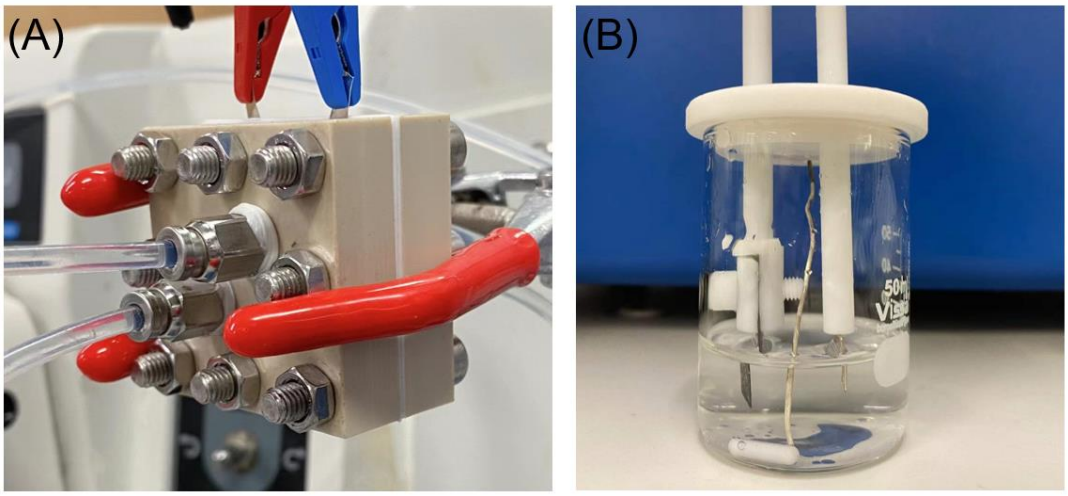


Figure S2. (A) Flow cell using in General Procedure A; **(B)** Set-up of undivided cell using in General Procedure B

General Procedure B (using undivided cell, Figure S2B) of Electrochemical oxidative rearrangement of indoles to 2-oxindoles: Reactions were conducted in a 20 mL vial with a stir bar and a carbon paper (10 mm*10 mm*0.6 mm) working electrode (anode), a platinum-plated (10 mm*10 mm*0.1 mm) counter-electrode (cathode) and Ag /AgBr reference electrode. Constant voltage mode with 2.0 V was applied by VMP-300. The Ag/AgBr electrode was made by Ag wire and HBr, the potential was calibrated with SCE and convert to -0.26 V vs. RHE. To a solution of **1** (0.1 mmol) in MeCN/AcOH/H_2O (15:2.4:2, 10 mL) was added KBr (0.05 mmol, 0.5 equiv.). The resistance in the set-up was measured 122 ohm by VMP-300. Reaction was monitored by TLC and stopped after complete conversion of **1a**. The reaction mixture was diluted with EtOAc (10 mL). Saturated Na_2CO_3 solution (10 mL) was added. The organic fraction was collected, and the aqueous fraction was extracted with EtOAc (2 x 10 mL). The combined organic fraction was washed with brine, dried over Na_2SO_4, filtered and concentrated under reduced pressure. The resulting residue was purified by flash column chromatography on silica gel using hexane/ethyl acetate eluents.

1. Faradaic Efficiency and Productivity

The faradaic efficiency (FE) of product formation and productivity were calculated using the following equation[1,2]:

$$Faradaic\ efficiency(\%) = \frac{n \times F \times mole\ of\ product\ formed}{total\ charge\ passed} \times 100\% \qquad \text{Equation (1)}$$

$$productivity(\mu mol/(h \times cm^2)) = \frac{mole\ of\ prodcut\ formed}{total\ time \times electrode\ area} \qquad \text{Equation (2)}$$

where n is the number of electron transfer for each product formation and F is the Faraday constant (96485 C mol^{-1}). Total time is the time achieved 100% conversion of substrate. Electrode area is the anode geometric area.

2. Flow cell set-up

2.1 Materials for zero-gap flow cell set-up using in General Procedure A:

2 x PEEK endplates (40 mm*40 mm*10 mm) with snake shape of flow channel, as Figure S3A&B shown

4 x PTFE Gasket (40 mm*40 mm*0.3 mm) with a hollow part (10 mm*10 mm) in the middle, as Figure S3C shown

2 x Celgard 3501 (12 mm* 12 mm), pretreatment with MeCN

1 x Proton-exchange membrane Nafion® 117 (12 mm* 12 mm, pre-treatment with MeCN)

Anode: 2 x Carbon papers (CP, SGL 39AA) (10 mm*10 mm*0.3 mm), heat treatment in 400 ˚C in air, 24 h

Cathode: 1 x 20% Pt/graphite carbon paper (10 mm*10 mm*0.3 mm), prepared by dripping 20% Pt/graphite (purchase from Sigma-Aldrich) Nafion® solution (5 wt% Nafion amount) on heat treated CP

Cuurent Collector: 2 x Platinum foil (3 mm*25 mm*0.1 mm)

8 x Screws and Screw caps

4 x SMC stainless steel air-tight connectors (tube: 1/8 to M5)

3 x PTFE tube (1/8)

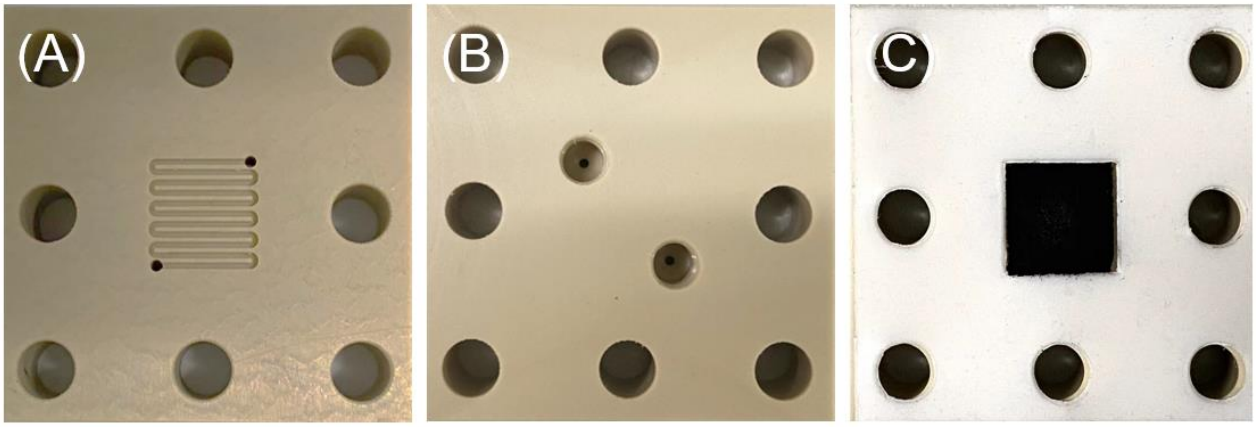


Figure S3. (A) PEEK endplates with flow channels, contact with PTFE gasket and electrode; **(B)** PEEK endplates with M5 holes connect with SMC air-tight connectors; (C) PTFE gasket with holes and CP in the hole.

2.2 Procedure for flow cell assembly:

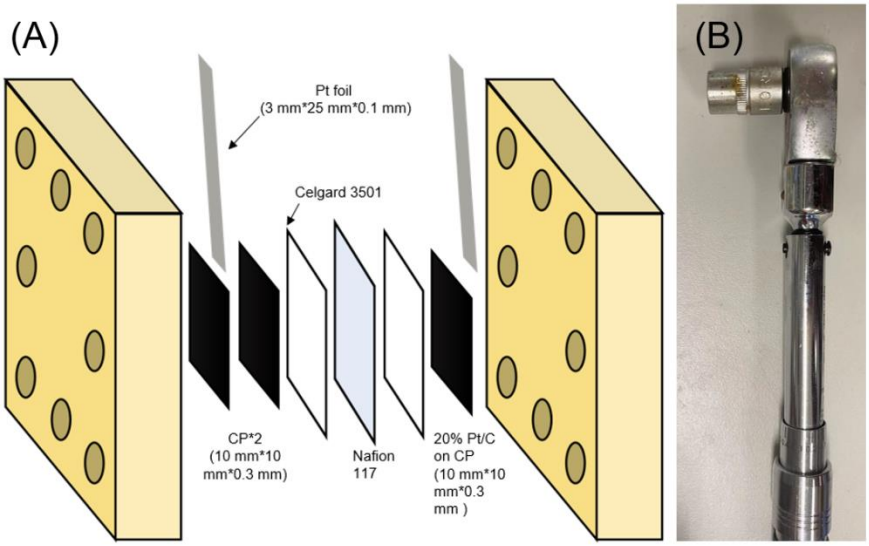
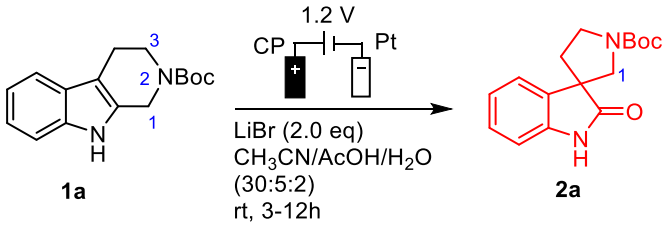


Figure S4. (A) Schematic of flow cell using in General Procedure A; **(B**) Torque wrench using to tight screw

A PEEK endplate with flow channel facing top was equipped with eight screws. The assemble schematic was presented in Figure S4A. A piece of PTFE gasket was placed on top of the endplate. Two pieces of carbon papers in the middle were fitted in the hollow part of the gasket. Current collector was placed beyond the edge of the set-up. A piece of celgard 3501 was placed on top covering the whole carbon paper electrode. Then additional two pieces of PTFE Gasket with a proton-exchange membrane Nafion 117 in the middle were placed on top. Another piece of celgard 3501 was placed to cover the hollow part. The 20% Pt/graphite CP and current collector were then placed, covered by the last PTFE Gasket and another PEEK endplates and tightened the screws with screw caps using torque wrench, as Figure S4B presented, with uniform force to prevent the deformation and leakage of using PTFE gasket. Finally, four SMC stainless steel air-tight connectors were screwed on two sides of the flow cell with Teflon tape to prevent corrosion and connected with four PTFE tube at the other end.

3. Other conditions screened

Table S1. Selected conditions for Br mediated electro-oxidative rearrangement[a]



Entry	Different from base case[a]	Current density (mA*cm^{-2})	Conv. (%)	Yield. (%)	Faradaic Efficiency (%)	Productivity (μmol*h^{-1}*cm^{-2})
S1	NaBr 40 mL*min^{-1}	5.2	100	82	82	78.7
S2	KBr 40 mL*min^{-1}	3.5	100	75	75	48.5
S3	TBAB 40 mL*min^{-1}	1.4	100	58	58	14.7
S4	LiBr 5 eq	7.0	100	87	87	113.6
S5	LiBr 2.5 eq	4.4	100	95	95	82.2
S6	LiBr 1.5 eq	2.0	100	73	73	37.2
S7	40 mL*min^{-1}	6.4	100	96	96	114.7
S8	5 mL*min^{-1}	2.0	100	89	89	33.2

[a] The reaction was carried out at room temperature with **1a** (0.2 mmol), LiBr (2.0 eq), solvents (20 mL) followed general procedure A. NMR yield was obtained

4. Electro-chemical test (undivided cell)

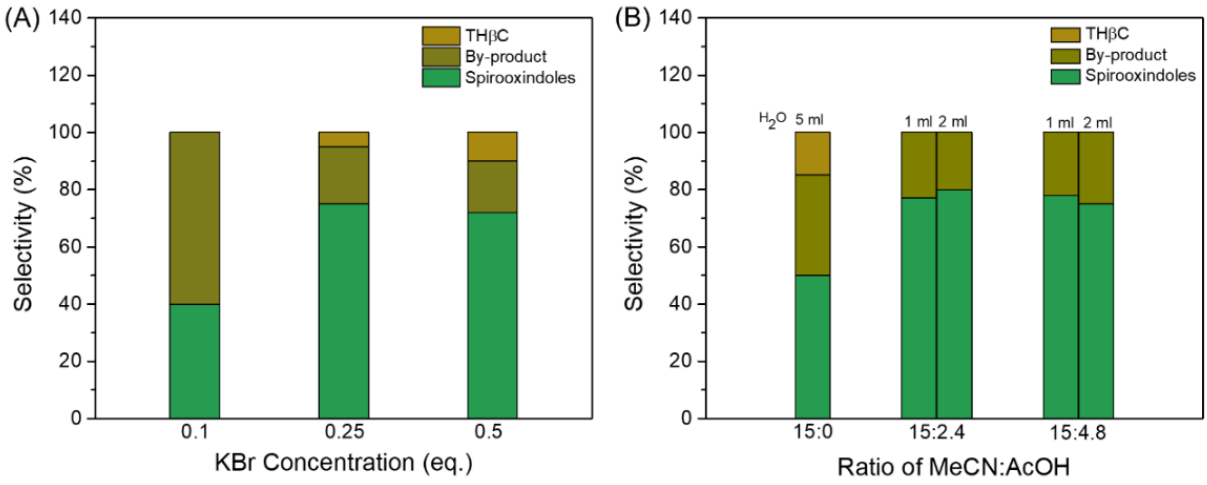


Figure S5. (A)Influence of KBr concentration in General Procedure B; (B) Influence of AcOH addition in General Procedure B

5. Electro-chemical test (flow cell)

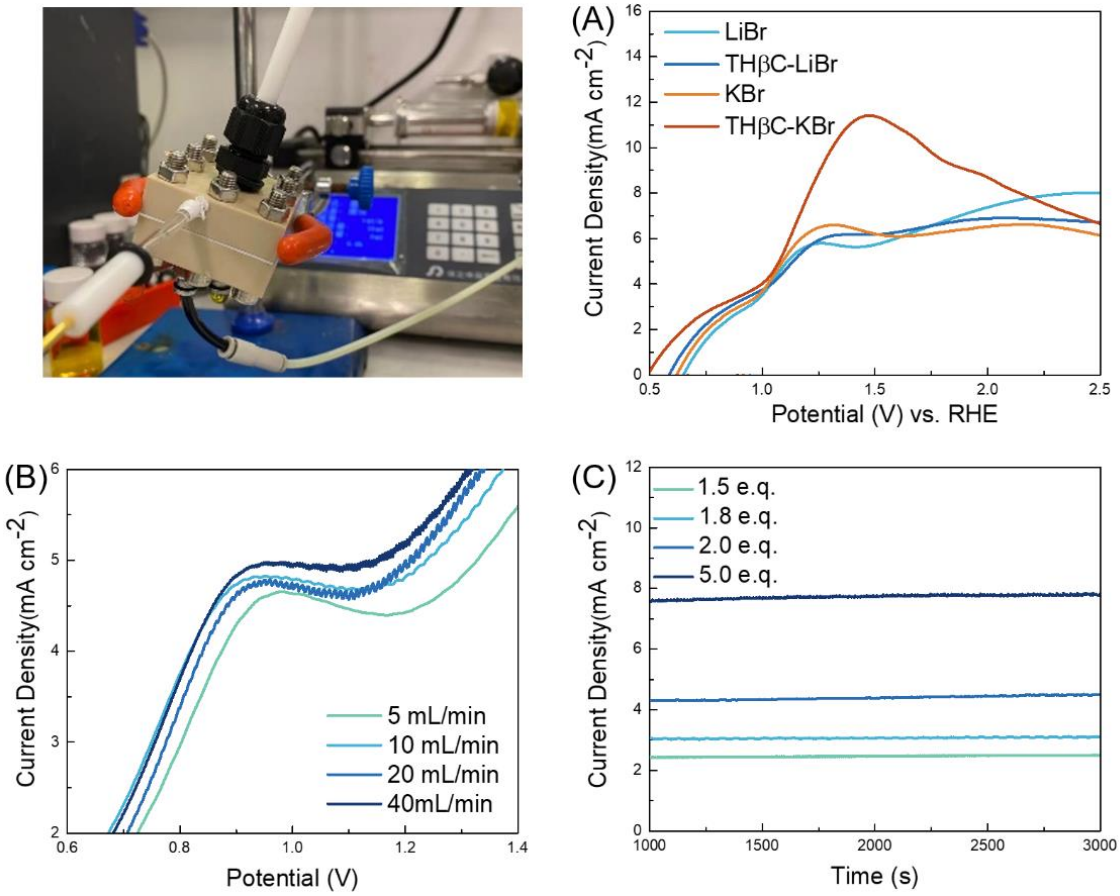


Figure S6. (A) LSV of half-flow cell (as photo present) with condition: Working electrode: CP; Counter electrode: Pt mesh; Reference electrode: Ag/AgCl, the E_{RHE} =E+0.0591*pH+0.197[3]; Scan rate 50 mV/s with 0.8 M $LiClO_4$ as supporting electrolyte, 0.2 mmol THβC, 2 e.q. LiBr or KBr; (B) LSV of different flow rate under General Procedure A. (C) Current density in different equivalent of LiBr under General procedure A.

6. Comparison with literature data

We also compared our results with other electrochemical oxidation reactions, in particular, of indole or related N-heterocycle compounds[4–9] in Tables S2-3, which including: (a) electrochemical 1,2-diarylation[4] of alkenes with a $CoCl_2$ catalyst; (b) electrochemical diazidation[5] reaction of alkenes with the $MnBr_2$ catalyst; (c) electro-oxidative [3+2] annulation[6] of phenol and indole; (d) electrochemical radical cascade cyclization[7] of N-methacryloyl-2-phenylbenzoimidazole and alkyl

boronic acid; (e) electrochemical flow microreactor[8] for efficient synthesis of isoquinoline-6(5H)-ones; and (f) flow Rhodaelectro-catalyzed alkyne annulations[9]. The comparison was based on terms of the yield and productivity, i.e., the number of mole of the desired product per unit of reaction time and per area of the cell [in μmol/(h*cm^2)]. The higher productivity, the shorter time and the smaller cell required, the more cost-effective. As shown in Figure S7, the yield (**2p**, 94%) of our reaction system was among the best, while the productivity (**2p**, 144 μmol/(h*cm^2) (also see Table S3 in Supporting Information) was much higher than all other electrochemical reactions. We attributed our outstanding performance to the following two factors: 1) the zero-gap flow cell minimizes the overpotential and mitigates the trade-off between a high current and a high selectivity, which is typically encountered in a cell with less forced convection; and 2) the use of bifunctional mediator LiBr with a suitable oxidation potential leaves a sufficiently wide potential window for the high selectivity.

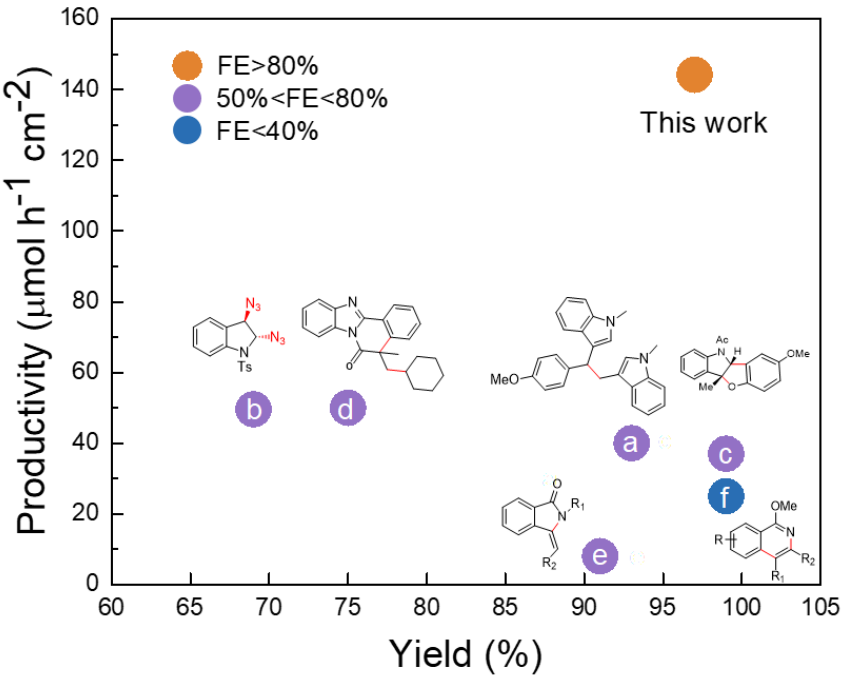


Figure S7. Comparison of productivity, Faradaic efficiencies and yield cycle life (Noted: all selected electro-oxidation of indole or N-heterocyclic compounds for a fair comparison, Table S3 summarizes the detail calculation; red bond represents new generated bond during the electrochemical reaction)[4–9]

Table S2. Summary of the detailed reactions in Figure S7 in the article.

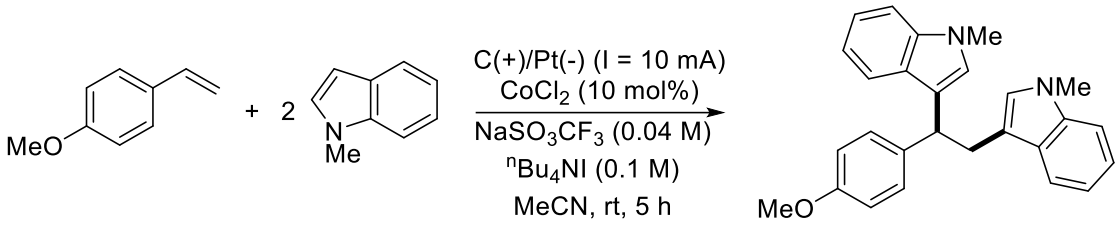
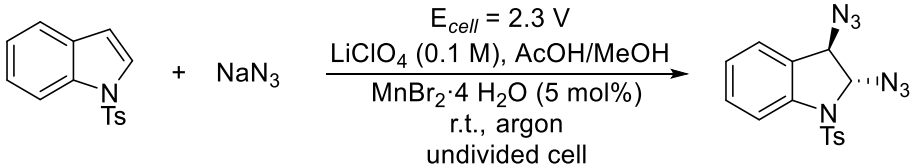
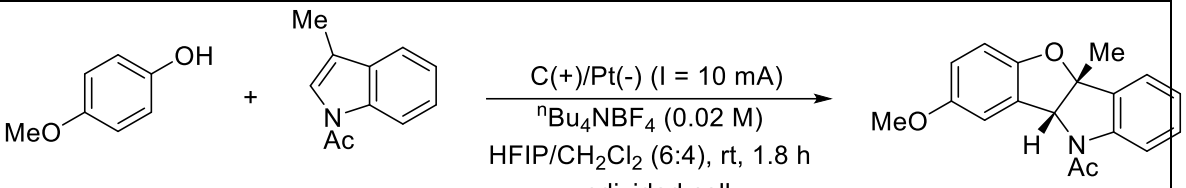
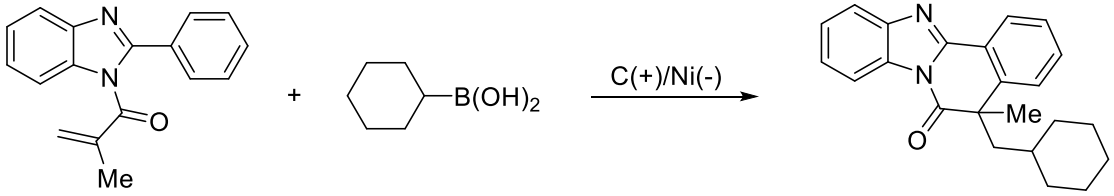
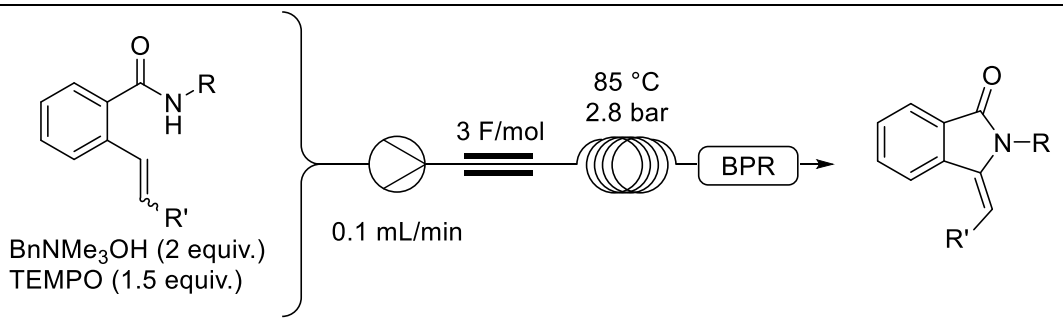
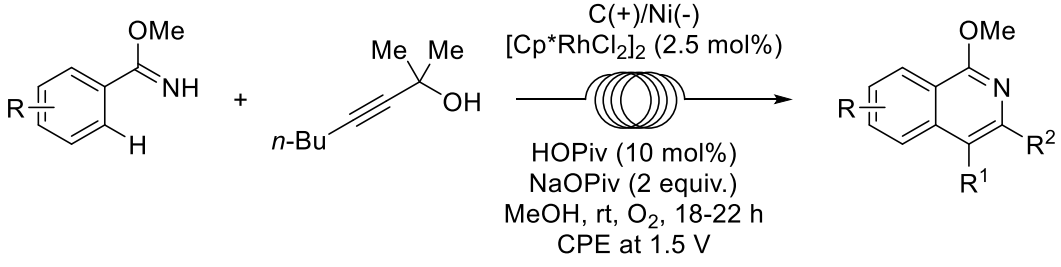
a[4]	
b[5]	
c[6]	
d[7]	
e[8]	
f[9]	

Table S3. Summary of productivity, Faradaic efficiency and yield in Figure S7

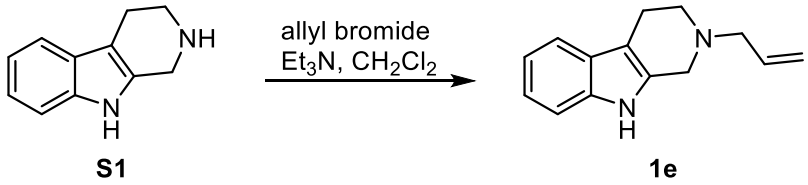
Entry	Reaction cell	Electrode	Yield. (%)	Faradaic Efficiency (%)	Productivity ($\mu mol*h^{-1}*cm^{-2}$)
Our work	Divided zero-gap flow cell	CP/Pt C	94	93	144
a[4]	Undivided beaker	CC[a]/Pt	93	64	40
b[5]	Undivided beaker	RVC[b]/Pt	69	62	49
c[6]	Undivided beaker	GR[c]/Pt	99	60	37
d[7]	Undivided beaker	CF[d]/Ni foam	75	54	50
e[8]	Single-pass flow	Pt/Pt	91	67	8
f[9]	Undivided flow cell	GF[d]/Ni foam	99	27	25

[a]Carbon Cloth; [b]Reticulated Vitreous Carbon; [c]Graphite Rod; [d]Carbon Felt

7. Preparation of indole substrates

Substrates **1a**[10], **1b**[11], **1c**[12], **1d**[13], **1f-1h**[14], **1i-1j**[15], **1l**[16], **1m**[17], **1o**[18], **1p**[19], **1q**[17], **1r**[20], and **1t**[17] were prepared according to the published procedures.

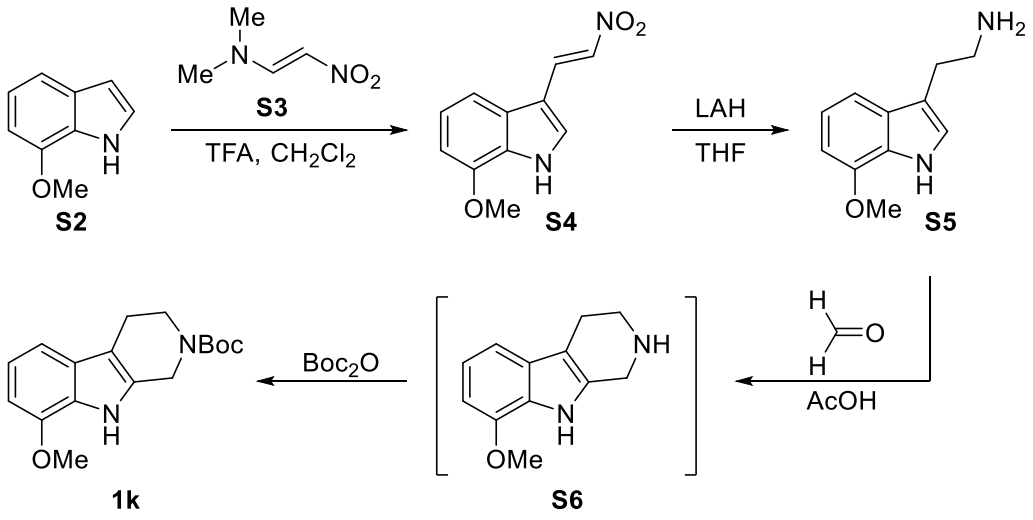
1. Preparation of 1e



S1 **1e**

To a solution of **S1** (516 mg, 3.0 mmol) and Et_3N (1.25 mL, 9.0 mmol) in CH_2Cl_2 (3 mL) was added allyl bromide (0.31 mL, 3.6 mmol) at 0 °C. The reaction mixture was stirred at rt for 2 h. Saturated $NaHCO_3$ (10mL) solution was added and extracted with CH_2Cl_2 (3 x 10 mL). The combined organic fraction was washed with brine, dried over Na_2SO_4, filtered, and concentrated under reduced pressure. The resulting residue was purified by flash column chromatography (hexane : EtOAc = 4 : 1) to give compound **1e** as a yellow solid (334 mg, 52%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.92 (s, 1H), 7.48 (d, J = 7.0 Hz, 1H), 7.27 (d, J = 6.8 Hz, 1H), 7.21 – 7.04 (m, 2H), 6.07 – 5.88 (m, 1H), 5.39 – 5.15 (m, 2H), 3.62 (s, 2H), 3.25 (d, J = 5.6 Hz, 2H), 2.97 – 2.74 (m, 4H). ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$) δ 136.2, 135.4, 131.8, 127.3, 121.4, 119.4, 118.3, 118.1, 110.8, 108.4, 60.9, 50.8, 50.2, 21.3. HRMS (ESI) *m/z*: Calcd for $C_{14}H_{17}N_2^+$ $[M+H]^+$ 213.1386; Found 213.1383.

2. Preparation of 1k

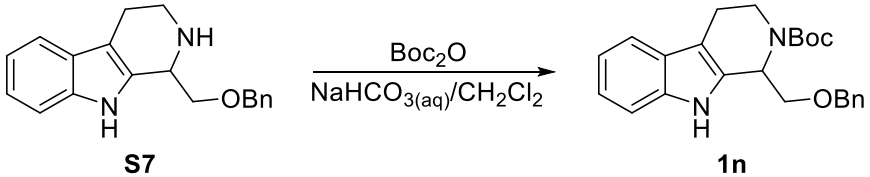


To a solution of **S3** (418 mg, 3.6 mmol) and TFA (0.53 mL, 6.9 mmol) in CH_2Cl_2 (10 mL) was added **S2** (0.39 mL, 3.0 mmol) at 0 °C and stirred at this temperature for 2 h. Diluted with water (10 mL) and extracted with CH_2Cl_2 (3 x 10 mL). The combined organic fraction was washed with saturated $NaHCO_3$ solution, brine, dried over Na_2SO_4, filtered, and concentrated under reduced pressure. The resulting residue was directly used in the next step without further purification.

To a solution of crude **S4** (269 mg) in THF (5 mL) at 0 °C was added LAH (280 mg, 7.4 mmol) portion wise and stirred for 18 h. Then, diluted with Et_2O (5 mL). Saturated sodium potassium tartrate solution (10mL) was added and stirred for another 2 h. The resulting mixture was extracted with EtOAc (3 x 10 mL). The combined organic fraction was acidified to pH 1-2 by addition of 1 M HCl. The collected aqueous fraction was then basified to pH 9-10 by addition of 3 N of NaOH. Extracted with EtOAc (3 x 10 mL). The combined organic fraction was washed with brine, dried over Na_2SO_4, filtered, and concentrated under reduced pressure. The resulting residue was directly used in the next step without further purification.

To a solution of crude **S5** (161mg) and paraformaldehyde (25 mg, 0.85 mmol) in AcOH (8.5 mL) was heated at at 80 °C for 1 h. After cooling down to rt, the reaction mixture was basified to pH 9-10 by addition of saturated Na_2CO_3 solution and diluted with CH_2Cl_2. Boc_2O (0.23 mL, 1.0 mmol) was added and stirred for another 3 h. The reaction mixture was extracted with CH_2Cl_2 (2 x 10mL). The combined organic fraction was washed with brine, dried over Na_2SO_4, filtered, and concentrated under reduced pressure. The resulting residue was purified by flash column chromatography (hexane : EtOAc = 12 : 1) to give compound **1k** as a white solid (105 mg, 12%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.28 – 7.97 (m, 1H), 7.10 (d, J = 7.8 Hz, 1H), 7.03 (t, J = 7.8 Hz, 1H), 6.64 (d, J = 7.7 Hz, 1H), 4.64 (br, 2H), 3.95 (s, 3H), 3.76 (br, 2H), 2.79 (br, 2H), 1.51 (s, 9H). ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$) δ 155.3, 146.0, 130.4, 128.4, 126.4, 120.1, 111.0, 109.7, 109.2, 102.2, 80.1, 55.5, 42.7, 41.8, 28.6, 21.6. HRMS (ESI) *m/z*: Calcd for $C_{17}H_{22}N_2O_3Na^+$ $[M+Na]^+$ 325.1523; Found 325.1525.

3. Preparation of 1n

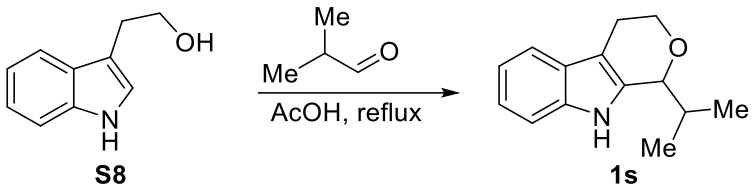


S7 **1n**

To a solution of **S7** (877 mg, 3.0 mmol) in a mixture of saturated $NaHCO_3$ solution/CH_2Cl_2 (1:4) (25 mL) was added Boc_2O (0.83 mL, 3.6 mmol) and stirred for 3 h. The reaction mixture was extracted with CH_2Cl_2 (2 x 30 mL). The combined organic fraction was washed with brine, dried over Na_2SO_4, filtered, and concentrated under reduced pressure. The resulting residue was purified by flash column

chromatography (hexane : EtOAc = 12 : 1) to give compound **1n** as pale-yellow oil (913mg, 78%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.79 – 8.48 (m, 1H), 7.60 (d, J = 7.6 Hz, 1H), 7.54 – 7.30 (m, 6H), 7.30 – 7.23 (m, 1H), 7.20 (t, J = 7.3 Hz, 1H), 5.70 – 5.29 (m, 1H), 4.79 – 4.40 (m, 3H), 3.92 (s, 1H), 3.75 (s, 1H), 3.33 – 3.05 (m, 1H), 2.98 – 2.87 (m, 1H), 2.87 – 2.76 (m, 1H), 1.75 – 1.53 (m, 9H). ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$) δ 154.7, 137.8, 136.1, 132.8, 128.5, 127.8, 126.9, 126.5, 121.7, 119.3, 118.1, 111.0, 108.8, 80.2, 73.5, 71.3, 50.7, 50.0, 40.4, 39.2, 28.5, 21.5. HRMS (ESI) *m/z*: Calcd for $C_{24}H_{28}N_2O_3Na^+$ $[M+Na]^+$ 415.1992; Found 415.1994.

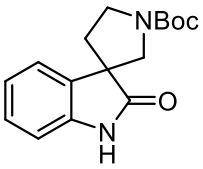
4. Preparation of 1s



S8 **1s**

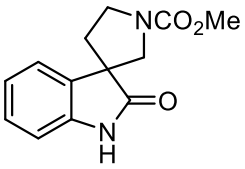
To a solution of **S8** (322 mg, 2.0 mmol) in AcOH (7 mL) was added isobutyraldehyde (0.18 mL, 2.0 mmol) and heated at reflux for 5 h. After cooling down to rt, the reaction mixture was basified to pH 9-10 by addition of saturated Na_2CO_3 solution and diluted with CH_2Cl_2. The reaction mixture was extracted with CH_2Cl_2 (2 x 20 mL). The combined organic fraction was washed with brine, dried over Na_2SO_4, filtered, and concentrated under reduced pressure. The resulting residue was purified by flash column chromatography (hexane : EtOAc = 15 : 1) to give compound **1s** as white solid (131 mg, 30%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.77 (br, 1H), 7.55 (d, J = 7.6 Hz, 1H), 7.36 (d, J = 7.8 Hz, 1H), 7.21 (t, J = 7.1 Hz, 1H), 7.16 (t, J = 7.2 Hz, 1H), 4.78 – 4.70 (m, 1H), 4.33 (ddd, J = 11.0, 5.4, 1.5 Hz, 1H), 3.80 (td, J = 10.8, 3.7 Hz, 1H), 2.98 (dddd, J = 15.9, 10.6, 5.4, 2.1 Hz, 1H), 2.76 – 2.67 (m, 1H), 2.16 (dtd, J = 13.7, 6.9, 3.3 Hz, 1H), 1.19 (d, J = 6.9 Hz, 3H), 0.91 (d, J = 6.9 Hz, 3H). ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$) δ 136.0, 134.4, 127.3, 121.8, 119.7, 118.2, 111.0, 109.4, 104.9, 77.6, 65.0, 32.6, 22.5, 19.1, 16.8, 16.5. HRMS (ESI) *m/z*: Calcd for $C_{14}H_{16}NO^-$ $[M-H]^-$ 214.1237; Found 214.1232

8. Electrochemical oxidative rearrangement of indoles to 2-oxindoles



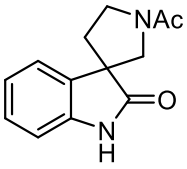
2a (95%)

2a was obtained by General Procedure A [54.7 mg, 95% yield, 89.55 μmol/(h*cm^{-2})] and by General Procedure B (23.1 mg, 80% yield). Eluent solvents: CH_2Cl_2/MeOH = 50:1. ^{1}H NMR (400 MHz, $CDCl_3$) (presence of rotamers) δ 9.27 – 9.03 (m, 1H), 7.23 (d, J = 7.7 Hz, 1H), 7.17 (br, 1H), 7.04 (t, J = 7.5 Hz, 1H), 6.96 (d, J = 7.7 Hz, 1H), 3.92 – 3.67 (m, 3H), 3.67 – 3.50 (m, 1H), 2.41 (dt, J = 12.6, 8.2 Hz, 1H), 2.07 (br, 2H), 1.57 – 1.40 (m, 9H). ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$) δ 180.2, 154.6, 140.3, 133.1, 128.5, 123.0, 122.8, 110.3, 80.0, 54.5, 53.5, 45.4, 35.7, 28.6. HRMS (ESI) *m/z*: Calcd for $C_{16}H_{20}N_2O_3Na$ $[M+Na]^+$ 311.1366; Found 311.1375.



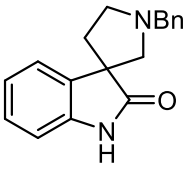
2b (84%)

2b was obtained by General Procedure A with flow rate of 40 mL/min [41.2 mg, 84% yield, 114.59 μmol/(h*cm^{-2})]. Eluent solvents: CH_2Cl_2/MeOH = 50:1. ^{1}H NMR (400 MHz, $CDCl_3$) (presence of rotamers) δ 8.49 – 8.36 (m, 1H), 7.24 (t, J = 7.7 Hz, 1H), 7.17 (d, J = 7.1 Hz, 1H), 7.05 (t, J = 7.5 Hz, 1H), 6.93 (d, J = 7.5 Hz, 1H), 3.97 – 3.57 (m, 7H), 2.48 – 2.38 (m, 1H), 2.18 – 2.04 (m, 1H). ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$) δ 179.8, 155.6, 140.2, 132.8, 132.4, 128.7, 123.2, 122.9, 110.2, 54.5, 54.1, 53.4, 52.8, 52.4, 45.8, 45.3, 36.5, 35.6. HRMS (ESI) *m/z*: Calcd for $C_{13}H_{14}N_2O_3Na^+$ $[M+Na]^+$ 269.0897; Found 269.0905.



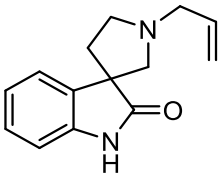
2c (79%)

2c was obtained by General Procedure A [25.5 mg, 79% yield, 0.1 mmol scale, 45.69 μmol/(h*cm^{-2})]. Eluent solvents: CH_2Cl_2/MeOH = 50:1. ^{1}H NMR (400 MHz, $CDCl_3$) (presence of rotamers) δ 8.49 – 8.24 (m, 1H), 7.31 – 7.20 (m, 2H), 7.20 – 7.11 (m, 1H), 7.11 – 7.00 (m, 1H), 7.00 – 6.88 (m, 1H), 4.06 – 3.93 (m, 1H), 3.93 – 3.80 (m, 2H), 3.80 – 3.59 (m, 1H), 2.53 – 2.38 (m, 1H), 2.29 – 2.02 (m, 4H). ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$) (presence of rotamers) δ 180.3, 179.2, 169.7, 169.5, 140.5, 140.2, 132.7, 131.8, 128.9, 128.8, 123.3, 123.1, 122.8, 110.4, 110.3, 55.6, 53.7, 53.7, 51.9, 46.8, 45.3, 36.6, 35.3, 22.7, 22.6. HRMS (ESI) *m/z*: Calcd for $C_{13}H_{14}N_2O_3Na^+$ $[M+Na]^+$ 269.0897; Found 269.0905.



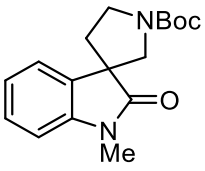
2d (71%)

2d was obtained by General Procedure A with 0.4 mmol LiBr [40.4 mg, 71% yield, 63.59 μmol/(h*cm^{-2})] and by General Procedure B (11.5mg, 41% yield). Eluent solvents: CH_2Cl_2/MeOH = 100:1-30:1. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.70 (br, 1H), 7.49 (d, J = 7.4 Hz, 1H), 7.39 (d, J = 7.2 Hz, 2H), 7.31 (t, J = 7.4 Hz, 2H), 7.26 – 7.21 (m, 1H), 7.19 (td, J = 7.7, 1.2 Hz, 1H), 7.06 (dd, J = 7.5, 0.8 Hz, 1H), 6.84 (d, J = 7.7 Hz, 1H), 3.75 (ABq, 2H, J = 13 Hz), 3.15 (dt, J = 8.4, 3.9 Hz, 1H), 2.92 (d, J = 9.1 Hz, 1H), 2.81 (d, J = 9.1 Hz, 1H), 2.73 (q, J = 8.3 Hz, 1H), 2.42 (ddd, J = 12.4, 8.3, 3.9 Hz, 1H), 2.09 (dt, J = 13.0, 7.8 Hz, 1H). ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$) δ 182.0, 139.8, 139.3, 137.0, 128.7, 128.4, 127.8, 127.1, 123.7, 123.1, 109.3, 77.5, 77.2, 76.8, 64.3, 59.8, 54.4, 53.3, 37.3. HRMS (ESI) *m/z*: Calcd for $C_{18}H_{19}N_2O^+$ $[M+H]^+$ 279.1492; Found 279.1494.



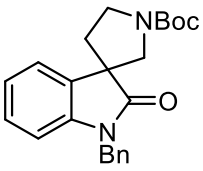
2e (73%)

2e was obtained by General Procedure A with 0.4 mmol LiBr [33.4 mg, 73% yield, 43.58 μmol/(h*cm^{-2})]. Eluent solvents: CH_2Cl_2/MeOH = 100:1-30:1. ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.16 (s, 1H), 7.41 (d, J = 7.3 Hz, 1H), 7.19 (td, J = 7.7, 1.2 Hz, 1H), 7.03 (td, J = 7.6, 0.9 Hz, 1H), 6.91 (d, J = 7.7 Hz, 1H), 5.95 (ddt, J = 16.6, 10.2, 6.3 Hz, 1H), 5.29 – 5.17 (m, 1H), 5.15 – 5.05 (m, 1H), 3.32 – 3.17 (m, 2H), 3.08 (ddd, J = 8.9, 7.6, 4.8 Hz, 1H), 2.96 – 2.86 (m, 2H), 2.81 (dd, J = 16.5, 7.7 Hz, 1H), 2.40 (ddd, J = 12.7, 7.8, 4.8 Hz, 1H), 2.09 (dt, J = 12.8, 7.4 Hz, 1H). ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$) δ 183.3, 140.3, 136.5, 135.8, 127.8, 123.4, 122.9, 117.1, 109.8, 64.1, 58.6, 54.5, 53.3, 37.4. HRMS (ESI) *m/z*: Calcd for $C_{14}H_{17}N_2O^+$ $[M+H]^+$ 229.1335; Found 229.1344.



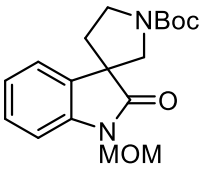
2f (89%)

2f was obtained by General Procedure A [53.5 mg, 89% yield, 39.85 μmol/(h*cm^{-2})] and by General Procedure B (18.9 mg, 63% yield). Eluent solvents: hexane/EtOAc = 5:1. ^{1}H NMR (400 MHz, $CDCl_3$) (presence of rotamers) δ 7.30 (t, J = 7.6 Hz, 1H), 7.18 (br, 1H), 7.06 (t, J = 7.4 Hz, 1H), 6.86 (d, J = 7.8 Hz, 1H), 3.88 – 3.63 (m, 3H), 3.62 – 3.47 (m, 1H), 3.22 (s, 3H), 2.46 – 2.33 (m, 1H), 2.00 (br, 1H), 1.55 – 1.38 (m, 9H). ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$) (presence of rotamers) δ 177.4, 154.5, 142.9, 132.9, 128.5, 123.1, 122.5, 108.3, 79.9, 54.5, 53.0, 45.3, 35.6, 28.6, 26.5. HRMS (ESI) *m/z*: Calcd for $C_{17}H_{22}N_2O_3Na^+$ $[M+Na]^+$ 325.1523; Found 325.1528.



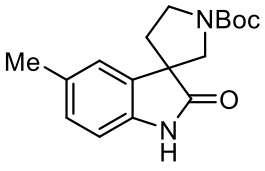
2g (**80%**)

2g was obtained by General Procedure A [60.2 mg, 80% yield, 41.79 μmol/(h*cm^{-2})] and by General Procedure B (22.7 mg, 60% yield). Eluent solvents: hexane/EtOAc = 5:1. ^{1}H NMR (400 MHz, $CDCl_3$) (presence of rotamers) δ 7.37 – 7.27 (m, 7H), 7.24 – 7.13 (m, 2H), 7.09 – 6.99 (m, 1H), 6.82 – 6.67 (m, 1H), 5.03 – 4.84 (m, 2H), 3.94 – 3.70 (m, 2H), 3.70 – 3.55 (m, 1H), 2.54 – 2.39 (m, 1H), 2.18 – 2.02 (m, 1H), 1.56 – 1.43 (m, 9H). ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$) (presence of rotamers) δ 177.6, 154.6, 142.0, 135.8, 132.9, 129.0, 128.4, 127.9, 127.4, 123.2, 122.7, 109.4, 80.0, 54.6, 54.2, 53.1, 52.1, 45.6, 45.4, 44.0, 36.6, 35.9, 28.7, 28.6. HRMS (ESI) *m/z*: Calcd for $C_{23}H_{26}N_2O_3Na^+$ $[M+Na]^+$ 401.1836; Found 401.1843.



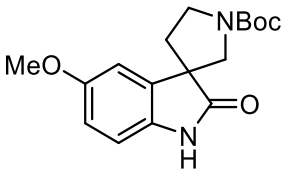
2h (**67%**)

2h was obtained by General Procedure A [44.6 mg, 67% yield, 25.01 μmol/(h*cm^{-2})]. Eluent solvents: hexane/EtOAc = 5:1. ^{1}H NMR (400 MHz, $CDCl_3$) (presence of rotamers) δ 7.30 (t, J = 7.6 Hz, 1H), 7.21 (t, J = 8.5 Hz, 1H), 7.11 (t, J = 7.5 Hz, 1H), 7.06 (d, J = 7.8 Hz, 1H), 5.14 (s, 2H), 3.90 – 3.67 (m, 3H), 3.65 – 3.53 (m, 1H), 3.33 (s, 3H), 2.48 – 2.34 (m, 1H), 2.16 – 2.00 (m, 1H), 1.55 – 1.41 (m, 9H). ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$) (presence of rotamers) δ 178.7, 178.2, 154.5, 141.3, 141.2, 132.3, 131.6, 128.7, 123.7, 123.7, 122.7, 109.8, 80.0, 71.6, 56.4, 54.8, 54.3, 53.3, 52.3, 45.6, 45.3, 36.8, 36.0, 28.6, 28.6. HRMS (ESI) *m/z*: Calcd for $C_{18}H_{24}N_2O_4Na^+$ $[M+Na]^+$ 355.1628; Found 355.1632.



2i (**85%**)

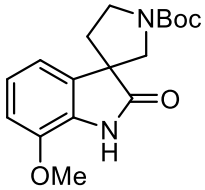
2i was obtained by General Procedure A [51.2 mg, 85% yield, 50.75 μmol/(h*cm^{-2})] and by General Procedure B (17.0 mg, 56% yield). Eluent solvents: hexane/EtOAc = 4:1-2:1. ^{1}H NMR (400 MHz, $CDCl_3$) (presence of rotamers) δ 9.35 – 9.15 (m, 1H), 7.09 – 6.93 (m, 2H), 6.84 (d, J = 7.7 Hz, 1H), 3.91 – 3.67 (m, 3H), 3.66 – 3.51 (m, 1H), 2.46 – 2.35 (m, 1H), 2.31 (s, 3H), 2.06 (br, 1H), 1.58 – 1.39 (m, 9H). ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$) (presence of rotamers) δ 180.3, 154.6, 137.8, 133.4, 132.6, 128.8, 123.6, 110.0, 80.0, 54.5, 53.6, 45.4, 35.6, 28.6, 21.3. HRMS (ESI) *m/z*: Calcd for $C_{17}H_{22}N_2O_3Na^+$ $[M+Na]^+$ 325.1523; Found 325.1527.



2j (**71%**)

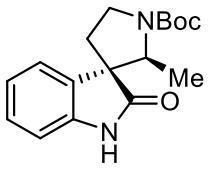
2j was obtained by General Procedure A [45.0 mg, 71% yield, 34.44 μmol/(h*cm^{-2})]. Eluent solvents: hexane/EtOAc = 4:1-2:1. ^{1}H NMR (400 MHz, $CDCl_3$) (presence of rotamers) δ 8.56 – 8.35 (m, 1H), 6.87 – 6.81 (m, 1H), 6.81 – 6.72 (m, 2H), 3.90 – 3.65 (m, 6H), 3.65 – 3.52 (m, 1H), 2.47 – 2.35 (m, 1H), 2.12 – 2.00 (m, 1H), 1.54 – 1.41 (m, 9H). ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$) (presence of rotamers) δ 180.0, 179.8, 156.3, 154.6, 134.6, 133.4, 112.8, 112.6, 110.5,

110.2, 80.0, 56.0, 54.5, 54.0, 53.9, 52.9, 45.5, 45.3, 36.5, 35.7, 28.7, 28.6. HRMS (ESI) *m/z*: Calcd for $C_{17}H_{22}N_2O_4Na^+$ $[M+Na]^+$ 341.1472; Found 341.1474.



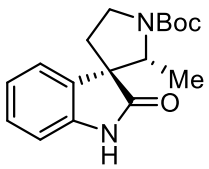
2k (**42%**)

2k was obtained by General Procedure A [13.3 mg, 42% yield, 0.1 mmol scale, 19.59 μmol/(h*cm^{-2})]. Eluent solvents: hexane/EtOAc = 4:1-2:1. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.66 – 7.55 (m, 1H), 7.06 – 6.98 (m, 1H), 6.87 – 6.76 (m, 2H), 3.88 (s, 3H), 3.86 – 3.66 (m, 3H), 3.66 – 3.51 (m, 1H), 2.47 – 2.35 (m, 1H), 2.14 – 2.00 (m, 1H), 1.54 – 1.41 (m, 9H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 178.9, 178.5, 154.6, 144.0, 134.0, 133.5, 128.7, 123.8, 123.7, 115.2, 110.9, 80.0, 55.9, 54.5, 54.1, 54.0, 53.1, 45.5, 45.3, 36.5, 35.7, 28.7, 28.6. HRMS (ESI) *m/z*: Calcd for $C_{17}H_{22}N_2O_4Na^+$ $[M+Na]^+$ 341.1472; Found 341.1470.



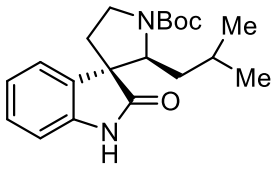
2l (**71%, *dr* 3:1**)

2l was obtained by General Procedure A [32.5 mg, 53% yield, 35.60 μmol/(h*cm^{-2})]. Eluent solvents: hexane/EtOAc = 6:1-2:1. ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.17 – 8.92 (m, 1H), 7.22 (t, J = 7.6 Hz, 1H), 7.11 (d, J = 7.4 Hz, 1H), 7.03 (t, J = 7.5 Hz, 1H), 6.93 (d, J = 7.7 Hz, 1H), 4.14 – 3.90 (m, 1H), 3.91 – 3.68 (m, 2H), 2.42 (br, 1H), 2.17 – 1.99 (m, 1H), 1.47 (s, 9H), 1.30 (d, J = 6.5 Hz, 3H). ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$) δ 179.1, 154.5, 140.4, 133.3, 128.4, 123.0, 122.8, 110.0, 79.9, 61.0, 56.8, 44.6, 33.4, 28.6, 16.7. HRMS (ESI) *m/z*: Calcd for $C_{17}H_{22}N_2O_3Na^+$ $[M+Na]^+$ 325.1523; Found 325.1523.



2l'

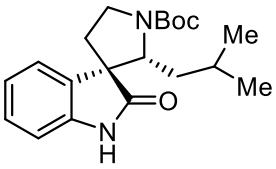
2l' was obtained by General Procedure A [10.6 mg, 18% yield, 12.09 μmol/(h*cm^{-2})]. Eluent solvents: hexane/EtOAc = 6:1-2:1. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.13 (br, 1H), 7.26 – 7.20 (m, 2H), 7.05 (td, J = 7.6, 0.9 Hz, 1H), 6.92 (d, J = 7.6 Hz, 1H), 4.19 – 4.08 (m, 1H), 3.95 (br, 1H), 3.74 – 3.62 (m, 1H), 2.32 (dt, J = 12.6, 8.1 Hz, 1H), 2.07 (br, 1H), 1.49 (s, 9H). ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$) δ 180.0, 155.1, 140.6, 130.0, 128.5, 125.3, 122.5, 110.1, 59.1, 46.0, 34.0, 28.7, 17.8. HRMS (ESI) *m/z*: Calcd for $C_{17}H_{22}N_2O_3Na^+$ $[M+Na]^+$ 325.1523; Found 325.1525.



2m (**83%, *dr* 1.6:1**)

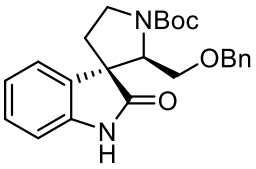
2m was obtained by General Procedure A with 20 mL solvent [29.3 mg, 56% yield, 17.76 μmol/(h*cm^{-2})] and by General Procedure B (<40%). Eluent solvents: hexane/EtOAc = 6:1-2:1. ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.33 – 9.12 (m, 1H), 7.22 (dd, J = 10.7, 5.4 Hz, 1H), 7.13 – 6.97 (m, 2H), 6.94 (d, J = 7.6 Hz, 1H), 4.19 – 3.61 (m, 3H), 2.62 – 2.34 (m, 1H), 2.08 – 1.97 (m, 1H), 1.86

(br, 1H), 1.59 – 1.16 (m, 11H), 0.81 (br, 6H). $^{13}C\{^{1}H\}$ NMR (100 MHz, $CDCl_3$) δ 178.8, 154.8, 139.7, 134.8, 128.2, 123.0, 122.6, 110.0, 80.3, 63.7, 55.9, 44.0, 40.3, 34.0, 28.7, 25.2, 23.1, 22.8. HRMS (ESI) *m/z*: Calcd for $C_{20}H_{28}N_2O_3Na^+$ $[M+Na]^+$ 367.1992; Found 367.1998.



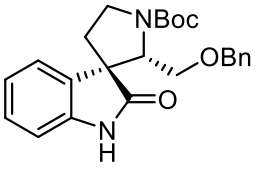
2m''

2m'' was obtained by General Procedure A with 20 mL solvent [18.4 mg, 27% yield, 8.57 μmol/(h*cm^{-2})]. Eluent solvents: hexane/EtOAc = 6:1-2:1. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.85 (s, 1H), 7.26 – 7.20 (m, 2H), 7.04 (t, J = 7.5 Hz, 1H), 6.94 (d, J = 7.9 Hz, 1H), 4.29 – 3.95 (m, 2H), 3.58 (dt, J = 11.2, 7.4 Hz, 1H), 2.31 (dt, J = 12.4, 8.5 Hz, 1H), 2.06 – 1.95 (m, 1H), 1.75 (br, 1H), 1.50 (s, 10H), 1.35 (br, 1H), 1.00 (br, 1H), 0.83 (d, J = 6.4 Hz, 3H), 0.58 (d, J = 6.3 Hz, 3H). $^{13}C\{^{1}H\}$ NMR (100 MHz, $CDCl_3$) δ 181.1, 155.4, 140.7, 130.1, 128.5, 125.3, 122.3, 110.3, 80.0, 61.2, 45.8, 41.0, 35.9, 31.1, 28.7, 24.7, 23.5, 22.0. HRMS (ESI) *m/z*: Calcd for $C_{20}H_{28}N_2O_3Na^+$ $[M+Na]^+$ 367.1992; Found 367.1997.



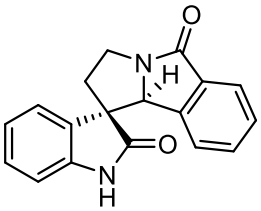
2n (**64%, *dr* 2:1**)

2n was obtained by General Procedure A [34.1 mg, 43% yield, 15.24 μmol/(h*cm^{-2})]. Eluent solvents: hexane/EtOAc = 6:1-2:1. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.66 – 8.40 (m, 1H), 7.25 – 7.06 (m, 6H), 7.06 – 6.93 (m, 2H), 6.83 (br, 1H), 4.52 – 3.69 (m, 7H), 2.45 (br, 1H), 2.07 (br, 1H), 1.64 – 1.32 (m, 9H). $^{13}C\{^{1}H\}$ NMR (100 MHz, $CDCl_3$) δ 178.7, 154.4, 140.0, 138.3, 134.3, 128.1, 127.4, 122.8, 122.4, 109.9, 80.3, 72.8, 68.6, 64.4, 55.1, 45.3, 34.7, 28.6. HRMS (ESI) *m/z*: Calcd for $C_{24}H_{28}N_2O_4Na^+$ $[M+Na]^+$ 431.1941; Found 431.1943.



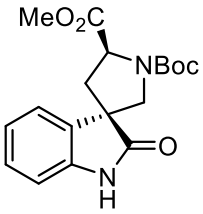
2n'

2n' was obtained by General Procedure A [16.9 mg, 21% yield, 7.44 μmol/(h*cm^{-2})]. Eluent solvents: hexane/EtOAc = 6:1-2:1. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.34 (br, 1H), 7.32 (d, J = 7.4 Hz, 1H), 7.29 – 7.16 (m, 4H), 7.11 – 6.95 (m, 3H), 6.88 (d, J = 7.7 Hz, 1H), 4.46 – 3.75 (m, 5H), 3.75 – 3.59 (m, 1H), 3.58 – 3.29 (m, 1H), 2.35 – 2.07 (m, 2H), 1.46 (s, 9H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 180.7, 155.0, 141.3, 138.1, 129.9, 128.4, 128.3, 127.5, 127.3, 124.9, 122.3, 110.1, 80.2, 73.3, 69.5, 62.2, 46.5, 35.2, 28.6. HRMS (ESI) *m/z*: Calcd for $C_{24}H_{28}N_2O_4Na^+$ $[M+Na]^+$ 431.1941; Found 431.1944.



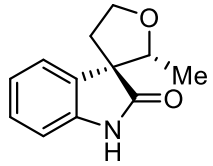
2o (81%, *dr* 5:1)

2o was obtained by General Procedure A with 20 mL solvent [23.4 mg, 81% yield, 0.1 mmol scale, 48.36 μmol/(h*cm^{-2})]. Eluent solvents: CH_2Cl_2/MeOH = 100:1. Data of the major isomer: ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.39 (s, 1H), 7.71 (d, J = 7.4 Hz, 1H), 7.30 (dt, J = 7.5, 3.8 Hz, 1H), 7.28 – 7.22 (m, 1H), 7.06 – 6.98 (m, 2H), 6.81 (d, J = 7.7 Hz, 1H), 6.68 (td, J = 7.6, 1.0 Hz, 1H), 6.49 (d, J = 7.5 Hz, 1H), 5.25 (s, 1H), 4.17 – 4.07 (m, 1H), 3.85 (ddd, J = 11.7, 9.9, 1.6 Hz, 1H), 3.11 (dt, J = 13.0, 9.8 Hz, 1H), 2.53 (ddd, J = 13.0, 8.1, 1.9 Hz, 1H). ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$) δ 178.0, 171.3, 142.1, 139.6, 134.4, 132.0, 129.2, 129.0, 128.4, 123.8, 123.5, 123.1, 122.0, 110.0, 71.0, 54.9, 41.6, 39.8. HRMS (ESI) *m/z*: Calcd for $C_{18}H_{14}N_2O_2Na^+$ $[M+Na]^+$ 313.0947; Found 313.0951.



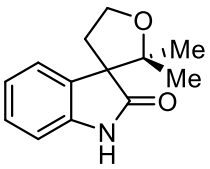
2p (94%, *dr* 5:1)

2p was obtained by General Procedure A with flow rate of 40 mL/min [65.3 mg, 94% yield, 144.21 μmol/(h*cm^{-2})] and by General Procedure B (25.3 mg, 73% yield). Eluent solvents: hexane/EtOAc = 10:1-3:1. Data of the major isomer: ^{1}H NMR (400 MHz, $CDCl_3$) (presence of rotamers) δ 8.93 – 8.82 (m, 1H), 7.29 – 7.20 (m, 1H), 7.13 – 7.08 (m, 1H), 7.08 – 7.01 (m, 1H), 6.99 – 6.92 (m, 1H), 4.79 – 4.57 (m, 1H), 3.89 – 3.66 (m, 1H), 2.63 – 2.50 (m, 1H), 2.44 – 2.34 (m, 1H), 1.51 – 1.40 (m, 1H). ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$) (presence of rotamers) δ 178.1, 178.0, 172.6, 172.3, 154.4, 153.5, 139.9, 139.9, 133.4, 133.2, 128.8, 123.3, 123.3, 122.6, 110.4, 81.0, 59.2, 58.8, 55.5, 54.9, 53.2, 52.6, 52.5, 52.3, 40.6, 39.7, 28.4. HRMS (ESI) *m/z*: Calcd for $C_{18}H_{22}N_2O_5Na^+$ $[M+Na]^+$ 369.1421; Found 369.1428.



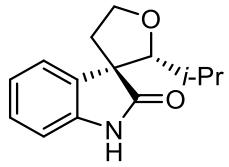
2q (63%, *dr* >20:1)

2q was obtained by General Procedure A [12.8 mg, 63% yield, 0.1 mmol scale, 65.68 μmol/(h*cm^{-2})]. Eluent solvents: hexane/EtOAc = 3:1-1:1. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.73 (br, 1H), 7.28 – 7.20 (m, 2H), 7.06 (t, J = 7.3 Hz, 1H), 6.95 (d, J = 7.6 Hz, 1H), 4.30 – 4.12 (m, 3H), 2.70 (ddd, J = 12.7, 9.4, 6.6 Hz, 1H), 2.20 (ddd, J = 10.0, 7.8, 3.7 Hz, 1H), 0.91 (d, J = 6.2 Hz, 3H). ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$) δ 180.0, 140.3, 131.8, 128.1, 124.8, 122.8, 110.0, 82.4, 67.3, 58.4, 38.2, 15.2. HRMS (ESI) *m/z*: Calcd for $C_{12}H_{13}NO_2Na^+$ $[M+Na]^+$ 226.0838; Found 226.0838.



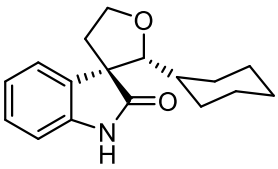
2r (74%)

2r was obtained by General Procedure A [32.2 mg, 74% yield, 80.08 μmol/(h*cm^{-2})]. Eluent solvents: hexane/EtOAc = 3:1-1:1. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.21 (br, 1H), 7.26 (d, J = 7.4 Hz, 2H), 7.22 (t, J = 7.6 Hz, 1H), 7.03 (t, J = 7.6 Hz, 1H), 6.90 (d, J = 7.7 Hz, 1H), 4.27 (td, J = 8.9, 5.6 Hz, 1H), 4.18 (td, J = 8.7, 6.0 Hz, 1H), 2.67 (ddd, J = 12.8, 9.3, 6.0 Hz, 1H), 2.31 (ddd, J = 12.9, 8.9, 5.4 Hz, 1H), 1.34 (s, 3H), 1.10 (s, 3H). ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$) δ 179.9, 140.6, 131.8, 128.2, 125.1, 122.5, 109.7, 84.9, 64.5, 60.0, 36.1, 24.4, 23.4. HRMS (ESI) *m/z*: Calcd for $C_{13}H_{15}NO_2Na^+$ $[M+Na]^+$ 240.0995; Found 240.0998.



2s (67%, *dr* >20:1)

2s was obtained by General Procedure A [31.0 mg, 67% yield, 23.75 μmol/(h*cm^{-2})]. Eluent solvents: hexane/EtOAc = 3:1-1:1. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.92 (s, 1H), 7.30 – 7.19 (m, 2H), 7.06 (t, J = 7.5 Hz, 1H), 6.97 (d, J = 7.7 Hz, 1H), 4.24 – 4.09 (m, 2H), 3.73 (d, J = 9.9 Hz, 1H), 2.68 (ddd, J = 12.4, 9.1, 7.6 Hz, 1H), 2.13 (ddd, J = 12.7, 7.8, 5.2 Hz, 1H), 1.59 (ddt, J = 13.2, 10.0, 6.6 Hz, 1H), 1.00 (d, J = 6.5 Hz, 3H), 0.43 (d, J = 6.7 Hz, 3H). ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$) δ 180.9, 140.2, 132.1, 128.1, 124.8, 122.7, 110.1, 91.6, 66.6, 56.9, 40.6, 30.3, 21.1, 17.6. HRMS (ESI) *m/z*: Calcd for $C_{14}H_{17}NO_2Na^+$ $[M+Na]^+$ 254.1151; Found 254.1148.

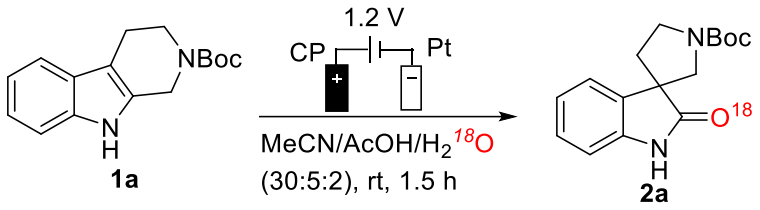


2t (71%, *dr* >20:1)

2**t** was obtained by General Procedure A [38.5 mg, 71% yield, 46.36 μmol/(h*cm^{-2})]. Eluent solvents: hexane/EtOAc = 3:1-1:1. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.62 (s, 1H), 7.29 – 7.21 (m, 2H), 7.06 (td, J = 7.6, 1.0 Hz, 1H), 6.94 (d, J = 7.7 Hz, 1H), 4.23 – 4.08 (m, 2H), 3.81 (d, J = 9.8 Hz, 1H), 2.64 (ddd, J = 12.4, 9.3, 7.5 Hz, 1H), 2.09 (ddd, J = 12.7, 7.8, 5.1 Hz, 1H), 2.03 – 1.96 (m, 1H), 1.69 – 1.60 (m, 1H), 1.52 – 1.43 (m, 1H), 1.43 – 1.29 (m, 2H), 1.14 – 0.96 (m, 3H), 0.90 – 0.81 (m, 1H), 0.81 – 0.71 (m, 2H). ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$) δ 180.6, 139.9, 132.1, 128.0, 124.7, 122.8, 110.1, 90.1, 66.4, 56.6, 40.6, 39.4, 31.1, 27.4, 26.2, 25.5. HRMS (ESI) *m/z*: Calcd for $C_{17}H_{21}NO_2Na^+$ $[M+Na]^+$ 294.1464; Found 294.1465.

9. Mechanistic study

9.1 Isotopic labelling experiment with ^{18}O

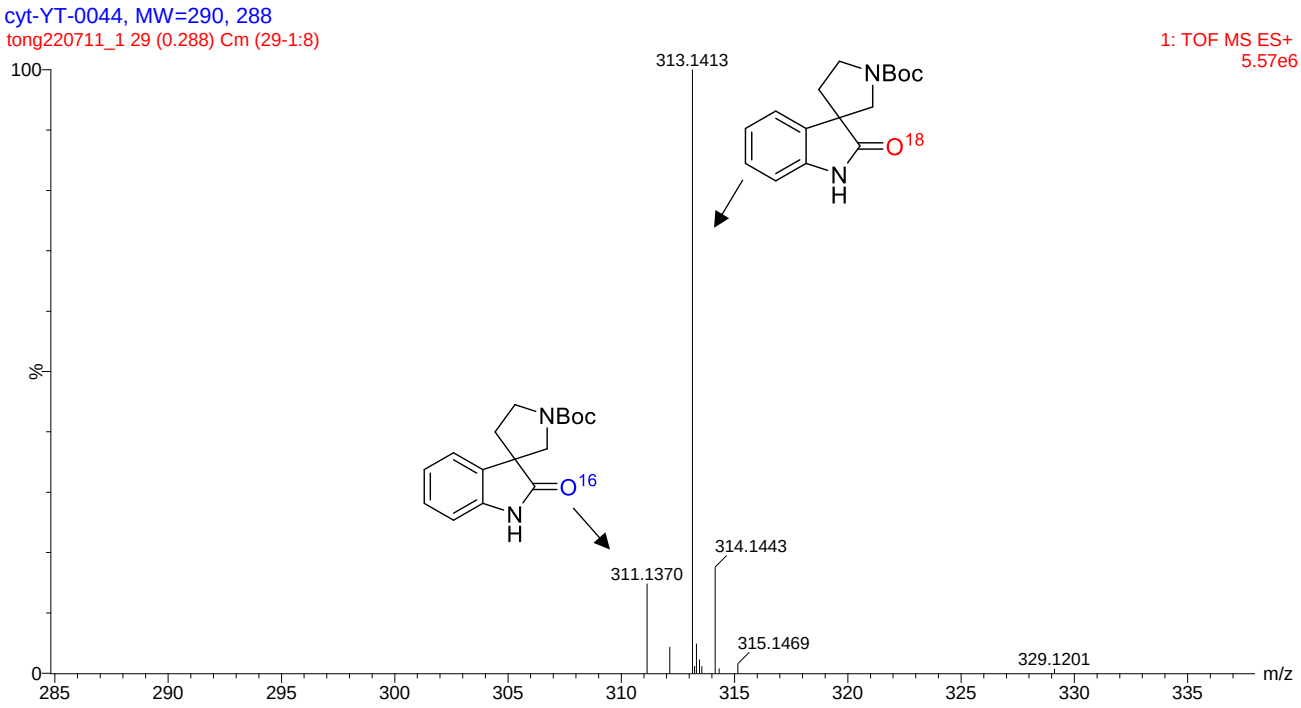


Reactions were conducted in two 4 mL vials with anolyte and catholyte. The assemble and detail information were discussed in **Flow cell setup.** The reaction was performed in the two PEEK housing flow cell at constant voltage 1.2 V using potentiostatic method in VMP-300. Electrolyte was prepared by the addition of LiBr (0.2 mmol, 2.0 equiv.) in MeCN/AcOH/$H_2{}^{18}O$ (30:5:2, 3 mL). **1a** (27.2mg, 0.1 mmol) was added in the anolyte. The anolyte was separated from the cathode electrolyte (3mL) by a proton-exchange membrane Nafion 117. The 20 mL/min was set for synthsis, and the cut-off charge was set 2 F/mol. Reaction was monitored by TLC. The reaction mixture was diluted with EtOAc (10 mL). Saturated aqueous Na_2CO_3 solution (10 mL) was added. The organic fraction was collected, and the aqueous fraction was extracted with EtOAc (2 x 10 mL). The combined organic fraction was washed with brine, dried over Na_2SO_4, filtered and concentrated under reduced pressure. The resulting residue was purified by flash column chromatography on silica gel (CH_2Cl_2/MeOH = 50:1) to give compound **2a** (26.9mg, 92.6%). ^{1}H NMR (400 MHz, $CDCl_3$) (presence of rotamers) δ 8.69 – 8.60 (m, 1H), 7.26 - 7.20 (m, 1H), 7.18 (t, J = 9.7 Hz, 1H), 7.05 (t, J = 7.4 Hz, 1H), 6.97 - 6.90 (m, 1H), 3.92 - 3.68 (m, 3H), 3.65 – 3.55 (m, 1H), 2.47 - 2.36 (m, 1H), 2.15 - 2.01 (m, 1H), 1.48 (d, J = 24.1 Hz, 9H). ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$) (presence of rotamers) δ 180.3, 180.0, 154.6, 140.2, 133.2, 128.5, 123.1, 122.9, 110.2, 80.0, 54.5, 54.0, 53.5, 52.5, 45.6, 45.3, 36.5, 35.7, 28.7, 28.6. HRMS (ESI) *m/z*: Calcd for $C_{16}H_{20}N_2O_2{}^{18}ONa^+$ $[M+Na]^+$ 313.1409; Found 313.1413. *m/z*: Calcd for $C_{16}H_{20}N_2O_3Na^+$ $[M+Na]^+$ 311.1366; Found 311.1370.

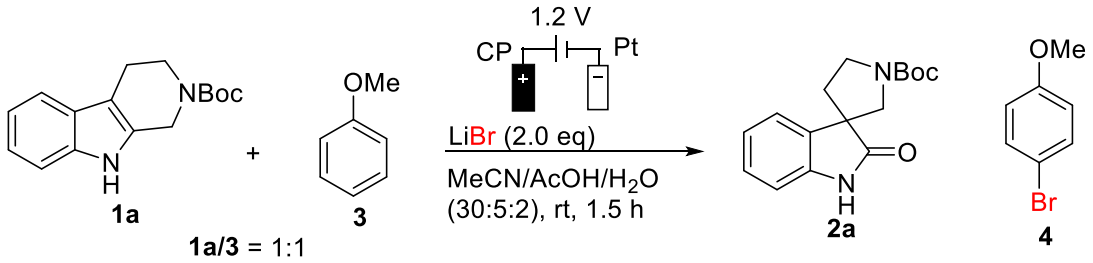
Intensity of peak *m/z* 313.1413 ($C_{16}H_{20}N_2O_2{}^{18}ONa^+$) : 5.572e6

Intensity of peak *m/z* 311.1370 ($C_{16}H_{20}N_2O_3Na^+$) : 8.255e5

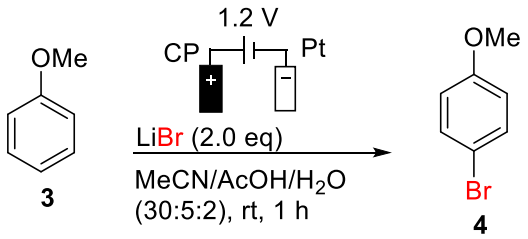
Percentage of ^{18}O substitution = $\frac{5.572e6}{5.572e6+8.255e5}$ = 87%



9.2 RBS trapping



The reaction was conducted following General Procedure A in 0.1 mmol scale with 1.0 equiv. **3** (0.1 mmol) added in anolyte. After workup, the ratio of **2a** and **4** was determined by NMR of the crude product. It was found that **2a**/**4** = 1:0.

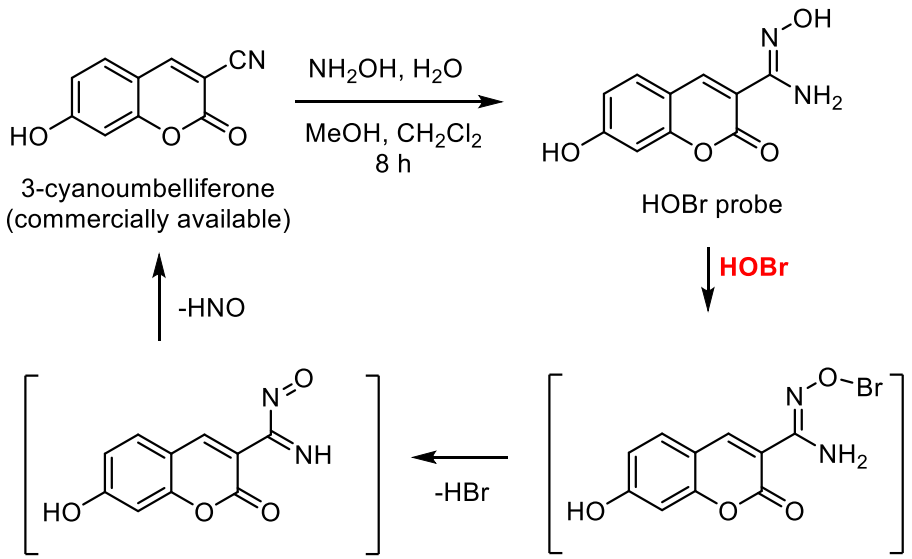


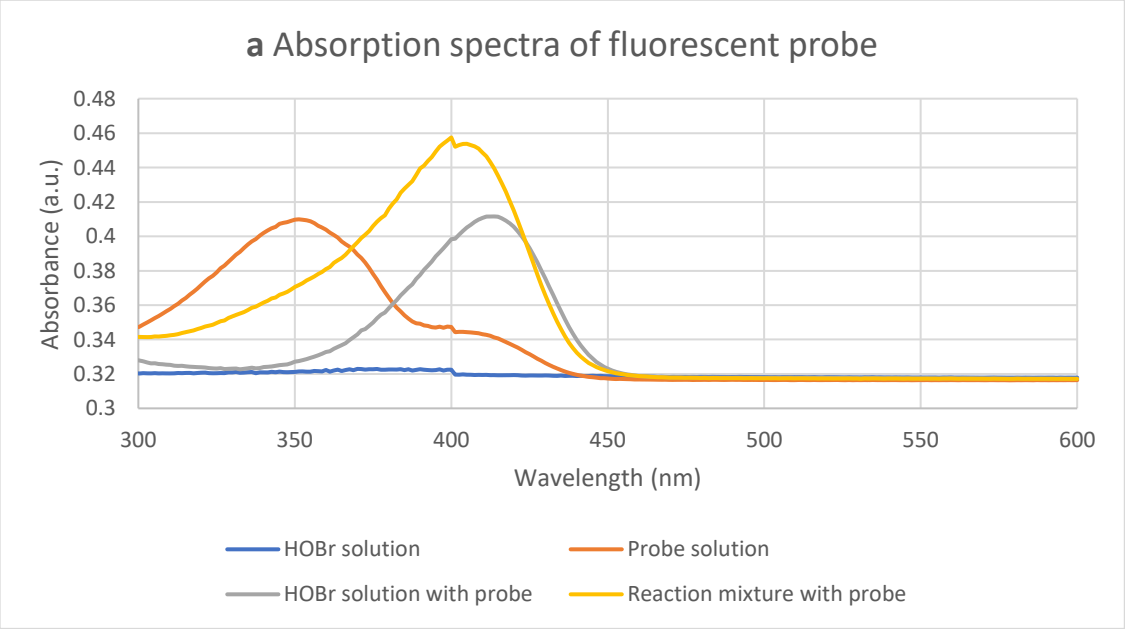
The reaction was conducted following General Procedure A in 0.1 mmol scale with **3** instead of **1a** added in anolyte. Compound **4** was obtained without further purification (16.9mg, 91%). ^{1}H NMR

(400 MHz, $CDCl_3$) δ 7.42 - 7.34 (m, 2H), 6.82 - 6.75 (m, 2H), 3.78 (s, 3H). $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$) δ 158.8, 132.4, 115.9, 113.0, 55.6. HRMS (CI) *m/z*: Calcd for $C_7H_7OBr^+$ $[M]^+$ 185.9675; Found 185.9687.

9.3 Detection of RBS

The fluorescent probe was prepared according to the phblished literature[21].





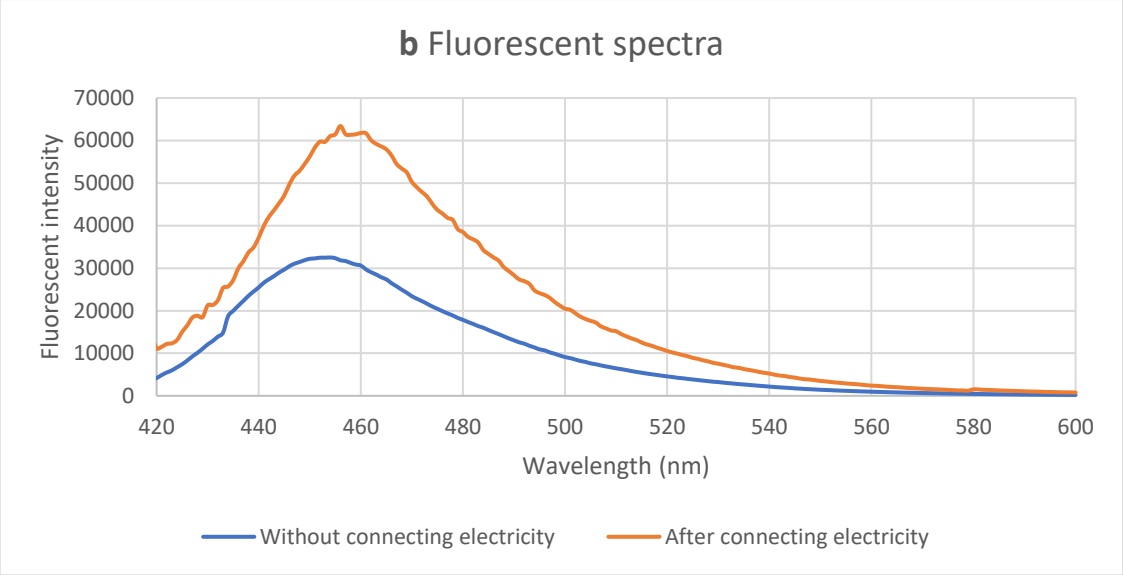


Figure S8. (a). Absorption spectra of fluorescent probe in different solution and HOBr solution. (b). Fluorescent spectra of reaction mixture before and during reaction.

Based on the results from Zeng[21], the shift in both UV absorption and fluosecent emission refer to the presence of HOBr (RBS) in the reaction mixture after connecting to electricity. This can support the *in situ* generation of HOBr after connecting electricity.

10. References

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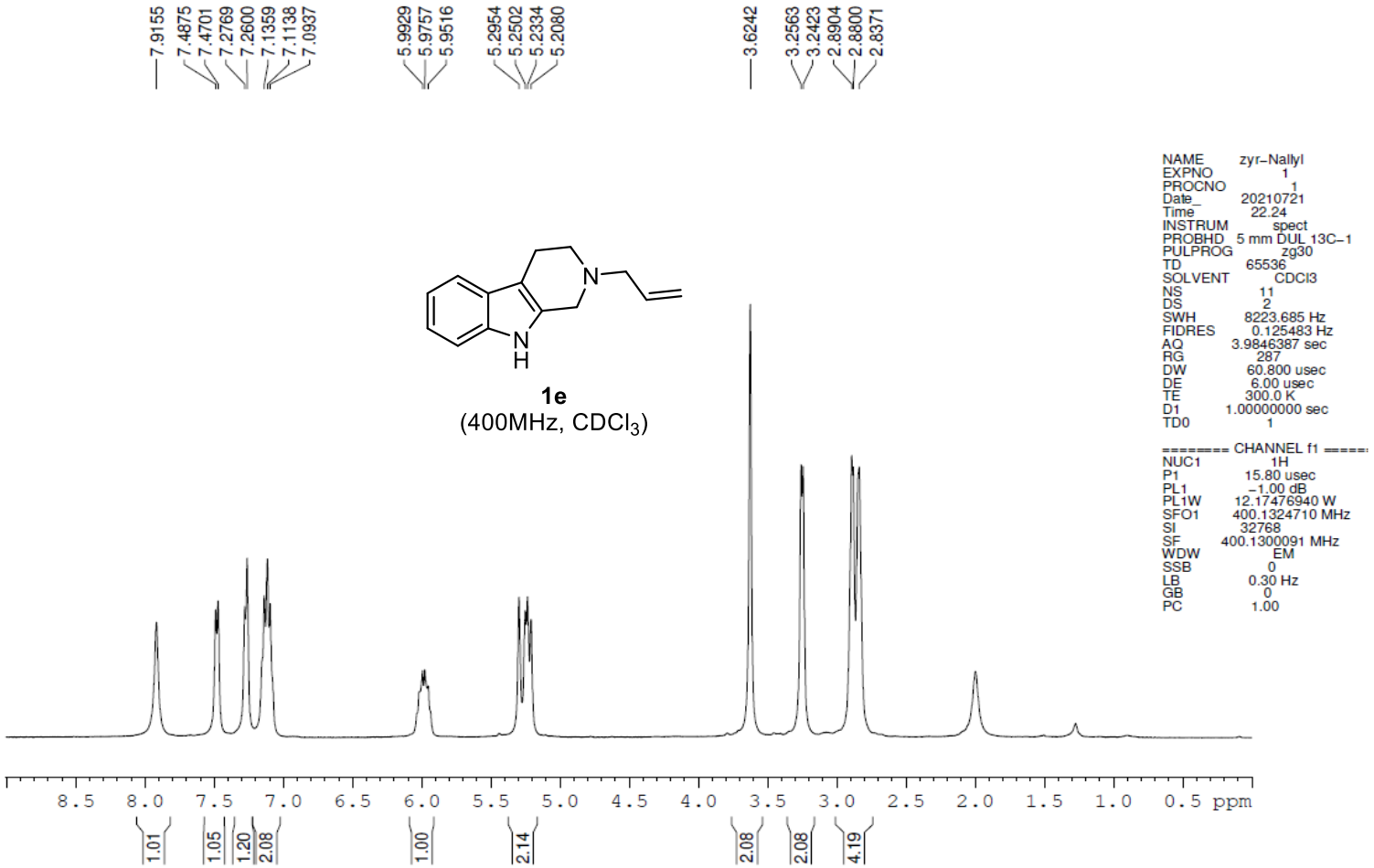
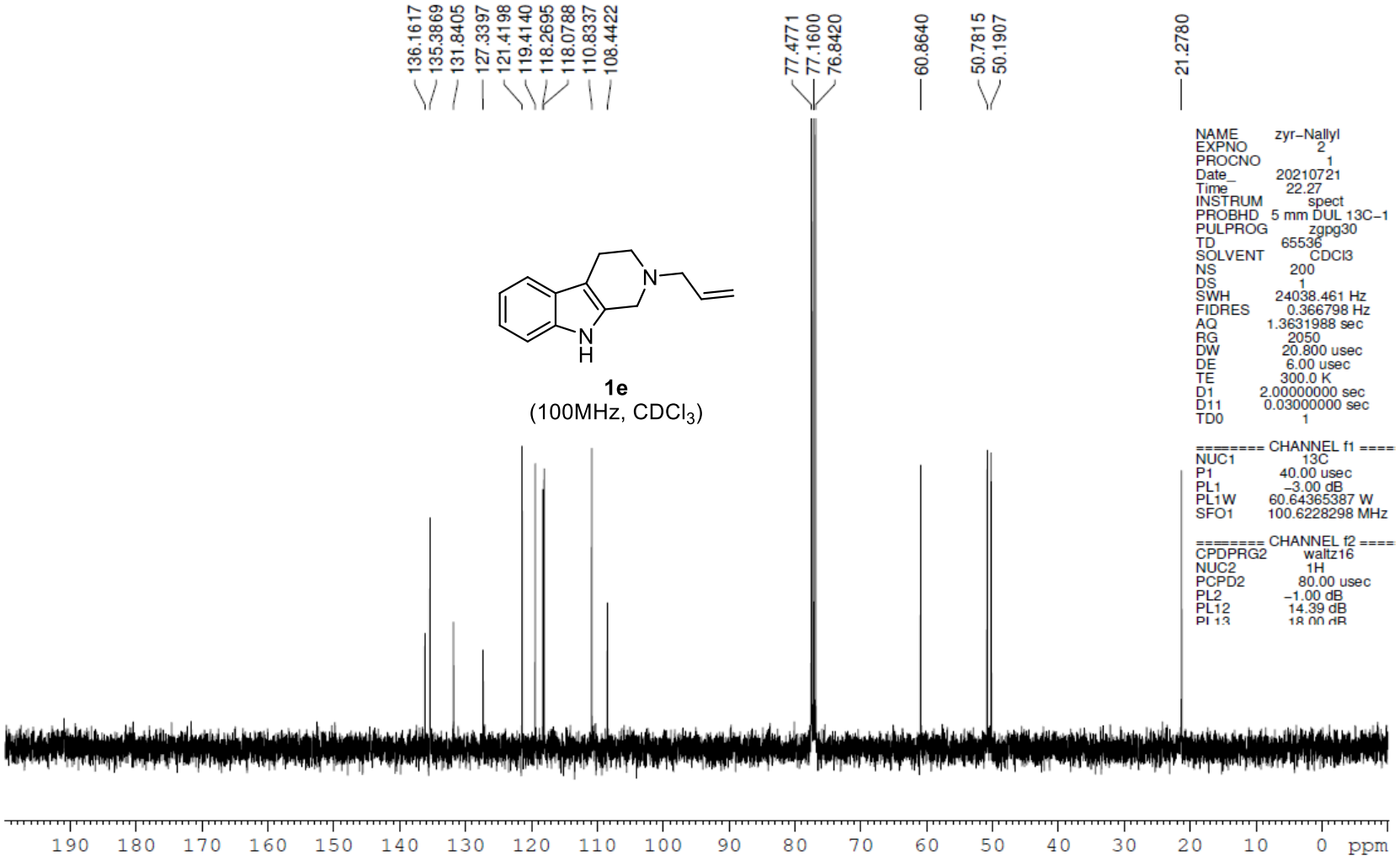
11. Copies of ^{1}H- and ^{13}C-NMR spectra (S28-S85)




136.1617
135.3869
131.8405
127.3397
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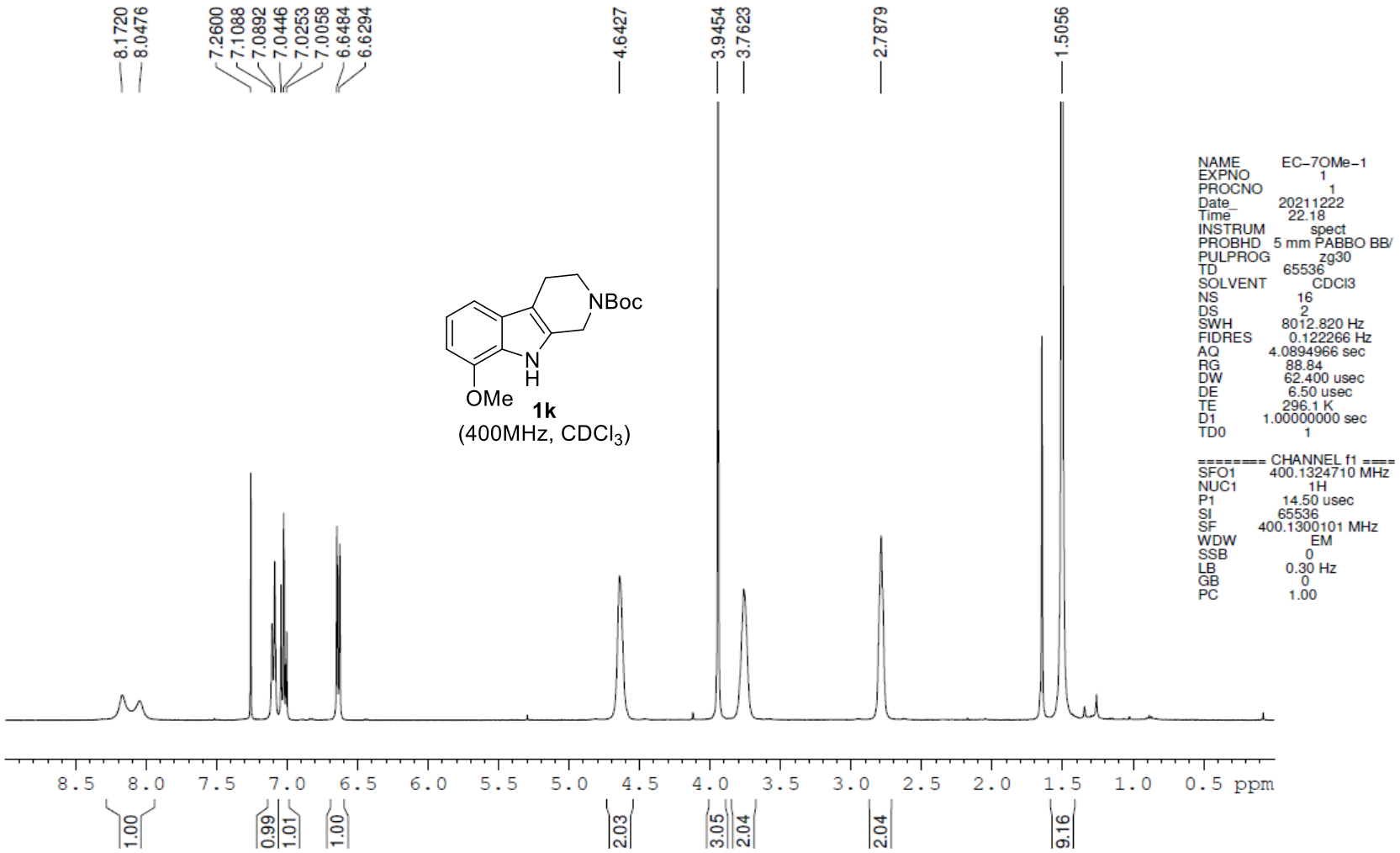
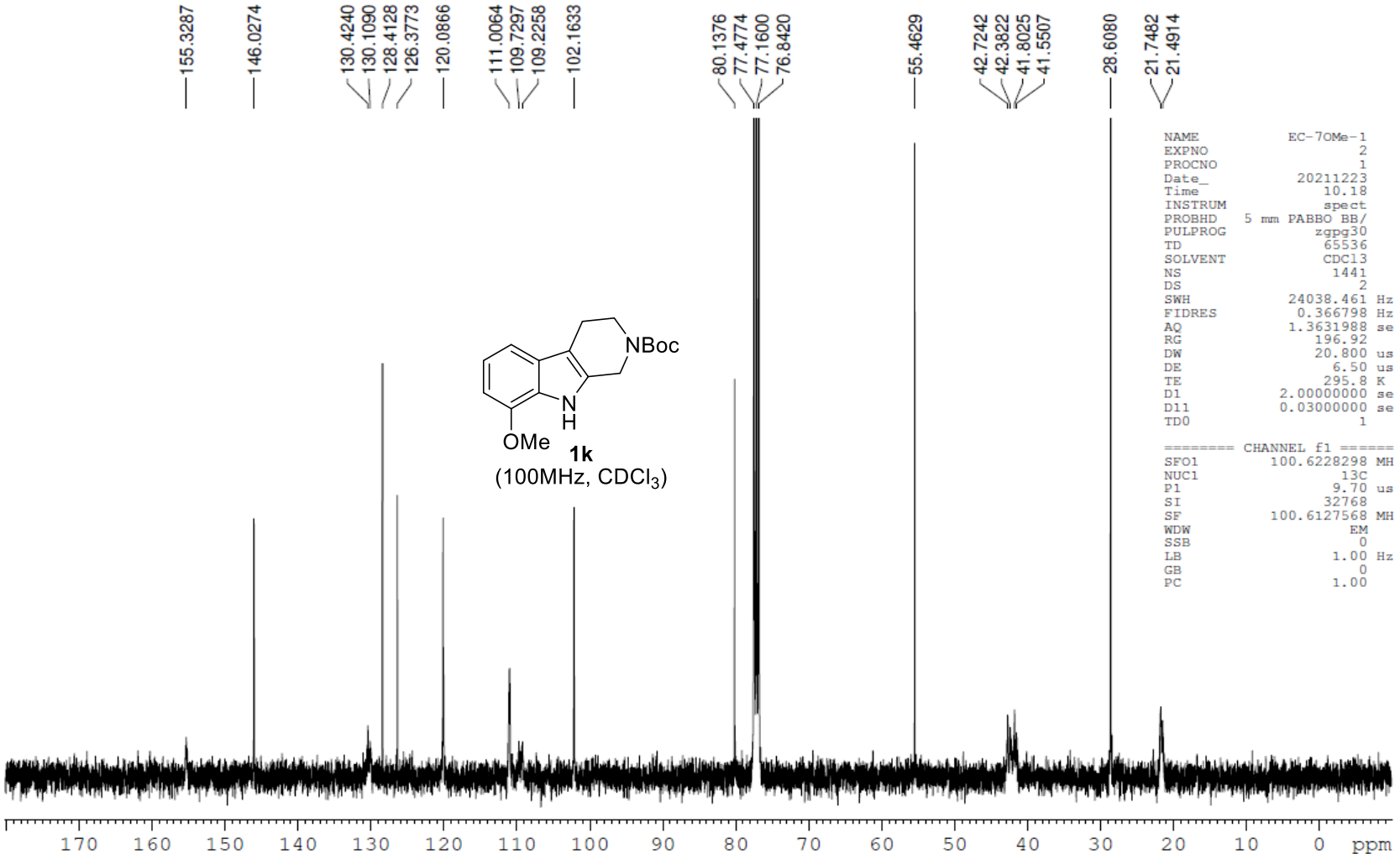
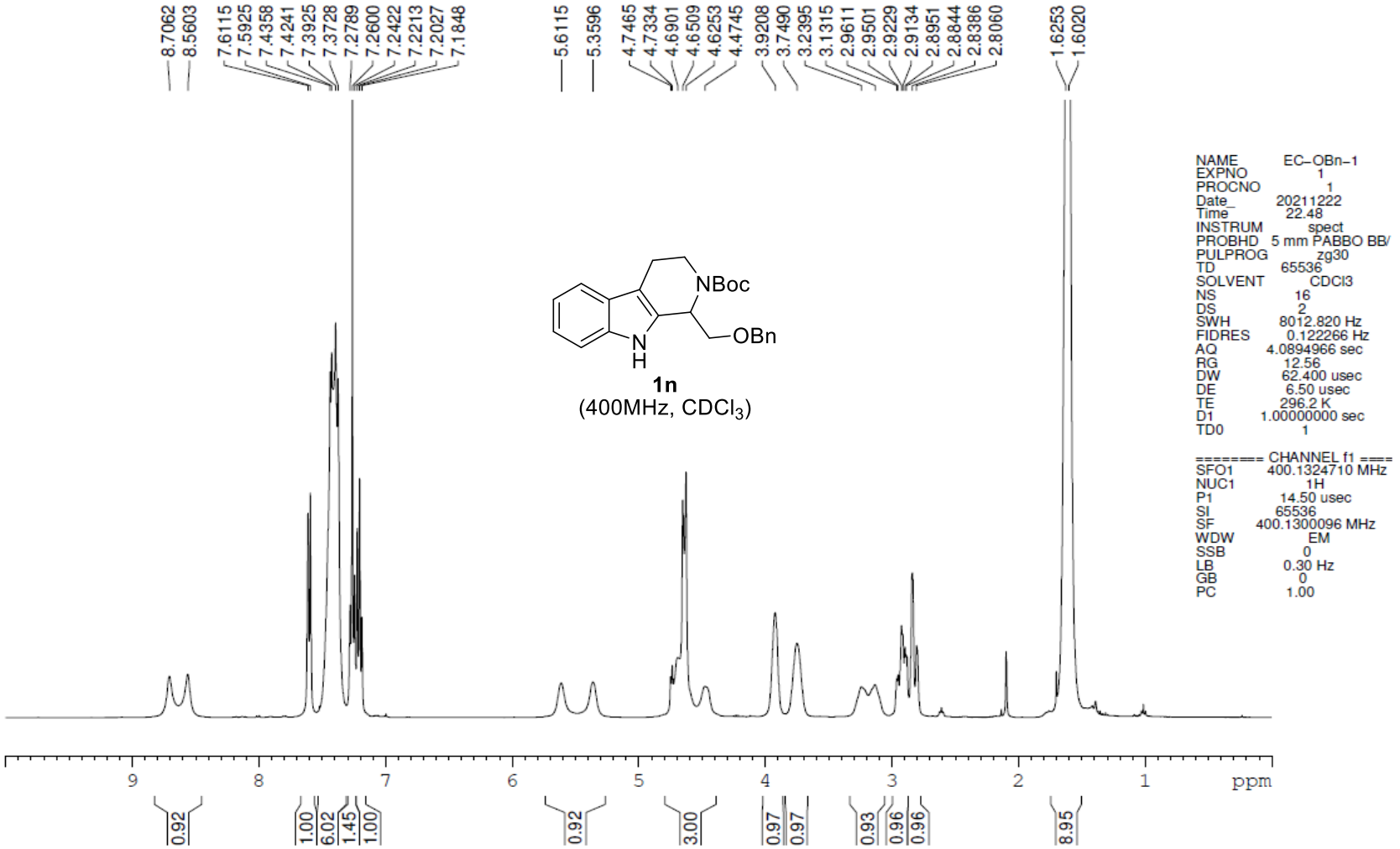
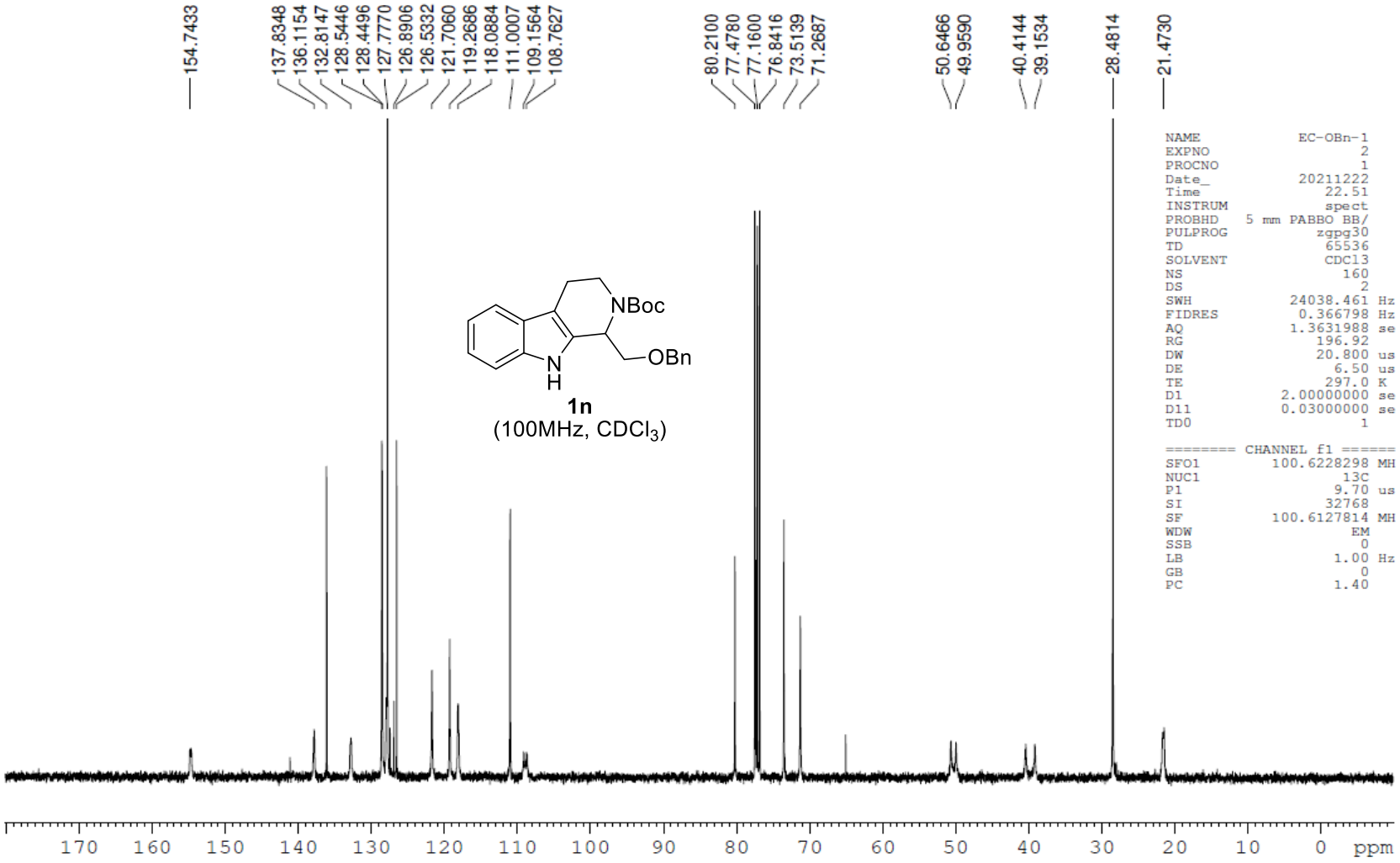
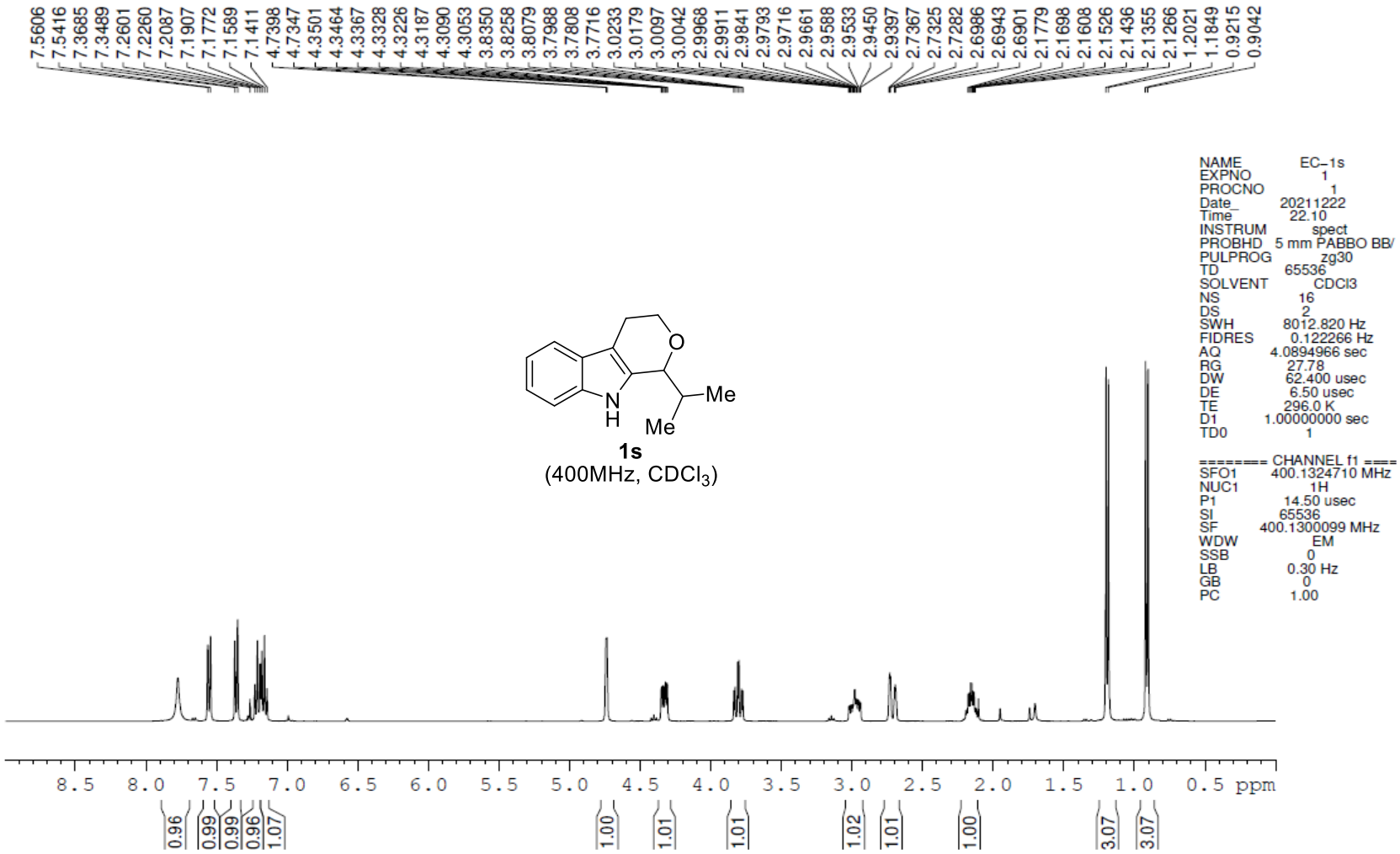
118.2695
118.0788
110.8337
108.4422
77.4771
77.1600
76.8420
60.8640
50.7815
50.1907
21.2780
N
N
H
1e
(100MHz, CDCl3)
NAME zyr-Nallyl
EXPNO 2
PROCNO 1
Date_ 20210721
Time 22.27
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 200
DS 1
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 2050
DW 20.800 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ====
NUC1 13C
P1 40.00 usec
PL1 -3.00 dB
PL1W 60.64365387 W
SFO1 100.6228298 MHz
======== CHANNEL f2 ====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -1.00 dB
PL12 14.39 dB
PL13 18.00 dB
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

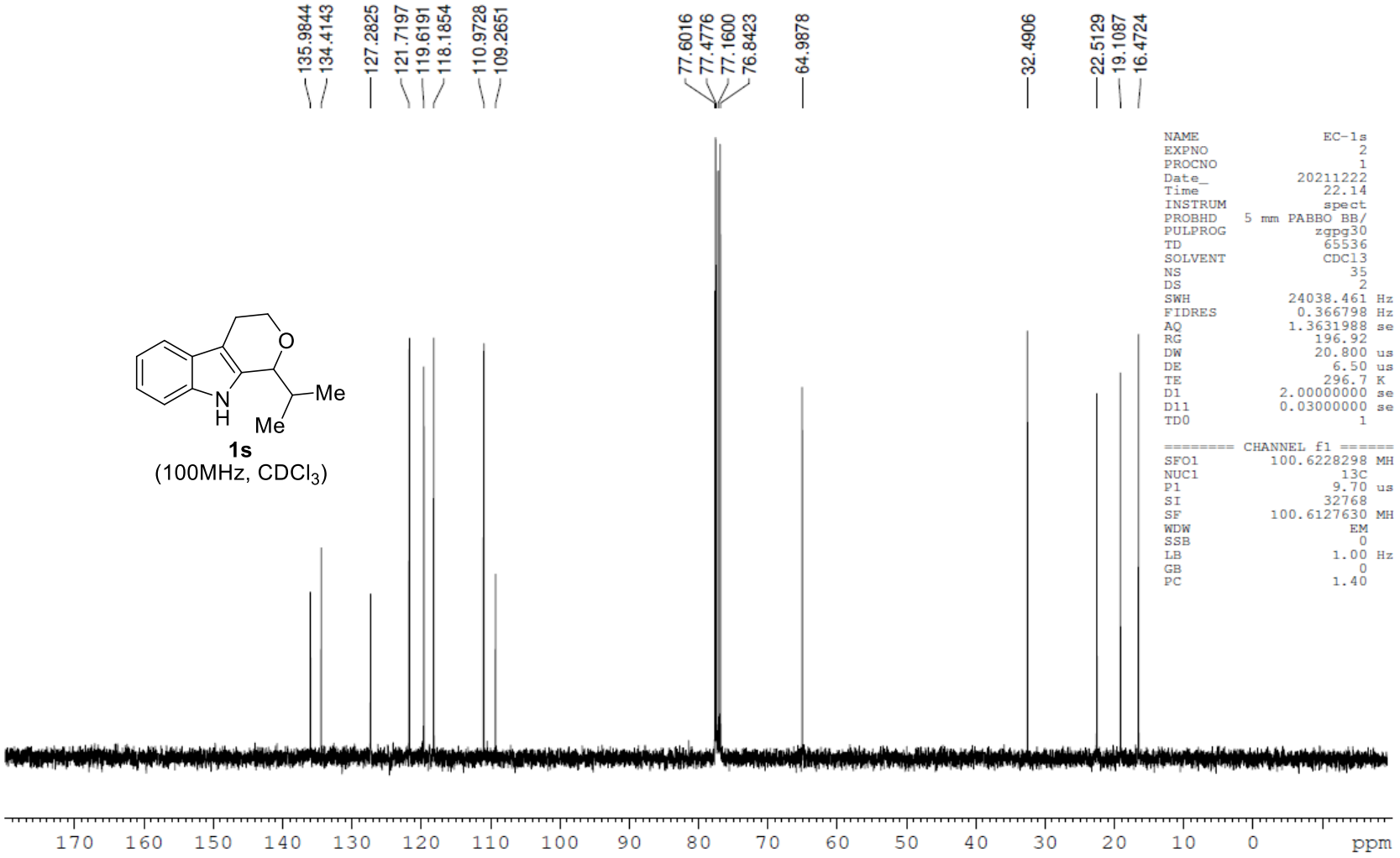

8.1720
8.0476
7.2600
7.1088
7.0892
7.0446
7.0253
7.0058
6.6484
6.6294
4.6427
3.9454
3.7623
2.7879
1.5056
NBoc
N
H
OMe
1k
(400MHz, CDCl3)
NAME EC-7OMe-1
EXPNO 1
PROCNO 1
Date_ 20211222
Time 22.18
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894966 sec
RG 88.84
DW 62.400 usec
DE 6.50 usec
TE 296.1 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ===
SFO1 400.1324710 MHz
NUC1 1H
P1 14.50 usec
SI 65536
SF 400.1300101 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
1.00
0.99
1.01
1.00
2.03
3.05
2.04
2.04
9.16

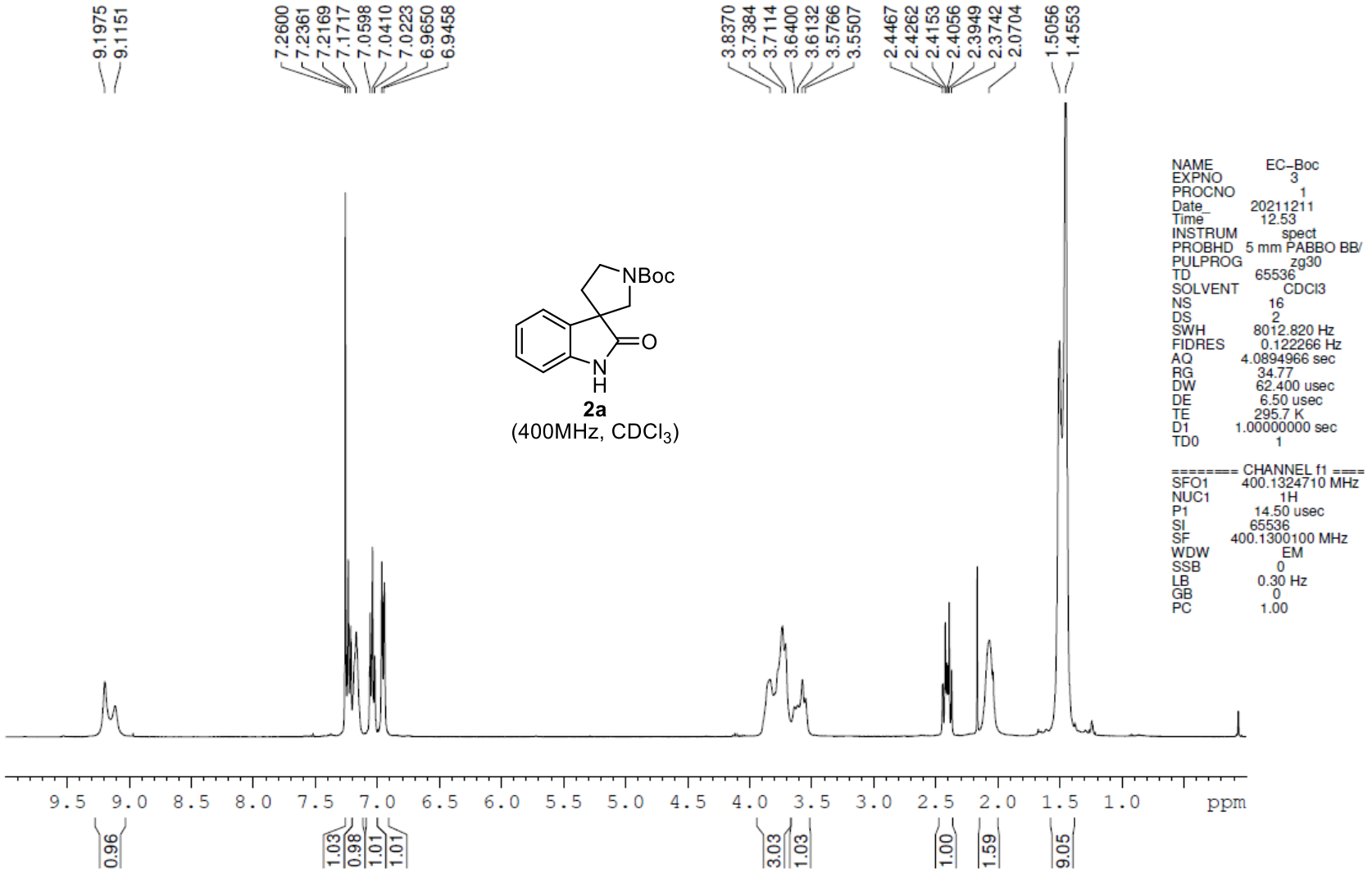

155.3287
146.0274
130.4240
130.1090
128.4128
126.3773
120.0866
111.0064
109.7297
109.2258
102.1633
80.1376
77.4774
77.1600
76.8420
55.4629
42.7242
42.3822
41.8025
41.5507
28.6080
21.7482
21.4914
NBoc
N
H
OMe
1k
(100MHz, CDCl3)
NAME EC-7OMe-1
EXPNO 2
PROCNO 1
Date_ 20211223
Time 10.18
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1441
DS 2
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 se
RG 196.92
DW 20.800 us
DE 6.50 us
TE 295.8 K
D1 2.00000000 se
D11 0.03000000 se
TD0 1
======== CHANNEL f1 ======
SFO1 100.6228298 MH
NUC1 13C
P1 9.70 us
SI 32768
SF 100.6127568 MH
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.00
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

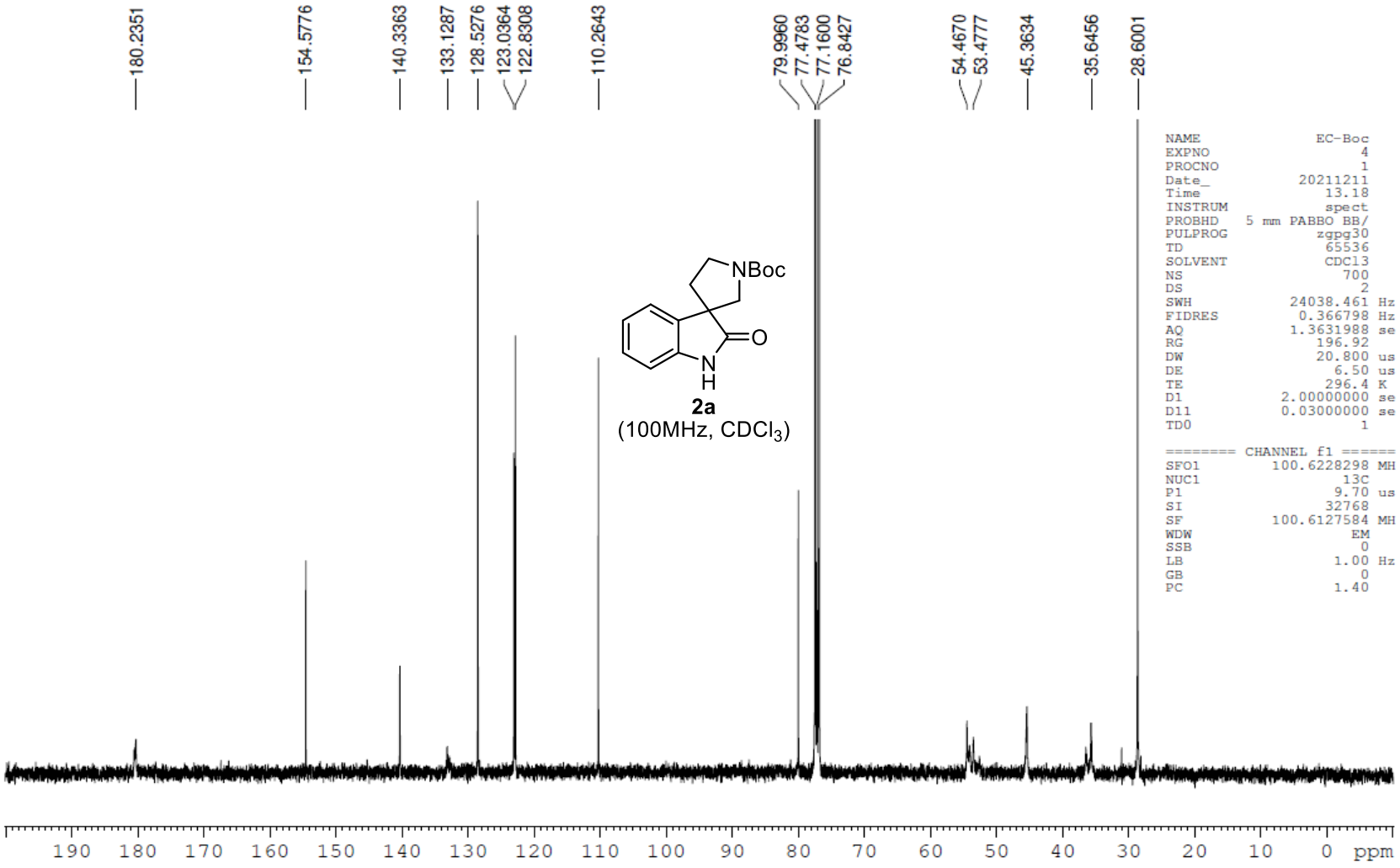

8.7062
8.5603
7.6115
7.5925
7.4358
7.4241
7.3925
7.3728
7.2789
7.2600
7.2422
7.2213
7.2027
7.1848
5.6115
5.3596
4.7465
4.7334
4.6901
4.6509
4.6253
4.4745
3.9208
3.7490
3.2395
3.1315
2.9611
2.9501
2.9229
2.9134
2.8951
2.8844
2.8386
2.8060
1.6253
1.6020
NBoc
OBn
N
H
1n
(400MHz, CDCl3)
NAME EC-OBn-1
EXPNO 1
PROCNO 1
Date_ 20211222
Time 22.48
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894966 sec
RG 12.56
DW 62.400 usec
DE 6.50 usec
TE 296.2 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ====
SFO1 400.1324710 MHz
NUC1 1H
P1 14.50 usec
SI 65536
SF 400.1300096 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
9
8
7
6
5
4
3
2
1
ppm
0.92
1.00
6.02
1.45
1.00
0.92
3.00
0.97
0.97
0.93
0.96
0.96
8.95

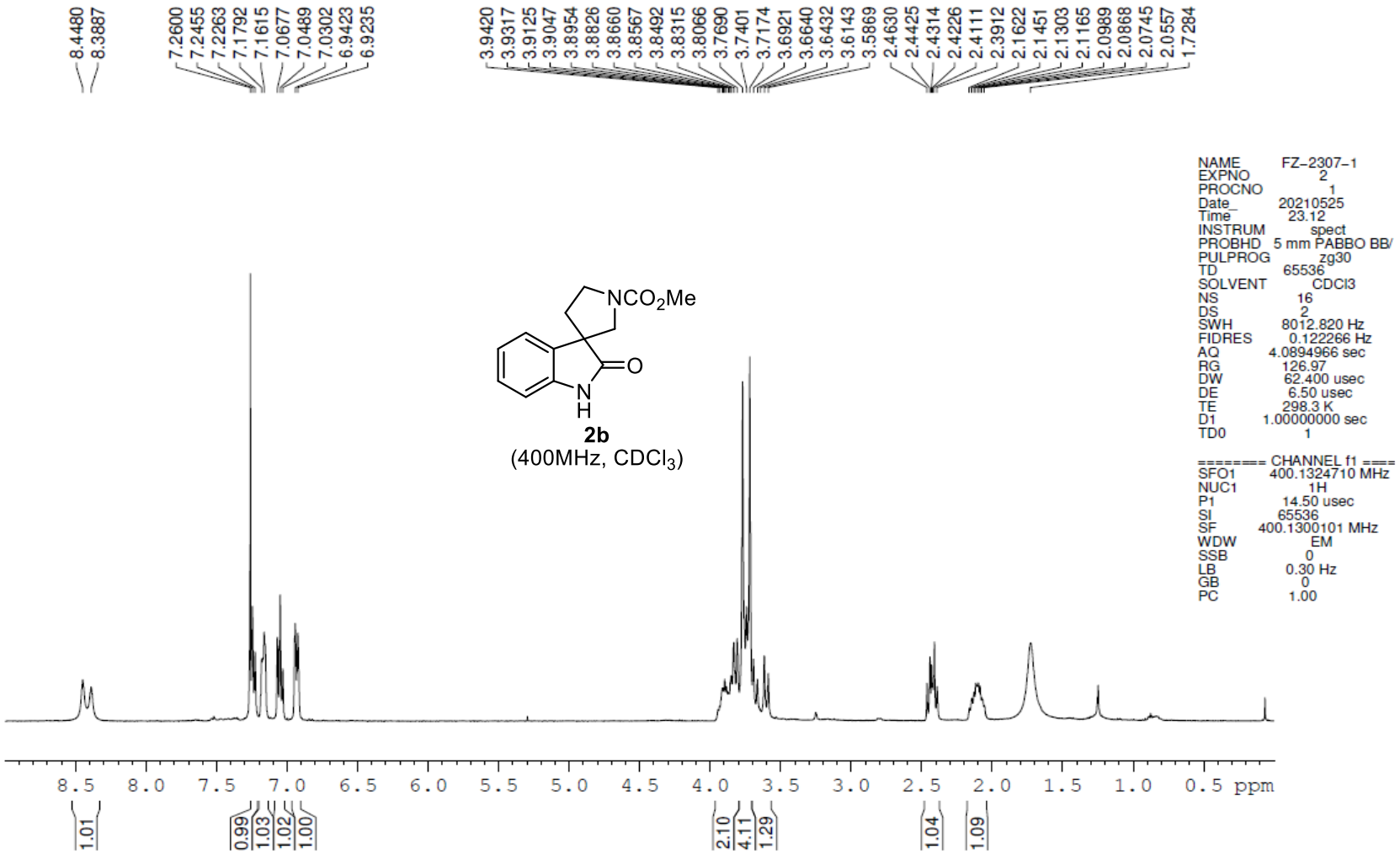

154.7433
137.8348
136.1154
132.8147
128.5446
128.4496
127.7770
126.8906
126.5332
121.7060
119.2686
118.0884
111.0007
109.1564
108.7627
80.2100
77.4780
77.1600
76.8416
73.5139
71.2687
50.6466
49.9590
40.4144
39.1534
28.4814
21.4730
NBoc
OBn
N
H
1n
(100MHz, CDCl3)
NAME EC-OBn-1
EXPNO 2
PROCNO 1
Date_ 20211222
Time 22.51
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 160
DS 2
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 se
RG 196.92
DW 20.800 us
DE 6.50 us
TE 297.0 K
D1 2.00000000 se
D11 0.03000000 se
TD0 1
======== CHANNEL f1 ======
SFO1 100.6228298 MH
NUC1 13C
P1 9.70 us
SI 32768
SF 100.6127814 MH
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

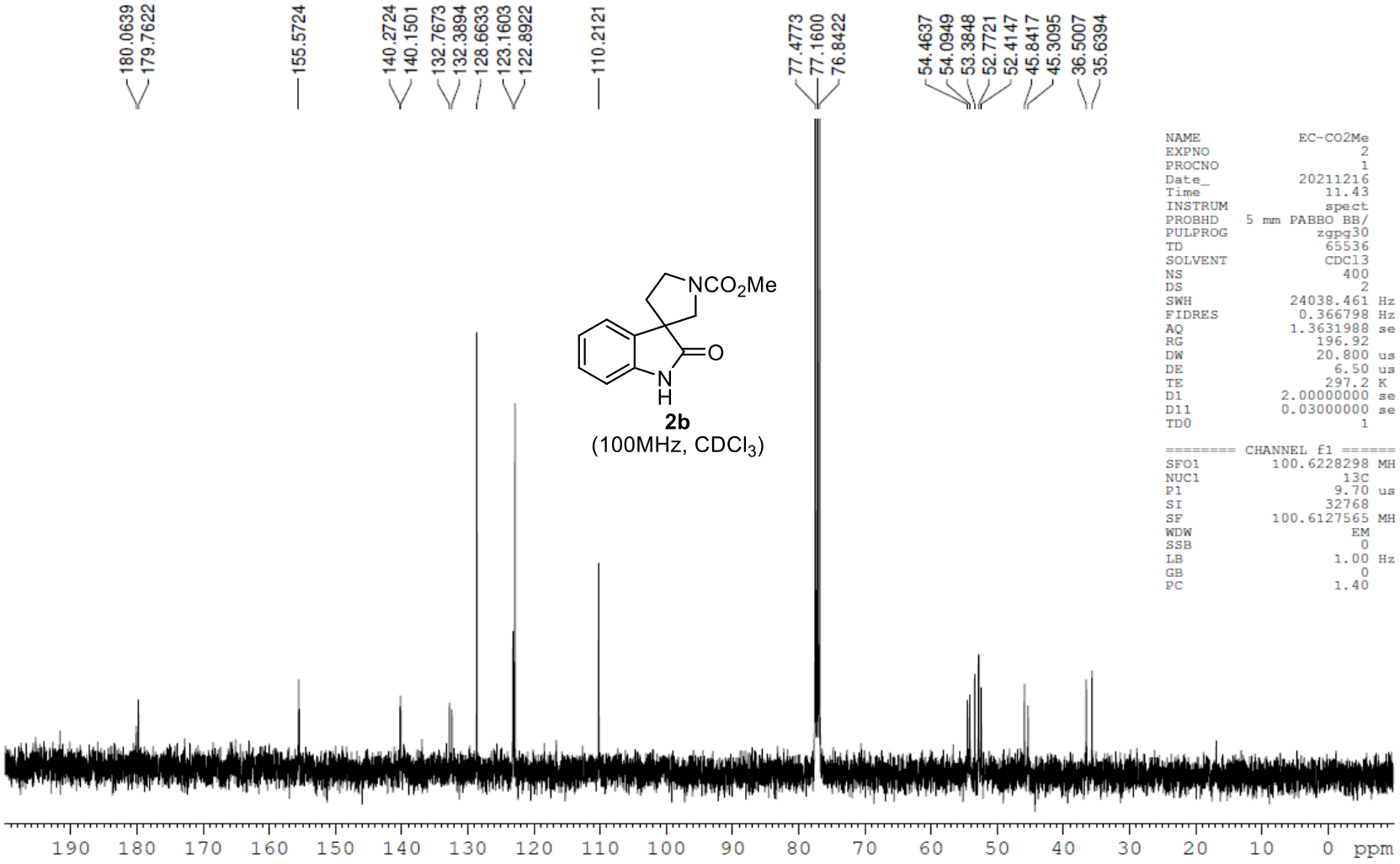

7.5606
7.5416
7.3685
7.3489
7.2601
7.2260
7.2087
7.1907
7.1772
7.1589
7.1411
4.7398
4.7347
4.3501
4.3464
4.3367
4.3328
4.3226
4.3187
4.3090
4.3053
3.8350
3.8258
3.8079
3.7988
3.7808
3.7716
3.0233
3.0179
3.0097
3.0042
2.9968
2.9911
2.9841
2.9793
2.9716
2.9661
2.9588
2.9533
2.9450
2.9397
2.7367
2.7325
2.7282
2.6986
2.6943
2.6901
2.1779
2.1698
2.1608
2.1526
2.1436
2.1355
2.1266
2.1021
1.1849
0.9215
0.9042
O
N
H
Me
Me
1s
(400MHz, CDCl3)
NAME EC-1s
EXPNO 1
PROCNO 1
Date_ 20211222
Time 22.10
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894966 sec
RG 27.78
DW 62.400 usec
DE 6.50 usec
TE 296.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ===
SFO1 400.1324710 MHz
NUC1 1H
P1 14.50 usec
SI 65536
SF 400.1300099 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
0.96
0.99
0.99
0.96
1.07
1.00
1.01
1.01
1.02
1.01
1.00
3.07
3.07

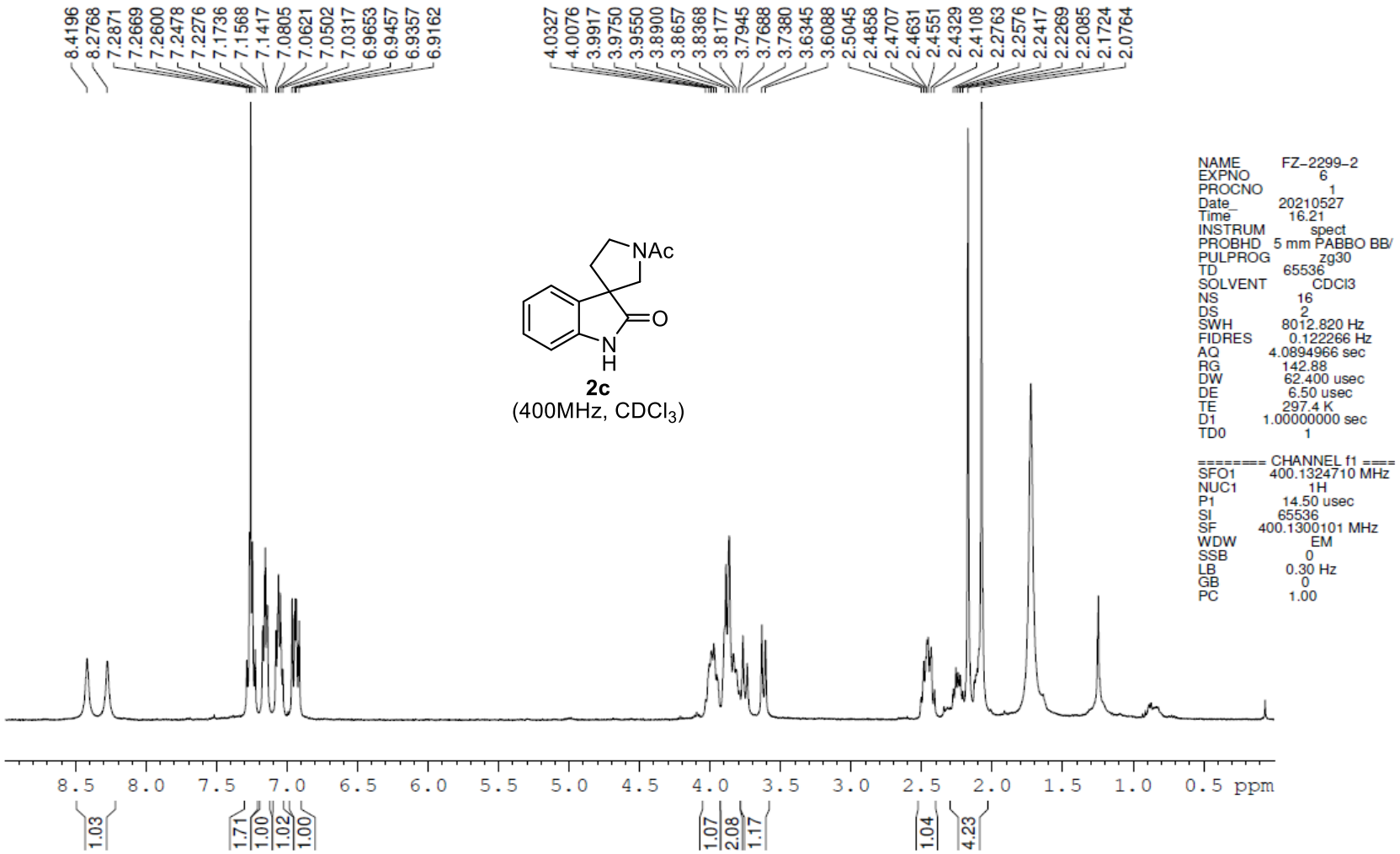

135.9844
134.4143
127.2825
121.7197
119.6191
118.1854
110.9728
109.2651
77.6016
77.4776
77.1600
76.8423
64.9878
32.4906
22.5129
19.1087
16.4724
O
N
H
Me
Me
1s
(100MHz, CDCl3)
NAME EC-1s
EXPNO 2
PROCNO 1
Date_ 20211222
Time 22.14
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 35
DS 2
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 se
RG 196.92
DW 20.800 us
DE 6.50 us
TE 296.7 K
D1 2.00000000 se
D11 0.03000000 se
TD0 1
======== CHANNEL f1 ======
SFO1 100.6228298 MH
NUC1 13C
P1 9.70 us
SI 32768
SF 100.6127630 MH
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

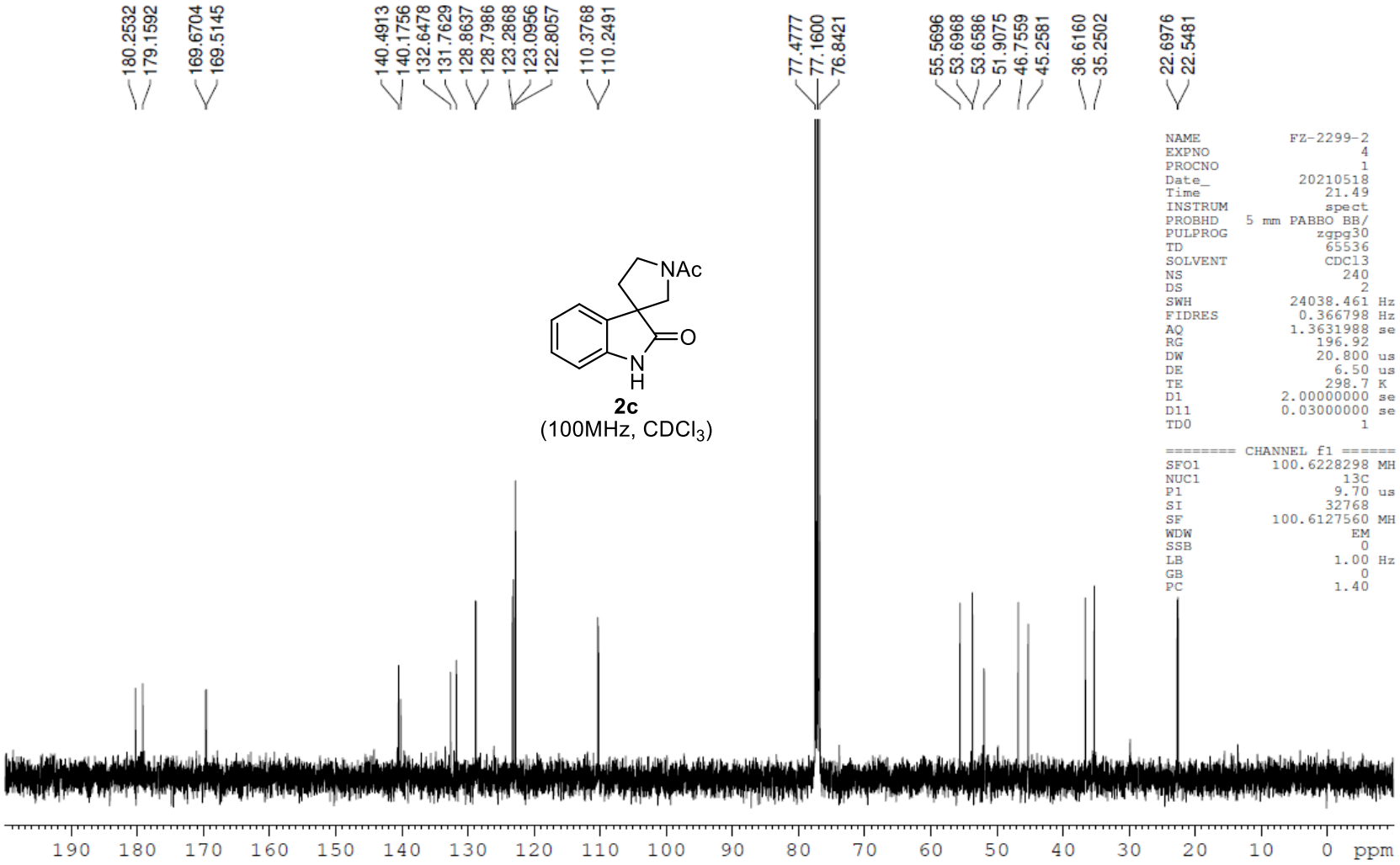

9.1975
9.1151
7.2600
7.2361
7.2169
7.1717
7.0598
7.0410
7.0223
6.9650
6.9458
3.8370
3.7384
3.7114
3.6400
3.6132
3.5766
3.5507
2.4467
2.4262
2.4153
2.4056
2.3949
2.3742
2.0704
1.5056
1.4553
NBoc
O
N
H
2a
(400MHz, CDCl3)
NAME EC--Boc
EXPNO 3
PROCNO 1
Date_ 20211211
Time 12.53
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894966 sec
RG 34.77
DW 62.400 usec
DE 6.50 usec
TE 295.7 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ====
SFO1 400.1324710 MHz
NUC1 1H
P1 14.50 usec
SI 65536
SF 400.1300100 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
1.03
0.98
1.01
1.01
3.03
1.03
1.00
1.59
9.05

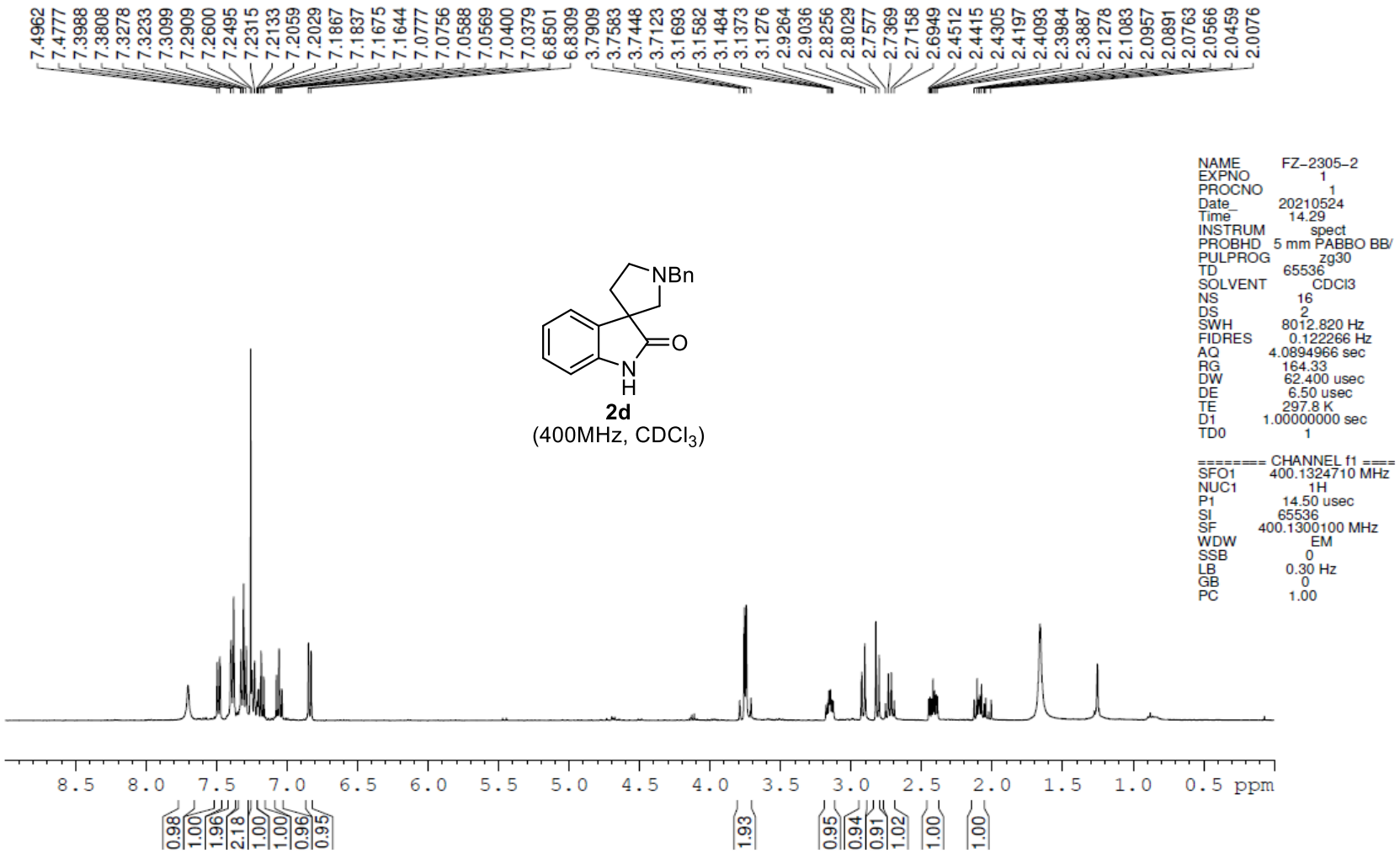

180.2351
154.5776
140.3363
133.1287
128.5276
123.0364
122.8308
110.2643
79.9960
77.4783
77.1600
76.8427
54.4670
53.4777
45.3634
35.6456
28.6001
NBoc
O
N
H
2a
(100MHz, CDCl3)
NAME EC-Boc
EXPNO 4
PROCNO 1
Date_ 20211211
Time 13.18
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 700
DS 2
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 se
RG 196.92
DW 20.800 us
DE 6.50 us
TE 296.4 K
D1 2.00000000 se
D11 0.03000000 se
TD0 1
======== CHANNEL f1 ======
SFO1 100.6228298 MH
NUC1 13C
P1 9.70 us
SI 32768
SF 100.6127584 MH
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 10 0 ppm

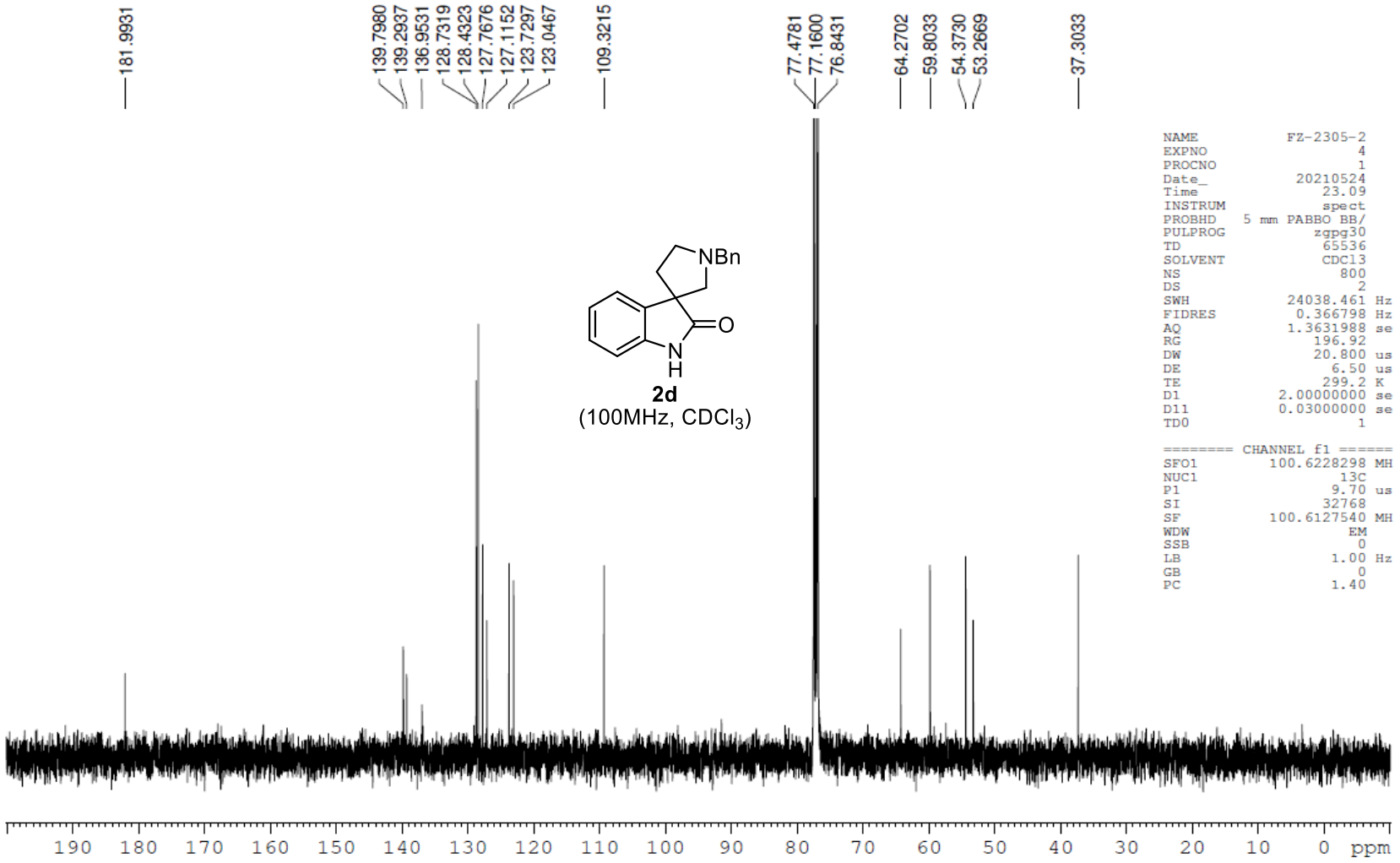

8.4480
8.3887
7.2600
7.2455
7.2263
7.1792
7.1615
7.0677
7.0489
7.0302
6.9423
6.9235
3.9420
3.9317
3.9125
3.9047
3.8954
3.8826
3.8660
3.8567
3.8492
3.8315
3.8066
3.7690
3.7401
3.7174
3.6921
3.6640
3.6432
3.6143
3.5869
2.4630
2.4425
2.4314
2.4226
2.4111
2.3912
2.1622
2.1451
2.1303
2.1165
2.0989
2.0868
2.0745
2.0557
1.7284
NCO2Me
O
N
H
2b
(400MHz, CDCl3)
NAME FZ-2307-1
EXPNO 2
PROCNO 1
Date_ 20210525
Time 23.12
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894966 sec
RG 126.97
DW 62.400 usec
DE 6.50 usec
TE 298.3 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ====
SFO1 400.1324710 MHz
NUC1 1H
P1 14.50 usec
SI 65536
SF 400.1300101 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5 ppm
1.01
0.99
1.03
1.02
1.00
2.10
4.11
1.29
1.04
1.09

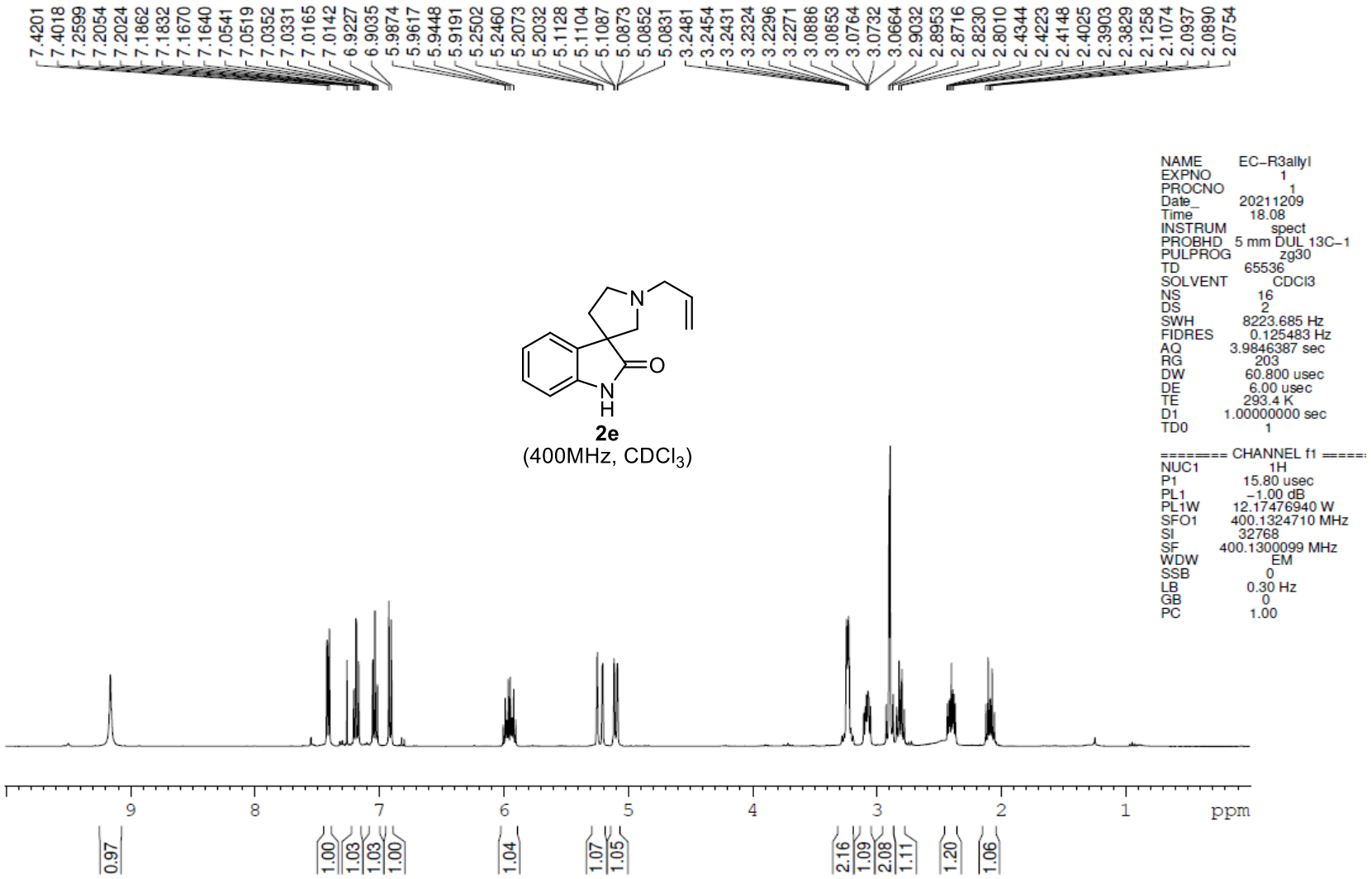

180.0639
179.7622
155.5724
140.2724
140.1501
132.7673
132.3894
128.6633
123.1603
122.8922
110.2121
77.4773
77.1600
76.8422
54.4637
54.0949
53.3848
52.7721
52.4147
45.8417
45.3095
36.5007
35.6394
NCO2Me
O
N
H
2b
(100MHz, CDCl3)
NAME EC-CO2Me
EXPNO 2
PROCNO 1
Date_ 20211216
Time 11.43
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 400
DS 2
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 se
RG 196.92
DW 20.800 us
DE 6.50 us
TE 297.2 K
D1 2.00000000 se
D11 0.03000000 se
TD0 1
======== CHANNEL f1 ======
SFO1 100.6228298 MH
NUC1 13C
P1 9.70 us
SI 32768
SF 100.6127565 MH
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 10 0 ppm

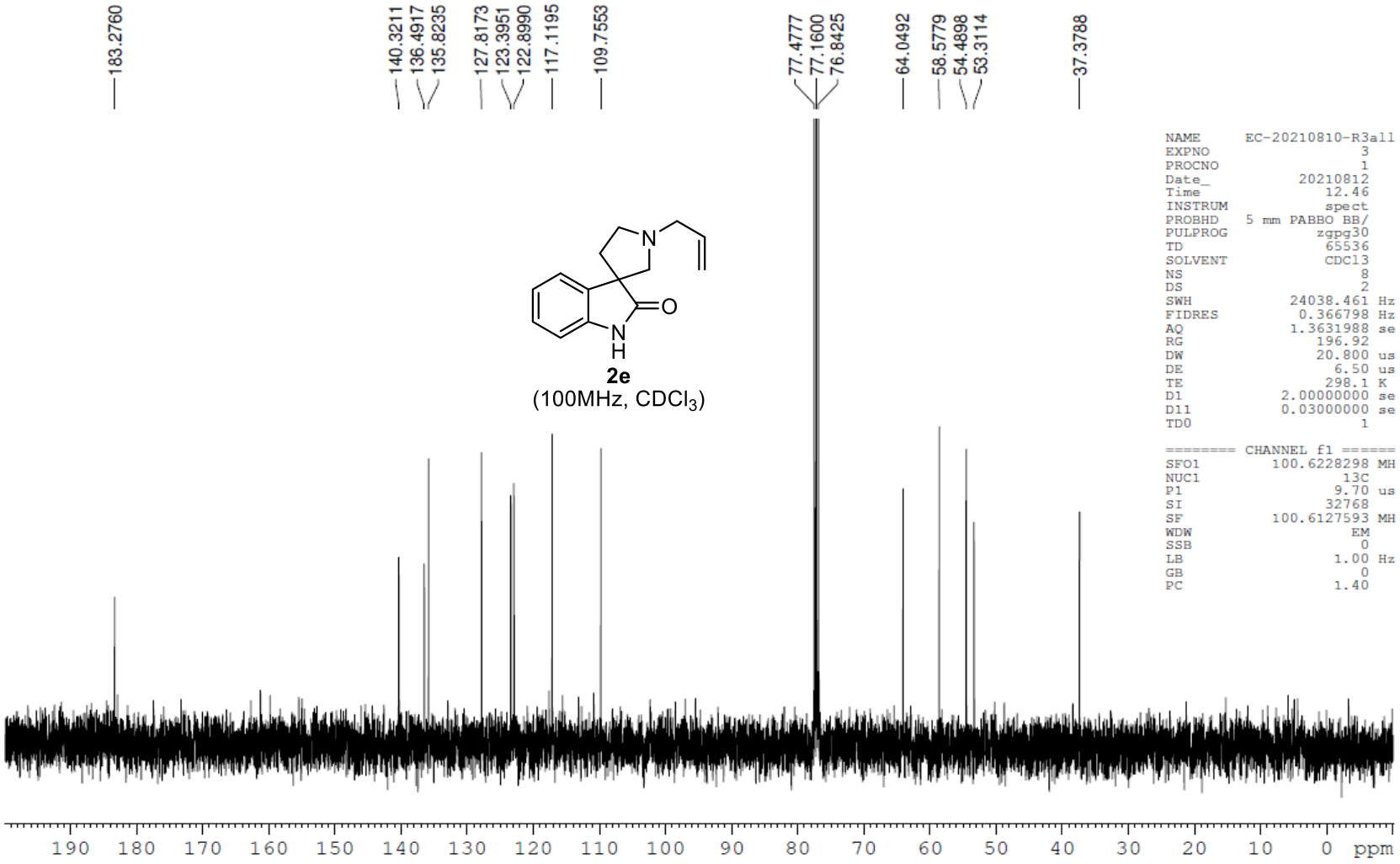

8.4196
8.2768
7.2871
7.2669
7.2600
7.2478
7.2276
7.1736
7.1568
7.1417
7.0805
7.0621
7.0502
7.0317
6.9653
6.9457
6.9357
6.9162
4.0327
4.0076
3.9917
3.9750
3.9550
3.8900
3.8657
3.8368
3.8177
3.7945
3.7688
3.7380
3.6345
3.6088
2.5045
2.4858
2.4707
2.4631
2.4551
2.4329
2.4108
2.2763
2.2576
2.2417
2.2269
2.2085
2.1724
2.0764
NAc
O
N
H
2c
(400MHz, CDCl3)
NAME FZ-2299-2
EXPNO 6
PROCNO 1
Date_ 20210527
Time 16.21
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894966 sec
RG 142.88
DW 62.400 usec
DE 6.50 usec
TE 297.4 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ====
SFO1 400.1324710 MHz
NUC1 1H
P1 14.50 usec
SI 65536
SF 400.1300101 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
1.03
1.71
1.00
1.02
1.00
1.07
2.08
1.17
1.04
4.23

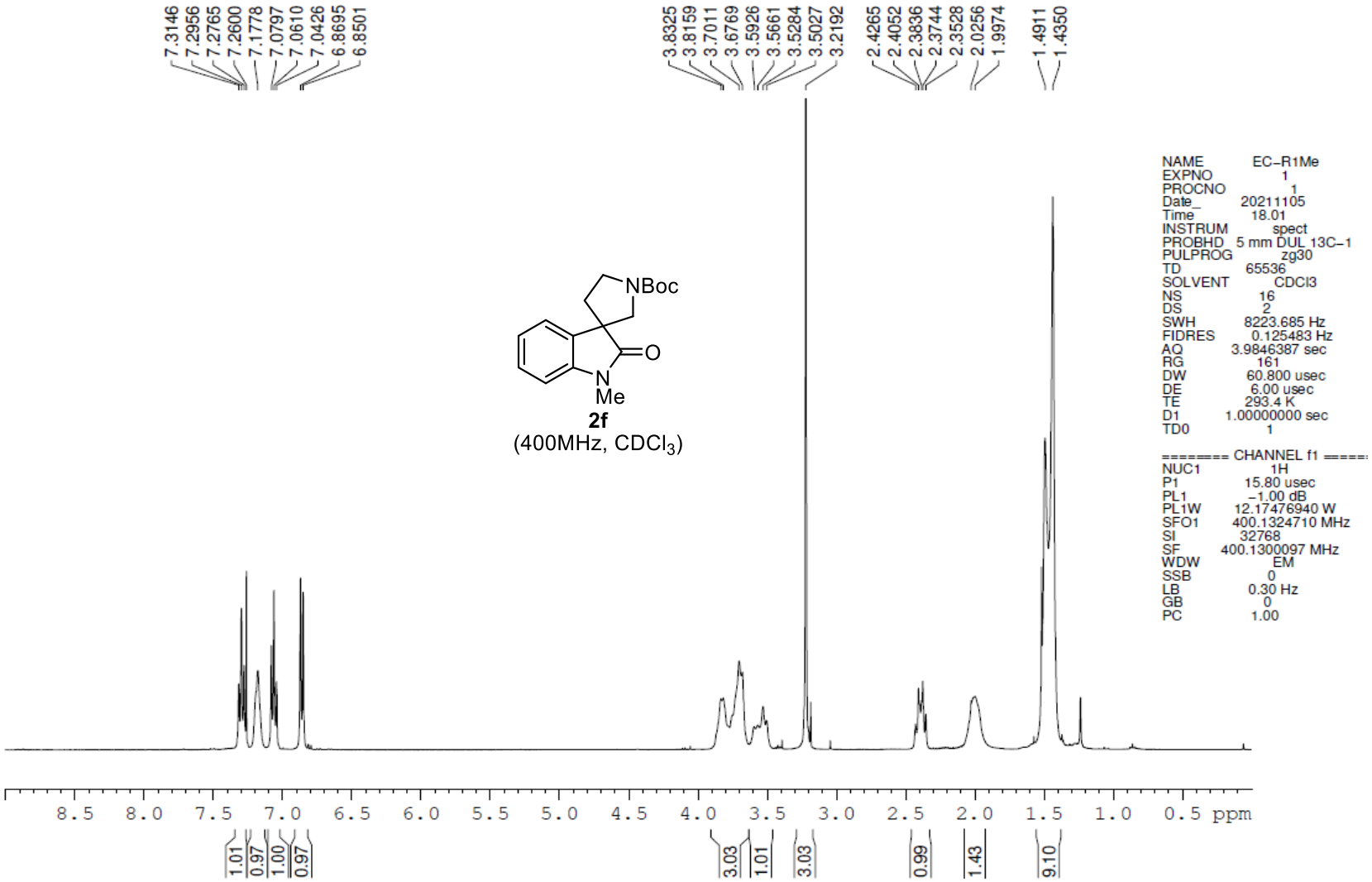

180.2532
179.1592
169.6704
169.5145
140.4913
140.1756
132.6478
131.7629
128.8637
128.7986
123.2868
123.0956
122.8057
110.3768
110.2491
77.4777
77.1600
76.8421
55.5696
53.6968
53.6586
51.9075
46.7559
45.2581
36.6160
35.2502
22.6976
22.5481
NAc
O
N
H
2c
(100MHz, CDCl3)
NAME FZ-2299-2
EXPNO 4
PROCNO 1
Date_ 20210518
Time 21.49
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 240
DS 2
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 se
RG 196.92
DW 20.800 us
DE 6.50 us
TE 298.7 K
D1 2.00000000 se
D11 0.03000000 se
TD0 1
======== CHANNEL f1 ======
SFO1 100.6228298 MH
NUC1 13C
P1 9.70 us
SI 32768
SF 100.6127560 MH
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 10 0 ppm

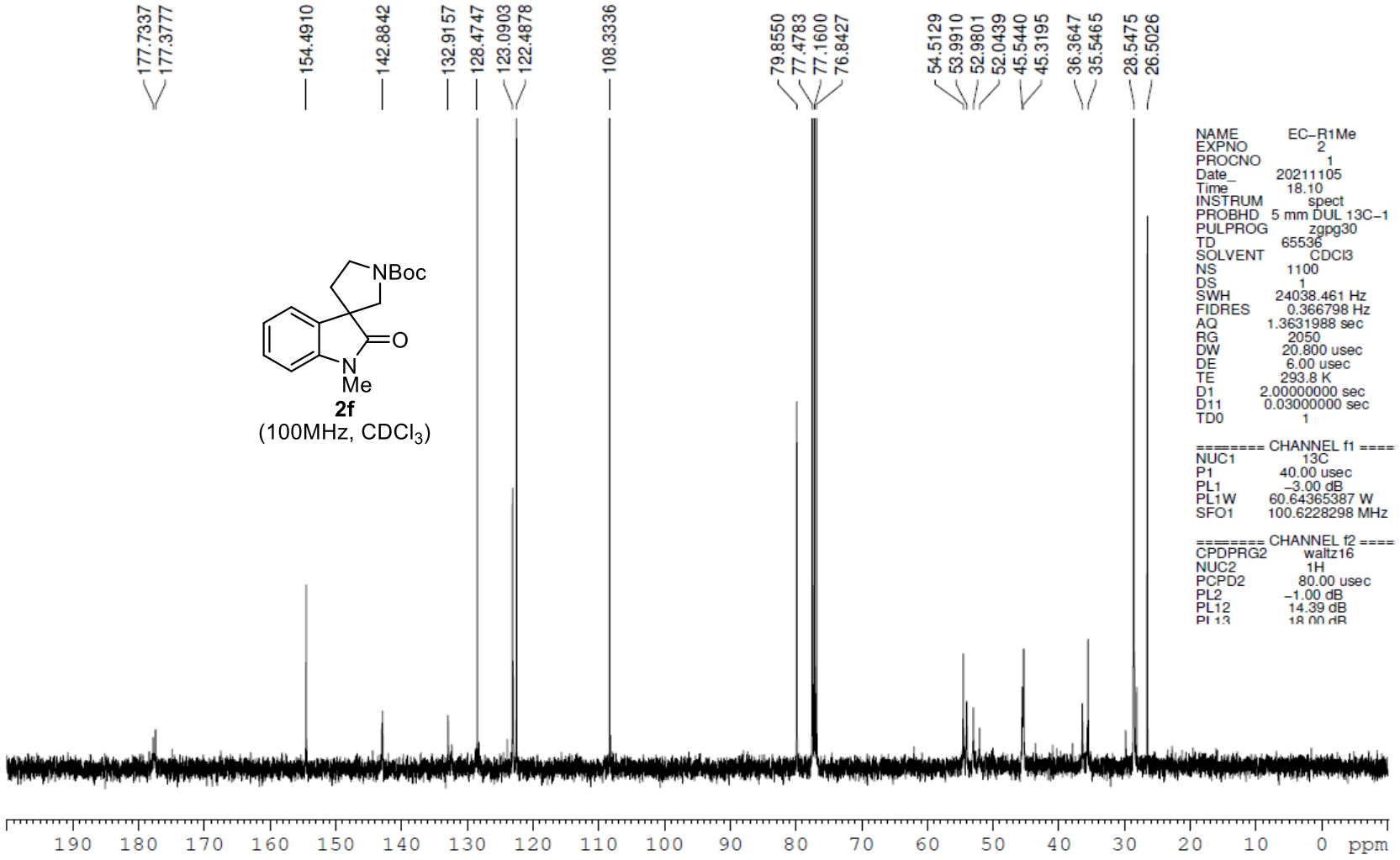

7.4962
7.4777
7.3988
7.3808
7.3278
7.3233
7.3099
7.2909
7.2600
7.2495
7.2315
7.2133
7.2059
7.2029
7.1867
7.1837
7.1675
7.1644
7.0777
7.0756
7.0588
7.0569
7.0400
7.0379
6.8501
6.8309
3.7909
3.7583
3.7448
3.7123
3.1693
3.1582
3.1484
3.1373
3.1276
2.9264
2.9036
2.8256
2.8029
2.7577
2.7369
2.7158
2.6949
2.4512
2.4415
2.4305
2.4197
2.4093
2.3984
2.3887
2.1278
2.1083
2.0957
2.0891
2.0763
2.0566
2.0459
2.0076
NBn
O
N
H
2d
(400MHz, CDCl3)
NAME FZ-2305-2
EXPNO 1
PROCNO 1
Date_ 20210524
Time 14.29
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894966 sec
RG 164.33
DW 62.400 usec
DE 6.50 usec
TE 297.8 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ====
SFO1 400.1324710 MHz
NUC1 1H
P1 14.50 usec
SI 65536
SF 400.1300100 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
0.98
1.00
1.96
2.18
1.00
1.00
0.96
0.95
1.93
0.95
0.94
0.91
1.02
1.00
1.00

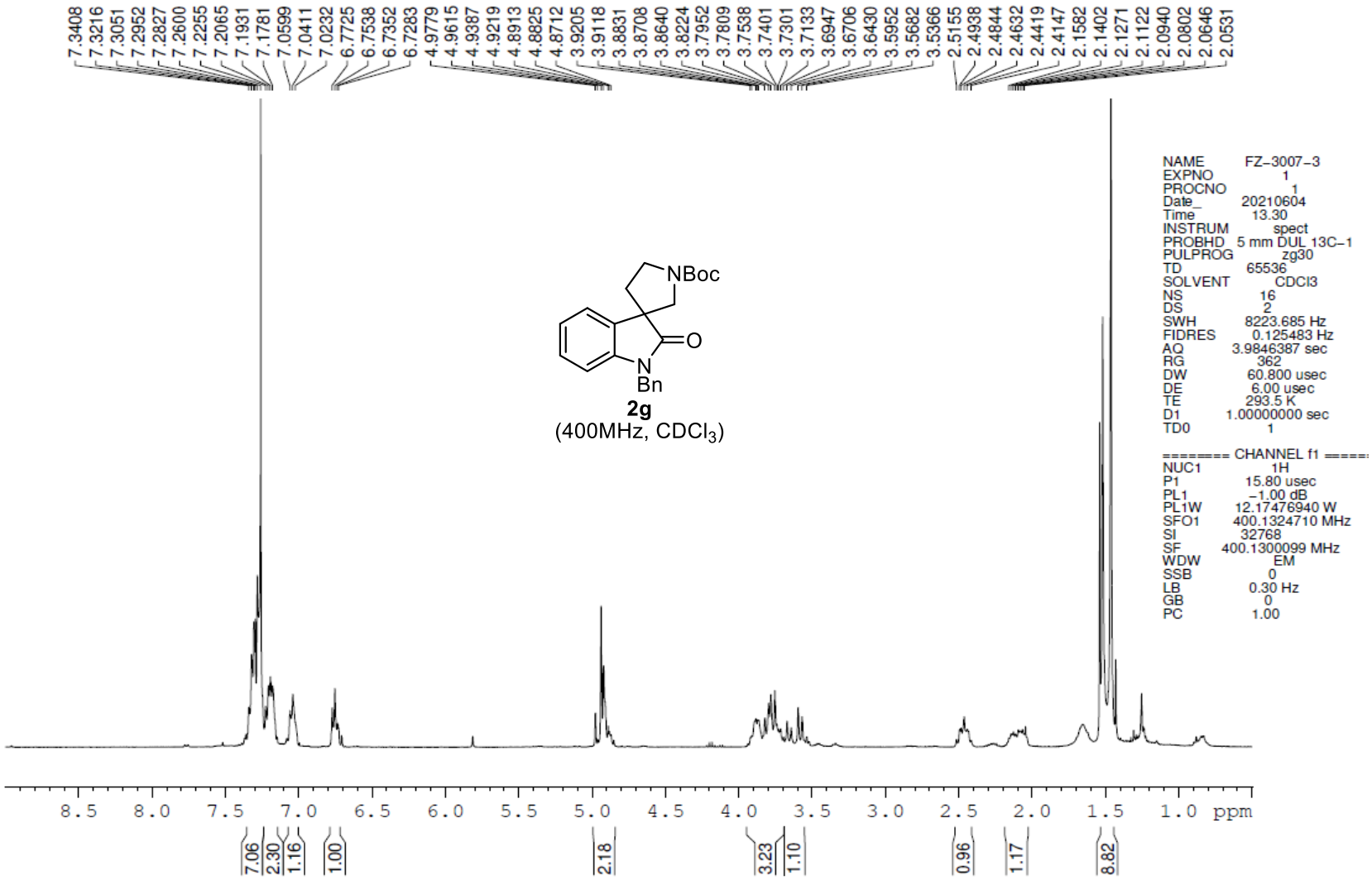

181.9931
139.7980
139.2937
136.9531
128.7319
128.4323
127.7676
127.1152
123.7297
123.0467
109.3215
77.4781
77.1600
76.8431
64.2702
59.8033
54.3730
53.2669
37.3033
NBn
O
N
H
2d
(100MHz, CDCl3)
NAME FZ-2305-2
EXPNO 4
PROCNO 1
Date_ 20210524
Time 23.09
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 800
DS 2
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 se
RG 196.92
DW 20.800 us
DE 6.50 us
TE 299.2 K
D1 2.00000000 se
D11 0.03000000 se
TD0 1
======== CHANNEL f1 ======
SFO1 100.6228298 MH
NUC1 13C
P1 9.70 us
SI 32768
SF 100.6127540 MH
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 10 0 ppm

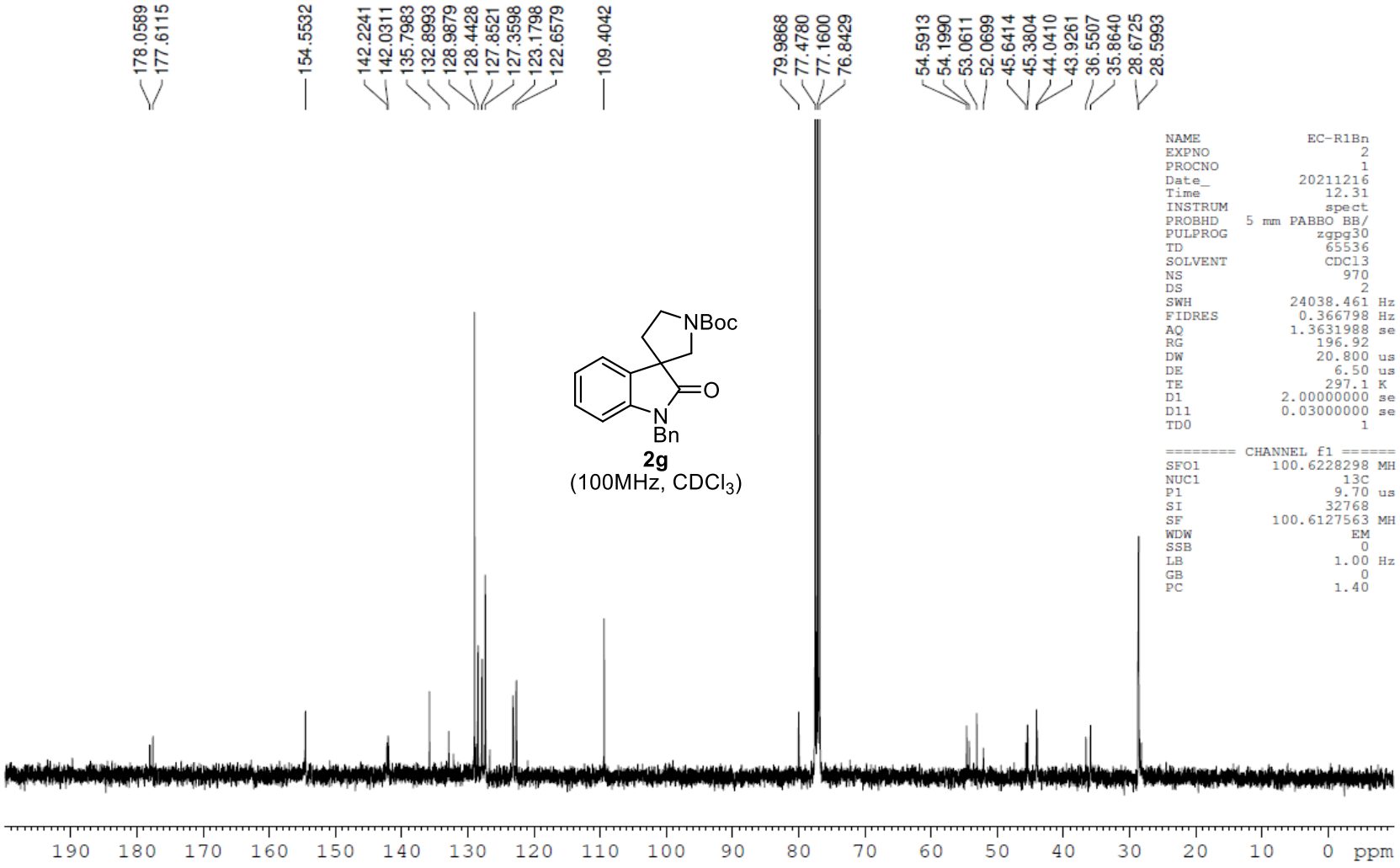

7.4201
7.4018
7.2599
7.2054
7.2024
7.1862
7.1832
7.1670
7.1640
7.0541
7.0519
7.0352
7.0331
7.0165
7.0142
6.9227
6.9035
5.9874
5.9617
5.9448
5.9191
5.2502
5.2460
5.2073
5.2032
5.1128
5.1104
5.1087
5.0873
5.0852
5.0831
3.2481
3.2454
3.2431
3.2324
3.2296
3.2271
3.0886
3.0853
3.0764
3.0732
3.0664
2.9032
2.8953
2.8716
2.8230
2.8010
2.4344
2.4223
2.4148
2.4025
2.3903
2.3829
2.1258
2.1074
2.0937
2.0890
2.0754
NAME EC-R3allyl
EXPNO 1
PROCNO 1
Date_ 20211209
Time 18.08
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 203
DW 60.800 usec
DE 6.00 usec
TE 293.4 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ======
NUC1 1H
P1 15.80 usec
PL1 -1.00 dB
PL1W 12.17476940 W
SFO1 400.1324710 MHz
SI 32768
SF 400.1300099 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
O
N
H
2e
(400MHz, CDCl3)
9
8
7
6
5
4
3
2
1
ppm
0.97
1.00
1.03
1.03
1.00
1.04
1.07
1.05
2.16
1.09
2.08
1.11
1.20
1.06

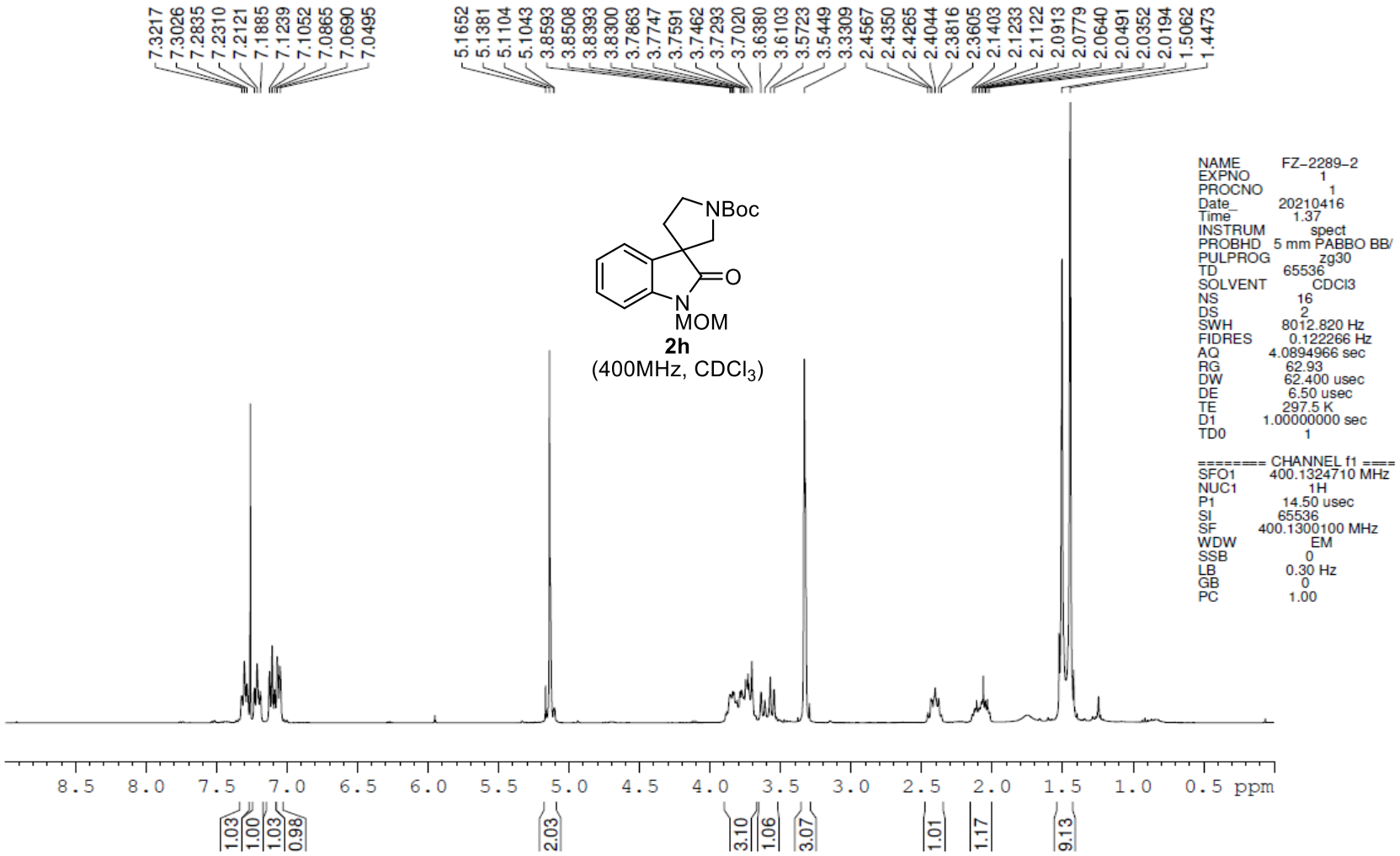

183.2760
140.3211
136.4917
135.8235
127.8173
123.3951
122.8990
117.1195
109.7553
77.4777
77.1600
76.8425
64.0492
58.5779
54.4898
53.3114
37.3788
N
O
N
H
2e
(100MHz, CDCl3)
NAME EC-20210810-R3all
EXPNO 3
PROCNO 1
Date_ 20210812
Time 12.46
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 se
RG 196.92
DW 20.800 us
DE 6.50 us
TE 298.1 K
D1 2.00000000 se
D11 0.03000000 se
TD0 1
======== CHANNEL f1 ======
SFO1 100.6228298 MH
NUC1 13C
P1 9.70 us
SI 32768
SF 100.6127593 MH
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 10 0 ppm

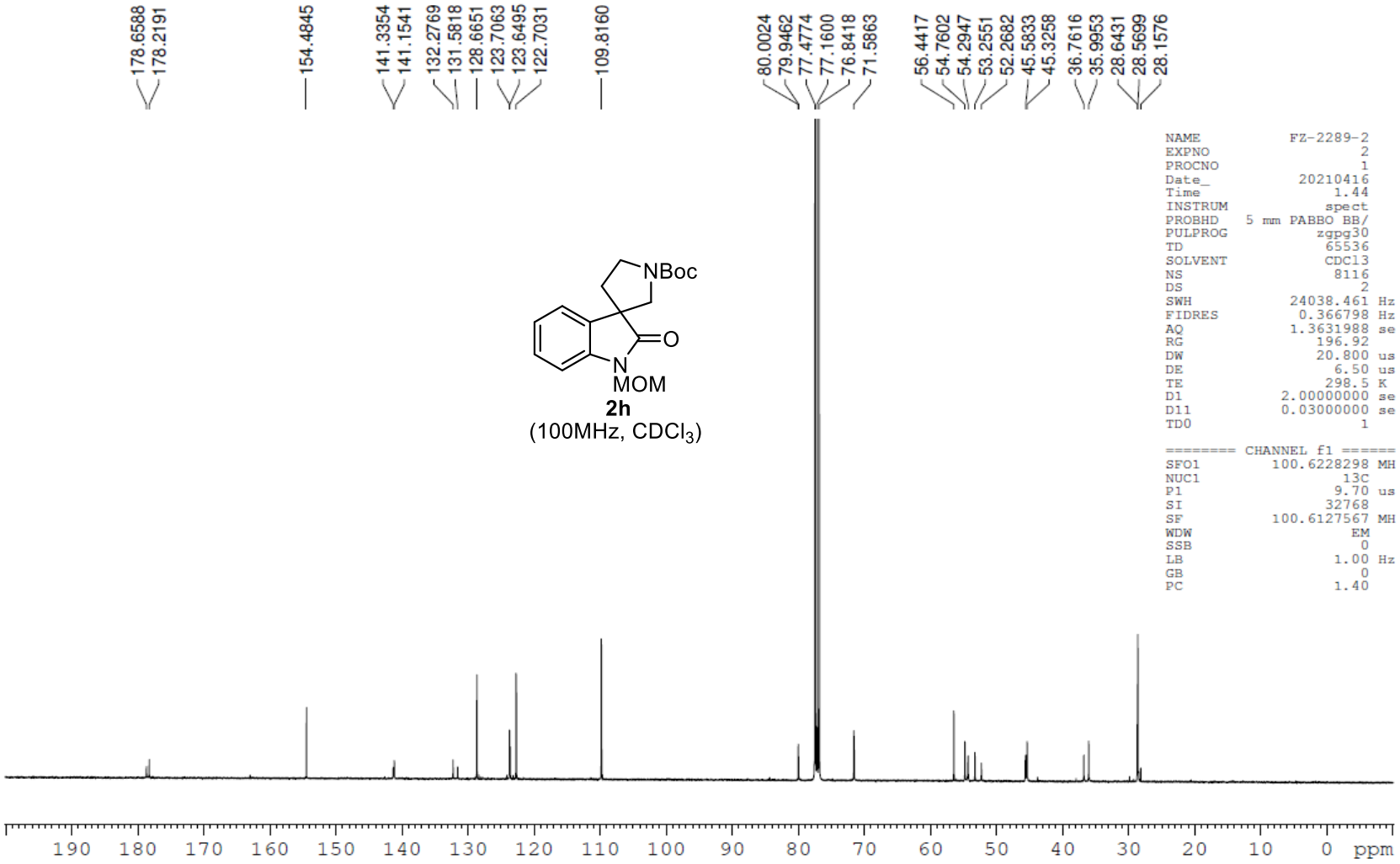

7.3146
7.2956
7.2765
7.2600
7.1778
7.0797
7.0610
7.0426
6.8695
6.8501
3.8325
3.8159
3.7011
3.6769
3.5926
3.5661
3.5284
3.5027
3.2192
2.4265
2.4052
2.3836
2.3744
2.3528
2.0256
1.9974
1.4911
1.4350
NBoc
O
N
Me
2f
(400MHz, CDCl3)
NAME EC-R1Me
EXPNO 1
PROCNO 1
Date_ 20211105
Time 18.01
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 161
DW 60.800 usec
DE 6.00 usec
TE 293.4 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ======
NUC1 1H
P1 15.80 usec
PL1 -1.00 dB
PL1W 12.17476940 W
SFO1 400.1324710 MHz
SI 32768
SF 400.1300097 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
1.01
0.97
1.00
0.97
3.03
1.01
3.03
0.99
1.43
9.10

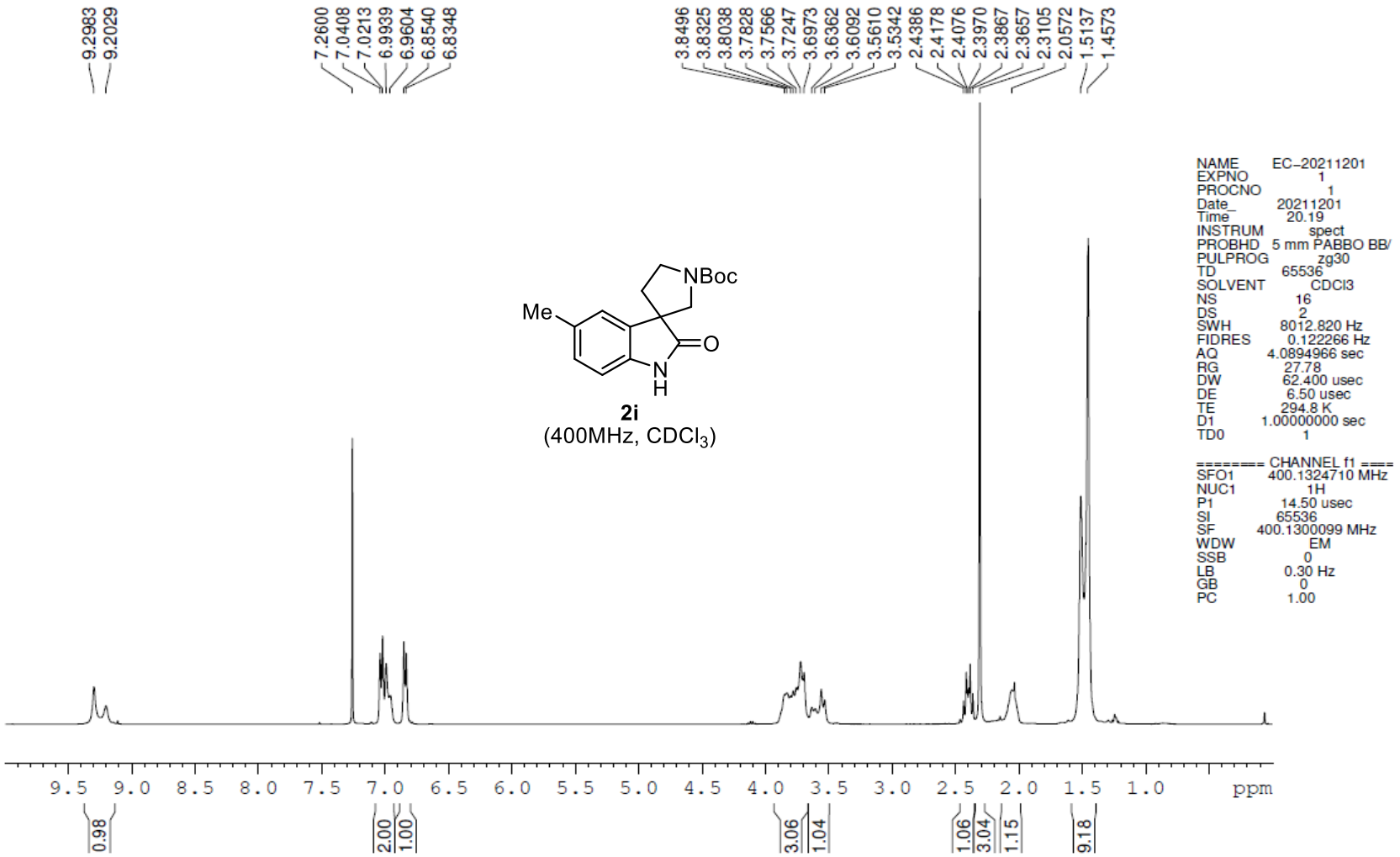

177.7337
177.3777
154.4910
142.8842
132.9157
128.4747
123.0903
122.4878
108.3336
79.8550
77.4783
77.1600
76.8427
54.5129
53.9910
52.9801
52.0439
45.5440
45.3195
36.3647
35.5465
28.5475
26.5026
NBoc
O
N
Me
2f
(100MHz, CDCl3)
NAME EC-R1Me
EXPNO 2
PROCNO 1
Date_ 20211105
Time 18.10
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1100
DS 1
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 2050
DW 20.800 usec
DE 6.00 usec
TE 293.8 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ====
NUC1 13C
P1 40.00 usec
PL1 -3.00 dB
PL1W 60.64365387 W
SFO1 100.6228298 MHz
======== CHANNEL f2 ====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -1.00 dB
PL12 14.39 dB
PL13 18.00 dB
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

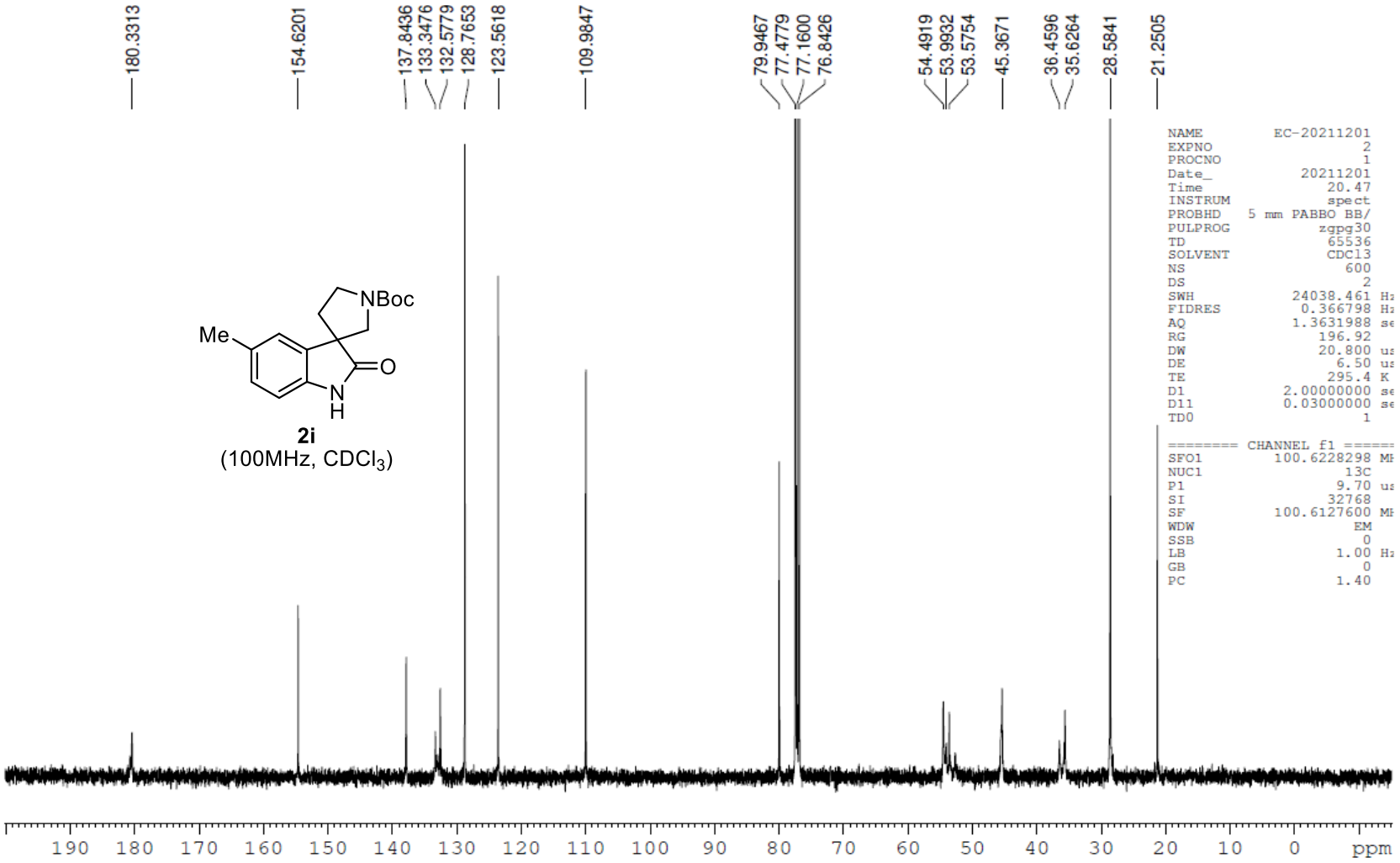

7.3408
7.3216
7.3051
7.2952
7.2827
7.2600
7.2255
7.2065
7.1931
7.1781
7.0599
7.0411
7.0232
6.7725
6.7538
6.7352
6.7283
4.9779
4.9615
4.9387
4.9219
4.8913
4.8825
4.8712
3.9205
3.9118
3.8831
3.8708
3.8640
3.8224
3.7952
3.7809
3.7538
3.7401
3.7301
3.7133
3.6947
3.6706
3.6430
3.5952
3.5682
3.5366
2.5155
2.4938
2.4844
2.4632
2.4419
2.4147
2.1582
2.1402
2.1271
2.1122
2.0940
2.0802
2.0646
2.0531
NBoc
O
N
Bn
2g
(400MHz, CDCl3)
NAME FZ-3007-3
EXPNO 1
PROCNO 1
Date_ 20210604
Time 13.30
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 362
DW 60.800 usec
DE 6.00 usec
TE 293.5 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ======
NUC1 1H
P1 15.80 usec
PL1 -1.00 dB
PL1W 12.17476940 W
SFO1 400.1324710 MHz
SI 32768
SF 400.1300099 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
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
7.06
2.30
1.16
1.00
2.18
3.23
1.10
0.96
1.17
8.82

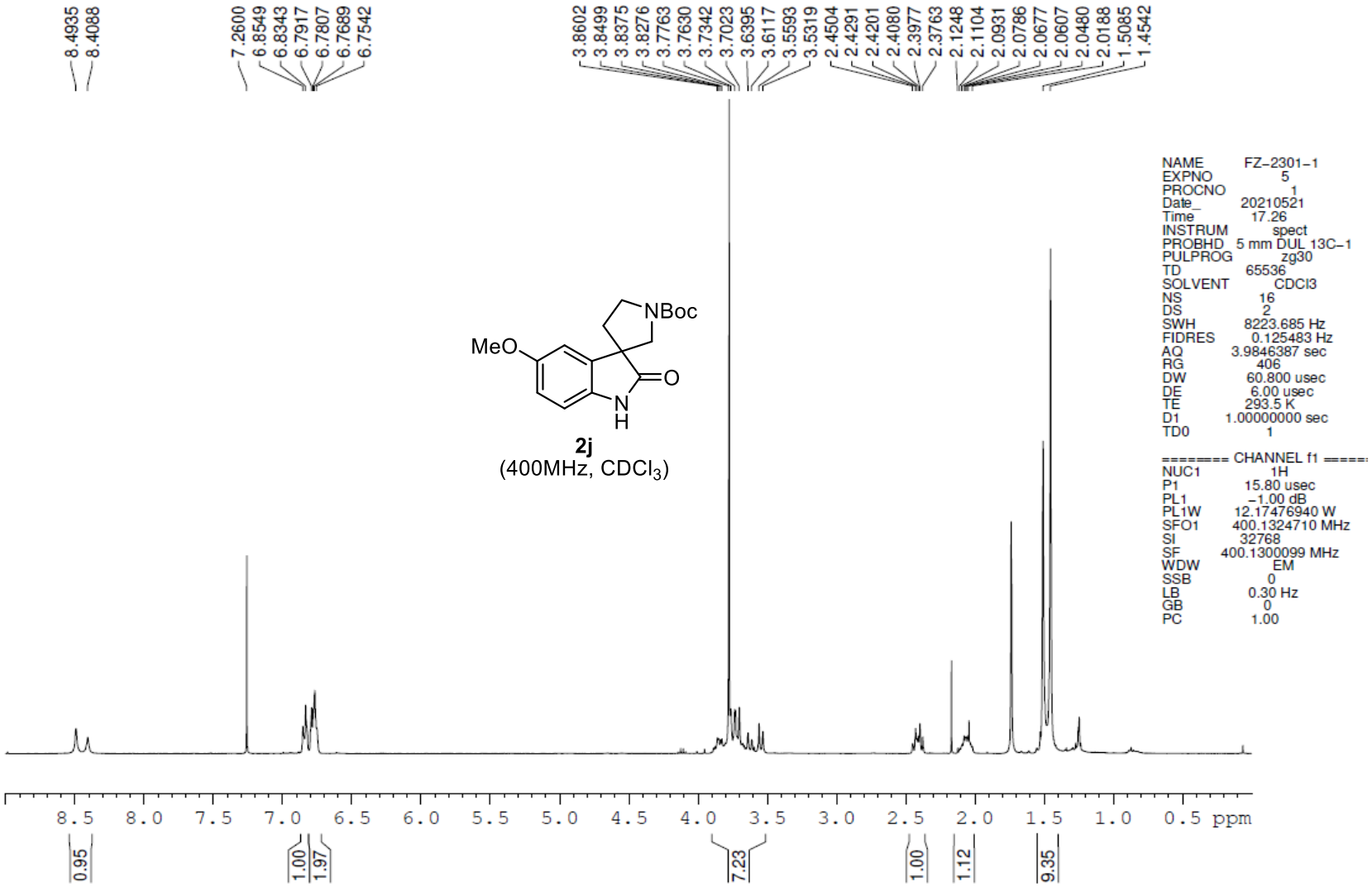

178.0589
177.6115
154.5532
142.2241
142.0311
135.7983
132.8993
128.9879
128.4428
127.8521
127.3598
123.1798
122.6579
109.4042
79.9868
77.4780
77.1600
76.8429
54.5913
54.1990
53.0611
52.0699
45.6414
45.3804
44.0410
43.9261
36.5507
35.8640
28.6725
28.5993
NBoc
O
N
Bn
2g
(100MHz, CDCl3)
NAME EC-R1Bn
EXPNO 2
PROCNO 1
Date_ 20211216
Time 12.31
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 970
DS 2
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 se
RG 196.92
DW 20.800 us
DE 6.50 us
TE 297.1 K
D1 2.00000000 se
D11 0.03000000 se
TD0 1
======== CHANNEL f1 ======
SFO1 100.6228298 MH
NUC1 13C
P1 9.70 us
SI 32768
SF 100.6127563 MH
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 10 0 ppm

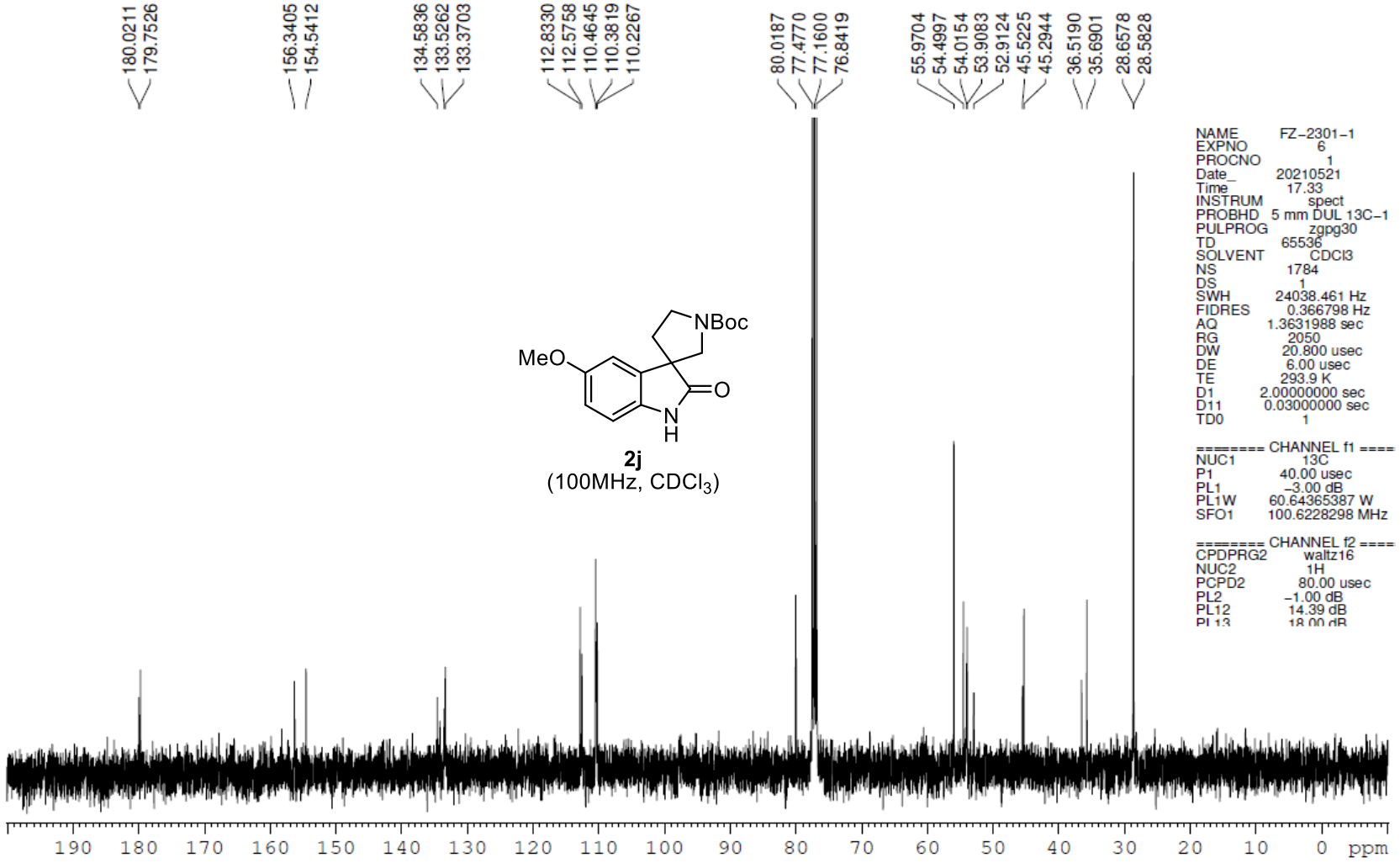

7.3217
7.3026
7.2835
7.2310
7.2121
7.1885
7.1239
7.1052
7.0865
7.0690
7.0495
5.1652
5.1381
5.1104
5.1043
3.8593
3.8508
3.8393
3.8300
3.7863
3.7747
3.7591
3.7462
3.7293
3.7020
3.6380
3.6103
3.5723
3.5449
3.3309
2.4567
2.4350
2.4265
2.4044
2.3816
2.3605
2.1403
2.1233
2.1122
2.0913
2.0779
2.0640
2.0491
2.0352
2.0194
1.5062
1.4473
NBoc
O
N
MOM
2h
(400MHz, CDCl3)
NAME FZ-2289-2
EXPNO 1
PROCNO 1
Date_ 20210416
Time 1.37
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894966 sec
RG 62.93
DW 62.400 usec
DE 6.50 usec
TE 297.5 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ====
SFO1 400.1324710 MHz
NUC1 1H
P1 14.50 usec
SI 65536
SF 400.1300100 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
1.03
1.00
1.03
0.98
2.03
3.10
1.06
3.07
1.01
1.17
9.13

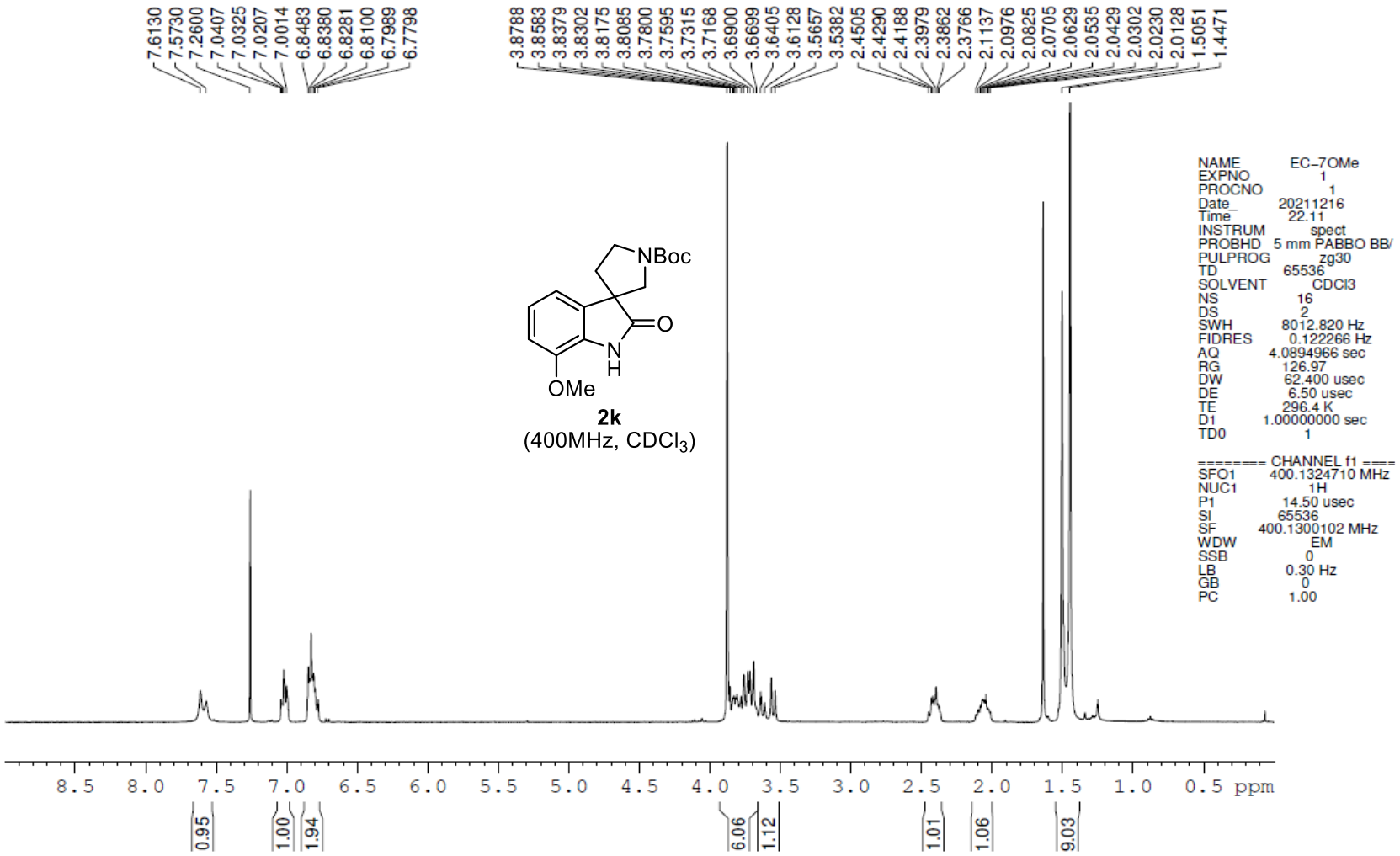

178.6588
178.2191
154.4845
141.3354
141.1541
132.2769
131.5818
128.6651
123.7063
123.6495
122.7031
109.8160
80.0024
79.9462
77.4774
77.1600
76.8418
71.5863
56.4417
54.7602
54.2947
53.2551
52.2682
45.5833
45.3258
36.7616
35.9953
28.6431
28.5699
28.1576
NBoc
O
N
MOM
2h
(100MHz, CDCl3)
NAME FZ-2289-2
EXPNO 2
PROCNO 1
Date_ 20210416
Time 1.44
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 8116
DS 2
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 se
RG 196.92
DW 20.800 us
DE 6.50 us
TE 298.5 K
D1 2.00000000 se
D11 0.03000000 se
TD0 1
======== CHANNEL f1 ======
SFO1 100.6228298 MH
NUC1 13C
P1 9.70 us
SI 32768
SF 100.6127567 MH
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 10 0 ppm

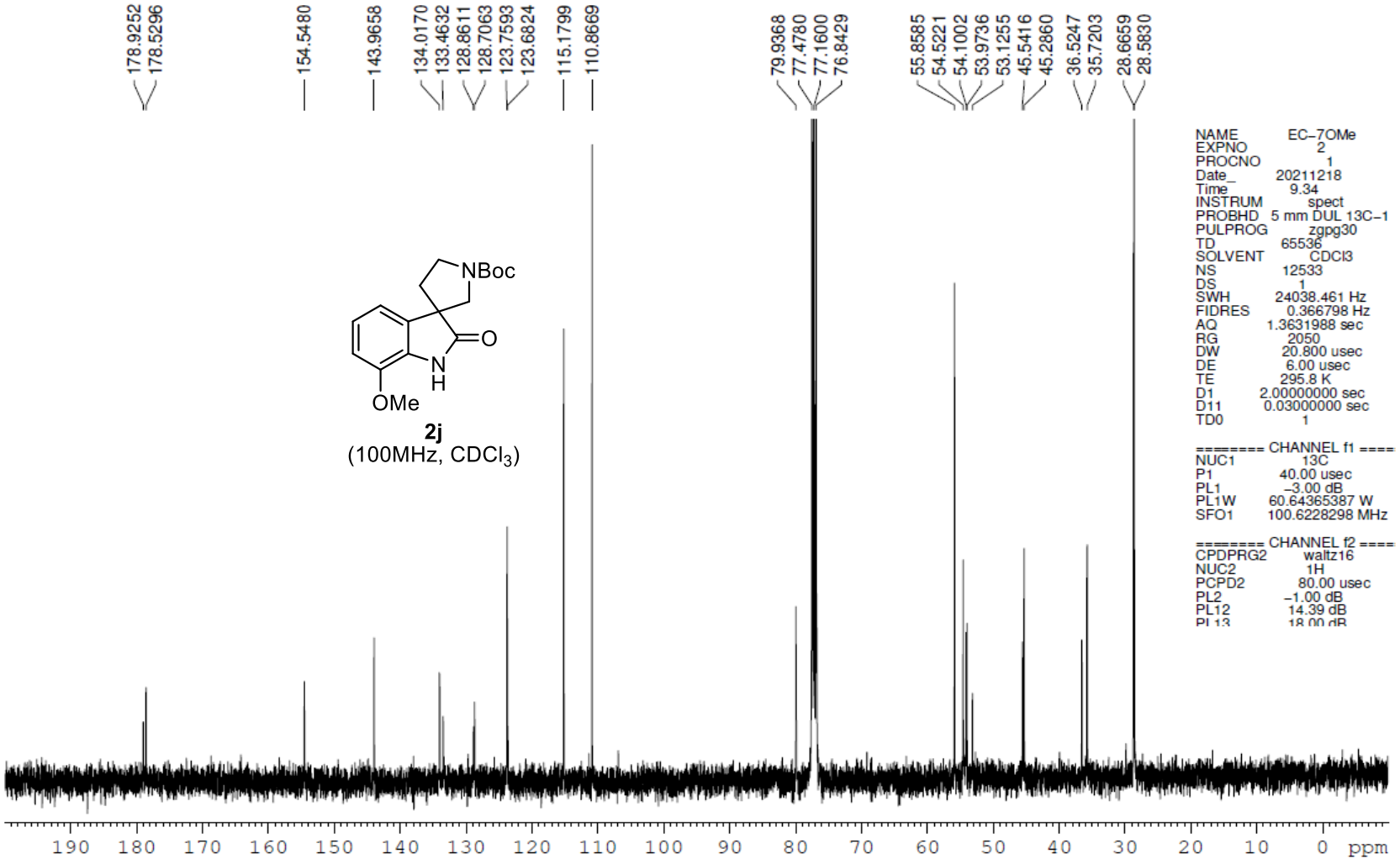

9.2983
9.2029
7.2600
7.0408
7.0213
6.9939
6.9604
6.8540
6.8348
3.8496
3.8325
3.8038
3.7828
3.7566
3.7247
3.6973
3.6362
3.6092
3.5610
3.5342
2.4386
2.4178
2.4076
2.3970
2.3867
2.3657
2.3105
2.0572
1.5137
1.4573
NBoc
Me
O
N
H
2i
(400MHz, CDCl3)
NAME EC-20211201
EXPNO 1
PROCNO 1
Date_ 20211201
Time 20.19
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894966 sec
RG 27.78
DW 62.400 usec
DE 6.50 usec
TE 294.8 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ====
SFO1 400.1324710 MHz
NUC1 1H
P1 14.50 usec
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
0.98
2.00
1.00
3.06
1.04
1.06
3.04
1.15
9.18

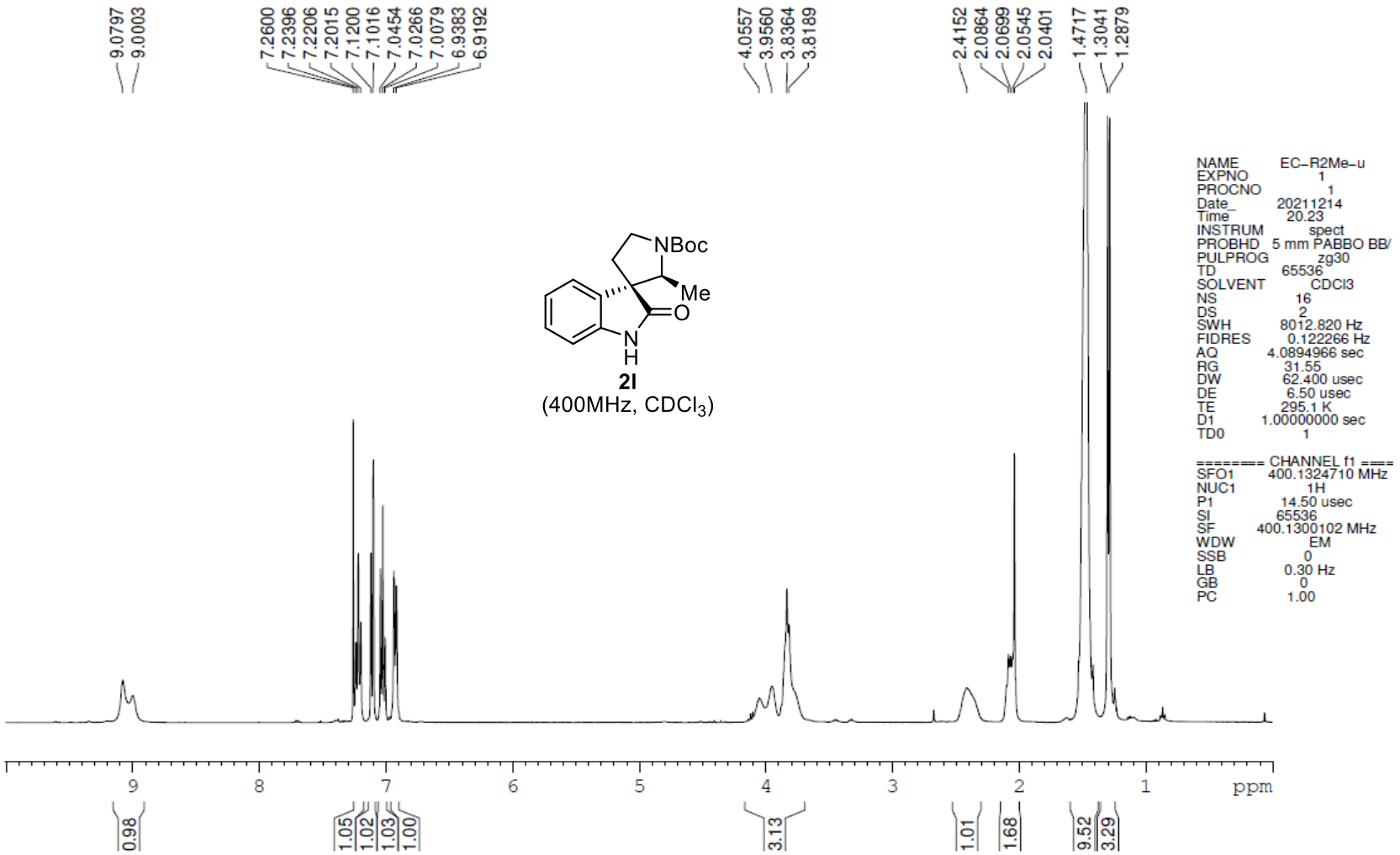

180.3313
154.6201
137.8436
133.3476
132.5779
128.7653
123.5618
109.9847
79.9467
77.4779
77.1600
76.8426
54.4919
53.9932
53.5754
45.3671
36.4596
35.6264
28.5841
21.2505
NBoc
Me
O
N
H
2i
(100MHz, CDCl3)
NAME EC-20211201
EXPNO 2
PROCNO 1
Date_ 20211201
Time 20.47
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 600
DS 2
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 se
RG 196.92
DW 20.800 us
DE 6.50 us
TE 295.4 K
D1 2.00000000 se
D11 0.03000000 se
TD0 1
======== CHANNEL f1 ======
SFO1 100.6228298 MH
NUC1 13C
P1 9.70 us
SI 32768
SF 100.6127600 MH
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 10 0 ppm

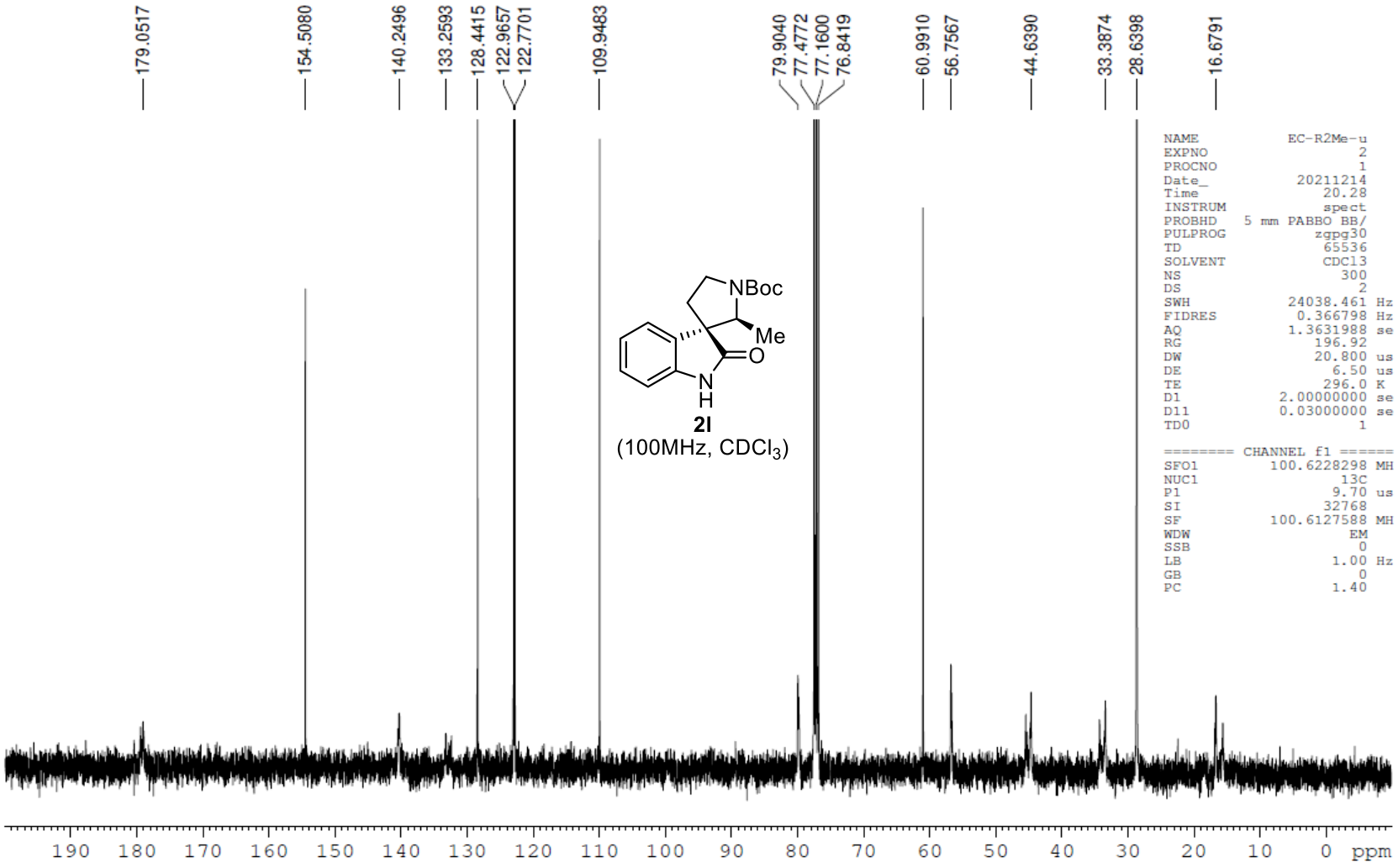

8.4935
8.4088
7.2600
6.8549
6.8343
6.7917
6.7807
6.7689
6.7542
3.8602
3.8499
3.8375
3.8276
3.7763
3.7630
3.7342
3.7023
3.6395
3.6117
3.5593
3.5319
2.4504
2.4291
2.4201
2.4080
2.3977
2.3763
2.1248
2.1104
2.0931
2.0786
2.0677
2.0607
2.0480
2.0188
1.5085
1.4542
NBoc
MeO
O
N
H
2j
(400MHz, CDCl3)
NAME FZ-2301-1
EXPNO 5
PROCNO 1
Date_ 20210521
Time 17.26
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 406
DW 60.800 usec
DE 6.00 usec
TE 293.5 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ======
NUC1 1H
P1 15.80 usec
PL1 -1.00 dB
PL1W 12.17476940 W
SFO1 400.1324710 MHz
SI 32768
SF 400.1300099 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
0.95
1.00
1.97
7.23
1.00
1.12
9.35

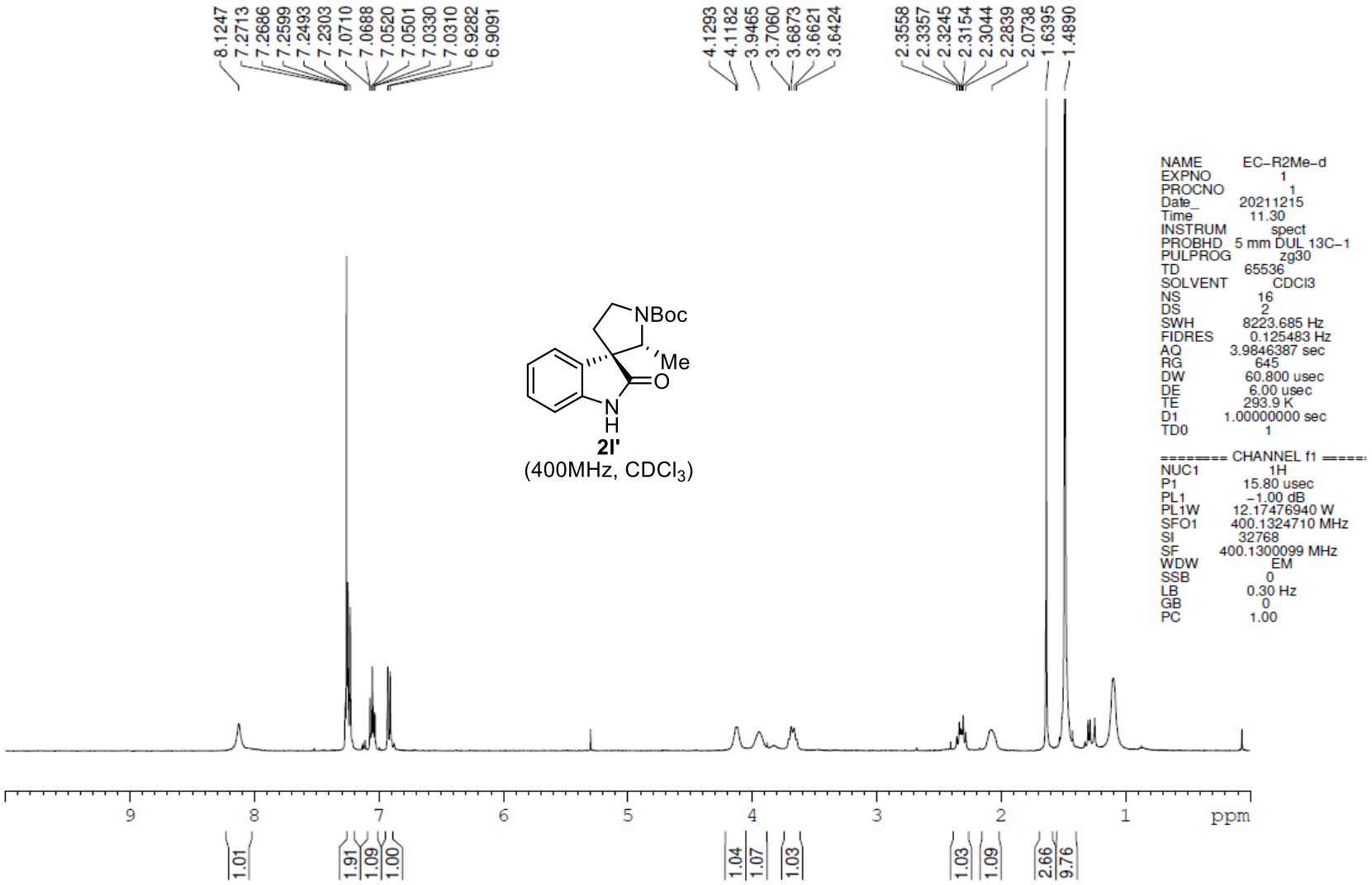

180.0211
179.7526
156.3405
154.5412
134.5836
133.5262
133.3703
112.8330
112.5758
110.4645
110.3819
110.2267
80.0187
77.4770
77.1600
76.8419
55.9704
54.4997
54.0154
53.9083
52.9124
45.5225
45.2944
36.5190
35.6901
28.6578
28.5828
NBoc
MeO
O
N
H
2j
(100MHz, CDCl3)
NAME FZ-2301-1
EXPNO 6
PROCNO 1
Date_ 20210521
Time 17.33
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1784
DS 1
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 2050
DW 20.800 usec
DE 6.00 usec
TE 293.9 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ====
NUC1 13C
P1 40.00 usec
PL1 -3.00 dB
PL1W 60.64365387 W
SFO1 100.6228298 MHz
======== CHANNEL f2 ====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -1.00 dB
PL12 14.39 dB
PL13 18.00 dB
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

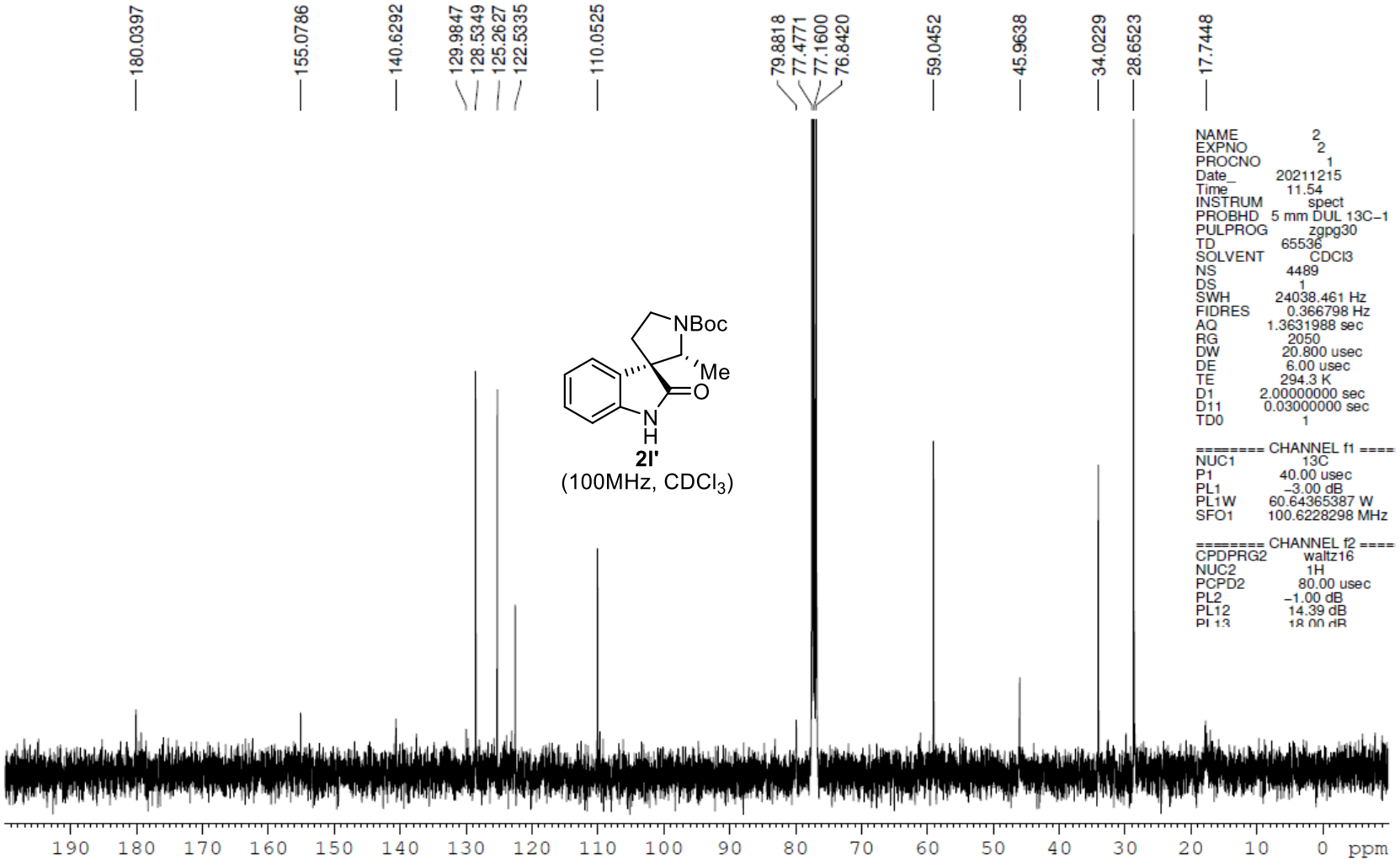

7.6130
7.5730
7.2600
7.0407
7.0325
7.0207
7.0014
6.8483
6.8380
6.8281
6.8100
6.7989
6.7798
3.8788
3.8583
3.8379
3.8302
3.8175
3.8085
3.7800
3.7595
3.7315
3.7168
3.6900
3.6699
3.6405
3.6128
3.5657
3.5382
2.4505
2.4290
2.4188
2.3979
2.3862
2.3766
2.1137
2.0976
2.0825
2.0705
2.0629
2.0535
2.0429
2.0302
2.0230
2.0128
1.5051
1.4471
NBoc
O
N
H
OMe
2k
(400MHz, CDCl3)
NAME EC-7OMe
EXPNO 1
PROCNO 1
Date_ 20211216
Time 22.11
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894966 sec
RG 126.97
DW 62.400 usec
DE 6.50 usec
TE 296.4 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ====
SFO1 400.1324710 MHz
NUC1 1H
P1 14.50 usec
SI 65536
SF 400.1300102 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
0.95
1.00
1.94
6.06
1.12
1.01
1.06
9.03

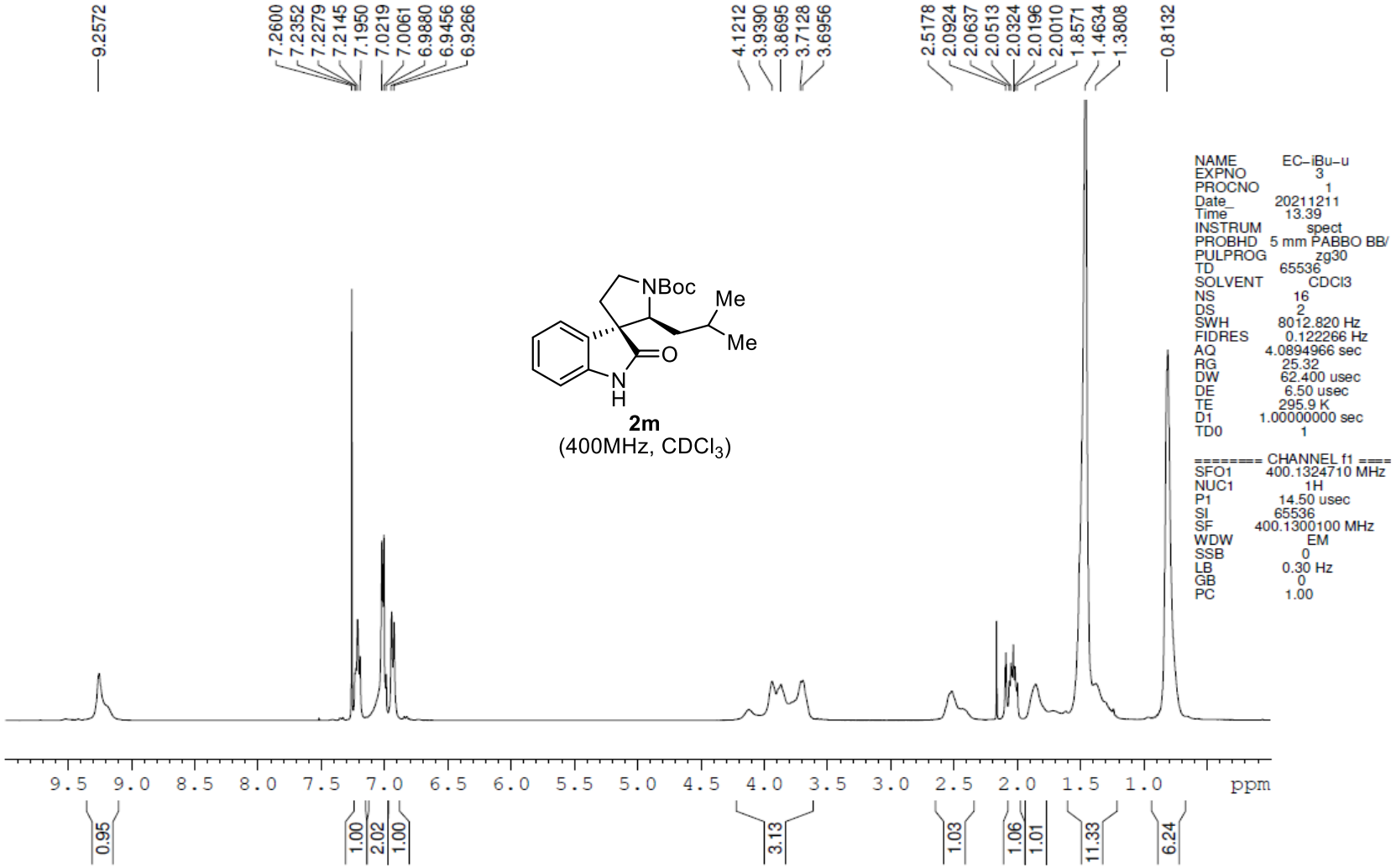

178.9252
178.5296
154.5480
143.9658
134.0170
133.4632
128.8611
128.7063
123.7593
123.6824
115.1799
110.8669
79.9368
77.4780
77.1600
76.8429
55.8585
54.5221
54.1002
53.9736
53.1255
45.5416
45.2860
36.5247
35.7203
28.6659
28.5830
NBoc
O
N
H
OMe
2j
(100MHz, CDCl3)
NAME EC-7OMe
EXPNO 2
PROCNO 1
Date_ 20211218
Time 9.34
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 12533
DS 1
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 2050
DW 20.800 usec
DE 6.00 usec
TE 295.8 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ====
NUC1 13C
P1 40.00 usec
PL1 -3.00 dB
PL1W 60.64365387 W
SFO1 100.6228298 MHz
======== CHANNEL f2 ====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -1.00 dB
PL12 14.39 dB
PL13 18.00 dB
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

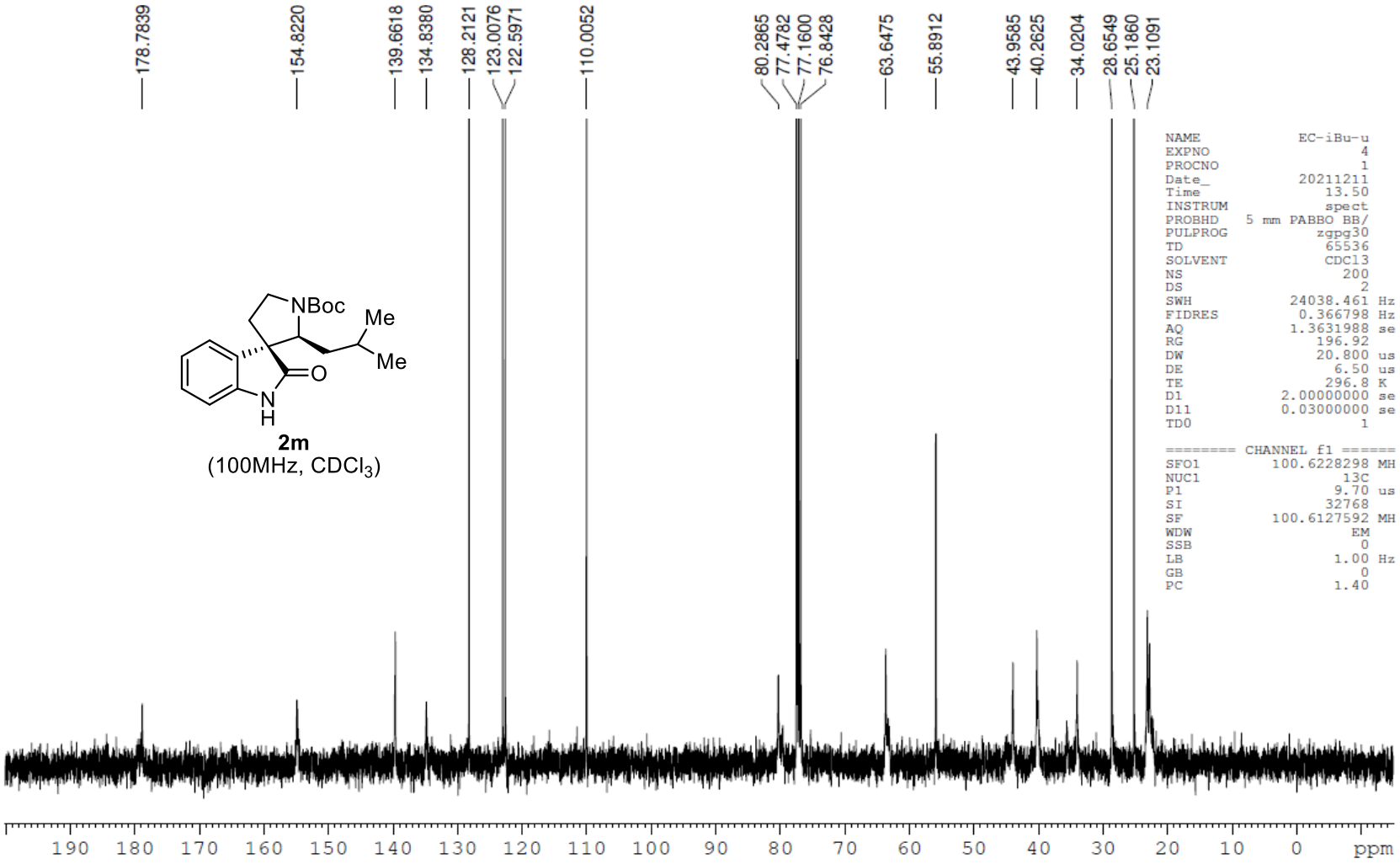

9.0797
9.0003
7.2600
7.2396
7.2206
7.2015
7.1200
7.1016
7.0454
7.0266
7.0079
6.9383
6.9192
4.0557
3.9560
3.8364
3.8189
2.4152
2.0864
2.0699
2.0545
2.0401
1.4717
1.3041
1.2879
NBoc
Me
O
N
H
2l
(400MHz, CDCl3)
NAME EC-R2Me-u
EXPNO 1
PROCNO 1
Date_ 20211214
Time 20.23
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894966 sec
RG 31.55
DW 62.400 usec
DE 6.50 usec
TE 295.1 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ===
SFO1 400.1324710 MHz
NUC1 1H
P1 14.50 usec
SI 65536
SF 400.1300102 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
1.05
1.02
1.03
1.00
3.13
1.01
1.68
9.52
3.29

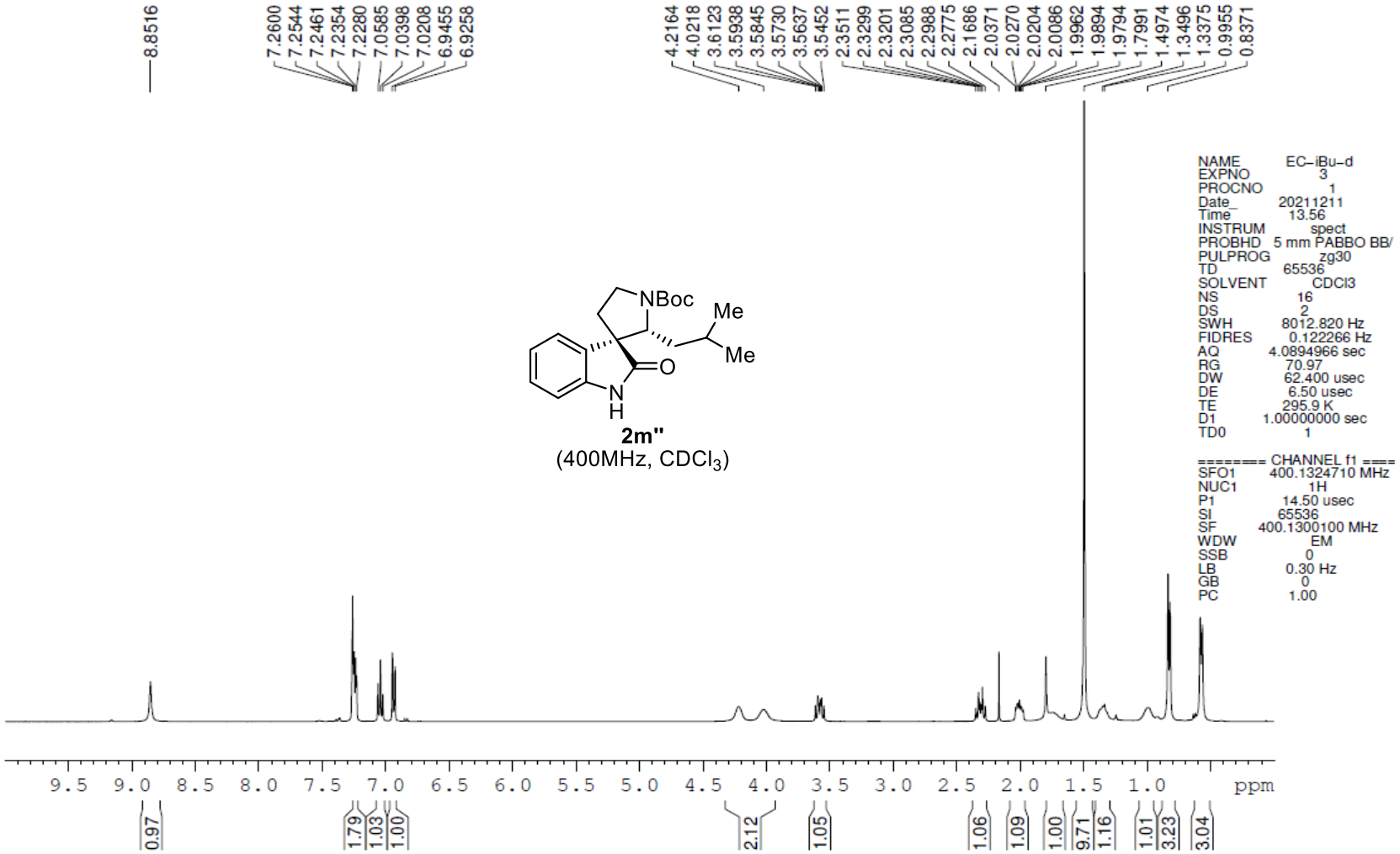

179.0517
154.5080
140.2496
133.2593
128.4415
122.9657
122.7701
109.9483
79.9040
77.4772
77.1600
76.8419
60.9910
56.7567
44.6390
33.3874
28.6398
16.6791
NBoc
Me
O
N
H
2l
(100MHz, CDCl3)
NAME EC-R2Me-u
EXPNO 2
PROCNO 1
Date_ 20211214
Time 20.28
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 300
DS 2
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 se
RG 196.92
DW 20.800 us
DE 6.50 us
TE 296.0 K
D1 2.00000000 se
D11 0.03000000 se
TD0 1
======== CHANNEL f1 ======
SFO1 100.6228298 MH
NUC1 13C
P1 9.70 us
SI 32768
SF 100.6127588 MH
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 10 0 ppm

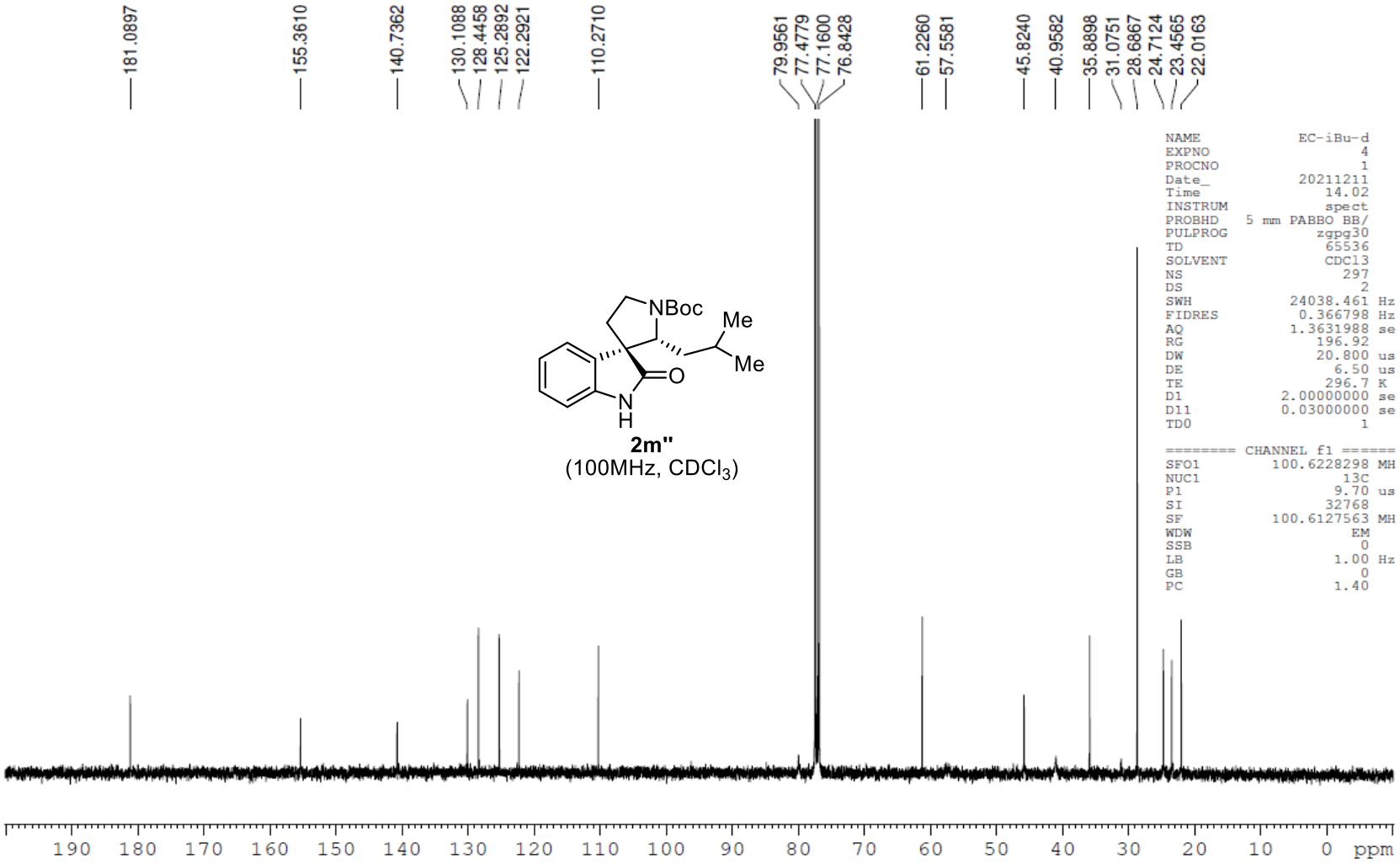

8.1247
7.2713
7.2686
7.2599
7.2493
7.2303
7.0710
7.0688
7.0520
7.0501
7.0330
7.0310
6.9282
6.9091
4.1293
4.1182
3.9465
3.7060
3.6873
3.6621
3.6424
2.3558
2.3357
2.3245
2.3154
2.3044
2.2839
2.0738
1.6395
1.4890
NBoc
Me
O
N
H
2l'
(400MHz, CDCl3)
NAME EC-R2Me-d
EXPNO 1
PROCNO 1
Date_ 20211215
Time 11.30
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 645
DW 60.800 usec
DE 6.00 usec
TE 293.9 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ======
NUC1 1H
P1 15.80 usec
PL1 -1.00 dB
PL1W 12.17476940 W
SFO1 400.1324710 MHz
SI 32768
SF 400.1300099 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
9
8
7
6
5
4
3
2
1
ppm
1.01
1.91
1.09
1.00
1.04
1.07
1.03
1.03
1.09
2.66
9.76

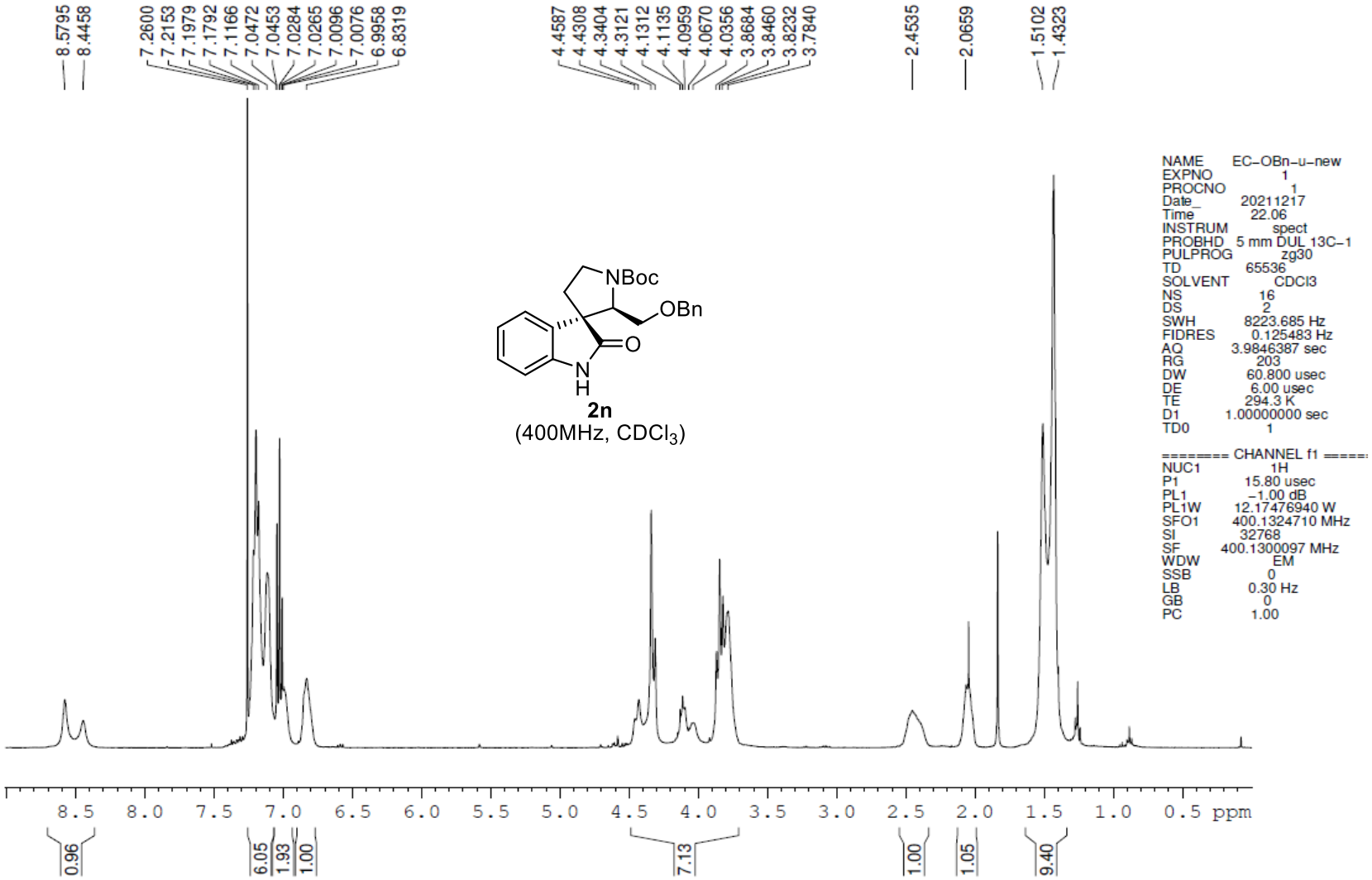

180.0397
155.0786
140.6292
129.9847
128.5349
125.2627
122.5335
110.0525
79.8818
77.4771
77.1600
76.8420
59.0452
45.9638
34.0229
28.6523
17.7448
NBoc
Me
O
N
H
2l'
(100MHz, CDCl3)
NAME 2
EXPNO 2
PROCNO 1
Date_ 20211215
Time 11.54
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 4489
DS 1
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 2050
DW 20.800 usec
DE 6.00 usec
TE 294.3 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ====
NUC1 13C
P1 40.00 usec
PL1 -3.00 dB
PL1W 60.64365387 W
SFO1 100.6228298 MHz
======== CHANNEL f2 ====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -1.00 dB
PL12 14.39 dB
PL13 18.00 dB
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

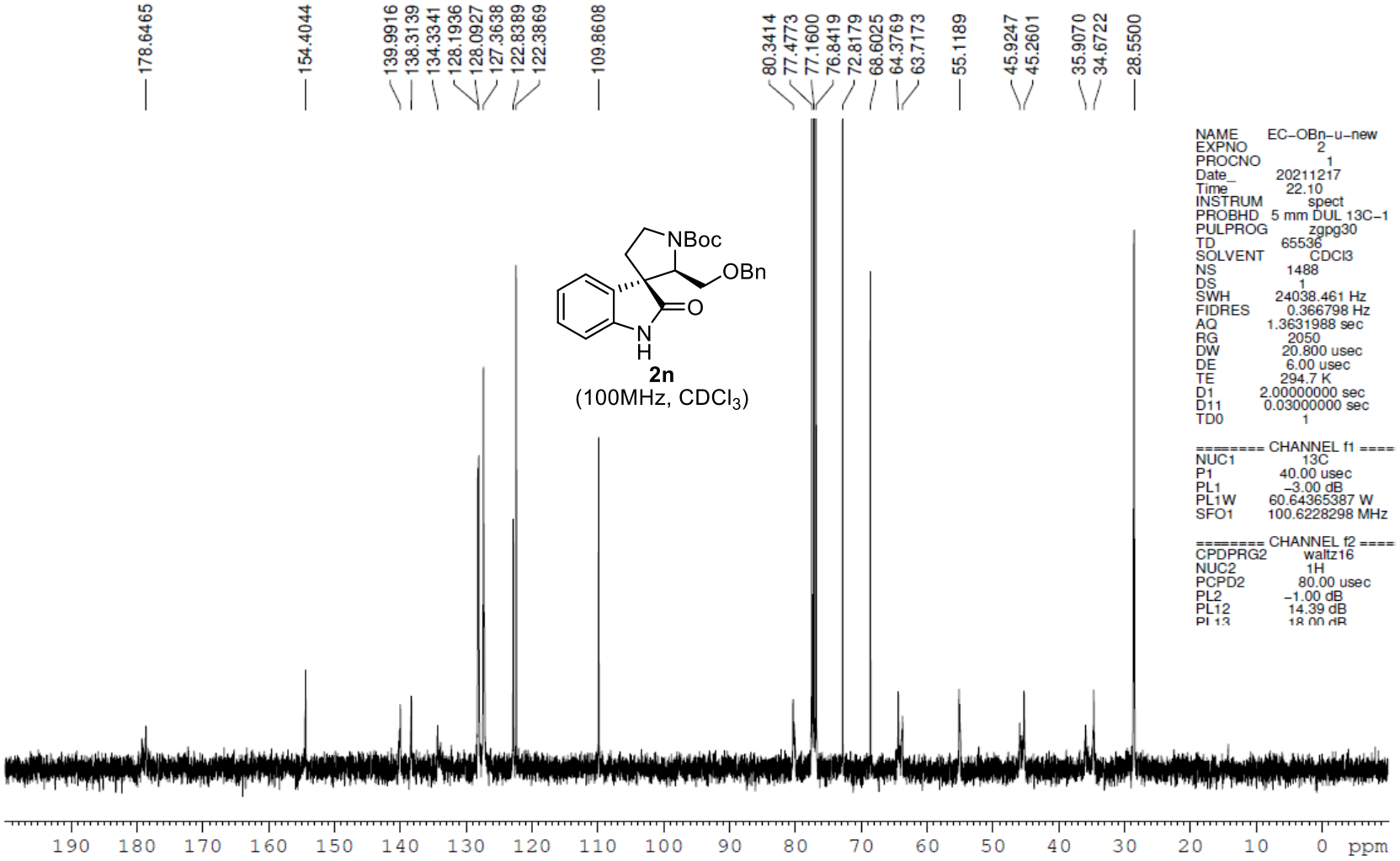

9.2572
7.2600
7.2352
7.2279
7.2145
7.1950
7.0219
7.0061
6.9880
6.9456
6.9266
4.1212
3.9390
3.8695
3.7128
3.6956
2.5178
2.0924
2.0637
2.0513
2.0324
2.0196
2.0010
1.8571
1.4634
1.3808
0.8132
NBoc
Me
Me
O
N
H
2m
(400MHz, CDCl3)
NAME EC-iBu-u
EXPNO 3
PROCNO 1
Date_ 20211211
Time 13.39
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894966 sec
RG 25.32
DW 62.400 usec
DE 6.50 usec
TE 295.9 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ====
SFO1 400.1324710 MHz
NUC1 1H
P1 14.50 usec
SI 65536
SF 400.1300100 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
2.02
1.00
3.13
1.03
1.06
1.01
11.33
6.24

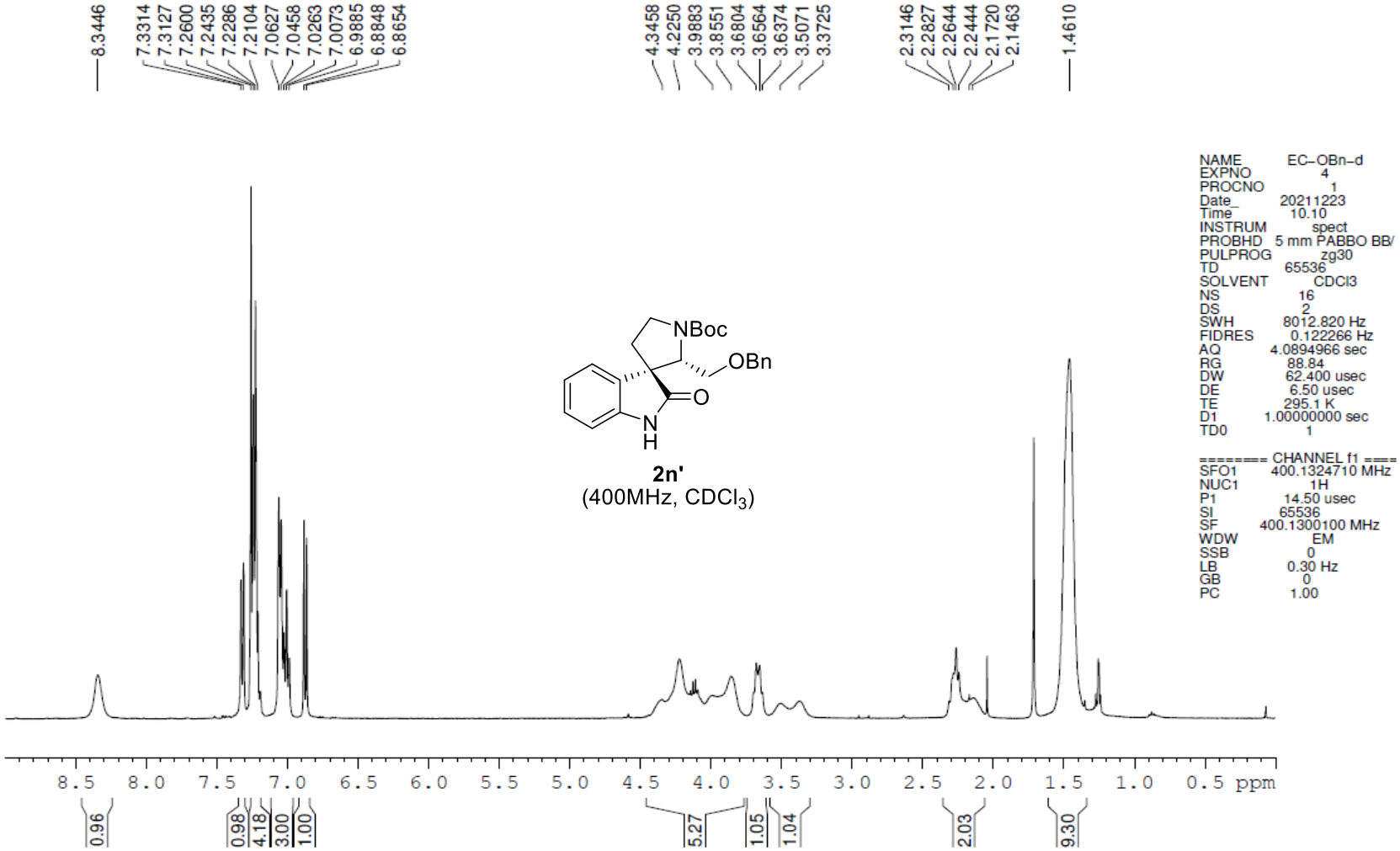

178.7839
154.8220
139.6618
134.8380
128.2121
123.0076
122.5971
110.0052
80.2865
77.4782
77.1600
76.8428
63.6475
55.8912
43.9585
40.2625
34.0204
28.6549
25.1860
23.1091
NBoc
Me
Me
O
N
H
2m
(100MHz, CDCl3)
NAME EC-iBu-u
EXPNO 4
PROCNO 1
Date_ 20211211
Time 13.50
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 200
DS 2
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 se
RG 196.92
DW 20.800 us
DE 6.50 us
TE 296.8 K
D1 2.00000000 se
D11 0.03000000 se
TD0 1
======== CHANNEL f1 ======
SFO1 100.6228298 MH
NUC1 13C
P1 9.70 us
SI 32768
SF 100.6127592 MH
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 10 0 ppm

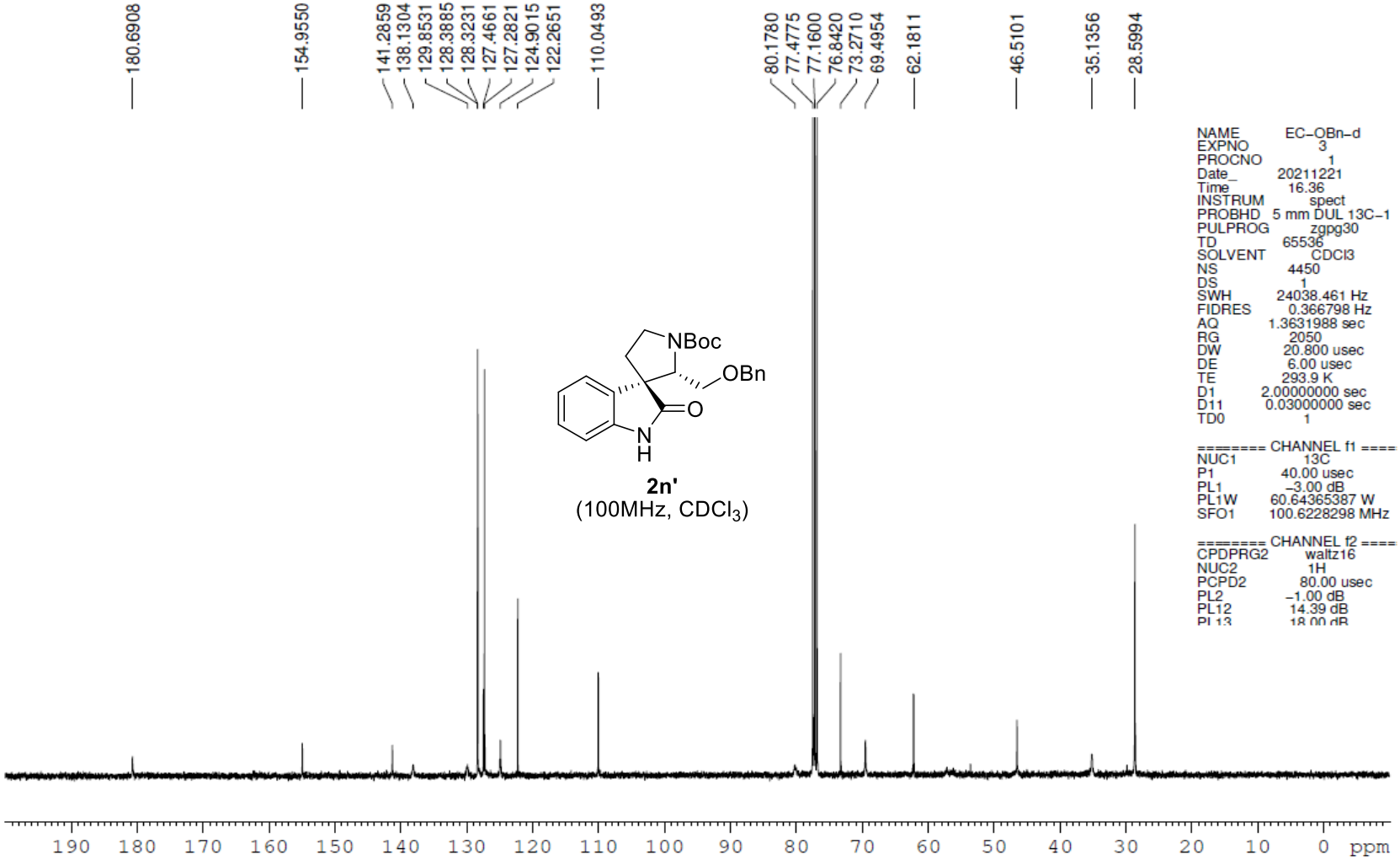

8.8516
7.2600
7.2544
7.2461
7.2354
7.2280
7.0585
7.0398
7.0208
6.9455
6.9258
4.2164
4.0218
3.6123
3.5938
3.5845
3.5730
3.5637
3.5452
2.3511
2.3299
2.3201
2.3085
2.2988
2.2775
2.1686
2.0371
2.0270
2.0204
2.0086
1.9962
1.9894
1.9794
1.7991
1.4974
1.3496
1.3375
0.9955
0.8371
NBoc
Me
Me
O
N
H
2m"
(400MHz, CDCl3)
NAME EC-iBu-d
EXPNO 3
PROCNO 1
Date_ 20211211
Time 13.56
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894966 sec
RG 70.97
DW 62.400 usec
DE 6.50 usec
TE 295.9 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ===
SFO1 400.1324710 MHz
NUC1 1H
P1 14.50 usec
SI 65536
SF 400.1300100 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
1.79
1.03
1.00
2.12
1.05
1.06
1.09
1.00
9.71
1.16
1.01
3.23
3.04

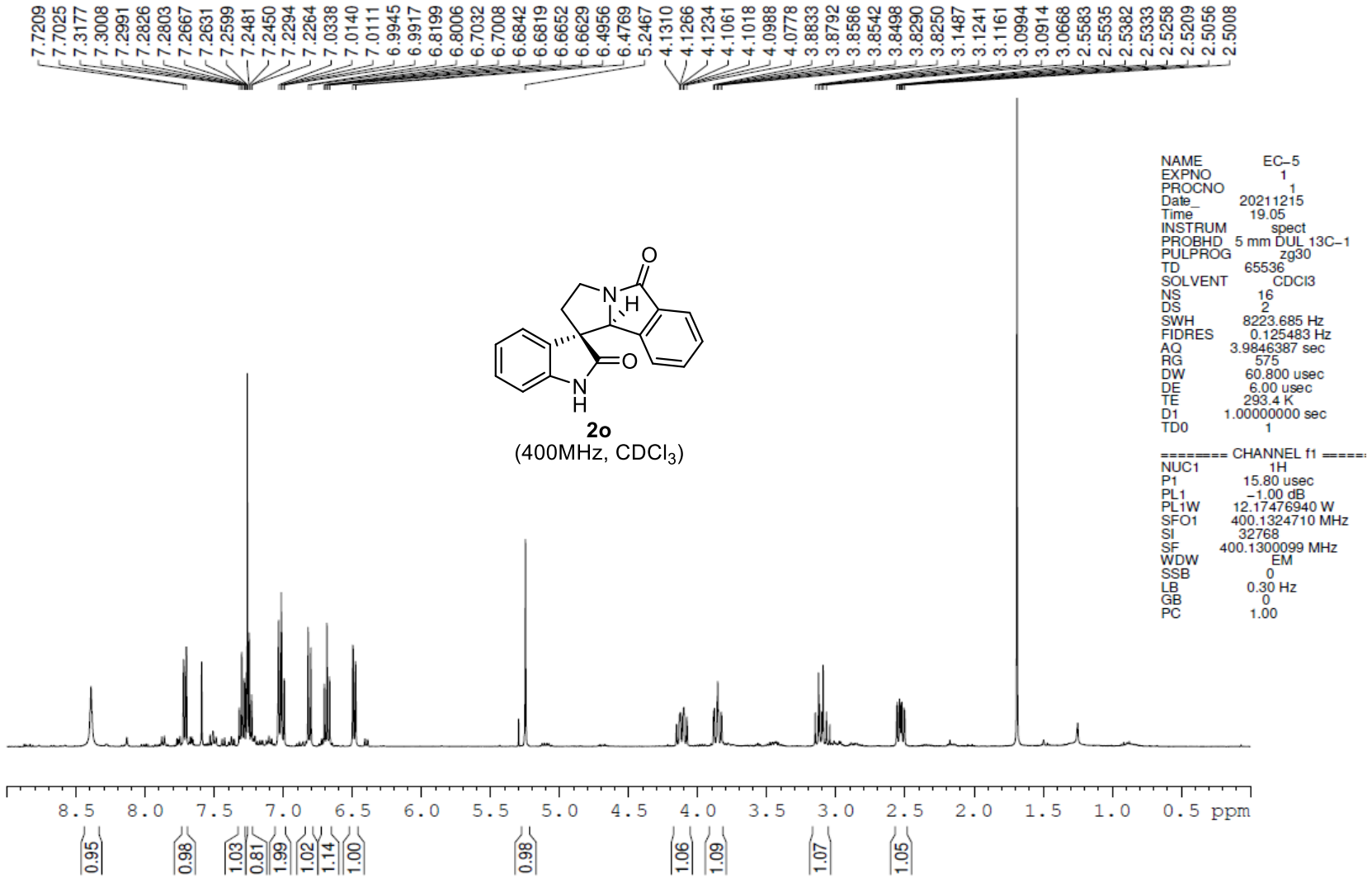

181.0897
155.3610
140.7362
130.1088
128.4458
125.2892
122.2921
110.2710
79.9561
77.4779
77.1600
76.8428
61.2260
57.5581
45.8240
40.9582
35.8898
31.0751
28.6867
24.7124
23.4565
22.0163
NBoc
Me
Me
O
N
H
2m"
(100MHz, CDCl3)
NAME EC-iBu-d
EXPNO 4
PROCNO 1
Date_ 20211211
Time 14.02
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 297
DS 2
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 se
RG 196.92
DW 20.800 us
DE 6.50 us
TE 296.7 K
D1 2.00000000 se
D11 0.03000000 se
TD0 1
======== CHANNEL f1 ======
SFO1 100.6228298 MH
NUC1 13C
P1 9.70 us
SI 32768
SF 100.6127563 MH
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 10 0 ppm

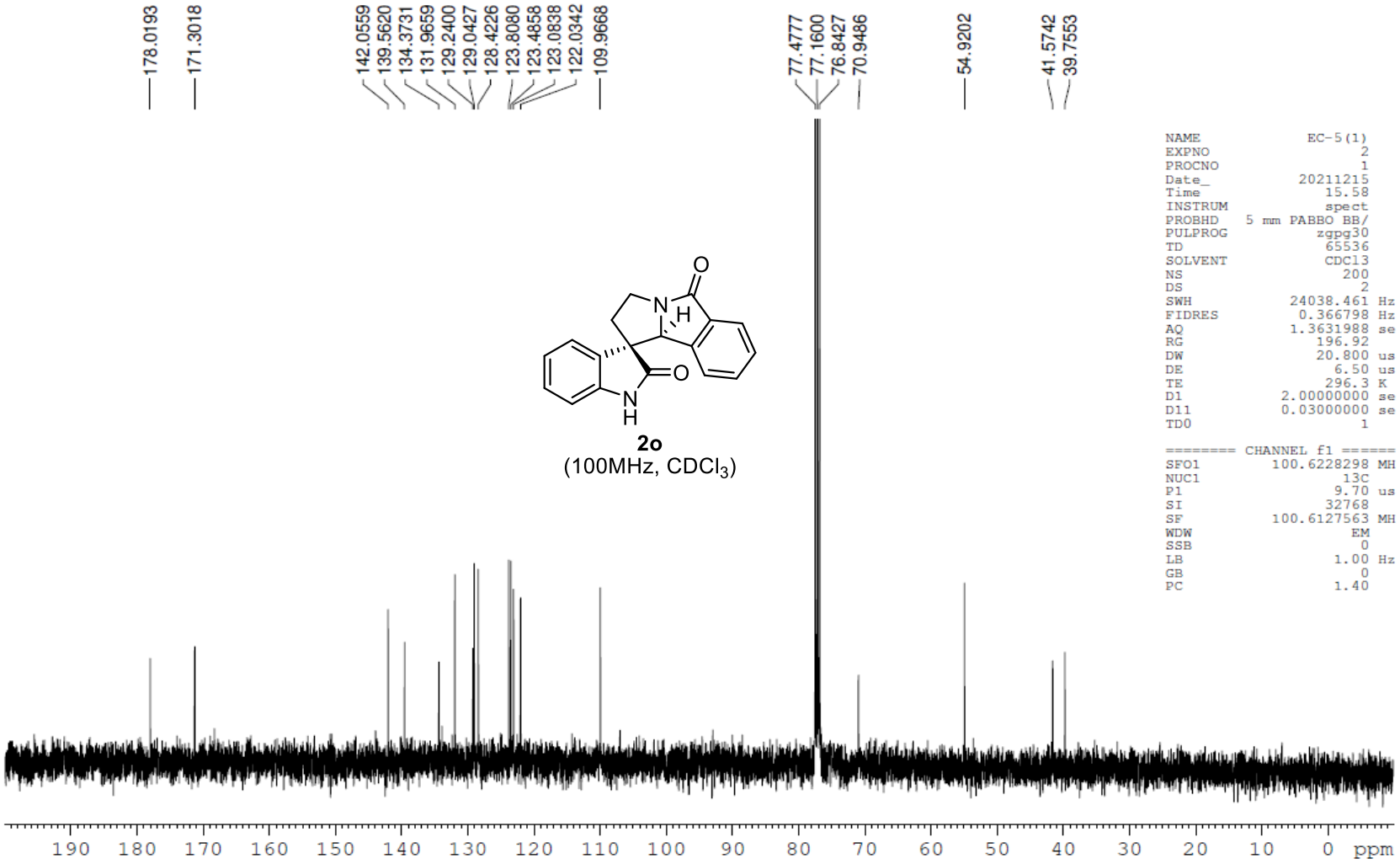

8.5795
8.4458
7.2600
7.2153
7.1979
7.1792
7.1166
7.0472
7.0453
7.0284
7.0265
7.0096
7.0076
6.9958
6.8319
4.4587
4.4308
4.3404
4.3121
4.1312
4.1135
4.0959
4.0670
4.0356
3.8684
3.8460
3.8232
3.7840
2.4535
2.0659
1.5102
1.4323
NBoc
OBn
O
N
H
2n
(400MHz, CDCl3)
NAME EC-OBn-u-new
EXPNO 1
PROCNO 1
Date_ 20211217
Time 22.06
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 203
DW 60.800 usec
DE 6.00 usec
TE 294.3 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ======
NUC1 1H
P1 15.80 usec
PL1 -1.00 dB
PL1W 12.17476940 W
SFO1 400.1324710 MHz
SI 32768
SF 400.1300097 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5 ppm
0.96
6.05
1.93
1.00
7.13
1.00
1.05
9.40

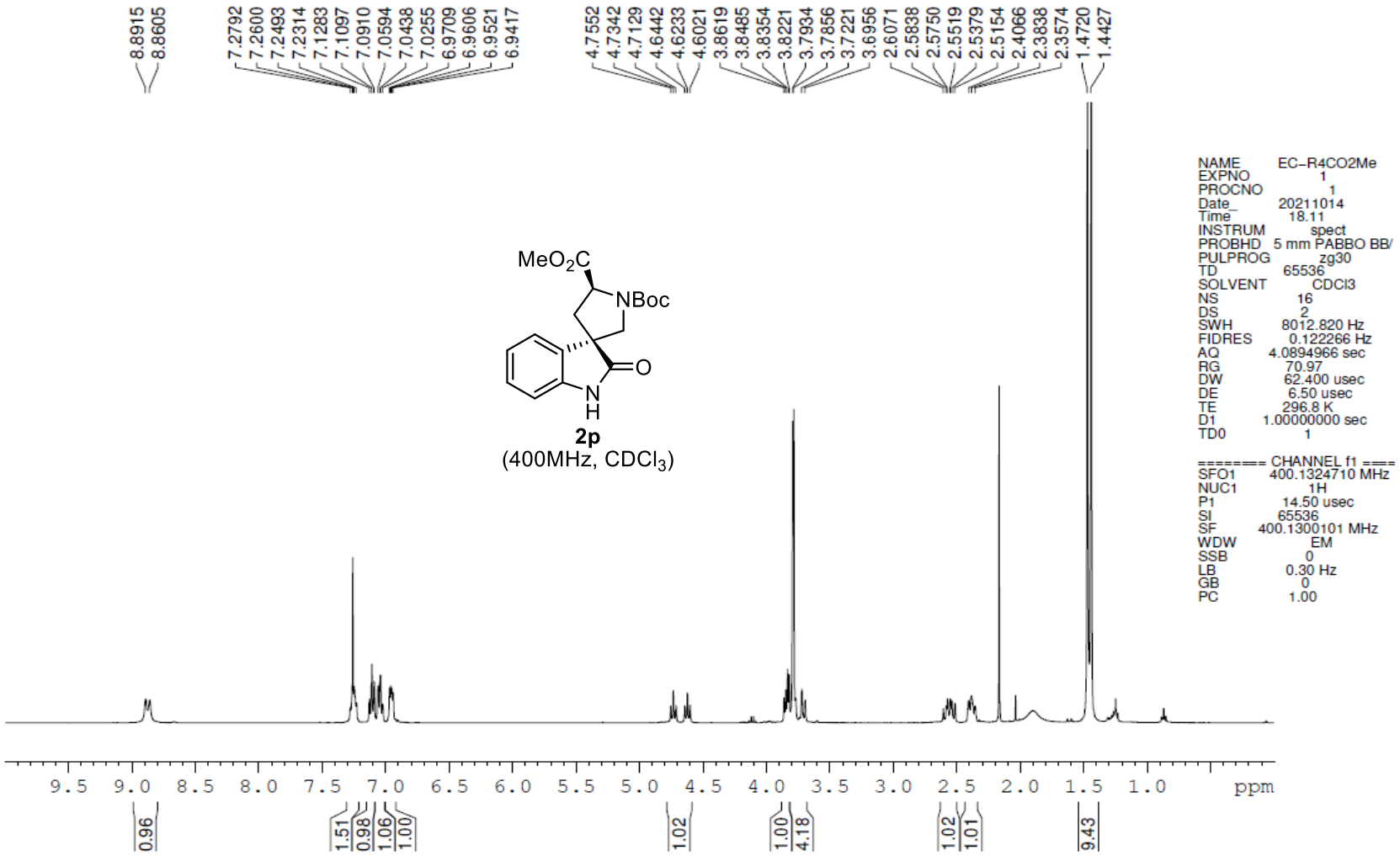

178.6465
154.4044
139.9916
138.3139
134.3341
128.1936
128.0927
127.3638
122.8389
122.3869
109.8608
80.3414
77.4773
77.1600
76.8419
72.8179
68.6025
64.3769
63.7173
55.1189
45.9247
45.2601
35.9070
34.6722
28.5500
NBoc
OBn
O
N
H
2n
(100MHz, CDCl3)
NAME EC-OBn-u-new
EXPNO 2
PROCNO 1
Date_ 20211217
Time 22.10
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1488
DS 1
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 2050
DW 20.800 usec
DE 6.00 usec
TE 294.7 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ====
NUC1 13C
P1 40.00 usec
PL1 -3.00 dB
PL1W 60.64365387 W
SFO1 100.6228298 MHz
======== CHANNEL f2 ====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -1.00 dB
PL12 14.39 dB
PL13 18.00 dB
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

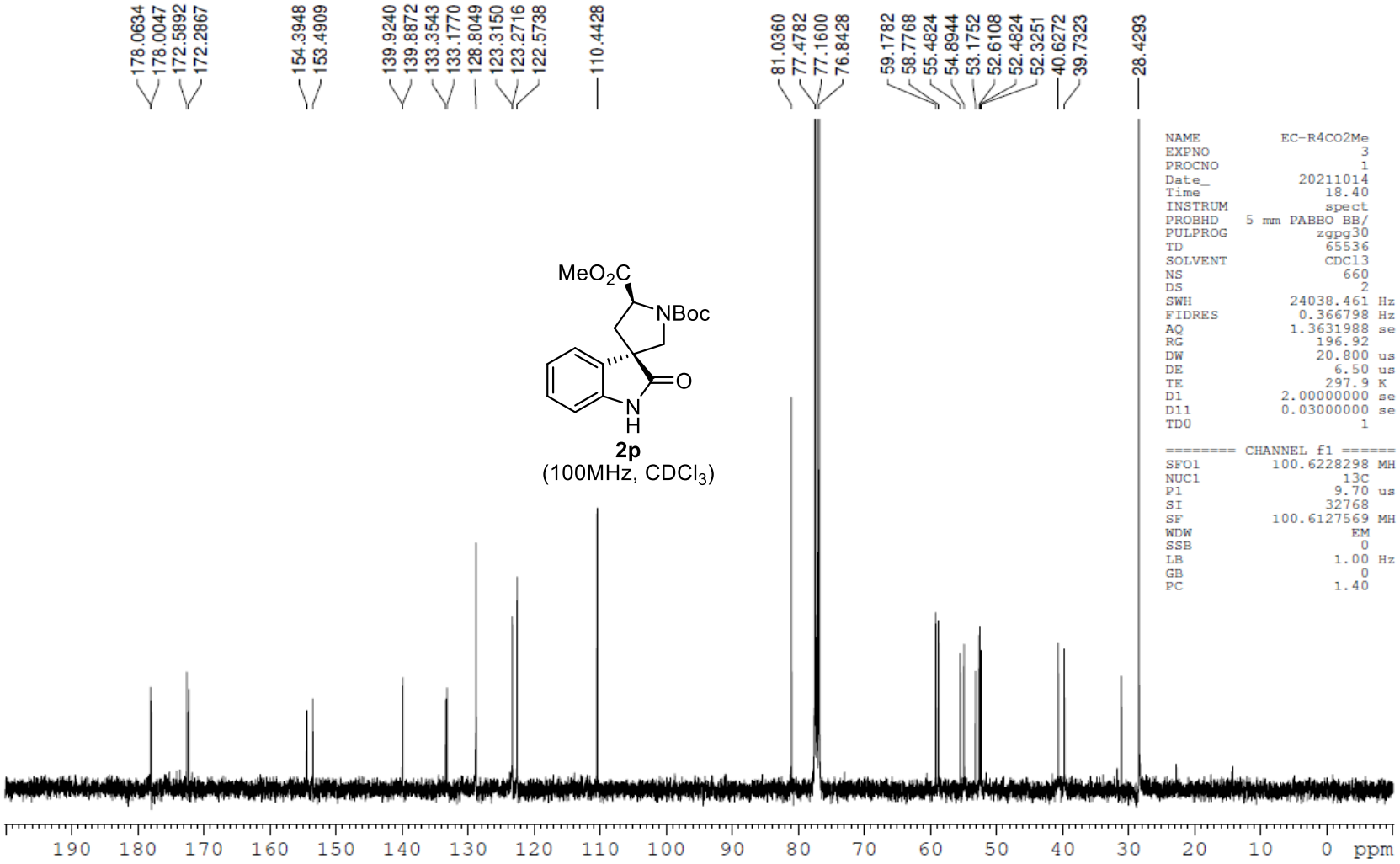

8.3446
7.3314
7.3127
7.2600
7.2435
7.2286
7.2104
7.0627
7.0458
7.0263
7.0073
6.9885
6.8848
6.8654
4.3458
4.2250
3.9883
3.8551
3.6804
3.6564
3.6374
3.5071
3.3725
2.3146
2.2827
2.2644
2.2444
2.1720
2.1463
1.4610
NBoc
OBn
O
N
H
2n'
(400MHz, CDCl3)
NAME EC-OBn-d
EXPNO 4
PROCNO 1
Date_ 20211223
Time 10.10
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894966 sec
RG 88.84
DW 62.400 usec
DE 6.50 usec
TE 295.1 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ====
SFO1 400.1324710 MHz
NUC1 1H
P1 14.50 usec
SI 65536
SF 400.1300100 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
0.96
0.98
4.18
3.00
1.00
5.27
1.05
1.04
2.03
9.30

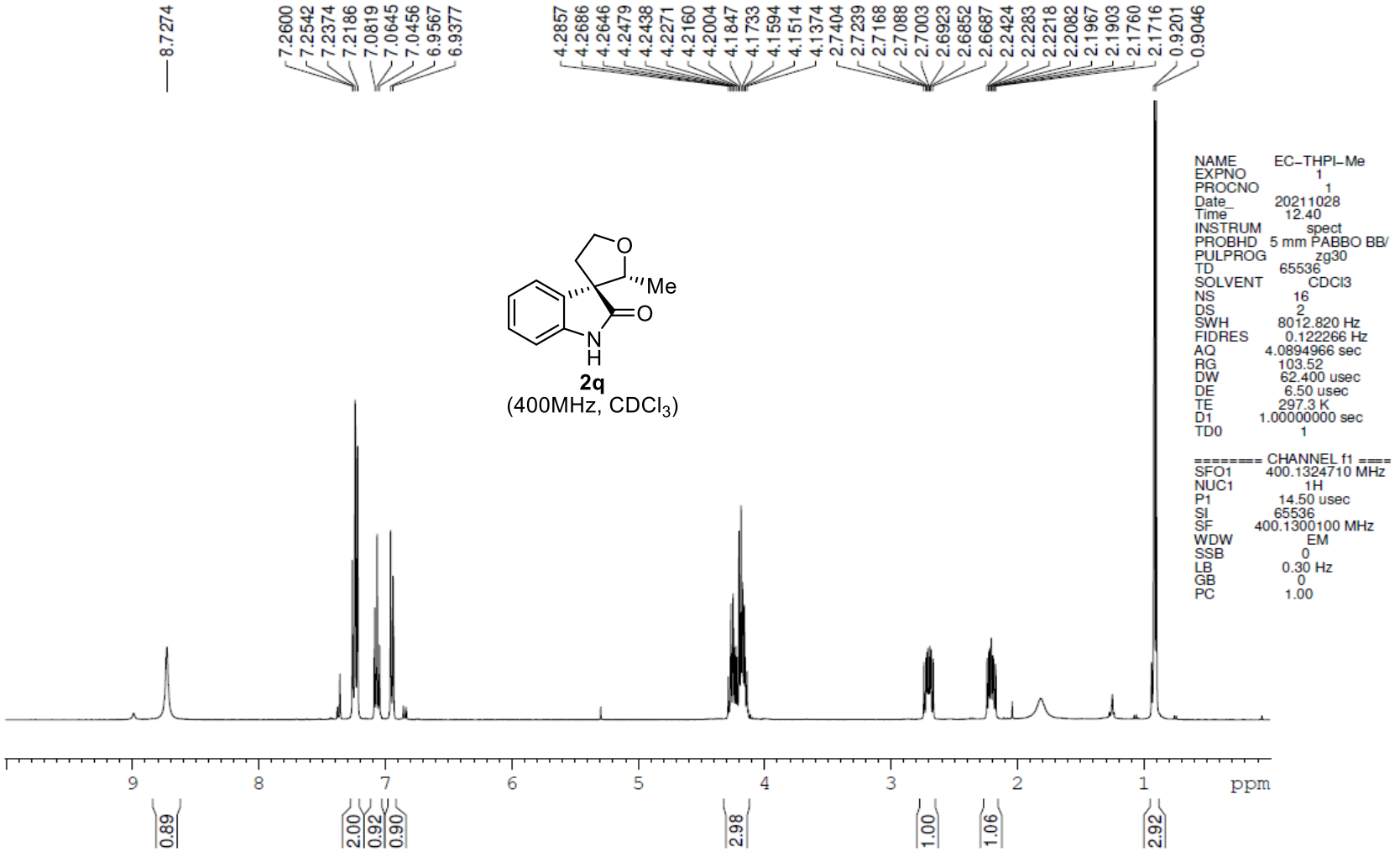

180.6908
154.9550
141.2859
138.1304
129.8531
128.3885
128.3231
127.4661
127.2821
124.9015
122.2651
110.0493
80.1780
77.4775
77.1600
76.8420
73.2710
69.4954
62.1811
46.5101
35.1356
28.5994
NBoc
OBn
O
N
H
2n'
(100MHz, CDCl3)
NAME EC-OBn-d
EXPNO 3
PROCNO 1
Date_ 20211221
Time 16.36
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 4450
DS 1
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 2050
DW 20.800 usec
DE 6.00 usec
TE 293.9 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ====
NUC1 13C
P1 40.00 usec
PL1 -3.00 dB
PL1W 60.64365387 W
SFO1 100.6228298 MHz
======== CHANNEL f2 ====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -1.00 dB
PL12 14.39 dB
PL13 18.00 dB
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

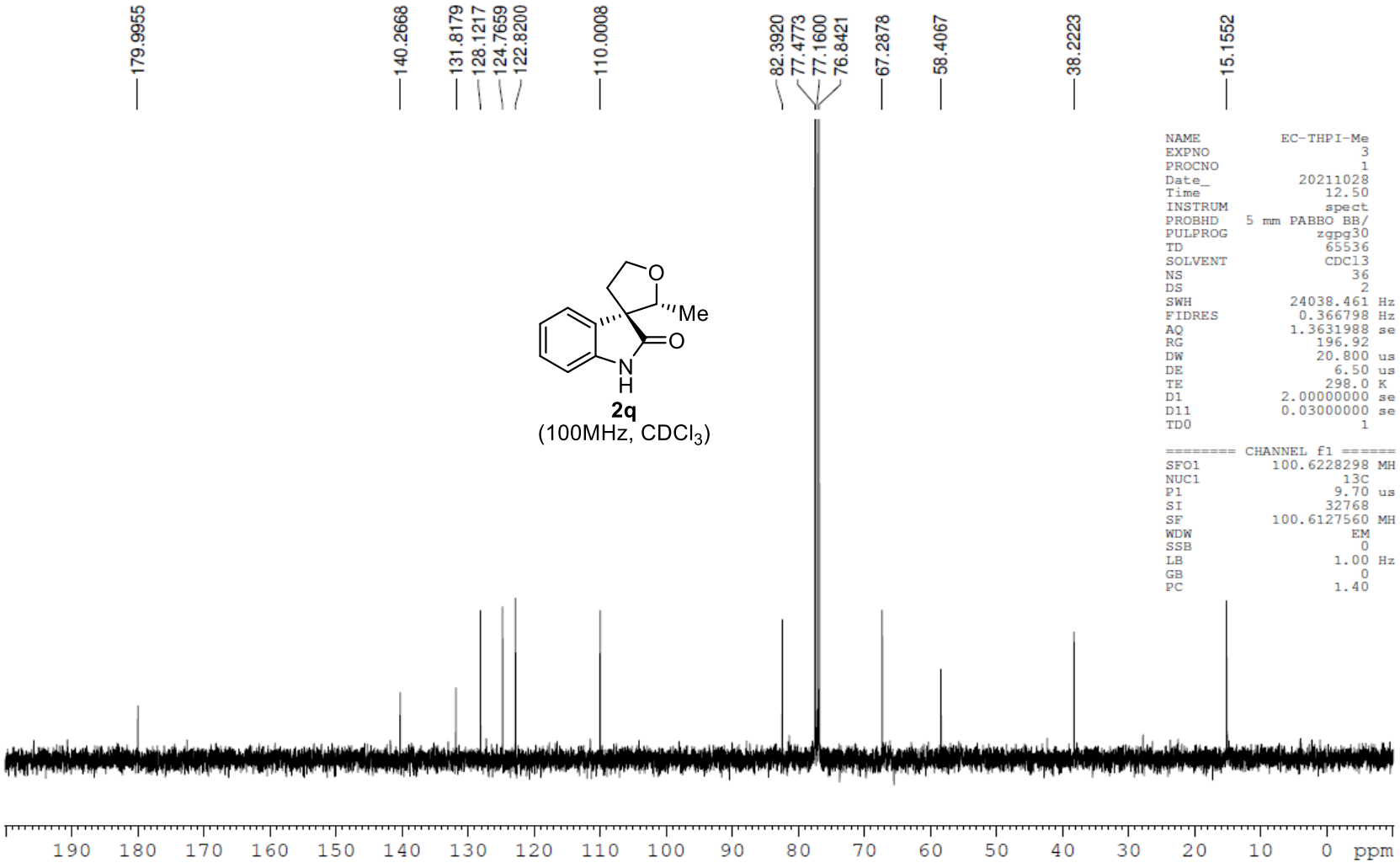

7.7209
7.7025
7.3177
7.3008
7.2991
7.2826
7.2803
7.2667
7.2631
7.2599
7.2481
7.2450
7.2294
7.2264
7.0338
7.0140
7.0111
6.9945
6.9917
6.8199
6.8006
6.7032
6.7008
6.6842
6.6819
6.6652
6.6629
6.4956
6.4769
5.2467
4.1310
4.1266
4.1234
4.1061
4.1018
4.0988
4.0778
3.8833
3.8792
3.8586
3.8542
3.8498
3.8290
3.8250
3.1487
3.1241
3.1161
3.0994
3.0914
3.0668
2.5583
2.5535
2.5382
2.5333
2.5258
2.5209
2.5056
2.5008
O
N
H
O
O
N
H
2o
(400MHz, CDCl3)
NAME EC-5
EXPNO 1
PROCNO 1
Date_ 20211215
Time 19.05
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 575
DW 60.800 usec
DE 6.00 usec
TE 293.4 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ======
NUC1 1H
P1 15.80 usec
PL1 -1.00 dB
PL1W 12.17476940 W
SFO1 400.1324710 MHz
SI 32768
SF 400.1300099 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5 ppm
0.95
0.98
1.03
0.81
1.99
1.02
1.14
1.00
0.98
1.06
1.09
1.07
1.05

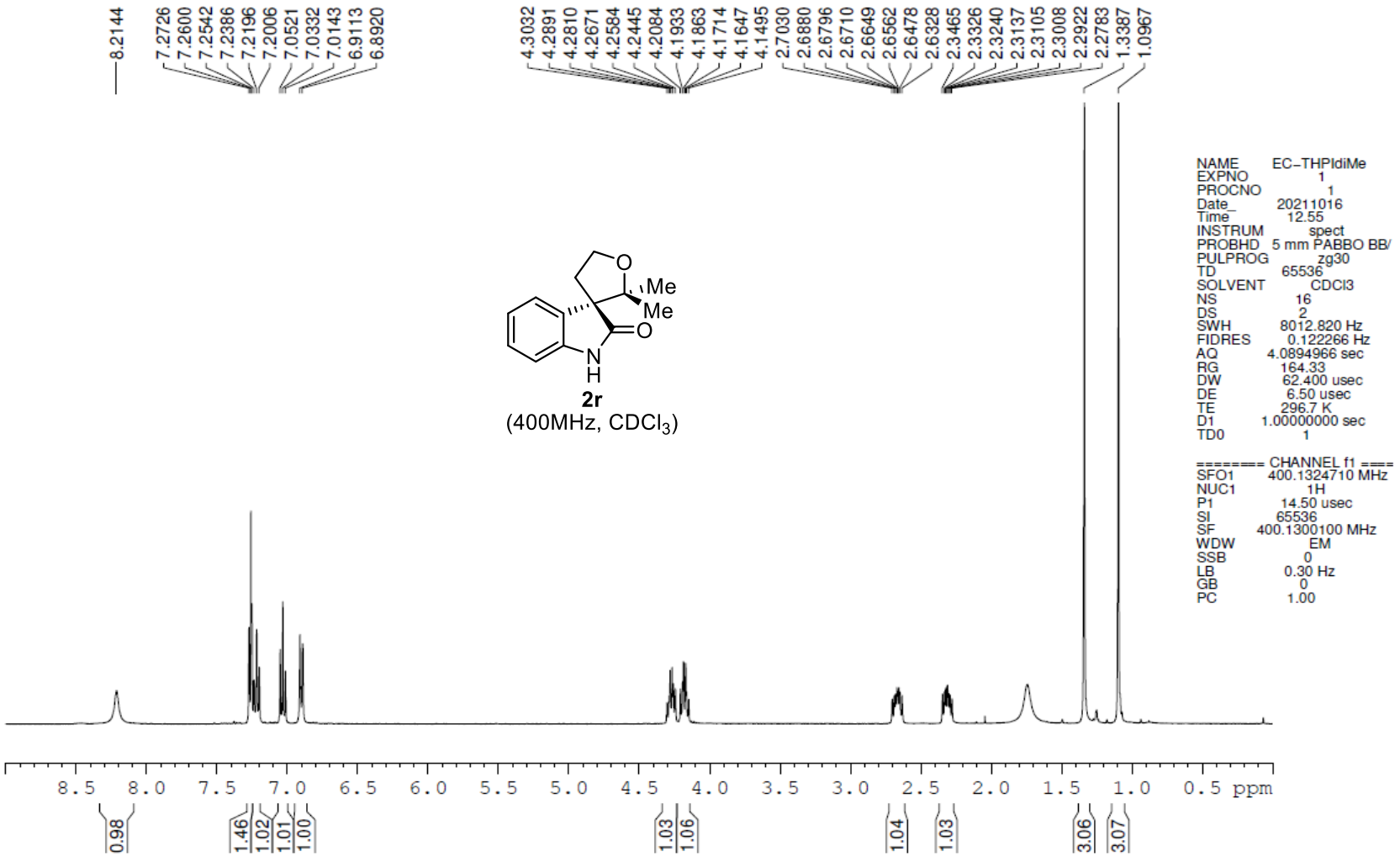

178.0193
171.3018
142.0559
139.5620
134.3731
131.9659
129.2400
129.0427
128.4226
123.8080
123.4858
123.0838
122.0342
109.9668
77.4777
77.1600
76.8427
70.9486
54.9202
41.5742
39.7553
NAME EC-5(1)
EXPNO 2
PROCNO 1
Date_ 20211215
Time 15.58
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 200
DS 2
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 se
RG 196.92
DW 20.800 us
DE 6.50 us
TE 296.3 K
D1 2.00000000 se
D11 0.03000000 se
TD0 1
======== CHANNEL f1 ======
SFO1 100.6228298 MH
NUC1 13C
P1 9.70 us
SI 32768
SF 100.6127563 MH
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
2o
(100MHz, CDCl3)
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

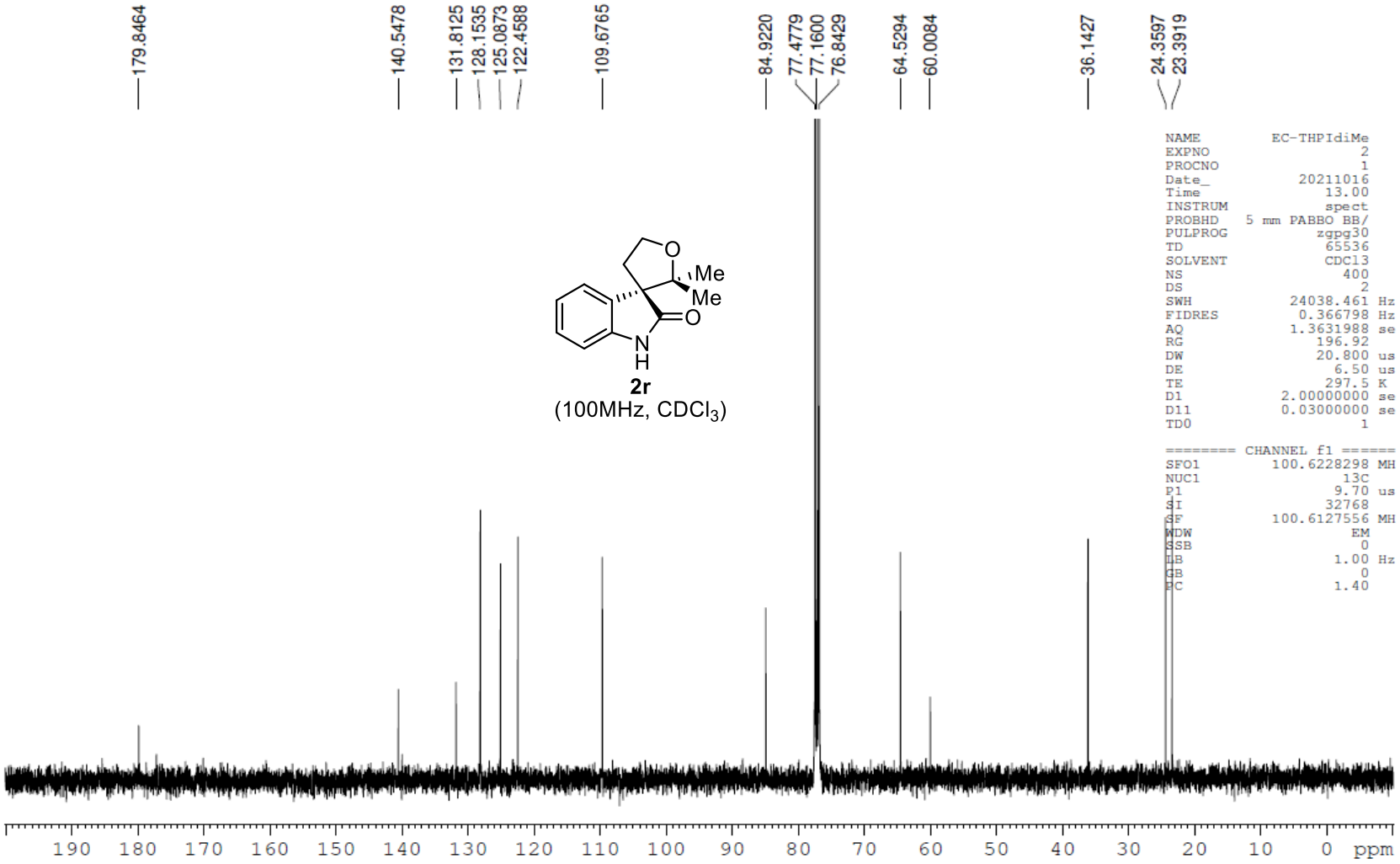

8.8915
8.8605
7.2792
7.2600
7.2493
7.2314
7.1283
7.1097
7.0910
7.0594
7.0438
7.0255
6.9709
6.9606
6.9521
6.9417
4.7552
4.7342
4.7129
4.6442
4.6233
4.6021
3.8619
3.8485
3.8354
3.8221
3.7934
3.7856
3.7221
3.6956
2.6071
2.5838
2.5750
2.5519
2.5379
2.5154
2.4066
2.3838
2.3574
1.4720
1.4427
MeO2C
NBoc
O
N
H
2p
(400MHz, CDCl3)
NAME EC-R4CO2Me
EXPNO 1
PROCNO 1
Date_ 20211014
Time 18.11
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894966 sec
RG 70.97
DW 62.400 usec
DE 6.50 usec
TE 296.8 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ====
SFO1 400.1324710 MHz
NUC1 1H
P1 14.50 usec
SI 65536
SF 400.1300101 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
1.51
0.98
1.06
1.00
1.02
1.00
4.18
1.02
1.01
9.43

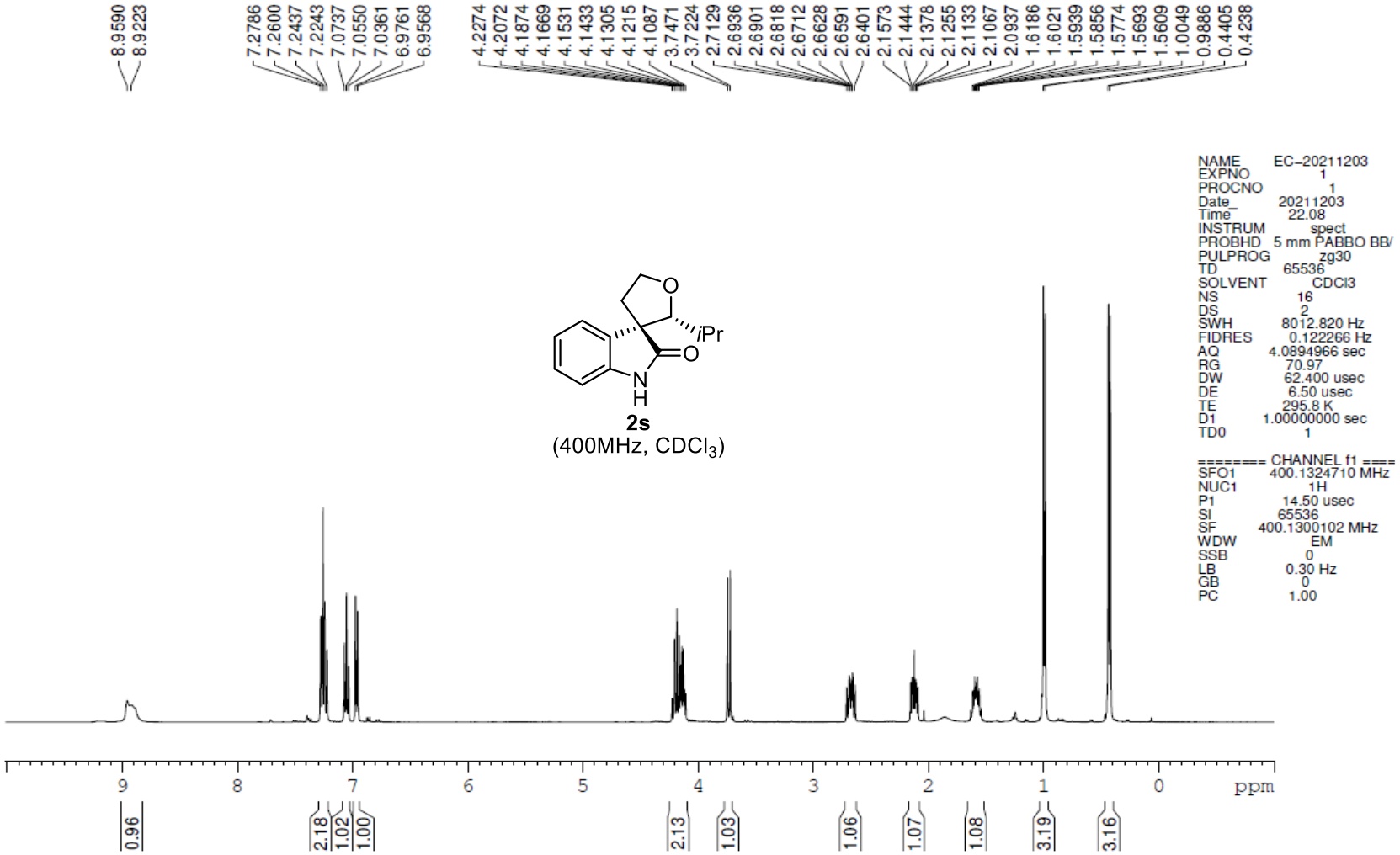

178.0634
178.0047
172.5892
172.2867
154.3948
153.4909
139.9240
139.8872
133.3543
133.1770
128.8049
123.3150
123.2716
122.5738
110.4428
81.0360
77.4782
77.1600
76.8428
59.1782
58.7768
55.4824
54.8944
53.1752
52.6108
52.4824
52.3251
40.6272
39.7323
28.4293
MeO2C
NBoc
O
N
H
2p
(100MHz, CDCl3)
NAME EC-R4CO2Me
EXPNO 3
PROCNO 1
Date_ 20211014
Time 18.40
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 660
DS 2
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 se
RG 196.92
DW 20.800 us
DE 6.50 us
TE 297.9 K
D1 2.00000000 se
D11 0.03000000 se
TD0 1
======== CHANNEL f1 ======
SFO1 100.6228298 MH
NUC1 13C
P1 9.70 us
SI 32768
SF 100.6127569 MH
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 10 0 ppm

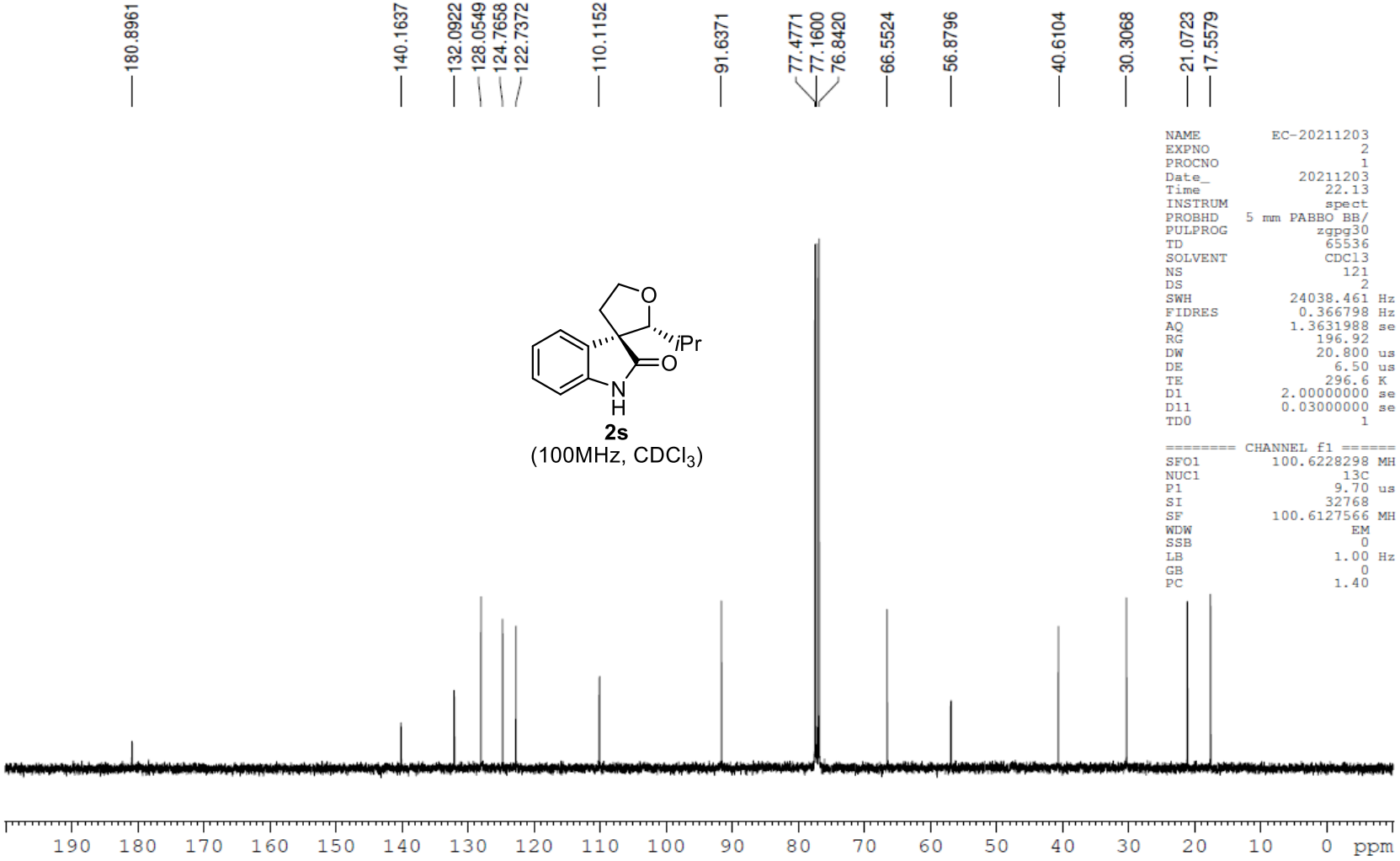

8.7274
7.2600
7.2542
7.2374
7.2186
7.0819
7.0645
7.0456
6.9567
6.9377
4.2857
4.2686
4.2646
4.2479
4.2438
4.2271
4.2160
4.2004
4.1847
4.1733
4.1594
4.1514
4.1374
2.7404
2.7239
2.7168
2.7088
2.7003
2.6923
2.6852
2.6687
2.2424
2.2283
2.2218
2.2082
2.1967
2.1903
2.1760
2.1716
0.9201
0.9046
Me
O
O
N
H
2q
(400MHz, CDCl3)
NAME EC-THPI-Me
EXPNO 1
PROCNO 1
Date_ 20211028
Time 12.40
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894966 sec
RG 103.52
DW 62.400 usec
DE 6.50 usec
TE 297.3 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ===
SFO1 400.1324710 MHz
NUC1 1H
P1 14.50 usec
SI 65536
SF 400.1300100 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
9
8
7
6
5
4
3
2
1
ppm
0.89
2.00
0.92
0.90
2.98
1.00
1.06
2.92

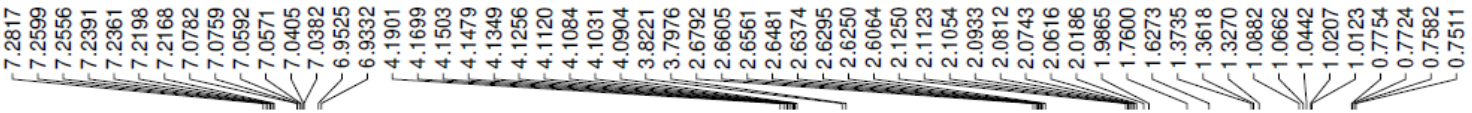

179.9955
140.2668
131.8179
128.1217
124.7659
122.8200
110.0008
82.3920
77.4773
77.1600
76.8421
67.2878
58.4067
38.2223
15.1552
O
Me
O
N
H
2q
(100MHz, CDCl3)
NAME EC-THPI-Me
EXPNO 3
PROCNO 1
Date_ 20211028
Time 12.50
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 36
DS 2
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 se
RG 196.92
DW 20.800 us
DE 6.50 us
TE 298.0 K
D1 2.00000000 se
D11 0.03000000 se
TD0 1
======== CHANNEL f1 ======
SFO1 100.6228298 MH
NUC1 13C
P1 9.70 us
SI 32768
SF 100.6127560 MH
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 10 0 ppm

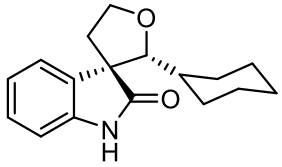

8.2144
7.2726
7.2600
7.2542
7.2386
7.2196
7.2006
7.0521
7.0332
7.0143
6.9113
6.8920
4.3032
4.2891
4.2810
4.2671
4.2584
4.2445
4.2084
4.1933
4.1863
4.1714
4.1647
4.1495
2.7030
2.6880
2.6796
2.6710
2.6649
2.6562
2.6478
2.6328
2.3465
2.3326
2.3240
2.3137
2.3105
2.3008
2.2922
2.2783
1.3387
1.0967
Me
Me
O
O
N
H
2r
(400MHz, CDCl3)
NAME EC-THPIdiMe
EXPNO 1
PROCNO 1
Date_ 20211016
Time 12.55
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894966 sec
RG 164.33
DW 62.400 usec
DE 6.50 usec
TE 296.7 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ====
SFO1 400.1324710 MHz
NUC1 1H
P1 14.50 usec
SI 65536
SF 400.1300100 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
0.98
1.46
1.02
1.01
1.00
1.03
1.06
1.04
1.03
3.06
3.07


179.8464
140.5478
131.8125
128.1535
125.0873
122.4588
109.6765
84.9220
77.4779
77.1600
76.8429
64.5294
60.0084
36.1427
24.3597
23.3919
Me
Me
O
O
N
H
2r
(100MHz, CDCl3)
NAME EC-THPIdiMe
EXPNO 2
PROCNO 1
Date_ 20211016
Time 13.00
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 400
DS 2
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 se
RG 196.92
DW 20.800 us
DE 6.50 us
TE 297.5 K
D1 2.00000000 se
D11 0.03000000 se
TD0 1
======== CHANNEL f1 ======
SFO1 100.6228298 MH
NUC1 13C
P1 9.70 us
SI 32768
SF 100.6127556 MH
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 10 0 ppm


8.9590
8.9223
7.2786
7.2600
7.2437
7.2243
7.0737
7.0550
7.0361
6.9761
6.9568
4.2274
4.2072
4.1874
4.1669
4.1531
4.1433
4.1305
4.1215
4.1087
3.7471
3.7224
2.7129
2.6936
2.6901
2.6818
2.6712
2.6628
2.6591
2.6401
2.1573
2.1444
2.1378
2.1255
2.1133
2.1067
2.0937
1.6186
1.6021
1.5939
1.5856
1.5774
1.5693
1.5609
1.0049
0.9886
0.4405
0.4238
iPr
O
O
N
H
2s
(400MHz, CDCl3)
NAME EC-20211203
EXPNO 1
PROCNO 1
Date_ 20211203
Time 22.08
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894966 sec
RG 70.97
DW 62.400 usec
DE 6.50 usec
TE 295.8 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ====
SFO1 400.1324710 MHz
NUC1 1H
P1 14.50 usec
SI 65536
SF 400.1300102 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
9
8
7
6
5
4
3
2
1
0
ppm
0.96
2.18
1.02
1.00
2.13
1.03
1.06
1.07
1.08
3.19
3.16

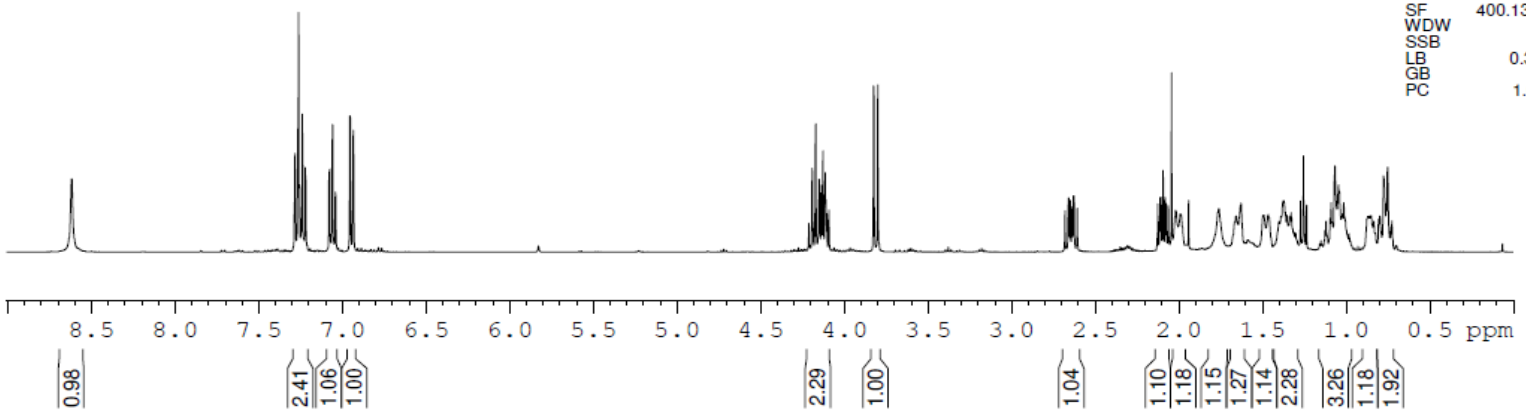

180.8961
140.1637
132.0922
128.0549
124.7658
122.7372
110.1152
91.6371
77.4771
77.1600
76.8420
66.5524
56.8796
40.6104
30.3068
21.0723
17.5579
NAME EC-20211203
EXPNO 2
PROCNO 1
Date_ 20211203
Time 22.13
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 121
DS 2
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 se
RG 196.92
DW 20.800 us
DE 6.50 us
TE 296.6 K
D1 2.00000000 se
D11 0.03000000 se
TD0 1
======== CHANNEL f1 ======
SFO1 100.6228298 MH
NUC1 13C
P1 9.70 us
SI 32768
SF 100.6127566 MH
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
O
iPr
O
N
H
2s
(100MHz, CDCl3)
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm



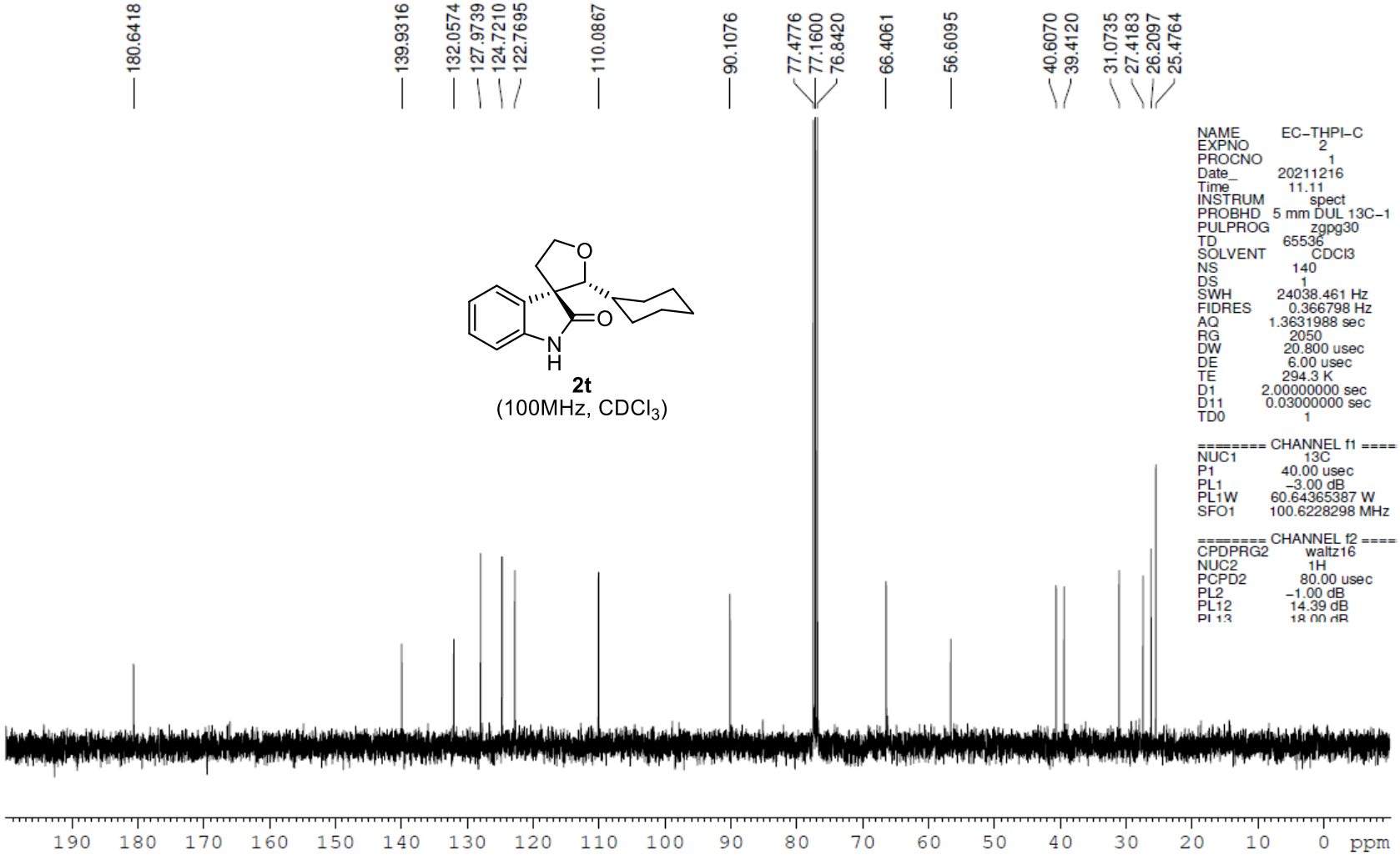


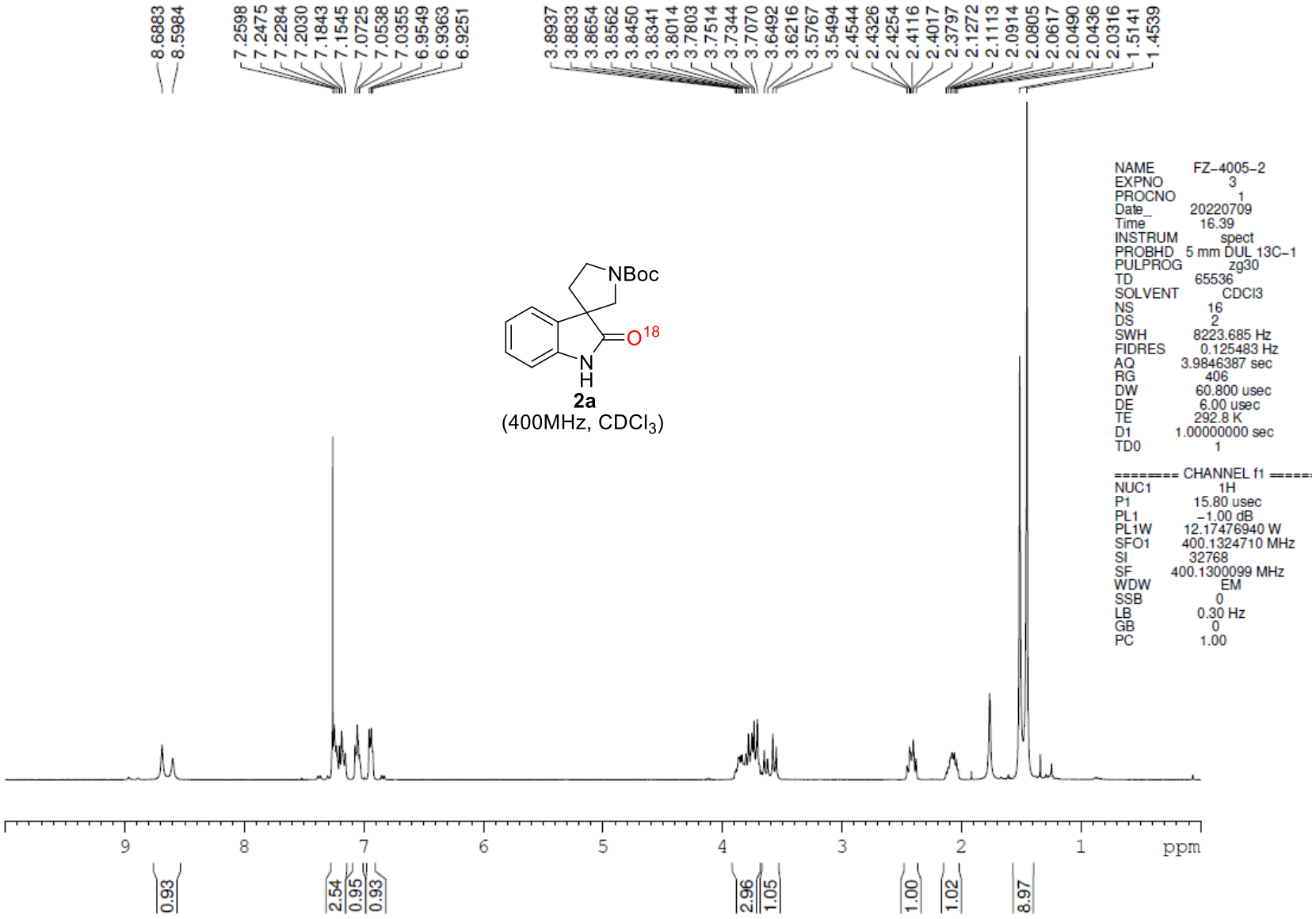
2t

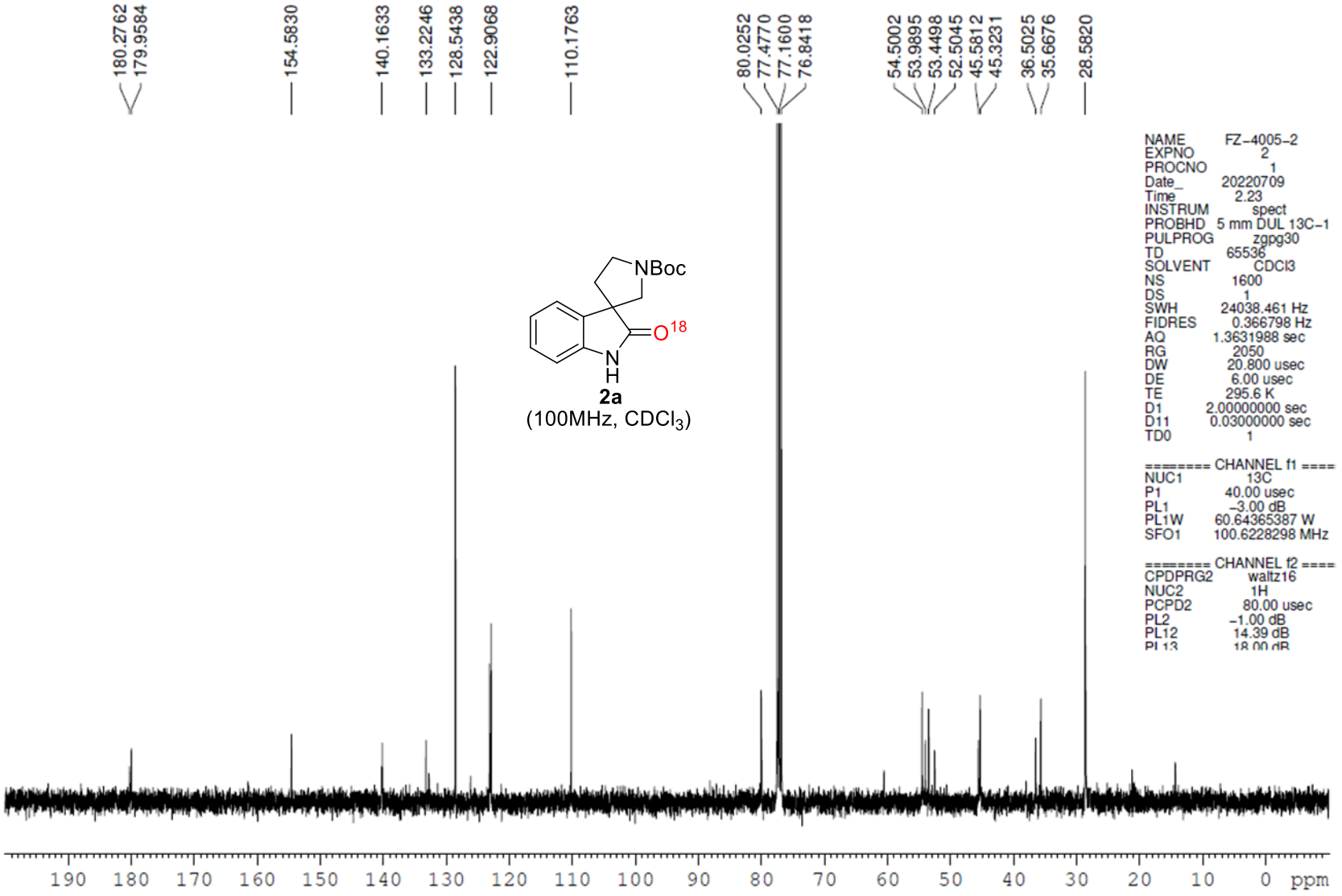
(400MHz, $CDCl_3$)

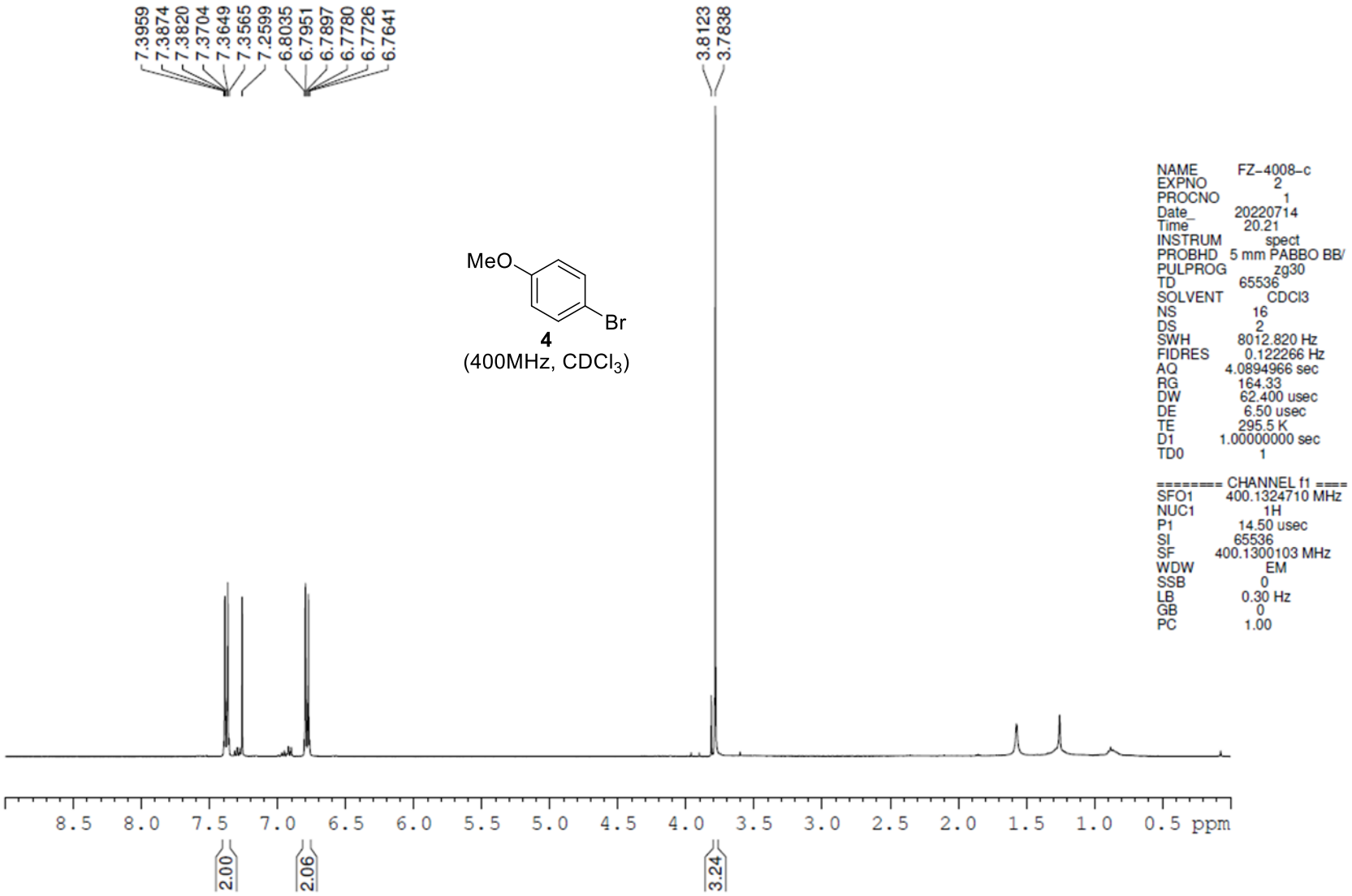


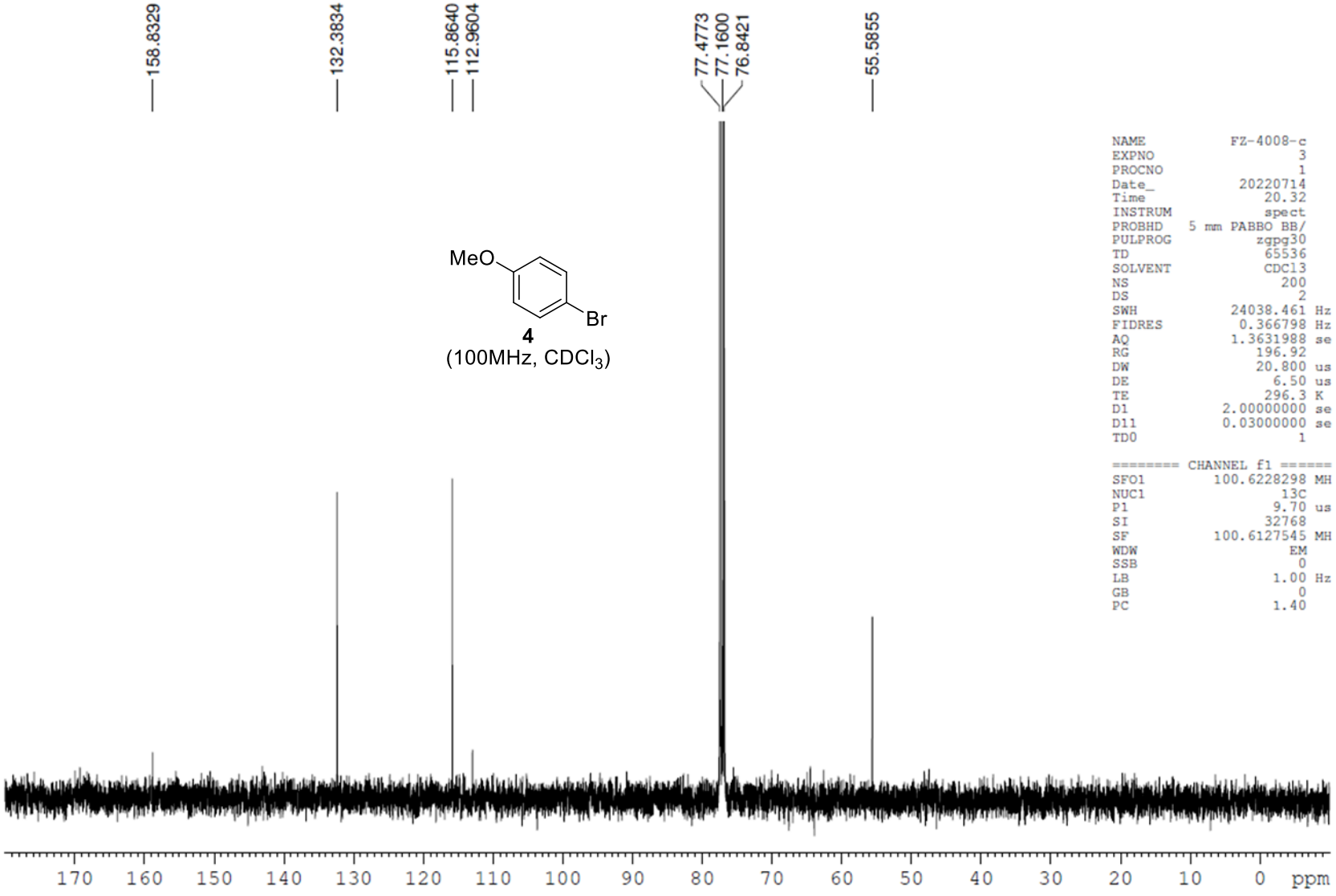

```
NAME          EC-THPI-C
EXPNO                 1
PROCNO                1
Date_          20211216
Time              11.07
INSTRUM           spect
PROBHD  5 mm DUL 13C-1
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                   16
DS                    2
SWH           8223.685 Hz
FIDRES       0.125483 Hz
AQ           3.9846387 sec
RG                  287
DW             60.800 usec
DE              6.00 usec
TE              294.0 K
D1          1.00000000 sec
TD0                   1

======== CHANNEL f1 ======
NUC1                1H
P1             15.80 usec
PL1             -1.00 dB
PL1W      12.17476940 W
SFO1      400.1324710 MHz
SI               32768
SF        400.1300099 MHz
WDW                EM
SSB                 0
LB              0.30 Hz
GB                  0
PC              1.00
```


180.6418
139.9316
132.0574
127.9739
124.7210
122.7695
110.0867
90.1076
77.4776
77.1600
76.8420
66.4061
56.6095
40.6070
39.4120
31.0735
27.4183
26.2097
25.4764
O
O
N
H
2t
(100MHz, CDCl3)
NAME EC-THPI-C
EXPNO 2
PROCNO 1
Date_ 20211216
Time 11.11
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 140
DS 1
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 2050
DW 20.800 usec
DE 6.00 usec
TE 294.3 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ====
NUC1 13C
P1 40.00 usec
PL1 -3.00 dB
PL1W 60.64365387 W
SFO1 100.6228298 MHz
======== CHANNEL f2 ====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -1.00 dB
PL12 14.39 dB
PL13 18.00 dB
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm


8.6883
8.5984
7.2598
7.2475
7.2284
7.2030
7.1843
7.1545
7.0725
7.0538
7.0355
6.9549
6.9363
6.9251
3.8937
3.8833
3.8654
3.8562
3.8450
3.8341
3.8014
3.7803
3.7514
3.7344
3.7070
3.6492
3.6216
3.5767
3.5494
2.4544
2.4326
2.4254
2.4116
2.4017
2.3797
2.1272
2.1113
2.0914
2.0805
2.0617
2.0490
2.0436
2.0316
1.5141
1.4539
NBoc
O18
N
H
2a
(400MHz, CDCl3)
NAME FZ-4005-2
EXPNO 3
PROCNO 1
Date_ 20220709
Time 16.39
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 406
DW 60.800 usec
DE 6.00 usec
TE 292.8 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ======
NUC1 1H
P1 15.80 usec
PL1 -1.00 dB
PL1W 12.17476940 W
SFO1 400.1324710 MHz
SI 32768
SF 400.1300099 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
9
8
7
6
5
4
3
2
1
ppm
0.93
2.54
0.95
0.93
2.96
1.05
1.00
1.02
8.97


180.2762
179.9584
154.5830
140.1633
133.2246
128.5438
122.9068
110.1763
80.0252
77.4770
77.1600
76.8418
54.5002
53.9895
53.4498
52.5045
45.5812
45.3231
36.5025
35.6676
28.5820
NBoc
O18
N
H
2a
(100MHz, CDCl3)
NAME FZ-4005-2
EXPNO 2
PROCNO 1
Date_ 20220709
Time 2.23
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1600
DS 1
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 2050
DW 20.800 usec
DE 6.00 usec
TE 295.6 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ====
NUC1 13C
P1 40.00 usec
PL1 -3.00 dB
PL1W 60.64365387 W
SFO1 100.6228298 MHz
======== CHANNEL f2 ====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -1.00 dB
PL12 14.39 dB
PL13 18.00 dB
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm


7.3959
7.3874
7.3820
7.3704
7.3649
7.3565
7.2599
6.8035
6.7951
6.7897
6.7780
6.7726
6.7641
3.8123
3.7838
MeO
Br
4
(400MHz, CDCl3)
NAME FZ-4008-c
EXPNO 2
PROCNO 1
Date_ 20220714
Time 20.21
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894966 sec
RG 164.33
DW 62.400 usec
DE 6.50 usec
TE 295.5 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ====
SFO1 400.1324710 MHz
NUC1 1H
P1 14.50 usec
SI 65536
SF 400.1300103 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
2.00
2.06
3.24


158.8329
132.3834
115.8640
112.9604
77.4773
77.1600
76.8421
55.5855
MeO
Br
4
(100MHz, CDCl3)
NAME FZ-4008-c
EXPNO 3
PROCNO 1
Date_ 20220714
Time 20.32
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 200
DS 2
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 se
RG 196.92
DW 20.800 us
DE 6.50 us
TE 296.3 K
D1 2.00000000 se
D11 0.03000000 se
TD0 1
======== CHANNEL f1 ======
SFO1 100.6228298 MH
NUC1 13C
P1 9.70 us
SI 32768
SF 100.6127545 MH
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm