Site-Specific Alkene Hydromethylation via Protonolysis of Titanacyclobutanes

James A. Law, Noah M. Bartfield, and James H. Frederich*

(corresponding author e-mail: jfrederich@fsu.edu)

Supporting Information

Table of Contents

1. Materials and Methods

Unless otherwise stated, reactions were conducted in oven-dried glassware (140 °C) under an atmosphere of nitrogen gas (N_2) using anhydrous solvents. Tetrahydrofuran (THF), methylene chloride (CH_2Cl_2), diethyl ether (Et_2O), and toluene (PhMe) were dried by passage through activated alumina using a solvent purification system. PhMe used for the synthesis of the Tebbe's Reagent (**1**) was degassed by freeze-pump-thaw cycling. Bis(cyclopentadienyl)titanium dichloride was purchased from TCI and stored in a nitrogen glovebox. Solutions of $AlMe_3$ (2.0 M, PhMe) were purchased from Sigma Aldrich and used as received. Starting materials prepared using literature procedures are listed in Figure S1. All other commercial reagents, including **9a**, **9d** and **9e**, were used as received.

One representative reaction and yield of the product is described in detail; isolated yields reported are the average of duplicate reactions, typically within ± 5% of the reported yield. Column chromatography was carried out using silica gel 60 (SiO_2, 240-400 mesh) as stationary phase. Thin layer chromatography (TLC) was performed using pre-coated, glass-backed plates (SiO_2, 60 PF254, 0.25 mm) and visualized by exposure to UV light (254 nm) or by anisaldehyde and/or potassium permanganate staining.

^{1}H NMR spectra were recorded at 400 MHz, 600 MHz or 700 MHz and are reported relative to deuterated solvent signals. Data for ^{1}H NMR spectra are reported as follows: chemical shift (ppm), multiplicity, coupling constant (Hz), and integration. Splitting patterns are abbreviated as follows: singlet (s), doublet (d), triplet (t), quartet (q), quintet (quint), multiplet (m), broad (br), apparent (app), and combinations thereof. ^{13}C NMR spectra were recorded at 100 MHz or 150 MHz. Data for ^{13}C NMR spectra are reported in order of carbon multiplicity (C = quaternary, CH = methine, CH_2 = methylene, CH_3 = methyl) and chemical shift. Carbon multiplicity was established by DEPT135 and/or HSQC experiments. Reported melting points of solids are uncorrected. IR spectra were recorded on an FT-IR spectrometer and reported in terms of frequency (cm^{-1}). Mass spectra were collected on an LCT spectrometer utilizing direct analysis in real time (DART) ionization.

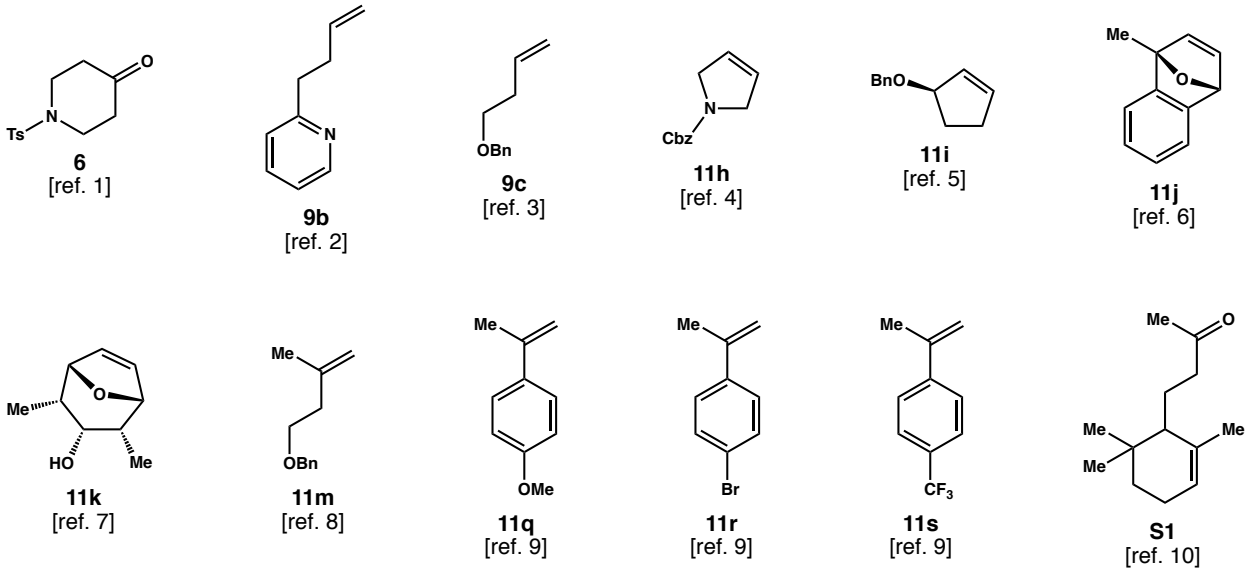


Figure S1. Starting materials prepared according to reported literature procedures.

2. Synthesis, Titration and Handling of Tebbe's Reagent (1).

Synthesis of $[Cp_2Ti(\mu_2\text{-}Cl)(\mu_2\text{-}CH_2)AlMe_2]$ (1). Following a modification of the procedure reported by Grubbs,[11] a flame-dried Schlenk tube (100 mL) equipped with a Teflon-coated stir bar was pumped into a glovebox. Under inert atmosphere (N_2), the flask was charged with bis(cyclopentadienyl)titanium dichloride (2.48 g, 10.0 mmol), capped with a yellow polyethylene stopper (Figure S2A), and removed from the glovebox. A 14/20 septa was used to seal the vessel and the flask was purged with a balloon of Ar using a vacuum line (Figure S2B). As shown in Figure S2C and D, the red solid was suspended in degassed PhMe (12 mL), protected from light, and treated with a solution of $AlMe_3$ (11 mL, 22 mmol) in PhMe (2.0 M) at ambient temperature. The resulting dark red slurry was maintained at ambient temperature for 60 h and then titrated as described below to establish the concentration of **1** (0.37 M, 85% yield). Solutions of **1** prepared in this way could be maintained on the benchtop ~120 h without complication.

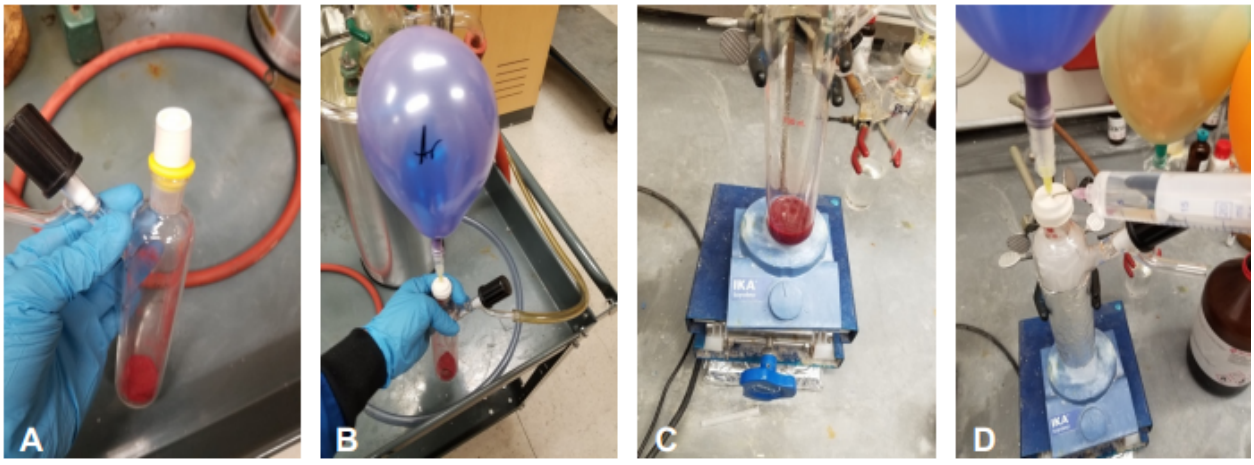


Figure S2. Tebbe Prep: (A) Schlenk tube charged with Cp_2TiCl_2 (red) and sealed with yellow PE cap/septa combination. (B) Solids placed under Ar using a Schlenk/vacuum line. (C) Stirred suspension of Cp_2TiCl_2

in degassed PhMe (12 mL). (D) The reaction vessel was protected from light and treated with 2.0 M solution of $AlMe_3$ in PhMe (commercial, used as received).

Titration of 1. The concentration of the solution of **1** prepared above was established in the following manner: A flame-dried Schlenk tube (25 mL) was charged with freshly distilled *p*-anisaldehyde (136 mg, 1.00 mmol). The reaction vessel was purged with nitrogen using a Schlenk line. The Schlenk tube was placed under a balloon of nitrogen and THF (0.3 mL) was added via syringe. The resulting solution was cooled to –40 ˚C and maintained for 15 min. An aliquot for the solution of **1** (0.60 mL, 0.260 mmol *theoretically*) was added stream-wise via syringe and the slurry was maintained at –40 ˚C (Figure S3A). After 15 min, the reaction mixture was treated with 0.1 M aq. NaOH (0.03 mL) at –40 ˚C and warmed to rt over 30 min. The resulting suspension was filtered over Celite (150 mg) using a pipette (Figure S3B) and concentrated under reduced pressure. The unpurified residue was analyzed by ^{1}H NMR ($CDCl_3$, 400 MHz) to determine the ratio of unreacted *p*-anisaldehyde and *p*-methoxystyrene. A representative ^{1}H NMR spectra is shown in Figure S3. The ratio of *p*-anisaldehyde (**AA**) to *p*-methoxystyrene (**MS**) (3.44:1, 22% conversion vs. 26% theoretical) was determined by integrating the signals at 7.84 ppm and 7.35 ppm, respectively.

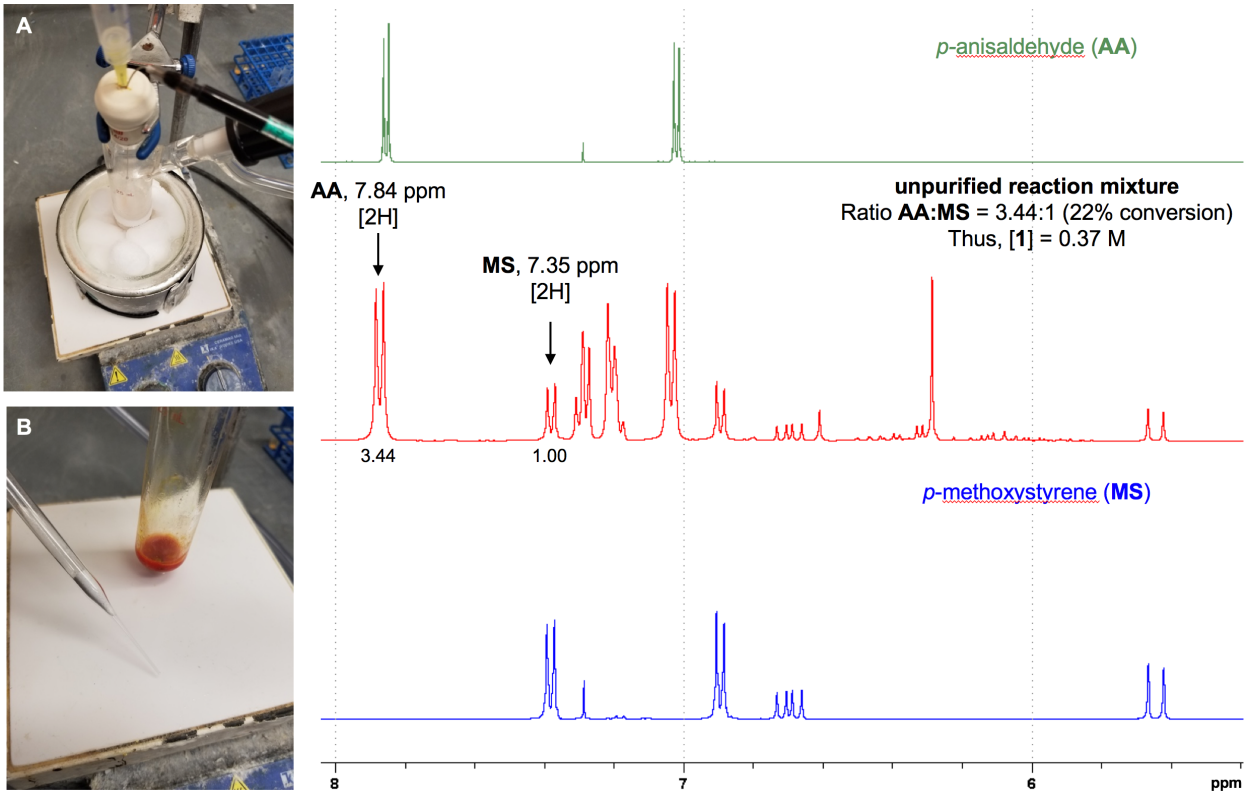


Figure S3. Titration Protocol: (A) General reaction setup. (B) Filtration of the unpurified reaction mixture. (C) Representative ^{1}H NMR of the unpurified reaction mixture to determine [**1**].

Handling solutions of 1. We observed that solutions of **1** prepared as described above could be maintained under a balloon of argon at ambient temperature for 5–7 days without issue. It was helpful to replace the argon balloon every 48 h to prolong the lifetime of the reagent. As shown in Figure S4, aged solutions of **1** turn noticeably brown. These old solutions contained active reagent ([**1**] ~ 0.25 M), but gave poor results in our hands. The optimal concentration of **1** was 0.4–0.3 M

based on the titration protocol above. **Caution** – solutions of **1** contain residual $AlMe_3$ and react vigorously with water.

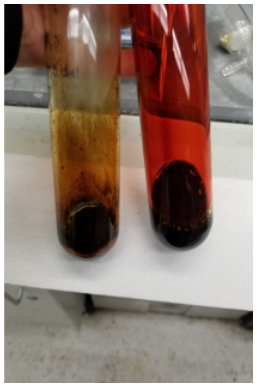
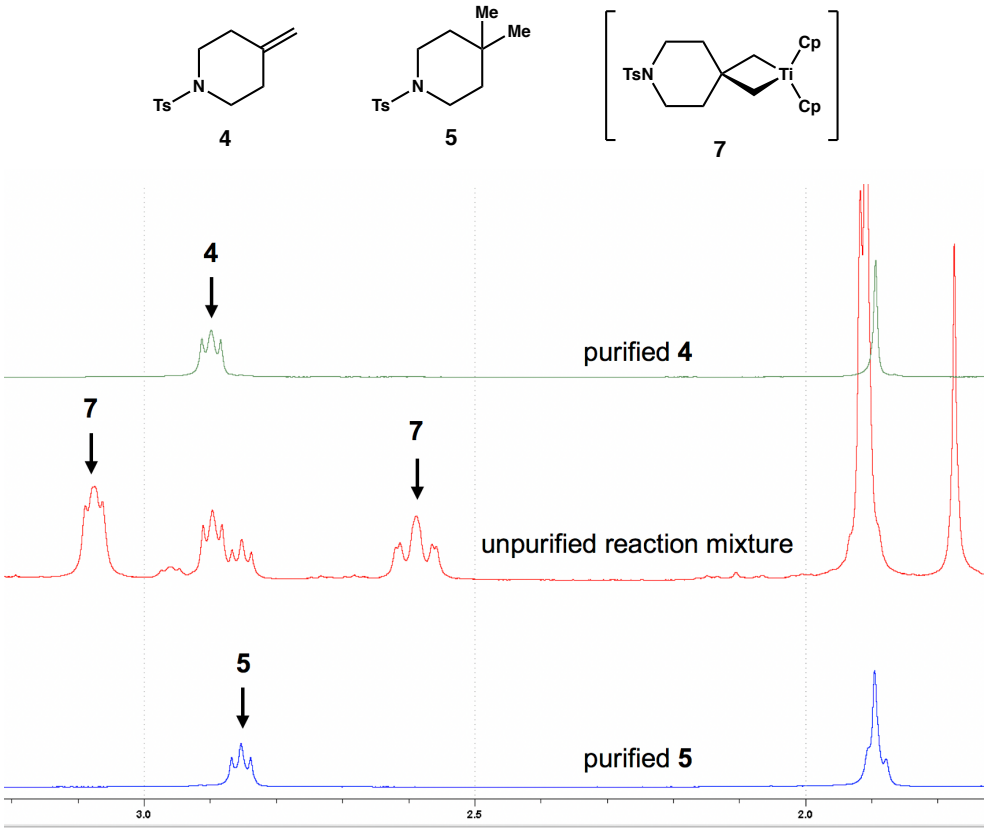


Figure S4. Active solution of **1** in PhMe (right) versus an aged solution of **1** in PhMe (left).

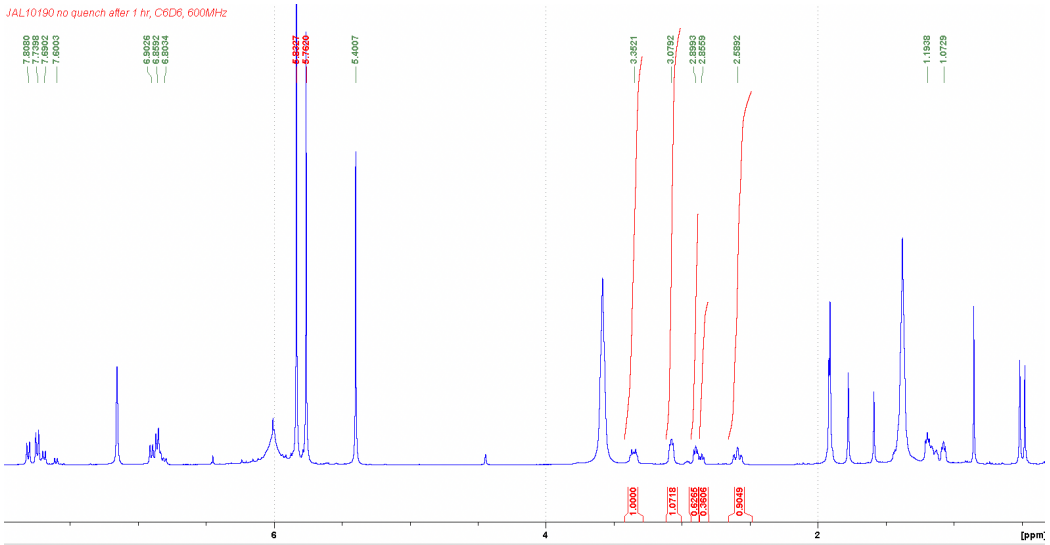
Cost analysis for the synthesis of 1. Titanocene dichloride (Cp_2TiCl_2, 1271-19-8) was purchased from TCI for $100 (50 g, 200 mmol). A 2.0 M solution of trimethylaluminum in PhMe was purchased from Sigma Aldrich for $378 (1 L, 2000 mmol). Based on these prices, the 0.37 M solution prepared above (8.5 mmol, 85% yield) cost $9.40 in reagents. Therefore, prepared as described here, reagent **1** costs ~$1.1/mmol. We note that the cost will vary depending on the choice of vendor and quantity of reagents ordered.

3. Temperature Profile of Titanacyclobutanes

Detection of transient titanacyclobutane 7 by ^{1}H NMR (C_6D_6).



Unpurified reaction mixture in C_6D_6 (expanded window)



Thermal stability of titanacyclobutane 7 *in situ*.

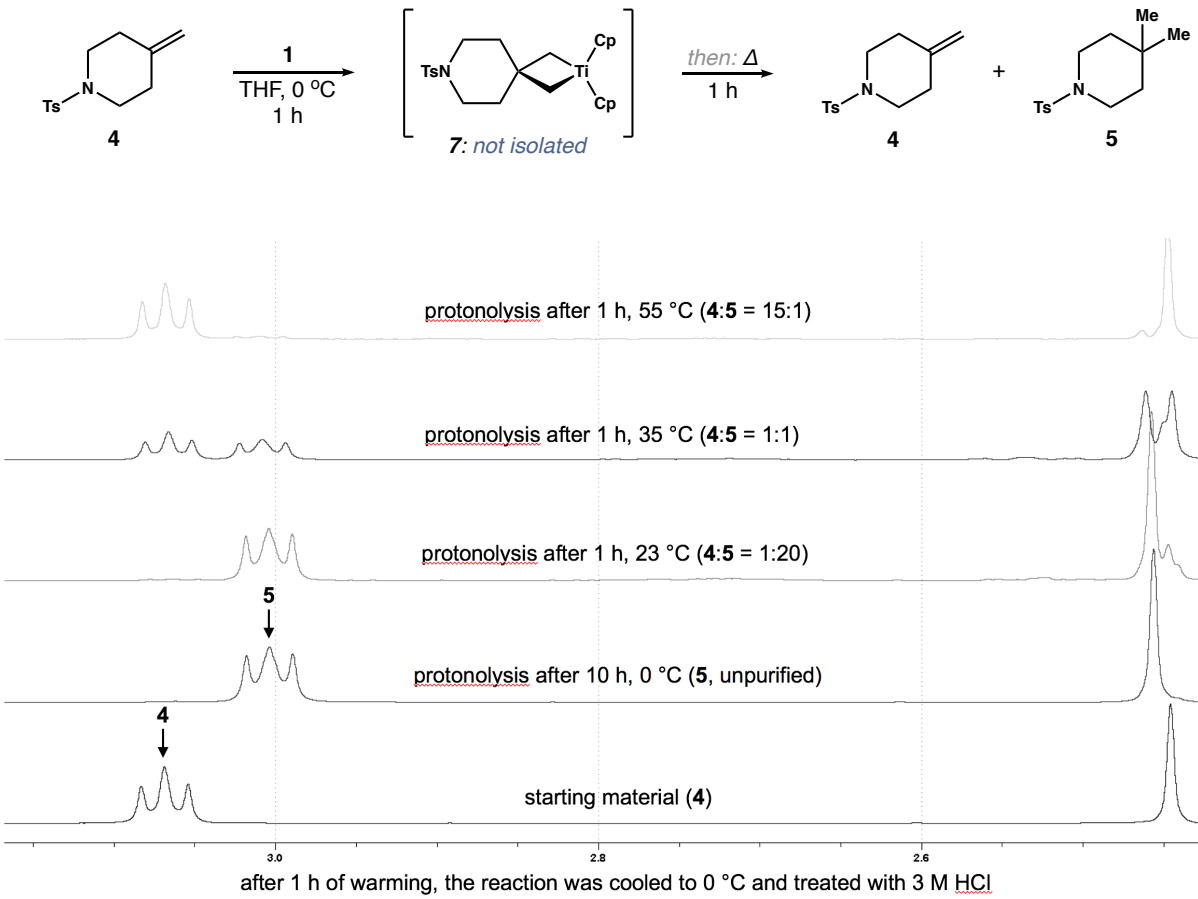
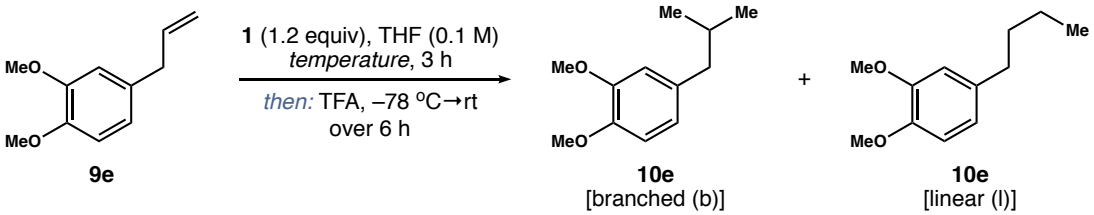


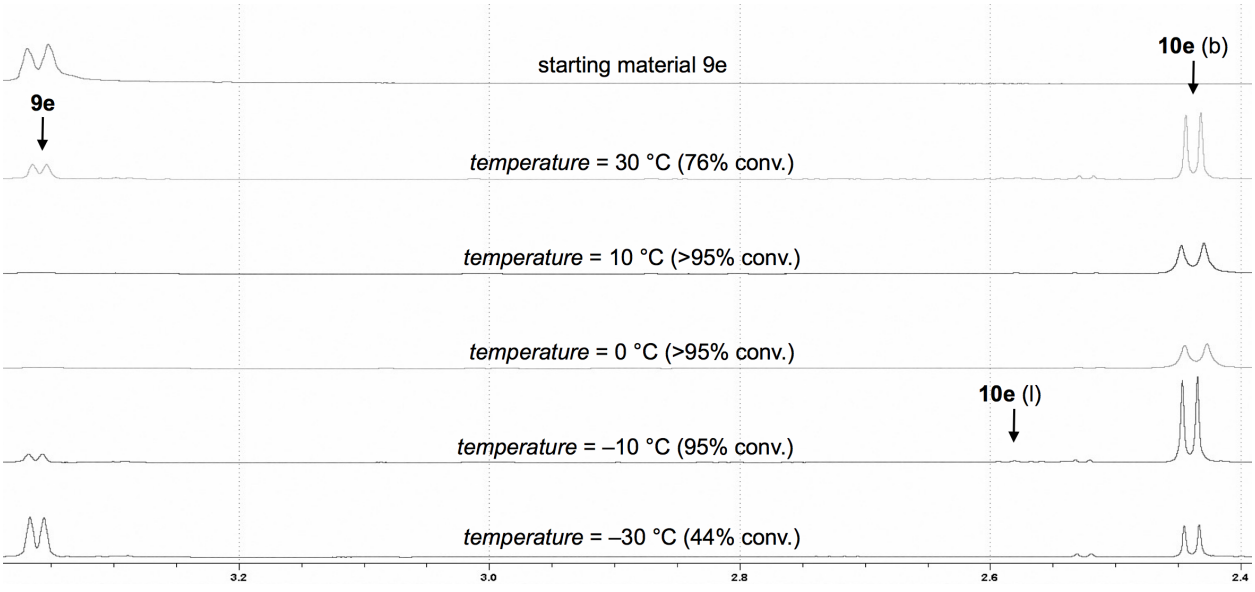
Figure S5. Supporting data for Scheme 1. The stability of titanacyclobutane **7** *in situ* at various temperatures was determined by ^{1}H NMR spectroscopy. As a representative experiment: **4** was reacted with 1.2 equiv **1** in THF (0.1 M) at 0 °C. After 1 h, the reaction mixture was warmed to rt. After 1 h, the reaction was cooled to 0 °C and treated sequentially with 3 M HCl (2 mL) and EtOAc (2 mL). The resulting slurry was rapidly stirred at 0 °C for 3 h, then warmed to rt and transferred to a separatory funnel with EtOAc (10 mL). The organic layer was washed with saturated aq. $NaHCO_3$ (2 x 10 mL) dried over $MgSO_4$, filtered, and concentrated. The unpurified residue was then analyzed by NMR.

Impact of reaction temperature of conversion and branch/linear (b/l) ratio.

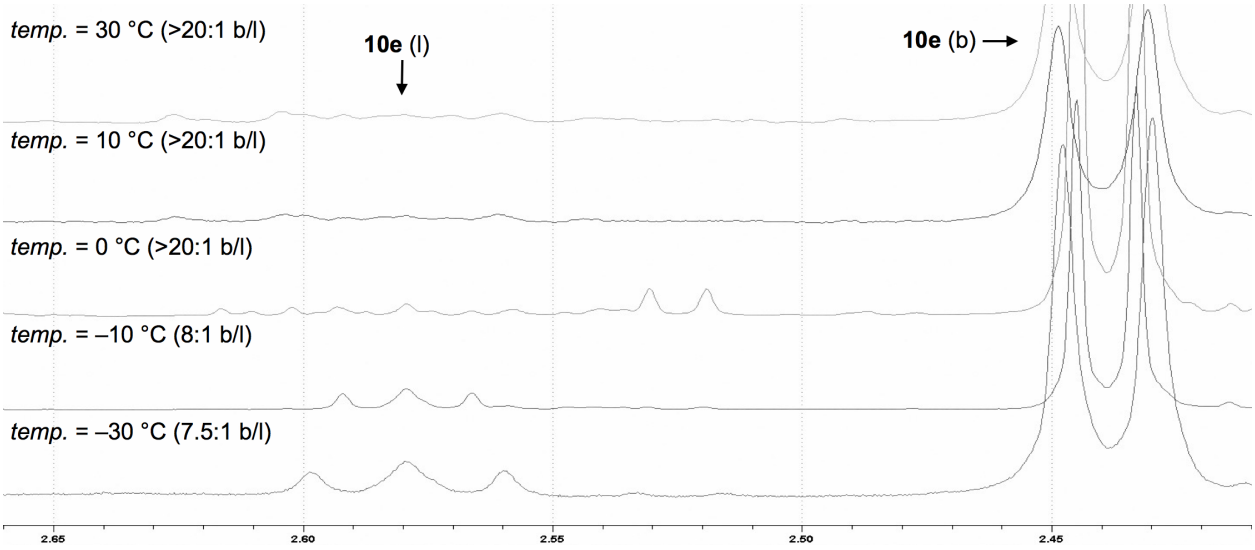


temperature (°C)	conversion to **10e** (%)	b/l ratio (^{1}H NMR)
–40	11	--
–30	44	7.5:1
–20	59	8:1
–10	95	8:1
0	99	>20:1
10	99	>20:1
23	88	>20:1
30	76	>20:1
40	46	>20:1
50	35	--

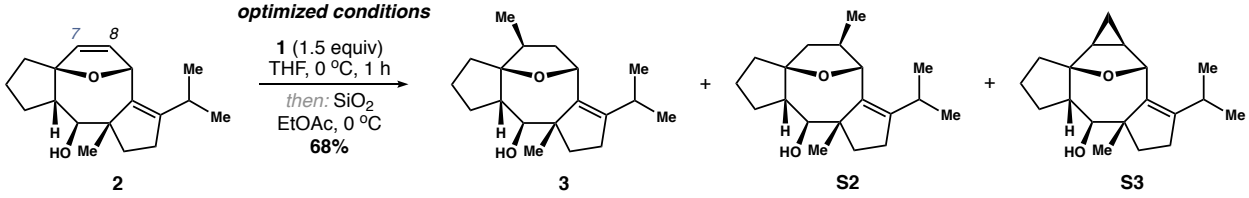
a. *reaction conversion as a function of temperature (^{1}H NMR spectroscopy, 600 MHz, $CDCl_3$)*



b. *regioselectivity as a function of temperature (^{1}H NMR spectroscopy, 600 MHz, $CDCl_3$)*

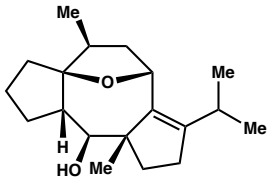


4. Hydromethylation of fusicoccane substrate 2.



deviation from optimal conditions	observations	3:S2:S3 (by NMR)	**3** (% yield)
none	**>95% conversion**	**21:1:0**	**68**
1.0 equiv. **1**	~20% conversion	1:0:20	n.d.
aq. HCl (3 M) as proton source	complex, intractable mixture	n.d.	n.d.
reaction temp = 50 °C	no rxn	n.d.	n.d.
reaction temp = 23 °C	47% conversion	5:1:3	8
reaction temp = –20 °C	87% conversion	19:1:0	46
PhMe/THF (2:1)	85% conversion	19:2:1	61
PhMe, 1 equiv DMAP	63% conversion	2:1:1	29

5. Experimental Procedures and Characterization Data



3: $C_{19}H_{30}O_2$
MW: 290.45

C3-*desmethyl*-fusioccane 3. A flask charged with a solution of **2**[12] (55 mg, 0.20 mmol) in THF (2.0 mL) was cooled to 0 °C. Separately, a Schlenk tube (25 mL) was charged with a 0.36 M (PhMe) solution of **1** (0.11 mL, 0.30 mmol) and concentrated under reduced pressure (ca. 1.0 Torr, 30 min). The Schlenk tube was backfilled with nitrogen and cooled to 0 °C. The solution of **2** in THF (2.0 mL) was added to the Schlenk flask via syringe and the reaction was maintained at 0 °C for 1 h. The reaction mixture was then transferred via cannula to a stirred suspension of SiO_2 (2 g) in EtOAc (5 mL) maintained at 0 °C. The resulting slurry was rapidly stirred for 6 h then filtered and the filter cake was washed with EtOAc (3 x 5 mL). The combined organic extracts were concentrated under reduced pressure. The resulting residue was purified by flash chromatography (SiO_2, 5:1 hexanes/Et_2O) to afford **3** (41 mg, 0.14 mmol, 68% yield) as a yellow oil: **^{1}H NMR** (600 MHz, $CDCl_3$) δ_H 4.61 (d, J = 7.6 Hz, 1H), 3.48 (d, J = 10.7 Hz, 1H), 2.52 (sept, J = 6.8 Hz, 1H), 2.27–2.18 (m, 3H), 2.18–2.10 (m, 2H), 2.02–1.95 (m, 2H), 1.94–1.89 (m, 1H),1.88–1.82 (m, 1H), 1.81–1.70 (m, 5H), 1.60–1.51 (m, 3H), 1.37–1.29 (m, 2H) 1.09 (s, 3H), 1.05 (d, J = 6.9 Hz, 3H), 0.98 (d, J = 6.9 Hz, 3H), 0.95 (d, J = 6.8 Hz, 3H); **^{13}C NMR** (150 MHz, $CDCl_3$) δ_c **C:** 148.7, 148.2, 117.4, 77.4; **CH:** 141.3, 113.4, 106.5; **CH_2:** 36.7, 24.3; **CH_3:** 26.1, 9.9; **IR** (thin film): 3443, 2930, 1466 cm^{-1}. **HRMS-DART** (*m*/z) $[M+H]^+$ calculated for $C_{19}H_{31}O_2$ = 291.2324; found 291.2302. The relative stereochemistry of structure **3** was confirmed by X-ray crystallography. These data have been deposited in the Cambridge Crystalographic Data Center (CCDC) under **Deposition Number 2059774**.

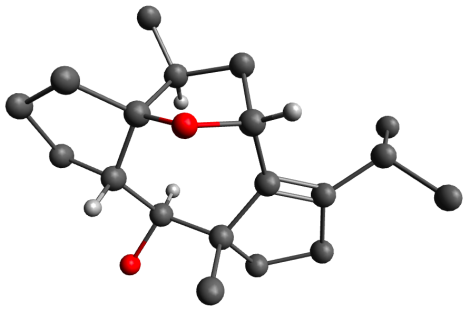
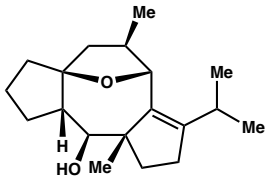
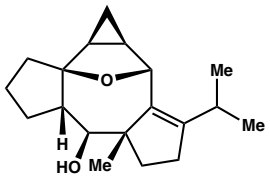


Figure S3. X-ray crystal structure of **3** [CCDC 2059774].



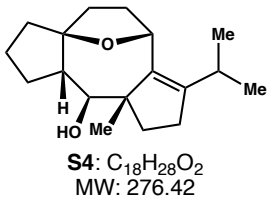
S2: $C_{19}H_{30}O_2$
MW: 290.45

C8-regioisomer of 3 (S2). An analytical sample of **S2** was prepared by preparatory TLC (9:1 hexanes/Et_2O): **^{1}H NMR** (600 MHz, $CDCl_3$) δ_H 4.06 (s, 1H), 3.33 (d, J = 10.5 Hz, 1H), 2.50 (sept, J = 6.7 Hz, 1H), 2.29–2.19 (m, 2H), 2.20–2.09 (m, 4H), 2.05–1.92 (m, 4H), 1.81–1.69 (m, 4H), 1.68–1.51 (m, 6H), 1.39–1.31 (m, 2H), 1.15 (d, J = 6.9 Hz, 3H), 1.10 (s, 3H), 1.00 (d, J = 6.7 Hz, 3H), 0.97 (d, J = 6.7 Hz, 3H); **^{13}C NMR** (150 MHz, $CDCl_3$) δ_C **C:** 148.7, 148.2, 117.4, 77.4; **CH:** 141.3, 113.4, 106.5; **CH_2:** 36.7, 24.3; **CH_3:** 26.1, 9.9; **IR** (thin film): 3443, 2930, 1466 cm^{-1}. **HRMS-DART** (*m*/z) $[M+H]^+$ calculated for $C_{19}H_{31}O_2$ = 291.2324; found 291.2302.



S3: $C_{19}H_{28}O_2$
MW: 288.43

Cyclopropyl fusicoccane analog S3. A Schlenk flask was added Et_2Zn (1.2 mL, 1.0 mmol, 0.83 M in hexanes) and 0.26 mL CH_2Cl_2. The reaction was cooled to 0 °C and a solution of freshly distilled CF_3CO_2H[2] (0.08 mL, 1 mmol) in 0.2 mL of CH_2Cl_2 was added via syringe over 5 min. The mixture was then stirred at 0 °C for 20 min during which time a white precipitate formed. The reaction mixture was warmed to 35 °C in a pre-heated oil bath for 10 min to create a homogenous solution, then cooled back down to 0 °C for 5 min. A solution of CH_2I_2 (0.08 mL, 1 mmol) in 0.2 mL of CH_2Cl_2 was added to the reaction at 0 °C over 5 min. The reaction mixture was maintained at 0 °C for 20 min, then a solution of **2** (0.055 g, 0.20 mmol) in CH_2Cl_2 (1.2 mL) was added over 5 min. The reaction was warmed to rt and maintained for 4 h. The reaction was then diluted with EtOAc (5 mL), transferred to a separatory funnel, and washed with sat. $NaHCO_3$ (10 mL). The aqueous layer was extracted EtOAc (3 x 10 mL). The combined organic extracts were washed sat. sodium thiosulfate (20 mL), then with brine (20 mL). The organic layer was then dried over Na_2SO_4, filtered and concentrated under reduced pressure. No further purification was necessary. This procedure returned an intractable mixture of **S3** and **2** (9:1, 0.055 g). Characterization data for **S3** = **^{1}H NMR** (400 MHz, $CDCl_3$) δ_H 4.81 (br s, 1H), 3.58 (dd, *J* = 10.8, 4.5 Hz, 1H), 2.66 (hep, *J* = 6.8 Hz, 1H), 2.29–2.12 (m, *3*H), 2.02–1.95 (m, 1H), 1.83–1.69 (m, 5H), 1.50–1.45 (m, 1H), 1.26 (d, *J* = 4.5 Hz, 1H), 1.22–1.17 (m, 1H), 1.05 (s, 3H), 1.02 (d, *J* = 6.8 Hz, 3H), 0.96 (d, *J* = 6.8 Hz, 3H), 0.52–0.47 (m, 1H), 0.39 (q, *J* = 4.3 Hz, 1H); **^{13}C NMR** (150 MHz, $CDCl_3$) δ_C C: 141.4, 141.3, 89.3, 54.9; CH: 79.2, 77.0, 49.9, 26.9, 25.2, 19.6; CH_2: 38.3, 33.3, 26.7, 25.1, 18.5, 6.8; CH_3: 21.2, 20.9,17.2**;** **IR** (thin film): 3481, 1447 cm^{-1}; **HRMS-DART** (m/z) $[M+H]^+$ calculated for $C_{19}H_{29}O_2$ = 289.2168; found 289.2173.



S4: $C_{18}H_{28}O_2$
MW: 276.42

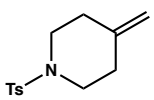
Net-hydrogenation product S4. An authentic sample of this compound was prepared by hydrogenation: A solution of $RhCl(PPh_3)_3$ (14 mg, 0.015 mmol) in PhH (0.9 mL) was treated with a solution of **2** (27 mg, 0.10 mmol) in PhH (2.4 mL). The reaction was equipped with a balloon of hydrogen gas and maintained at rt. After 1.5 h, an additional quantity of $RhCl(PPh_3)_3$ (14 mg, 0.015 mmol) was added. The reaction was continued at rt. After 12 h, the reaction was concentrated under reduced pressure. The resulting crude residue was purified by flash column chromatography (SiO_2; 4:1 hexanes/EtOAc) to afford **S4** (11 mg, 0.040 mmol, 40% yield) as a white foam; **^{1}H NMR** (600 MHz, $CDCl_3$) δ_H 5.04 (d, *J* = 7.3 Hz, 1H), 3.32 (d, *J* = 10.6 Hz, 1H), 2.54 (heptet, *J* = 6.7 Hz, 1H), 2.24–2.11 (m, 4H), 1.99–1.89 (m, 3H), 1.78–1.69 (m, 2H), 1.61–1.53 (m, 4H), 1.52–1.46 (m, 1H), 1.39–1.32 (m, 2H), 1.08 (s, 3H), 0.97 (d, *J* = 6.8 Hz, 3H), 0.94 (d, *J* = 6.8 Hz, 1H); **^{13}C NMR** (150 MHz, $CDCl_3$) δ_C C: 141.9, 141.7, 89.4, 55.6; CH: 80.9, 76.8, 50.3, 27.0; CH_2: 38.5, 38.1, 35.2, 29.6, 26.6, 25.4, 18.4; CH_3: 20.9, 20.7, 17.1; **IR** (thin film): 3438, 1466, 1453 cm^{-1}; **HRMS**-DART (m/z) $[M+NH_4]^+$ calculated for $C_{18}H_{32}O_2N$ = 294.2428; found 294.2422.

Substrate Synthesis

Alkenes **6, 11a, 11c**–**11g**, **11o** and **11p** were prepared via Wittig olefination of the corresponding ketones as described below.

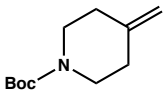
General procedure: A freshly titrated solution of *n*-BuLi (1.1 equiv) in THF was added dropwise to a stirred suspension of Ph_3PMeI (1.4 equiv) in Et_2O (0.5 M) at 0°C. The resulting solution was maintained at 0°C for 1 h, then treated with a solution of ketone (1 equiv) in Et_2O (1 M). The ketone was generally consumed within 1–6 h as judged by TLC of the unpurified reaction mixture. At this point, saturated aq. NH_4Cl (1 mL/mmol) was added via syringe and the reaction vessel was warmed to rt. The resulting slurry was added to a separatory funnel and extracted with Et_2O

(3 x 10 mL/mmol). The combined organic extracts were washed with water and brine, dried over $MgSO_4$, filtered, and concentrated under reduced pressure. The resulting residues were purified as indicated.



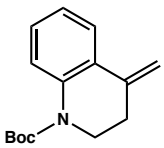
4: $C_{13}H_{17}NO_2S$
MW: 251.34

4-Methylene-1-tosylpiperdine (4). The title compound was prepared from **6** using the General Procedure. Purification by flash chromatography (SiO_2, 4:1 hexanes/EtOAc) afforded **4** (7.23 g, 28.8 mmol, 61% yield): **^{1}H NMR** (400 MHz, $CDCl_3$) δ_H 7.64 (d, J = 8.2 Hz, 2H), 7.31 (d, J = 7.9 Hz, 2H), 4.69 (s, 2H), 3.04 (t, J = 5.8 Hz, 4H), 2.42 (s, 3H), 2.30 (t, J = 5.8 Hz, 4H). All other characterization data was identical to previously reported values.[13]



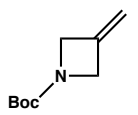
11a: $C_{11}H_{19}NO_2$
MW: 197.28

***tert*-Butyl-4-methylene-1-tosylpiperdine (11a).** The title compound was prepared from commercial 1-(*tert*-butoxycarbonyl)-4-piperdone [79099-07-3] using the General Procedure. Purification by flash chromatography (SiO_2, 10:1 hexanes/EtOAc) afforded **11a** (1.91 g, 9.60 mmol, 96% yield): **^{1}H NMR** (400 MHz, $CDCl_3$) δ_H 4.76, (s, 2H), 3.44 (t, J = 5.8 Hz, 4H), 2.20 (t, J = 5.7 Hz, 4H), 1.50 (s, 9H). All other characterization data was identical to previously reported values.[14]



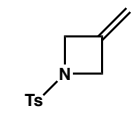
11c: $C_{15}H_{19}NO_2$
MW: 245.32

***tert*-Butyl-4-methylene-3,4-dihydroquinoline-1(*2H*)-carboxylate (11c).** The title compound was prepared from commercial *tert*-butyl-4-oxo-3,4-dihydroquinoline-1(2H)-carboxylate [179898-00-1] using the General Procedure. Purification by flash chromatography (SiO_2, 10:1 hexanes/EtOAc) afforded **11c** (335 mg, 1.37 mmol, 68% yield): **^{1}H NMR** (400 MHz, $CDCl_3$) δ_H 7.68 (d, J = 8.0 Hz, 1H), 7.62 (d, J = 8.2 Hz, 1H), 7.23 (t, J = 8.4 Hz, 1H), 7.07 (t, J = 8.2 Hz, 1H), 5.64 (s, 1H), 5.00 (s, 1H); 3.82 (t, J = 6.0 Hz, 2H), 2.74 (t, J = 6.0 Hz, 2H), 1.55 (s, 9H). **^{13}C NMR** (150 MHz, $CDCl_3$) δ_C **C**: 153.6, 139.0, 138.3, 81.1; CH: 127.8, 127.3 124.8, 124.2, 123.9; **CH_2**: 109.3, 44.2, 32.8; **CH_3**: 28.5; **IR** (thin film): 1365, 1694, 2977 cm$^{-1;}$ **HRMS-DART** (m/z) $[M+H]^+$ calculated for $C_{15}H_{19}NO_2H$ = 246.1494; found 246.1494.



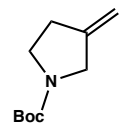
11d: $C_9H_{15}NO_2$
MW: 169.22

***tert*-Butyl-3-methyleneazetidine-1-carboxylate (11d).** The title compound was prepared from commercial 1-Boc-3-azetidinone [398489-26-4] using the General Procedure. Purification by flash chromatography (SiO_2, 9:1 hexanes/EtOAc) afforded **11d** (247 mg, 1.64 mmol, 82% yield): **^{1}H NMR** (400 MHz, $CDCl_3$) δ_H 5.00 (m, J = 2.4 Hz, 2H), 4.50 (t, J = 2.5 Hz, 4H), 1.47 (s, 9H). All other characterization data was identical to previously reported values.[15]



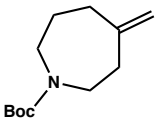
11e: $C_{11}H_{13}NO_2S$
MW: 223.29

3-Methylene-1-tosylazetidine (11e). The title compound was prepared from commercial *N*-tosyl-3-azetidinone [76543-27-6] using the General Procedure. Purification by flash chromatography (SiO_2, 7:1 hexanes/EtOAc) afforded **11e** (328 mg, 1.47 mmol, 76% yield): **^{1}H NMR** (400 MHz, $CDCl_3$) δ_H 7.78 (d, J = 8.4 Hz, 2H), 7.39 (d, J = 8.0 Hz, 2H), 4.94 (app quint, J = 2.5 Hz, 2H), 4.41 (t, J = 2.4 Hz, 4H), 2.48 (s, 3H); **^{13}C NMR** (150 MHz, $CDCl_3$) δ_C **C**: 144.1, 134.4, 131.2; **CH**: 129.7, 128.1; **CH_2**: 108.2, 59.6; **CH_3**: 21.3; **IR** (thin film): 1152, 1338, 2967 cm^{-1}; **HRMS-DART** (m/z) $[M+H]^+$ calculated for $C_{11}H_{14}NO_2S$ = 224.0745; found 224.0737.



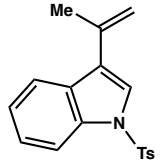
11f: $C_{10}H_{17}NO_2$
MW: 183.25

***tert*-Butyl-3-methylenepyrrolidine-1-carboxylate (11f).** The title compound was prepared from commercial *N*-Boc-3-pyrrolidinone [101385-93-7] using the General Procedure. Purification by flash chromatography (SiO_2, 9:1 hexanes/Et_2O) afforded **11f** (217 mg, 1.18 mmol, 59% yield): **^{1}H NMR** (400 MHz, $CDCl_3$) δ_H 4.99–4.91 (m, 2H), 3.94 (s, 2H), 3.48 (t, *J* = 7.2 Hz, 3H), 2.57 (t, *J* = 7.3 Hz, 2H), 1.49 (s, 9H). All other characterization data was identical to previously reported values.[16]



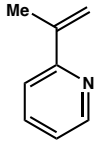
11g: $C_{12}H_{21}NO_2$
MW: 211.31

***tert*-Butyl-4-methyleneazepane-1-carboxylate (11g).** The title compound was prepared from commercial *N*-Boc-hexahydro-1H-azepin-4-one [188975-88-4] using the General Procedure. Purification by flash chromatography (SiO_2, 9:1 hexanes/Et_2O) afforded **11g** (513 mg, 2.43 mmol, 97% yield): **^{1}H NMR** (400 MHz, $CDCl_3$) δ_H 4.89–4.69 (m, 2H), 3.44–3.30 (m, 4H), 2.43 (br s, 2H), 2.25 (br s, 2H), 1.71–1.66 (m, 2H), 1.48 (s, 9H). All other characterization data was identical to previously reported values.[17]



11o: $C_{18}H_{17}NO_2S$
MW: 311.40

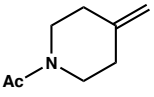
3-(Prop-1-en-2-yl)-1-tosyl-1*H*-indole (11o). The title compound was prepared from commercial 3-acetyl-*N*-(p-toluenesulfonyl)indole [104142-24-7] using the General Procedure. Purification by flash chromatography (SiO_2, 4:1 hexanes/EtOAc) afforded **11o** (737 mg, 2.37 mmol, 79% yield): **^{1}H NMR** (400 MHz, $CDCl_3$) δ_H 7.99 (dq, *J* = 0.7, 0.6 Hz 1H), 7.81 (d, *J* = 7.6 Hz, 1H), 7.77 (d, *J* = 8.3 Hz, 2H), 7.56 (s, 1H), 7.36-7.20 (m, 5H), 5.51 (s, 1H), 5.22 (t, *J* = 1.4 Hz, 1H), 2.33 (s, 3H), 2.17 (q, *J* = 0.7 Hz, 3H); **^{13}C NMR** (150 MHz, $CDCl_3$) δ_C **C**: 145.1, 136.3, 135.7, 135.2, 129.0, 124.0; **CH**: 129.9, 126.9, 124.7, 123.6, 123.5, 121.4, 113.7; **CH_2**: 113.8; **CH_3**: 23.1, 21.5; **IR** (thin film): 1173, 1370, 1597 cm^{-1}; **HRMS-DART** (m/z) $[M+H]^+$ calculated for $C_{18}H_{18}NO_2S$ = 312.1058; found 312.1068.



11p: C_8H_9N
MW: 119.17

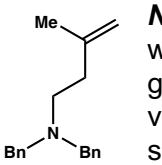
2-(prop-1-en-2-yl)pyridine (11p). The title compound was prepared from commercial 2-acylpyridine [1122-62-9] using the General Procedure. Purification by flash chromatography (SiO_2, 99:1 pentane/Et_2O) afforded **11g** (388 mg, 3.24 mmol, 81% yield): **^{1}H NMR** (400 MHz, $CDCl_3$) δ_H 8.59 (ddd, *J* = 4.8, 1.8, 0.9, 1H), 7.65 (ddd, *J* = 15.5, 1.8, 1.8, 1H), 7.46–7.51 (m, 1H), 7.16 (ddd, *J* = 7.4, 4.8, 1.1, 1H), 5.85 (d, *J* = 0.7, 1H), 5.30 (app q, *J* = 1.4, 1H), 2.22 (d, *J* = 0.7, 3H). All other characterization data was identical to previously reported values.[18]

Other Substrates



11b: $C_8H_{13}NO$
MW: 139.20

1-(4-methylenepiperidin-1-yl)ethan-1-one (11b). The title compound was prepared from **11a** using the procedure described by Li.[19] **^{1}H NMR** (400 MHz, $CDCl_3$) δ_H 4.78, (s, 2H), 3.59 (t, *J* = 5.9 Hz, 2H), 3.43 (t, *J* = 5.8 Hz, 2H), 2.22 (dt, J = 6.2 Hz, 4H), 2.12 (s, 3H). All characterization data was identical to previously reported values.[19]

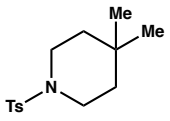


11n: $C_{19}H_{23}N$
MW: 265.40

***N,N*-Dibenzyl-3-methylbut-3-en-1-amine (11n).** Sodium iodide (1.49 g, 10.0 mmol) was added in a single portion to a solution of (*O*-tosyl)-3-methyl-3-butene-1-ol (1.20 g, 5.00 mmol) in acetone (10 mL) at rt. Dibenzylamine (1.0 mL, 10 mmol) was added via syringe and the reaction mixture was heated to 70 °C. After 18 h, the resulting slurry was cooled to rt and filtered through a pad of Celite. The filter cake was washed with acetone (3 x 10 mL) and the combined filtrate fractions were concentrated under reduced pressure. The resulting crude residue was purified by flash chromatography (SiO_2, benzene) to afford **11n** (1.09 g, 4.11, 82% yield) as a colorless oil: **^{1}H NMR** (400 MHz,

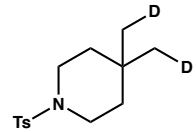
$CDCl_3$) δ_H 7.40–7.25 (m, 10H), 4.73 (br s, 1H), 4.66 (br s), 1H), 3.61 (s, 4H), 2.59 (t, J = 6.7 Hz, 2H), 2.28 (t, J = 6.7 Hz) 1.63 (s, 3H); **^{13}C NMR** (150 MHz, $CDCl_3$) δ_C **C**: 144.4, 140.0; **CH**: 128.9, 128.3, 126.9; **CH_2**: 111.0, 58.2, 51.8, 35.4; **CH_3**: 22.5; **IR** (thin film): 3051, 2928, 1097 cm^{-1}; **HRMS-DART** (m/z) $[M+H]^+$ calculated for $C_{19}H_{24}NO$ = 266.1908; found 266.1909.

Data for structures in Table 1 and Scheme 1



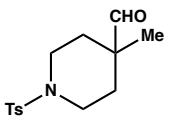
5: $C_{14}H_{21}NO_2S$
MW: 267.39

4,4-Dimethyl-1-tosylpiperdine (5). Gram-scale procedure: A two-neck flask equipped with a Schlenk adapter was flushed with nitrogen and then charged with a solution of **1** (0.36 M in PhMe, 13.2 mL, 4.78 mmol). Under a positive pressure of nitrogen, the flask was equipped with a distillation head. PhMe was removed by distillation and trace PhMe was removed under vacuum (0.1 torr, 30 min). The flask was backfilled with nitrogen and cooled to 0 °C. After 10 min, a pre-cooled (0 °C) solution of **4** (1.00 g, 3.98 mmol) in THF (4.0 mL) was added stream-wise via syringe. The red solution was maintained at 0 °C for 1 h, then treated sequentially with HCl (20 mL of a 3.0 M in H_2O) and EtOAc (20 mL). After 1 h, the reaction mixture was warmed to rt. After 3 h, the mixture was transferred to a separatory funnel using EtOAc (50 mL) and washed with saturated $NaHCO_3$ (2 x 50 mL). The organic layer was dried over $MgSO_4$, filtered and concentrated. The crude residue was purified by flash chromatography (SiO_2, 4:1 hexanes:EtOAc) to provide **5** (947 mg, 3.54 mmol, 89% yield) as a colorless oil: **^{1}H NMR** (400 MHz, $CDCl_3$) δ_H 7.64, (d, J = 8.3 Hz, 2H), 7.31 (d, J = 8.0 Hz, 2H), 2.98 (app t, J = 5.7 Hz, 4H), 2.43 (s, 3H), 1.43 (app t, J = 5.8 Hz, 4H), 0.83 (s, 6H); **^{13}C NMR** (150 MHz, $CDCl_3$) δ_C **C**: 143.3, 133.3, 28.2; **CH**: 129.6, 127.6; **CH_2**: 42.6, 37.8; **CH_3**: 27.5, 21.5; **IR** (thin film): 1456, 1410, 2928 cm^{-1}. **HRMS-DART** (m/z) $[M+H]^+$ calculated for $C_{14}H_{22}NO_2S$ = 268.1371; found 268.1381.



5-*d*$_2$: $C_{14}H_{19}D_2NO_2S$
MW: 269.40

4,4-Bis(dimethyl-*d*)-1-tosylpiperdine (5-*d*$_2$). A solution of **1** (0.36 M in PhMe, 0.67 mL, 0.24 mmol) was concentrated in a Schlenk tube under reduced pressure (0.1 torr, 30 min). The flask as backfilled with nitrogen and cooled to 0 °C. After 10 min, a pre-cooled (0 °C) solution of **4** (50 mg, 0.20 mmol) in THF (2.0 mL) was added stream-wise via syringe. The red solution was maintained at 0 °C for 1 h, then treated sequentially with DCl (2.0 mL of a 3.0 M in D_2O) and EtOAc (4 mL). The reaction mixture was warmed to rt. After 3 h, the mixture was transferred to a separatory funnel using EtOAc (10 mL) and washed with saturated $NaHCO_3$ (2 x 10 mL). The organic layer was dried over $MgSO_4$, filtered and concentrated. The crude residue was purified by flash chromatography (SiO_2, 4:1 hexanes:EtOAc) to provide **5-*d*$_2$** (49 mg, 0.18 mmol, 90% yield): **^{1}H NMR** (400 MHz, $CDCl_3$) δ_H 7.67, (d, J = 8.3 Hz, 2H), 7.34 (d, J = 8.0 Hz, 2H), 3.00 (app t, J = 5.7 Hz, 4H), 2.46 (s, 3H), 1.46 (app t, J = 5.8 Hz, 4H), 0.83 (s, 4H); **HRMS-DART** (m/z) $[M+H]^+$ calculated for $C_{14}H_{20}D_2NO_2S$ = 270.1527; found = 270.1544. The percent deuterium incorporation was determined by integration of distinct signals at 0.81 ppm (**5**) and 0.79 ppm (**5-*d*$_2$**). This analysis revealed a >90% deuterium incorporation.



8: $C_{14}H_{19}NO_3S$
MW: 281.37

4-Methyl-1-tosylpiperdine-4-carbaldehyde (8). A solution of **1** (0.36 M in PhMe, 1.67 mL, 0.60 mmol) was concentrated in a Schlenk tube under reduced pressure (0.1 torr, 30 min). The flask as backfilled with nitrogen and cooled to 0 °C. After 10 min, a pre-cooled solution of **4** (126 mg, 0.50 mmol) in THF (5.0 mL) at 0 °C was added stream-wise via syringe. The red solution was maintained at 0 °C for 1 h, then purged with O_2. The reaction mixture was maintained under a balloon of O_2 at 0 °C. After 3 h, the reaction was treated with 3 M aq. HCl (5 mL) and the stirred slurry was warmed to rt. After 5 h, the reaction mixture was transferred to a separatory funnel using EtOAc (10 mL) and washed with saturated aq. $NaHCO_3$ (2 x 10 mL) and brine (10 mL). The organic layer

was separated, dried over $MgSO_4$, filtered and concentrated under reduced pressure. Analysis of the unpurified reaction mixture by ^{1}H NMR spectroscopy reveled ~42% conversion of **4** to **8**, alongside several other intractable impurities and net hydromethylation product **5**. An analytical sample of **8** (23 mg, 0.08 mmol, 16% yield) was prepared by flash chromatography (SiO_2, 2:1 hexanes/EtOAc): **^{1}H NMR** (400 MHz, $CDCl_3$) δ_H 9.32 (s, 1H), 7.59 (d, *J* = 8.1 Hz, 2H), 7.30 (d, *J* = 8.1 Hz, 1H), 3.43 (m, 2H), 2.51 (td, *J* = 3.2, 2.9, 2.0 Hz, 2H), 2.42 (s, 3H), 2.04 (m, 2H), 1.60 (td, *J* = 4.3, 3.5, 3.4 Hz, 2H), 1.0q (s, 3H); **^{13}C NMR** (150 MHz, $CDCl_3$) δ_C **C**: 143.8, 133.0, 44.4; **CH**: 204.5; **CH_2**: 43.2, 31.4; **CH_3**: 129.7, 127.6, 21.7, 21.5; **IR** (thin film): 1161, 1721, 2924 cm^{-1}; **HRMS-DART** (m/z) $[M+H]^+$ calculated for $C_{14}H_{20}NO_3S$ = 282.1164; found 282.1151.

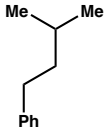
General Procedure: A solution of **1** (ca. 0.36 M in PhMe, 1.2 equiv) was concentrated in a Schlenk tube under reduced pressure (0.1 torr, 30 min). The flask as backfilled with nitrogen and cooled to 0 °C. After 10 min, a pre-cooled solution of alkene (1 equiv) in THF (0.1 M) at 0 °C was added stream-wise via syringe. The resulting red solution was maintained at 0 °C for the indicated amount of time, then treated with a proton source following one of the workup procedures listed below:

Workup A (HCl): Following the indicated reaction time, the reaction mixture was transferred via cannula to a cooled slurry of 3 M aq. HCl (5 mL/mmol alkene) and EtOAc (5 mL/mmol alkene) maintained at –10 °C. The resulting rapidly stirred slurry was allowed warm to rt over 6 h, then added to a solution of saturated aq. $NaHCO_3$ (10 mL/mmol of alkene). The resulting slurry was rapidly stirred for 4 h. A gradual color change from red to yellow to colorless indicated breakdown of titanium & aluminum impurities. The slurry was added to a separatory funnel and extracted with EtOAc (2 x 10 mL). The combined organic extracts were washed with brine (25 mL), dried over $MgSO_4$ and concentrated under reduced pressure. The resulting crude reside was then purified as indicated.

Workup B (SiO_2): Following the indicated reaction time, the reaction mixture was transferred via cannula to a cooled slurry of SiO_2 (3 g/mmol alkene) and EtOAc (3 mL/mmol alkene) maintained at –10 °C. The resulting rapidly stirred slurry was allowed warm to rt over 6 h, then filtered over a pad of celite. The filter cake was washed with EtOAc (2 x 5 mL). The combined organic extracts were added to a solution of saturated aq. $NaHCO_3$ (10 mL/mmol of alkene). The resulting slurry was rapidly stirred for 4 h. A gradual color change from red to yellow to colorless indicated breakdown of titanium & aluminum impurities. The slurry was added to a separatory funnel and extracted with EtOAc (2 x 10 mL). The combined organic extracts were washed with brine (25 mL), dried over $MgSO_4$ and concentrated under reduced pressure. The resulting crude reside was then purified as indicated.

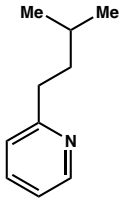
Workup C (TFA): Following the indicated reaction time, the reaction mixture was cooled to –78 °C and TFA (15 equiv) was added via syringe. The reaction mixture was maintained at –78 °C for 15 min, then allowed to warm to rt over the course of 6 h. The resulting mixture was exposed to air and added to a solution of saturated aq. $NaHCO_3$ (10 mL/mmol of alkene). The resulting slurry was rapidly stirred for 4 h. A gradual color change from red to yellow to colorless indicated breakdown of titanium & aluminum impurities. The slurry was added to a separatory funnel and extracted with EtOAc (2 x 10 mL). The combined organic extracts were washed with brine (25 mL), dried over $MgSO_4$ and concentrated under reduced pressure. The resulting crude reside was then purified as indicated.

Data for structures in Scheme 2



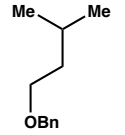
10a: $C_{11}H_{16}$
MW: 148.25

Isopentylbenzene (10a). The title compound was prepared from **9a** (44 mg, 0.33 mmol) following the General Procedure. After 1 h, the reaction was completed using Workup A. The resulting crude residue was purified by flash chromatography (SiO_2, hexane) to afford **10a** (35 mg, 0.23 mmol, 71% yield, >20:1 b/l ratio) as a colorless oil: **^{1}H NMR** (400 MHz, $CDCl_3$) δ_H 7.38–7.26 (m, 2H), 7.25–7.17 (m, 3H), 2.64 (app t, J = 8.0 Hz, 2H), 1.62 (app. sept., J = 6.7 Hz, 1H), 1.53 (q, J = 7.9 Hz, 2H), 0.96 (d, J =6.4 Hz, 6H). All other characterization was identical to reported values.[20]



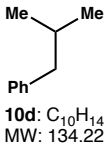
10b: $C_{10}H_{15}N$
MW: 149.24

2-Isopentylpyridine (10b). The title compound was prepared from **9b** (67 mg, 0.50 mmol) following the General Procedure. After 3 h, the reaction was completed using Workup A. The resulting crude residue was purified by flash chromatography (SiO_2, 10:1 hexanes/EtOAc) to afford **10b** (63 mg, 0.42 mmol, 71% yield, 6:1 b/l ratio) as a yellow oil: **^{1}H NMR** (400 MHz, $CDCl_3$) **major isomer** δ_H 8.51 (d, J = 4.1 Hz, 1H), 7.56 (t, J = 7.5 Hz, 1H), 7.13 (d, J = 7.1 Hz, 1H), 7.07 (t, J = 5.6 Hz, 1H), 2.81–2.72 (m, 2H), 1.65–1.55 (m, 3H), 0.94 (d, J = 5.9 Hz, 6H). **minor isomer** δ_H 8.51 (d, J = 4.1 Hz, 1H), 7.56 (t, J = 7.5 Hz, 1H), 7.13 (d, J = 7.1 Hz, 1H), 7.07 (t, J = 5.6 Hz, 1H), 2.81–2.72 (m, 2H), 1.79–1.67 (m, 2H), 1.65–1.55 (m, 2H), 1.37–1.30 (m, 2H), 0.88 (t, J = 6.6 Hz, 3H). All other characterization was identical to reported values.[21]



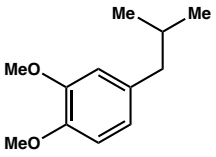
10c: $C_{12}H_{18}O$
MW: 178.28

Benzyl isoamyl ether (10c). The title compound was prepared from **9c** (82 mg, 0.50 mmol) following the General Procedure. After 1 h, the reaction was completed using Workup A. The resulting crude residue was purified by flash chromatography (SiO_2, 10:1 hexane/Et_2O) to afford **10c** (46 mg, 26 mmol, 52% yield, >20 b/l ratio) as a yellow oil: **^{1}H NMR** (400 MHz, $CDCl_3$) δ_H 7.39–7.33 (m, 4H), 7.33–7.29 (m, 1H), 4.53 (s, 2H), 3.52 (d, J = 6.7 Hz, 2H), 1.76 (app. sept., J = 6.7 Hz, 1H), 1.54 (q, J = 6.7 Hz, 2H), 0.93 (d, J = 6.6 Hz, 6H). All other characterization was identical to reported values.[22]



10d: $C_{10}H_{14}$
MW: 134.22

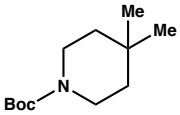
Isobutylbenzene (10d). The title compound was prepared from **9d** (0.07 mL, 0.50 mmol) following the General Procedure. After 6 h, the reaction was completed using Workup A. The resulting crude residue was purified by flash chromatography (SiO_2, 20:1 pentanes/Et_2O) to afford **10d** (52 mg, 0.39 mmol, 78% yield) as a colorless oil: **^{1}H NMR** (400 MHz, $CDCl_3$) δ_H 7.34–7.25 (m, 2H), 7.23–7.14 (m, 3H), 2.50 (d, J = 7.2 Hz, 2H), 1.89 (app. sept., J = 6.8 Hz), 0.93 (d, J = 6.6 Hz, 6H). All other characterization was identical to reported values.[23]



10e: $C_{12}H_{18}O_2$
MW: 194.27

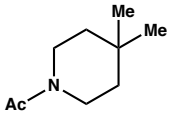
4-Isobutyl-1,2-dimethoxybenzene (10e). The title compound was prepared from **9e** (0.36 mg, 0.20 mmol) following the General Procedure. After 3 h, the reaction was completed using Workup A. The resulting crude residue was purified by flash chromatography (SiO_2, 99:1 hexanes/Et_2O) to afford **10e** (33 mg, 0.17 mmol, 87% yield, >20:1 b/l ratio) as a colorless oil: **^{1}H NMR** (400 MHz, $CDCl_3$) δ_H 6.85–6.77 (m, 1H), 6.74–6.66 (m, 2H), 3.90 (s, 3H), 3.88 (s, 3H), 2.44 (d, J = 7.2 Hz, 2H), 1.86 (app. sept., J = 6.8 Hz, 1H), 0.93 (d, J = 6.6 Hz, 6H). All other characterization was identical to reported values.[24]

Data for structures in Scheme 3



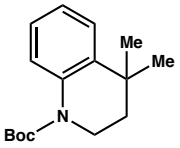
12a: $C_{12}H_{23}NO_2$
MW: 213.32

***tert*-Butyl-4,4,-dimethylpiperidine-1-carboxylate (12a).** The title compound was prepared from **11a** (99 mg, 0.5 mmol) following the General Procedure. After 1 h, the reaction was completed using Workup A. The resulting crude residue was purified by flash chromatography (SiO_2, 20:1 hexanes/EtOAc) to afford **12a** (95 mg, 0.45 mmol, 89% yield) as a colorless oil: **^{1}H NMR** (400 MHz, $CDCl_3$) δ_H 3.36 (t, J = 5.8 Hz, 2H), 1.45 (s, 9H), 1.39 (t, J = 5.8 Hz, 4H), 0.94 (s, 6H); **^{13}C NMR** (150 MHz, $CDCl_3$) δ_C **C**: 155.0, 79.1, 40.3; **CH_2**: 38.3, 28.8; **CH_3**: 28.5, 27.7; **IR** (thin film): 1262, 1695, 2921 cm^{-1}. **HRMS-DART** (m/z) $[M+H]^+$ calculated for $C_{12}H_{24}NO_2$ = 214.1807; found 214.1815.



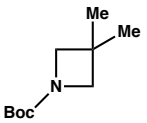
12b: $C_9H_{17}NO$
MW: 155.24

1-(4,4-Dimethylpiperidin-1-yl)ethan-1-one (12b). The title compound was prepared from **11b** (0.28 mg, 0.20 mmol) following the General Procedure. After 1 h, the reaction was completed using Workup A. The resulting crude residue was purified by flash chromatography (SiO_2, 4:1 hexanes/EtOAc) to afford **12b** (24 mg, 0.15 mmol, 77%) as a yellow oil: **^{1}H NMR** (400 MHz, $CDCl_3$) δ_H 3.55, (t, J = 5.9 Hz, 2H), 3.38 (t, J = 5.9 Hz, 2H), 2.07 (s, 3H), 1.34 (m, J = 6.0 Hz, 4H), 0.97 (s, 6H); **^{13}C NMR** (150 MHz, CDCl3) δ_C **C:** 169.0; **CH_2**: 43.3, 39.2, 38.3, 38.2, 32.2, 31.6, 29.7; **CH_3**: 29.2, 27.7, 21.5; **IR** (thin film): 1262, 1695, 2921 cm^{-1}; **HRMS-DART** (m/z) $[M+H]^+$ calculated for $C_9H_{18}NO$ = 156.1388; found 156.1326.



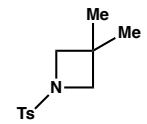
12c: $C_{16}H_{23}NO_2$
MW: 261.37

***tert*-Butyl-4,4-dimethyl-3,4-dihydroquinoline-1(*2H*)-carboxylate (12c).** The title compound was prepared from **11c** (28 mg, 0.20 mmol) following a modification of General Procedure. After 1 h, the reaction was completed using Workup A. The resulting crude residue consisting of an inseparable mixture of **11c** and **12c** was dissolved in CH_2Cl_2 (3 mL) and treated with *m*-CBPA (34 mg, 0.20 mmol) at rt. After 2 h, the reaction mixture was filtered over a pad of Celite and concentrated under reduced pressure. The resulting residue was purified by flash chromatography (SiO_2, 10:1 hexanes/Et_2O) to afford **12c** (38 mg, 0.16 mmol, 31% yield) as a colorless oil: **^{1}H NMR** (400 MHz, $CDCl_3$) δ_H 7.61 (d, J = 8.0 Hz, 1H), 7.28 (d, J = 7.8 Hz, 1H), 7.12 (t, J = 7.8 Hz, 1H), 7.02 (t, J = 7.4 Hz, 1H), 3.74 (t, J = 6.1 Hz, 2H), 1.75 (t, J = 6.1 Hz, 2H), 1.52 (s, 9H), 1.30 (s, 6H). **^{13}C NMR** (150 MHz, $CDCl_3$) δ_C **C**: 154.1, 138.0, 137.3, 80.9, 33.2; **CH**: 125.9, 125.7, 124.5, 123.6, 41.9, 38.7; **CH_2**: 30.4, 28.6; **CH_3**: 41.9, 38.7; **IR** (thin film): 1155, 1698, 2966 cm^{-1}; **HRMS-DART** (m/z) $[M+H]^+$ calculated for $C_{16}H_{24}NO_2$ = 262.1807; found 262.1793.



12d: $C_{10}H_{19}NO_2$
MW: 185.27

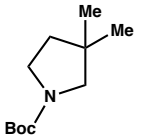
***tert*-Butyl-3,3-dimethylazetidine-1-carboxylate (12d).** The title compound was prepared from **11d** (34 mg, 0.20 mmol) following the General Procedure. After 1 h, the reaction was completed using Workup A. The resulting crude residue was purified by flash chromatography (SiO_2, 9:1 pentanes/Et_2O) to afford **12d** (31 mg, 0.17 mmol, 83% yield) as a colorless oil: **^{1}H NMR** (400 MHz, $CDCl_3$) δ_H 3.61 (s, 4H), 1.46 (s, 9H), 1.26 (s, 6H); **^{13}C NMR** (150 MHz, $CDCl_3$) δ_C **C**: 156.9, 79.2, 30.8; **CH_2**: 61.5; **CH_3**: 28.6, 27.1; **IR** (thin film): 1396, 1704, 2924 cm^{-1}; **HRMS-DART** (m/z) $[M+H]^+$ calculated for $C_{10}H_{20}NO_2$ = 186.1494; found 186.1491.



12e: $C_{12}H_{17}NO_2S$
MW: 239.33

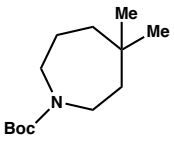
3,3-Dimethyl-1-tosylazetidine (12e). The title compound was prepared from **11e** (45 mg, 0.20 mmol) following the General Procedure. After 1 h, the reaction was completed using Workup A. The resulting crude residue was purified by flash chromatography (SiO_2, 9:1 hexanes/EtOAc) to afford **12e** (36 mg, 0.18 mmol, 92% yield) as a yellow oil**: ^{1}H NMR** (400 MHz, $CDCl_3$) δ_H 7.72 (d, J = 8.0 Hz, 2H), 7.36 (d, J = 7.9 Hz, 2H), 3.44 (s, 4H), 2.46 (s, 3H), 1.06 (s, 6H); **^{13}C NMR** (150 MHz, $CDCl_3$) δ_C **C**:

143.8, 131.8, 30.4; **CH**: 129.6, 128.3; **CH_2**: 62.5; **CH_3**: 26.6, 21.6 **IR** (thin film): 1229, 1341, 2959 cm^{-1}; **HRMS-DART** (m/z) $[M+H]^+$ calculated for $C_{12}H_{18}NO_2S$ = 240.1058; found 240.1052.



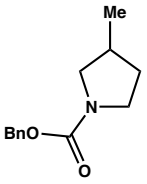
12f: $C_{11}H_{21}NO_2$
MW: 199.29

***tert*-Butyl-3,3-dimethylpyrrolidine-1-carboxylate (12f).** The title compound was prepared from **11f** (37 mg, 0.20 mmol) following the General Procedure. After 1 h, the reaction was completed using Workup A. The resulting crude residue was purified by flash chromatography (SiO_2, 9:1 hexanes/EtOAc) to afford **12f** (35 mg, 017 mmol, 87% yield) as a colorless oil. **12g** was isolated as a mixture of rotational isomers carbamate bond: **^{1}H NMR** (400 MHz, $CDCl_3$) **isomer 1**: δ_H 3.43 (t, *J* = 6.9 Hz, 2H), 3.11 (br s, 2H), 1.48 (s, 9H), 1.08 (s, 6H);) **isomer 2**: δ_H 3.38 (t, *J* = 6.9 Hz, 1H), 3.05 (br s, 1H), 1.48 (s, 9H), 1.08 (s, 6H). The remaining ^{1}H resonances could not be resolved; **^{13}C NMR** (150 MHz, $CDCl_3$) **isomer 1**: δ_C **C**: 154.9, 79.0, 29.7; **CH_2**: 58.9, 45.2, 39.3; **CH_3**: 28.6, 26.2; **isomer 2**: δ_C **C**: 154.9, 79.0, 29.7; **CH_2**: 58.3, 44.9, 38.6; **CH_3**: 28.4, 26.2 **IR** (thin film): 1399, 1699, 2959 cm^{-1}; **HRMS-DART** (m/z) $[M+H]^+$ calculated for $C_{11}H_{22}NO_2$ = 200.1651; found 200.1644.



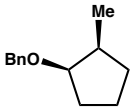
12g: $C_{13}H_{25}NO_2$
MW: 227.35

***tert*-Butyl-4,4-dimethylazepane-1-carboxylate (12g).** The title compound was prepared from **11g** (42 mg, 0.20 mmol) following the General Procedure. After 3 h, the reaction was completed using Workup A. The resulting crude residue was purified by flash chromatography (SiO_2, 10:1 hexanes/Et_2O) to afford **12g** (29 mg, 0.16 mmol, 81% yield) as a yellow oil. **12g** was isolated as a mixture of rotational isomers carbamate bond: **^{1}H NMR** (400 MHz, $CDCl_3$) **isomers 1 & 2 are indistinguishable by ^{1}H NMR:** δ_H 3.39, (t, *J* = 6.0 Hz, 1H), 3.34 (dd, *J* = 6.2 Hz, 2H), 3.28 (t, *J* = 5.3 Hz, 1H), 1.65 (m, 2H), 1.50 (t, *J* = 5.6 Hz, 2H), 1.46 (s, 9H), 1.37 (t, *J* = 5.9 Hz, 2H), 0.92 (s, 6H); **^{13}C NMR** (150 MHz, $CDCl_3$) **isomer 1**: δ_C **C** 155.7, 78.9, 32.9, 28.2; **CH_2**: 46.1, 42.6, 41.5, 41.0, 24.1; **CH_3**: 29.2, 28.5;) **isomer 2**: δ_C **C** 155.7, 78.9, 32.9, 28.18; **CH_2**: 45.4, 42.0, 41.1, 40.6, 24.1; **CH_3**: 29.0, 28.5; **IR** (thin film): 1165, 1692, 2929 cm^{-1}; **HRMS-DART** (m/z) $[M+H]^+$ calculated for $C_{13}H_{26}NO_2$ = 228.1964; found 228.1956.



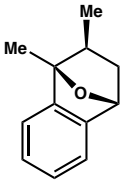
12h: $C_{13}H_{17}NO_2$
MW: 219.28

Benzyl-3-methylpyrrolidine-1-carboxylate (12h). The title compound was prepared from **11h** (102 mg, 0.50 mmol) following the General Procedure. After 3 h, the reaction was completed using Workup B. The resulting crude residue was purified by flash chromatography (SiO_2, 9:1 hexanes/Et_2O) to afford **12h** (79 mg, 0.36 mmol, 72% yield) as a yellow oil: **^{1}H NMR** (400 MHz, $CDCl_3$) δ_H 7.44–7.27 (m, 5H), 5.13 (s, 2H), 3.68–3.45 (m, 2H), 3.35 (app. q, *J* = 8.0 Hz, 1H), 2.99–2.82 (m, 1H), 2.24 (dt, *J* = 14.6, 7.3 Hz, 1H), 2.05–1.91 (m, 1H), 1.57–1.39 (m, 1H), 1.05 (d, *J* = 6.6 Hz, 3H). All other characterization data was identical to previously reported values.[25]



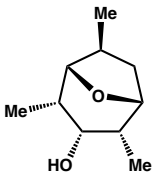
12i: $C_{13}H_{18}O$
MW: 190.29

***cis*-(((2-Methylcyclopentyl)oxy)methyl)benzene (12i).** The title compound was prepared from **11i** (86 mg, 0.50 mmol) following the General Procedure. After 3 h, the reaction was completed using Workup B. The resulting crude residue was purified by flash chromatography (SiO_2, hexanes) to afford **12i** (56 mg, 0.36 mmol, 60% yield) as an inseparable mixture of proximal (α) and distal (β) isomers (13:1 ratio): **^{1}H NMR** (400 MHz, $CDCl_3$) δ_H 7.41–7.21 (m, 5H), 4.50 (app. q., *J* = 17.8 Hz, 2H) 3.49 (app q, *J* =5.7 Hz, 1H), 2.08–1.97 (m, 1H), 1.96–1.83 (m, 2H), 1.79–1.60 (m, 3H), 1.22–1.11 (dd *J* = 12.4, 7.6 Hz, 1H), 1.02 (d, *J* = 6.8 Hz, 3H), All other characterization data was identical to previously reported values.[26]



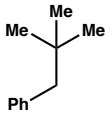
12j: $C_{12}H_{14}O$
MW: 174.24

***cis*-1,2-Dimethyl-1,2,3,4-tetrahydro-1,4-epoxynaphthalene (12j).** The title compound was prepared from **11j** (32 mg, 0.20 mmol) following the General Procedure. After 3 h, the reaction was completed using Workup B. The resulting crude residue was purified by flash chromatography (SiO_2, benzene) to afford **12j** (17 mg, 0.10 mmol, 50% yield) as a yellow oil: **^{1}H NMR** (600 MHz, $CDCl_3$) δ_H 7.25–7.11 (m, 4H), 5.27 (d, J = 4.9 Hz, 1H), 1.84–1.68 (m, 5H), 1.64 (ddd, J = 10.9, 4.9, 3.5 Hz, 1H), 1.13 (d, J = 6.7 Hz, 3H); **^{13}C NMR** (150 MHz, $CDCl_3$) δ_C **C:** 149.0, 146.7, 78.1; **CH:** 126.4, 126.2, 118.5, 117.6, 87.0, 38.7; **CH_2:** 36.3; **CH_3:** 17.6, 14.2; **IR** (thin film): 3048, 2958, 1363 cm^{-1}. **HRMS-ESI** (*m*/z) $[M+H]^+$ calculated for $C_{12}H_{15}O$ = 175.1123; found 175.1126.



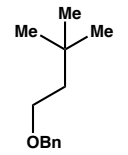
12k: $C_{10}H_{18}O_2$
MW: 170.25

2,4,6-timethyl-8-oxabicyclo[3.2.1]octan-3-ol (12k). The title compound was prepared from **11k** (30 mg, 0.20 mmol) following the General Procedure. After 3 h, the reaction was completed using Workup B. The resulting crude residue was purified by flash chromatography (SiO_2, 10:1 benzene/Et_2O) to afford **12k** (16 mg, 0.09 mmol, 47%) as a yellow oil: **^{1}H NMR** (600 MHz, $CDCl_3$) δ_H 4.05 (dd, J = 7.4, 3.3 Hz, 1H), 3.71 (br s, 1H), 3.55 (d, J = 3.2 Hz, 1H), 2.64–2.55 (m, 1H), 2.41 (dd, J = 12.1, 8.6 Hz, 1H), 2.05–1.96 (m, 2H), 1.25–1.19 (m, 1H), 1.04 (d, J = 7.1 Hz, 3H), 0.99 (d, J = 7.3 Hz, 3H), 0.95 (d, J = 7.3 Hz, 3H); **^{13}C NMR** (150 MHz, $CDCl_3$) δ_C **CH:** 85.4, 79.2, 71.9, 38.9, 31.9, 29.7; **CH_2:** 34.5; **CH_3:** 23.4, 12.9, 12.8. **IR** (thin film): 3446 (br), 2930, 2871 cm^{-1}; **HRMS-ESI** (*m*/z) $[M+H]^+$ calculated for $C_{10}H_{19}O_2$ = 171.1139; found 171.1141.



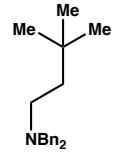
12l: $C_{11}H_{16}$
MW: 148.25

Neopentylbenzene (12l). The title compound was prepared from **11l** (59 mg, 0.50 mmol) following a modification of General Procedure employing 2.0 equiv of **1** at –10°C. After 3 h, the reaction was completed using Workup C. The resulting crude residue consisting of an inseparable mixture of **11l** and **12l** was dissolved in CH_2Cl_2 (3 mL) and treated with *m*-CBPA (34 mg, 0.20 mmol) at rt. After 2 h, the reaction mixture was filtered over a pad of Celite and concentrated under reduced pressure. The resulting residue was purified by flash chromatography (SiO_2, 99:1 pentane/Et_2O) to afford **12l** (45 mg, 0.31 mmol, 61% yield) as a colorless oil: **^{1}H NMR** (400 MHz, $CDCl_3$) δ_H 7.28–7.11 (m, 5H), 2.50 (s, 2H), 0.90 (s, 9H). All other characterization data was identical to previously reported values.[27]



12m: $C_{13}H_{20}O$
MW: 192.30

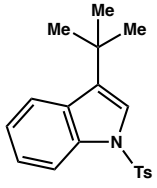
((3,3-Dimethylbutoxy)methyl)benzene (12m). The title compound was prepared from **11m** (53 mg, 0.20 mmol) following a modification of General Procedure employing 2.0 equiv of **1** at –10°C. After 3 h, the reaction was completed using Workup C. The resulting crude residue consisting of an inseparable mixture of **11m** and **12m** was dissolved in CH_2Cl_2 (3 mL) and treated with *m*-CBPA (34 mg, 0.20 mmol) at rt. After 2 h, the reaction mixture was filtered over a pad of Celite and concentrated under reduced pressure. The resulting residue was purified by flash chromatography (SiO_2, benzene) to afford **12m** (42 mg, 0.15 mmol, 75% yield) as a colorless oil: δ_H 7.40–7.33 (m, 4H), 7.33–7.29 (m, 1H), 4.52 (s, 2H), 3.57 (t, J = 7.5 Hz, 2H), 1.61 (t, J = 7.7 Hz, 2H), 0.95 (s, 9H). All other characterization data was identical to previously reported values.[28]



12n: $C_{20}H_{27}N$
MW: 281.44

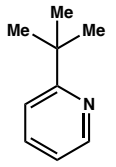
***N,N*-Dibenzyl-3,3-dimethylbutan-1-amine (12n).** The title compound was prepared from **11n** (53 mg, 0.20 mmol) following a modification of General Procedure employing 2.0 equiv of **1** at –10°C. After 3 h, the reaction was completed using Workup C. The resulting crude residue consisting of an inseparable mixture of **11n** and **12n** (1:3 ratio, 34 mg of **12n**, 0.12 mmol, est. 61% yield based on NMR). An analytical sample of **12n** was prepared by pTLC (benzene): **^{1}H NMR** (400 MHz, $CDCl_3$) δ_H 7.40–7.25 (m, 10H),

3.59 (s, 4H), 2.47 (app t, *J* = 8.0 Hz, 2H), 1.49 (app t, *J* = 7.9 Hz, 2H), 0.84 (s, 9H); **^{13}C NMR** (150 MHz, $CDCl_3$) δ_C **C:** 140.1, 29.9; **CH_2:** 58.2, 49.3, 40.32; **CH_3:** 29.5; **IR** (thin film): 3078, 2958 cm^{-1}; **HRMS-ESI** (*m*/*z*) $[M+H]^+$ calculated for $C_{20}H_{28}N$ = 282.2222; found 282.2224.



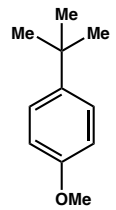
12o: $C_{19}H_{21}NO_2S$
MW: 327.44

3-(*tert*-Butyl)-1-tosyl-1*H*-indole (12o). The title compound was prepared from **11o** (62 mg, 0.20 mmol) following a modification of General Procedure employing 2.0 equiv of **1** at –10°C. After 3 h, the reaction was completed using Workup C. The resulting crude residue consisting of an inseparable mixture of **11o** and **12o** was dissolved in CH_2Cl_2 (3 mL) and treated with *m*-CBPA (34 mg, 0.20 mmol) at rt. After 2 h, the reaction mixture was filtered over a pad of Celite and concentrated under reduced pressure. The resulting residue was purified by flash chromatography (SiO_2, 4:1 pentanes/Et_2O) to afford **12o** (29 mg, 0.09 mmol, 44% yield) as a colorless oil: **^{1}H NMR** (400 MHz, $CDCl_3$) δ_H 7.98 (d, *J* = 8.1 Hz, 1H), 7.74 (d, *J* = 8.3 Hz, 2H), 7.70 (d, *J* = 7.9 Hz, 1H), 7.27 (m, 2H), 7.21 (d, *J* = 8.0 Hz, 3H), 2.34 (s, 3H), 1.40 (s, 9H). **^{13}C NMR** (150 MHz, $CDCl_3$) δ_C **C**: 144.8, 136.2, 135.6, 132.8, 129.8, 32.0; **CH**: 129.9, 126.9, 124.1, 122.7, 122.0, 121.2; **CH_3**: 30.3, 21.7; **IR** (thin film): 1172, 1367, 2960 cm^{-1}; **HRMS-DART** (m/z) $[M+H]^+$ calculated for $C_{19}H_{22}NO_2S$ = 328.1371; found 327.1395.



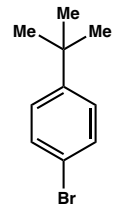
12p: $C_9H_{13}N$
MW: 135.21

2-(*tert*-Butyl)pyridine (12p). The title compound was prepared from **11p** (60 mg, 0.50 mmol) following a modification of General Procedure employing 2.0 equiv of **1** at –10°C. After 3 h, the reaction was completed using Workup C. The resulting residue was purified by flash chromatography (SiO_2, pentanes) to afford **12p** (55 mg, 0.41 mmol, 81% yield) as a colorless oil: : **^{1}H NMR** (400 MHz, $CDCl_3$) δ_H 8.57 (d, *J* = 3.9 Hz, 1H), 7.61 (td, *J* = 7.7, 1.9 Hz, 1H), 7.34 (d, *J* = 8.0 Hz, 1H), 7.08 (ddd, *J* = 7.0, 4.9, 0.9 Hz, 1H), 1.37 (s, 9H). All other characterization data was identical to previously reported values.[29]



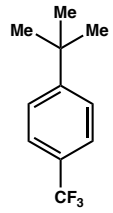
12q: $C_{11}H_{16}O$
MW: 164.25

1-(*tert*-Butyl)-4-methoxybenzene (12q). The title compound was prepared from **11q** (74 mg, 0.50 mmol) following a modification of General Procedure employing 2.0 equiv of **1** at –10°C. After 3 h, the reaction was completed using Workup C. The resulting crude residue consisting of an inseparable mixture of **11q** and **12q** was dissolved in CH_2Cl_2 (3 mL) and treated with *m*-CBPA (34 mg, 0.20 mmol) at rt. After 2 h, the reaction mixture was filtered over a pad of Celite and concentrated under reduced pressure. The resulting residue was purified by flash chromatography (SiO_2, 10:1 pentanes/Et_2O) to afford **12q** (31 mg, 0.19 mmol, 38% yield) as a yellow oil: **^{1}H NMR** (400 MHz, $CDCl_3$) δ_H 7.31 (d, *J* = 8.8 Hz, 2H), 6.85 (d, *J* = 9.2 Hz, 2H), 3.80 (s, 3H), 1.30 (s, 9H). All other characterization data was identical to previously reported values.[30]



12r: $C_{10}H_{13}Br$
MW: 213.12

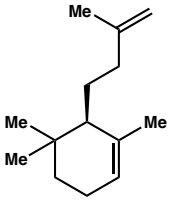
1-(*tert*-Butyl)-4-bromobenzene (12r). The title compound was prepared from **11r** (98 mg, 0.50 mmol) following a modification of General Procedure employing 2.0 equiv of **1** at –10°C. After 3 h, the reaction was completed using Workup C. The resulting crude residue consisting of an inseparable mixture of **11r** and **12r** was dissolved in CH_2Cl_2 (3 mL) and treated with *m*-CBPA (34 mg, 0.20 mmol) at rt. After 2 h, the reaction mixture was filtered over a pad of Celite and concentrated under reduced pressure. The resulting residue was purified by flash chromatography (SiO_2, pentanes) to afford **12r** (65 mg, 0.31 mmol, 61% yield) as a colorless oil: **^{1}H NMR** (400 MHz, $CDCl_3$) δ_H 7.43 (dd, *J* = 8.5, 4.7 Hz, 2H), 7.32–7.25 (m, 2H), 1.32 (s, 9H). All other characterization data was identical to previously reported values.[31]



12s: $C_{11}H_{13}F_3$
MW: 202.22

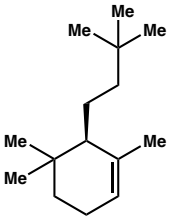
1-(*tert*-Butyl)-4-trifluoromethyl-benzene (12s). The title compound was prepared from **11s** (93 mg, 0.50 mmol) following a modification of General Procedure employing 2.0 equiv of **1** at –10°C. After 3 h, the reaction was completed using Workup C. The resulting crude residue consisting of an inseparable mixture of **11s** and **12s** was dissolved in CH_2Cl_2 (3 mL) and treated with *m*-CBPA (34 mg, 0.20 mmol) at rt. After 2 h, the reaction mixture was filtered over a pad of Celite and concentrated under reduced pressure. The resulting residue was purified by flash chromatography (SiO_2, pentanes) to afford **12s** (76 mg, 0.35 mmol, 70% yield) as a colorless oil: **^{1}H NMR** (400 MHz, $CDCl_3$) δ_H 7.52 (dd, J = 10.7 Hz, 2H), 1.37 (s, 9H). All other characterization data was identical to previously reported values.[32]

Data for structures Scheme 4



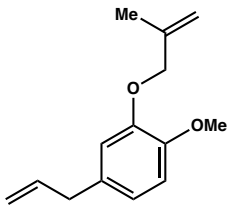
13: $C_{14}H_{24}$
MW: 192.35

1,5,5-Trimethyl-6-(3-methylbut-3-en-1-yl)-cyclohex-1-ene (13). The title compound was prepared from **S1** using the protocol described by Shoenebeck:[33] **^{1}H NMR** (400 MHz, $CDCl_3$) **^{1}H NMR** (400 MHz, $CDCl_3$) δ_H 5.32 (s, 1H), 4.70 (s, 1H), 4.69 (s, 1H), 2.09–2.04 (m, 2H), 2.01–1.96 (m, 2H), 1.75 (s, 3H), 1.71 (s, 3H), 1.63–1.54 (m, 2H), 1.49–1.40 (m, 2H), 1.17–1.11 (m, 1H), 0.95 (s, 3H), 0.90 (s, 3H). All other characterization data was identical to reported values.[33]



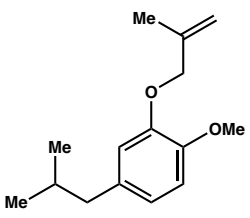
14: $C_{15}H_{28}$
MW: 208.39

6-(3,3-Dimethylbutyl)-1,5,5-trimethylcyclohex-1-ene (14). The title compound was prepared from **13** (96 mg, 0.50 mmol) following a modification of General Procedure employing 2.0 equiv of **1** at –10°C. After 3 h, the reaction was completed using Workup C. The resulting crude residue was purified by flash chromatography (SiO_2, 20:1 hexanes/Et_2O) to afford an inseparable mixture of **14** and unreacted **13** (3:1 ratio, 73% yield combined, 40% yield of **14**). An analytical sample of **14** was prepared by pTLC (benzene): **^{1}H NMR** (600 MHz, $CDCl_3$) δ_H 5.29 (s, 1H), 2.04–1.97 (m, 2H), 1.68 (s, 3H), 1.69 (app d, J = 13.7 Hz, 2H), 1.53–1.38 (m, 2H), 1.33–1.19 (m, 4H), 1.16–1.09 (d, J = 12.3 Hz, 1H), 0.93 (s, 6H), 0.89 (s, 9H). **^{13}C NMR** (150 MHz, $CDCl_3$) δ_C **C:** 137.2, 31.7, 30.7; **CH:** 119.5, 31.8; **CH_2:** 49.8, 29.3, 27.7, 27.5; **CH_3:** 44.9, 25.9, 23.0; **IR** (thin film): 3073, 2868, 1508 cm^{-1}; **HRMS-ESI** (*m*/z) $[M+H]^+$ calculated for $C_{15}H_{29}$ = 209.2269; found 209.2271.



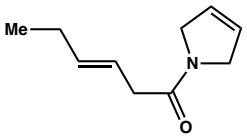
15: $C_{14}H_{18}O_2$
MW: 218.30

4-Allyl-1-methoxy-2-((2-methylallyl)oxy)benzene (15). The title compound was prepared from eugenol using the protocol described by Shoenebeck:[33] **^{1}H NMR** (400 MHz, $CDCl_3$) δ_H 6.80 (d, J = 8.0 Hz, 1H), 6.79 (s, 1H), 6.68 (d, J = 8.1 Hz, 1H), 6.01–5.88 (m, 1H), 5.11–5.01 (m, 2H), 4.96 (br s, 1H), 4.49 (s, 2H), 3.86 (s, 3H), 3.32 (d, J = 6.64 Hz, 2H), 1.82 (s, 3H). All other characterization data was identical to reported values.



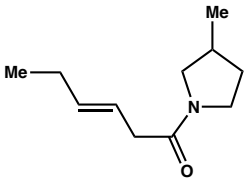
16: $C_{15}H_{22}O_2$
MW: 234.34

4-Isobutyl-1-methoxy-2-((2-methylallyl)oxy)benzene (16). The title compound was prepared from **15** (44 mg, 0.20 mmol) following the General Procedure. After 6 h, the reaction was completed using Workup C. The resulting crude residue was purified by flash chromatography (SiO_2, hexanes) to afford **16** (40 mg, 0.17 mmol, 84% yield) as a 15:1 mixture of branched and linear regioisomers. The major (branched) regioisomer was characterized: **^{1}H NMR** (600 MHz, $CDCl_3$) δ_H 6.78 (d, J = 8.1 Hz, 1H), 6.67 (s, 1H), 6.62 (d, J = 8.2 Hz, 1H), 5.08 (br s, 1H), 4.96 (br s, 1H), 4.48 (s, 2H), 3.86 (s, 3H), 2.40 (d, J = 7.2 Hz, 1H), 1.88–1.75 (m, 4H), 0.89 (d, J = 6.6 Hz, 1H); **^{13}C NMR** (150 MHz, $CDCl_3$) δ **C:** 149.2, 146.3, 141.2, 134.8; **CH:** 121.0, 113.6, 113.0, 19.4; **CH_2:** 112.5, 72.9, 45.1; **CH_3:** 56.0, 30.3, 22.4; **IR** (thin film): 3084, 2943, 1108 cm^{-1}; **HRMS-ESI** (*m*/z) $[M+H]^+$ calculated for $C_{15}H_{23}O_2$ = 235.1698; found 235.1699.



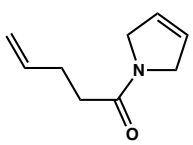
17: $C_{10}H_{15}NO$
MW: 165.24

(*E*)-1-(2,5-Dihydro-1*H*-pyrrol-1-yl)hex-3-en-1-one (17). A solution of (*E*)-3-hexenoic acid (0.1 mL, 1 mmol) in CH_2Cl_2 (3 mL) was treated sequentially with Et_3N (0.3 mL, 2 mmol), HATU (370 mg, 1.20 mmol) and 3-pyrroline (0.1 mL, 1 mmol) at rt. After 18 h, the reaction mixture was treated with water (10 mL) and transferred to a separatory funnel with CH_2Cl_2 (10 mL). The mixture was extracted with CH_2Cl_2 (3 x 10 mL). The combined organic extracts were washed with water (2 x 10 mL) and brine (10 mL), dried over $MgSO_4$, filtered and concentrated under reduced pressure. The resulting crude residue was purified by flash chromatography (SiO_2, 2:1 PhH/EtOAc) to afford **17** (126 mg, 0.76 mmol, 76% yield) as a colorless oil: **^{1}H NMR** (400 MHz, $CDCl_3$) δ_H 5.89 (d, J = 6.0 Hz, 1H), 5.81 (d, J = 6.0 Hz, 1H), 5.67–5.53 (m, 2H), 4.27 (s, 4H), 3.05 (d, J = 4.8 Hz, 2H), 2.13 –2.02 (m, 2H), 1.01 (t, J = 7.4 Hz, 3H). **^{13}C NMR** (150 MHz, $CDCl_3$) δ_C **C:** 170.1; **CH**: 135.9, 126.4, 124.9, 121.2; **CH_2**: 53.4, 53.1, 38.8, 25.6; **CH_3**: 13.6; **IR**: 2926, 1618, 1358. **HRMS-DART** (m/z) [M+1] calculated for $C_{10}H_{16}NO$ = 166.1232; found 166.1232.



18: $C_{11}H_{19}NO$
MW: 181.28

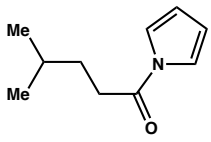
(*E*)-1-(3-methylpyrrolidin-1-yl)hex-3-en-1-one (18). The title compound was prepared from **17** (83 mg, 0.50 mmol) following the General Procedure. After 3 h, the reaction was completed using Workup C. The resulting crude residue was purified by flash chromatography (SiO_2, 6:1 hexanes/EtOAc) to afford **18** (62 mg, 0.34 mmol, 68% yield) as a 1:1 mixture of isomers about the amide bond: **^{1}H NMR** (600 MHz, $CDCl_3$) **isomer 1**, δ_H 3.68 (dd, J = 11.8, 7.3 Hz, 1H), 3.62 (dddd, J = 12.1, 7.9, 3.3, 0.5 Hz, 1H), 2.29 (app. sext, J = 7.0 Hz, 1H), 1.56 (qd, 12.3, 8.6 Hz, 1H), 1.06 (d, J = 6.9 Hz, 3H); **isomer 2**, δ_H 3.57 (dd, J = 9.5, 7.3 Hz, 1H), 3.55–3.49 (m, 1H), 2.20 (app. sext, J = 7.0 Hz, 1H), 1.45 (qd, 12.3, 8.6 Hz, 1H), 1.05 (d, J = 6.9 Hz, 3H); remaining ^{1}H resonances could not be resolved.; **^{13}C NMR** (150 MHz, $CDCl_3$) δ_C **isomer 1**, δ_C **C:** 170.1; **CH:** 135.4, 121.7, 34.0; **CH_2:** 54.0, 46.4, 39.1, 32.3, 25.6; **CH_3:** 17.7, 13.6; **isomer 2**, δ_C **C:** 170.1; **CH:** 135.4, 121.7, 33.9; **CH_2:** 52.7, 45.5, 38.9, 32.1, 25.6; **CH_3:** 17.6, 13.6 **IR**: 2936, 1641, 1438. **HRMS-ESI** (*m*/z) $[M+H]^+$ calculated for $C_{11}H_{20}NO$ = 182.1545; found 182.1545.



19: $C_9H_{13}NO$
MW: 151.21

1-(2,5-Dihydro-1*H*-pyrrol-1-yl)pent-4-en-1-one (19). A solution of 4-pentenoic acid (200 mg, 2.00 mmol) in CH_2Cl_2 (6 mL) was treated sequentially with Et_3N (0.6 mL, 4 mmol), HATU (740 mg, 2.40 mmol) and 3-pyrroline (0.2 mL, 2 mmol) at rt. After 18 h, the reaction mixture was treated with water (20 mL) and transferred to a separatory funnel with CH_2Cl_2 (20 mL). The mixture was extracted with CH_2Cl_2 (3 x 20 mL). The combined organic extracts were washed with water (2 x 20 mL) and brine (20 mL), dried over $MgSO_4$, filtered and concentrated under reduced pressure. The resulting crude residue was purified by flash chromatography (SiO_2, 4:1

hexanes/EtOAc) to afford **19** (257 mg, 1.70 mmol, 85% yield) as a colorless oil: **^{1}H NMR** (600 MHz, $CDCl_3$) δ_H 5.99–5.85 (m, 2H), 5.81 (d, J = 6.1 Hz, 1H), 5.08 (d, J = 17.1 Hz, 1H), 5.01 (d, J = 10.2 Hz, 1H), 4.26 (s, 4H), 2.50–2.33 (m, 4H); **^{13}C NMR** (150 MHz, $CDCl_3$) δ_C **C:** 171.0; **CH**: 137.6, 126.5, 124.8; **CH_2**: 115.2, 53.3, 52.9, 33.65, 28.8; **IR** (thin film): 3008, 2870, 1655 cm^{-1}; **HRMS-DART** (m/z) $[M+H]^+$ calculated for $C_9H_{14}NO$ = 152.1075; found 152.1068.



20: $C_{10}H_{15}NO$
MW: 165.24

4-Methyl-1-(1*H*-pyrrol-1-yl)pentan-1-one (20). The title compound was prepared from **19** (76 mg, 0.50 mmol) following the General Procedure. After 3 h, the reaction was completed using Workup C. The resulting crude residue was digested in $CHCl_3$ (3 mL) and stirred under a balloon of O_2 (1 atm). After 16 h, the mixture was concentrated and purified by flash chromatography (SiO_2, 9:1 hexanes/EtOAc) to afford **20** (59 mg, 0.36 mmol, 72% yield) as a yellow oil: **^{1}H NMR** (600 MHz, $CDCl_3$) δ_H 7.35 (br s, 2H), 6.33 (app t, J = 2.3 Hz, 2H), 2.85 (app t, J = 8.4 Hz, 2H), 1.72–1.60 (m, 2H), 1.43–1.34 (m, 1H) 0.98 (d, J = 6.3 Hz, 6H); **^{13}C NMR** (150 MHz, $CDCl_3$) δ_C **C:** 171.0; **CH**: 119.0, 113.0, 27.7; **CH_2**: 33.4, 32.6, **CH_3**: 22.3; **IR**: 2963, 1641, 1438. **HRMS-DART** (m/z) $[M+H]^+$ calculated for $C_{10}H_{15}NO$ = 165.1154; found 165.1155.

6. References

(1) Abd Rabo Moustafa, M. M.; Pagenkopf, B. L. *Org. Lett.* **2010**, *12*, 3168–3171.

(2) Alacid, E.; Najera, C. *Org. Lett.* **2008**, *10*, 5011–5014.

(3) Xu, Y.-C.; Kohlman, D. T.; Liang, S.X.; Erikkson, C. *Org. Lett.* **1999**, *1*, 1599–1602.

(4) Davis, B. G.; Maughan, M. A. T.; Champman, T. M.; Villard, R.; Courtney, S. *Org. Lett.* **2002**, *4*, 103–106.

(5) Crotti, P.; Dussolo, V. D.; Favero, L.; Macchia, F.; Pinechi, M. *Eur. J. Org. Chem.* **1998**, 1675–1686.

(6) Zhou, H.; Li, J.; Yang, H.; Xia, C.; Jiang, G. *Org. Lett.* **2015**, *17*, 4628–4631.

(7) Yadav, J.S.; Hossain, S.; Madhu, M.; Mohapatra, D.K. *J. Org. Chem.* **2009**, *74*, 8822–8825.

(8) Cleary, P. A.; Woerpel, K. A. *Org. Lett.* **2005**, *7*, 5531–5533.

(9) (a) Zhang, M.-M.; Liu, F. *Org. Chem. Front.* **2018**, *5*, 3343–3347; (b) Lux, M.; Klussmann, M. *Org. Lett.* **2020**, *22*, 3697–3701.

(10) Baker, B. A.; Boskovic, Z. C.; Lipshutz, B. H. *Org. Lett.* **2008**, *10*, 289–292.

(11) Cannizzo, L. F.; Grubbs, R. H. *J. Org. Chem.* **1985**, *50*, 2386–2387.

(12) The synthesis and characterization data for **2** will be reported as part of a separate manuscript that is currently in progress.

(13) Romanov-Michailidis, F; Sedillo, K.F.; Neely, J.M; Rovis, T. *J. Am. Chem. Soc.* **2015**, *137*, 8892–8895.

(14) Siu, J.C.; Parry, J.B.; Lin, S. *J. Am. Chem. Soc*. **2019**, *141*, 2825–2831.

(15) Matos, J.L.M.; Vásquez Céspedes, S.; Gu, J.; Oguma, T.; Shenvi, R.A. *J. Am. Chem. Soc.* **2018**, *140*, 16976–16981.

(16) Green, S.A.; Vásquez-Céspedes, S.; Shenvi, R.A. *J. Am. Chem. Soc*, **2018**, 140, 11317–11324.

(17) Tran, N.C.; Dhondt, H.; Flipo, M.; Deprez, B.; Willand, N. *Tetrahedron Lett.* **2015***, 56,* 4119–4123.

(18) Watson, D.W.; Gill, M.; Kemmitt, P.; Lamont, S.G.; Popescu, M.V.; Simpson, I. *Tetrahedron Lett*. **2018**, *59*, 4479–4482.

(19) Zhang, C.; Li, Z.; Zhu, L.; Yu, L.; Wang, Z.; Li, C. *J. Am. Chem. Soc.* **2013**, *135*, 14082–14085.

(20) Stakem, F. G.; Heck, R. F. *J. Org. Chem.* **1980,** *45*, 3584–3593.

(21) Fukuyama, T.; Nishikawa, T.; Yamada, K.; Ravelli, D.; Fagnoni, M.l Ryu, I. *Org. Lett.*, **2017**, *19*, 6436–6439.

(22) Zhao, C.; Sojdak, C. A.; Seidel, D. *J. Am. Chem. Soc.,* **2017**, *139*, 10224–10227.

(23) Baldwin, J. *Tetrahedron Lett.* **1986**, *42,* 4235–4236.

(24) Khrimian, A. P.; DeMilo, A. B.; Waters, R. M.; Cunningham, R. T.; Leonhardt, B. A. *J. Chem. Ecol.* **1993**, *19*, 2935–2946.

(25) Xue, W.; Xu, H.; Liang, Z.; Qian, Q.; Gong, H. *Org*. *Lett*. **2014**, *16*, 4984–4987.

(26) Molander; G. A.; Hoberg, J. O. *J. Am. Chem. Soc.*, **1992**, *114*, 3123–3125.

(27) Furstner, A.; Martin, R.; Krause, H.; Seidel, G.; Goddard, R.; Lehmann, C.W. *J. Am. Chem. Soc.* **2008**, *130*, 8773–8787.

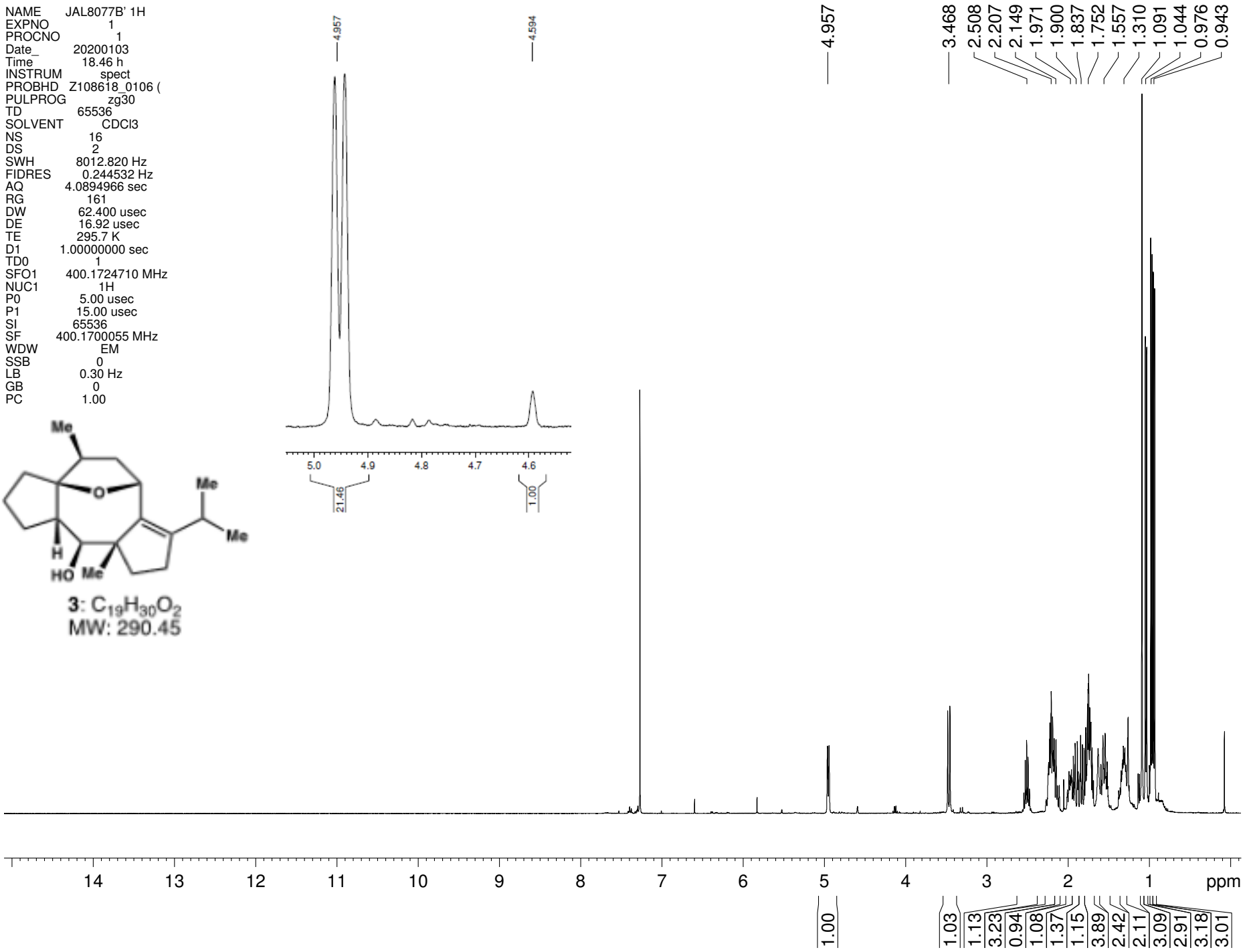
(28) Urgnoitia, G.; SanMartin, R.; Herrero, M. T.; Domigues, E. *Adv. Synth. & Catalysis,* **2016**, *358,* 3307–3312.

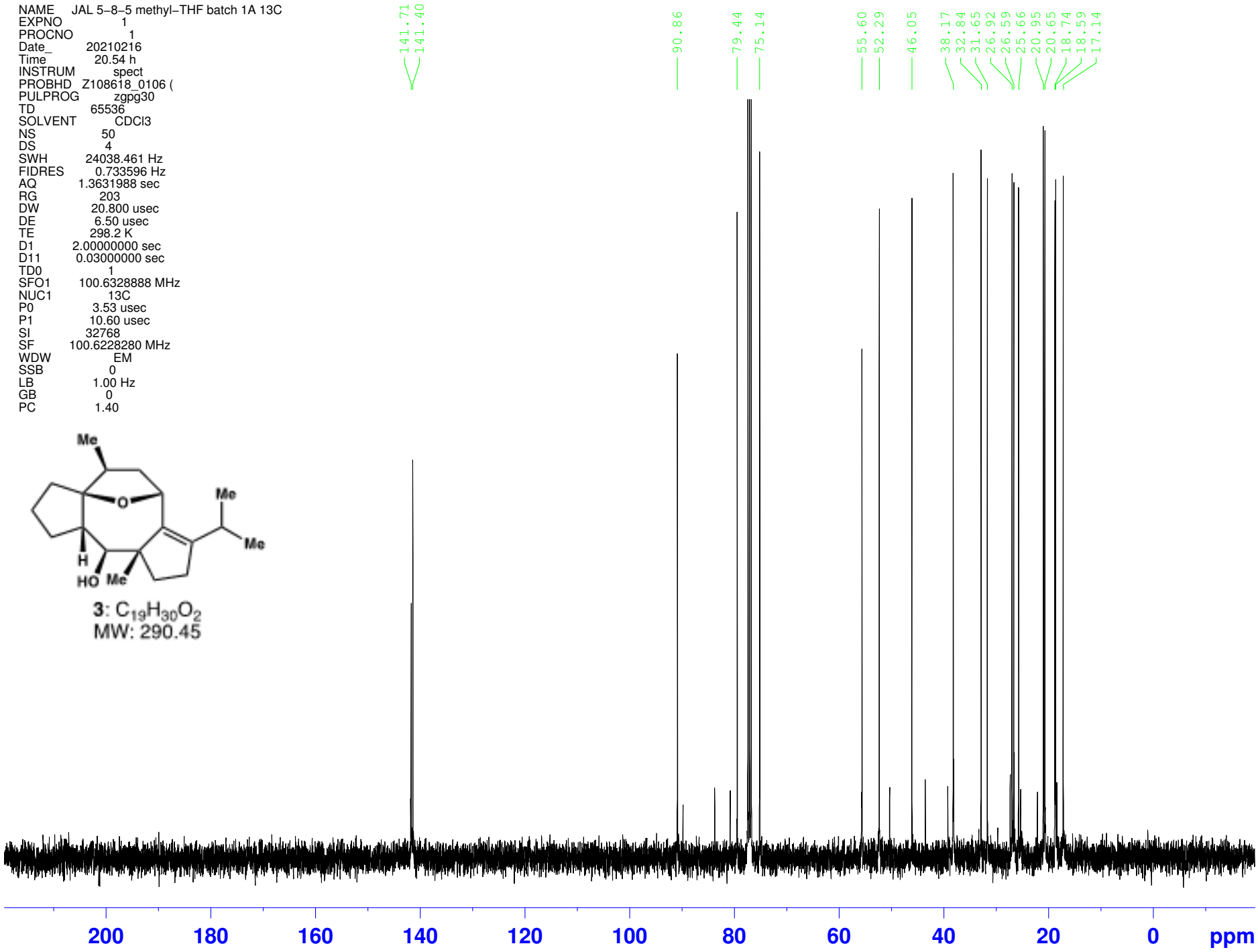
(29) Mita, T.; Michigami, K.; Sato, Y. *Chem. Asian. J.* **2013**, *8*, 2970–2973.

(30) Cheung, C. W.; Buchwald, S. L. *Org. Lett.* **2013**, *15*, 3998–4001.

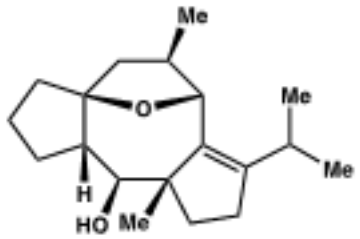
(31) Patra, T.; Mukherjee, S.; Ma, J.; Strieth-Kalthoff, F.; Glorius, F. *Angew. Chem. Int. Ed.* **2019**, *58*, 10514–10520.

(32) Huang, Y.; Fang, X.; Lin, X.; Li, H.; He, W.; Huang, K.-W.; Yuan, Y; Weng, Z. *Tetrahedron* **2012**, *68*, 9949–9953.

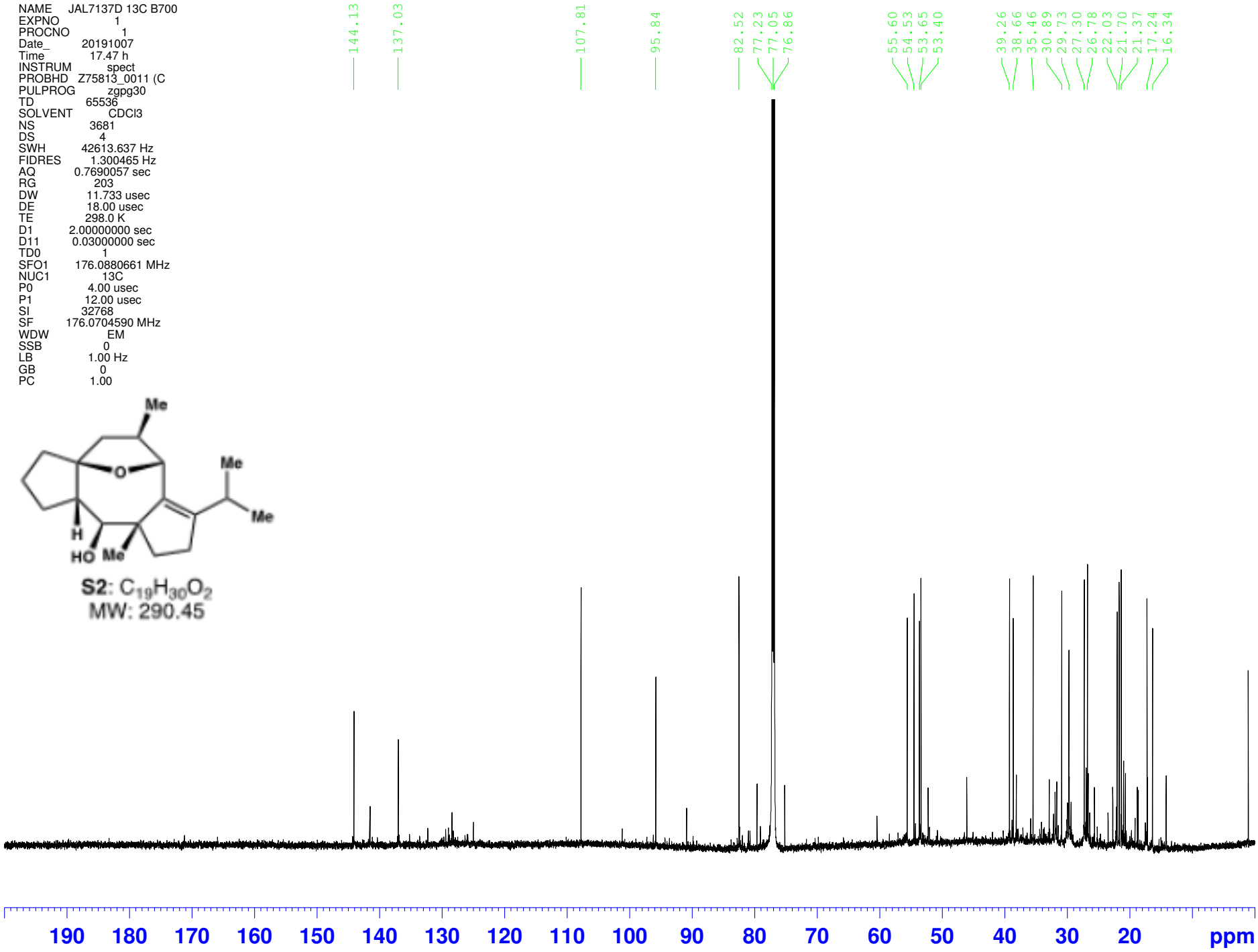

NAME JAL8077B' 1H
EXPNO 1
PROCNO 1
Date_ 20200103
Time 18.46 h
INSTRUM spect
PROBHD Z108618_0106 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.244532 Hz
AQ 4.0894966 sec
RG 161
DW 62.400 usec
DE 16.92 usec
TE 295.7 K
D1 1.00000000 sec
TD0 1
SFO1 400.1724710 MHz
NUC1 1H
P0 5.00 usec
P1 15.00 usec
SI 65536
SF 400.1700055 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
Me
O
Me
Me
H
HO
Me
3: C19H30O2
MW: 290.45
4.957
4.594
21.46
1.00
4.957
3.468
2.508
2.207
2.149
1.971
1.900
1.837
1.752
1.557
1.310
1.091
1.044
0.976
0.943
ppm
1.00
1.03
1.13
3.23
0.94
1.08
1.37
1.15
3.89
2.42
2.11
3.09
2.91
3.18
3.01


NAME JAL 5–8–5 methyl–THF batch 1A 13C
EXPNO 1
PROCNO 1
Date_ 20210216
Time 20.54 h
INSTRUM spect
PROBHD Z108618_0106 (
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 50
DS 4
SWH 24038.461 Hz
FIDRES 0.733596 Hz
AQ 1.3631988 sec
RG 203
DW 20.800 usec
DE 6.50 usec
TE 298.2 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 100.6328888 MHz
NUC1 13C
P0 3.53 usec
P1 10.60 usec
SI 32768
SF 100.6228280 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
141.71
141.40
90.86
79.44
75.14
55.60
52.29
46.05
38.17
32.84
31.65
26.92
26.59
25.66
20.95
20.65
18.74
18.59
17.14
Me
O
Me
Me
H
HO
Me
3: C19H30O2
MW: 290.45
200
180
160
140
120
100
80
60
40
20
0
ppm

NAME JAL7229C 1H
EXPNO 1
PROCNO 1
Date_ 20191112
Time 22.44 h
INSTRUM spect
PROBHD Z847801_0093 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 12019.230 Hz
FIDRES 0.366798 Hz
AQ 2.7263477 sec
RG 203
DW 41.600 usec
DE 10.42 usec
TE 293.6 K
D1 1.00000000 sec
TD0 1
SFO1 600.1337058 MHz
NUC1 1H
P0 5.00 usec
P1 15.00 usec
SI 65536
SF 600.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

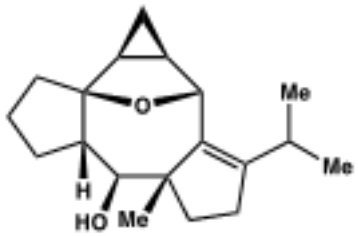


S2: $C_{19}H_{30}O_2$
MW: 290.45

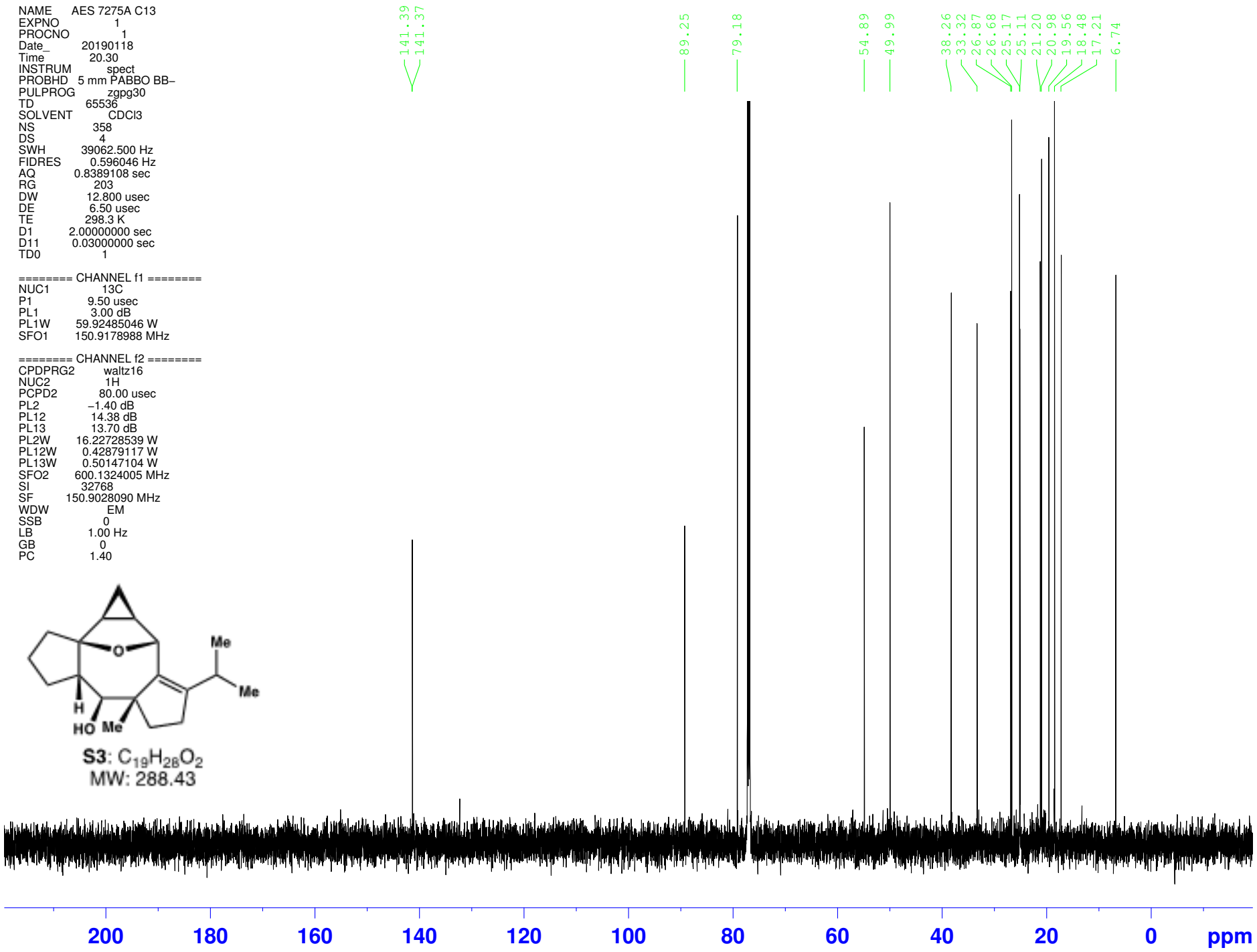

NAME JAL7137D 13C B700
EXPNO 1
PROCNO 1
Date_ 20191007
Time 17.47 h
INSTRUM spect
PROBHD Z75813_0011 (C
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 3681
DS 4
SWH 42613.637 Hz
FIDRES 1.300465 Hz
AQ 0.7690057 sec
RG 203
DW 11.733 usec
DE 18.00 usec
TE 298.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 176.0880661 MHz
NUC1 13C
P0 4.00 usec
P1 12.00 usec
SI 32768
SF 176.0704590 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.00
144.13
137.03
107.81
95.84
82.52
77.23
77.05
76.86
55.60
54.53
53.65
53.40
39.26
38.66
35.46
30.89
29.73
27.30
26.78
22.03
21.70
21.37
17.24
16.34
Me
O
Me
Me
H
HO
Me
S2: C19H30O2
MW: 290.45
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm

NAME AES 7275A 1H
EXPNO 1
PROCNO 1
Date_ 20190118
Time 20.22
INSTRUM spect
PROBHD 5 mm PABBO BB–
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 12335.526 Hz
FIDRES 0.188225 Hz
AQ 2.6564426 sec
RG 181
DW 40.533 usec
DE 6.50 usec
TE 298.2 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 13.00 usec
PL1 –1.40 dB
PL1W 16.22728539 W
SFO1 600.1337060 MHz
SI 32768
SF 600.1300167 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

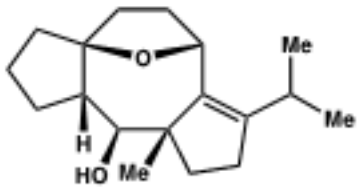


S3: $C_{19}H_{28}O_2$
MW: 288.43

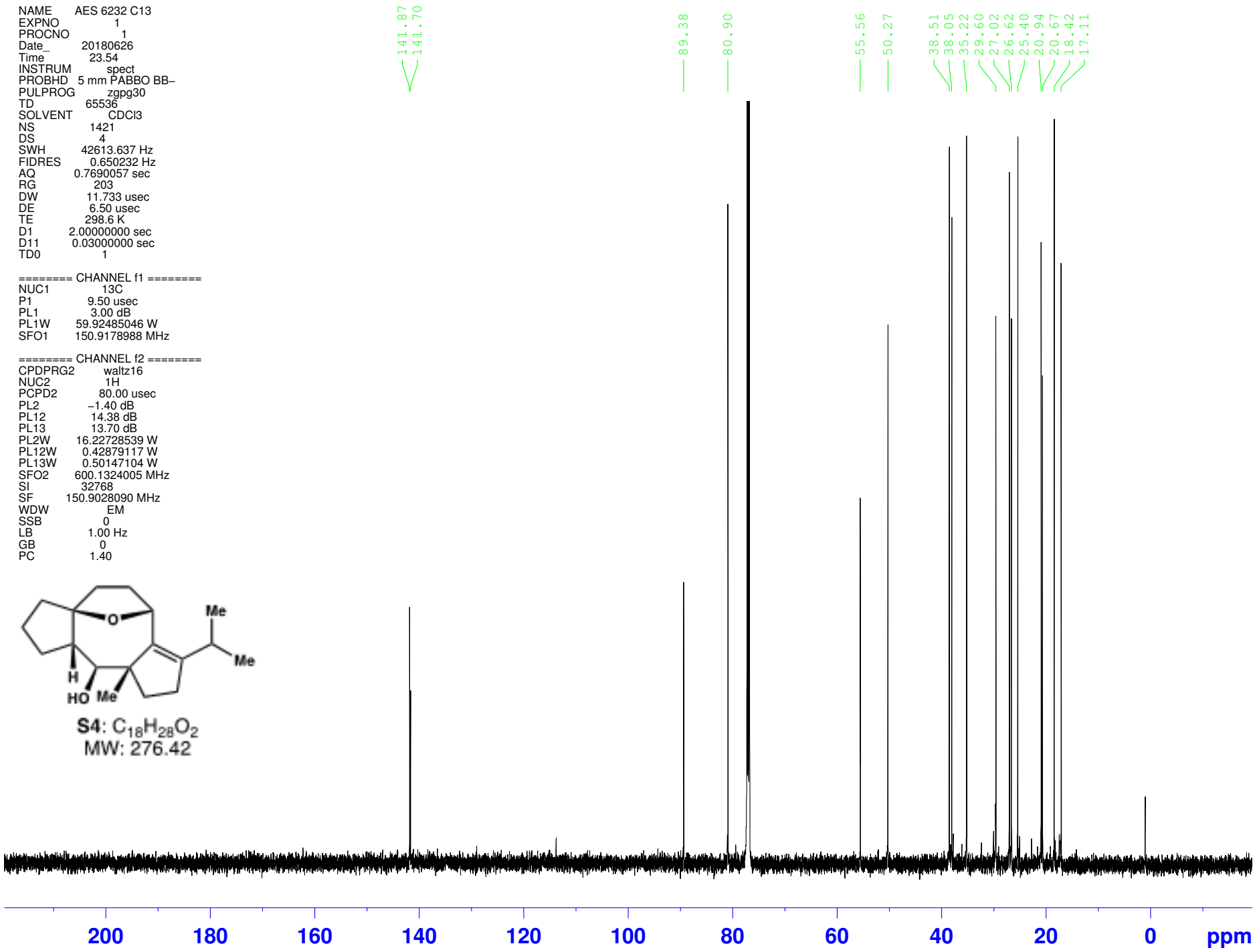

NAME AES 7275A C13
EXPNO 1
PROCNO 1
Date_ 20190118
Time 20.30
INSTRUM spect
PROBHD 5 mm PABBO BB–
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 358
DS 4
SWH 39062.500 Hz
FIDRES 0.596046 Hz
AQ 0.8389108 sec
RG 203
DW 12.800 usec
DE 6.50 usec
TE 298.3 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.50 usec
PL1 3.00 dB
PL1W 59.92485046 W
SFO1 150.9178988 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 –1.40 dB
PL12 14.38 dB
PL13 13.70 dB
PL2W 16.22728539 W
PL12W 0.42879117 W
PL13W 0.50147104 W
SFO2 600.1324005 MHz
SI 32768
SF 150.9028090 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
141.39
141.37
89.25
79.18
54.89
49.99
38.26
33.32
26.87
26.68
25.17
25.11
21.20
20.98
19.56
18.48
17.21
6.74
O
Me
Me
H
HO
Me
S3: C19H28O2
MW: 288.43
200
180
160
140
120
100
80
60
40
20
0
ppm

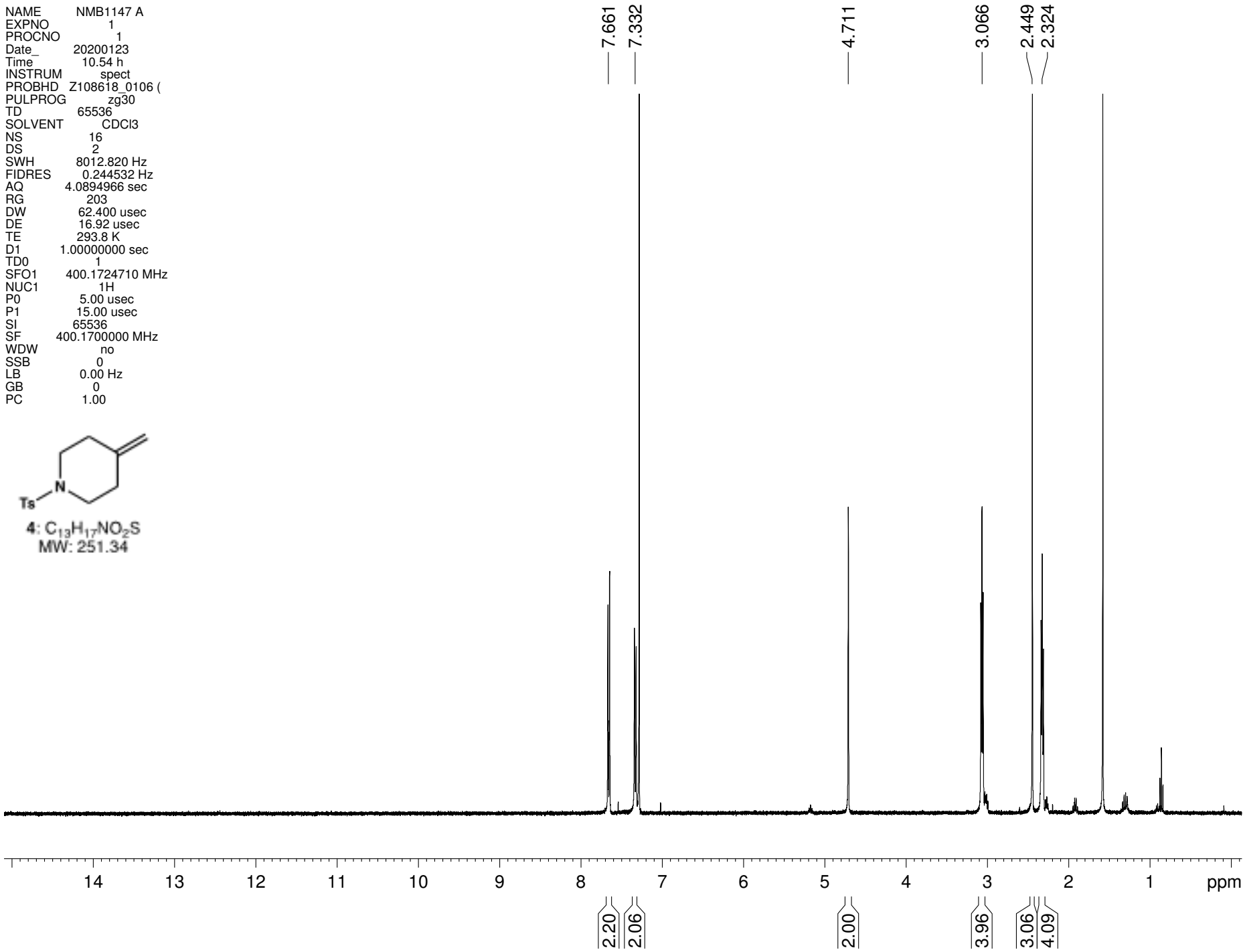
NAME JAL7027A 1H
EXPNO 1
PROCNO 1
Date_ 20190804
Time 21.03
INSTRUM spect
PROBHD 5 mm PABBO BB–
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 5
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 203
DW 60.800 usec
DE 6.50 usec
TE 297.8 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 10.00 usec
PL1 –2.78 dB
PL1W 27.10892296 W
SFO1 400.1724712 MHz
SI 32768
SF 400.1700082 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

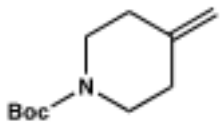


S4: $C_{18}H_{28}O_2$
MW: 276.42


NAME AES 6232 C13
EXPNO 1
PROCNO 1
Date_ 20180626
Time 23.54
INSTRUM spect
PROBHD 5 mm PABBO BB–
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1421
DS 4
SWH 42613.637 Hz
FIDRES 0.650232 Hz
AQ 0.7690057 sec
RG 203
DW 11.733 usec
DE 6.50 usec
TE 298.6 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.50 usec
PL1 3.00 dB
PL1W 59.92485046 W
SFO1 150.9178988 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 –1.40 dB
PL12 14.38 dB
PL13 13.70 dB
PL2W 16.22728539 W
PL12W 0.42879117 W
PL13W 0.50147104 W
SFO2 600.1324005 MHz
SI 32768
SF 150.9028090 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
141.87
141.70
89.38
80.90
55.56
50.27
38.51
38.05
35.22
29.60
27.02
26.62
25.40
20.94
20.67
18.42
17.11
O
Me
Me
H
HO
Me
S4: C18H28O2
MW: 276.42
200
180
160
140
120
100
80
60
40
20
0
ppm

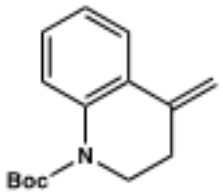

NAME NMB1147 A
EXPNO 1
PROCNO 1
Date_ 20200123
Time 10.54 h
INSTRUM spect
PROBHD Z108618_0106 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.244532 Hz
AQ 4.0894966 sec
RG 203
DW 62.400 usec
DE 16.92 usec
TE 293.8 K
D1 1.00000000 sec
TD0 1
SFO1 400.1724710 MHz
NUC1 1H
P0 5.00 usec
P1 15.00 usec
SI 65536
SF 400.1700000 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
Ts
N
4: C13H17NO2S
MW: 251.34
7.661
7.332
4.711
3.066
2.449
2.324
14
13
12
11
10
9
8
7
6
5
4
3
2
1
ppm
2.20
2.06
2.00
3.96
3.06
4.09

NAME NMB SKNBocPiperidine Olefin
EXPNO 1
PROCNO 1
Date_ 20200629
Time 21.18 h
INSTRUM spect
PROBHD Z108618_0106 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.244532 Hz
AQ 4.0894966 sec
RG 161
DW 62.400 usec
DE 16.92 usec
TE 297.6 K
D1 1.00000000 sec
TD0 1
SFO1 400.1724710 MHz
NUC1 1H
P0 5.00 usec
P1 15.00 usec
SI 65536
SF 400.1700000 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

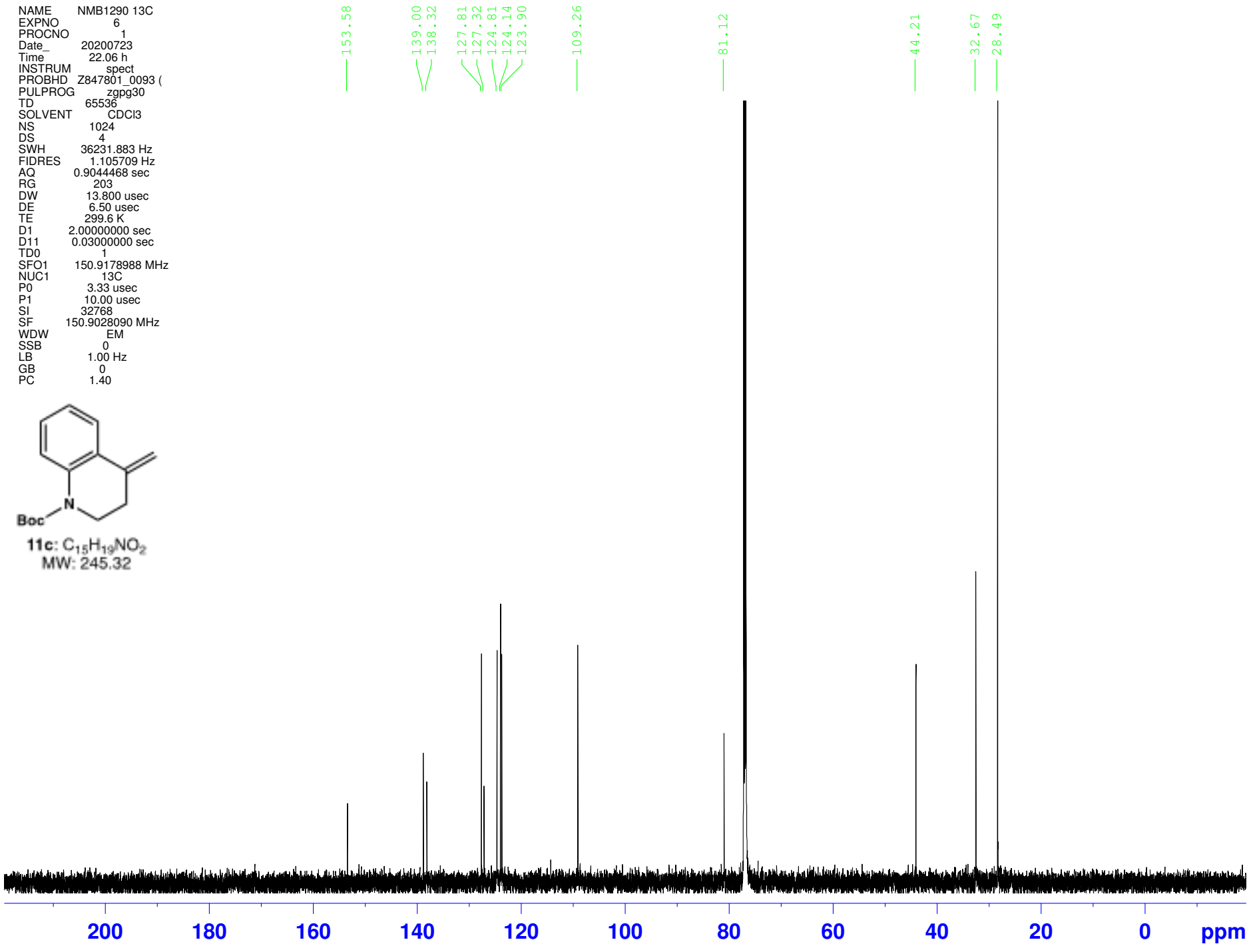


11a: $C_{11}H_{19}NO_2$
MW: 197.28

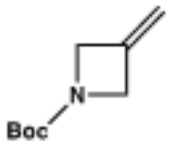
NAME	NMB2116 1H
EXPNO	1
PROCNO	1
Date_	20201224
Time	10.21 h
INSTRUM	spect
PROBHD	Z108618_0106 (
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	16
DS	2
SWH	8012.820 Hz
FIDRES	0.244532 Hz
AQ	4.0894966 sec
RG	203
DW	62.400 usec
DE	16.92 usec
TE	297.6 K
D1	1.00000000 sec
TD0	1
SFO1	400.1724710 MHz
NUC1	1H
P0	5.00 usec
P1	15.00 usec
SI	65536
SF	400.1700000 MHz
WDW	no
SSB	0
LB	0.00 Hz
GB	0
PC	1.00



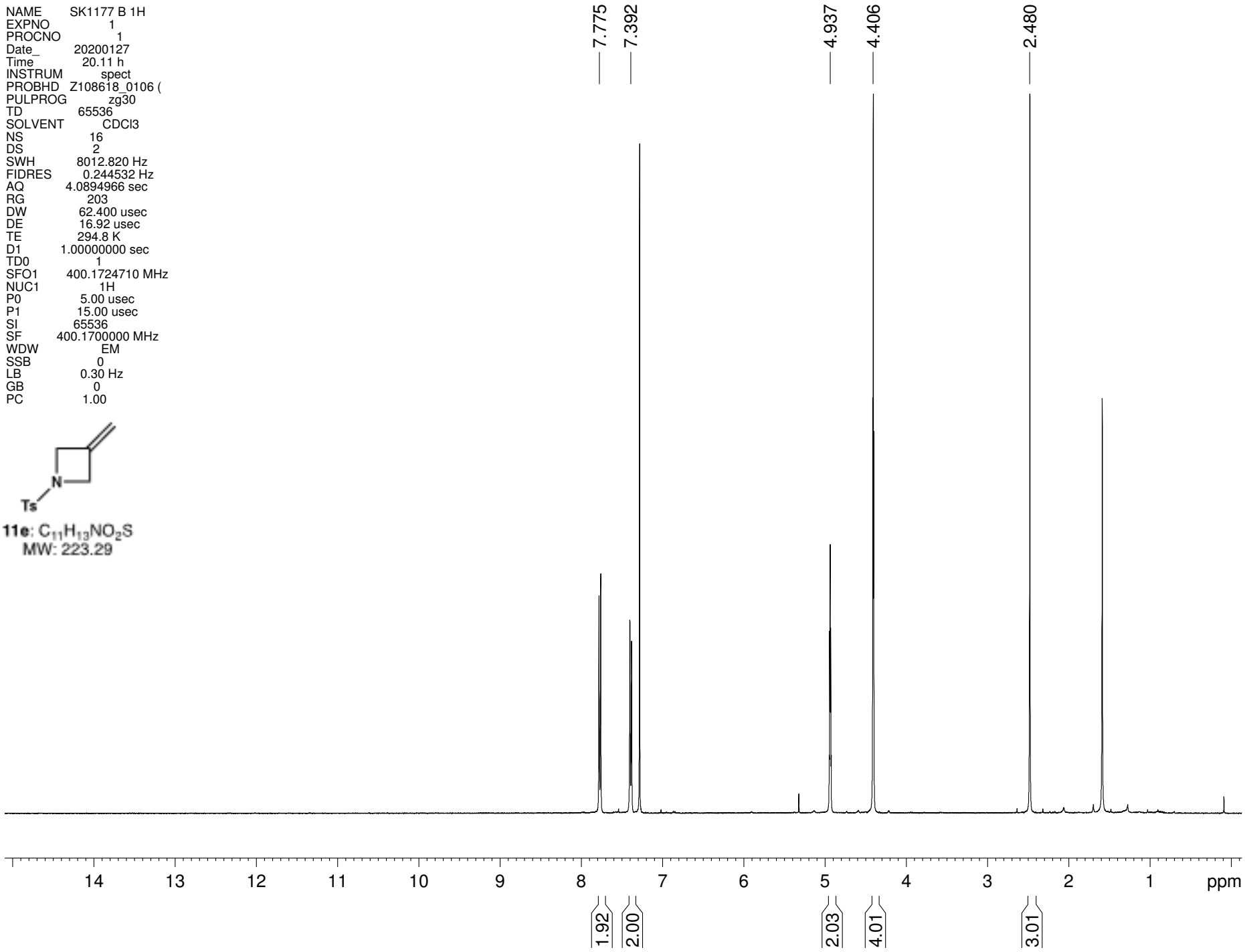
11c: $C_{15}H_{19}NO_2$
MW: 245.32


NAME NMB1290 13C
EXPNO 6
PROCNO 1
Date_ 20200723
Time 22.06 h
INSTRUM spect
PROBHD Z847801_0093 (
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 36231.883 Hz
FIDRES 1.105709 Hz
AQ 0.9044468 sec
RG 203
DW 13.800 usec
DE 6.50 usec
TE 299.6 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 150.9178988 MHz
NUC1 13C
P0 3.33 usec
P1 10.00 usec
SI 32768
SF 150.9028090 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
153.58
139.00
138.32
127.81
127.32
124.81
124.14
123.90
109.26
81.12
44.21
32.67
28.49
N
Boc
11c: C15H19NO2
MW: 245.32
200
180
160
140
120
100
80
60
40
20
0
ppm

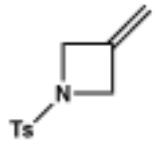
NAME	NMB1283
EXPNO	6
PROCNO	1
Date_	20200722
Time	15.03 h
INSTRUM	spect
PROBHD	Z108618_0106 (
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	16
DS	2
SWH	8012.820 Hz
FIDRES	0.244532 Hz
AQ	4.0894966 sec
RG	203
DW	62.400 usec
DE	16.92 usec
TE	297.5 K
D1	1.00000000 sec
TD0	1
SFO1	400.1724710 MHz
NUC1	1H
P0	5.00 usec
P1	15.00 usec
SI	65536
SF	400.1700000 MHz
WDW	no
SSB	0
LB	0.00 Hz
GB	0
PC	1.00



11d: $C_9H_{15}NO_2$
MW: 169.22

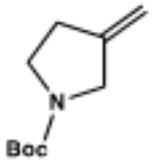

NAME SK1177 B 1H
EXPNO 1
PROCNO 1
Date_ 20200127
Time 20.11 h
INSTRUM spect
PROBHD Z108618_0106 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.244532 Hz
AQ 4.0894966 sec
RG 203
DW 62.400 usec
DE 16.92 usec
TE 294.8 K
D1 1.00000000 sec
TD0 1
SFO1 400.1724710 MHz
NUC1 1H
P0 5.00 usec
P1 15.00 usec
SI 65536
SF 400.1700000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
N
Ts
11e: C11H13NO2S
MW: 223.29
7.775
7.392
4.937
4.406
2.480
14
13
12
11
10
9
8
7
6
5
4
3
2
1
ppm
1.92
2.00
2.03
4.01
3.01

NAME NMB1227 13C
EXPNO 1
PROCNO 1
Date_ 20210121
Time 16.29 h
INSTRUM spect
PROBHD Z108618_0106 (
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 29
DS 4
SWH 24038.461 Hz
FIDRES 0.733596 Hz
AQ 1.3631988 sec
RG 203
DW 20.800 usec
DE 6.50 usec
TE 297.9 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 100.6328888 MHz
NUC1 13C
P0 3.53 usec
P1 10.60 usec
SI 32768
SF 100.6228280 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



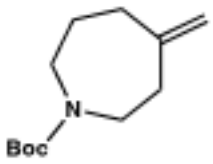
11e: $C_{11}H_{13}NO_2S$
MW: 223.29

NAME	NMB1284
EXPNO	2
PROCNO	1
Date_	20200715
Time	19.46 h
INSTRUM	spect
PROBHD	Z108618_0106 (
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	16
DS	2
SWH	8012.820 Hz
FIDRES	0.244532 Hz
AQ	4.0894966 sec
RG	203
DW	62.400 usec
DE	16.92 usec
TE	297.8 K
D1	1.00000000 sec
TD0	1
SFO1	400.1724710 MHz
NUC1	1H
P0	5.00 usec
P1	15.00 usec
SI	65536
SF	400.1700000 MHz
WDW	no
SSB	0
LB	0.00 Hz
GB	0
PC	1.00

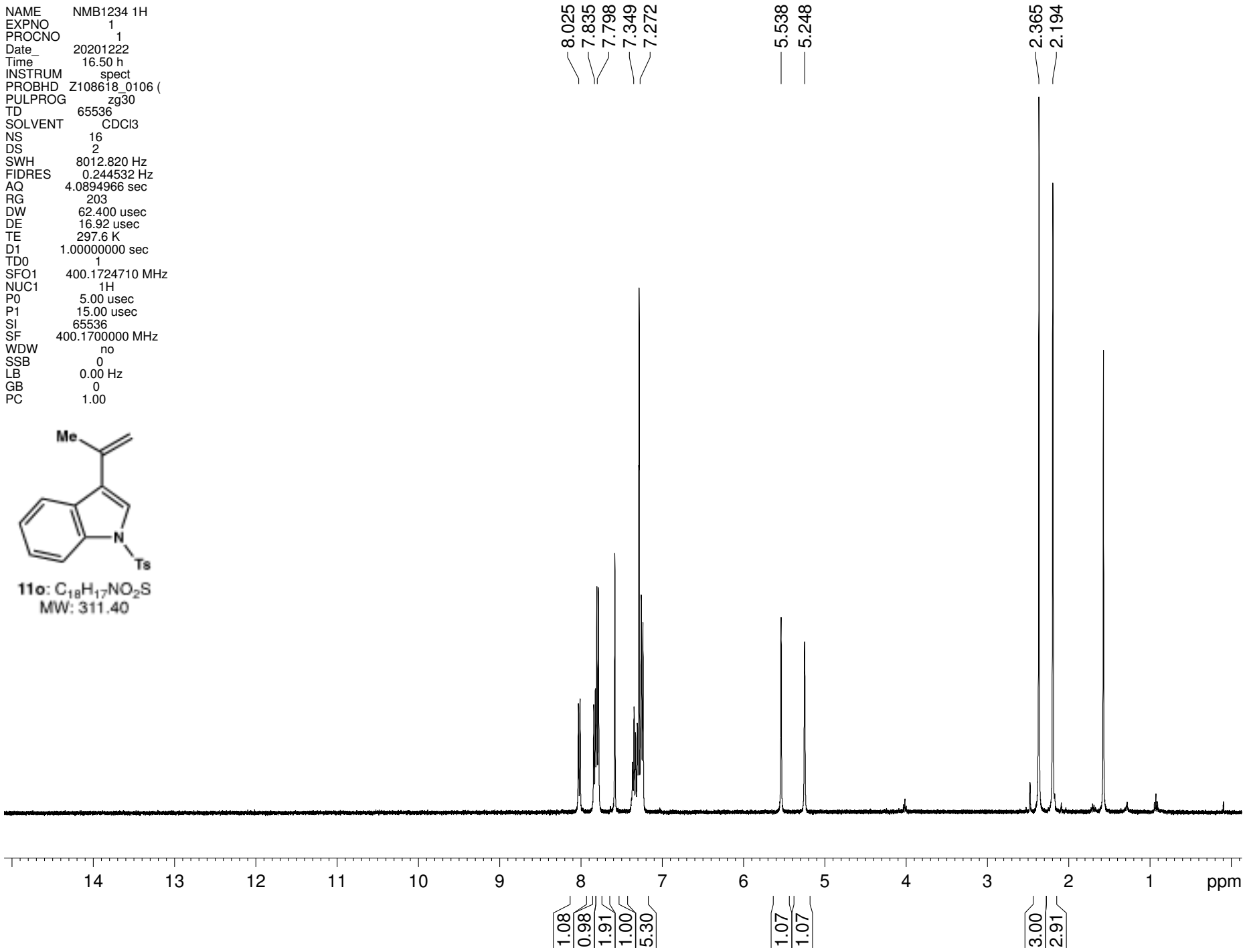


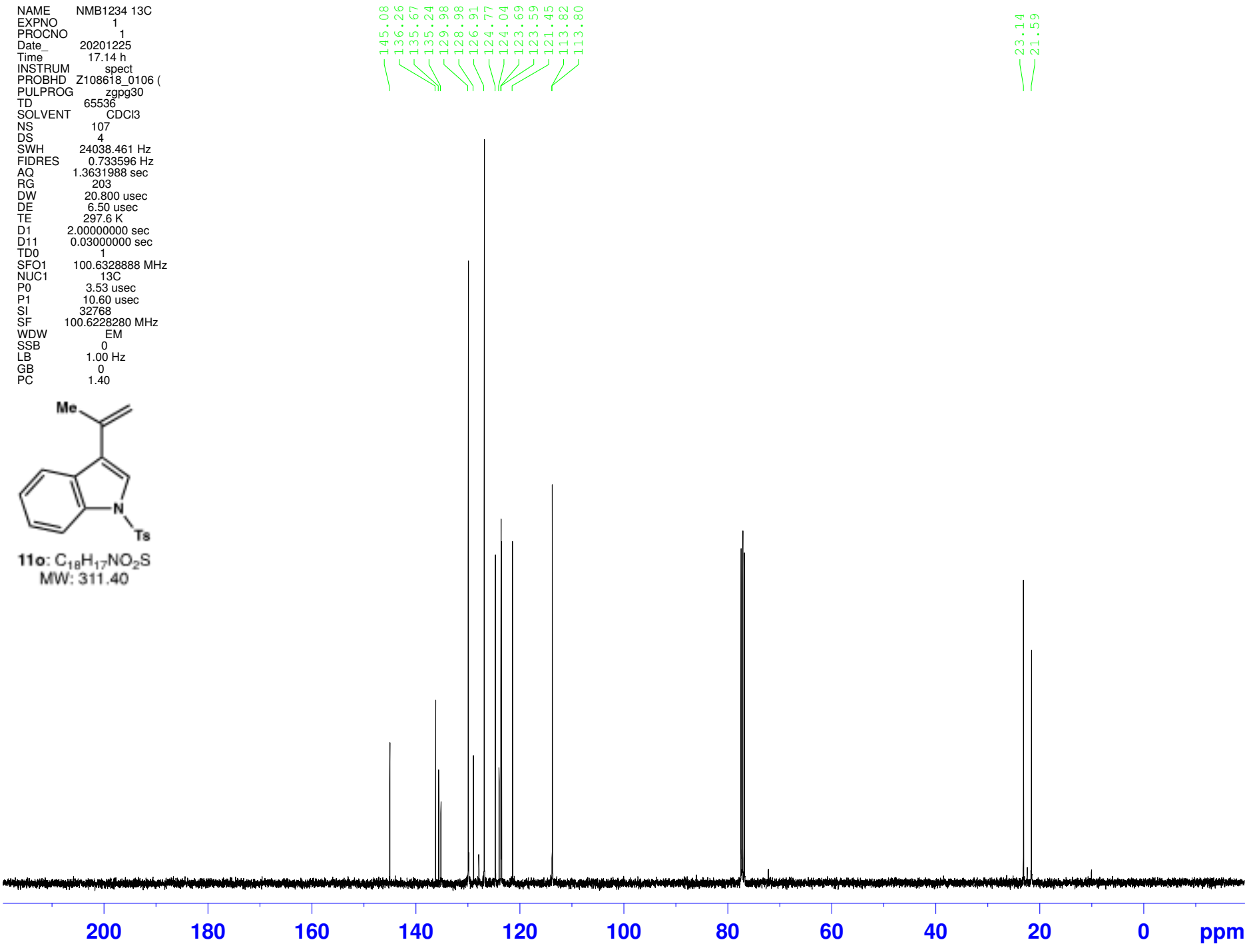
11f: $C_{10}H_{17}NO_2$
MW: 183.25

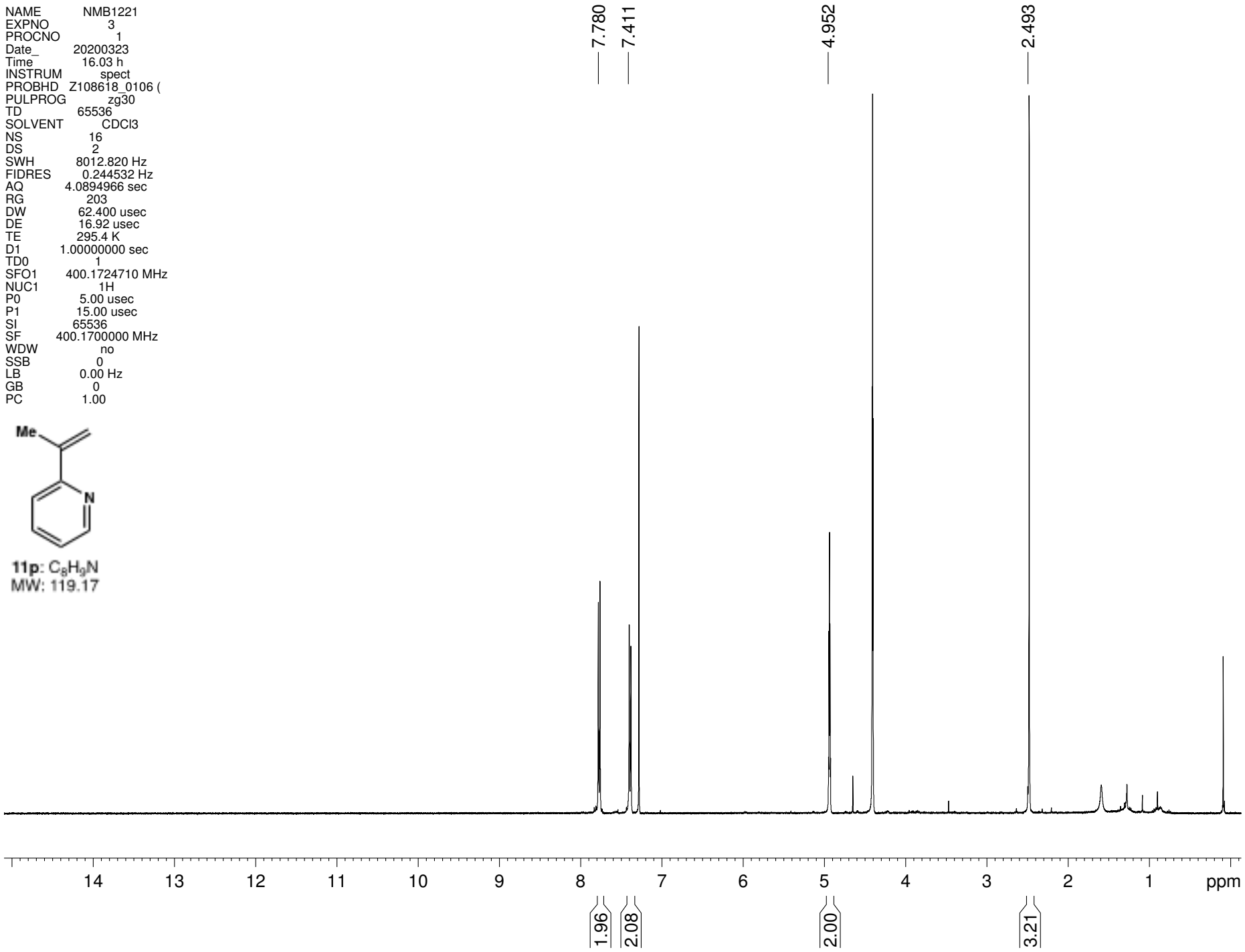
NAME	NMB1304
EXPNO	6
PROCNO	1
Date_	20200824
Time	13.23 h
INSTRUM	spect
PROBHD	Z847801_0093 (
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	16
DS	2
SWH	12019.230 Hz
FIDRES	0.366798 Hz
AQ	2.7263477 sec
RG	203
DW	41.600 usec
DE	10.42 usec
TE	299.0 K
D1	1.00000000 sec
TD0	1
SFO1	600.1337058 MHz
NUC1	1H
P0	5.00 usec
P1	15.00 usec
SI	65536
SF	600.1300000 MHz
WDW	no
SSB	0
LB	0.00 Hz
GB	0
PC	1.00



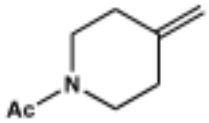
11g: $C_{12}H_{21}NO_2$
MW: 211.31


NAME NMB1234 1H
EXPNO 1
PROCNO 1
Date_ 20201222
Time 16.50 h
INSTRUM spect
PROBHD Z108618_0106 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.244532 Hz
AQ 4.0894966 sec
RG 203
DW 62.400 usec
DE 16.92 usec
TE 297.6 K
D1 1.00000000 sec
TD0 1
SFO1 400.1724710 MHz
NUC1 1H
P0 5.00 usec
P1 15.00 usec
SI 65536
SF 400.1700000 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
Me
N
Ts
11o: C18H17NO2S
MW: 311.40
8.025
7.835
7.798
7.349
7.272
5.538
5.248
2.365
2.194
14
13
12
11
10
9
8
7
6
5
4
3
2
1
ppm
1.08
0.98
1.91
1.00
5.30
1.07
1.07
3.00
2.91

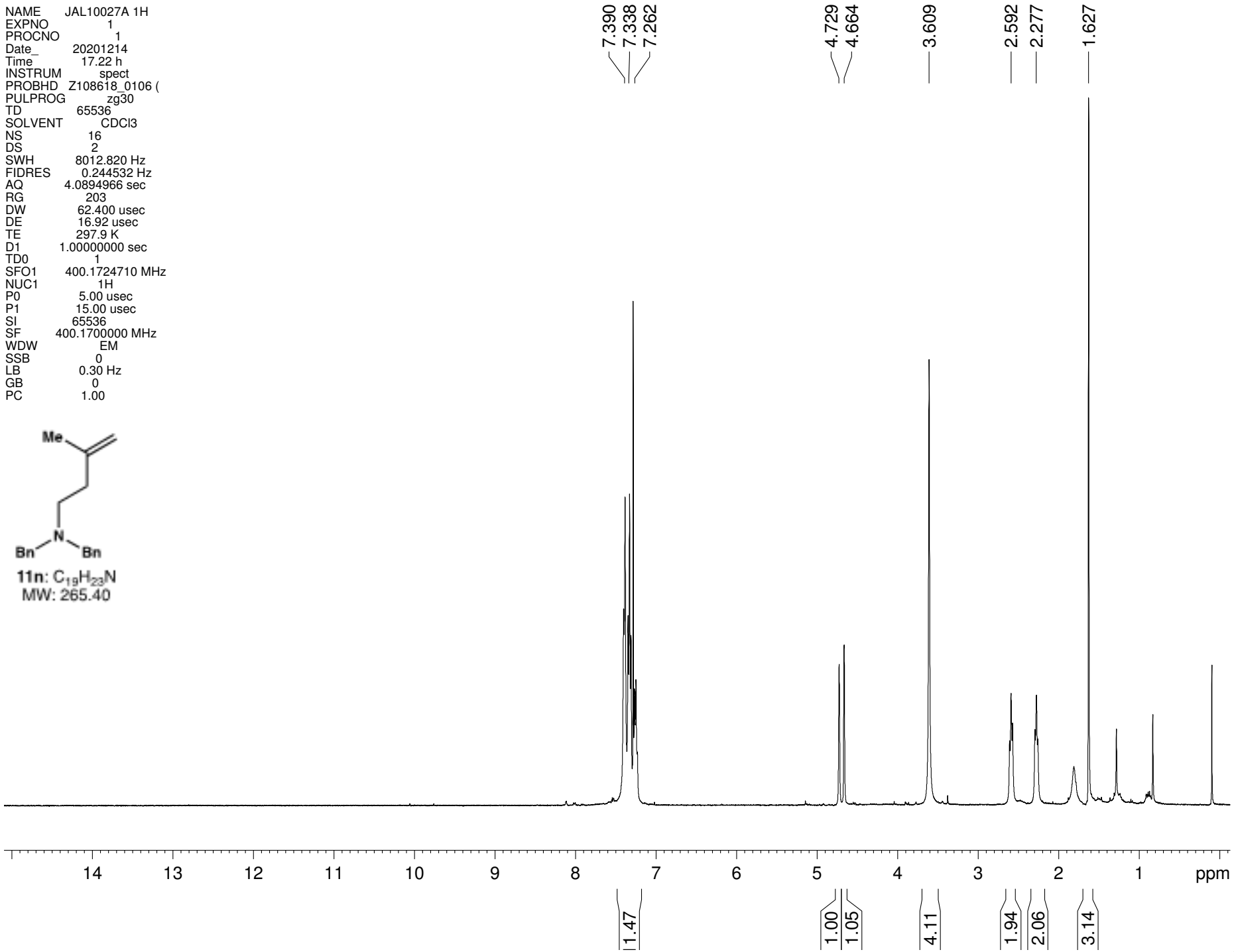

NAME NMB1234 13C
EXPNO 1
PROCNO 1
Date_ 20201225
Time 17.14 h
INSTRUM spect
PROBHD Z108618_0106 (
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 107
DS 4
SWH 24038.461 Hz
FIDRES 0.733596 Hz
AQ 1.3631988 sec
RG 203
DW 20.800 usec
DE 6.50 usec
TE 297.6 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 100.6328888 MHz
NUC1 13C
P0 3.53 usec
P1 10.60 usec
SI 32768
SF 100.6228280 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
145.08
136.26
135.67
135.24
129.98
128.98
126.91
124.77
124.04
123.69
123.59
121.45
113.82
113.80
23.14
21.59
Me
N
Ts
11o: C18H17NO2S
MW: 311.40
200
180
160
140
120
100
80
60
40
20
0
ppm

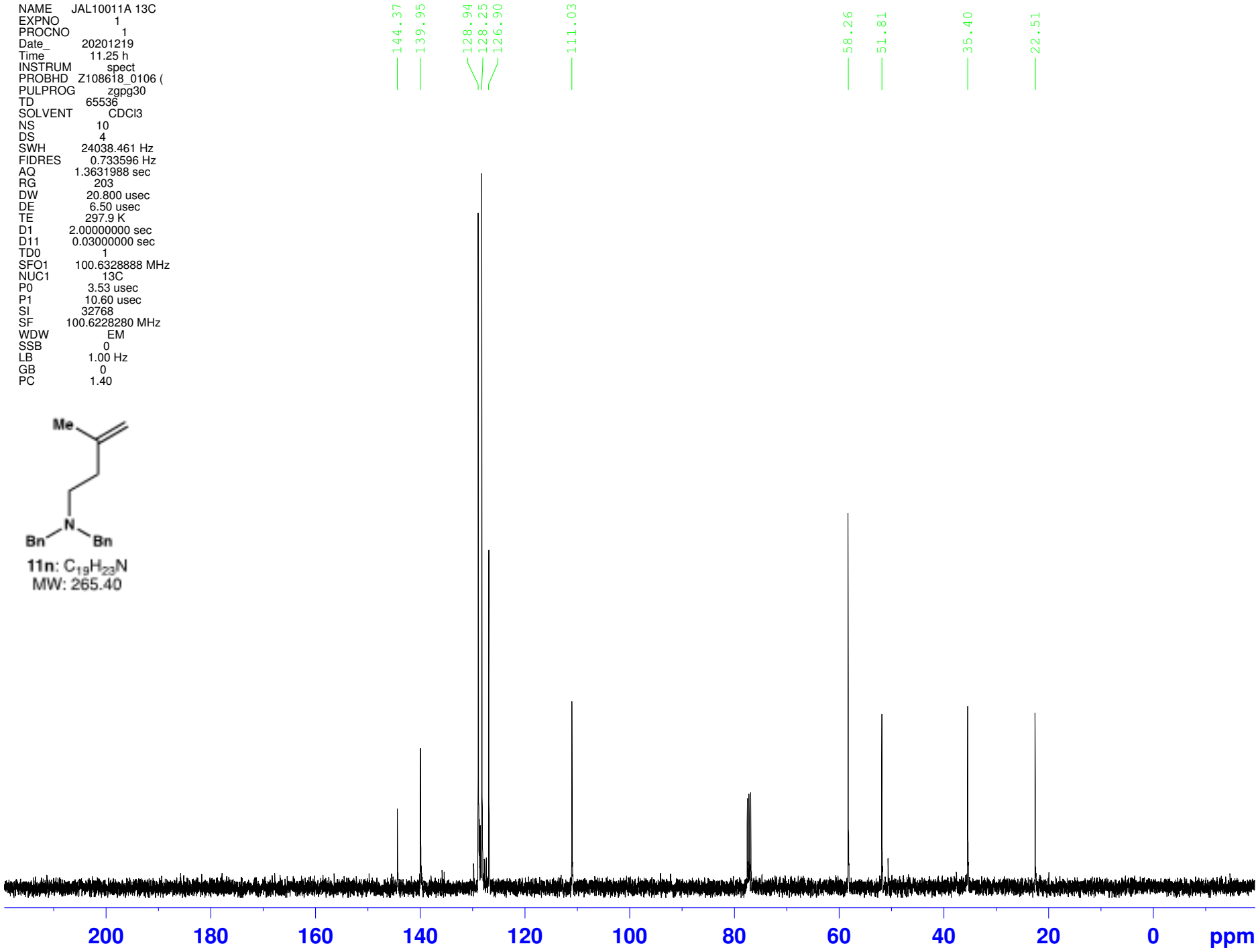

NAME NMB1221
EXPNO 3
PROCNO 1
Date_ 20200323
Time 16.03 h
INSTRUM spect
PROBHD Z108618_0106 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.244532 Hz
AQ 4.0894966 sec
RG 203
DW 62.400 usec
DE 16.92 usec
TE 295.4 K
D1 1.00000000 sec
TD0 1
SFO1 400.1724710 MHz
NUC1 1H
P0 5.00 usec
P1 15.00 usec
SI 65536
SF 400.1700000 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
Me
N
11p: C8H9N
MW: 119.17
7.780
7.411
4.952
2.493
14
13
12
11
10
9
8
7
6
5
4
3
2
1
ppm
1.96
2.08
2.00
3.21

NAME	NMB2115 B
EXPNO	1
PROCNO	1
Date_	20201214
Time	17.04 h
INSTRUM	spect
PROBHD	Z108618_0106 (
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	16
DS	2
SWH	8012.820 Hz
FIDRES	0.244532 Hz
AQ	4.0894966 sec
RG	203
DW	62.400 usec
DE	16.92 usec
TE	297.6 K
D1	1.00000000 sec
TD0	1
SFO1	400.1724710 MHz
NUC1	1H
P0	5.00 usec
P1	15.00 usec
SI	65536
SF	400.1700000 MHz
WDW	no
SSB	0
LB	0.00 Hz
GB	0
PC	1.00

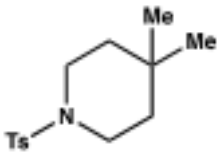


11b: $C_8H_{13}NO$
MW: 139.20

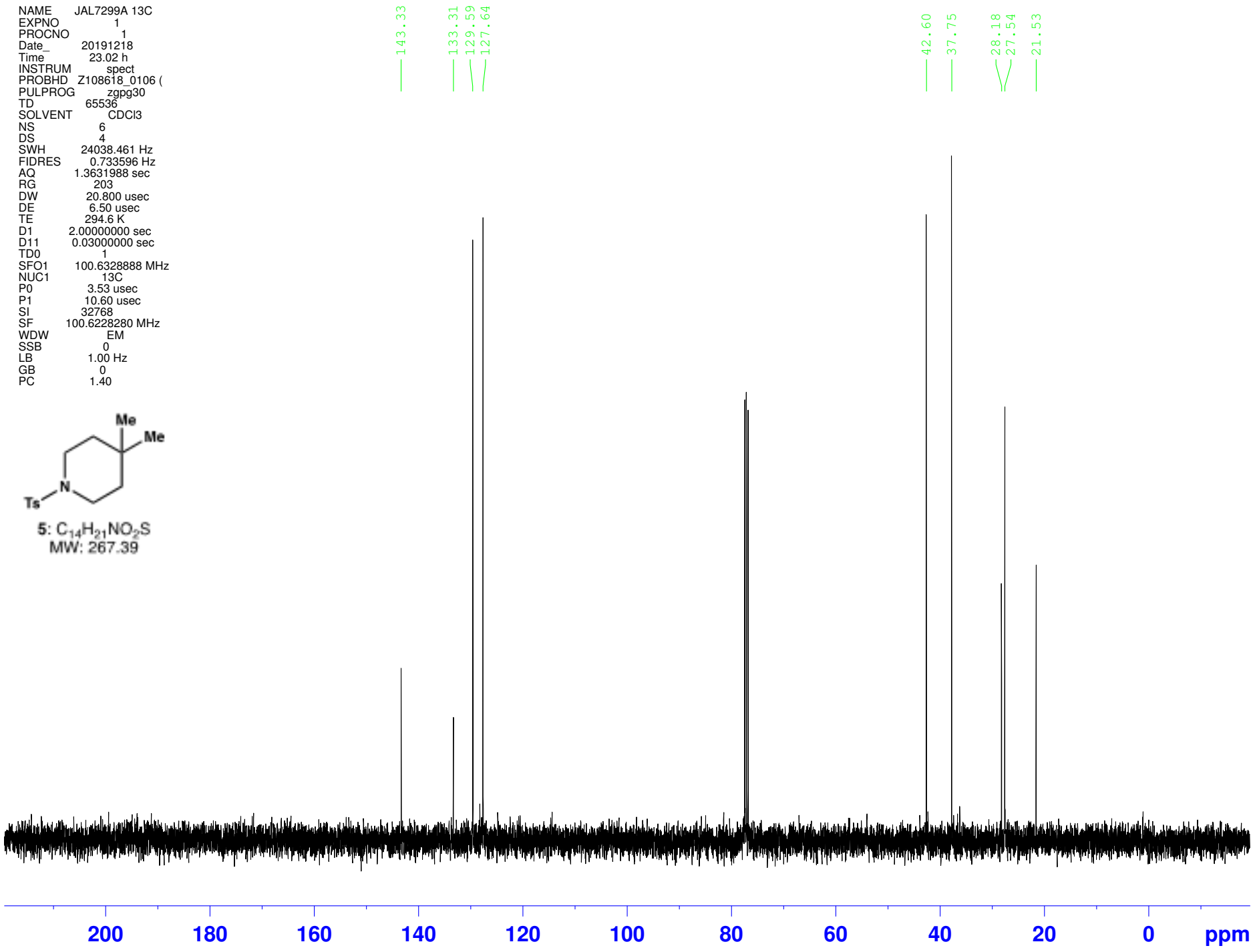

NAME JAL10027A 1H
EXPNO 1
PROCNO 1
Date_ 20201214
Time 17.22 h
INSTRUM spect
PROBHD Z108618_0106 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.244532 Hz
AQ 4.0894966 sec
RG 203
DW 62.400 usec
DE 16.92 usec
TE 297.9 K
D1 1.00000000 sec
TD0 1
SFO1 400.1724710 MHz
NUC1 1H
P0 5.00 usec
P1 15.00 usec
SI 65536
SF 400.1700000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
Me
N
Bn
Bn
11n: C19H23N
MW: 265.40
7.390
7.338
7.262
4.729
4.664
3.609
2.592
2.277
1.627
14
13
12
11
10
9
8
7
6
5
4
3
2
1
ppm
1.47
1.00
1.05
4.11
1.94
2.06
3.14

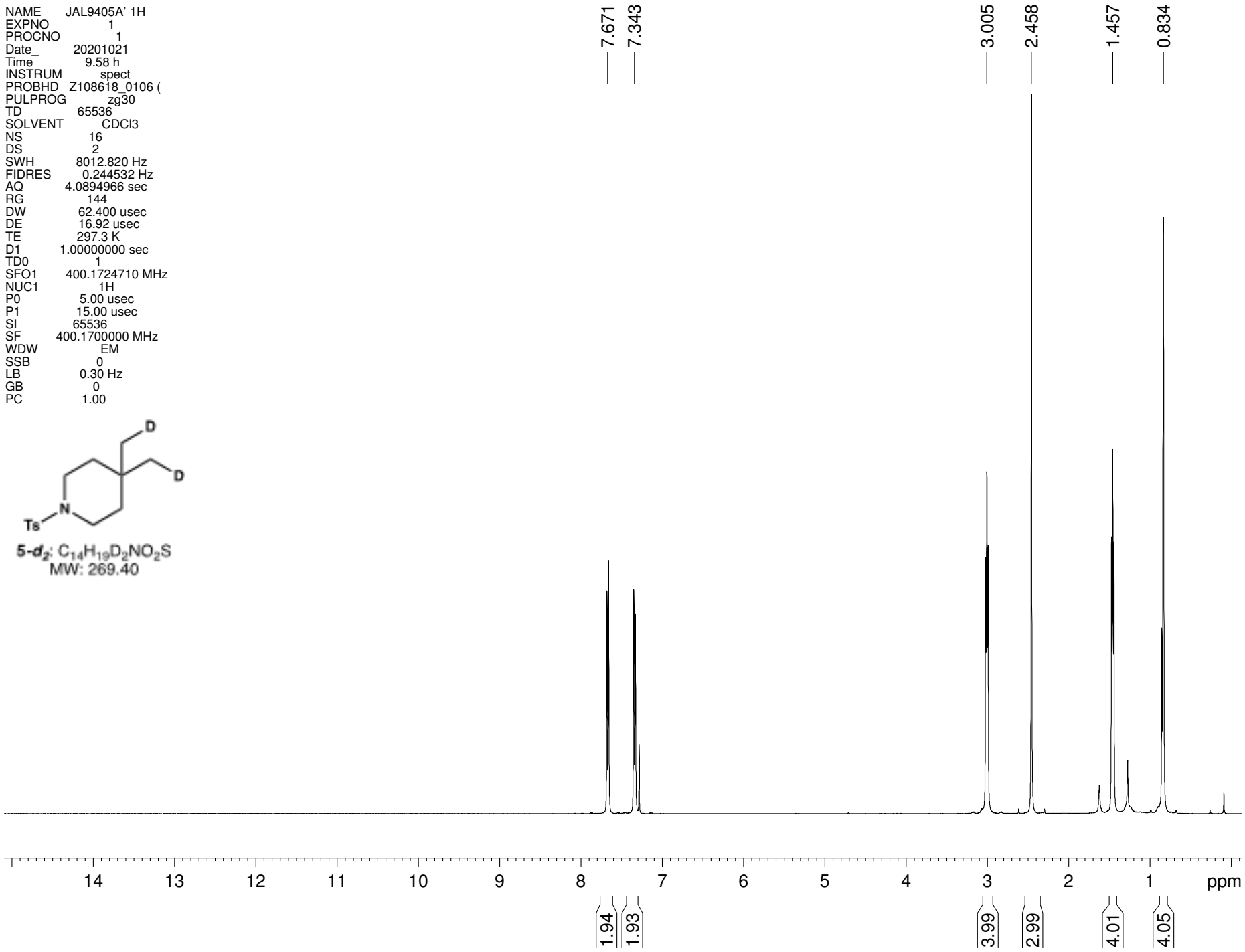

NAME JAL10011A 13C
EXPNO 1
PROCNO 1
Date_ 20201219
Time 11.25 h
INSTRUM spect
PROBHD Z108618_0106 (
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 10
DS 4
SWH 24038.461 Hz
FIDRES 0.733596 Hz
AQ 1.3631988 sec
RG 203
DW 20.800 usec
DE 6.50 usec
TE 297.9 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 100.6328888 MHz
NUC1 13C
P0 3.53 usec
P1 10.60 usec
SI 32768
SF 100.6228280 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
144.37
139.95
128.94
128.25
126.90
111.03
58.26
51.81
35.40
22.51
Me
N
Bn
Bn
11n: C19H23N
MW: 265.40
200
180
160
140
120
100
80
60
40
20
0
ppm

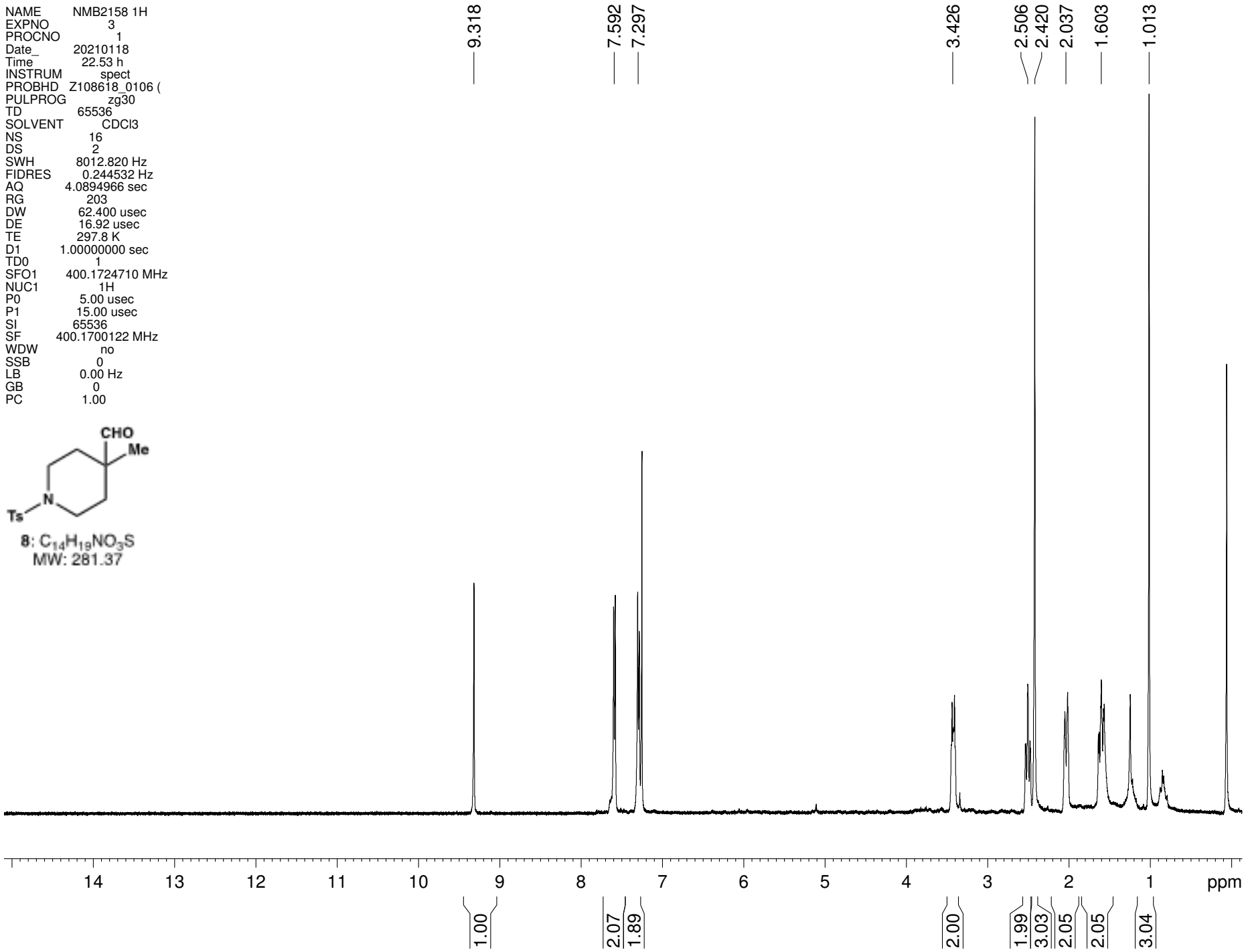
NAME JAL7295Ai 1H
EXPNO 1
PROCNO 1
Date_ 20191217
Time 20.32 h
INSTRUM spect
PROBHD Z108618_0106 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.244532 Hz
AQ 4.0894966 sec
RG 32
DW 62.400 usec
DE 16.92 usec
TE 295.3 K
D1 1.00000000 sec
TD0 1
SFO1 400.1724710 MHz
NUC1 1H
P0 5.00 usec
P1 15.00 usec
SI 65536
SF 400.1700000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

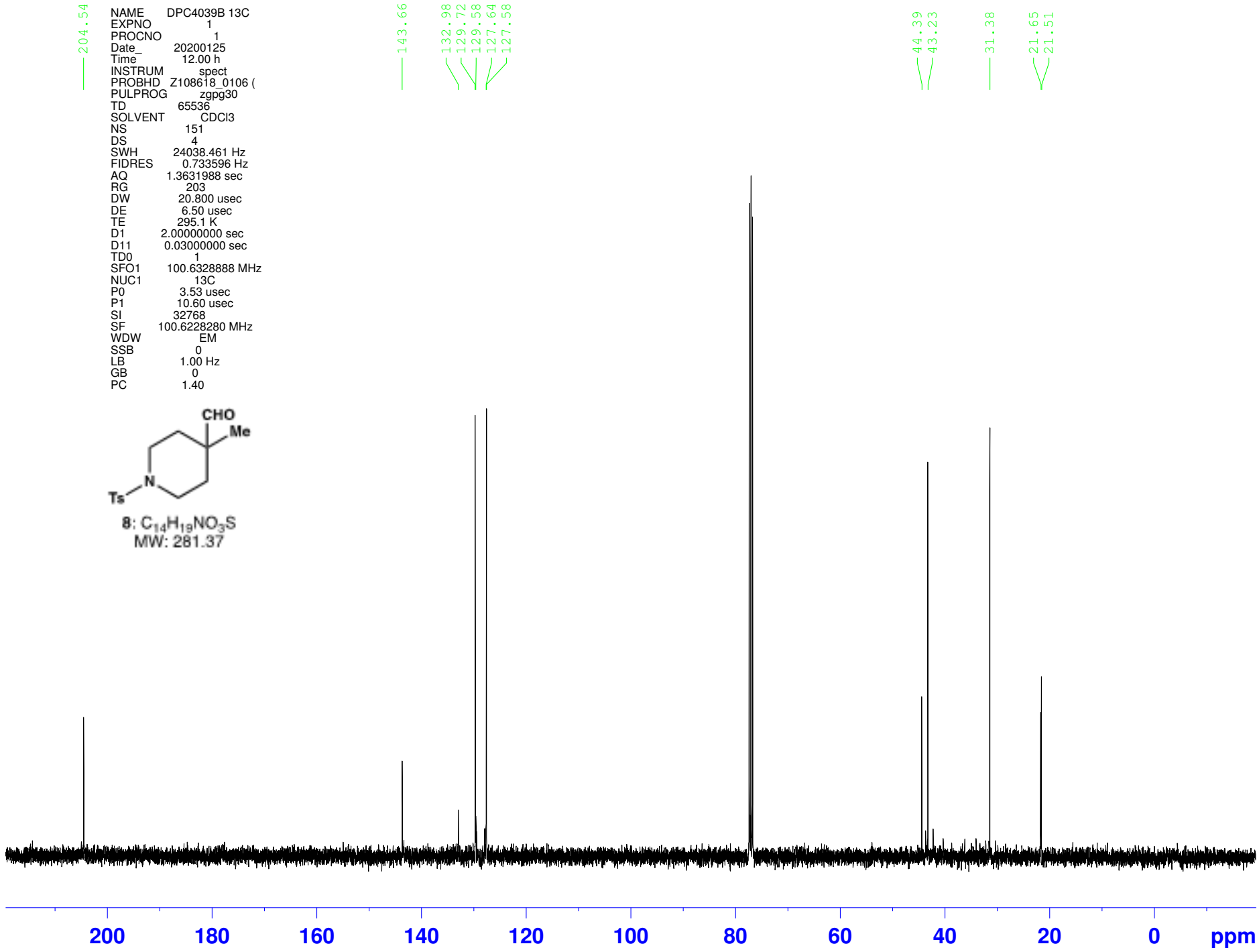


5: $C_{14}H_{21}NO_2S$
MW: 267.39

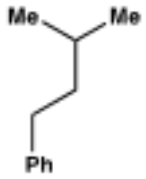

NAME JAL7299A 13C
EXPNO 1
PROCNO 1
Date_ 20191218
Time 23.02 h
INSTRUM spect
PROBHD Z108618_0106 (
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 6
DS 4
SWH 24038.461 Hz
FIDRES 0.733596 Hz
AQ 1.3631988 sec
RG 203
DW 20.800 usec
DE 6.50 usec
TE 294.6 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 100.6328888 MHz
NUC1 13C
P0 3.53 usec
P1 10.60 usec
SI 32768
SF 100.6228280 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
143.33
133.31
129.59
127.64
42.60
37.75
28.18
27.54
21.53
Me
Me
N
Ts
5: C14H21NO2S
MW: 267.39
200
180
160
140
120
100
80
60
40
20
0
ppm


NAME JAL9405A' 1H
EXPNO 1
PROCNO 1
Date_ 20201021
Time 9.58 h
INSTRUM spect
PROBHD Z108618_0106 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.244532 Hz
AQ 4.0894966 sec
RG 144
DW 62.400 usec
DE 16.92 usec
TE 297.3 K
D1 1.00000000 sec
TD0 1
SFO1 400.1724710 MHz
NUC1 1H
P0 5.00 usec
P1 15.00 usec
SI 65536
SF 400.1700000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
D
D
N
Ts
5-d2: C14H19D2NO2S
MW: 269.40
7.671
7.343
3.005
2.458
1.457
0.834
14
13
12
11
10
9
8
7
6
5
4
3
2
1
ppm
1.94
1.93
3.99
2.99
4.01
4.05

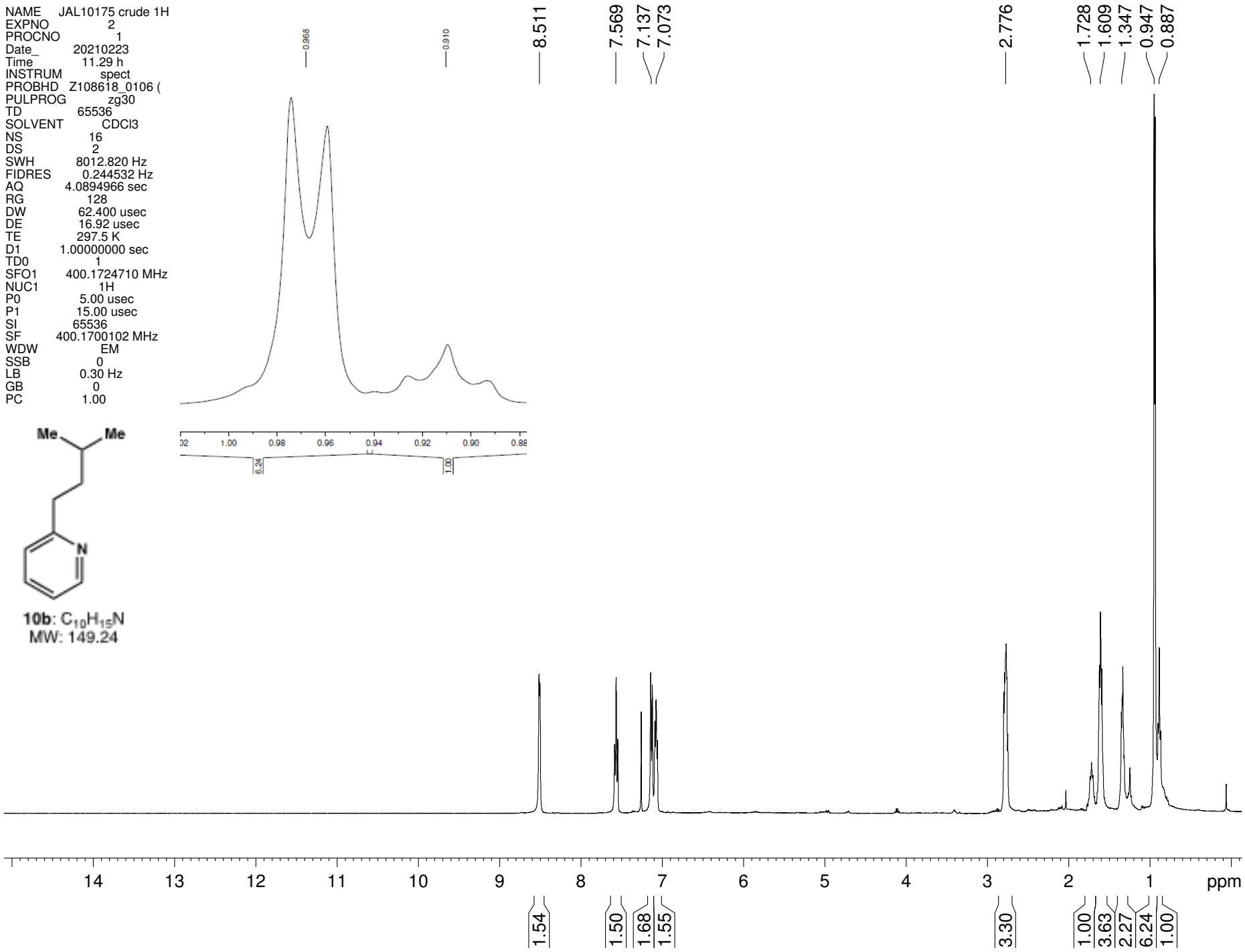

NAME NMB2158 1H
EXPNO 3
PROCNO 1
Date_ 20210118
Time 22.53 h
INSTRUM spect
PROBHD Z108618_0106 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.244532 Hz
AQ 4.0894966 sec
RG 203
DW 62.400 usec
DE 16.92 usec
TE 297.8 K
D1 1.00000000 sec
TD0 1
SFO1 400.1724710 MHz
NUC1 1H
P0 5.00 usec
P1 15.00 usec
SI 65536
SF 400.1700122 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
CHO
Me
N
Ts
8: C14H19NO3S
MW: 281.37
9.318
7.592
7.297
3.426
2.506
2.420
2.037
1.603
1.013
14
13
12
11
10
9
8
7
6
5
4
3
2
1
ppm
1.00
2.07
1.89
2.00
1.99
3.03
2.05
2.05
3.04

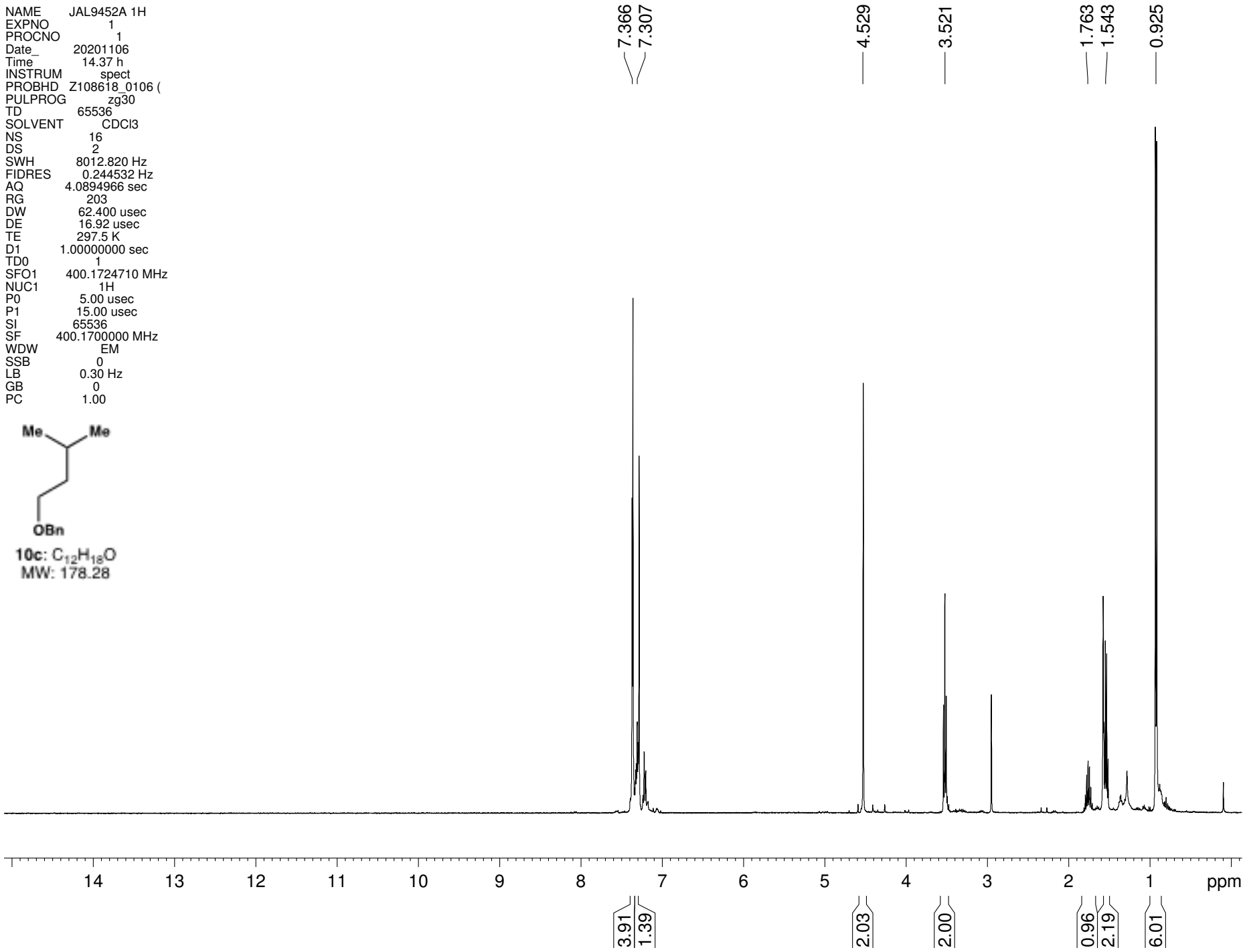

204.54
143.66
132.98
129.72
129.58
127.64
127.58
44.39
43.23
31.38
21.65
21.51
NAME DPC4039B 13C
EXPNO 1
PROCNO 1
Date_ 20200125
Time 12.00 h
INSTRUM spect
PROBHD Z108618_0106 (
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 151
DS 4
SWH 24038.461 Hz
FIDRES 0.733596 Hz
AQ 1.3631988 sec
RG 203
DW 20.800 usec
DE 6.50 usec
TE 295.1 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 100.6328888 MHz
NUC1 13C
P0 3.53 usec
P1 10.60 usec
SI 32768
SF 100.6228280 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
CHO
Me
N
Ts
8: C14H19NO3S
MW: 281.37
200
180
160
140
120
100
80
60
40
20
0
ppm

NAME NMB2166 A
EXPNO 1
PROCNO 1
Date_ 20210201
Time 22.15 h
INSTRUM spect
PROBHD Z108618_0106 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.244532 Hz
AQ 4.0894966 sec
RG 203
DW 62.400 usec
DE 16.92 usec
TE 297.8 K
D1 1.00000000 sec
TD0 1
SFO1 400.1724710 MHz
NUC1 1H
P0 5.00 usec
P1 15.00 usec
SI 65536
SF 400.1700000 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

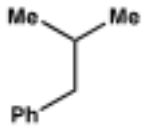


10a: $C_{11}H_{16}$
MW: 148.25


NAME JAL10175 crude 1H
EXPNO 2
PROCNO 1
Date_ 20210223
Time 11.29 h
INSTRUM spect
PROBHD Z108618_0106 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.244532 Hz
AQ 4.0894966 sec
RG 128
DW 62.400 usec
DE 16.92 usec
TE 297.5 K
D1 1.00000000 sec
TD0 1
SFO1 400.1724710 MHz
NUC1 1H
P0 5.00 usec
P1 15.00 usec
SI 65536
SF 400.1700102 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
0.968
0.910
8.511
7.569
7.137
7.073
2.776
1.728
1.609
1.347
0.947
0.887
Me
Me
N
10b: C10H15N
MW: 149.24
ppm
1.54
1.50
1.68
1.55
3.30
1.00
3.63
2.27
6.24
1.00

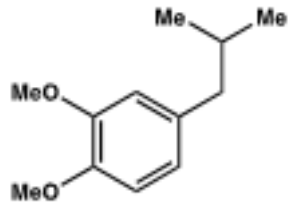

NAME JAL9452A 1H
EXPNO 1
PROCNO 1
Date_ 20201106
Time 14.37 h
INSTRUM spect
PROBHD Z108618_0106 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.244532 Hz
AQ 4.0894966 sec
RG 203
DW 62.400 usec
DE 16.92 usec
TE 297.5 K
D1 1.00000000 sec
TD0 1
SFO1 400.1724710 MHz
NUC1 1H
P0 5.00 usec
P1 15.00 usec
SI 65536
SF 400.1700000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
Me
Me
OBn
10c: C12H18O
MW: 178.28
7.366
7.307
4.529
3.521
1.763
1.543
0.925
14
13
12
11
10
9
8
7
6
5
4
3
2
1
ppm
3.91
1.39
2.03
2.00
0.96
2.19
6.01

NAME JAL10170A 1H
EXPNO 1
PROCNO 1
Date_ 20210220
Time 15.19 h
INSTRUM spect
PROBHD Z108618_0106 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.244532 Hz
AQ 4.0894966 sec
RG 203
DW 62.400 usec
DE 16.92 usec
TE 297.6 K
D1 1.00000000 sec
TD0 1
SFO1 400.1724710 MHz
NUC1 1H
P0 5.00 usec
P1 15.00 usec
SI 65536
SF 400.1700000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



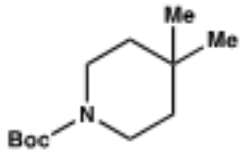
10d: $C_{10}H_{14}$
MW: 134.22

NAME JAL10136A–A 1H
EXPNO 1
PROCNO 1
Date_ 20210202
Time 17.25 h
INSTRUM spect
PROBHD Z108618_0106 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 8012.820 Hz
FIDRES 0.244532 Hz
AQ 4.0894966 sec
RG 203
DW 62.400 usec
DE 16.92 usec
TE 297.7 K
D1 1.00000000 sec
TD0 1
SFO1 400.1724710 MHz
NUC1 1H
P0 5.00 usec
P1 15.00 usec
SI 65536
SF 400.1700000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



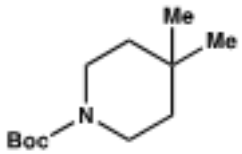
10e: $C_{12}H_{18}O_2$
MW: 194.27

NAME	NMB1281
EXPNO	3
PROCNO	1
Date_	20200712
Time	22.49 h
INSTRUM	spect
PROBHD	Z108618_0106 (
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	16
DS	2
SWH	8012.820 Hz
FIDRES	0.244532 Hz
AQ	4.0894966 sec
RG	203
DW	62.400 usec
DE	16.92 usec
TE	297.7 K
D1	1.00000000 sec
TD0	1
SFO1	400.1724710 MHz
NUC1	1H
P0	5.00 usec
P1	15.00 usec
SI	65536
SF	400.1700000 MHz
WDW	no
SSB	0
LB	0.00 Hz
GB	0
PC	1.00



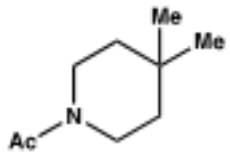
12a: $C_{12}H_{23}NO_2$
MW: 213.32

NAME	NMB1281
EXPNO	8
PROCNO	1
Date_	20200713
Time	9.51 h
INSTRUM	spect
PROBHD	Z108618_0106 (
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	200
DS	4
SWH	24038.461 Hz
FIDRES	0.733596 Hz
AQ	1.3631988 sec
RG	203
DW	20.800 usec
DE	6.50 usec
TE	298.3 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
SFO1	100.6328888 MHz
NUC1	13C
P0	3.53 usec
P1	10.60 usec
SI	32768
SF	100.6228280 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

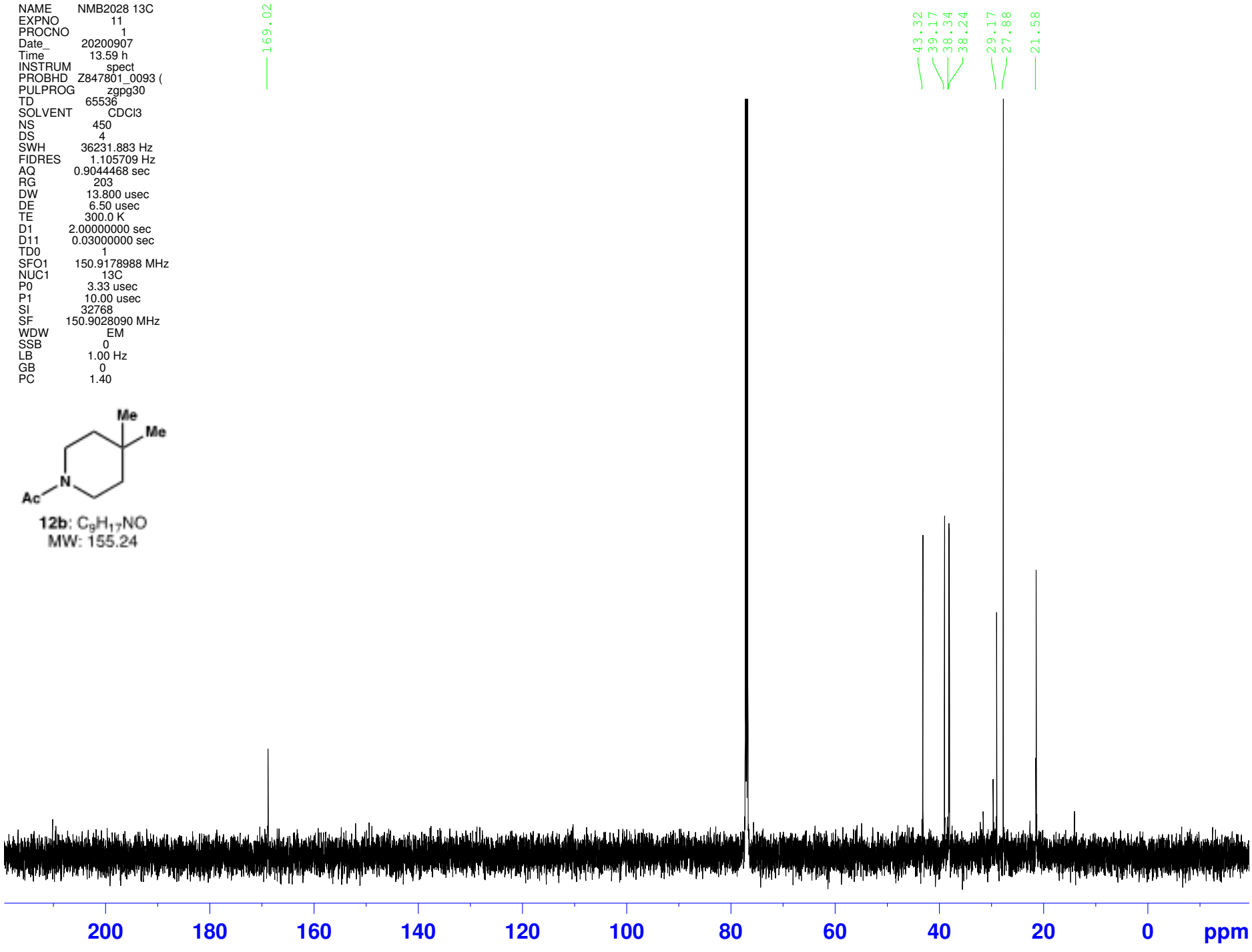


12a: $C_{12}H_{23}NO_2$
MW: 213.32

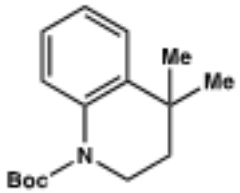
NAME	NMB2132 1H
EXPNO	1
PROCNO	1
Date_	20201230
Time	14.09 h
INSTRUM	spect
PROBHD	Z108618_0106 (
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	16
DS	2
SWH	8012.820 Hz
FIDRES	0.244532 Hz
AQ	4.0894966 sec
RG	203
DW	62.400 usec
DE	16.92 usec
TE	298.1 K
D1	1.00000000 sec
TD0	1
SFO1	400.1724710 MHz
NUC1	1H
P0	5.00 usec
P1	15.00 usec
SI	65536
SF	400.1700000 MHz
WDW	no
SSB	0
LB	0.00 Hz
GB	0
PC	1.00



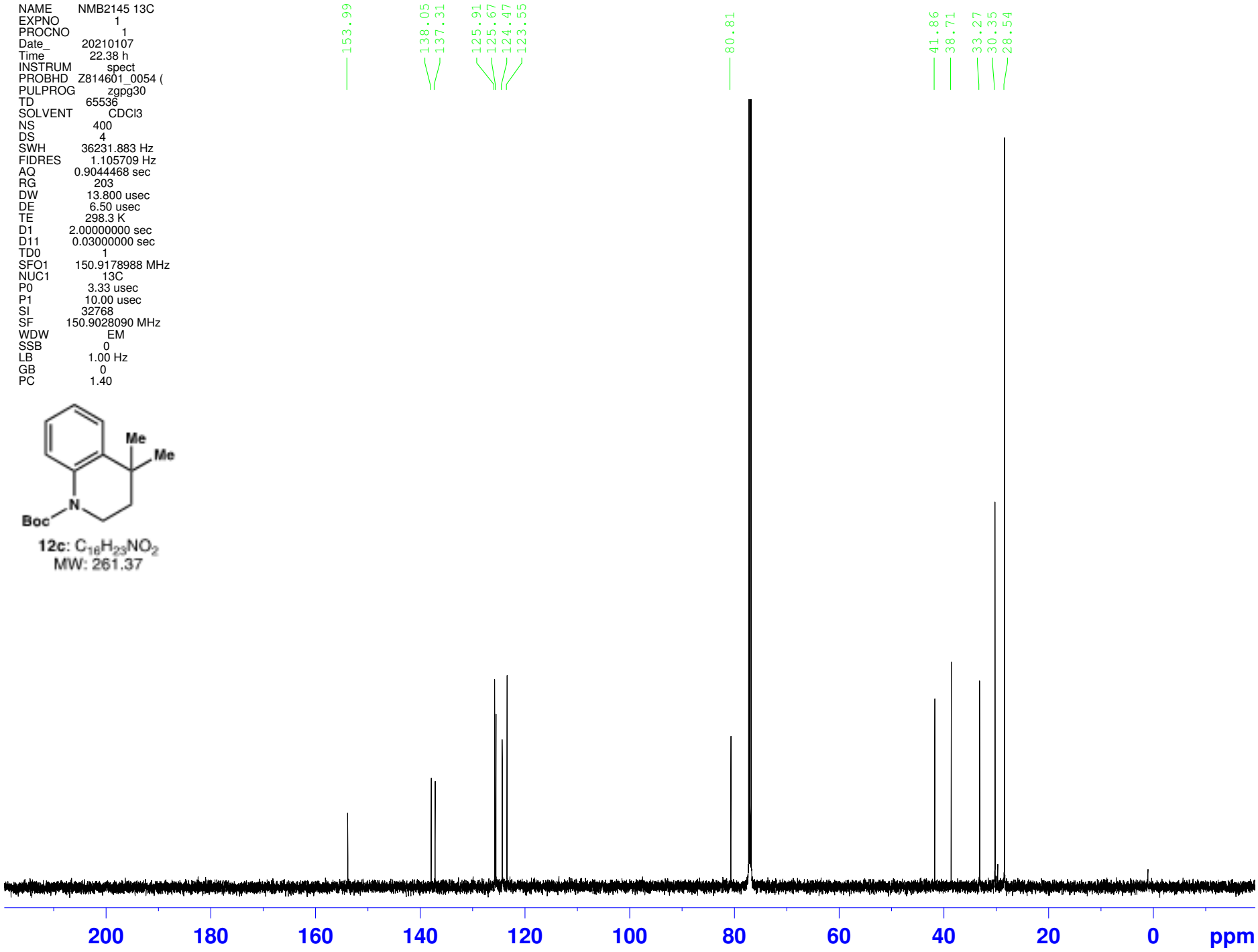
12b: $C_9H_{17}NO$
MW: 155.24

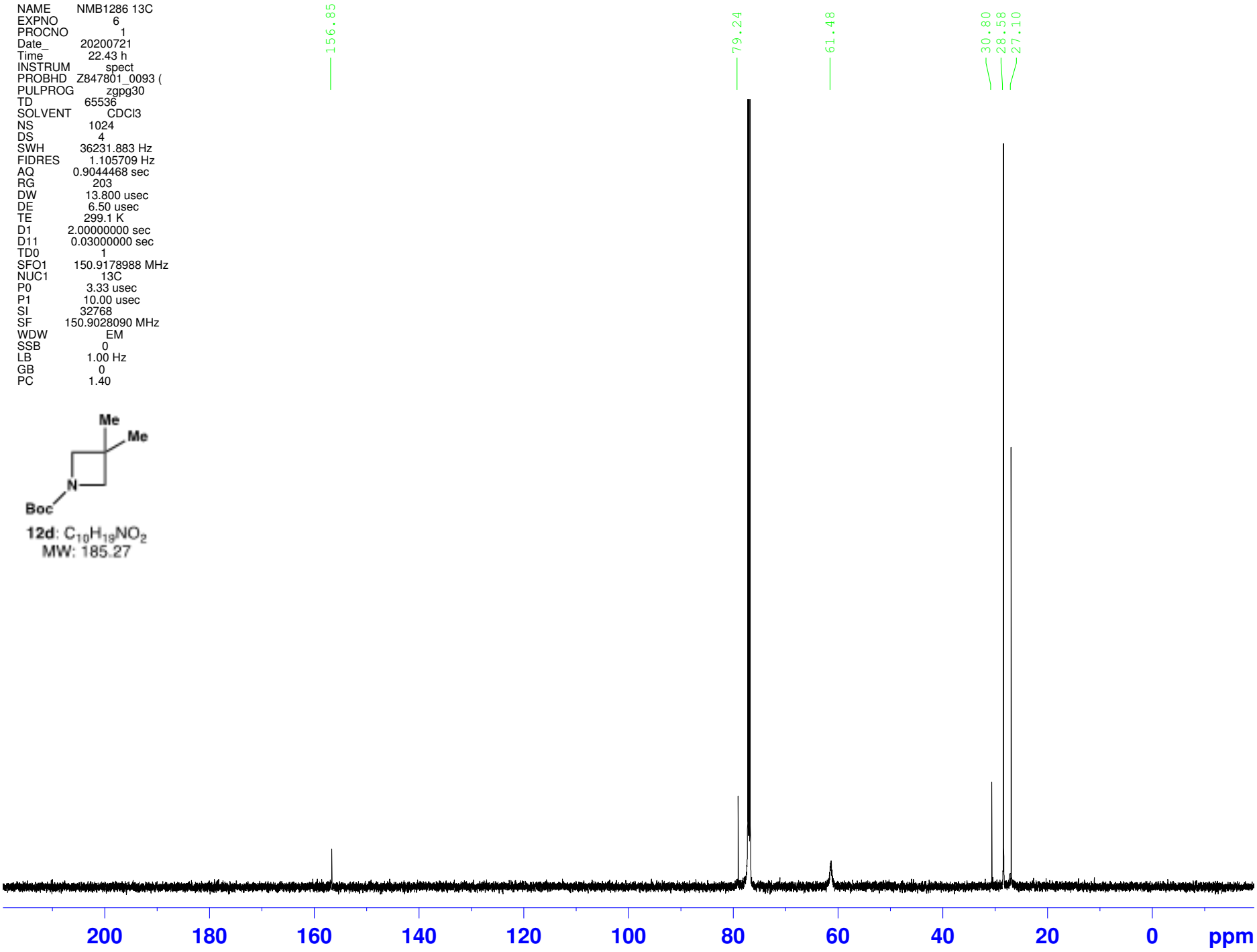

NAME NMB2028 13C
EXPNO 11
PROCNO 1
Date_ 20200907
Time 13.59 h
INSTRUM spect
PROBHD Z847801_0093 (
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 450
DS 4
SWH 36231.883 Hz
FIDRES 1.105709 Hz
AQ 0.9044468 sec
RG 203
DW 13.800 usec
DE 6.50 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 150.9178988 MHz
NUC1 13C
P0 3.33 usec
P1 10.00 usec
SI 32768
SF 150.9028090 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
169.02
43.32
39.17
38.34
38.24
29.17
27.88
21.58
Me
Me
N
Ac
12b: C9H17NO
MW: 155.24
200
180
160
140
120
100
80
60
40
20
0
ppm

NAME	NMB2145 1H
EXPNO	1
PROCNO	1
Date_	20210107
Time	22.04 h
INSTRUM	spect
PROBHD	Z108618_0106 (
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	16
DS	2
SWH	8012.820 Hz
FIDRES	0.244532 Hz
AQ	4.0894966 sec
RG	203
DW	62.400 usec
DE	16.92 usec
TE	297.5 K
D1	1.00000000 sec
TD0	1
SFO1	400.1724710 MHz
NUC1	1H
P0	5.00 usec
P1	15.00 usec
SI	65536
SF	400.1700099 MHz
WDW	no
SSB	0
LB	0.00 Hz
GB	0
PC	1.00

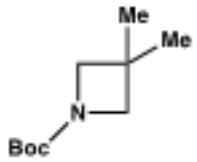


12c: $C_{16}H_{23}NO_2$
MW: 261.37


NAME NMB2145 13C
EXPNO 1
PROCNO 1
Date_ 20210107
Time 22.38 h
INSTRUM spect
PROBHD Z814601_0054 (
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 400
DS 4
SWH 36231.883 Hz
FIDRES 1.105709 Hz
AQ 0.9044468 sec
RG 203
DW 13.800 usec
DE 6.50 usec
TE 298.3 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 150.9178988 MHz
NUC1 13C
P0 3.33 usec
P1 10.00 usec
SI 32768
SF 150.9028090 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
153.99
138.05
137.31
125.91
125.67
124.47
123.55
80.81
41.86
38.71
33.27
30.35
28.54
Me
Me
N
Boc
12c: C16H23NO2
MW: 261.37
200
180
160
140
120
100
80
60
40
20
0
ppm

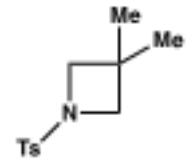

NAME NMB1286 13C
EXPNO 6
PROCNO 1
Date_ 20200721
Time 22.43 h
INSTRUM spect
PROBHD Z847801_0093 (
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 36231.883 Hz
FIDRES 1.105709 Hz
AQ 0.9044468 sec
RG 203
DW 13.800 usec
DE 6.50 usec
TE 299.1 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 150.9178988 MHz
NUC1 13C
P0 3.33 usec
P1 10.00 usec
SI 32768
SF 150.9028090 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
156.85
79.24
61.48
30.80
28.58
27.10
Me
Me
N
Boc
12d: C10H19NO2
MW: 185.27
200
180
160
140
120
100
80
60
40
20
0
ppm

NAME	NMB1286
EXPNO	7
PROCNO	1
Date_	20200717
Time	19.22 h
INSTRUM	spect
PROBHD	Z108618_0106 (
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	16
DS	2
SWH	8012.820 Hz
FIDRES	0.244532 Hz
AQ	4.0894966 sec
RG	161
DW	62.400 usec
DE	16.92 usec
TE	297.9 K
D1	1.00000000 sec
TD0	1
SFO1	400.1724710 MHz
NUC1	1H
P0	5.00 usec
P1	15.00 usec
SI	65536
SF	400.1700000 MHz
WDW	no
SSB	0
LB	0.00 Hz
GB	0
PC	1.00

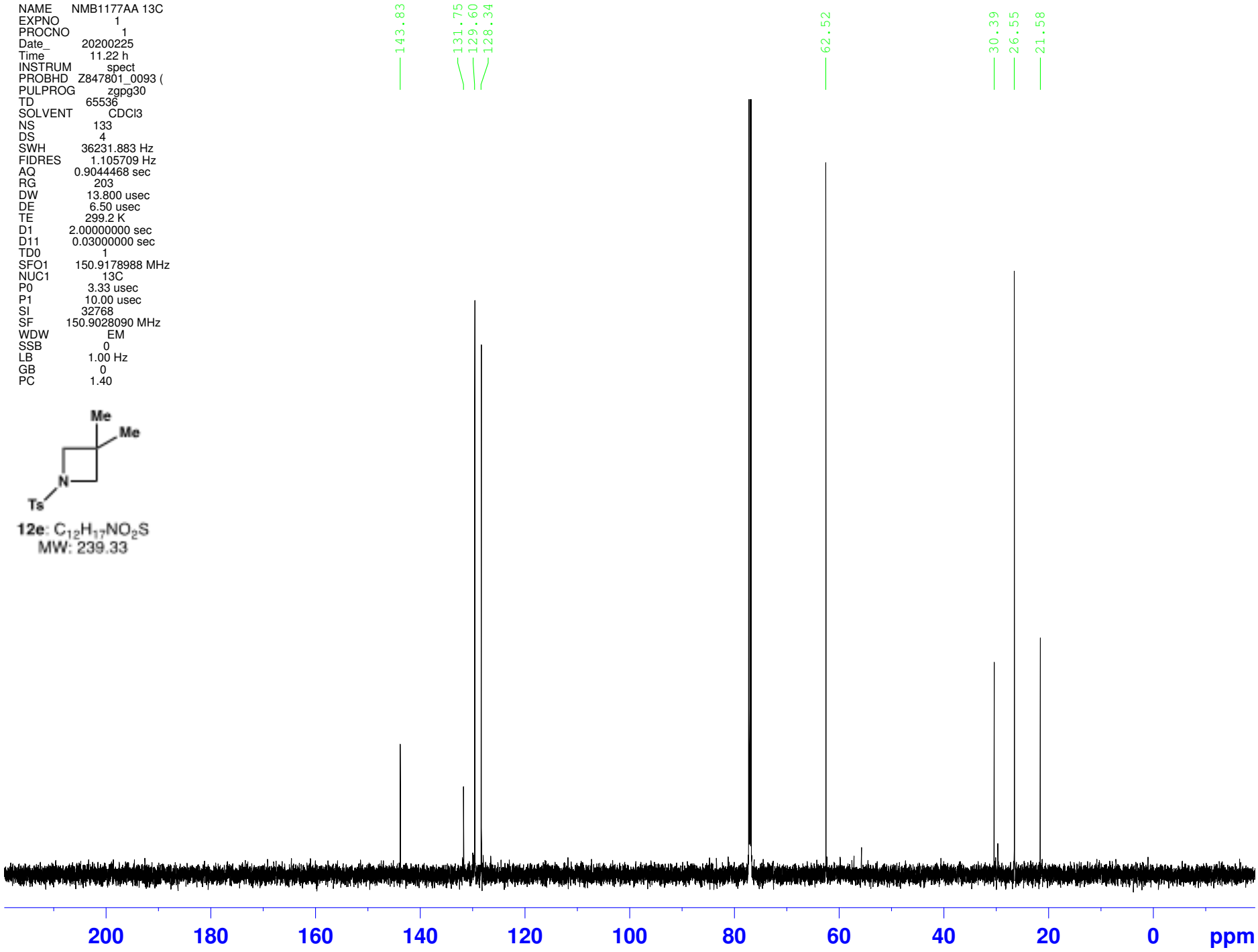


12d: $C_{10}H_{18}NO_2$
MW: 185.27

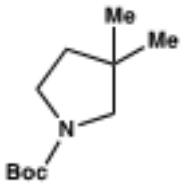
NAME NMB1177AA solvent free
EXPNO 1
PROCNO 1
Date_ 20200301
Time 22.16 h
INSTRUM spect
PROBHD Z108618_0106 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.244532 Hz
AQ 4.0894966 sec
RG 203
DW 62.400 usec
DE 16.92 usec
TE 294.3 K
D1 1.00000000 sec
TD0 1
SFO1 400.1724710 MHz
NUC1 1H
P0 5.00 usec
P1 15.00 usec
SI 65536
SF 400.1700113 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00



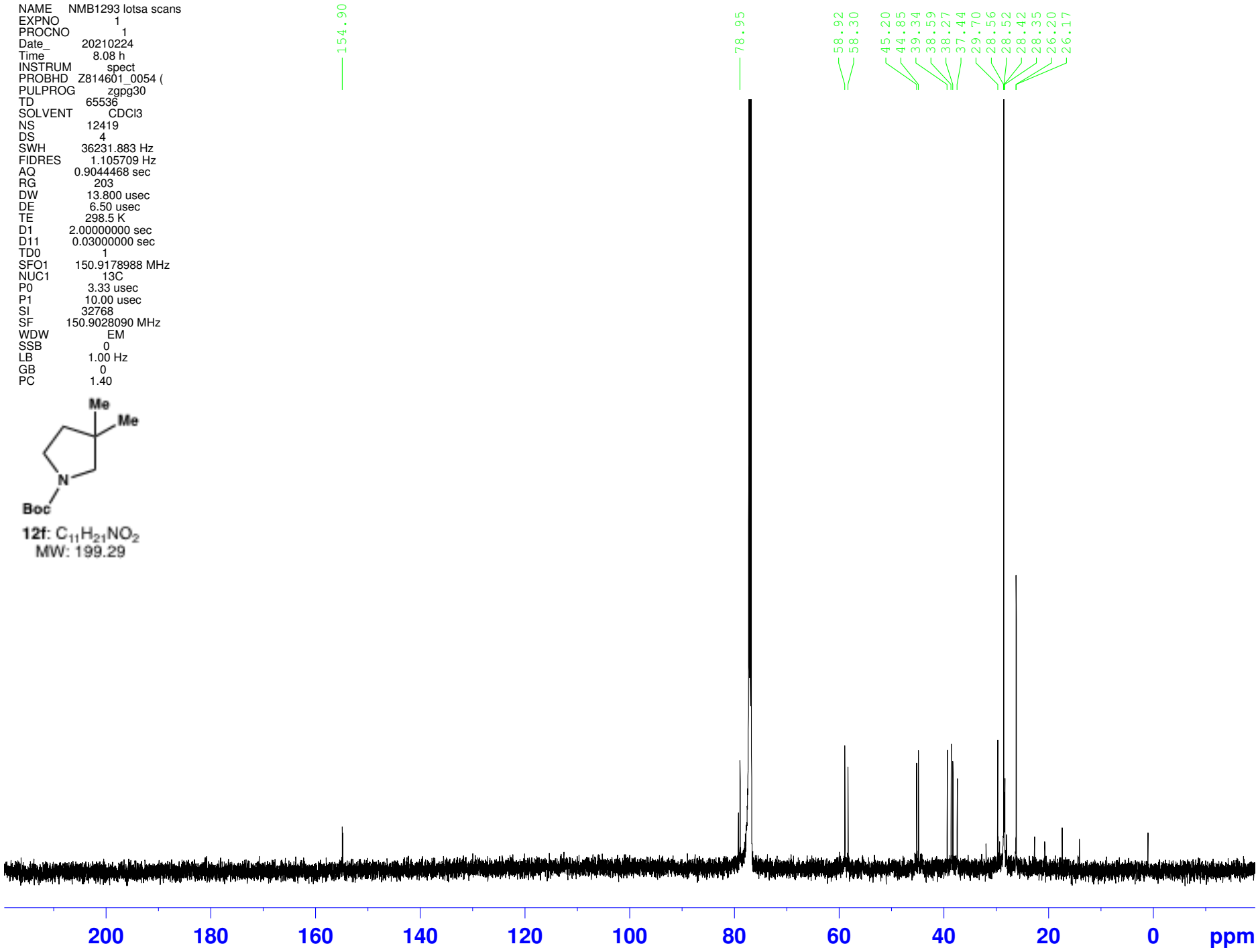
12e: $C_{12}H_{17}NO_2S$
MW: 239.33


NAME NMB1177AA 13C
EXPNO 1
PROCNO 1
Date_ 20200225
Time 11.22 h
INSTRUM spect
PROBHD Z847801_0093 (
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 133
DS 4
SWH 36231.883 Hz
FIDRES 1.105709 Hz
AQ 0.9044468 sec
RG 203
DW 13.800 usec
DE 6.50 usec
TE 299.2 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 150.9178988 MHz
NUC1 13C
P0 3.33 usec
P1 10.00 usec
SI 32768
SF 150.9028090 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
143.83
131.75
129.60
128.34
62.52
30.39
26.55
21.58
Me
Me
N
Ts
12e: C12H17NO2S
MW: 239.33
200
180
160
140
120
100
80
60
40
20
0
ppm

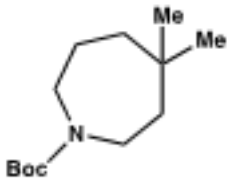
NAME NMB1293 A 1H
EXPNO 1
PROCNO 1
Date_ 20210123
Time 9.32 h
INSTRUM spect
PROBHD Z108618_0106 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.244532 Hz
AQ 4.0894966 sec
RG 203
DW 62.400 usec
DE 16.92 usec
TE 297.8 K
D1 1.00000000 sec
TD0 1
SFO1 400.1724710 MHz
NUC1 1H
P0 5.00 usec
P1 15.00 usec
SI 65536
SF 400.1700000 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00



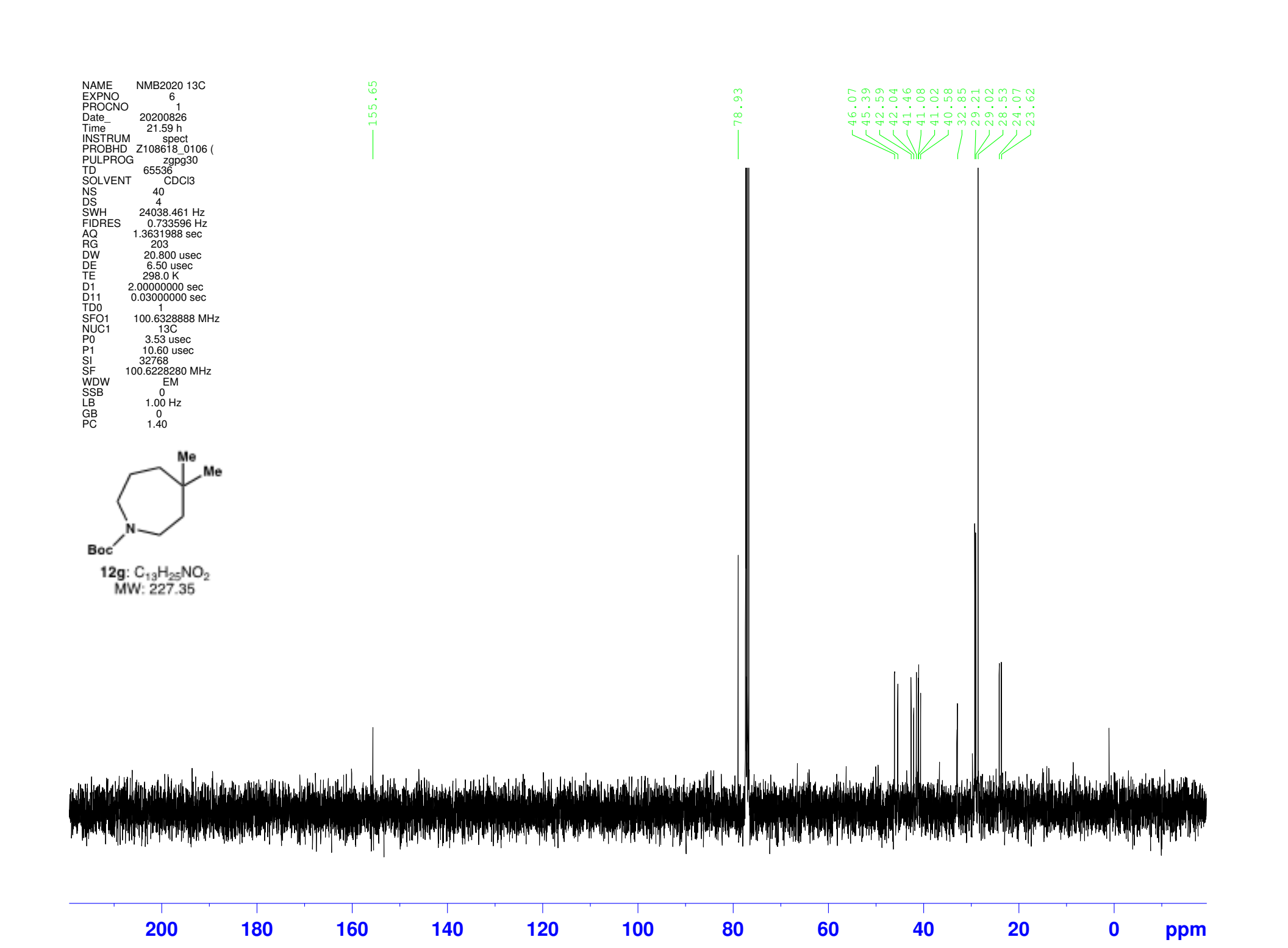
12f: $C_{11}H_{21}NO_2$
MW: 199.29

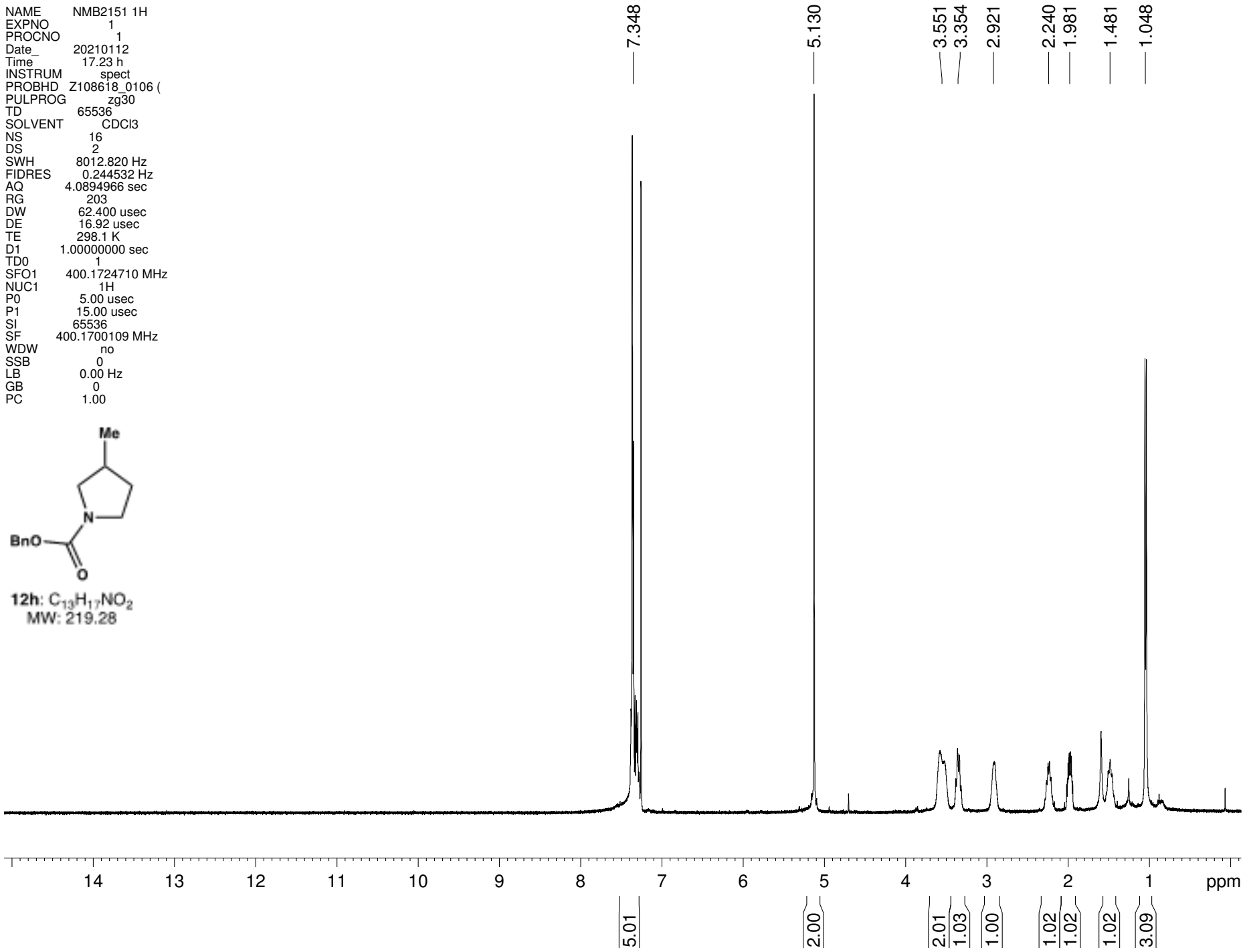

NAME NMB1293 lotsa scans
EXPNO 1
PROCNO 1
Date_ 20210224
Time 8.08 h
INSTRUM spect
PROBHD Z814601_0054 (
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 12419
DS 4
SWH 36231.883 Hz
FIDRES 1.105709 Hz
AQ 0.9044468 sec
RG 203
DW 13.800 usec
DE 6.50 usec
TE 298.5 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 150.9178988 MHz
NUC1 13C
P0 3.33 usec
P1 10.00 usec
SI 32768
SF 150.9028090 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
Me
Me
N
Boc
12f: C11H21NO2
MW: 199.29
154.90
78.95
58.92
58.30
45.20
44.85
39.34
38.59
38.27
37.44
29.70
28.56
28.52
28.42
28.35
26.20
26.17
200
180
160
140
120
100
80
60
40
20
0
ppm

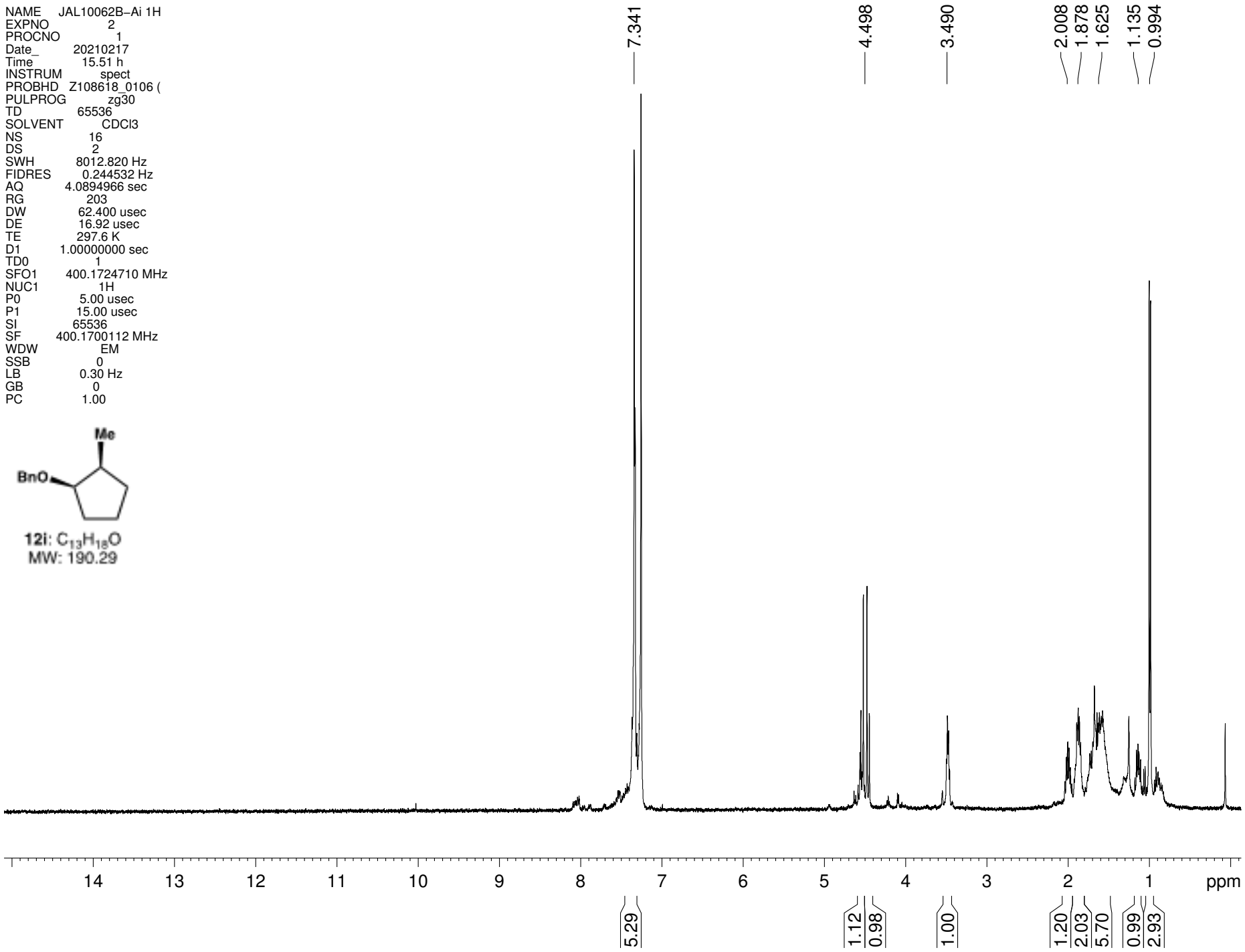
NAME	NMB2020
EXPNO	8
PROCNO	1
Date_	20200904
Time	15.52 h
INSTRUM	spect
PROBHD	Z847801_0093 (
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	16
DS	2
SWH	12019.230 Hz
FIDRES	0.366798 Hz
AQ	2.7263477 sec
RG	203
DW	41.600 usec
DE	10.42 usec
TE	298.6 K
D1	1.00000000 sec
TD0	1
SFO1	600.1337058 MHz
NUC1	1H
P0	5.00 usec
P1	15.00 usec
SI	65536
SF	600.1300143 MHz
WDW	no
SSB	0
LB	0.00 Hz
GB	0
PC	1.00



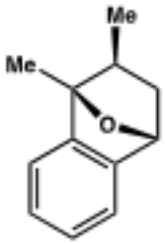
12g: $C_{13}H_{25}NO_2$
MW: 227.35


NAME NMB2020 13C
EXPNO 6
PROCNO 1
Date_ 20200826
Time 21.59 h
INSTRUM spect
PROBHD Z108618_0106 (
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 40
DS 4
SWH 24038.461 Hz
FIDRES 0.733596 Hz
AQ 1.3631988 sec
RG 203
DW 20.800 usec
DE 6.50 usec
TE 298.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 100.6328888 MHz
NUC1 13C
P0 3.53 usec
P1 10.60 usec
SI 32768
SF 100.6228280 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
155.65
78.93
46.07
45.39
42.59
42.04
41.46
41.08
41.02
40.58
32.85
29.21
29.02
28.53
24.07
23.62
Me
Me
N
Boc
12g: C13H25NO2
MW: 227.35
200
180
160
140
120
100
80
60
40
20
0
ppm

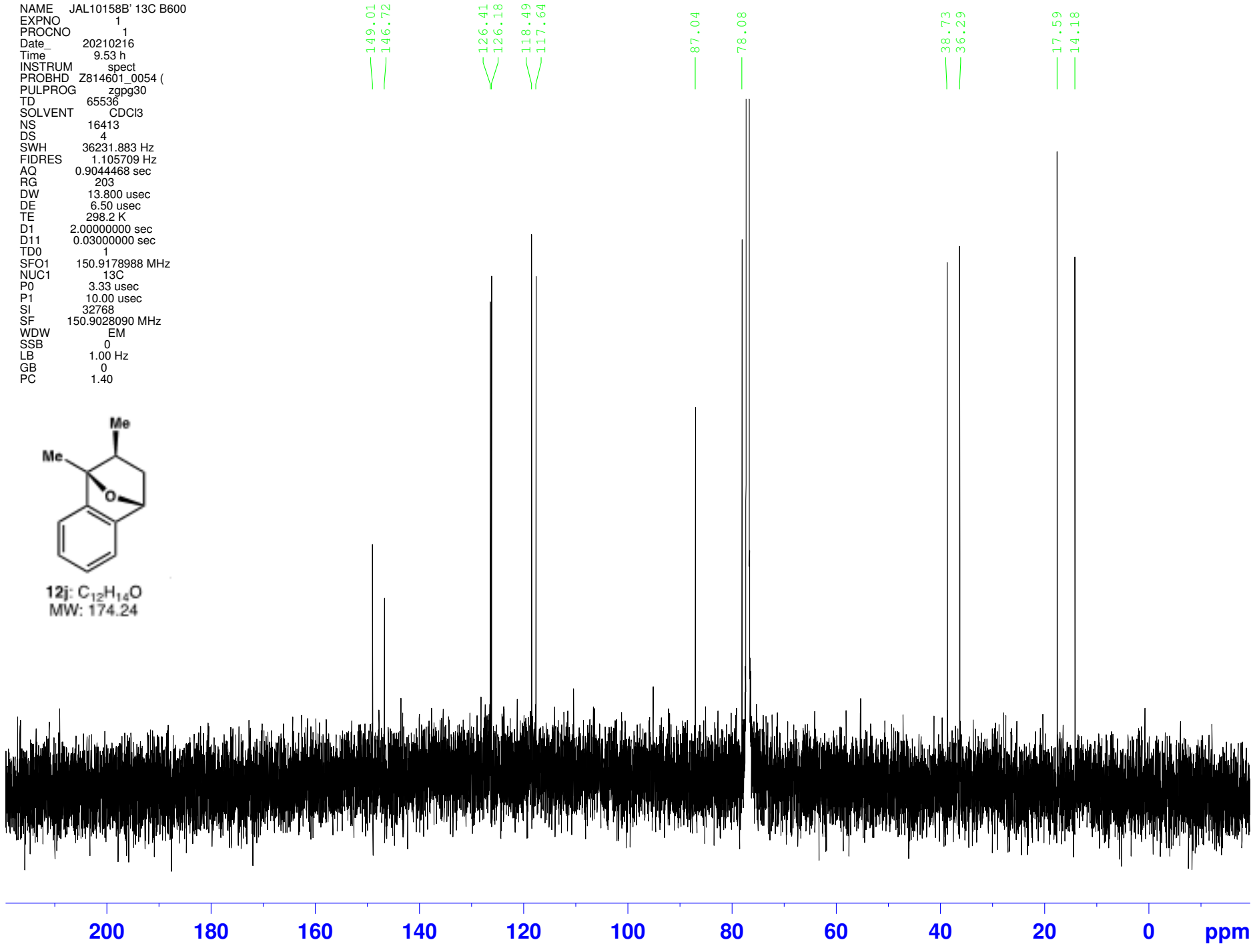

NAME NMB2151 1H
EXPNO 1
PROCNO 1
Date_ 20210112
Time 17.23 h
INSTRUM spect
PROBHD Z108618_0106 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.244532 Hz
AQ 4.0894966 sec
RG 203
DW 62.400 usec
DE 16.92 usec
TE 298.1 K
D1 1.00000000 sec
TD0 1
SFO1 400.1724710 MHz
NUC1 1H
P0 5.00 usec
P1 15.00 usec
SI 65536
SF 400.1700109 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
Me
N
BnO
O
12h: C13H17NO2
MW: 219.28
7.348
5.130
3.551
3.354
2.921
2.240
1.981
1.481
1.048
14
13
12
11
10
9
8
7
6
5
4
3
2
1
ppm
5.01
2.00
2.01
1.03
1.00
1.02
1.02
1.02
3.09


NAME JAL10062B–Ai 1H
EXPNO 2
PROCNO 1
Date_ 20210217
Time 15.51 h
INSTRUM spect
PROBHD Z108618_0106 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.244532 Hz
AQ 4.0894966 sec
RG 203
DW 62.400 usec
DE 16.92 usec
TE 297.6 K
D1 1.00000000 sec
TD0 1
SFO1 400.1724710 MHz
NUC1 1H
P0 5.00 usec
P1 15.00 usec
SI 65536
SF 400.1700112 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
Me
BnO
12i: C13H18O
MW: 190.29
7.341
4.498
3.490
2.008
1.878
1.625
1.135
0.994
14
13
12
11
10
9
8
7
6
5
4
3
2
1
ppm
5.29
1.12
0.98
1.00
1.20
2.03
5.70
0.99
2.93

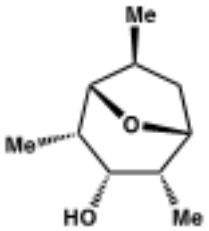
NAME JAL10158B–3 1H B600
EXPNO 1
PROCNO 1
Date_ 20210215
Time 19.41 h
INSTRUM spect
PROBHD Z108618_0106 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.244532 Hz
AQ 4.0894966 sec
RG 203
DW 62.400 usec
DE 16.92 usec
TE 298.0 K
D1 1.00000000 sec
TD0 1
SFO1 400.1724710 MHz
NUC1 1H
P0 5.00 usec
P1 15.00 usec
SI 65536
SF 400.1700000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



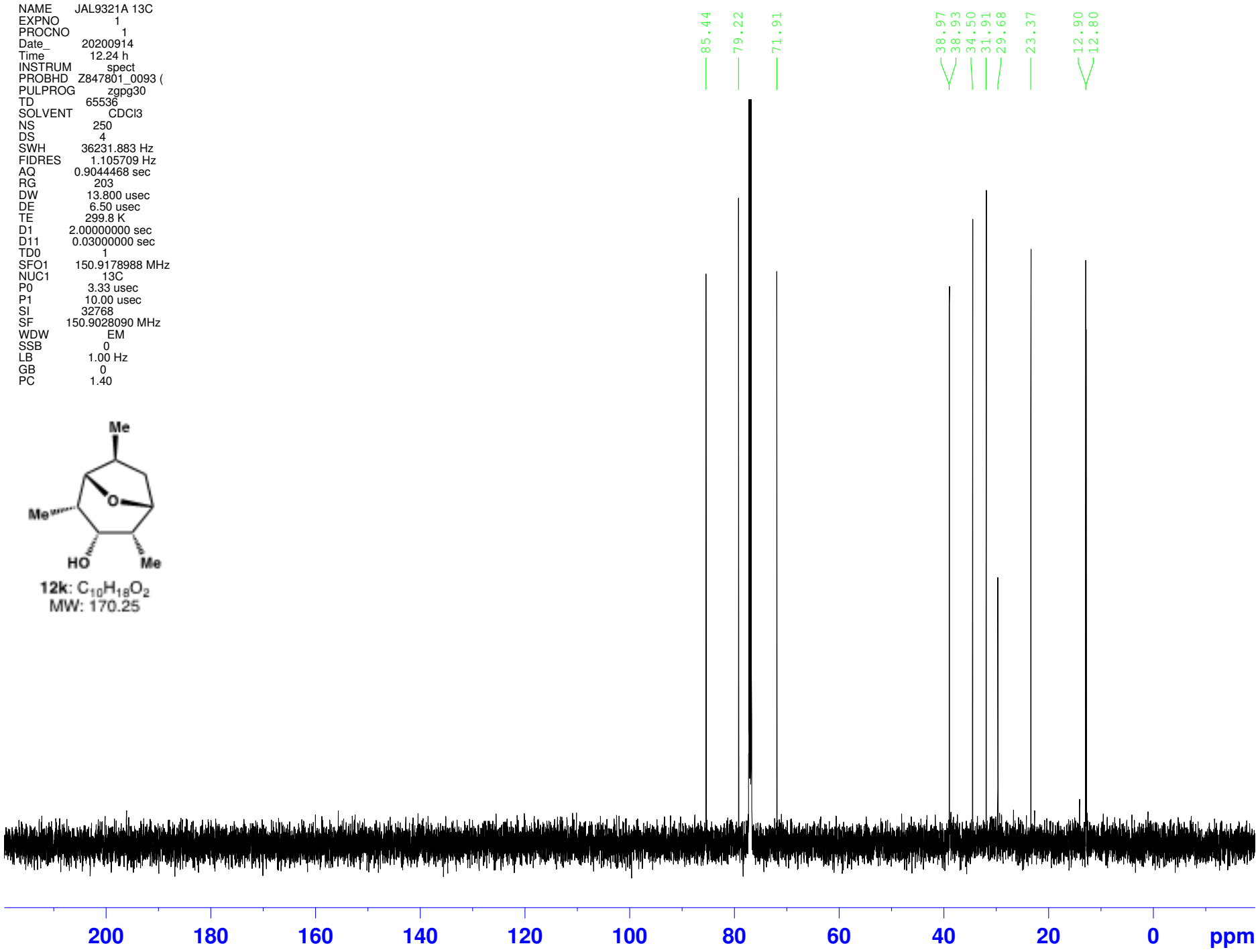
12j: $C_{12}H_{14}O$
MW: 174.24

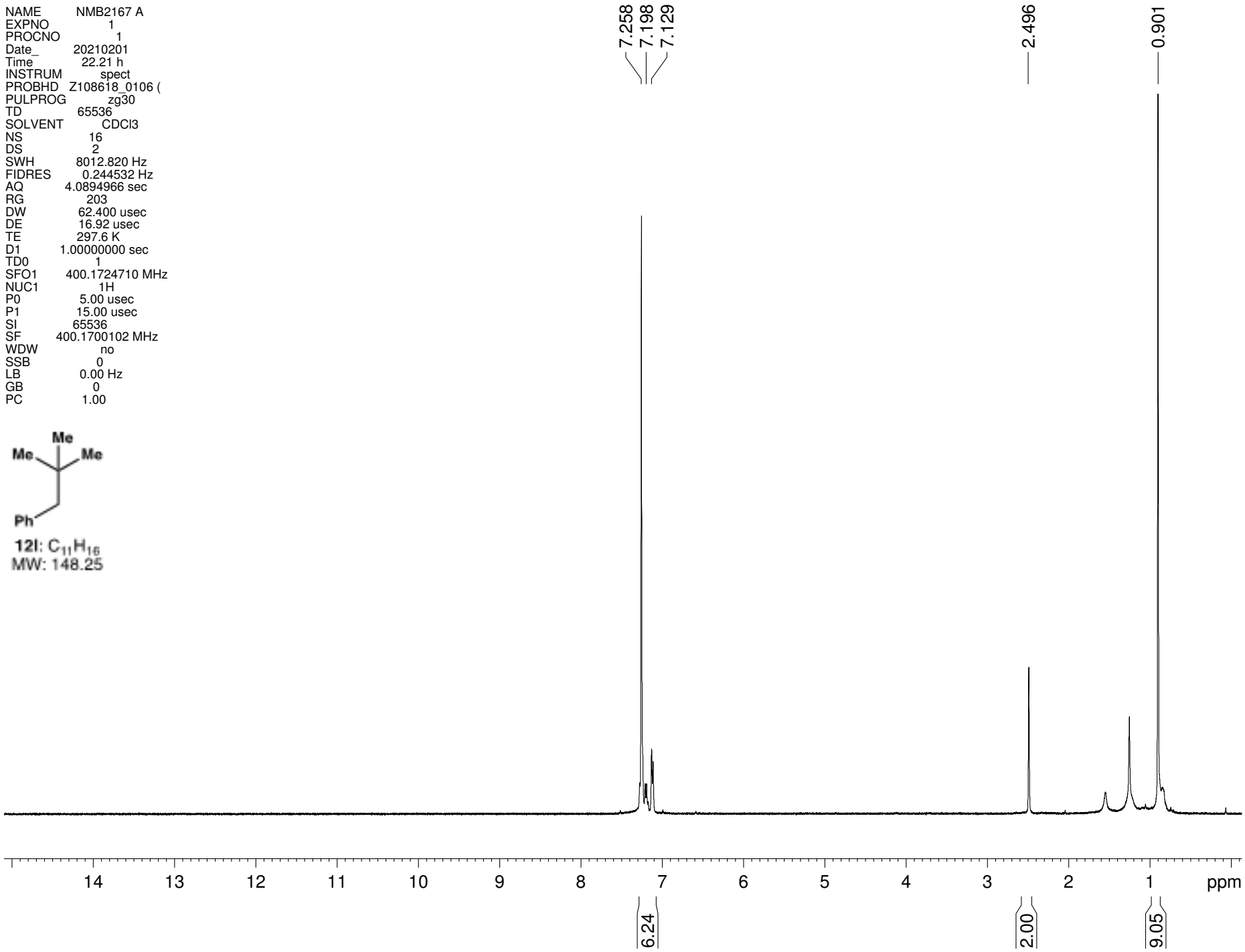

NAME JAL10158B' 13C B600
EXPNO 1
PROCNO 1
Date_ 20210216
Time 9.53 h
INSTRUM spect
PROBHD Z814601_0054 (
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 16413
DS 4
SWH 36231.883 Hz
FIDRES 1.105709 Hz
AQ 0.9044468 sec
RG 203
DW 13.800 usec
DE 6.50 usec
TE 298.2 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 150.9178988 MHz
NUC1 13C
P0 3.33 usec
P1 10.00 usec
SI 32768
SF 150.9028090 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
149.01
146.72
126.41
126.18
118.49
117.64
87.04
78.08
38.73
36.29
17.59
14.18
Me
Me
O
12j: C12H14O
MW: 174.24
200
180
160
140
120
100
80
60
40
20
0
ppm

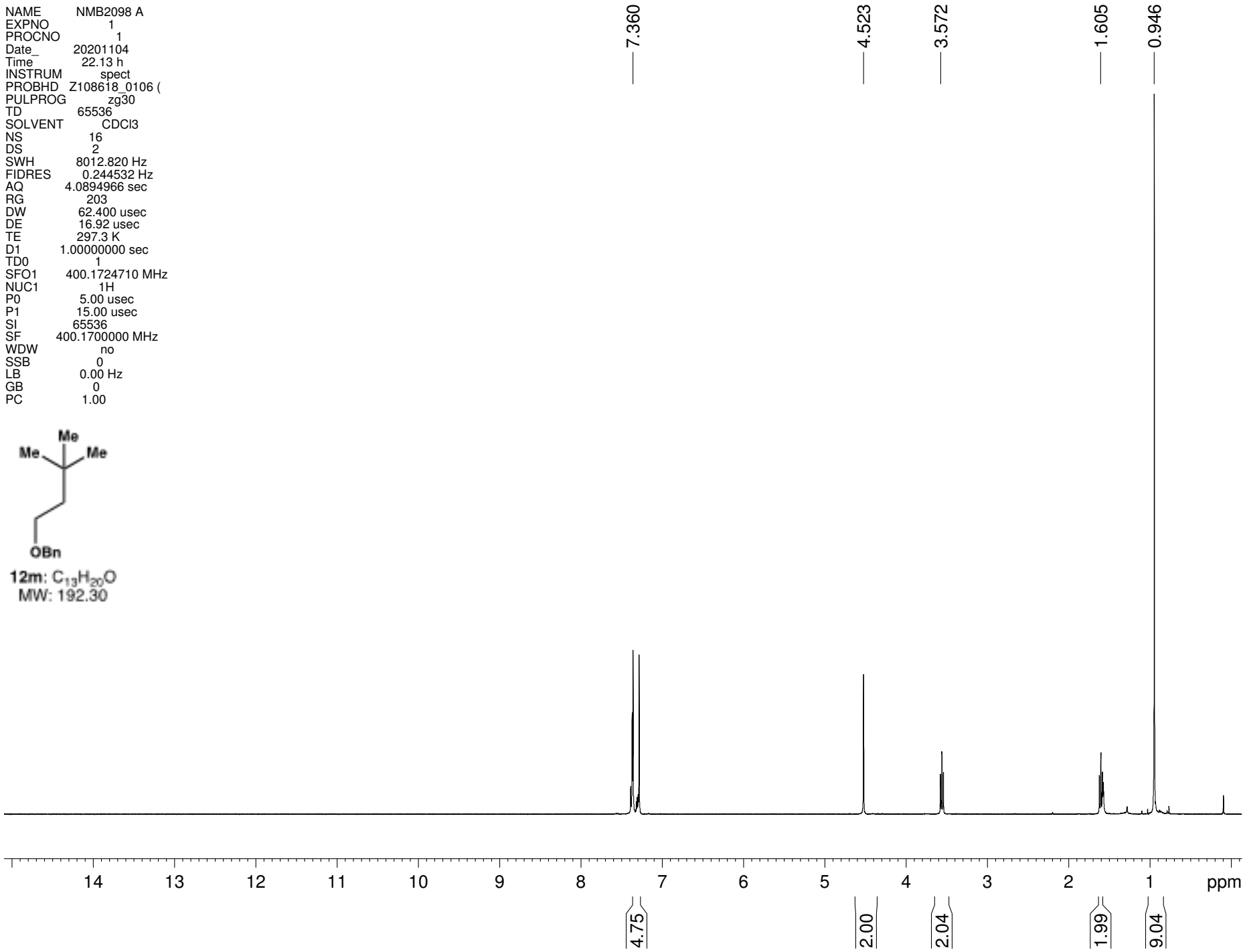
NAME JAL9321A 1H
EXPNO 1
PROCNO 1
Date_ 20200914
Time 12.10 h
INSTRUM spect
PROBHD Z847801_0093 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 12019.230 Hz
FIDRES 0.366798 Hz
AQ 2.7263477 sec
RG 144
DW 41.600 usec
DE 10.42 usec
TE 298.5 K
D1 1.00000000 sec
TD0 1
SFO1 600.1337058 MHz
NUC1 1H
P0 5.00 usec
P1 15.00 usec
SI 65536
SF 600.1300141 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



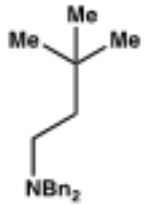
12k: $C_{10}H_{18}O_2$
MW: 170.25


NAME JAL9321A 13C
EXPNO 1
PROCNO 1
Date_ 20200914
Time 12.24 h
INSTRUM spect
PROBHD Z847801_0093 (
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 250
DS 4
SWH 36231.883 Hz
FIDRES 1.105709 Hz
AQ 0.9044468 sec
RG 203
DW 13.800 usec
DE 6.50 usec
TE 299.8 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 150.9178988 MHz
NUC1 13C
P0 3.33 usec
P1 10.00 usec
SI 32768
SF 150.9028090 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
85.44
79.22
71.91
38.97
38.93
34.50
31.91
29.68
23.37
12.90
12.80
Me
Me
O
HO
Me
12k: C10H18O2
MW: 170.25
200
180
160
140
120
100
80
60
40
20
0
ppm

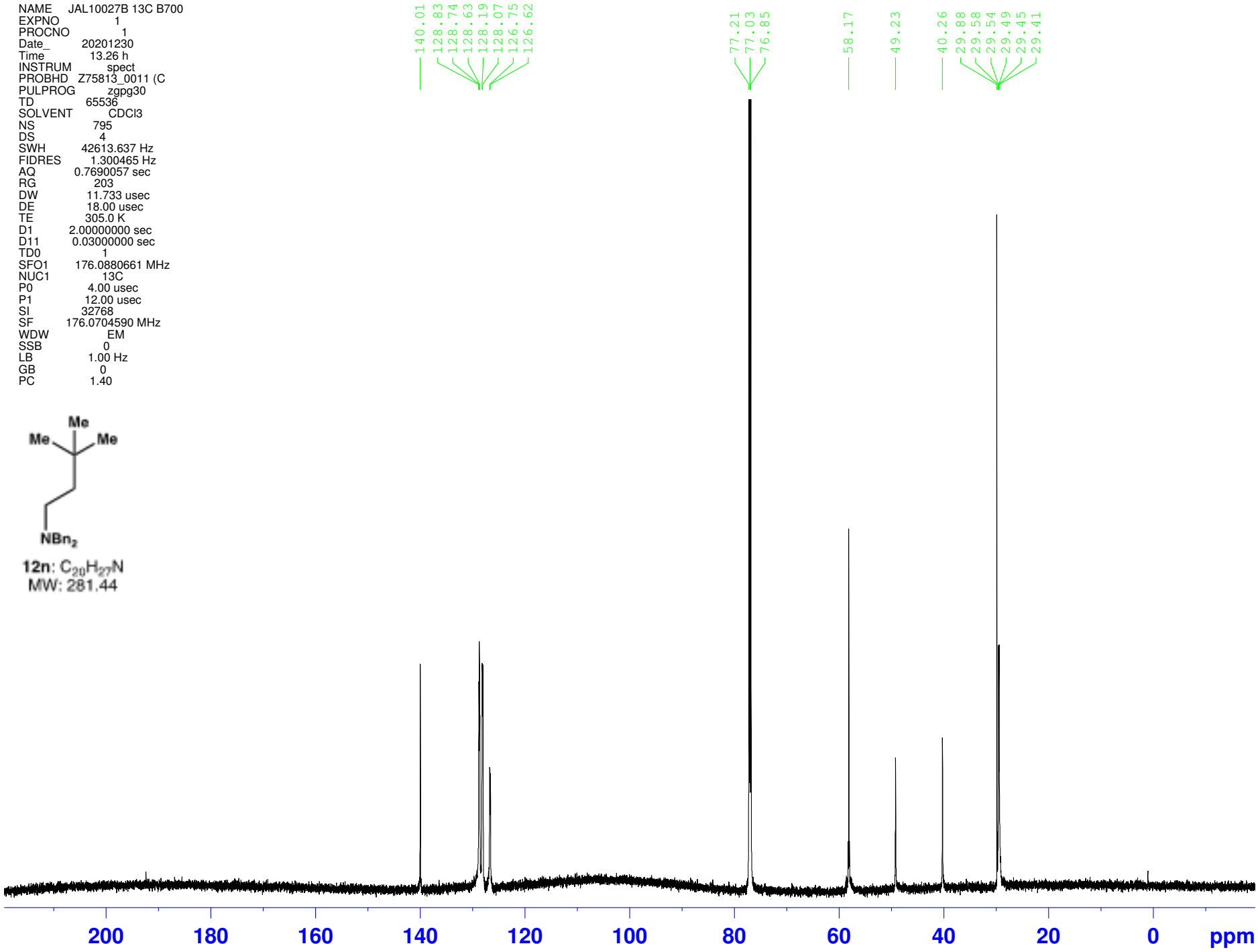

NAME NMB2167 A
EXPNO 1
PROCNO 1
Date_ 20210201
Time 22.21 h
INSTRUM spect
PROBHD Z108618_0106 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.244532 Hz
AQ 4.0894966 sec
RG 203
DW 62.400 usec
DE 16.92 usec
TE 297.6 K
D1 1.00000000 sec
TD0 1
SFO1 400.1724710 MHz
NUC1 1H
P0 5.00 usec
P1 15.00 usec
SI 65536
SF 400.1700102 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
Me
Me
Me
Ph
12l: C11H16
MW: 148.25
7.258
7.198
7.129
2.496
0.901
14
13
12
11
10
9
8
7
6
5
4
3
2
1
ppm
6.24
2.00
9.05


NAME NMB2098 A
EXPNO 1
PROCNO 1
Date_ 20201104
Time 22.13 h
INSTRUM spect
PROBHD Z108618_0106 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.244532 Hz
AQ 4.0894966 sec
RG 203
DW 62.400 usec
DE 16.92 usec
TE 297.3 K
D1 1.00000000 sec
TD0 1
SFO1 400.1724710 MHz
NUC1 1H
P0 5.00 usec
P1 15.00 usec
SI 65536
SF 400.1700000 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
Me
Me
Me
OBn
12m: C13H20O
MW: 192.30
7.360
4.523
3.572
1.605
0.946
14
13
12
11
10
9
8
7
6
5
4
3
2
1
ppm
4.75
2.00
2.04
1.99
9.04

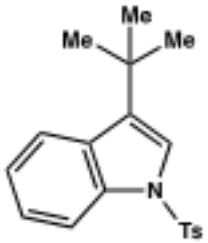
NAME JAL10027B 1H
EXPNO 1
PROCNO 1
Date_ 20201214
Time 17.27 h
INSTRUM spect
PROBHD Z108618_0106 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.244532 Hz
AQ 4.0894966 sec
RG 181
DW 62.400 usec
DE 16.92 usec
TE 297.7 K
D1 1.00000000 sec
TD0 1
SFO1 400.1724710 MHz
NUC1 1H
P0 5.00 usec
P1 15.00 usec
SI 65536
SF 400.1700000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



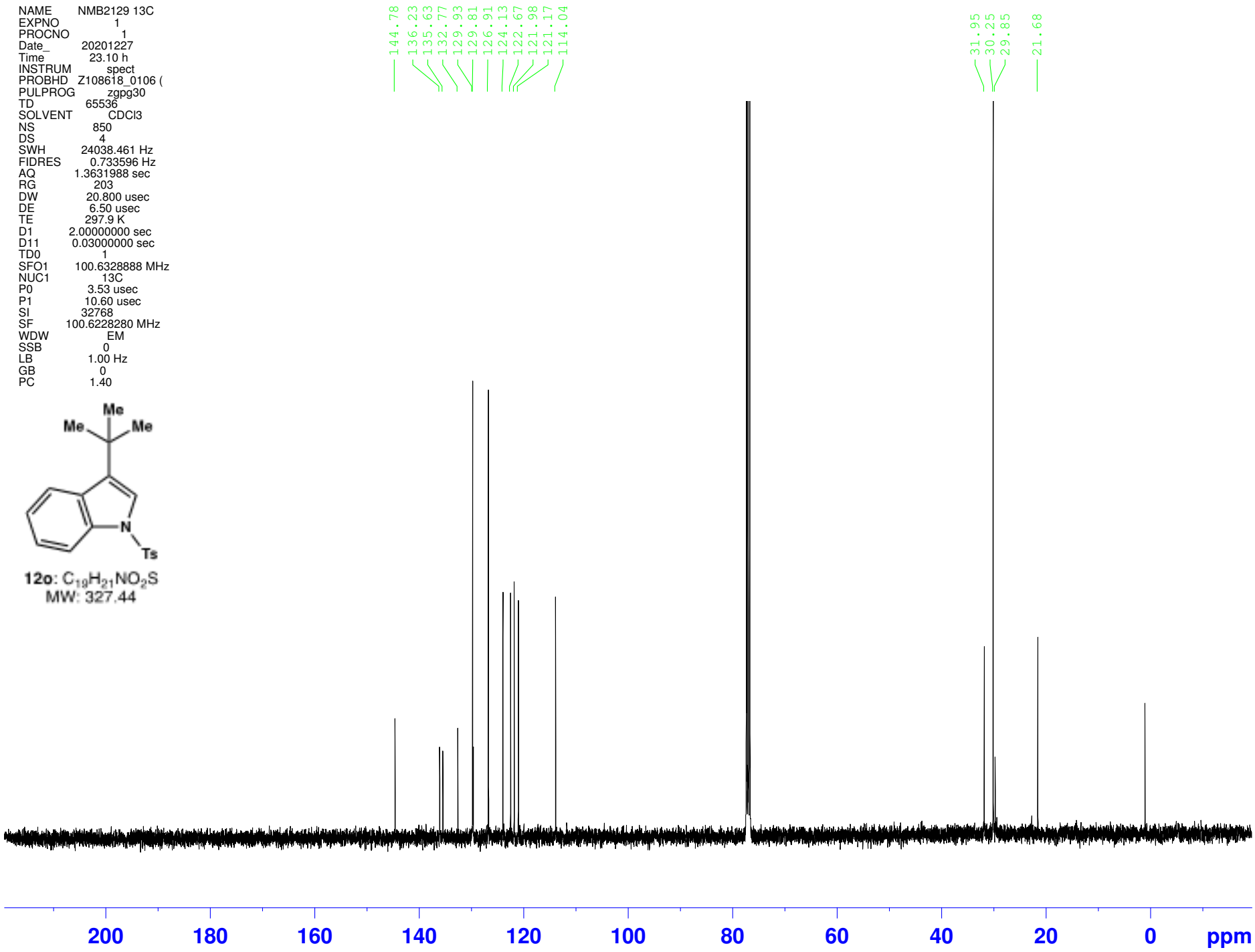
12n: $C_{20}H_{27}N$
MW: 281.44


NAME JAL10027B 13C B700
EXPNO 1
PROCNO 1
Date_ 20201230
Time 13.26 h
INSTRUM spect
PROBHD Z75813_0011 (C
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 795
DS 4
SWH 42613.637 Hz
FIDRES 1.300465 Hz
AQ 0.7690057 sec
RG 203
DW 11.733 usec
DE 18.00 usec
TE 305.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 176.0880661 MHz
NUC1 13C
P0 4.00 usec
P1 12.00 usec
SI 32768
SF 176.0704590 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
140.01
128.83
128.74
128.63
128.19
128.07
126.75
126.62
77.21
77.03
76.85
58.17
49.23
40.26
29.88
29.58
29.54
29.49
29.45
29.41
Me
Me
Me
NBn2
12n: C20H27N
MW: 281.44
200
180
160
140
120
100
80
60
40
20
0
ppm

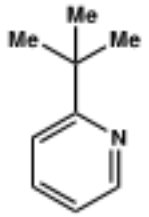
NAME NMB2129 1H
EXPNO 1
PROCNO 1
Date_ 20201227
Time 23.42 h
INSTRUM spect
PROBHD Z108618_0106 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.244532 Hz
AQ 4.0894966 sec
RG 203
DW 62.400 usec
DE 16.92 usec
TE 297.5 K
D1 1.00000000 sec
TD0 1
SFO1 400.1724710 MHz
NUC1 1H
P0 5.00 usec
P1 15.00 usec
SI 65536
SF 400.1700000 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00



12o: $C_{19}H_{21}NO_2S$
MW: 327.44

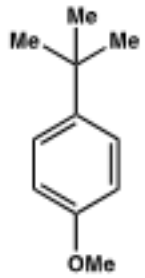

NAME NMB2129 13C
EXPNO 1
PROCNO 1
Date_ 20201227
Time 23.10 h
INSTRUM spect
PROBHD Z108618_0106 (
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 850
DS 4
SWH 24038.461 Hz
FIDRES 0.733596 Hz
AQ 1.3631988 sec
RG 203
DW 20.800 usec
DE 6.50 usec
TE 297.9 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 100.6328888 MHz
NUC1 13C
P0 3.53 usec
P1 10.60 usec
SI 32768
SF 100.6228280 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
144.78
136.23
135.63
132.77
129.93
129.81
126.91
124.13
122.67
121.98
121.17
114.04
31.95
30.25
29.85
21.68
Me
Me
Me
N
Ts
12o: C19H21NO2S
MW: 327.44
200
180
160
140
120
100
80
60
40
20
0
ppm

NAME NMB2146 1H
EXPNO 1
PROCNO 1
Date_ 20210105
Time 16.36 h
INSTRUM spect
PROBHD Z108618_0106 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.244532 Hz
AQ 4.0894966 sec
RG 203
DW 62.400 usec
DE 16.92 usec
TE 297.8 K
D1 1.00000000 sec
TD0 1
SFO1 400.1724710 MHz
NUC1 1H
P0 5.00 usec
P1 15.00 usec
SI 65536
SF 400.1700100 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00



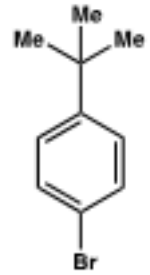
12p: $C_9H_{13}N$
MW: 135.21

NAME NMB2148 1H
EXPNO 1
PROCNO 1
Date_ 20210110
Time 19.14 h
INSTRUM spect
PROBHD Z108618_0106 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.244532 Hz
AQ 4.0894966 sec
RG 203
DW 62.400 usec
DE 16.92 usec
TE 297.7 K
D1 1.00000000 sec
TD0 1
SFO1 400.1724710 MHz
NUC1 1H
P0 5.00 usec
P1 15.00 usec
SI 65536
SF 400.1700100 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00



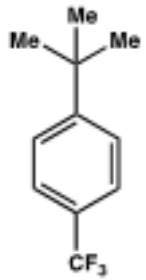
12q: $C_{11}H_{16}O$
MW: 164.25

NAME NMB2123 1H
EXPNO 1
PROCNO 1
Date_ 20201221
Time 18.24 h
INSTRUM spect
PROBHD Z108618_0106 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.244532 Hz
AQ 4.0894966 sec
RG 203
DW 62.400 usec
DE 16.92 usec
TE 297.8 K
D1 1.00000000 sec
TD0 1
SFO1 400.1724710 MHz
NUC1 1H
P0 5.00 usec
P1 15.00 usec
SI 65536
SF 400.1700000 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00



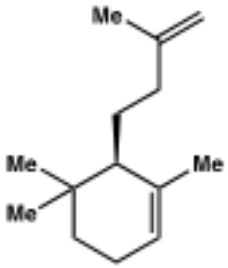
12r: $C_{10}H_{13}Br$
MW: 213.12

NAME	NMB2131 1H
EXPNO	1
PROCNO	1
Date_	20201228
Time	12.38 h
INSTRUM	spect
PROBHD	Z108618_0106 (
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	16
DS	2
SWH	8012.820 Hz
FIDRES	0.244532 Hz
AQ	4.0894966 sec
RG	203
DW	62.400 usec
DE	16.92 usec
TE	297.7 K
D1	1.00000000 sec
TD0	1
SFO1	400.1724710 MHz
NUC1	1H
P0	5.00 usec
P1	15.00 usec
SI	65536
SF	400.1700000 MHz
WDW	no
SSB	0
LB	0.00 Hz
GB	0
PC	1.00



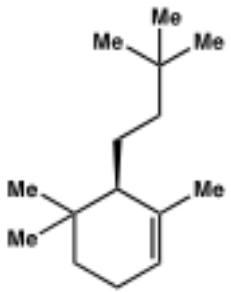
12s: $C_{11}H_{13}F_3$
MW: 202.22

NAME JAL10072A 1H B600
EXPNO 1
PROCNO 1
Date_ 20210109
Time 13.08 h
INSTRUM spect
PROBHD Z814601_0054 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 12019.230 Hz
FIDRES 0.366798 Hz
AQ 2.7263477 sec
RG 114
DW 41.600 usec
DE 11.27 usec
TE 298.2 K
D1 1.00000000 sec
TD0 1
SFO1 600.1337058 MHz
NUC1 1H
P0 3.33 usec
P1 10.00 usec
SI 65536
SF 600.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

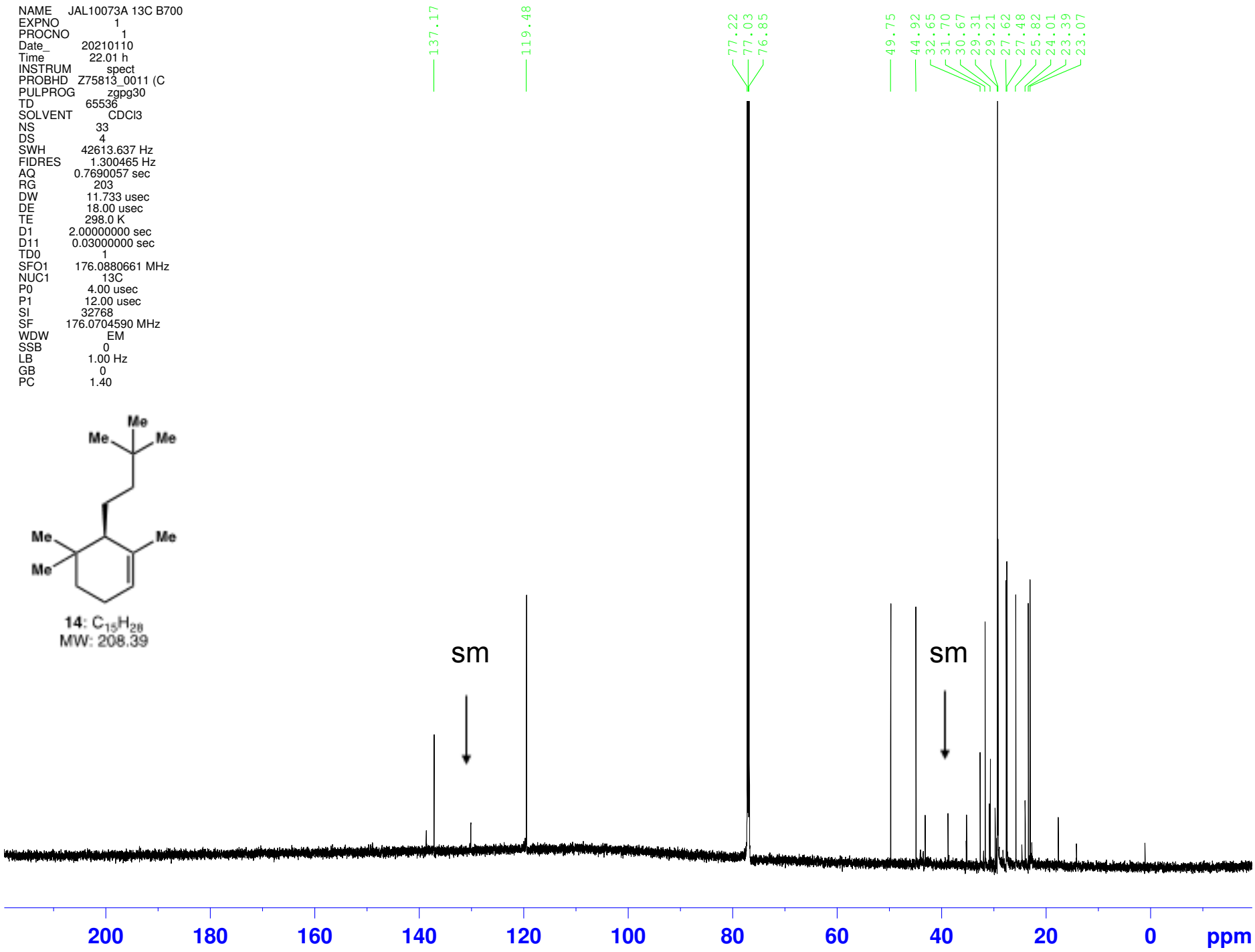


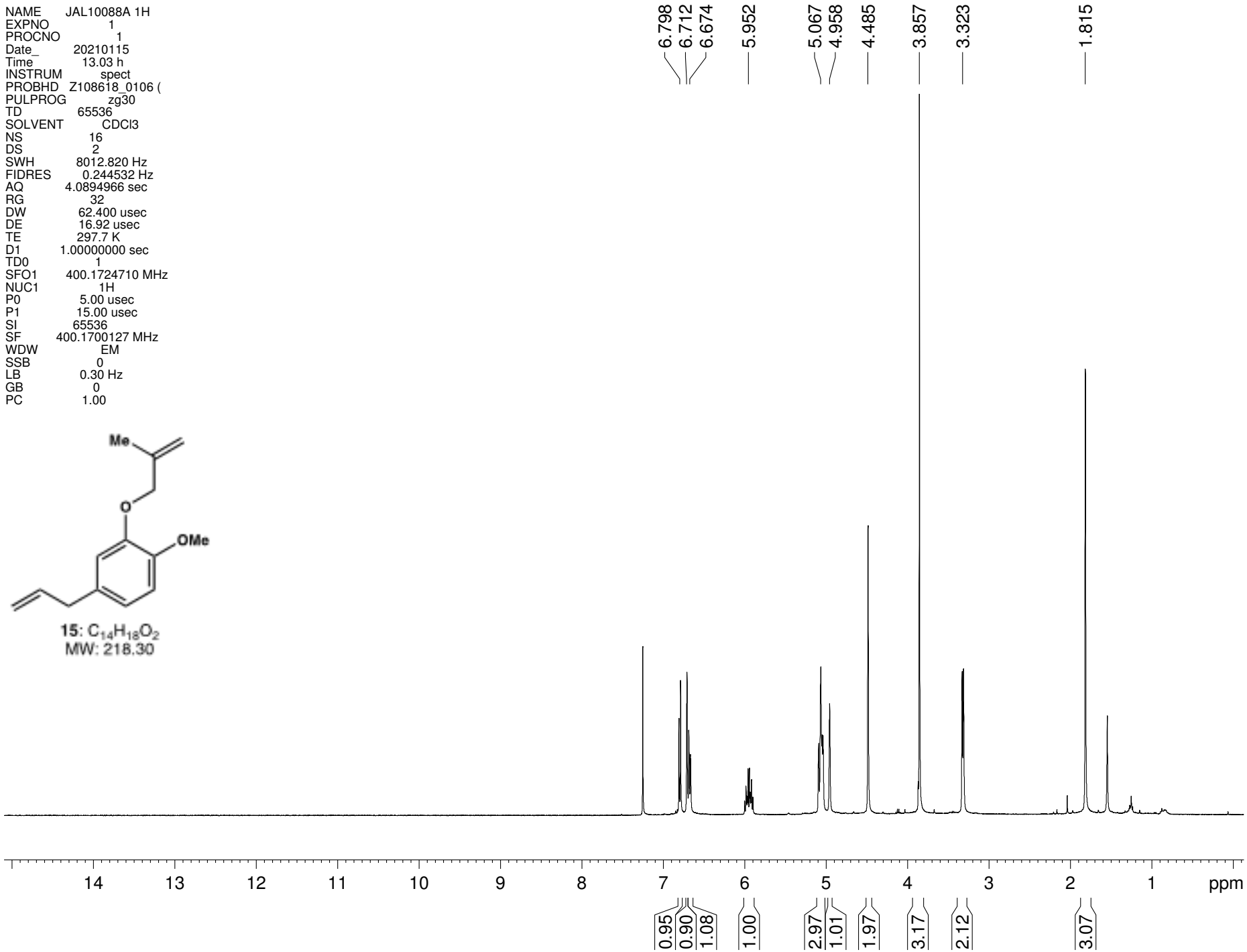
13: $C_{14}H_{24}$
MW: 192.35

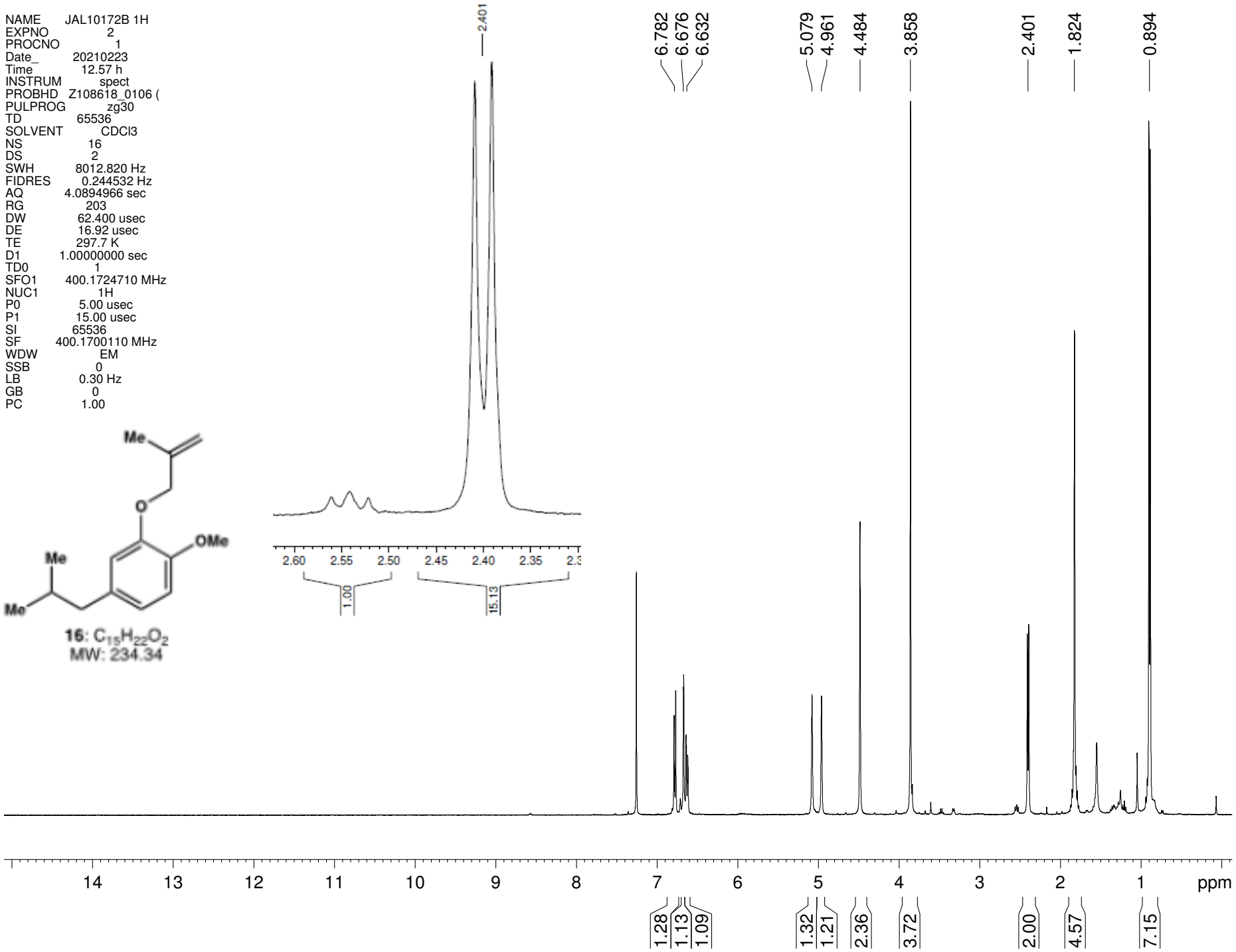
NAME JAL10073A 1H B700
EXPNO 1
PROCNO 1
Date_ 20210110
Time 21.56 h
INSTRUM spect
PROBHD Z75813_0011 (C
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 14097.744 Hz
FIDRES 0.430229 Hz
AQ 2.3243935 sec
RG 3.56
DW 35.467 usec
DE 14.10 usec
TE 298.0 K
D1 1.00000000 sec
TD0 1
SFO1 700.2243239 MHz
NUC1 1H
P0 3.33 usec
P1 10.00 usec
SI 65536
SF 700.2200000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

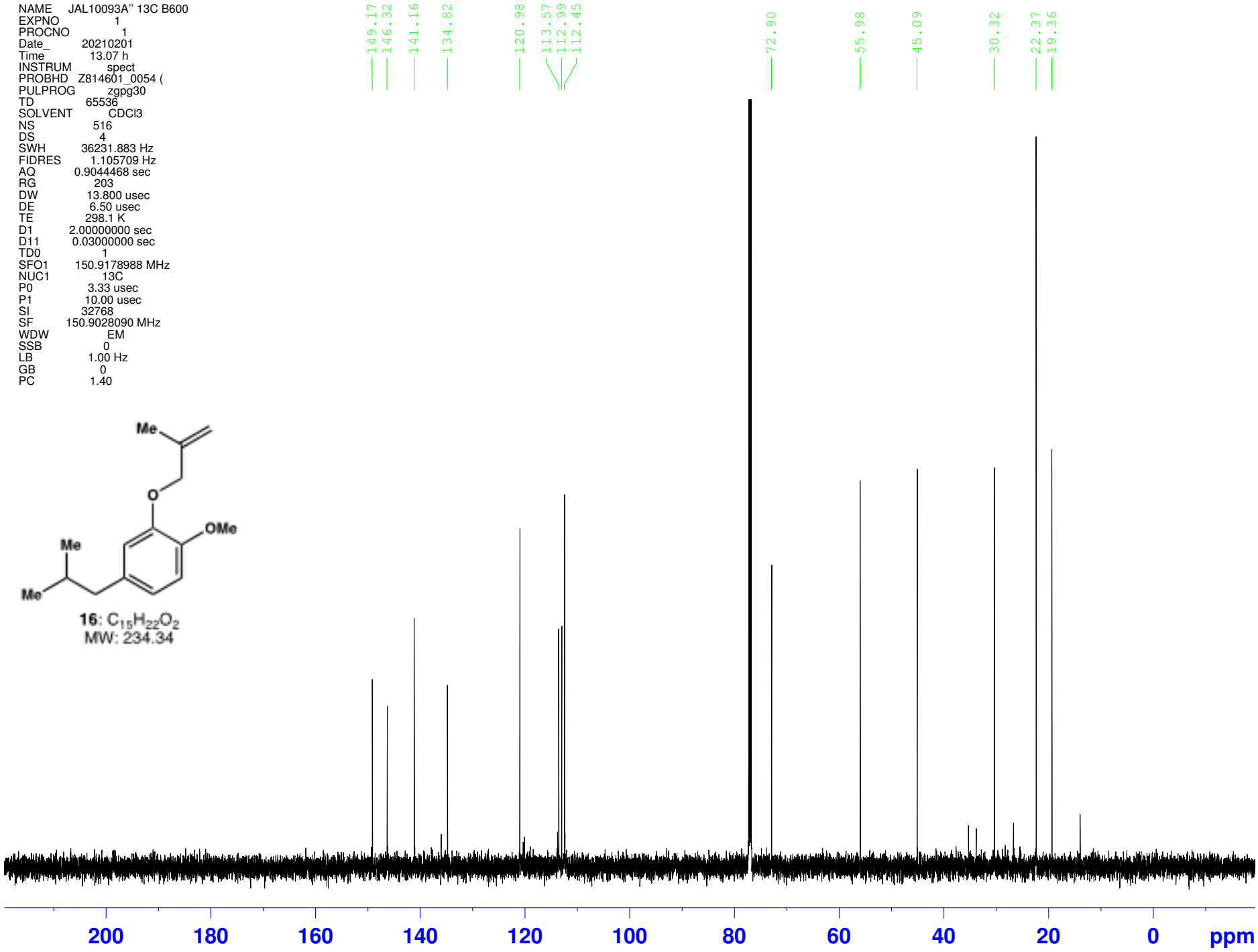


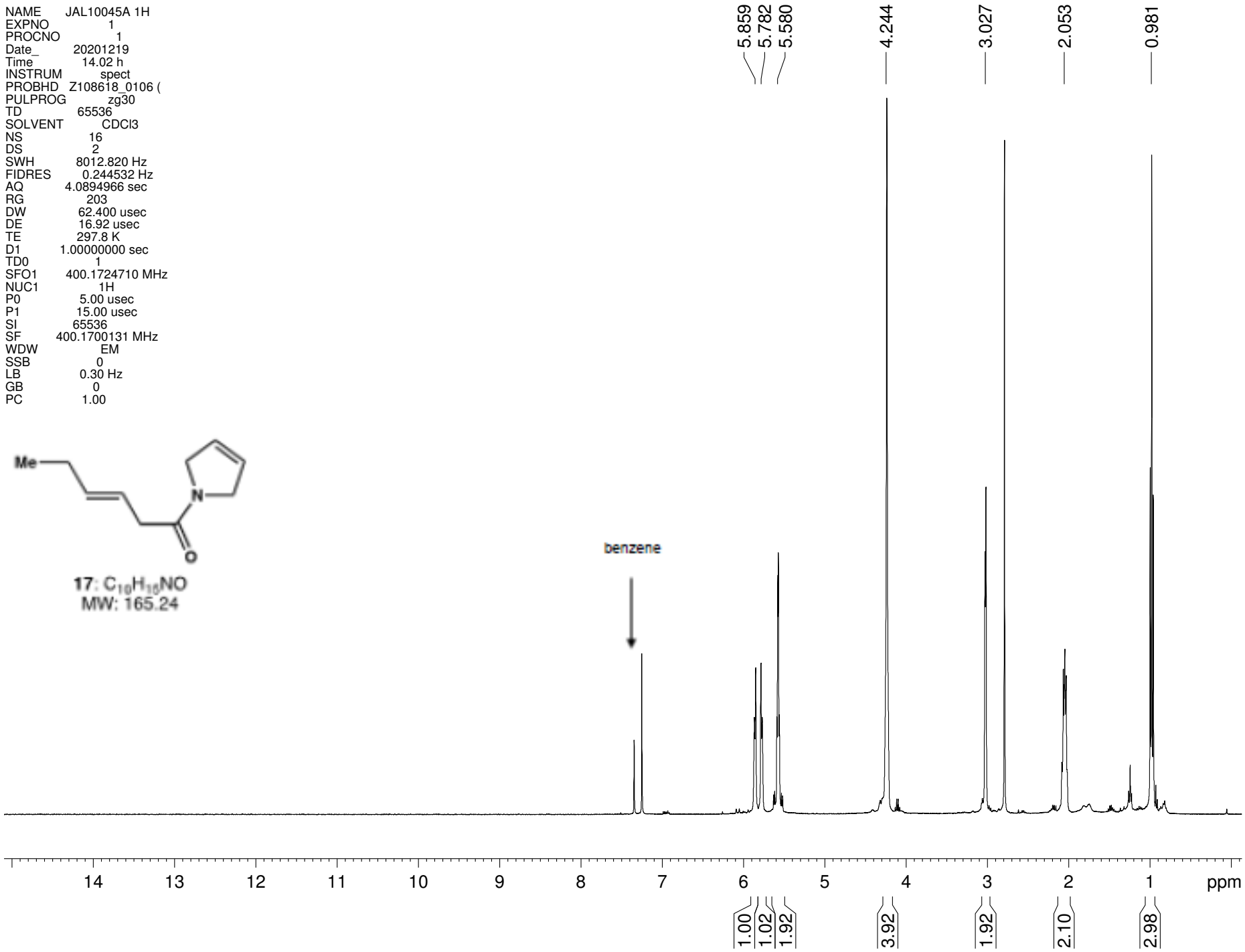
14: $C_{15}H_{28}$
MW: 208.39

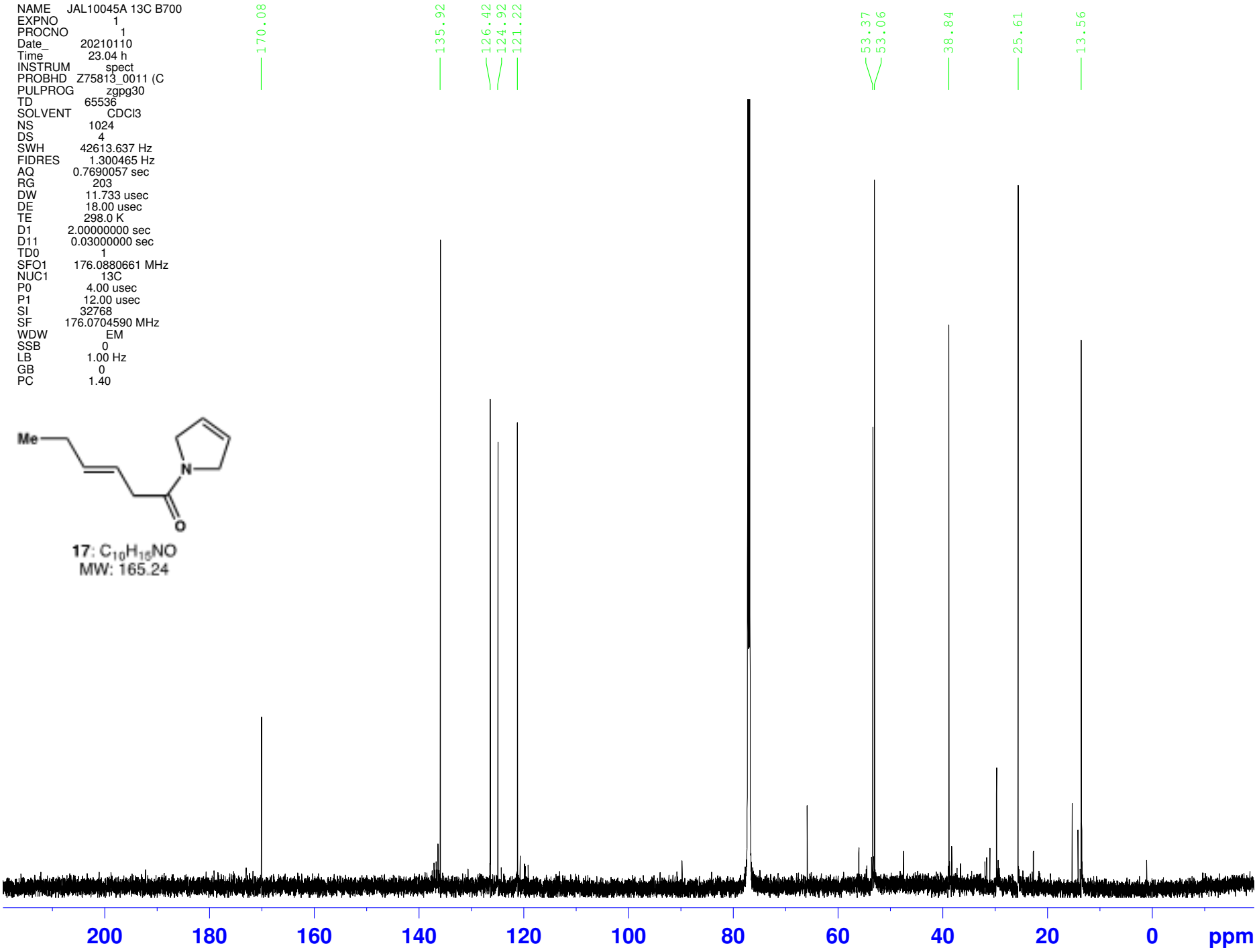

NAME JAL10073A 13C B700
EXPNO 1
PROCNO 1
Date_ 20210110
Time 22.01 h
INSTRUM spect
PROBHD Z75813_0011 (C
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 33
DS 4
SWH 42613.637 Hz
FIDRES 1.300465 Hz
AQ 0.7690057 sec
RG 203
DW 11.733 usec
DE 18.00 usec
TE 298.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 176.0880661 MHz
NUC1 13C
P0 4.00 usec
P1 12.00 usec
SI 32768
SF 176.0704590 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
137.17
119.48
77.22
77.03
76.85
49.75
44.92
32.65
31.70
30.67
29.31
29.21
27.62
27.48
25.82
24.01
23.39
23.07
Me
Me
Me
Me
Me
14: C15H28
MW: 208.39
sm
sm
200
180
160
140
120
100
80
60
40
20
0
ppm


NAME JAL10088A 1H
EXPNO 1
PROCNO 1
Date_ 20210115
Time 13.03 h
INSTRUM spect
PROBHD Z108618_0106 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.244532 Hz
AQ 4.0894966 sec
RG 32
DW 62.400 usec
DE 16.92 usec
TE 297.7 K
D1 1.00000000 sec
TD0 1
SFO1 400.1724710 MHz
NUC1 1H
P0 5.00 usec
P1 15.00 usec
SI 65536
SF 400.1700127 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
Me
O
OMe
15: C14H18O2
MW: 218.30
6.798
6.712
6.674
5.952
5.067
4.958
4.485
3.857
3.323
1.815
14
13
12
11
10
9
8
7
6
5
4
3
2
1
ppm
0.95
0.90
1.08
1.00
2.97
1.01
1.97
3.17
2.12
3.07

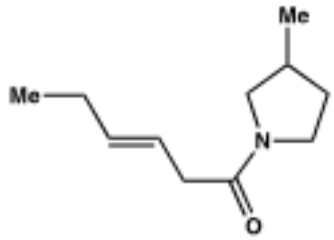

NAME JAL10172B 1H
EXPNO 2
PROCNO 1
Date_ 20210223
Time 12.57 h
INSTRUM spect
PROBHD Z108618_0106 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.244532 Hz
AQ 4.0894966 sec
RG 203
DW 62.400 usec
DE 16.92 usec
TE 297.7 K
D1 1.00000000 sec
TD0 1
SFO1 400.1724710 MHz
NUC1 1H
P0 5.00 usec
P1 15.00 usec
SI 65536
SF 400.1700110 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
Me
O
OMe
Me
Me
16: C15H22O2
MW: 234.34
2.401
6.782
6.676
6.632
5.079
4.961
4.484
3.858
2.401
1.824
0.894
2.60
2.55
2.50
2.45
2.40
2.35
1.00
15.13
14
13
12
11
10
9
8
7
6
5
4
3
2
1
ppm
1.28
1.13
1.09
1.32
1.21
2.36
3.72
2.00
4.57
7.15


NAME JAL10093A" 13C B600
EXPNO 1
PROCNO 1
Date_ 20210201
Time 13.07 h
INSTRUM spect
PROBHD Z814601_0054 (
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 516
DS 4
SWH 36231.883 Hz
FIDRES 1.105709 Hz
AQ 0.9044468 sec
RG 203
DW 13.800 usec
DE 6.50 usec
TE 298.1 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 150.9178988 MHz
NUC1 13C
P0 3.33 usec
P1 10.00 usec
SI 32768
SF 150.9028090 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
149.17
146.32
141.16
134.82
120.98
113.57
112.99
112.45
72.90
55.98
45.09
30.32
22.37
19.36
Me
O
OMe
Me
Me
16: C15H22O2
MW: 234.34
200
180
160
140
120
100
80
60
40
20
0
ppm

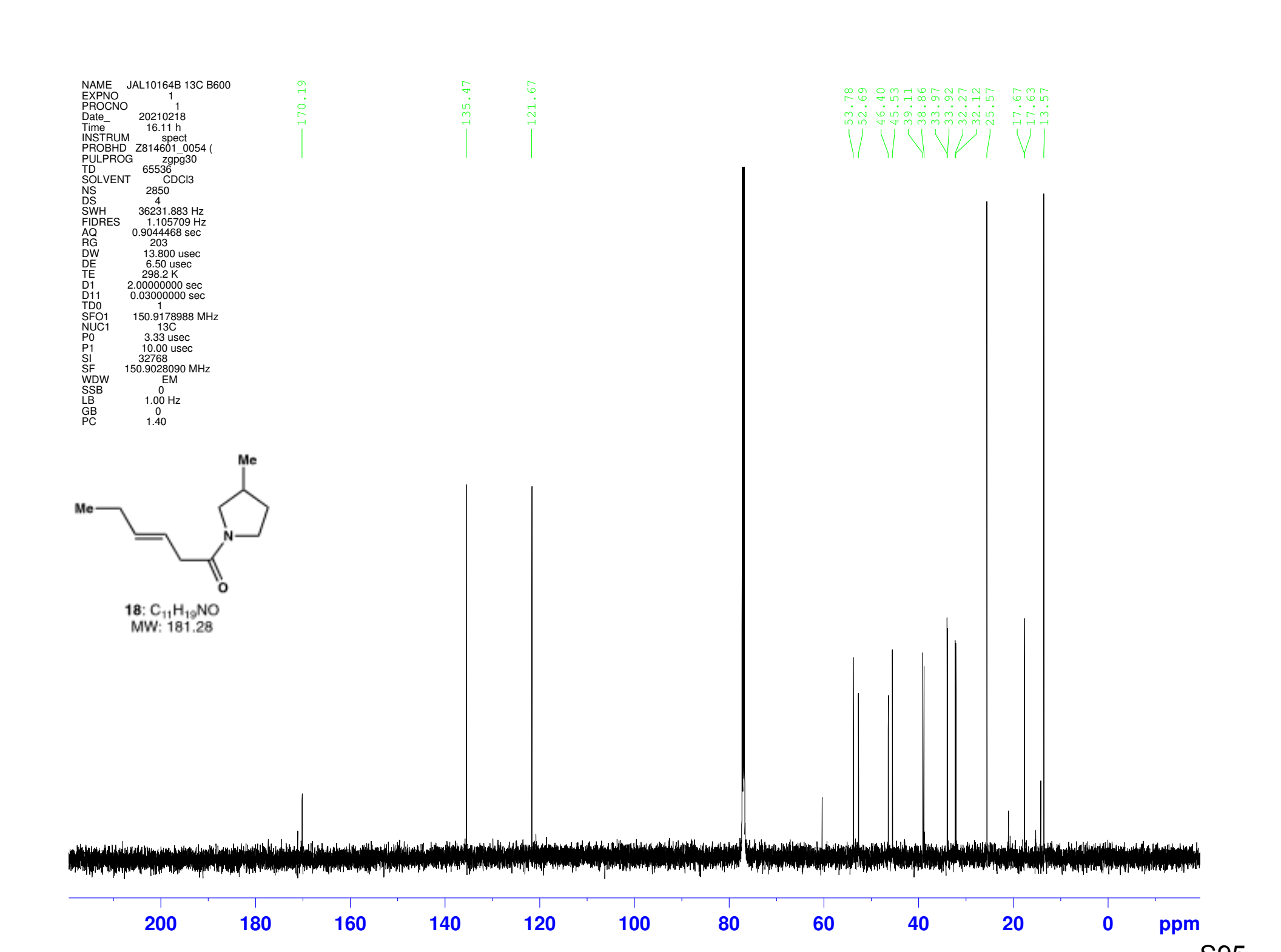

NAME JAL10045A 1H
EXPNO 1
PROCNO 1
Date_ 20201219
Time 14.02 h
INSTRUM spect
PROBHD Z108618_0106 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.244532 Hz
AQ 4.0894966 sec
RG 203
DW 62.400 usec
DE 16.92 usec
TE 297.8 K
D1 1.00000000 sec
TD0 1
SFO1 400.1724710 MHz
NUC1 1H
P0 5.00 usec
P1 15.00 usec
SI 65536
SF 400.1700131 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
Me
N
O
17: C10H15NO
MW: 165.24
benzene
5.859
5.782
5.580
4.244
3.027
2.053
0.981
14
13
12
11
10
9
8
7
6
5
4
3
2
1
ppm
1.00
1.02
1.92
3.92
1.92
2.10
2.98


NAME JAL10045A 13C B700
EXPNO 1
PROCNO 1
Date_ 20210110
Time 23.04 h
INSTRUM spect
PROBHD Z75813_0011 (C
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 42613.637 Hz
FIDRES 1.300465 Hz
AQ 0.7690057 sec
RG 203
DW 11.733 usec
DE 18.00 usec
TE 298.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 176.0880661 MHz
NUC1 13C
P0 4.00 usec
P1 12.00 usec
SI 32768
SF 176.0704590 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
170.08
135.92
126.42
124.92
121.22
53.37
53.06
38.84
25.61
13.56
Me
N
O
17: C10H15NO
MW: 165.24
200
180
160
140
120
100
80
60
40
20
0
ppm

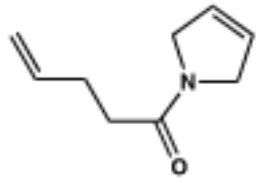
NAME JAL10164B' 1H B600
EXPNO 1
PROCNO 1
Date_ 20210301
Time 8.45 h
INSTRUM spect
PROBHD Z814601_0054 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 12019.230 Hz
FIDRES 0.366798 Hz
AQ 2.7263477 sec
RG 203
DW 41.600 usec
DE 11.27 usec
TE 298.3 K
D1 1.00000000 sec
TD0 1
SFO1 600.1337058 MHz
NUC1 1H
P0 3.33 usec
P1 10.00 usec
SI 65536
SF 600.1300199 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



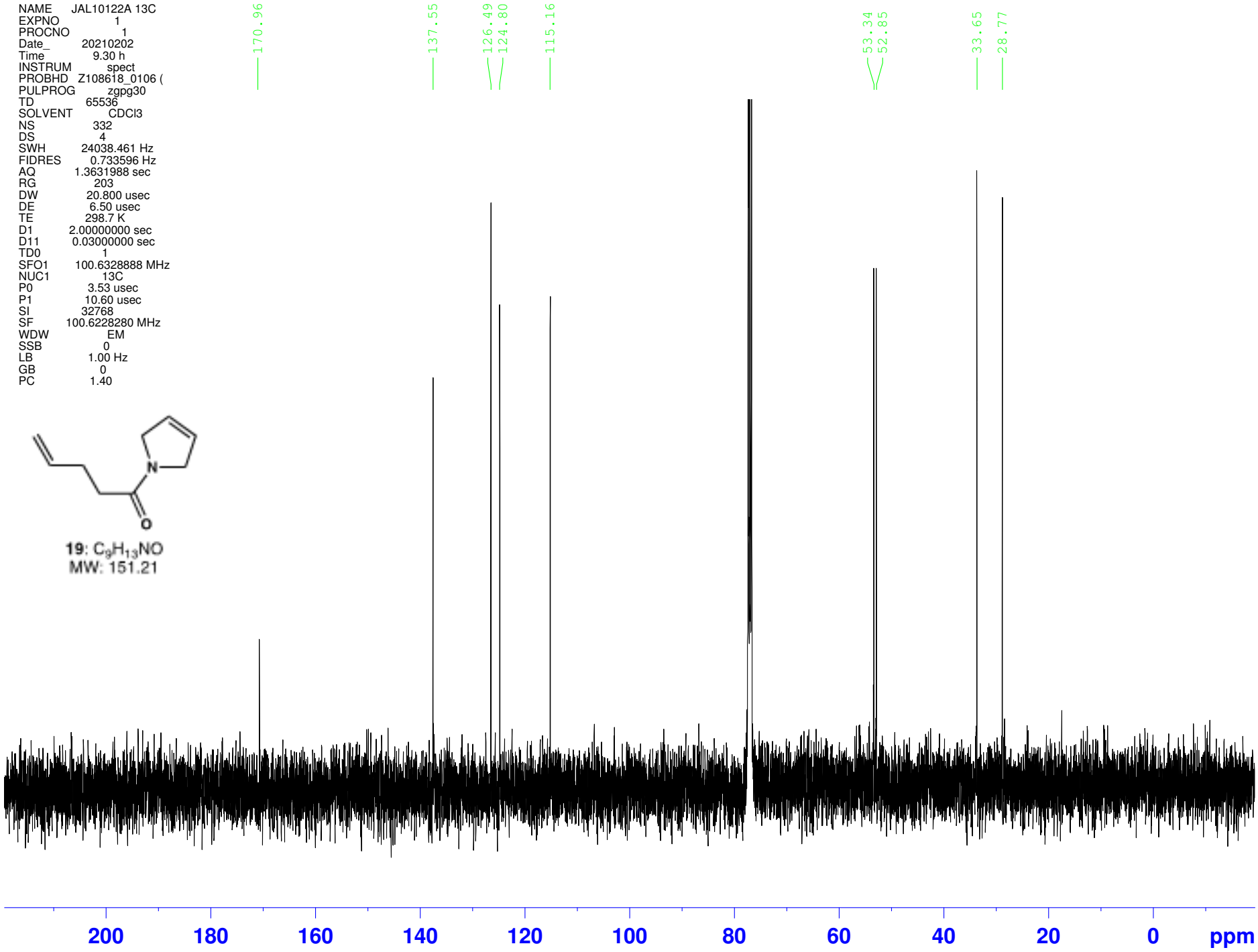
18: $C_{11}H_{19}NO$
MW: 181.28

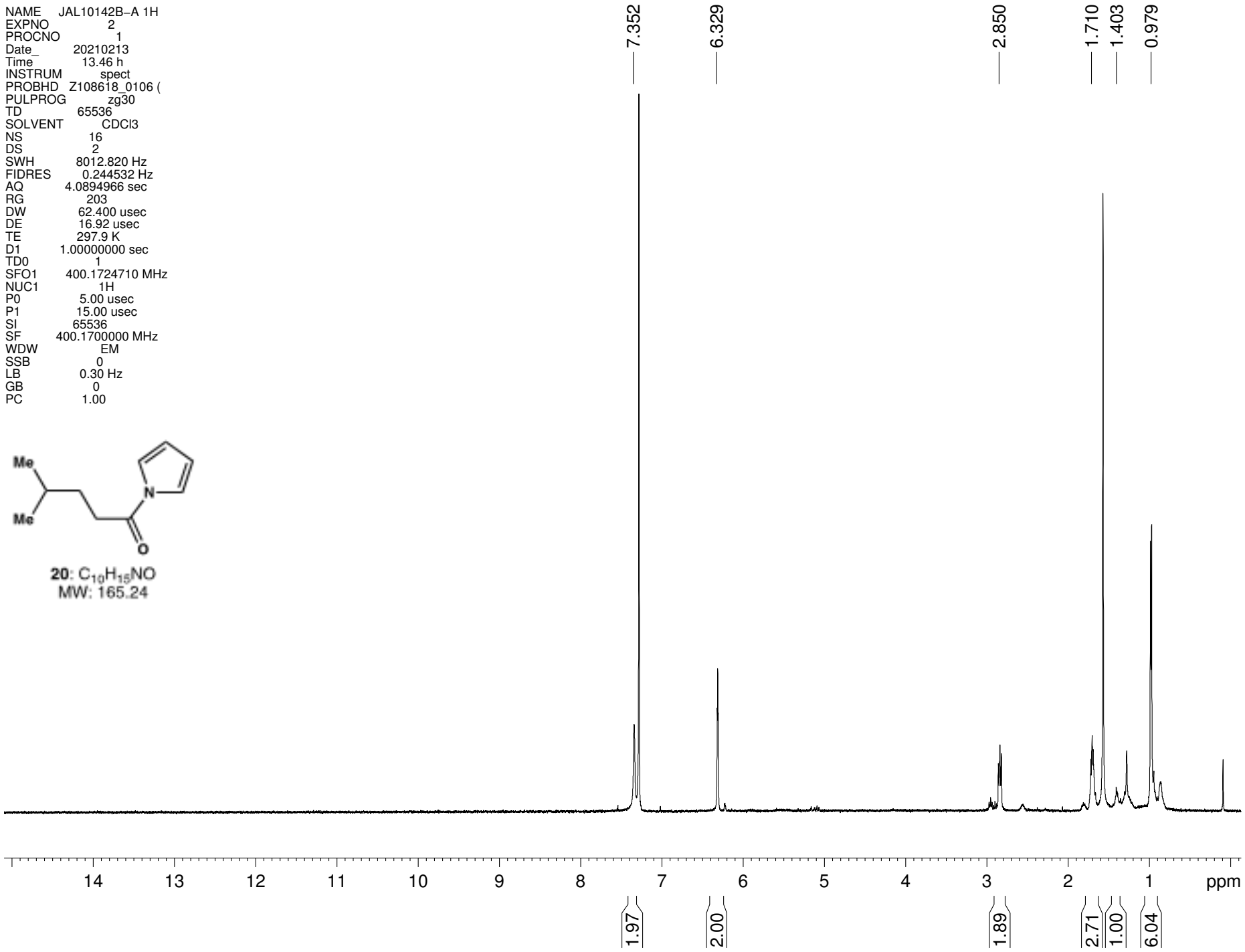

NAME JAL10164B 13C B600
EXPNO 1
PROCNO 1
Date_ 20210218
Time 16.11 h
INSTRUM spect
PROBHD Z814601_0054 (
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 2850
DS 4
SWH 36231.883 Hz
FIDRES 1.105709 Hz
AQ 0.9044468 sec
RG 203
DW 13.800 usec
DE 6.50 usec
TE 298.2 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 150.9178988 MHz
NUC1 13C
P0 3.33 usec
P1 10.00 usec
SI 32768
SF 150.9028090 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
170.19
135.47
121.67
53.78
52.69
46.40
45.53
39.11
38.86
33.97
33.92
32.27
32.12
25.57
17.67
17.63
13.57
Me
Me
N
O
18: C11H19NO
MW: 181.28
200
180
160
140
120
100
80
60
40
20
0
ppm

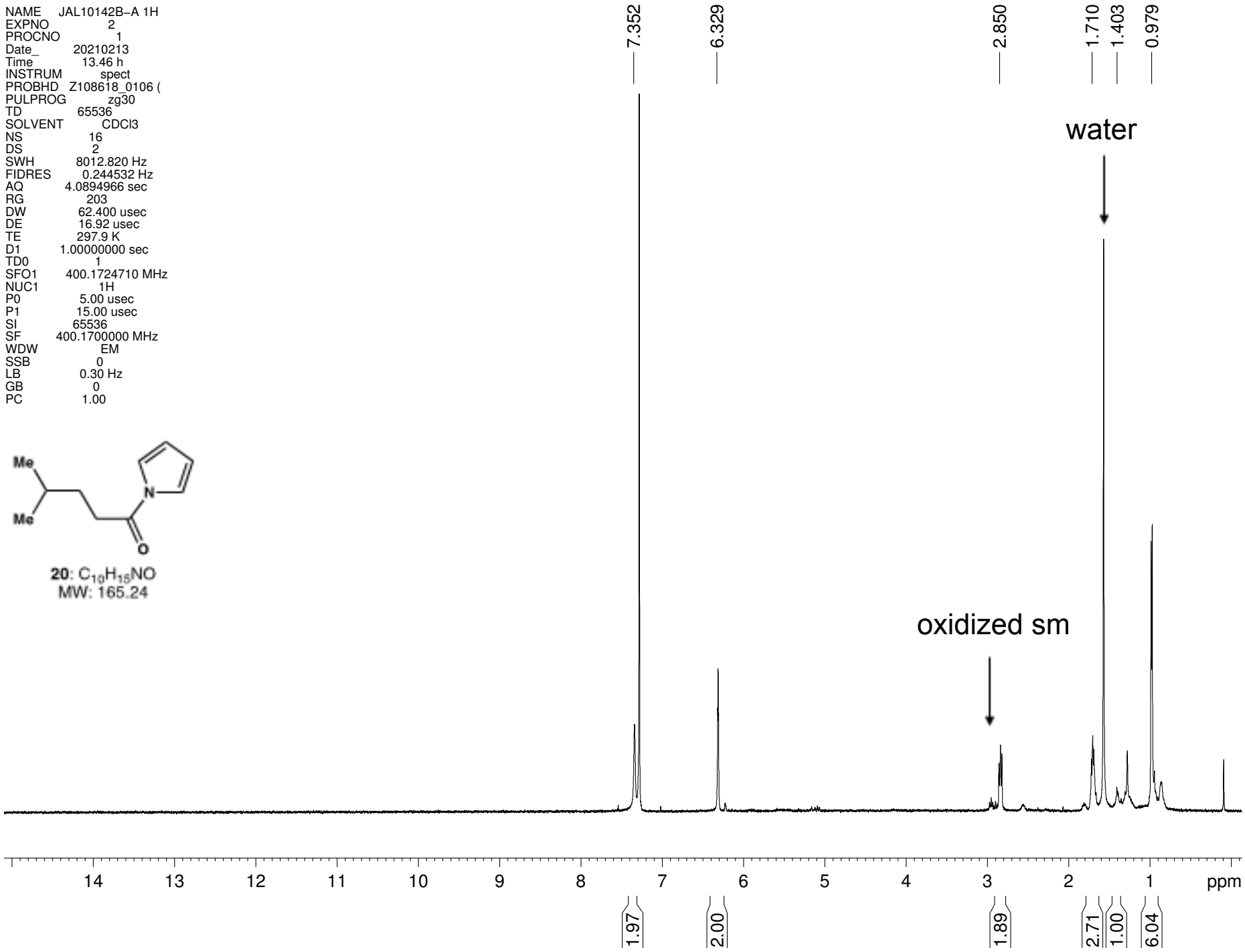
NAME JAL10122A 1H
EXPNO 2
PROCNO 1
Date_ 20210202
Time 8.55 h
INSTRUM spect
PROBHD Z108618_0106 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.244532 Hz
AQ 4.0894966 sec
RG 203
DW 62.400 usec
DE 16.92 usec
TE 298.0 K
D1 1.00000000 sec
TD0 1
SFO1 400.1724710 MHz
NUC1 1H
P0 5.00 usec
P1 15.00 usec
SI 65536
SF 400.1700110 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



19: $C_9H_{13}NO$
MW: 151.21


NAME JAL10122A 13C
EXPNO 1
PROCNO 1
Date_ 20210202
Time 9.30 h
INSTRUM spect
PROBHD Z108618_0106 (
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 332
DS 4
SWH 24038.461 Hz
FIDRES 0.733596 Hz
AQ 1.3631988 sec
RG 203
DW 20.800 usec
DE 6.50 usec
TE 298.7 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 100.6328888 MHz
NUC1 13C
P0 3.53 usec
P1 10.60 usec
SI 32768
SF 100.6228280 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
170.96
137.55
126.49
124.80
115.16
53.34
52.85
33.65
28.77
N
O
19: C9H13NO
MW: 151.21
200
180
160
140
120
100
80
60
40
20
0
ppm


NAME JAL10142B–A 1H
EXPNO 2
PROCNO 1
Date_ 20210213
Time 13.46 h
INSTRUM spect
PROBHD Z108618_0106 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.244532 Hz
AQ 4.0894966 sec
RG 203
DW 62.400 usec
DE 16.92 usec
TE 297.9 K
D1 1.00000000 sec
TD0 1
SFO1 400.1724710 MHz
NUC1 1H
P0 5.00 usec
P1 15.00 usec
SI 65536
SF 400.1700000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
Me
Me
N
O
20: C10H15NO
MW: 165.24
7.352
6.329
2.850
1.710
1.403
0.979
14
13
12
11
10
9
8
7
6
5
4
3
2
1
ppm
1.97
2.00
1.89
2.71
1.00
6.04


NAME JAL10142B–A 1H
EXPNO 2
PROCNO 1
Date_ 20210213
Time 13.46 h
INSTRUM spect
PROBHD Z108618_0106 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.244532 Hz
AQ 4.0894966 sec
RG 203
DW 62.400 usec
DE 16.92 usec
TE 297.9 K
D1 1.00000000 sec
TD0 1
SFO1 400.1724710 MHz
NUC1 1H
P0 5.00 usec
P1 15.00 usec
SI 65536
SF 400.1700000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
Me
Me
N
O
20: C10H15NO
MW: 165.24
7.352
6.329
2.850
1.710
1.403
0.979
water
oxidized sm
14
13
12
11
10
9
8
7
6
5
4
3
2
1
ppm
1.97
2.00
1.89
2.71
1.00
6.04