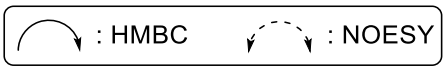
Supporting Information

Table of Contents

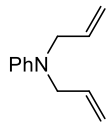
General remarks:

All reactions were performed with dry solvents and the reagents were purified by the usual methods. Reactions were monitored by thin-layer chromatography carried out on 0.25 mm Merck silica gel plates (60F-254). Column chromatography was performed with silica gel (Fuji Silysia, PSQ-60B) or NH-silica (Fuji Silysia, DM2035). NMR spectra were recorded on spectrometers of JEOL JMN-ECS-400, ECP-400, ECZ-400, ECZ-600, and ECA-600 operating at 400 and 600 MHz for ^{1}H NMR and 100 or 150 MHz for ^{13}C NMR with calibration using residual undeuterated solvent as an internal reference. IR spetra were recorded on JASCO FT/IR-4700. High resolution mass spectra were measured by The AccuTOFLC-plus JMS-T100LP (Ionization method: ESI).

General procedure for hydrocyanative cyclization: EtOH (0.2 M for diene) was added to the mixture of diene **1** (1.0 eq.), Co (2 mol%), TsCN (1.2 eq.) under an argon atmosphere at rt, then $PhSiH_3$ (1.2 eq.) was added at rt and the solution was stirred at room temperature. Then, the solvent was removed under reduced pressure, and the residue was purified by flash column chromatography.



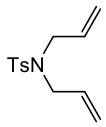
N,N-diallylaniline (**1a**)



(CAS-Reg# 6247-00-3)

^{1}H-NMR ($CDCl_3$, 400 MHz) δ: 3.79 (d, 2H, *J* = 4.8 Hz, 2 H), 5.17-5.33 (m, 2 H), 5.98 (m, 1 H) ,6.63-6.65 (m, 2 H), 6.71-6.75 (m, 1 H), 7.18-7.22 (m, 2 H)

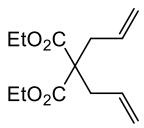
N,N-diallyl-4-methylbenzenesulfonamide (**1b**)



(CAS-Reg# 50487-72-4)

^{1}H-NMR ($CDCl_3$, 400 MHz) δ: 2.39 (s, 3H), 3.77 (d, 4H, *J* = 6.4 Hz), 5.08-5.51 (m, 4H), 5.56 (ddt, 2H, *J* = 16.4 Hz, 10.0 Hz, 6.4 Hz), 7.27 (d, 2H, *J* = 8.0 Hz), 7.67 (d, 1H, *J* = 8.4 Hz)

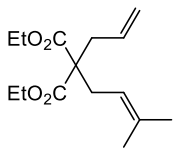
diethyl 2,2-diallylmalonate (**1c**)



(CAS-Reg# 3195-24-2)

^{1}H-NMR ($CDCl_3$, 400 MHz) δ: 1.23 (t, 6 H, *J* = 7.2 Hz), 2.62 (d, 4 H, *J* = 7.2 Hz), 4.17 (q, 4 H, *J* = 7.2 Hz), 5.08 (d, 1 H, *J* = 11.2 Hz), 5.09 (d, 1 H, *J* = 16.0 Hz), 5.65 (m, 1 H)

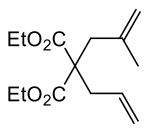
diethyl 2-allyl-2-(3-methylbut-2-en-1-yl)malonate (**1d**)



(CAS-Reg# 408333-38-0)

^{1}H-NMR ($CDCl_3$, 400 MHz) δ:1.23 (t, *J* = 7.2 Hz, 6H), 1.61 (s, 3H), 1.69 (s, 3H), 2.58-2.64 (m, 2H), 4.12-4.22 (m, 4H), 4.94-5.00 (m, 1H), 5.01 (s, 1H), 5.08-5.12 (m, 1H), 5.60-5.72 (m, 1H)

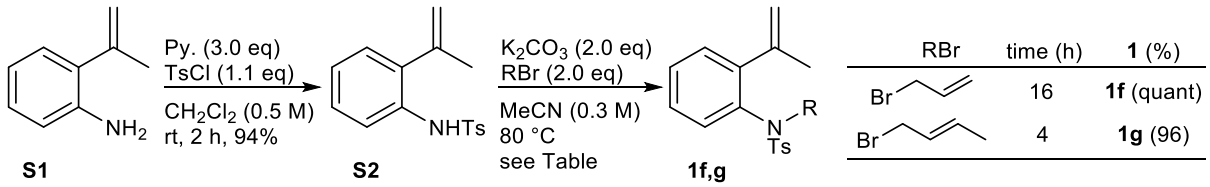
diethyl 2-allyl-2-(2-methylallyl)malonate (**1e**)



(CAS-Reg# 5309-50-2)

^{1}H-NMR ($CDCl_3$, 400 MHz) δ: 1.25 (t, 6H, *J* = 7.2 Hz), 1.67 (s, 3H), 2.67 (d, 2H, *J* = 7.2 Hz), 2.69-2.71 (m, 2H), 4.13-4.22 (m, 4H), 4.75-4.77 (m, 1H), 4.86-4.89 (m, 1H), 5.06-5.08 (m, 1H), 5.11 (d, 1H, *J* = 6.0 Hz), 5.64-5.75 (m, 1H).

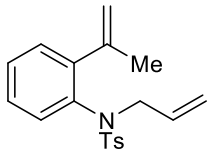
Synthesis of 1f,g



S1 to **S2**: To a stirred solution of **S1** (2 mL, 14.7 mmol) in CH_2Cl_2 (29 mL) was added pyridine (3.6 mL, 44.1 mmol), and TsCl (3.1 g, 16.2 mmol) at 0 °C, and the reaction mixture was stirred for 2 h at rt. The reaction was quenched with 1 N HCl, extracted with CH_2Cl_2, dried over Na_2SO_4, filtered, and concentrated in vacuo. Purification by flash column chromatography (SiO_2, n-hexane/AcOEt = 2/1) afforded **S2** (3.96 g, 94%)

S2 to **1f,g**: To a stirred solution of **S2** (1.0 eq.) in MeCN (0.3 M for **S2**) was added K_2CO_3 (2.0 eq.), and RX (2.0 eq.), and the reaction mixture was stirred at 80 °C. The reaction was quenched with H_2O, extracted with AcOEt, dried over Na_2SO_4, filtered, and concentrated in vacuo. Purification by flash column chromatography afforded **1f,g**.

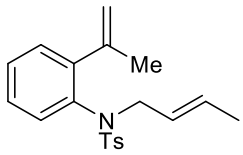
N-allyl-4-methyl-N-(2-(prop-1-en-2-yl)phenyl)benzenesulfonamide (1f)



(CAS-Reg# 402822-74-6)

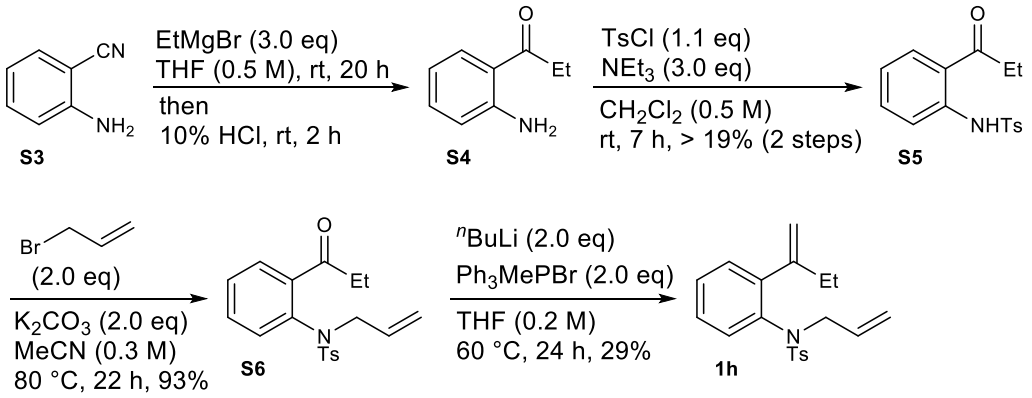
^{1}H-NMR ($CDCl_3$, 400 MHz) δ: 2.18 (s, 3H), 2.44 (s, 3H), 4.12 (d, 2H, *J* = 6.8 Hz), 4.94 (dd, 1H, *J* = 11.2, 1.6 Hz), 4.98 (d, 1H, *J* = 3.2 Hz), 5.05 (dd, 1H, *J* = 1.2, 0.8 Hz), 5.22 (dd, 1H, *J* = 1.6, 1.2 Hz), 5.69 (dddd, 1H, *J* = 17.2, 11.2, 6.8, 6.8 Hz), 6.74 (d, 1H, *J* = 8.0 Hz), 7.12 (ddd, 1H, *J* = 7.8, 6.4, 2.4 Hz), 7.27-7.31 (m, 4H), 7.67 (d, 2H, *J* = 8.4 Hz); (675 mg, quant.)

(*E*)-N-(but-2-en-1-yl)-4-methyl-N-(2-(prop-1-en-2-yl)phenyl)benzenesulfonamide (1g)



^{1}H-NMR ($CDCl_3$, 400 MHz) δ: 1.49 (d, 3H, *J* = 5.6 Hz), 2.17(s, 3H), 2.43 (s, 3H), 4.05 (brs, 2H), 5.05 (s, 1H), 5.21 (s, 1H), 5.26-5.42 (m, 2H), 6.71 (d, 1H, *J* = 8.4 Hz), 7.11 (dd, 1H, *J* = 7.6, 2.0 Hz), 7.21-7.29 (m, 4H), 7.65 (d, 2H, *J* = 8.4 Hz); ^{13}C-NMR ($CDCl_3$, 100 MHz) δ: 17.5, 21.4, 24.1, 24.2, 54.0, 116.3, 125.0, 127.1, 127.9, 128.0, 128.5, 129.3, 129.8, 130.6, 136.4, 136.8, 143.2, 143.6, 144.9 11; IR (ATR) ν: 2982, 1736, 1237, 1044 cm^{-1}; HRMS (ESI) Calcd for $C_{20}H_{23}NNaO_2S$, $[M+Na]^+$ 364.1347, found; 364.1360; yellow oil (126.2 mg, 96%).

Synthesis of 1h



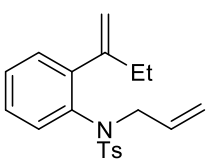
S3 to **S4**: Ethylmagnesium bromide (10 mL, 3.0 M in Et_2O, 30 mmol) was added to a solution of **S3** (1.18 g, 10 mmol) in THF (20 mL) at 0 °C. Then the reaction was allowed to warm to ambient temperature and allowed to stir at this temperature for 20 h. The reaction was quenched by slow addition of 10% HCl and made basic by the addition of NaOH at 0 °C. The organic layer was separated and the remaining aqueous layer was extracted with AcOEt for three times. The combined organic layers were washed with brine, dried over Na_2SO_4 and concentrated. The crude **S4** (1.68 g) was used in the next step without further purification.

S4 to **S5**: To a stirred solution of crude **S4** (1.68 g) in CH_2Cl_2 (20 mL) was added NEt_3 (4.2 mL, 30 mmol), and TsCl (1.9 g, 10 mmol) at 0 °C, and the reaction mixture was stirred for 7 h at rt. The reaction was quenched with 1 N HCl, extracted with CH_2Cl_2, dried over Na_2SO_4, filtered, and concentrated in vacuo. Purification by flash column chromatography (SiO_2, n-hexane/AcOEt = 4/1) afforded **S5** (573 mg, 19%, 2 steps)

S5 to **S6**: To a stirred solution of **S5** (150 mg, 0.49 mmol) in MeCN (1.6 mL) was added K_2CO_3 (136 mg, 0.99 mmol), and allyl bromide (0.08 mL, 0.99 mmol), and the reaction mixture was stirred at 80 °C for 22 h. The reaction was quenched with 1N HCl, extracted with AcOEt, dried over Na_2SO_4, filtered, and concentrated in vacuo. Purification by flash column chromatography afforded **S6** (158 mg, 93%).

S6 to **1h**: To a stirred solution of Ph_3PMeBr (357 mg, 1.0 mmol) in THF (2.5 mL) was added *n*-BuLi (0.63 mL, 1.6 M in hexane, 1.0 mmol) at 0 °C, and the reaction mixture was stirred at rt for 1 h. Then, **S6** (158 mg, 0.46 mmol)in THF (1.0 mL) was added at 0 °C, and the reaction mixture was stirred at 60 °C for 24 h. The reaction was quenched with H_2O, extracted with AcOEt, dried over Na_2SO_4, filtered, and concentrated in vacuo. Purification by flash column chromatography afforded **1h** (45.6 mg, 29%).

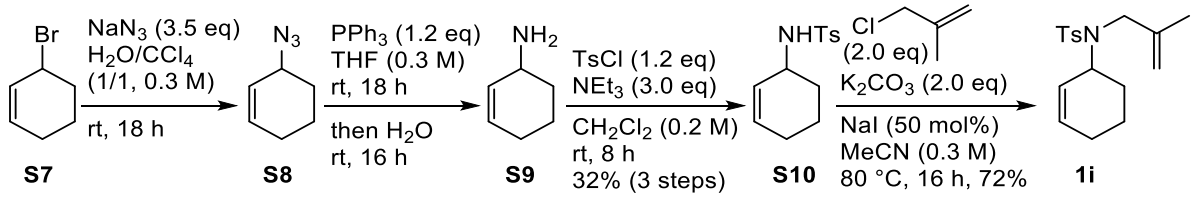
N-allyl-N-(2-(but-1-en-2-yl)phenyl)-4-methylbenzenesulfonamide (**1h**)



^{1}H-NMR ($CDCl_3$, 400 MHz) δ: 1.07 (t, 3H, *J* = 7.2 Hz), 2.45 (s, 3H), 2.50 (q, 2H, *J* = 7.2 Hz), 4.11 (d, 2H, *J* = 6.8 Hz), 4.92-4.98 (m, 2H), 5.04 (s, 1H), 5.21 (d, 1H, *J* = 6.0 Hz), 5.66 (ddd, 1H, *J* = 16.8, 10.4, 6.8 Hz), 6.79 (d, 1H, *J* = 8.0 Hz), 7.14 (ddd, 1H, *J*

= 7.6, 7.6, 2.0 Hz), 7.20-7.27 (m, 2H), 7.30 (d, 2H, *J* = 8.0 Hz), 7.69 (d, 2H, *J* = 8.0 Hz); ^{13}C-NMR ($CDCl_3$, 100 MHz) δ: 12.5, 21.5, 29.7, 54.6, 114.2, 119.1, 127.2, 128.0, 128.2, 128.8, 129.5, 130.7, 132.6, 136.5, 137.1, 143.4, 144.6, 149.8; IR (ATR) ν: 2965, 1349, 1165, 1091 cm^{-1}; HRMS (ESI) Calcd for $C_{20}H_{23}NNaO_2S$, $[M+Na]^+$ 364.1347, found 364.1356; yellow oil

Synthesis of 1i



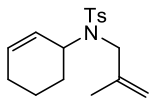
S7 to **S8**: To a stirred solution of **S7** (1.0 mL, 8.6 mmol) in CCl_4 (15 mL) at rt was added NaN_3 (1.95 g, 30 mmol) in H_2O (15 mL), and the reaction mixture was stirred for 18 h at rt. The reaction mixture was extracted with CH_2Cl_2, dried over Na_2SO_4, filtered, and concentrated in vacuo gave crude **S8** which was used in the next step without further purification.

S8 to **S9**: A mixture of the crude **S8** (approximately 8.6 mmol), PPh_3 (2.7 g, 10.32 mmol), THF (29 mL) was stirred for 16 h. The mixture was extracted with 1 N HCl (3 times). The combined aqueous phases were made basic with NaOH. The aqueous solution was extracted with CH_2Cl_2. The organic phases were combined, washed with water and brine, dried over sodium sulfate, filtered, and concentrated under reduced pressure to yield crude **S9** (444 mg) which was used in the next step without further purification.

S9 to **S10**: To a stirred solution of crude **S9** (444 mg) in CH_2Cl_2 (23 mL) was added NEt_3 (1.9 mL, 13.71 mmol), and TsCl (1.05 g, 5.48 mmol) at 0 °C, and the reaction mixture was stirred for 8 h at rt. The reaction was quenched with 1 N HCl, extracted with CH_2Cl_2, dried over Na_2SO_4, filtered, and concentrated in vacuo. Purification by flash column chromatography (SiO_2, *n*-hexane/AcOEt = 5/1) afforded **S10** (686 mg, 32%, 3 steps)

S10 to **1i**: A solution of crude **S10** (234 mg), K_2CO_3 (259 mg, 1.88 mmol), 4-chloro-2-methyl-1-butene (0.18 mL, 1.88 mmol), NaI (71 mg, 0.47 mmol) in MeCN (3.1 mL) was stirred for 8 h at 80 °C. The reaction was quenched with aqueous sodium thiosulfate, extracted with AcOEt, dried over Na_2SO_4, filtered, and concentrated in vacuo. Purification by flash column chromatography (SiO_2, *n*-hexane/AcOEt = 10/1) afforded **1i** (219.9 mg, 72%)

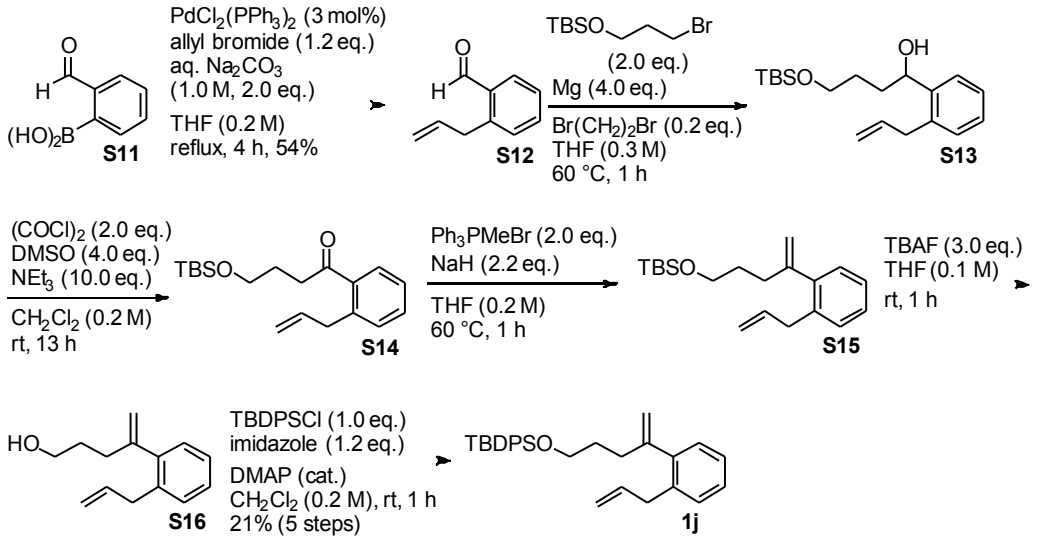
N-(cyclohex-2-en-1-yl)-4-methyl-N-(2-methylallyl)benzenesulfonamide (**1i**)



^{1}H-NMR ($CDCl_3$, 400 MHz) δ: 1.52-1.65 (m, 2H), 1.71-1.75 (m, 1H), 1.77 (s, 3H), 1.85-1.93 (m, 3H), 2.43 (s, 3H), 3.44 (16.4 Hz), 3.80 (d, 1H, *J* = 16.4 Hz), 4.48-4.55 (m, 1H), 4.88 (s, 1H), 4.93 (d, 1H, *J* = 10.8 Hz), 5.00 (s, 1H), 5.69-5.81 (m, 1H), 7.28 (d, 2H, *J* = 8.0 Hz), 7.71 (d, 2H, *J* = 8.0 Hz); ^{13}C-NMR ($CDCl_3$, 100 MHz) δ:20.0, 21.3, 21.6, 24.2, 28.5, 49.6,

55.4, 112.0, 126.6, 126.8, 129.5, 132.3, 137.8, 142.7, 142.9; IR (ATR) ν: 2935, 1337, 1161, 759 cm^{-1}; HRMS (ESI) Calcd for $C_{17}H_{23}NNaO_2S$, $[M+Na]^+$ 328.1347, found 328.1343; yellow oil

Synthesis of 1j



S12 to **S13**: A solution of Mg (566 mg, 23.28 mmol) and 1,2-dibromoethane (0.1 mL, 1.16 mmol) in THF (3.6 mL) was stirred at 60 °C for 15 min, then cooled to rt, and a solution of $Br(CH_2)_3OTBS$ (2.95 g, 11.64 mmol) in THF (6 mL) was added slowly at rt, and the mixture was stirred for 10 min at rt. Then the solution was stirred at 60 °C for 1 h, the solution of **S12** (850.8 mg, 5.82 mmol) in THF (6 mL) was added at 0 °C, and the solution was stirred for another 2 h at room temperature. The solution was dissolved in AcOEt, sat. NH_4Cl aq. was added and the aqueous phase was extracted with AcOEt. The combined organic phases were washed with water and sat. sodium chloride solution and dried over Na_2SO_4. The solvent was removed in vacuum to leave a crude mixture, which was filtered through a short silica gel column (eluting with hexane/AcOEt = 50/1) to afford crude **S13** (940 mg).

S13 to **S14**: To an oven-dried 100 mL round-bottomed flask equipped with a stir bar was added 13 mL of CH_2Cl_2 and dimethyl sulfoxide (0.89 mL, 12.48 mmol), and the reaction mixture was cooled in a -78 °C. Oxalyl chloride (0.54 mL, 6.24 mmol) was added carefully, and the reaction stirred for 30 min at -78 °C, then crude **S13** (940 mg) in CH_2Cl_2 (3 mL) was added, and the reaction mixture was stirred 30 min at -78 °C. Triethylamine (4.3 mL, 31.2 mmol) was added, and the reaction was allowed to warm to r.t. over 13 h. The reaction mixture was concentrated in vacuo and partitioned between EtOAc and 1 M HCl (aq). The layers were separated, and the aqueous layer was extracted with EtOAc (×1). The combined organic layers were washed with sat. $NaHCO_3$ (aq), water, brine, dried over $MgSO_4$, and concentrated in vacuo. This crude mixture was filtered through a short silica gel column (eluting with hexane/AcOEt = 20/1) to afford crude **S14** (614.6 mg)

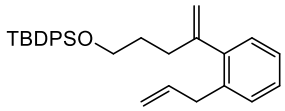
S14 to **S15**: To a suspension of $MePPh_3Br$ (1.38 g, 3.86 mmol) in THF (8 mL) was added NaH in one portion (204 mg, 4.25 mmol) at 0 °C, and the resulting mixture was stirred at 60 °C for 1 h. Then, crude **S14** (614.6 mg) in THF (2 mL) was added at 60 °C, and the mixture was stirred 1 h at 60 °C. A saturated ammonium chloride solution was added and the aqueous phase was extracted with AcOEt. The organic

phase was dried over Na_2SO_4, filtered and concentrated. The compound was purified by flash (10:1 Hex:EtOAc) to give crude **S15** (497 mg)

S15 to **S16**: TBAF (4.7 mL, 1.0 M in THF, 4.7 mmol) was added to the solution of crude **S15** (497 mg) in THF (15.7 mL) at 0 °C, and the solution was stirred at rt for 1 h. H_2O was added and the aqueous phase was extracted with AcOEt. The organic phase was dried over Na_2SO_4, filtered and concentrated. The compound was purified by flash (150:1 Hex:EtOAc) to give crude **S16** (220 mg)

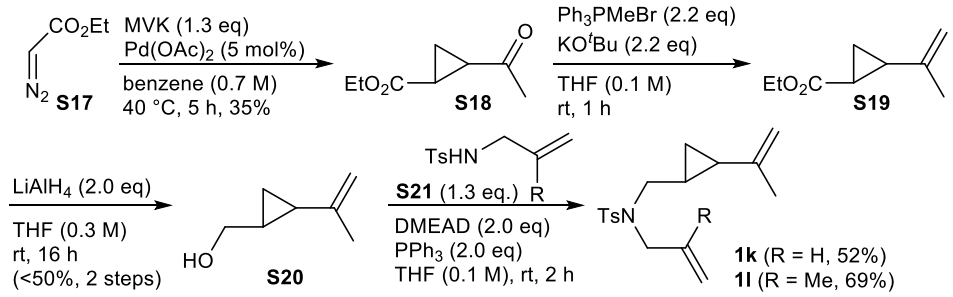
S16 to **1j**: To a solution of crude **S16** (78.2 mg), 1H-imidazole (31.6 mg, 0.46 mmol), DMAP (2 blocks) in CH_2Cl_2 (1.9 mL) was added TBDPSCl (0.1 mL, 0.387 mmol) at 0 °C, and the mixture was stirred at rt for 1 h. sat. NH_4Cl aq. was added and the aqueous phase was extracted with CH_2Cl_2. The organic phase was dried over Na_2SO_4, filtered and concentrated. The compound was purified by flash (75:1 Hex:EtOAc) to give **1j** (147.1 mg, 21%, 5 steps)

((4-(2-allylphenyl)pent-4-en-1-yl)oxy)(tert-butyl)diphenylsilane (**1j**)



^{1}H-NMR ($CDCl_3$, 400 MHz) δ: 1.03 (s, 9H), 1.66 (tt, 2H, *J* = 8.0, 6.4 Hz), 2.47 (t, 2H, *J* = 8.0 Hz), 3.36 (d, 2H, *J* = 6.4 Hz), 3.66 (t, 2H, *J* = 6.4 Hz), 4.86 (d, 1H, *J* = 1.2 Hz), 4.96-5.04 (m, 2H), 5.16 (d, 1H, *J* = 1.2 Hz), 5.87-5.97 (m, 1H), 7.03-7.47 (m, 10H), 7.64 (dd, 4H, *J* = 8.0, 2.0 Hz); ^{13}C-NMR ($CDCl_3$, 100 MHz) δ: 19.2, 26.8, 30.7, 34.5, 37.1, 63.4, 114.0, 115.6, 125.7, 126.8, 127.6, 128.5, 129.4, 129.5, 133.9, 135.5, 136.6, 138.0, 142.9, 149.2; IR (ATR) ν: 2938, 1740, 1239, 1047 cm^{-1}; HRMS (ESI) Calcd for $C_{30}H_{36}NaO_{Si,}$ $[M+Na]^+$ 463.2433,found 463.2435; yellow oil

Synthesis of 1k,l



S17 to **S18**: To a stirred solution of MVK (10.1 mL, 123.1 mmol), $Pd(OAc)_2$ (500 mg, 2.23 mmol) in benzene (50 mL) was added **S17** (5 mL, 47.3 mmol) in benzene (40 mL) at 40 °C using dropping funnel over 1 h. Then, $Pd(OAc)_2$ (500 mg, 2.23 mmol) was added and the solution of **S18** (5 mL, 47.3 mmol) in benzene (40 mL) was added at 40 °C using dropping funnel over 1 h. The reaction mixture was stirred for 5 h at 40 °C, and the reaction was quenched with sat. Na_2CO_3, extracted with Et_2O, dried over Na_2SO_4, filtered, and concentrated in vacuo. Purification by flash column chromatography (SiO_2, n-hexane/AcOEt = 10/1) afforded **S18** as a yellow oil (5.21 g, 35%).

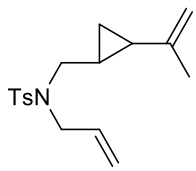
S18 to **S19**: To a stirred solution of Ph_3PMeBr (5.5 g, 15.4 mmol) in THF (46 mL) was added *t*-BuOK

(1.73 g, 15.4 mmol) at 0 °C, and the reaction mixture was stirred at 0 °C for 30 min. Then, **S18** (1.0 g, 6.4 mmol) in THF (18 mL) was added at 0 °C, and the reaction mixture was stirred at rt for 1 h. The reaction was quenched with H_2O, extracted with Et_2O, dried over Na_2SO_4, filtered, and concentrated in vacuo ***at 0 °C***. Purification by flash column chromatography (Et_2O : pentane = 1 : 4) afforded crude **S19** (925 mg) as a colorless liquid.

S19 to **S20**: $LiAlH_4$ (456 mg, 12 mmol) was added to a solution of crude **S19** (925 mg) in Et_2O (20 mL) at 0 °C under argon atmosphere. After addition, the mixture was warmed to rt, and the mixture was stirred for 16 h. Then, worked up by precipitation with 0.5 mL of water and 0.5 mL of 15% NaOH and 1.5 mL of water at 0 °C, and filtration through celite and washed thoroughly with Et_2O. The solvent was reduced under vacuum ***at 0 °C***. Purification by flash column chromatography (Et_2O) afforded crude **S20** (362 mg) as a yellow oil.

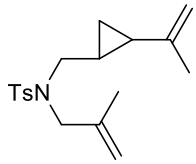
S20 to **1k,l**: To a stirred solution of **S20** (1.0 eq.), **S21** (1.3 eq.), PPh_3 (2.0 eq.) in THF (0.1 M for **S20**) was added DMEAD (2.0 eq.) at 0 °C, and the reaction mixture was stirred for 2 h at rt. Then, the reaction mixture was concentrated in vacuo. Purification by flash column chromatography (hexane/AcOEt = 20/1) gave **1k,l**.

N-allyl-4-methyl-N-((2-(prop-1-en-2-yl)cyclopropyl)methyl)benzenesulfonamide (**1k**)



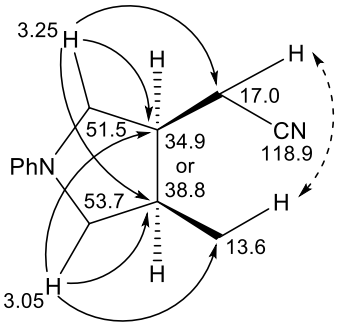
^{1}H-NMR ($CDCl_3$, 400 MHz) δ: 0.47-0.52 (m, 1H), 0.68-0.72 (m, 1H), 0.97-1.05 (m, 1H), 1.56 (s, 3H), 2.42 (s, 3H), 2.99 (dd, 1H, *J* = 14.0, 7.2 Hz), 3.18 (dd, 1H, *J* = 14.0, 6.4 Hz), 3.84 (dd, 1H, *J* = 15.6, 6.4 Hz), 3.93 (dd, 1H, *J* = 15.6, 6.4 Hz), 4.62 (s, 1H), 4.65 (s, 1H), 5.13 (d, 1H, *J* =11.6 Hz), 5.20 (d, 1H, *J* = 16.8 Hz), 5.60-5.70 (m, 1H), 7.28 (d, 2H, *J* = 8.4 Hz), 7.69 (d, 2H, *J* = 8.4 Hz); ^{13}C-NMR ($CDCl_3$, 100 MHz) δ: 11.0, 17.2, 20.7, 21.3, 24.6, 49.8, 50.6, 108.7, 118.3, 126.9, 129.5, 133.2, 137.3, 142.9, 144.5; IR (ATR) ν: 2921, 1737, 1338, 1153, 1090, 658 cm^{-1}; HRMS (ESI) Calcd for $C_{17}H_{23}NNaO_2S$, $[M+Na]^+$ 328.1347, found 328.1342; yellow oil (241.3 mg, 52%).

4-methyl-N-(2-methylallyl)-N-((2-(prop-1-en-2-yl)cyclopropyl)methyl)benzenesulfonamide (**1l**)



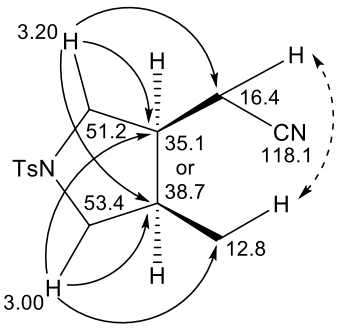
^{1}H-NMR ($CDCl_3$, 400 MHz) δ:0.43 (br, 1H), 0.63 (br, 1H), 0.98 (br, 1H), 1.18 (br, 1H), 1.50 (s, 3H), 1.71 (s, 3H), 2.40 (s, 3H), 2.90-2.96 (m, 1H), 3.15-3,19 (m, 2H), 3.68-3.86 (m, 3H), 4.57 (d, 1H, *J* = 6.4 Hz), 4.63 (d, 1H, *J* = 6.4 Hz), 4.89 (s, 2H), 7.28 (d, 2H, *J* = 8.0 Hz), 7.72 (d, 2H, *J* = 8.0 Hz); ^{13}C-NMR ($CDCl_3$, 100 MHz) δ:11.1, 16.8, 19.8, 19.9, 21.4, 24.8, 51.1, 53.7, 108.7, 114.0, 127.0, 129.5, 137.3, 140.7, 143.0, 144.7; IR (ATR) ν: 2981, 1735, 1337, 1239, 1156 cm^{-1}; HRMS (ESI) Calcd for $C_{18}H_{25}NNaO_2S$, $[M+Na]^+$ 342.1504, found 342.1515; yellow oil (301.6 mg, 69%).

2-4-methyl-1-phenylpyrrolidin-3-yl)acetonitrile (2a)



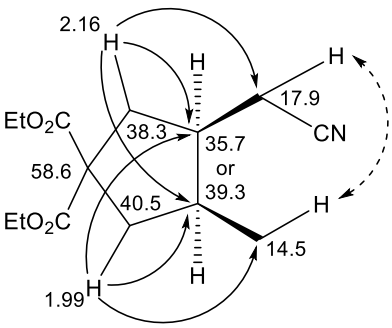
^{1}H-NMR ($CDCl_3$, 400 MHz) δ: 1.05 (dd, 2.1H, *J* = 6.8, 1.6 Hz), 1.16 (d, 0.9H, *J* = 4.4 Hz), 2.10-2.72 (m, 4H), 2.97 (dd, 0.3H, *J* = 7.6, 7.6 Hz), 3.05 (dd, 0.7H, *J* = 7.2, 7.2 Hz), 3.17 (dd, 0.3 H, *J* = 7.2, 7.2 Hz), 3.25 (dd, 0.7H, *J* = 8.4, 5.6 Hz), 3.49 (ddd, 1.4 H, *J* = 14.4, 7.2, 7.2 Hz), 3.58 (ddd, 0.6H, *J* = 14.4, 7.2, 7.2 Hz), 6.52 (d, 2H, *J* = 7.6 Hz), 6.69 (dd, 1H, *J* = 7.6, 7.6 Hz), 7.23 (dd, 2H, *J* = 7.6, 7.6 Hz); ^{13}C-NMR ($CDCl_3$, 100 MHz) δ:13.6, (16.8), 17.0, (19.6), 34.9, (38.0), 38.8, (41.8), 51.5, (52.4), 53.7, (54.7), 111.4, 116.0, 118.9, 129.2, 147.2; IR (ATR) ν: 2974, 1736, 1345 cm^{-1}; HRMS (ESI) Calcd for $C_{13}H_{16}N_2Na$, $[M+Na]^+$ 223.1211, found 223.1203; yellow oil (28.9 mg, 47%); *( ) in ^{13}C NMR is peak of *trans*-**2a**

2-4-methyl-1-tosylpyrrolidin-3-yl)acetonitrile (2b)



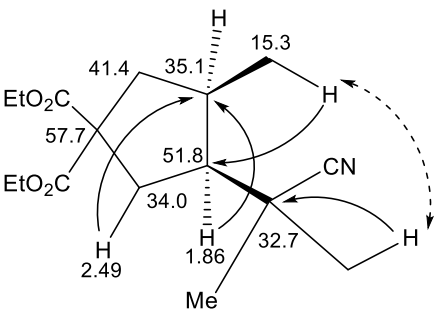
^{1}H-NMR ($CDCl_3$, 400 MHz) δ: 0.87 (d, 2.1H, *J* = 6.4 Hz), 0.98 (d, 0.9H, *J* = 6.4 Hz), 1.86-2.48 (m, 7H), 2.85 (dd, 0.3H, *J* = 10.4, 8.0 Hz), 3.00 (dd, 0.7H, *J* = 10.4, 5.6 Hz), 3.06 (dd, 0.3H, *J* = 10.4, 8.0 Hz), 3.20 (dd, 0.7H, *J* = 10.4, 5.6 Hz), 3.41-3.58 (m, 2H), 7.34 (d, 2H, *J* = 8.4 Hz), 7.72 (d, 2H, *J* = 8.4 Hz); ^{13}C-NMR ($CDCl_3$, 100 MHz)* δ: 12.8, (16.2), 16.4, (19.1), 21.5, 35.1, (38.1), 38.7, (41.7), 51.2, (52.0), 53.4, (54.3), 118.1, 127.4, 129.8, 133.4, 143.8; IR (ATR) ν: 2970, 1736, 1345, 1167 cm^{-1}; HRMS (ESI) Calcd for $C_{14}H_{18}N_2NaO_2S$, $[M+Na]^+$ 301.0987, found 301.0998; yellow oil (35.2 mg, 68%); *( ) in ^{13}C NMR is peak of *trans*-**2b**

diethyl-3-(cyanomethyl)-4-methylcyclopentane-1,1-dicarboxylate (2c)



^{1}H-NMR ($CDCl_3$, 400 MHz) δ: 0.94 (dd, 3H, *J* = 7.2, 2.4 Hz), 1.25 (t, 6H, *J* = 7.2 Hz), 1.99 (ddd, 1H, *J* = 14.0, 7.2, 2.0 Hz), 2.16 (ddd, 1H, *J* = 14.0, 7.2, 2.0 Hz), 2.25-2.65 (m, 6H), 4.19 (q, 4H, *J* = 7.2 Hz); ^{13}C-NMR ($CDCl_3$, 100 MHz) δ: 13.9, 14.5, 17.9, 35.7, 38.3, 39.3, 40.5, 58.6, 61.6, 119.1172.1, 172.2; IR (ATR) ν: 2984, 1711, 1360, 1219 cm^{-1}; HRMS (ESI) Calcd for $C_{14}H_{21}NNaO_4$, $[M+Na]^+$ 290.1368, found 290.1372; colorless oil (60.3 mg, 82%).

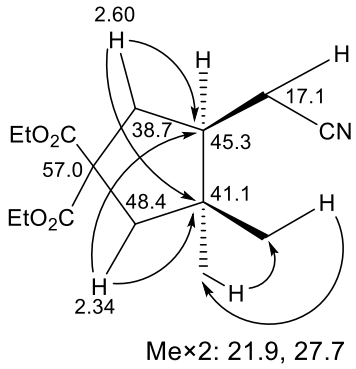
diethyl-3-(2-cyanopropan-2-yl)-4-methylcyclopentane-1,1-dicarboxylate (2d)



^{1}H-NMR ($CDCl_3$, 400 MHz) δ: 1.06 (d, 3H, *J* = 6.8 Hz), 1.24 (t, 3H, *J* = 7.6 Hz), 1.26 (t, 3H, *J* = 7.2 Hz), 1.41 (d, 6H, *J* = 4.8 Hz), 1.86 (ddd, 1H, *J* = 13.6, 6.8, 6.8 Hz), 2.24-2.41 (m, 4H), 2.49 (dd, 1H, *J* = 13.6, 6.8 Hz), 4.14-4.26 (m, 4H); ^{13}C-NMR ($CDCl_3$, 100 MHz) δ: 14.0, 15.3, 25.7, 28.6, 32.7, 34.0, 35.1, 41.4, 51.8, 57.7, 61.6, 124.2, 172.0, 172.9; IR (ATR) ν: 2980, 1728, 1253 cm^{-1};

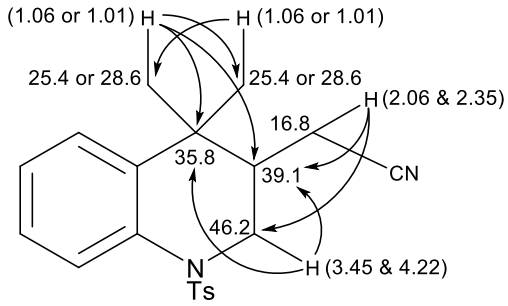
HRMS (ESI) Calcd for $C_{16}H_{25}NNaO_4$, $[M+Na]^+$ 318.1681, found 318.1684; colorless oil (22.6 mg, 62%).

diethyl-4-(cyanomethyl)-3,3-dimethylcyclopentane-1,1-dicarboxylate (2e)



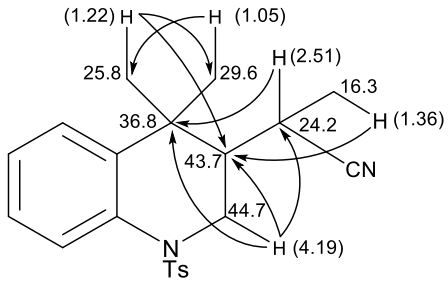
^{1}H-NMR ($CDCl_3$, 400 MHz) δ: 0.85 (d, 3H, *J* = 2.0 Hz), 1.10 (d, 3H, *J* = 2.0 Hz), 1.25 (t, 3H, *J* = 7.2 Hz), 1.26 (t, 3H, *J* = 7.2 Hz), 1.90-2.08 (m, 1H), 2.12-2.29 (m, 4H), 2.34 (ddd, 1H, *J* = 16.4, 5.6, 2.0 Hz), 2.60 (ddd, 1H, *J* = 13.6, 6.8, 2.0 Hz), 4.18 (q, 2H, *J* = 7.2 Hz), 4.19 (q, 2H, *J* = 7.2 Hz); ^{13}C-NMR ($CDCl_3$, 100 MHz) δ:13.9, 17.1, 21.9, 27.7, 38.7, 41.1, 45.3, 48.4, 57.0, 61.6, 61.7, 119.0, 172.1, 172.4; IR (ATR) ν: 2981, 1716, 1239 cm^{-1}; HRMS (ESI) Calcd for $C_{15}H_{23}NNaO_4$, $[M+Na]^+$ 304.1525, found 304.1520; colorless oil (24.4 mg, 72%).

2-(2,4,4-trimethyl-1-tosyl-1,2,3,4-tetrahydroquinolin-3-yl)acetonitrile (2f)



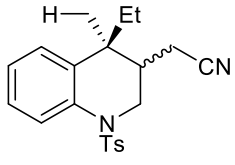
^{1}H-NMR ($CDCl_3$, 400 MHz) δ: 1.01 (s, 3H), 1.06 (s, 3H), 1.60-1.72 (m, 1H), 2.06 (dd, 1H, *J* = 16.8, 10.0 Hz), 2.35 (dd, 1H, *J* = 16.8, 4.8 Hz), 2.39 (s, 3H), 3.45 (dd, 1H, *J* = 14.0, 10.4 Hz), 4.22 (dd, 1H, *J* = 14.0, 3.6 Hz), 7.13-7.26 (m, 5H), 7.53 (d, 2H, *J* = 8.8 Hz), 7.83 (d, 1H, *J* = 8.4 Hz); ^{13}C-NMR ($CDCl_3$, 100 MHz) δ: 16.8, 21.5, 25.4, 28.6, 35.8, 39.1, 46.2, 118.3, 124.1, 125.3, 126.8, 127.1, 127.2, 129.8, 134.8, 136.2, 136.5, 144.1; IR (ATR) ν: 3000, 1709, 1357, 1219 cm^{-1}; HRMS (ESI) Calcd for $C_{20}H_{22}N_2NaO_2S$, $[M+Na]^+$ 377.1300, found 377.1297; yellow solid (31.1 mg, 71%).

2-(4,4-dimethyl-1-tosyl-1,2,3,4-tetrahydroquinolin-3-yl)propanenitrile (2g)



^{1}H-NMR ($CDCl_3$, 400 MHz) δ: 1.05 (s, 3H), 1.22 (s, 3H), 1.34 (d, 3H, *J* = 7.2 Hz), 1.60-1.66 (m, 1H), 2.38 (s, 3H), 2.51 (q, 1H, *J* = 7.2 Hz), 3.26 (dd, 1H, *J* = 14.0, 10.0 Hz), 4.19 (dd, 1H, *J* = 14.0, 3.6 Hz), 7.14-7.30 (m, 5H), 7.46 (dd, 2H, *J* = 8.0, 2.8 Hz), 7.84 (dd, 1H, *J* = 8.0, 1.6 Hz); ^{13}C-NMR ($CDCl_3$, 100 MHz) δ: 16.3, 18.9, 23.2, 24.2, 25.8, 26.0, 28.1, 29.6, 36.8, 37.0, 43.7, 43.8, 44.6, 44.7, 120.7, 122.8, 124.2, 124.7, 125.6, 125.7, 126.7, 126.7, 127.2, 127.2, 127.5, 129.6, 129.7, 135.0, 136.1, 136.8, 137.9, 138.1, 144.0, 144.1; IR (ATR) ν: 2984, 1716, 1344, 1213 cm^{-1}; HRMS (ESI) Calcd for $C_{21}H_{24}N_2NaO_2S$, $[M+Na]^+$ 391.1456, found 391.1469; yellow oil (31.7 mg, 90%).

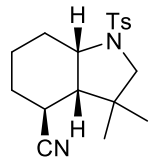
2-((4S)-4-ethyl-4-methyl-1-tosyl-1,2,3,4-tetrahydroquinolin-3-yl)acetonitrile (2h)



^{1}H-NMR ($CDCl_3$, 400 MHz) δ: 0.05 (t, 2.3H, *J* = 8.0 Hz), 0.60 (t, 3H, *J* = 7.6 Hz), 0.98 (s, 3H), 1.03 (s, 2.3H), 1.20-1.39 (m, 2+1.5H), 1.53-1.67 (m, 2+1.5H), 2.36 (s, 2.3H), 2.38 (s, 3H), 3.43 (dd, 0.8H, *J* = 14.0, 9.6 Hz), 3.65 (dd, 1H, *J* = 14.0, 9.6 Hz), 4.09 (dd, 1H, *J* = 13.6, 4.4 Hz), 4.34 (dd, 0.8H, *J* = 14.0, 3.2 Hz), 7.10-7.24 (m, 5+3.8H), 7.54 (d, 2H, *J* = 8.0 Hz), 7.59 (d, 1.5H, *J* = 8.4 Hz), 7.88 (d, 1H, *J* = 8.0 Hz), 7.94

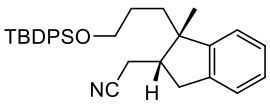
(d, 0.8H, J = 8.4 Hz); ^{13}C-NMR ($CDCl_3$, 100 MHz) δ: 7.6, 9.1, 16.4, 16.6, 21.5, 25.5, 25.8, 28.8, 31.0, 33.8, 38.3, 39.1, 39.7, 45.9, 46.9, 118.4, 123.7, 124.5, 124.7, 125.4, 126.4, 126.8, 127.0, 127.1, 127.4, 129.7, 129.9, 135.3, 136.2, 144.0; IR (ATR) ν: 2682, 1721, 1366, 1238 cm^{-1}; HRMS (ESI) Calcd for $C_{21}H_{24}N_2NaO_2S$, $[M+Na]^+$ 391.1456, found 391.1469; yellow oil (11.4 mg, 58%).

3,3-dimethyl-1-tosyloctahydro-1H-indole-4-carbonitrile (**2i**)



^{1}H-NMR ($CDCl_3$, 400 MHz) δ: 0.62 (s, 3H), 1.15 (s, 3H), 1.56-1.68 (m, 4H), 1.76 (dd, 1H, J = 9.6, 6.0 Hz), 2.05-2.14 (m, 1H), 2.44 (s, 3H), 2.41-2.48 (m, 2H), 3.06 (d, 1H, J = 10.8 Hz), 3.27 (d, 1H, J = 10.8 Hz), 3.61 (brs, 1H), 7.33 (d, 2H, J = 7.6 Hz), 7.68 (d, 2H, J = 7.6 Hz); ^{13}C-NMR ($CDCl_3$, 100 MHz) δ: 18.2, 21.5, 23.0, 26.6, 27.1, 27.9, 28.8, 39.6, 49.6, 57.8, 60.2, 122.8, 127.5, 129.7, 133.1, 143.8; IR (ATR) ν: 2938, 1337, 1159, 669 cm^{-1}; HRMS (ESI) Calcd for $C_{18}H_{24}N_2NaO_2S$, $[M+Na]^+$ 355.1456, found 355.1450; colorless solid (30.7 mg, 70%, mp: 115-118 °C).

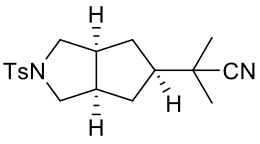
2-(1-(3-((tert-butyldiphenylsilyl)oxy)propyl)-1-methyl-2,3-dihydro-1H-inden-2-yl)acetonitrile (**2j**)



To the solution of **1j** (74.3 mg, 0.169 mmol), TsCN (36.7 mg, 0.203 mmol), Co (2.0 mg, 0.034 mmol) in EtOH (0.85 mL), $PhSiH_3$ (25 μl, 0.203 mmol) was added at rt, and the solution was stirred for 16 h at rt. Then, the solvent was removed under reduced pressure, and the residue was purified by flash column chromatography to give *cis*-**2j** (43.2 mg, 55%), and *trans*-**2j** (20.1 mg, 25%).

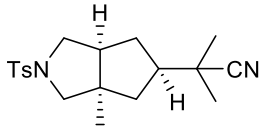
^{1}H-NMR ($CDCl_3$, 400 MHz) δ: 1.00 (s, 9H), 1.32 (s, 3H), 1.24-1.45 (m, 4H), 2.33 (dd, 1H, J = 15.6, 8.8 Hz), 2.44 (dddd, 1H, J = 9.6, 8.8, 7.2, 5.2 Hz), 2.50 (dd, 1H, J = 15.6, 5.2 Hz), 2.71 (dd, 1H, J = 15.2, 9.6 Hz), 3.11 (dd, 1H, J = 15.2, 7.2 Hz), 3.56 (dd, 2H, J = 10.4, 5.6 Hz), 7.07 (dd, 1H, J = 8.8, 3.2 Hz), 7.17-7.21 (m, 3H), 7.34-7.44 (m, 6H), 7.58 (d, 4H, J = 7.6 Hz); ^{13}C-NMR ($CDCl_3$, 100 MHz) δ: 17.2, 19.1, 24.5, 26.8, 27.5, 31.3, 36.4, 47.9, 48.0, 64.0, 119.5, 123.5, 124.6, 126.4, 126.8, 127.6, 129.6, 133.8, 135.5, 140.1, 149.2; IR (ATR) ν: 2930, 2857, 1736, 1427, 1241, 1105, 755, 700 cm^{-1}; HRMS (ESI) Calcd for $C_{31}H_{37}NnaO_{Si}$, $[M+Na]^+$ 490.2542, found 490.2529; yellow oil

2-methyl-2-2-tosyloctahydrocyclopenta[c]pyrrol-5-yl)propanenitrile (**2k**)



^{1}H-NMR ($CDCl_3$, 400 MHz) δ: 1.21-1.27 (m, 2H), 1.30 (s, 6H), 1.79-1.88 (m, 1H), 1.98-2.08 (m, 2H), 2.44 (s, 3H), 2.54-2.63 (m, 2H), 3.02 (dd, 2H, J = 9.6, 6.8 Hz), 3.09 (dd, 1H, J = 9.6, 1.6 Hz), 7.32 (d, 2H, J = 8.0 Hz), 7.69 (d, 2H, J = 8.0 Hz); ^{13}C-NMR ($CDCl_3$, 100 MHz) δ: 21.5, 25.6, 34.5, 35.0, 41.9, 50.5, 53.5, 123.9, 128.0, 129.6, 132.4, 143.6; IR (ATR) ν: 2963, 1736, 1240, 1163, 1045, 664 cm^{-1}; HRMS (ESI) Calcd for $C_{18}H_{24}N_2NaO_2S$, $[M+Na]^+$ 355.1456, found 355.1451; colorless solid (18.4 mg, 38%, mp: 155-158 °C)

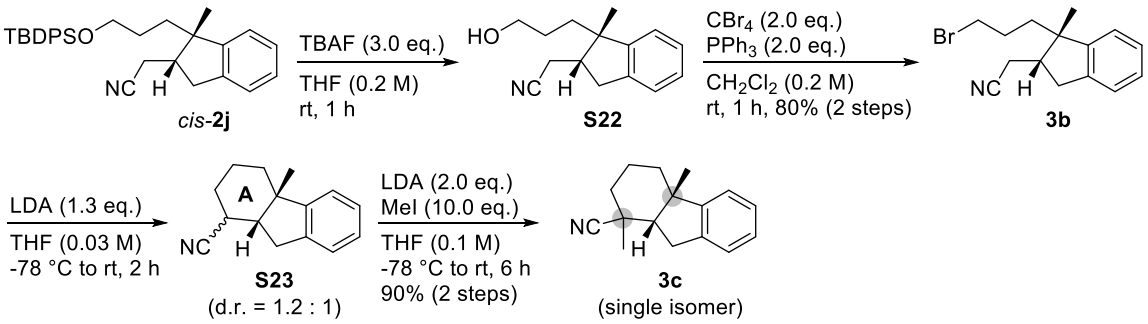
2-methyl-2-3a-methyl-2-tosyloctahydrocyclopenta[c]pyrrol-5-yl)propanenitrile (**2l**)



^{1}H-NMR ($CDCl_3$, 400 MHz) δ: 1.05 (s, 3H), 1.21-1.31 (m. 2H), 1.29 (s, 6H), 1.54 (dd, 1H, J = 12.6, 12.6 Hz), 1.67 (dd, 1H, J = 12.6, 7.2 Hz), 1.90-1.99 (m, 1H), 2.01-2.10 (m, 2H), 2.44 (s, 3H), 2.71 (d, 1H, J = 9.0 Hz), 3.10 (d, 2H, J =

4.8 Hz), 3.20 (d, 1H, *J* = 9.0 Hz), 7.33 (d, 2H, *J* = 8.4 Hz), 7.70 (d, 2H, *J* = 8.4 Hz); ^{13}C-NMR ($CDCl_3$, 100 MHz) δ:21.6, 25.5, 25.6, 26.1, 35.2, 41.2, 48.8, 49.1, 49.2, 53.4, 60.1, 124.0, 128.0, 129.6, 132.6, 143.6; IR (ATR) ν: 2969, 1736, 1344, 1158, 664 cm^{-1}; HRMS (ESI) Calcd for $C_{19}H_{26}N_2NaO_2S$, $[M+Na]^+$ 369.1613, found 369.1607; colorless solid (16.9 mg, 31%, 124-127 °C)

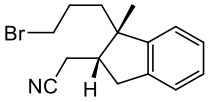
Synthesis of 3c



Cis-**2j** to **S22**: TBAF (1.23 mL, 1.0 M in THF, 1.23 mmol) was added to the solution of *cis*-**2j** (191.9 mg) in THF (2.1 mL) at 0 °C, and the solution was stirred at rt for 1 h. H_2O was added and the aqueous phase was extracted with AcOEt. The organic phase was dried over Na_2SO_4, filtered and concentrated. The compound was purified by flash (10:1 Hex:EtOAc) to give crude **S22** (79.8 mg)

S22 to **3b**: To a solution of crude **S22** (79.8 mg) in CH_2Cl_2 (1.7 mL) was added CBr_4 (23.1 mg, 0.07 mmol) and PPh_3 (18.4 mg, 0.07 mmol) at 0 °C, and the solution was stirred at rt for 1 h. The solution was directly pored to flash column chromatography to give **3b** (95.3 mg, 80%, 2 steps)

2-(1-(3-bromopropyl)-1-methyl-2,3-dihydro-1H-inden-2-yl)acetonitrile (**3b**)



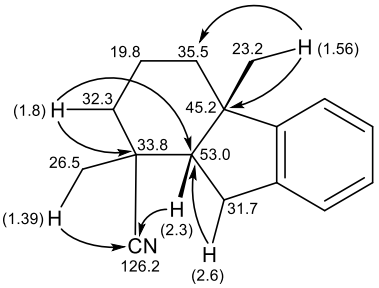
^{1}H-NMR ($CDCl_3$, 400 MHz) δ: 1.39 (s, 3H), 1.44-1.83 (m, 4H), 2.48-2.54 (m, 2H), 2.58-2.65 (m, 1H), 2.78 (dd, 1H, *J* = 16.0, 9.6 Hz), 3.14 (dd, 1H, *J* = 16.0, 7.2 Hz), 7.09-7.26 (m, 4H); ^{13}C-NMR ($CDCl_3$, 100 MHz) δ: 17.1, 24.2, 27.8, 33.7, 34.0, 36.3, 47.8, 48.0, 119.2, 123.5, 124.7, 126.5, 127.1, 140.0, 148.5; IR (ATR) ν: 2959, 1740, 1477, 1216, 909, 754, 729 cm^{-1}; HRMS (ESI) Calcd for $C_{15}H_{18}BrNNa$, $[M+Na]^+$ 314.0520, found 314.0520; yellow oil

3b to **S23**: Preparation of LDA: *n*-BuLi (0.92 mL, 1.43 mmol) was added to a solution of *i*Pr_2NH (0.2 mL, 1.43 mmol) in THF (6.02 mL) at -78 °C, and the solution was stirred at 0 °C for 1 h. Resulting LDA was added to the solution of **3b** (26.4 mg, 0.09 mmol) in THF (3 mL) at -78 °C, and the solution was slowly warmed to rt, and stirred for 2 h. Then, sat. NH_4Cl aq. was added and the aqueous phase was extracted with AcOEt. The organic phase was dried over Na_2SO_4, filtered and concentrated. The compound was purified by flash (20:1 Hex:EtOAc) to give crude **S23** (16.9 mg)

S23 to **3c**: LDA (prepared by the same scale for the synthesis of **S23**) was added to the solution of crude **S23** (16.9 mg) in THF (0.82 mL) at -78 °C, and stirred for 1h at the same temperature. Then, MeI (50 μL, 0.803 mmol) was added to the solution at -78 °C, and the solution was slowly warmed to rt, stirred for 6 h. Then, sat. NH_4Cl aq. was added and the aqueous phase was extracted with AcOEt. The organic phase was

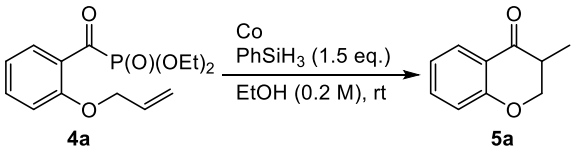
dried over Na_2SO_4, filtered and concentrated. The compound was purified by flash (20:1 Hex:EtOAc) to give **3c** (15.2 mg) as a single diastereomer.

1,4a-dimethyl-2,3,4,4a,9,9a-hexahydro-1H-fluorene-1-carbonitrile (3c)

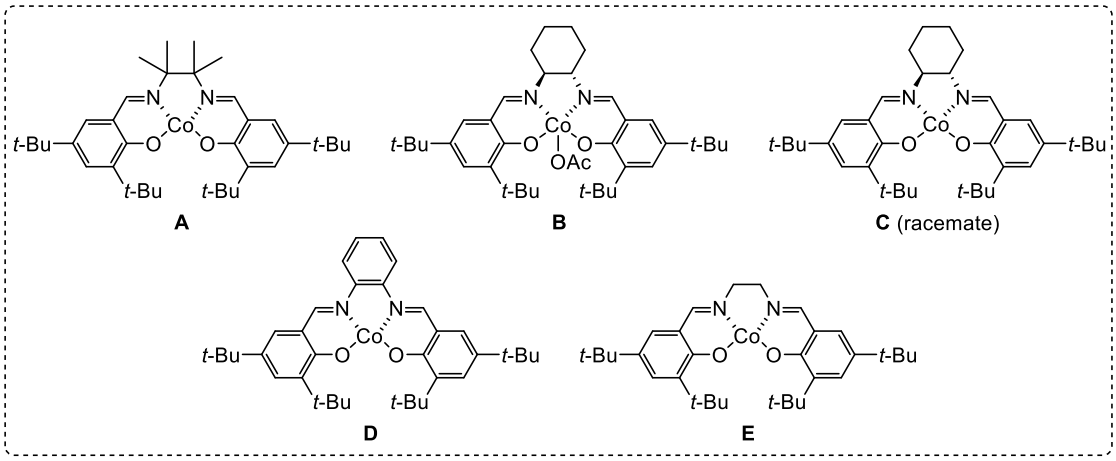


^{1}H-NMR ($CDCl_3$, 600 MHz) δ: 1.10-1.29 (m, 3H), 1.39 (s, 3H), 1.43-1.62 (m, 2H), 1.56 (s, 3H), 1.77-1.84 (m, 1H), 2.33-2.36 (m, 1H), 2.64-2.69 (m, 1H), 2.72-2.77 (m, 1H), 7.02-7.19 (m, 4H) ; ^{13}C-NMR ($CDCl_3$, 150 MHz) δ: 19.8, 23.2, 26.5, 31.7, 32.3, 33.8, 35.5, 45.2, 53.0, 121.5, 124.7, 126.2, 126.5, 126.8, 138.6, 153.0; IR (ATR) ν: 2980, 1755, 1234 cm^{-1}; HRMS (ESI) Calcd for $C_{16}H_{19}NNa$, $[M+Na]^+$ 248.1452, found 248.1414; yellow oil

Optimization of Catalyst

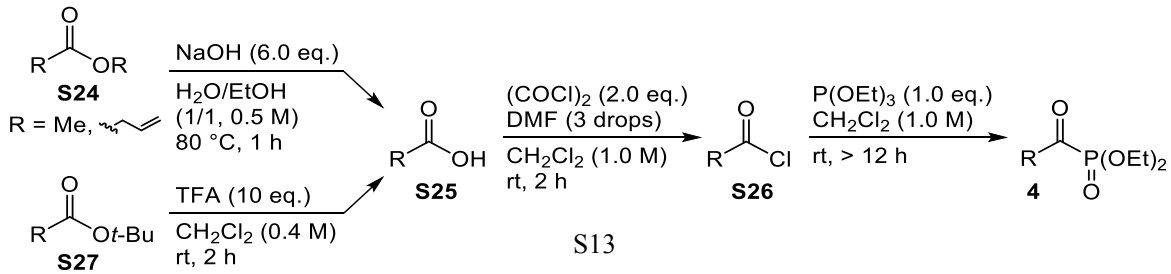


entry	Co (mol%)	time (h)	**5a** (%)	% ee
1	**A** (5)	4	65	-
2	**B** (racemate, 5)	24	24	-
3	**C** (5)	24	trace	-
4	**D** (5)	24	trace	-
5	**E** (5)	9	trace	-
6	**B** (chiral, 5)	24	20	0
7	**A** (2)	9	36	-



General procedure for hydroacylation: EtOH (0.2 M for alkenylacylphosphonate) was added to the mixture of alkenylacylphosphonate **4** (1.0 eq.), Co **A** (5 mol%) under an argon atmosphere at rt, then $PhSiH_3$ (1.5 eq.) was added at rt and the solution was stirred at rt. Then, the solvent was removed under reduced pressure, and the residue was purified by flash column chromatography.

General procedure for the synthesis of acylphosphonate 4



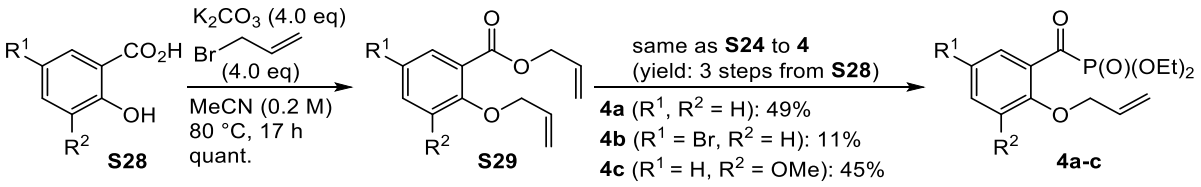
S24 to **S25**: To a solution of **S24** (1.0 eq.) in EtOH (1.0 M for **S24**) was added H_2O (1.0 M for **S24**) and NaOH (6.0 eq.) at rt, and the reaction mixture was stirred at 80 °C for 1 h. The reaction was quenched with 1 N HCl (pH = 1), extracted with CH_2Cl_2, dried over Na_2SO_4, filtered, and concentrated in vacuo. ***Purification by flash column chromatography*** afforded **S25**. (***Caution!!***: Purification of **S25** is necessary even if crude **S25** is seems to be pure.)

S27 to **S25**: To a solution of **S27** (1.0 eq.) in CH_2Cl_2 (0.4 M for **S27**) was added TFA (10 eq.) at 0 °C, and the reaction mixture was stirred at rt for 2 h. The reaction mixture was evaporated under reduced pressure and diluted with CH_2Cl_2 and 1 N HCl. The organic layer was separated and the mixture was further extracted with CH_2Cl_2. The combined organic extract was then dried over anhydrous sodium sulfate, concentrated under reduced pressure. ***Purification by flash column chromatography*** afforded **S25**. (***Caution!!***: Purification of **S25** is necessary even if crude **S25** is seems to be pure.)

S25 to **S26**: To a solution of **S25** (1.0 eq.) in CH_2Cl_2 (1.0 M for **S25**), $(COCl)_2$ (2.0 eq.) followed by DMF (3 drops) was added at 0 °C, and the reaction mixture was stirred at 0 °C for 15 min. and then, the reaction mixture was allowed to warm to rt. After 2 h, the reaction mixture was evaporated under reduced pressure gave crude **S26**, and ***the resulting volatiles were removed under reduced pressure (at 50 °C, 1 h)*** to give **S26** which was used in the next step without further purification. (***Caution!!***: Careful removal of volatiles under heating condition is necessary even if crude **S26** seems to be pure.)

S26 to **4**: $P(OEt)_3$ (***1.0 eq.***) was added to the solution of **S26** (1.0 eq.) in CH_2Cl_2 (1.0 M) at 0 °C, and stirred for 1 h at the same temperature. Then, the reaction mixture was allowed to warm to rt, and ***stirred for overnight***. Any volatiles were removed under reduced pressure and the crude **4** was purified by column chromatography. (***Caution!!***: Excess addition of triehylphosphite gave complex mixture. // Even if TLC indicates the generation of **4** only in a few hours, ***stirring for overnight is necessary***.)

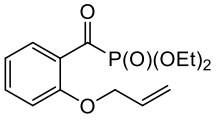
Synthesis of 4a-c



S28 to **S29**: To a stirred solution of **S28** (1.0 eq.) in MeCN (0.2 M for **S28**) was added K_2CO_3 (4.0 eq.), and allyl bromide (4.0 eq.), and the reaction mixture was stirred at 80 °C for 17 h. The reaction was quenched with H_2O, extracted with AcOEt, dried over Na_2SO_4, filtered, and concentrated in vacuo. Purification by flash column chromatography afforded **S29** (quant.).

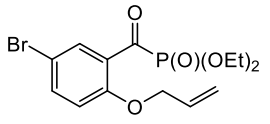
S29 to **4a-c**: Same as general procedure (**S24** to **4**)

diethyl (2-(allyloxy)benzoyl)phosphonate (**4a**)



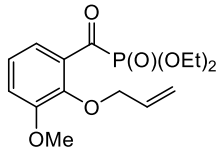
(CAS-Reg# 1454663-48-9); ^{1}H-NMR ($CDCl_3$, 400 MHz) δ: 1.35 (t, 6H, J = 7.1 Hz), 4.23 (m, 4H), 4.69 (d, 2H, J = 5.1 Hz), 5.38 (m, 2H), 6.13 (m, 1H), 7.02 (m, 2H), 7.50 (m, 1H), 7.92 (dd, 1H, J = 7.8 Hz, J = 1.4 Hz); (2.11 g, 49%)

diethyl (2-(allyloxy)-5-bromobenzoyl)phosphonate (**4b**)



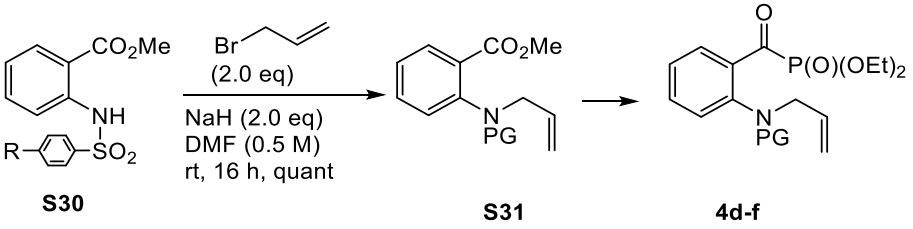
^{1}H-NMR ($CDCl_3$, 400 MHz) δ: 1.36 (t, 6H, J = 7.2 Hz), 4.24 (q, 4H, J = 7.2 Hz), 4.67 (d, 2H, J = 5.2 Hz), 5.31 (d, 1H, J = 9.6 Hz), 5.43 (d, 1H, J = 17.2 Hz), 6.06-6.16 (m, 1H), 6.87 (d, 1H, J = 8.8 Hz), 7.57 (dd, 1H, J = 8.8, 2.8 Hz), 7.90 (d, 1H, J = 2.8 Hz); ^{13}C-NMR ($CDCl_3$, 100 MHz) δ: 16.2, 16.2, 63.7, 63.8, 70.1, 112.9, 115.1, 118.1, 128.2, 128.9, 132.1, 132.9, 137.3, 157.3, 198.7, 200.5; IR (ATR) ν: 2984, 1727, 1482, 1227, 1012 cm^{-1}; HRMS (ESI) Calcd for $C_{14}H_{18}BrNaO_5P$, $[M+Na]^+$ 398.9973, found 398.9963; yellow oil (195 mg, 11%).

diethyl (2-(allyloxy)-3-methoxybenzoyl)phosphonate (**4c**)



^{1}H-NMR ($CDCl_3$, 400 MHz) δ:1.34 (t, 6H, J = 7.2 Hz), 3.89 (s, 3H), 4.25 (q, 4H, J = 7.2 Hz), 4.63 (d, 2H, J = 5.6 Hz), 5.20 (d, 1H, J = 10.0 Hz), 5.31 (dd, 1H, J = 17.2, 1.2 Hz), 6.09-6.21 (m, 1H), 7.10-7.17 (m, 2H), 7.48 (dd, 1H, J = 7.2, 2.4 Hz); ^{13}C-NMR ($CDCl_3$, 100 MHz) δ: 16.2, 16.2, 55.9, 63.7, 63.8, 75.0, 117.0, 118.0, 121.4, 123.8, 131.9, 132.5, 133.8, 147.2, 152.9, 152.9, 200.2, 202.0; IR (ATR) ν: 2982, 1724, 1474, 1016 cm^{-1}; HRMS (ESI) Calcd for $C_{15}H_{21}NaO_6P$, $[M+Na]^+$ 351.0973, found 351.0963; yellow oil (492 mg, 45%).

Synthesis of 4d-f



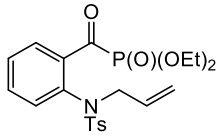
R	**4** (%)*
Me	**4d** (88)
MeO	**4e** (42)
F	**4f** (40)

*3 steps from **S31**

S30 to **S31**: To a solution of **S30** (1.0 eq.) in DMF (0.5 M for **S30**) was added 60% sodium hydride (2.0 eq.) at 0 °C and the mixture was stirred at room temperature for 30 minutes. Allyl bromide (2.0 eq.) was then added and the mixture was stirred for 16 h. Water was added and the mixture was diluted with ethyl acetate. The layers were separated and the organic layer was washed with water and brine, dried over anhydrous Na_2SO_4 and concentrated under reduced pressure. The crude product was purified by silica gel column chromatography to afford the corresponding **S31** (quant.).

S31 to **4d-f**: Same as general procedure (**S24** to **4**)

diethyl (2-((N-allyl-4-methylphenyl)sulfonamido)benzoyl)phosphonate (**4d**)

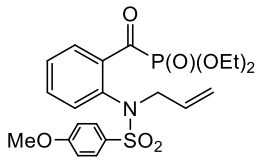


(CAS-Reg# 1454663-49-0)

^{1}H-NMR ($CDCl_3$, 400 MHz) δ: 1.36 (m, 6H), 2.42 (s, 3H), 4.27 (m, 6H), 5.02 (m, 2H), 5.94 (m, 1H), 6.86 (m, 1H), 7.23 (d, 2H, J = 8.4 Hz), 7.44 (m, 4H), 8.27 (m,

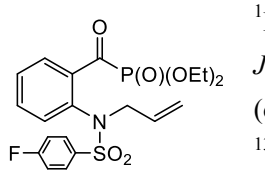
1H); (354.2 mg, 88%)

diethyl (2-((N-allyl-4-methoxyphenyl)sulfonamido)benzoyl)phosphonate (4e)



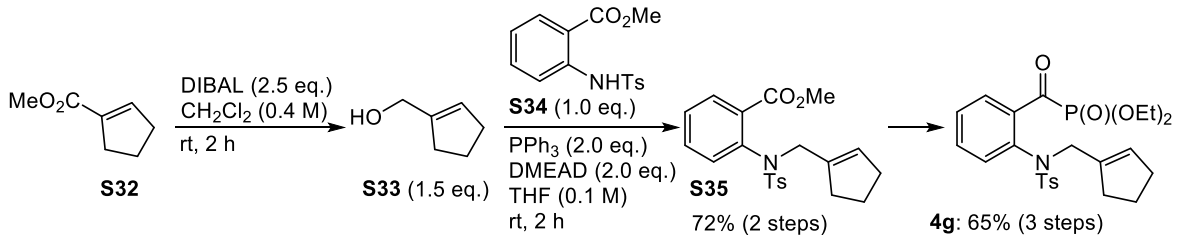
^{1}H-NMR ($CDCl_3$, 400 MHz) δ: 1.37 (t, 6H, *J* = 6.8 Hz), 3.86 (s, 3H), 4.21-4.32 (m, 6H), 5.02 (d, 1H, *J* = 16.0 Hz), 5.03 (d, 1H, *J* = 10.8 Hz), 5.90-6.00 (m, 1H), 6.88-6.91 (m, 3H), 7.41-7.47 (m, 2H), 7.51 (d, 2H, *J* = 8.8 Hz), 8.24-8.27 (m, 1H); ^{13}C-NMR ($CDCl_3$, 100 MHz) δ: 16.0, 16.1, 54.3, 55.3, 63.9, 113.7, 118.8, 127.8, 129.5, 130.0, 130.2, 131.0, 132.5, 132.8, 136.8, 136.9, 137.0, 137.6, 162.7, 199.2, 201.0; IR (ATR) ν: 2969, 1733, 1594, 1158, 1019 cm^{-1}; HRMS (ESI) Calcd for $C_{21}H_{26}NNaO_7PS$, $[M+Na]^+$ 490.1065, found 490.1053; yellow oil (610.6 mg, 42%).

diethyl (2-((N-allyl-4-fluorophenyl)sulfonamido)benzoyl)phosphonate (4f)



^{1}H-NMR ($CDCl_3$, 400 MHz) δ: 1.37 (t, 6H, *J* = 6.8 Hz), 4.26 (brs, 6H), 5.03 (d, 1H, *J* = 18.4 Hz), 5.05 (d, 1H, *J* = 9.2 Hz), 5.88-5.98 (m, 1H), 6.94-6.96 (m, 1H), 7.12 (dd, 2H, *J* = 8.4, 8.4 Hz), 7.45-7.50 (m, 2H), 7.60-7.63 (m, 2H), 8.30-8.32 (m, 1H); ^{13}C-NMR ($CDCl_3$, 100 MHz) δ: 16.0, 16.1, 54.5, 64.0, 115.7, 115.9, 119.1, 128.2, 130.0, 130.1, 130.8, 131.5, 132.5, 132.8, 134.8, 134.8, 136.4, 136.4, 137.0, 163.5, 166.1, 198.8, 200.6; IR (ATR) ν: 2984, 1719, 1492, 1154, 1011, 839 cm^{-1}; HRMS (ESI) Calcd for $C_{20}H_{23}FNNaO_6PS$, $[M+Na]^+$ 478.0865, found 478.0861; yellow oil (588 mg, 40%).

Synthesis of 4g

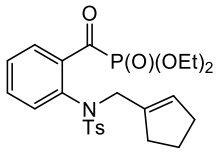


S32 to **S33**: To a stirred solution of **S32** (1.0 eq.) in CH_2Cl_2 (0.4 M for **S32**) at 0 °C was added DIBAL (2.5 eq., 1.0 M in toluene) dropwise. The mixture was stirred at 0 °C to rt for 2 h then treated with ice cold Rochelles salt (saturated Na/K tartrate) and allowed to stir at 0 °C open to air. The reaction became a gray-white gelatinous suspension that solubilized over the course of 30 min to 1 h with stirring at room temperature open to air. The mixture was poured into diethyl ether and H_2O. The layers were separated, and the aqueous layer was extracted twice more with diethyl ether. The organic layers were combined, dried over Na_2SO_4, and concentrated to afford a crude **S33** which was used without further purification.

S33 to **S35**: To a stirred solution of **S33** (1.5 eq.), **S34** (1.0 eq.), PPh_3 (2.0 eq.) in THF (0.1 M for **S34**) was added DMEAD (2.0 eq.) at 0 °C, and the reaction mixture was stirred for 2 h at rt. Then, the reaction mixture was concentrated in vacuo. Purification by flash column chromatography gave **S35**.

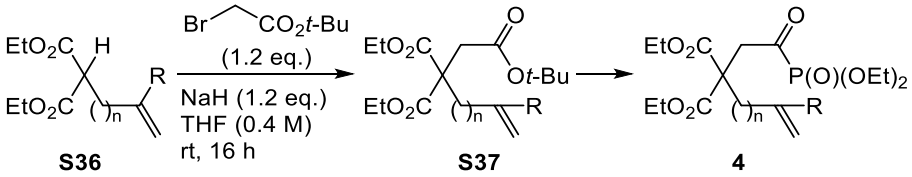
S35 to **4g**: Same as general procedure (**S24** to **4**)

diethyl (2-((N-(cyclopent-1-en-1-ylmethyl)-4-methylphenyl)sulfonamido)benzoyl)phosphonate (4g)



^{1}H-NMR ($CDCl_3$, 400 MHz) δ:1.36 (t, 6H, *J* = 7.2 Hz), 1.75 (dt, 2H, *J* = 15.2, 7.2 Hz), 2.11-2.24 (m, 4H), 2.41 (s, 3H), 4.23-4.31 (m, 6H), 5.42 (s, 1H), 6.95-6.98 (m, 1H), 7.21 (d, 2H, *J* = 8.0 Hz), 7.42-7.48 (m, 4H), 8.36 (m, 1H); ^{13}C-NMR ($CDCl_3$, 100 MHz) δ: 16.3, 21.5, 23.4, 32.2, 33.6, 51.5, 64.2, 127.6, 127.7, 129.2, 130.4, 131.7, 132.7, 135.9, 136.5, 136.9, 138.8, 143.2, 198.3, 200.2; IR (ATR) ν: 2982, 1670, 1159, 1018, 750 cm^{-1}; HRMS (ESI) Calcd for $C_{24}H_{30}NNaO_6PS$, $[M+Na]^+$ 514.1429, found 514.1430; yellow oil (301.5 mg, 65%).

Synthesis of 4h,i,l



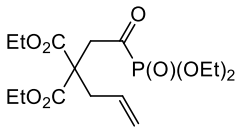
n	R	**4** (%)*
1	H	**4h** (84)
1	Me	**4i** (25)
2	H	**4l** (51)

*4 steps from **S36**

S36 to **S37**: To a solution of **S36** (1.0 eq.) in THF (0.4 M for **S36**) was added 60% sodium hydride (1.2 eq.) at 0 °C and the mixture was stirred at room temperature for 30 minutes. *tert*-Butyl Bromoacetate (1.2 eq.) was then added and the mixture was stirred for 16 h. Water was added and the mixture was diluted with ethyl acetate. The layers were separated and the organic layer was washed with water and brine, dried over anhydrous Na_2SO_4 and concentrated under reduced pressure. The crude product was purified by silica gel column chromatography to afford the corresponding **S37**.

S37 to **4**: Same as general procedure (**S24** to **4**)

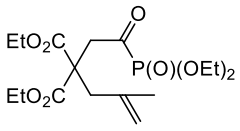
diethyl 2-allyl-2-(2-(diethoxyphosphoryl)-2-oxoethyl)malonate (4h)



(CAS-Reg# 593254-57-0)

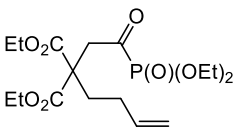
^{1}H-NMR ($CDCl_3$, 400 MHz) δ: 1.25 (t, 6H, *J* = 7.2 Hz), 1.37 (t, 6H, *J* = 7.2 Hz), 2.76 (d, 2H, *J* = 7.6 Hz), 3.50 (d, 2H, *J* = 2.8 Hz), 4.21 (m, 8H), 5.10 (m, 2H), 5.66 (m, 1H); (1.60 g, 84%)

diethyl 2-(2-((diethylperoxy)(oxo)-l4-phosphanyl)-2-oxoethyl)-2-(2-methylallyl)malonate (4i)



^{1}H-NMR ($CDCl_3$, 400 MHz) δ: 1.25 (t, 6H, *J* = 7.2 Hz), 1.37 (t, 6H, *J* = 1.62 (s, 2.4H), 1.65 (s, 0.6H, s 0.6H), 2.84 (s, 1.6H), 2.88 (s, 0.4H), 3.05 (s, 0.6H), 3.55 (s, 1.6H), 4.14-4.24 (m, 8H), 4.65 (s, 0.8H), 4.74 (s, 0.2H), 4.87 (s, 0.8H), 4.90 (s, 0.2H); ^{13}C-NMR ($CDCl_3$, 100 MHz) δ: 13.7, 16.1, 16.2, 22.7, 40.5, 44.8, 45.4, 54.1, 54.2, 61.7, 63.8, 63.9, 116.4, 140.3, 169.8, 170.2, 207.1, 208.8; IR (ATR) ν: 2982, 1731, 1183, 1019 cm^{-1}; HRMS (ESI) Calcd for $C_{17}H_{29}NaO_8P$, $[M+Na]^+$ 415.1498, found 415.1493; yellow oil (357 mg, 25%).

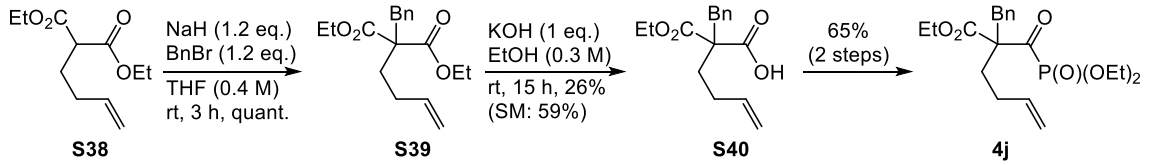
diethyl 2-(but-3-en-1-yl)-2-(2-(diethoxyphosphoryl)-2-oxoethyl)malonate (4l)



^{1}H-NMR ($CDCl_3$, 400 MHz) δ: 1.25 (t, 6H, *J* = 6.8 Hz), 1.38 (t, 6H, *J* = 7.2 Hz), 1.92-2.15 (m, 4H), 3.52 (s, 2H), 4.14-4.27 (m, 8H), 4.96 (d, 1H, *J* = 10.0 Hz), 5.00 (d, 1H, *J* = 17.2 Hz), 5.68-5.81 (m, 1H); ^{13}C-NMR ($CDCl_3$, 100 MHz) δ: 13.4, 15.8,

28.5, 32.1, 44.8, 45.4, 54.3, 61.2, 63.5, 114.9, 136.5, 169.4, 206.5, 208.2; IR (ATR) ν: 2983, 1731, 1184, 1012 cm^{-1}; HRMS (ESI) Calcd for $C_{17}H_{29}NaO_8P$, $[M+Na]^+$ 415.1498, found 415.1508; colorless oil (794.5 mg, 51%).

Synthesis of 4j

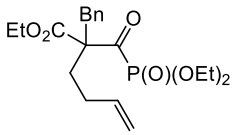


S38 to **S39**: To a solution of **S38** (549 mg, 2.56 mmol) in THF (6.4 mL) was added 60% sodium hydride (123 mg, 3.07 mmol) at 0 °C and the mixture was stirred at room temperature for 30 minutes. Benzyl bromide (0.36 mL, 3.07 mmol) was then added and the mixture was stirred for 3 h. Water was added and the mixture was diluted with ethyl acetate. The layers were separated and the organic layer was washed with water and brine, dried over anhydrous Na_2SO_4 and concentrated under reduced pressure. The crude product was purified by silica gel column chromatography to afford the corresponding **S39** (779 mg, quant.).

S39 to **S40**: To a stirred solution of **S39** (779 mg, 2.56 mmol) in EtOH (8.5 mL) at rt, KOH (143 mg, 2.56 mmol) was added. After 15 h, the mixture was acidified with 1 N HCl and filtered. The filter cake (KCl) was washed with EtOH. The combined filtrate was concentrated and residual liquid was subjected to column chromatography to afford **S40** (186 mg, 26%) and unreacted **S39** (481 mg, 59%).

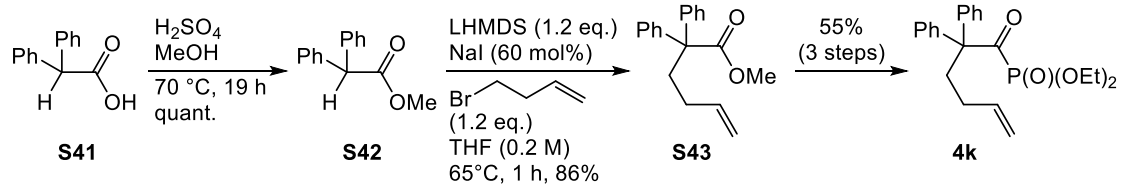
S40 to **4j**: Same as general procedure (**S25** to **4**).

ethyl 2-benzyl-2-(((diethylperoxy)(oxo)-l4-phosphanyl)carbonyl)hex-5-enoate (4j)



^{1}H-NMR ($CDCl_3$, 400 MHz) δ: 1.19 (t, 3H, *J* = 7.6 Hz), 1.36 (t, 6H, *J* = 7.2 Hz), 1.90-2.05 (m, 4H), 3.22 (d, 1H, *J* = 14.0 Hz), 3.29 (d, 1H, *J* = 14.0 Hz), 4.10-4.18 (m, 2H), 4.22-4.28 (m, 4H), 4.96 (d, 1H, *J* = 10.0 Hz), 5.01 (d, 1H, *J* = 17.2 Hz), 5.69-5.83 (m, 1H), 7.08 (d, 2H, *J* = 8.0 Hz), 7.20-7.25 (m, 3H); ^{13}C-NMR ($CDCl_3$, 100 MHz) δ: 13.6, 16.1, 16.1, 27.7, 29.2, 36.2, 61.4, 63.8, 63.8, 63.9, 64.1, 64.6, 115.1, 126.8, 128.0, 129.9, 135.2, 136.8, 169.9, 207.1, 208.8; IR (ATR) ν: 2981, 1731, 1197, 1011, 700 cm^{-1}; HRMS (ESI) Calcd for $C_{20}H_{29}NaO_6P$, $[M+Na]^+$ 419.1599, found 419.1604; yellow oil (165 mg, 55%).

Synthesis of 4k



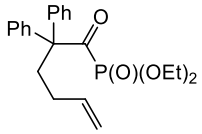
S41 to **S42**: To a solution of **S41** (2.0 g) in MeOH (100 mL) was added H_2SO_4 (1.0 mL) at rt and the mixture was stirred at 70 °C for 19 h, then MeOH was removed under reduced pressure. sat. $NaHCO_3$ aq. was added and the mixture was diluted with CH_2Cl_2. The layers were separated and the organic layer was

washed with water and brine, dried over anhydrous Na_2SO_4 and concentrated under reduced pressure. The crude **S42** was used in the next step without further purification.

S42 to **S43**: To a solution of crude **S42** (500 mg, 2.21 mmol) in THF (11 mL) was added LHMDS (2.7 mL, 1.0 M in THF, 2.7 mmol) at -78 °C and the mixture was stirred at 0 °C for 1 h. 4-Bromo-1-butene (0.27 mL, 2.65 mmol) and NaI (199 mg, 1.33 mmol) was then added at -78 °C and the mixture was stirred for 1 h at 65 °C. Water was added and the mixture was diluted with ethyl acetate. The layers were separated and the organic layer was washed with water and brine, dried over anhydrous Na_2SO_4 and concentrated under reduced pressure. The crude product was purified by silica gel column chromatography to afford the corresponding **S43** (528 mg, 86%).

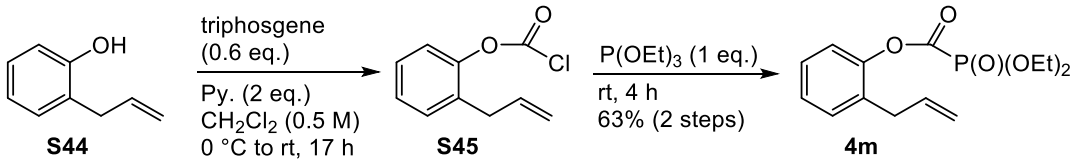
S43 to **4k**: Same as general procedure (**S24** to **4**).

1-((diethylperoxy)(oxo)-l4-phosphanyl)-2,2-diphenylhex-5-en-1-one (**4k**)



^{1}H-NMR ($CDCl_3$, 400 MHz) δ: 1.14 (t, 6H, *J* = 7.2 Hz), 1.76-1.83 (m, 2H), 2.65 (dt, 2H, *J* = 8.0, 4.0 Hz), 3.71-3.92 (m, 4H), 5.73-5.83 (m, 1H), 7.27-7.37 (m, 10H); ^{13}C-NMR ($CDCl_3$, 100 MHz) δ: 16.0, 16.0, 29.2, 29.6, 34.9, 37.1, 63.5, 63.6, 66.6, 67.2, 114.4, 114.8, 126.7, 127.4, 127.7, 128.2, 128.9, 129.8, 137.7, 138.3, 142.7, 207.6, 209.2; IR (ATR) ν: 2981, 1736, 1237, 1016, 700 cm^{-1}; HRMS (ESI) Calcd for $C_{22}H_{27}NaO_4P$, $[M+Na]^+$ 409.1545, found 409.1532; yellow oil (399.8 mg, 55%).

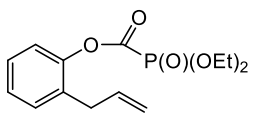
Synthesis of 4m



S44 to **S45**: To a solution of **S44** (1.0 mL, 7.6 mmol) and triphosgene (1.35 g, 4.56 mmol) in CH_2Cl_2 (7 mL), pyridine (1.2 mL, 15.2 mmol) was added using dropping funnel over 1 h at 0 °C. The temperature was allowed to rise to room temperature gradually after the addition of pyridine and the solutions was stirred for 17 h at room temperature (color of the solution: clear yellow to clear dark orange). The reaction was quenched with 1N HCl, extracted with CH_2Cl_2, dried over Na_2SO_4, filtered, and concentrated in vacuo gave crude **S45** as a deep red oil which was used without further purification.

S45 to **4m**: $P(OEt)_3$ (0.18 mL, 1.07 mmol) was directly added to the crude **S45** (210 mg, 1.07 mmol) at 0 °C, and the mixture was stirred at rt for 4 h. Any volatiles were removed under reduced pressure and the crude **4m** was purified by column chromatography (201 mg, 63%, 2 steps).

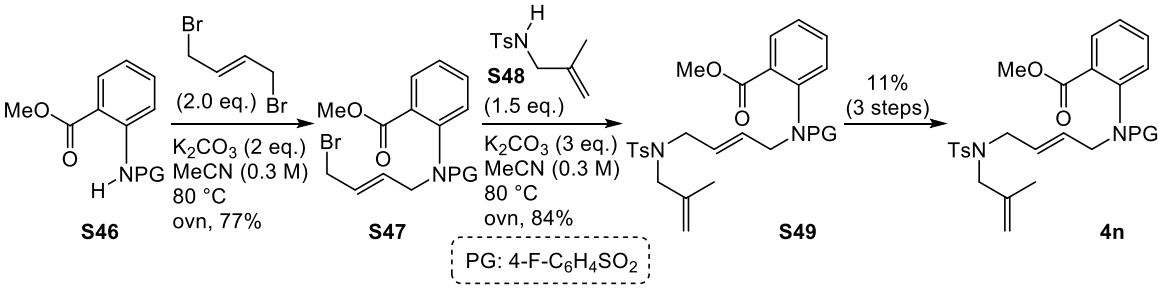
2-allylphenyl (diethoxyphosphoryl)formate (**4m**)



^{1}H-NMR ($CDCl_3$, 400 MHz) δ:1.45 (t, 6H, *J* = 7.2 Hz), 3.31 (d, 2H, *J* = 6.4 Hz), 4.40 (q, 4H, *J* = 7.2 Hz), 5.05 (d, 1H, *J* = 17.2 Hz), 5.10 (d, 1H, *J* = 10.8 Hz), 5.84-5.94 (m, 1H), 7.08 (d, 1H, *J* = 8.0 Hz), 7.21-7.29 (m, 3H); ^{13}C-NMR

($CDCl_3$, 100 MHz) δ:16.3, 16.3, 34.3, 64.8, 64.9, 116.7, 121.9, 126.9, 127.5, 130.6, 131.6, 135.3, 147.9, 164.1; IR (ATR) ν: 2982, 1721, 1011, 772 cm^{-1}; HRMS (ESI) Calcd for $C_{14}H_{19}NaO_5P$, $[M+Na]^+$ 321.0868, found 321.0857; yellow oil

Synthesis of 4n

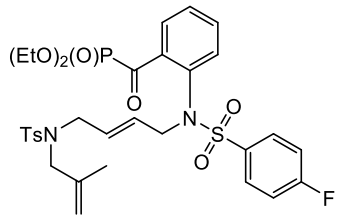


S46 to **S47**: To a stirred solution of **S46** (1.0 g, 3.23 mmol) in MeCN (11 mL) was added K_2CO_3 (891 mg, 6.46 mmol), and *trans*-1,4-dibromo-2-butene (1.38 g, 6.46 mmol), and the reaction mixture was stirred at 80 °C. The reaction was quenched with H_2O, extracted with AcOEt, dried over Na_2SO_4, filtered, and concentrated in vacuo. Purification by flash column chromatography afforded **S47** (1.1 g, 77%).

S47 to **S49**: To a stirred solution of **S47** (442.3 mg, 1.0 mmol) in MeCN (3.3 mL) was added K_2CO_3 (414 mg, 3.0 mmol), and **S48** (338 mg, 1.5 mmol), and the reaction mixture was stirred at 80 °C. The reaction was quenched with H_2O, extracted with AcOEt, dried over Na_2SO_4, filtered, and concentrated in vacuo. Purification by flash column chromatography afforded **S49** (491 mg, 84%).

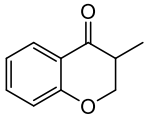
S49 to **4n**: Same as general procedure (**S24** to **4**)

diethyl(E)-(2-((4-fluoro-N-(4-((4-methyl-N-(2-methylallyl)phenyl)sulfonamido)but-2-en-1-yl)phenyl)sulfonamido)benzoyl)phosphonate (**4n**)



^{1}H-NMR ($CDCl_3$, 400 MHz) δ: 1.31-1.43 (m, 6H), 1.52 (s, 3H), 2.42 (s, 3H), 3.31-3.69 (br, 4H), 4.15 (q, 4H, *J* = 6.8 Hz), 4.26 (brs, 2H), 4.63 (s, 1H), 4.79 (s, 1H), 5.30 (dt, 1H, *J* = 15.2, 6.8 Hz), 5.68 (d, 1H, *J* = 15.2, 6.8 Hz), 6.84 (d, 1H, *J* = 6.8 Hz), 7.09 (d, 1H, *J* = 8.4 Hz), 7.14 (d, 1H, *J* = 8.8 Hz), 7.21-7.39 (m, 2H), 7.44-7.49 (m, 2H), 7.54 (d, 1H, *J* = 8.8 Hz), 7.58 (d, 1H, *J* = 8.8 Hz), 7.61 (d, 2H, *J* = 8.0 Hz), 8.26 (dd, 1H, *J* = 6.8, 2.8 Hz); ^{13}C-NMR ($CDCl_3$, 100 MHz) δ: 16.2, 16.3, 19.7, 21.4, 47.9, 52.7, 53.3, 114.4, 115.9, 116.2, 127.1, 128.3, 129.2, 129.5, 129.5, 130.2, 130.3, 130.7, 131.6, 133.0, 134.5, 136.5, 136.5, 136.7, 136.9, 137.3, 139.8, 143.2,163.8, 166.3, 199.2, 201.0; IR (ATR) ν: 2809, 1348, 1158, 1022 cm^{-1}; HRMS (ESI) Calcd for $C_{32}H_{38}FN_2NaO_8PS$, $[M+Na]^+$ 715.1689, found 715.1685; yellow oil (100.2 mg, 11%).

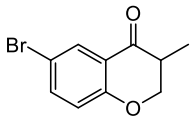
3-methylchroman-4-one (**5a**)



(CAS-Reg# 16982-86-8)

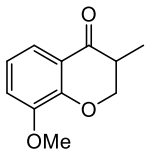
^{1}H-NMR ($CDCl_3$, 400 MHz) δ: 1.19 (d, 3H, *J* = 6.8 Hz), 2.78-2.91 (m, 1H), 4.13 (dd, 1H, *J* = 11.2, 11.2 Hz), 4.46 (dd, 1H, *J* = 11.2, 5.2 Hz), 6.93 (d, 1H, *J* = 8.4 Hz), 6.99 (dd, 1H, *J* = 7.6, 7.2 Hz), 7.43 (dd, 1H, *J* = 8.4, 7.2 Hz), 7.89 (d, 1H, *J* = 7.6 Hz); (28.5 mg, 65%)

6-bromo-3-methylchroman-4-one (5b)



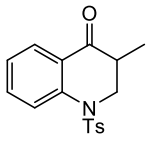
^{1}H-NMR ($CDCl_3$, 400 MHz) δ: 1.21 (d, 3H, *J* = 7.2 Hz), 2.82-2.91 (m, 1H), 4.15 (dd, 1H, *J* = 11.2, 11.2 Hz), 4.50 (dd, 1H, *J* = 11.2, 5.2 Hz), 6.87 (d, 1H, *J* = 8.4 Hz), 7.53 (dd, 1H, *J* = 8.4, 2.8 Hz), 8.00 (d, 1H, *J* = 2.8 Hz); ^{13}C-NMR ($CDCl_3$, 100 MHz) δ: 10.6, 40.4, 72.2, 114.0, 119.8, 121.7, 129.7, 138.3, 160.6, 193.5; IR (ATR) ν: 2991, 1738, 1239, 822 cm^{-1}; HRMS (ESI) Calcd for $C_{10}H_9BrNaO_2$, $[M+Na]^+$ 262.9684, found 262.9681; colorless solid (26.4 mg, 59%).

8-methoxy-3-methylchroman-4-one (5c)



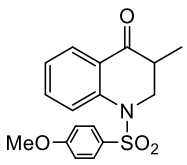
^{1}H-NMR ($CDCl_3$, 400 MHz) δ: 1.23 (d, 3H, *J* = 7.2 Hz), 2.84-2.93 (m, 1H), 3.92 (s, 3H), 4.23 (dd, 1H, *J* = 10.8, 10.8 Hz), 4.61 (dd, 1H, *J* = 10.8, 4.8 Hz), 6.96 (dd, 1H, *J* = 7.6, 7.6 Hz), 7.04 (d, 1H, *J* = 7.6 Hz), 7.49 (d, 1H, *J* = 7.6 Hz); ^{13}C-NMR ($CDCl_3$, 100 MHz) δ: 10.7, 40.5, 56.1, 72.7, 116.2, 118.4, 120.8, 121.0, 148.6, 151.5, 194.7; IR (ATR) ν: 2983, 1736, 1373, 1237, 1045 cm^{-1}; HRMS (ESI) Calcd for $C_{11}H_{12}NaO_3$, $[M+Na]^+$ 215.0684, found 215.0678; colorless solid (15.5 mg, 69%).

3-methyl-1-tosyl-2,3-dihydroquinolin-4(1H)-one (5d)



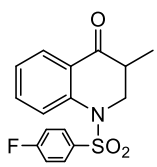
(CAS-Reg# 30504-93-9); ^{1}H-NMR ($CDCl_3$, 400 MHz) δ: 1.13 (d, 3H, *J* = 7.2 Hz), 2.39 (s, 3H), 2.42-2.49 (ddq, 1H), 3.64 (dd, 1H, *J* = 13.8, 13.8 Hz), 4.46 (dd, 1H, *J* = 13.8, 4.8 Hz), 7.22-7.27 (m, 3H), 7.53 (dd, 1H, *J* = 8.4, 8.4 Hz), 7.60 (d, 2H, *J* = 8.4 Hz), 7.84 (d, 1H, *J* = 8.4 Hz), 7.95 (d, 1H, *J* = 8.4 Hz); (28.5 mg, 57%)

1-((4-methoxyphenyl)sulfonyl)-3-methyl-2,3-dihydroquinolin-4(1H)-one (5e)



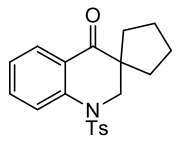
^{1}H-NMR ($CDCl_3$, 400 MHz) δ: 1.13 (d, 3H, *J* = 7.2 Hz), 2.41-2.51 (m, 1H), 3.63 (dd, 1H, *J* = 13.6, 13.6 Hz), 3.83 (s, 3H), 4.45 (dd, 1H, *J* = 13.6, 4.8 Hz), 6.89 (d, 2H, *J* = 9.2 Hz), 7.22 (dd, 1H, *J* = 7.6, 7.6 Hz), 7.53 (ddd, 1H, *J* = 8.4, 7.6, 1.6 Hz), 7.64 (d, 2H, *J* = 9.2 Hz), 7.84 (d, 1H, *J* = 8.4 Hz), 7.95 (dd, 1H, *J* = 7.6, 1.6 Hz); ^{13}C-NMR ($CDCl_3$, 100 MHz) δ: 12.3, 40.2, 52.2, 55.6, 114.5, 123.1, 124.6, 125.0, 128.1, 128.9, 131.1, 134.4, 142.4, 163.4, 195.5; IR (ATR) ν: 2983, 1736, 1372, 1233, 1043 cm^{-1}; HRMS (ESI) Calcd for $C_{17}H_{17}NNaO_4S$, $[M+Na]^+$ 354.0776, found 354.0785; yellow oil (32.6 mg, 56%).

1-((4-fluorophenyl)sulfonyl)-3-methyl-2,3-dihydroquinolin-4(1H)-one (5f)



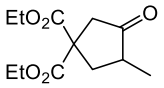
^{1}H-NMR ($CDCl_3$, 400 MHz) δ: 1.14 (d, 3H, *J* = 6.8 Hz), 2.42-2.52 (m, 1H), 3.65 (dd, 1H, *J* = 13.6, 13.6 Hz), 4.46 (dd, 1H, *J* = 13.6, 4.8 Hz), 7.15 (dd, 2H, *J* = 8.8, 8.8 Hz), 7.23 (dd, 1H, *J* = 8.0, 8.0 Hz), 7.55 (ddd, 1H, *J* = 8.8, 8.0, 1.6 Hz), 7.75 (dd, 2H, *J* = 8.8, 4.8 Hz), 7.81 (d, 1H, *J* = 8.8 Hz), 7.97 (dd, 1H, *J* = 8.0, 1.6 Hz); ^{13}C-NMR ($CDCl_3$, 100 MHz) δ: 12.4, 40.4, 52.4, 116.7, 116.9, 122.9, 124.7, 125.4, 128.3, 129.5, 129.6, 134.5, 135.7, 142.0, 164.5, 166.2, 195.2; IR (ATR) ν: 2932, 1690, 1355, 1170, 734 cm^{-1}; HRMS (ESI) Calcd for $C_{16}H_{14}FNNaO_3S$, $[M+Na]^+$ 342.0576, found 342.0586; colorless solid (20.2 mg, 46%).

1'-tosyl-1',2'-dihydro-4'H-spiro[cyclopentane-1,3'-quinolin]-4'-one (5g)



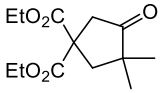
^{1}H-NMR ($CDCl_3$, 400 MHz) δ: 1.68-1.87 (m, 6H), 2.00-2.11 (m, 2H), 2.41 (s, 3H), 3.99 (s, 2H), 7.08 (dd, 1H, *J* = 7.2, 7.2 Hz), 7.32 (d, 2H, *J* = 8.4 Hz), 7.39 (dd, 1H, *J* = 7.8, 7.8 Hz), 7.59 (d, 1H, *J* = 8.4 Hz), 7.78 (d, 2H, *J* = 8.4 Hz), 8.00 (d, 1H, *J* = 7.8 Hz); ^{13}C-NMR ($CDCl_3$, 100 MHz) δ: 21.5, 25.7, 33.2, 53.9, 54.9, 118.4, 122.3, 123.3, 126.8, 129.0, 130.0, 134.2, 136.9, 142.3, 144.4, 197.6; IR (ATR) ν: 2953, 1683, 1351, 1161 cm^{-1}; HRMS (ESI) Calcd for $C_{20}H_{21}NNaO_3S$, $[M+Na]^+$ 378.1140, found 378.1153; colorless solid (19.5 mg, 30%, mp: 117-119 °C)

diethyl 3-methyl-4-oxocyclopentane-1,1-dicarboxylate (**5h**)



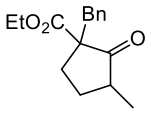
^{1}H-NMR ($CDCl_3$, 400 MHz) δ: 1.11 (d, 3H, *J* = 7.6 Hz), 1.27 (t, 6H, *J* = 7.2 Hz), 1.97 (dd, 1H, *J* = 12.8, 12.8 Hz), 2.41 (m, 1H), 2.72 (d, 1H, *J* = 18.8 Hz), 2.84 (dd, 1H, *J* = 12.8, 8.8 Hz), 2.95 (d, 1H, *J* = 18.8 Hz), 4.22 (q, 4H, *J* = 7.2 Hz); ^{13}C-NMR ($CDCl_3$, 100 MHz) δ: 13.9, 38.1, 42.5, 44.4, 54.8, 61.9, 62.0, 170.7, 171.0, 215.8; IR (ATR) ν: 2984, 1731, 1238, 1044 cm^{-1}; HRMS (ESI) Calcd for $C_{12}H_{18}NaO_5$, $[M+Na]^+$ 265.1052, found 265.1060; yellow oil (56 mg, 81%).

diethyl 3,3-dimethyl-4-oxocyclopentane-1,1-dicarboxylate (**5i**)



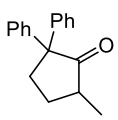
^{1}H-NMR ($CDCl_3$, 400 MHz) δ: 1.07 (s, 6H), 1.27 (t, 6H, *J* = 7.2 Hz), 2.52 (s, 2H), 2.98 (s, 2H), 4.23 (m, 4H); ^{13}C-NMR ($CDCl_3$, 100 MHz) δ: 13.9, 25.2, 43.9, 44.3, 45.2, 53.9, 62.1, 171.4, 218.5; IR (ATR) ν: 2970, 1731, 1366, 1184 cm^{-1}; HRMS (ESI) Calcd for $C_{13}H_{20}NaO_5$, $[M+Na]^+$ 279.1208, found 279.1203; yellow oil (25.1 mg, 54%).

ethyl 1-benzyl-3-methyl-2-oxocyclopentane-1-carboxylate (**5j**)



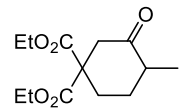
^{1}H-NMR ($CDCl_3$, 400 MHz) δ: 0.62-0.73 (m, 1H), 0.88 (d, 3H, *J* = 7.2 Hz), 1.25 (t, 3H, *J* = 7.2 Hz), 1.89-2.01 (m, 1H), 2.06-2.12 (m, 1H), 2.24-2.41 (m, 2H), 3.11 (d, 1H, *J* = 14.0 Hz), 3.17 (d, 1H, *J* = 14.0 Hz), 4.16 (q, 2H, *J* = 7.2 Hz), 7.11 (d, 2H, *J* = 7.6 Hz), 7.21-7.26 (m, 3H); ^{13}C-NMR ($CDCl_3$, 100 MHz) δ: 26.4, 26.6, 37.6, 39.2, 46.6, 50.5, 64.1, 64.6, 116.9, 118.0, 119.6, 124.6, 152.8, 188.6; IR (ATR) ν: 2964, 1747, 1722, 1454, 1156, 702 cm^{-1}; HRMS (ESI) Calcd for $C_{16}H_{20}NaO_3$, $[M+Na]^+$ 283.1310, found 283.1303; yellow oil (20.4 mg, 50%)

5-methyl-2,2-diphenylcyclopentan-1-one (**5k**)



(CAS-Reg# 1912-08-9); ^{1}H-NMR ($CDCl_3$, 400 MHz) δ: 1.16 (d, 3H, *J* = 7.6 Hz), 1.56-1.62 (m, 1H), 2.18-2.26 (m, 1H), 2.37-2.45 (m, 1H), 2.52-2.59 (m, 1H), 2.78-2.84 (m, 1H), 7.18-7.32 (m, 10H); (24.9 mg, 53%)

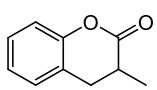
diethyl 4-methyl-3-oxocyclohexane-1,1-dicarboxylate (**5l**)



^{1}H-NMR ($CDCl_3$, 400 MHz) δ: 1.06 (d, 3H, *J* = 6.4 Hz), 1.22-1.27 (m, 6H), 1.40-1.51 (m, 1H), 2.02-2.11 (m, 1H), 2.18 (ddd, 1H, *J* = 13.6, 13.6, 3.6 Hz), 2.32 (qd, 1H, *J* = 6.4, 6.4 Hz), 2.41 (dd, 1H, *J* = 13.6, 2.8 Hz), 2.51 (d, 1H, *J* = 14.8 Hz), 2.96 (d, 1H, *J* = 14.8 Hz), 4.13-4.26 (m, 4H); ^{13}C-NMR ($CDCl_3$, 100 MHz) δ: 13.9, 13.9, 14.5, 29.9, 30.2, 44.0, 45.1, 57.6, 61.8, 61.8, 170.3, 170.4, 208.2; IR (ATR) ν: 2980, 1735, 1229, 1045 cm^{-1}; HRMS (ESI)

Calcd for $C_{13}H_{20}NaO_5$, $[M+Na]^+$ 279.1208, found 279. 1214; yellow oil (26.4 mg, 37%).

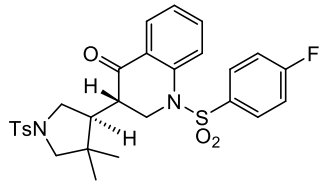
3-methylchroman-2-one (**5m**)



(CAS-Reg# 66122-70-1)

^{1}H-NMR ($CDCl_3$, 400 MHz) δ: 1.54 (d, 3H, *J* = 6.8 Hz), 2.89-2.99 (m, 2H), 4.64-4.73 (m, 1H), 7.26 (t, 1H, *J* = 6.8 Hz), 7.39 (t, 1H, *J* = 7.6 Hz), 7.53 (dt, 1H, *J* = 7.6, 1.6 Hz), 8.10 (d, 1H, *J* = 7.6 Hz); (17.8 mg, 52%)

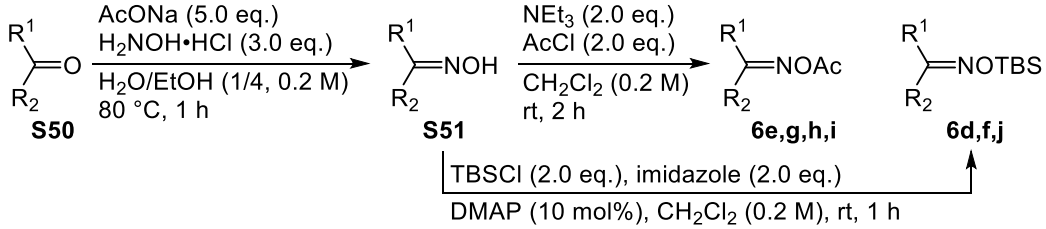
3-(4,4-dimethyl-1-tosylpyrrolidin-3-yl)-1-((4-fluorophenyl)sulfonyl)-2,3-dihydroquinolin-4(1H)-one (*trans*-**5n**)



^{1}H-NMR ($CDCl_3$, 400 MHz) δ: 0.74 (s, 3H), 1.07 (s, 3H), 2.44 (s, 3H), 2.59-2.68 (m, 1H), 2.87 (d, 1H, *J* = 9.6 Hz), 3.10 (dd, 1H, *J* = 10.8, 7.6 Hz), 3.20 (d, 1H, *J* = 9.6 Hz), 3.57-3.62 (m, 2H), 3.92 (dd, 1H, *J* = 13.6, 9.6 Hz), 4.20 (dd, 1H, *J* = 13.6, 4.4 Hz), 7.14-7.22 (m, 3H), 7.32 (d, 2H, *J* = 8.4 Hz), 7.48-7.56 (m, 2H), 7.70 (d, 2H, *J* = 8.4 Hz), 7.76-7.84 (m, 2H), 7.92 (d, 1H, *J* = 8.0 Hz); ^{13}C-NMR ($CDCl_3$, 100 MHz) δ: 21.5, 21.6, 26.0, 40.4, 44.0, 45.5, 49.2, 49.4, 61.2, 116.8, 117.0, 120.8, 123.9, 124.8, 127.4, 128.7, 129.6, 129.7, 129.8, 133.4, 134.9, 141.7, 143.6, 193.9; IR (ATR) ν: 1734, 1345, 1158, 634 cm^{-1}; HRMS (ESI) Calcd for $C_{28}H_{29}FN_2NaO_5S_2$, $[M+Na]^+$ 579.1400, found 579.1399; colorless solid (23.5 mg, 28%, 135-137 °C).

General procedure for cyclization: EtOH (0.2 M for alkenyloxime) was added to the mixture of alkenyloxime **6** (1.0 eq.), Co (5 mol%) under an argon atmosphere at rt, then $PhSiH_3$ (1.5 eq.) was added at rt and the solution was stirred at rt. Then, the solvent was removed under reduced pressure, and the residue was purified by flash column chromatography.

General procedure for the synthesis of alkenyloxime 6



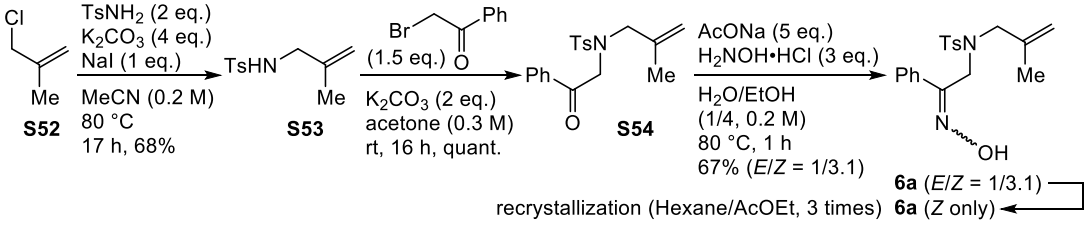
S50 to **S51**: To a solution of **S50** (1.0 eq.) in H_2O/EtOH (1/4, 0.2 M for **S50**), H_2NOH•HCl (5.0 eq.) and AcONa (5.0 eq.) was added at rt, and the mixture was stirred for 1 h at 80 °C. Then, the mixture was diluted with water and the aqueous layer was extracted with CH_2Cl_2. The combined organic layers were washed with brine, dried over Na_2SO_4, filtered, and concentrated on a rotary evaporator gave crude **S51** which was purified by column chromatography.

S51 to **6e,g,h,i**: To a solution of **S51** (1.0 eq.) in CH_2Cl_2 (0.2 M for **S51**), NEt_3 (2.0 eq.) and AcCl (2.0 eq.) was added at 0 °C, and the mixture was stirred for 2 h at rt. Then, the mixture was diluted with 1N HCl and the aqueous layer was extracted with CH_2Cl_2. The combined organic layers were washed with brine, dried

over Na_2SO_4, filtered, and concentrated on a rotary evaporator gave crude **6e,g,h,i** which was purified by column chromatography.

S51 to **6d,f,j**: To a solution of **S51** (1.0 eq.) in CH_2Cl_2 (0.2 M for **S51**), imidazole (2.0 eq.) and TBSCl (2.0 eq.) was added at 0 °C, and the mixture was stirred for 1 h at rt. Then, the mixture was diluted with H_2O and the aqueous layer was extracted with CH_2Cl_2. The combined organic layers were washed with brine, dried over Na_2SO_4, filtered, and concentrated on a rotary evaporator gave crude **6d,f,j** which was purified by column chromatography.

Synthesis of 6a

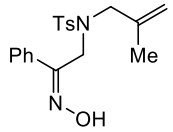


S52 to **S53**: To a stirred solution of **S52** (3.0 mL, 30.81 mmol) in MeCN (154 mL) was added K_2CO_3 (17.0 g, 123.2 mmol), NaI (4.6 g, 30.81 mmol) and $TsNH_2$ (10.5 g, 61.6 mmol), and the reaction mixture was stirred at 80 °C. The reaction was quenched with H_2O, extracted with AcOEt, dried over Na_2SO_4, filtered, and concentrated in vacuo. Purification by flash column chromatography afforded **S53** (4.5 g, 65%).

S53 to **S54**: To a stirred solution of **S53** (2.55 g, 10.0 mmol) in acetone (33 mL) was added K_2CO_3 (2.76 g, 20 mmol), phenayl bromide (2.99 g, 15.0 mmol) and the reaction mixture was stirred at rt for 16 h. The reaction was quenched with H_2O, extracted with AcOEt, dried over Na_2SO_4, filtered, and concentrated in vacuo. Purification by flash column chromatography afforded **S54** (3.42 g, quant.).

S54 to **6a**: Same as general procedure (**S50** to **S51**).

1-(4-isopropyl-2,3,5-trimethoxyphenyl)-5-methylhex-5-en-1-ol (**6a**)



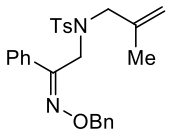
^{1}H-NMR ($CDCl_3$, 400 MHz) δ: 1.33 (s, 3H), 2.43 (s, 3H), 3.51 (s, 2H), 4.50 (s, 2H), 4.72 (s, 1H), 4.76 (s, 1H), 7.28 (d, 2H, J = 8.4 Hz), 7.33-7.40 (m, 3H), 7.60-7.69 (m, 4H), 9.30 (brs, 1H); ^{13}C-NMR ($CDCl_3$, 100 MHz) δ: 19.8, 21.5, 42.4, 56.0, 114.4, 127.3, 127.5, 128.3, 129.4, 129.7, 133.7, 135.0, 140.0, 143.6, 154.9; IR (ATR) ν: 3245, 3069, 2906, 1327, 1155, 752, 661 cm^{-1}; HRMS (ESI) Calcd for $C_{19}H_{22}N_2NaO_3S$, [M+Na] $^+$381.1249, found 381.1252; colorless solid (323 mg, 67%, mp: 140-143 °C)

Synthesis of 6b

To a stirred solution of **6a** (103.3 mg, 0.289 mmol) in THF (1.4 mL) was added NaH (35 mg, 0.88 mmol) at 0 °C, and the reaction mixture was stirred at 0 °C for 30 min. Then, benzyl bromide (0.1 mL, 0.84 mmol) was added at 0 °C, and the mixture was stirred for 2 h at rt. The reaction mixture was quenched with H_2O, extracted with AcOEt, dried over Na_2SO_4, filtered, and concentrated in vacuo. Purification by flash column chromatography afforded **6b** (*E*/*Z* mixture) which was recrystallized from hexane and AcOEt

to give **6b** (*Z* only, 45.9 mg, 35%).

(*Z*)-N-(2-((benzyloxy)imino)-2-phenylethyl)-4-methyl-N-(2-methylallyl)benzenesulfonamide (6b)

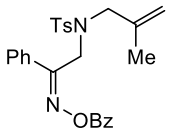


^{1}H-NMR ($CDCl_3$, 400 MHz) δ:1.31 (s, 3H), 2.42 (s, 3H), 3.47 (s, 2H), 4.46 (s, 2H), 4.62 (s, 1H), 4.67 (s, 1H), 5.14 (s, 2H), 7.20-7.41 (m, 10H), 7.57-7.72 (m, 4H); ^{13}C-NMR ($CDCl_3$, 100 MHz) δ:19.7, 21.5, 42.7, 55.7, 76.6, 114.4, 127.4, 127.5, 128.0, 128.2, 128.4, 128.5, 129.2, 129.6, 1334.0, 135.3, 137.2, 139.9, 143.4, 153.9; IR (ATR) ν: 2970, 1738, 1365, 1216, 612 cm^{-1}; HRMS (ESI) Calcd for $C_{26}H_{28}N_2NaO_3Si$, $[M+Na]^+$ 471.1718, found 471.1716; colorless solid

Synthesis of 6c

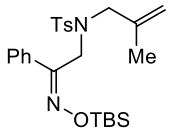
To a solution of **6a** (319 mg, 0.89 mmol) in CH_2Cl_2 (1.8 mL), NEt_3 (0.25 mL, 1.78 mmol) and BzCl (0.21 mL, 1.78 mmol) was added at 0 °C, and the mixture was stirred for 2 h at rt. Then, the mixture was diluted with water and the aqueous layer was extracted with CH_2Cl_2. The combined organic layers were washed with brine, dried over Na_2SO_4, filtered, and concentrated on a rotary evaporator gave crude **6c** which was purified by column chromatography (378 mg, 92%).

(*Z*)-N-(2-((benzoyloxy)imino)-2-phenylethyl)-4-methyl-N-(2-methylallyl)benzenesulfonamide (6c)



^{1}H-NMR ($CDCl_3$, 400 MHz) δ:1.39 (s, 3H), 2.44 (s, 3H), 3.54 (s, 2H), 4.70 (s, 2H), 4.80 (s, 1H), 4.84 (s, 1H), 7.24 (d, 2H, *J* = 7.6 Hz), 7.38 (dd, 2H, *J* = 7.6, 7.6 Hz), 7.44-7.66 (m, 8H), 8.07 (d, 2H, *J* = 7.6 Hz); ^{13}C-NMR ($CDCl_3$, 100 MHz) δ: 19.5, 21.2, 43.0, 55.4, 115.3, 127.2, 128.1, 128.1, 128.3, 128.4, 129.4, 129.5, 130.3, 132.2, 133.4, 134.8, 139.0, 143.6, 162.6, 163.0; IR (ATR) ν: 2942, 1763, 1344, 1212 cm^{-1}; HRMS (ESI) Calcd for $C_{26}H_{26}N_2NaO_4S$, $[M+Na]^+$ 485.1510, found 485.1506; colorless solid

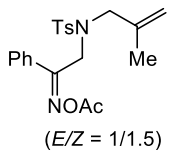
(*Z*)-N-(2-(((tert-butyldimethylsilyl)oxy)imino)-2-phenylethyl)-4-methyl-N-(2-methylallyl)benzenesulfonamide (6d)



6a to **6d**: Same as general procedure (**S51** to **6d**).

^{1}H-NMR ($CDCl_3$, 400 MHz) δ: 0.17 (s, 6H), 0.91 (s, 9H), 1.34 (s, 3H), 2.43 (s, 3H), 3.50 (s, 2H), 4.53 (s, 2H), 4.69 (s, 1H), 4.72 (s, 1H), 7.28 (d, 2H, *J* = 8.4 Hz), 7.32-7.35 (m, 3H), 7.66-7.70 (m, 4H); ^{13}C-NMR ($CDCl_3$, 100 MHz) δ: -5.1, 18.0, 19.8, 21.5, 26.0, 42.1, 55.3, 113.9, 127.2, 127.5, 128.1, 129.1, 129.6, 134.3, 135.2, 140.0, 143.4, 157.0; IR (ATR) ν: 2954, 1737, 1372, 1238, 1045, 744, 666 cm^{-1}; HRMS (ESI) Calcd for $C_{25}H_{36}N_2NaO_3SSi$, $[M+Na]^+$ 495.2114, found 495.2096; colorless oil (143.8 mg, quant.).

N-(2-(acetoxyimino)-2-phenylethyl)-4-methyl-N-(2-methylallyl)benzenesulfonamide (6e)



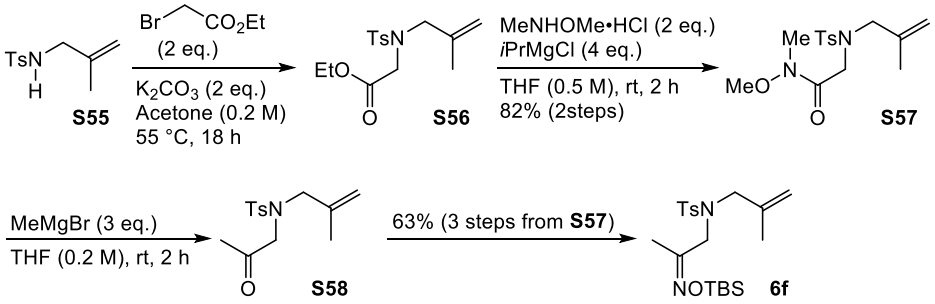
(*E*/*Z* = 1/1.5)

6a to **6e**: Same as general procedure (**S51** to **6e**).

^{1}H-NMR ($CDCl_3$, 400 MHz) δ: 1.36 (s, 1.8 H), 1.41 (s, 1.2H), 2.03 (s, 1.2H), 2.24 (s, 1.8 H), 2.42 (s, 1.2H), 2.44 (s, 1.8H), 3.51 (s, 1.2H), 3.63 (s, 0.8H), 4.31 (s, 0.8H), 4.54 (s, 1.2H), 4.75 (s, 0.6H), 4.81 (s, 0.6H), 4.83 (s, 0.4H), 4.86 (s, 0.4H), 7.23 (dd, 2H, *J* = 8.4, 8.4 Hz), 7.36-7.46 (m, 4H), 7.62-7.73 (m, 3H); ^{13}C-NMR ($CDCl_3$, 100 MHz) δ: 19.4, 19.6, 19.7, 19.7, 21.5, 43.6, 50.8, 55.1, 55.9, 115.2, 115.8, 127.4, 127.5, 128.1, 128.2, 128.3, 128.4, 129.6, 129.7, 130.0, 130.1,

130.5, 132.3, 135.1, 135.3, 139.5, 139.8, 143.6, 143.8, 161.4, 161.5, 168.2, 168.9; IR (ATR) ν: 2928, 1739, 1349, 1160, 812, 782 cm^{-1}; HRMS (ESI) Calcd for $C_{21}H_{24}N_2NaO_4Si$, $[M+Na]^+$ 423.1255, found 423.1347; yellow oil (45.1 mg, 80%).

Synthesis of 6f



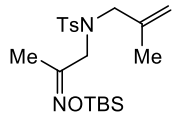
S55 to **S56**: To a stirred solution of **S55** (1.0 g, 4.44 mmol) in acetone (22 mL) was added K_2CO_3 (1.23 g, 8.88 mmol), ethyl bromoacetate (0.98 mL, 8.88 mmol) and the reaction mixture was stirred at 55 °C for 18 h. The reaction was quenched with H_2O, extracted with AcOEt, dried over Na_2SO_4, filtered, and concentrated in vacuo. Short column chromatography gave crude **S56**.

S56 to **S57**: To a solution of crude **S56** and MeNHOMe•HCl (866 mg, 8.88 mmol) in THF (9 mL) was added *i*PrMgCl (2.0 M in THF, 8.9 mL, 17.8 mmol) at -20 °C and the mixture was stirred at room temperature for 2 h. Sat. NH_4Cl aq. was added and the mixture was diluted with ethyl acetate. The layers were separated and the organic layer was washed with water and brine, dried over anhydrous Na_2SO_4 and concentrated under reduced pressure. The crude product was purified by silica gel column chromatography to afford the corresponding **S57** as a yellow oil (1.19 g, 82%, 2 steps).

S57 to **S58**: To a solution of **S57** (136.4 mg, 0.42 mmol) in THF (2 mL) was added MeMgBr (3.0 M in Et_2O, 0.42 mL, 1.3 mmol) at 0 °C and the mixture was stirred at room temperature for 2 h. Sat. NH_4Cl aq. was added and the mixture was diluted with ethyl acetate. The layers were separated and the organic layer was washed with water and brine, dried over anhydrous Na_2SO_4 and concentrated under reduced pressure. The crude **S58** was used in the next step without further purification.

S58 to **6f**: Same as general procedure (**S50** to **6f**). (108.2 mg, 63%, 3 steps)

N-(2-(((tert-butyldimethylsilyl)oxy)imino)propyl)-4-methyl-N-(2-methylallyl)benzenesulfonamide (6f)

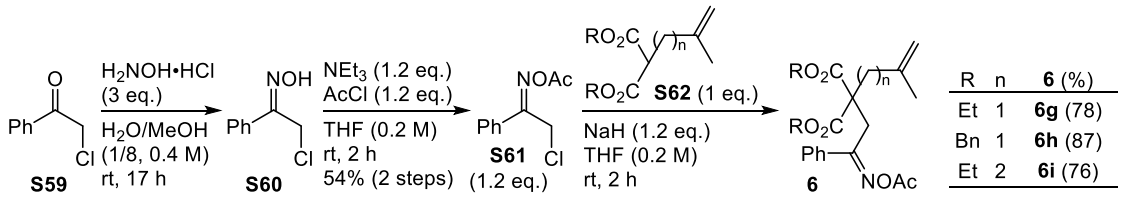


(*E*/*Z* = 1/7.3)

^{1}H-NMR ($CDCl_3$, 400 MHz) δ: 0.13 (s, 5.3H), (s, 0.7H), 0.90 (s, 1.1H), 0.91 (s, 7.9H), 1.65 (s, 2.6H), 1.68 (s, 0.4H), 1.84 (s, 2.6H), 1.92 (s, 0.4H), 2.43 (s, 2.6H), 2.44 (s, 0.4H), 3.62 (s, 0.2H), 3.64 (s, 1.8H), 3.78 (s, 1.8H), 4.04 (s, 0.2H), 4.76 (s. 0.1H), 4.80 (s, 0.9H), 4.87 (s, 1H), 7.29 (d, 2H, *J* = 8.0 Hz), 7.69(d, 2H, *J* = 8.0 Hz); ^{13}C-NMR ($CDCl_3$, 100 MHz) δ: -5.3, 12.3, 18.0, 20.0, 21.5, 26.0, 51.9, 54.3, 114.8, 127.3, 129.6, 136.2, 139.7, 143.3, 157.7; IR (ATR) ν: 2928, 1342, 1160, 916, 777, 655 cm^{-1}; HRMS (ESI) Calcd for $C_{20}H_{34}N_2NaO_3Si$,

$[M+Na]^+$ 433.1957, found 433.1953; colorless oil

Synthesis of 6g,h,i

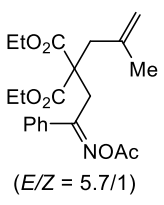


S59 to **S60**: To a slurry of phenacyl chloride (3.0 g, 19.4 mmol) and hydroxylamine hydrochloride (4.02 g, 58.2 mmol) in 5 mL of water was added with stirring portionwise methanol (40 mL) to yield a clear solution. The clear solution was stirred for 17 h at room temperature and was added cold water, whereupon an orange solid precipitated out. The oxime product was filtered out, washed several times with water, and dried under reduced pressure gave crude **S60** which was used in the next step without further purification.

S60 to **S61**: To a solution of crude **S60** in THF (116 mL), NEt_3 (3.3 mL, 23.3 mmol) and AcCl (1.7 mL, 23.3 mmol) was added at 0 °C, and the mixture was stirred for 2 h at rt. Then, the mixture was diluted with H_2O and the aqueous layer was extracted with AcOEt. The combined organic layers were washed with brine, dried over Na_2SO_4, filtered, and concentrated on a rotary evaporator gave crude **S61** which was purified by column chromatography (2.2 g, 54%, 2 steps).

S61 to **6g,h,i**: To a solution of **S61** (1.0 eq.) in THF (0.2 M for **S61**) was added 60% sodium hydride (1.2 eq.) at 0 °C and the mixture was stirred at room temperature for 30 minutes. **S62** (1.2 eq.) was then added and the mixture was stirred for 2 h. Water was added and the mixture was diluted with ethyl acetate. The layers were separated and the organic layer was washed with water and brine, dried over anhydrous Na_2SO_4 and concentrated under reduced pressure. The crude product was purified by silica gel column chromatography to afford the corresponding **6g,h,i**.

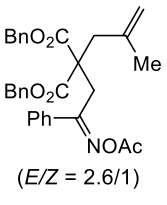
diethyl 2-(2-(acetoxyimino)-2-phenylethyl)-2-(2-methylallyl)malonate (**6g**)



(*E*/*Z* = 5.7/1)

^{1}H-NMR ($CDCl_3$, 400 MHz) δ: 1.13 (t, 0.9H, *J* = 6.8 Hz), 1.22 (t, 5.1H, *J* = 6.8 Hz), 1.62 (s, 0.45H), 1.65 (s, 2.55H), 2.02 (s, 2.55H), 2.21 (s, 0.45H), 2.65 (s, 0.3H), 2.97 (s, 1.7H), 3.35 (s, 1.7H), 3.61 (s. 0.3H), 3.86-4.18 (m, 4H), 4.72 (s, 0.15H), 4.80 (s, 0.85H), 4.85 (s, 0.15H), 4.88 (s, 0.85H), 7.28-7.59 (m, 5H); ^{13}C-NMR ($CDCl_3$, 100 MHz) δ: 13.7, 19.6, 23.0, 37.6, 39.6, 55.4, 61.5, 116.0, 127.3, 128.0, 129.5, 132.9, 140.6, 162.5, 168.6, 170.3; IR (ATR) ν: 2981, 1770, 1732, 1202, 760 cm^{-1}; HRMS (ESI) Calcd for $C_{21}H_{27}NNaO_6$, $[M+Na]^+$ 412.1736, found 412.1725; yellow oil (284.2 mg, 78%).

dibenzyl 2-(2-(acetoxyimino)-2-phenylethyl)-2-(2-methylallyl)malonate (**6h**)

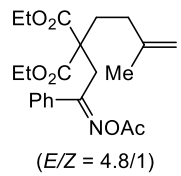


(*E*/*Z* = 2.6/1)

^{1}H-NMR ($CDCl_3$, 400 MHz) δ: 1.56 (s, 3H), 1.98 (s, 2.17H), 2.17 (s, 0.83H), 2.69 (s, 0.56H), 3.03 (s, 1.44H), 3.37 (s, 1.44H), 3.68 (s, 0.56H), 4.58-5.06 (m, 6H), 7.11-7.55 (m, 15H); ^{13}C-NMR ($CDCl_3$, 100 MHz) δ: 14.1, 19.5, 20.9, 22.9, 23.8, 31.3, 37.7, 39.8, 40.9, 55.5, 56.0, 60.2, 67.3, 67.3, 113.8, 116.2, 127.3, 128.0, 128.1, 128.1, 128.3, 129.5, 130.2, 132.7, 134.1, 134.6, 135.1, 140.3, 140.4, 162.0, 163.5, 168.7, 169.8, 169.9; IR (ATR) ν:

2943, 1730, 1194, 903, 736 cm^{-1}; HRMS (ESI) Calcd for $C_{31}H_{31}NNaO_6$, $[M+Na]^+$ 536.2049, found 536.2049; yellow oil (504.5 mg, 87%).

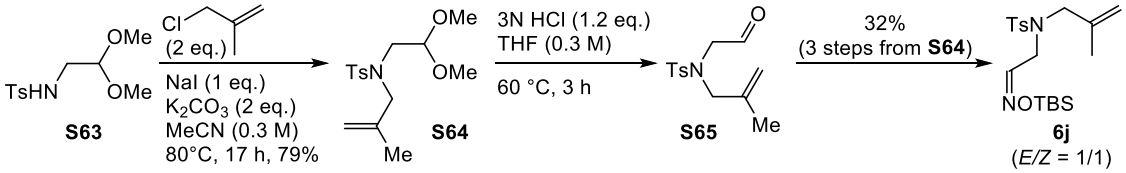
diethyl 2-(2-(acetoxyimino)-2-phenylethyl)-2-(3-methylbut-3-en-1-yl)malonate (6i)



(*E*/*Z* = 4.8/1)

^{1}H-NMR ($CDCl_3$, 400 MHz) δ: 1.17 (t, 6H, *J* = 6.8 Hz), 1.63 (s, 0.52H), 1.67 (s, 2.48H), 1.82-2.31 (m, 7H), 3.39 (s, 1.66H), 3.57 (s, 0.34H), 3.80-4.20 (m, 4H), 4.58 (s, 0.17H), 4.61 (s, 0.17H), 4.64 (s, 1.83H), 4.68 (s, 1.83H), 7.29-7.82 (m, 5H); ^{13}C-NMR ($CDCl_3$, 100 MHz) δ: 13.8, 19.5, 22.2, 30.4, 32.0, 37.3, 56.0, 61.4, 110.5, 127.7, 127.9, 129.7, 132.2, 144.5, 162.4, 168.4, 170.1; IR (ATR) ν: 2982, 1728, 1183, 903, 697 cm^{-1}; HRMS (ESI) Calcd for $C_{22}H_{29}NNaO_6$, $[M+Na]^+$ 426.1893, found 426.1902; yellow oil (404.0 mg, 76%).

Synthesis of 6j

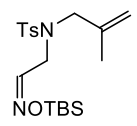


S63 to **S64**: To a stirred solution of **S63** (1.25 g, 4.83 mmol) in MeCN (16 mL) was added K_2CO_3 (1.33 g, 9.65 mmol), NaI (724 mg, 4.83 mmol) and 3-chloro-2-methyl-1-propene (0.94 mL, 9.65 mmol), and the reaction mixture was stirred at 80 °C for 17 h. The reaction was quenched with H_2O, extracted with AcOEt, dried over Na_2SO_4, filtered, and concentrated in vacuo. Purification by flash column chromatography afforded **S64** (1.2 g, 79%).

S64 to **S65**: To a stirred solution of **S64** (1.2 g, 3.83 mmol) in THF (12 mL) was added 3N HCl (1.5 mL, 4.5 mmol) at rt, and the reaction mixture was stirred at 60 °C for 3 h. The reaction was quenched with H_2O, extracted with AcOEt, dried over Na_2SO_4, filtered, and concentrated in vacuo. Purification by flash column chromatography afforded crude **S65** (864 mg) and **S64** (223 mg, 19%).

S65 to **6j**: Same as general procedure (**S50** to **6j**).

N-(2-(((tert-butyldimethylsilyl)oxy)imino)ethyl)-4-methyl-N-(2-methylallyl)benzenesulfonamide (6j)

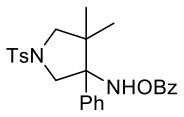


(*E*/*Z* = 1/1)

^{1}H-NMR ($CDCl_3$, 400 MHz) δ: 0.11 (s, 3H), 0.13 (s, 3H), 0.90 (s, 9H), 1.69 (s, 1.5H), 1.75 (s, 1.5H), 2.43 (s, 3H), 3.68 (s, 1H), 3.69 (s, 1H), 3.85 (d, 1H, *J* = 6.0 Hz), 4.03 (d, 1H, *J* = 3.6 Hz), 4.86 (s, 1H), 4.93 (s, 1H), 6.74 (t, 0.5H, *J* = 4.0 Hz), 7.28-7.33 (m, 2.5H), 7.69 (d, 2H, *J* = 8.0 Hz); ^{13}C-NMR ($CDCl_3$, 100 MHz) δ: -5.5, -5.5, 17.9, 19.7, 21.4, 25.8, 25.8,

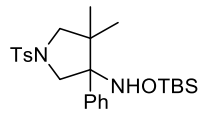
42.5, 45.5, 53.7, 55.2, 115.0, 115.5, 127.1, 129.7, 129.8, 135.8, 136.4, 139.3, 139.3, 143.4, 143.6, 149.9, 152.1; IR (ATR) ν: 2929, 1739, 1350, 1249, 1160, 912, 754 cm^{-1}; HRMS (ESI) Calcd for $C_{19}H_{32}N_2NaO_3SSi$, $[M+Na]^+$ 419.1801, found 419.1791; colorless oil (250 mg, 32%).

O-benzoyl-N-(4,4-dimethyl-3-phenyl-1-tosylpyrrolidin-3-yl)hydroxylamine (7c)



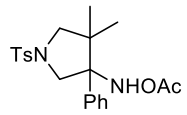
^{1}H-NMR ($CDCl_3$, 400 MHz) δ: 0.70 (s, 3H), 1.10 (s, 3H), 2.28 (s, 3H), 3.37 (d, 1H, *J* = 9.2 Hz), 3.46 (d, 1H, *J* = 9.2 Hz), 3.94 (d, 1H, *J* = 11.2 Hz), 4.21 (d, 1H, *J* = 11.2 Hz), 7.17 (d, 2H, *J* = 8.4 Hz), 7.28-7.38 (m, 7H), 7.53 (dd, 1H, *J* = 7.2, 7.2 Hz), 7.64 (d, 2H, *J* = 8.4 Hz), 7.76 (d, 2H, *J* = 7.6 Hz), 7.92 (s, 1H); ^{13}C-NMR ($CDCl_3$, 100 MHz) δ: 19.6, 21.4, 24.9, 44.8, 53.1, 60.0, 72.8, 126.5, 127.4, 127.8, 128.3, 128.4, 129.0, 129.6, 133.3, 143.5, 165.5; IR (ATR) ν: 2924, 1718, 1343, 1157, 705 cm^{-1}; HRMS (ESI) Calcd for $C_{26}H_{28}N_2NaO_3Si$, $[M+Na]^+$ 487.1662, found 487.1160; colorless solid (20.9 mg, 35%).

O-(tert-butyldimethylsilyl)-N-(4,4-dimethyl-3-phenyl-1-tosylpyrrolidin-3-yl)hydroxylamine (7d)



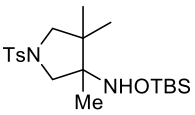
^{1}H-NMR ($CDCl_3$, 400 MHz) δ: -0.21 (s, 3H), -0.09 (s, 3H), 0.46 (s, 3H), 0.71 (s, 9H), 1.05 (s, 3H), 2.43 (s, 3H), 3.26 (d, 1H, *J* = 9.6 Hz), 3.30 (d, 1H, *J* = 9.6 Hz), 3.87 (d, 1H, *J* = 10.0 Hz), 4.19 (d, 1H, *J* = 10.0 Hz), 4.97 (brs, 1H), 7.19-7.30 (m, 5H), 7.33 (d, 2H, *J* = 8.0 Hz), 7.79 (d, 2H, *J* = 8.0 Hz); ^{13}C-NMR ($CDCl_3$, 100 MHz) δ: -5.53, -5.45, 17.8, 19.6, 21.5, 25.6, 25.9, 44.0, 51.2, 59.9, 73.5, 127.1, 127.3, 127.5, 127.6, 129.7, 134.5, 138.4, 143.4; IR (ATR) ν: 2926, 1713, 1345, 1158, 1092, 826 cm^{-1}; HRMS (ESI) Calcd for $C_{25}H_{38}N_2NaO_3Si$, $[M+Na]^+$ 497.2270, found 497.2257; colorless solid (23.7 mg, 60%).

O-acetyl-N-(4,4-dimethyl-3-phenyl-1-tosylpyrrolidin-3-yl)hydroxylamine (7e)



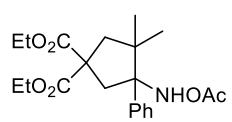
^{1}H-NMR ($CDCl_3$, 400 MHz) δ: 0.58 (s, 3H), 1.04 (s, 3H), 1.80 (s, 3H), 2.44 (s, 3H), 3.28 (d, 1H, *J* = 9.6 Hz), 3.40 (d, 1H, *J* = 9.6 Hz), 3.83 (d, 1H, *J* = 11.2 Hz), 4.08 (d, 1H, *J* = 11.2 Hz), 7.25-7.31 (m, 4H), 7.33 (d, 2H, *J* = 8.0 Hz), 7.79-7.81 (m, 3H); ^{13}C-NMR ($CDCl_3$, 100 MHz) δ: 18.8, 19.7, 21.5, 24.7, 44.6, 53.1, 59.8, 72.4, 126.5, 127.4, 127.7, 128.2, 129.5, 134.2, 136.9, 143.5, 169.7; IR (ATR) ν: 2969, 1739, 1343, 1220, 1157, 813 cm^{-1}; HRMS (ESI) Calcd for $C_{21}H_{26}N_2NaO_4Si$, $[M+Na]^+$ 425.1511, found 425.1504; colorless solid (32.8 mg, 61%).

O-(tert-butyldimethylsilyl)-N-(3,4,4-trimethyl-1-tosylpyrrolidin-3-yl)hydroxylamine (7f)



^{1}H-NMR ($CDCl_3$, 400 MHz) δ: -0.02 (s, 3H), 0.02 (s, 3H), 0.78 (s, 3H), 0.84 (s, 3H), 0.87 (s, 3H), 0.96 (s, 3H), 2.42 (s, 3H), 3.04 (d, 1H, *J* = 10.8 Hz), 3.16 (brs, 2H), 3.64 (d, 1H, *J* = 10.8 Hz), 4.42 (brs, 1H), 7.30 (d, 2H, *J* = 7.6 Hz), 7.72 (d, 2H, *J* = 7.6 Hz); ^{13}C-NMR ($CDCl_3$, 100 MHz) δ: -5.6, -5.5, 16.0, 17.9, 19.1, 21.5, 24.3, 26.1, 43.0, 54.2, 59.9, 68.1, 127.3, 129.6, 134.4, 143.2; IR (ATR) ν: 2952, 1542, 1344, 1158, 1094, 665 cm^{-1}; HRMS (ESI) Calcd for $C_{20}H_{36}N_2NaO_3SSi$, $[M+Na]^+$ 435.2114, found 435.2107; yellow oil (25.7 mg, 66%).

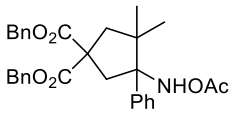
diethyl 3-(acetoxyamino)-4,4-dimethyl-3-phenylcyclopentane-1,1-dicarboxylate (7g)



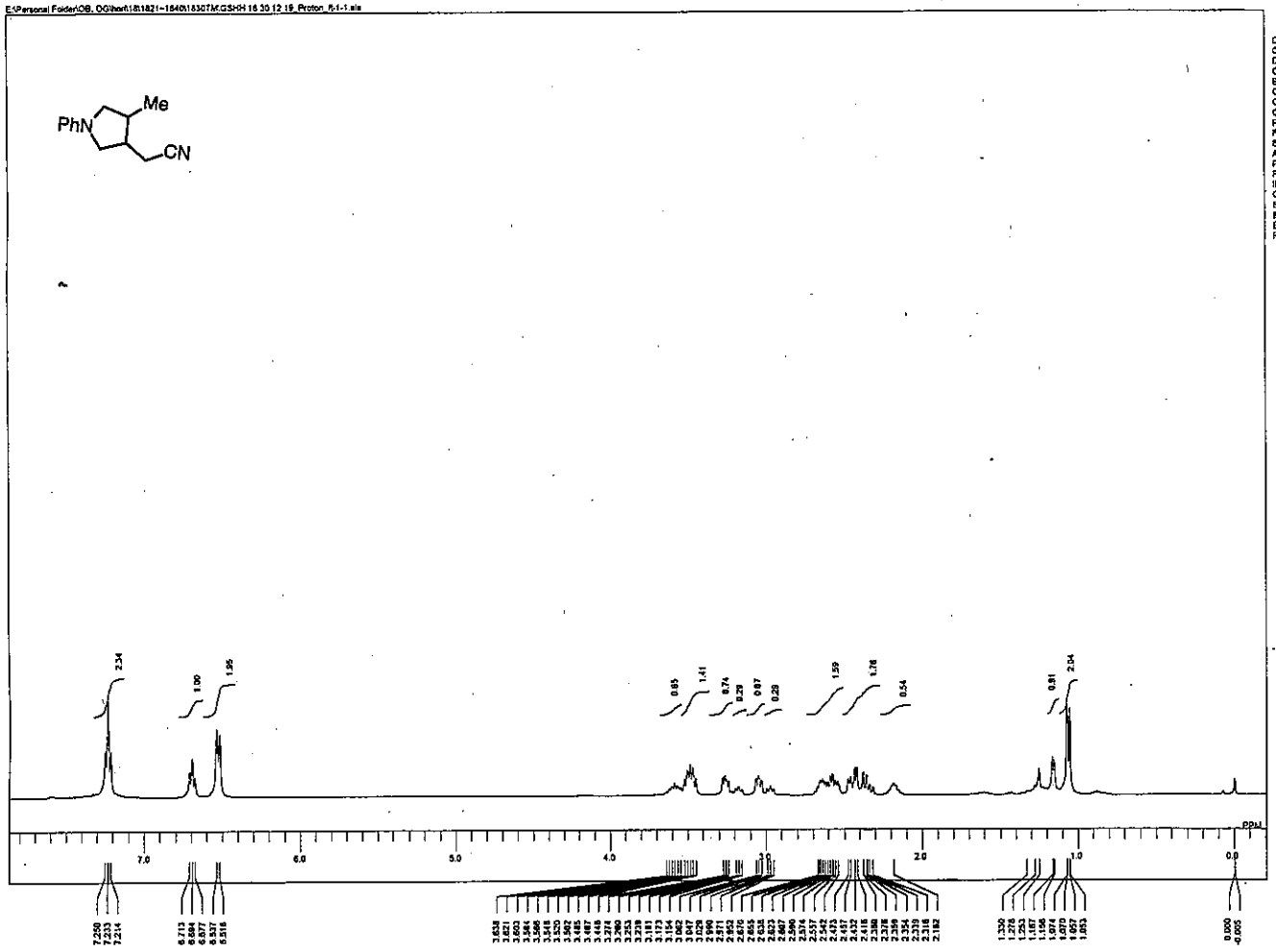
^{1}H-NMR ($CDCl_3$, 400 MHz) δ: 0.66 (s, 3H), 1.15 (s, 3H), 1.28 (t, 6H, *J* = 7.2 Hz), 1.79 (s, 3H), 2.17 (d, 1H, *J* = 14.4 Hz), 2.91 (d, 1H, *J* = 14.4 Hz), 3.06 (d, 1H, *J* = 14.4 Hz), 3.35 (d, 1H, *J* = 14.4 Hz), 4.16-4.32 (m, 4H), 7.24-7.40 (m, 5H), 8.09 (brs,

1H); ^{13}C-NMR ($CDCl_3$, 100 MHz) δ: 14.0, 18.9, 21.4, 27.4, 39.3, 45.7, 47.4, 56.7, 61.5, 61.7, 74.6, 126.5, 127.1, 127.9, 138.8, 169.8, 172.1, 173.0; IR (ATR) ν: 2980, 1730, 1369, 1238, 1044, 700 cm^{-1}; HRMS (ESI) Calcd for $C_{21}H_{29}NNaO_6$, $[M+Na]^+$ 414.1893, found 414.1906; orange oil (57.5 mg, 96%).

dibenzyl 3-(acetoxyamino)-4,4-dimethyl-3-phenylcyclopentane-1,1-dicarboxylate (7h)



^{1}H-NMR ($CDCl_3$, 400 MHz) δ: 0.63 (s, 3H), 1.13 (s, 3H), 1.64 (s, 3H), 2.18 (d, 1H, *J* = 14.4 Hz), 2.97 (d, 1H, *J* = 14.4 Hz), 3.11 (d, 1H, *J* = 14.4 Hz), 3.37 (d, 1H, *J* = 14.4 Hz), 5.06-5.20 (m, 4H), 7.24-7.36 (m, 15H), 8.14 (s, 1H)); ^{13}C-NMR ($CDCl_3$, 100 MHz) δ: 18.8, 21.4, 27.5, 39.5, 45.6, 47.5, 56.9, 67.3, 67.4, 74.7, 126.4, 127.1, 127.9, 127.9, 128.0, 128.0, 128.2, 128.4, 128.5, 135.4, 135.8, 138.6, 169.9, 171.8, 172.7; IR (ATR) ν: 2969, 1731, 1239, 698 cm^{-1}; HRMS (ESI) Calcd for $C_{31}H_{33}NNaO_6$, $[M+Na]^+$ 538.2206, found 538.2193; yellow oil (69.2 mg, 83%).


Me
PhN
CN
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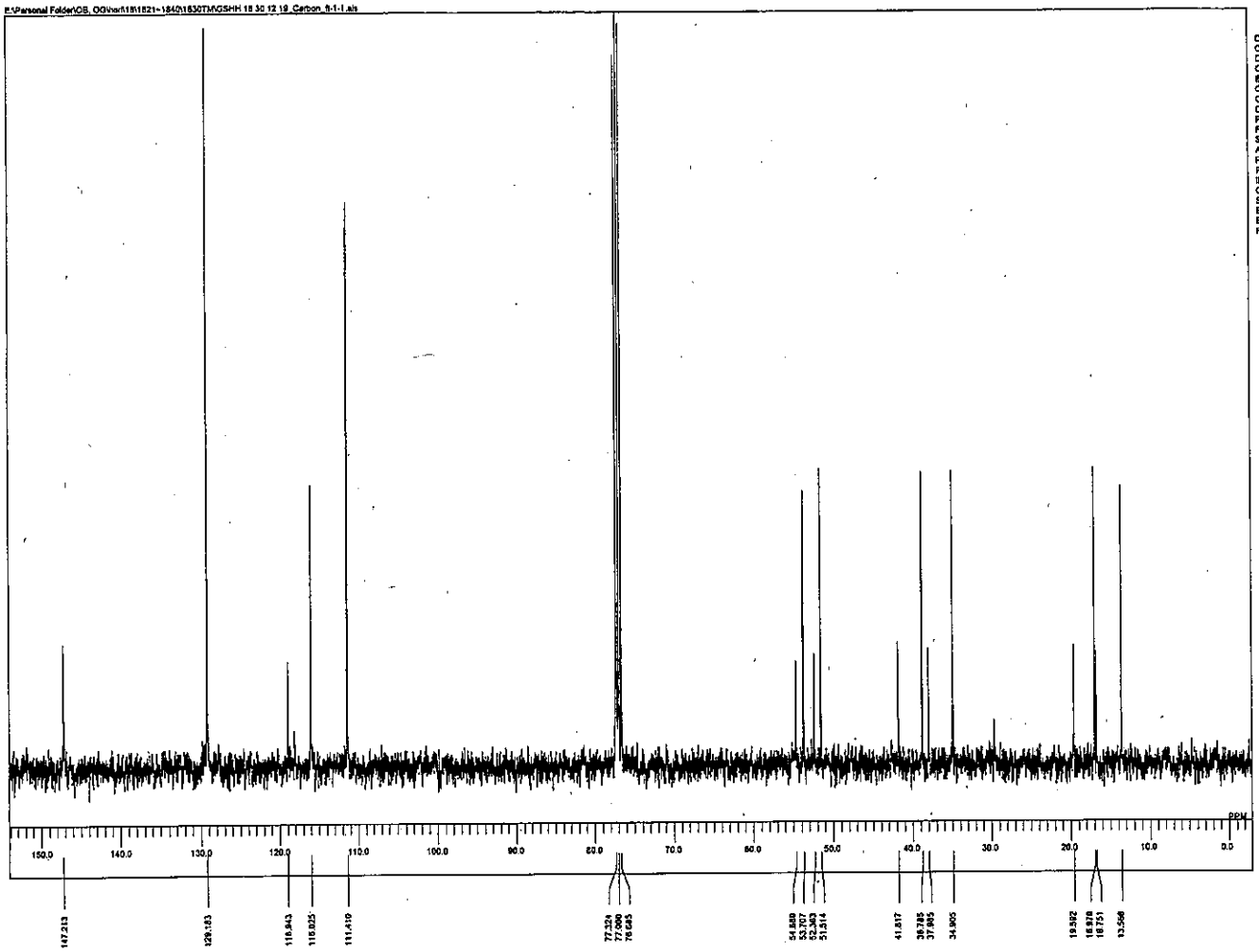
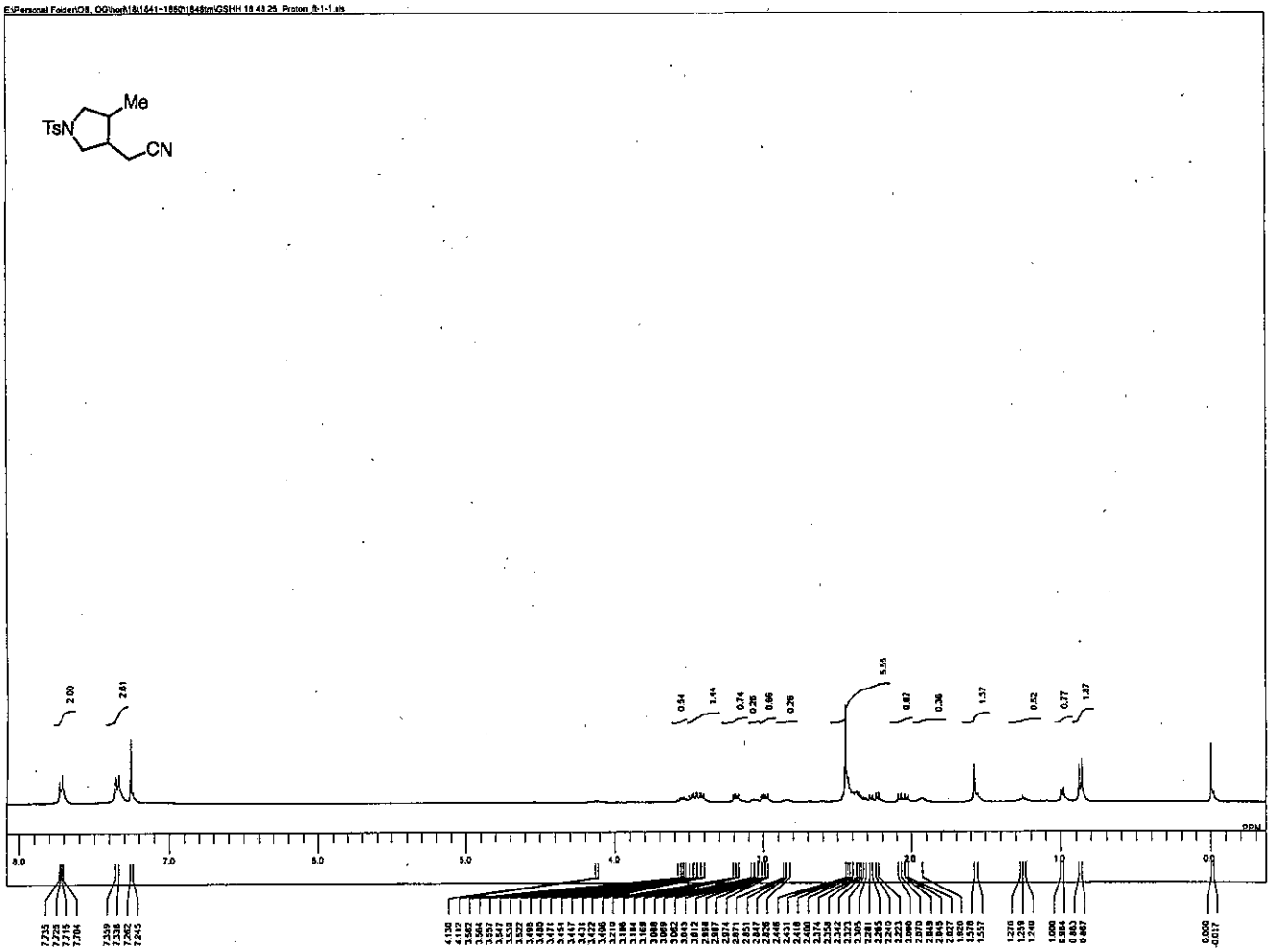
1.00
1.95
0.85
1.41
0.74
0.29
0.97
0.29
1.59
1.78
0.54
0.81
2.04
PPM


PPM


Me
TsN
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2.61
0.54
1.44
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0.26
5.55
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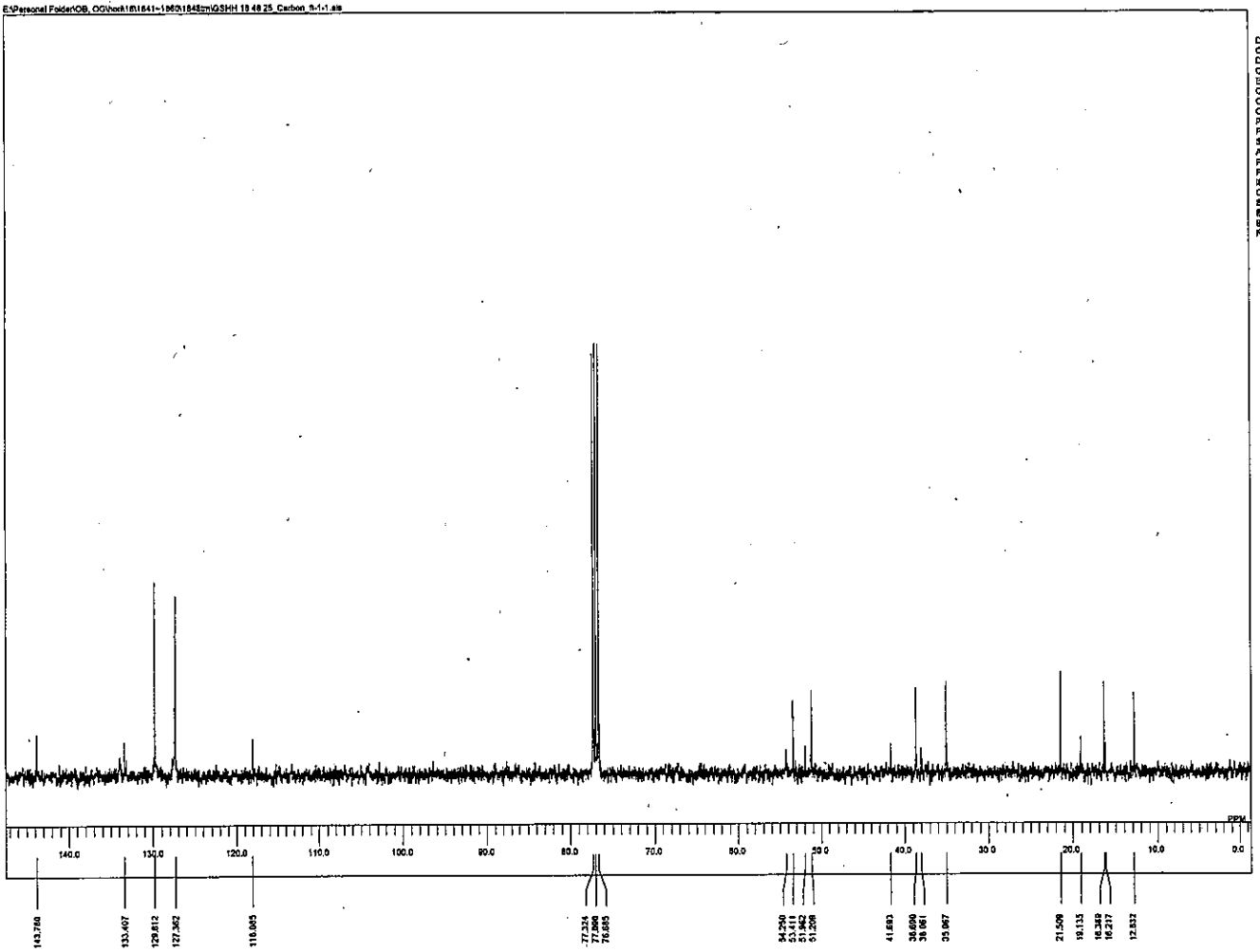
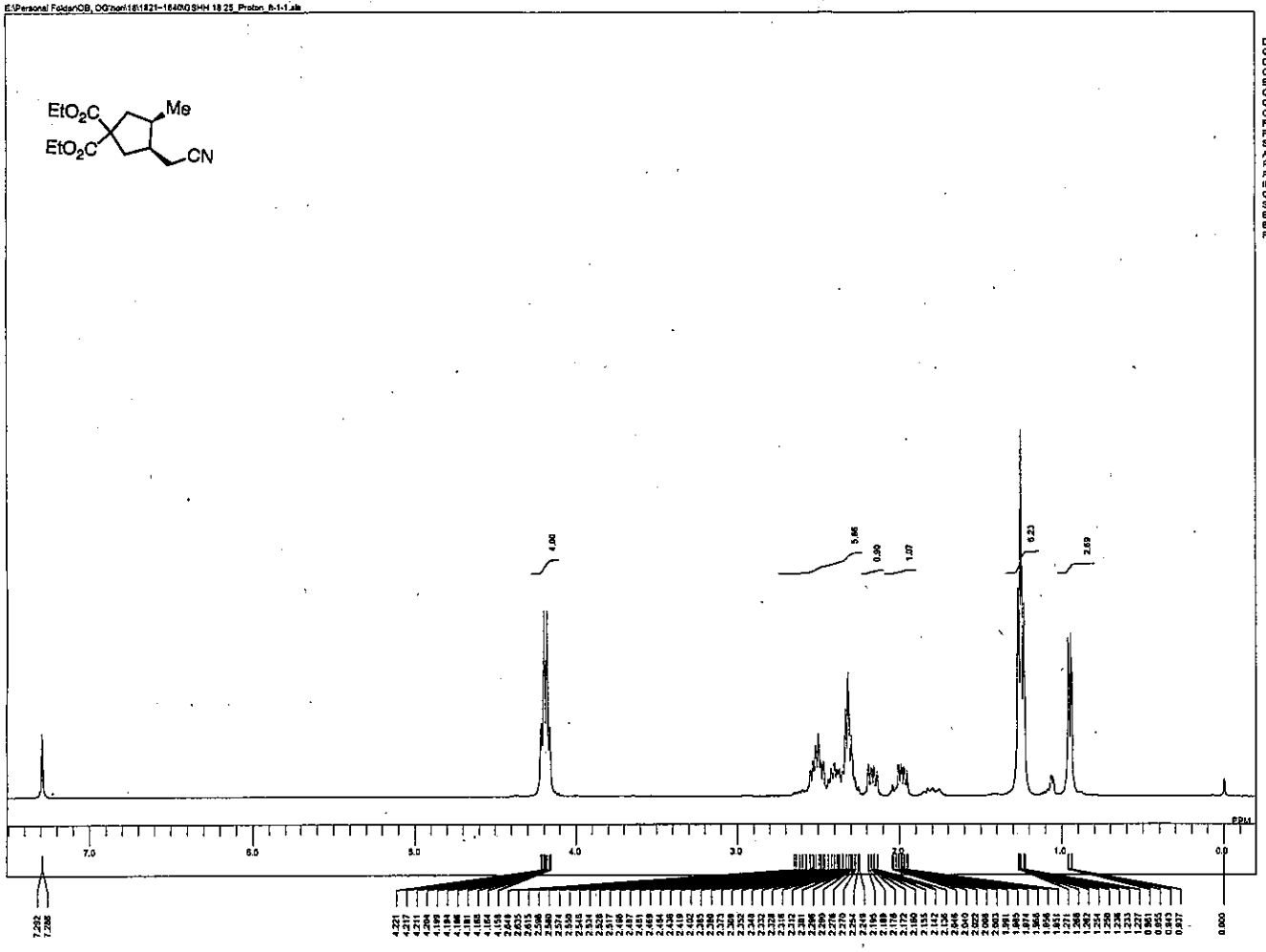
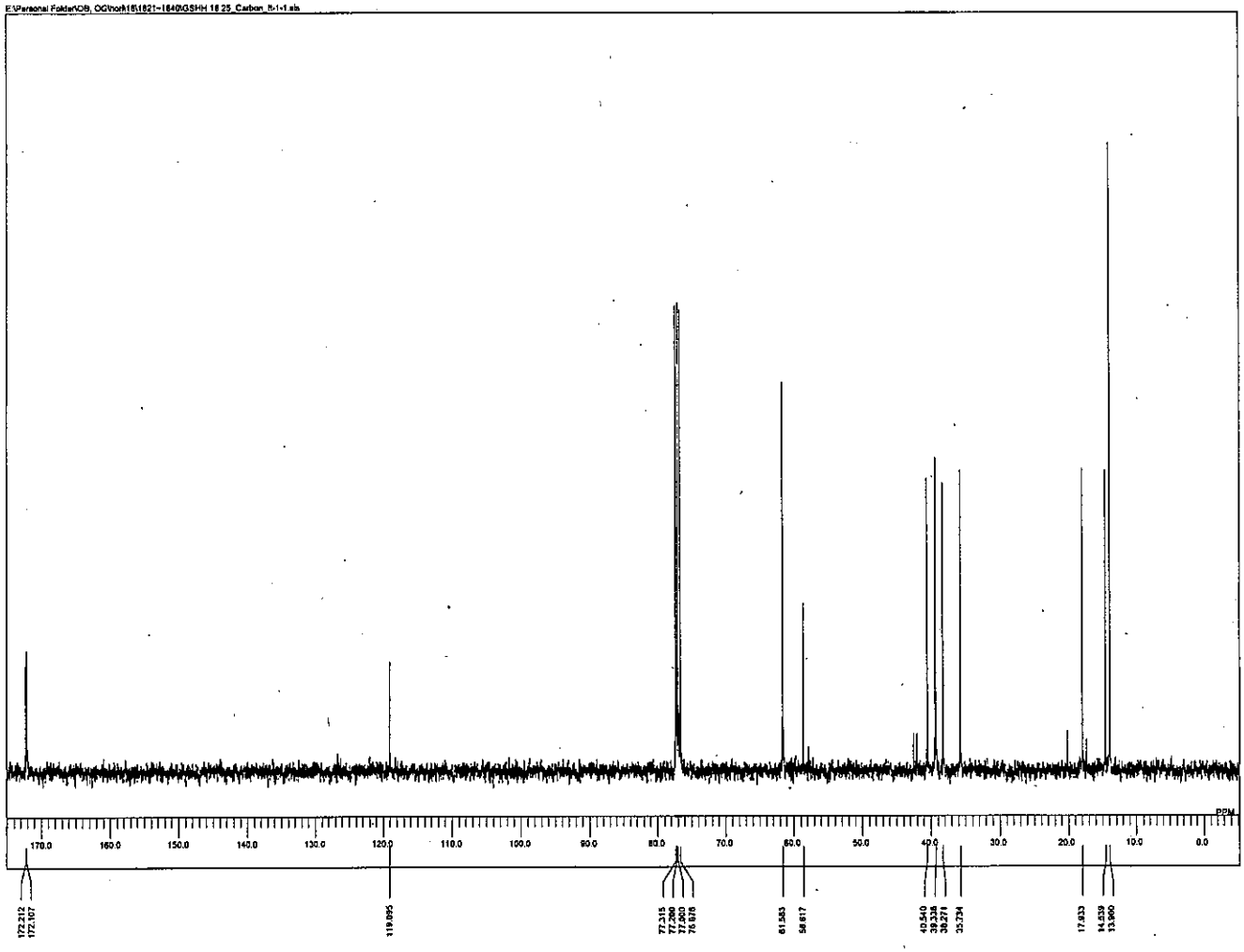
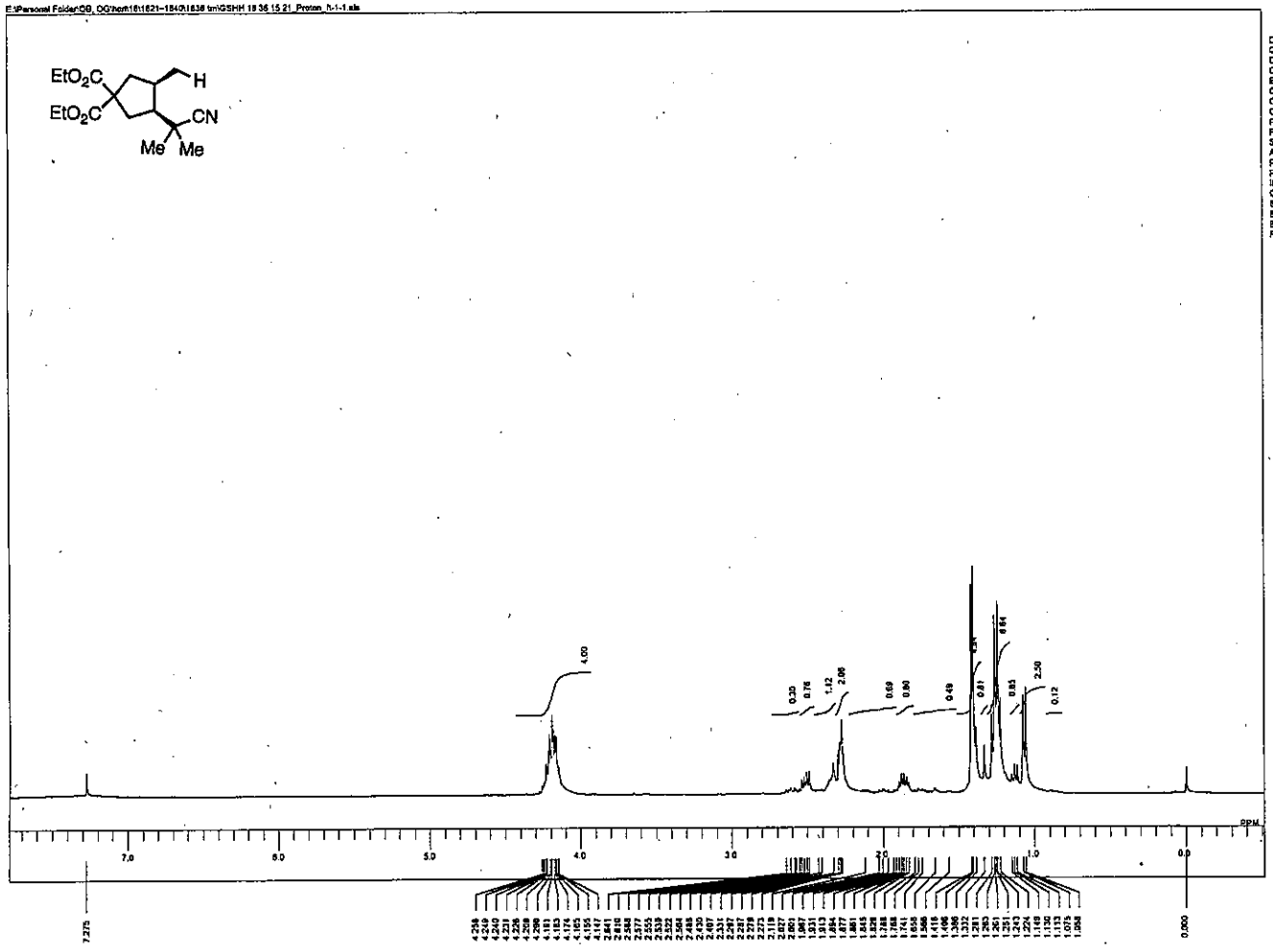
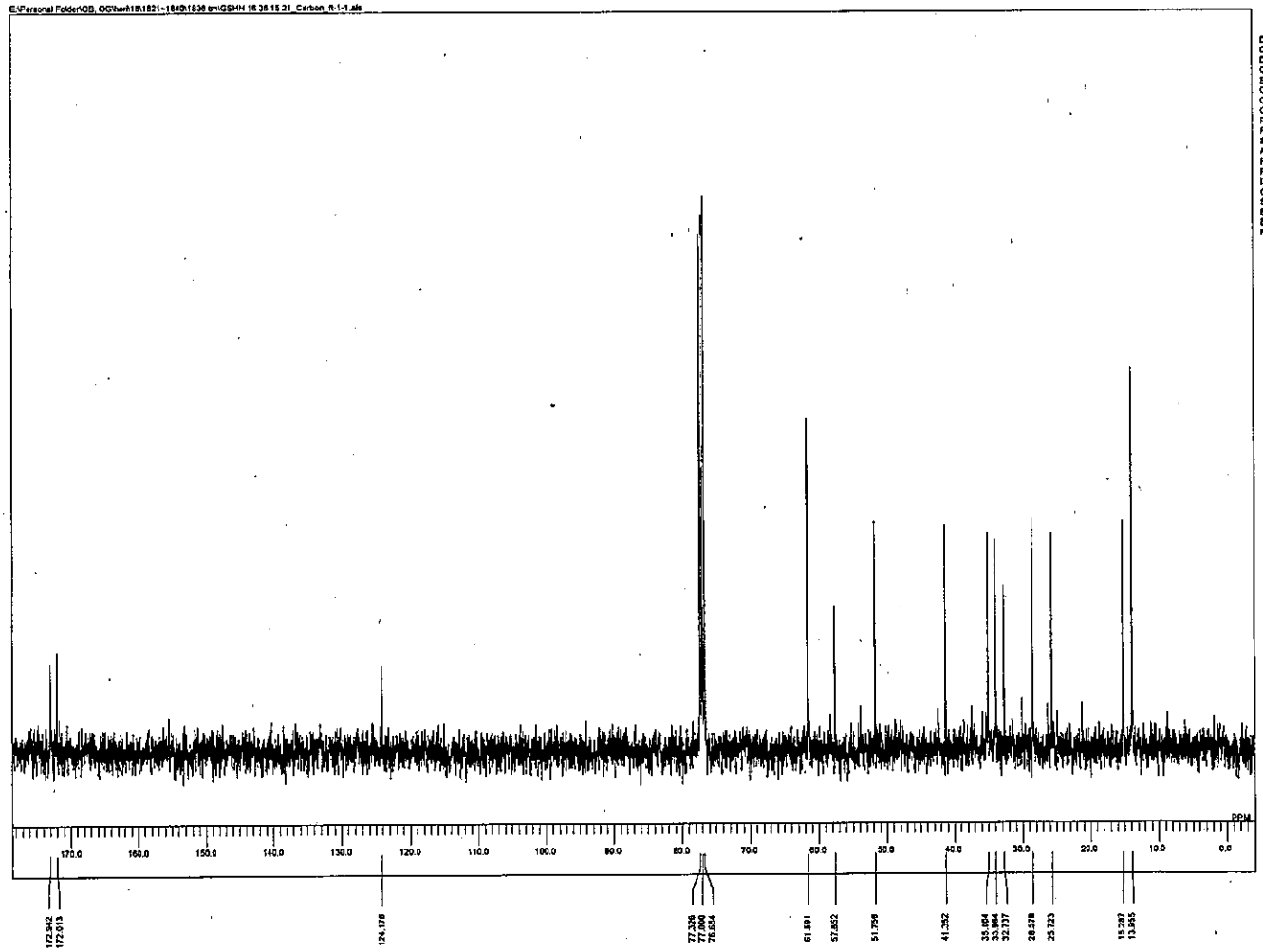
1.57
0.52
0.77
1.87
PPM

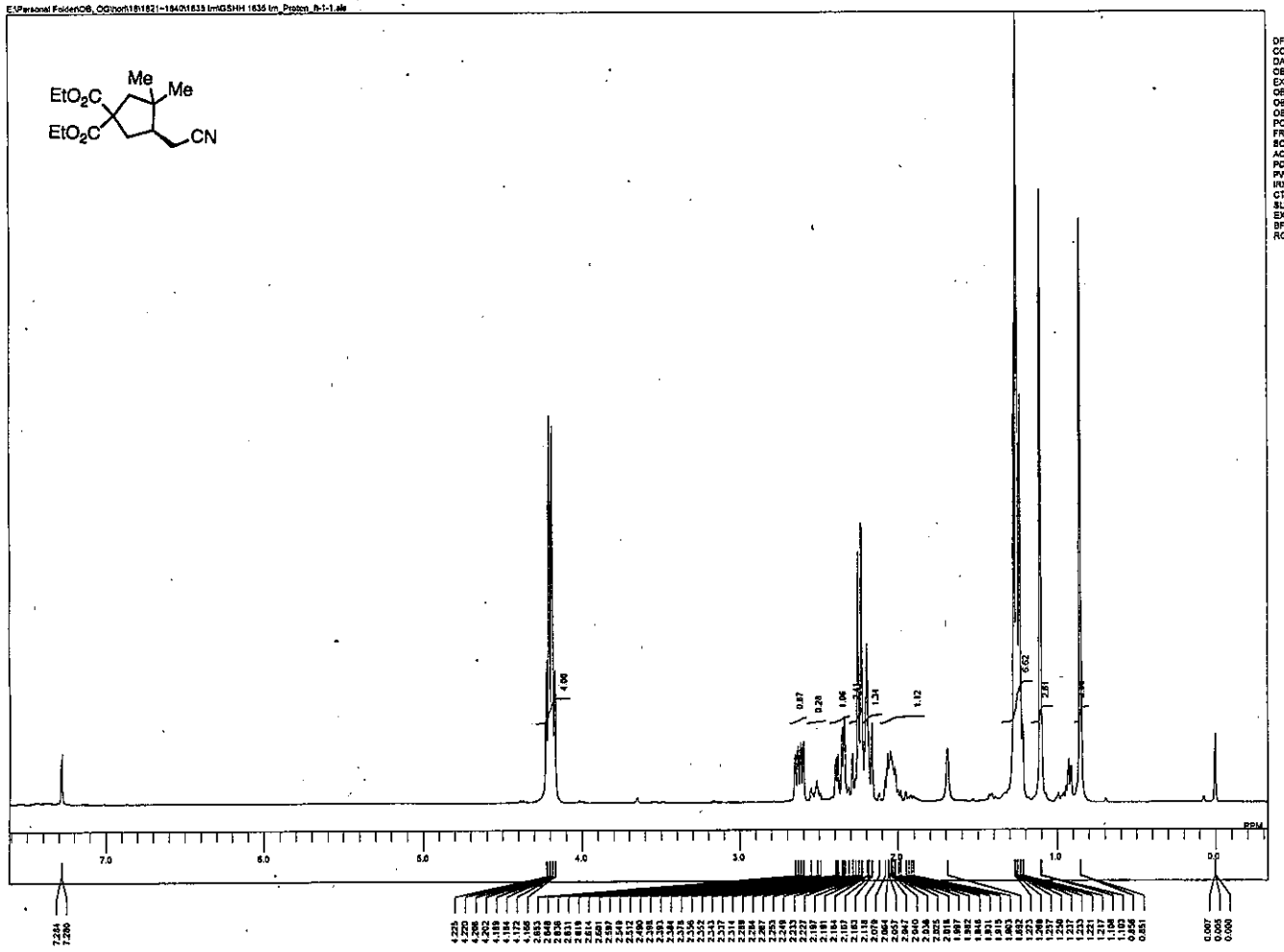

PPM

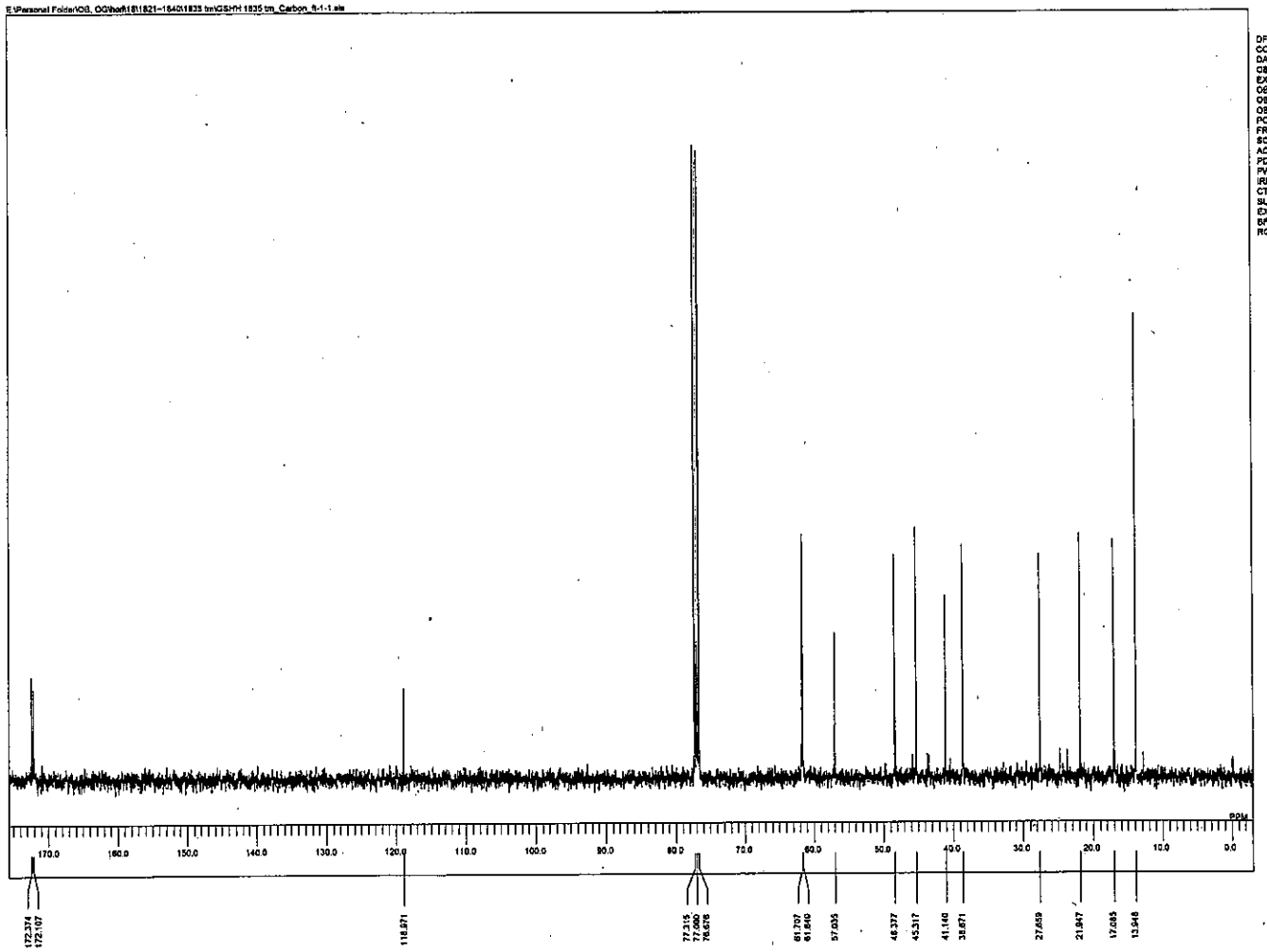

EtO2C
EtO2C
Me
CN
4.00
5.46
0.89
1.07
6.23
2.69
PPM
7.292
7.266
0.000


PPM
172.212
172.107
119.895
77.315
77.200
77.000
76.676
61.583
58.617
40.540
39.338
30.271
25.734
17.933
14.539
13.990


EtO2C
EtO2C
H
CN
Me Me
4.00
0.30
0.76
1.12
2.06
0.69
0.80
0.49
0.81
0.84
0.85
2.50
0.12
PPM
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
7.275
0.000

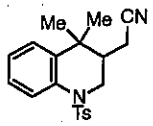

PPM
170.0
160.0
150.0
140.0
130.0
120.0
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0.0
172.342
172.019
124.178
77.326
77.000
76.674
61.591
57.852
51.759
41.352
35.184
33.984
32.737
28.578
25.723
15.287
13.935


Me
Me
EtO2C
EtO2C
CN
PPM

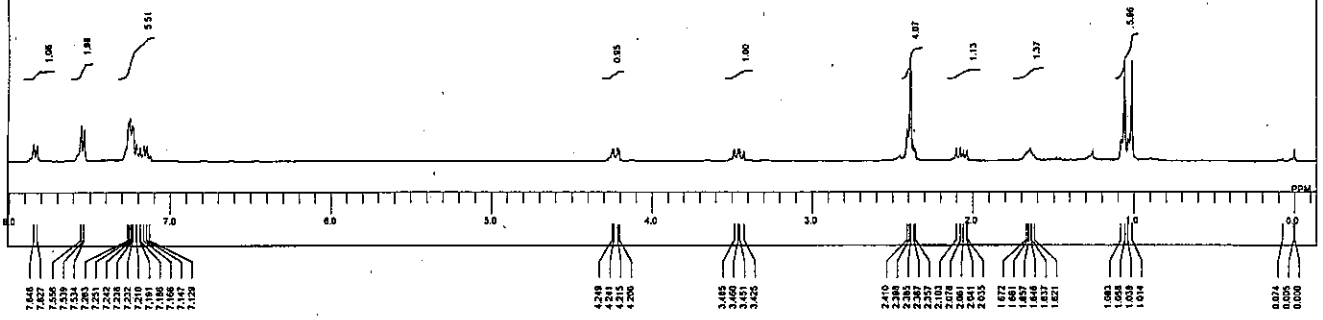

PPM

single_pulse

E:\Personal Folder\OB, OG\Ihor\181661~188015 72 tm\GSHH 18 72 tm_Proton_fi-1-1.als



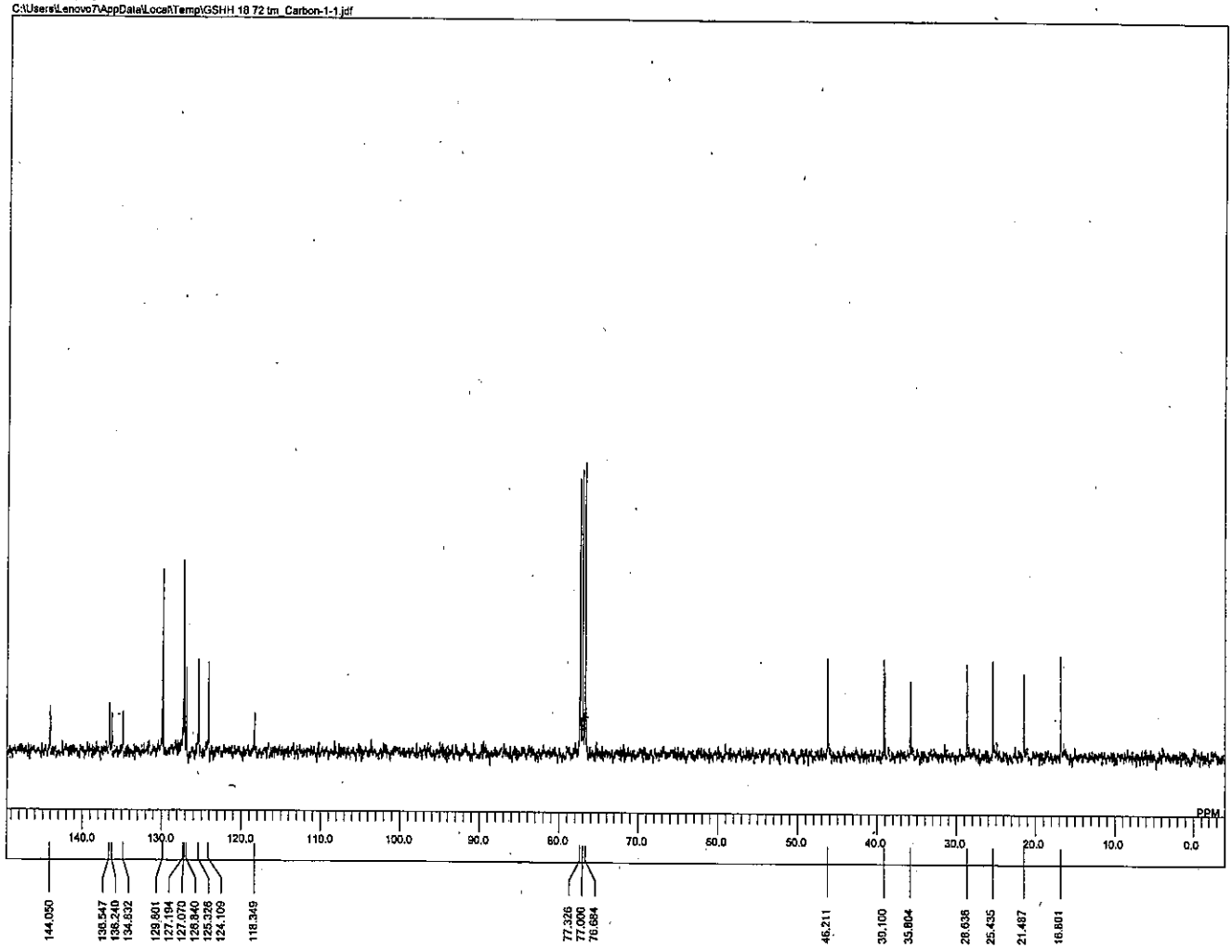
DFILE GSHH 18 72 tm_Proton_fi-1-1
COMNT single_pulse
DATIM 2015-06-29 19:06:32
OBNUC 1H
EXMOD proton.jxp
OBFRQ 399.78 MHz
OBSET 4.19 KHz
OBFIN 7.28 Hz
POINT 18364
FREQU 7494.00 Hz
SCANS 8
ACQTM 2.1883 sec
PD 5.0000 sec
PW1 2.72 usec
IRNUC 1H
CTEMP 23.5 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 3.00 Hz
RGAIN 36

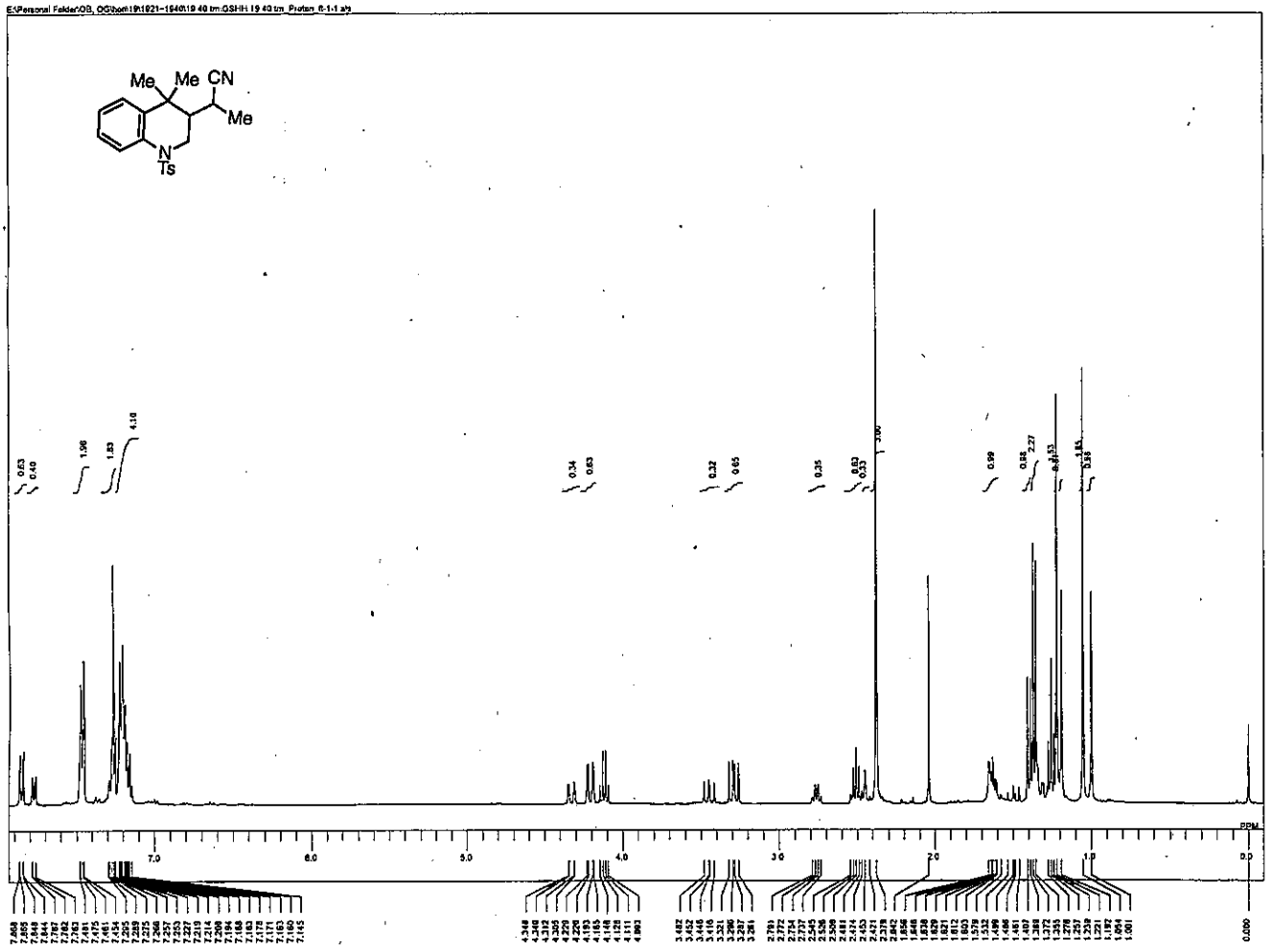


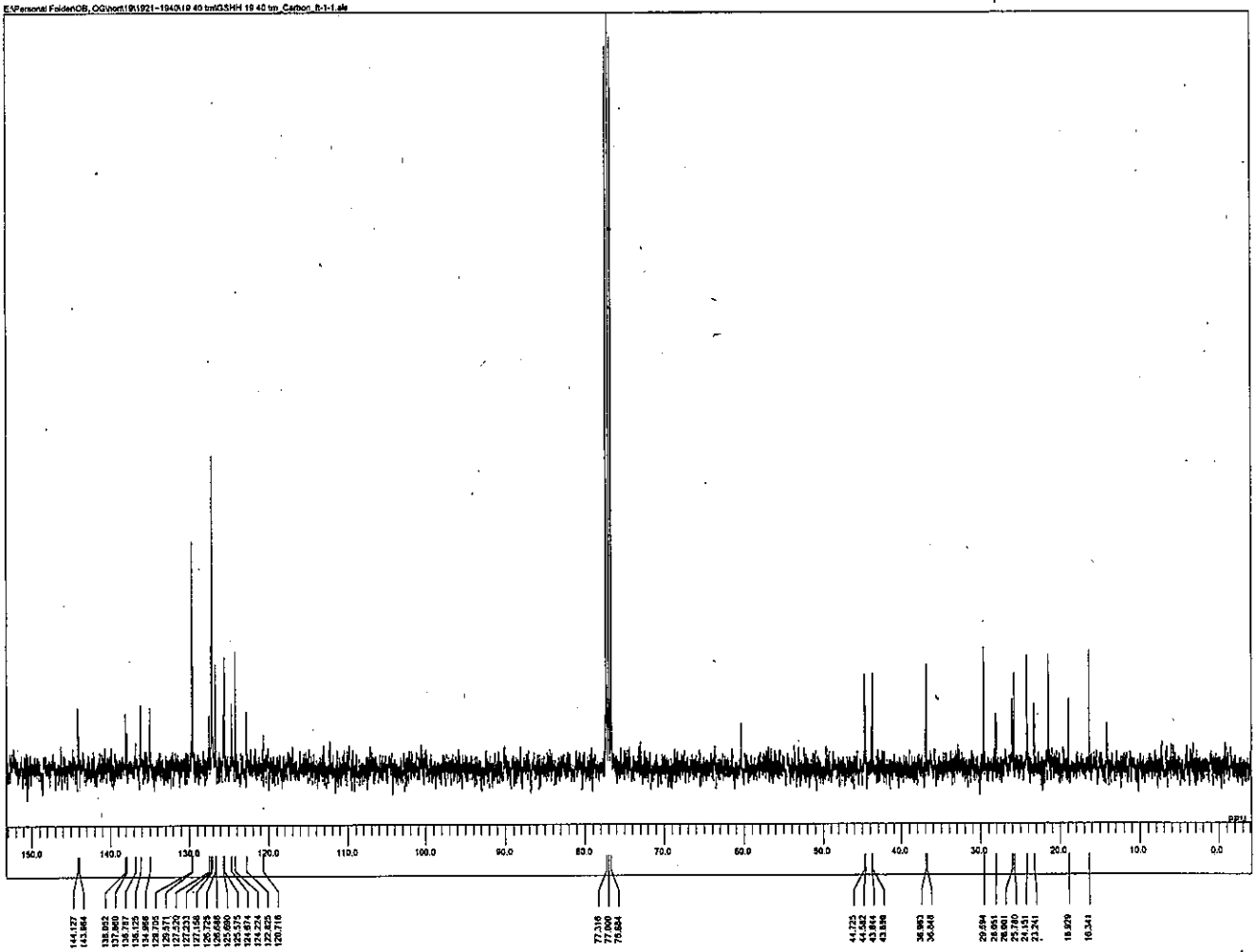
single pulse decoupled gated NOE

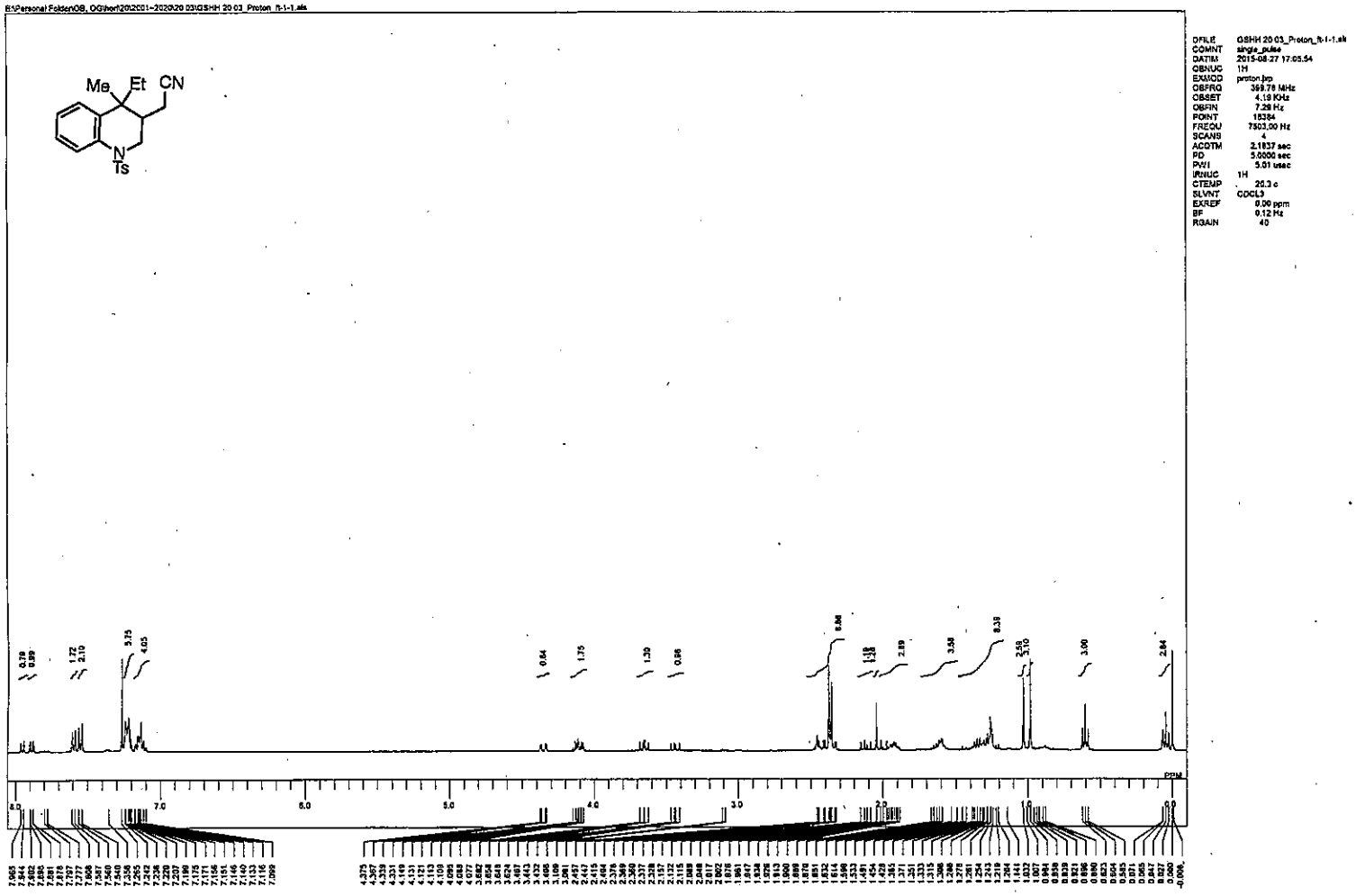
C:\Users\Lenovo7\AppData\Local\Temp\GSHH 18 72 tm_Carbon-1-1.jdf

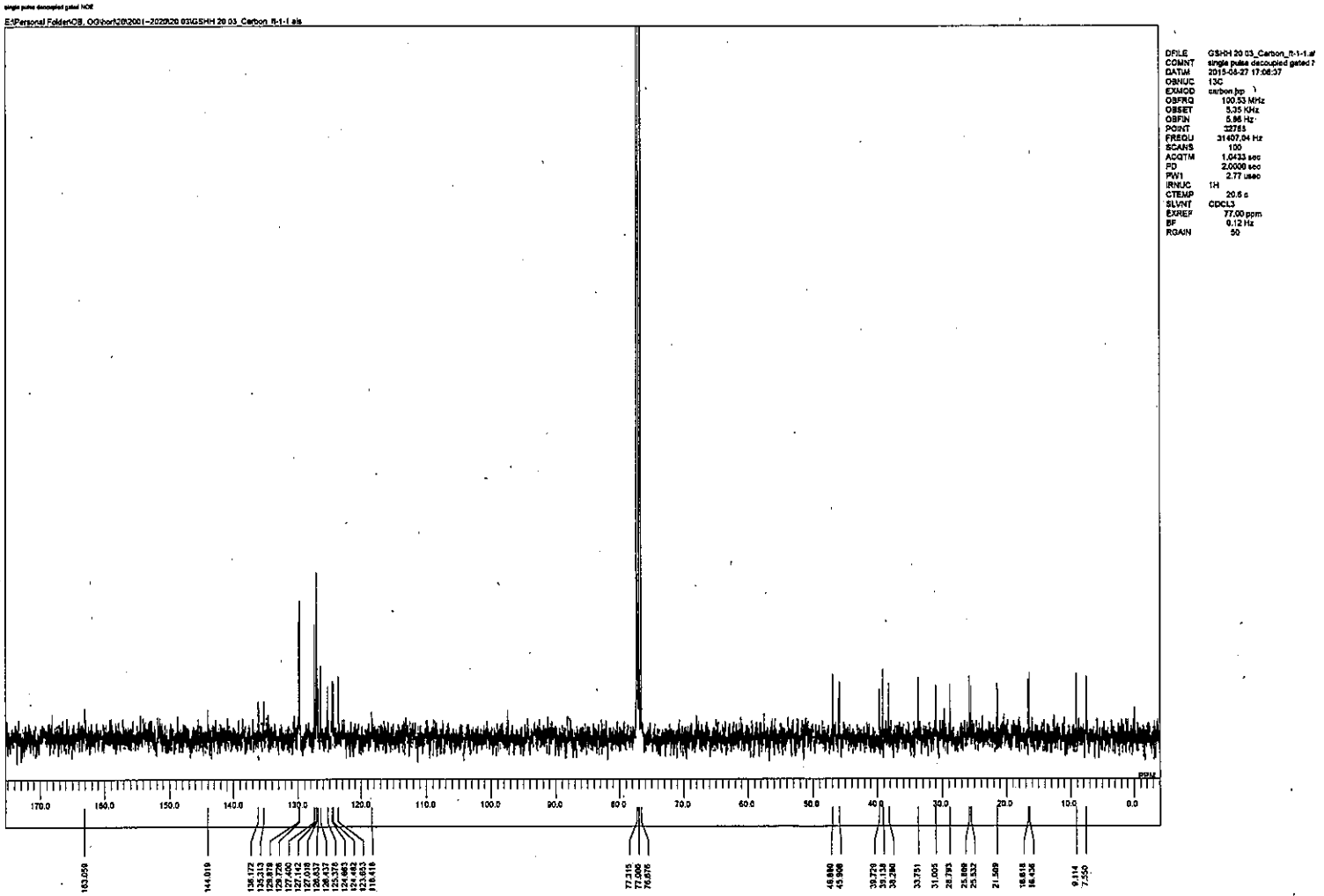
DFILE GSHH 18 72 tm_C
COMNT single pulse decou
DATIM 2015-06-29 19:07:
OBNUC 13C
EXMOD carbon.jxp
OBFRQ 100.53 MHz
OBSET 5.35 KHz
OBFIN 5.86 Hz
POINT 32767
FREQU 31565.66 Hz
SCANS 50
ACQTM 1.0381 sec
PD 2.0000 sec
PW1 3.52 usec
IRNUC 1H
CTEMP 23.4 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 3.00 Hz
RGAIN 50

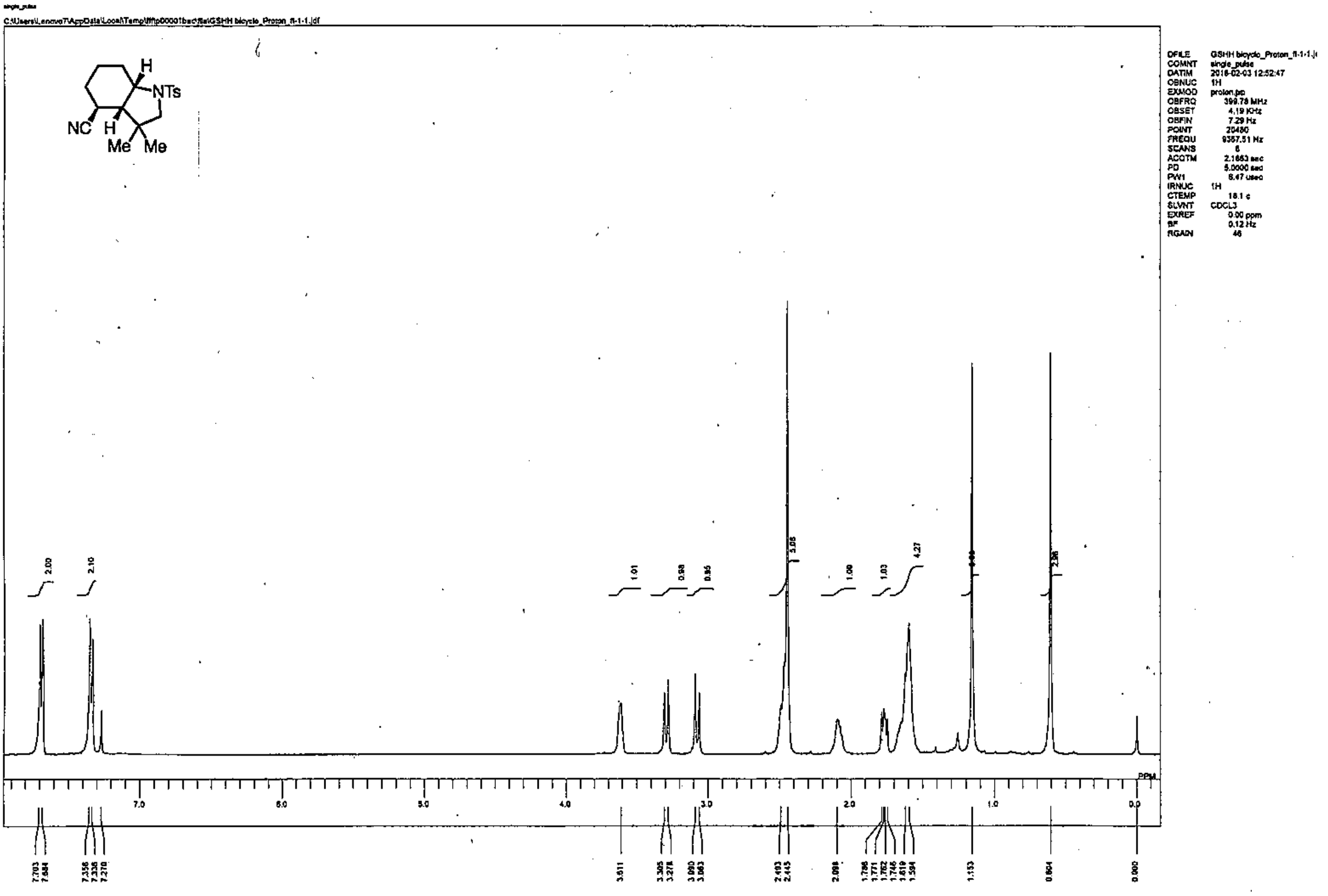


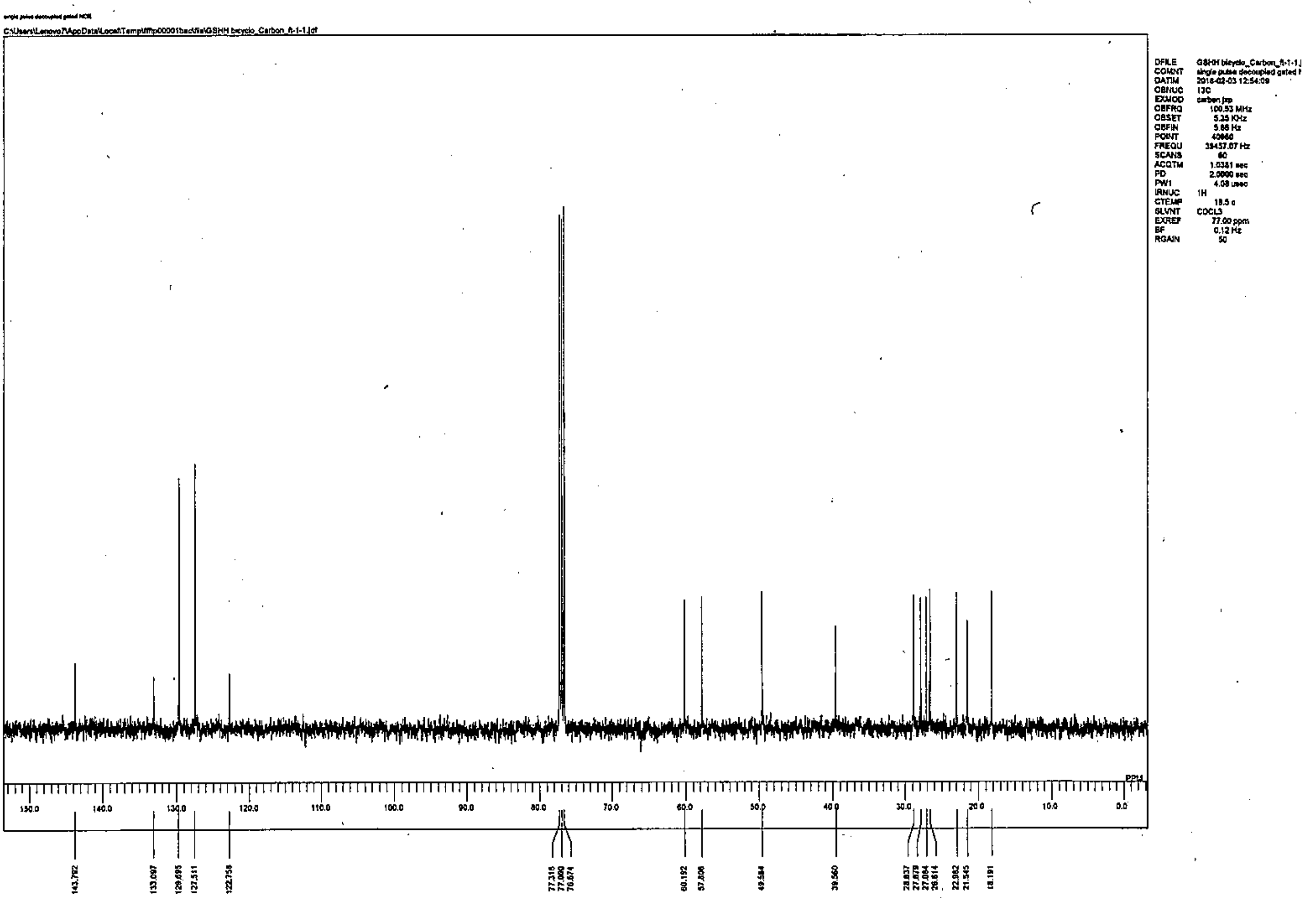

Me
Me
CN
Me
N
Ts
PPM

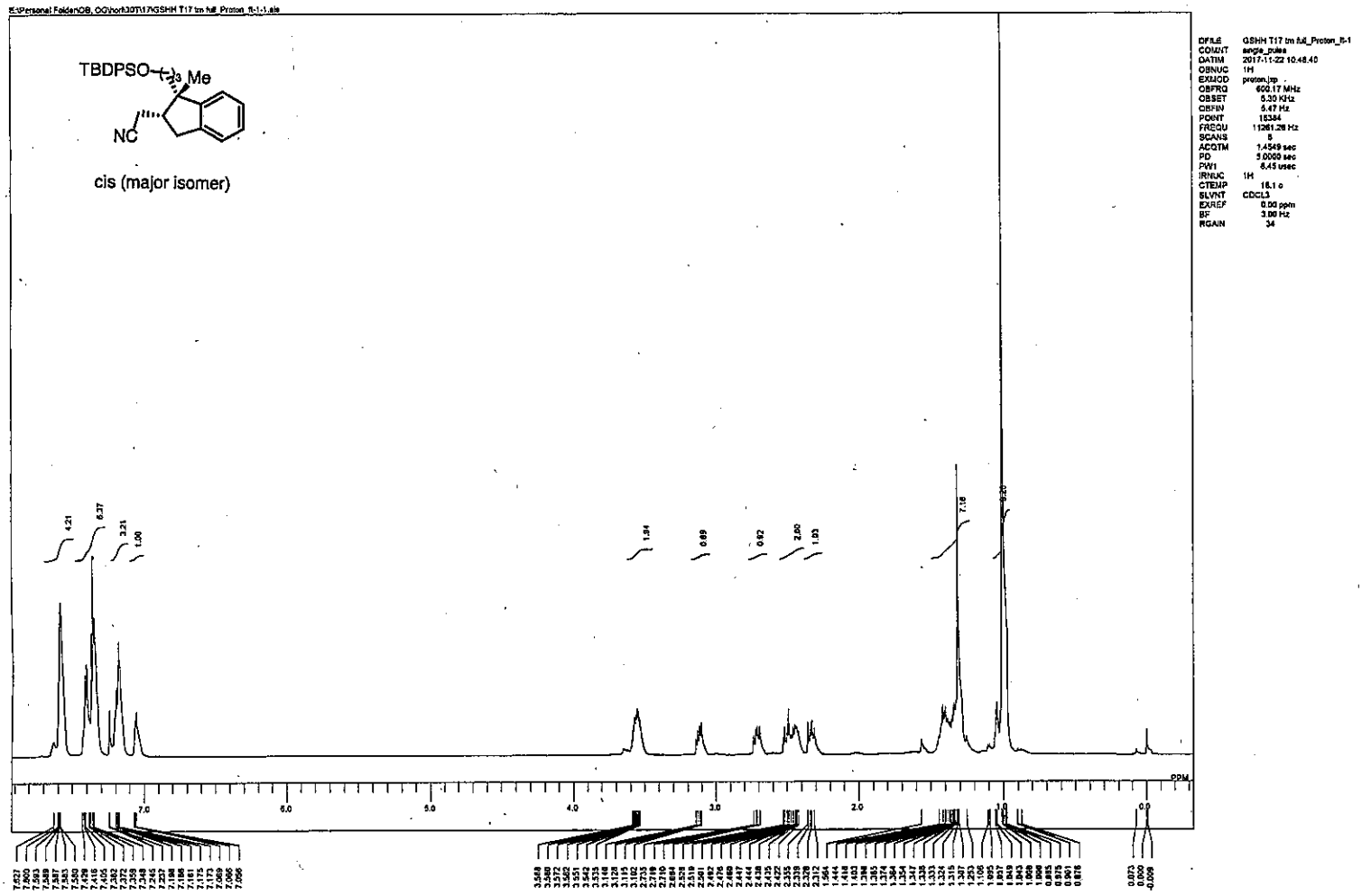

PPM

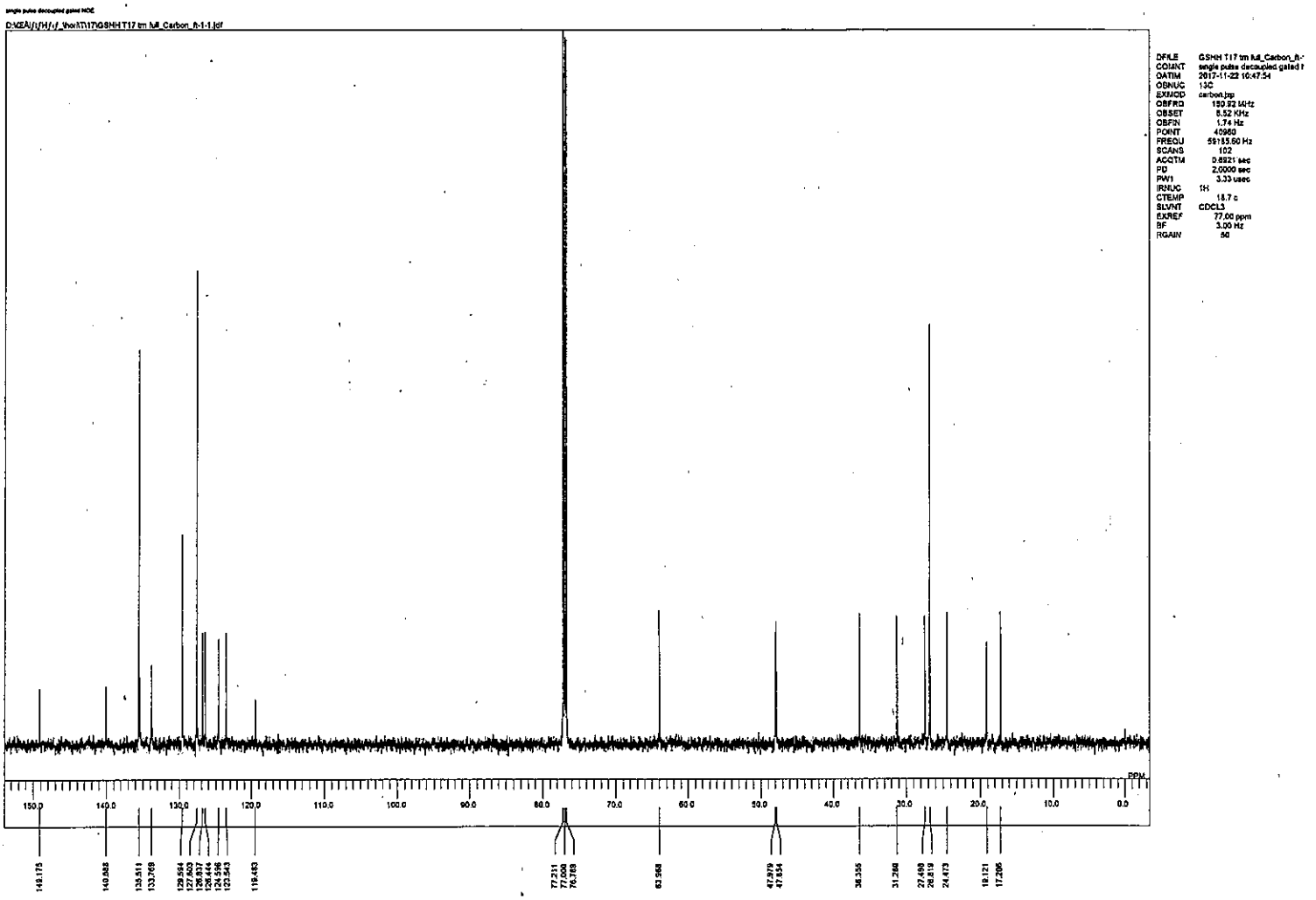

Me
Et
CN
N
Ts
PPM


PPM


single_pulse
C:\Users\Lenovo7\AppData\Local\Temp\ftp00001bac\fis\GSHH bicyclo_Proton_fl-1-1.jdf
NC
H
H
NTs
Me
Me
DFILE GSHH bicyclo_Proton_fl-1-1.j
COMNT single_pulse
DATIM 2016-02-03 12:52:47
OBNUC 1H
EXMOD proton.jxp
OBFRQ 399.78 MHz
OBSET 4.19 KHz
OBFIN 7.29 Hz
POINT 20480
FREQU 9357.51 Hz
SCANS 8
ACQTM 2.1863 sec
PD 5.0000 sec
PW1 6.47 usec
IRNUC 1H
CTEMP 18.1 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 46
2.00
2.10
1.01
0.98
0.95
3.05
1.00
1.03
4.27
2.96
PPM
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
7.703
7.684
7.356
7.336
7.270
3.611
3.305
3.278
3.090
3.063
2.463
2.445
2.096
1.786
1.771
1.762
1.746
1.619
1.594
1.153
0.604
0.000

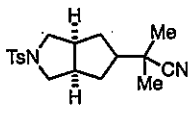

single pulse decoupled gated NOE
C:\Users\Lenovo7\AppData\Local\Temp\ftp00001bac\fis\GSHH bicyclo_Carbon_fl-1-1.jdf
DFILE GSHH bicyclo_Carbon_fl-1-1.j
COMNT single pulse decoupled gated
DATIM 2016-02-03 12:54:09
OBNUC 13C
EXMOD carbon.jxp
OBFRQ 100.53 MHz
OBSET 5.25 KHz
OBFIN 5.86 Hz
POINT 40960
FREQU 33457.07 Hz
SCANS 60
ACQTM 1.0381 sec
PD 2.0000 sec
PW1 4.08 usec
IRNUC 1H
CTEMP 18.5 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 50
PPM
150.0
140.0
130.0
120.0
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0.0
143.792
133.097
129.695
127.511
122.759
77.315
77.000
76.674
60.182
57.806
49.584
39.560
28.637
27.678
27.064
26.614
22.982
21.545
18.191


TBDPSO
Me
NC
cis (major isomer)
DFILE GSHH T17 tm full_Proton_fi-1
COMNT single_pulse
DATIM 2017-11-22 10:48:40
OBNUC 1H
EXMOD proton.jxp
OBFRQ 600.17 MHz
OBSET 5.39 KHz
OBFIN 5.47 Hz
POINT 16384
FREQU 11261.26 Hz
SCANS 8
ACQTM 1.4549 sec
PD 5.0000 sec
PW1 8.45 usec
IRNUC 1H
CTEMP 18.1 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 3.00 Hz
RGAIN 34
4.21
6.37
3.21
1.00
1.84
0.89
0.92
2.00
1.93
7.16
6.20
PPM

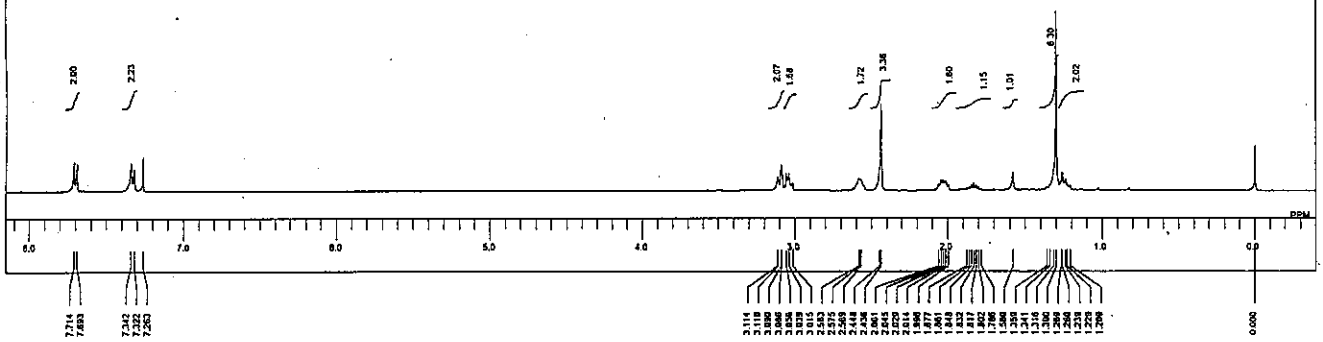

DFILE GSHH T17 tm full_Carbon_fi-
COMNT single pulse decoupled gated
DATIM 2017-11-22 10:47:54
OBNUC 13C
EXMOD carbon.jxp
OBFRQ 150.92 MHz
OBSET 8.52 KHz
OBFIN 1.74 Hz
POINT 40960
FREQU 59155.60 Hz
SCANS 102
ACQTM 0.8521 sec
PD 2.0000 sec
PW1 3.33 usec
IRNUC 1H
CTEMP 18.7 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 3.00 Hz
RGAIN 50
PPM

single_pulse

E:\Personal Folder\DS, GS\hoit24\GSHH 24 57 37 39_Proton_ft-1-1.als



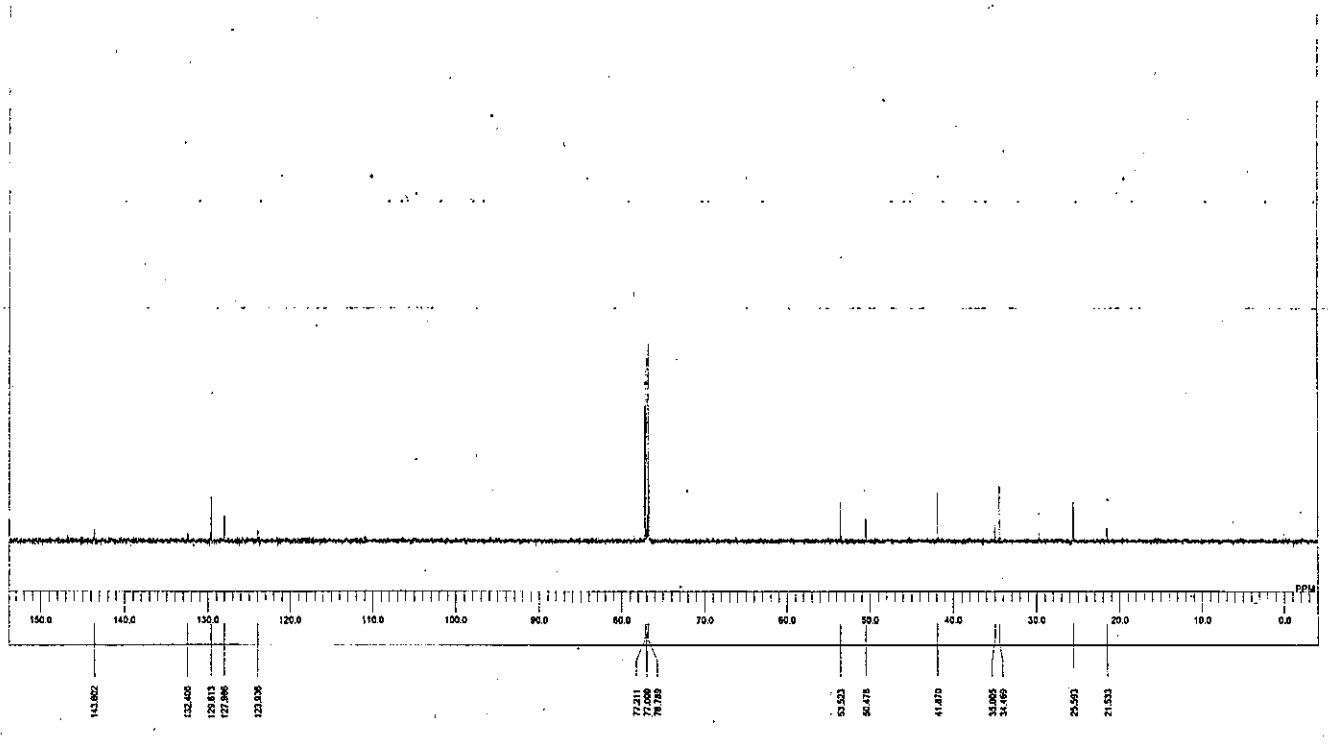
DFILE GSHH 24 57 37 39_Proton_ft-
COMNT single_pulse
DATIM 2016-08-03 19:45:32
OBNUC 1H
EXMOD proton.jxp
OBFRQ 399.78 MHz
OBSET 4.19 KHz
OBFIN 7.29 Hz
POINT 16384
FREQU 7494.00 Hz
SCANS 4
ACQTM 2.1863 sec
PD 5.0000 sec
PW1 2.72 usec
INNUC 1H
CTEMP 22.4 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 58



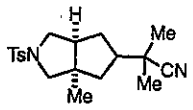
single pulse decoupled gated NOE

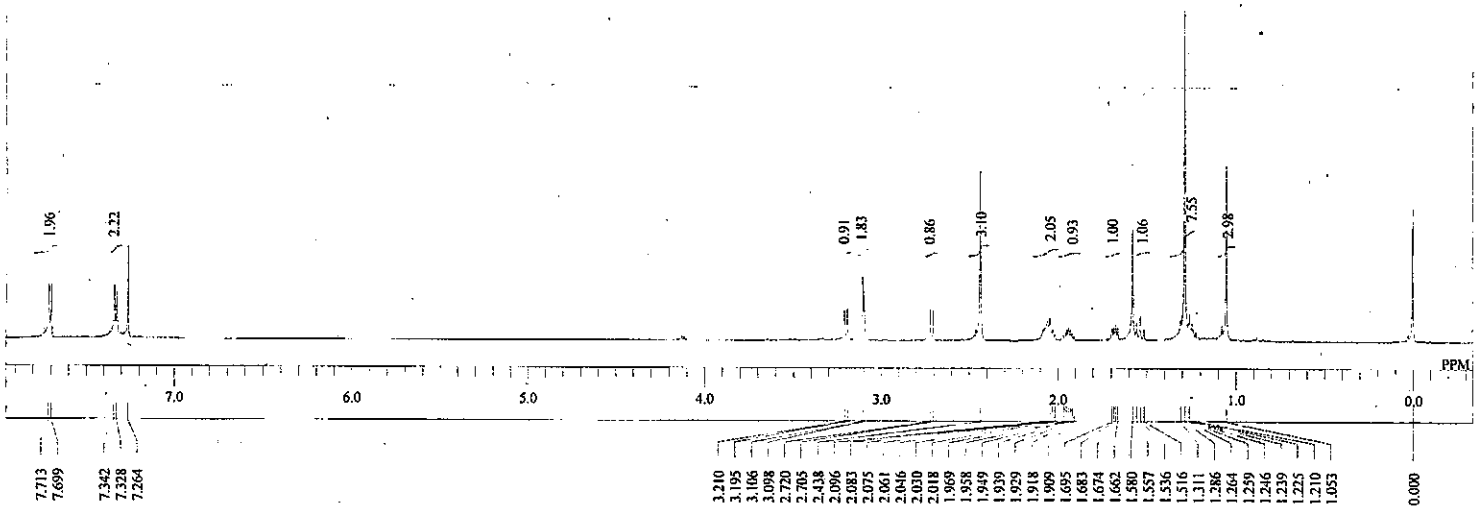
C:\Users\Lenovo7\AppData\Local\Temp\ffftp000023\hoit\GSHH 24 57 tm rec_Carbon-1-1.jdf

DFILE GSHH 24 57 tm rec_Carbo
COMNT single pulse decoupled gate
DATIM 2016-08-13 15:44:46
OBNUC 13C
EXMOD carbon.jxp
OBFRQ 150.92 MHz
OBSET 8.52 KHz
OBFIN 1.74 Hz
POINT 32767
FREQU 47348.48 Hz
SCANS 200
ACQTM 0.6921 sec
PD 2.0000 sec
PW1 3.21 usec
INNUC 1H
CTEMP 22.4 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 50

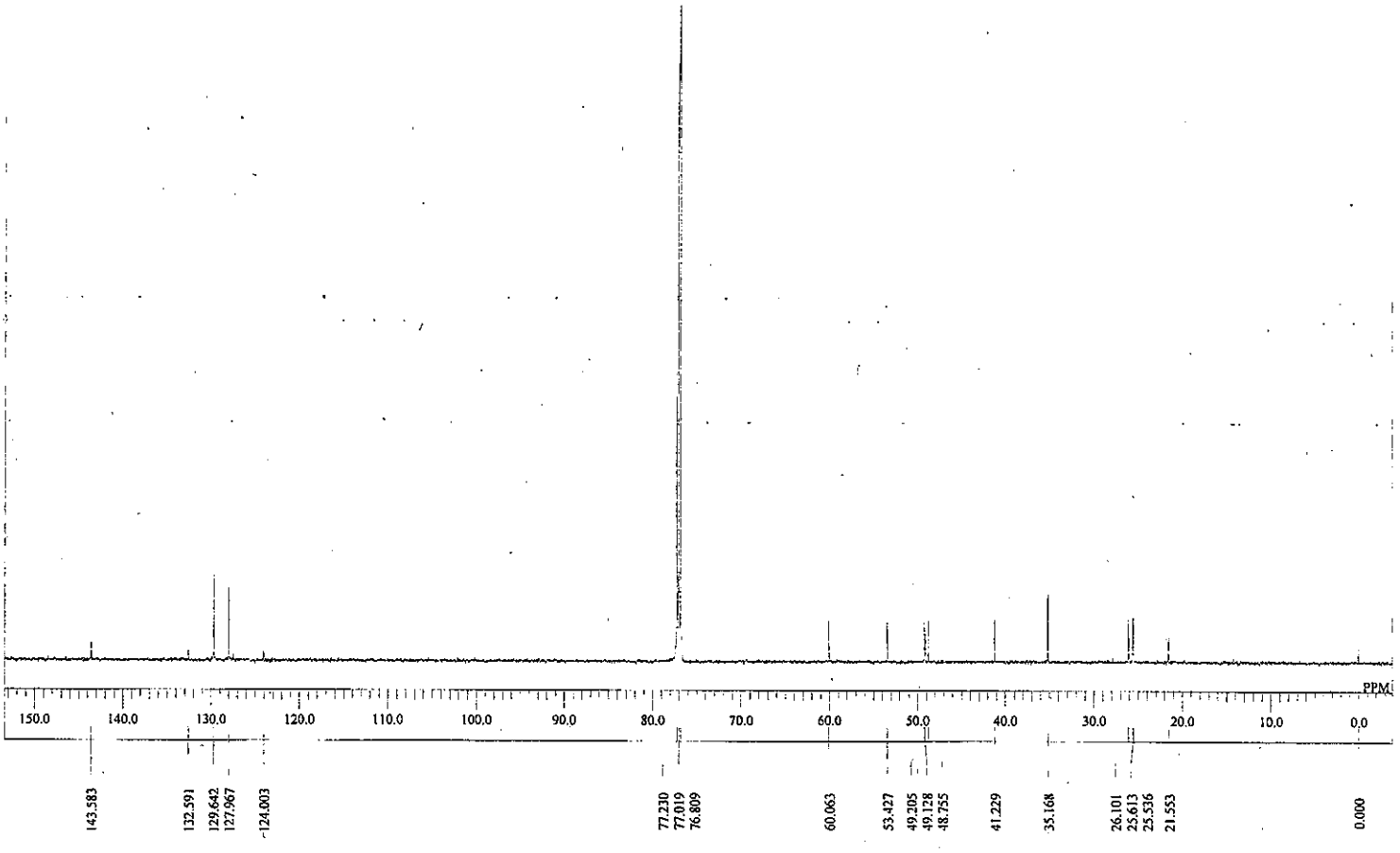


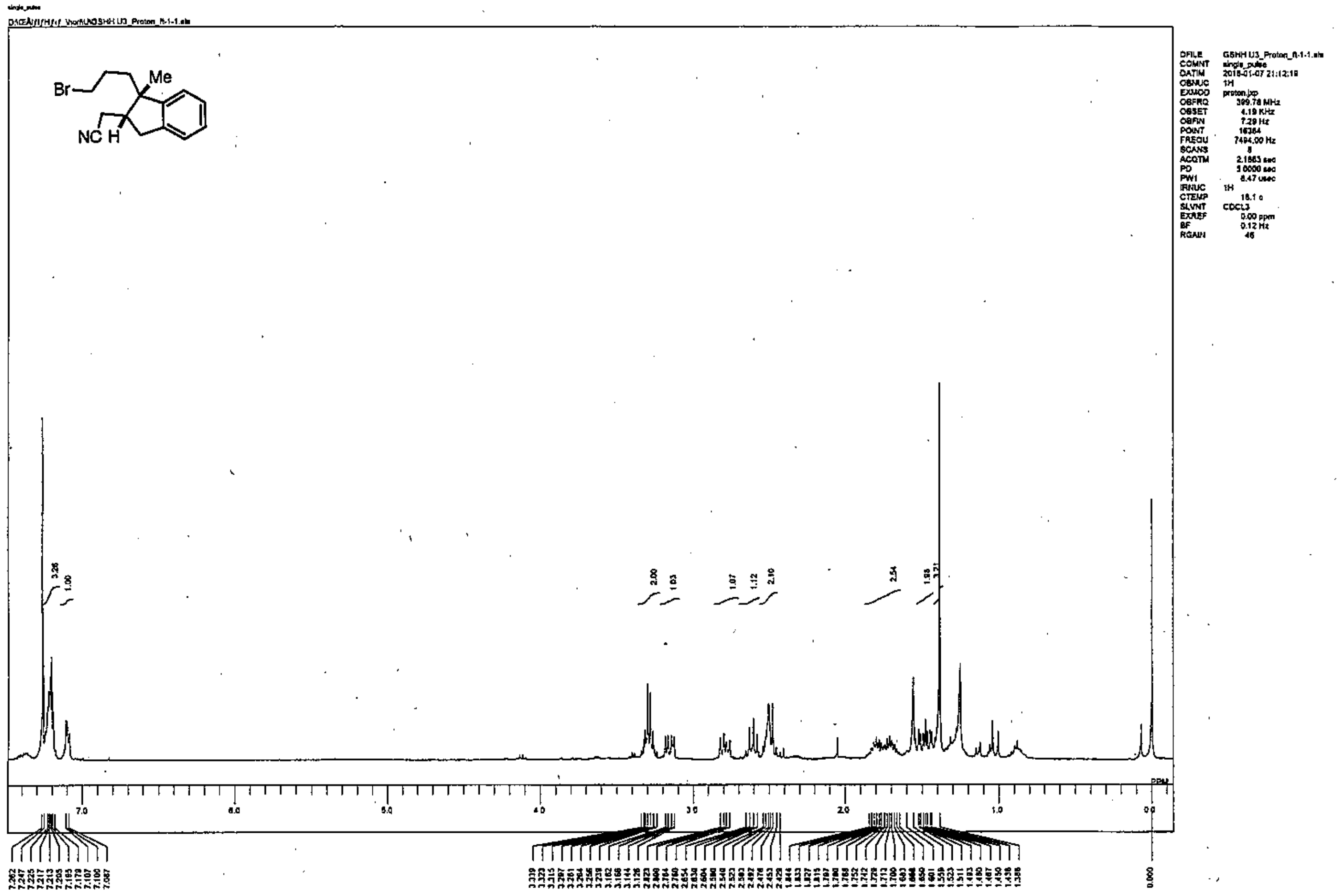
single_pulse
C:\Users\Lenovo7\AppData\Local\Temp\fffp00001e98\file\GSHH 25 42 re_Proton_ft-1-1.jdf



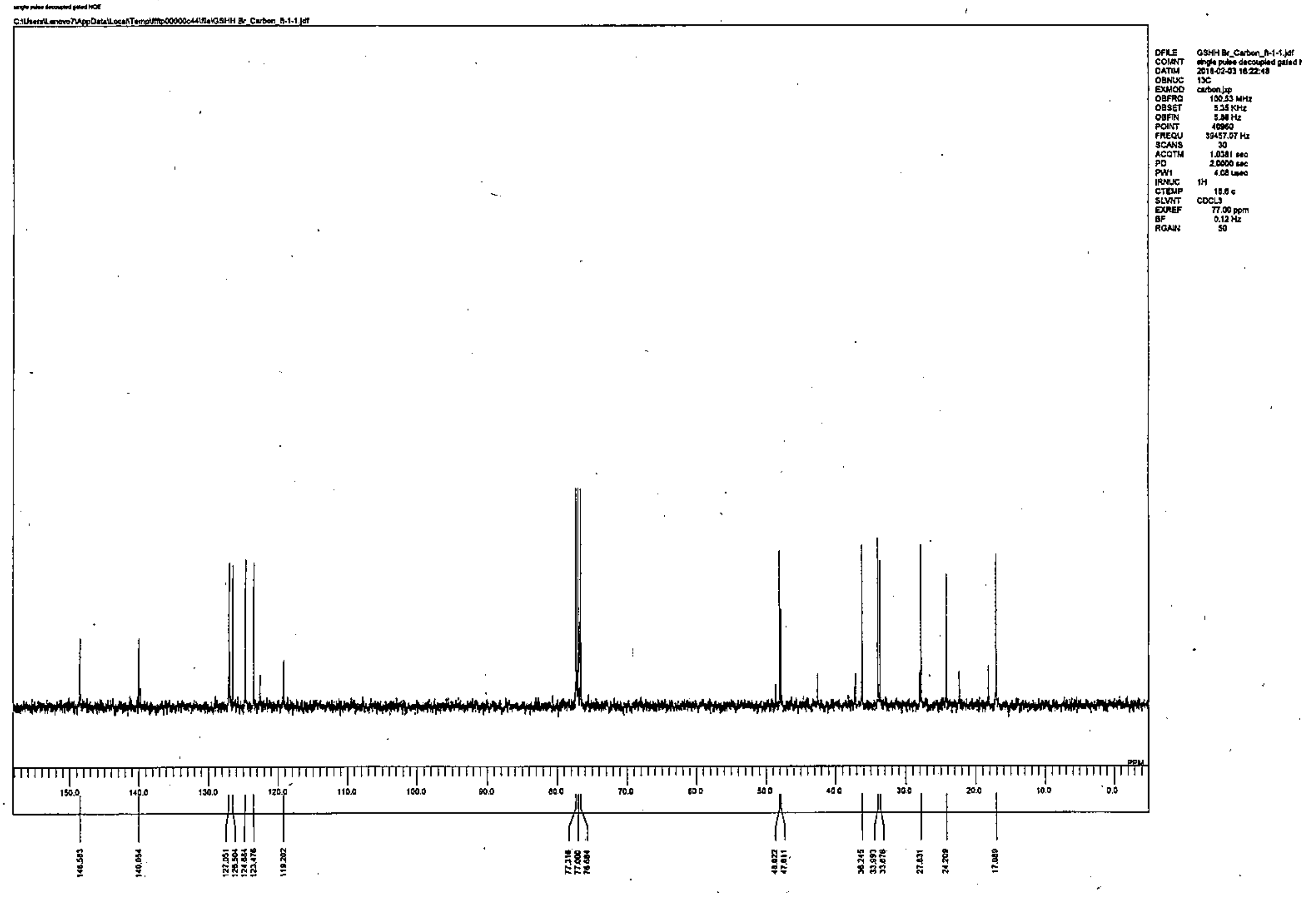


single pulse decoupled gated NOE
C:\Users\Lenovo7\AppData\Local\Temp\fffp00001e98\file\GSHH 25 42 re_Carbon_ft-1-1.jdf





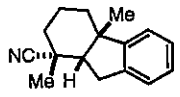
Br
Me
NC H
DFILE GSHH U3_Proton_ft-1-1.als
COMNT single_pulse
OBNUC 1H
SLVNT CDCL3
EXREF 0.00 ppm
3.26
1.00
2.00
1.03
1.07
1.12
2.10
2.54
1.98
3.21
PPM



DFILE GSHH Br_Carbon_ft-1-1.jdf
OBNUC 13C
SLVNT CDCL3
EXREF 77.00 ppm
PPM
146.583
140.054
127.251
126.504
124.684
123.476
119.202
77.316
77.000
76.684
48.622
47.811
36.245
33.993
33.676
27.831
24.209
17.089

single_pulse

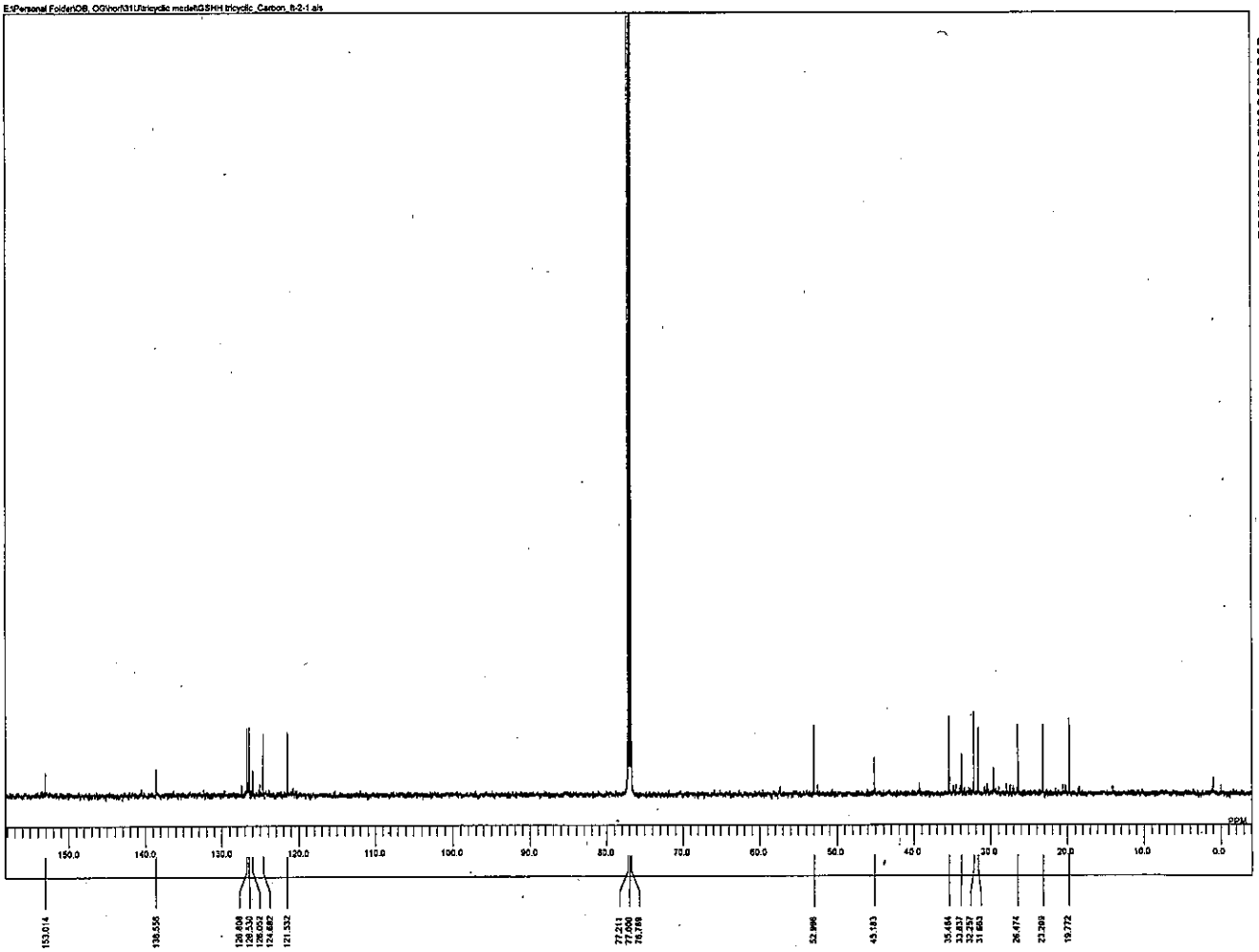
E:\Personal Folder\OB, OG\non\31\Tricyclic model\GSHH tricyclic_Proton_ft-1-1.als



DFILE GSHH tricyclic_Proton_ft-1-1.
COMNT single_pulse
DATIM 2016-02-05 12:45:14
OBNUC 1H
EXMOD proton.jxp
OBFRQ 600.17 MHz
OBSET 5.30 KHz
OBFIN 5.47 Hz
POINT 16384
FREQU 11261.26 Hz
SCANS 8
ACQTM 1.4549 sec
PD 5.0000 sec
PW1 8.45 usec
IRNUC 1H
CTEMP 16.4 c
SLVNT CDCl3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 42

single pulse decoupled gated NOE

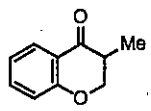
E:\Personal Folder\OB, OG\non\31\Tricyclic model\GSHH tricyclic_Carbon_ft-2-1.als



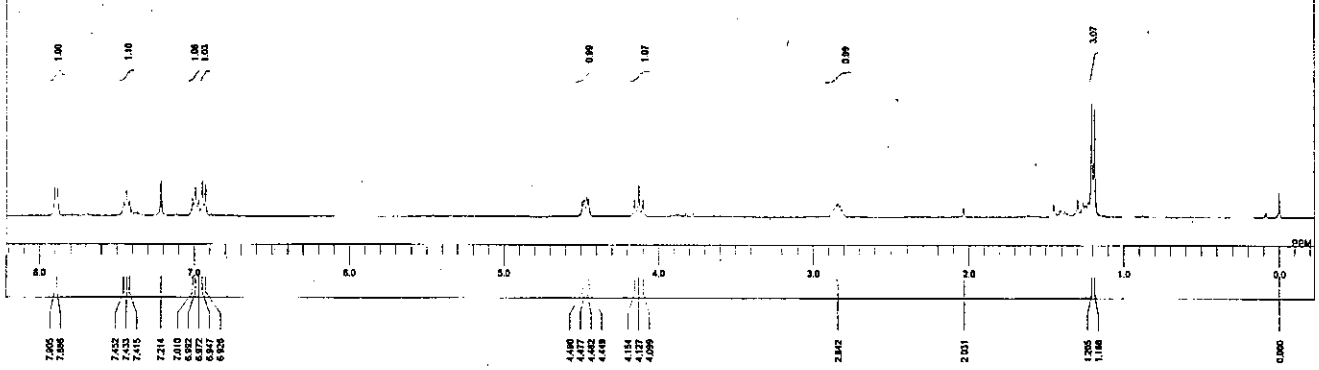
DFILE GSHH tricyclic_Carbon_ft-2-1.
COMNT single pulse decoupled gated
DATIM 2016-02-05 12:51:12
OBNUC 13C
EXMOD carbon.jxp
OBFRQ 150.92 MHz
OBSET 6.52 KHz
OBFIN 1.74 Hz
POINT 32768
FREQU 47348.48 Hz
SCANS 560
ACQTM 0.6921 sec
PD 2.0000 sec
PW1 3.33 usec
IRNUC 1H
CTEMP 17.4 c
SLVNT CDCl3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 50

single_pulse

C:\Users\Lenovo7\AppData\Local\Temp\GSHH 22 04_Proton_ft-1-1.jdf



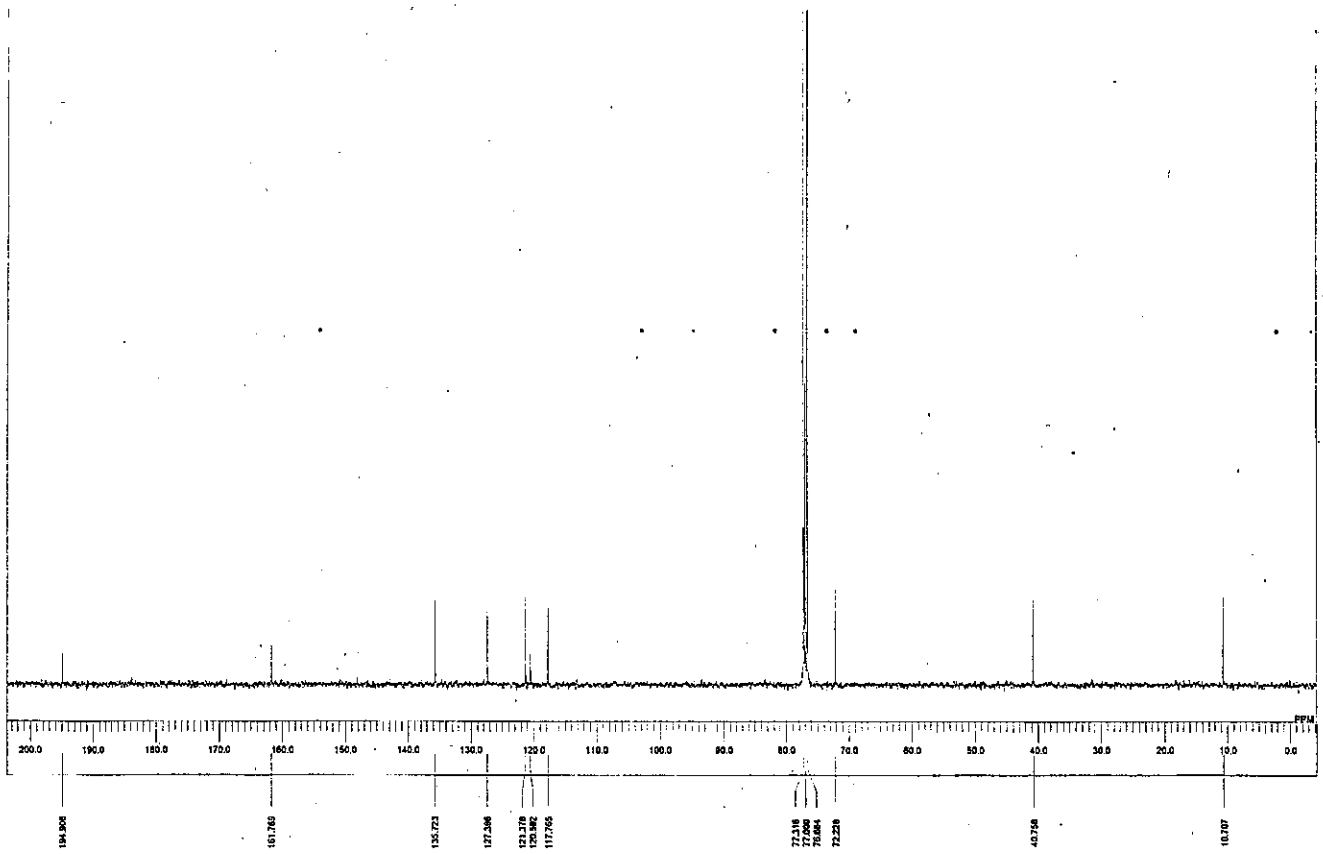

```
DFILE   GSHH 22 04_Proton_ft-1-
COMNT   single_pulse
DATIM   2015-12-09 20:32:48
OBNUC   1H
EXMOD   proton.jxp
OBFRQ     399.78 MHz
OBSET       4.19 KHz
OBFIN       7.29 Hz
POINT      20480
FREQU    9367.51 Hz
SCANS          8
ACQTM      2.1863 sec
PD         5.0000 sec
PW1        2.72 usec
IRNUC      1H
CTEMP      20.7 c
SLVNT      CDCL3
EXREF      0.00 ppm
BF         0.12 Hz
RGAIN        36
```



single pulse decoupled gated NOE

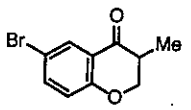
C:\Users\Lenovo7\AppData\Local\Temp\GSHH 22 04_Carbon_ft-1-1.jdf

```
DFILE   GSHH 22 04_Carbon_ft-1
COMNT   single pulse decoupled gat
DATIM   2015-12-09 20:34:08
OBNUC   13C
EXMOD   carbon.jxp
OBFRQ     100.53 MHz
OBSET       5.35 KHz
OBFIN       5.85 Hz
POINT      40960
FREQU    39457.97 Hz
SCANS        100
ACQTM      1.0381 sec
PD         2.0000 sec
PW1        3.52 usec
IRNUC      1H
CTEMP      29.8 c
SLVNT      CDCL3
EXREF      77.00 ppm
BF         0.12 Hz
RGAIN        30
```



single_pulse

C:\Users\Lenovo7\AppData\Local\Temp\GSHH 22 59 tm pure_Proton_ft-1-1.jdf

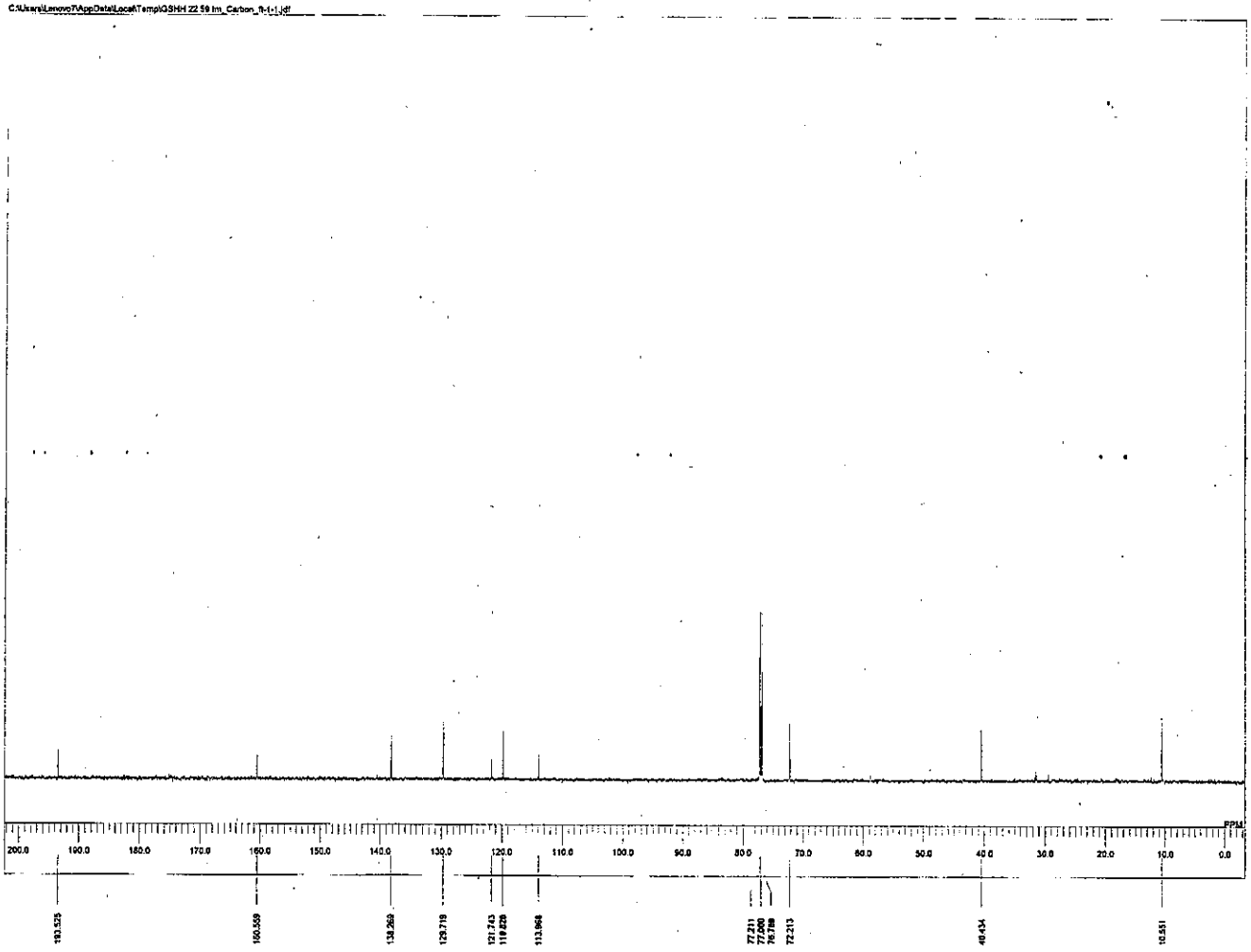


DFILE GSHH 22 59 tm pure_Pro
COMNT single_pulse
DATIM 2018-01-19 22:15:36
OBNUC 1H
EXMOD proton.jxp
OBFRQ 399.78 MHz
OBSET 4.19 KHz
OBFIN 7.29 Hz
POINT 26480
FREQU 9367.51 Hz
SCANS 8
ACQTM 2.1863 sec
PD 5.0000 sec
PW1 2.72 usec
IRNUC 1H
CTEMP 18.0 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 66

single pulse decoupled gated NOE

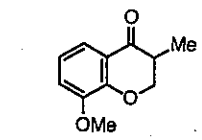
C:\Users\Lenovo7\AppData\Local\Temp\GSHH 22 59 tm_Carbon_ft-1-1.jdf

DFILE GSHH 22 59 tm_Carbon_
COMNT single pulse decoupled ga
DATIM 2018-01-19 19:00:13
OBNUC 13C
EXMOD carbon.jxp
OBFRQ 150.92 MHz
OBSET 8.52 KHz
OBFIN 1.74 Hz
POINT 40960
FREQU 59185.50 Hz
SCANS 100
ACQTM 0.6921 sec
PD 2.0000 sec
PW1 3.40 usec
IRNUC 1H
CTEMP 18.4 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 50

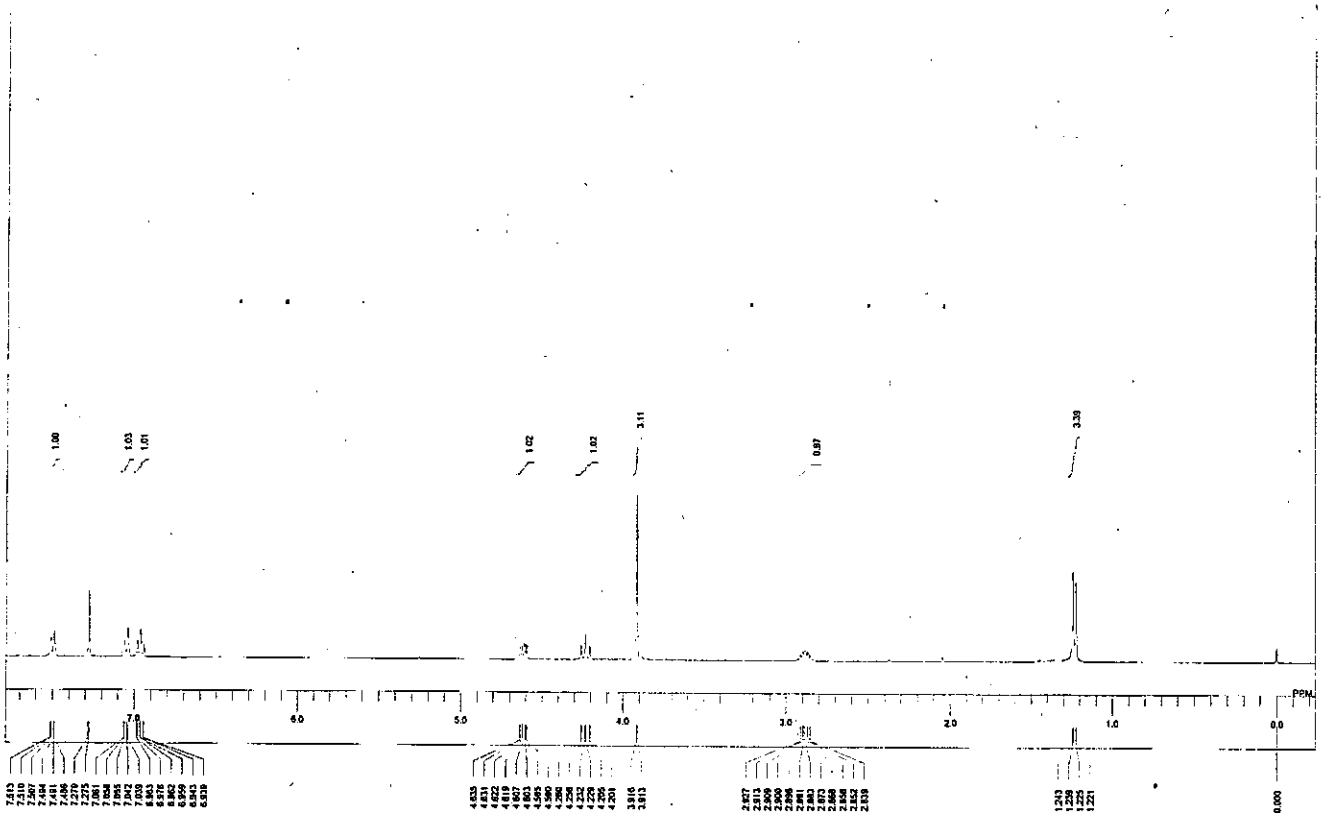


single_pulse

C:\Users\Lenovo7\AppData\Local\Temp\GSHH 22 60 tm_Proton_ft-1-1.jdf



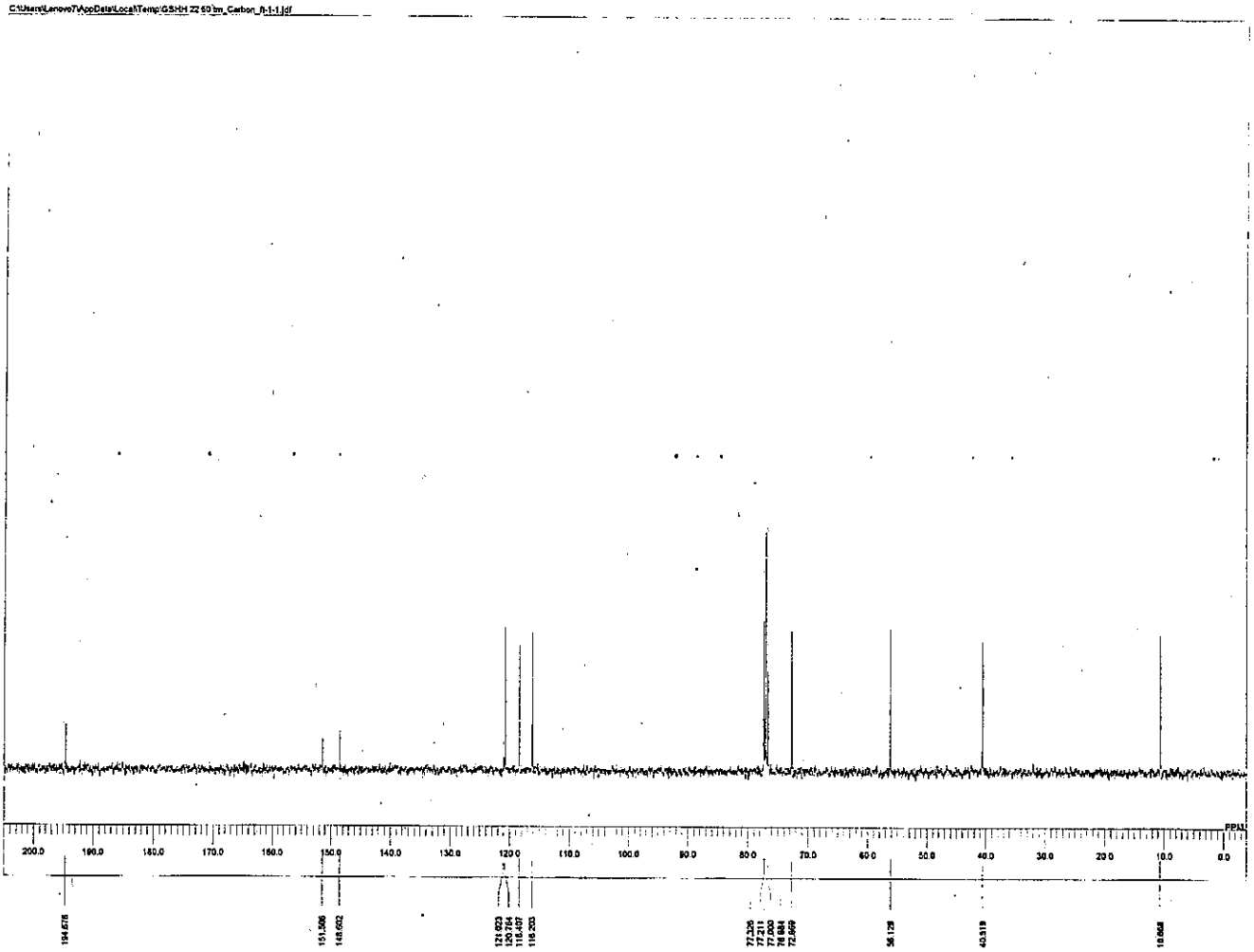

```
DFILE   GSHH 22 60 tm_Proton_f
COMNT   single_pulse
DATIM   2015-01-20 12:17:21
OBNUC   1H
EXMOD   proton.jxp
OBFRQ     399.78 MHz
OBSET       4.19 KHz
OBFIN       7.28 Hz
POINT      25480
FREQU    9387.51 Hz
SCANS          8
ACQTM      2.1663 sec
PD         5.0000 sec
PW1        2.72 usec
IRNUC      1H
CTEMP      17.6 c
SLVNT      CDCL3
EXREF      0.00 ppm
RF         0.12 Hz
RGAIN        36
```



single pulse decoupled gated NOE

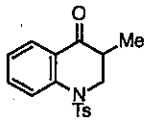
C:\Users\Lenovo7\AppData\Local\Temp\GSHH 22 60 tm_Carbon_ft-1-1.jdf

```
DFILE   GSHH 22 60 tm_Carbon_
COMNT   single pulse decoupled ga
DATIM   2015-01-20 12:18:41
OBNUC   13C
EXMOD   carbon.jxp
OBFRQ     100.53 MHz
OBSET       5.35 KHz
OBFIN       5.86 Hz
POINT      40960
FREQU    39457.07 Hz
SCANS         50
ACQTM      1.0381 sec
PD         2.0000 sec
PW1        3.52 usec
IRNUC      1H
CTEMP      17.8 c
SLVNT      CDCL3
EXREF      77.00 ppm
RF         0.12 Hz
RGAIN        50
```

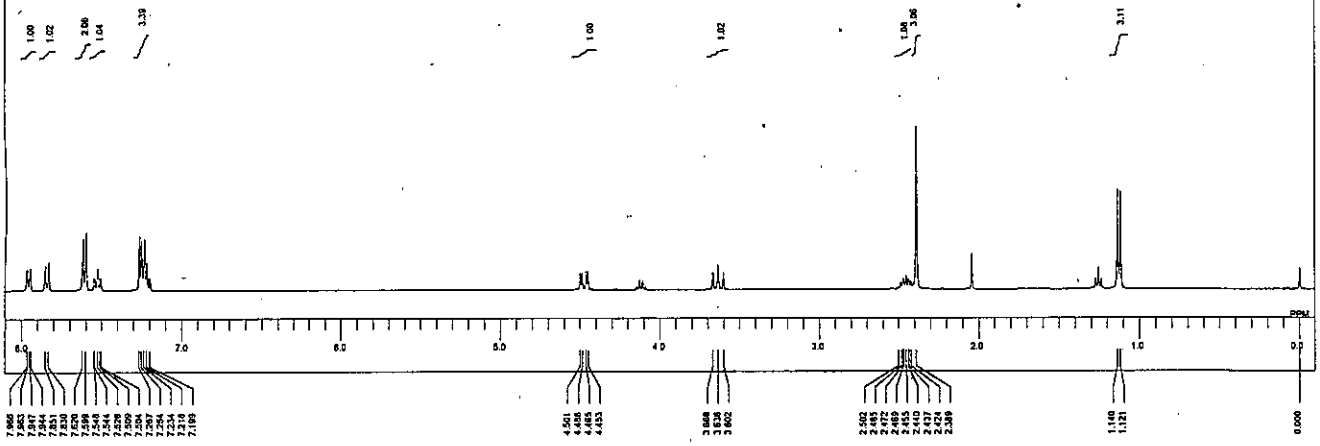


single_pulse

E:\Personal Folder\OB_OG\hari\21\41-60\GSHH 21 50_Proton_fl-1-1.als

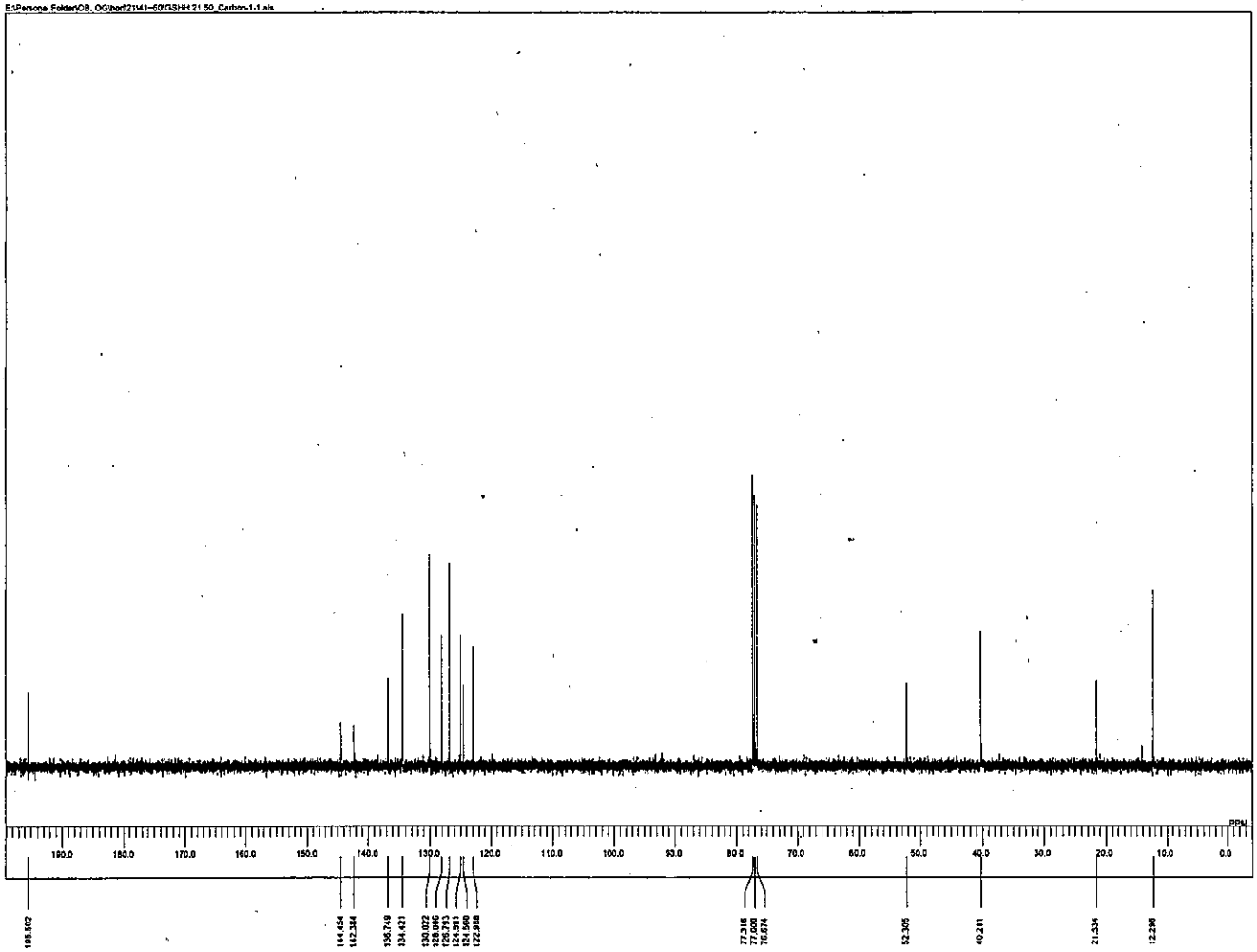


DFILE GSHH 21 50_Proton_fl-1-1.als
COMNT single_pulse
DATIM 2019-11-25 21:32:23
OBNUC 1H
EXMOD proton.jxp
OBFRQ 399.78 MHz
OBSET 4.19 KHz
OBFIN 7.29 Hz
POINT 16384
FREQU 7494.00 Hz
SCANS 8
ACQTM 2.1853 sec
PD 5.0000 sec
PW1 2.72 usec
IRNUC 1H
CTEMP 22.3 c
SLVNT CDCl3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 48



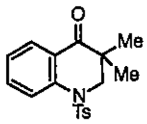
single pulse decoupled gated NOE

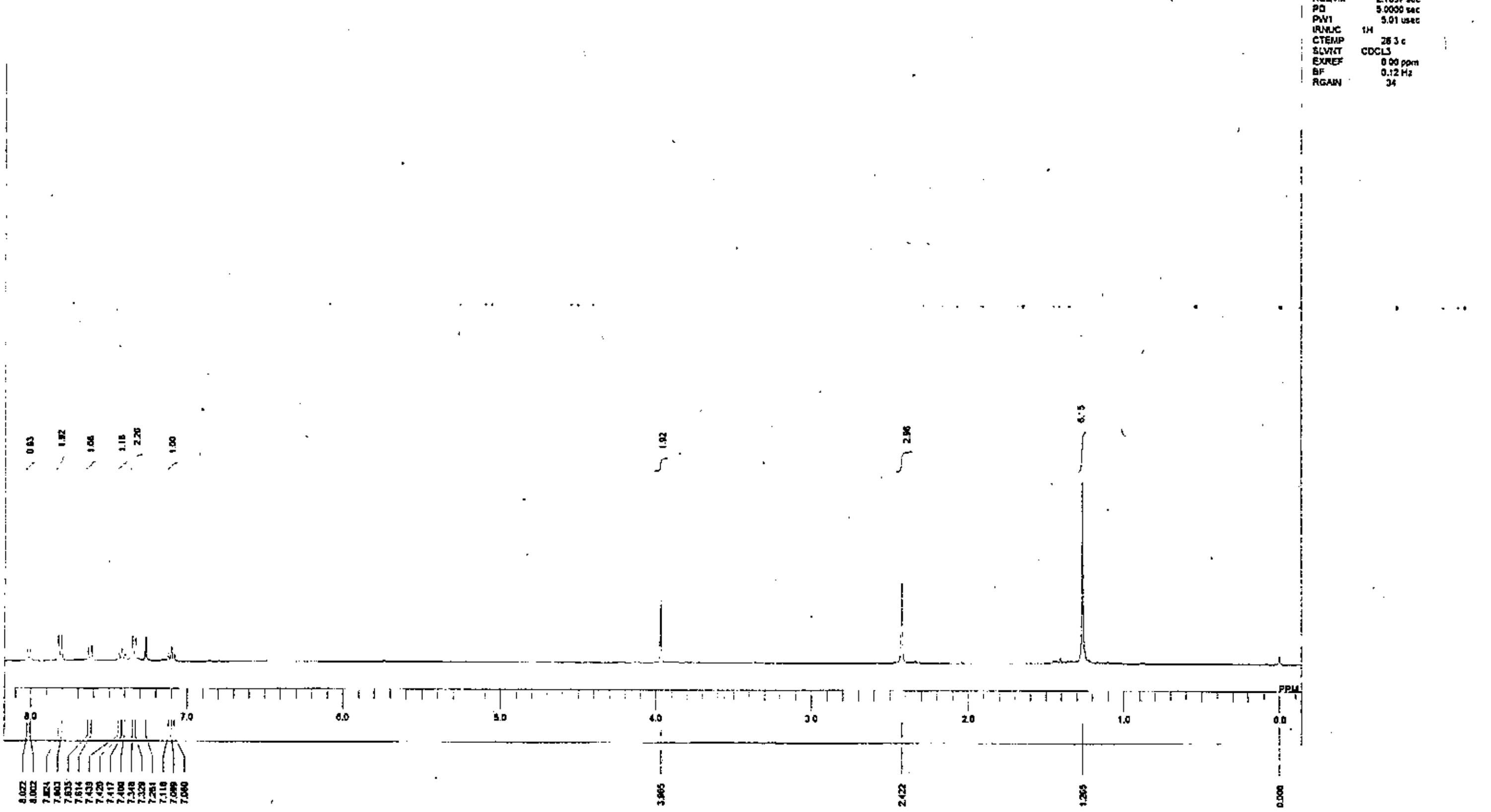
E:\Personal Folder\OB_OG\hari\21\41-60\GSHH 21 50_Carbon-1-1.als



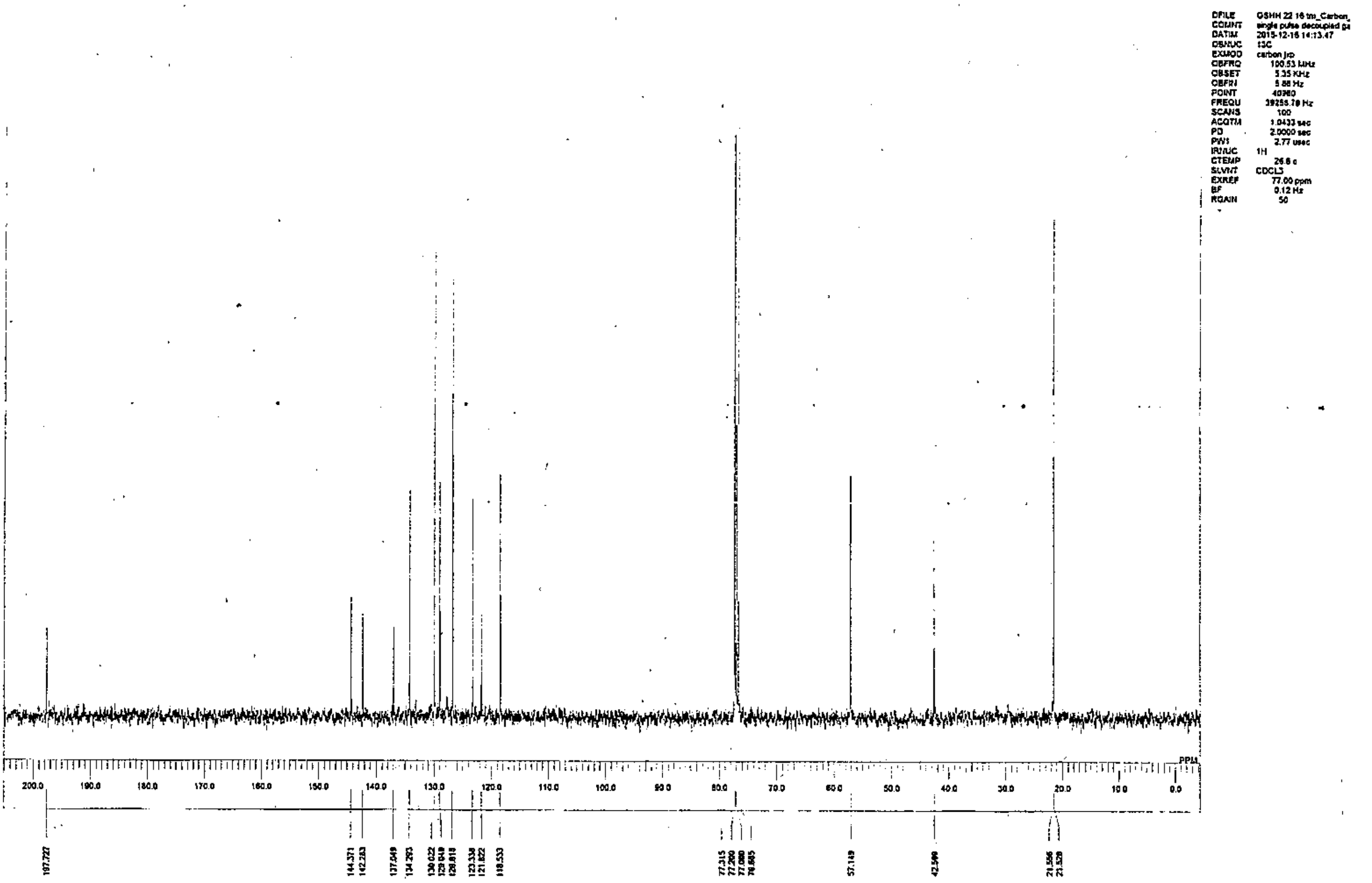
DFILE GSHH 21 50_Carbon-1-1.als
COMNT single pulse decoupled gated
DATIM 2019-11-25 21:33:48
OBNUC 13C
EXMOD carbon.jxp
OBFRQ 100.53 MHz
OBSET 5.55 KHz
OBFIN 5.86 Hz
POINT 26214
FREQU 25252.53 Hz
SCANS 102
ACQTM 1.0381 sec
PD 2.0000 sec
PW1 3.52 usec
IRNUC 1H
CTEMP 22.3 c
SLVNT CDCl3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 50

single_pulse
C:\Users\Lenovo7\AppData\Local\Temp\GSHH 22 16 tm_Proton_ft-1-1.jdf

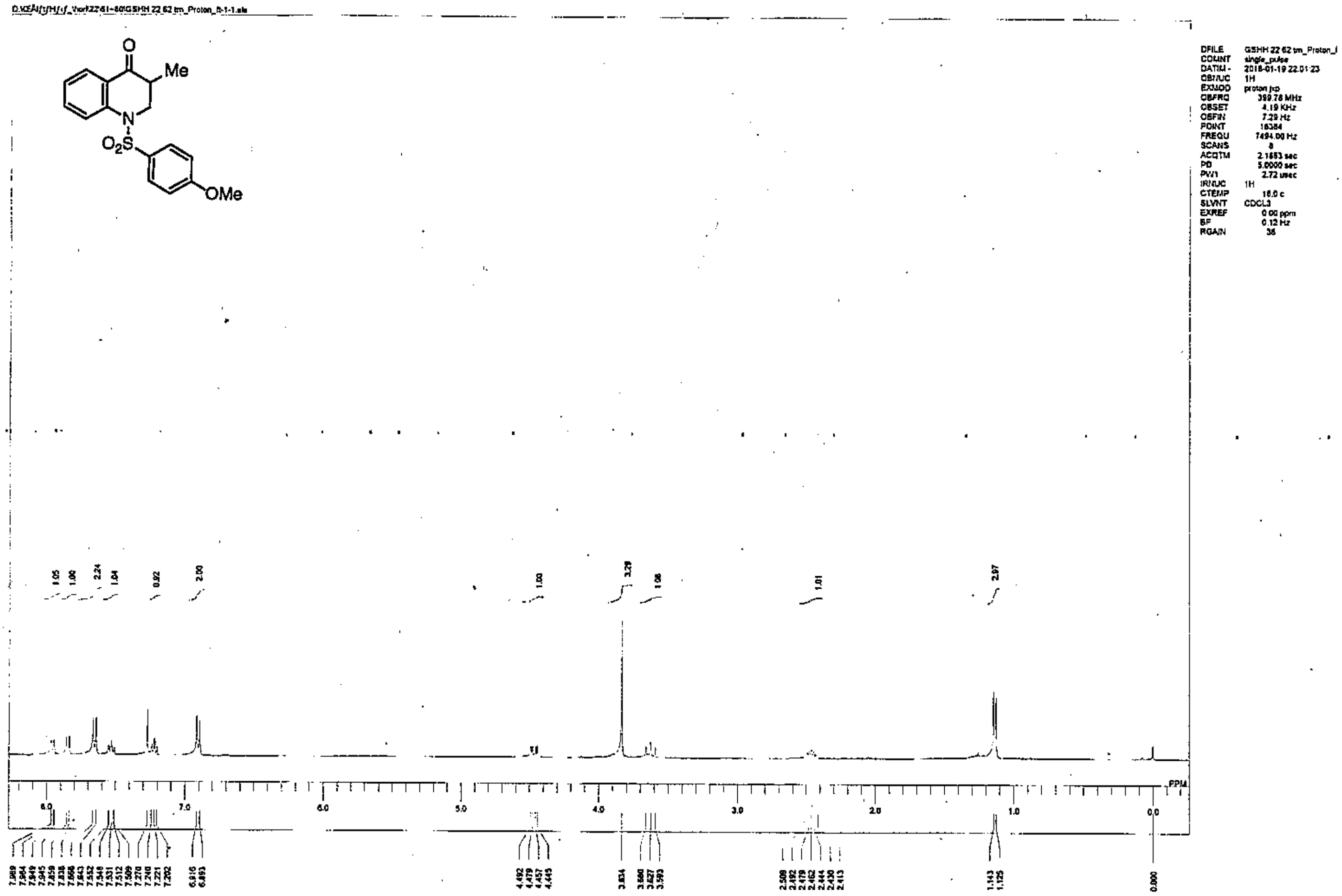



DFILE GSHH 22 16 tm_Proton_ft
COMNT single_pulse
DATIM 2015-12-16 14:12:34
OBNUC 1H
EXMOD proton.jxp
OBFRQ 399.78 MHz
OBSET 4.19 KHz
OBFIN 7.29 Hz
POINT 20480
FREQU 9378.75 Hz
SCANS 8
ACQTM 2.1837 sec
PD 5.0000 sec
PW1 5.01 usec
IRNUC 1H
CTEMP 26.3 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 34
0.93
1.92
1.06
1.18
2.20
1.00
1.92
2.96
6.15
PPM
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
8.022
8.002
7.824
7.843
7.635
7.614
7.439
7.420
7.417
7.408
7.348
7.329
7.261
7.118
7.099
7.080
3.965
2.422
1.265
0.000

single pulse decoupled gated NOE
C:\Users\Lenovo7\AppData\Local\Temp\GSHH 22 16 tm_Carbon_ft-1-1.jdf

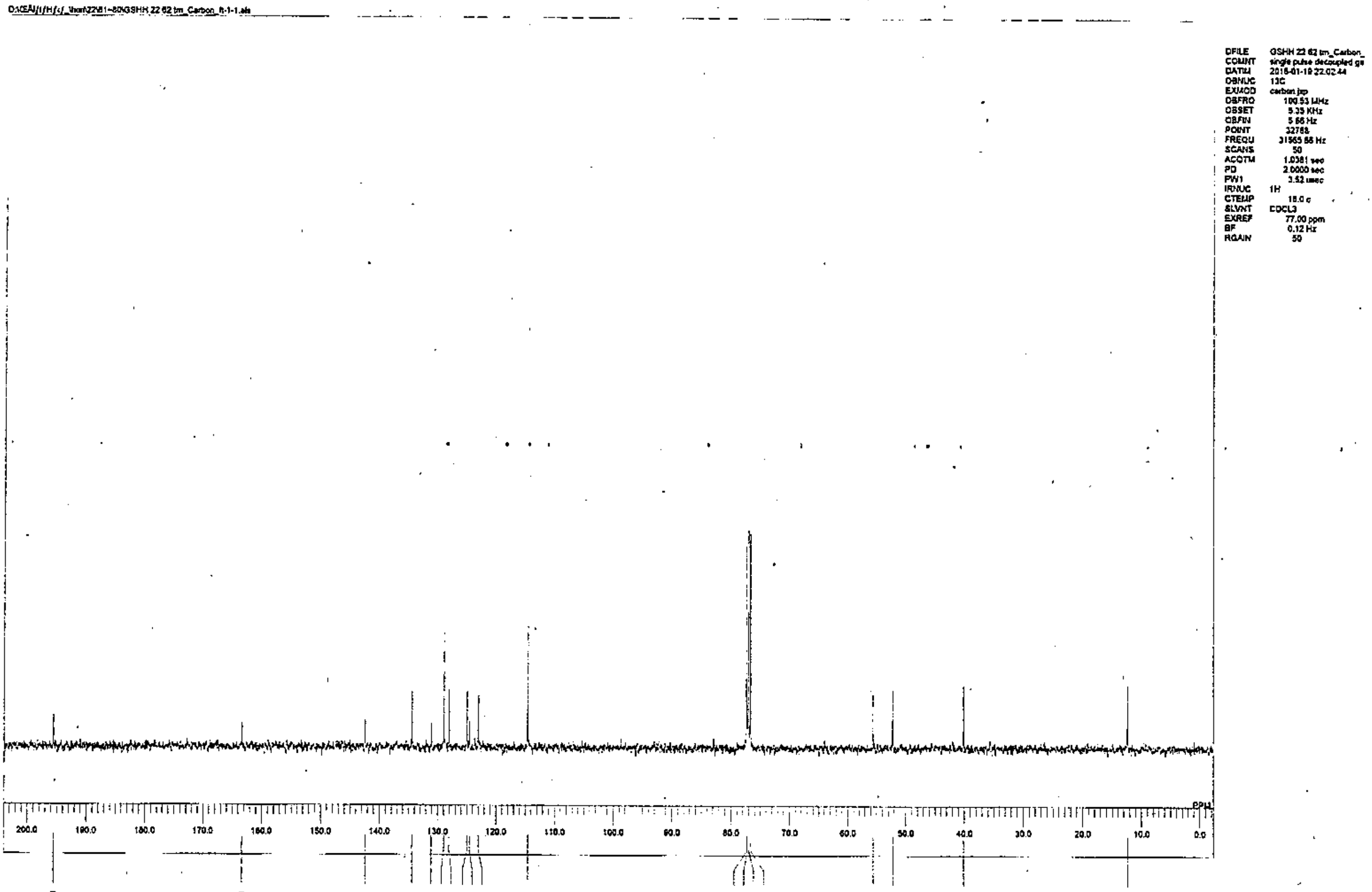

DFILE GSHH 22 16 tm_Carbon_
COMNT single pulse decoupled ga
DATIM 2015-12-16 14:13:47
OBNUC 13C
EXMOD carbon.jxp
OBFRQ 100.53 MHz
OBSET 5.35 KHz
OBFIN 5.88 Hz
POINT 40960
FREQU 39255.78 Hz
SCANS 100
ACQTM 1.0433 sec
PD 2.0000 sec
PW1 2.77 usec
IRNUC 1H
CTEMP 26.6 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 50
PPM
200.0
190.0
180.0
170.0
160.0
150.0
140.0
130.0
120.0
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0.0
197.727
144.371
142.283
137.049
134.293
130.022
129.949
126.818
123.338
121.822
118.533
77.315
77.200
77.080
76.885
57.149
42.996
21.556
21.529

single_pulse
D:\CEAJ/tj/H/cf_\hon\22\61~80\GSHH 22 62 tm_Proton_ft-1-1.als

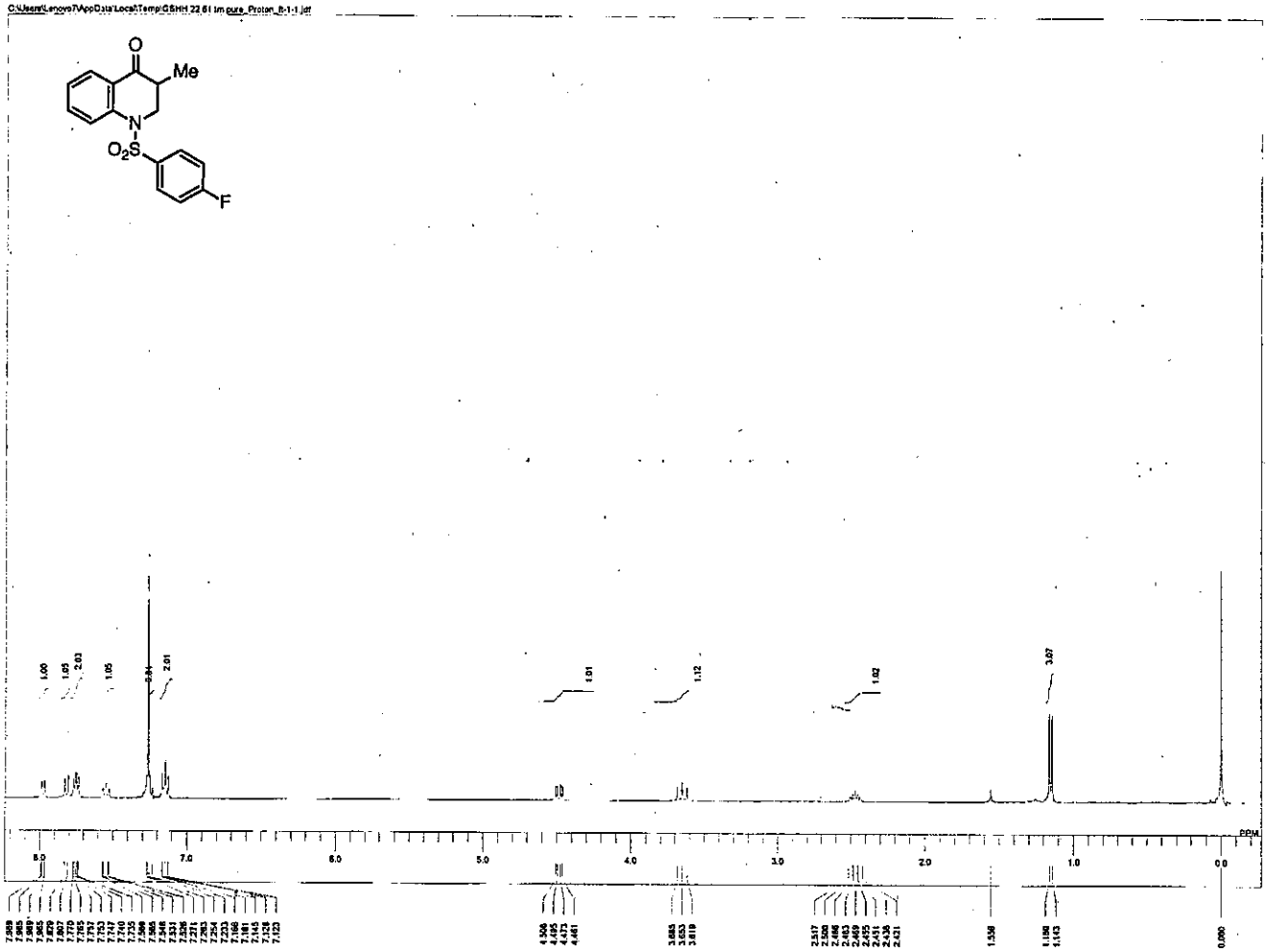


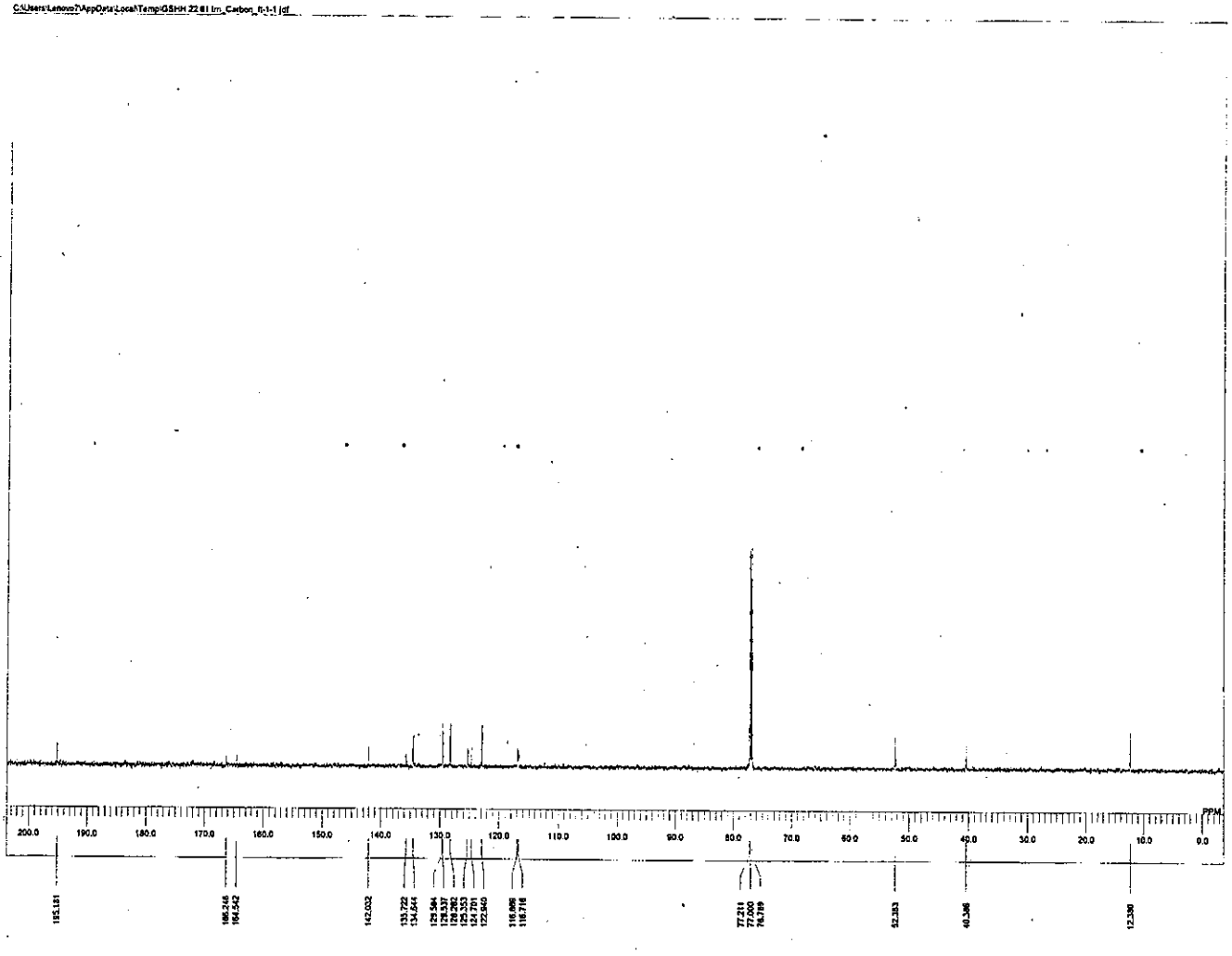
Me
O
N
O_2S
OMe
DFILE GSHH 22 62 tm_Proton_
COMNT single_pulse
DATIM - 2018-01-19 22:01:23
OBNUC 1H
EXMOD proton.jxp
OBFRQ 399.78 MHz
OBSET 4.19 KHz
OBFIN 7.29 Hz
POINT 16384
FREQU 7494.00 Hz
SCANS 8
ACQTM 2.1853 sec
PD 5.0000 sec
PW1 2.72 usec
IRNUC 1H
CTEMP 18.0 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 36
PPM
4.492 4.479 4.457 4.445
3.834
3.660 3.627 3.593
2.508 2.492 2.479 2.462 2.444 2.430 2.413
1.143 1.125
0.000
6.916 6.893

single pulse decoupled gated NOE
D:\CEAJ/tj/H/cf_\hon\22\61~80\GSHH 22 62 tm_Carbon_ft-1-1.als

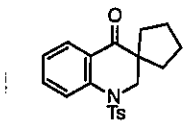


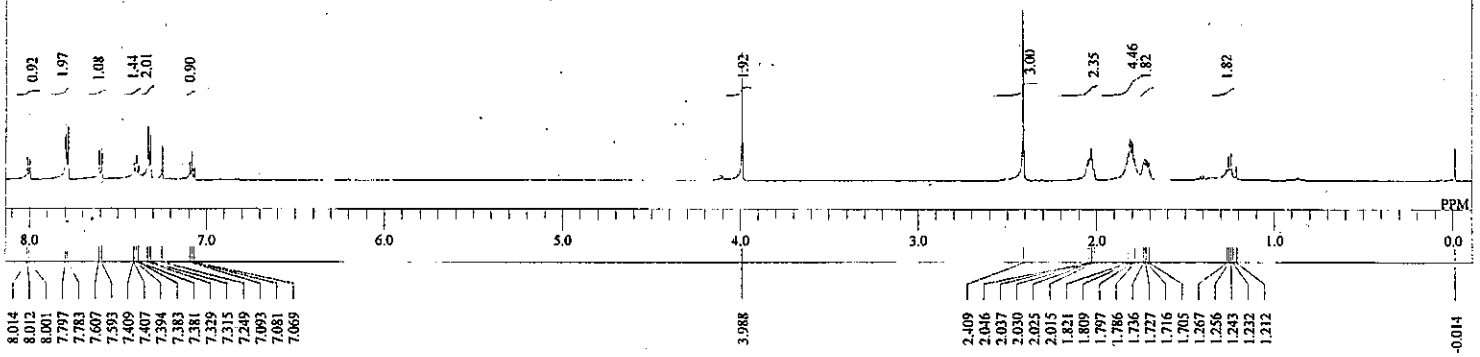
DFILE GSHH 22 62 tm_Carbon_
COMNT single pulse decoupled ge
DATIM 2018-01-19 22:02:44
OBNUC 13C
EXMOD carbon.jxp
OBFRQ 100.53 MHz
OBSET 5.35 KHz
OBFIN 5.66 Hz
POINT 32768
FREQU 31565.66 Hz
SCANS 50
ACQTM 1.0381 sec
PD 2.0000 sec
PW1 3.52 usec
IRNUC 1H
CTEMP 18.0 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 50
PPM
195.548
163.368
142.421
134.429
131.095 128.934 128.066 124.871 124.568 123.074
114.545
77.316 77.211 77.000 76.684
55.592
52.248
40.154
12.336


Me
O
N
O2S
F
DFILE GSHH 22 61 tm pure_Pro
COMNT single_pulse
DATIM 2015-01-19 22:20:42
OBNUC 1H
EXMOD proton.jxp
OBFRQ 399.78 MHz
OBSET 4.19 KHz
OBFIN 7.29 Hz
PCNT 20480
FREQU 6387.51 Hz
SCANS 8
ACQTM 2.1853 sec
PD 5.0000 sec
PW1 2.72 usec
IRNUC 1H
CTEMP 18.0 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 66
PPM

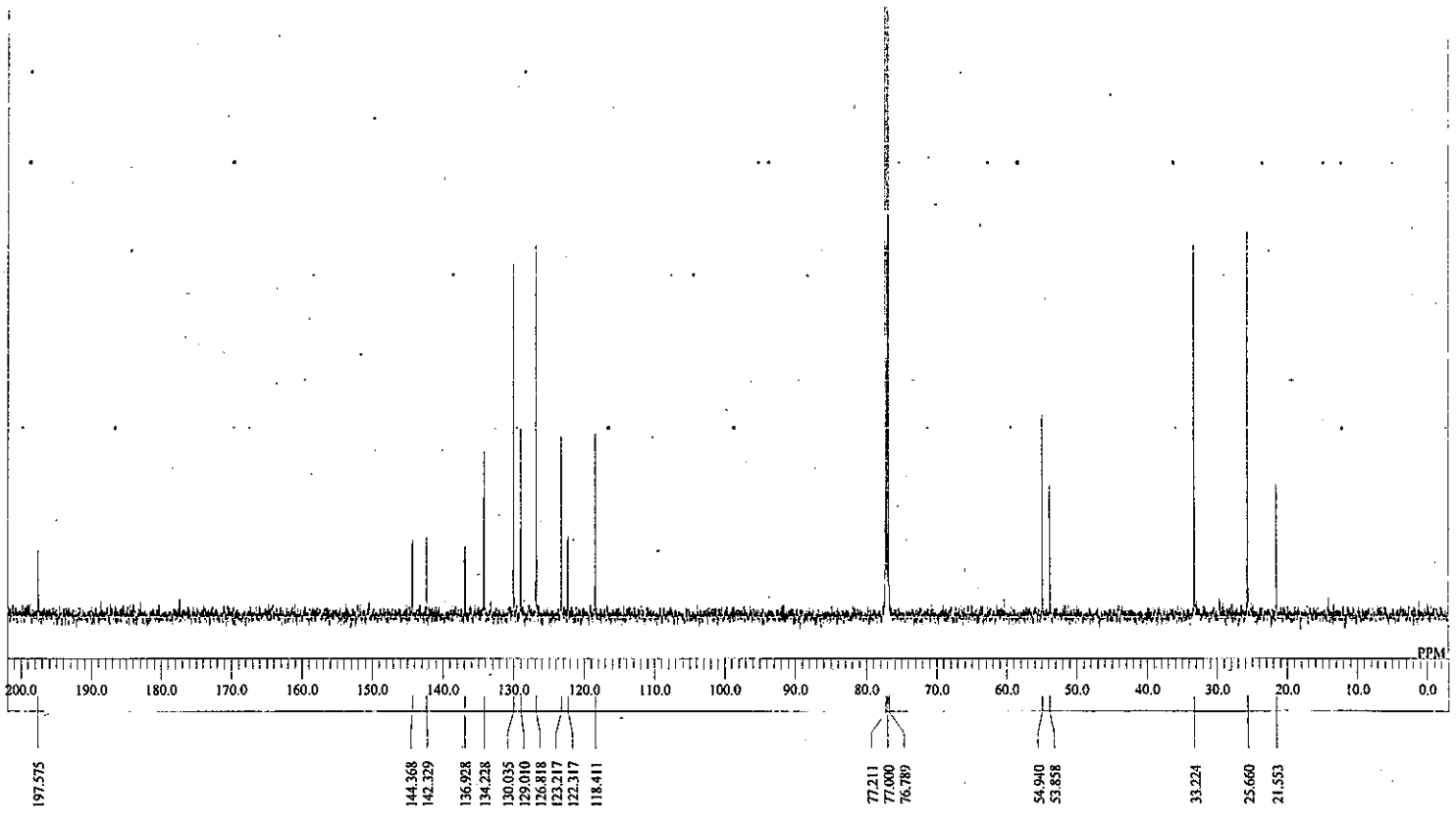

DFILE GSHH 22 61 tm_Carbon
COMNT single pulse decoupled g
DATIM 2015-01-19 19:09:01
OBNUC 13C
EXMOD carbon.jxp
OBFRQ 150.92 MHz
OBSET 8.32 KHz
OBFIN 1.74 Hz
PCNT 40960
FREQU 59185.80 Hz
SCANS 100
ACQTM 0.6921 sec
PD 2.0000 sec
PW1 3.40 usec
IRNUC 1H
CTEMP 18.5 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 52
PPM

single pulse
C:\Users\Lenovo7\AppData\Local\Temp\ffp000002a8\file\GSHH 25 71_Proton_ft-1-1.jdf



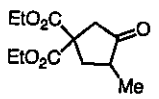


single pulse decoupled gated NOE
C:\Users\Lenovo7\AppData\Local\Temp\ffp000002a8\file\GSHH 25 71_Carbon_ft-1-1.jdf

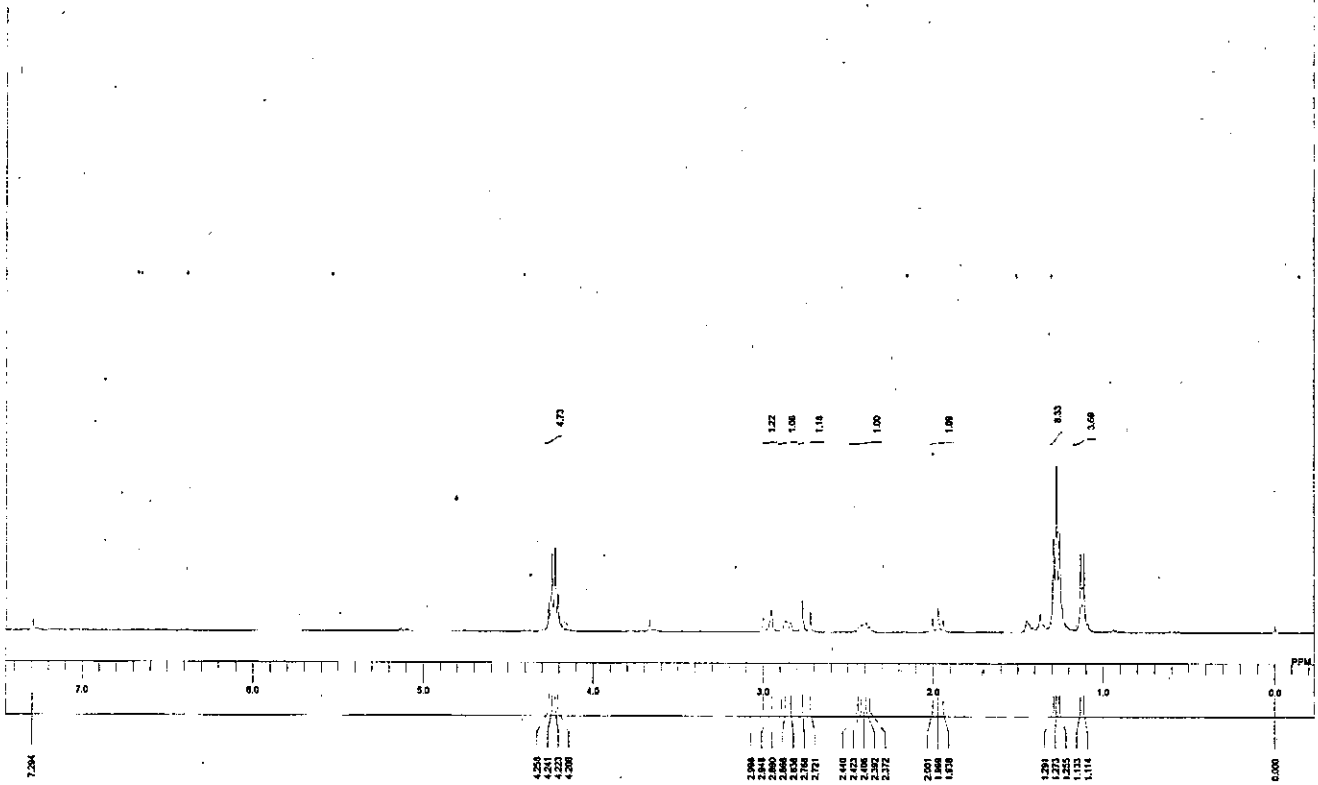


single_pulse

C:\Users\Lenovo7\AppData\Local\Temp\GSHH 23 01 tm_Proton_fi-1-1.jdf



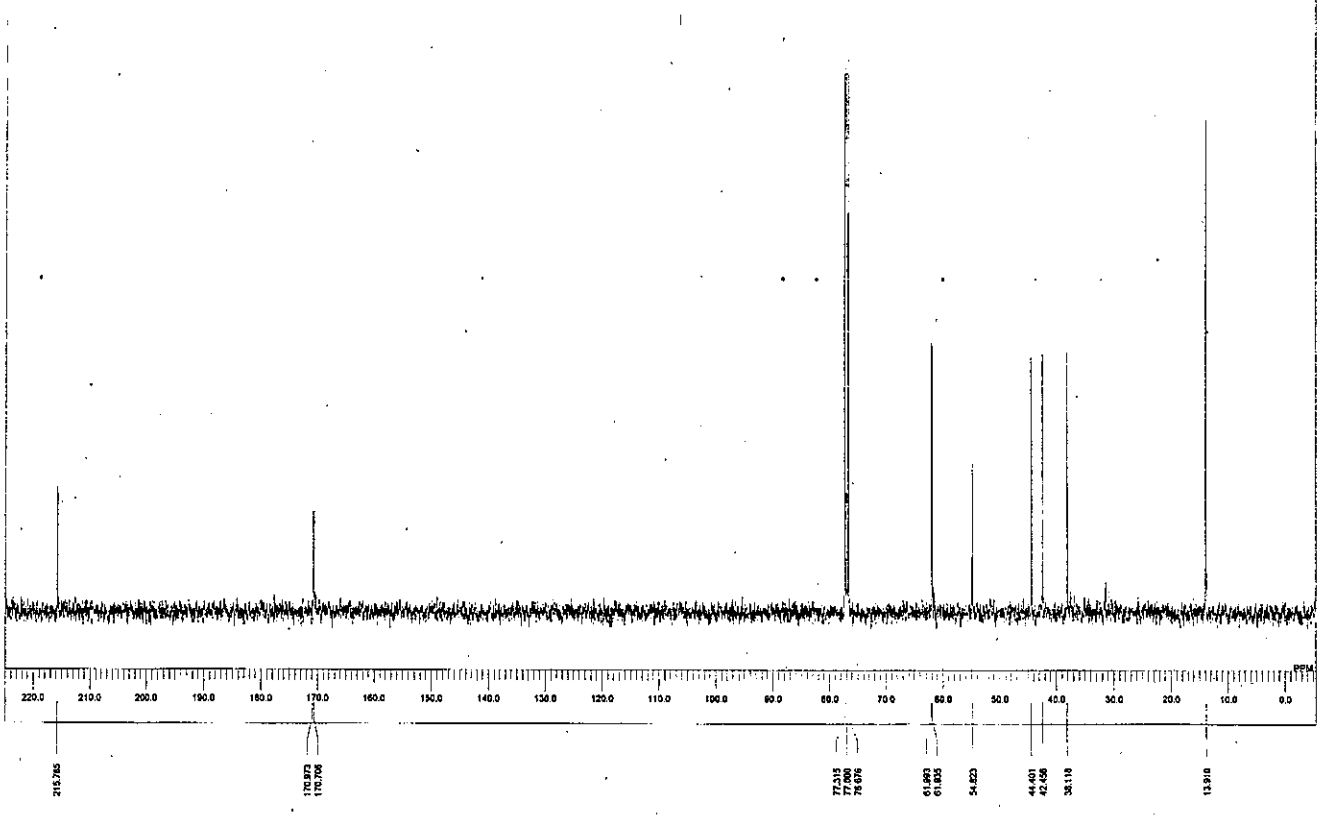
DFILE GSHH 23 01 tm_Proton_f
COMNT single_pulse
DATIM 2018-02-02 13:49:30
OBNUC 1H
EXMOD proton.jxp
OBFRQ 399.78 MHz
OBSET 4.19 KHz
OBFIN 7.29 Hz
POINT 20480
FREQU 9367.51 Hz
SCANS 8
ACQTM 2.1853 sec
PD 5.0000 sec
PW1 2.72 usec
IRNUC 1H
CTEMP 17.1 c
SLVNT CDCL3
EXREF 0.00 ppm
RF 0.12 Hz
RGAIN 36



single pulse decoupled gated NOE

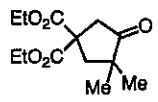
C:\Users\Lenovo7\AppData\Local\Temp\GSHH 23 01 tm_Carbon_fi-1-1.jdf

DFILE GSHH 23 01 tm_Carbon_
COMNT single pulse decoupled gat
DATIM 2018-02-02 14:41:29
OBNUC 13C
EXMOD carbon.jxp
OBFRQ 100.53 MHz
OBSET 5.35 KHz
OBFIN 5.86 Hz
POINT 40960
FREQU 39256.79 Hz
SCANS 50
ACQTM 1.0433 sec
PD 2.0000 sec
PW1 2.77 usec
IRNUC 1H
CTEMP 20.8 c
SLVNT CDCL3
EXREF 77.00 ppm
RF 0.12 Hz
RGAIN 50

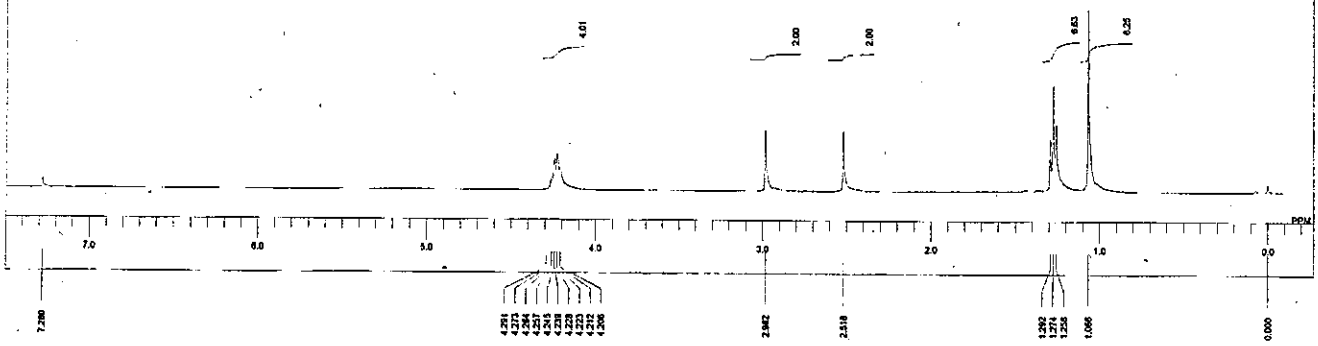


single_pulse

C:\Users\Lenovo7\AppData\Local\Temp\GSHH 23 10 tm_Proton_ft-1-1.jdf



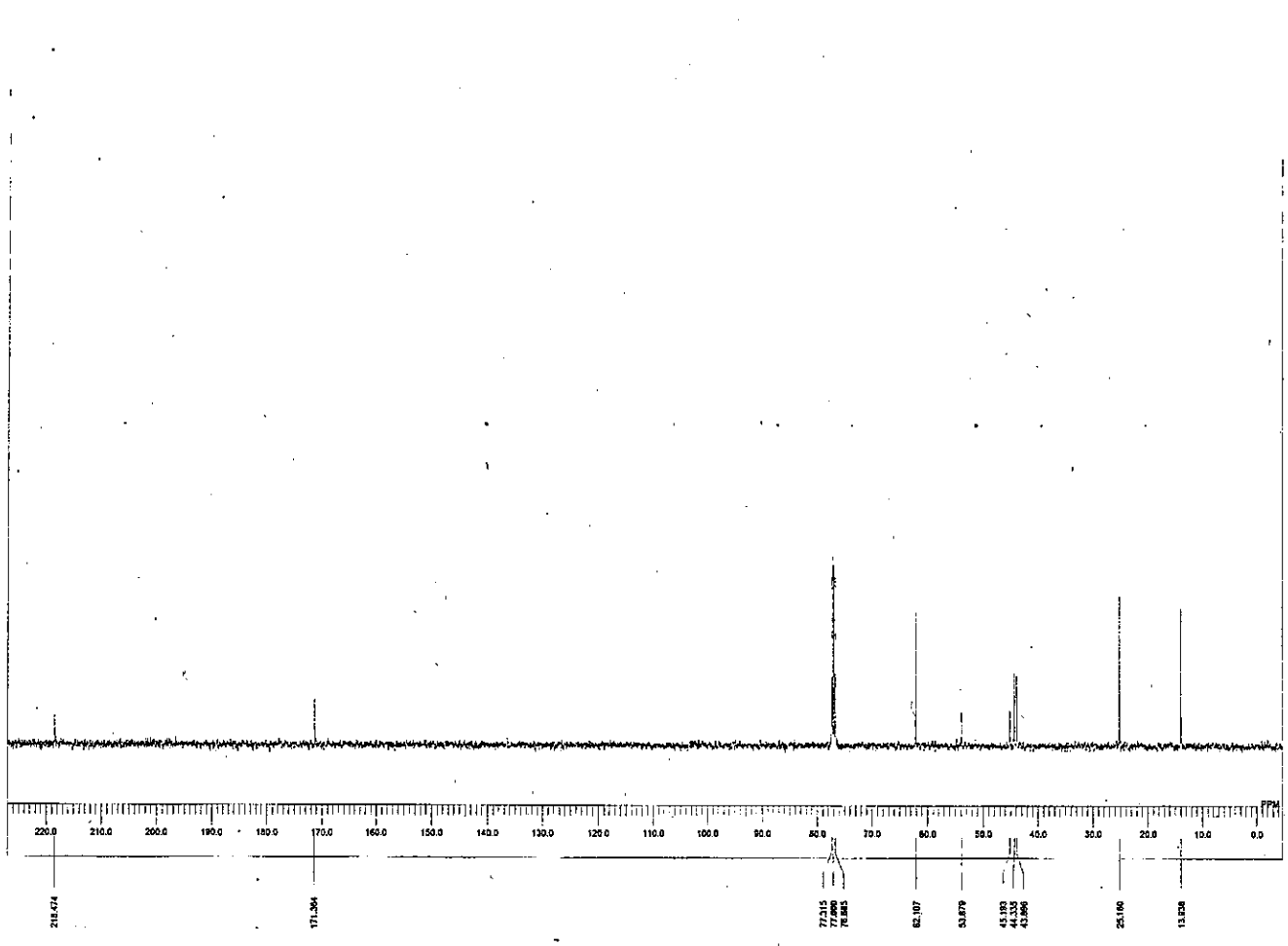
DFILE GSHH 23 10 tm_Proton_1
COMNT single_pulse
DATIM 2016-02-08 21:38:36
OBNUC 1H
EXMOD proton.jxp
OBFRQ 399.78 MHz
OBSET 4.19 KHz
OBFIN 7.39 Hz
POINT 32488
FREQU 9378.75 Hz
SCANS 8
ACQTM 2.1837 sec
PD 5.0000 sec
PW1 5.01 usec
IRNUC 1H
CTEMP 20.3 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 32



single pulse decoupled post NOE

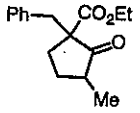
C:\Users\Lenovo7\AppData\Local\Temp\GSHH 23 10 tm_Carbon_ft-1-1.jdf

DFILE GSHH 23 10 tm_Carbon_
COMNT single pulse decoupled pi
DATIM 2016-02-08 21:39:49
OBNUC 13C
EXMOD carbon.jxp
OBFRQ 100.53 MHz
OBSET 9.35 KHz
OBFIN 5.86 Hz
POINT 40960
FREQU 39238.78 Hz
SCANS 50
ACQTM 1.0433 sec
PD 2.0000 sec
PW1 2.77 usec
IRNUC 1H
CTEMP 20.5 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 50

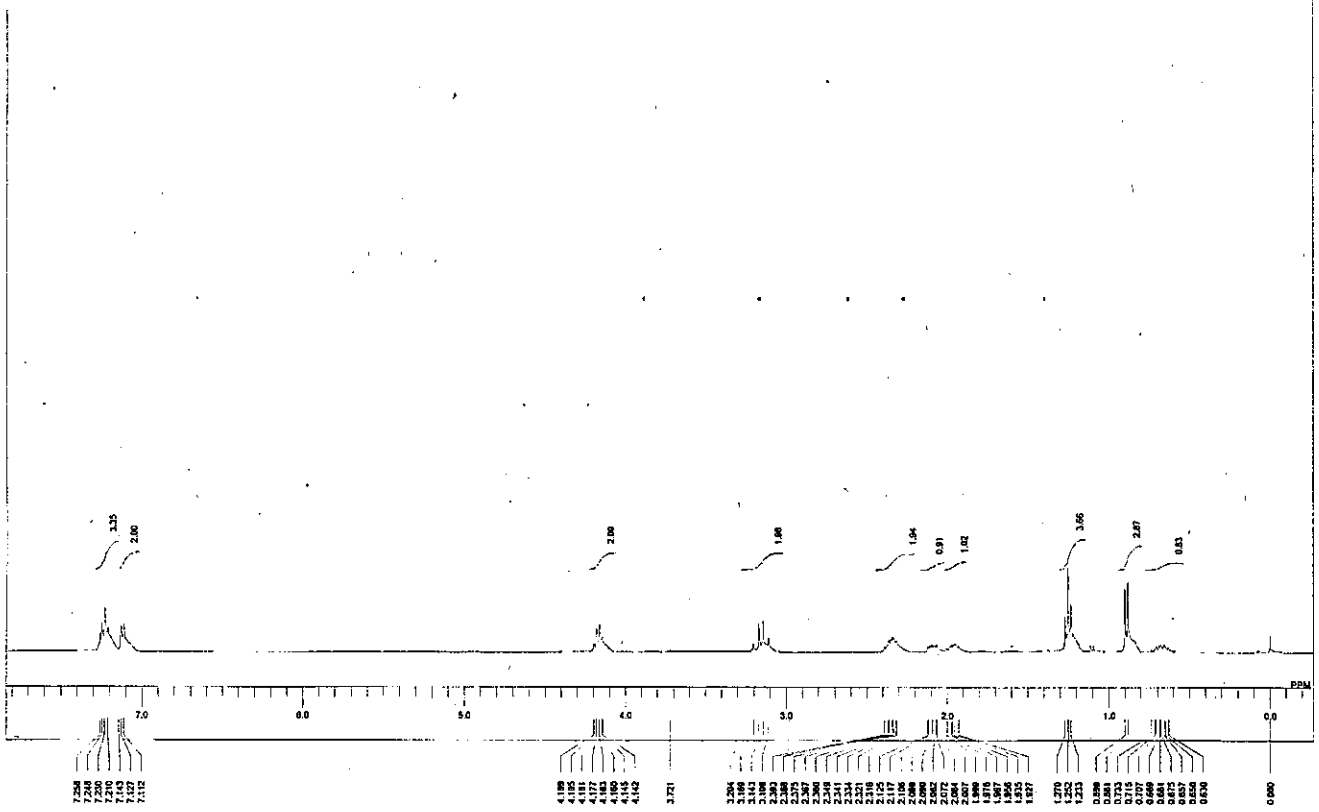


single_pulse

C:\Users\Lenovo7\AppData\Local\Temp\GSHH 23 28 tm_Proton_ft-1-1.jdf



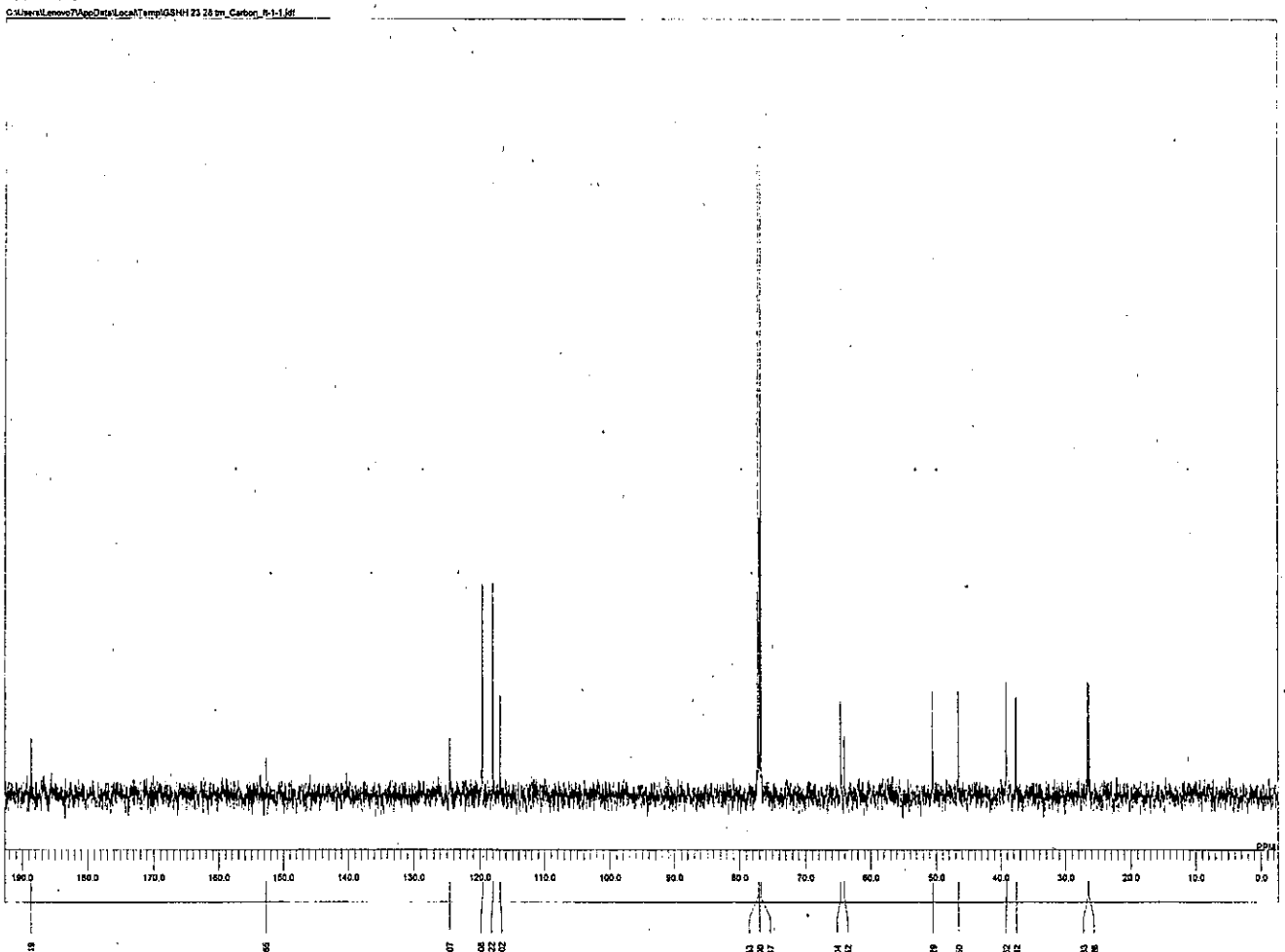
5j: 50% (10 h)



DFILE GSHH 23 28 tm_Proton_ft
COMNT single_pulse
DATIM 2016-03-02 15:35:10
OBNUC 1H
EXMOD proton.jxp
OBFRQ 399.78 MHz
OBSET 4.19 KHz
OBFIN 7.29 Hz
POINT 20480
FREQU 9367.51 Hz
SCANS 8
ACQTM 2.1883 sec
PD 5.0000 sec
PW1 2.72 usec
IRNUC 1H
CTEMP 19.0 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 36

single pulse decoupled gated NOE

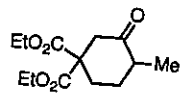
C:\Users\Lenovo7\AppData\Local\Temp\GSHH 23 28 tm_Carbon_ft-1-1.jdf



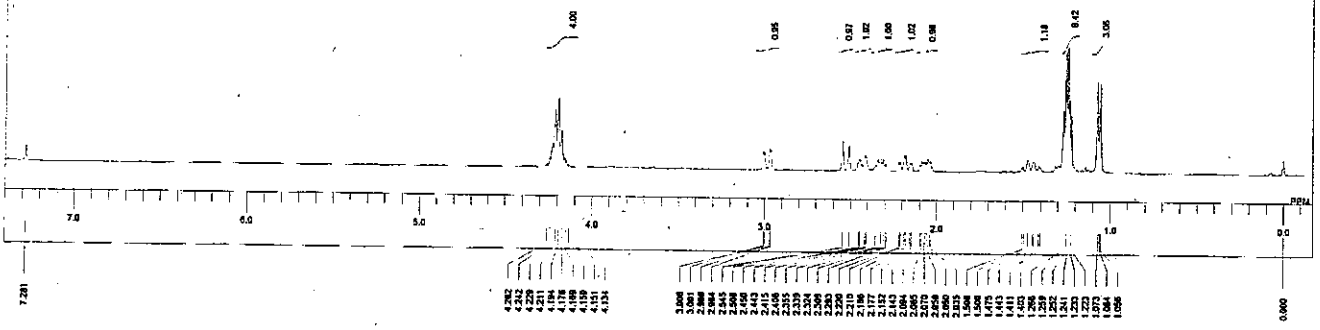
DFILE GSHH 23 28 tm_Carbon_f
COMNT single pulse decoupled gat
DATIM 2016-03-02 15:36:29
OBNUC 13C
EXMOD carbon.jxp
OBFRQ 100.53 MHz
OBSET 5.35 KHz
OBFIN 5.96 Hz
POINT 40960
FREQU 31949.66 Hz
SCANS 50
ACQTM 1.0381 sec
PD 2.0000 sec
PW1 3.52 usec
IRNUC 1H
CTEMP 18.1 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 50

single_pulse

C:\Users\Lenovo7\AppData\Local\Temp\GSHH 23 23 tm_Proton_1-1-1.jdf



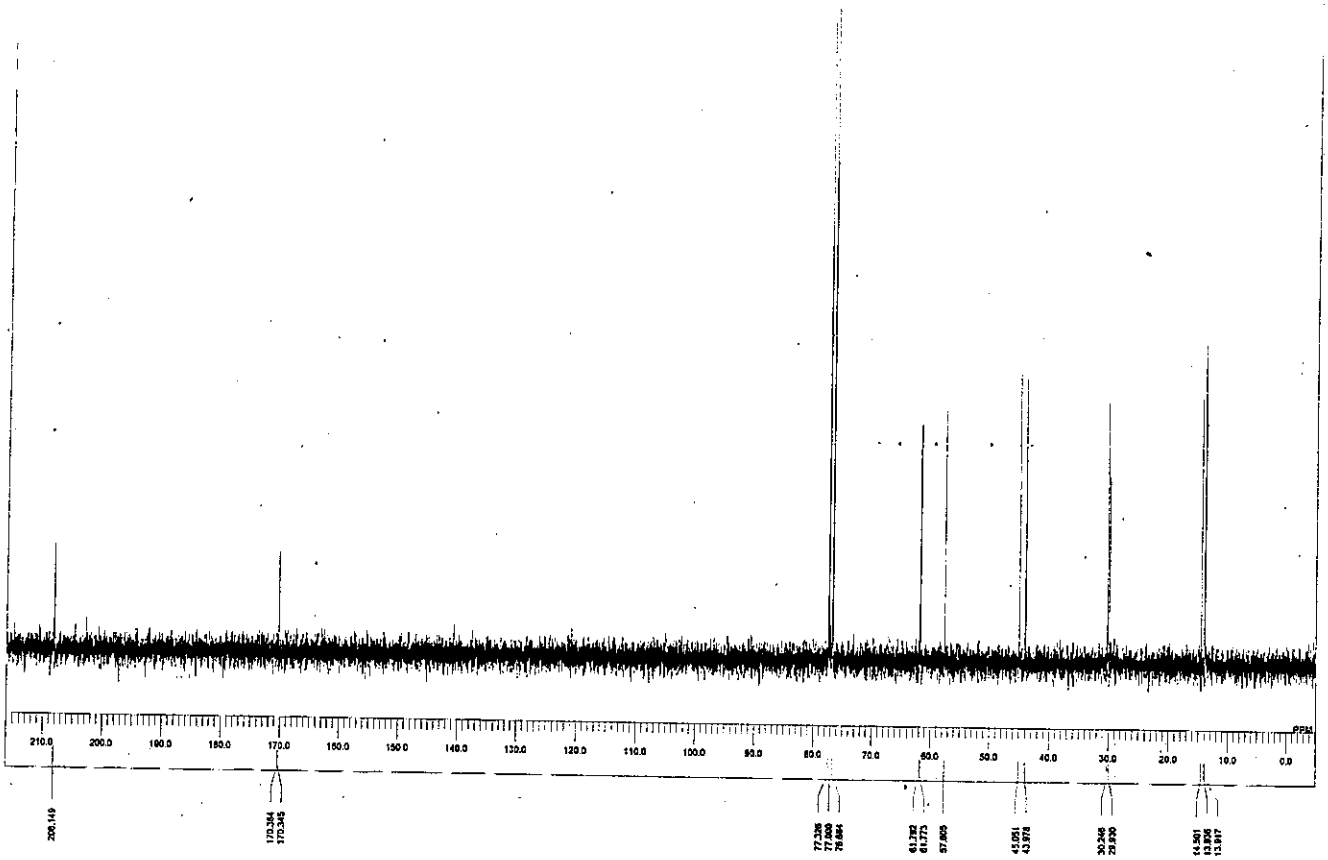
DFILE GSHH 23 23 tm_Proton_1
COMNT single_pulse
DATIM 2018-02-20 16:02:10
OBNUC 1H
EXMOD proton.jxp
OBFRQ 399.78 MHz
OBSET 4.19 KHz
OBFIN 7.29 Hz
POINT 20480
FREQU 9367.51 Hz
SCANS 8
ACQTM 2.1963 sec
PD 5.0000 sec
PW1 2.72 usec
IRNUC 1H
CTEMP 19.8 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 48

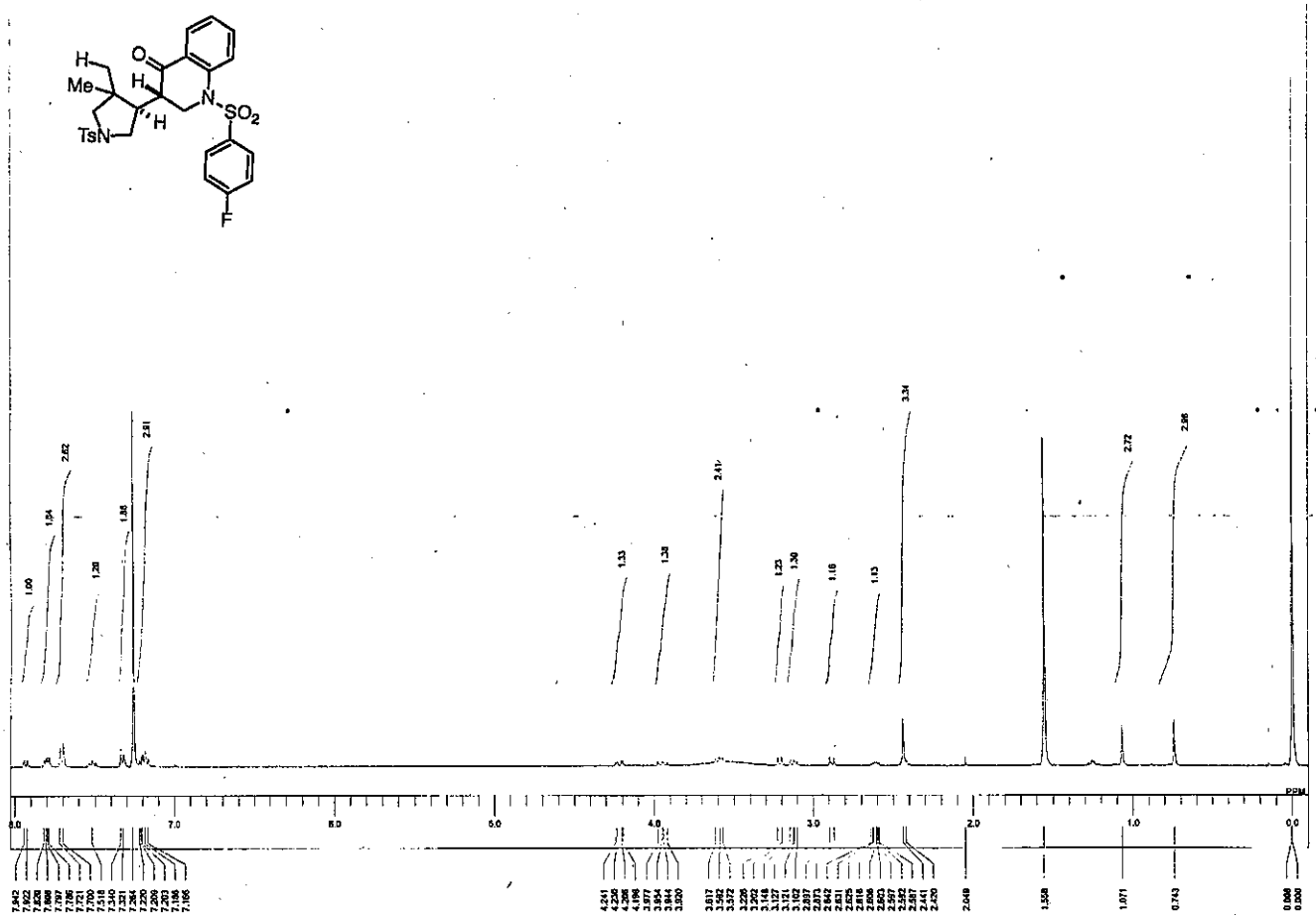


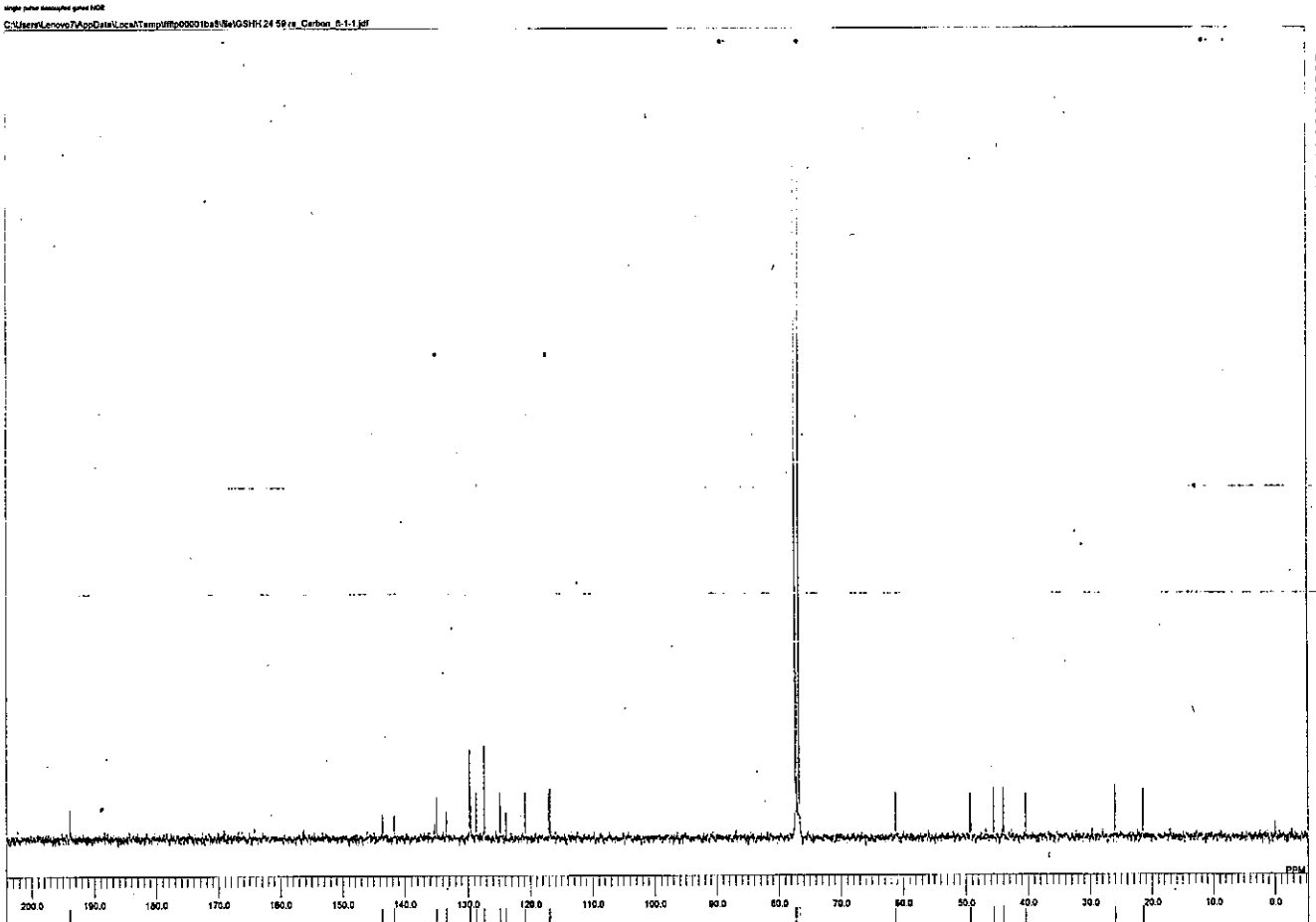
single pulse decoupled gated NOE

C:\Users\Lenovo7\AppData\Local\Temp\GSHH 23 23 tm_Carbon-1-1.jdf

DFILE GSHH 23 23 tm_Carbon
COMNT single pulse decoupled ga
DATIM 2018-02-23 16:03:32
OBNUC 13C
EXMOD carbon.jxp
OBFRQ 100.53 MHz
OBSET 9.35 KHz
OBFIN 3.66 Hz
POINT 32767
FREQU 31565.66 Hz
SCANS 100
ACQTM 1.0381 sec
PD 2.0000 sec
PW1 3.52 usec
IRNUC 1H
CTEMP 18.8 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 50

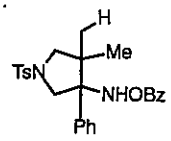



DFILE GSHH 24 59 re_Proton_fi-
COMNT single_pulse
DATIM 2018-06-20 21:56:29
OBNUC 1H
EXMOD proton.jxp
OBFRQ 399.78 MHz
OBSET 4.19 KHz
OBFIN 7.29 Hz
POINT 16384
FREQU 7503.00 Hz
SCANS 8
ACQTM 2.1837 sec
PD 5.0000 sec
PW1 5.00 usec
IRNUC 1H
CTEMP 20.2 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 50
PPM


DFILE GSHH 24 59 re_Carbon_fi-
COMNT single pulse decoupled gate
DATIM 2018-06-20 22:16:38
OBNUC 13C
EXMOD carbon.jxp
OBFRQ 100.53 MHz
OBSET 5.35 KHz
OBFIN 5.56 Hz
POINT 40960
FREQU 39259.78 Hz
SCANS 400
ACQTM 1.0433 sec
PD 2.0000 sec
PW1 2.97 usec
IRNUC 1H
CTEMP 20.4 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 50
PPM

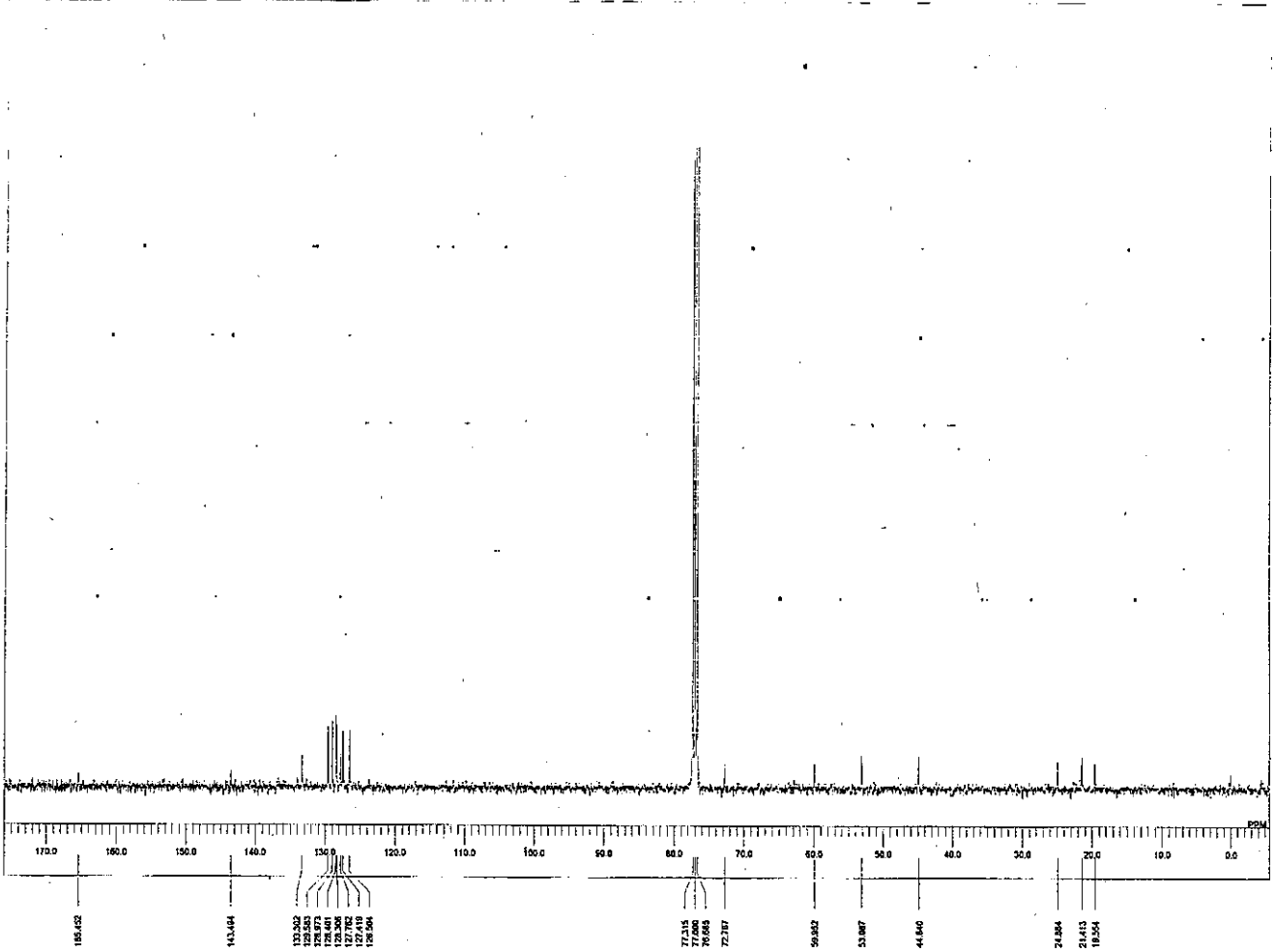
single_pulse

C:\Users\Lenovo7\AppData\Local\Temp\ffp0000125b0\file\GSHH 25 54 re_Proton_ft-1-1.jdf



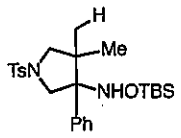
single pulse decoupled gated NOE

C:\Users\Lenovo7\AppData\Local\Temp\ffp0000125b0\file\GSHH 25 54 re_Carbon_ft-1-1.jdf

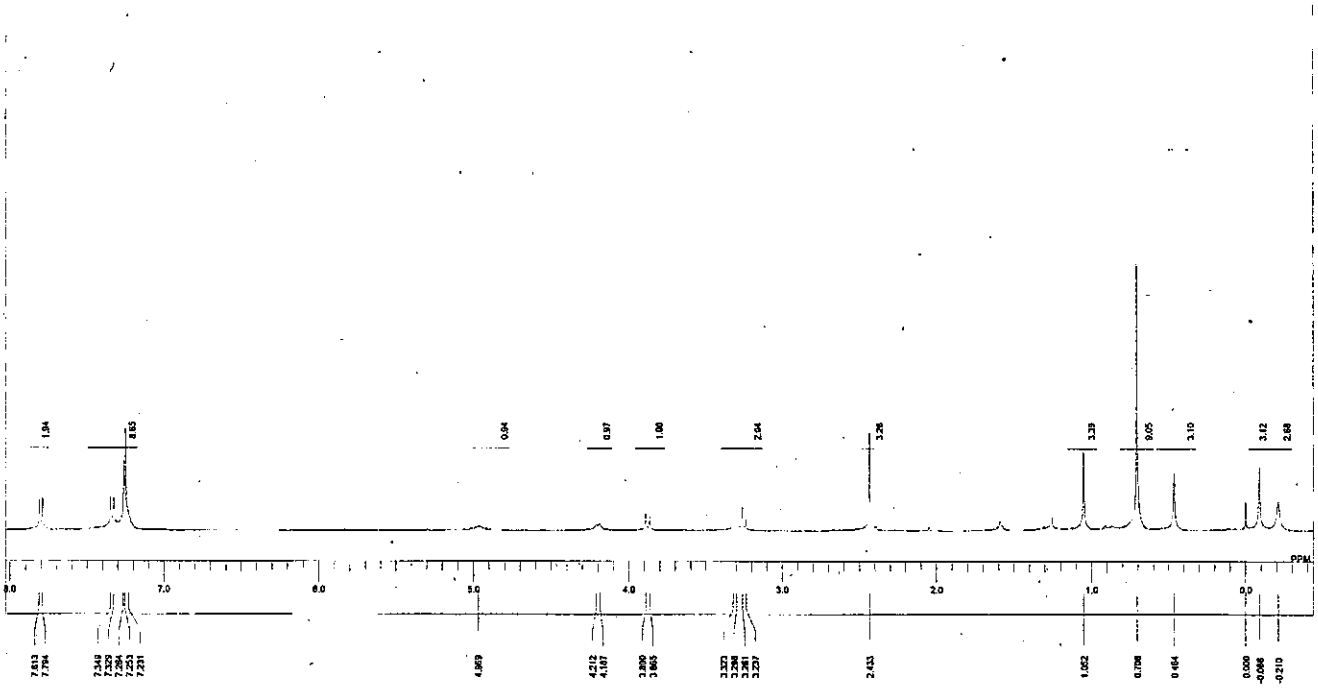


single_pulse

C:\Users\Lenovo7\AppData\Local\Temp\Rtmp00001658\file\GSHH 26 21 3 8 pure_Proton_fi-1-1.jdf



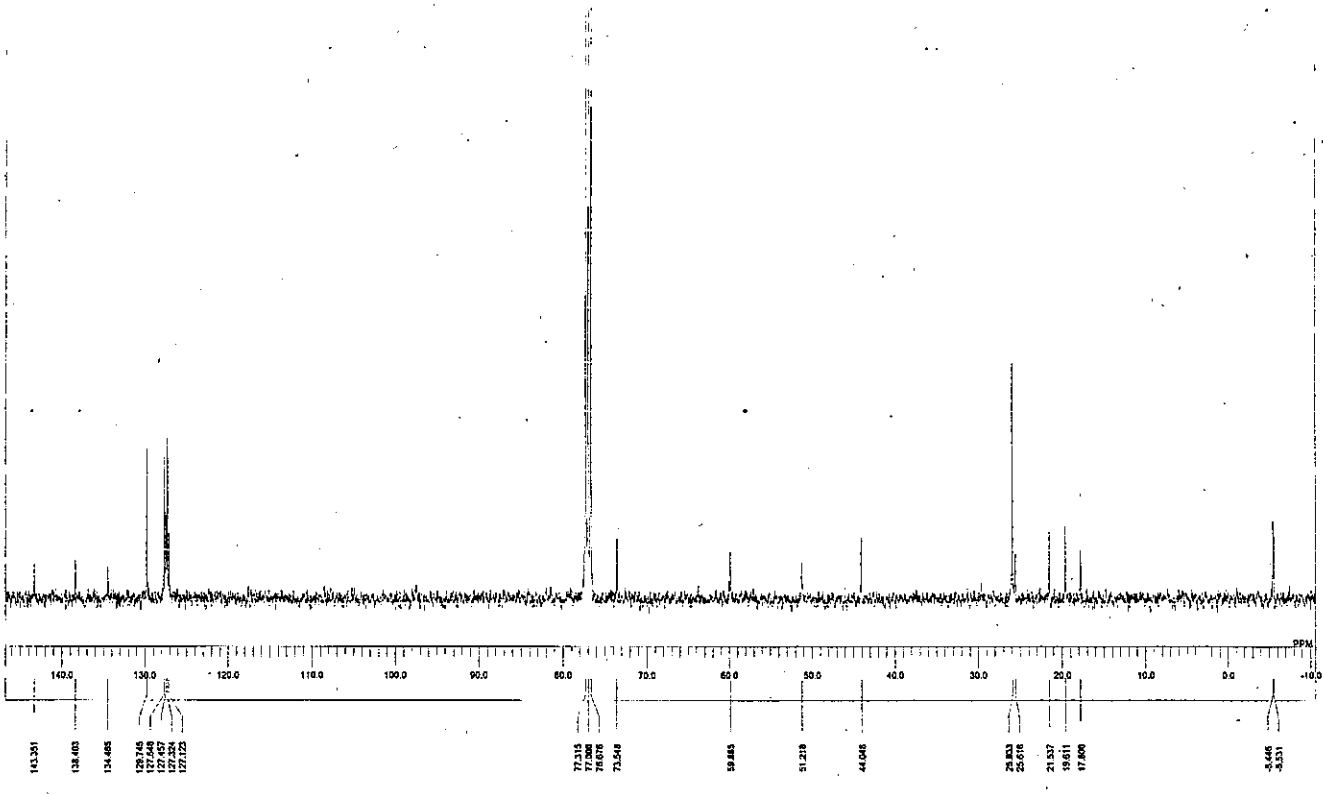

```
DFILE   GSHH 26 21 3 8 pure_Pro
COMNT   single_pulse
DATIM   2016-11-17 19:29:48
OBSNUC  1H
EXMOD   proton.jxp
OBFRQ     399.78 MHz
OBSET       4.19 KHz
OBFIN       7.29 Hz
POINT      20480
FREQU    9376.75 Hz
SCANS          8
ACQTM      2.1837 sec
PD         5.0000 sec
PW1        5.00 usec
IRNUC      1H
CTEMP      29.5 c
SLVNT      CDCL3
EXREF      0.00 ppm
BF         0.12 Hz
RGAIN        38
```



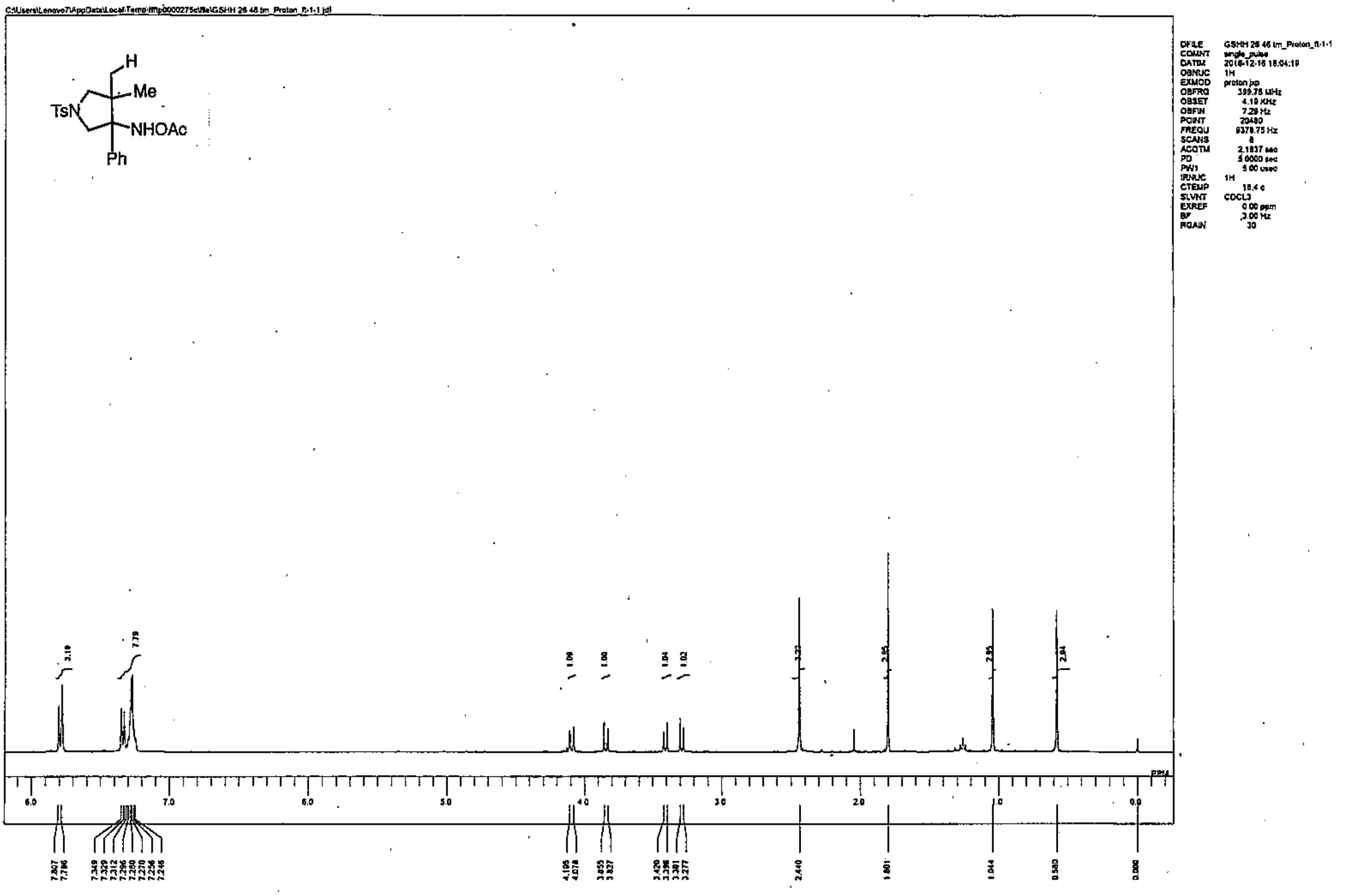
single pulse decoupled gated NOE

C:\Users\Lenovo7\AppData\Local\Temp\Rtmp00001658\file\GSHH 26 21 3 8 pure_Carbon_fi-1-1.jdf

```
DFILE   GSHH 26 21 3 8 pure_Car
COMNT   single pulse decoupled gat
DATIM   2016-11-17 19:31:00
OBSNUC  13C
EXMOD   carbon.jxp
OBFRQ     100.53 MHz
OBSET       5.35 KHz
OBFIN       5.80 Hz
POINT      40960
FREQU    39238.79 Hz
SCANS        200
ACQTM      1.0433 sec
PD         2.0000 sec
PW1        2.87 usec
IRNUC      1H
CTEMP      20.4 c
SLVNT      CDCL3
EXREF      77.00 ppm
BF         0.12 Hz
RGAIN        50
```

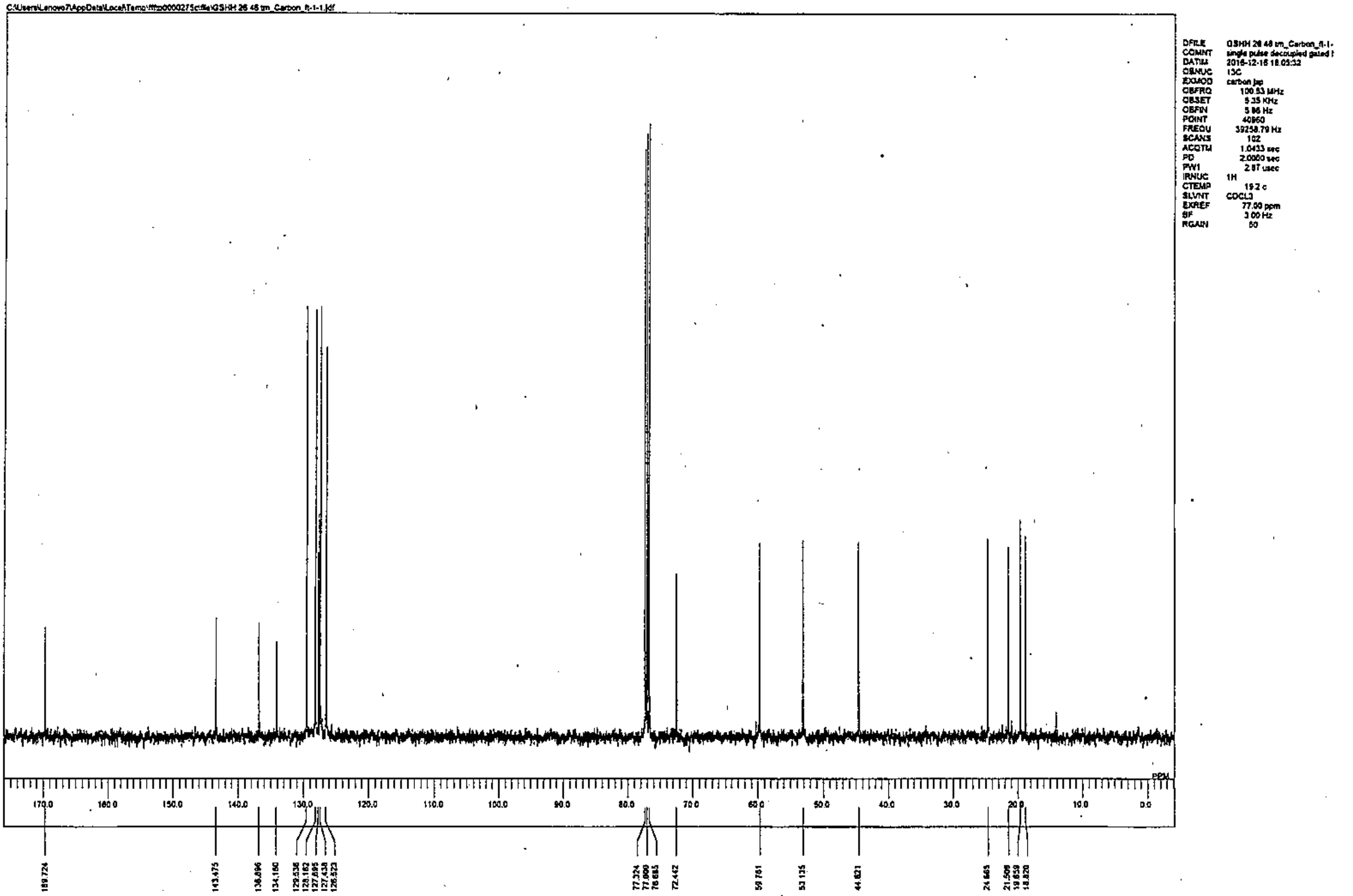


single_pulse
C:\Users\Lenovo7\AppData\Local\Temp\fftp0000275ct\file\GSHH 26 46 tm_Proton_ft-1-1.jdf

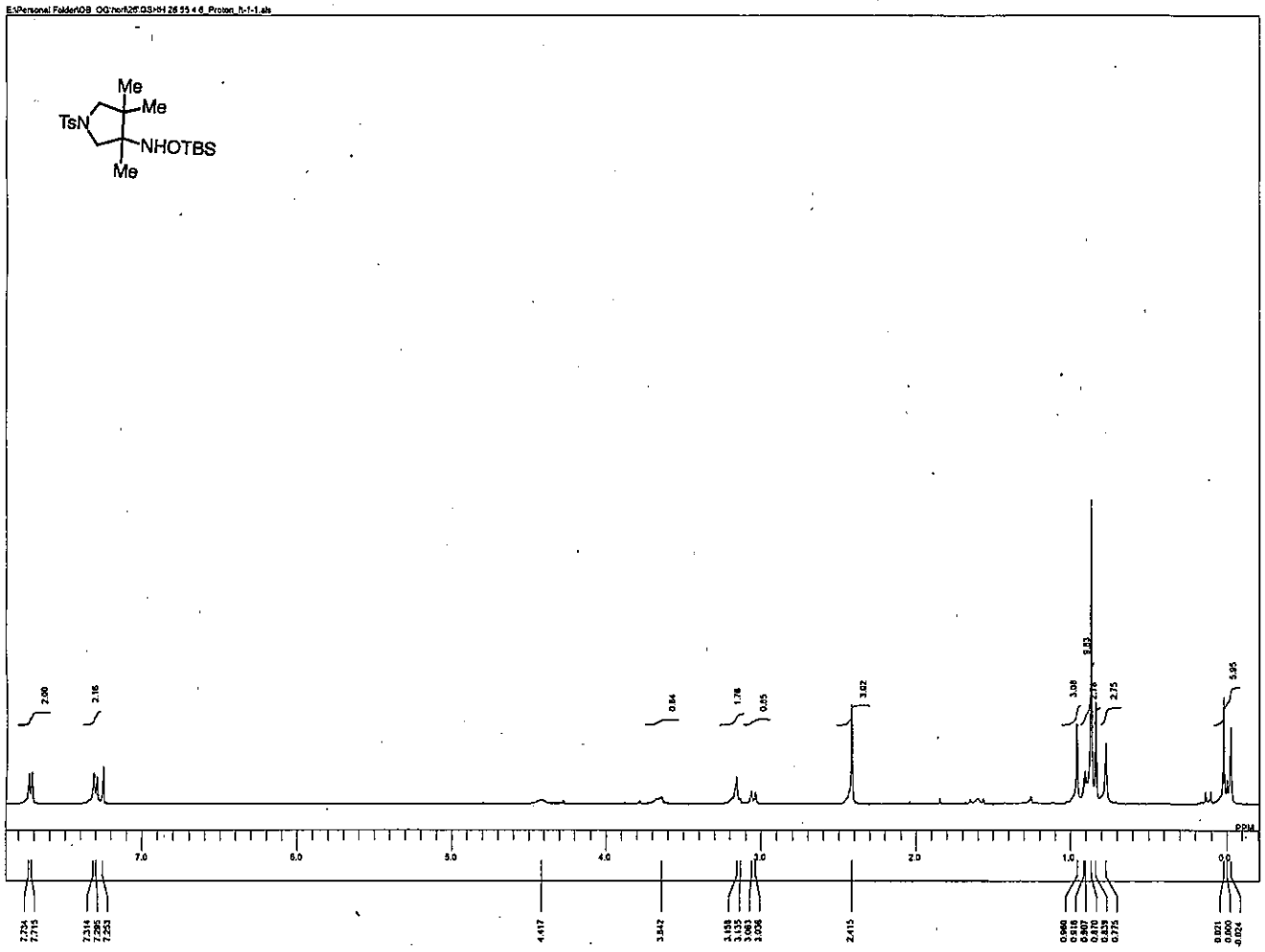


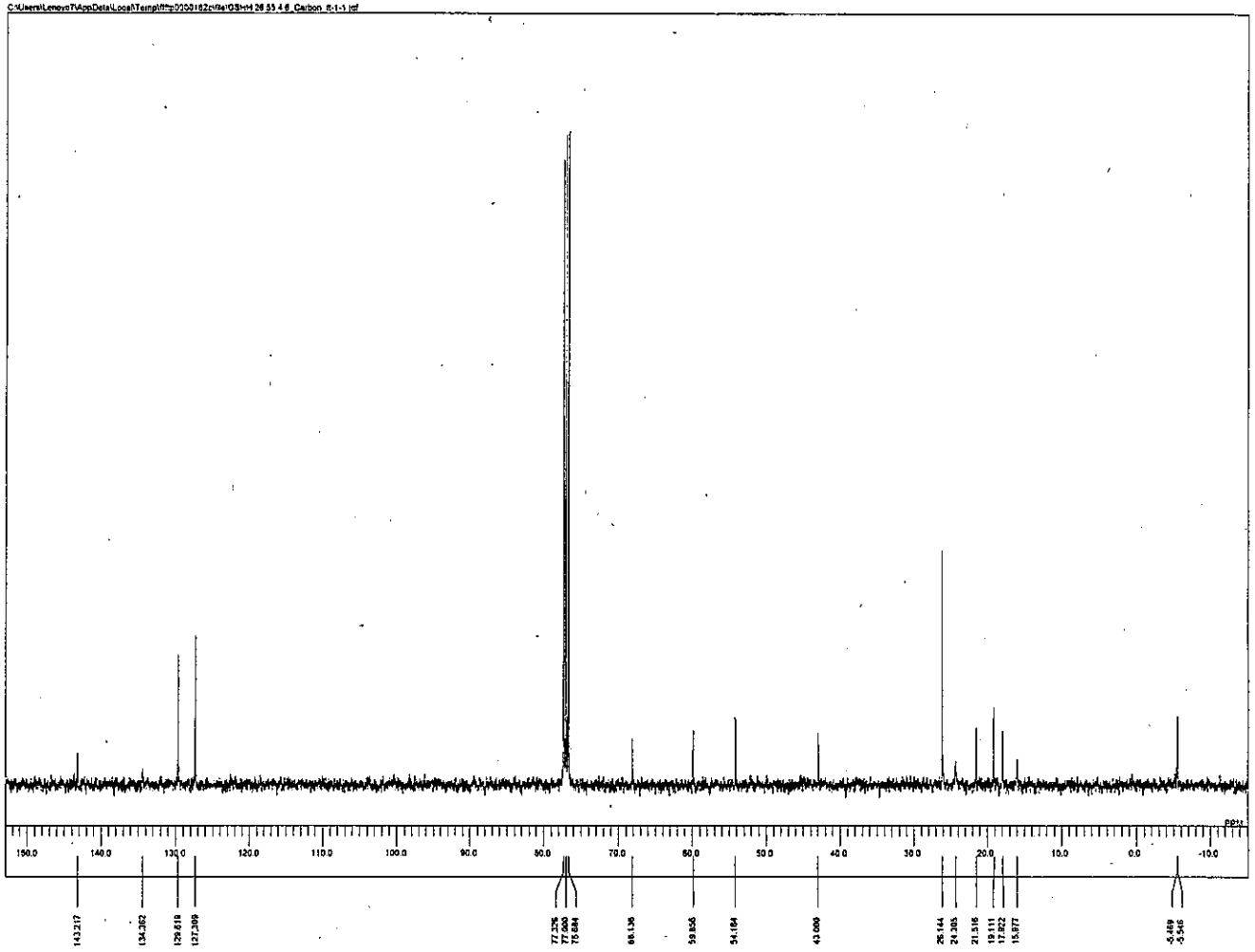
H
Me
TsN
NHOAc
Ph
DFILE GSHH 26 46 tm_Proton_ft-1-1
COMNT single_pulse
DATIM 2016-12-16 18:04:19
OBNUC 1H
EXMOD proton.jxp
OBFRQ 399.78 MHz
OBSET 4.19 KHz
OBFIN 7.29 Hz
POINT 20480
FREQU 9378.75 Hz
SCANS 8
ACQTM 2.1837 sec
PD 5.0000 sec
PW1 5.00 usec
IRNUC 1H
CTEMP 18.4 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 3.00 Hz
RGAIN 30
3.19
7.79
1.09
1.00
1.04
1.02
3.22
2.95
2.95
2.94
PPM
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
7.807
7.786
7.349
7.329
7.312
7.296
7.290
7.270
7.256
7.246
4.105
4.078
3.855
3.827
3.420
3.396
3.301
3.277
2.440
1.801
1.044
0.580
0.000

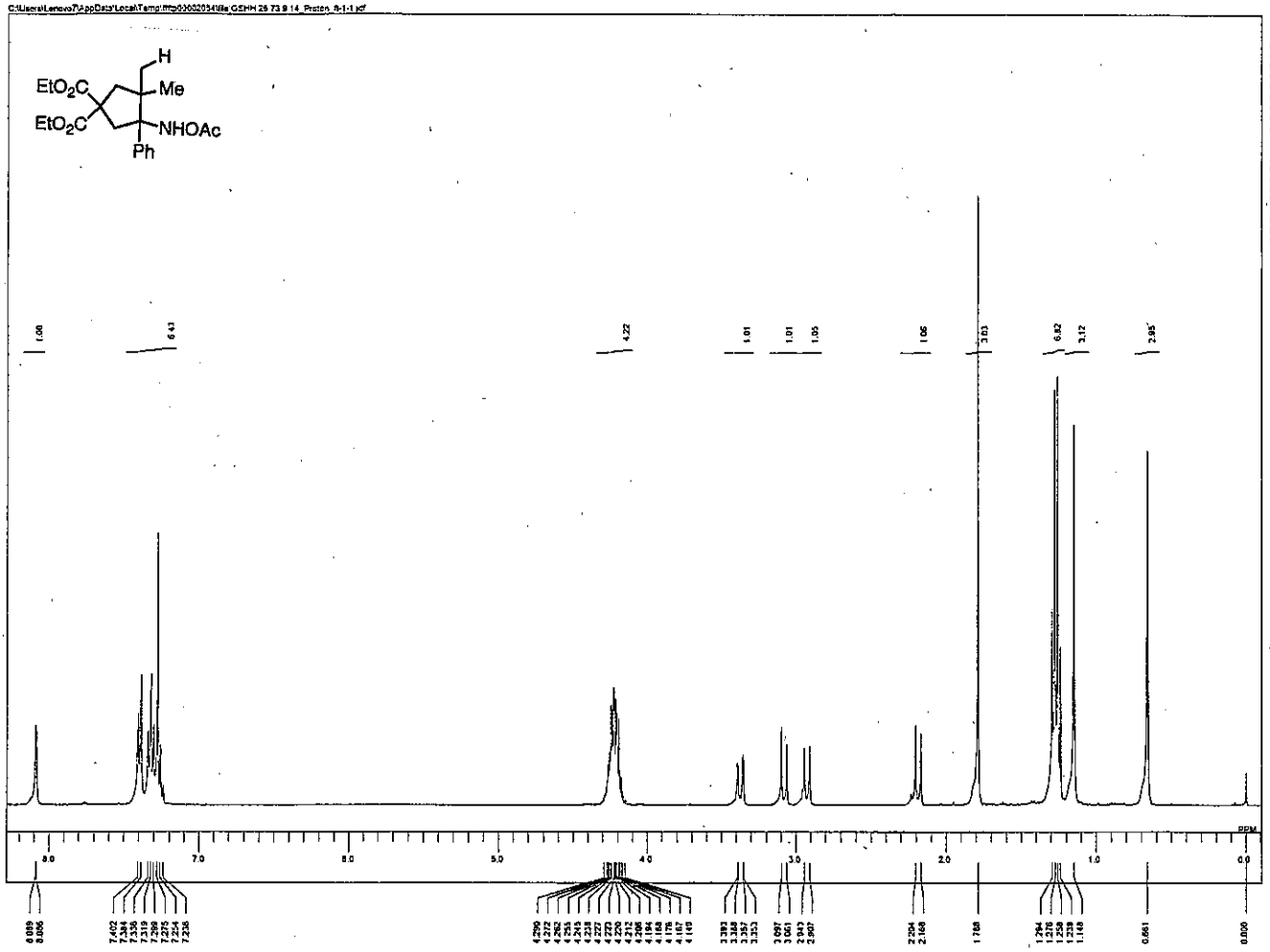
single pulse decoupled gated NOE
C:\Users\Lenovo7\AppData\Local\Temp\fftp0000275ct\file\GSHH 26 46 tm_Carbon_ft-1-1.jdf

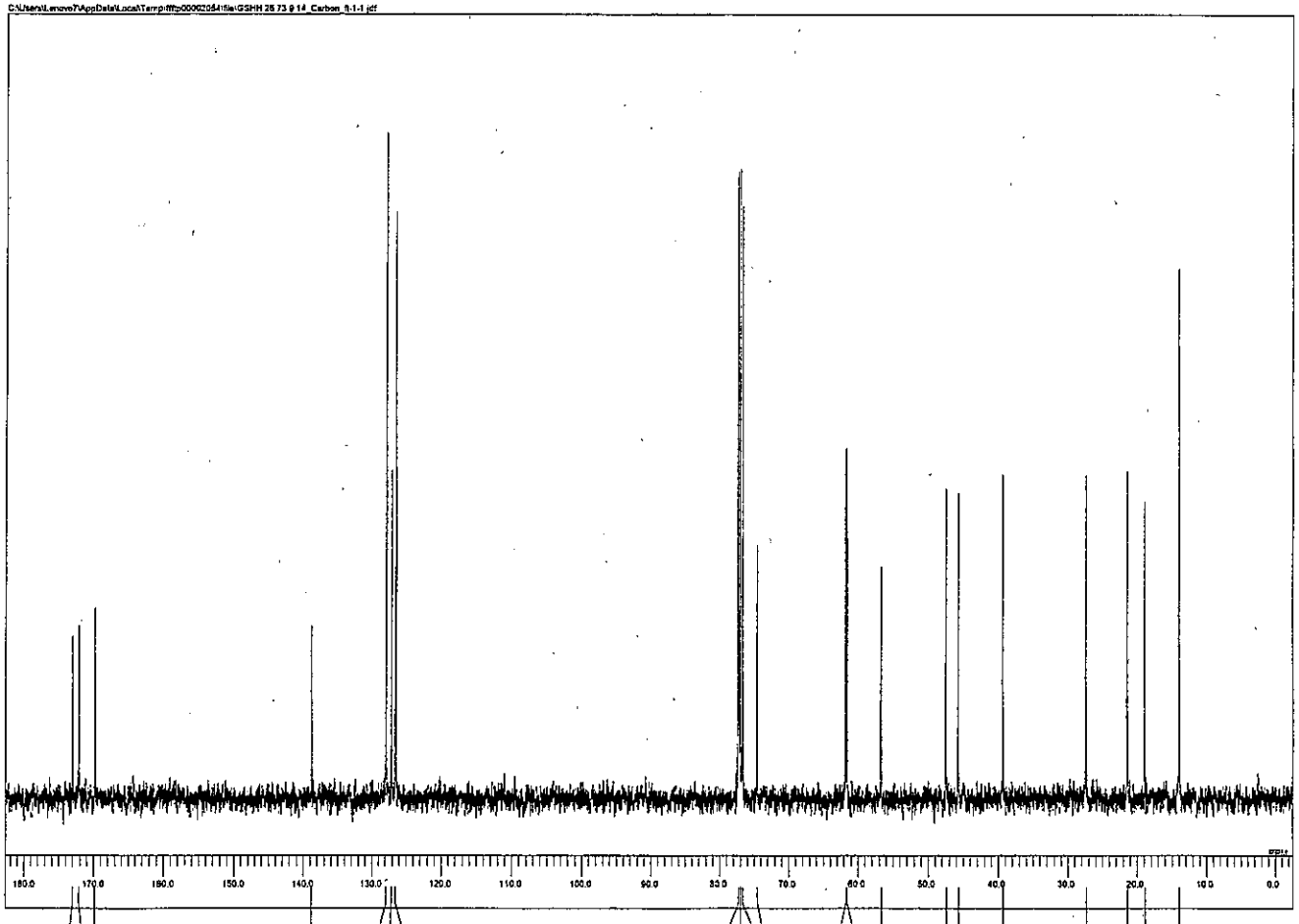


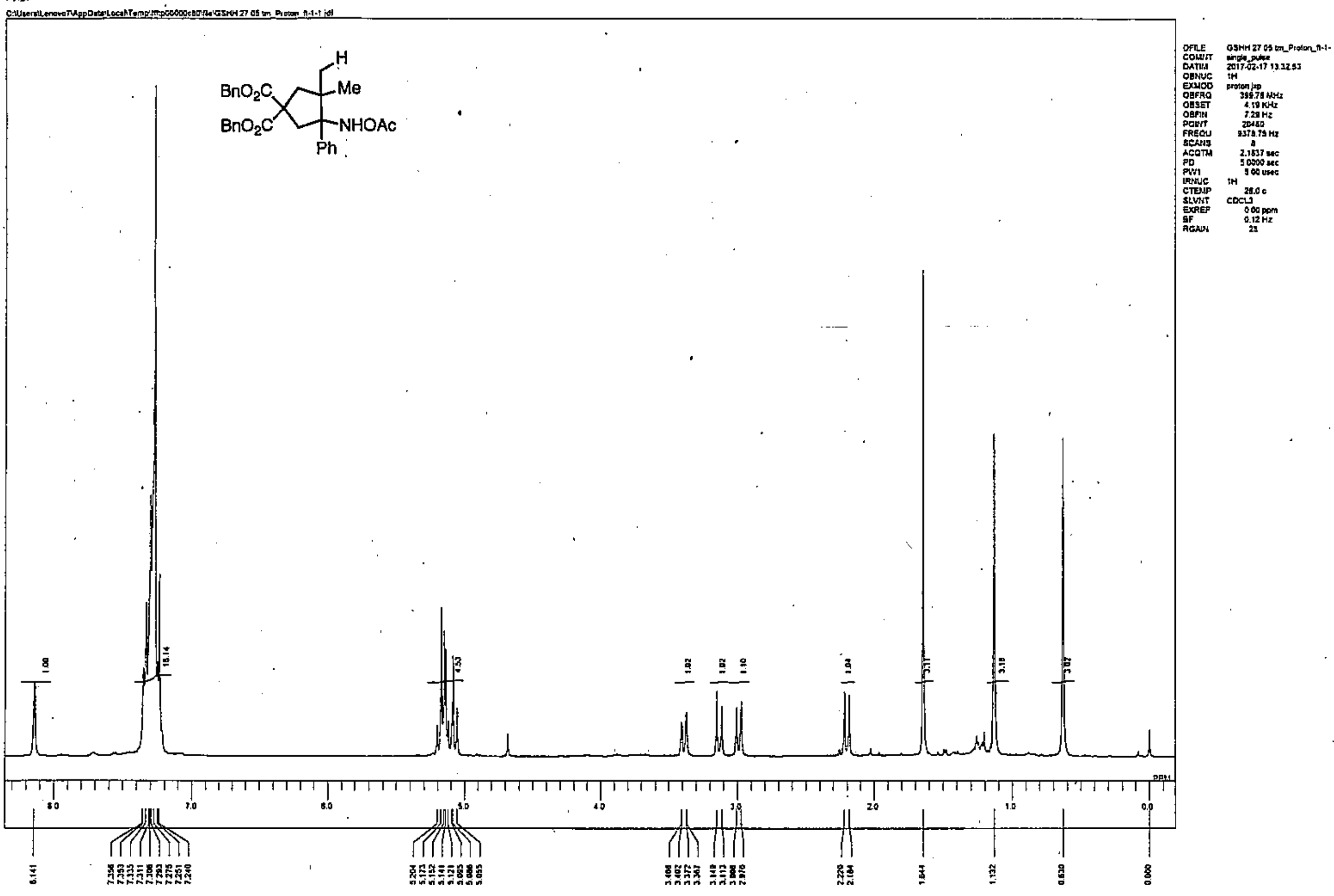
DFILE GSHH 26 46 tm_Carbon_ft-1-
COMNT single pulse decoupled gated
DATIM 2016-12-16 18:05:32
OBNUC 13C
EXMOD carbon.jxp
OBFRQ 100.53 MHz
OBSET 5.35 KHz
OBFIN 5.86 Hz
POINT 40960
FREQU 39258.79 Hz
SCANS 102
ACQTM 1.0433 sec
PD 2.0000 sec
PW1 2.87 usec
IRNUC 1H
CTEMP 19.2 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 3.00 Hz
RGAIN 50
PPM
170.0
160.0
150.0
140.0
130.0
120.0
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0.0
169.724
143.475
136.896
134.160
129.536
128.182
127.695
127.430
126.523
77.324
77.000
76.683
72.442
59.761
53.135
44.621
24.665
21.506
19.650
18.820


Me
Me
TsN
NHOTBS
Me
DFILE GSHH 26 55 4 6_Proton_fl-1-
COMNT single_pulse
DATIM 2016-12-22 12:14:44
OBNUC 1H
EXMOD proton.jxp
OBFRQ 399.78 MHz
OBSET 4.19 KHz
OBFIN 7.29 Hz
POINT 16384
FREQU 7404.00 Hz
SCANS 8
ACQTM 2.1663 sec
PD 5.0000 sec
PW1 2.72 usec
IRNUC 1H
CTEMP 21.4 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 1.00 Hz
RGAIN 36
2.00
2.16
0.84
1.78
0.55
3.02
3.08
5.83
2.76
2.75
5.95
PPM
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
7.724
7.715
7.314
7.295
7.253
4.417
3.842
3.158
3.135
3.083
3.056
2.415
0.960
0.818
0.807
0.800
0.839
0.775
0.931
0.900
-0.024

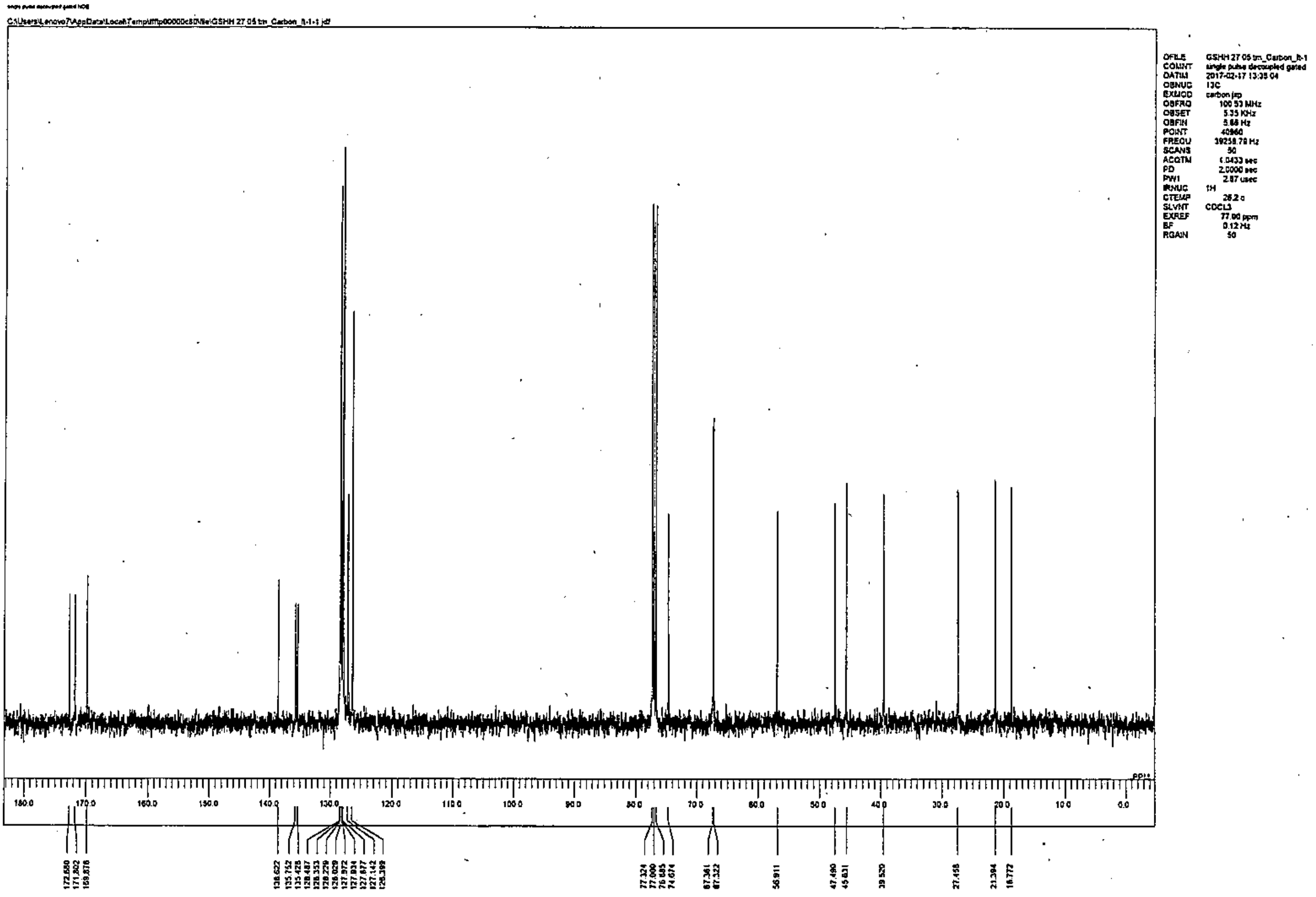

DFILE GSHH 26 55 4 6_Carbon_fl-1-
COMNT single pulse decoupled gated r
DATIM 2016-12-22 12:18:04
OBNUC 13C
EXMOD carbon.jxp
OBFRQ 100.53 MHz
OBSET 9.35 KHz
OBFIN 5.56 Hz
POINT 40960
FREQU 39457.97 Hz
SCANS 70
ACQTM 1.0381 sec
PD 2.0000 sec
PW1 3.52 usec
IRNUC 1H
CTEMP 21.4 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 1.00 Hz
RGAIN 50
PPM
150.0
140.0
130.0
120.0
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0.0
-10.0
143.217
134.362
129.619
127.399
77.326
77.000
76.684
66.136
59.855
54.184
43.000
26.144
24.385
21.516
19.111
17.822
15.877
-5.469
-5.545


EtO2C
EtO2C
Me
H
NHOAc
Ph
PPM

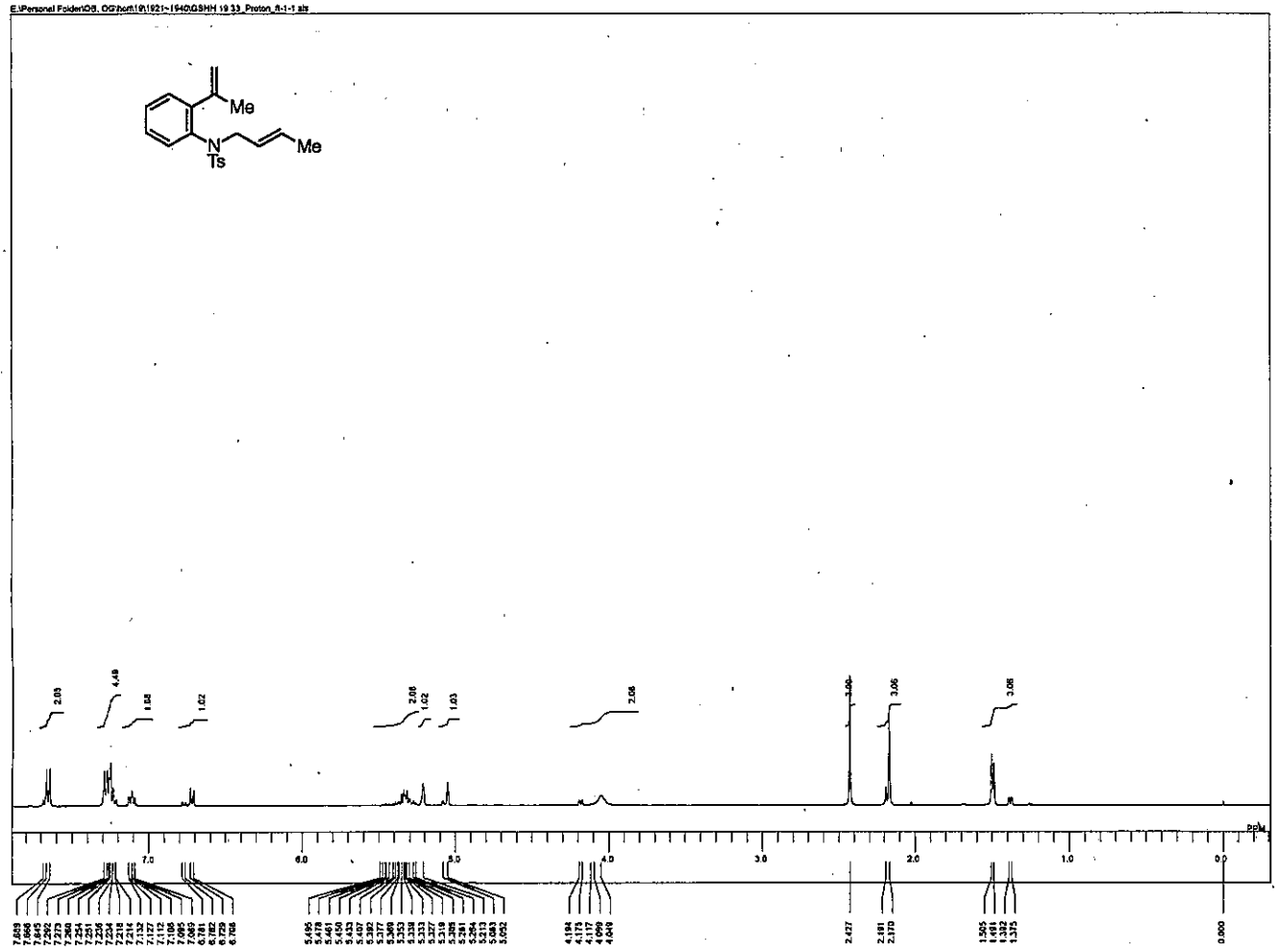

PPM

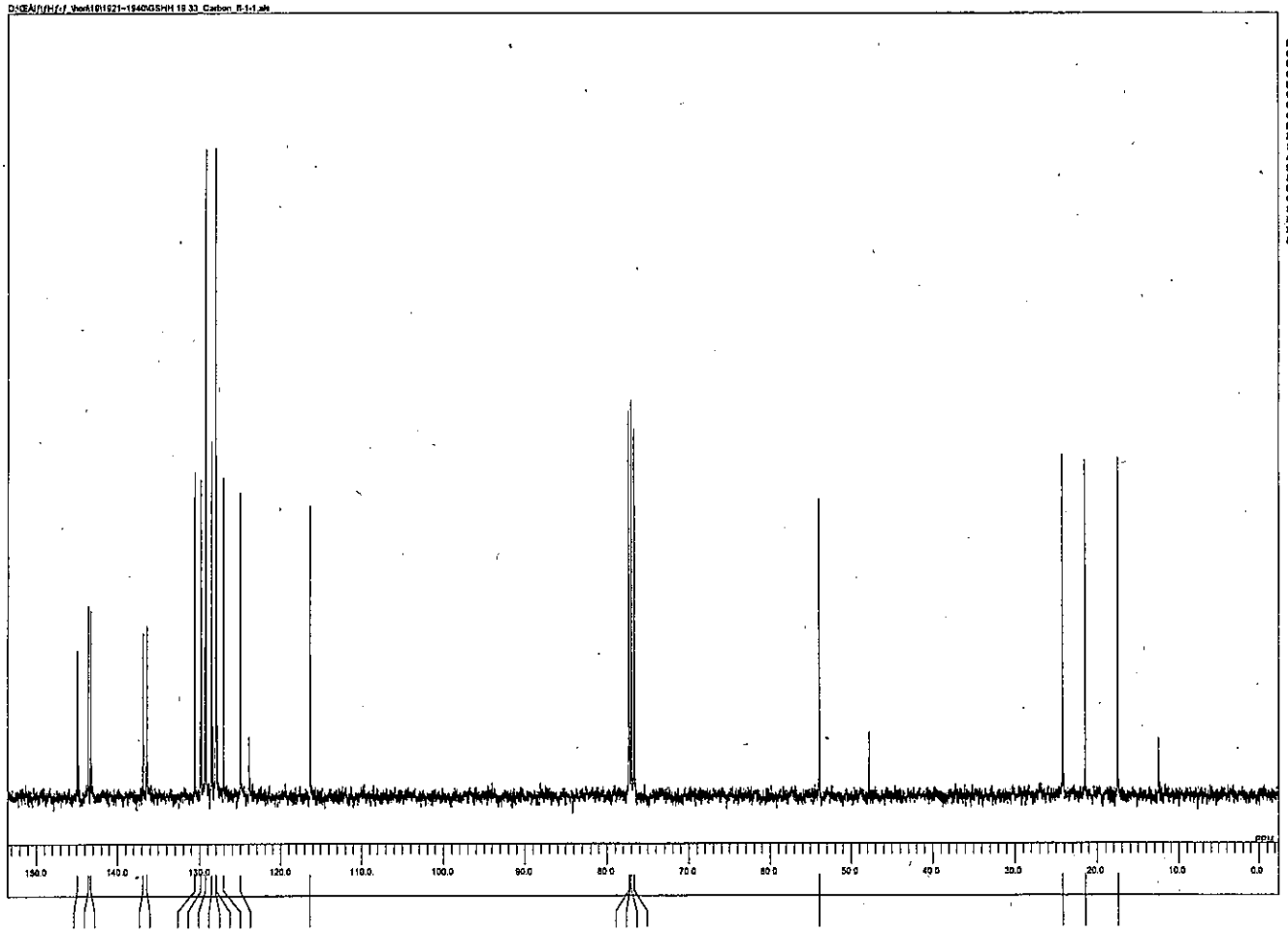


BnO2C
BnO2C
H
Me
NHOAc
Ph
DFILE GSHH 27 05 tm_Proton_ft-1-
COMNT single_pulse
DATIM 2017-02-17 13:32:53
OBNUC 1H
EXMOD proton.jxp
OBFRQ 399.78 MHz
SCANS 8
SLVNT CDCL3
1.00
15.14
4.53
1.92
1.92
1.10
1.94
3.11
3.18
3.02
8.141
1.644
1.132
0.630
0.000
ppm

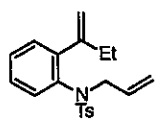


DFILE GSHH 27 05 tm_Carbon_ft-1
COMNT single pulse decoupled gated
DATIM 2017-02-17 13:35:04
OBNUC 13C
EXMOD carbon.jxp
OBFRQ 100.53 MHz
SCANS 50
SLVNT CDCL3
EXREF 77.00 ppm
172.680
171.802
169.878
136.622
135.752
135.428
77.324
77.000
76.685
74.674
67.361
67.322
56.911
47.490
45.631
39.520
27.468
21.394
18.772
ppm


Me
N
Ts
Me
2.00
4.49
1.58
1.02
2.06
1.02
1.03
2.06
3.00
3.06
3.08
ppm
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0


ppm
150.0
140.0
130.0
120.0
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0.0

single pulse
C:\Users\Lenovo7\AppData\Local\Temp\GSHH 19 71_Proton_ft-1-1.jdf



2.00 4.42 1.00 0.96 0.92 1.00 1.02 1.99 1.99 5.08 3.05

PPM
7.0 6.0 5.0 4.0 3.0 2.0 1.0 0.0

7.708 7.687 7.315 7.295 7.272 7.258 7.239 7.235 7.225 7.221 7.207 7.201 7.159 7.154 7.139 7.135 7.122 7.116 6.810 6.790 5.721 5.703 5.694 5.685 5.677 5.660 5.652 5.643 5.635 5.618 5.215 5.210 5.043 4.977 4.965 4.962 4.953 4.923 4.920 4.123 4.106
2.523 2.504 2.486 2.450
1.088 1.070 1.050
0.000

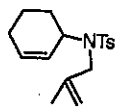
single pulse decoupled gated NOE
C:\Users\Lenovo7\AppData\Local\Temp\GSHH 19 71_Carbon_ft-1-1.jdf

PPM
150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0 0.0

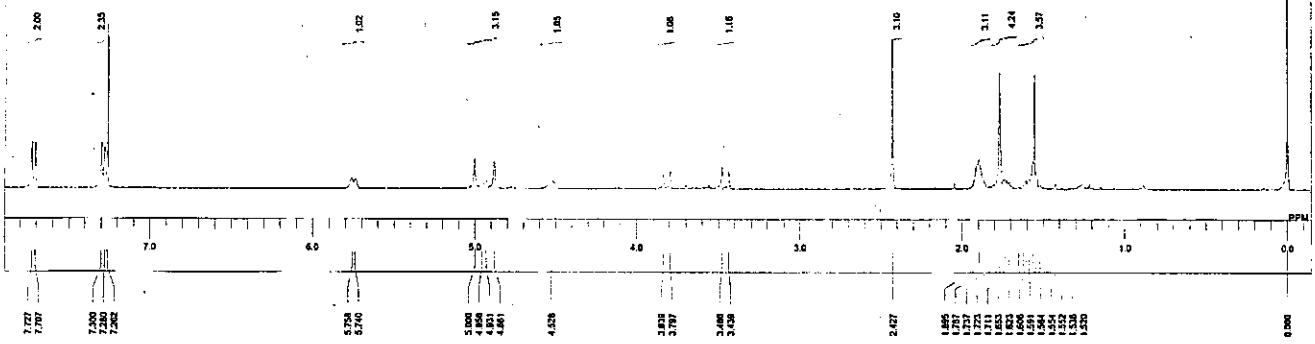
49.762 44.635 43.408 37.112 36.490 32.618 30.721 29.465 28.785 28.162 28.028 27.156 19.107 14.191
77.326 77.000 76.684
64.557
29.671 21.545 12.527

single_pulse

C:\Users\Lenovo\AppData\Local\Temp\ftp00002\f0\fid\GSHH 25 61 last_Proton_fi-1-1.jdf



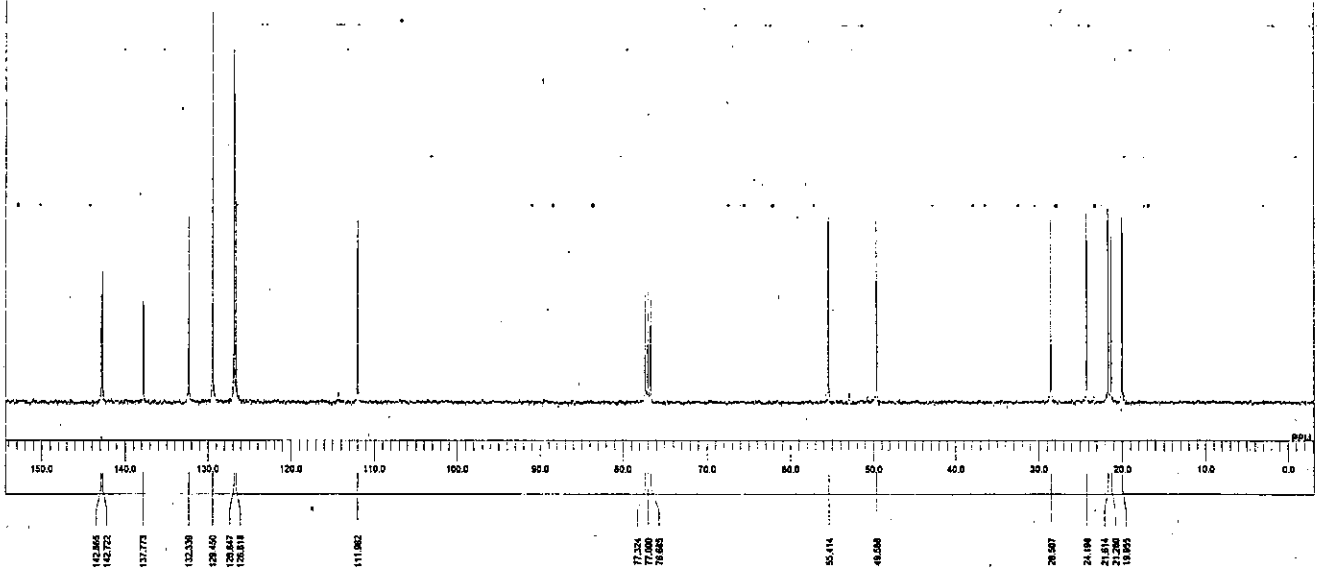

```
DFILE     GSHH 25 61 last_Proton_
COMNT     single_pulse
DATIM     2016-09-30 17:03:15
OBNUC     1H
EXMOD     proton.jxp
OBFRQ     399.78 MHz
OBSET     4.19 KHz
OBFIN     7.29 Hz
POINT     20480
FREQU     9378.75 Hz
SCANS     8
ACQTM     2.1837 sec
PD        5.0000 sec
PW1       5.00 usec
IRNUC     1H
CTEMP     21.0 c
SLVNT     CDCL3
EXREF     0.00 ppm
BF        1.00 Hz
RGAIN     48
```



single pulse decoupled gated NOE

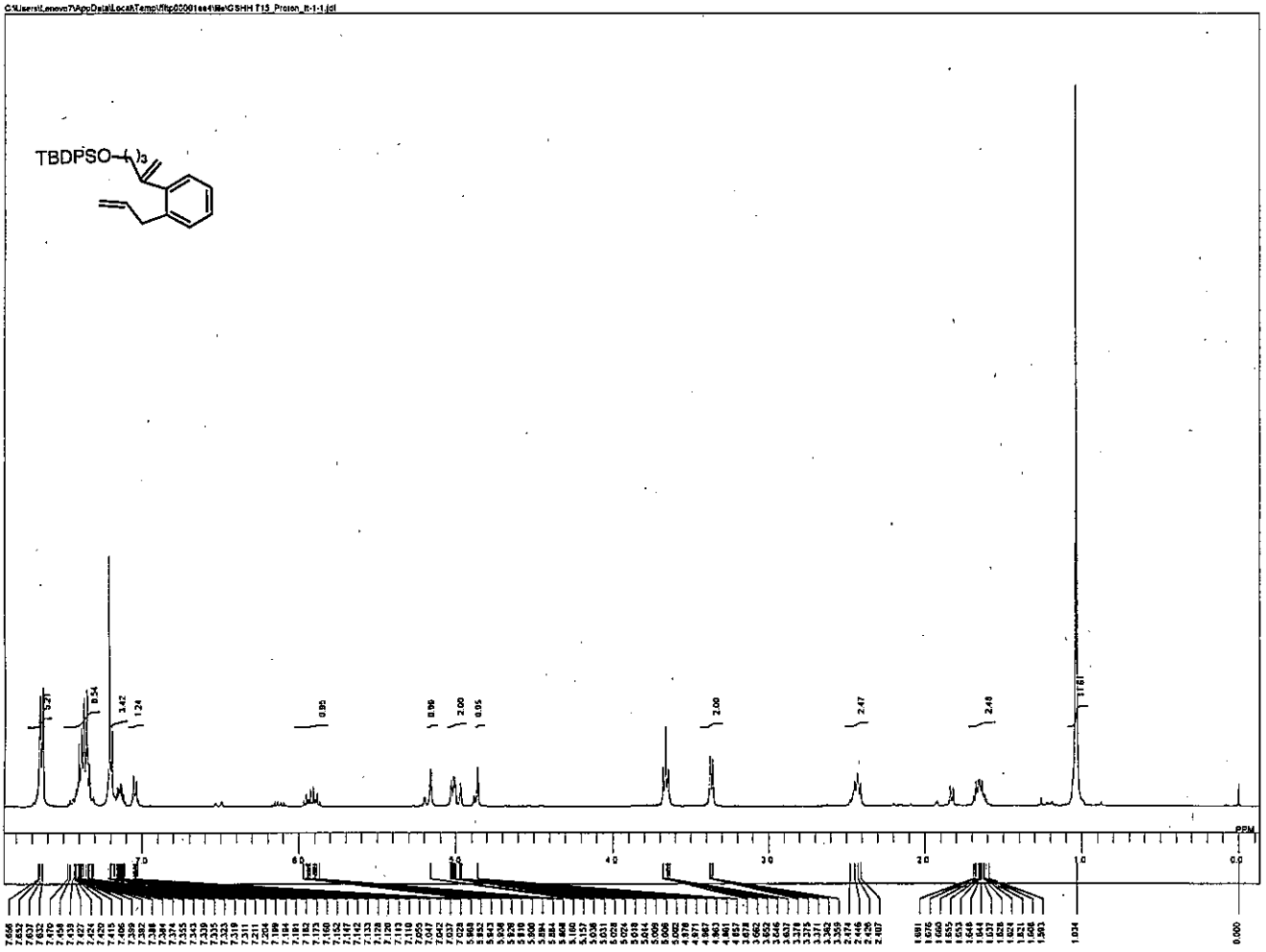
C:\Users\Lenovo\AppData\Local\Temp\ftp00002\f0\fid\GSHH 25 61 last_Carbon_fi-1-1.jdf

```
DFILE     GSHH 25 61 last_Carbon_
COMNT     single pulse decoupled gat
DATIM     2016-09-30 17:24:15
OBNUC     13C
EXMOD     carbon.jxp
OBFRQ     100.53 MHz
OBSET     5.35 KHz
OBFIN     5.66 Hz
POINT     40960
FREQU     39256.79 Hz
SCANS     50
ACQTM     1.0433 sec
PD        2.0000 sec
PW1       2.87 usec
IRNUC     1H
CTEMP     21.2 c
SLVNT     CDCL3
EXREF     77.00 ppm
BF        1.00 Hz
RGAIN     50
```



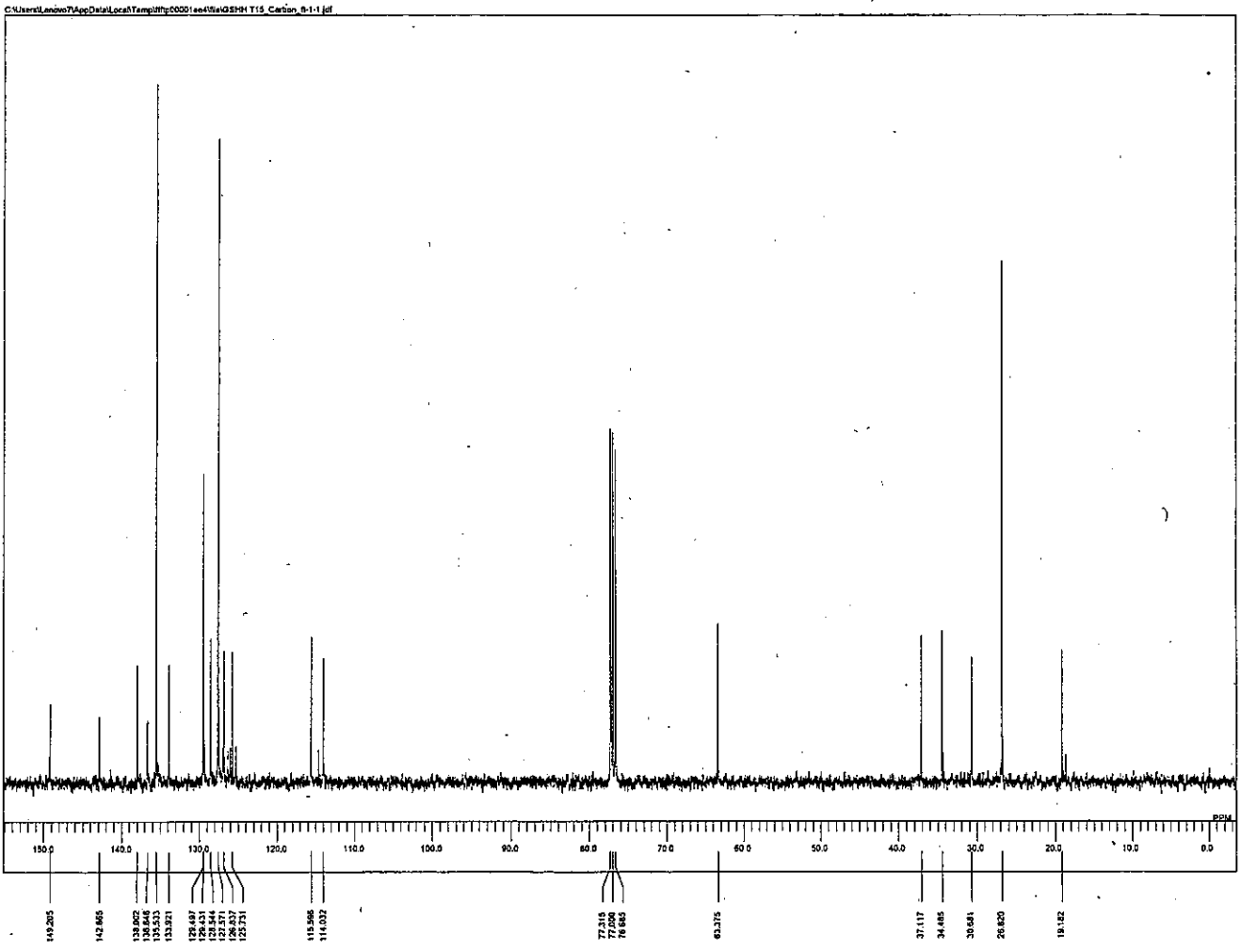
single_pulse

C:\Users\Lenovo7\AppData\Local\Temp\ffpc00001ee4\file\GSHH T15_Proton_ft-1-1.jdf

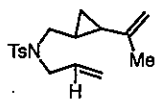


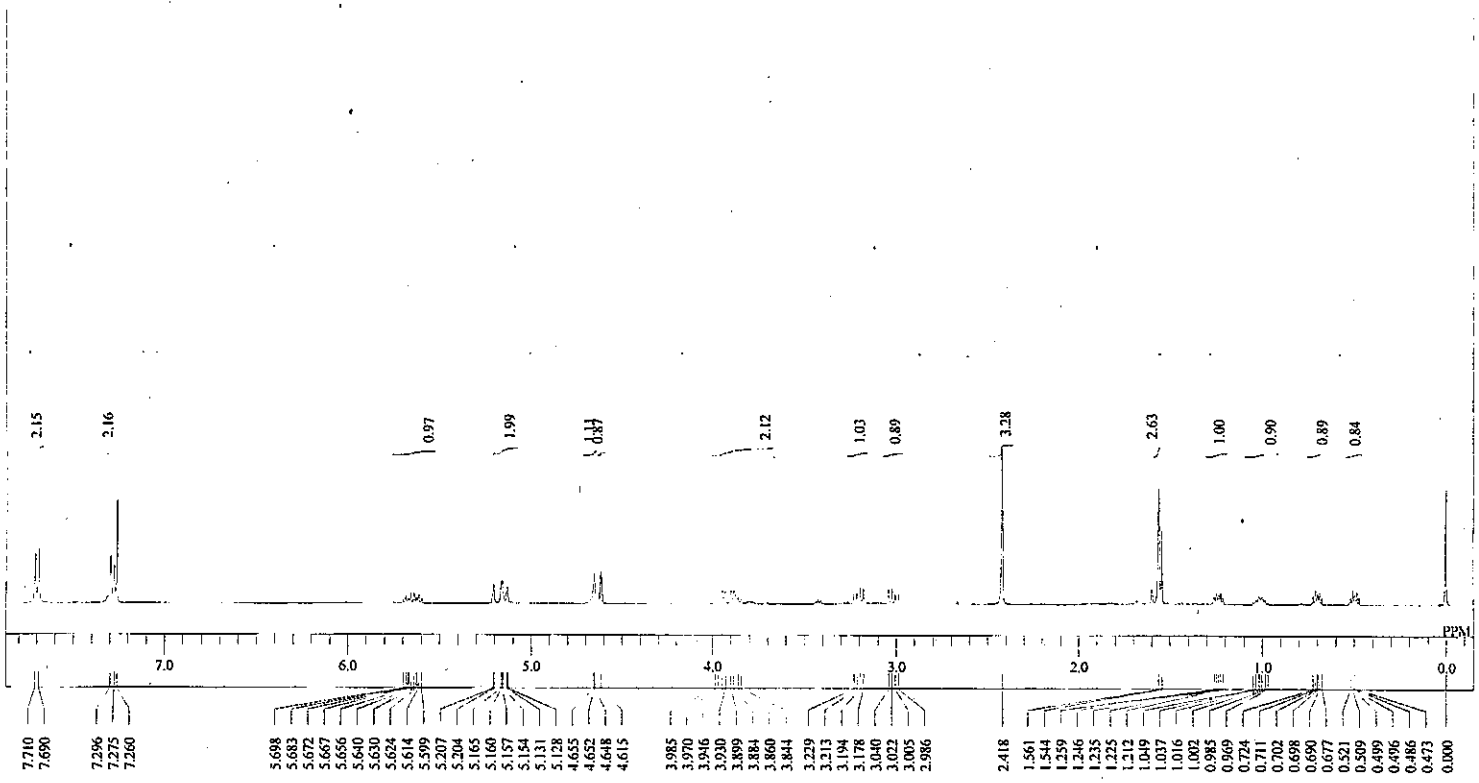
single pulse decoupled gated NOE

C:\Users\Lenovo7\AppData\Local\Temp\ffpc00001ee4\file\GSHH T15_Carbon_ft-1-1.jdf

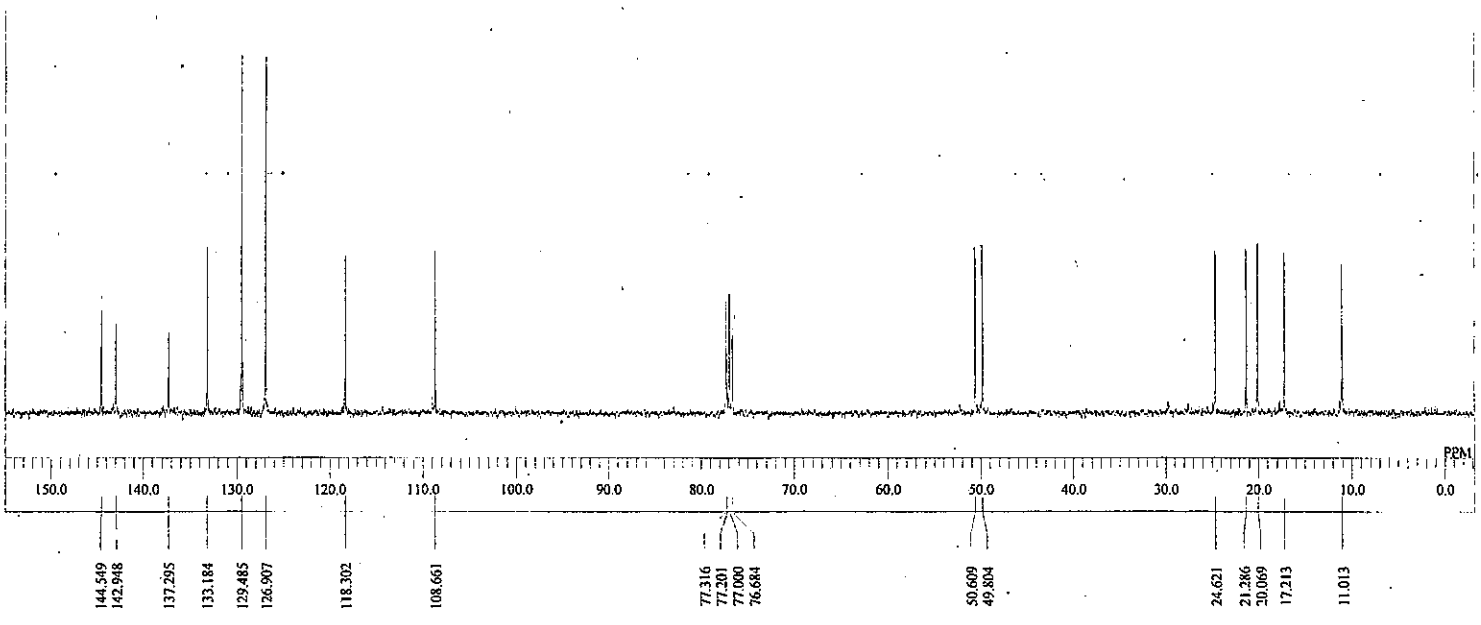


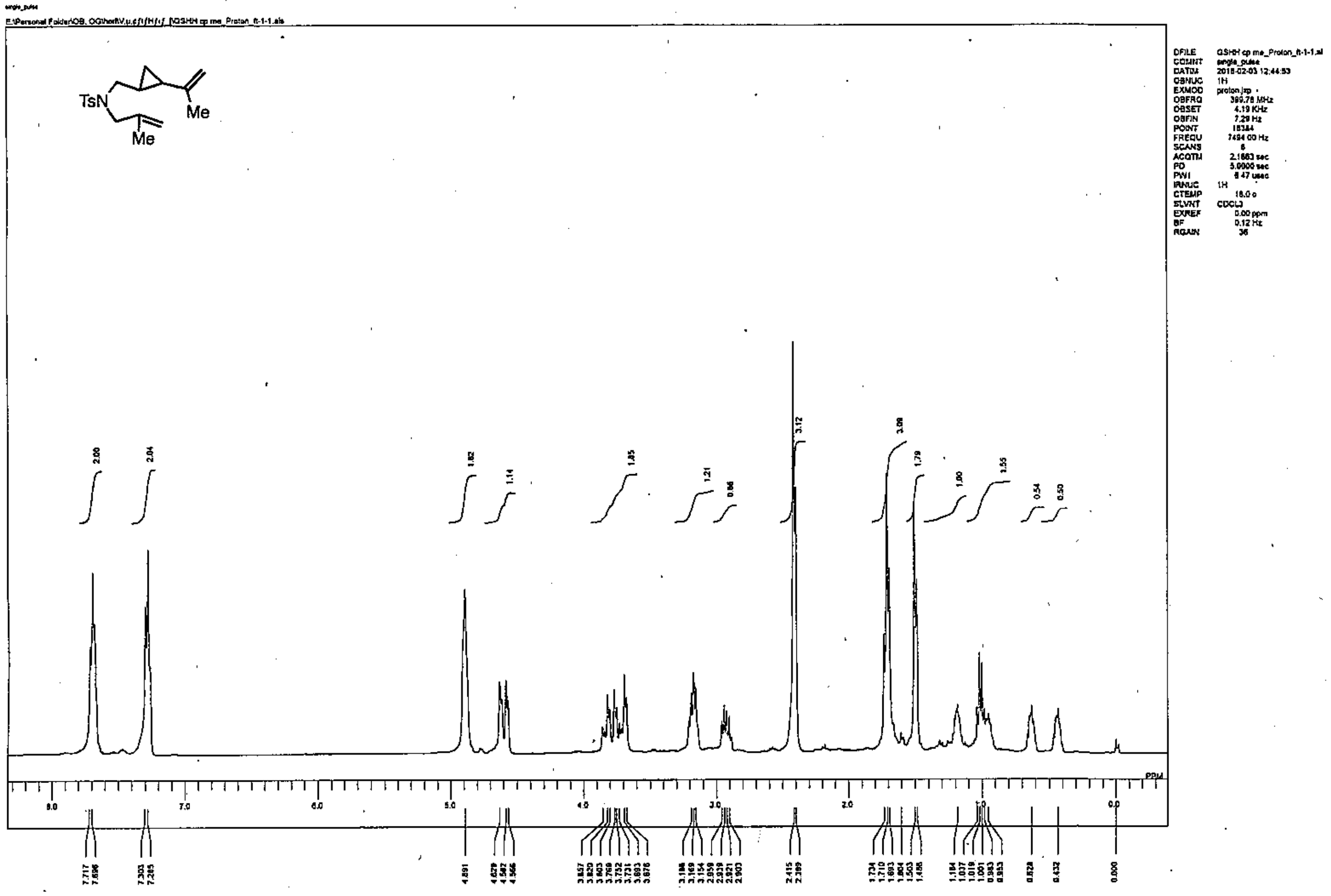
single_pulse
C:\Users\Lenovo7\AppData\Local\Temp\ffffp000016ac\file\GSHH 24 55_Proton_ft-1-1.jdf

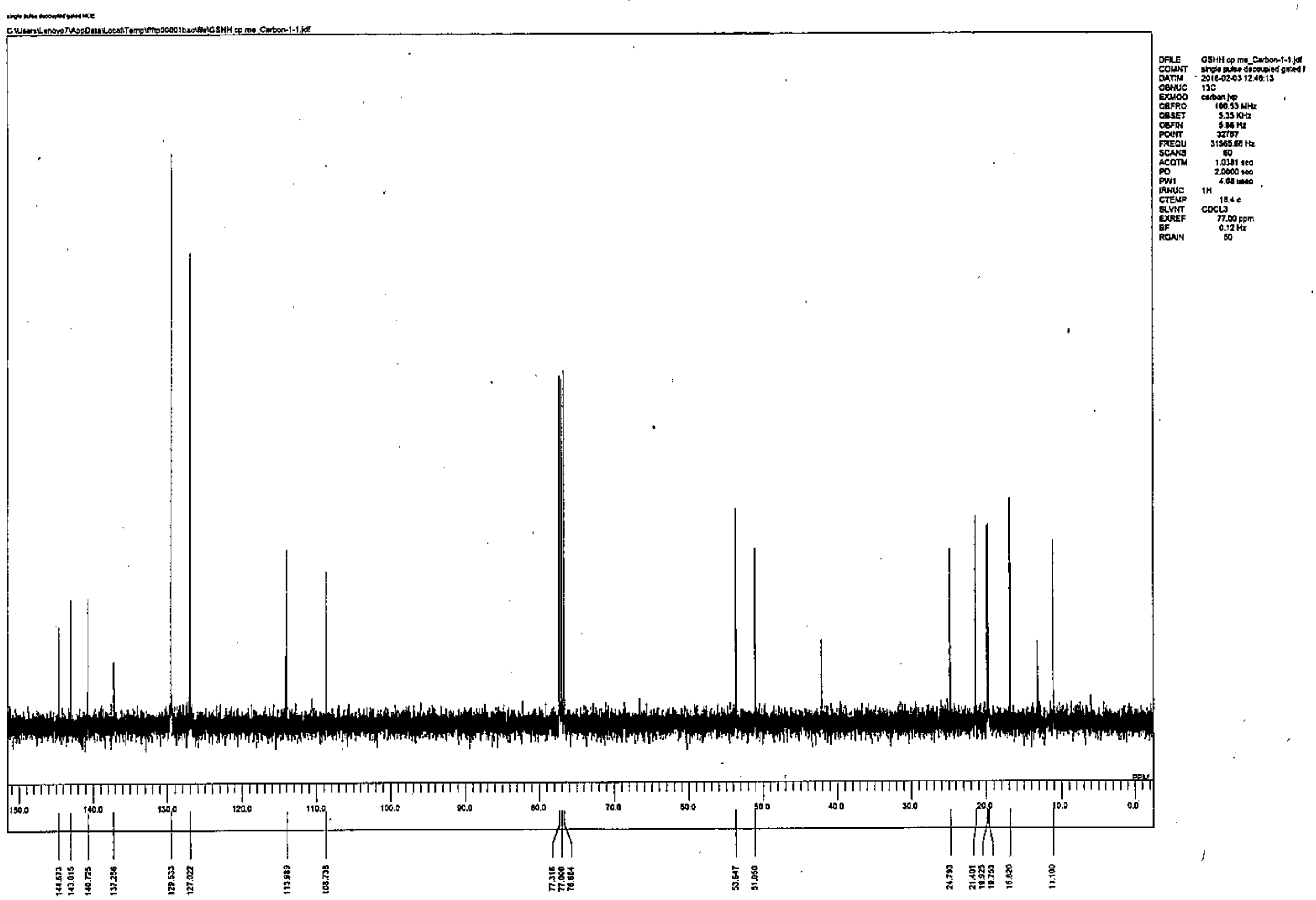




single pulse decoupled gated NOE
C:\Users\Lenovo7\AppData\Local\Temp\ffffp000010c8\file\GSHH 24 55 13c_Carbon_ft-1-1.jdf

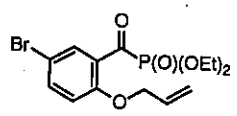



single_pulse
TsN
Me
Me
2.00
2.04
1.82
1.14
1.85
1.21
0.86
3.12
3.08
1.79
1.90
1.55
0.54
0.50
PPM
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
7.717
7.696
7.303
7.285
4.891
4.629
4.582
4.566
2.415
2.399
0.528
0.432
0.000
DFILE GSHH cp me_Proton_ft-1-1.al
COMNT single_pulse
DATIM 2018-02-03 12:44:53
OBNUC 1H
EXMOD proton.jxp
OBFRQ 399.78 MHz
OBSET 4.19 KHz
OBFIN 7.29 Hz
POINT 16384
FREQU 7494.00 Hz
SCANS 8
ACQTM 2.1863 sec
PD 5.0000 sec
PW1 6.47 usec
IRNUC 1H
CTEMP 18.0 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 36

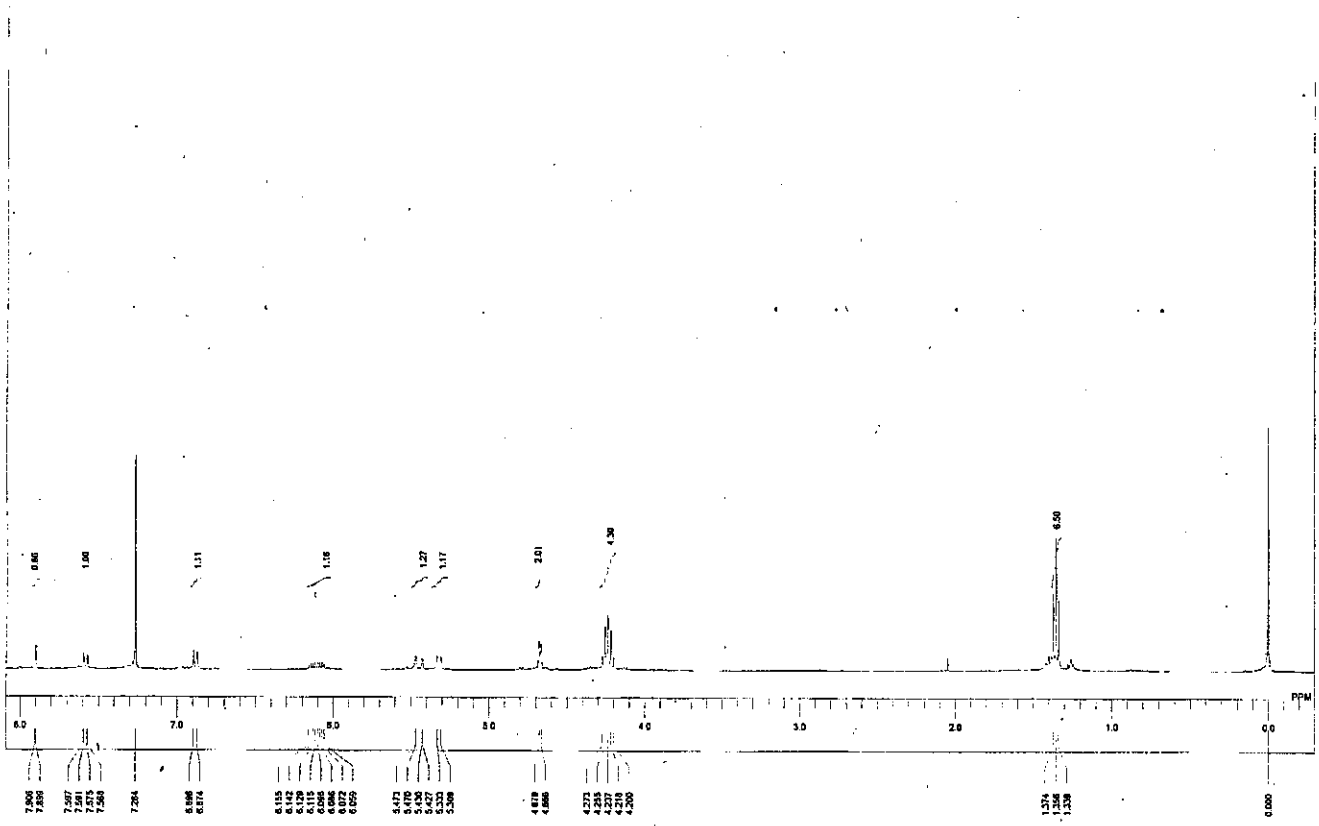

single pulse decoupled gated NOE
PPM
150.0
140.0
130.0
120.0
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0.0
144.573
143.015
140.725
137.256
129.533
127.022
113.989
108.738
77.316
77.000
76.684
53.647
51.050
24.793
21.401
19.925
19.753
15.820
11.100
DFILE GSHH cp me_Carbon-1-1.jdf
COMNT single pulse decoupled gated I
DATIM 2018-02-03 12:46:13
OBNUC 13C
EXMOD carbon.jxp
OBFRQ 100.53 MHz
OBSET 5.35 KHz
OBFIN 5.86 Hz
POINT 32767
FREQU 31565.66 Hz
SCANS 60
ACQTM 1.0381 sec
PD 2.0000 sec
PW1 4.08 usec
IRNUC 1H
CTEMP 18.4 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 50

single_pulse

C:\Users\Lenovo7\AppData\Local\Temp\GSHH 22 33 Br last_Proton_ft-1-1.jdf



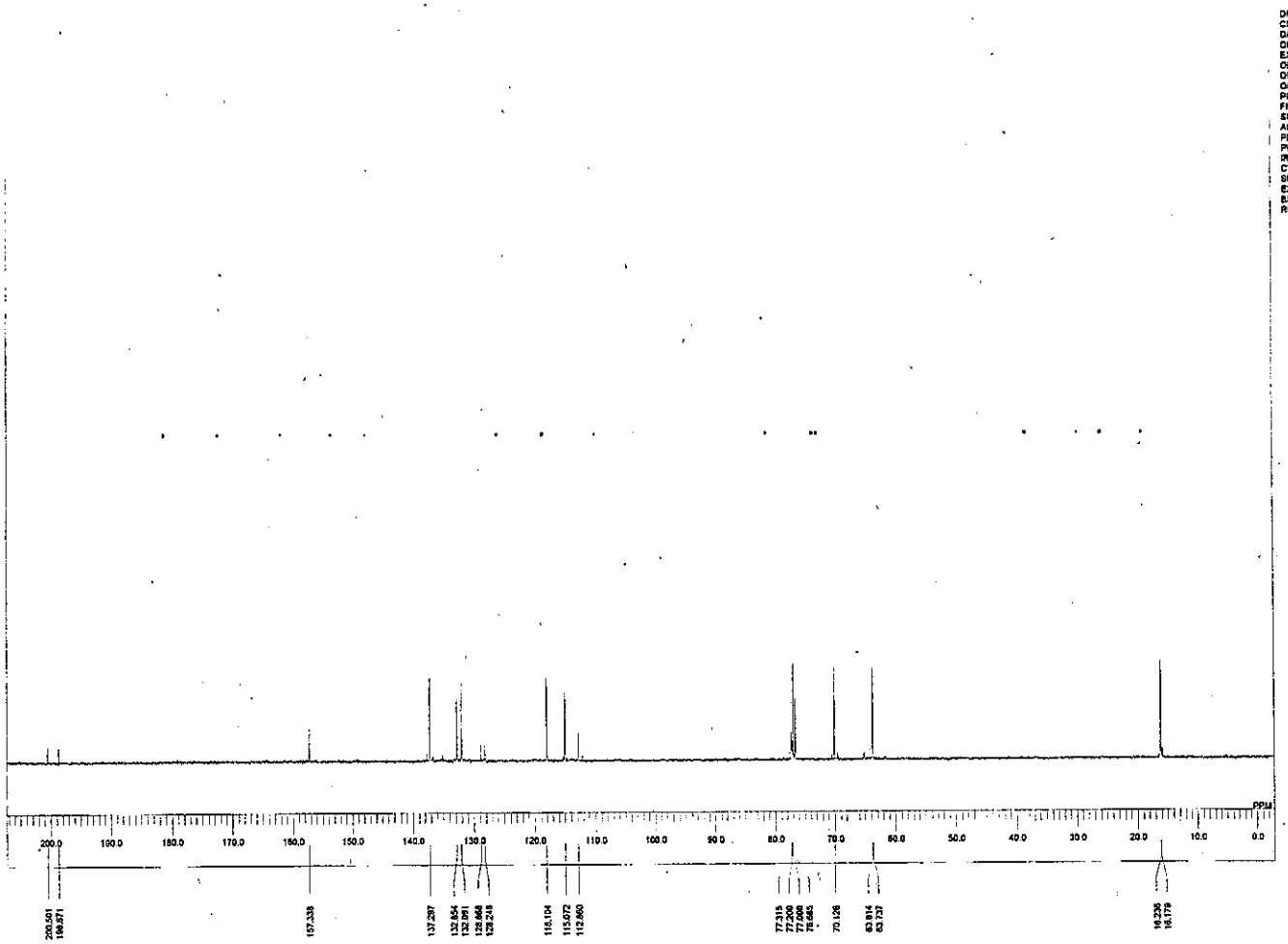
DFILE	GSHH 22 33 Br last_Proto
COMNT	single_pulse
DATIM	2018-01-09 19:41:58
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	399.76 MHz
OBSET	4.19 KHz
OBFIN	7.29 Hz
POINT	20480
FREQU	9367.51 Hz
SCANS	4
ACQTM	2.1863 sec
PD	5.0000 sec
PW1	2.72 usec
IRNUC	1H
CTEMP	19.3 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.12 Hz
RGAIN	56



single pulse decoupled gated NOE

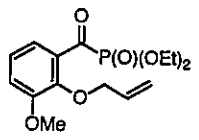
C:\Users\Lenovo7\AppData\Local\Temp\GSHH 22 xx br last_Carbon_ft-1-1.jdf

DFILE	GSHH 22 xx br last_Carbon
COMNT	single pulse decoupled gate
DATIM	2018-01-13 12:50:22
OBNUC	13C
EXMOD	carbon.jxp
OBFRQ	100.53 MHz
OBSET	5.35 KHz
OBFIN	5.86 Hz
POINT	40960
FREQU	39258.79 Hz
SCANS	70
ACQTM	1.0433 sec
PD	2.0000 sec
PW1	2.77 usec
IRNUC	1H
CTEMP	22.1 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	50

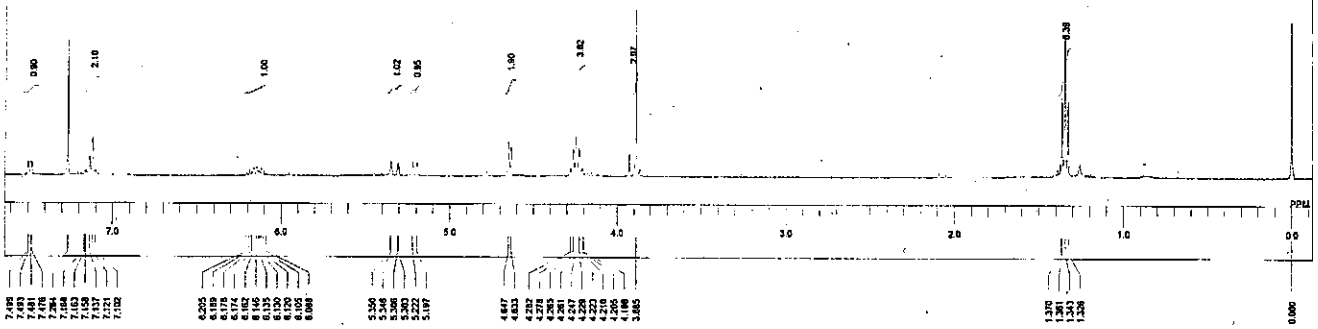


single_pulse

C:\Users\Lenovo7\AppData\Local\Temp\GSHH 22 37 ome last impure_Proton_fi-1-1.fid



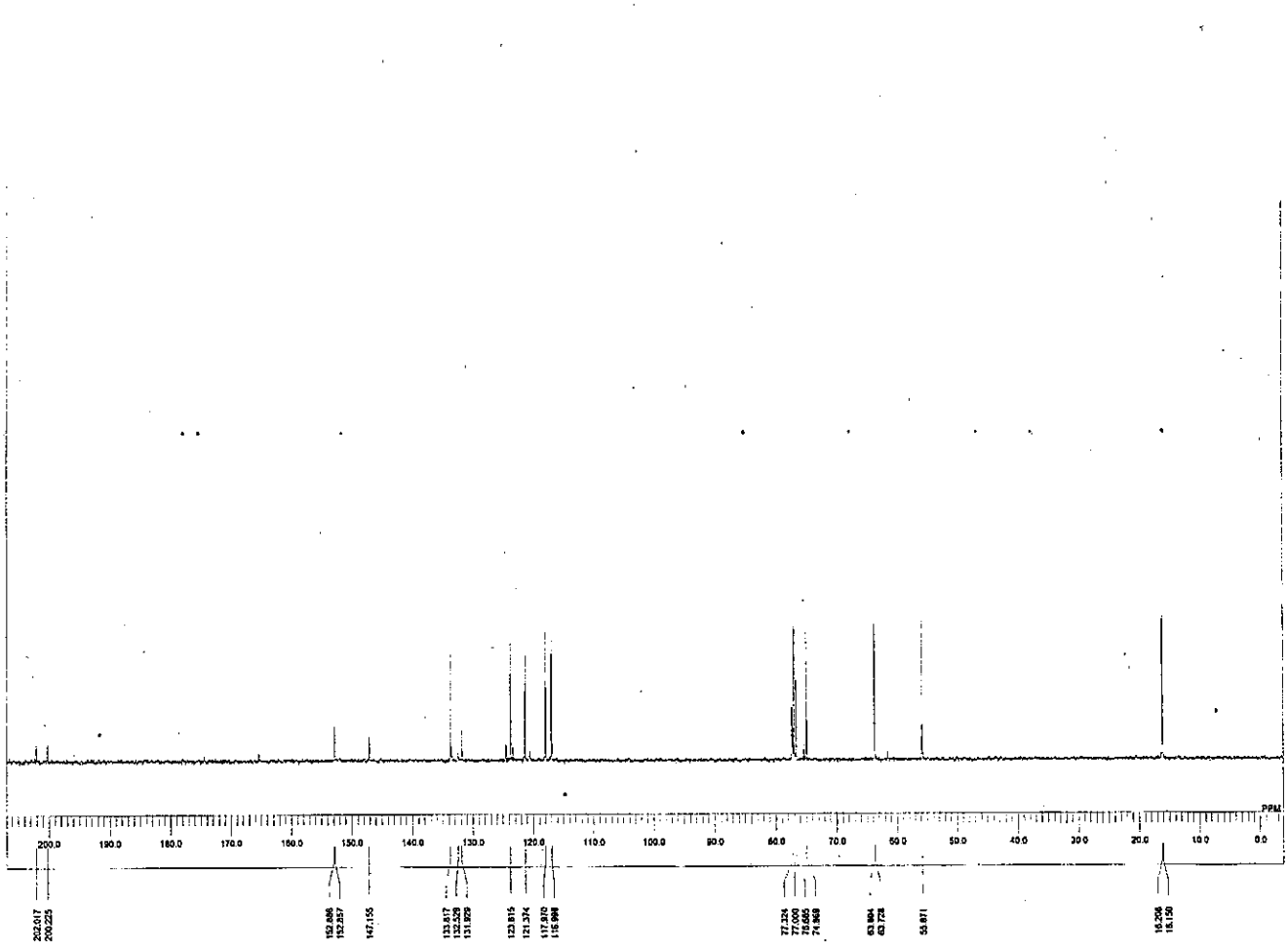
DFILE GSHH 22 37 ome last imp
COMNT single_pulse
DATIM 2016-01-09 19:38:04
OBNUC 1H
EXMOD proton.jxp
OBFRQ 399.78 MHz
OBSET 4.19 KHz
OBFIN 7.29 Hz
POINT 20480
FREQU 9367.51 Hz
SCANS 4
ACQTM 2.1863 sec
PD 5.0000 sec
PW1 2.72 usec
IRNUC 1H
CTEMP 19.3 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 56



single pulse decoupled gated NOE

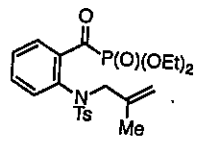
C:\Users\Lenovo7\AppData\Local\Temp\GSHH 22 xx ome last_Carbon_fi-1-1.fid

DFILE GSHH 22 xx ome last_Carl
COMNT single pulse decoupled gat
DATIM 2016-01-13 12:45:19
OBNUC 13C
EXMOD carbon.jxp
OBFRQ 100.53 MHz
OBSET 9.33 KHz
OBFIN 5.66 Hz
POINT 40960
FREQU 39258.78 Hz
SCANS 50
ACQTM 1.0433 sec
PD 2.0000 sec
PW1 2.77 usec
IRNUC 1H
CTEMP 22.1 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 50



single_pulse

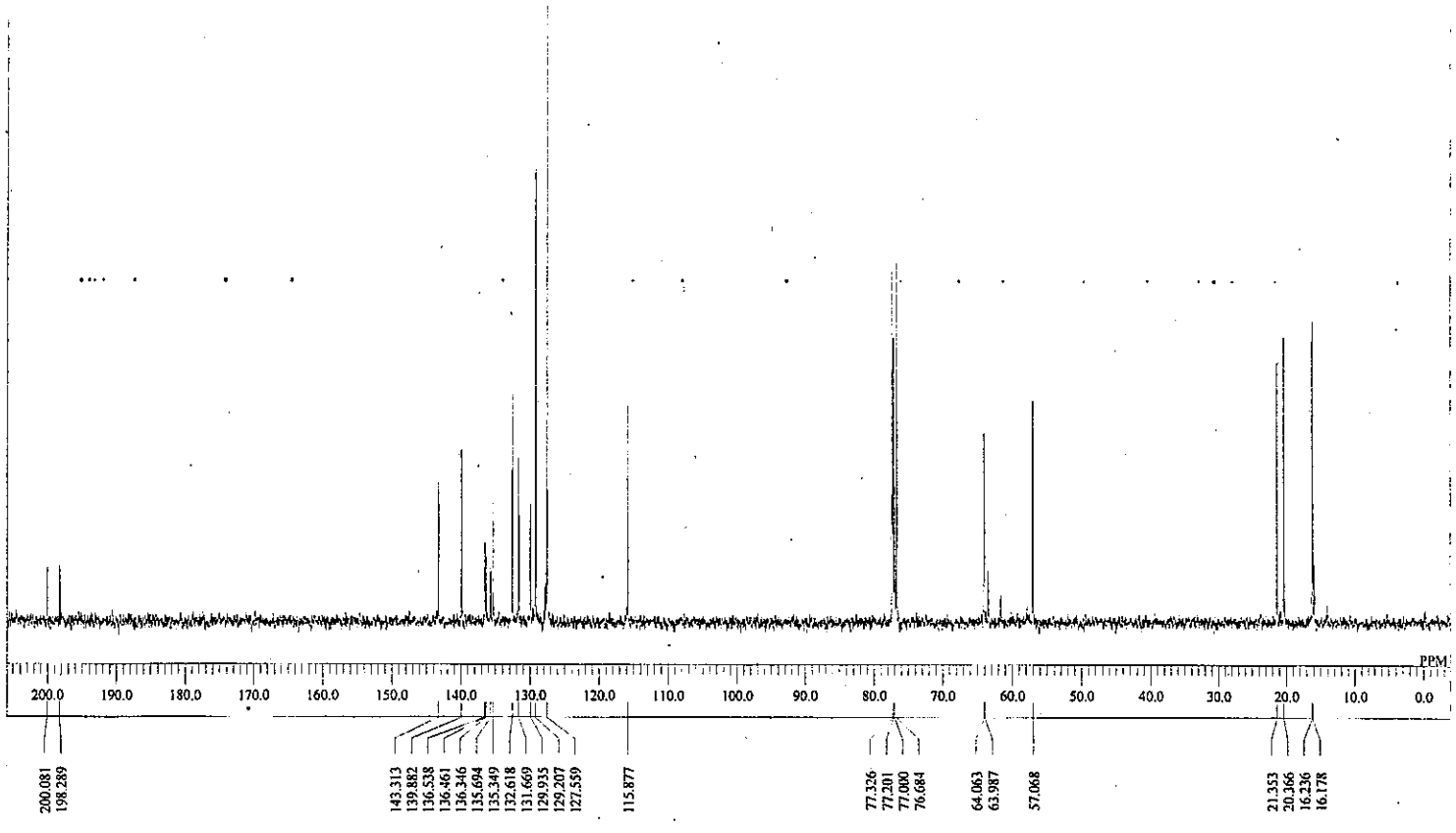
C:\Users\Lenovo7\AppData\Local\Temp\GSHH 22 05 last_Proton_ft-1-1.jdf



DFILE GSHH 22 05 last_Proton_
COMNT single_pulse
DATIM 2018-01-19 22:10:16
OBNUC 1H
EXMOD proton.jxp
OBFRQ 399.78 MHz
OBSET 4.19 KHz
OBFIN 7.29 Hz
POINT 20480
FREQU 9387.51 Hz
SCANS 8
ACQTM 2.1805 sec
PD 5.0000 sec
PW1 2.72 usec
IRNUC 1H
CTEMP 18.0 c
SLVNT CDCL3
EXREF 0.00 ppm
RF 0.12 Hz
RGAIN 56

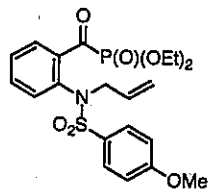
single pulse decoupled gated NOE

C:\Users\Lenovo7\AppData\Local\Temp\GSHH 22 05 last_Carbon_ft-1-1.jdf

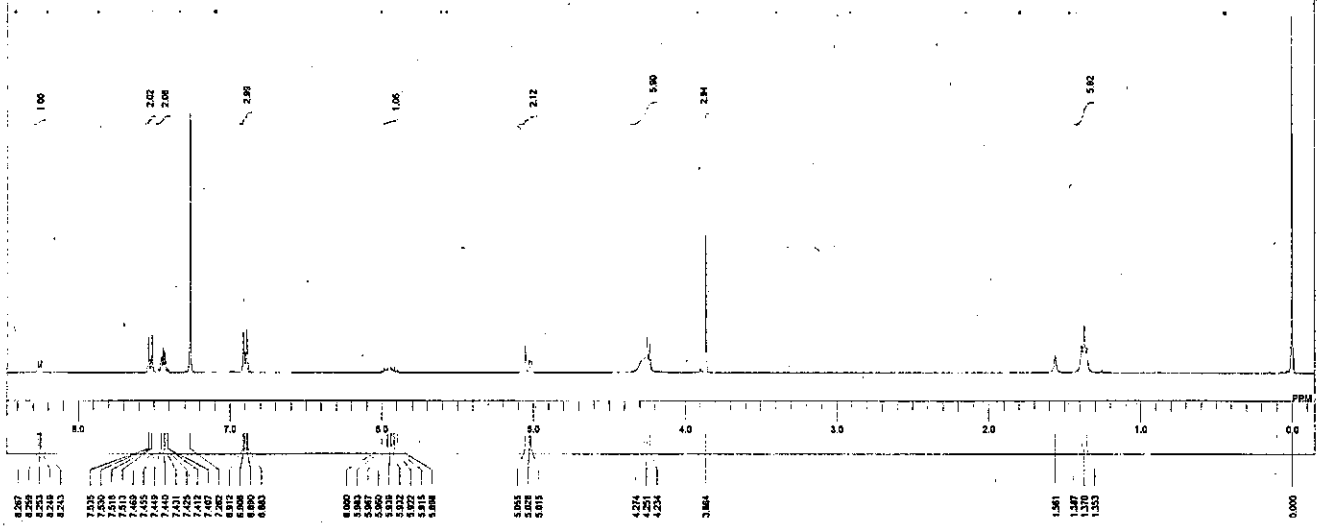


single_pulse

C:\Users\Lenovo7\AppData\Local\Temp\GSHH 22 09 pure h_Proton_fi-1-1.jdf



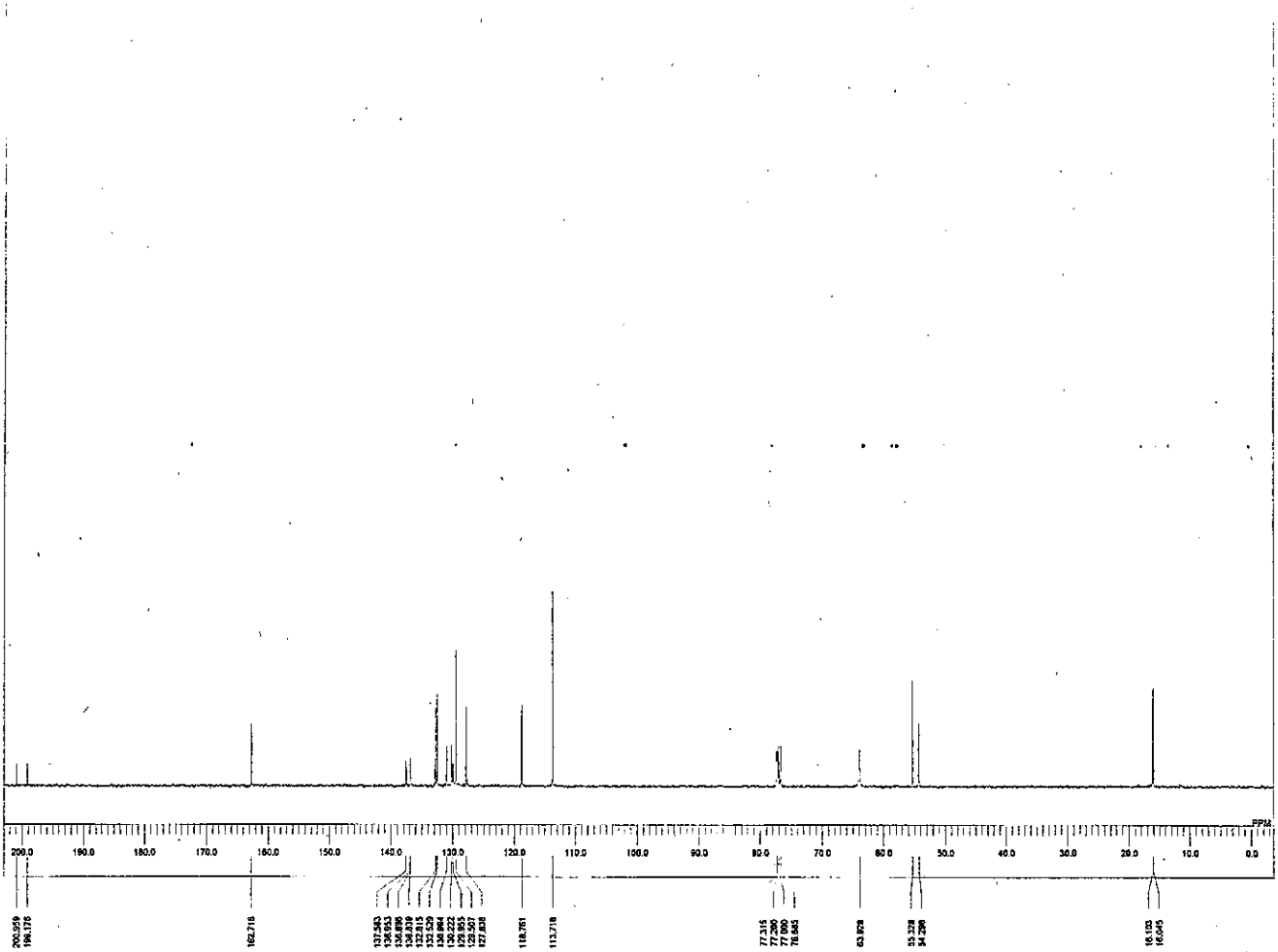
DFILE GSHH 22 09 pure h_Proton
COMNT single_pulse
DATIM 2016-01-05 09:46:21
OBNUC 1H
EXMOD proton.jxp
OBFRQ 399.78 MHz
OBSET 4.19 KHz
OBFIN 7.29 Hz
POINT 26480
FREQU 9578.75 Hz
SCANS 8
ACQTM 2.1837 sec
PD 5.0000 sec
PW1 5.01 usec
IRNUC 1H
CTEMP 21.6 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 54



single pulse decoupled gated NOE

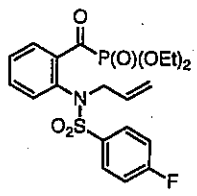
C:\Users\Lenovo7\AppData\Local\Temp\GSHH 22 09_Carbon_fi-1-1.jdf

DFILE GSHH 22 09_Carbon_fi-1
COMNT single pulse decoupled ga
DATIM 2015-12-22 14:45:06
OBNUC 13C
EXMOD carbon.jxp
OBFRQ 100.53 MHz
OBSET 6.35 KHz
OBFIN 5.65 Hz
POINT 40960
FREQU 39258.79 Hz
SCANS 60
ACQTM 1.0433 sec
PD 2.0000 sec
PW1 2.77 usec
IRNUC 1H
CTEMP 24.5 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 50

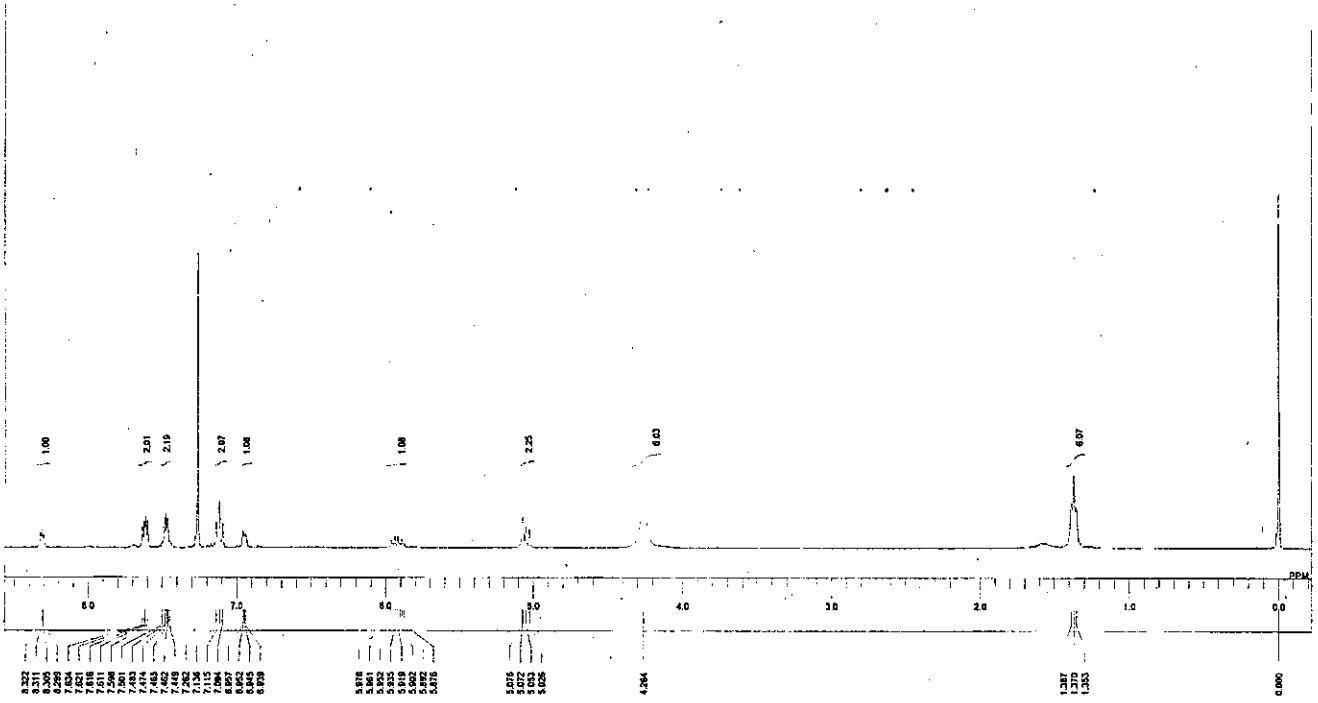


single pulse

C:\Users\Lenovo7\AppData\Local\Temp\GSHH 22 08 pure h_Proton_fl-1-1.jdf



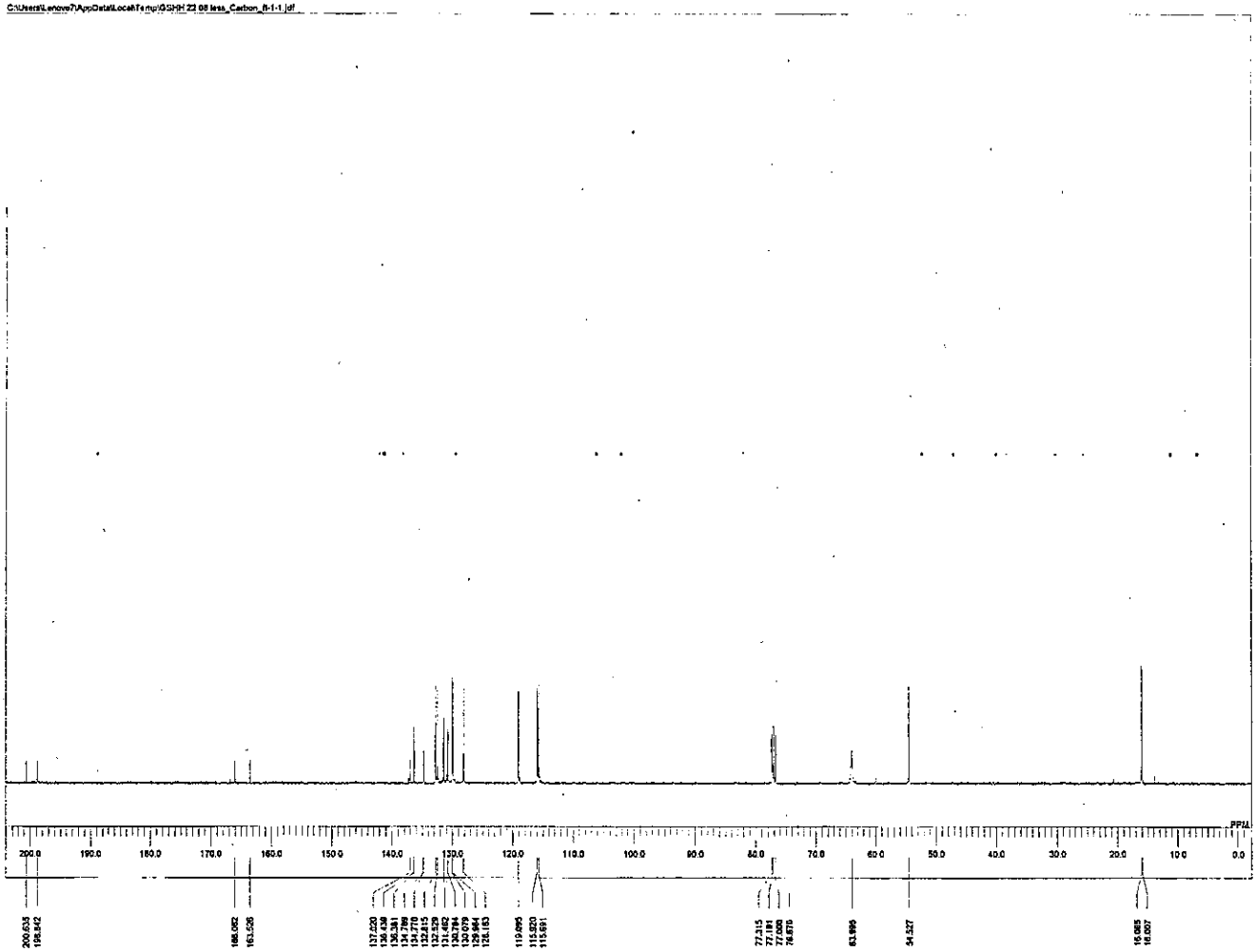
DFILE GSHH 22 08 pure h_Proto
COMNT single_pulse
DATIM 2016-01-05 09:44:18
OBNUC 1H
EXMOD proton.jxp
OBFRQ 399.78 MHz
OBSET 4.19 KHz
OBFIN 7.29 Hz
POINT 29480
FREQU 8378.75 Hz
SCANS 8
ACQTM 2.1837 sec
PD 5.0000 sec
PW1 5.01 usec
IRNUC 1H
CTEMP 21.7 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 50



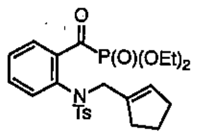
single pulse decoupled gated NOE

C:\Users\Lenovo7\AppData\Local\Temp\GSHH 22 08 less_Carbon_fl-1-1.jdf

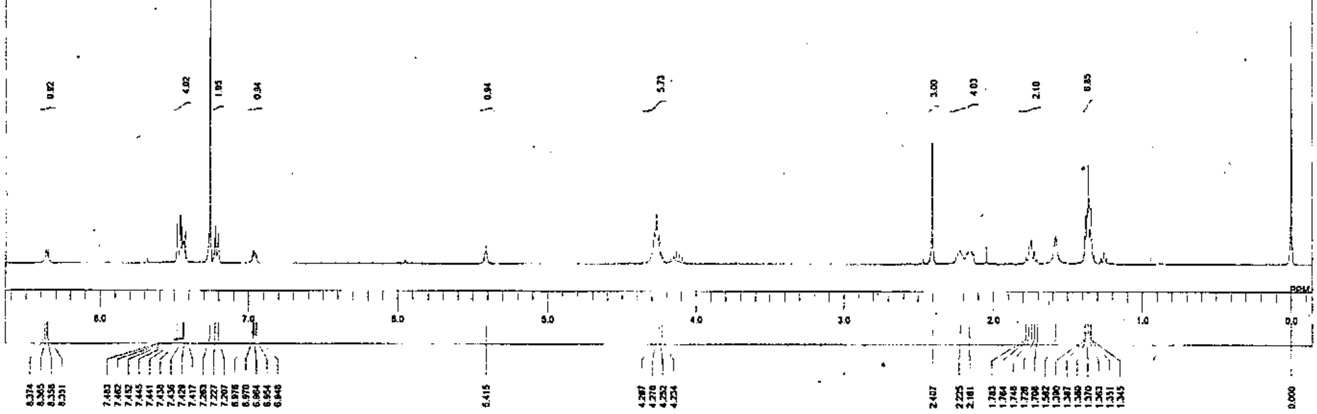
DFILE GSHH 22 08 less_Carbor
COMNT single pulse decoupled ga
DATIM 2015-12-22 14:35:47
OBNUC 13C
EXMOD carbon.jxp
OBFRQ 100.53 MHz
OBSET 5.35 KHz
OBFIN 5.86 Hz
POINT 40960
FREQU 39258.79 Hz
SCANS 60
ACQTM 1.0433 sec
PD 2.0000 sec
PW1 2.77 usec
IRNUC 1H
CTEMP 24.5 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 50



single_pulse
D:\...\GSHH 25 69 last_Proton_ft-1-1.als

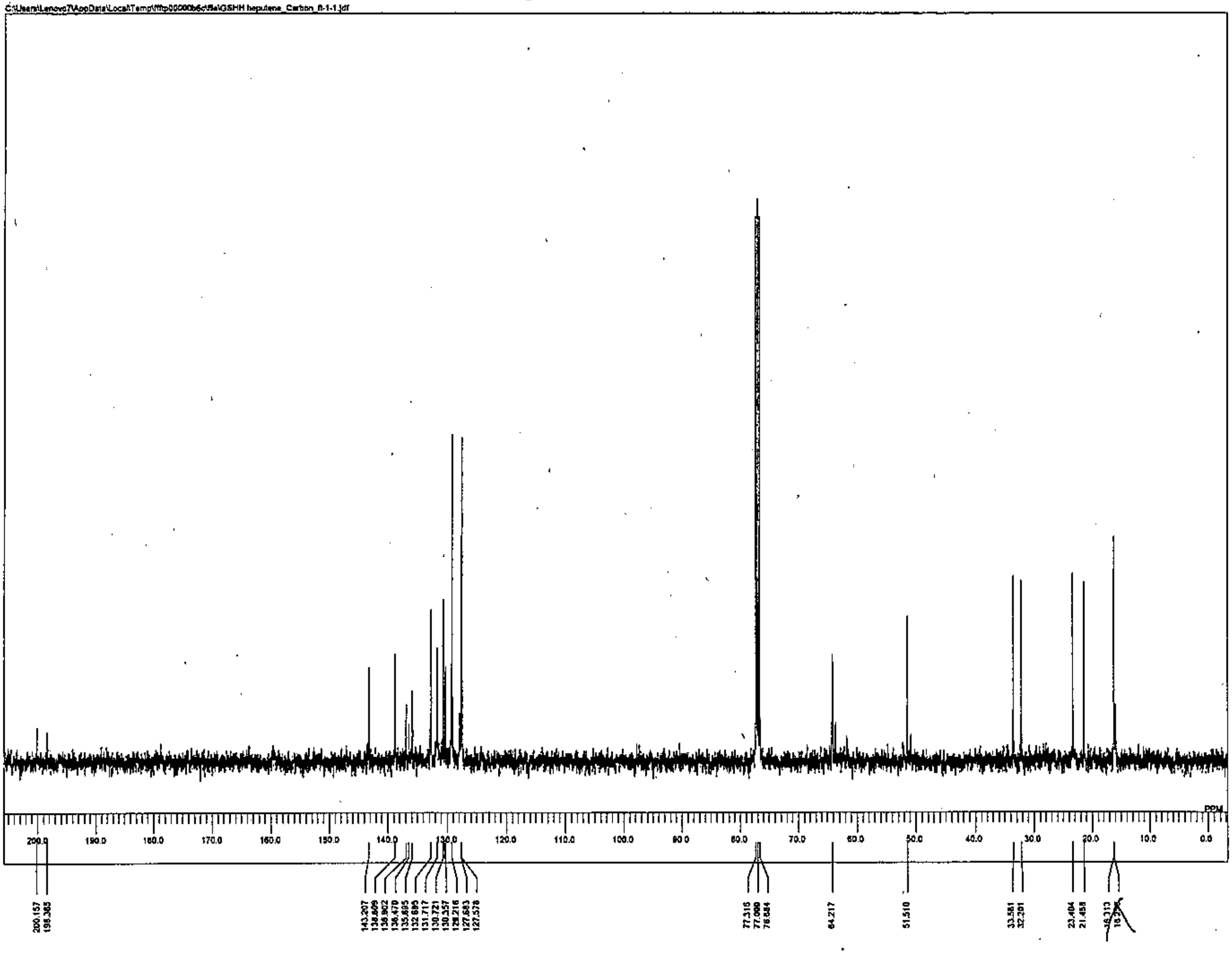


DFILE GSHH 25 69 last_Proton_I
COMNT single_pulse
DATIM 2016-10-08 14:25:02
OBNUC 1H
EXMOD proton.jxp
OBFRQ 399.78 MHz
OBSET 4.19 KHz
OBFIN 7.29 Hz
POINT 16384
FREQU 7494.00 Hz
SCANS 8
ACQTM 2.1863 sec
PD 5.0000 sec
PW1 2.72 usec
IRNUC 1H
CTEMP 23.0 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 68



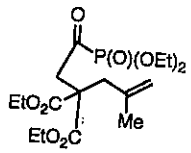
single pulse decoupled gated NOE
C:\Users\Lenovo7\AppData\Local\Temp\...\GSHH hepulene_Carbon_ft-1-1.jdf

DFILE GSHH hepulene_Carbon_ft-1
COMNT single pulse decoupled gated
DATIM 2016-02-03 14:51:37
OBNUC 13C
EXMOD carbon.jxp
OBFRQ 100.53 MHz
OBSET 5.35 KHz
OBFIN 5.68 Hz
POINT 40960
FREQU 39457.07 Hz
SCANS 60
ACQTM 1.0381 sec
PD 2.0000 sec
PW1 4.08 usec
IRNUC 1H
CTEMP 18.7 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 50



single_pulse

C:\Users\Lenovo7\AppData\Local\Temp\GSHH 23 03 last pure 4_Proton_fi-1-1.jdf

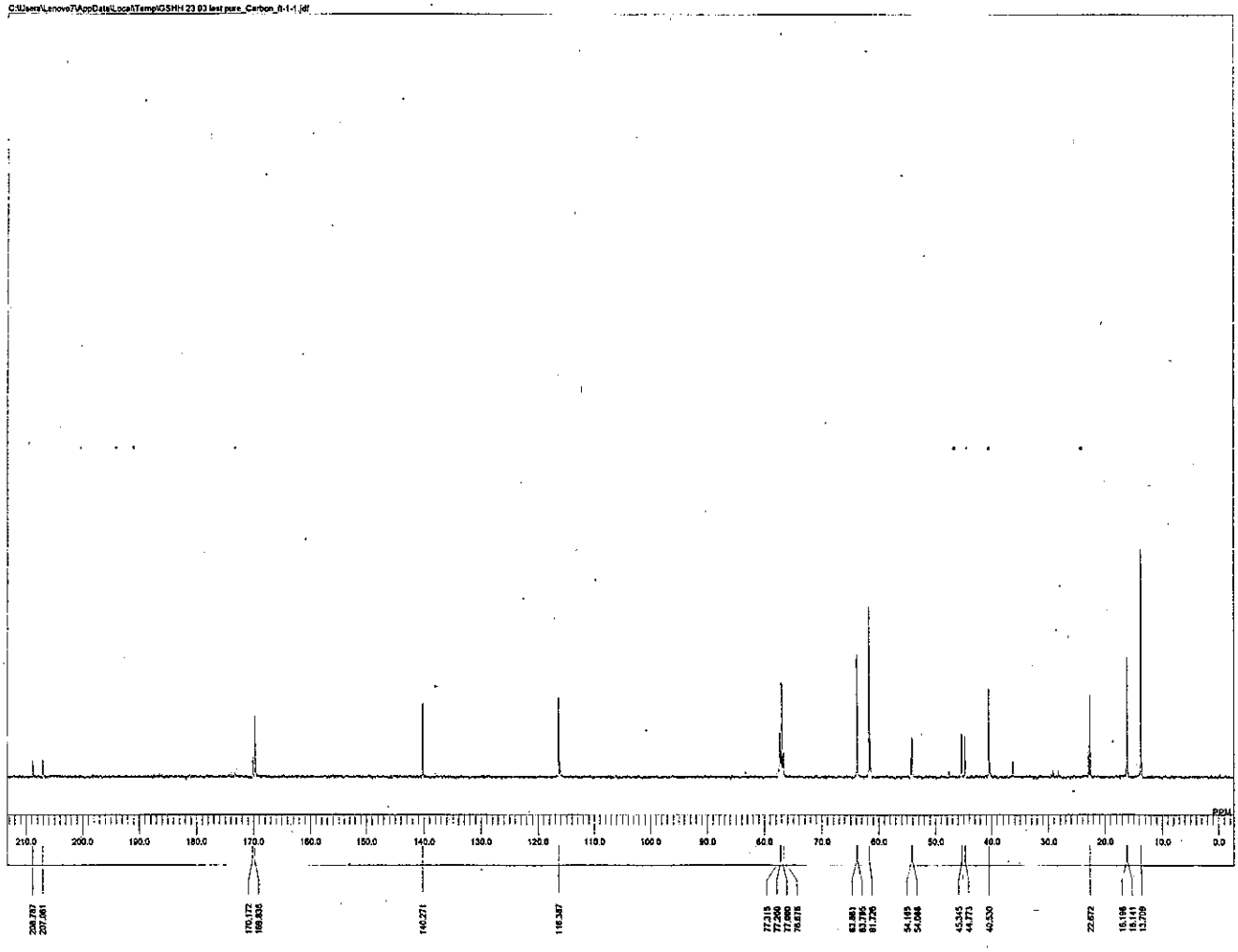


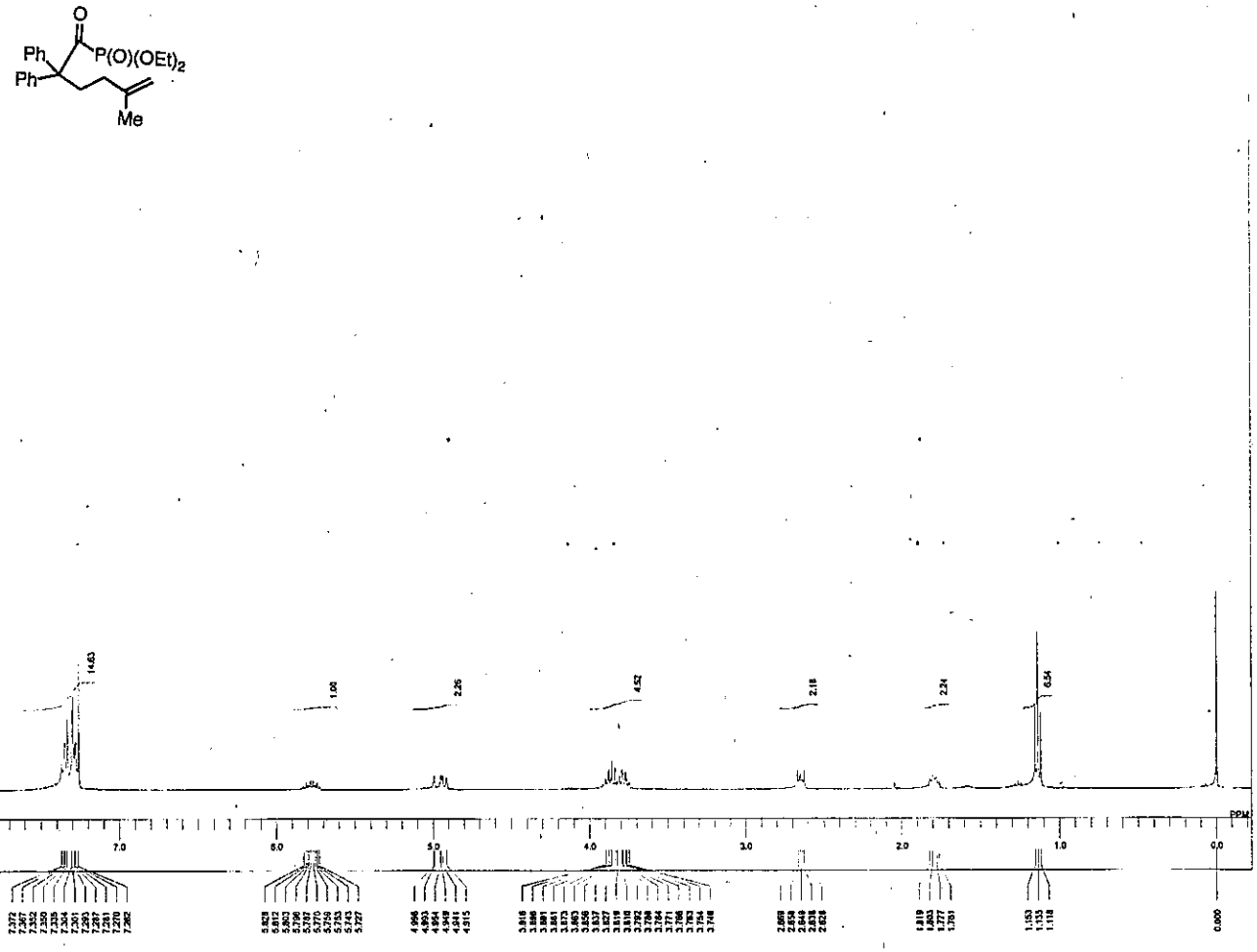

```
DFILE   GSHH 23 03 last pure 4_P
COMNT   single_pulse
DATIM   2016-02-05 17:40:32
OBNUC   1H
EXMOD   proton.jxp
OBFRQ     600.17 MHz
OBSET       5.30 KHz
OBFIN       5.47 Hz
POINT      20480
FREQU   14101.98 Hz
SCANS          8
ACQTM     1.4523 sec
PD        5.0000 sec
PW1       4.30 usec
IRNUC      1H
CTEMP      18.3 c
SLVNT      CDCL3
EXREF      0.00 ppm
SF         1.00 Hz
RGAIN        56
```

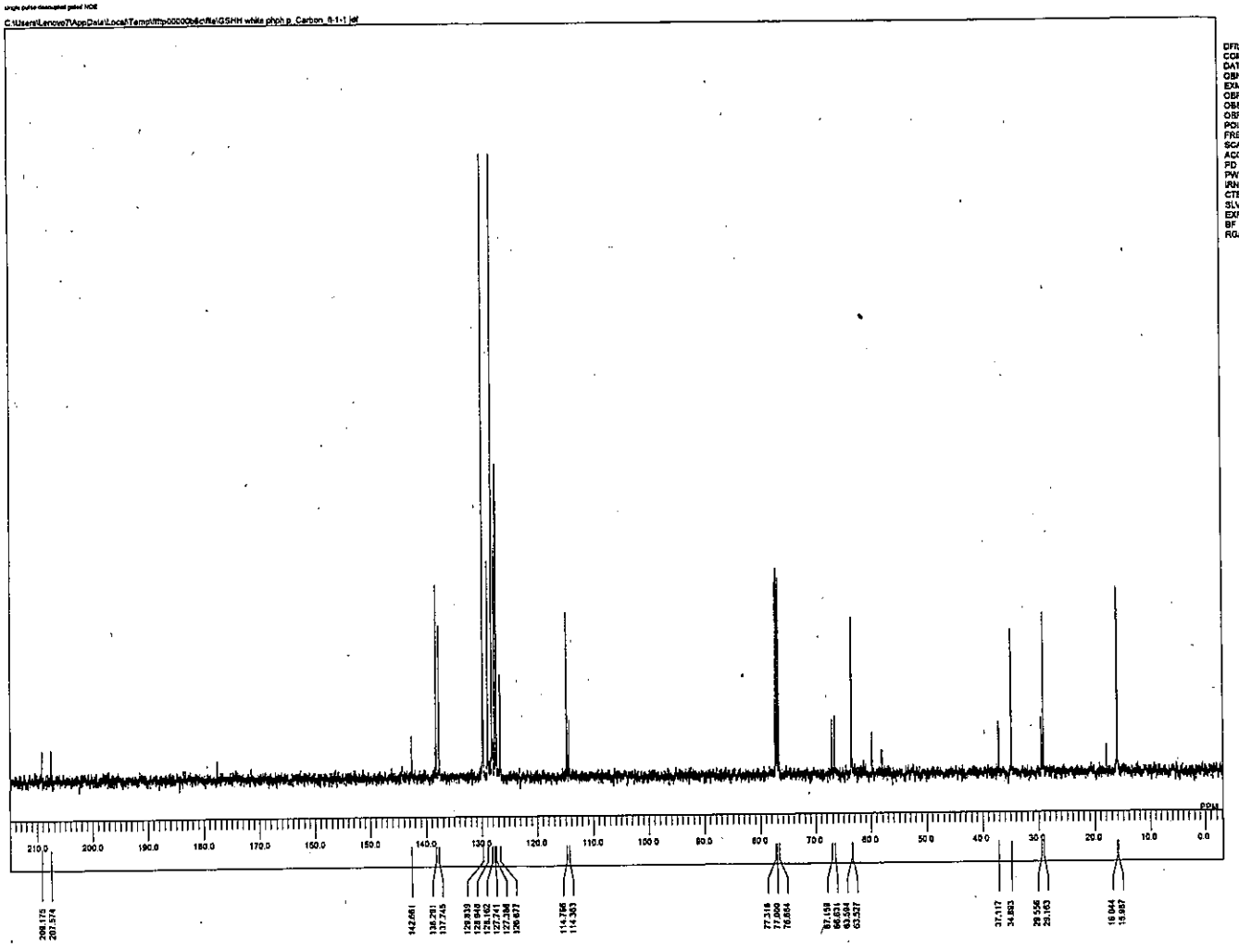
single pulse decoupled gated NOE

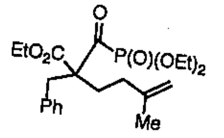
C:\Users\Lenovo7\AppData\Local\Temp\GSHH 23 03 last pure_Carbon_fi-1-1.jdf

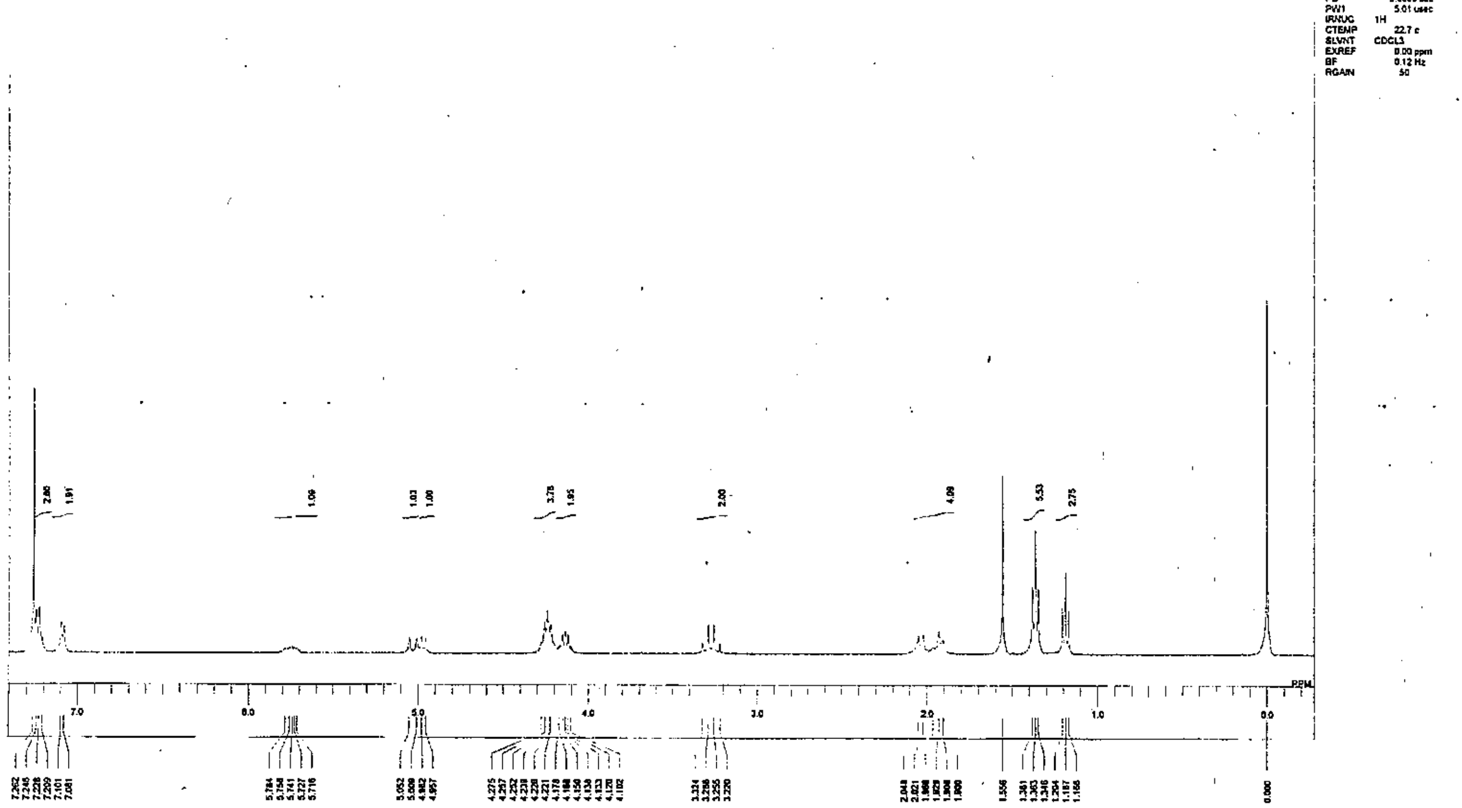
```
DFILE   GSHH 23 03 last pure_Ca
COMNT   single pulse decoupled gat
DATIM   2016-02-05 18:50:40
OBNUC   13C
EXMOD   carbon.jxp
OBFRQ     100.53 MHz
OBSET       6.39 KHz
OBFIN       5.86 Hz
POINT      40960
FREQU   39235.79 Hz
SCANS        100
ACQTM     1.0433 sec
PD        2.0000 sec
PW1       2.77 usec
IRNUC      1H
CTEMP      21.9 c
SLVNT      CDCL3
EXREF      77.00 ppm
SF         1.00 Hz
RGAIN        50
```

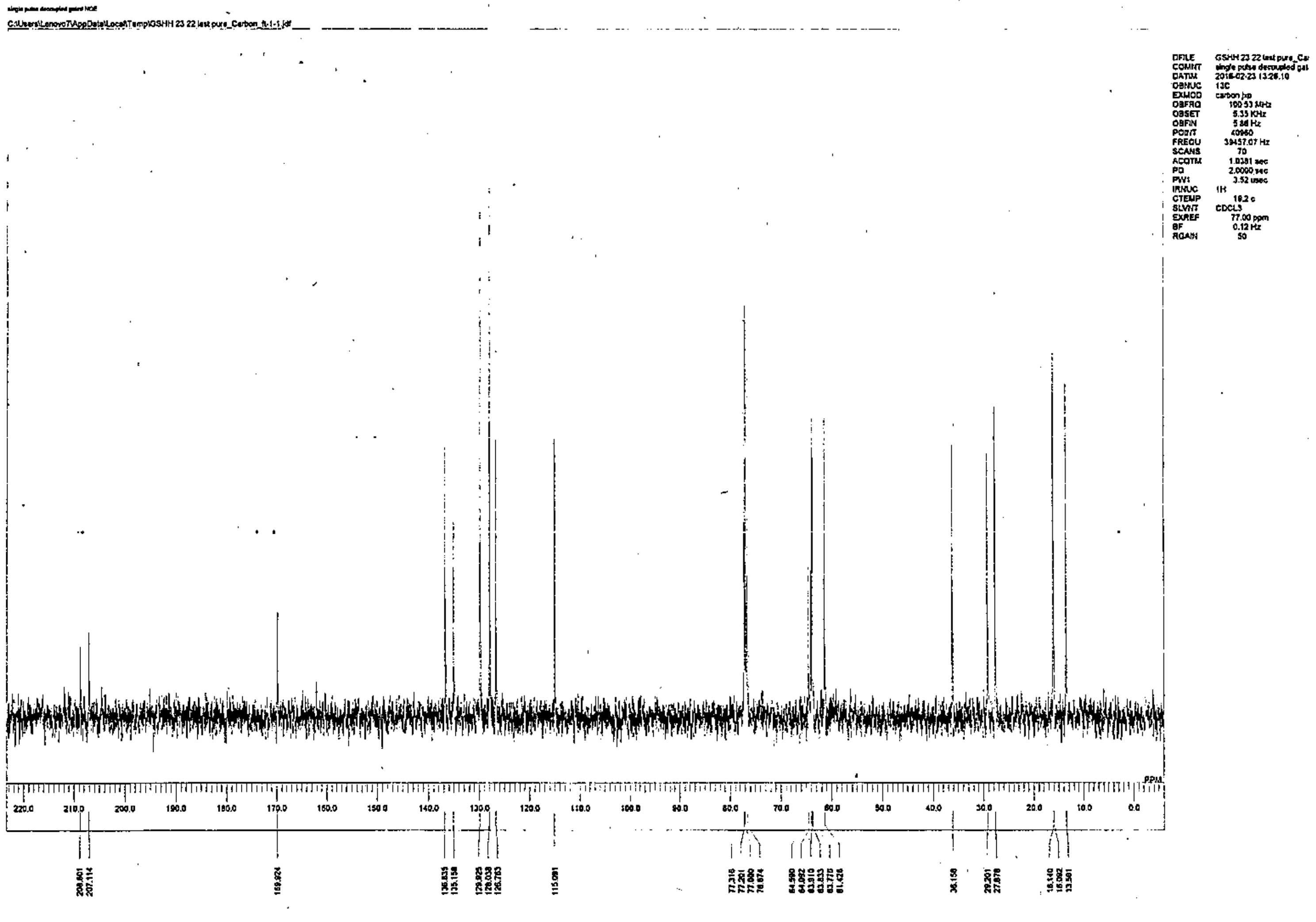



Ph
Ph
O
P(O)(OEt)2
Me
DFILE GSHH23 38 last_Proton_f
COUNT single_pulse
DATIM 2018-03-17 18:40:50
OBNUC 1H
EXMOD proton.jxp
OBFRQ 399.78 MHz
OBSET 4.19 KHz
OBFIN 7.29 Hz
POINT 20480
FREQU 9387.51 Hz
SCANS 3
ACQTM 2.1883 sec
PD 5.0000 sec
PW1 2.72 usec
IRNUC 1H
CTEMP 20.8 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 56
14.63
1.00
2.25
4.52
2.18
2.24
6.54
PPM

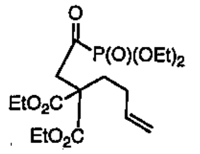

DFILE GSHH white phph p_Carbon_1
COUNT single pulse decoupled gated f
DATIM 2018-02-03 14:57:53
OBNUC 13C
EXMOD carbon.jxp
OBFRQ 100.53 MHz
OBSET 9.35 KHz
OBFIN 5.86 Hz
POINT 40960
FREQU 39457.07 Hz
SCANS 40
ACQTM 1.0381 sec
PD 2.0000 sec
PW1 4.05 usec
IRNUC 1H
CTEMP 18.6 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 50
PPM

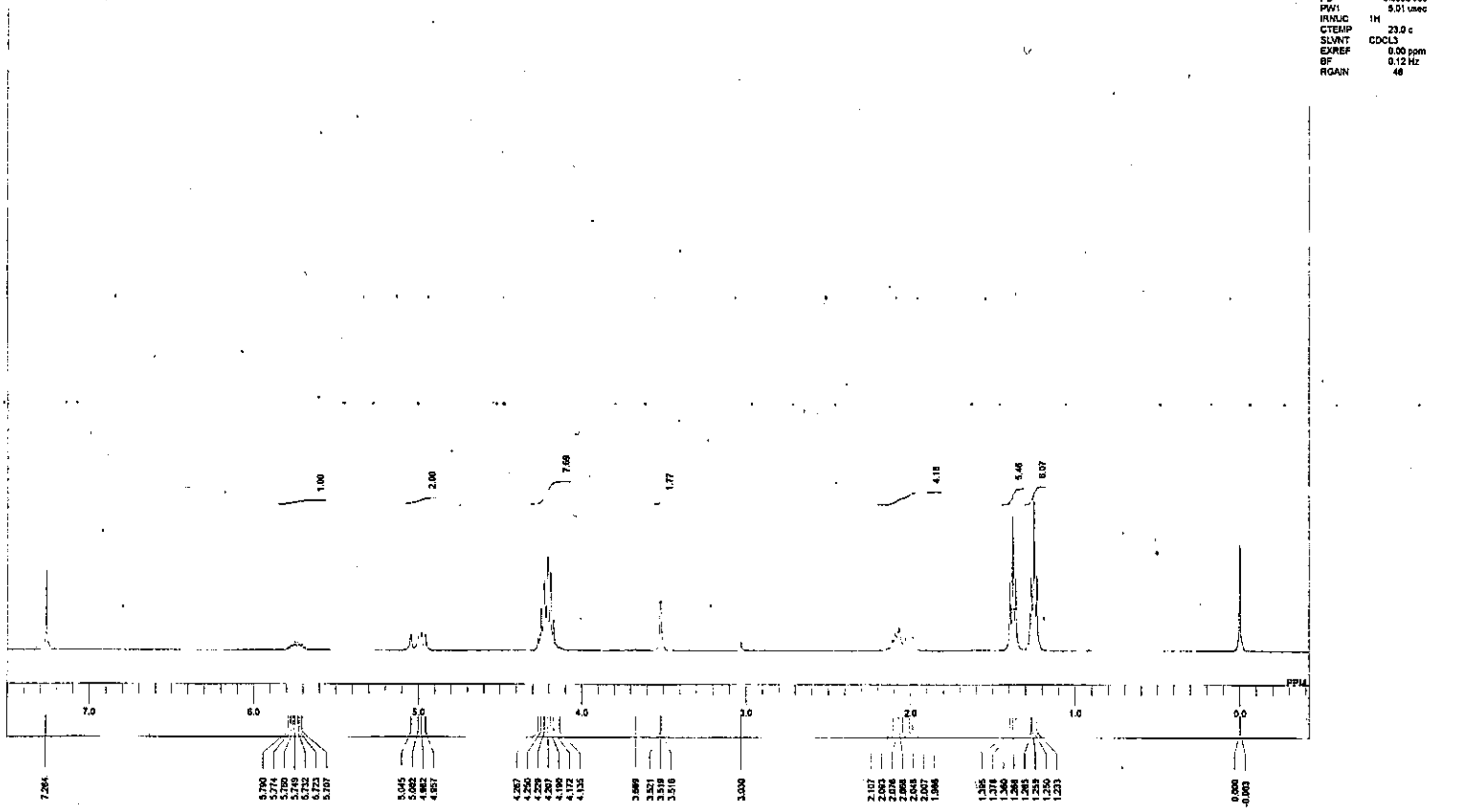

EtO2C
O
P(O)(OEt)2
Ph
Me


single_pulse
DFILE GSHH 23 22 last 3_Proton
COMNT single_pulse
DATIM 2016-02-23 13:57:38
OBNUC 1H
EXMOD proton.jxp
OBFRQ 399.78 MHz
OBSET 4.19 KHz
OBFIN 7.29 Hz
POINT 16384
FREQU 7503.00 Hz
SCANS 8
ACQTM 2.1837 sec
PD 5.0000 sec
PW1 5.01 usec
IRNUC 1H
CTEMP 22.7 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 50
PPM

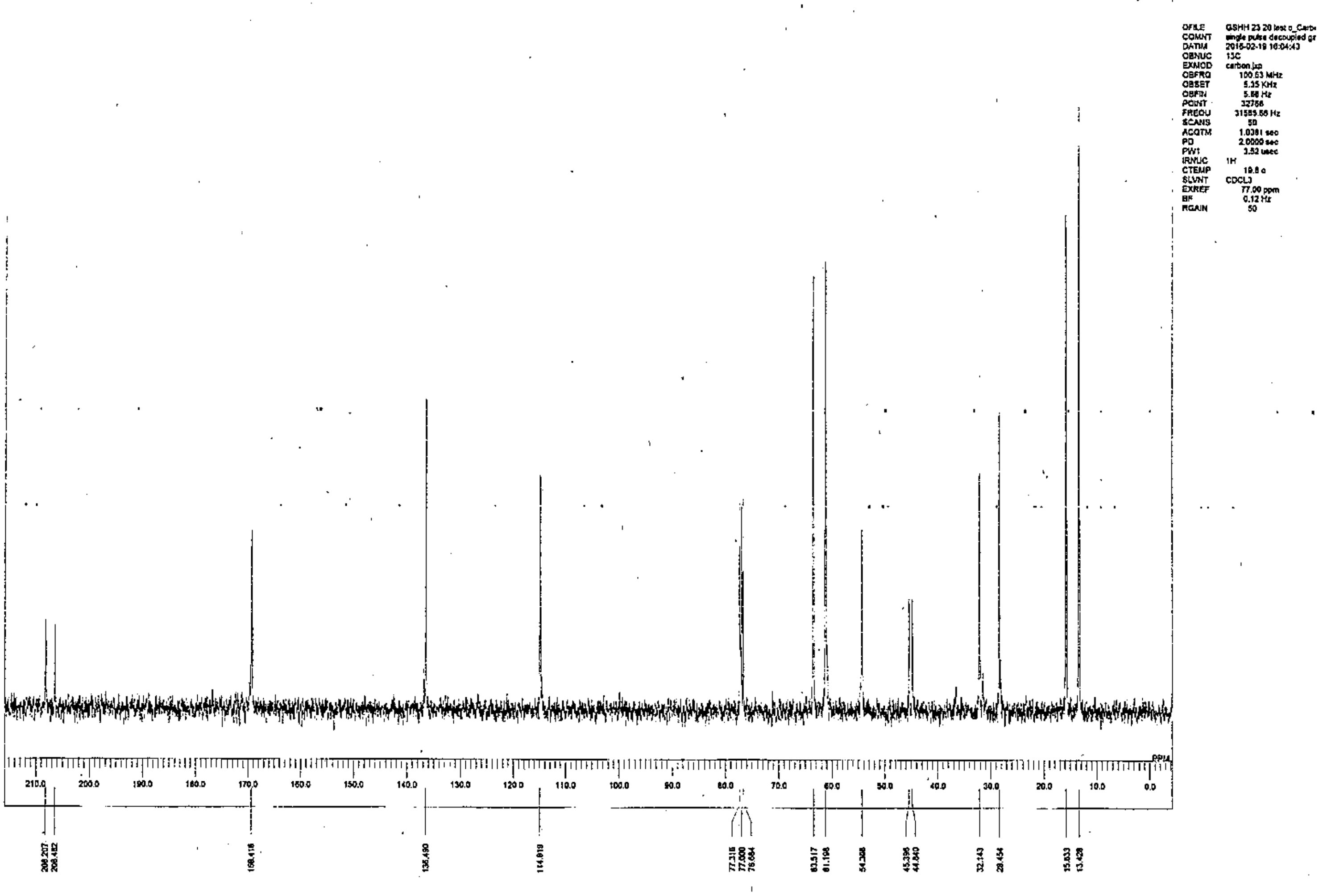

single pulse decoupled gated NOE
DFILE GSHH 23 22 last pure_Car
COMNT single pulse decoupled gat
DATIM 2016-02-23 13:26:10
OBNUC 13C
EXMOD carbon.jxp
OBFRQ 100.53 MHz
OBSET 5.35 KHz
OBFIN 5.86 Hz
POINT 40960
FREQU 39457.07 Hz
SCANS 70
ACQTM 1.0381 sec
PD 2.0000 sec
PW1 3.52 usec
IRNUC 1H
CTEMP 19.2 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 50
PPM

single_pulse
D:\CEA\/1\/H/\/_\hori23\01~20\GSHH 23 20 last impure_Proton_ft-1-1.als



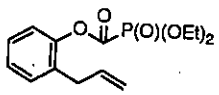

DFILE GSHH 23 20 last impure_I
COMNT single_pulse
DATIM 2016-02-19 13:14:46
OBNUC 1H
EXMOD proton.jxp
OBFRQ 399.78 MHz
OBSET 4.19 KHz
OBFIN 7.29 Hz
POINT 16384
FREQU 7503.00 Hz
SCANS 4
ACQTM 2.1837 sec
PD 5.0000 sec
PW1 5.01 usec
IRNUC 1H
CTEMP 23.0 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 46
1.00
2.00
7.69
1.77
4.18
5.46
6.07
PPM
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
7.264
5.790 5.774 5.760 5.749 5.732 5.723 5.707
5.045 5.002 4.982 4.957
4.267 4.250 4.229 4.207 4.190 4.172 4.135
3.569 3.521 3.519 3.516
3.000
2.107 2.093 2.076 2.068 2.048 2.007 1.996
1.395 1.378 1.360 1.288 1.265 1.259 1.250 1.233
0.000 -0.003

single pulse decoupled gated NOE
D:\CEA\/1\/H/\/_\hori23\01~20\GSHH 23 20 last c_Carbon_ft-1-1.als

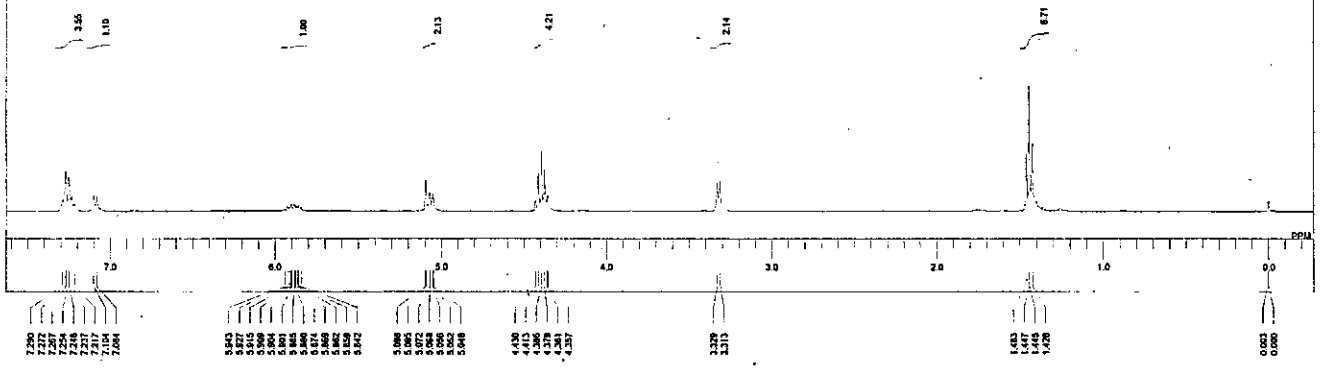

DFILE GSHH 23 20 last c_Carb
COMNT single pulse decoupled ga
DATIM 2016-02-19 16:04:43
OBNUC 13C
EXMOD carbon.jxp
OBFRQ 100.53 MHz
OBSET 5.35 KHz
OBFIN 5.56 Hz
POINT 32768
FREQU 31589.56 Hz
SCANS 50
ACQTM 1.0381 sec
PD 2.0000 sec
PW1 3.52 usec
IRNUC 1H
CTEMP 19.8 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 50
PPM
210.0 200.0 190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0 0.0
208.207
206.482
169.418
136.490
114.919
77.316
77.000
76.684
63.517
61.198
54.268
45.396
44.840
32.143
28.454
15.833
13.429

single_pulse

C:\Users\Lenovo7\AppData\Local\Temp\GSHH 23 44 pure_Proton_fr-1-1.jdf



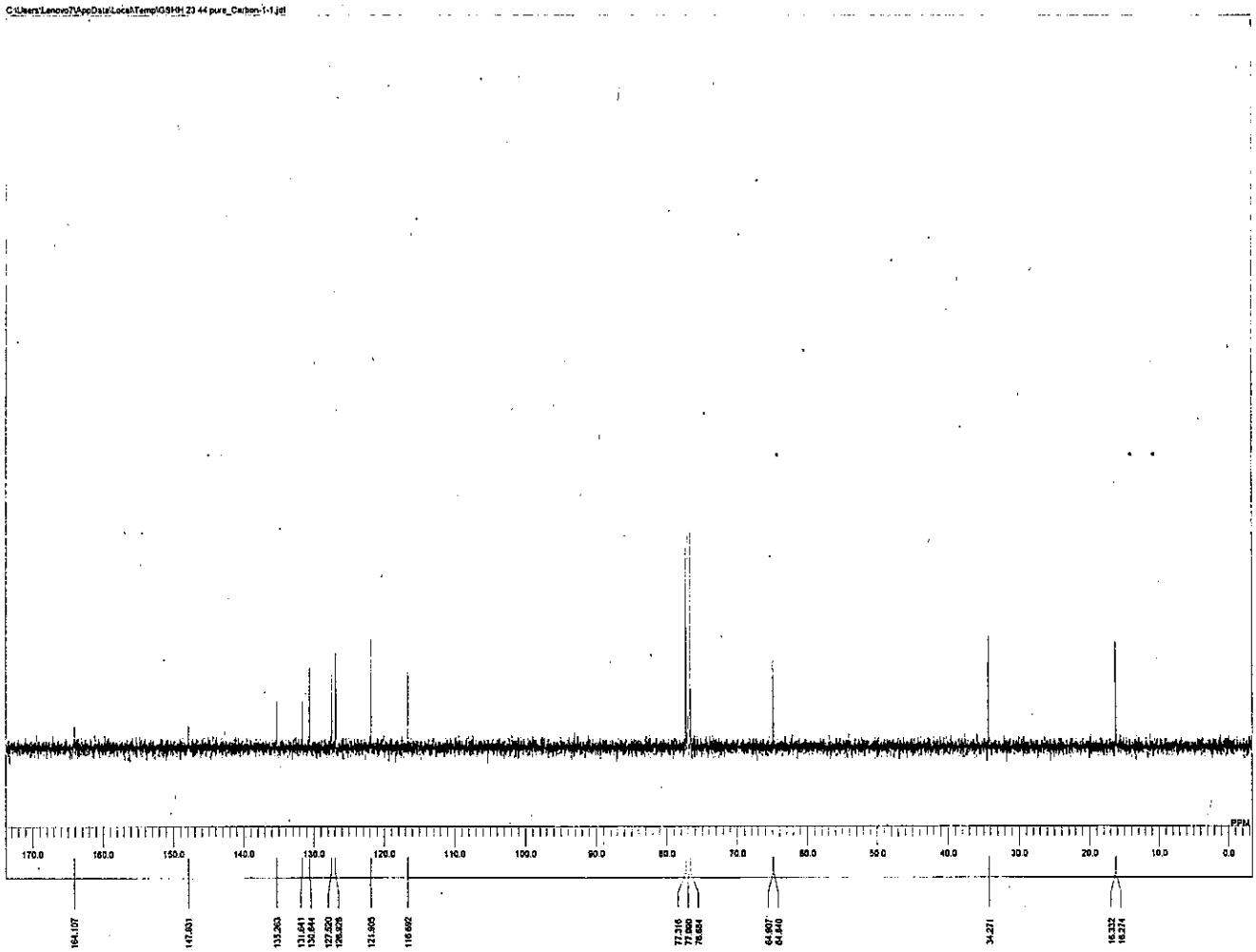

```
DFILE   GSHH 23 44 pure_Proton
COMNT   single_pulse
DATIM   2016-03-05 22:21:27
OBNUC   1H
EXMOD   proton.jxp
OBFRQ     399.78 MHz
OBSET       4.19 KHz
OBFIN       7.29 Hz
POINT      20480
FREQU    9387.51 Hz
SCANS          4
ACQTM      2.1863 sec
PD         5.0000 sec
PW1        2.72 usec
IRNUC      1H
CTEMP     29.0 c
SLVNT     CDCL3
EXREF      0.00 ppm
BF         0.12 Hz
RGAIN        48
```



single pulse decoupled gated NOE

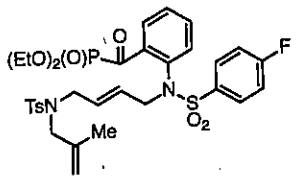
C:\Users\Lenovo7\AppData\Local\Temp\GSHH 23 44 pure_Carbon-1-1.jdf

```
DFILE   GSHH 23 44 pure_Carbon
COMNT   single pulse decoupled ga
DATIM   2016-03-05 22:22:18
OBNUC   13C
EXMOD   carbon.jxp
OBFRQ     100.53 MHz
OBSET       5.35 KHz
OBFIN       5.86 Hz
POINT      32767
FREQU    31565.68 Hz
SCANS         50
ACQTM      1.0381 sec
PD         2.0000 sec
PW1        3.52 usec
IRNUC      1H
CTEMP     29.0 c
SLVNT     CDCL3
EXREF      77.00 ppm
BF         0.12 Hz
RGAIN        50
```

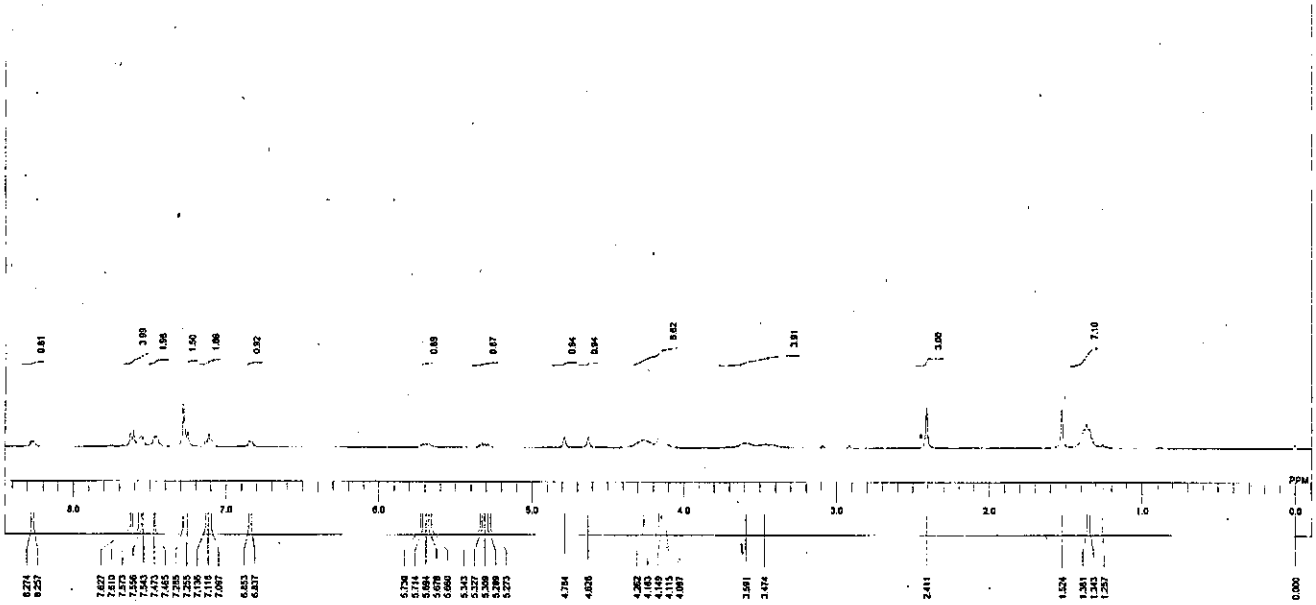


single_pulse

C:\Users\Lenovo7\AppData\Local\Temp\ffp00001e60\file\GSHH 24 51 last pure_Proton_fi-1-1.jdf



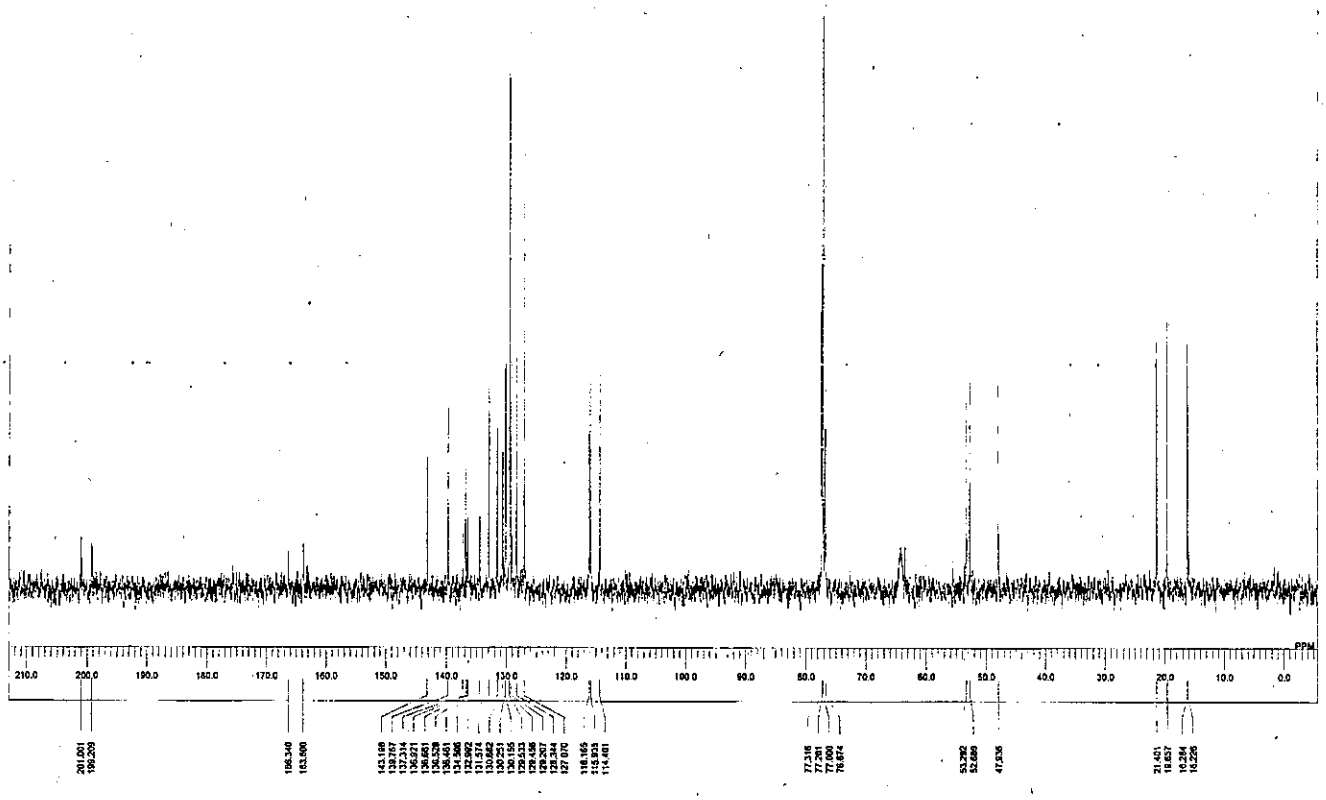

```
DFILE   GSHH 24 51 last pure_Pro
COMNT   single_pulse
DATIM   2016-06-02 21:16:07
OBNUC   1H
EXMOD   proton.jxp
OBFRQ     399.78 MHz
OBSET       4.19 KHz
OBFIN       7.29 Hz
POINT      20480
FREQU    8387.51 Hz
SCANS         8
ACQTM     2.1863 sec
PD        3.0000 sec
PW1       2.72 usec
IRNUC     1H
CTEMP     23.2 c
SLVNT     CDCL3
EXREF     0.00 ppm
BF        0.12 Hz
RGAIN       36
```

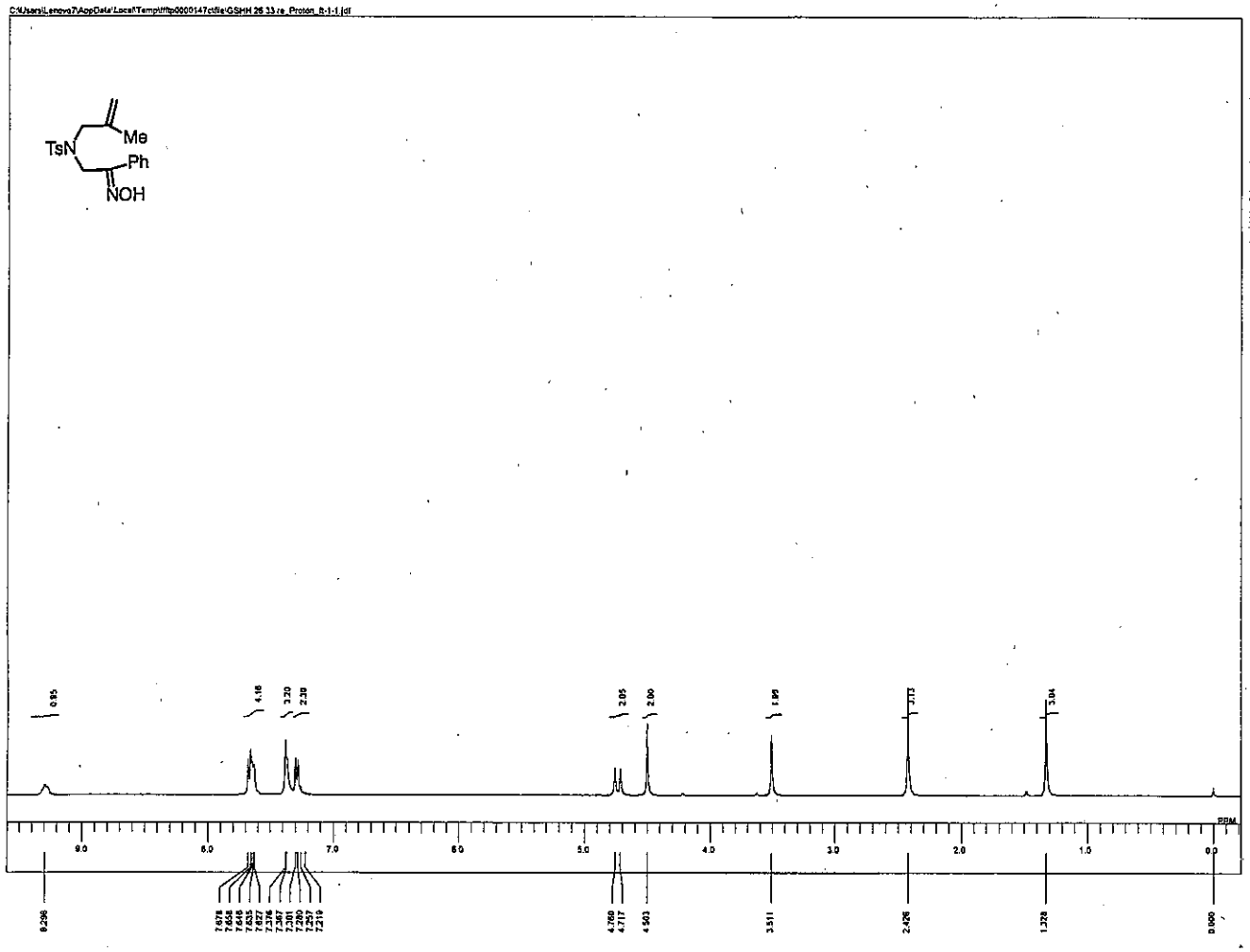


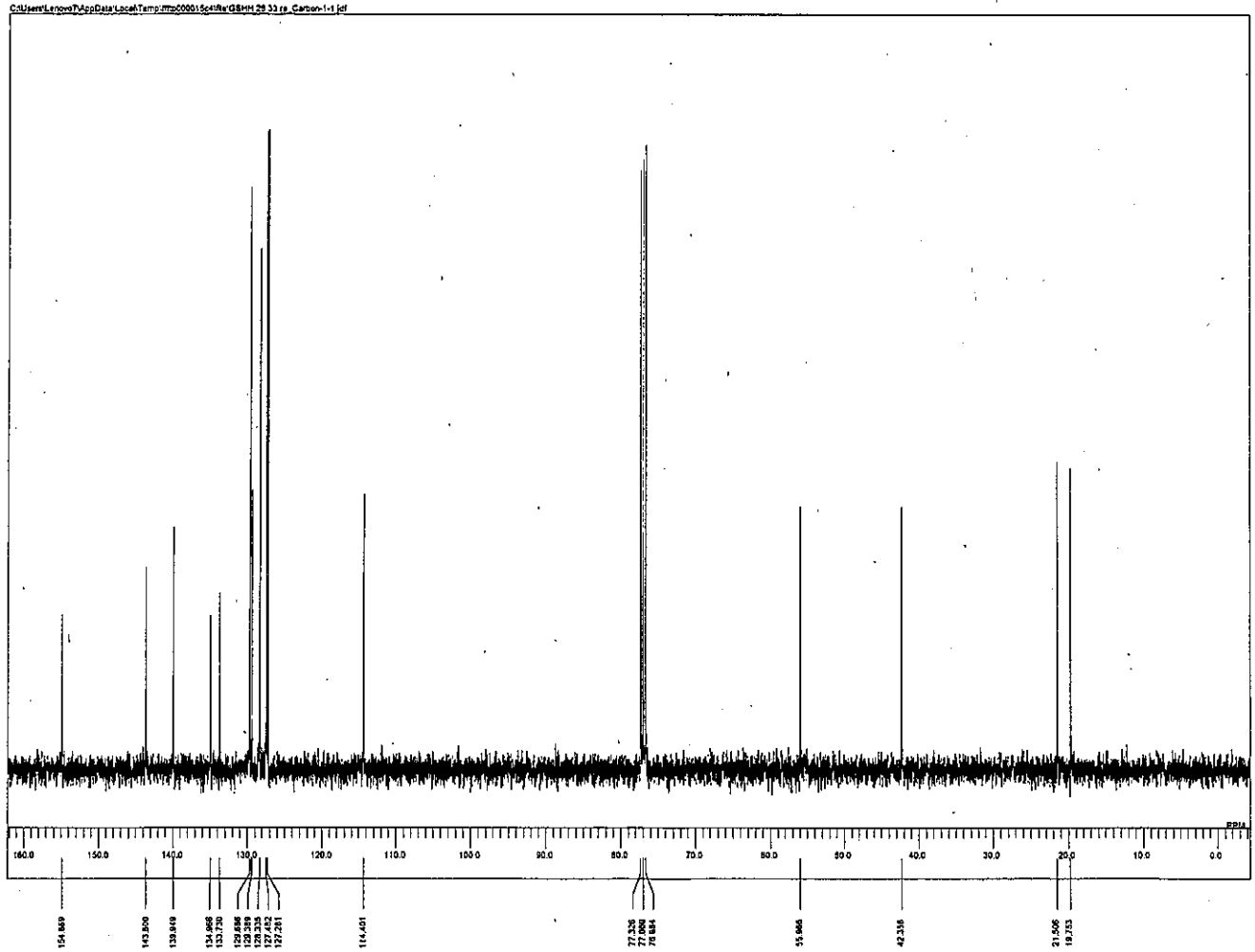
single pulse decoupled gated NOE

C:\Users\Lenovo7\AppData\Local\Temp\ffp00001e60\file\GSHH 24 51 last pure_Carbon_fi-1-1.jdf

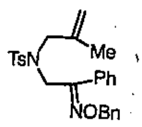
```
DFILE   GSHH 24 51 last pure_Ca
COMNT   single pulse decoupled gat
DATIM   2016-06-02 21:16:27
OBNUC   13C
EXMOD   carbon.jxp
OBFRQ     100.53 MHz
OBSET       5.35 KHz
OBFIN       5.66 Hz
POINT      40960
FREQU    39457.07 Hz
SCANS        100
ACQTM     1.0381 sec
PD        2.0000 sec
PW1       3.32 usec
IRNUC     1H
CTEMP     23.3 c
SLVNT     CDCL3
EXREF     77.00 ppm
BF        0.12 Hz
RGAIN       50
```



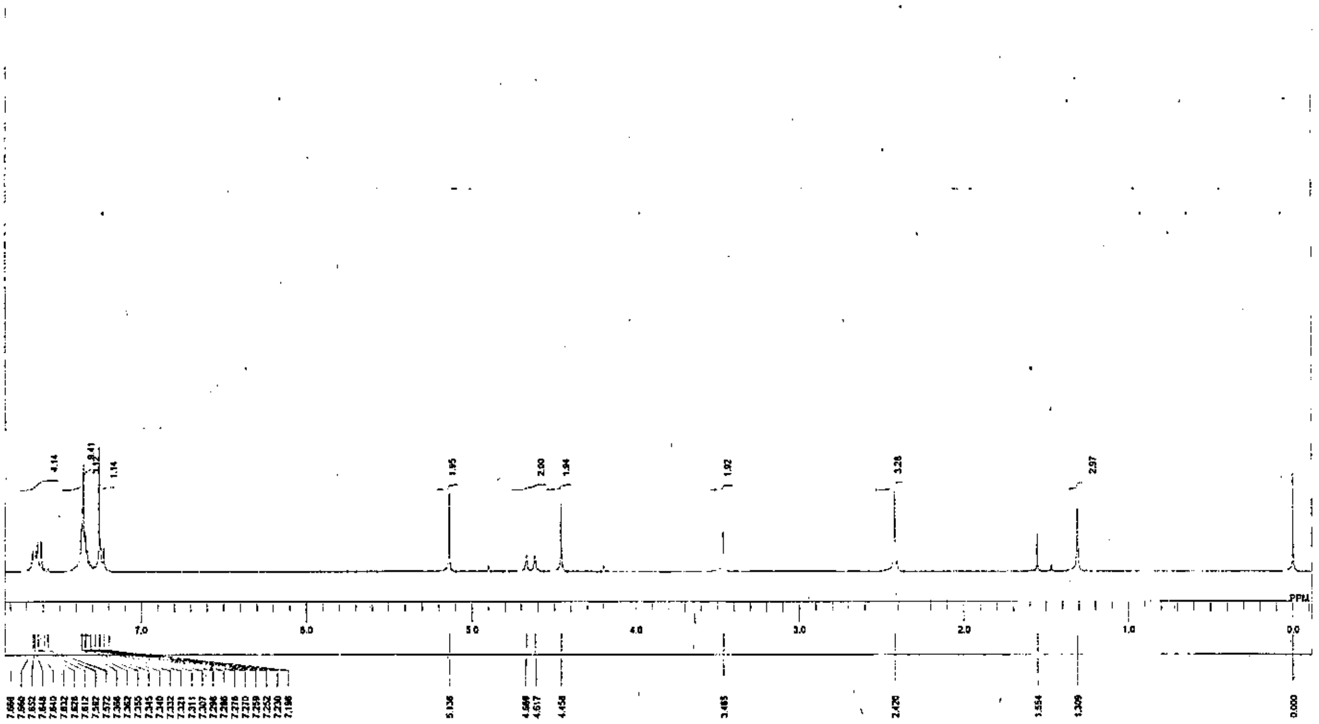

TsN
Me
Ph
NOH
0.95
4.16
3.20
2.39
2.05
2.00
1.89
3.13
3.04
PPM
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
8.296
7.678
7.658
7.646
7.635
7.627
7.376
7.367
7.355
7.280
7.257
7.219
4.769
4.717
4.503
3.511
2.426
1.328
0.000


PPM
160.0
150.0
140.0
130.0
120.0
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0.0
154.669
143.500
139.949
134.966
133.730
129.686
128.389
128.335
127.452
127.261
114.401
77.326
77.000
76.684
55.968
42.356
21.506
19.753

single_pulse
D:\KEAN\JH\..\bori\26\GSHH 26 14 re_Proton_ft-1-1.als

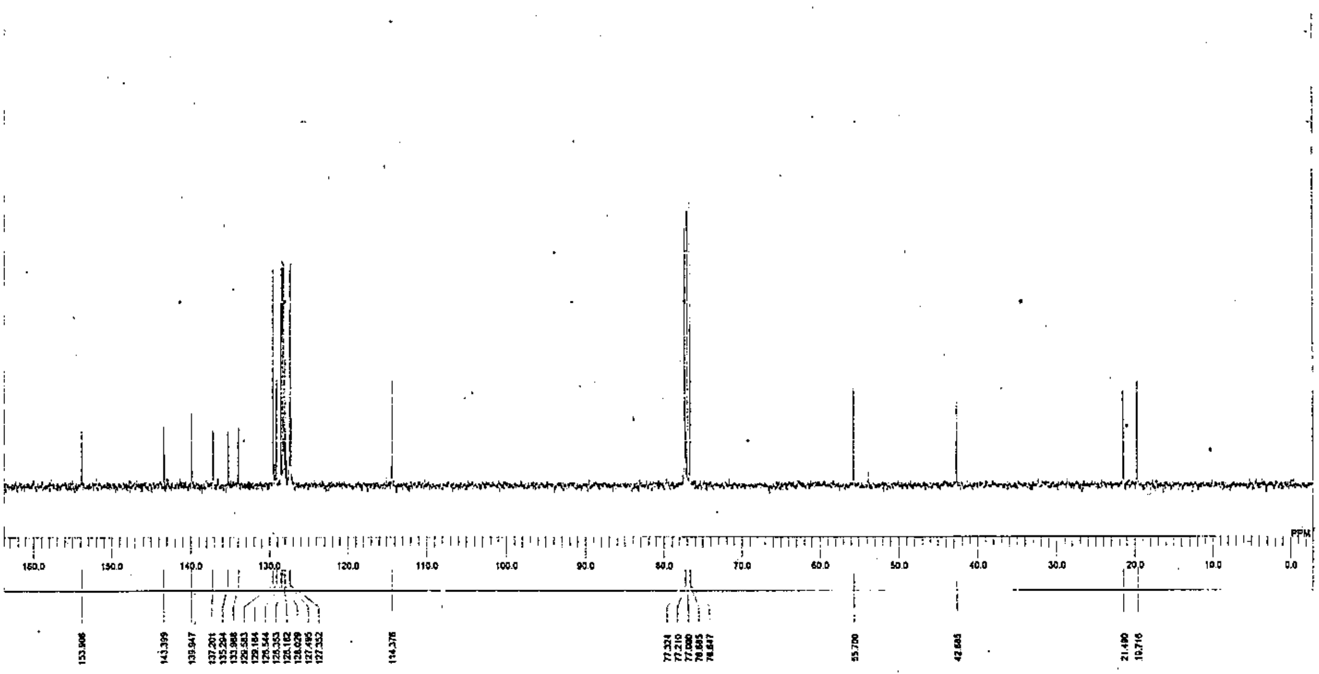


DFILE GSHH 26 14 re_Proton_ft-
COMNT single_pulse
DATIM 2016-11-15 21:18:59
OBNUC 1H
EXMOD proton.jxp
OBFRQ 399.78 MHz
OBSET 4.19 KHz
OBFIN 7.29 Hz
POINT 16384
FREQU 7503.00 Hz
SCANS 8
ACQTM 2.1837 sec
PD 5.0000 sec
PW1 5.00 usec
IRNUC 1H
CTEMP 20.5 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 44

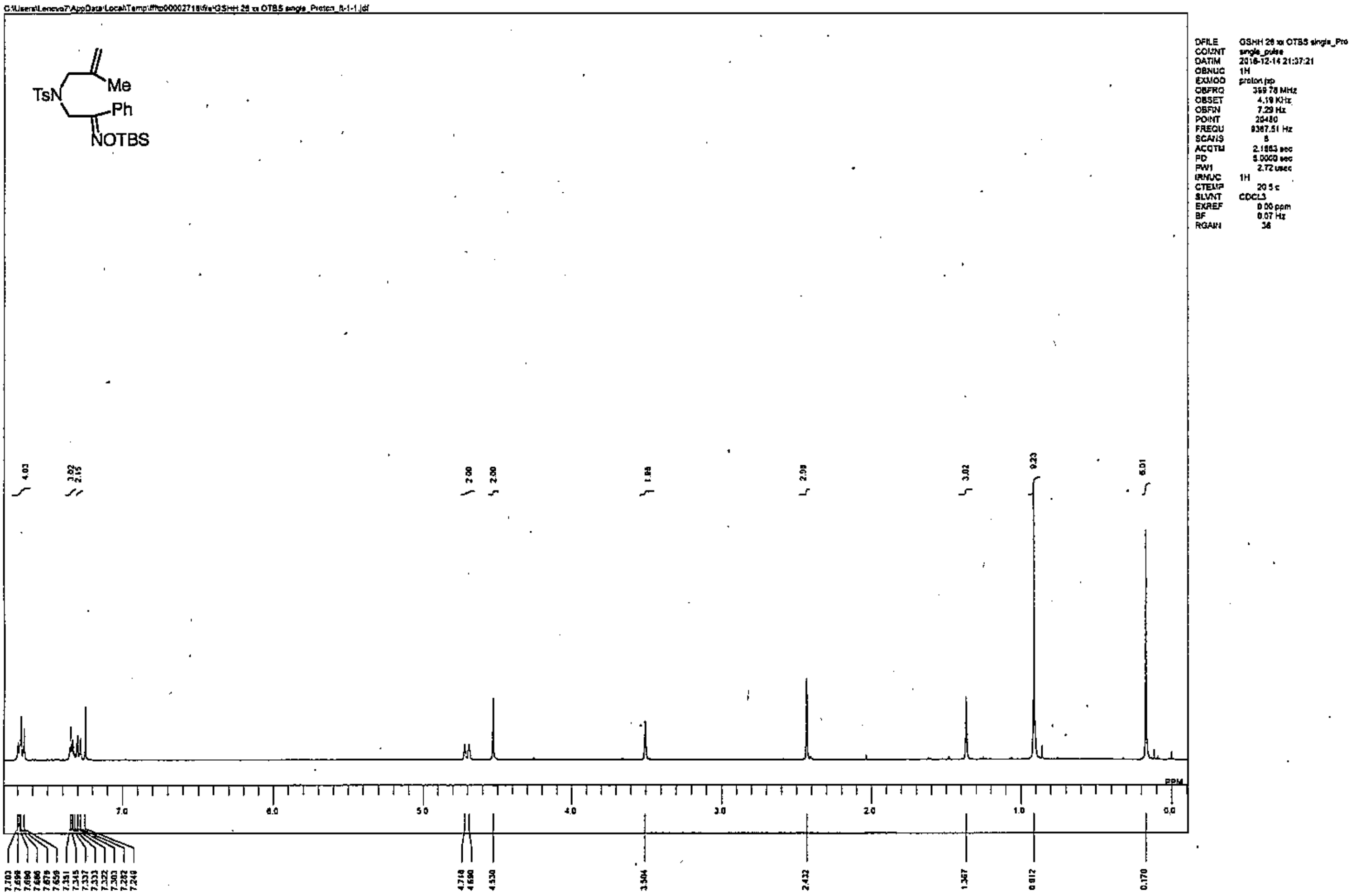


single pulse decoupled gated NOE
C:\Users\Lenovo7\AppData\Local\Temp\...\GSHH 26 14 re_Carbon_ft-1-1.jdf

DFILE GSHH 26 14 re_Carbon_ft
COMNT single pulse decoupled gat
DATIM 2016-11-15 21:21:23
OBNUC 13C
EXMOD carbon.jxp
OBFRQ 100.53 MHz
OBSET 5.35 KHz
OBFIN 5.86 Hz
POINT 40960
FREQU 39259.79 Hz
SCANS 50
ACQTM 1.0433 sec
PD 2.0000 sec
PW1 2.87 usec
IRNUC 1H
CTEMP 20.7 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 50

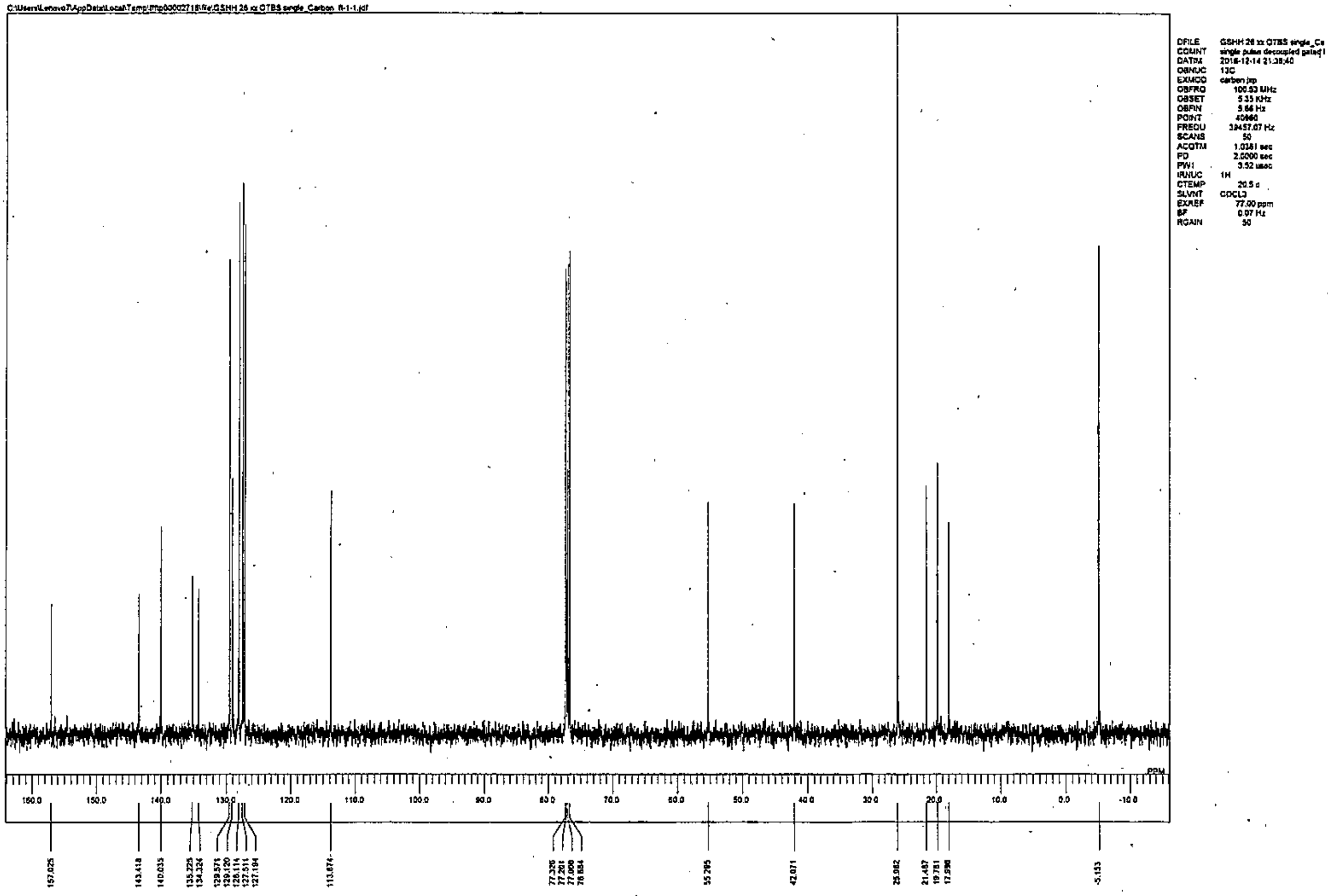


single_pulse
C:\Users\Lenovo7\AppData\Local\Temp\fftp00002718\fe\GSHH 26 xx OTBS single_Proton_ft-1-1.jdf



TsN
Me
Ph
NOTBS
DFILE GSHH 26 xx OTBS single_Pro
COMNT single_pulse
DATIM 2016-12-14 21:37:21
OBNUC 1H
EXMOD proton.jxp
OBFRQ 399.78 MHz
OBSET 4.19 KHz
OBFIN 7.29 Hz
POINT 26480
FREQU 8367.51 Hz
SCANS 8
ACQTM 2.1863 sec
PD 5.0000 sec
PW1 2.72 usec
IRNUC 1H
CTEMP 20.5 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.07 Hz
RGAIN 36
4.03
3.92
2.15
2.00
2.00
1.98
2.98
3.02
9.23
6.01
PPM
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
7.703
7.699
7.696
7.686
7.679
7.659
7.351
7.345
7.337
7.333
7.322
7.303
7.282
7.248
4.718
4.690
4.530
3.504
2.432
1.367
0.912
0.170

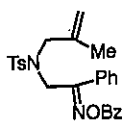
single pulse decoupled gated NOE
C:\Users\Lenovo7\AppData\Local\Temp\fftp00002718\fe\GSHH 26 xx OTBS single_Carbon_ft-1-1.jdf



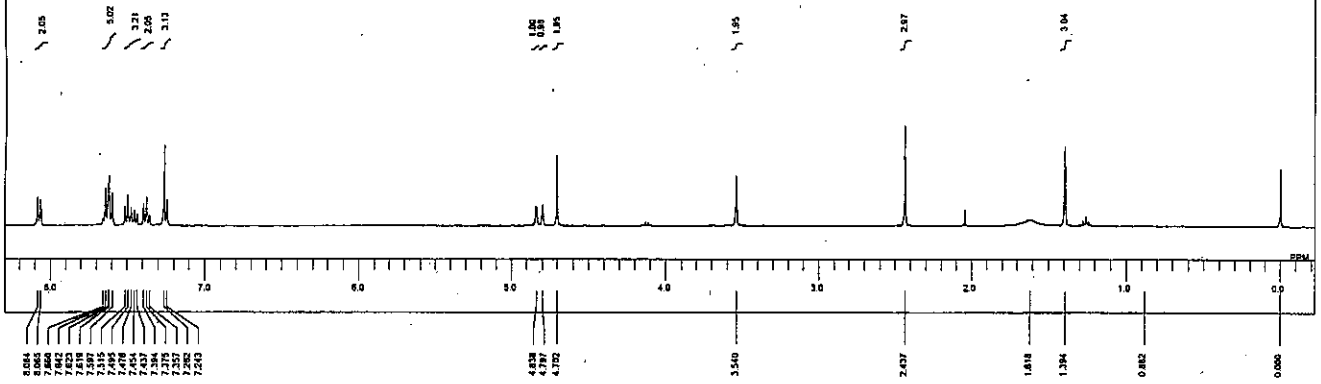
DFILE GSHH 26 xx OTBS single_Ca
COMNT single pulse decoupled gated I
DATIM 2016-12-14 21:38:40
OBNUC 13C
EXMOD carbon.jxp
OBFRQ 100.53 MHz
OBSET 5.35 KHz
OBFIN 5.64 Hz
POINT 40960
FREQU 39457.07 Hz
SCANS 50
ACQTM 1.0381 sec
PD 2.0000 sec
PW1 3.52 usec
IRNUC 1H
CTEMP 20.5 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.07 Hz
RGAIN 50
PPM
160.0
150.0
140.0
130.0
120.0
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0.0
-10.0
157.025
143.418
140.035
135.225
134.324
129.571
129.120
128.114
127.511
127.194
113.874
77.320
77.201
77.000
76.684
55.295
42.071
25.982
21.487
19.751
17.598
-5.153

single_pulse

E:\Personal Folder\OB, OC\hori25\GSHH 25 52 OBz_Proton_fi-1-1.als



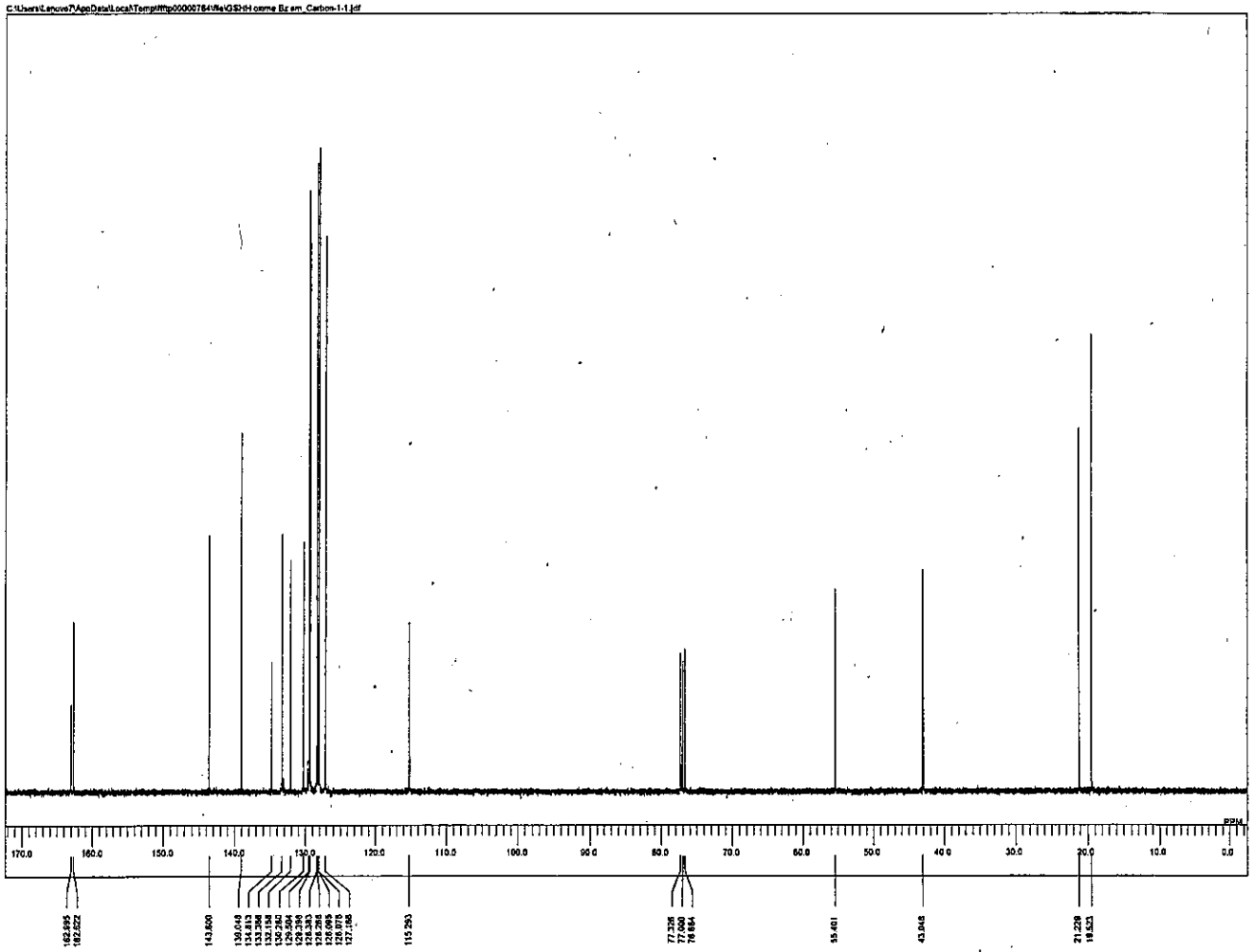
DFILE GSHH 25 52 OBz_Proton_fi-
COMNT single_pulse
DATIM 2018-06-29 22:08:08
OBNUC 1H
EXMOD proton.jxp
OBFRQ 399.78 MHz
OBSET 4.19 KHz
OBFIN 7.29 Hz
POINT 16384
FREQU 7494.60 Hz
SCANS 8
ACQTM 2.1863 sec
PD 5.0000 sec
PW1 2.72 usec
IRNUC 1H
CTEMP 22.4 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 1.00 Hz
RGAIN 56

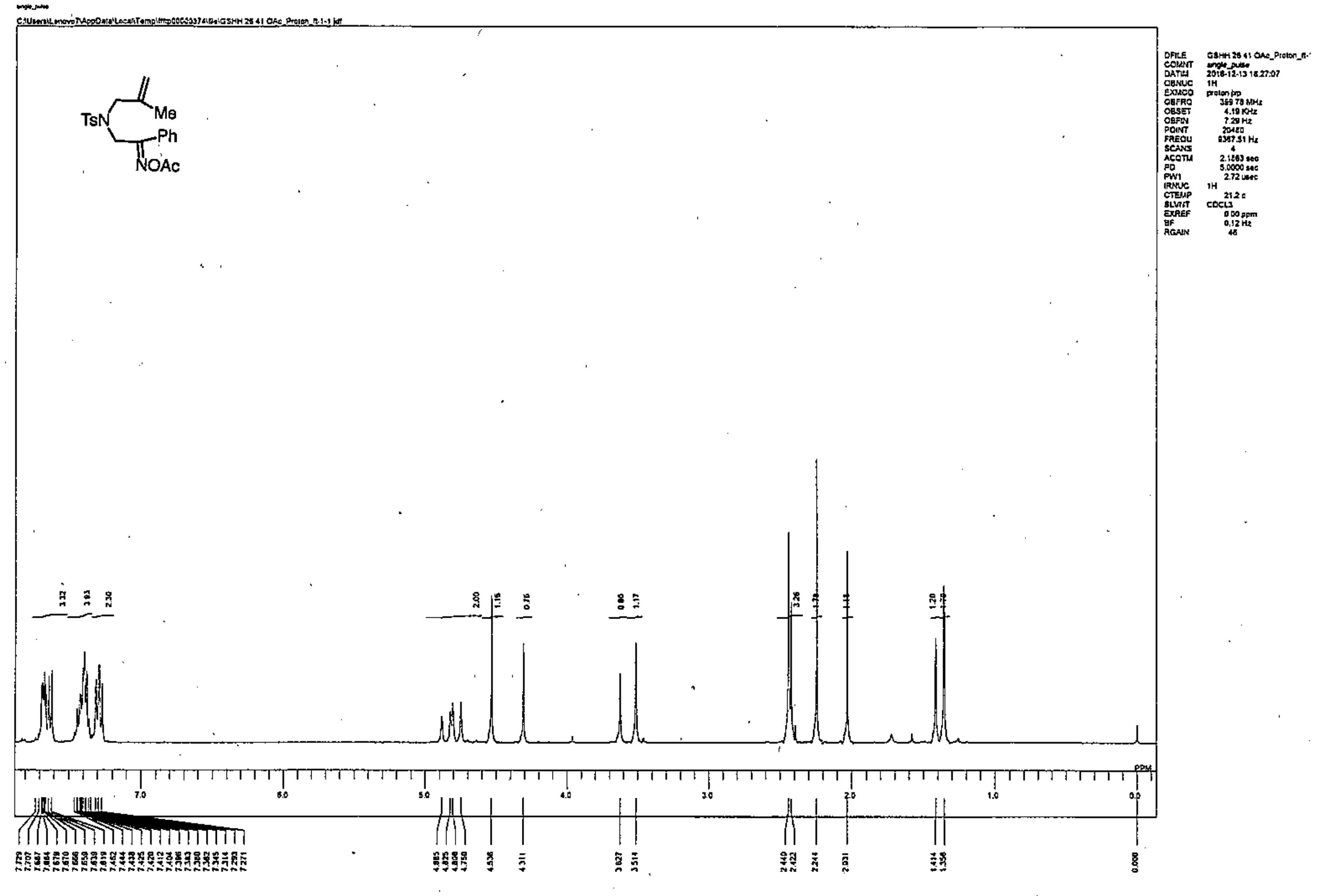


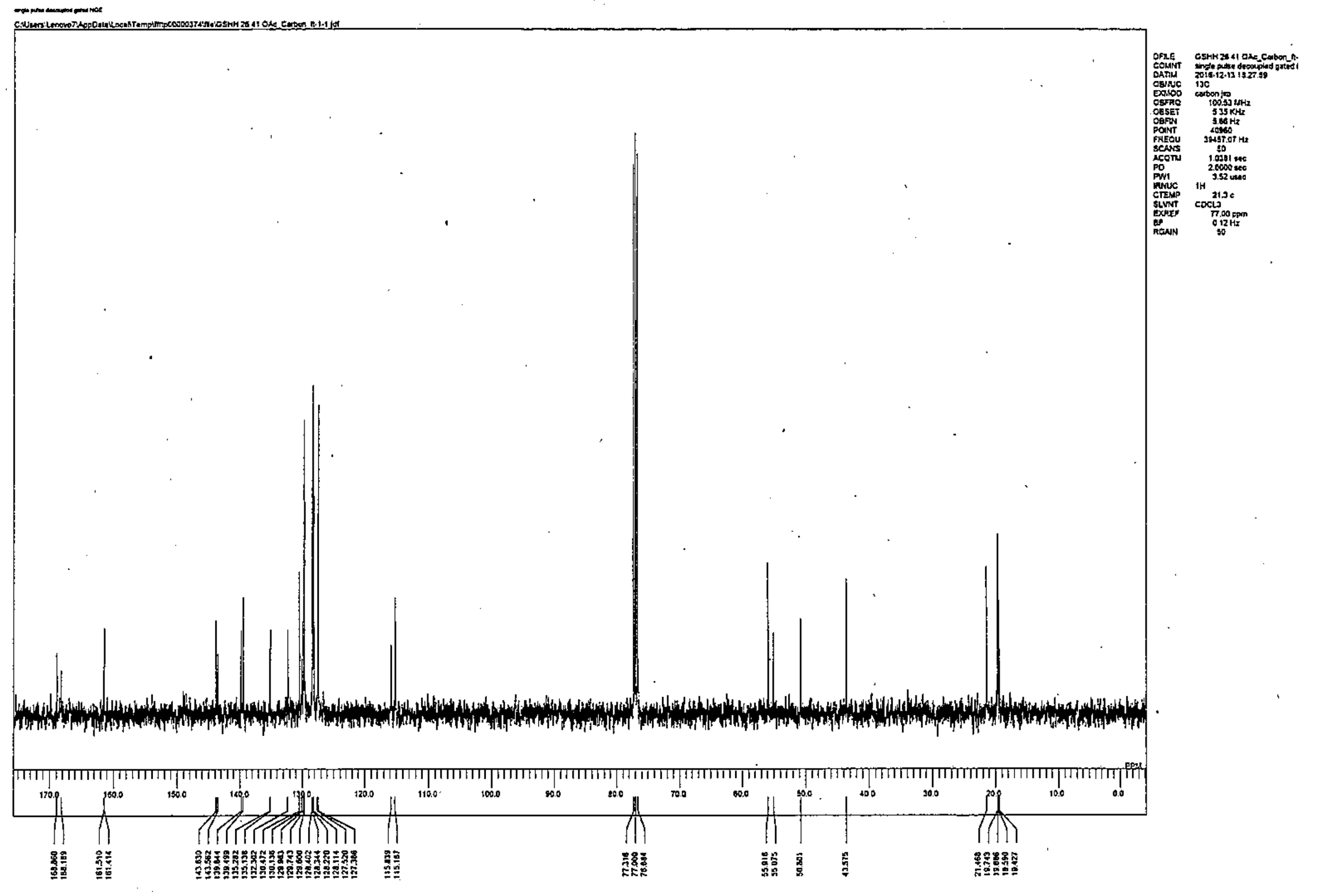
single pulse decoupled gated NOE

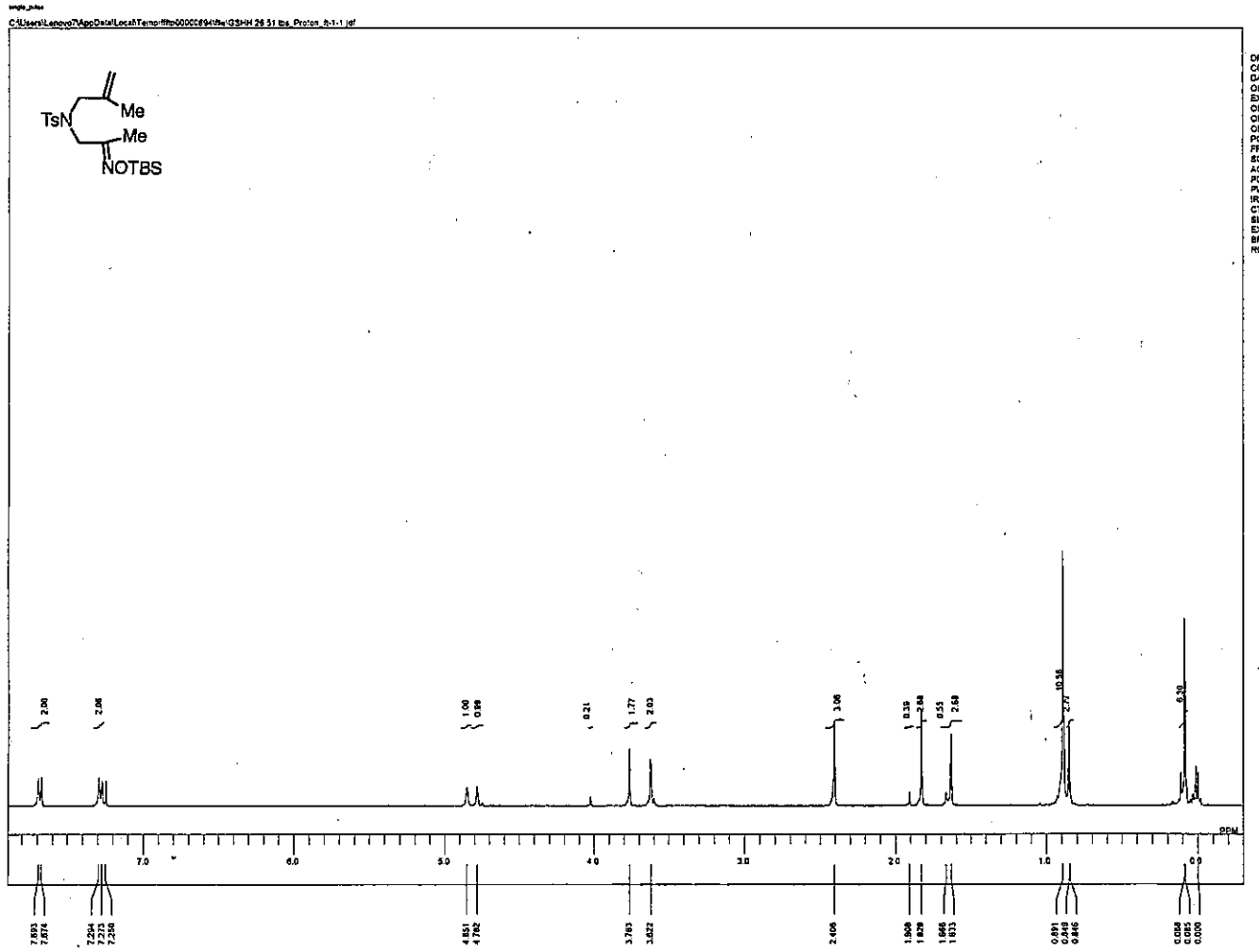
C:\Users\Lenovo7\AppData\Local\Temp\Rtmp00000784\file\GSHH oxime Bz sm_Carbon-1-1.jdf

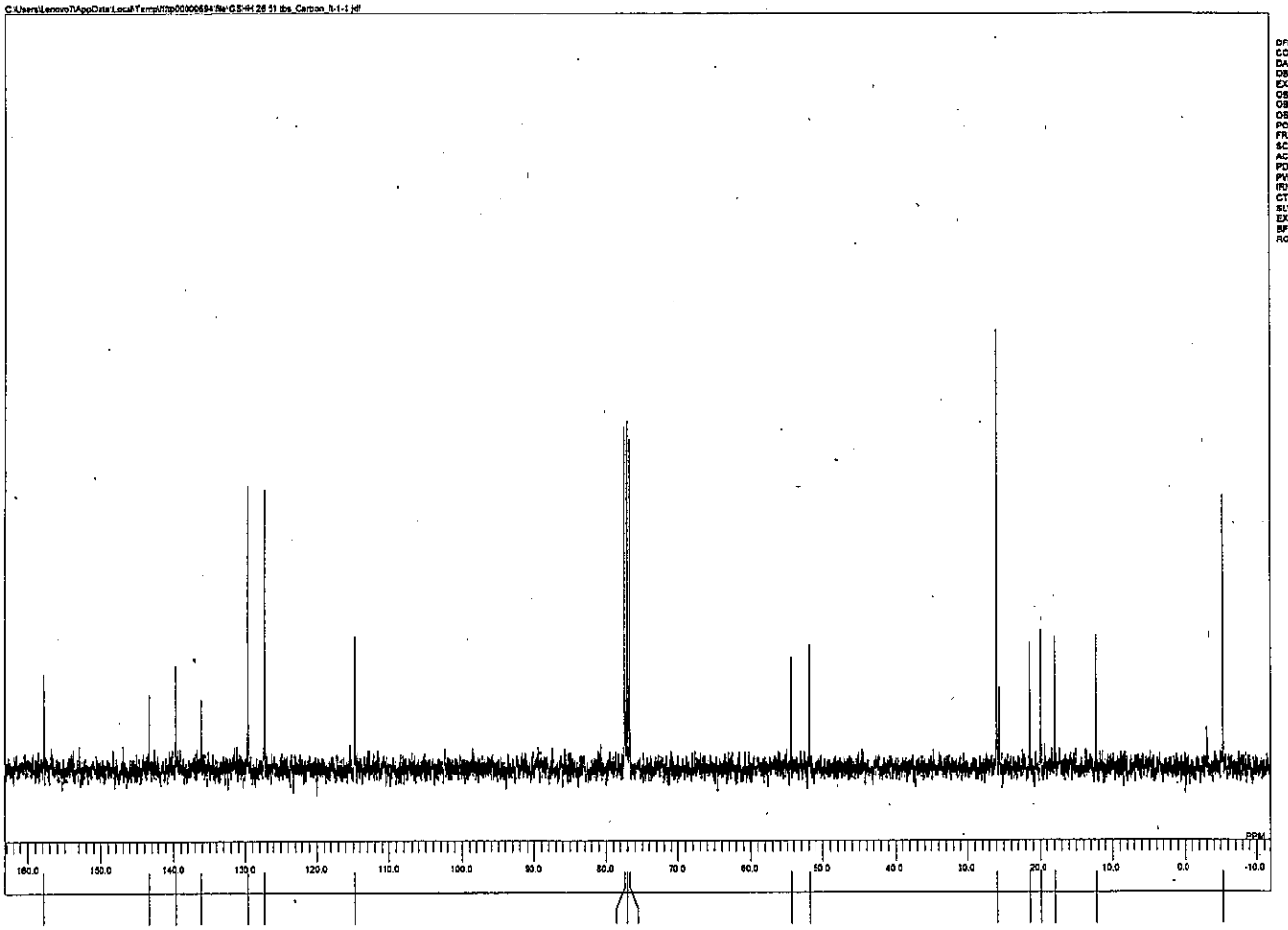
DFILE GSHH oxime Bz sm_Carbon-
COMNT single pulse decoupled gated I
DATIM 2018-02-27 12:14:47
OBNUC 13C
EXMOD carbon.jxp
OBFRQ 100.53 MHz
OBSET 5.35 KHz
OBFIN 5.86 Hz
POINT 32767
FREQU 31565.66 Hz
SCANS 1fd
ACQTM 1.0381 sec
PD 2.0000 sec
PW1 4.08 usec
IRNUC 1H
CTEMP 19.0 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 60

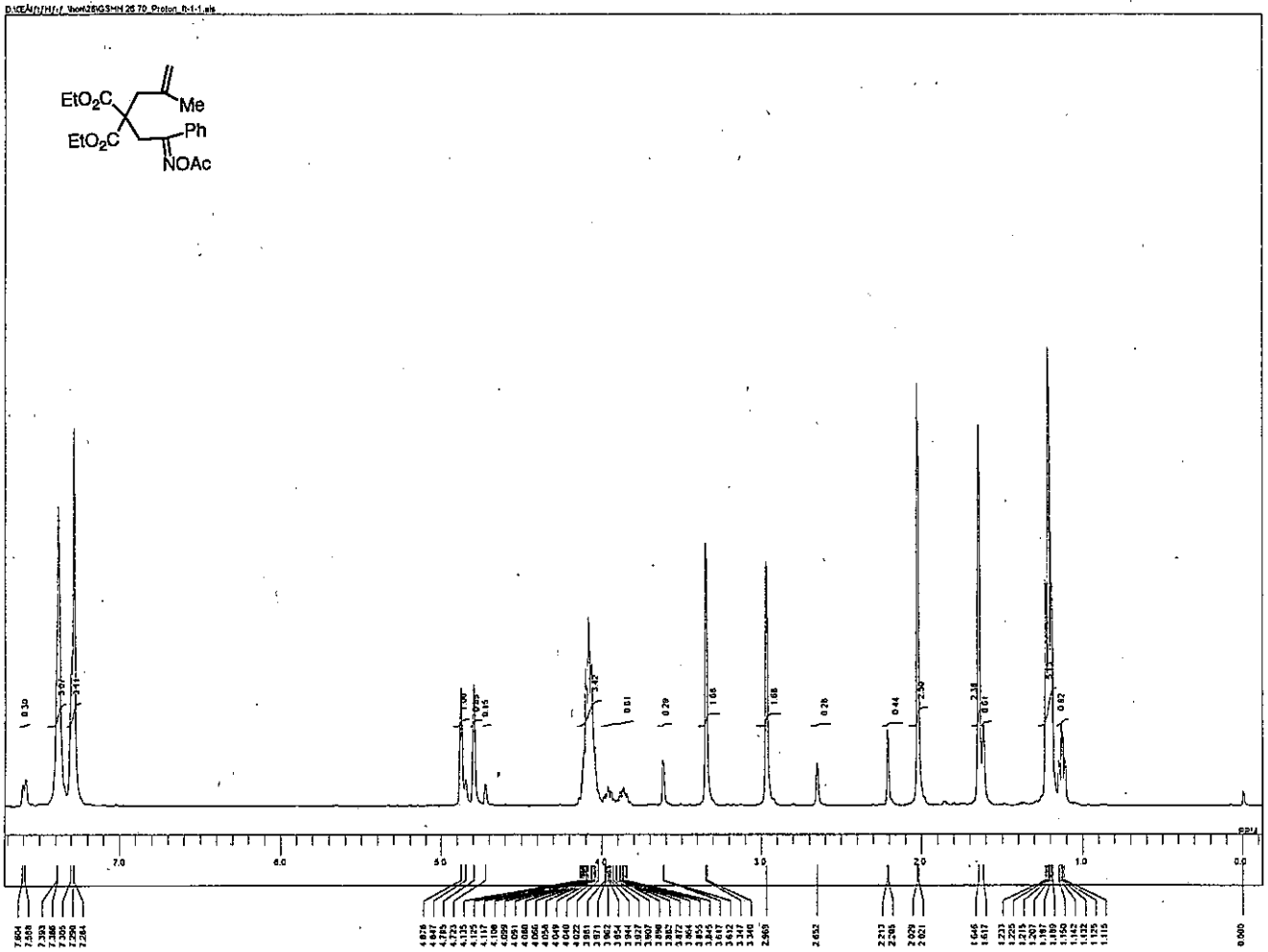


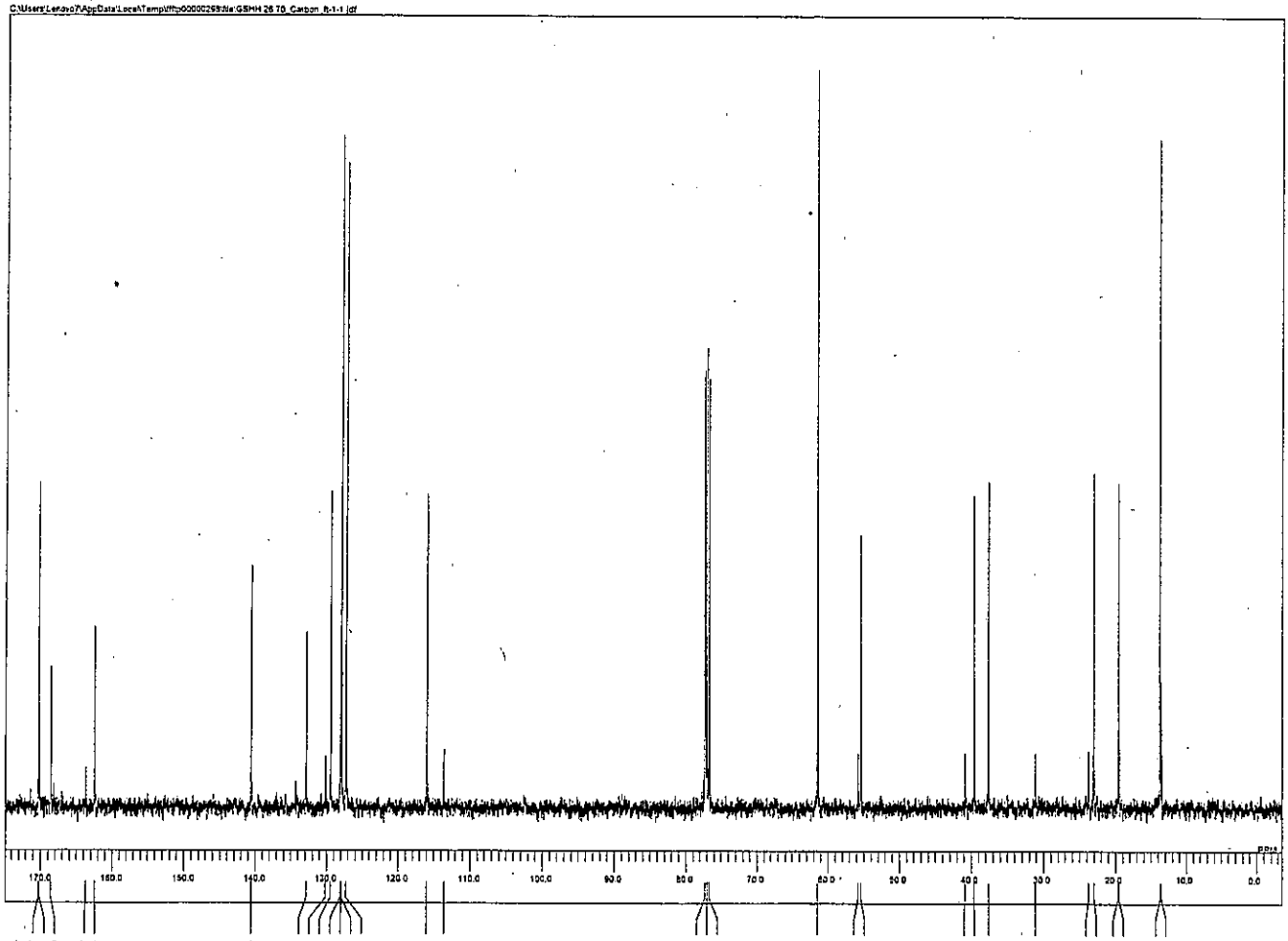

C:\Users\Lenovo7\AppData\Local\Temp\fmp00000374\fe\GSHH 26 41 OAc_Proton_ft-1-1.jdf
TsN
Me
Ph
NOAc
DFILE GSHH 26 41 OAc_Proton_ft-
COMNT single_pulse
DATIM 2016-12-13 15:27:07
OBNUC 1H
EXMOD proton.jxp
OBFRQ 399.78 MHz
OBSET 4.19 KHz
OBFIN 7.29 Hz
POINT 26480
FREQU 8367.51 Hz
SCANS 4
ACQTM 2.1863 sec
PD 5.0000 sec
PW1 2.72 usec
IRNUC 1H
CTEMP 21.2 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 46
PPM
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
4.885
4.825
4.808
4.758
4.536
4.311
3.627
3.514
2.440
2.422
2.244
2.031
1.414
1.356
0.000

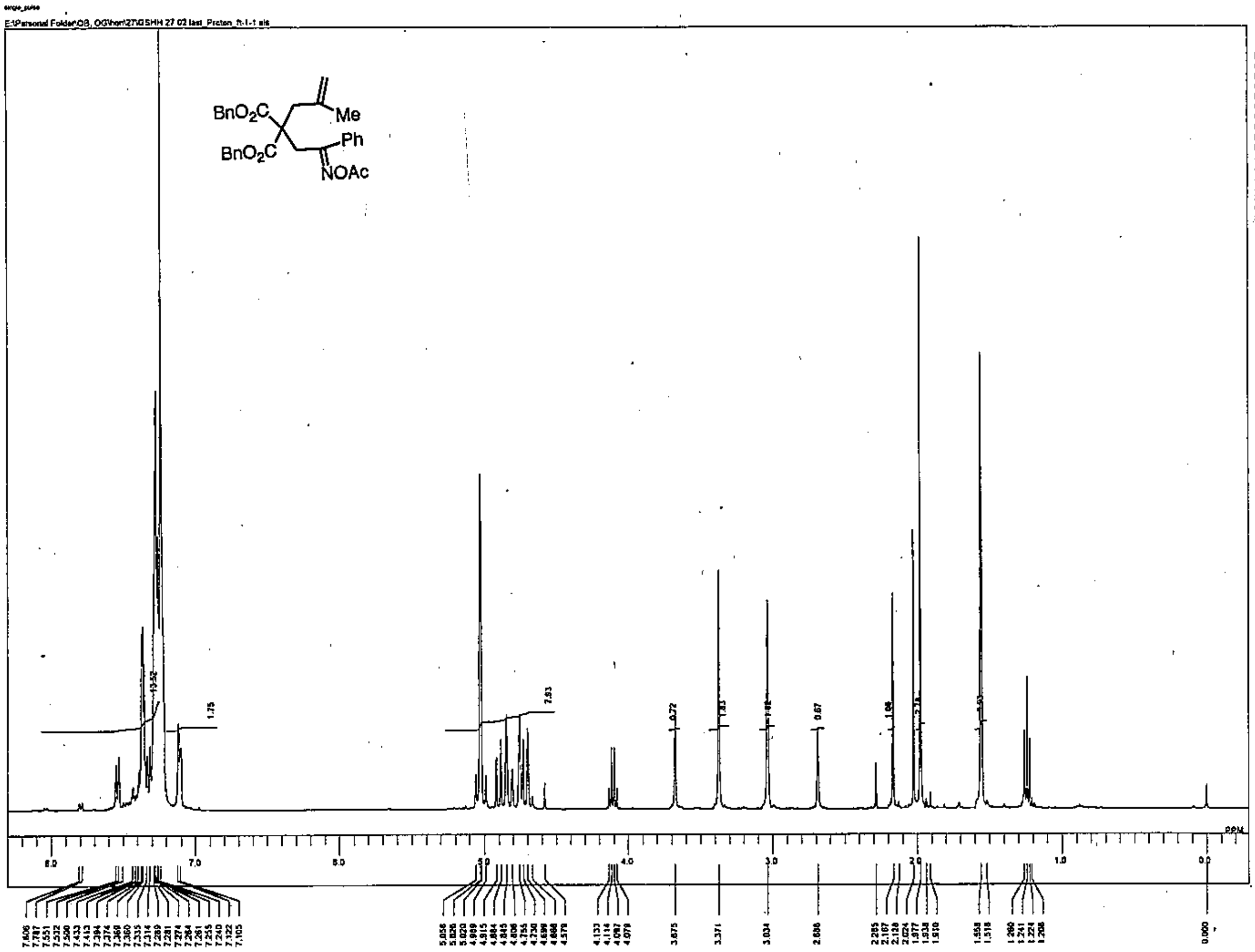

C:\Users\Lenovo7\AppData\Local\Temp\fmp00000374\fe\GSHH 26 41 OAc_Carbon_ft-1-1.jdf
DFILE GSHH 26 41 OAc_Carbon_ft-
COMNT single pulse decoupled gated
DATIM 2016-12-13 15:27:59
OBNUC 13C
EXMOD carbon.jxp
OBFRQ 100.53 MHz
OBSET 5.35 KHz
OBFIN 5.86 Hz
POINT 40960
FREQU 39457.07 Hz
SCANS 50
ACQTM 1.0381 sec
PD 2.0000 sec
PW1 3.52 usec
IRNUC 1H
CTEMP 21.3 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 50
PPM
170.0
160.0
150.0
140.0
130.0
120.0
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0.0
168.860
168.189
161.510
161.414
143.630
143.562
139.844
139.499
135.282
135.136
132.342
130.472
130.136
129.983
129.743
129.600
128.402
128.344
128.220
128.114
127.520
127.386
115.839
115.187
77.316
77.000
76.684
55.916
55.075
50.801
43.575
21.468
19.743
19.696
19.590
19.427


single_pulse
C:\Users\Lenovo7\AppData\Local\Temp\fftp00000694\file\GSHH 26 51 tbs_Proton_fi-1-1.jdf
TsN
Me
Me
NOTBS
DFILE GSHH 26 51 tbs_Proton_fi-1-
COMNT single_pulse
DATIM 2016-12-17 17:00:31
OBNUC 1H
EXMOD proton.jxp
OBFRQ 399.78 MHz
OBSET 4.19 KHz
OBFIN 7.29 Hz
POINT 20480
FREQU 9367.51 Hz
SCANS 8
ACQTM 2.1883 sec
PD 5.0000 sec
PW1 2.72 usec
IRNUC 1H
CTEMP 19.5 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 3.00 Hz
RGAIN 36
2.00
2.06
1.00
0.89
0.21
1.77
2.03
3.06
0.35
2.84
0.55
2.68
10.58
2.79
6.30
PPM
7.693
7.674
7.264
7.273
7.250
4.851
4.782
3.763
3.622
2.406
1.908
1.829
1.666
1.633
0.891
0.848
0.846
0.058
0.052
0.000

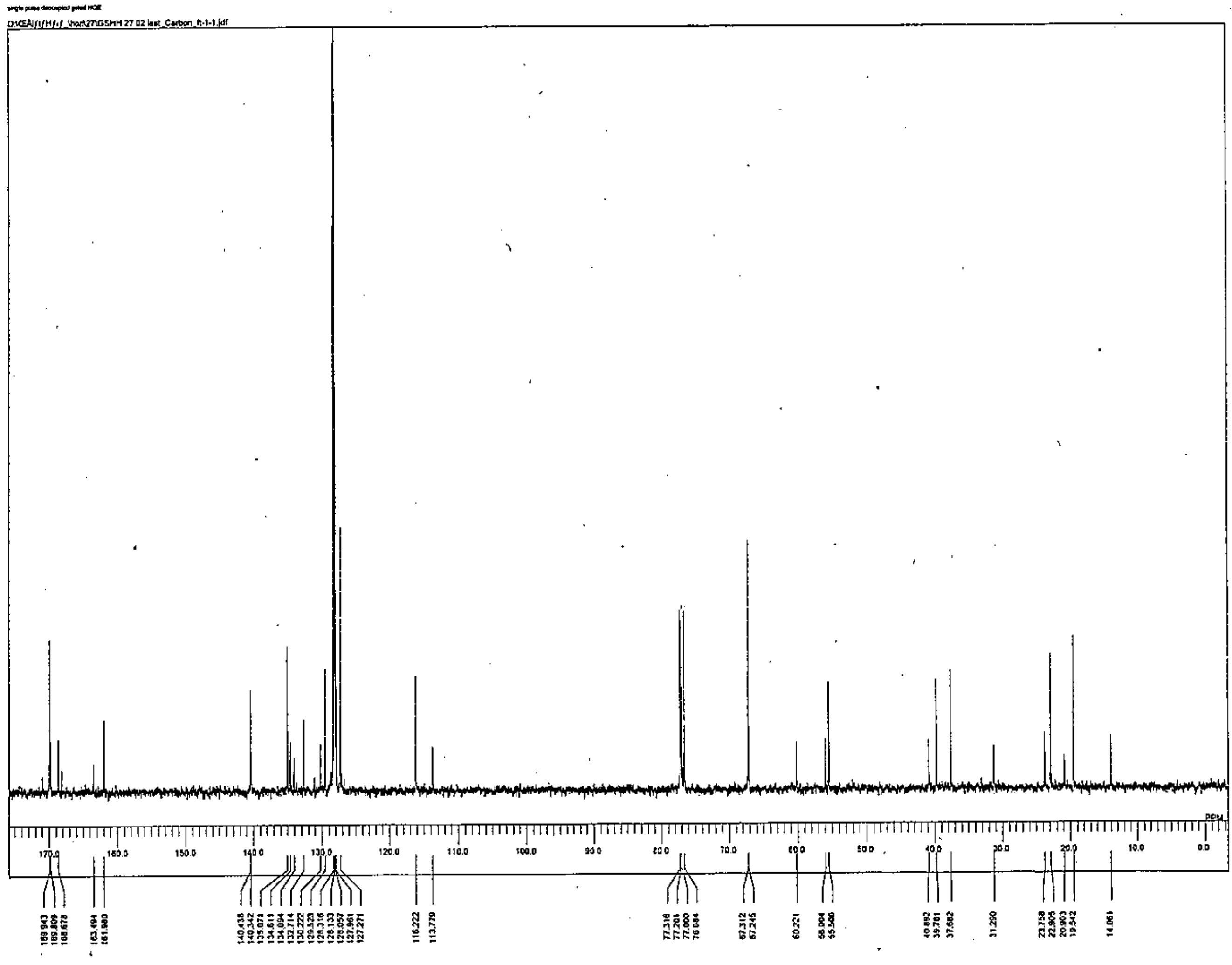

single pulse decoupled gated NOE
C:\Users\Lenovo7\AppData\Local\Temp\fftp00000694\file\GSHH 26 51 tbs_Carbon_fi-1-1.jdf
DFILE GSHH 26 51 tbs_Carbon_fi-1-
COMNT single pulse decoupled gated N
DATIM 2016-12-17 17:02:31
OBNUC 13C
EXMOD carbon.jxp
OBFRQ 100.53 MHz
OBSET 5.39 KHz
OBFIN 5.86 Hz
POINT 40960
FREQU 35457.07 Hz
SCANS 30
ACQTM 1.0361 sec
PD 2.0000 sec
PW1 3.52 usec
IRNUC 1H
CTEMP 19.5 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 3.00 Hz
RGAIN 50
PPM
157.725
143.341
139.681
136.164
129.600
127.335
114.786
77.316
77.000
76.684
54.346
51.922
25.862
21.455
19.354
17.970
12.345
-5.257


EtO2C
Me
Ph
EtO2C
NOAc
DFILE GSHH 26 70_Proton_R-1-1.als
COMNT single_pulse
DATIM 2017-01-30 16:11:44
OBNUC 1H
EXMOD proton.jxp
OBFRQ 399.78 MHz
OBSET 4.19 KHz
OBFIN 7.29 Hz
POINT 16384
FREQU 7503.00 Hz
SCANS 8
ACQTM 2.1837 sec
PD 5.0000 sec
PW1 5.00 usec
IRNUC 1H
CTEMP 26.2 c
SLVNT CDCL3
EXREF 0.00 ppm
SF 0.12 Hz
RGAIN 24

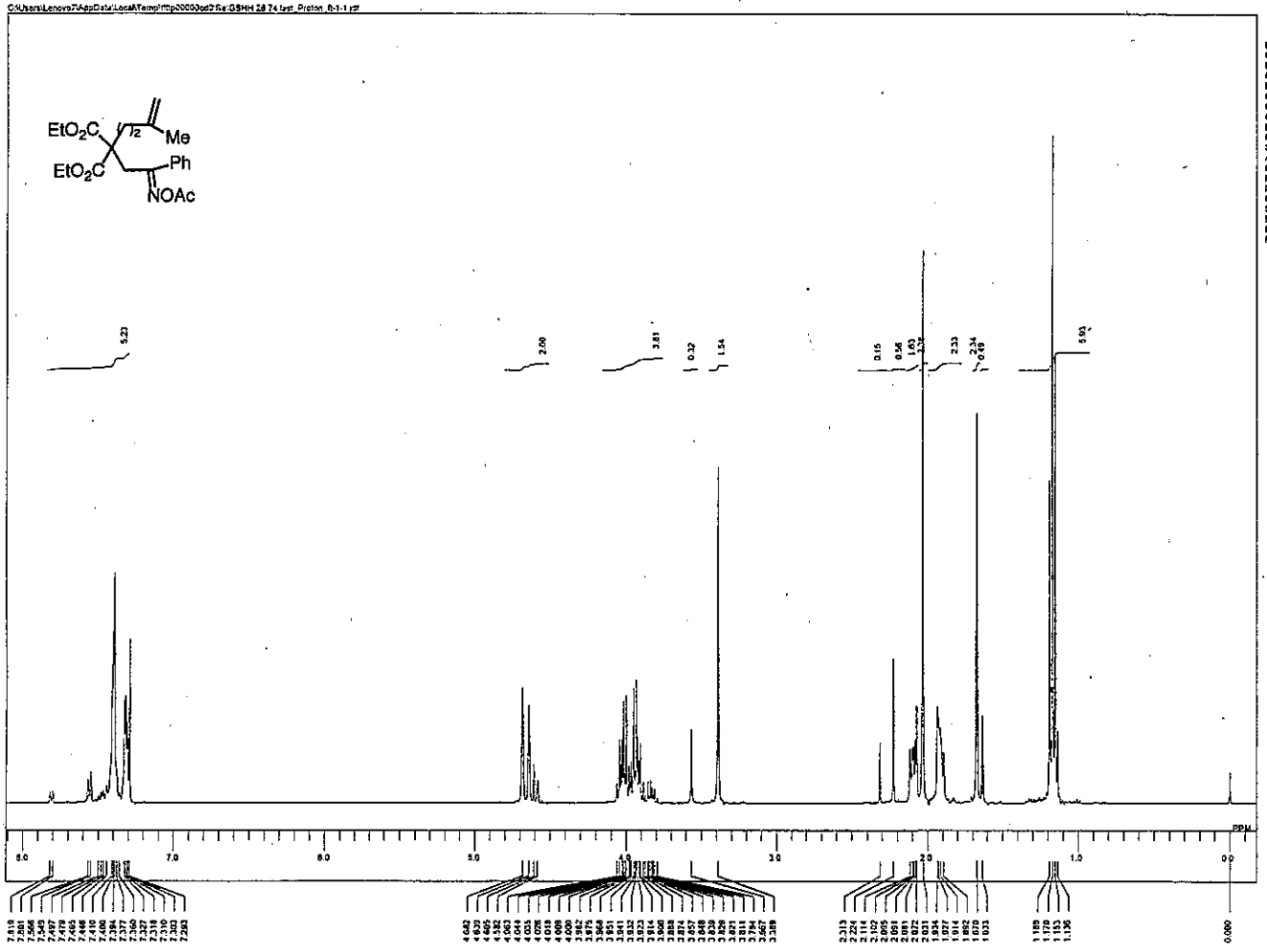

DFILE GSHH 26 70_Carbon_R-1-1
COMNT single pulse decoupled gated
DATIM 2017-01-30 18:12:57
OBNUC 13C
EXMOD carbon.jxp
OBFRQ 100.53 MHz
OBSET 9.33 KHz
OBFIN 5.86 Hz
POINT 40960
FREQU 39258.79 Hz
SCANS 40
ACQTM 1.0433 sec
PD 2.0000 sec
PW1 2.87 usec
IRNUC 1H
CTEMP 26.4 c
SLVNT CDCL3
EXREF 77.00 ppm
SF 0.12 Hz
RGAIN 50

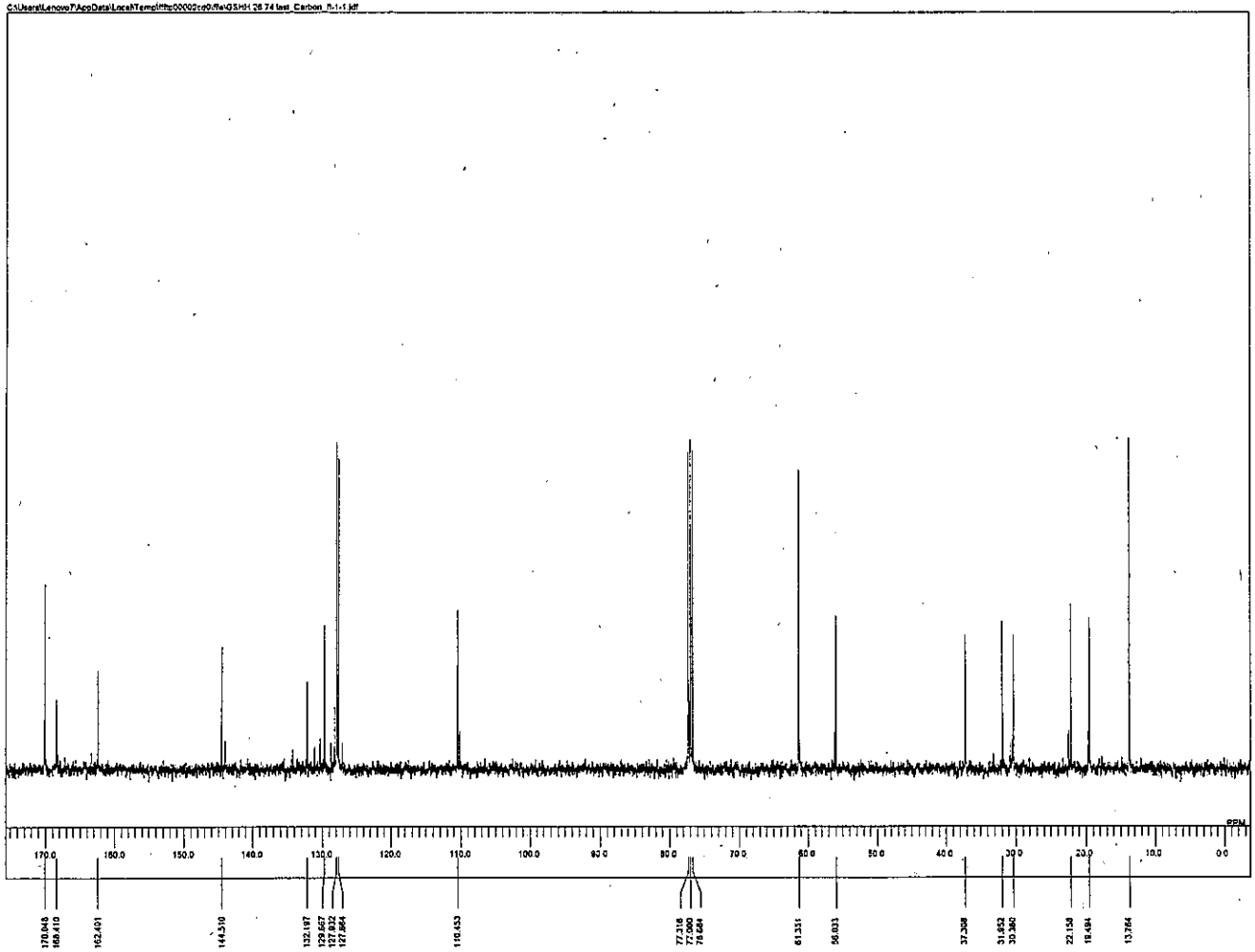


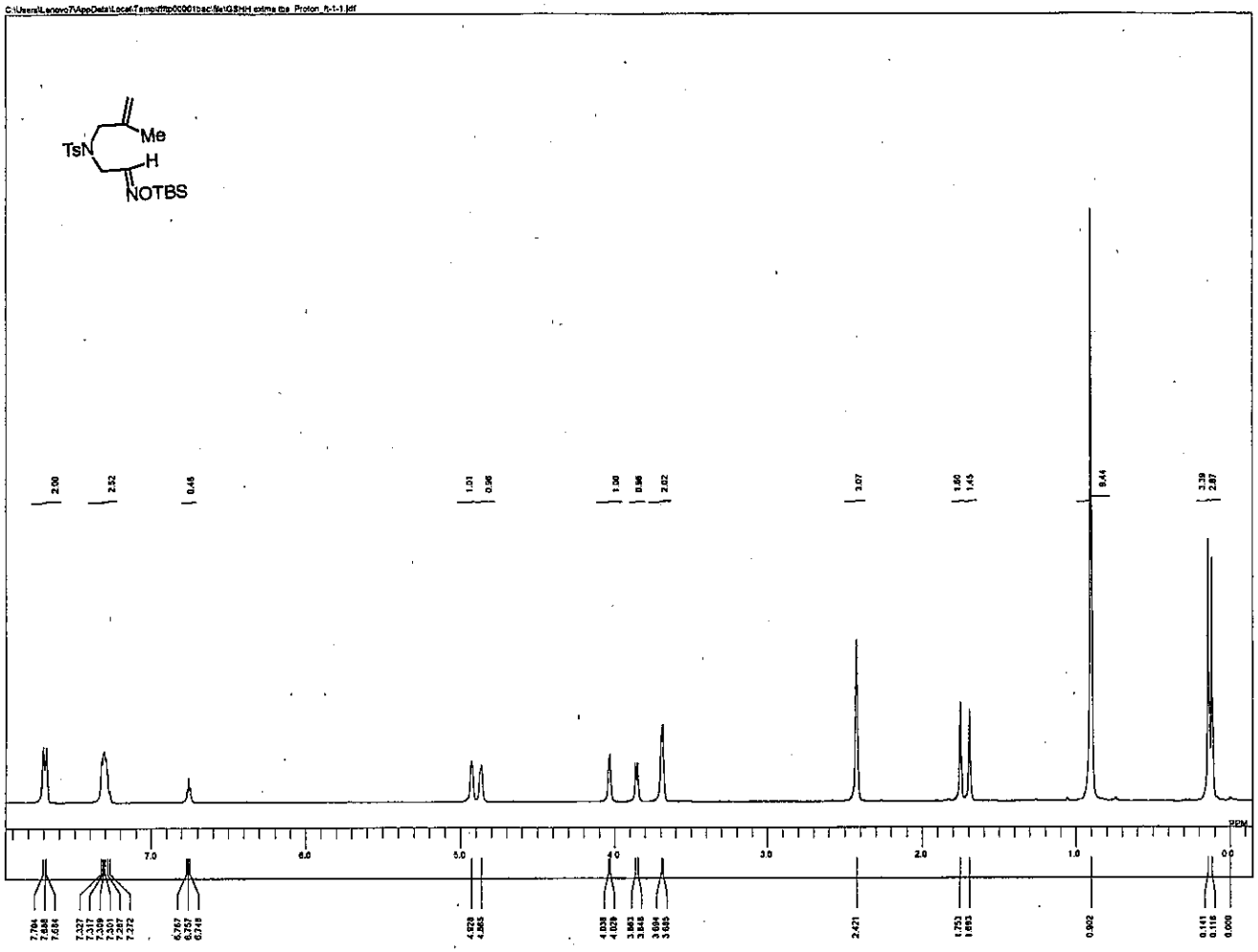
DFILE GSHH 27 02 last_Proton_ft-1-
COMNT single_pulse
DATIM 2017-02-15 21:32:31
OBNUC 1H
EXMOD proton.jxp
OBFRQ 399.78 MHz
OBSET 4.19 KHz
OBFIN 7.29 Hz
POINT 16384
FREQU 7494.00 Hz
SCANS 8
ACQTM 2.1863 sec
PD 5.0000 sec
PW1 2.72 usec
IRNUC 1H
CTEMP 18.6 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 26

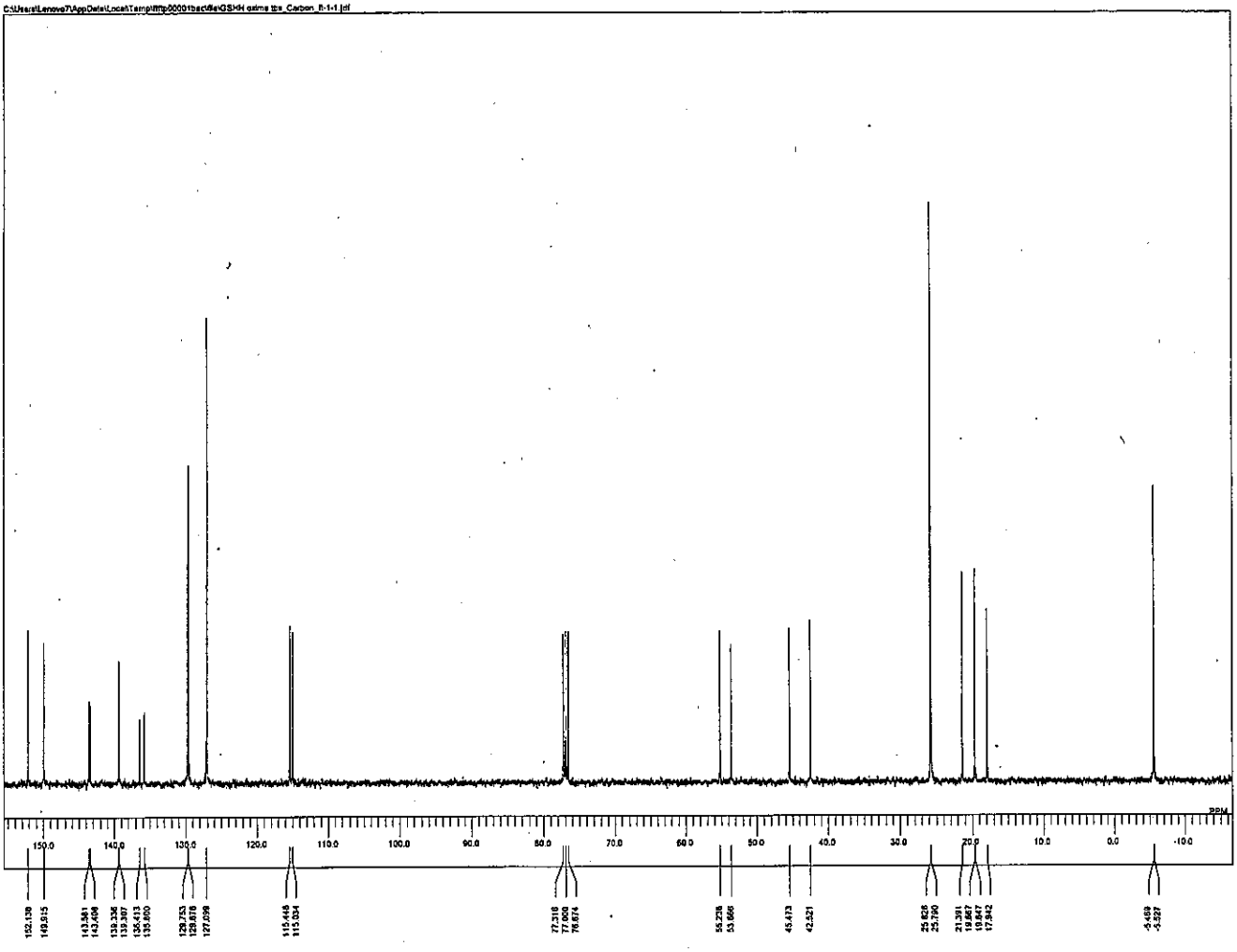


DFILE GSHH 27 02 last_Carbon_ft-1
COMNT single pulse decoupled gated N
DATIM 2017-02-15 21:33:50
OBNUC 13C
EXMOD carbon.jxp
OBFRQ 100.53 MHz
OBSET 5.35 KHz
OBFIN 5.86 Hz
POINT 40960
FREQU 39457.07 Hz
SCANS 50
ACQTM 1.0381 sec
PD 2.0000 sec
PW1 3.52 usec
IRNUC 1H
CTEMP 18.6 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 50


EtO2C
EtO2C
Me
Ph
NOAc
DFILE GSHH 26 74 last_Proton_fl-1-
COMNT single_pulse
DATIM 2017-02-04 12:56:54
OBNUC 1H
EXMOD proton.jxp
OBFRQ 399.78 MHz
OBSET 4.19 KHz
OBFIN 7.29 Hz
POINT 20480
FREQU 6387.51 Hz
SCANS 8
ACQTM 2.1663 sec
PD 5.0000 sec
PW1 2.72 usec
IRNUC 1H
CTEMP 18.4 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 1.00 Hz
RGAIN 38
5.23
2.00
3.81
0.32
1.54
0.15
0.56
1.00
1.43
2.33
2.34
0.48
5.93
PPM


DFILE GSHH 26 74 last_Carbon_fl-
COMNT single pulse decoupled gated
DATIM 2017-02-04 12:58:14
OBNUC 13C
EXMOD carbon.jxp
OBFRQ 100.53 MHz
OBSET 5.35 KHz
OBFIN 5.56 Hz
POINT 40960
FREQU 39457.07 Hz
SCANS 50
ACQTM 1.0381 sec
PD 2.0000 sec
PW1 9.52 usec
IRNUC 1H
CTEMP 18.5 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 1.00 Hz
RGAIN 50
PPM


TsN
Me
H
NOTBS
PPM


PPM