

Supporting Information

Rh-Catalyzed Ortho-Selective C–H Borylation of *N*-Functionalized Arenes with Silica-Supported Bridgehead Monophosphine Ligands

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Instrumentation and Chemicals

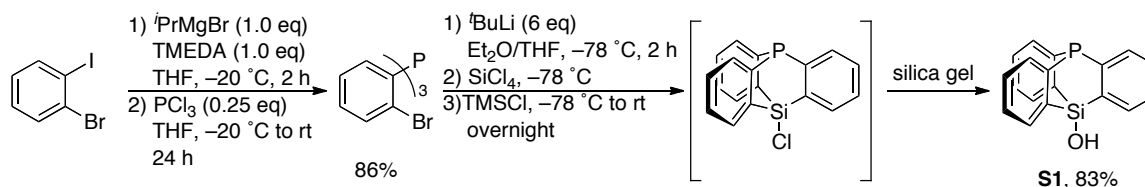
Solution NMR spectra were recorded on a Varian Gemini 2000 spectrometer, operating at 300 MHz for ^1H NMR, 75.4 MHz for ^{13}C NMR and 121.4 MHz for ^{31}P NMR. Chemical shift values for ^1H and ^{13}C are reference to Me_4Si and the residual solvent resonances respectively, and for ^{31}P chemical shift values are reference to 85% phosphoric acid. Chemical shifts are reported in δ ppm. Magic angle spinning (MAS) NMR spectra were recorded on a Bruker ESL-400 spectrometer, operating at 161.9 MHz for ^{31}P NMR. Elemental analysis was performed at the Instrumental Analysis Division, Equipment Management Center, Creative Research Institution, Hokkaido University. High-resolution mass spectra were recorded on a Thermo Fisher Scientific Exactive, JEOL JMS-T100LP mass spectrometer or JEOL JMS-T100 GC mass spectrometer at the Instrumental Analysis Division, Equipment Management Center, Creative Research Institution, Hokkaido University. IR spectrum was recorded on a JASCO FT/IR-4100 spectrophotometer. TLC analyses were performed on commercial glass plates bearing 0.25-mm layer of Merck Silica gel

60F₂₅₄. Silica gel (Kanto Chemical Co., Silica gel 60 N, spherical, neutral) was used for column chromatography. Gel permeation chromatography (GPC) was performed by LC-908 (Japan Analytical Industry Ltd., two in-line JAIGEL-2H, CHCl₃, 3.5 mL/min, UV and RI detectors). GLC analyses were conducted on a Shimadzu GC-14B equipped with a flame ionization detector. Melting points were determined on a micro melting point apparatus (Yanaco: MP-500D) using micro cover glass.

All reactions were carried out under nitrogen atmosphere. Materials were obtained from commercial suppliers or prepared according to standard procedures unless otherwise noted. [Rh(OH)(cod)]₂ and [Ir(OMe)(cod)]₂ were prepared according to the reported procedure.¹ [Rh(OMe)(cod)]₂ was purchased from Aldrich Co., Ltd. Silica-SMAP was prepared with CARiACT Q-10[®] according to the reported procedure.² CARiACT Q-10 was dehydrated by heating at 120 °C for 10 h and stored in a glovebox before use. All solvents for catalytic reactions were degassed via four freeze–pump–thaw cycles before use. 2-Phenylpyridine was purchased from Aldrich Co., Ltd., and purified by distillation with CaH₂ before use. Bis(pinacolato)diboron was purchased from AllyChem Co., Ltd., and recrystallized from pentane before use.

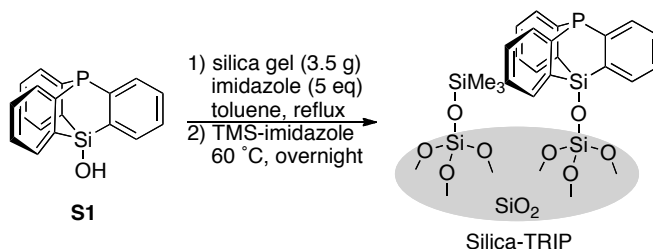
Preparation of Ligands

Preparation of 10-Hydroxy-9-phospha-10-silatriptycene (S1). The reported procedure for the preparation of 10-chloro-9-phospha-10-silatriptycene was modified as follows:³ the magnesiation of 2-iodobromobenzene (3.4 g, 12.0 mmol) through the Mg/I exchange followed by the reaction with phosphorous trichloride gave tri-(2-bromophenyl)phosphine in 86% yield. The trilithiatoin with ^tBuLi followed by the reaction with tetrachlorosilane gave 10-chloro-9-phospha-10-silatriptycene. After quenching the excess organolithium species with TMSCl, crude material was treated by silica gel column chromatography (hexane/diethyl ether 100:0–80:20), to give **S1** in 83% yield.



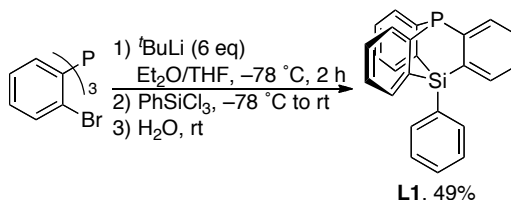
White solid. **M.p.** 320.8 °C, decomp. ¹H NMR (CDCl₃) δ 3.35 (bs, 1H), 7.19–7.34 (m, 6H), 7.80–7.91 (m, 6H). ¹³C NMR (CDCl₃) δ 127.90 (d, *J* = 1.7 Hz), 128.10 (d, *J* = 15.4 Hz), 131.77, 134.74 (d, *J* = 46.3 Hz), 141.50 (d, *J* = 2.3 Hz), 146.24 (d, *J* = 8.5 Hz). ³¹P NMR (CDCl₃) δ –54.5. IR (CDCl₃): 3110 (br, SiO–H), 3055, 1573, 1428 cm^{–1}. HRMS–EI (*m/z*): [M+H]⁺ calcd for C₁₈H₁₄OPSi, 305.05460; found, 305.0549.

Preparation of Silica-TRIP. 10-Hydroxy-9-phospha-10-silatriptycene (**S1**) (0.34 mmol, 109.9 mg), CARIACT Q-10[®] silica gel (3.5 g), imidazole (1.7 mmol, 120.2 mg) and anhydrous, degassed toluene (6 mL) were placed in a 50 mL glass tube containing a magnetic stirring bar. The tube was a screw cap, and suspension was refluxed for 16 h. The mixture was filtered through a cotton, and was washed successively with degassed toluene, degassed MeOH, degassed water, and again with degassed MeOH. Drying in vacuum at 120 °C overnight. The obtained gel was treated with *N*-trimethylsilylimidazole (8.5 ml, excess) at 60 °C for 12 h. A mixture and was filtered through a cotton, and was washed with degassed MeOH. Drying in vacuum at 120 °C overnight gave 3.12 g of Silica-TRIP (loading amount: 0.070 mmol P/g, 64% yield from **S1**).^{2a}



Preparation of 10-Phenyl-9-phospha-10-silatriptycene (**L1**).

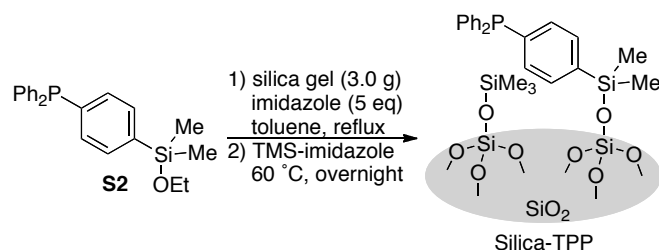
10-Phenyl-9-phospha-10-silatriptycene (**L1**) was synthesized with 248.2 mg (0.5 mmol) of tris(2-bromophenyl)phosphine according to the procedure for the preparation of **S1**, except for using trichlorophenylsilane instead of tetrachlorosilane. A crude material was purified by GPC to give pure **L1** in 49% yield.



White solid. **M.p.** 244.6–246.3 °C. ¹H NMR (CDCl₃) δ 7.20–7.28 (m, 6H), 7.70–7.72 (m, 3H), 7.76–7.78 (m, 3H), 7.94–8.00 (m, 3H), 8.26–8.30 (m, 2H). ¹³C NMR (CDCl₃) δ 127.13, 127.84 (d, *J* = 1.7 Hz), 128.05 (d, *J* = 15.4 Hz), 128.94, 131.16, 133.18 (d, *J* = 1.1 Hz), 135.02 (d, *J* = 47.3 Hz), 136.51, 142.16 (d, *J* = 2.8 Hz), 146.81 (d, *J* = 8.0 Hz). ³¹P NMR (C₆D₆) δ –44.51. **HRMS–ESI** (*m/z*): [M+H]⁺ calcd for C₂₄H₁₈PSi, 365.09099; found, 365.09119.

Preparation of Silica-TPP. [4-(Ethoxydimethylsilyl)phenyl]diphenylphosphine (**S2**) was prepared according to reported procedure.⁴ **S2** (0.34 mmol, 129.8 mg), silica gel (3.0 g), imidazole (1.7 mmol, 120.2 mg), and anhydrous, degassed toluene (12 mL) were placed in a 50 mL glass tube containing a magnetic stirring bar, and the tube was sealed with a screw cap. The suspension was refluxed for 16 h. A mixture was filtered through a cotton, and was washed successively with degassed toluene, degassed MeOH, degassed water, and again with degassed MeOH. After drying in vacuum at 120°C overnight, the gel was treated with *N*-trimethylsilylimidazole (8.5 ml, excess)

at 60°C for 12 h, and was filtered through a cotton and washed with degassed MeOH. Drying in vacuum at 120°C overnight gave 3.1 g of Silica-TPP (loading amount: 0.053 mmol P/g, 47% yield from S.M.).^{2a}



Experimental Procedures for Borylation

Typical Procedure for the Borylation of 2-Phenylpyridine (1a**) with Immobilized Ligand (Figure 1, with Silica-SMAP).** In a glove box, Silica-SMAP (0.069 mmol/g, 0.0025 mmol, 0.5 mol%), bis(pinacolato)diboron (**2**) (127.0 mg, 0.50 mmol), dibenzyl (50 mg, as internal standard) and anhydrous, degassed THF (0.72 mL) were placed in a 10 mL glass tube containing a magnetic stirring bar. A solution of [Rh(OH)(cod)]₂ (0.58 mg, 0.00125 mmol, 0.25 mol%) in THF (0.28 mL) and 2-phenylpyridine (**1a**) (155.2 mg, 0.50 mmol) were added. The tube was sealed with a screw cap and was removed from the glovebox. The mixture was stirred at room temperature for 1 h, and filtered through a glass pipet equipped with a cotton filter. Solvent was removed under reduced pressure. The yield of the product was determined by GC (103%). The crude material was purified by Kugelrohr distillation to give the corresponding arylboronate **3a** (131.3 mg, 0.47 mmol) in 93% isolated yield.

Typical Procedure for the Borylation of 2-Phenylpyridine (1a) with Soluble Ligand (Figure 1, with Ph-SMAP). In a glove box, bis(pinacolato)diboron (**2**) (127.0 mg, 0.50 mmol), dibenzyl (50 mg, as internal standard) and anhydrous, degassed THF (0.58 mL) were placed in a 10 mL glass tube containing a magnetic stirring bar. A solution of Ph-SMAP (0.55 mg, 0.0025 mmol, 0.5 mol%) in THF (0.14 mL), a solution of [Rh(OH)(cod)]₂ (0.58 mg, 0.00125 mmol, 0.25 mol%) in THF (0.28 mL) and 2-phenylpyridine (**1a**) (155.2 mg, 0.50 mmol) were added in this order. The tube was sealed with a screw cap and was removed from the glovebox. The mixture was stirred at room temperature for 1 h. Solvent was removed under reduced pressure. The yield of the product was determined by GC (17%).

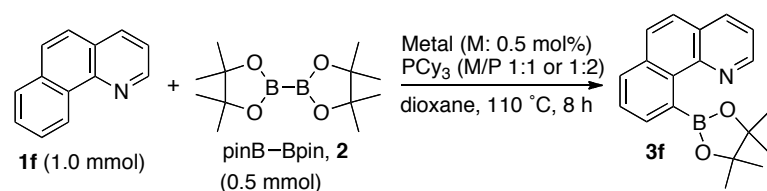
Typical Procedure for the Borylation of Functionalized Arenes (Table 1, entry 1). In a glove box, Silica-SMAP (0.069 mmol/g, 0.0025 mmol, 0.5 mol%), bis(pinacolato)diboron (**2**) (127.0 mg, 0.50 mmol) and anhydrous, degassed THF (0.72 mL) were placed in a 10 mL glass tube containing

a magnetic stirring bar. A solution of $[\text{Rh}(\text{OH})(\text{cod})]_2$ (0.58 mg, 0.00125 mmol, 0.25 mol%) in THF (0.28 mL) and 2-(3-methylphenyl)pyridine (**1b**) (169.2 mg, 0.50 mmol) were added. The tube was sealed with a screw cap and was removed from the glove box. The mixture was stirred at room temperature for 8 h, and filtered through a glass pipet equipped with a cotton filter. Solvent was removed under reduced pressure. An internal standard (1,1,2,2-tetrachloroethane) was added to the residue. The yield of the product was determined by ^1H NMR (95%, C6'/C2'=46:1). The crude material was then purified by Kugelrohr distillation to give the corresponding arylboronate **3b** (110.1 mg, 0.37 mmol in 75% isolated yield).

Metal Screening for the Borylation

In preliminary research, the screening of various transition metal catalyst was performed in the reaction of borylation of benzo[*h*]quinoline (**1f**) with PCy_3 (Table S1).

Table S1. Metal Screening for Various Metal-Catalyzed Borylation of Benzo[*h*]quinoline (**1f**).

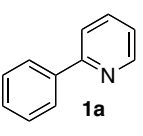
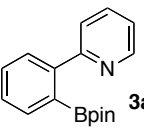
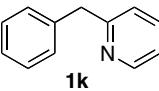
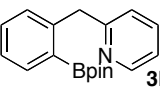
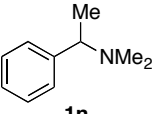
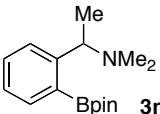
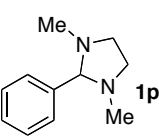
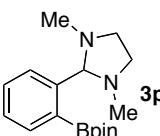


Entry	Metal precursor	[M]/[L]	GC conv. (%)	NMR yield (%)
1	$[\text{PtCl}_2(\text{H}_2\text{C}=\text{CH}_2)_2]_2$	1:1	17	18
2	$[\text{PtCl}_2(\text{H}_2\text{C}=\text{CH}_2)_2]_2$	1:2	0	0
3	$\text{Pd}(\text{OAc})_2$	1:1	0	0
4	$\text{Pd}(\text{OAc})_2$	1:2	0	0
5	$[\text{Ir}(\text{OMe})(\text{cod})]_2$	1:1	74	0*
6	$[\text{Ir}(\text{OMe})(\text{cod})]_2$	1:2	15	13
7	$[\text{Rh}(\text{OMe})(\text{cod})]_2$	1:1	100	99
8	$[\text{Rh}(\text{OMe})(\text{cod})]_2$	1:2	99	99

*The borylation afforded unidentified isomer.

Ligand Effects (Table S2).

Table S2. Rh-catalyzed Borylation of Various Substrates with Silica-SMAP or PCy₃ Ligands or with Ligand-Free systems.^a

Entry	Substrate	Product	Ligand	P/Rh	Temp. (°C)	Time (h)	NMR yield (%)
1	 1a	 3a	Silica-SMAP	1	rt	1	103 ^b
2			PCy ₃	1	rt	1	35 ^b
3			PCy ₃	2	rt	1	6 ^b
4			None	-	rt	1	10 ^b
5			None	-	rt	12	106 ^b
6	 1k	 3k	Silica-SMAP	1	rt	2	108
7			PCy ₃	1	rt	2	trace
8			PCy ₃	2	rt	2	trace
9			PCy ₃	1	60	17	16
10			PCy ₃	2	60	17	5
11			None	-	60	17	trace
12	 1n	 3n	Silica-SMAP	1	40	6	99
13			PCy ₃	1	60	14	3
14			PCy ₃	2	60	14	6
15			None	-	60	14	2
16	 1p	 3p	Silica-SMAP	1	40	0.5	92
17			PCy ₃	1	60	13	9
19			PCy ₃	2	60	13	1
15			None	-	60	13	56

^a Conditions: **1** (1.0 mmol), **2** (0.5 mmol), [Rh(OH)(cod)]₂ (Rh: 0.0025 mmol), ligand (P: 0.0025 mmol or 0.0050 mmol), THF (1.0 mL). ^b GC yield.

Complementary Use of the Silica-SMAP–Rh and Silica-SMAP–Ir systems

Scheme S1. The Another Example of Complementary Borylation; The Borylation of 3-Acetylphenyl Diethylcarbamate Derivatives with Silica-TRIP–Rh and Silica-SMAP–Ir systems.

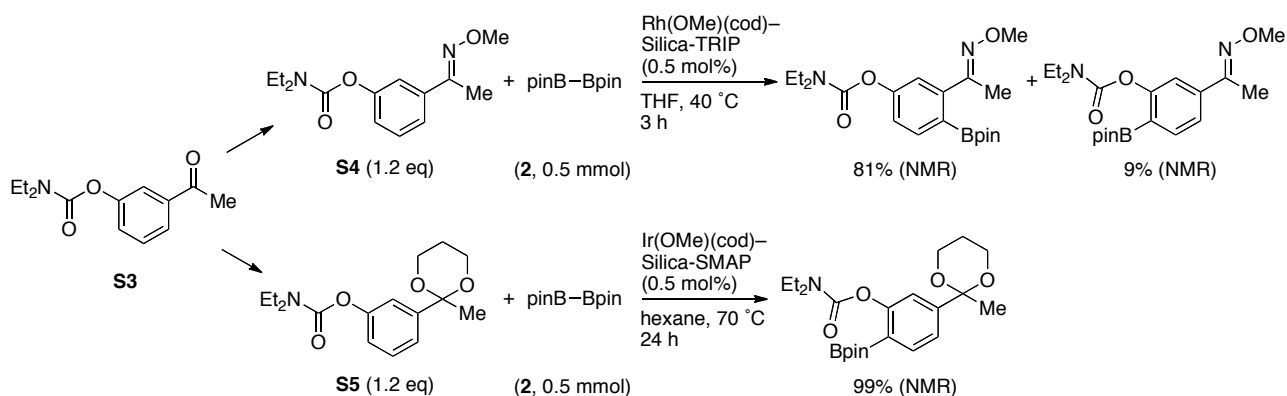
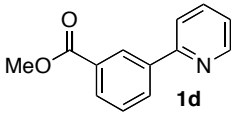
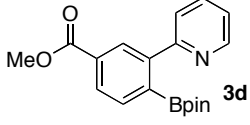
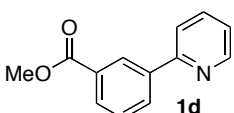
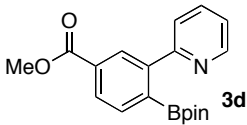
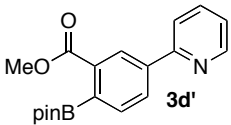
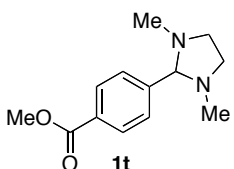
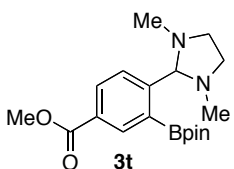
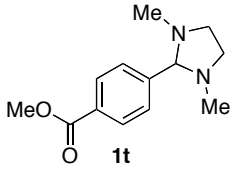
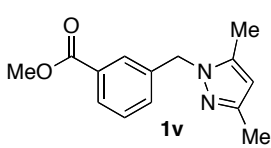
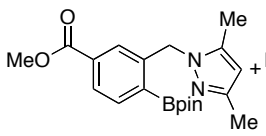
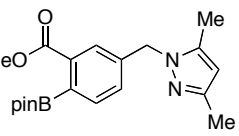
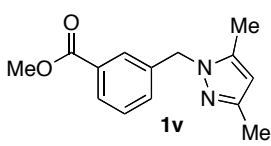
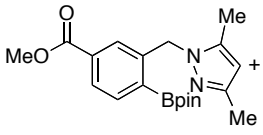
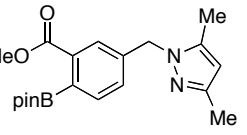


Table S3. Limitations of Complementary *N*- or *O*-Directed Borylation of Arenes.

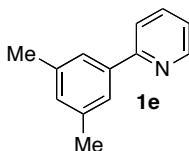
$ \begin{array}{c} \text{O-DG} \text{---} \text{C}_6\text{H}_4 \text{---} \text{N-DG} \\ \text{(Ar, 1 or 2 eq)} \end{array} + \text{pinB-Bpin} \xrightarrow[\text{conditions}]{\text{Rh or Ir (0.5 mol\%)}, \text{Silica-SMAP (0.5 mol\%)}} \begin{array}{c} \text{O-DG} \text{---} \text{C}_6\text{H}_3 \text{---} \text{N-DG} \\ \text{Bpin} \end{array} $ (2, 0.5 mmol)			
Entry	Substrate	Conditions	Products (NMR yield)
1	 1d	[Rh(OH)(cod)] ₂ THF, 40 °C, 2 h Ar = 1.0 eq	 3d >99%
2	 1d	[Ir(OMe)(cod)] ₂ hexane, 40 °C 15 h Ar = 1.0 eq	 3d trace +  3d' trace
3	 1t	[Rh(OH)(cod)] ₂ THF, 40 °C, 13 h Ar = 2.0 eq	 3t 83%
4	 1t	[Ir(OMe)(cod)] ₂ hexane, 60 °C, 4 h Ar = 2.0 eq	N.R.
5	 1v	[Rh(OH)(cod)] ₂ THF, 40 °C, 3 h Ar = 1.0 eq	 3v N.D. +  3v' 91%
6	 1v	[Ir(OMe)(cod)] ₂ hexane, 60 °C 1 h Ar = 1.0 eq	 3v N.D. +  3v' >99%

With the Silica-SMAP–Rh system, the borylation of **1d** and **1t** occurred at the position *ortho* to the nitrogen-based directing group (Table S3, entries 1 and 3). However, the Silica-SMAP–Ir system was not applicable to the borylation of these substrate (entries 2 and 4). The reaction might be inhibited by the strong coordination ability of the nitrogen-based substitution. On the other hands, the borylation of methyl 3-(3,5-dimethylpyrazolyl)methylbenzoate (**1v**) occurred cleanly at the position *ortho* to the ester group irrespective of using the Rh or Ir catalyst systems (entries 5 and 6).

Preparation of Substrates

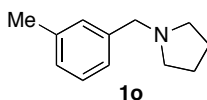
The starting materials **1a**, **1f** and **1k** are known compounds. The starting materials **1b**⁵, **1c**⁶, **1d**⁷, **1g**⁸, **1h**⁹, **1i**¹⁰, **1j**¹¹, **1l**¹², **1m**¹³, **1n**¹⁴, **1p**¹⁵, **1q**¹⁶, **1r**¹⁷, **1s**¹⁸ and **1u**¹⁹ shown in Tables 1 and 2 are known compounds.

Preparation of 2-(3,5-Dimethylphenyl)pyridine (1e). 2-(3,5-Dimethylphenyl)pyridine (**1e**) was synthesized by the Suzuki-Miyaura coupling reaction of 3,5-dimethylphenylboronic acid (900 mg. 6.0 mmol) with 2-bromopyridine (790 mg. 5.0 mmol) according to the reported procedure.²⁰ Purification by silica gel column chromatography (hexane/diethyl ether = 100:0–90:10) gave pure **1e** in 96% yield.



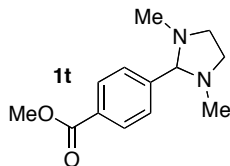
Colorless oil. ¹H NMR (CDCl₃) δ 2.40 (s, 6H), 7.07 (s, 1H), 7.22 (m, 1H), 7.60 (s, 2H), 7.70–7.77 (m, 2H), 8.69 (d, *J* = 4.5 Hz, 1H). ¹³C NMR (CDCl₃) δ 21.26, 120.70, 121.97, 124.80, 130.67, 136.72, 138.35, 139.42, 149.63, 157.86. **Anal.** Calcd for C₁₃H₁₃N: C, 85.21%; H, 7.15%; N, 7.64%. Found: C, 85.16%; H, 7.23%; N, 7.47%.

Preparation of 1-(3-Methylbenzyl)pyrrolidine (1o). 1-(3-Methylbenzyl)pyrrolidine (**1o**) was synthesized by of 1-(bromomethyl)-3-methylbenzene (3.7 g. 20.0 mmol) with pyrrolidine (1.8 g. 25.0 mmol) according to the reported procedure.²¹ Purification by silica gel column chromatography (hexane/diethyl ether = 100:0–90:20) gave pure **1o** in 31% yield.



Colorless oil. ¹H NMR (CDCl₃) δ 1.79 (quin, *J* = 3.3 Hz, 4H), 2.34 (s, 3H), 2.48–2.53 (m, 4H), 3.58 (s, 2H), 7.06 (d, *J* = 7.5 Hz, 1H), 7.12 (d, *J* = 7.5 Hz, 1H), 7.16 (s, 1H), 7.20 (d, *J* = 7.5 Hz, 1H). ¹³C NMR (CDCl₃) δ 21.28, 23.28, 54.17, 60.75, 126.04, 127.67, 128.14, 129.73, 137.91, 139.35. **HRMS–ESI** (*m/z*): [M+H]⁺ Calcd for C₁₂H₁₈N, 176.14338; found, 176.14352.

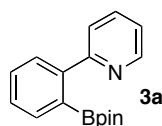
Preparation of Methyl 4-(1,3-Dimethylimidazolidin-2-yl)benzoate (1t). Methyl 4-(1,3-dimethylimidazolidin-2-yl)benzoate (**1t**) was prepared by the protection of methyl 4-formylbenzoate (2.5 g. 15.0 mmol) with *N,N'*-dimethylethylenediamine (1.5 g. 16.5 mmol) according to the reported procedure.²² The obtained crude material was purified by kugelrohr distillation to give pure **1t** in 74% yield.



Pale yellow solid. **M.p.** 35.9–36.3 °C. **¹H NMR** (CDCl₃) δ 2.18 (s, 6H), 2.56–2.62 (m, 2H), 3.33 (s, 1H), 3.39–3.45 (m, 2H), 3.92 (s, 3H), 7.53 (d, *J* = 8.1 Hz, 2H), 8.04 (d, *J* = 8.1 Hz, 2H). **¹³C NMR** (CDCl₃) δ 39.33, 51.97, 53.32, 91.81, 128.92, 129.65, 130.40, 145.42, 167.14. **Anal.** Calcd for C₁₃H₁₈N₂O₂: C, 66.64%; H, 7.74%; N, 11.96%. Found: C, 66.71%; H, 7.91%; N, 11.81%.

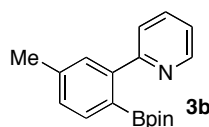
Characterization of Borylation Products

2-[2-(4,4,5,5-Tetramethyl-1,3,2-dioxaborolan-2-yl)phenyl]pyridine (3a)



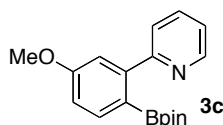
White solid. **M.p.** 136.3–138.3 °C. **¹H NMR** (CDCl₃) δ 1.43 (s, 12H), 7.27–7.44 (m, 3H), 7.66 (d, *J* = 7.8 Hz, 1H), 7.73 (d, *J* = 6.9 Hz, 1H), 7.81 (d, *J* = 8.1 Hz, 1H), 7.97 (td, *J* = 7.8, 1.5 Hz, 1H), 8.68 (d, *J* = 5.4 Hz, 1H). **¹³C NMR** (CDCl₃) δ 26.87, 80.12, 117.42, 121.20, 122.73, 127.84, 131.45, 131.51, 137.13, 141.93, 142.12, 156.59. A signal for the carbon directly attached to the boron atom was not observed. **HRMS–ESI** (*m/z*): [M+Na]⁺ Calcd for C₁₇H₂₀N¹⁰BO₂Na, 303.15156; found, 303.15125.

2-[5-Methyl-2-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)phenyl]pyridine (3b)



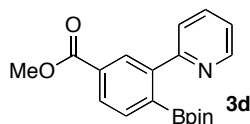
White solid. **M.p.** 147.7–149.1 °C. **¹H NMR** (CDCl₃) δ 1.41 (s, 12H), 2.37 (s, 3H), 6.87 (d, *J* = 7.2 Hz, 1H), 7.34 (d, *J* = 5.4 Hz, 1H), 7.46 (s, 1H), 7.66 (d, *J* = 7.5 Hz, 1H), 7.77 (d, *J* = 7.5 Hz, 1H), 7.94 (td, *J* = 7.5, 1.5 Hz, 1H), 8.65 (d, *J* = 5.4 Hz, 1H). **¹³C NMR** (CDCl₃) δ 21.23, 26.78, 80.04, 117.30, 121.82, 122.55, 131.32, 132.47, 137.38, 137.46, 141.75, 143.24, 156.75. A signal for the carbon directly attached to the boron atom was not observed. **HRMS–ESI** (*m/z*): [M+H]⁺ Calcd for C₁₈H₂₃N¹⁰BO₂, 295.18527; found, 295.18495.

2-[5-Methoxy-2-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)phenyl]pyridine (3c)



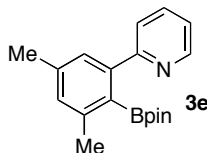
White solid. **M.p.** 130.6–131.6 °C. **¹H NMR** (CDCl₃) δ 1.41 (s, 12H), 3.85 (s, 3H), 6.99 (dd *J* = 8.1, 2.1 Hz, 1H), 7.17 (d, *J* = 2.1 Hz, 1H), 7.36 (tm, *J* = 5.7 Hz, 1H), 7.62 (d, *J* = 8.1 Hz, 1H), 7.75 (d, *J* = 7.8 Hz, 1H), 7.95 (td, *J* = 7.8, 1.5 Hz, 1H), 8.66 (d, *J* = 5.7 Hz, 1H). **¹³C NMR** (CDCl₃) δ 26.68, 55.22, 80.04, 106.83, 117.15, 117.45, 122.78, 132.46, 138.69, 141.75, 143.23, 156.13, 159.97. A signal for the carbon directly attached to the boron atom was not observed. **HRMS–ESI** (*m/z*): [M+Na]⁺ Calcd for C₁₈H₂₂N¹⁰BO₃Na, 333.16213; found, 333.16223.

2-[5-Methoxycarbonyl-2-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)phenyl]pyridine (3d)



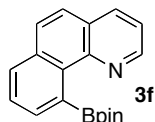
White solid. **M.p.** 173.8–174.2 °C. **¹H NMR** (CDCl₃) δ 1.44 (s, 12H), 3.95 (s, 3H), 7.45 (tm *J* = 5.7 Hz, 1H), 7.81 (d, *J* = 7.5 Hz, 1H), 7.93 (d, *J* = 7.9 Hz, 1H), 8.01–8.10 (m, 2H), 8.35 (s, 1H), 8.70 (d, *J* = 5.7 Hz, 1H). **¹³C NMR** (CDCl₃) δ 26.90, 52.10, 80.40, 117.90, 122.31, 123.44, 129.90, 131.55, 132.40, 137.51, 142.38, 143.33, 155.58, 167.27. A signal for the carbon directly attached to the boron atom was not observed. **HRMS–ESI** (*m/z*): [M+Na]⁺ Calcd for C₁₉H₂₂N¹⁰BO₄Na, 361.15704; found, 361.15724.

2-[3,5-Dimethyl-2-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)phenyl]pyridine (3e)



White solid. **M.p.** 147.1–147.9 °C. **¹H NMR** (CDCl₃) δ 1.46 (s, 12H), 2.32 (s, 3H), 2.53 (s, 3H), 7.00 (s, 1H), 7.28–7.31 (m, 2H), 7.75 (d, *J* = 7.8 Hz, 1H), 7.86 (td, *J* = 7.8, 1.2 Hz, 1H), 8.60 (d, *J* = 5.4 Hz, 1H). **¹³C NMR** (CDCl₃) δ 20.93, 21.90, 28.16, 81.04, 117.69, 120.01, 122.26, 133.43, 137.69, 138.57, 140.40, 141.88, 143.25, 156.50. A signal for the carbon directly attached to the boron atom was not observed. **HRMS–ESI** (*m/z*): [M+H]⁺ Calcd for C₁₉H₂₅N¹⁰BO₂, 309.20092; found, 309.20054.

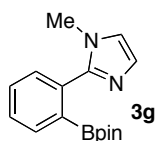
10-(4,4,5,5-Tetramethyl-1,3,2-dioxaborolan-2-yl)benzo[*h*]quinoline (3f)



White solid. **M.p.** 171.2–172.8 °C. **¹H NMR** (CDCl₃) δ 1.55 (s, 12H), 7.52 (dd, *J* = 7.8, 4.5 Hz, 1H), 7.65–7.72 (m, 2H), 7.77 (dd, *J* = 6.9, 1.5 Hz, 1H), 7.81 (d, *J* = 8.7 Hz, 1H), 7.88 (dd, *J* = 7.5, 1.5 Hz, 1H), 8.19 (dd, *J* = 7.8, 1.5 Hz, 1H), 8.90 (dd, *J* = 4.5, 1.5 Hz, 1H). **¹³C NMR** (CDCl₃) δ 25.10, 83.10, 121.66, 124.42, 126.12, 127.80, 128.02, 128.12, 130.69, 132.53, 134.26, 135.95,

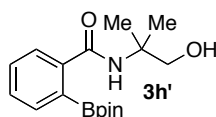
146.29, 146.57. A signal for the carbon directly attached to the boron atom was not observed. **HRMS–ESI** (m/z): $[M]^+$ Calcd for $C_{19}H_{20}N^{10}BO_2$, 304.16234; found, 304.16165.

1-Methyl-2-[2-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)phenyl]-1H-imidazole (3g)



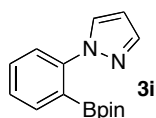
White solid. **M.p.** 150.0–150.3 °C. **1H NMR** ($CDCl_3$) δ 1.33 (s, 12H), 3.85 (s, 3H), 6.84 (d, J = 1.5 Hz, 1H), 7.13 (d, J = 1.5 Hz, 1H), 7.28 (m, 1H), 7.36 (td, J = 7.2, 1.2 Hz, 1H), 7.43 (d, J = 7.2 Hz, 1H), 7.71 (d, J = 6.9 Hz, 1H). **^{13}C NMR** ($CDCl_3$) δ 25.53, 34.41, 80.34, 121.17, 121.20, 122.43, 123.00, 127.63, 129.87, 131.28, 132.14. A signal for the carbon directly attached to the boron atom was not observed. **HRMS–FD** (m/z): $[M]^+$ Calcd for $C_{16}H_{21}N_2^{11}BO_2$, 284.16961; found, 284.16952.

N-(1-Hydroxy-2-methylpropan-2-yl)-2-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)benzamide (3h')



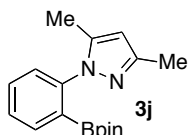
White solid. **M.p.** 152.1–153.1 °C. **1H NMR** ($CDCl_3$) δ 1.38 (s, 12H), 1.40 (s, 6H), 3.78 (d, J = 6.9 Hz, 2H), 4.34 (bt, J = 6.9 Hz, 1H), 6.02 (bs, 1H), 7.37–7.48 (m, 3H), 7.69 (d, J = 6.6 Hz, 1H). **^{13}C NMR** ($CDCl_3$) δ 24.35, 24.69, 56.80, 68.44, 83.12, 123.88, 129.17, 131.02, 133.18, 138.65, 170.79. A signal for the carbon directly attached to the boron atom was not observed. **HRMS–ESI** (m/z): $[M+Na]^+$ Calcd for $C_{17}H_{26}N^{10}BO_4Na$, 341.18834; found, 341.18815

1-[2-(4,4,5,5-Tetramethyl-1,3,2-dioxaborolan-2-yl)phenyl]-1H-pyrazole (3i)



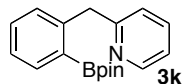
White solid. **M.p.** 46.3–47.3 °C. **1H NMR** ($CDCl_3$) δ 1.36 (s, 12H), 6.45 (t, J = 2.1 Hz, 1H), 7.30 (dd, J = 6.9, 1.5 Hz, 1H), 7.37–7.44 (m, 2H), 7.59 (d, J = 6.3 Hz, 1H), 7.66 (d, J = 2.1 Hz, 1H), 7.87 (d, J = 2.1 Hz, 1H). **^{13}C NMR** ($CDCl_3$) δ 24.67, 83.32, 107.65, 117.53, 126.33, 126.37, 129.93, 133.79, 138.98, 142.67. A signal for the carbon directly attached to the boron atom was not observed. **HRMS–ESI** (m/z): $[M+Na]^+$ Calcd for $C_{15}H_{19}N_2^{10}BO_2Na$, 292.14681; found, 292.14664.

3,5-Dimethyl-1-[2-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)phenyl]-1H-pyrazole (3j)



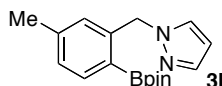
Colorless oil. $^1\text{H NMR}$ (CDCl_3) δ 1.22 (s, 12H), 2.19 (s, 3H), 2.27 (s, 3H), 5.93 (s, 1H), 7.27 (m, 1H), 7.37 (td, $J = 7.5, 1.2$ Hz, 1H), 7.47 (td, $J = 7.8, 1.5$ Hz, 1H), 7.74 (dd, $J = 7.5, 1.2$ Hz, 1H). $^{13}\text{C NMR}$ (CDCl_3) δ 11.75, 13.28, 24.53, 83.37, 105.61, 125.49, 127.32, 130.50, 134.90, 139.89, 143.31, 147.70. A signal for the carbon directly attached to the boron atom was not observed. **HRMS–ESI** (m/z): $[\text{M}+\text{Na}]^+$ Calcd for $\text{C}_{17}\text{H}_{23}\text{N}_2^{10}\text{BO}_2\text{Na}$, 320.17811; found, 320.17809.

2-[2-(4,4,5,5-Tetramethyl-1,3,2-dioxaborolan-2-yl)benzyl]pyridine (3k)



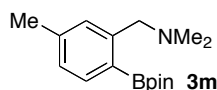
White solid. **M.p.** 125.7–127.0 °C. $^1\text{H NMR}$ (CDCl_3) δ 1.25 (s, 12H), 4.34 (s, 2H), 7.12–7.29 (m, 3H), 7.39 (t, $J = 6.6$ Hz, 1H), 7.48 (d, $J = 7.5$ Hz, 1H), 7.70 (d, $J = 6.6$ Hz, 1H), 7.83 (td, $J = 7.5, 1.5$ Hz, 1H), 9.11 (d, $J = 4.8$ Hz, 1H). $^{13}\text{C NMR}$ (CDCl_3) δ 25.89, 39.68, 80.76, 122.02, 125.20, 126.11, 126.54, 126.82, 130.93, 138.99, 139.72, 143.70, 158.76. A signal for the carbon directly attached to the boron atom was not observed. **HRMS–ESI** (m/z): $[\text{M}+\text{H}]^+$ Calcd for $\text{C}_{18}\text{H}_{23}\text{N}^{10}\text{BO}_2$, 295.18527; found, 295.18491.

1-[5-Methyl-2-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)benzyl]-1H-pyrazole (3l)



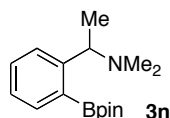
Colorless oil. $^1\text{H NMR}$ (CDCl_3) δ 1.33 (s, 12H), 2.29 (s, 3H), 5.63 (s, 2H), 6.24 (t, $J = 1.8$ Hz, 1H), 6.88 (s, 1H), 7.10 (d, $J = 7.8$ Hz, 1H), 7.45 (d, $J = 1.8$ Hz, 1H), 7.55 (d, $J = 1.8$ Hz, 1H), 7.76 (d, $J = 7.8$ Hz, 1H). $^{13}\text{C NMR}$ (CDCl_3) δ 21.43, 24.69, 54.89, 83.59, 105.29, 127.92, 129.11, 129.55, 136.46, 139.06, 141.79, 143.04. A signal for the carbon directly attached to the boron atom was not observed. **HRMS–ESI** (m/z): $[\text{M}+\text{H}]^+$ Calcd for $\text{C}_{17}\text{H}_{24}\text{N}_2^{11}\text{BO}_2$, 299.19254; found, 299.19257.

***N,N*-Dimethyl-1-[5-methyl-2-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)phenyl]methanamine (3m)**



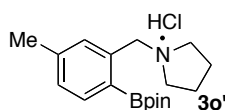
White solid. **M.p.** 86.6–88.8 °C. $^1\text{H NMR}$ (CDCl_3) δ 1.30 (s, 12H), 2.29 (s, 3H), 2.53 (s, 6H), 3.80 (s, 2H), 6.83 (s, 1H), 7.03 (d, $J = 7.5$ Hz, 1H), 7.42 (d, $J = 7.5$ Hz, 1H). $^{13}\text{C NMR}$ (CDCl_3) δ 21.19, 26.26, 45.51, 65.11, 80.34, 123.92, 128.09, 131.51, 137.12, 140.21. A signal for the carbon directly attached to the boron atom was not observed. **HRMS–ESI** (m/z): $[\text{M}+\text{H}]^+$ Calcd for $\text{C}_{16}\text{H}_{27}\text{N}^{10}\text{BO}_2$, 275.21657; found, 275.21715.

***N,N*-Dimethyl-1-[2-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)phenyl]ethanamine (3n)**



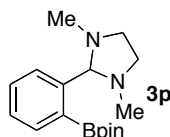
Colorless oil. **¹H NMR** (CDCl₃) δ 1.29 (s, 6H), 1.32 (s, 6H), 1.44 (d, *J* = 6.9 Hz, 3H), 2.43 (s, 6H), 4.14 (q, *J* = 6.9 Hz, 1H), 7.01 (m, 1H), 7.16–7.25 (m, 2H), 7.54 (m, 1H). **¹³C NMR** (CDCl₃) δ 11.87, 26.32, 27.17, 40.44, 65.58, 79.97, 121.64, 127.36 (x2C), 130.63, 144.10. A signal for the carbon directly attached to the boron atom was not observed. **HRMS–ESI** (*m/z*): [M+Na]⁺ Calcd for C₁₆H₂₆N¹⁰BO₂Na, 297.19851; found, 297.19907.

1-[5-Methyl-2-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)benzyl]pyrrolidine hydrochloride salt (3o')



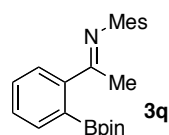
White solid. **M.p.** 197.3–200.3 °C. **¹H NMR** (CDCl₃) δ 1.36 (s, 12H), 1.98–2.03 (m, 2H), 2.20–2.23 (m, 2H), 2.42 (s, 3H), 2.86–2.95 (m, 2H), 3.57 (sext, *J* = 5.1 Hz, 2H), 4.59 (d, *J* = 6.0 Hz, 2H), 7.22 (d, *J* = 7.8 Hz, 1H), 7.79 (d, *J* = 7.8 Hz, 1H), 7.90 (s, 1H), 12.28 (bs, 1H). **¹³C NMR** (CDCl₃) δ 21.38, 23.01, 24.79, 52.34, 55.70, 84.09, 129.80, 132.93, 136.03, 136.90, 143.14. A signal for the carbon directly attached to the boron atom was not observed. **HRMS–ESI** (*m/z*): [M–Cl]⁺ Calcd for C₁₈H₂₉N¹⁰BO₂, 301.23222; found, 301.23240.

1,3-Dimethyl-2-[2-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)phenyl]imidazolidine (3p)



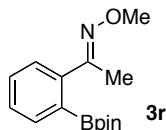
White solid. **M.p.** 107.6–108.3 °C. **¹H NMR** (CDCl₃) δ 1.29 (s, 12H), 2.46 (s, 6H), 2.72 (q, *J* = 3.6 Hz, 2H), 3.77 (q, *J* = 3.6 Hz, 2H), 3.87 (s, 1H), 7.09 (d, *J* = 7.5 Hz, 1H), 7.20 (td, *J* = 7.5, 1.5 Hz, 1H), 7.31 (td, *J* = 7.5, 1.2 Hz, 1H), 7.56 (d, *J* = 7.5 Hz, 1H). **¹³C NMR** (CDCl₃) δ 25.90, 41.58, 53.67, 80.26, 95.18, 124.04, 127.06, 128.92, 131.93, 139.05. A signal for the carbon directly attached to the boron atom was not observed. **HRMS–ESI** (*m/z*): [M+H]⁺ Calcd for C₁₇H₂₈N₂¹⁰BO₂, 302.22747; found, 302.22715.

(E)-2,4,6-Trimethyl-N-{1-[2-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)phenyl]ethylidene}aniline (3q)



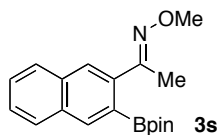
White solid. M.p. 201.2–202.4 °C. $^1\text{H NMR}$ (CDCl_3) δ 1.02 (s, 12H), 2.19 (s, 9H), 2.32 (s, 3H), 6.92 (s, 2H), 7.30 (d, $J = 7.2$ Hz, 1H), 7.49–7.56 (m, 2H), 7.68 (d, $J = 7.2$ Hz, 1H). $^{13}\text{C NMR}$ (CDCl_3) δ 14.21, 18.21, 20.79, 25.74, 79.92, 123.84, 127.45, 128.86, 130.44, 132.42, 133.37, 136.52, 137.96, 138.43, 177.71. A signal for the carbon directly attached to the boron atom was not observed. **HRMS–ESI** (m/z): $[\text{M}+\text{Na}]^+$ calcd for $\text{C}_{23}\text{H}_{30}^{11}\text{BNO}_2\text{Na}$, 385.22981; found, 385.22946.

(*E*)-1-[2-(4,4,5,5-Tetramethyl-1,3,2-dioxaborolan-2-yl)phenyl]ethanone *O*-Methyloxime (3r)



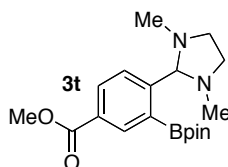
Colorless oil. $^1\text{H NMR}$ (CDCl_3) δ 1.34 (s, 12H), 2.22 (s, 3H), 3.97 (s, 3H), 7.31–7.44 (m, 3H), 7.69 (dm, $J = 7.5$ Hz, 1H). $^{13}\text{C NMR}$ (CDCl_3) δ 16.16, 24.69, 61.57, 83.79, 127.33, 127.90, 130.28, 134.79, 143.07, 158.54. A signal for the carbon directly attached to the boron atom was not observed. **HRMS–ESI** (m/z): $[\text{M}+\text{Na}]^+$ Calcd for $\text{C}_{15}\text{H}_{22}\text{N}^{10}\text{BO}_3\text{Na}$, 297.16213; found, 297.16162.

(*E*)-1-[3-(4,4,5,5-Tetramethyl-1,3,2-dioxaborolan-2-yl)naphthalen-2-yl]ethanone *O*-Methyloxime (3s)



White solid. M.p. 140.6–142.1 °C. $^1\text{H NMR}$ (CDCl_3) δ 1.39 (s, 12H), 2.30 (s, 3H), 4.01 (s, 3H), 7.47–7.51 (m, 2H), 7.80–7.87 (m, 3H), 8.23 (s, 1H). $^{13}\text{C NMR}$ (CDCl_3) δ 16.14, 24.71, 61.58, 83.81, 126.42, 126.60, 127.19, 128.01, 128.17, 132.61, 134.03, 136.25, 139.06, 158.36. A signal for the carbon directly attached to the boron atom was not observed. **HRMS–ESI** (m/z): $[\text{M}+\text{Na}]^+$ Calcd for $\text{C}_{19}\text{H}_{24}^{11}\text{BNO}_3\text{Na}$, 347.17778; found, 347.17812.

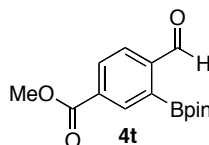
Methyl 4-(1,3-Dimethylimidazolidin-2-yl)-3-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)benzoate (3t)



White solid. **M.p.** 98.7–99.8 °C. $^1\text{H NMR}$ (CDCl_3) δ 1.31 (s, 12H), 2.47 (s, 6H), 2.76 (q, $J = 4.2$ Hz, 2H), 3.78 (q, $J = 4.2$ Hz, 2H), 3.89 (s, 3H), 3.91 (s, 1H), 7.17 (d, $J = 7.8$ Hz, 1H), 7.92 (d, $J = 7.8$ Hz, 1H), 8.22 (s, 1H). $^{13}\text{C NMR}$ (CDCl_3) δ 25.90, 41.61, 51.74, 53.76, 80.49, 94.61, 124.06, 128.75, 130.56, 133.45, 144.08, 167.67. A signal for the carbon directly attached to the boron atom

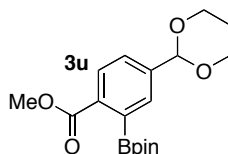
was not observed. **HRMS–ESI** (m/z): $[M+Na]^+$ Calcd for $C_{19}H_{29}N_2^{10}BO_4Na$, 382.21489; found, 382.21475.

Methyl 4-Formyl-3-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)benzoate (4t)



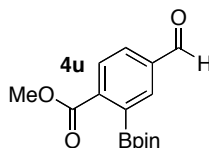
White solid. **M.p.** 108.6–109.5 °C. **1H NMR** ($CDCl_3$) δ 1.41 (s, 12H), 3.96 (s, 3H), 8.02 (d, J = 8.1 Hz, 1H), 8.21 (dd, J = 8.1, 1.5 Hz, 1H), 8.53 (d, J = 1.5 Hz, 1H), 10.63 (s, 1H). **^{13}C NMR** ($CDCl_3$) δ 24.77, 54.43, 84.74, 127.78, 131.86, 133.65, 136.87, 144.22, 166.41, 194.35. A signal for the carbon directly attached to the boron atom was not observed. **HRMS–FD** (m/z): $[M]^+$ Calcd for $C_{15}H_{19}^{10}BO_5$, 289.13619; found, 289.13603.

Methyl 4-(1,3-Dioxan-2-yl)-2-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)benzoate (3u)



Colorless wax. **1H NMR** ($CDCl_3$) δ 1.46 (m, 13H), 2.22 (m, 1H), 3.90 (s, 3H), 3.99 (td, J = 12.0, 2.4 Hz, 2H), 4.28 (dd, J = 10.2, 5.1 Hz, 2H), 5.52 (s, 1H), 7.55 (dd, J = 7.8, 1.8 Hz, 1H), 7.60 (s, 1H), 7.94 (d, J = 7.8 Hz, 1H). **^{13}C NMR** ($CDCl_3$) δ 24.79, 25.59, 52.24, 67.35, 84.01, 100.93, 126.68, 128.87, 130.06, 133.83, 142.00, 168.41. A signal for the carbon directly attached to the boron atom was not observed. **HRMS–ESI** (m/z): $[M+Na]^+$ Calcd for $C_{18}H_{25}^{10}BO_6Na$, 370.16727; found, 370.16710.

Methyl 4-Formyl-2-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)benzoate (4u)



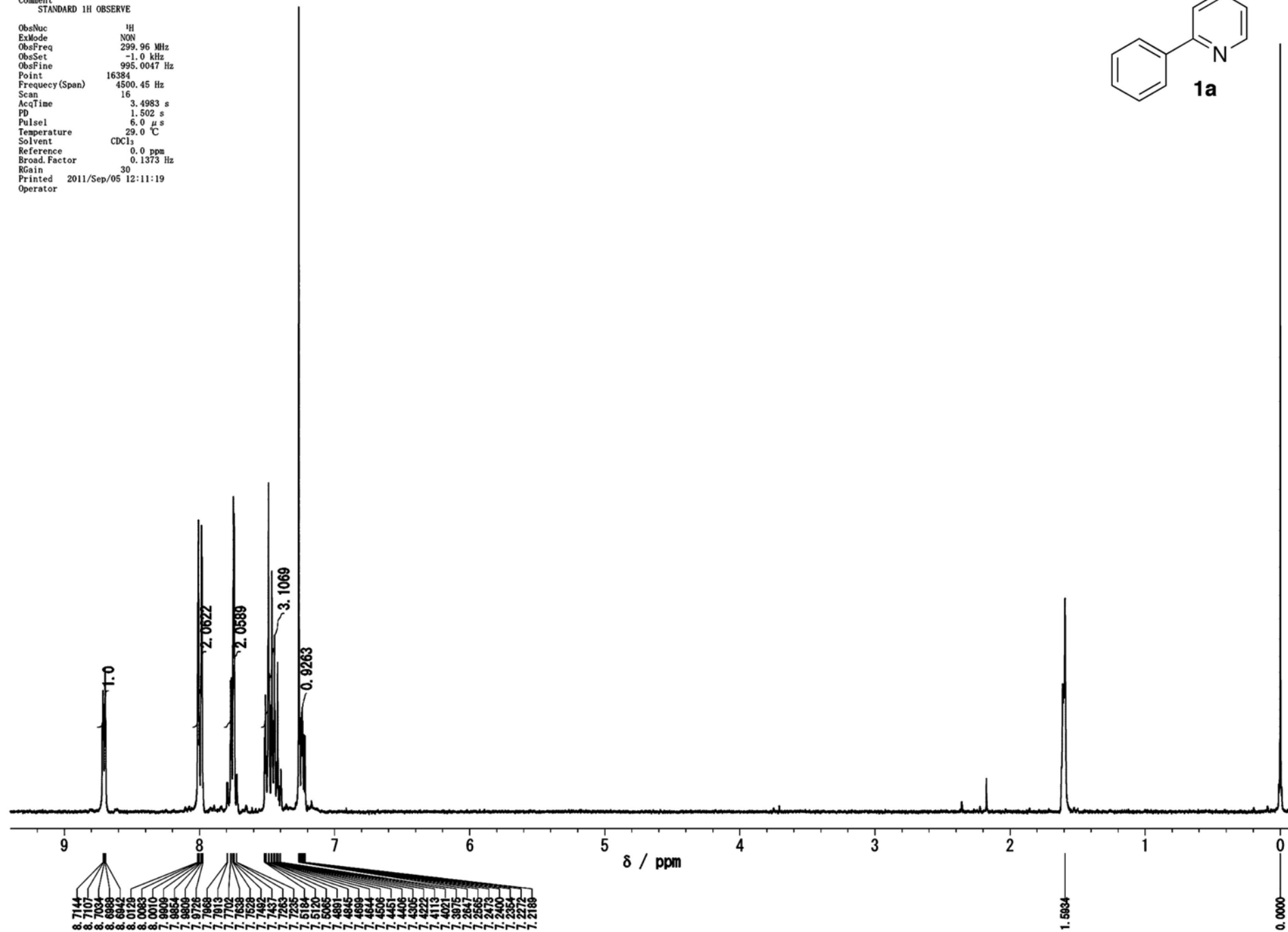
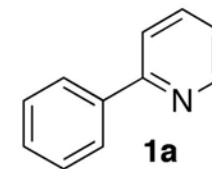
White solid. **M.p.** 102.6–105.5 °C. **1H NMR** ($CDCl_3$) δ 1.44 (s, 12H), 3.96 (s, 3H), 7.94 (dd, J = 8.1, 1.5 Hz, 1H), 8.02 (d, J = 1.5 Hz, 1H), 8.09 (d, J = 8.1 Hz, 1H), 10.10 (s, 1H). **^{13}C NMR** ($CDCl_3$) δ 24.76, 52.72, 84.50, 129.36, 130.07, 133.87, 137.94, 138.63, 167.75, 192.06. A signal for the carbon directly attached to the boron atom was not observed. **HRMS–ESI** (m/z): $[M+H]^+$ Calcd for $C_{15}H_{20}^{10}BO_5$, 290.14346; found, 290.14368.

References

- (1) Uson, R.; Oro, L. A.; Cabeza, J. A. *Inorg. Synth.* **1985**, *23*, 126.
- (2) (a) Hamasaka, G.; Ochida, A.; Hara, K.; Sawamura, M. *Angew. Chem., Int. Ed.* **2007**, *46*, 5381–5383. (b) Hamasaka, G.; Kawamorita, S.; Ochida, A.; Akiyama, R.; Hara, K.; Fukuoka, A.; Asakura, K.; Chun, W. J.; Ohmiya, H.; Sawamura, M. *Organometallics* **2008**, *27*, 6495–6506. (c) Kawamorita, S.; Ohmiya, H.; Hara, K.; Fukuoka, A.; Sawamura, M. *J. Am. Chem. Soc.* **2009**, *131*, 5058–5059.
- (3) Tsuji, H.; Inoue, T.; Kaneta, Y.; Sase, S.; Kawachi, A.; Tamao, K. *Organometallics* **2006**, *25*, 6142–6148.
- (4) Takuma, Y.; Imaki, N. *J. Organomet. Chem.* **1989**, *379*, 27–32.
- (5) Li, M.; Hua, R. *Tetrahedron Letters* **2009**, *50*, 1478–1481.
- (6) Campeau, L.-C.; Rouseaux, S.; Fagnou, K. *J. Am. Chem. Soc.* **2005**, *127*, 18020–18021.
- (7) Gosmini, C.; Bassene-Ernst, C.; Durandetti, M. *Tetrahedron* **2009**, *65*, 6141–6146.
- (8) Do, H.-Q.; Khan, R. M. K.; Dauguilis, O. *J. Am. Chem. Soc.* **2008**, *130*, 15185–15192.
- (9) Lewis, J. C.; Berman, A. M.; Bergman, R. G.; Ellman, J. A. *J. Am. Chem. Soc.* **2008**, *130*, 2493–2500.
- (10) Taillefer, M.; Xia, N.; Ouali, A. *Angew. Chem., Int. Ed.* **2007**, *46*, 934–936.
- (11) Antilla, J. C.; Maskin, J. M.; Barder, T. E.; Buchwald, S. L. *J. Org. Chem.* **2004**, *69*, 5578–5587.
- (12) Desai, L. V.; Stowers, K. J.; Sanford, M. S. *J. Am. Chem. Soc.* **2008**, *130*, 13285–13293.
- (13) Cai, G.; Fu, Y.; Li, Y.; Wan, X.; Shi, Z. *J. Am. Chem. Soc.* **2007**, *129*, 7666–7673.
- (14) Ebden, M. R.; Simpkins, N. S.; Fox, D. N. A. *Tetrahedron* **1998**, *54*, 12923–12952.
- (15) Lambert, J. B.; Wang, G.-T.; Huseland, D. E.; Takiff, L. C. *J. Org. Chem.* **1987**, *52*, 68–71.
- (16) Barluenga, J.; Fernández, M. A.; Aznar, F.; Valdés, C. *Chem. Eur. J.* **2004**, *10*, 494–507.
- (17) Chu, Y.; Shan, Z.; Liu, D.; Sun, N. *J. Org. Chem.* **2006**, *71*, 3998–4001.
- (18) Padwa, A.; Albrecht, F. *J. Org. Chem.* **1974**, *39*, 2361–2366.
- (19) Marconi, G.; Pertici, P.; Evangelisti, C.; Caporusso, A. M.; Vitulli, G.; Capannelli, G.; Hoang, M.; Turney, T. W. *J. Organomet. Chem.* **2004**, *689*, 639–646.
- (20) Liu, C.; Yang, W. *Chem. Commun.* **2009**, 6267–6269.
- (21) Sperotto, E.; van Klink, G.; de Vries, J. G.; van Koten, G. *Tetrahedron* **2010**, *66*, 3478–3484.
- (22) Chikashita, H.; Ishimoto, N.; Komazawa, S.; Itoh, K. *Heterocycles*, **1985**, *23*, 2509.

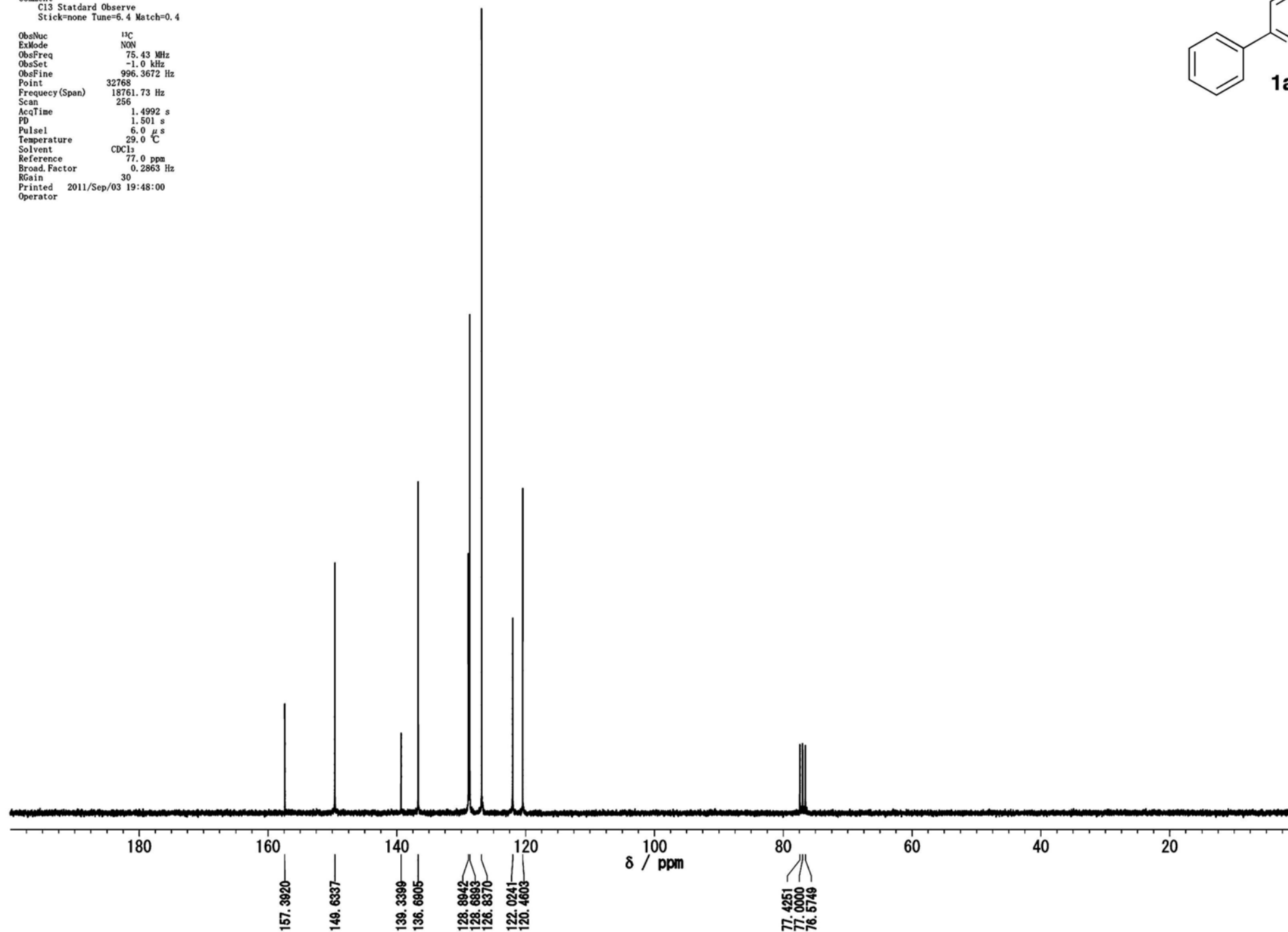
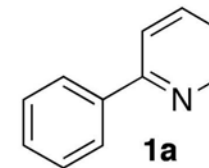
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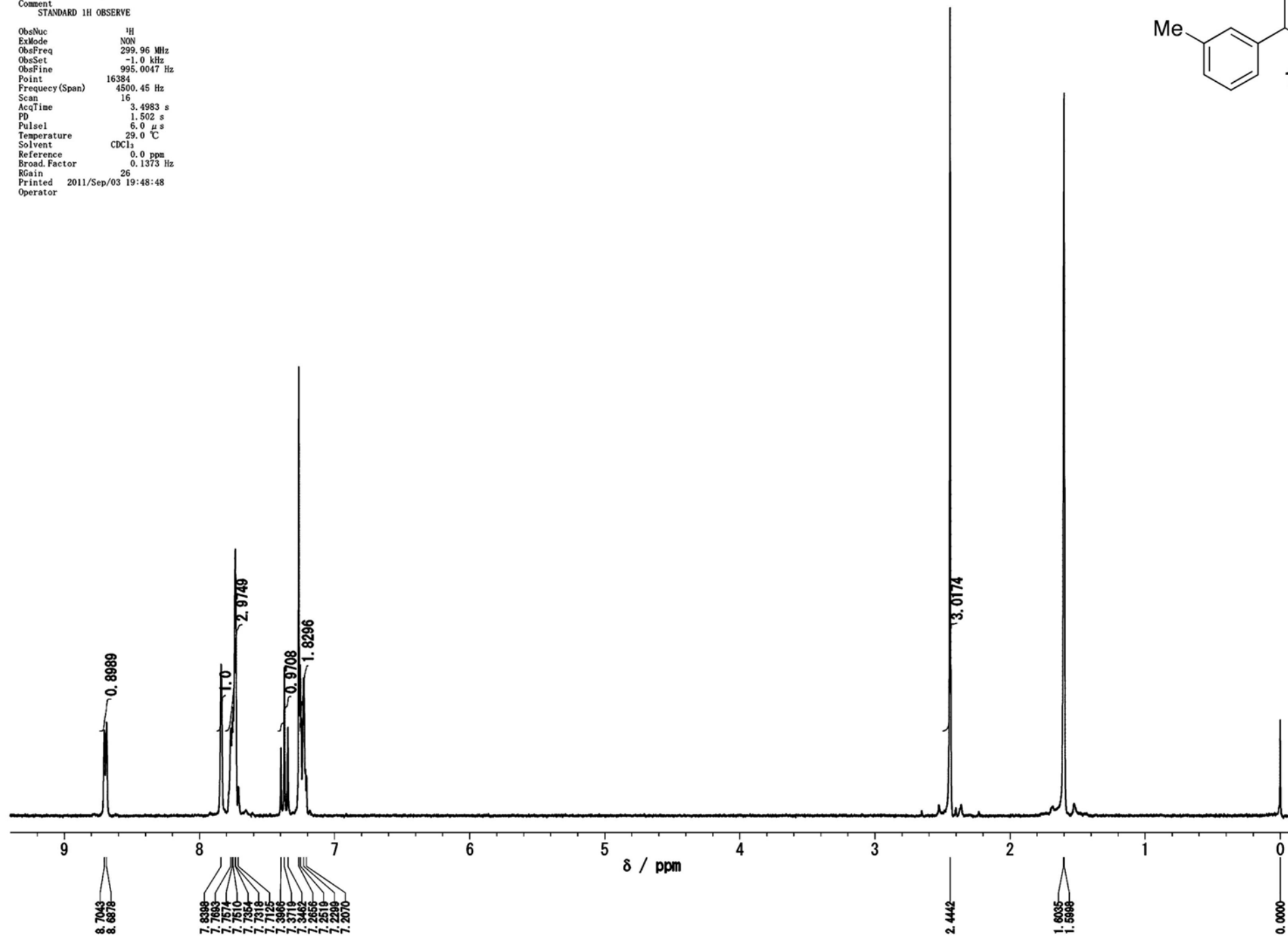
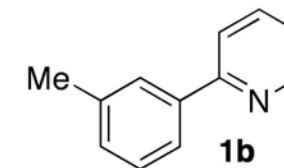
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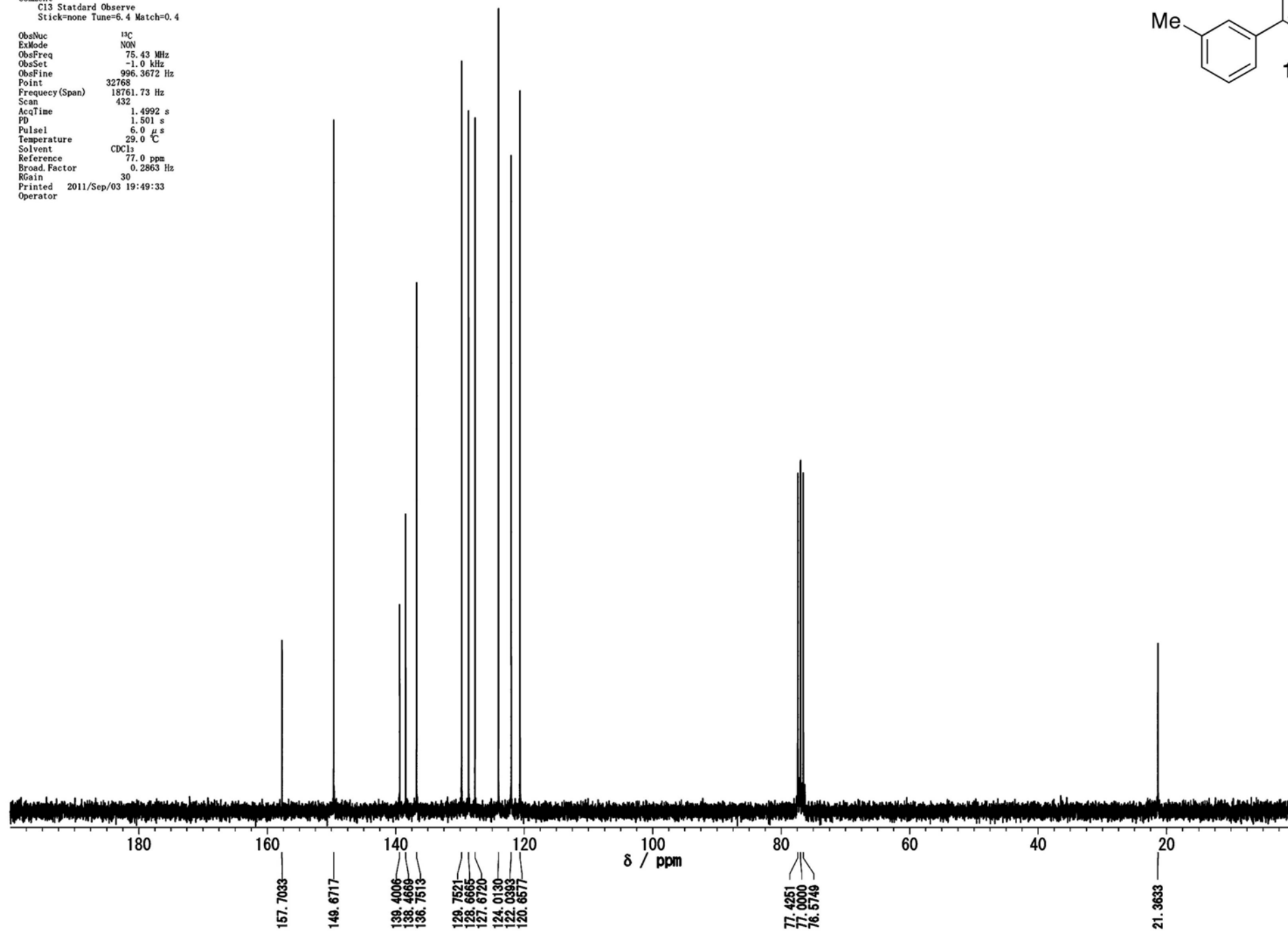
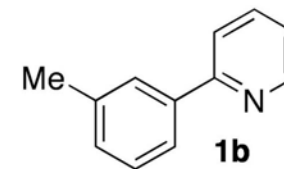
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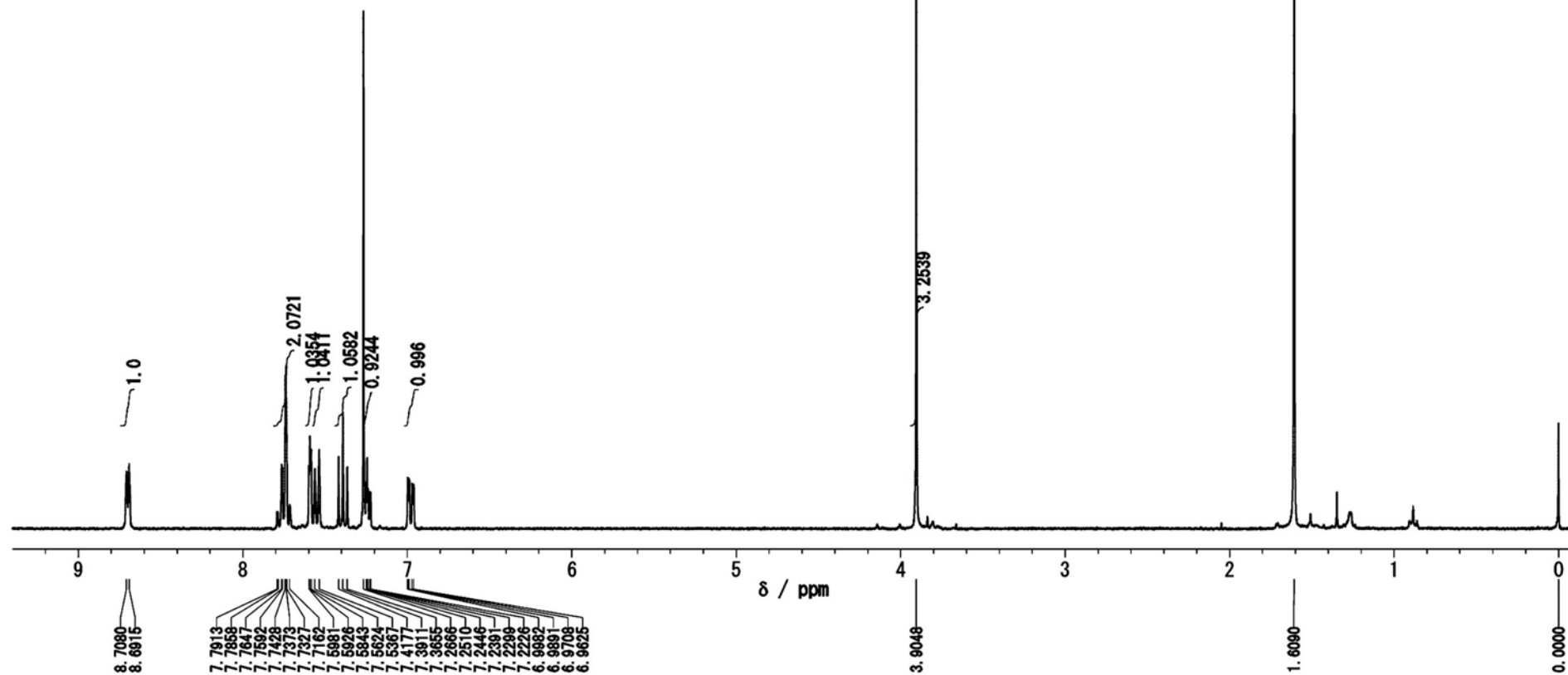
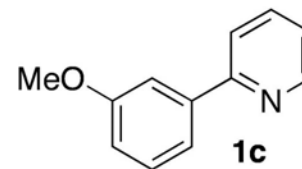


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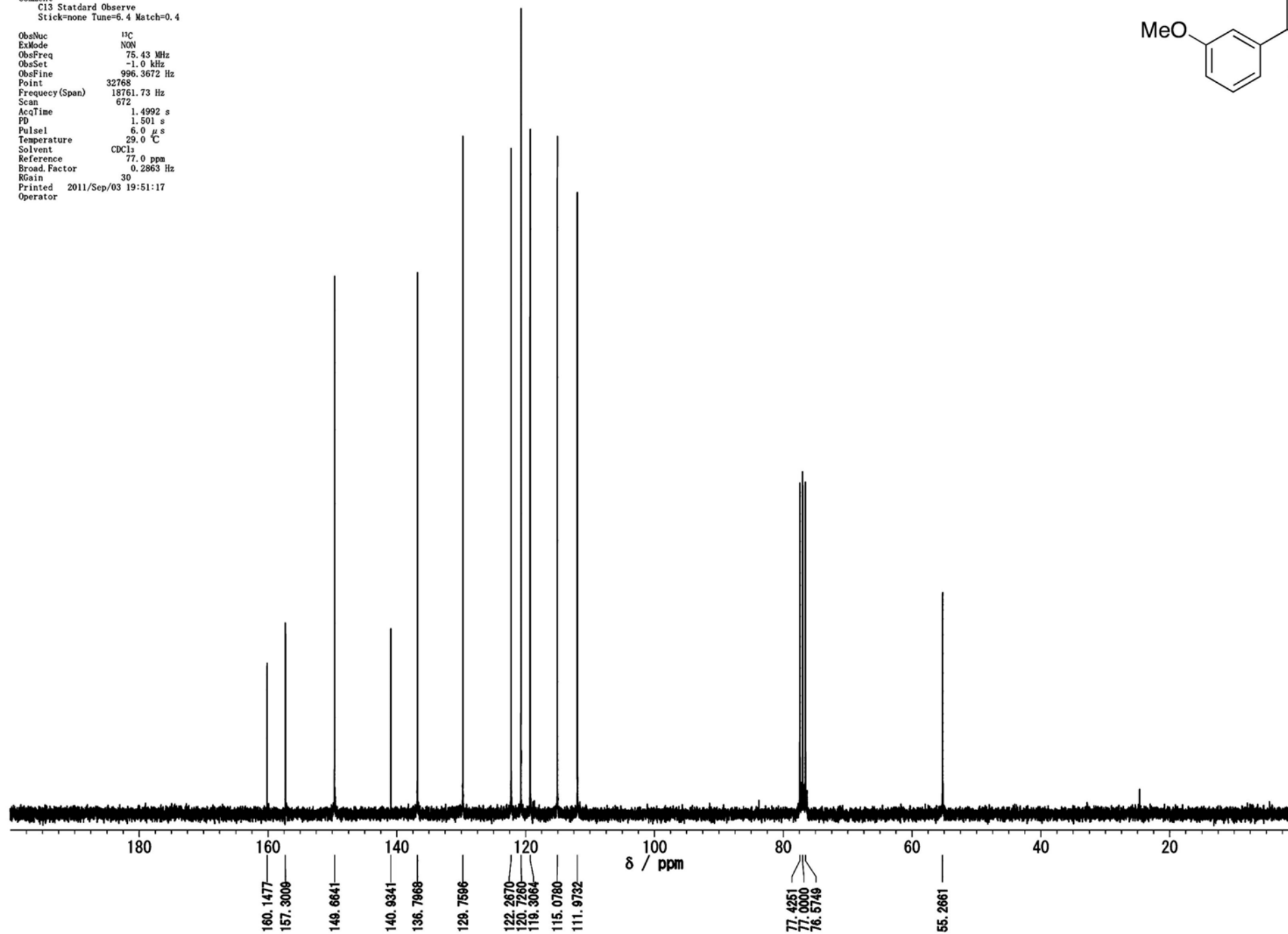
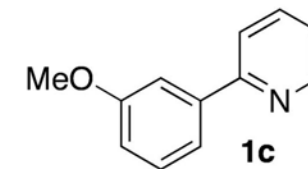


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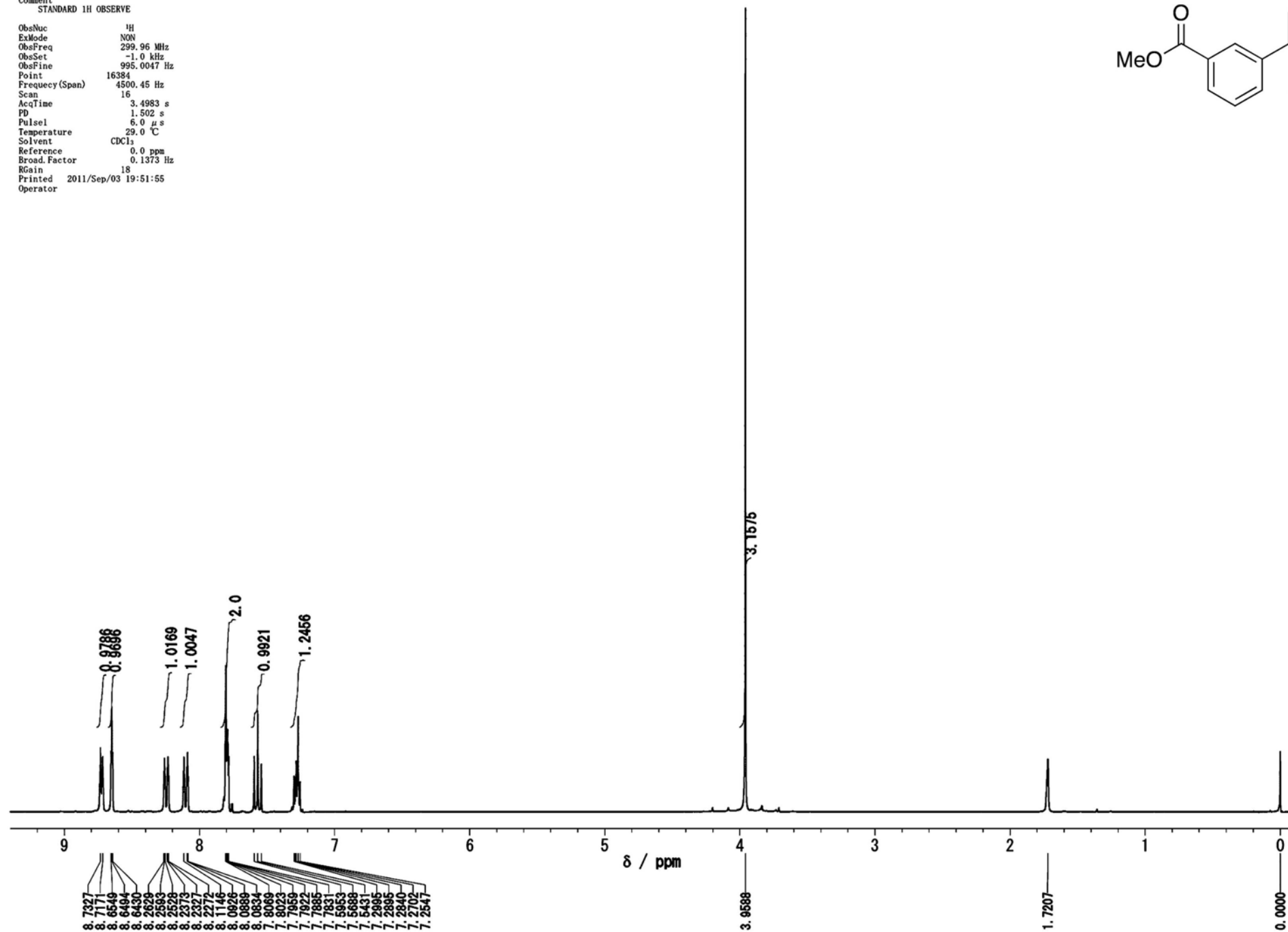
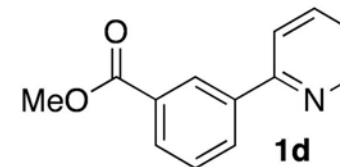
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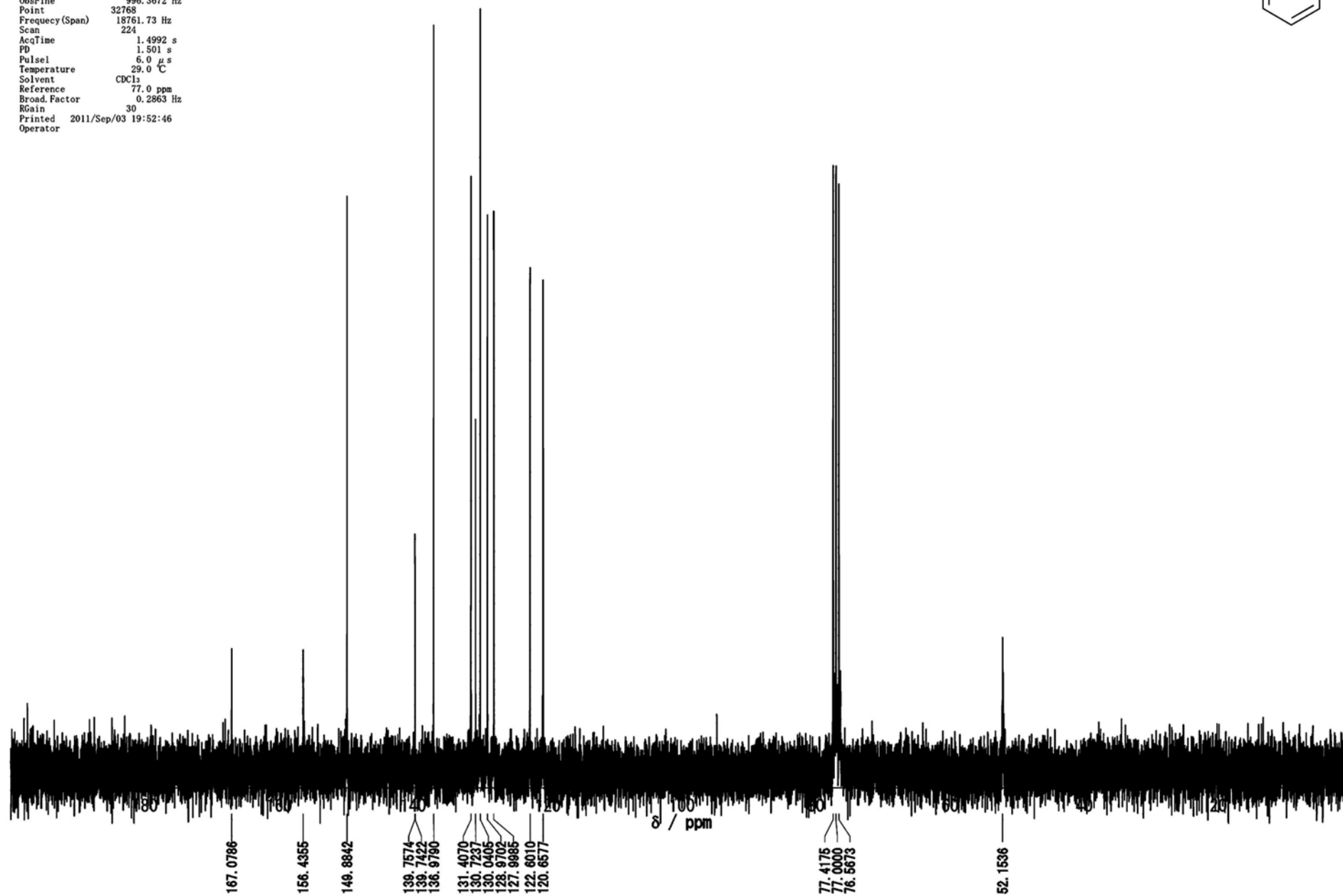
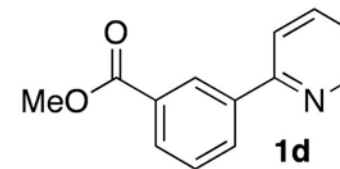
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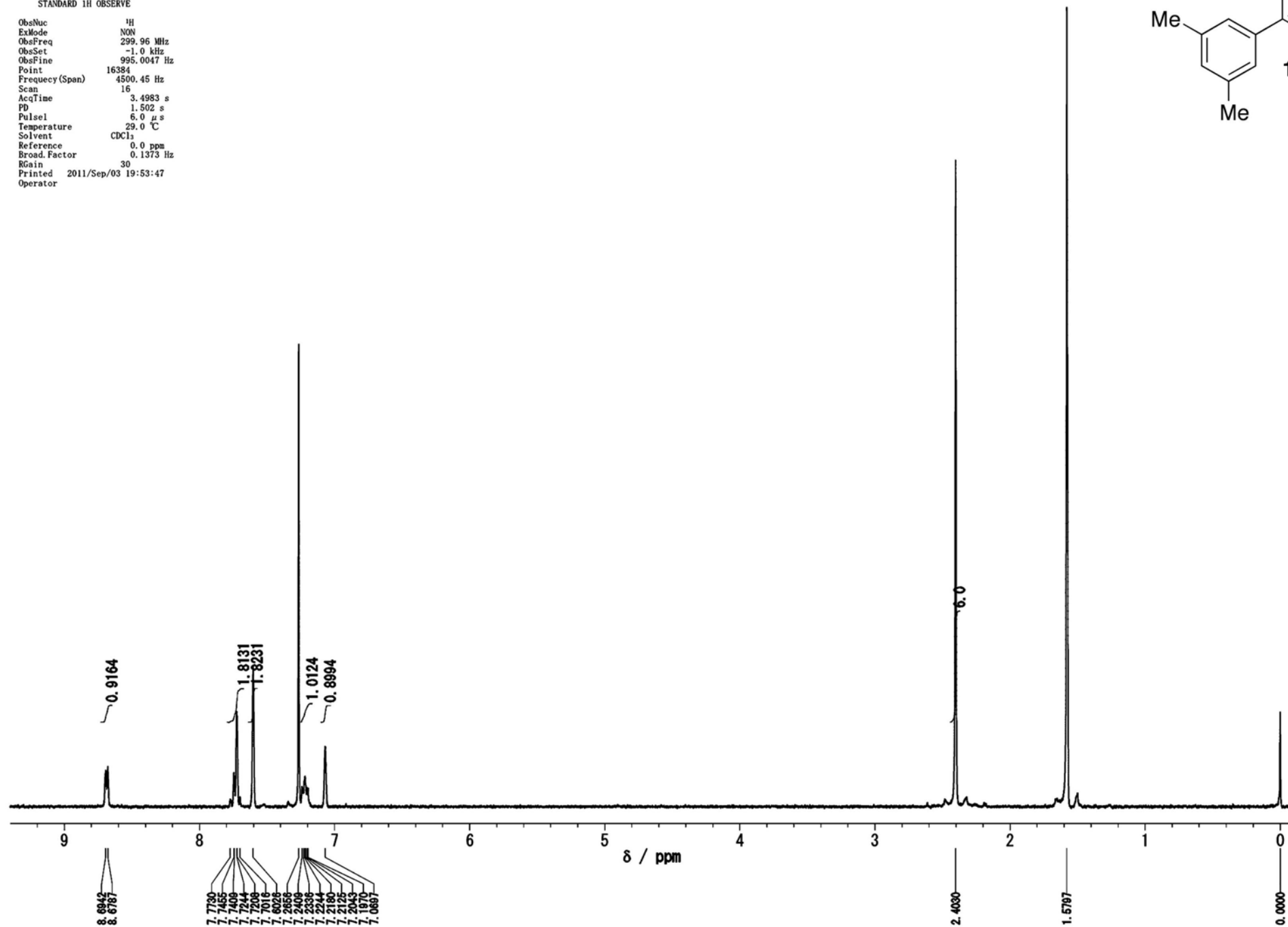
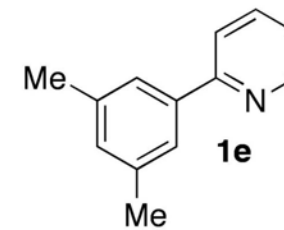


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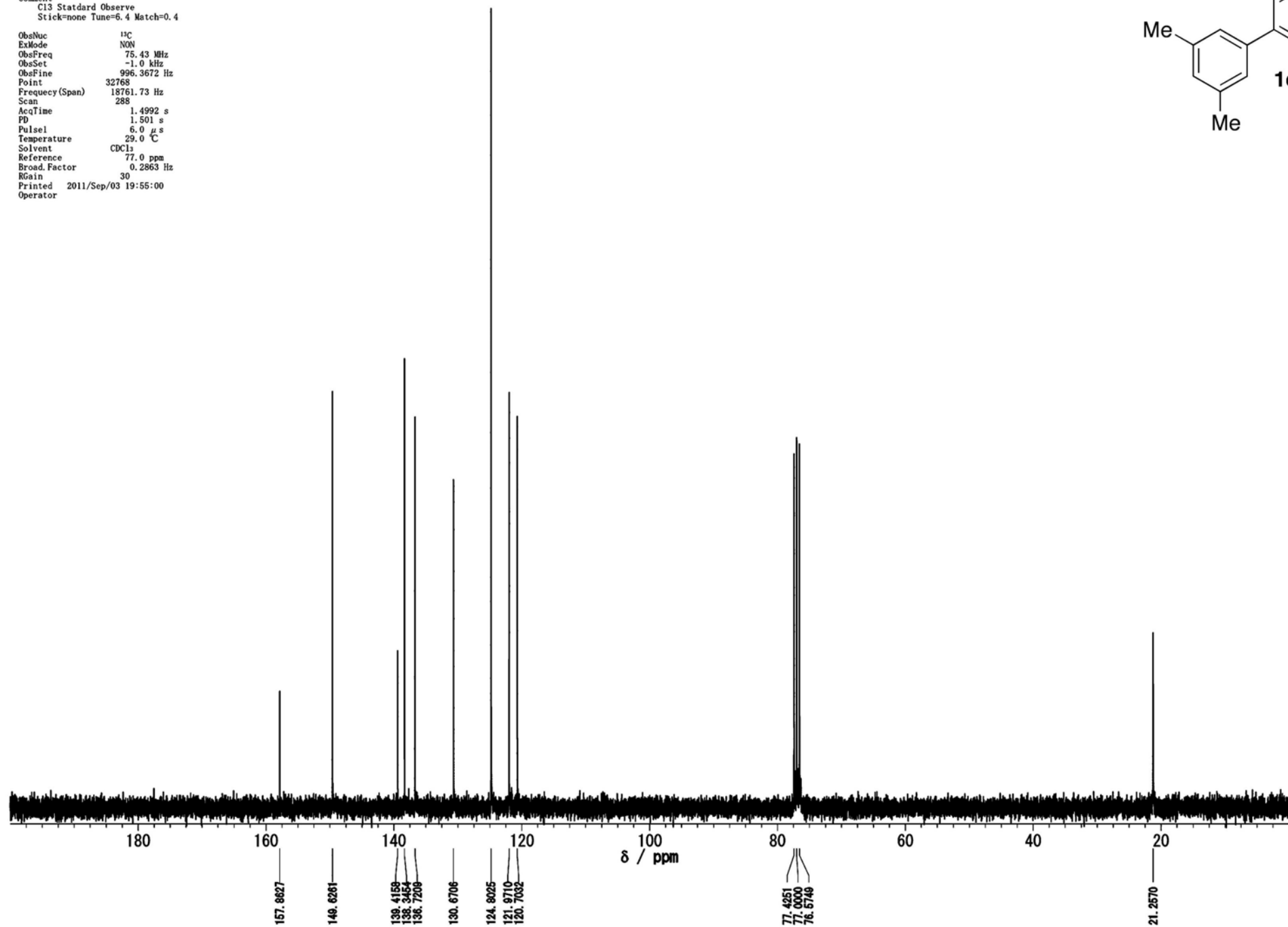
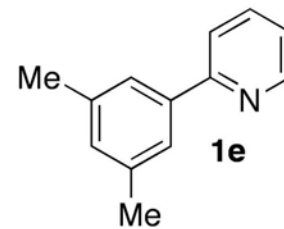


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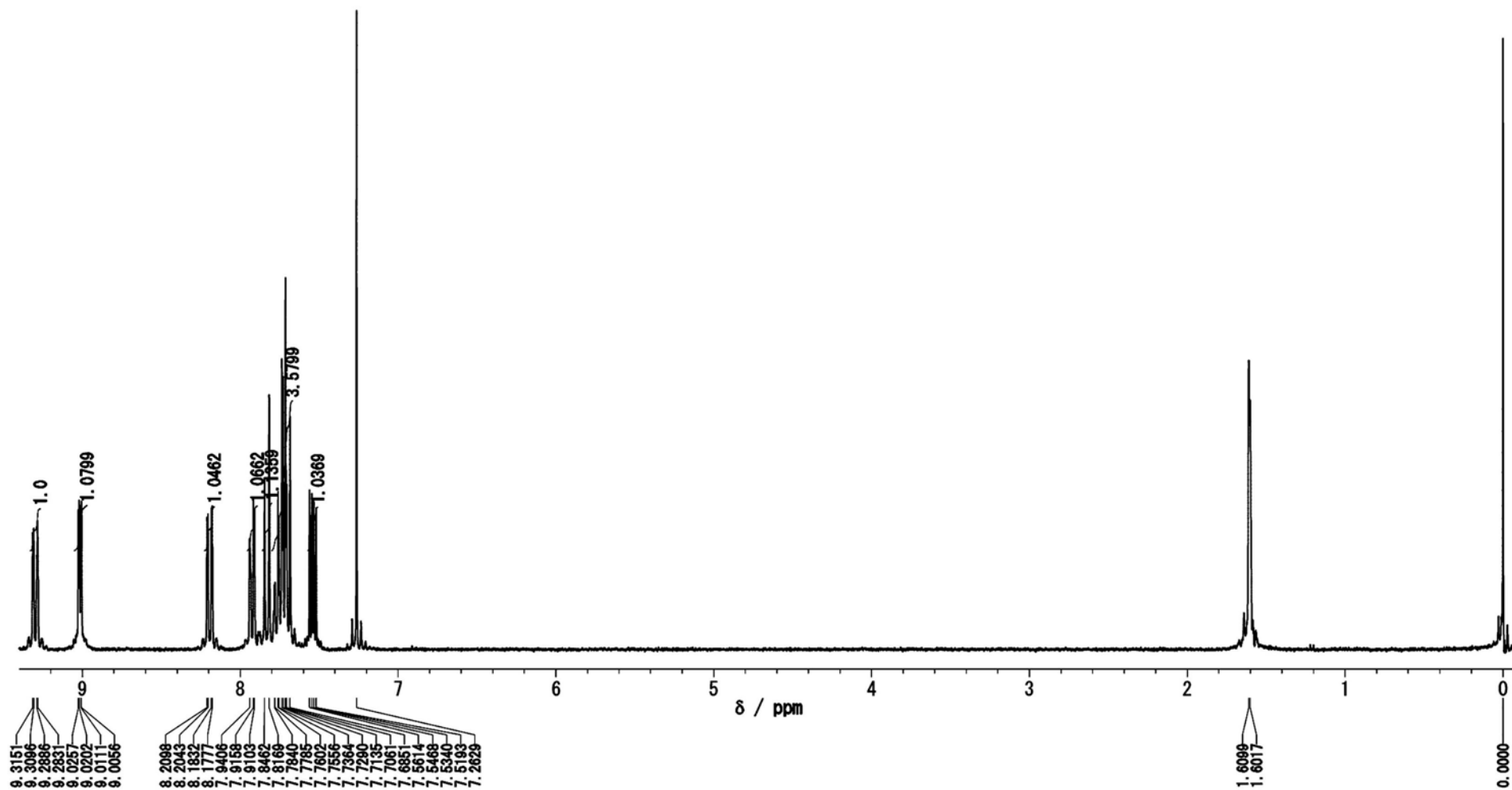
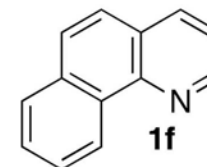
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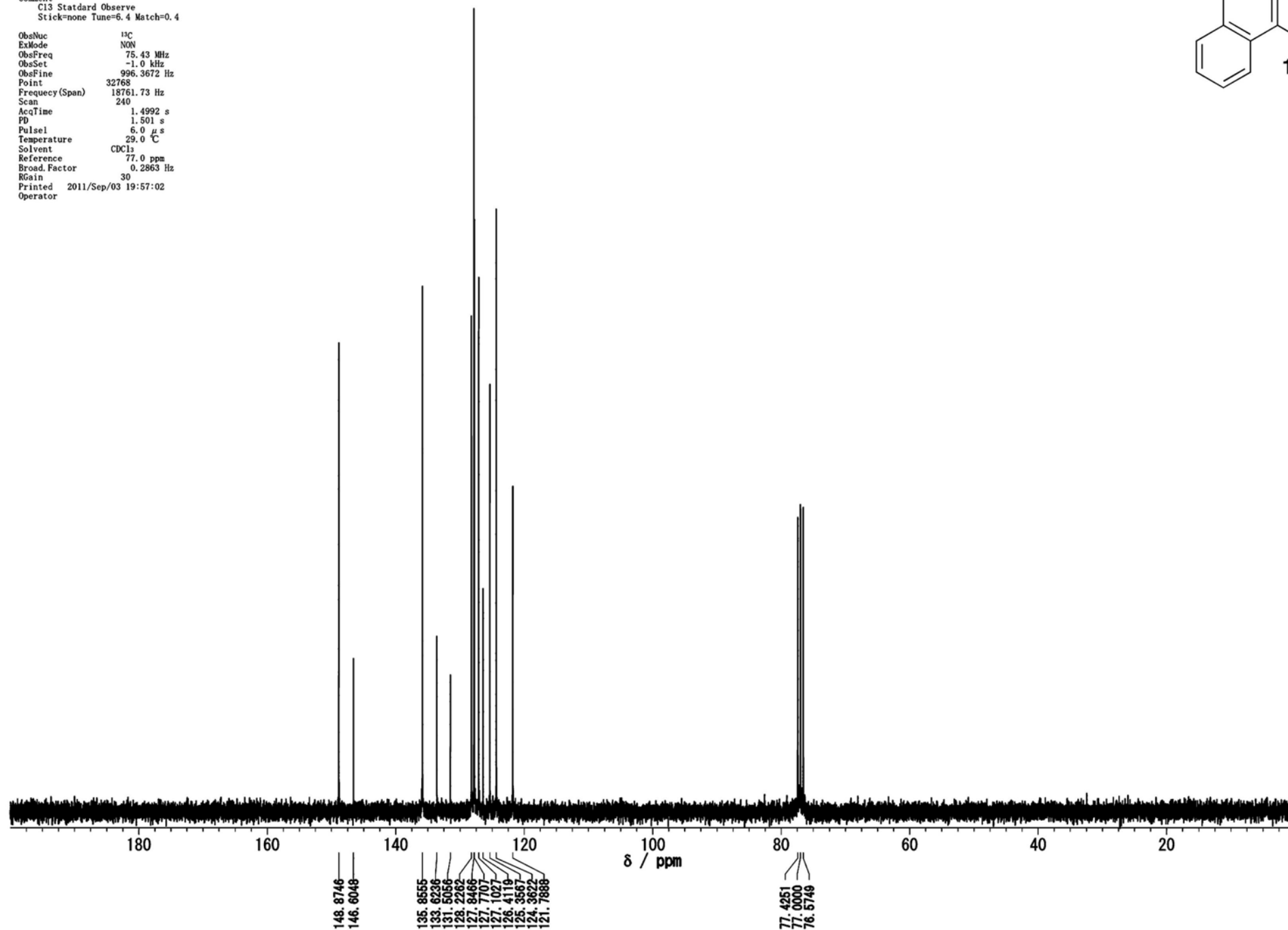
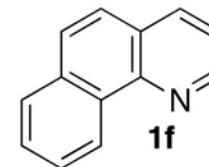
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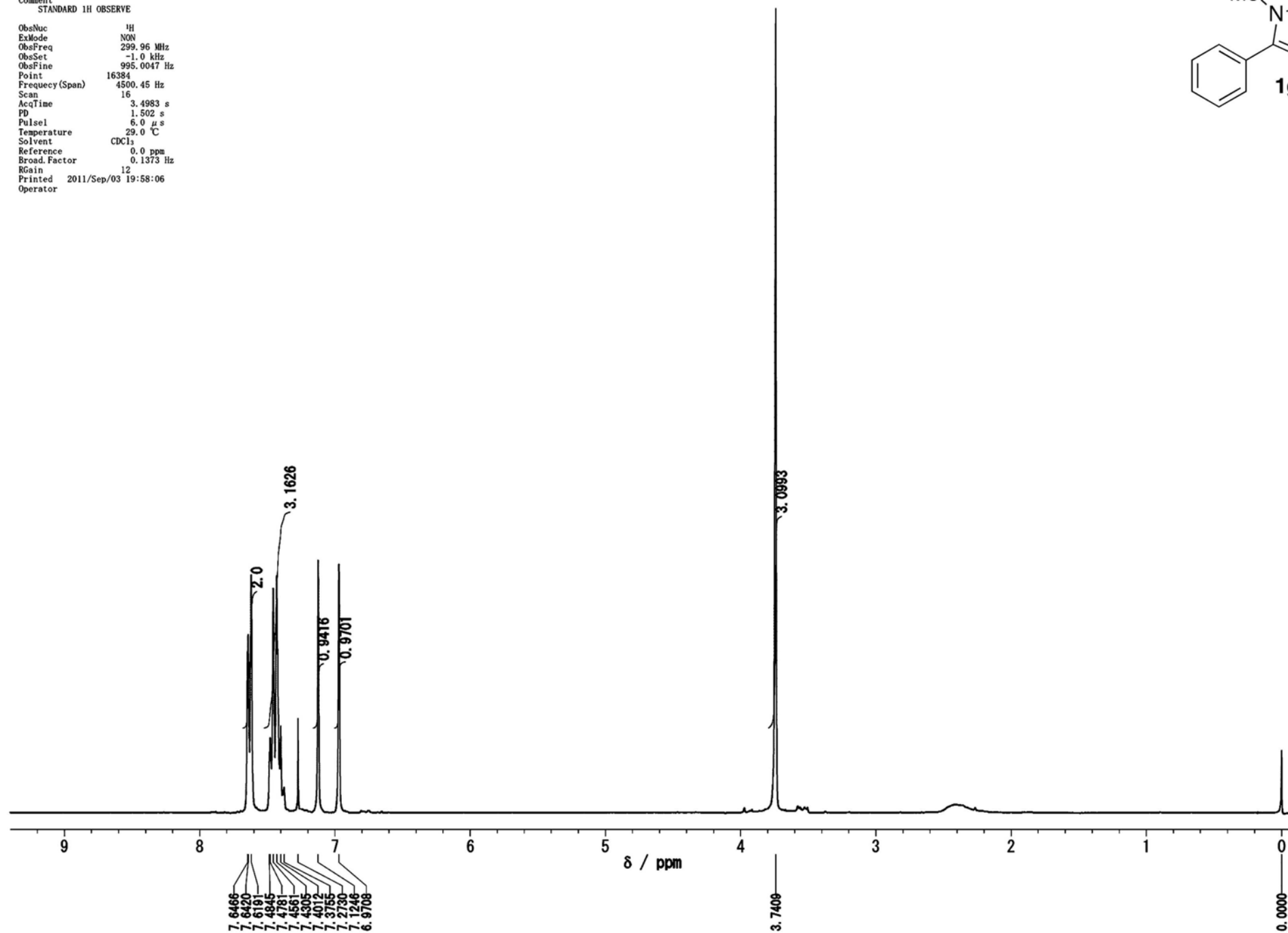
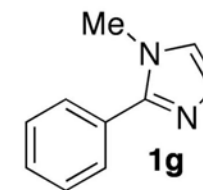
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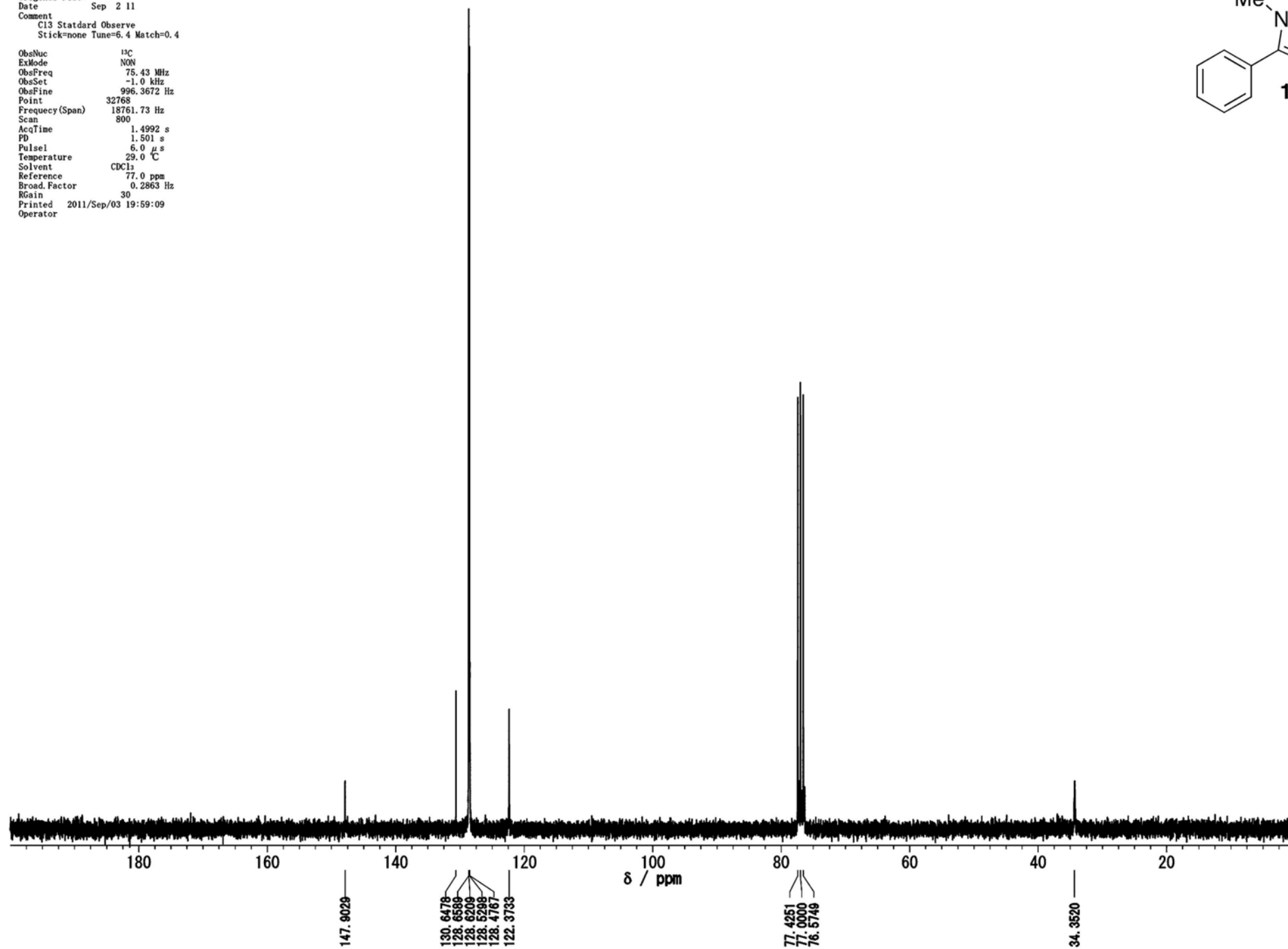
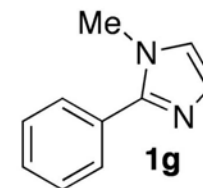
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Printed	2011/Sep/03 19:58:06
Operator	



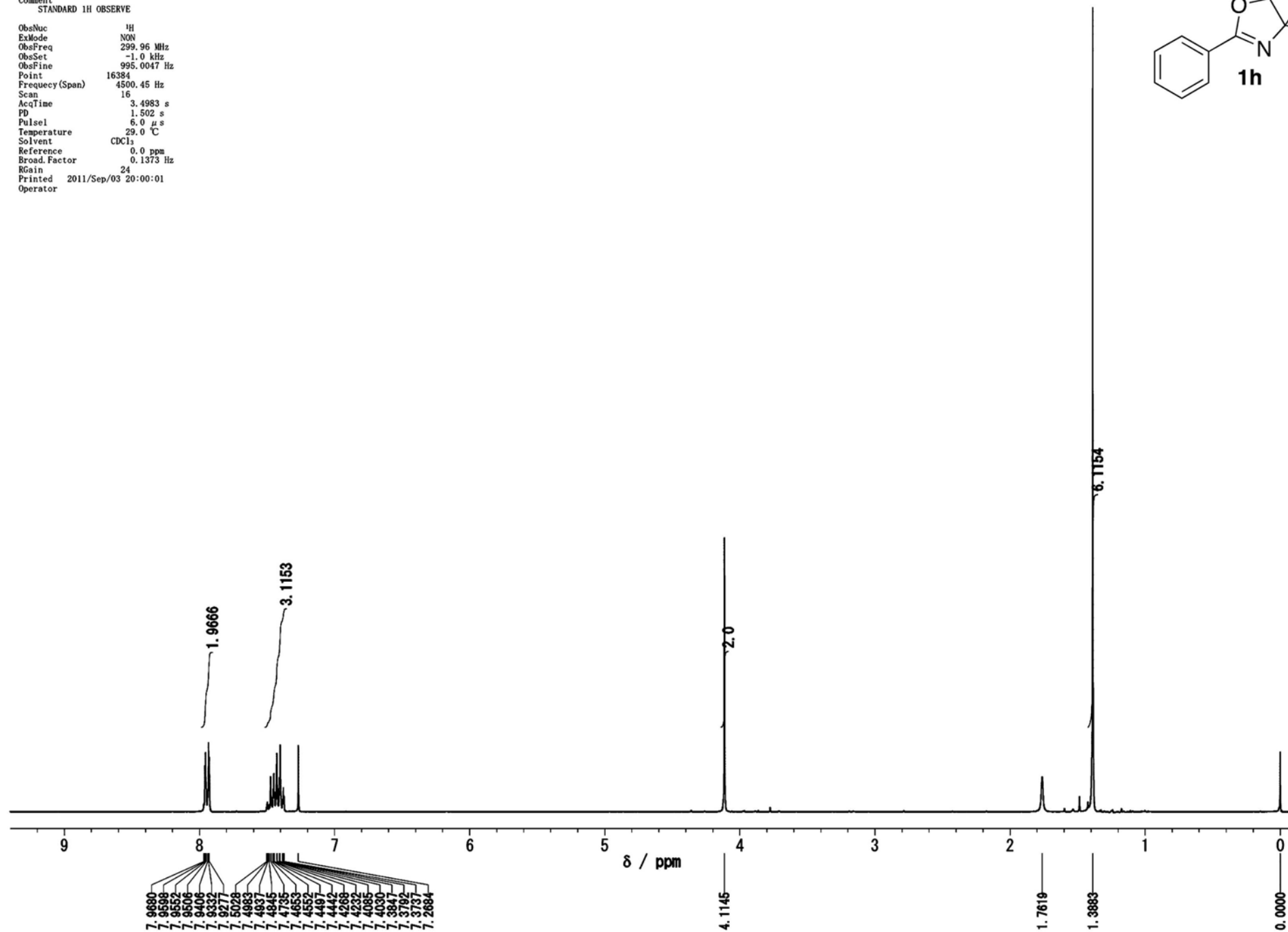
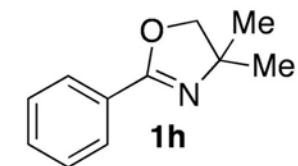
Original File:
 Date Sep 2 11
 Comment
 C13 Statdard Observe
 Stick=none Tune=6.4 Match=0.4

ObsNuc ¹³C
 ExMode NON
 ObsFreq 75.43 MHz
 ObsSet -1.0 kHz
 ObsFine 996.3672 Hz
 Point 32768
 Frequency (Span) 18761.73 Hz
 Scan 800
 AcqTime 1.4992 s
 PD 1.501 s
 Pulsel 6.0 μ s
 Temperature 29.0 $^{\circ}$ C
 Solvent CDCl₃
 Reference 77.0 ppm
 Broad.Factor 0.2863 Hz
 RGain 30
 Printed 2011/Sep/03 19:59:09
 Operator



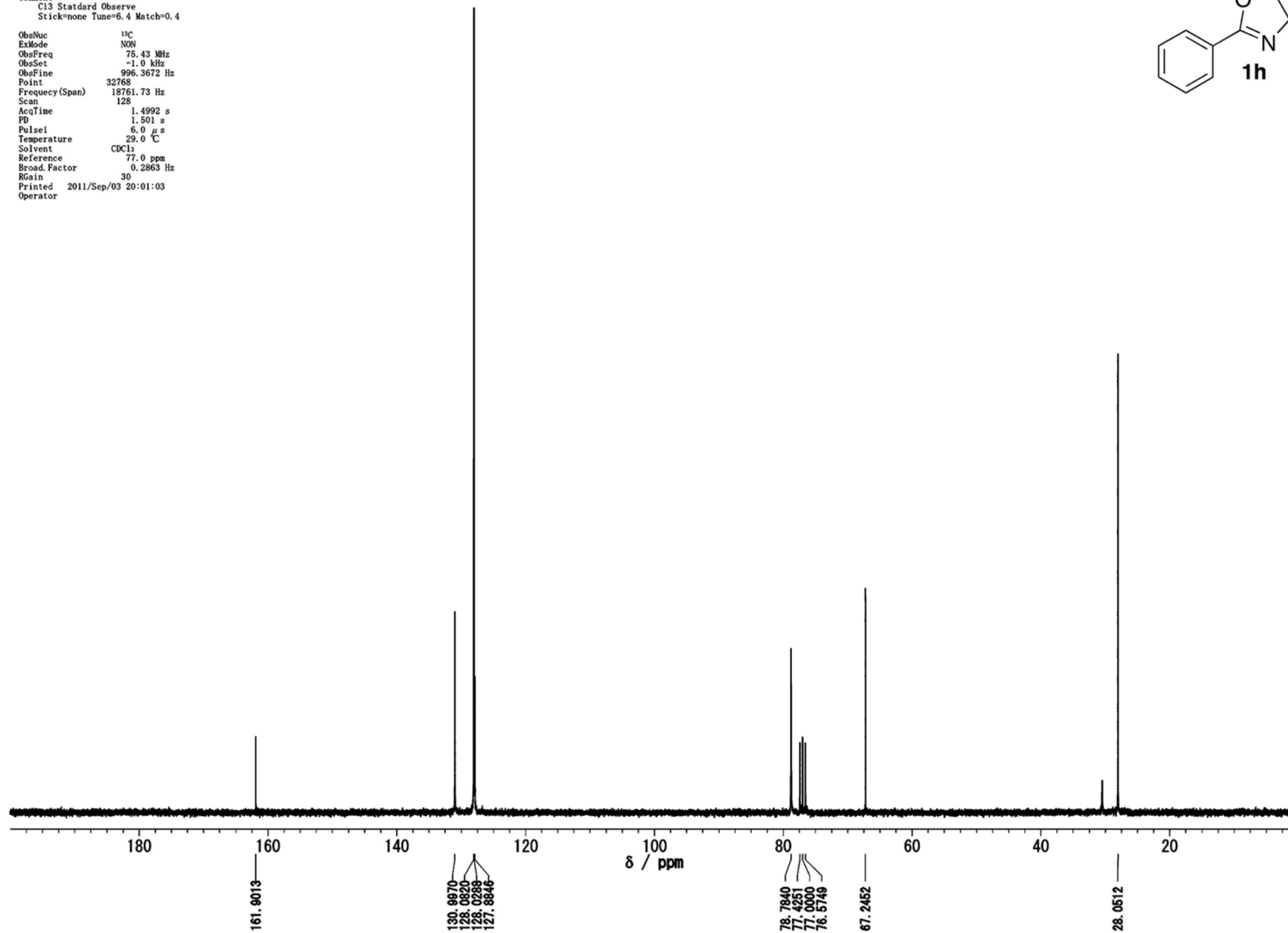
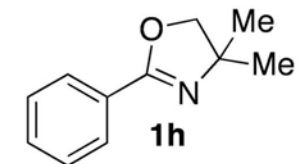
Original File:
 Date Sep 2 11
 Comment
 STANDARD 1H OBSERVE

ObsNuc	¹ H
ExMode	NON
ObsFreq	299.96 MHz
ObsSet	-1.0 kHz
ObsPine	995.0047 Hz
Point	16384
Frequency (Span)	4500.45 Hz
Scan	16
AcqTime	3.4983 s
PD	1.502 s
Pulse1	6.0 μ s
Temperature	29.0 $^{\circ}$ C
Solvent	CDCl ₃
Reference	0.0 ppm
Broad. Factor	0.1373 Hz
RGain	24
Printed	2011/Sep/03 20:00:01
Operator	



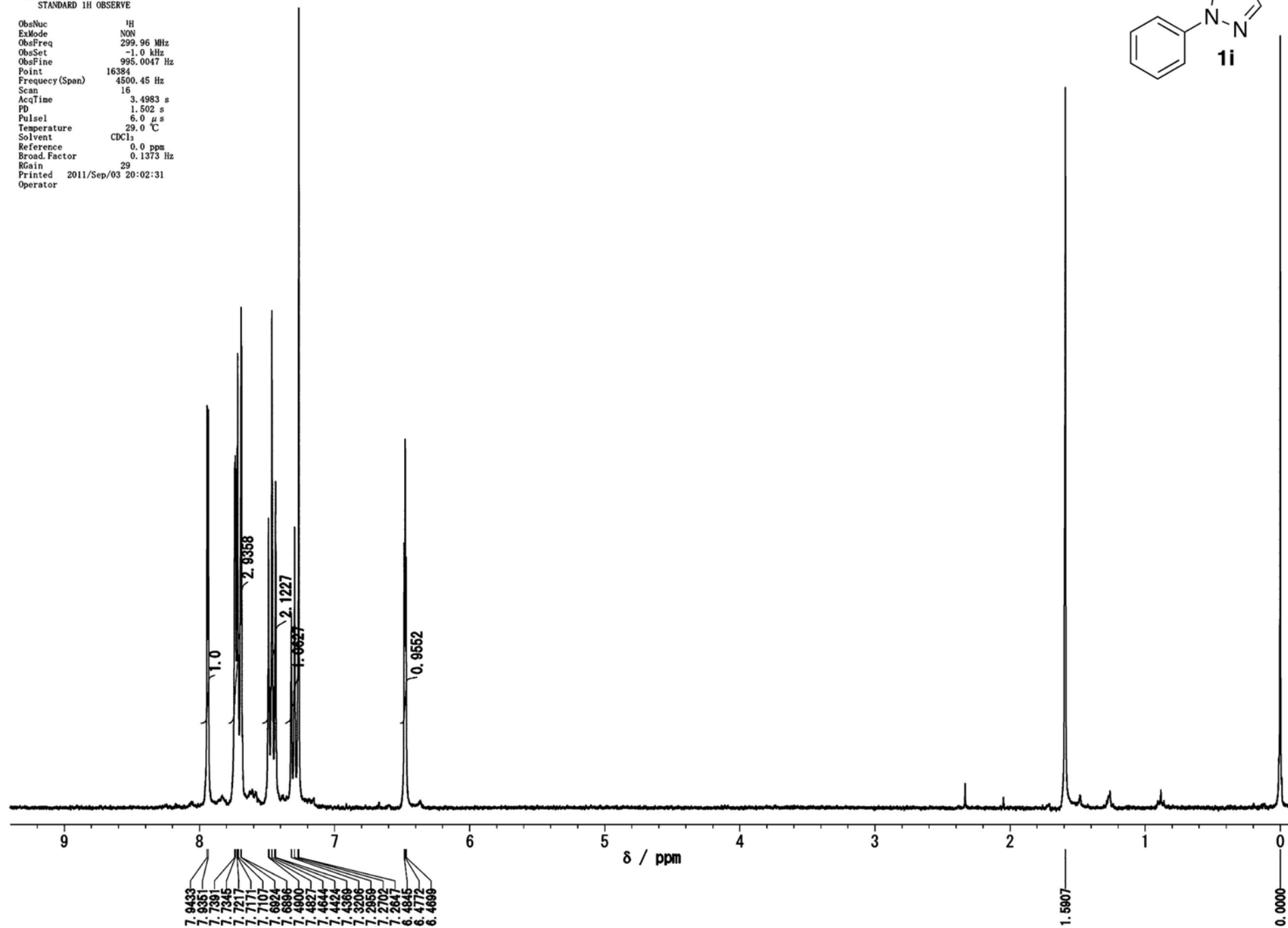
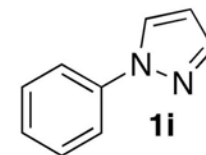
Original File:
 Date Sep 2 11
 Comment
 C13 Statdard Observe
 Stick=none Tune=6.4 Match=0.4

ObsNuc ¹³C
 ExMode NON
 ObsFreq 75.43 MHz
 ObsSet -1.0 kHz
 ObsFine 996.3672 Hz
 Point 32768
 Frequency (Span) 18761.73 Hz
 Scan 128
 AcqTime 1.4992 s
 PD 1.501 s
 Pulsel 6.0 μ s
 Temperature 29.0 $^{\circ}$ C
 Solvent CDCl₃
 Reference 77.0 ppm
 Broad Factor 0.2863 Hz
 RGain 30
 Printed 2011/Sep/03 20:01:03
 Operator



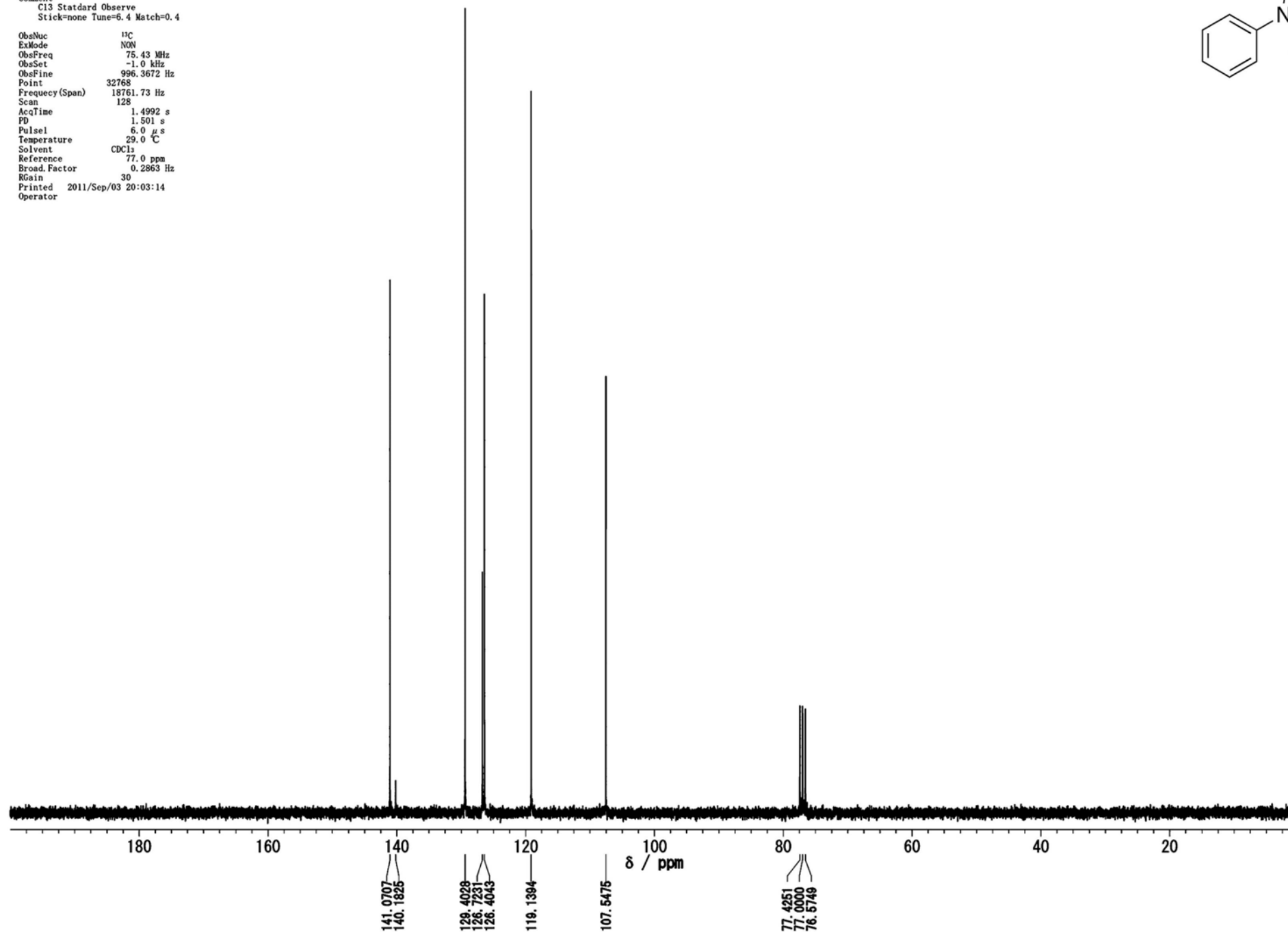
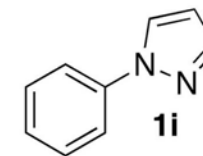
Original File:
 Date Aug 23 11
 Comment
 STANDARD 1H OBSERVE

ObsNuc	¹ H
ExMode	NON
ObsFreq	299.96 MHz
ObsSet	-1.0 kHz
ObsPine	995.0047 Hz
Point	16384
Frequency (Span)	4500.45 Hz
Scan	16
AcqTime	3.4983 s
PD	1.502 s
Pulse1	6.0 μs
Temperature	29.0 °C
Solvent	CDCl ₃
Reference	0.0 ppm
Broad. Factor	0.1373 Hz
RGain	29
Printed	2011/Sep/03 20:02:31
Operator	



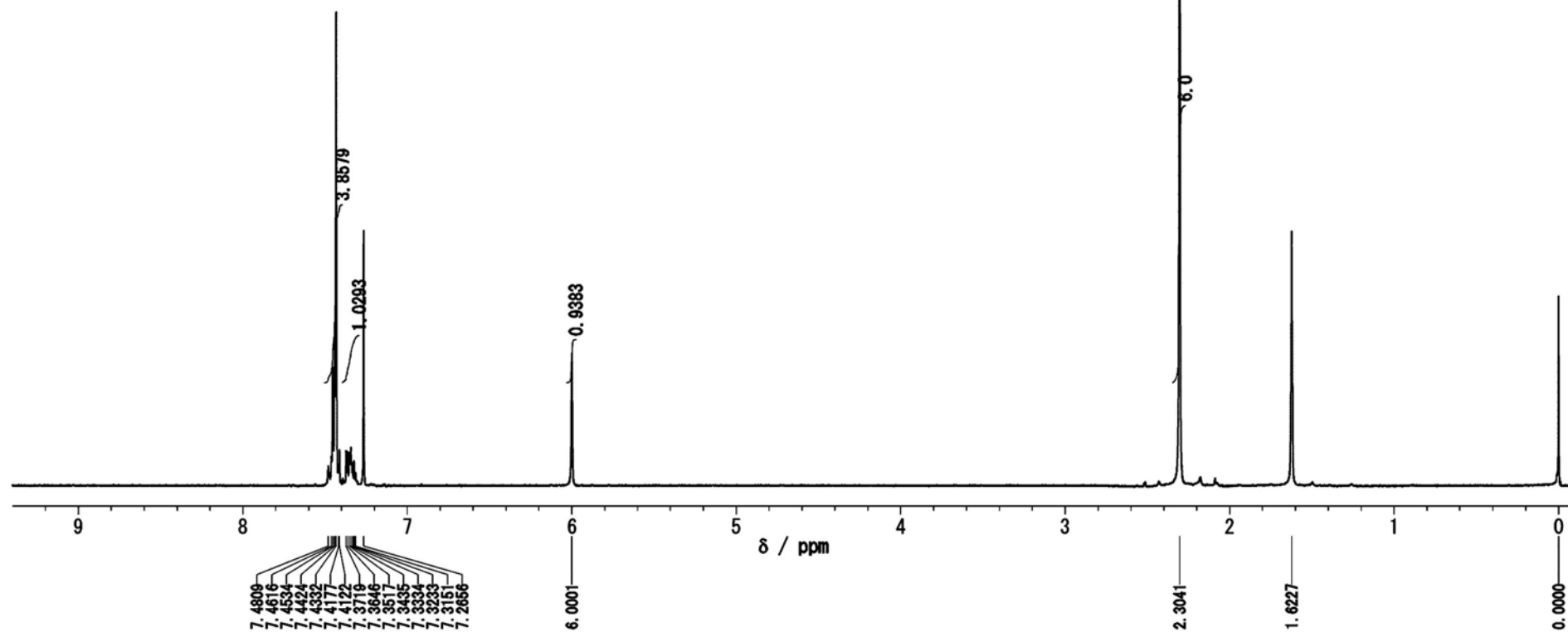
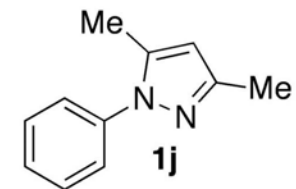
Original File:
Date Sep 2 11
Comment
C13 Statdard Observe
Stick=none Tune=6.4 Match=0.4

ObsNuc ¹³C
ExMode NON
ObsFreq 75.43 MHz
ObsSet -1.0 kHz
ObsFine 996.3672 Hz
Point 32768
Frequency (Span) 18761.73 Hz
Scan 128
AcqTime 1.4992 s
PD 1.501 s
Pulse 6.0 μ s
Temperature 29.0 $^{\circ}$ C
Solvent CDCl₃
Reference 77.0 ppm
Broad Factor 0.2863 Hz
RGain 30
Printed 2011/Sep/03 20:03:14
Operator

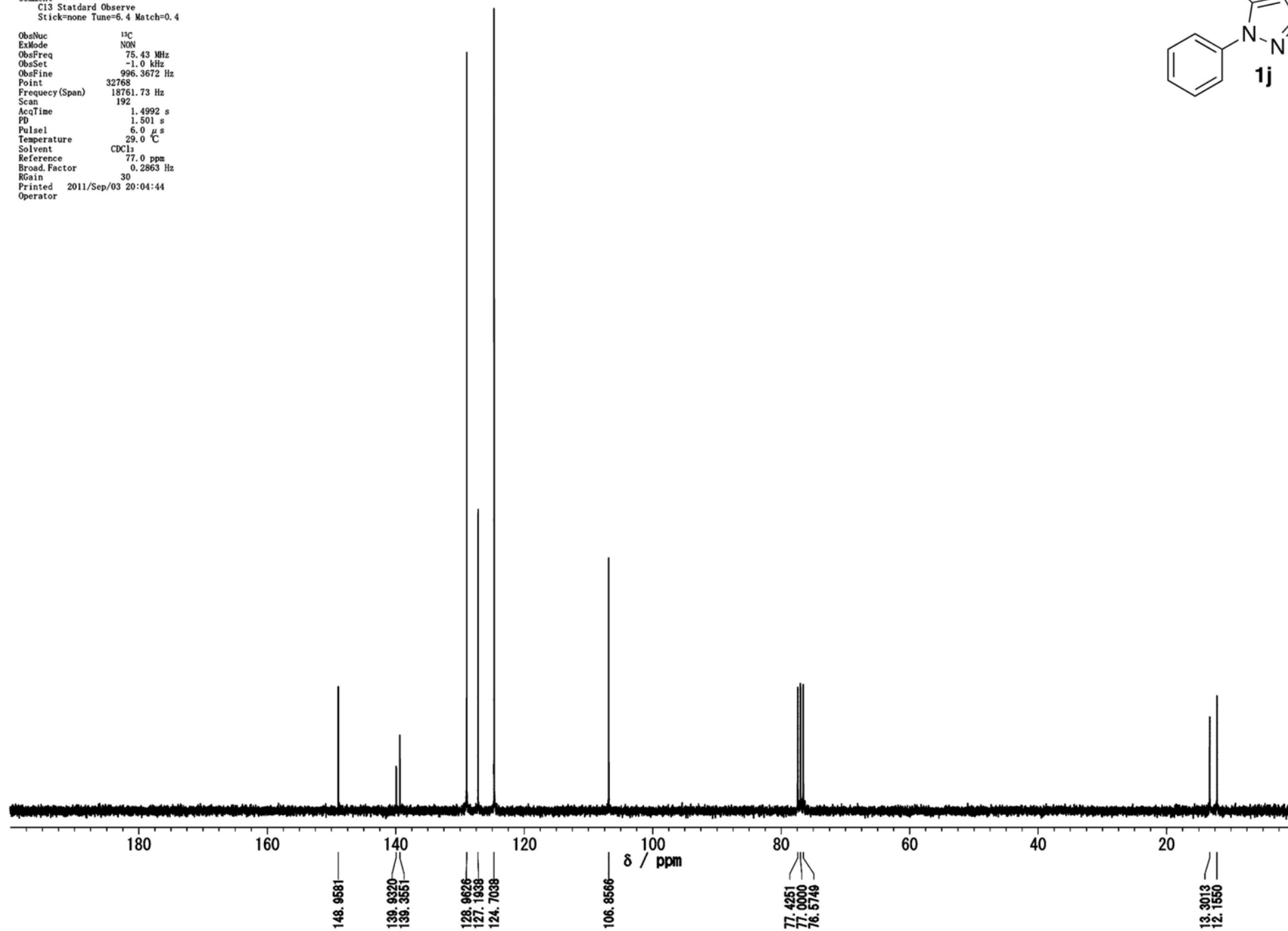
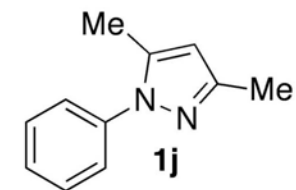


Original File: Jun 23 11
 Date
 Comment
 STANDARD 1H OBSERVE

ObsNuc	¹ H
ExMode	NON
ObsFreq	299.96 MHz
ObsSet	-1.0 kHz
ObsPine	995.0047 Hz
Point	16384
Frequency (Span)	4500.45 Hz
Scan	16
AcqTime	3.4983 s
PD	1.502 s
Pulse1	6.0 μ s
Temperature	29.0 $^{\circ}$ C
Solvent	CDCl ₃
Reference	0.0 ppm
Broad. Factor	0.1373 Hz
RGain	29
Printed	2011/Sep/03 20:03:57
Operator	

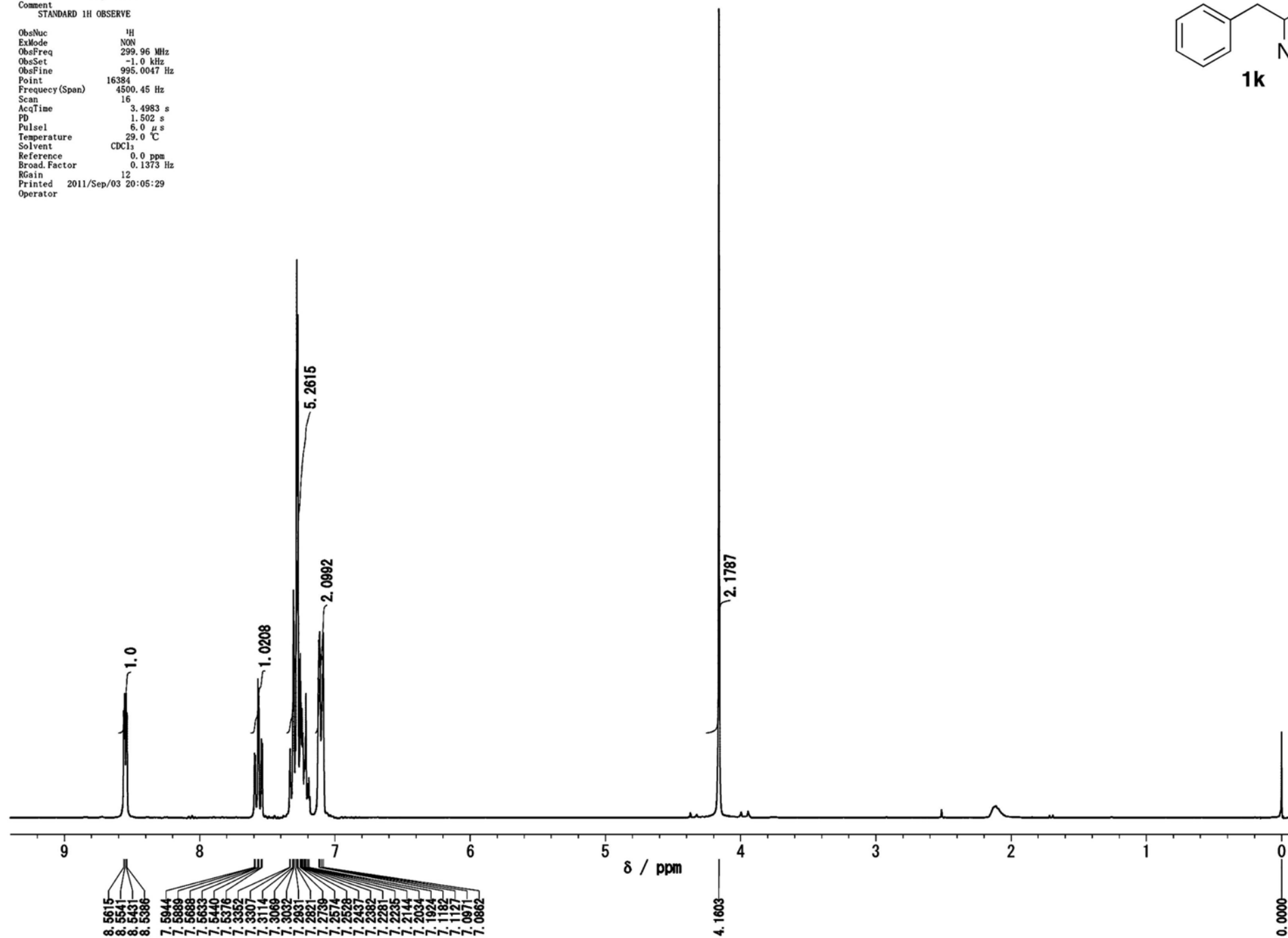
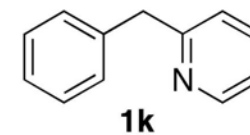


Original File:
 Date Sep 3 11
 Comment
 C13 Statdard Observe
 Stick=none Tune=6.4 Match=0.4
 ObsNuc ¹³C
 ExMode NON
 ObsFreq 75.43 MHz
 ObsSet -1.0 kHz
 ObsFine 996.3672 Hz
 Point 32768
 Frequency (Span) 18761.73 Hz
 Scan 192
 AcqTime 1.4992 s
 PD 1.501 s
 Pulsel 6.0 μ s
 Temperature 29.0 $^{\circ}$ C
 Solvent CDCl₃
 Reference 77.0 ppm
 Broad.Factor 0.2863 Hz
 RGain 30
 Printed 2011/Sep/03 20:04:44
 Operator



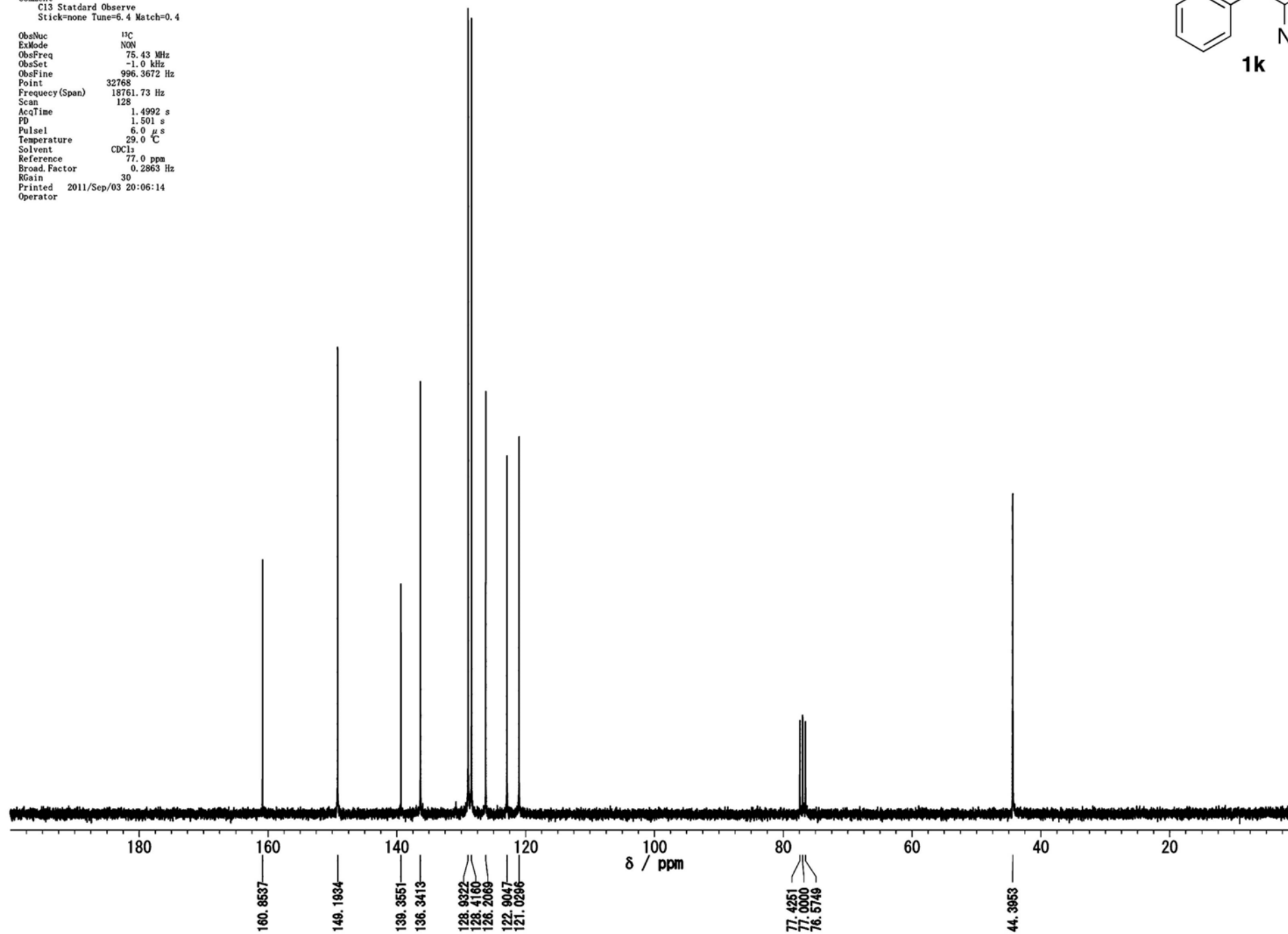
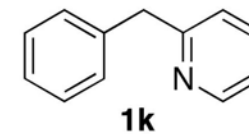
Original File:
 Date Sep 2 11
 Comment
 STANDARD 1H OBSERVE

ObsNuc	1H
ExMode	NON
ObsFreq	299.96 MHz
ObsSet	-1.0 kHz
ObsPine	995.0047 Hz
Point	16384
Frequency (Span)	4500.45 Hz
Scan	16
AcqTime	3.4983 s
PD	1.502 s
Pulse1	6.0 μ s
Temperature	29.0 $^{\circ}$ C
Solvent	CDCl ₃
Reference	0.0 ppm
Broad.Factor	0.1373 Hz
RGain	12
Printed	2011/Sep/03 20:05:29
Operator	

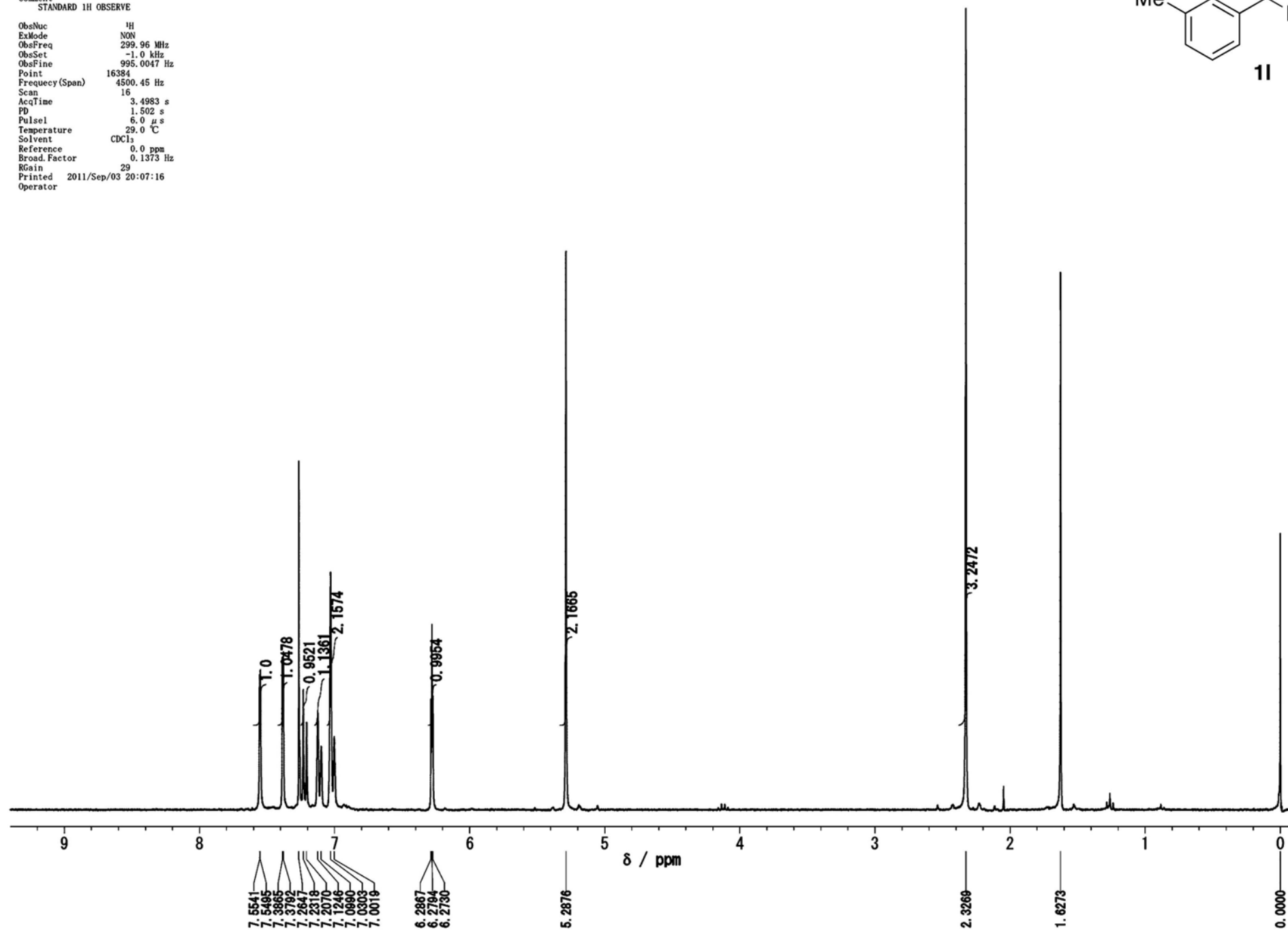
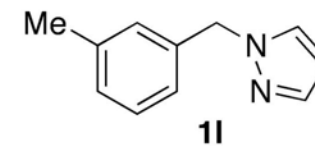


Original File:
 Date Sep 2 11
 Comment
 C13 Statdard Observe
 Stick=none Tune=6.4 Match=0.4

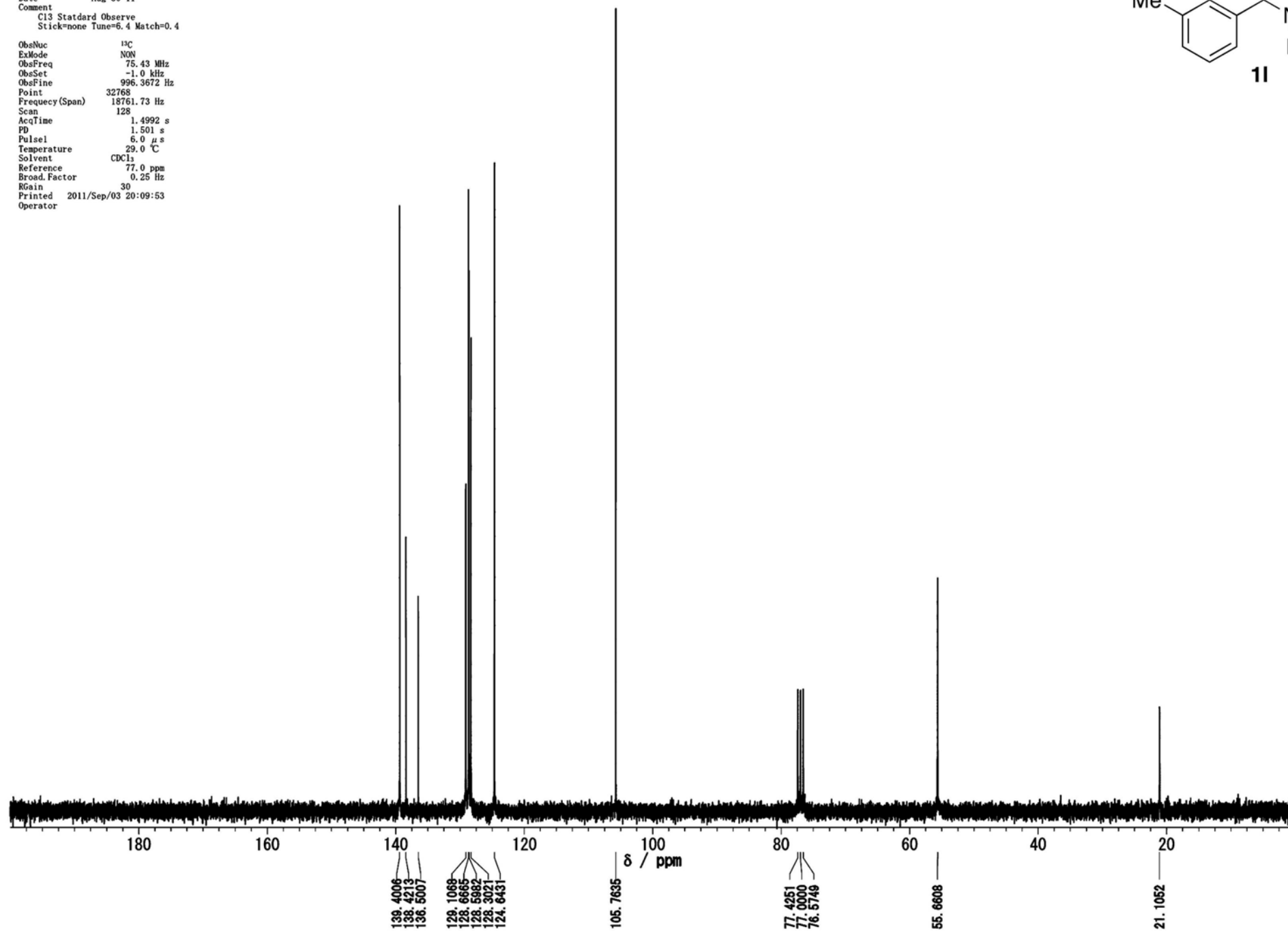
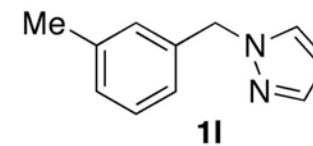
ObsNuc ^{13}C
 ExMode NON
 ObsFreq 75.43 MHz
 ObsSet -1.0 kHz
 ObsFine 996.3672 Hz
 Point 32768
 Frequency (Span) 18761.73 Hz
 Scan 128
 AcqTime 1.4992 s
 PD 1.501 s
 Pulsel 6.0 μs
 Temperature 29.0 $^{\circ}\text{C}$
 Solvent CDCl_3
 Reference 77.0 ppm
 Broad.Factor 0.2863 Hz
 RGain 30
 Printed 2011/Sep/03 20:06:14
 Operator



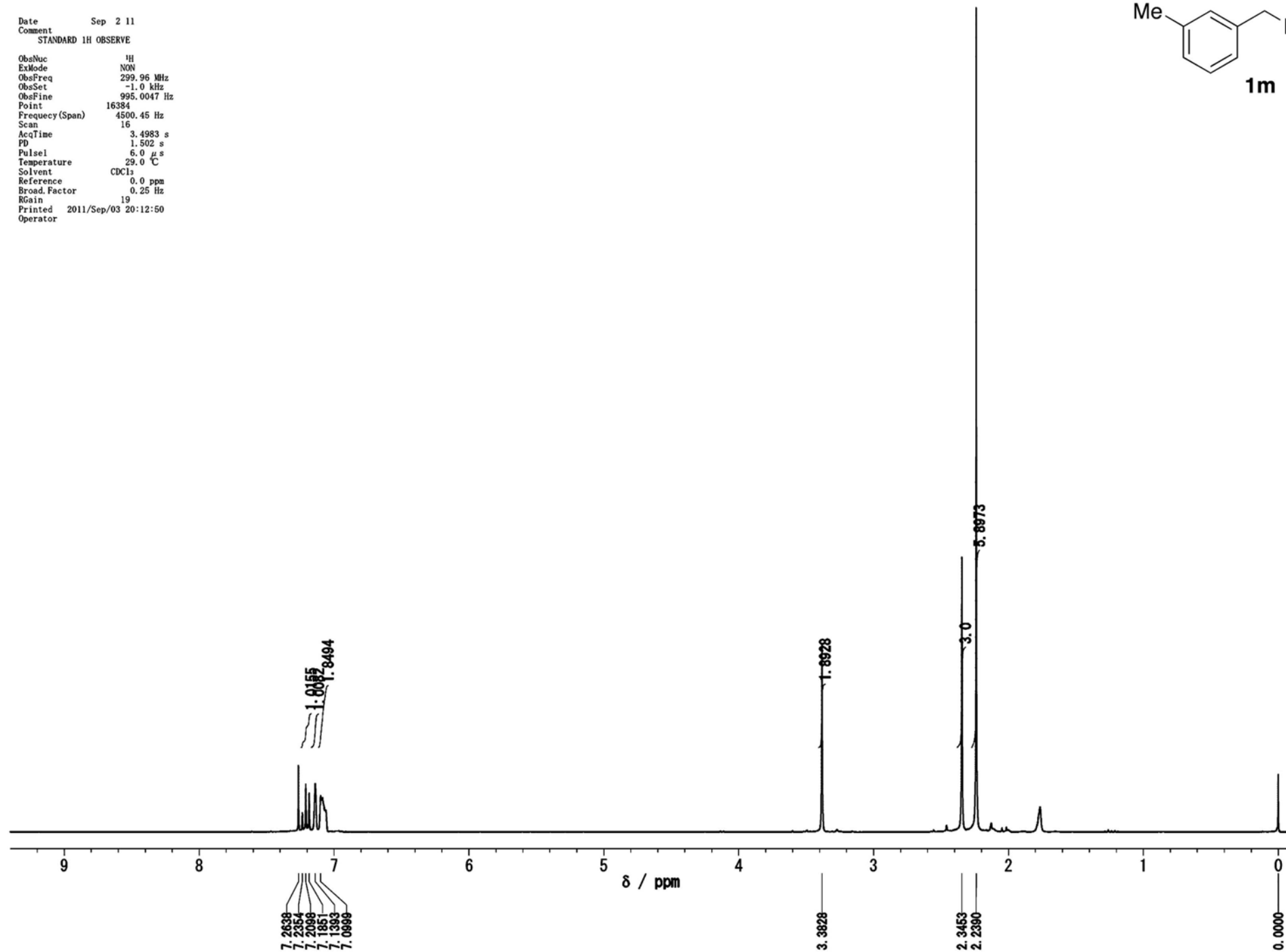
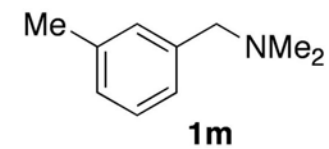
Original File: Jun 21 11
 Date: Jun 21 11
 Comment: STANDARD 1H OBSERVE
 ObsNuc: ¹H
 ExMode: NON
 ObsFreq: 299.96 MHz
 ObsSet: -1.0 kHz
 ObsPine: 995.0047 Hz
 Point: 16384
 Frequency (Span): 4500.45 Hz
 Scan: 16
 AcqTime: 3.4983 s
 PD: 1.502 s
 Pulse1: 6.0 μ s
 Temperature: 29.0 $^{\circ}$ C
 Solvent: CDCl₃
 Reference: 0.0 ppm
 Broad Factor: 0.1373 Hz
 RGain: 29
 Printed: 2011/Sep/03 20:07:16
 Operator:



Date Aug 30 11
 Comment C13 Statdard Observe
 Stick=none Tune=6.4 Match=0.4
 ObsNuc ¹³C
 ExMode NQV
 ObsFreq 75.43 MHz
 ObsSet -1.0 kHz
 ObsFine 996.3672 Hz
 Point 32768
 Frequency (Span) 18761.73 Hz
 Scan 128
 AcqTime 1.4992 s
 PD 1.501 s
 Pulse 6.0 μ s
 Temperature 29.0 $^{\circ}$ C
 Solvent CDCl₃
 Reference 77.0 ppm
 Broad. Factor 0.25 Hz
 RGain 30
 Printed 2011/Sep/03 20:09:53
 Operator

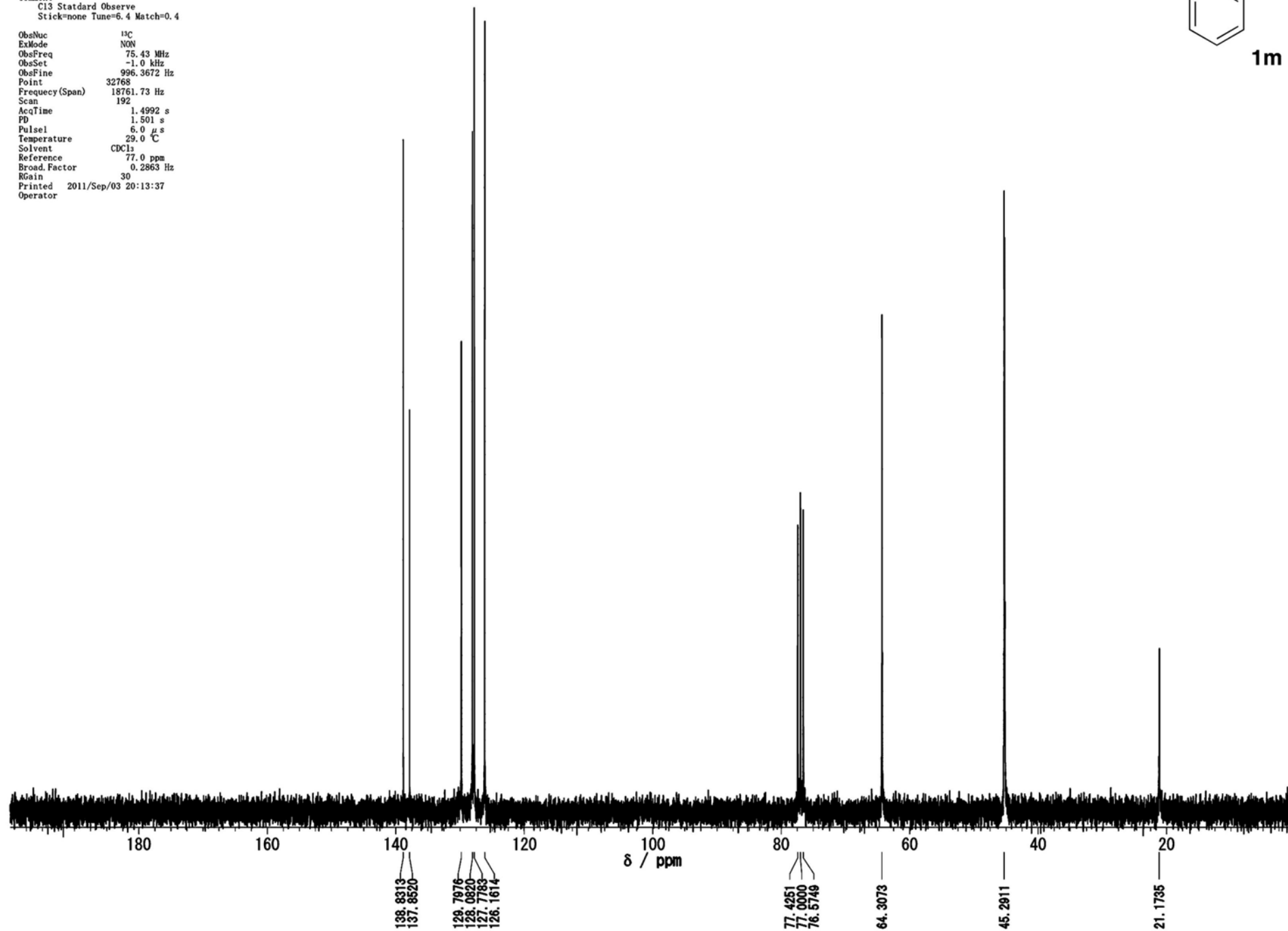
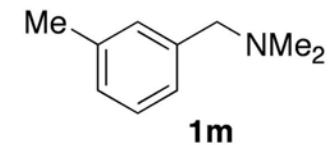


Date Sep 2 11
 Comment STANDARD 1H OBSERVE
 ObsNuc ¹H
 ExMode NON
 ObsFreq 299.96 MHz
 ObsSet -1.0 kHz
 ObsFine 995.0047 Hz
 Point 16384
 Frequency (Span) 4500.45 Hz
 Scan 16
 AcqTime 3.4983 s
 PD 1.502 s
 Pulse 6.0 μ s
 Temperature 29.0 $^{\circ}$ C
 Solvent CDCl₃
 Reference 0.0 ppm
 Broad Factor 0.25 Hz
 RGain 19
 Printed 2011/Sep/03 20:12:50
 Operator



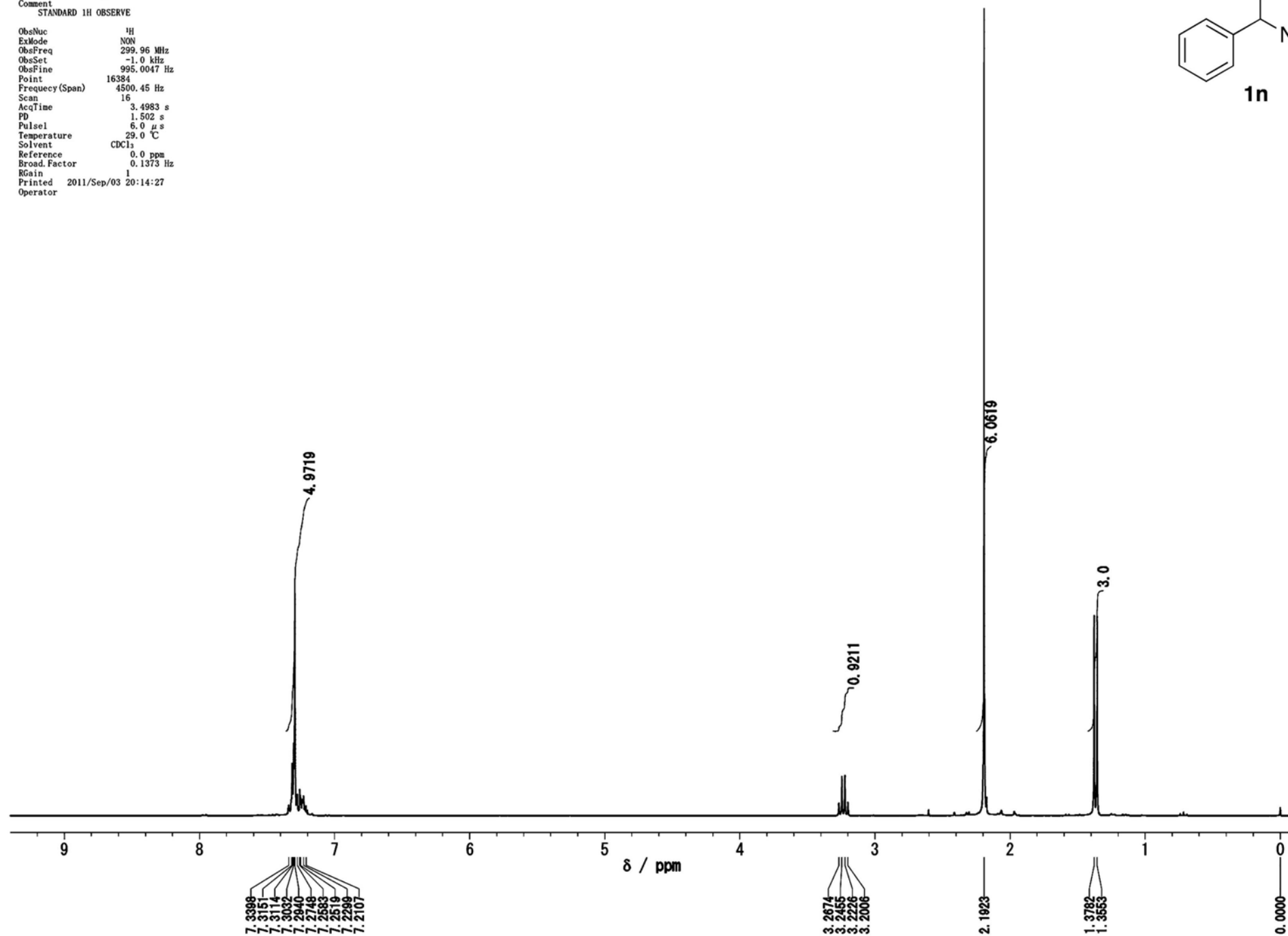
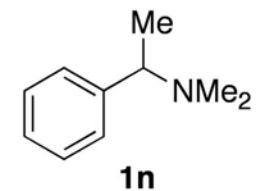
Original File:
 Date Sep 2 11
 Comment
 C13 Statdard Observe
 Stick=none Tune=6.4 Match=0.4

ObsNuc ^{13}C
 ExMode NON
 ObsFreq 75.43 MHz
 ObsSet -1.0 kHz
 ObsFine 996.3672 Hz
 Point 32768
 Frequency (Span) 18761.73 Hz
 Scan 192
 AcqTime 1.4992 s
 PD 1.501 s
 Pulsel 6.0 μs
 Temperature 29.0 $^{\circ}\text{C}$
 Solvent CDCl_3
 Reference 77.0 ppm
 Broad.Factor 0.2863 Hz
 RGain 30
 Printed 2011/Sep/03 20:13:37
 Operator



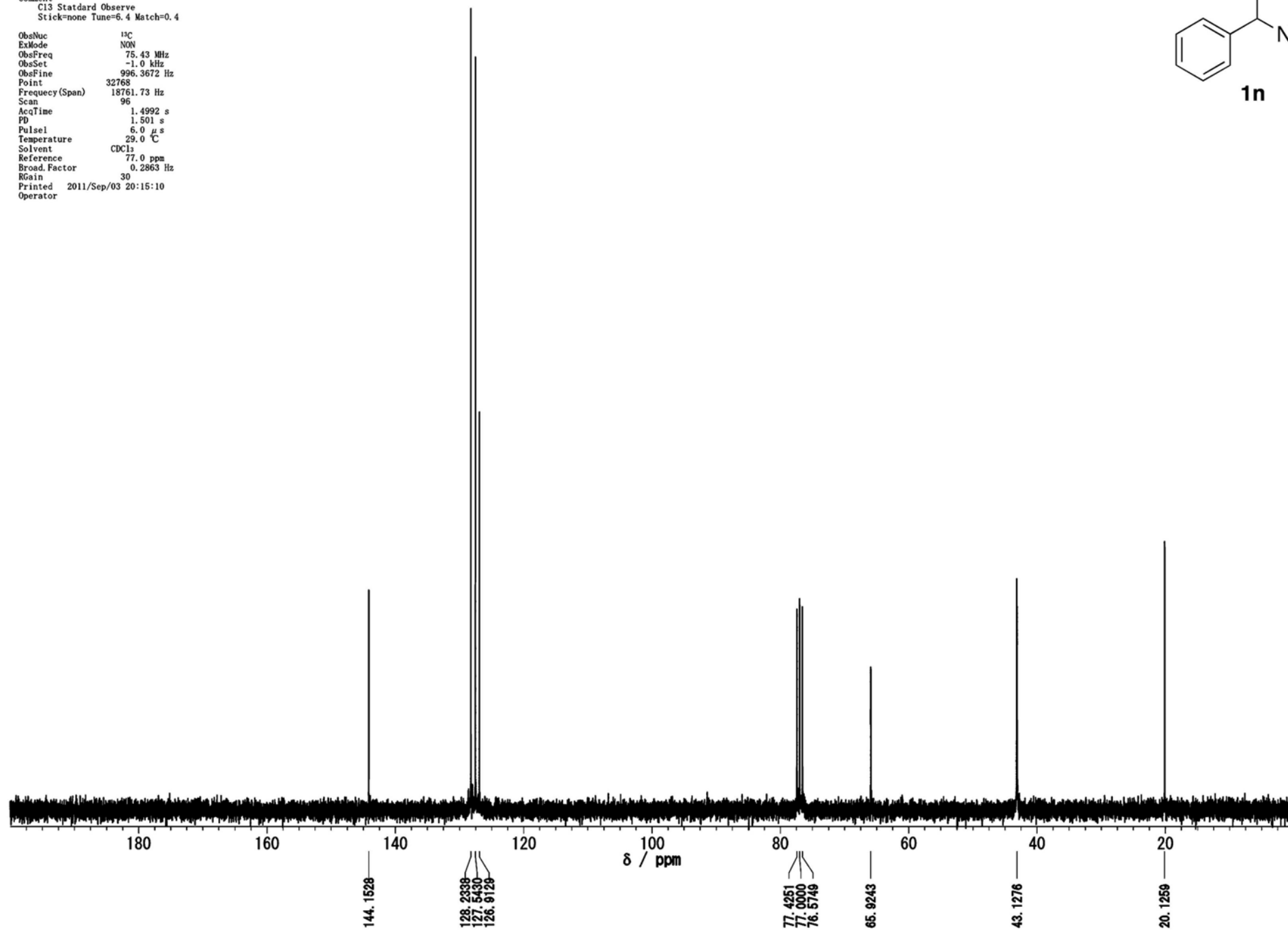
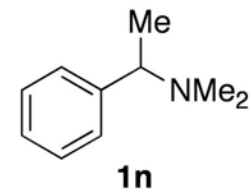
Original File:
 Date Aug 31 11
 Comment
 STANDARD 1H OBSERVE

ObsNuc	¹ H
ExMode	NON
ObsFreq	299.96 MHz
ObsSet	-1.0 kHz
ObsPine	995.0047 Hz
Point	16384
Frequency (Span)	4500.45 Hz
Scan	16
AcqTime	3.4983 s
PD	1.502 s
Pulse1	6.0 μ s
Temperature	29.0 $^{\circ}$ C
Solvent	CDCl ₃
Reference	0.0 ppm
Broad.Factor	0.1373 Hz
RGain	1
Printed	2011/Sep/03 20:14:27
Operator	



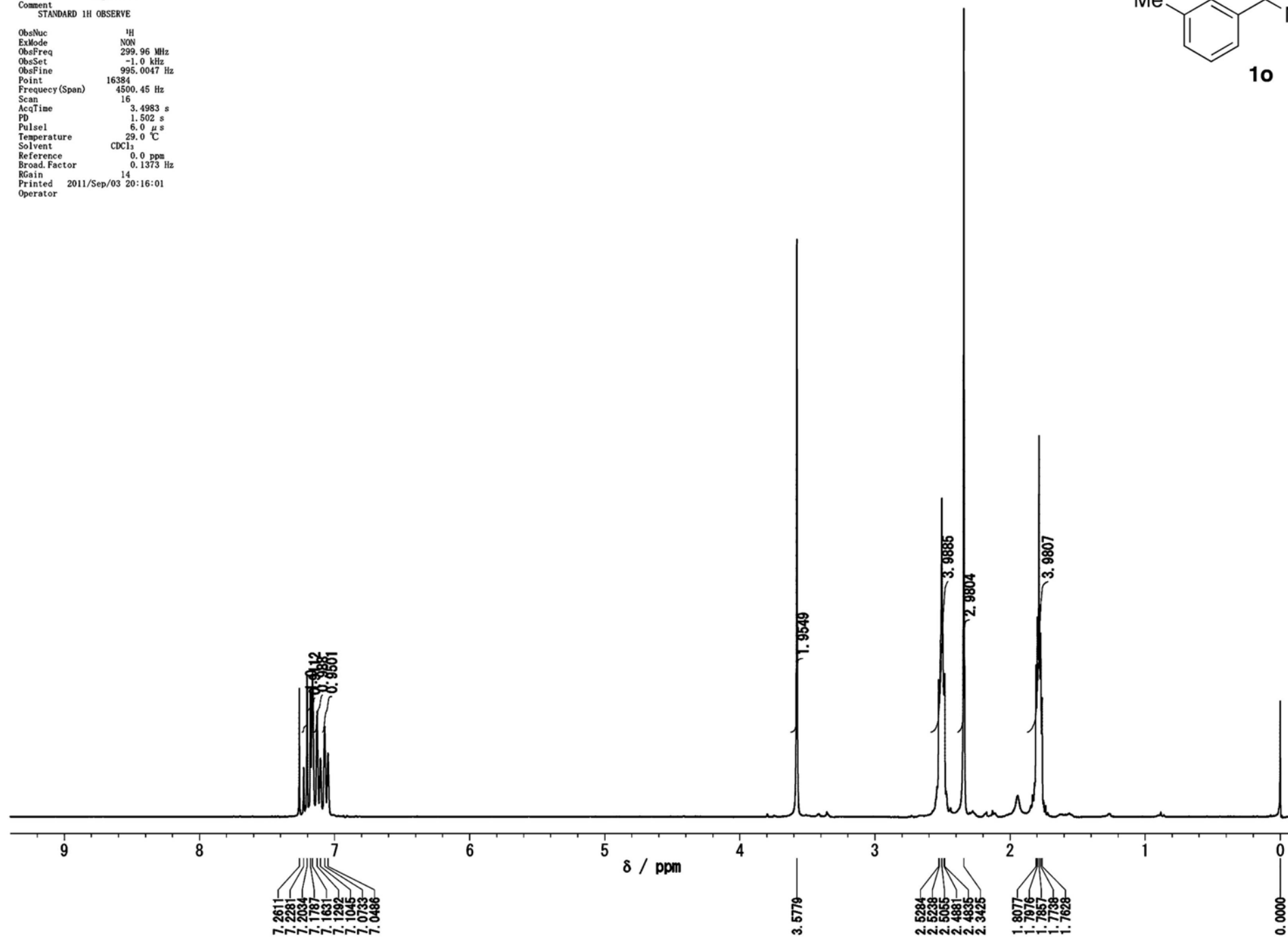
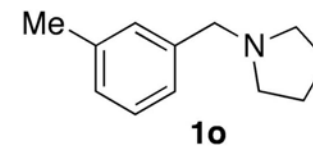
Original File:
Date Aug 31 11
Comment
C13 Statdard Observe
Stick=none Tune=6.4 Match=0.4

ObsNuc ¹³C
ExMode NON
ObsFreq 75.43 MHz
ObsSet -1.0 kHz
ObsFine 996.3672 Hz
Point 32768
Frequency (Span) 18761.73 Hz
Scan 96
AcqTime 1.4992 s
PD 1.501 s
Pulse 6.0 μ s
Temperature 29.0 $^{\circ}$ C
Solvent CDCl₃
Reference 77.0 ppm
Broad Factor 0.2863 Hz
RGain 30
Printed 2011/Sep/03 20:15:10
Operator



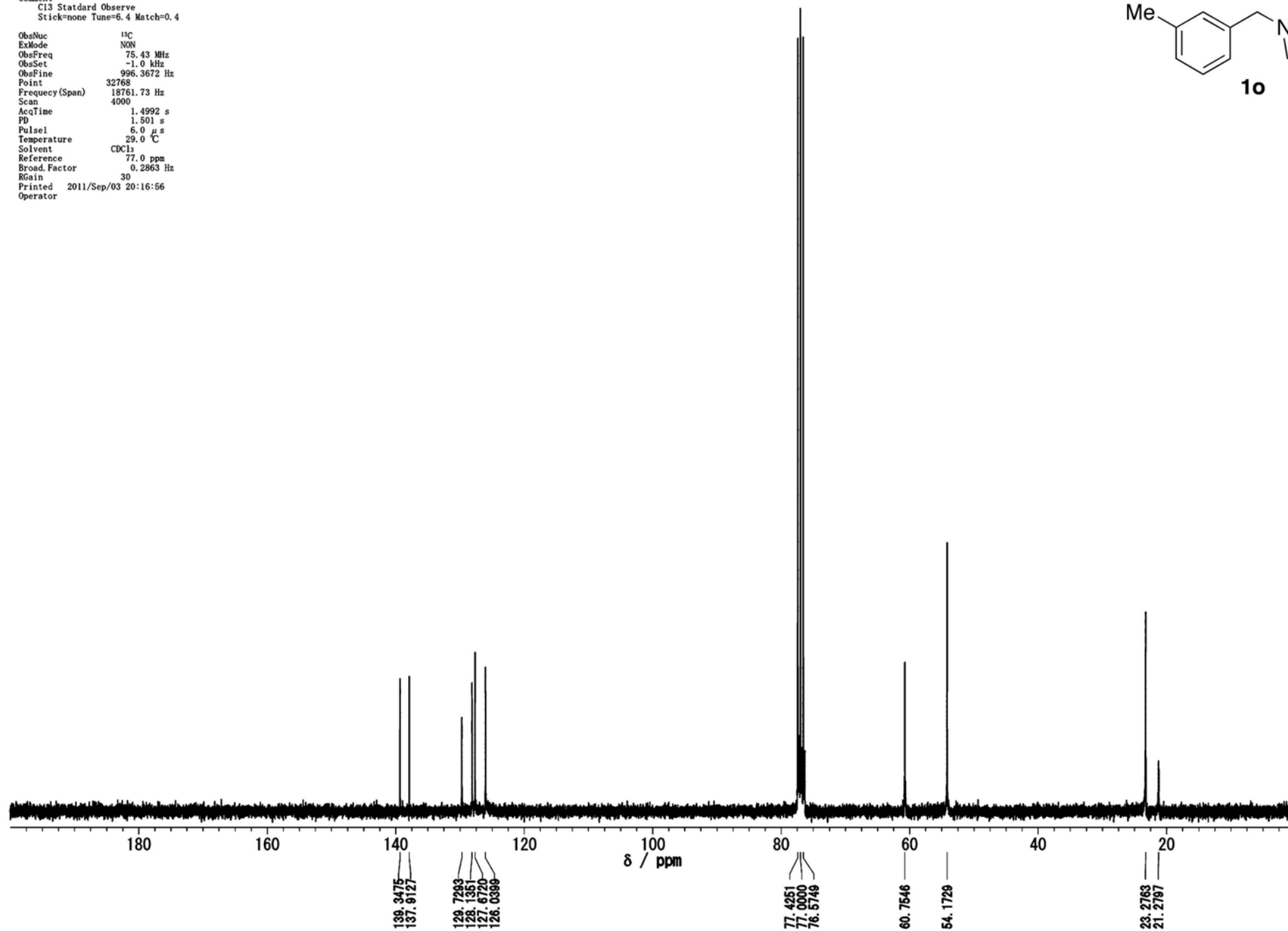
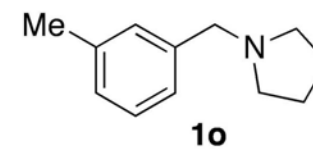
Original File:
 Date Aug 31 11
 Comment
 STANDARD 1H OBSERVE

ObsNuc	¹ H
ExMode	NON
ObsFreq	299.96 MHz
ObsSet	-1.0 kHz
ObsPine	995.0047 Hz
Point	16384
Frequency (Span)	4500.45 Hz
Scan	16
AcqTime	3.4983 s
PD	1.502 s
Pulse1	6.0 μ s
Temperature	29.0 $^{\circ}$ C
Solvent	CDCl ₃
Reference	0.0 ppm
Broad.Factor	0.1373 Hz
RGain	14
Printed	2011/Sep/03 20:16:01
Operator	

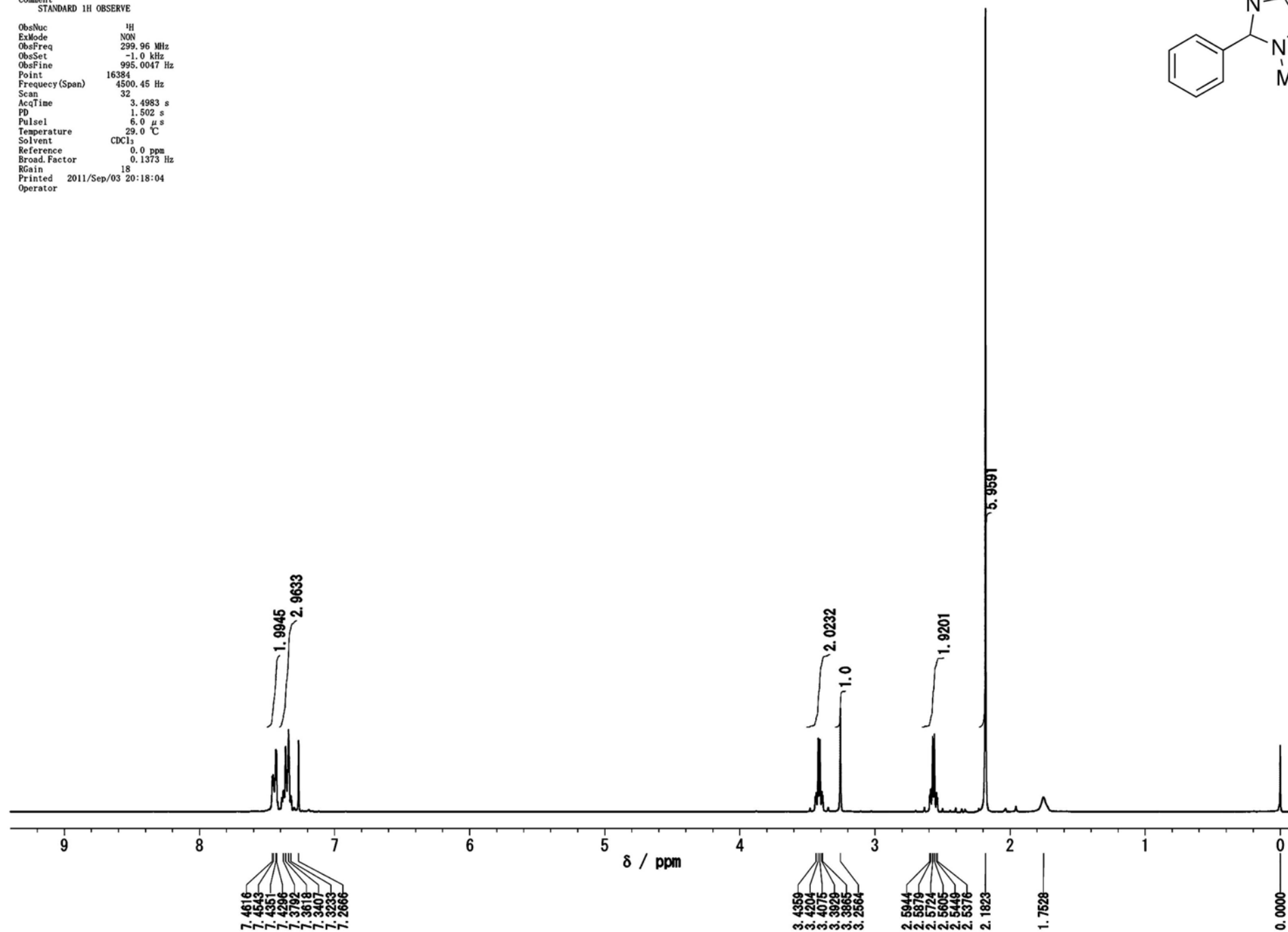
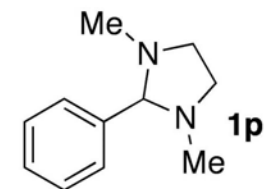


Original File:
 Date Aug 31 11
 Comment
 C13 Statdard Observe
 Stick=none Tune=6.4 Match=0.4

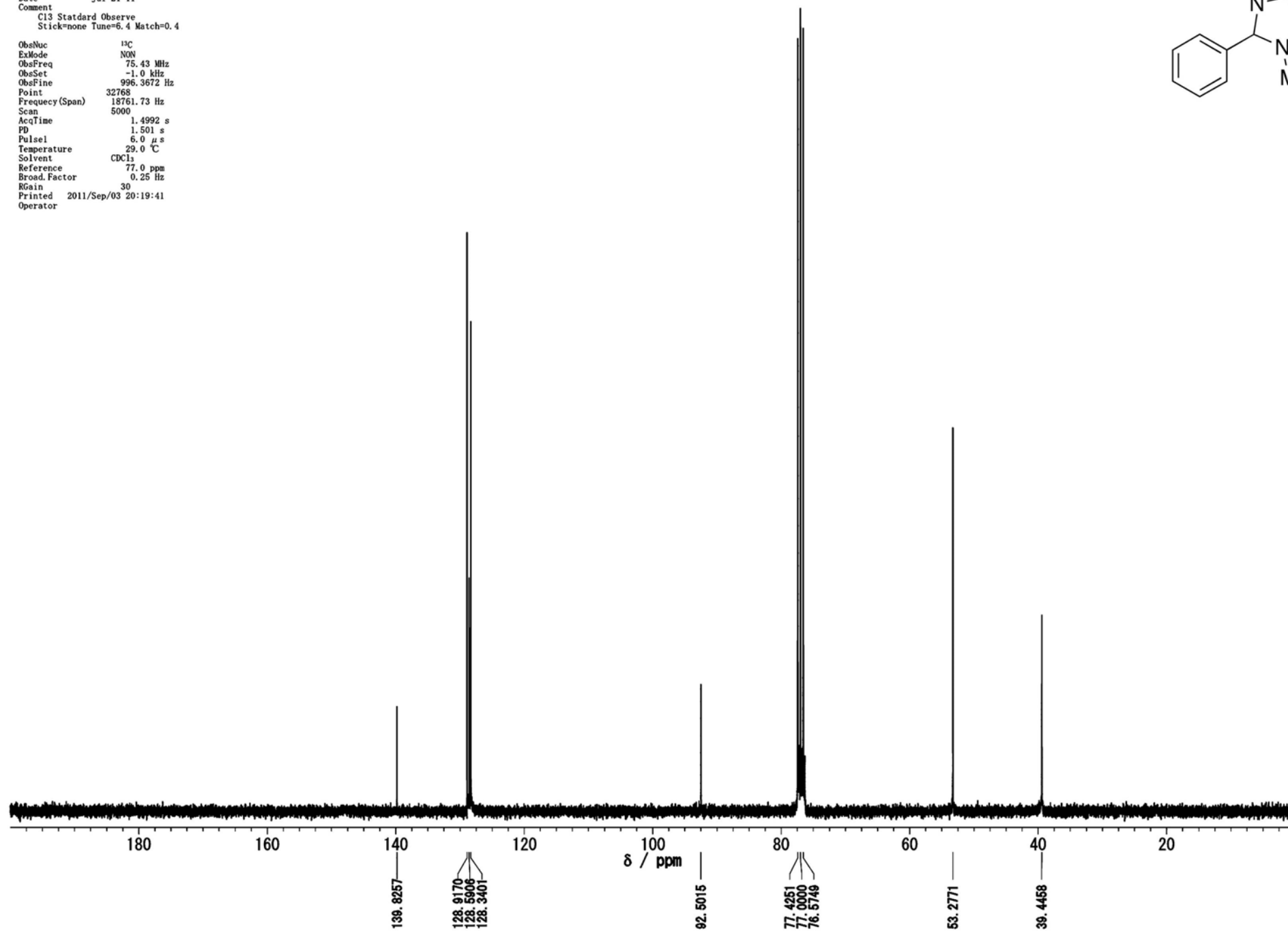
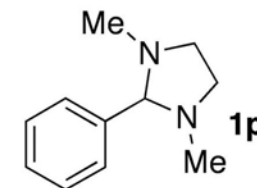
ObsNuc ¹³C
 ExMode NON
 ObsFreq 75.43 MHz
 ObsSet -1.0 kHz
 ObsFine 996.3672 Hz
 Point 32768
 Frequency (Span) 18761.73 Hz
 Scan 4000
 AcqTime 1.4992 s
 PD 1.501 s
 Pulsel 6.0 μ s
 Temperature 29.0 $^{\circ}$ C
 Solvent CDCl₃
 Reference 77.0 ppm
 Broad.Factor 0.2863 Hz
 RGain 30
 Printed 2011/Sep/03 20:16:56
 Operator



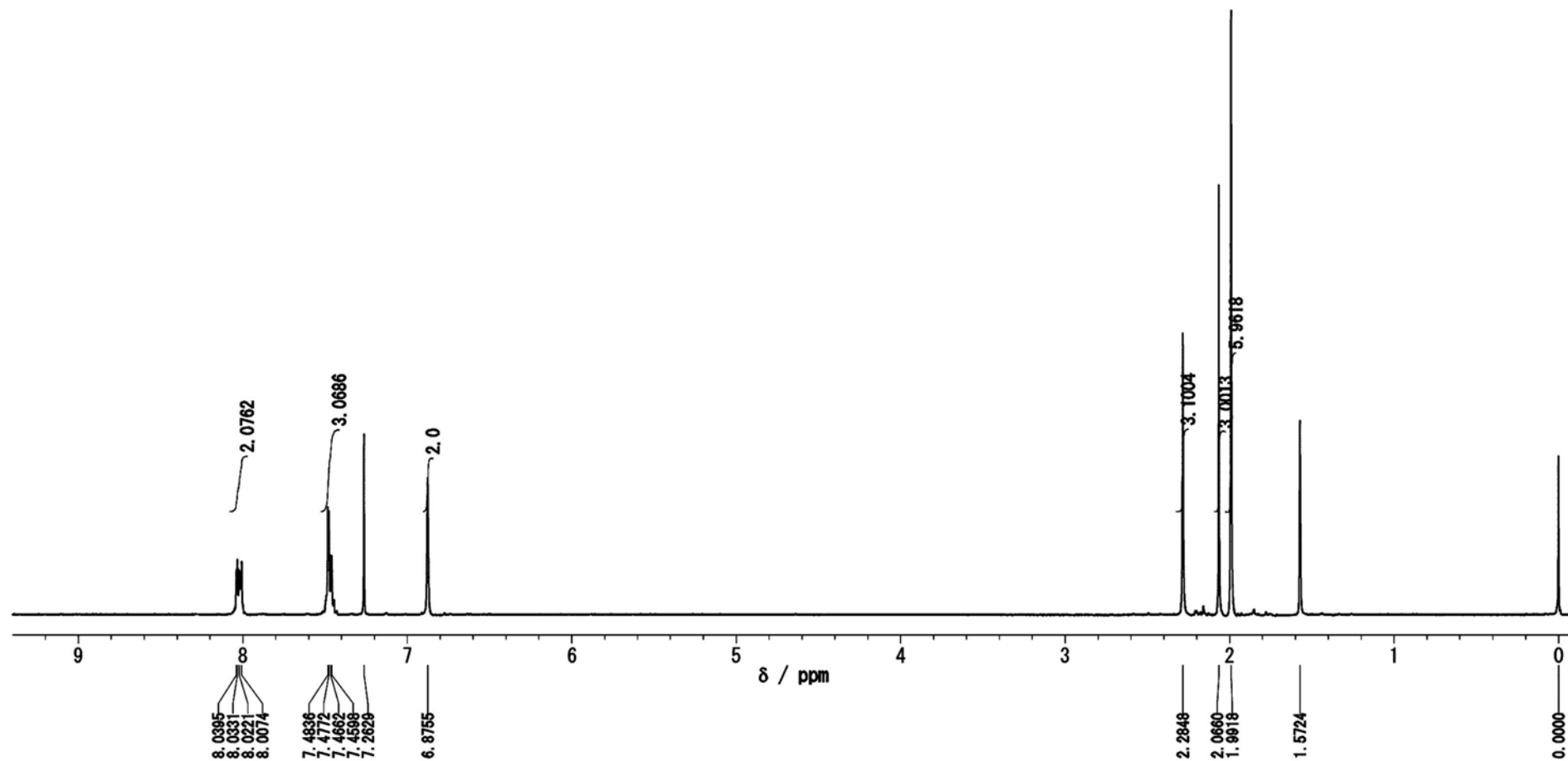
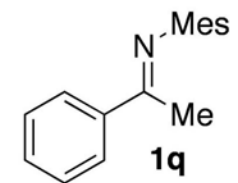
Original File:
 Date Jul 19 11
 Comment STANDARD 1H OBSERVE
 ObsNuc 1H
 ExMode NON
 ObsFreq 299.96 MHz
 ObsSet -1.0 kHz
 ObsPine 995.0047 Hz
 Point 16384
 Frequency (Span) 4500.45 Hz
 Scan 32
 AcqTime 3.4983 s
 PD 1.502 s
 Pulse 6.0 μ s
 Temperature 29.0 $^{\circ}$ C
 Solvent CDCl₃
 Reference 0.0 ppm
 Broad.Factor 0.1373 Hz
 RGain 18
 Printed 2011/Sep/03 20:18:04
 Operator



Date Jul 21 11
 Comment C13 Statdard Observe
 Stick=none Tune=6.4 Match=0.4
 ObsNuc ¹³C
 ExMode NGV
 ObsFreq 75.43 MHz
 ObsSet -1.0 kHz
 ObsFine 996.3672 Hz
 Point 32768
 Frequency (Span) 18761.73 Hz
 Scan 5000
 AcqTime 1.4992 s
 PD 1.501 s
 Pulse 6.0 μ s
 Temperature 29.0 $^{\circ}$ C
 Solvent CDCl₃
 Reference 77.0 ppm
 Broad. Factor 0.25 Hz
 RGain 30
 Printed 2011/Sep/03 20:19:41
 Operator



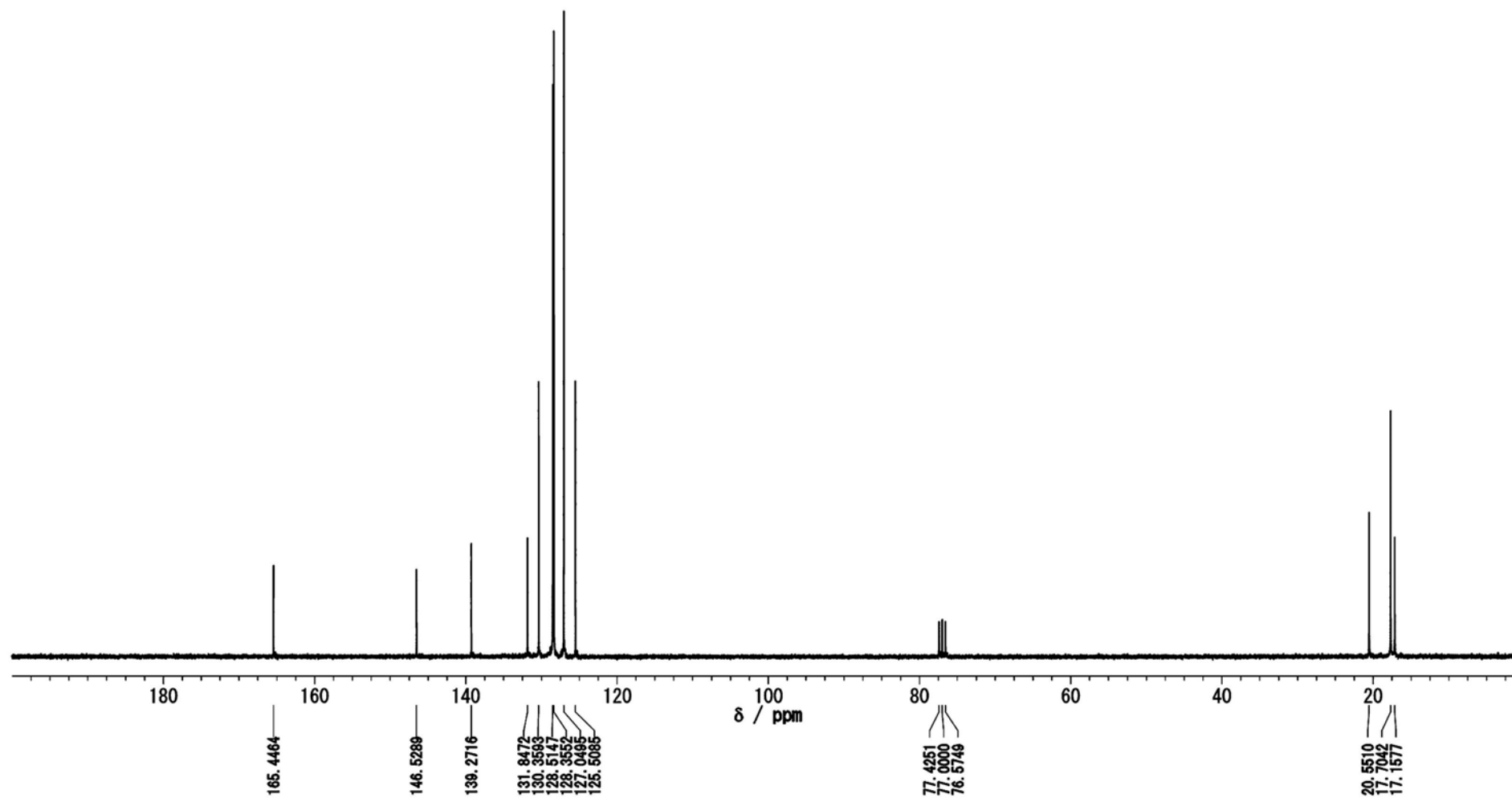
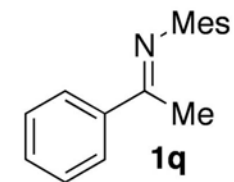
Original File: Jul 16 11
 Date: Jul 16 11
 Comment: STANDARD 1H OBSERVE
 ObsNuc: ¹H
 ExMode: NON
 ObsFreq: 299.96 MHz
 ObsSet: -1.0 kHz
 ObsPine: 995.0047 Hz
 Point: 16384
 Frequency (Span): 4500.45 Hz
 Scan: 16
 AcqTime: 3.4983 s
 PD: 1.502 s
 Pulse1: 6.0 μs
 Temperature: 29.0 °C
 Solvent: CDCl₃
 Reference: 0.0 ppm
 Broad Factor: 0.1373 Hz
 RGain: 24
 Printed: 2011/Jul/28 15:24:53
 Operator:



```

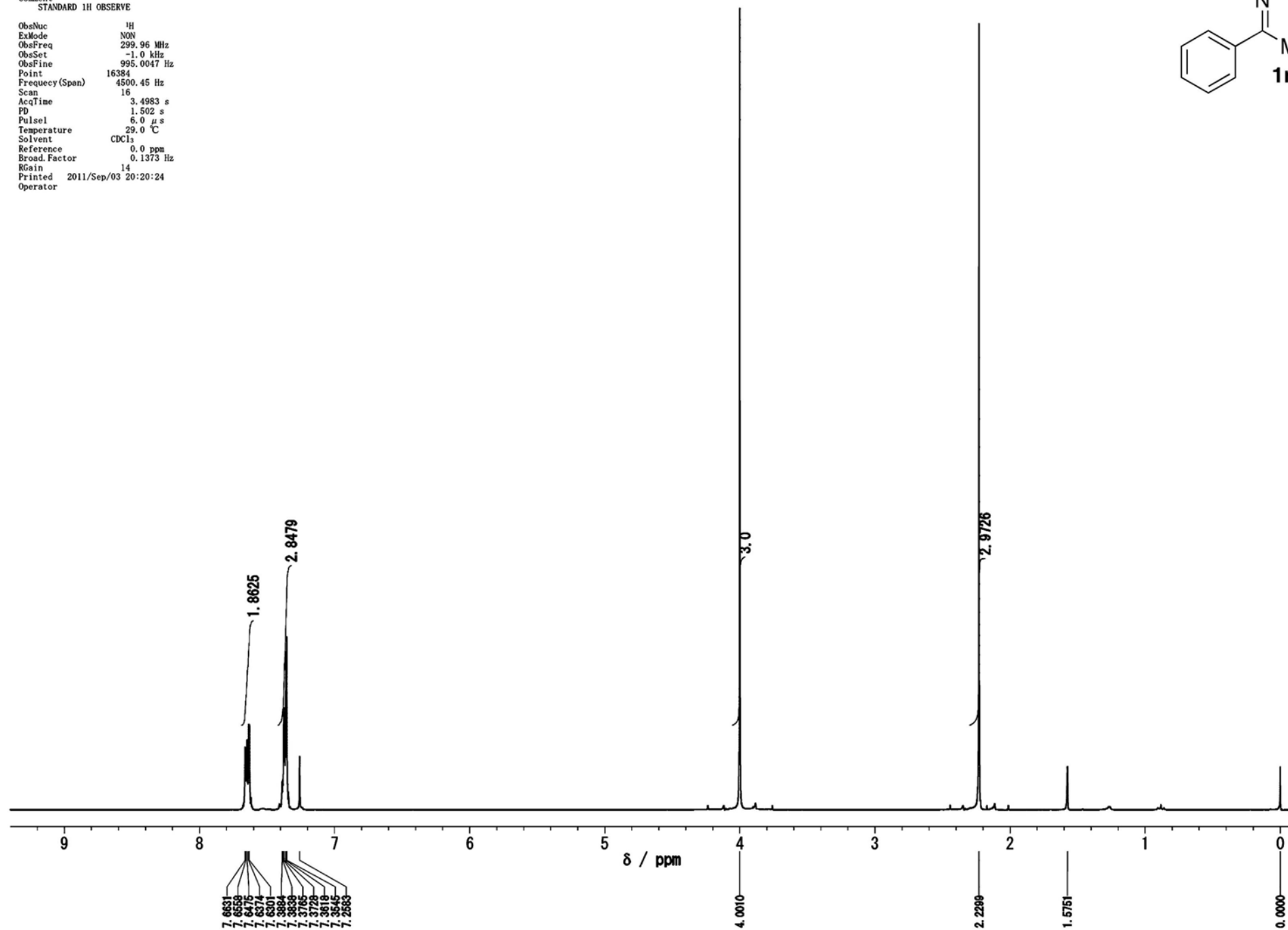
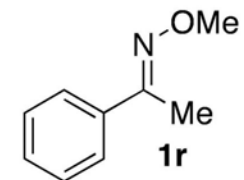
ObsNuc      13C
ExHmode      NON
ObsFreq      75.43 MHz
ObsSet      -1.0 kHz
ObsFine      996.3672 Hz
Point      32768
Frequency(Span) 18761.73 Hz
Scan      300
AcqTime      1.4992 s
PD      1.501 s
Puls1      6.0  $\mu$ s
Temperature  29.0  $^{\circ}$ C
Solvent      CDCl3
Reference    77.0 ppm
Broad_Factor 0.2863 Hz
RGain      30
Printed      2011/jul/28 15:37:56
Operator

```



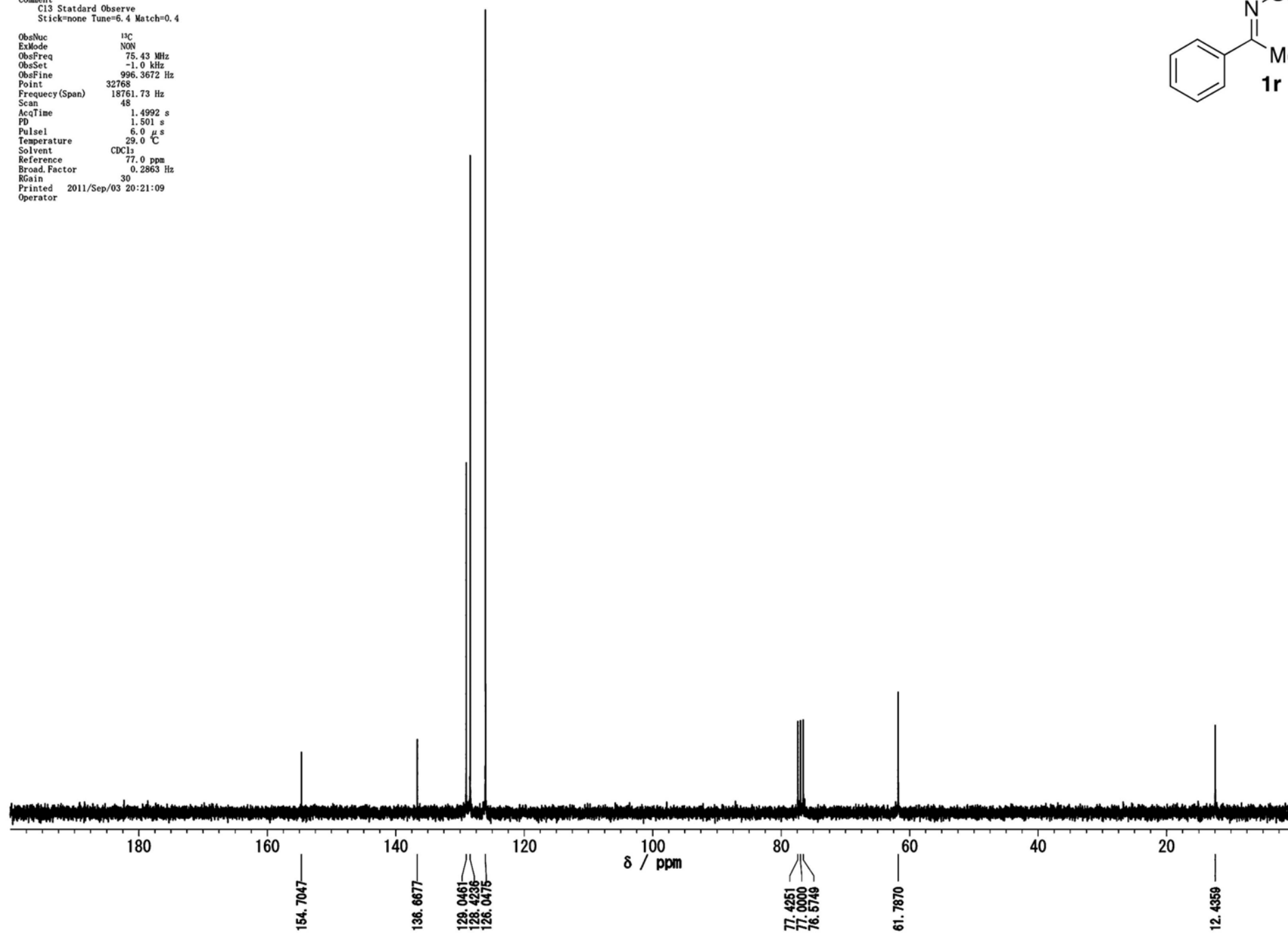
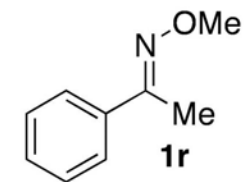
Original File:
Date 11/May/2016 00:00:00
Comment
STANDARD 1H OBSERVE

ObsNuc ¹H
ExMode NON
ObsFreq 299.96 MHz
ObsSet -1.0 kHz
ObsPine 995.0047 Hz
Point 16384
Frequency (Span) 4500.45 Hz
Scan 16
AcqTime 3.4983 s
PD 1.502 s
Pulse 6.0 μ s
Temperature 29.0 $^{\circ}$ C
Solvent CDCl₃
Reference 0.0 ppm
Broad Factor 0.1373 Hz
RGain 14
Printed 2011/Sep/03 20:20:24
Operator



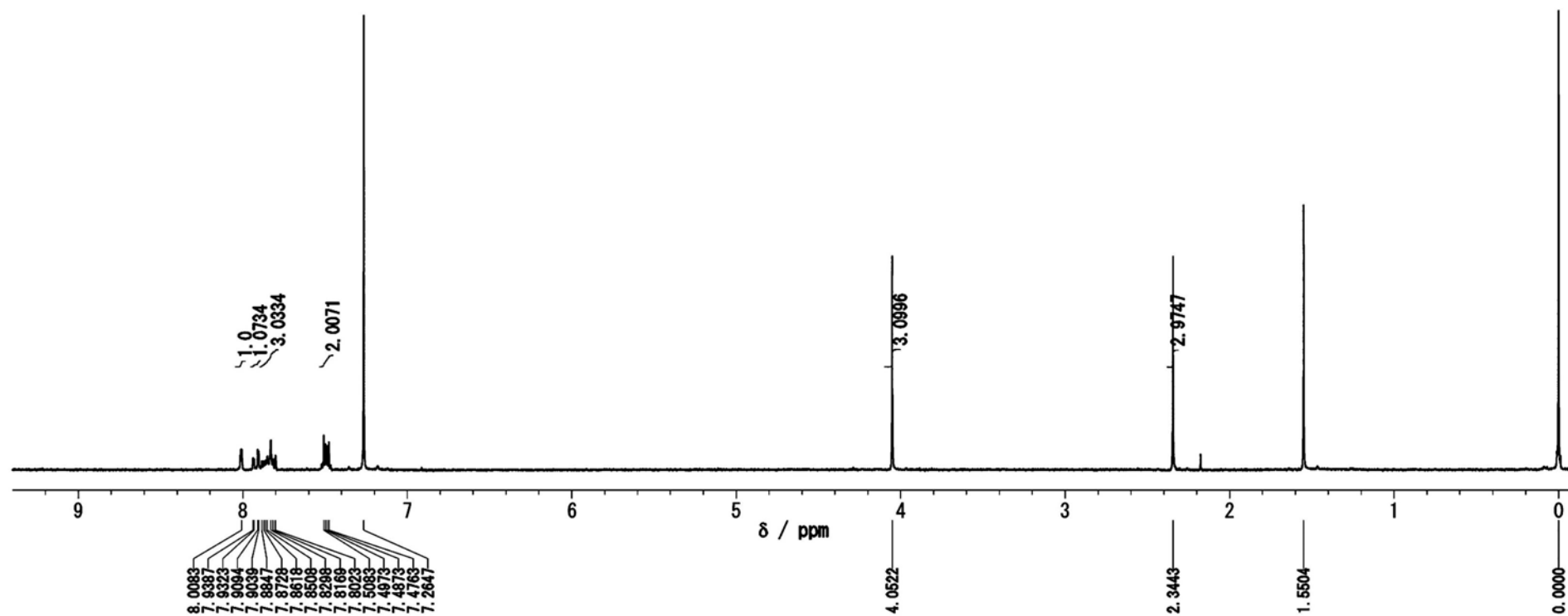
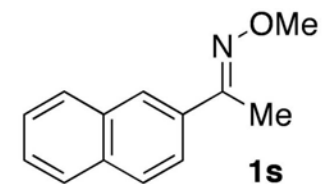
Original File:
 Date Sep 2 11
 Comment
 C13 Statdard Observe
 Stick=none Tune=6.4 Match=0.4

ObsNuc ^{13}C
 ExMode NON
 ObsFreq 75.43 MHz
 ObsSet -1.0 kHz
 ObsFine 996.3672 Hz
 Point 32768
 Frequency (Span) 18761.73 Hz
 Scan 48
 AcqTime 1.4992 s
 PD 1.501 s
 Pulse 6.0 μs
 Temperature 29.0 $^{\circ}\text{C}$
 Solvent CDCl_3
 Reference 77.0 ppm
 Broad Factor 0.2863 Hz
 RGain 30
 Printed 2011/Sep/03 20:21:09
 Operator



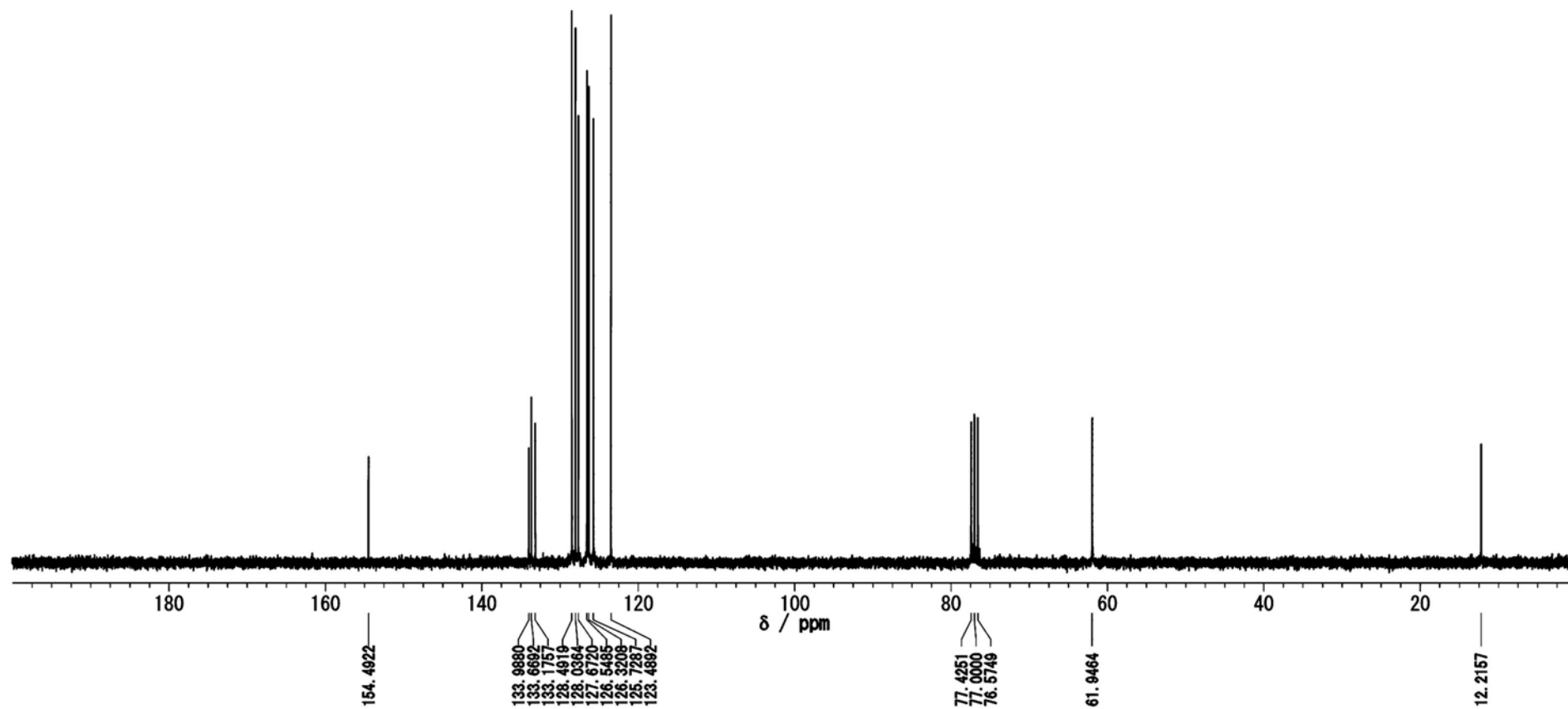
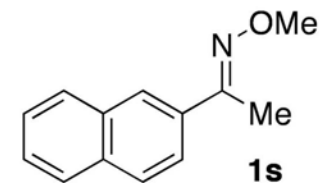
Original File:
 Date Jun 30 11
 Comment
 STANDARD 1H OBSERVE

ObsNuc	¹ H
ExMode	NON
ObsFreq	299.96 MHz
ObsSet	-1.0 kHz
ObsPine	995.0047 Hz
Point	16384
Frequency (Span)	4500.45 Hz
Scan	16
AcqTime	3.4983 s
PD	1.502 s
Pulse1	6.0 μ s
Temperature	29.0 $^{\circ}$ C
Solvent	CDCl ₃
Reference	0.0 ppm
Broad. Factor	0.1373 Hz
RGain	30
Printed	2011/Jul/28 15:33:11
Operator	



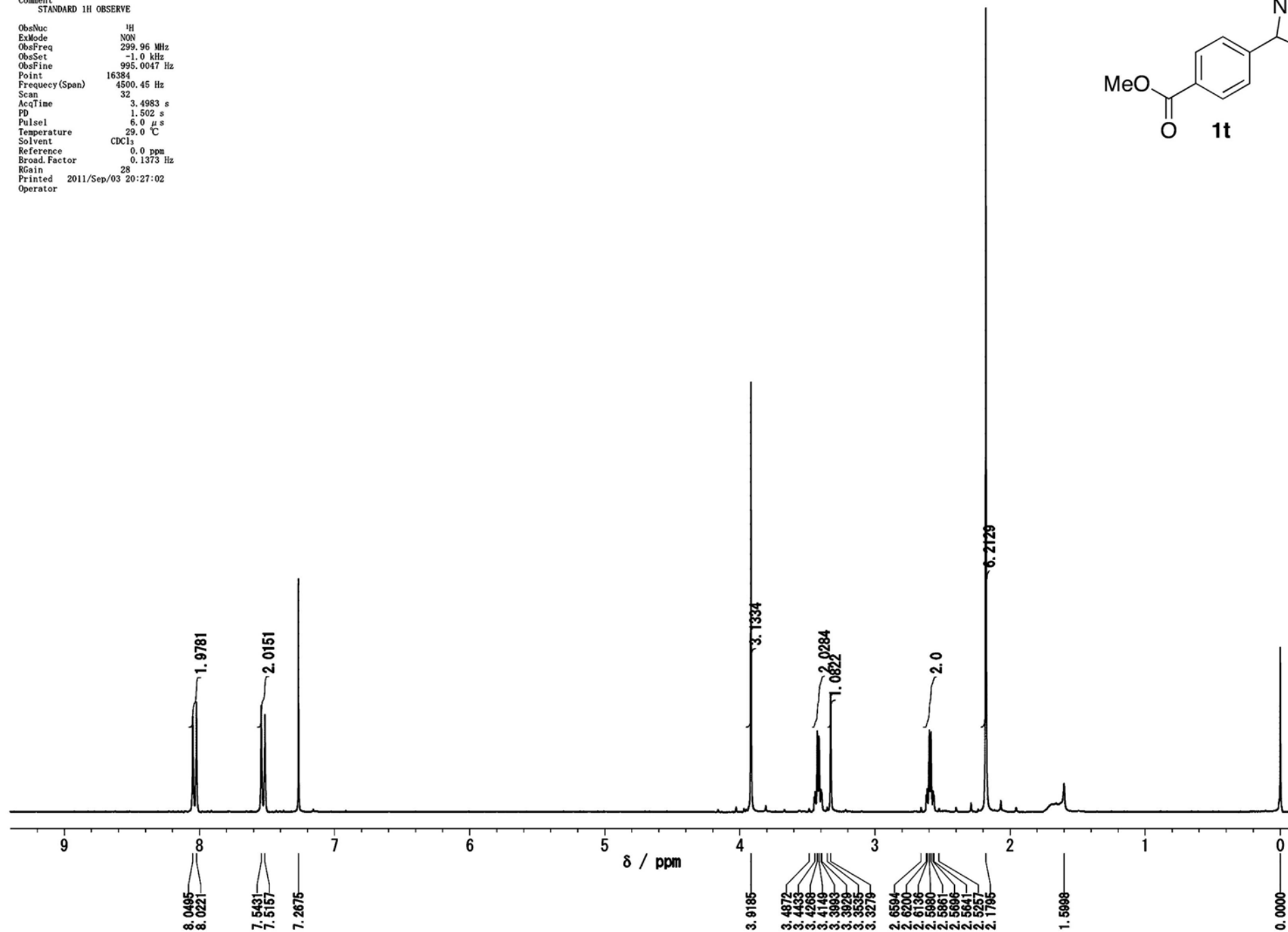
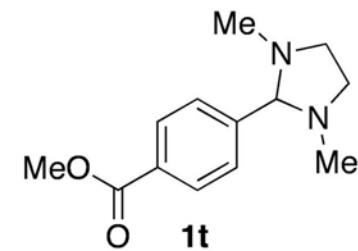
Original File:
 Date Jul 22 11
 Comment
 C13 Statdard Observe
 Stick=none Tune=6.4 Match=0.4

ObsNuc ¹³C
 ExMode NON
 ObsFreq 75.43 MHz
 ObsSet -1.0 kHz
 ObsFine 996.3672 Hz
 Point 32768
 Frequency (Span) 18761.73 Hz
 Scan 256
 AcqTime 1.4992 s
 PD 1.501 s
 Pulsel 6.0 μ s
 Temperature 29.0 $^{\circ}$ C
 Solvent CDCl₃
 Reference 77.0 ppm
 Broad Factor 0.2863 Hz
 RGain 30
 Printed 2011/Jul/28 15:35:54
 Operator



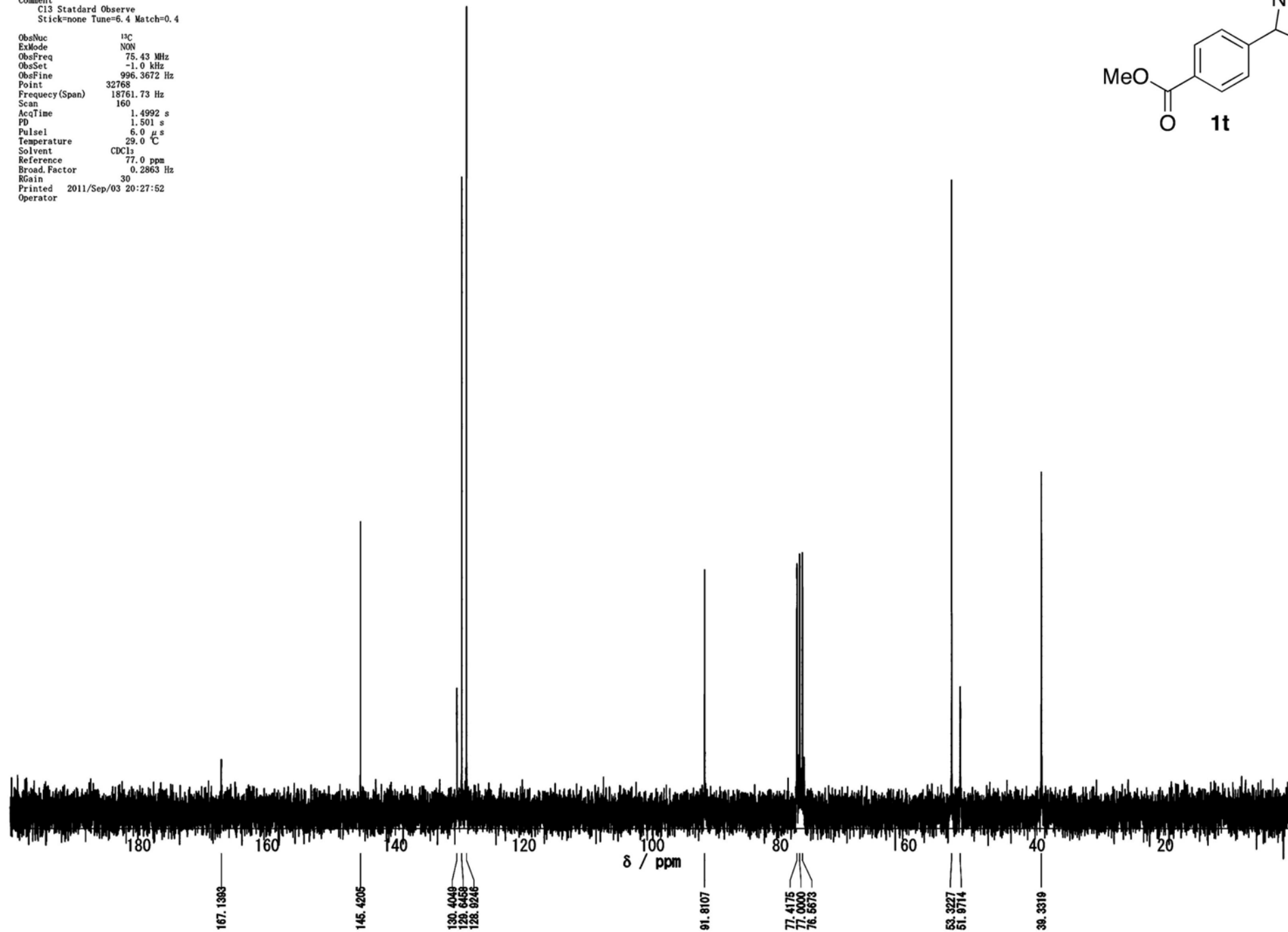
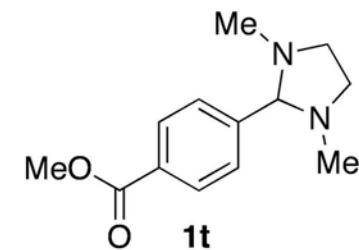
Original File:
 Date Aug 30 11
 Comment
 STANDARD 1H OBSERVE

ObsNuc	¹ H
ExMode	NON
ObsFreq	299.96 MHz
ObsSet	-1.0 kHz
ObsPine	995.0047 Hz
Point	16384
Frequency (Span)	4500.45 Hz
Scan	32
AcqTime	3.4983 s
PD	1.502 s
Pulse1	6.0 μs
Temperature	29.0 °C
Solvent	CDCl ₃
Reference	0.0 ppm
Broad. Factor	0.1373 Hz
RGain	28
Printed	2011/Sep/03 20:27:02
Operator	



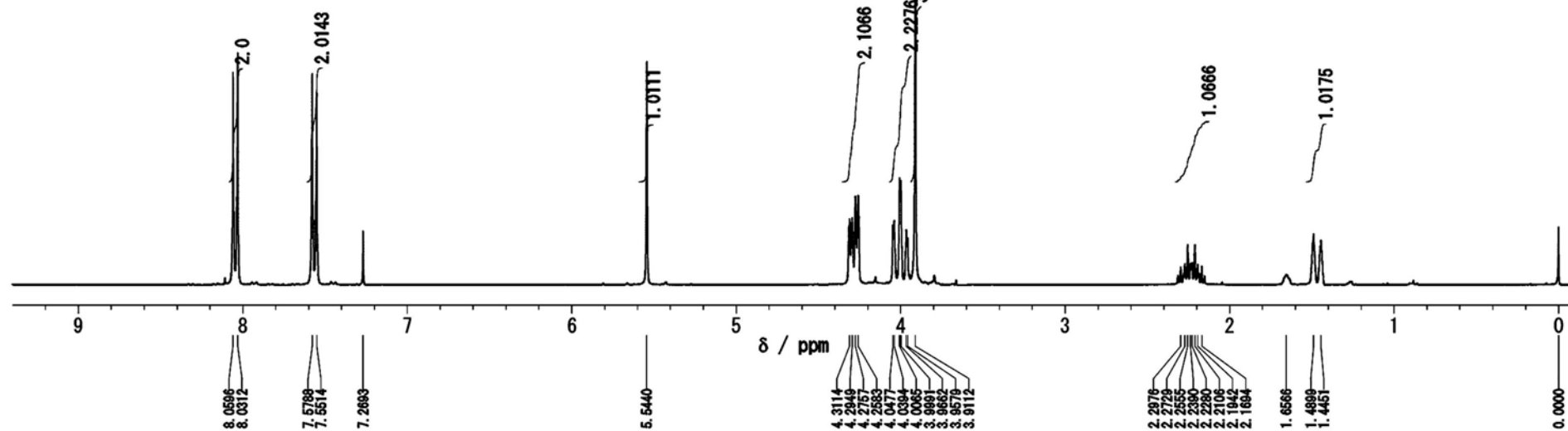
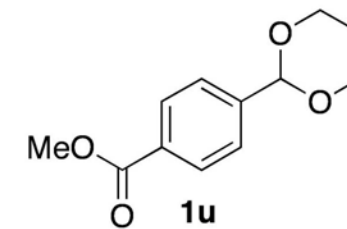
Original File:
 Date Aug 30 11
 Comment
 C13 Statdard Observe
 Stick=none Tune=6.4 Match=0.4

ObsNuc ¹³C
 ExMode NON
 ObsFreq 75.43 MHz
 ObsSet -1.0 kHz
 ObsFine 996.3672 Hz
 Point 32768
 Frequency (Span) 18761.73 Hz
 Scan 160
 AcqTime 1.4992 s
 PD 1.501 s
 Pulsel 6.0 μ s
 Temperature 29.0 $^{\circ}$ C
 Solvent CDCl₃
 Reference 77.0 ppm
 Broad.Factor 0.2863 Hz
 RGain 30
 Printed 2011/Sep/03 20:27:52
 Operator



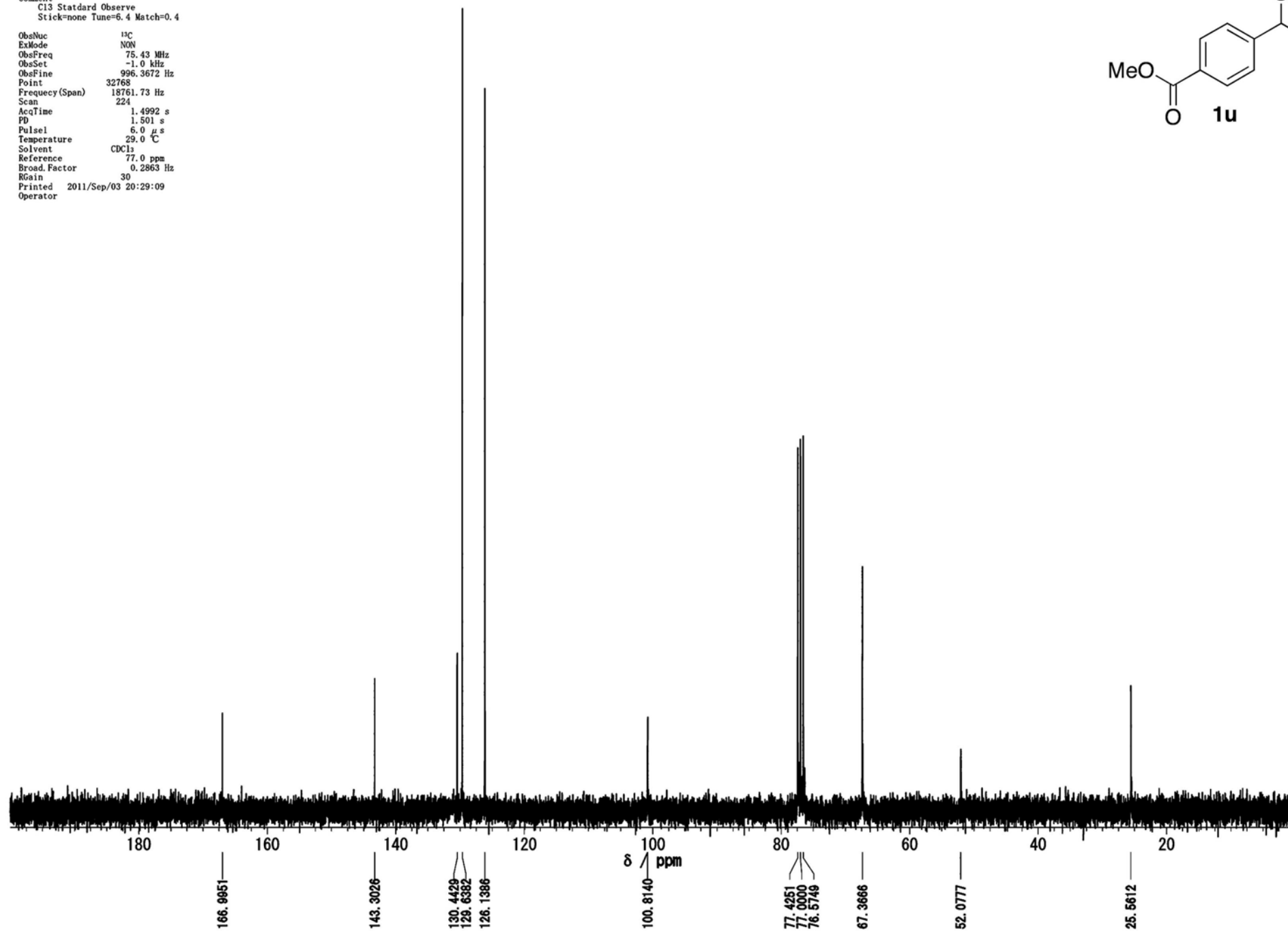
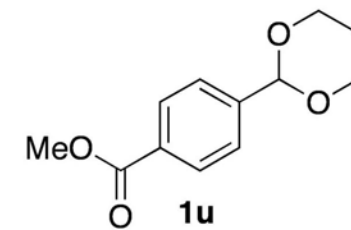
Original File:
 Date Aug 31 11
 Comment
 STANDARD 1H OBSERVE

ObsNuc ¹H
 ExMode NON
 ObsFreq 299.96 MHz
 ObsSet -1.0 kHz
 ObsPine 995.0047 Hz
 Point 16384
 Frequency (Span) 4500.45 Hz
 Scan 16
 AcqTime 3.4983 s
 PD 1.502 s
 Pulse 6.0 μ s
 Temperature 29.0 $^{\circ}$ C
 Solvent CDCl₃
 Reference 0.0 ppm
 Broad Factor 0.1373 Hz
 RGain 12
 Printed 2011/Sep/03 20:29:56
 Operator



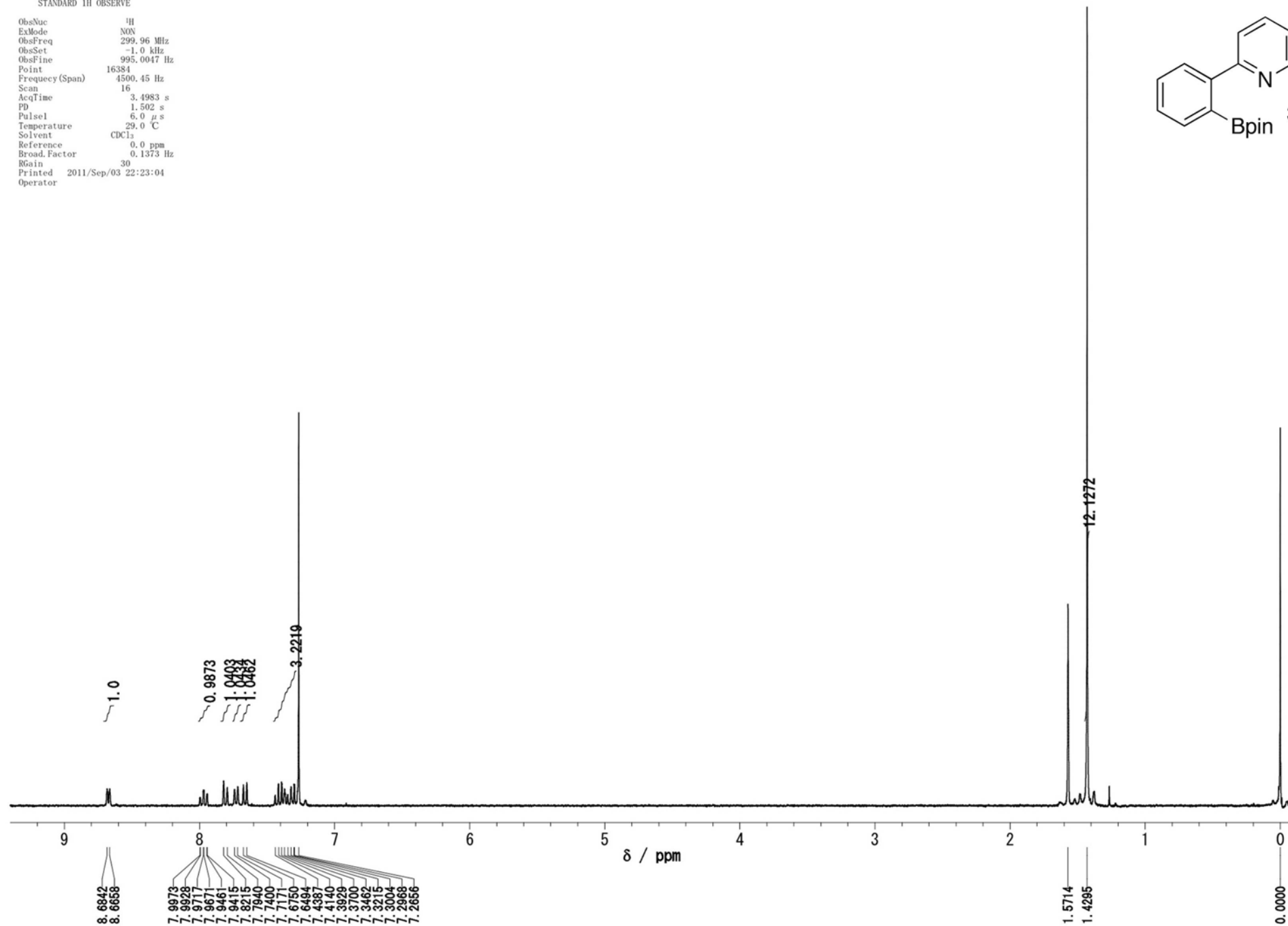
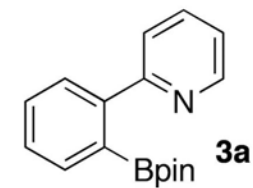
Original File:
 Date Aug 31 11
 Comment
 C13 Statdard Observe
 Stick=none Tune=6.4 Match=0.4

ObsNuc ¹³C
 ExMode NON
 ObsFreq 75.43 MHz
 ObsSet -1.0 kHz
 ObsFine 996.3672 Hz
 Point 32768
 Frequency (Span) 18761.73 Hz
 Scan 224
 AcqTime 1.4992 s
 PD 1.501 s
 Pulse 6.0 μs
 Temperature 29.0 °C
 Solvent CDCl₃
 Reference 77.0 ppm
 Broad Factor 0.2863 Hz
 RGain 30
 Printed 2011/Sep/03 20:29:09
 Operator



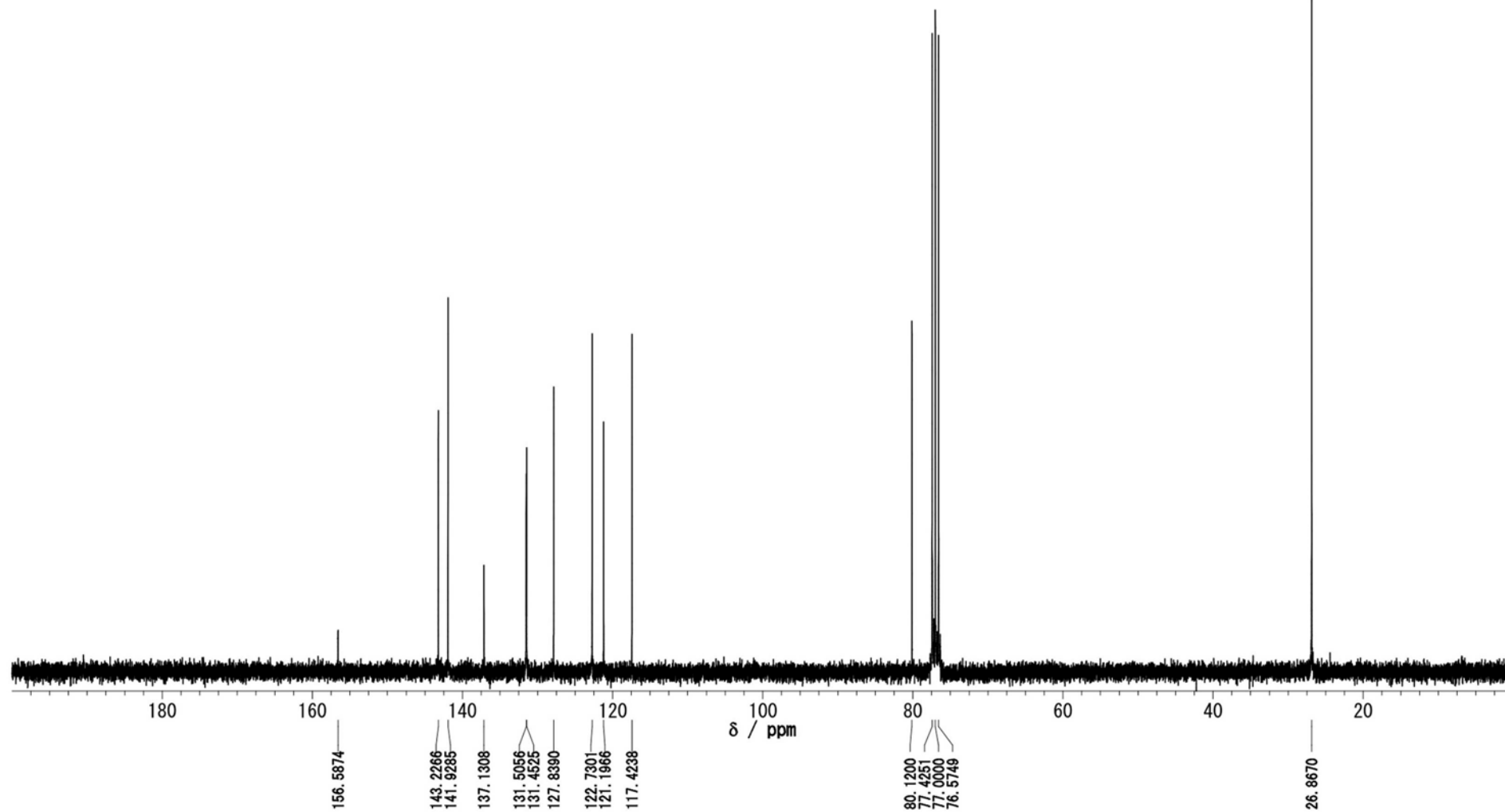
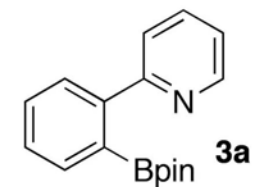
Original File:
 Date Jul 11 11
 Comment
 STANDARD 1H OBSERVE

ObsNuc	1H
ExMode	NON
ObsFreq	299.96 MHz
ObsSet	-1.0 kHz
ObsFine	995.0047 Hz
Point	16384
Frequency (Span)	4500.45 Hz
Scan	16
AcqTime	3.4983 s
PD	1.502 s
Pulse1	6.0 μ s
Temperature	29.0 $^{\circ}$ C
Solvent	CDCl ₃
Reference	0.0 ppm
Broad. Factor	0.1373 Hz
RGain	30
Printed	2011/Sep/03 22:23:04
Operator	



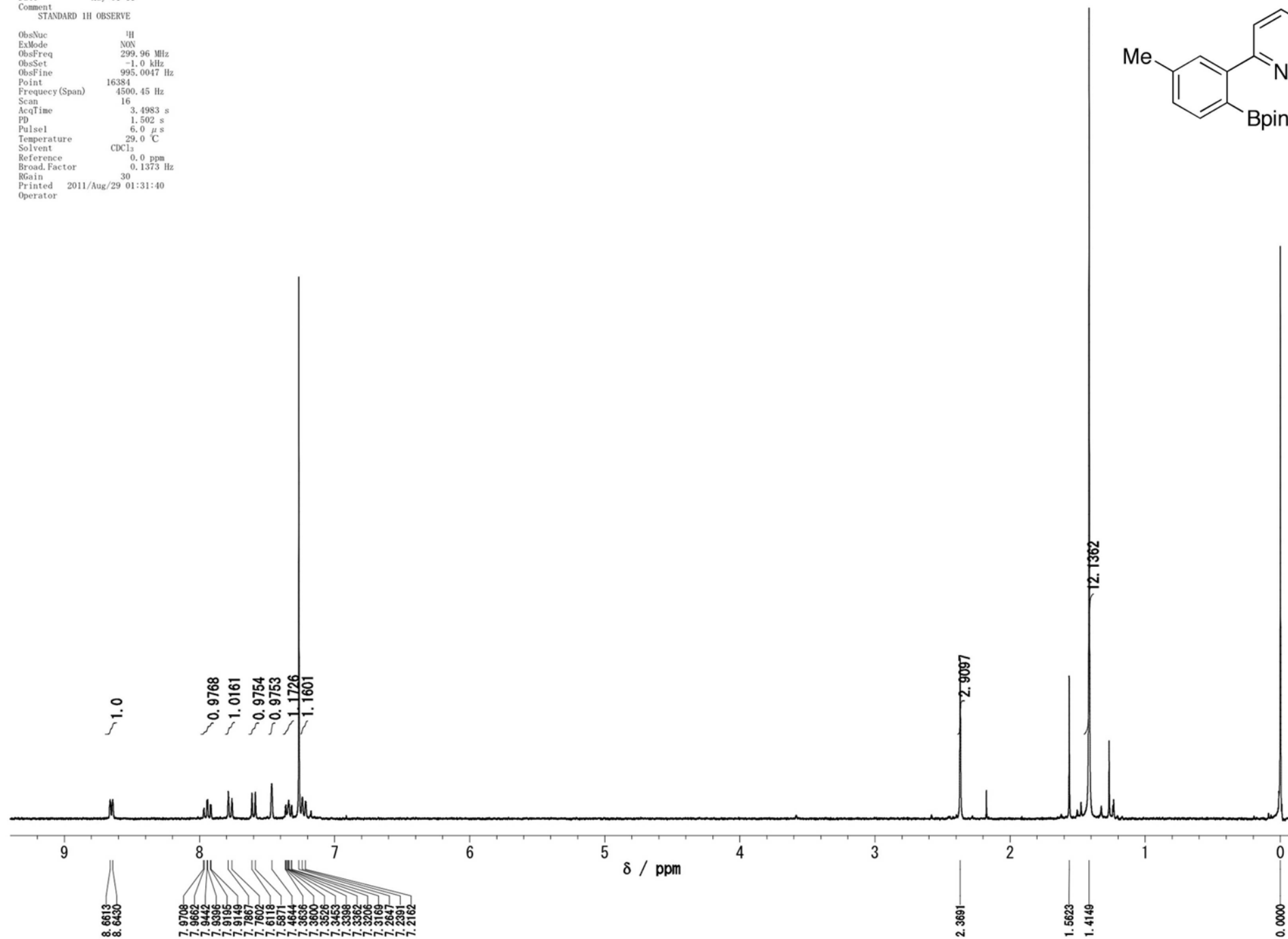
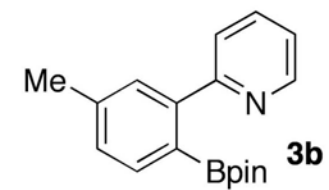
Original File:
Date Jul 11 11
Comment
C13 Statdard Observe
Stick=none Tune=6.4 Match=0.4

ObsNuc ^{13}C
ExMode NON
ObsFreq 75.43 MHz
ObsSet -1.0 kHz
ObsFine 996.3672 Hz
Point 32768
Frequency (Span) 18761.73 Hz
Scan 896
AcqTime 1.4992 s
PD 1.501 s
Pulse1 6.0 μs
Temperature 29.0 $^{\circ}\text{C}$
Solvent CDCl_3
Reference 77.0 ppm
Broad.Factor 0.2863 Hz
RGain 30
Printed 2011/Aug/29 01:29:50
Operator



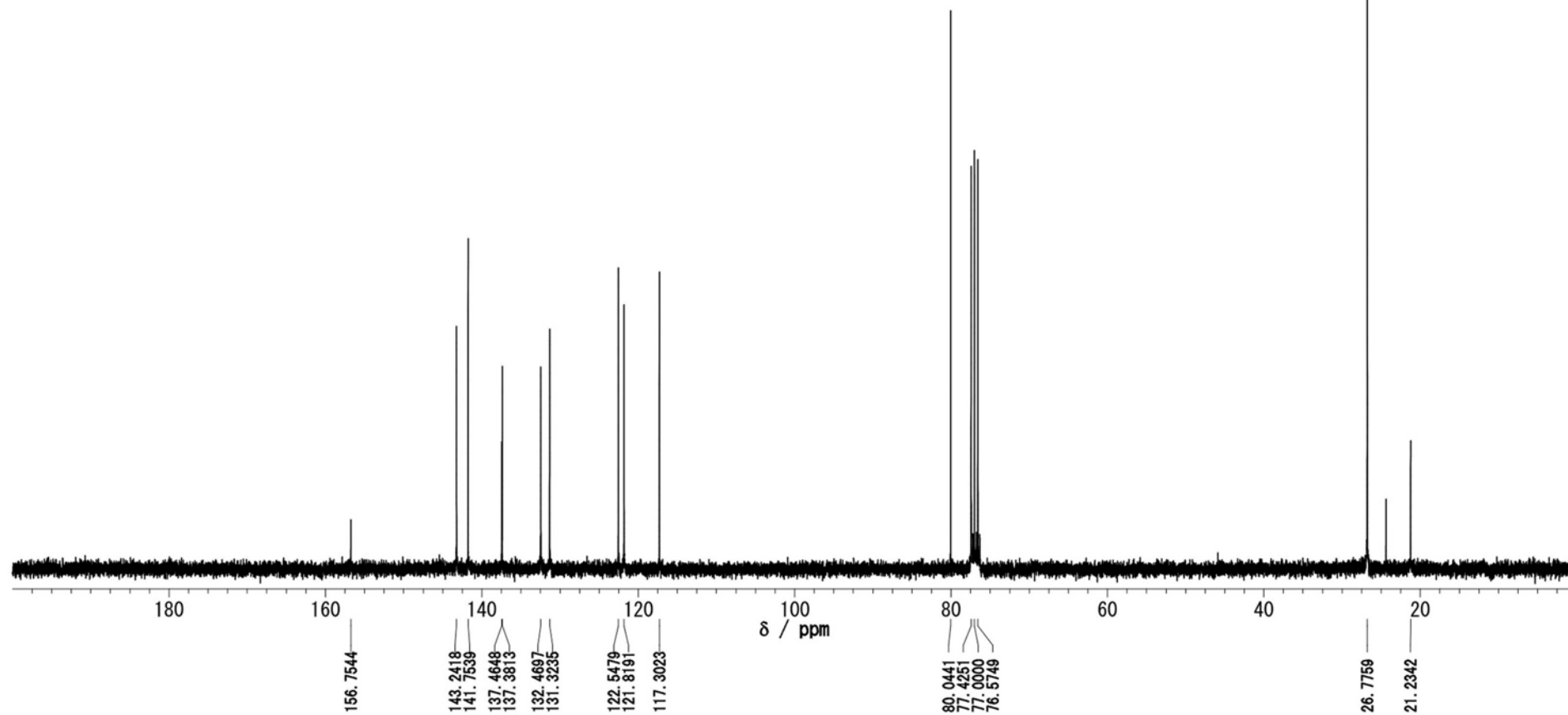
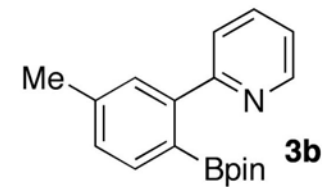
Original File:
 Date May 31 11
 Comment
 STANDARD 1H OBSERVE

ObsNuc	¹ H
ExMode	NON
ObsFreq	299.96 MHz
ObsSet	-1.0 kHz
ObsFine	995.0047 Hz
Point	16384
Frequency (Span)	4500.45 Hz
Scan	16
AcqTime	3.4983 s
PD	1.502 s
Pulse1	6.0 μ s
Temperature	29.0 $^{\circ}$ C
Solvent	CDCl ₃
Reference	0.0 ppm
Broad.Factor	0.1373 Hz
RGain	30
Printed	2011/Aug/29 01:31:40
Operator	

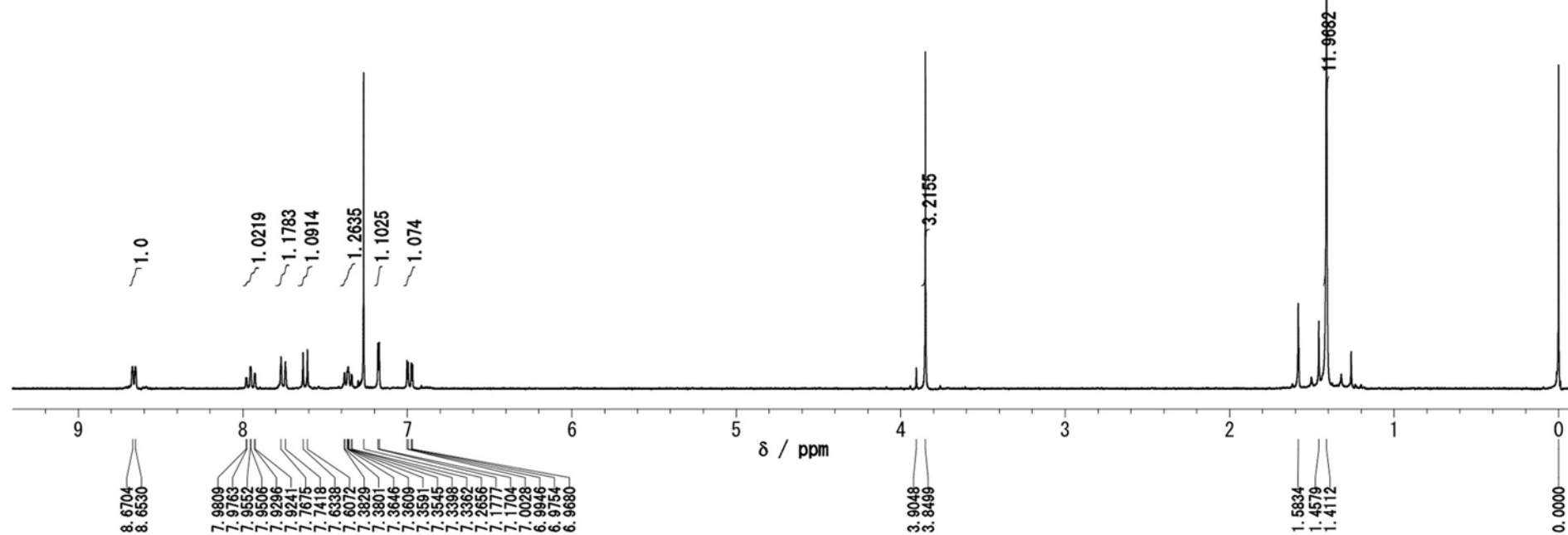
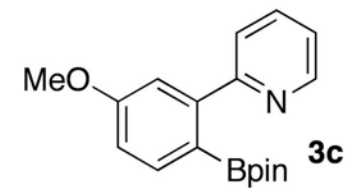


Original File:
 Date Aug 27 11
 Comment
 C13 Statdard Observe
 Stick=none Tune=6.4 Match=0.4

ObsNuc ^{13}C
 ExMode NON
 ObsFreq 75.43 MHz
 ObsSet -1.0 kHz
 ObsFine 996.3672 Hz
 Point 32768
 Frequency (Span) 18761.73 Hz
 Scan 912
 AcqTime 1.4992 s
 PD 1.501 s
 Pulse1 6.0 μs
 Temperature 29.0 $^{\circ}\text{C}$
 Solvent CDCl_3
 Reference 77.0 ppm
 Broad.Factor 0.2863 Hz
 RGain 30
 Printed 2011/Aug/29 01:33:37
 Operator

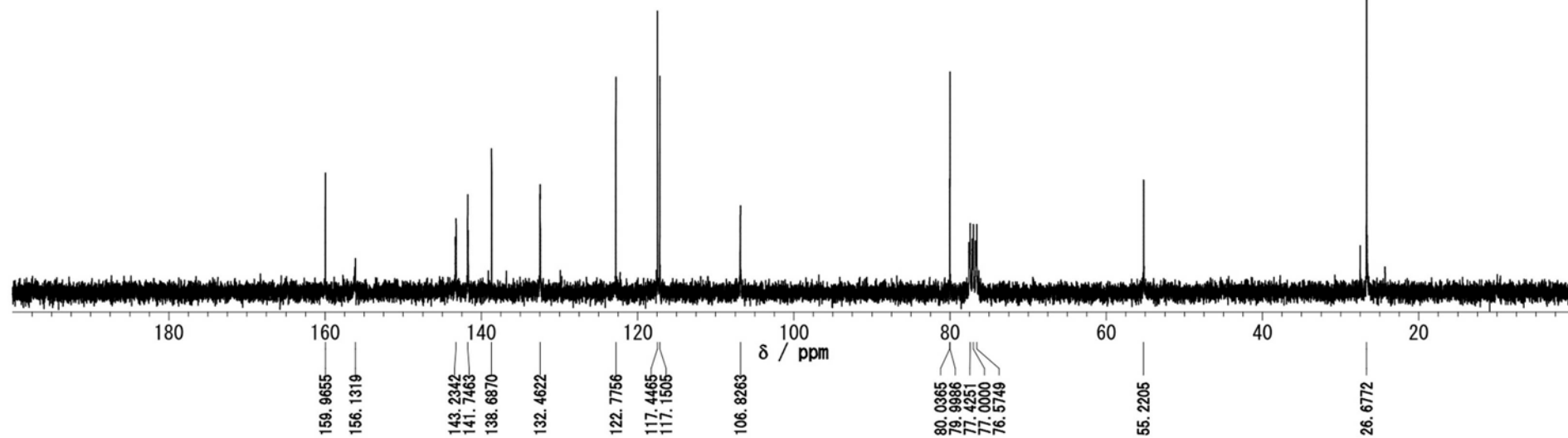
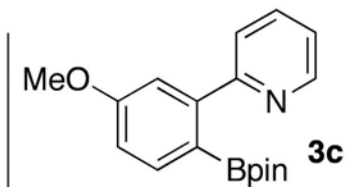


Original File: Jul 7 11
 Date: Jul 7 11
 Comment: STANDARD 1H OBSERVE
 ObsNuc: ¹H
 ExMode: NON
 ObsFreq: 299.96 MHz
 ObsSet: -1.0 kHz
 ObsFine: 995.0047 Hz
 Point: 16384
 Frequency (Span): 4500.45 Hz
 Scan: 16
 AcqTime: 3.4983 s
 PD: 1.502 s
 Pulse1: 6.0 μ s
 Temperature: 29.0 $^{\circ}$ C
 Solvent: CDCl₃
 Reference: 0.0 ppm
 Broad.Factor: 0.1373 Hz
 RGain: 30
 Printed: 2011/Aug/29 01:35:33
 Operator:



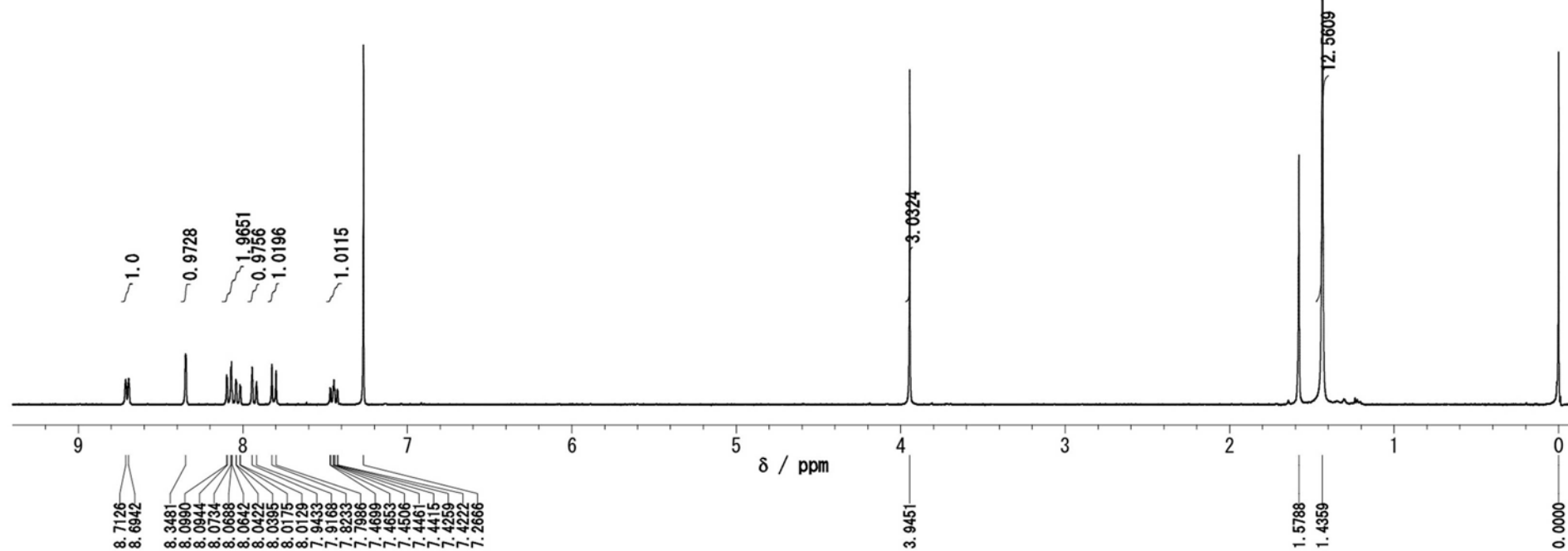
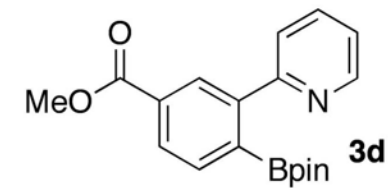
Original File:
 Date Aug 27 11
 Comment
 C13 Statdard Observe
 Stick=none Tune=6.4 Match=0.4

ObsNuc ^{13}C
 ExMode NON
 ObsFreq 75.43 MHz
 ObsSet -1.0 kHz
 ObsFine 996.3672 Hz
 Point 32768
 Frequency (Span) 18761.73 Hz
 Scan 80
 AcqTime 1.4992 s
 PD 1.501 s
 Pulse 6.0 μs
 Temperature 29.0 $^{\circ}\text{C}$
 Solvent CDCl_3
 Reference 77.0 ppm
 Broad.Factor 0.2863 Hz
 RGain 30
 Printed 2011/Aug/29 01:37:38
 Operator

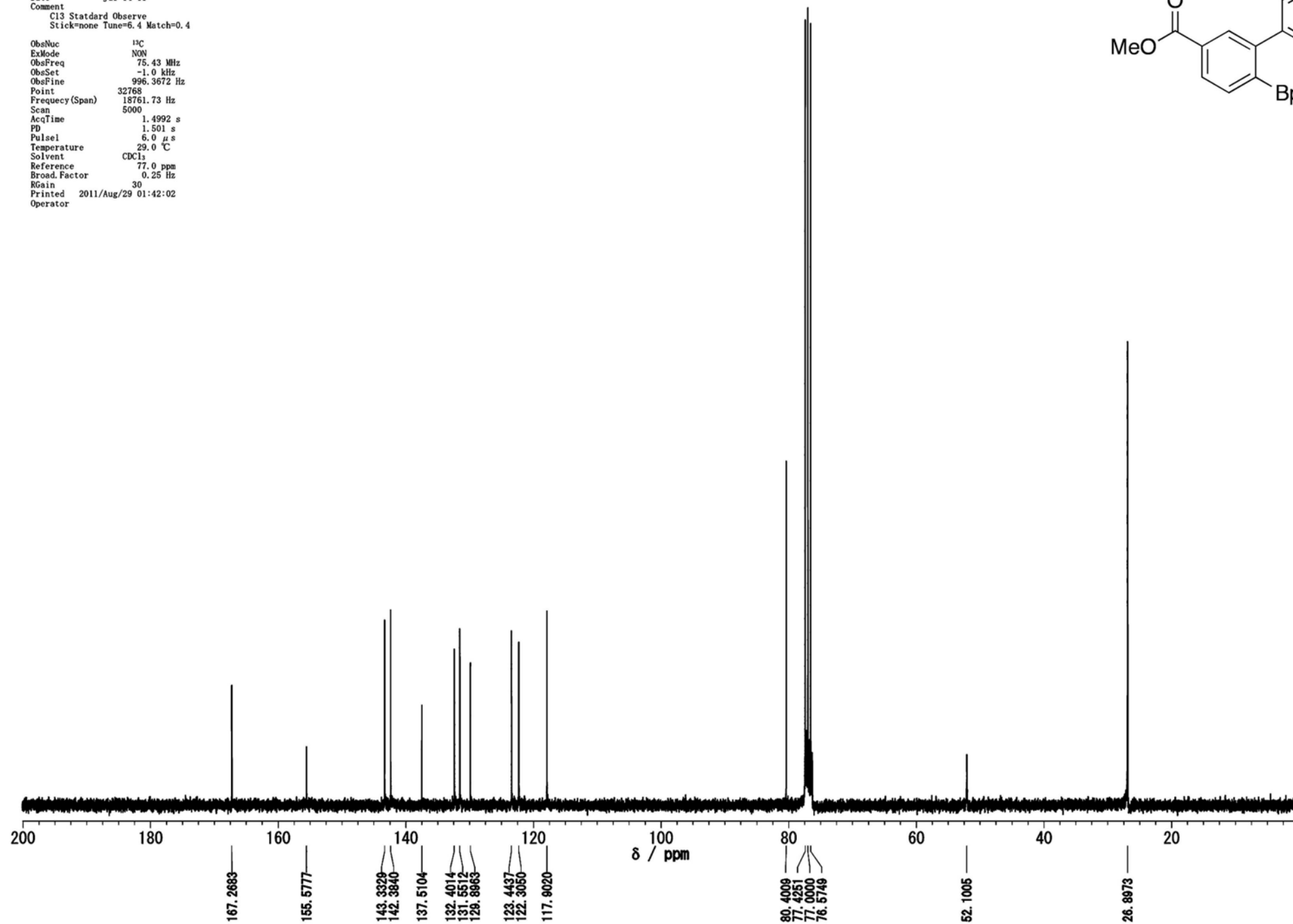
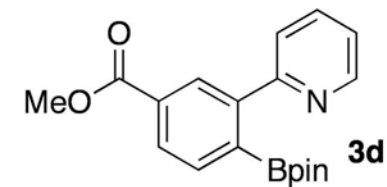


Original File:
 Date Jul 13 11
 Comment
 STANDARD 1H OBSERVE

ObsNuc	¹ H
ExMode	NON
ObsFreq	299.96 MHz
ObsSet	-1.0 kHz
ObsFine	995.0047 Hz
Point	16384
Frequency (Span)	4500.45 Hz
Scan	32
AcqTime	3.4983 s
PD	1.502 s
Pulse1	6.0 μ s
Temperature	29.0 $^{\circ}$ C
Solvent	CDCl ₃
Reference	0.0 ppm
Broad. Factor	0.1373 Hz
RGain	30
Printed	2011/Aug/29 01:39:31
Operator	

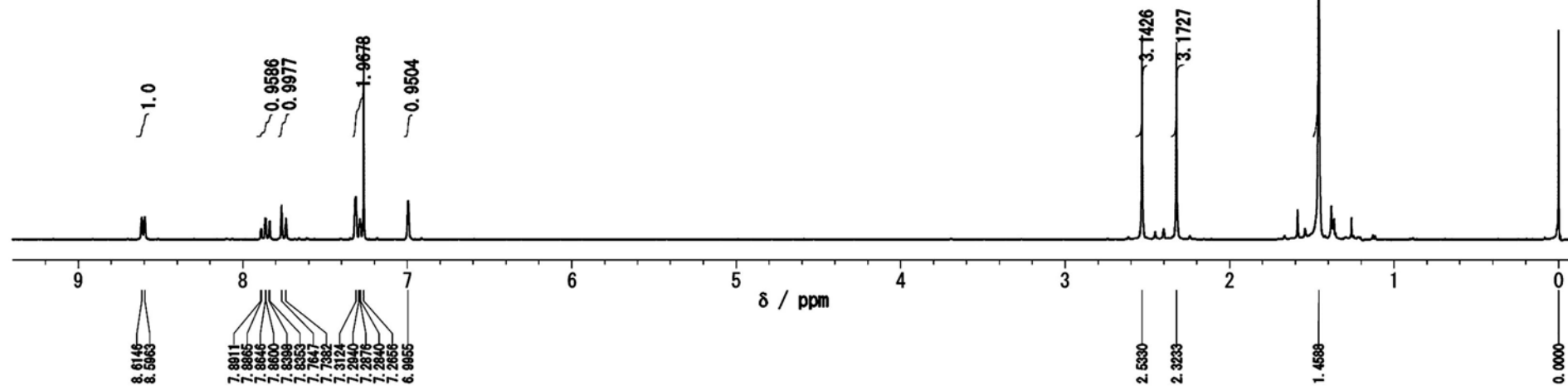
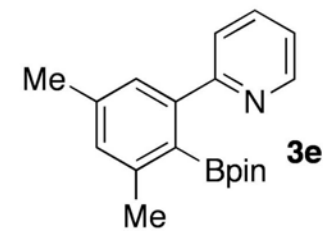


Date Jul 14 11
 Comment C13 Statdard Observe
 Stick=none Tune=6.4 Match=0.4
 ObsNuc ¹³C
 ExMode NQV
 ObsFreq 75.43 MHz
 ObsSet -1.0 kHz
 ObsFine 996.3672 Hz
 Point 32768
 Frequency (Span) 18761.73 Hz
 Scan 5000
 AcqTime 1.4992 s
 PD 1.501 s
 Pulse 6.0 μ s
 Temperature 29.0 $^{\circ}$ C
 Solvent CDCl₃
 Reference 77.0 ppm
 Broad Factor 0.25 Hz
 RGain 30
 Printed 2011/Aug/29 01:42:02
 Operator



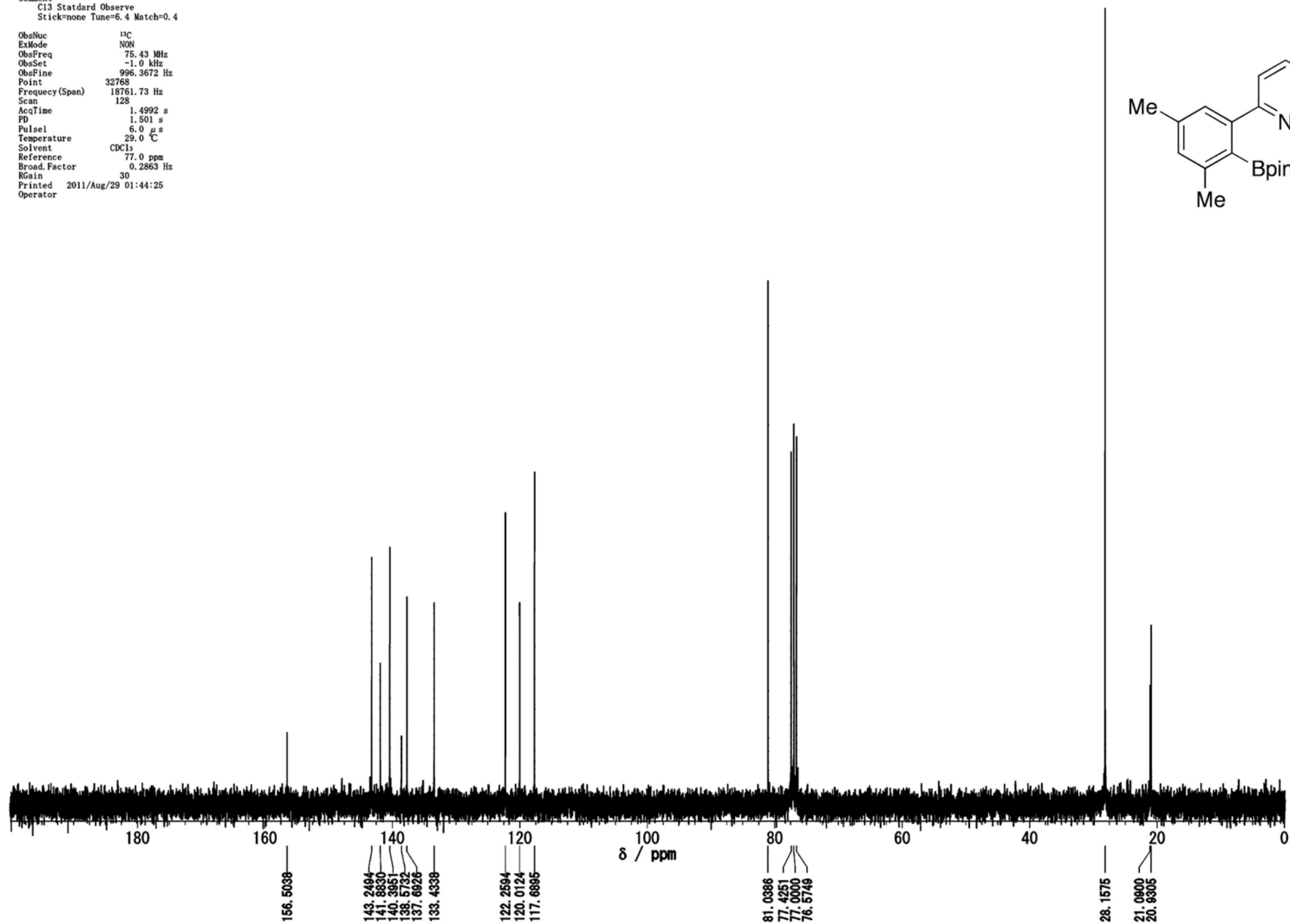
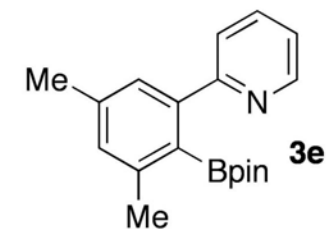
Original File:
 Date May 30 11
 Comment
 STANDARD 1H OBSERVE

ObsNuc ¹H
 ExMode NON
 ObsFreq 299.96 MHz
 ObsSet -1.0 kHz
 ObsPine 995.0047 Hz
 Point 16384
 Frequency (Span) 4500.45 Hz
 Scan 16
 AcqTime 3.4983 s
 PD 1.502 s
 Pulse 6.0 μ s
 Temperature 29.0 $^{\circ}$ C
 Solvent CDCl₃
 Reference 0.0 ppm
 Broad. Factor 0.1373 Hz
 RGain 24
 Printed 2011/Aug/29 01:43:02
 Operator



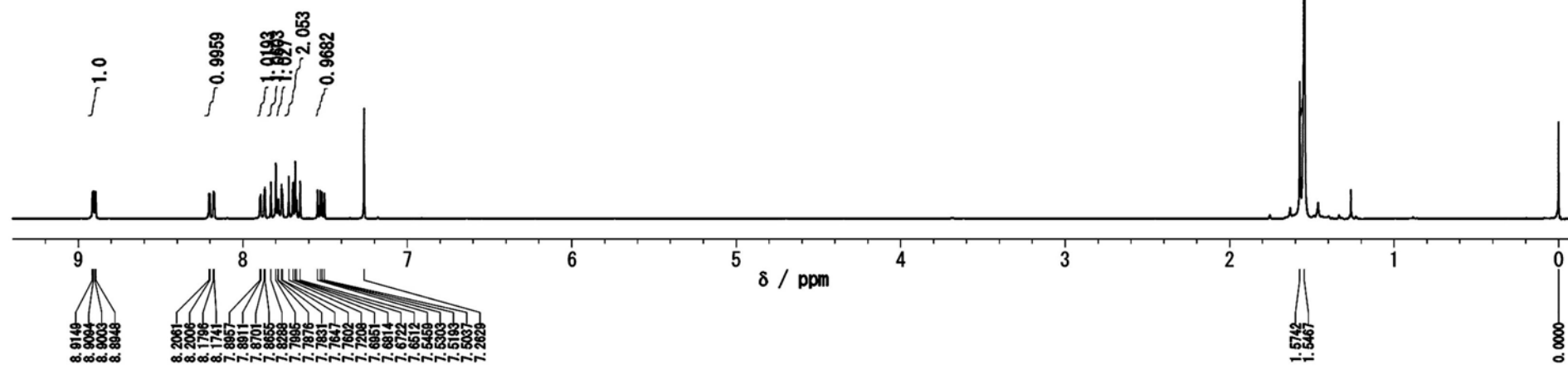
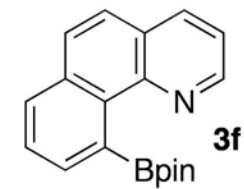
Original File:
 Date Aug 27 11
 Comment
 C13 Statdard Observe
 Stick=none Tune=6.4 Match=0.4

ObsNuc ¹³C
 ExMode NON
 ObsFreq 75.43 MHz
 ObsSet -1.0 kHz
 ObsFine 996.3672 Hz
 Point 32768
 Frequency (Span) 18761.73 Hz
 Scan 128
 AcqTime 1.4992 s
 PD 1.501 s
 Pulse 6.0 μ s
 Temperature 29.0 $^{\circ}$ C
 Solvent CDCl₃
 Reference 77.0 ppm
 Broad Factor 0.2863 Hz
 RGain 30
 Printed 2011/Aug/29 01:44:25
 Operator



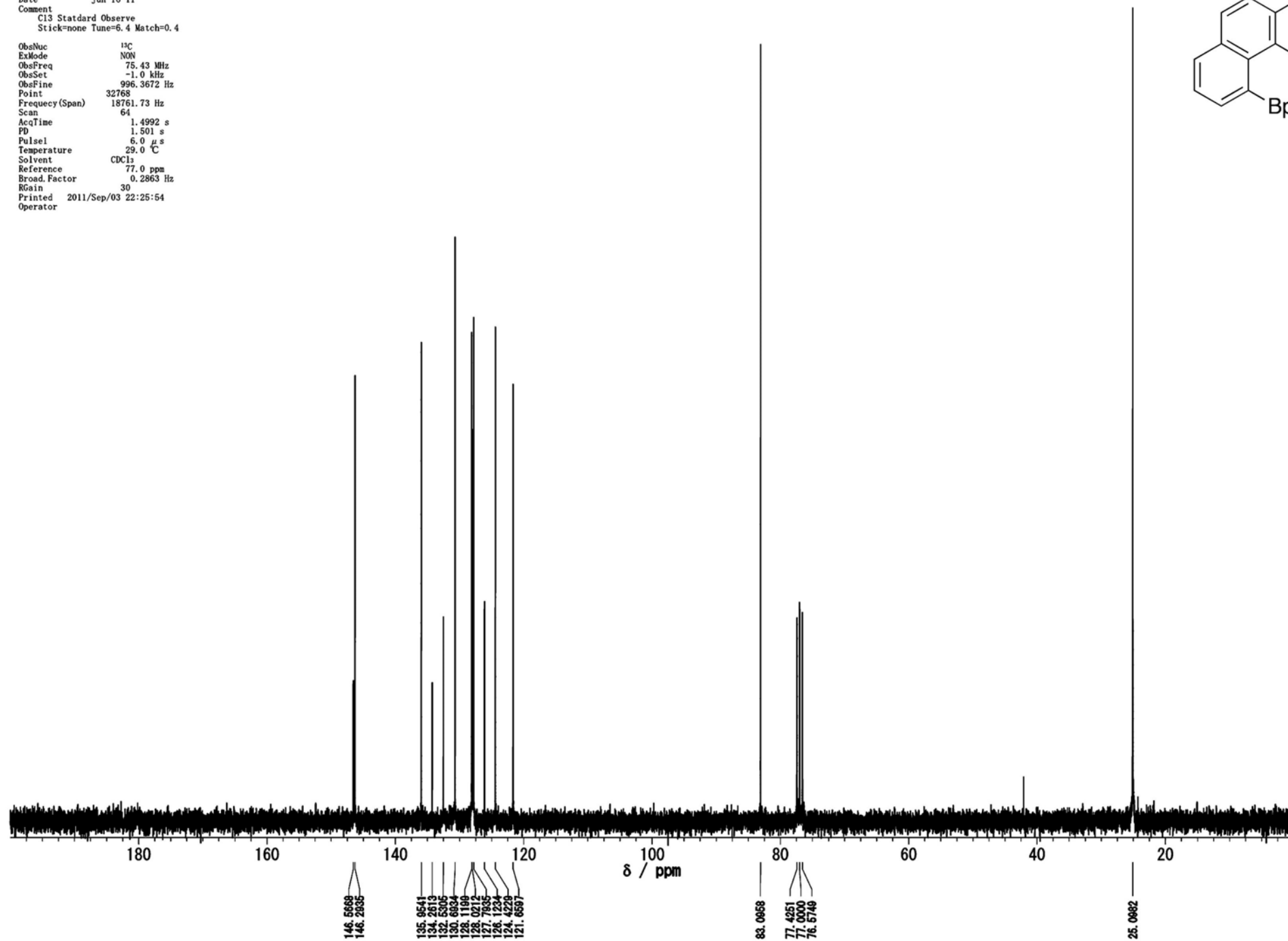
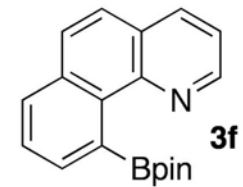
Original File:
Date Jun 16 11
Comment
STANDARD 1H OBSERVE

ObsNuc ¹H
ExMode NON
ObsFreq 299.96 MHz
ObsSet -1.0 kHz
ObsPine 995.0047 Hz
Point 16384
Frequency (Span) 4500.45 Hz
Scan 16
AcqTime 3.4983 s
PD 1.502 s
Pulse 6.0 μ s
Temperature 29.0 $^{\circ}$ C
Solvent CDCl₃
Reference 0.0 ppm
Broad Factor 0.1373 Hz
RGain 20
Printed 2011/Sep/03 22:24:50
Operator



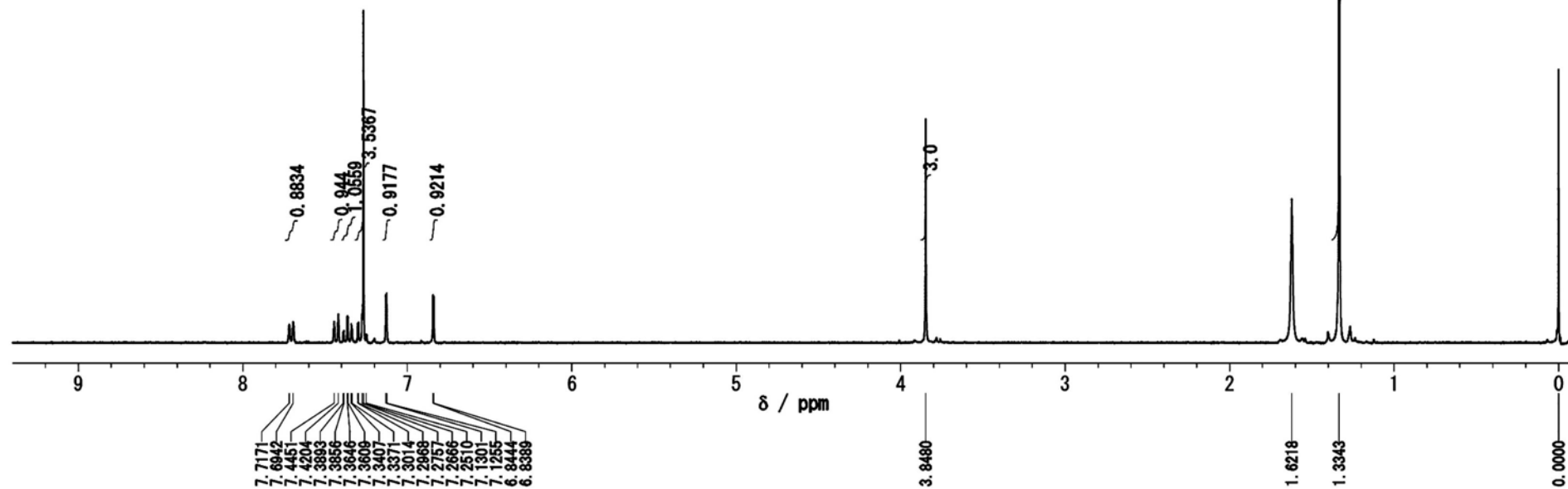
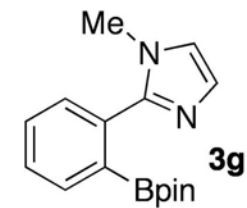
Original File:
 Date Jun 16 11
 Comment
 C13 Statdard Observe
 Stick=none Tune=6.4 Match=0.4

ObsNuc ^{13}C
 ExMode NON
 ObsFreq 75.43 MHz
 ObsSet -1.0 kHz
 ObsFine 996.3672 Hz
 Point 32768
 Frequency (Span) 18761.73 Hz
 Scan 64
 AcqTime 1.4992 s
 PD 1.501 s
 Pulse 6.0 μs
 Temperature 29.0 $^{\circ}\text{C}$
 Solvent CDCl_3
 Reference 77.0 ppm
 Broad.Factor 0.2863 Hz
 RGain 30
 Printed 2011/Sep/03 22:25:54
 Operator



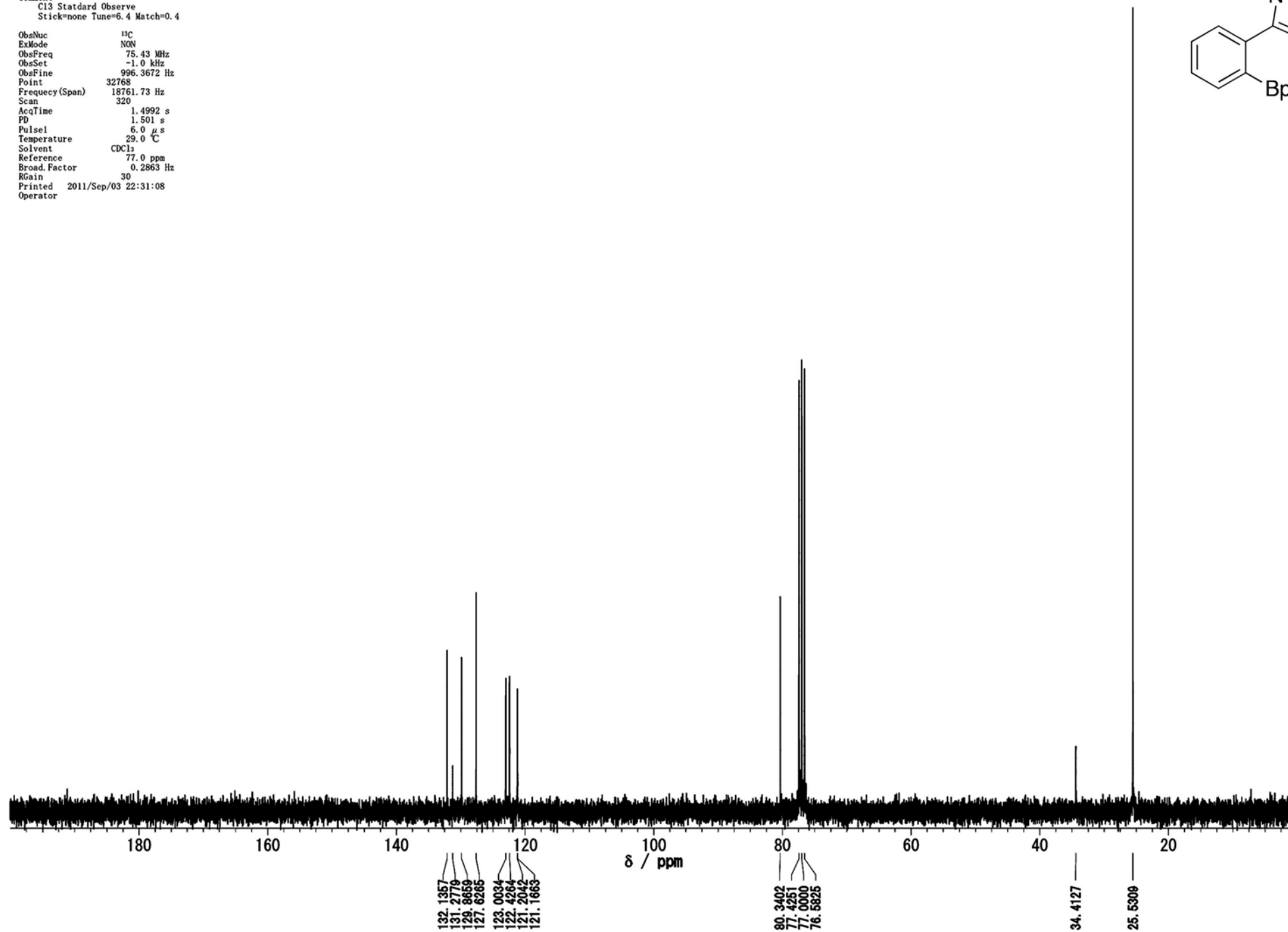
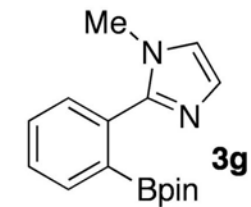
Original File:
 Date Jun 19 11
 Comment
 STANDARD 1H OBSERVE

ObsNuc	¹ H
ExMode	NON
ObsFreq	299.96 MHz
ObsSet	-1.0 kHz
ObsPine	995.0047 Hz
Point	16384
Frequency (Span)	4500.45 Hz
Scan	16
AcqTime	3.4983 s
PD	1.502 s
Pulse1	6.0 μ s
Temperature	29.0 $^{\circ}$ C
Solvent	CDCl ₃
Reference	0.0 ppm
Broad. Factor	0.1373 Hz
RGain	30
Printed	2011/Sep/03 22:27:09
Operator	



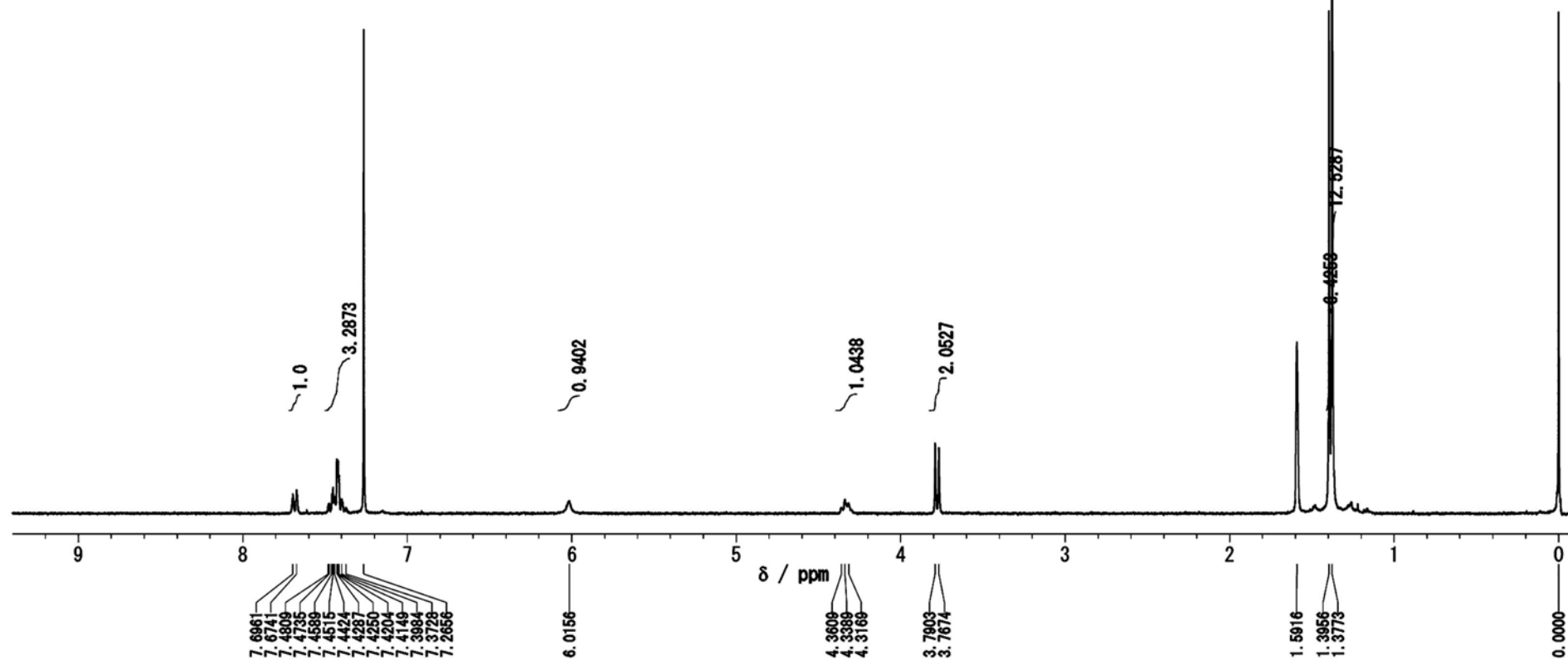
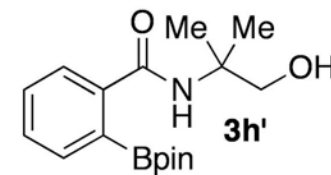
Original File:
 Date Jun 20 11
 Comment
 C13 Statdard Observe
 Stick=none Tune=6.4 Match=0.4

ObsNuc ¹³C
 ExMode NON
 ObsFreq 75.43 MHz
 ObsSet -1.0 kHz
 ObsFine 996.3672 Hz
 Point 32768
 Frequency (Span) 18761.73 Hz
 Scan 320
 AcqTime 1.4992 s
 PD 1.501 s
 Pulsel 6.0 μ s
 Temperature 29.0 $^{\circ}$ C
 Solvent CDCl₃
 Reference 77.0 ppm
 Broad.Factor 0.2863 Hz
 RGain 30
 Printed 2011/Sep/03 22:31:08
 Operator



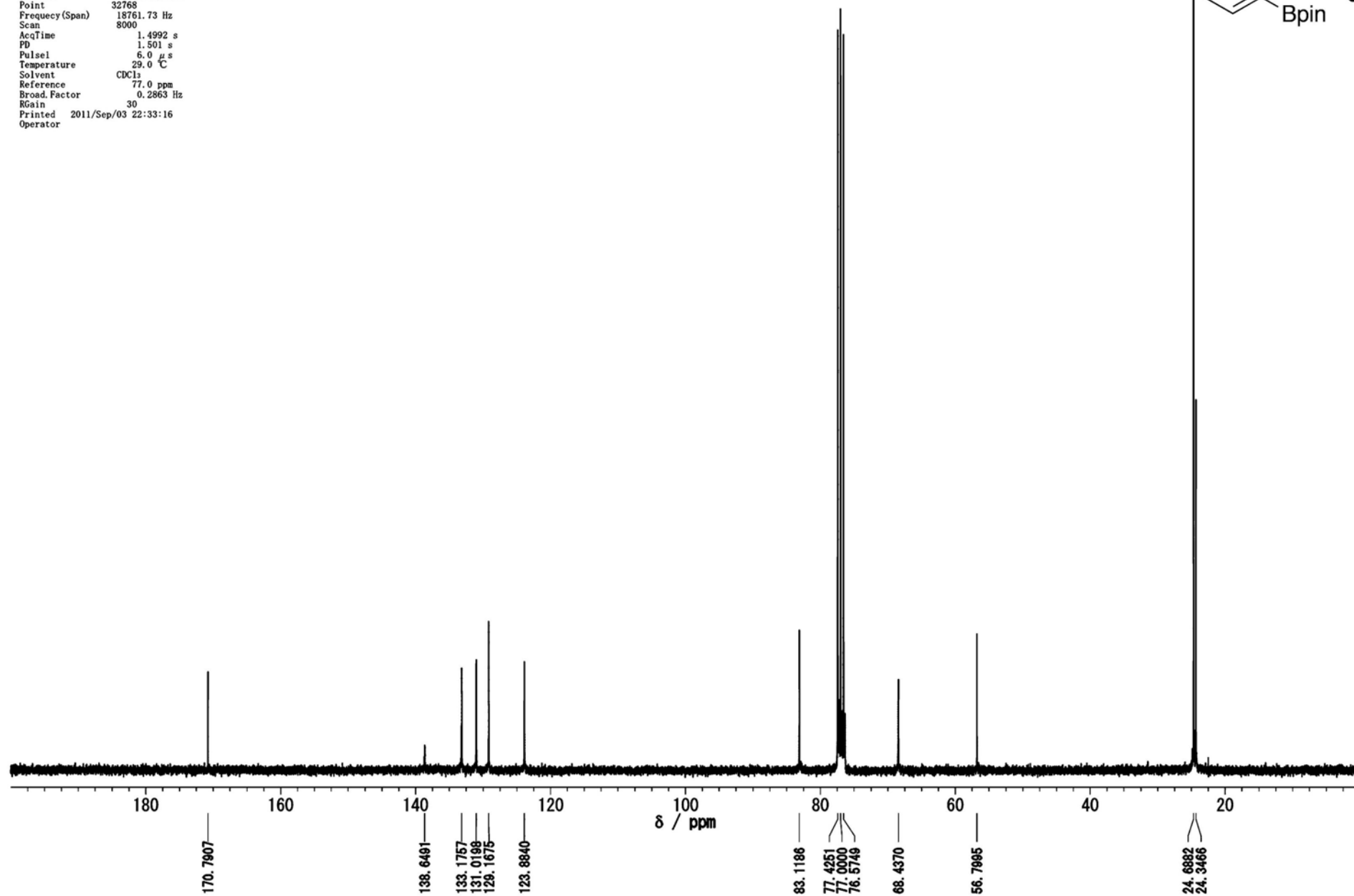
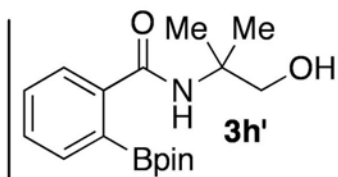
Original File:
 Date Aug 1 11
 Comment
 STANDARD 1H OBSERVE

ObsNuc ¹H
 ExMode NON
 ObsFreq 299.96 MHz
 ObsSet -1.0 kHz
 ObsPine 995.0047 Hz
 Point 16384
 Frequency (Span) 4500.45 Hz
 Scan 16
 AcqTime 3.4983 s
 PD 1.502 s
 Pulse 6.0 μ s
 Temperature 29.0 $^{\circ}$ C
 Solvent CDCl₃
 Reference 0.0 ppm
 Broad. Factor 0.1373 Hz
 RGain 30
 Printed 2011/Sep/03 22:32:05
 Operator

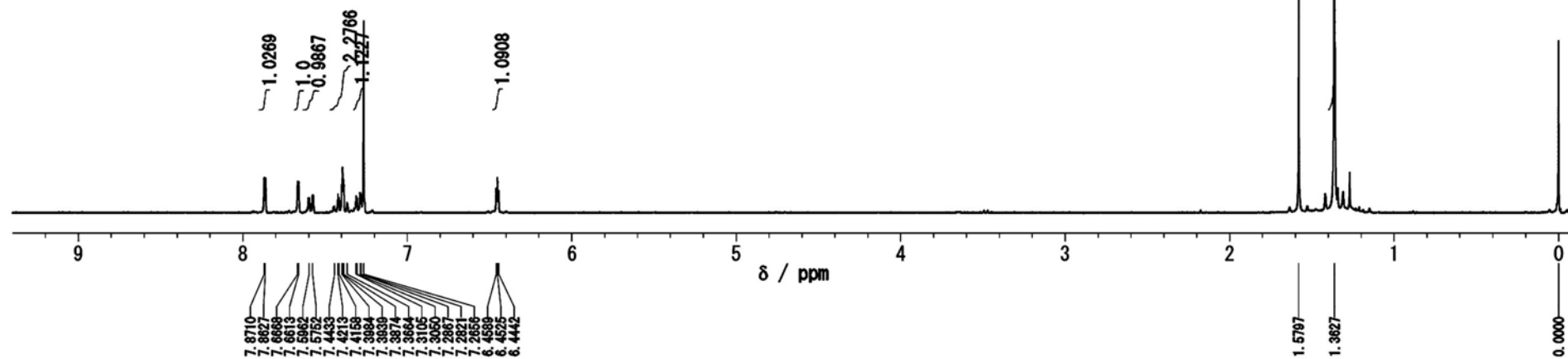
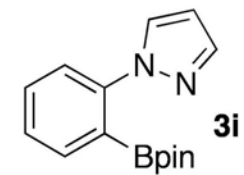


Original File:
 Date Aug 2 11
 Comment
 C13 Statdard Observe
 Stick=none Tune=6.4 Match=0.4

ObsNuc ¹³C
 ExMode NON
 ObsFreq 75.43 MHz
 ObsSet -1.0 kHz
 ObsFine 996.3672 Hz
 Point 32768
 Frequency (Span) 18761.73 Hz
 Scan 8000
 AcqTime 1.4992 s
 PD 1.501 s
 Pulsel 6.0 μ s
 Temperature 29.0 $^{\circ}$ C
 Solvent CDCl₃
 Reference 77.0 ppm
 Broad.Factor 0.2863 Hz
 RGain 30
 Printed 2011/Sep/03 22:33:16
 Operator

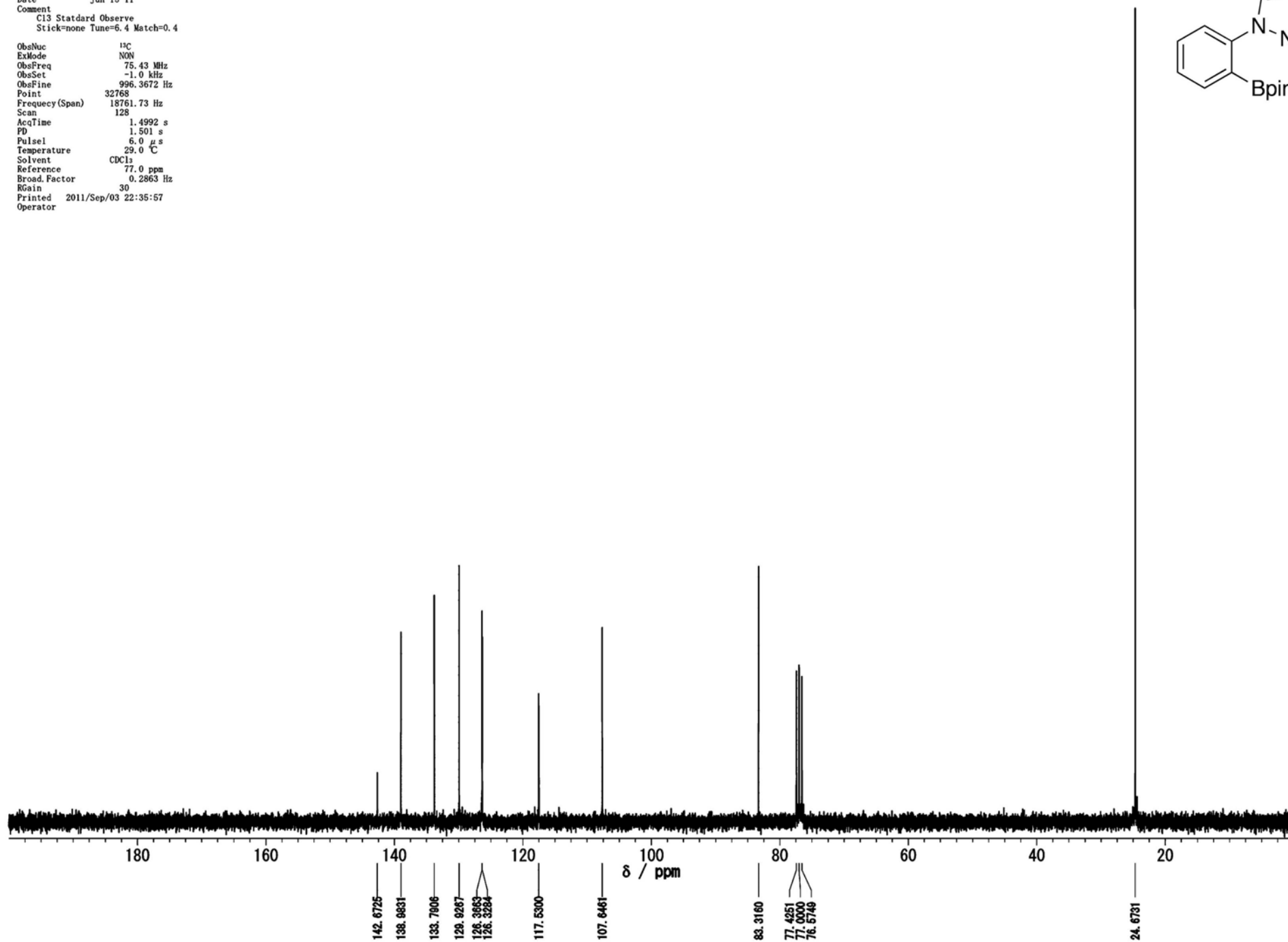
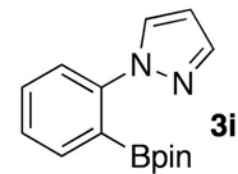


Original File: Jun 15 11
 Date: Jun 15 11
 Comment: STANDARD 1H OBSERVE
 ObsNuc: ¹H
 ExMode: NON
 ObsFreq: 299.96 MHz
 ObsSet: -1.0 kHz
 ObsPine: 995.0047 Hz
 Point: 16384
 Frequency (Span): 4500.45 Hz
 Scan: 16
 AcqTime: 3.4983 s
 PD: 1.502 s
 Pulse: 6.0 μ s
 Temperature: 29.0 $^{\circ}$ C
 Solvent: CDCl₃
 Reference: 0.0 ppm
 Broad Factor: 0.1373 Hz
 RGain: 26
 Printed: 2011/Sep/03 22:34:45
 Operator:

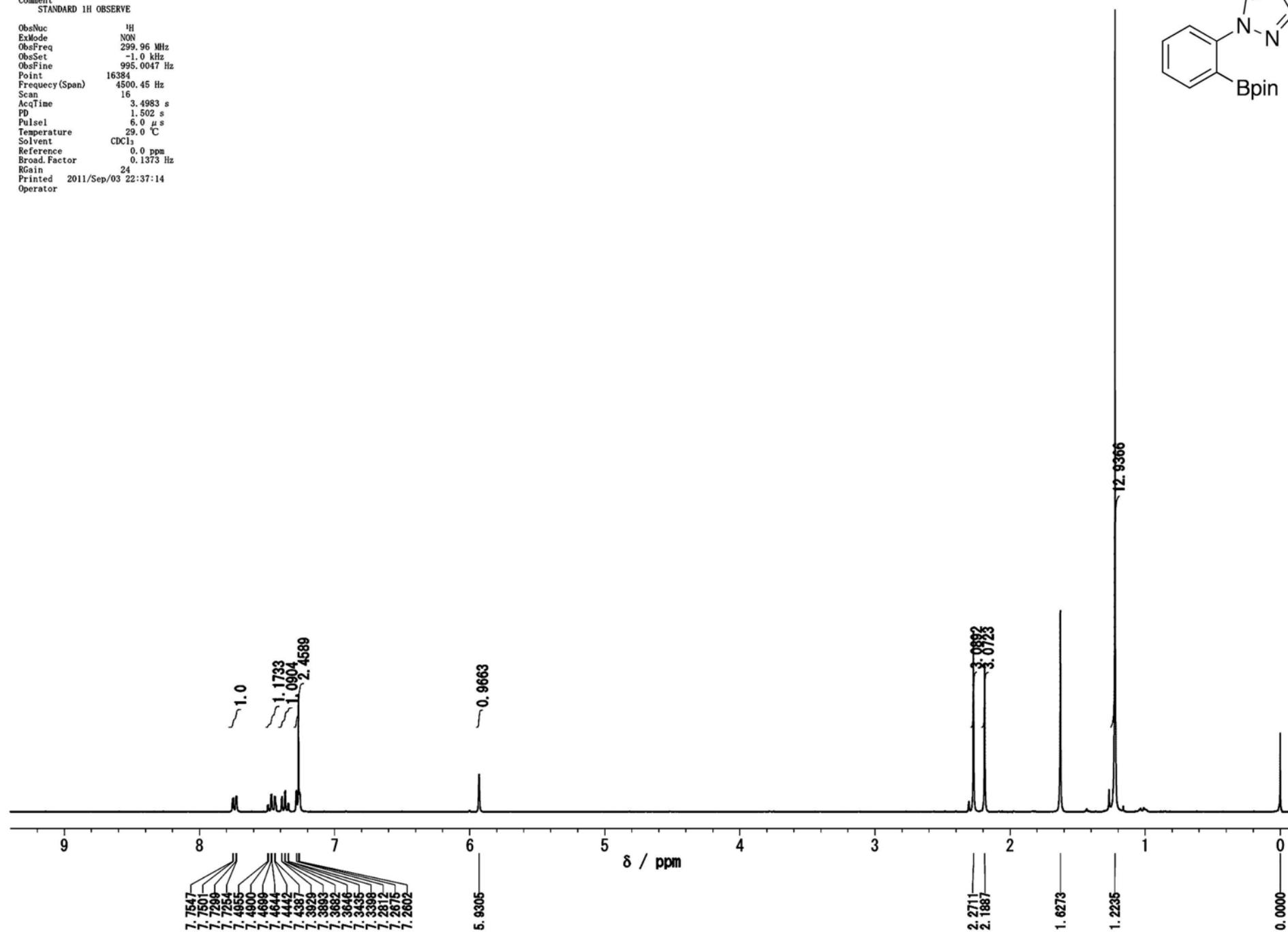
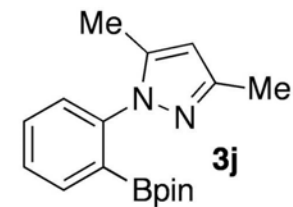


Original File: Jun 15 11
 Date Jun 15 11
 Comment C13 Statdard Observe
 Stick=none Tune=6.4 Match=0.4

ObsNuc ^{13}C
 ExMode NON
 ObsFreq 75.43 MHz
 ObsSet -1.0 kHz
 ObsFine 996.3672 Hz
 Point 32768
 Frequency (Span) 18761.73 Hz
 Scan 128
 AcqTime 1.4992 s
 PD 1.501 s
 Pulse 6.0 μs
 Temperature 29.0 $^{\circ}\text{C}$
 Solvent CDCl_3
 Reference 77.0 ppm
 Broad Factor 0.2863 Hz
 RGain 30
 Printed 2011/Sep/03 22:35:57
 Operator

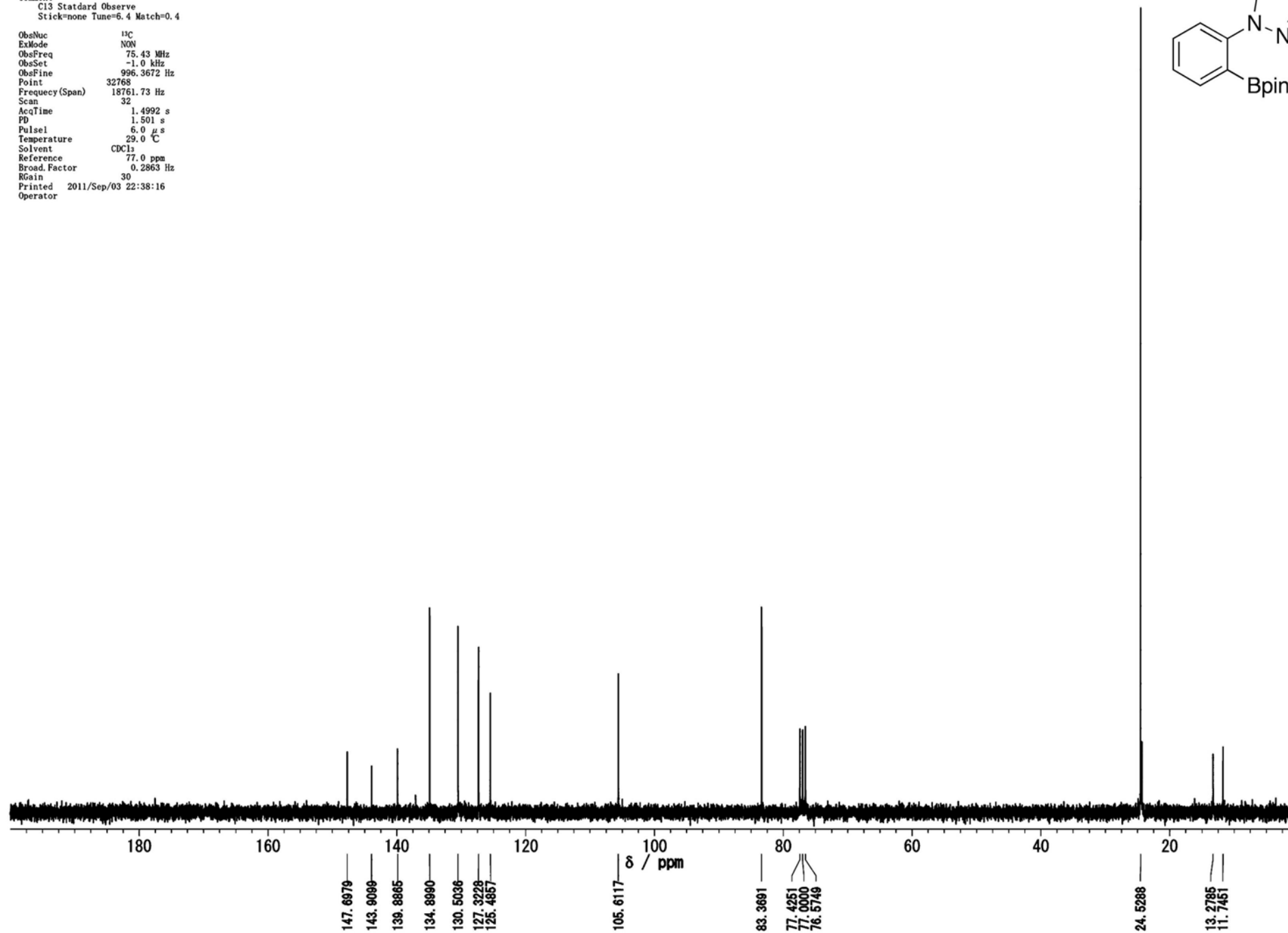
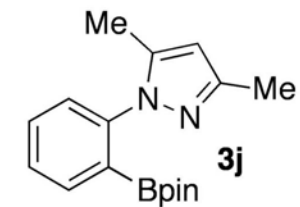


Original File:
 Date Jul 26 11
 Comment
 STANDARD 1H OBSERVE
 ObsNuc ¹H
 ExMode NON
 ObsFreq 299.96 MHz
 ObsSet -1.0 kHz
 ObsPine 995.0047 Hz
 Point 16384
 Frequency (Span) 4500.45 Hz
 Scan 16
 AcqTime 3.4983 s
 PD 1.502 s
 Pulse 6.0 μs
 Temperature 29.0 °C
 Solvent CDCl₃
 Reference 0.0 ppm
 Broad Factor 0.1373 Hz
 RGain 24
 Printed 2011/Sep/03 22:37:14
 Operator



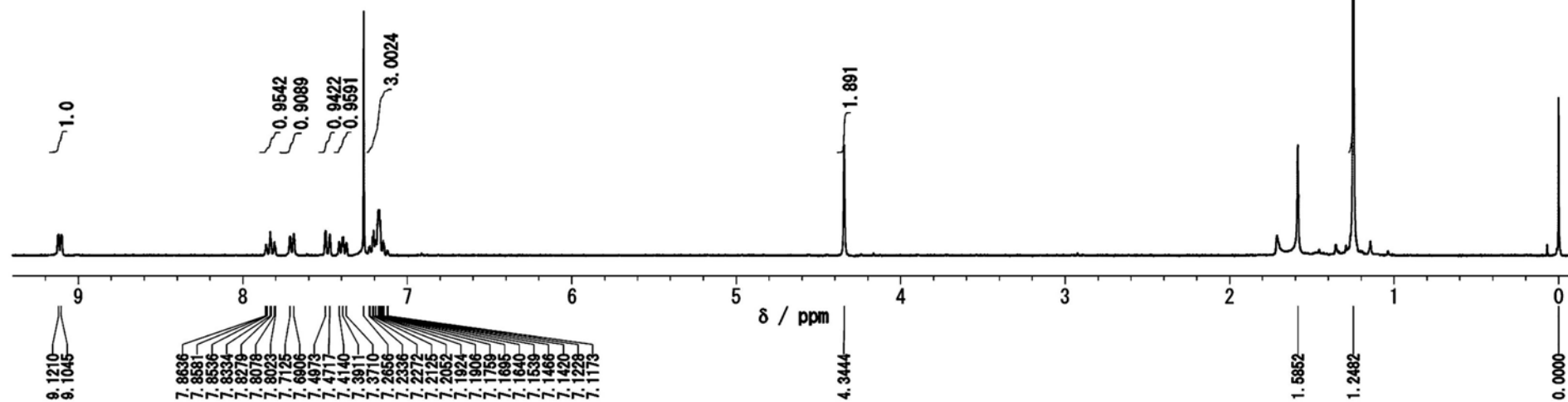
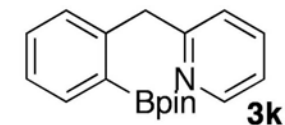
Original File: Jul 26 11
 Date
 Comment
 C13 Statdard Observe
 Stick=none Tune=6.4 Match=0.4

ObsNuc ¹³C
 ExMode NON
 ObsFreq 75.43 MHz
 ObsSet -1.0 kHz
 ObsFine 996.3672 Hz
 Point 32768
 Frequency (Span) 18761.73 Hz
 Scan 32
 AcqTime 1.4992 s
 PD 1.501 s
 Pulsel 6.0 μ s
 Temperature 29.0 $^{\circ}$ C
 Solvent CDCl₃
 Reference 77.0 ppm
 Broad.Factor 0.2863 Hz
 RGain 30
 Printed 2011/Sep/03 22:38:16
 Operator



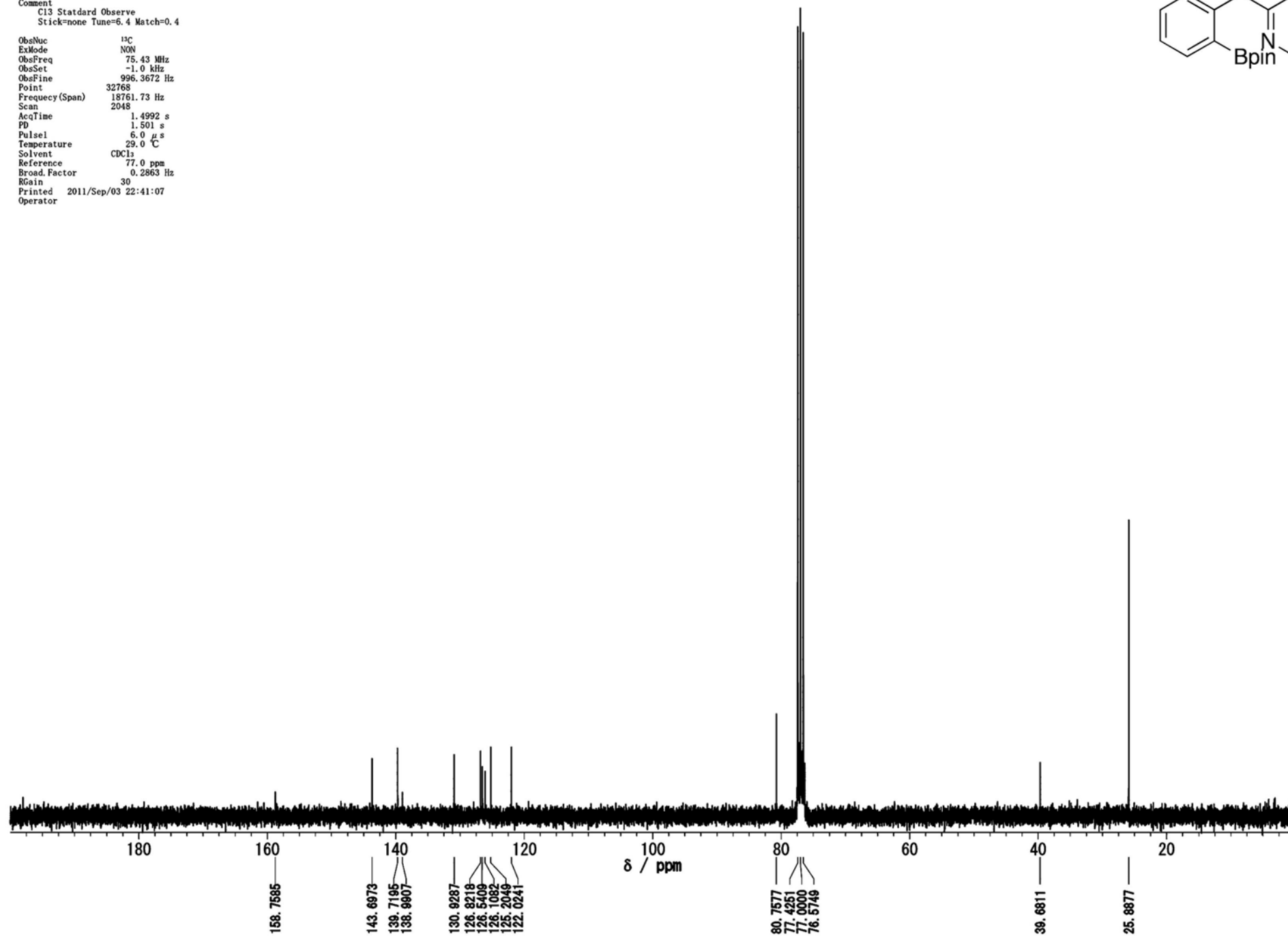
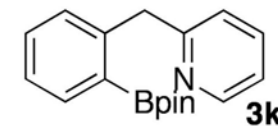
Original File:
 Date May 19 11
 Comment
 STANDARD 1H OBSERVE

ObsNuc H
 ExMode NON
 ObsFreq 299.96 MHz
 ObsSet -1.0 kHz
 ObsPine 995.0047 Hz
 Point 16384
 Frequency (Span) 4500.45 Hz
 Scan 16
 AcqTime 3.4983 s
 PD 1.502 s
 Pulse 6.0 μ s
 Temperature 29.0 $^{\circ}$ C
 Solvent CDCl₃
 Reference 0.0 ppm
 Broad Factor 0.1373 Hz
 RGain 26
 Printed 2011/Sep/03 22:40:05
 Operator



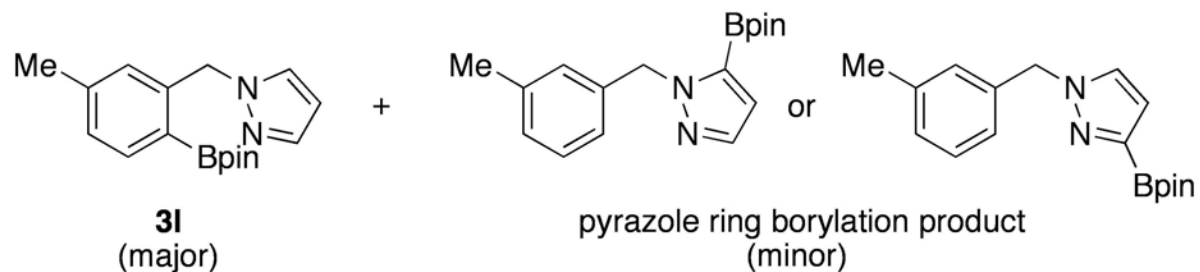
Original File:
 Date May 19 11
 Comment
 C13 Statdard Observe
 Stick=none Tune=6.4 Match=0.4

ObsNuc ¹³C
 ExMode NON
 ObsFreq 75.43 MHz
 ObsSet -1.0 kHz
 ObsFine 996.3672 Hz
 Point 32768
 Frequency (Span) 18761.73 Hz
 Scan 2048
 AcqTime 1.4992 s
 PD 1.501 s
 Pulsel 6.0 μs
 Temperature 29.0 °C
 Solvent CDCl₃
 Reference 77.0 ppm
 Broad Factor 0.2863 Hz
 RGain 30
 Printed 2011/Sep/03 22:41:07
 Operator



Original File:
Date Aug 30 11
Comment
STANDARD 1H OBSERVE

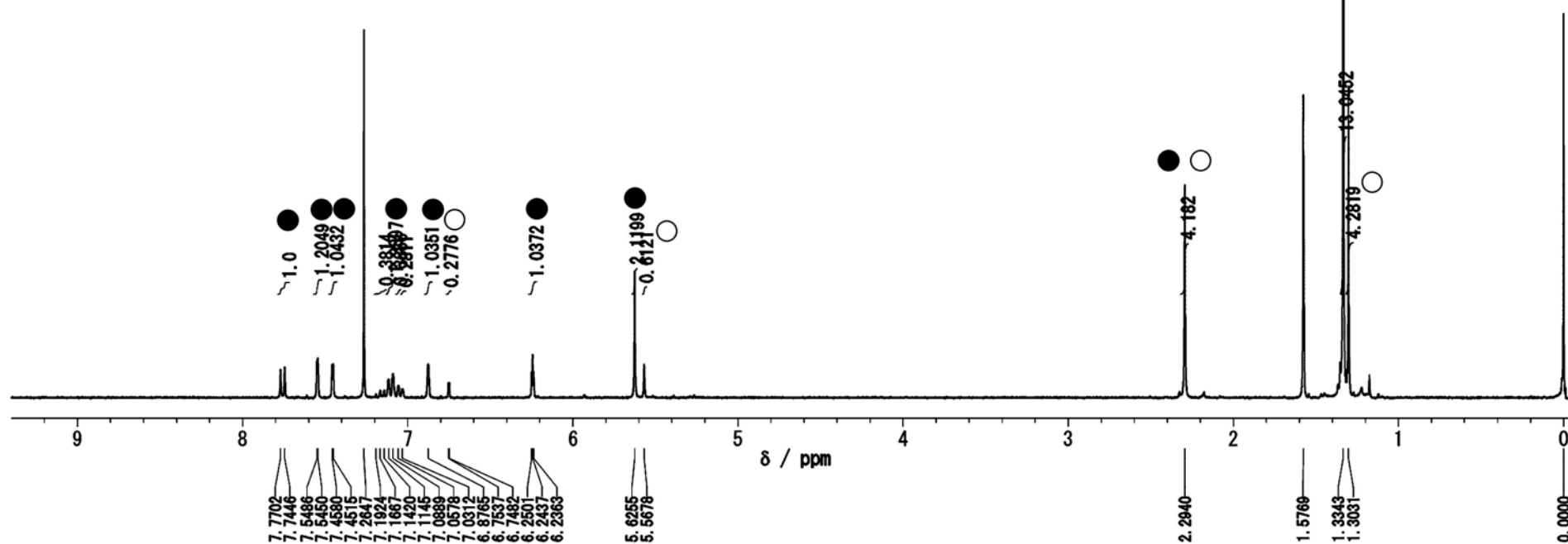
ObsNuc H
ExMode NOV
ObsFreq 299.96 MHz
ObsSet -1.0 kHz
ObsFine 995.0047 Hz
Point 16384
Frequency(Span) 4500.45 Hz
Scan 16
AcqTime 3.4983 s
PD 1.502 s
Pulse 6.0 μ s
Temperature 29.0 $^{\circ}$ C
Solvent CDCl₃
Reference 0.0 ppm
Broad.Factor 0.1373 Hz
RGain 30
Printed 2011/Sep/03 22:42:49
Operator



ratio of **3I**/pyrazole ring borylation =79:21

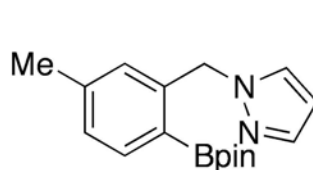
● peaks: **3I**

○ peaks: pyrazole ring borylation

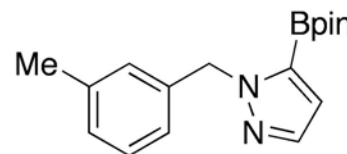


Original File:
Date Aug 30 11
Comment
C13 Statdard Observe
Stick=none Tune=6.4 Match=0.4

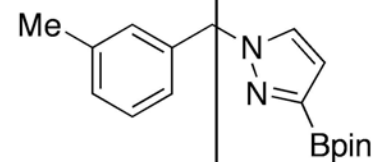
ObsNuc ¹³C
ExMode NON
ObsFreq 75.43 MHz
ObsSet -1.0 kHz
ObsFine 996.3672 Hz
Point 32768
Frequency (Span) 18761.73 Hz
Scan 224
AcqTime 1.4992 s
PD 1.501 s
Pulse 6.0 μs
Temperature 29.0 °C
Solvent CDCl₃
Reference 77.0 ppm
Broad.Factor 0.2863 Hz
RGain 30
Printed 2011/Sep/03 22:44:05
Operator



3I
(major)



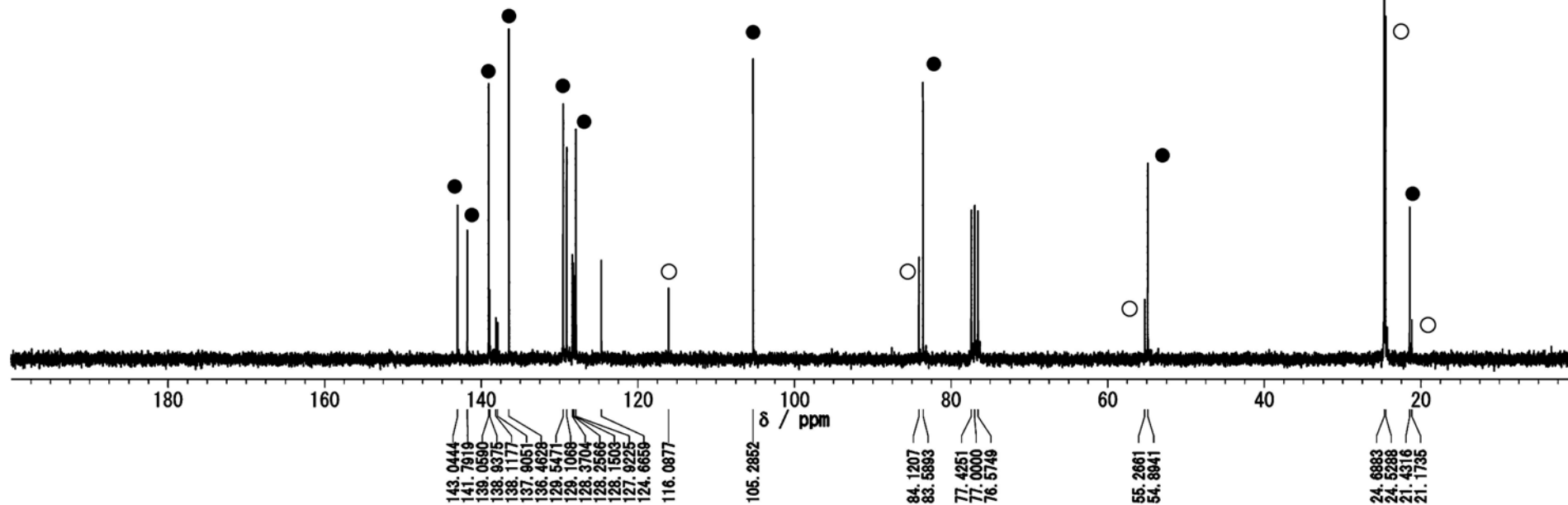
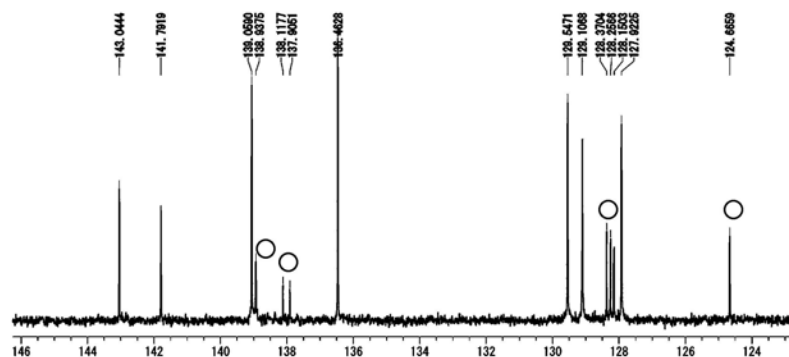
pyrazole ring borylation product
(minor)



ratio of **3I**/pyrazole ring borylation = 79:21

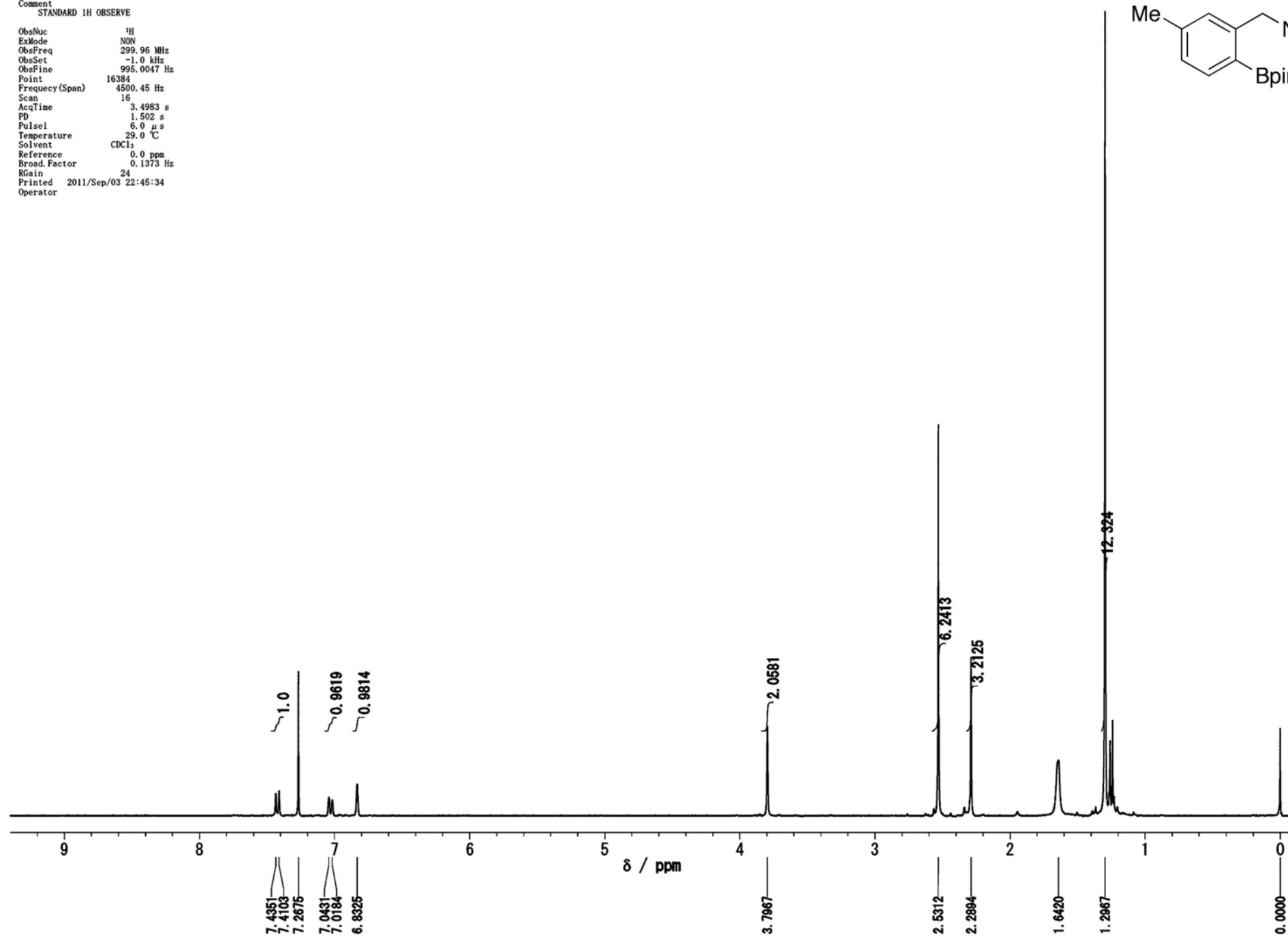
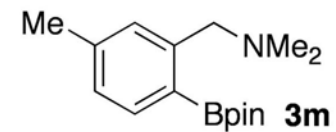
● peaks: **3I**

○ peaks: pyrazole ring borylation



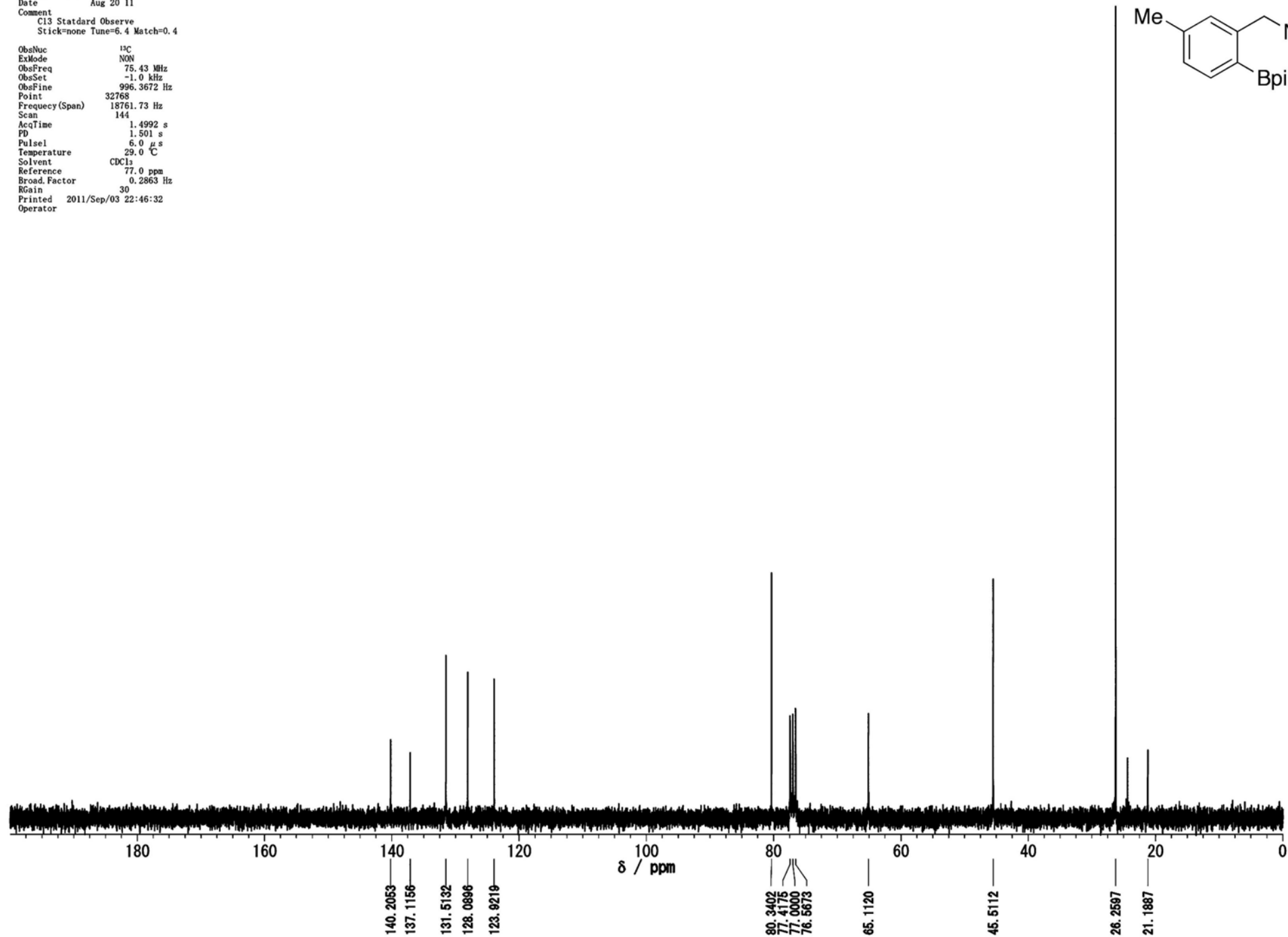
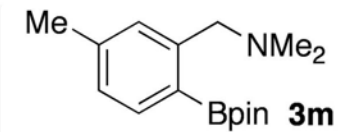
Original File:
 Date Aug 20 11
 Comment
 STANDARD 1H OBSERVE

ObsNuc	¹ H
ExMode	NON
ObsFreq	299.96 MHz
ObsSet	-1.0 kHz
ObsPine	995.0047 Hz
Point	16384
Frequency (Span)	4500.45 Hz
Scan	16
AcqTime	3.4983 s
PD	1.502 s
Pulse1	6.0 μ s
Temperature	29.0 $^{\circ}$ C
Solvent	CDCl ₃
Reference	0.0 ppm
Broad. Factor	0.1373 Hz
RGain	24
Printed	2011/Sep/03 22:45:34
Operator	



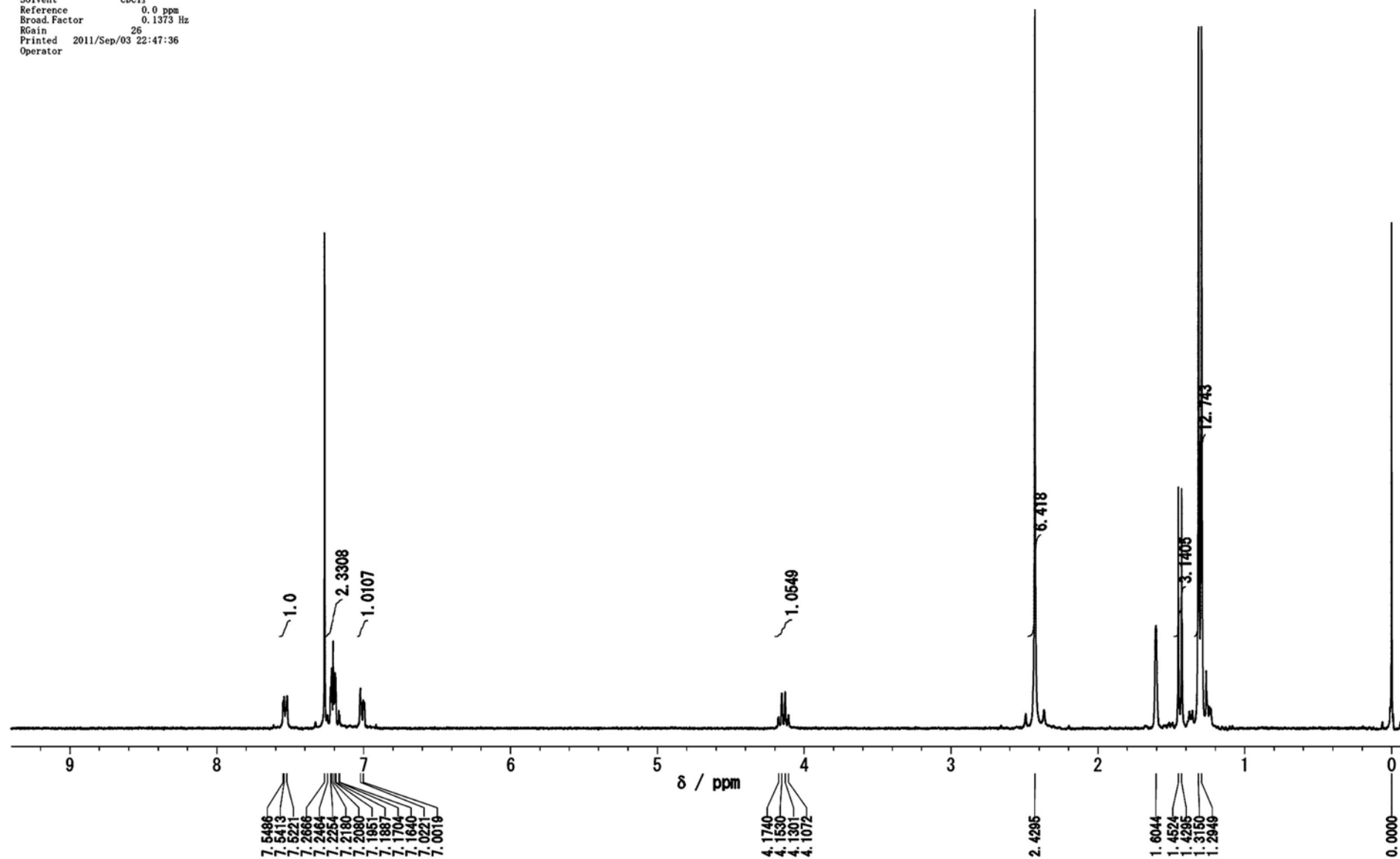
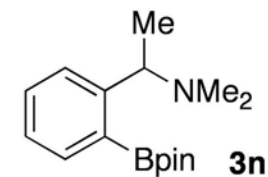
Original File:
 Date Aug 20 11
 Comment
 C13 Statdard Observe
 Stick=none Tune=6.4 Match=0.4

ObsNuc ¹³C
 ExMode NON
 ObsFreq 75.43 MHz
 ObsSet -1.0 kHz
 ObsFine 996.3672 Hz
 Point 32768
 Frequency (Span) 18761.73 Hz
 Scan 144
 AcqTime 1.4992 s
 PD 1.501 s
 Pulsel 6.0 μ s
 Temperature 29.0 $^{\circ}$ C
 Solvent CDCl₃
 Reference 77.0 ppm
 Broad.Factor 0.2863 Hz
 RGain 30
 Printed 2011/Sep/03 22:46:32
 Operator



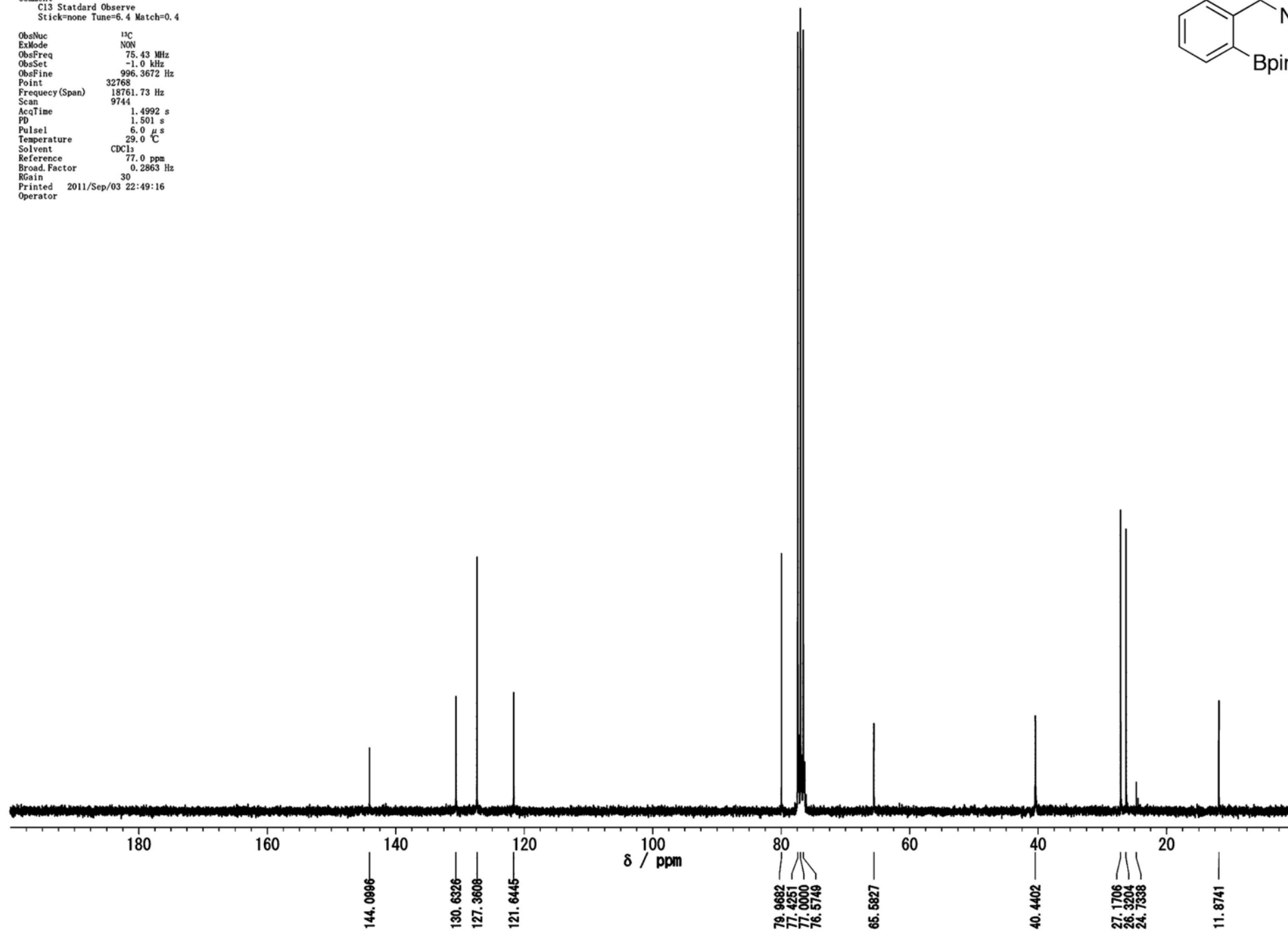
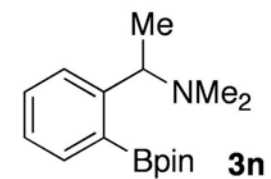
Original File:
 Date Aug 25 11
 Comment
 STANDARD 1H OBSERVE

ObsNuc ¹H
 ExMode NON
 ObsFreq 299.96 MHz
 ObsSet -1.0 kHz
 ObsPine 995.0047 Hz
 Point 16384
 Frequency (Span) 4500.45 Hz
 Scan 16
 AcqTime 3.4983 s
 PD 1.502 s
 Pulse 6.0 μ s
 Temperature 29.0 $^{\circ}$ C
 Solvent CDCl₃
 Reference 0.0 ppm
 Broad Factor 0.1373 Hz
 RGain 26
 Printed 2011/Sep/03 22:47:36
 Operator



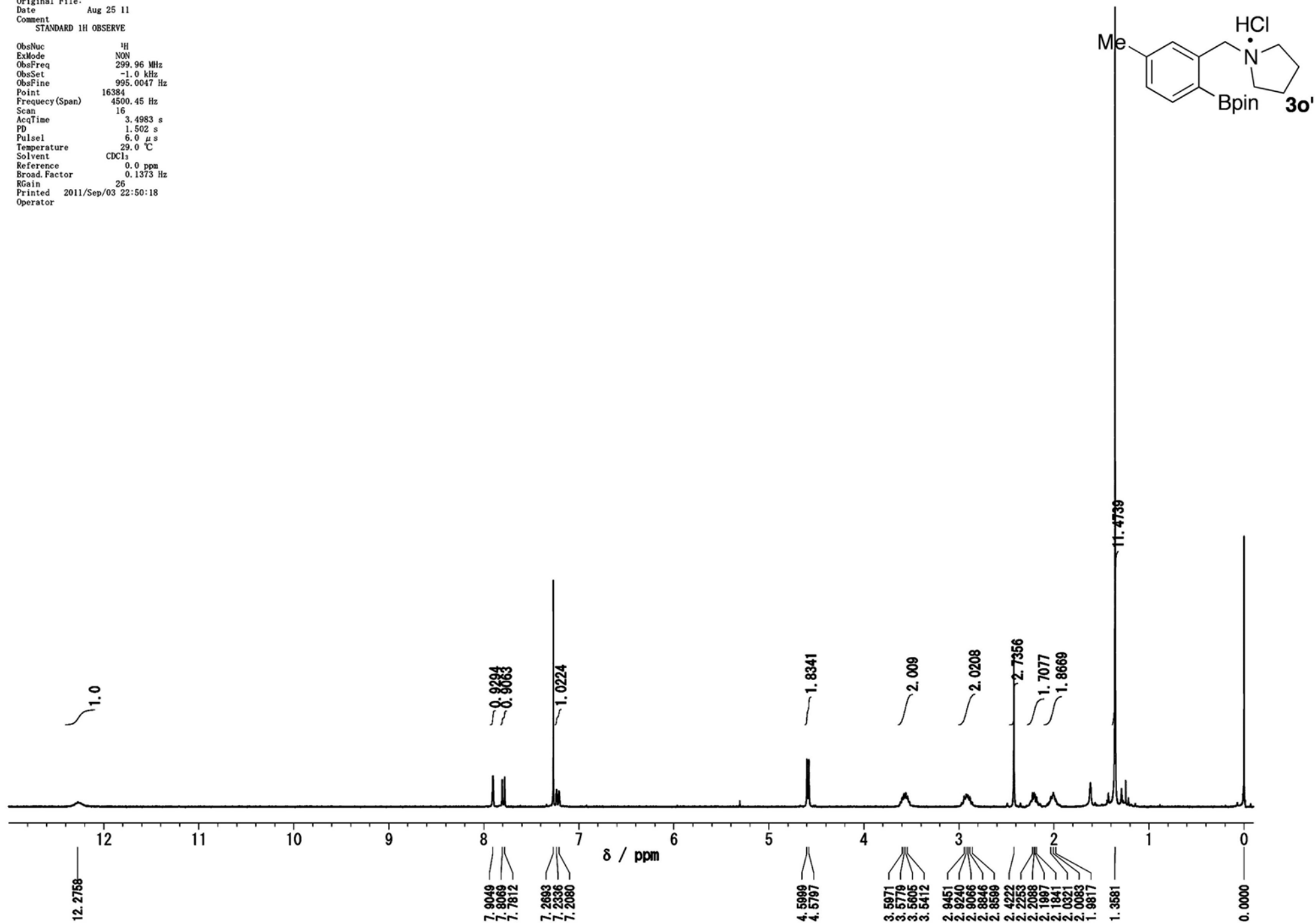
Original File:
 Date Aug 28 11
 Comment
 C13 Statdard Observe
 Stick=none Tune=6.4 Match=0.4

ObsNuc ^{13}C
 ExMode NON
 ObsFreq 75.43 MHz
 ObsSet -1.0 kHz
 ObsFine 996.3672 Hz
 Point 32768
 Frequency (Span) 18761.73 Hz
 Scan 9744
 AcqTime 1.4992 s
 PD 1.501 s
 Pulsel 6.0 μs
 Temperature 29.0 $^{\circ}\text{C}$
 Solvent CDCl_3
 Reference 77.0 ppm
 Broad.Factor 0.2863 Hz
 RGain 30
 Printed 2011/Sep/03 22:49:16
 Operator



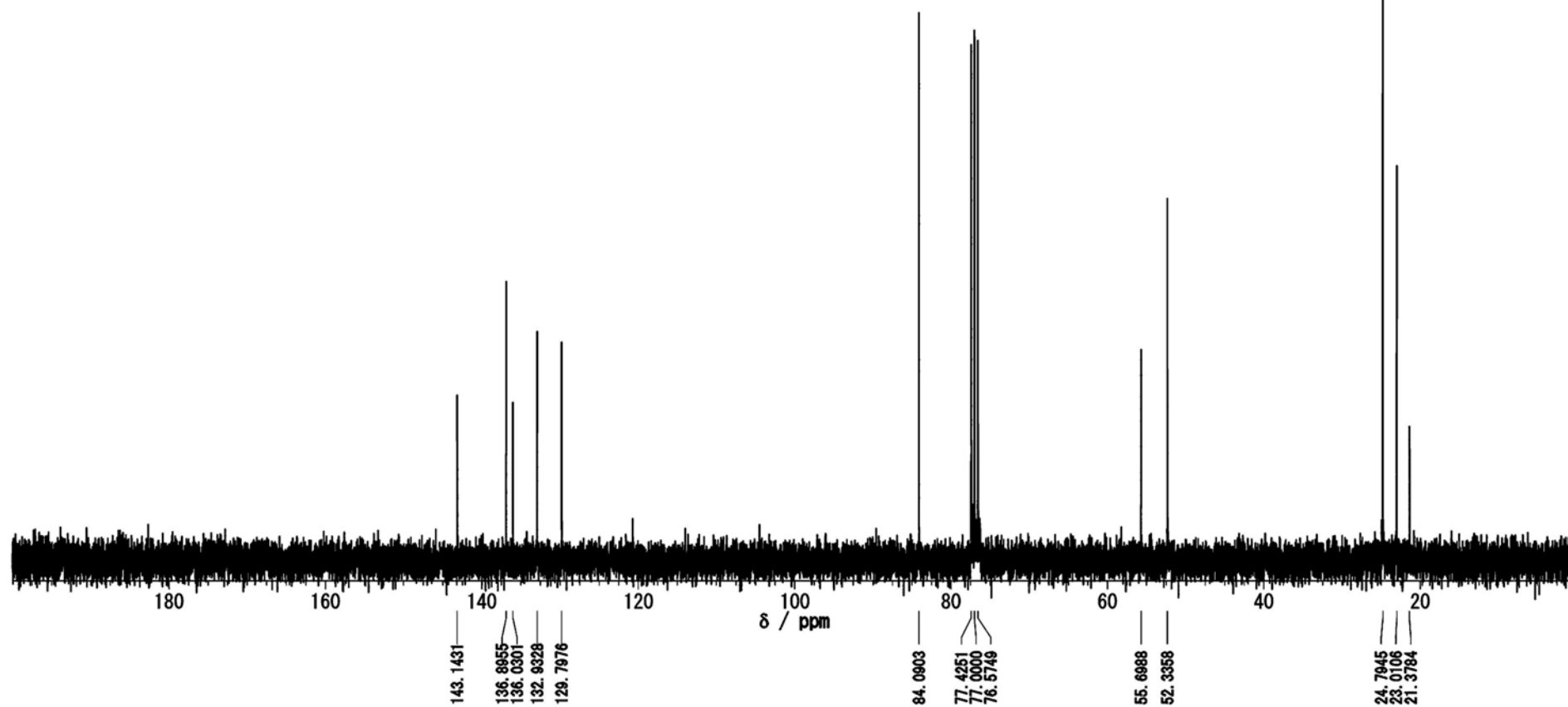
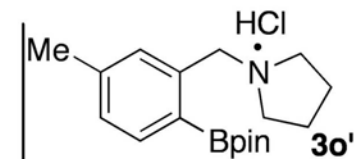
Original File:
 Date Aug 25 11
 Comment
 STANDARD 1H OBSERVE

ObsNuc ¹H
 ExMode NON
 ObsFreq 299.96 MHz
 ObsSet -1.0 kHz
 ObsPine 995.0047 Hz
 Point 16384
 Frequency (Span) 4500.45 Hz
 Scan 16
 AcqTime 3.4983 s
 PD 1.502 s
 Pulse 6.0 μ s
 Temperature 29.0 $^{\circ}$ C
 Solvent CDCl₃
 Reference 0.0 ppm
 Broad Factor 0.1373 Hz
 RGain 26
 Printed 2011/Sep/03 22:50:18
 Operator

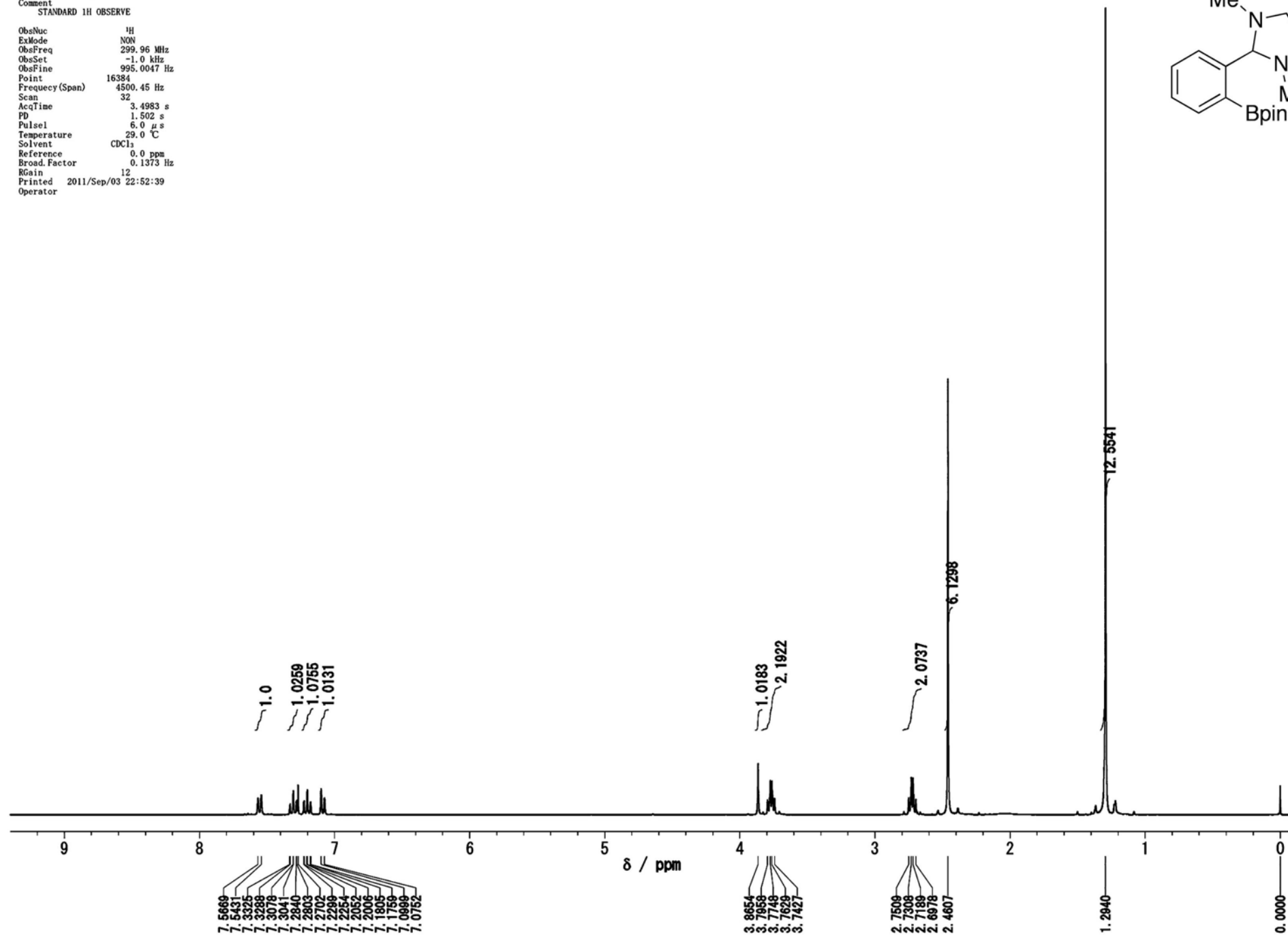
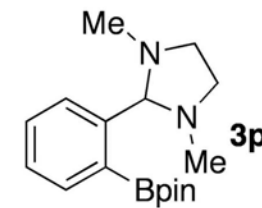


Original File:
 Date Aug 27 11
 Comment
 C13 Statdard Observe
 Stick=none Tune=6.4 Match=0.4

ObsNuc ¹³C
 ExMode NON
 ObsFreq 75.43 MHz
 ObsSet -1.0 kHz
 ObsFine 996.3672 Hz
 Point 32768
 Frequency (Span) 18761.73 Hz
 Scan 160
 AcqTime 1.4992 s
 PD 1.501 s
 Pulsel 6.0 μ s
 Temperature 29.0 $^{\circ}$ C
 Solvent CDCl₃
 Reference 77.0 ppm
 Broad.Factor 0.2863 Hz
 RGain 30
 Printed 2011/Sep/03 22:51:31
 Operator

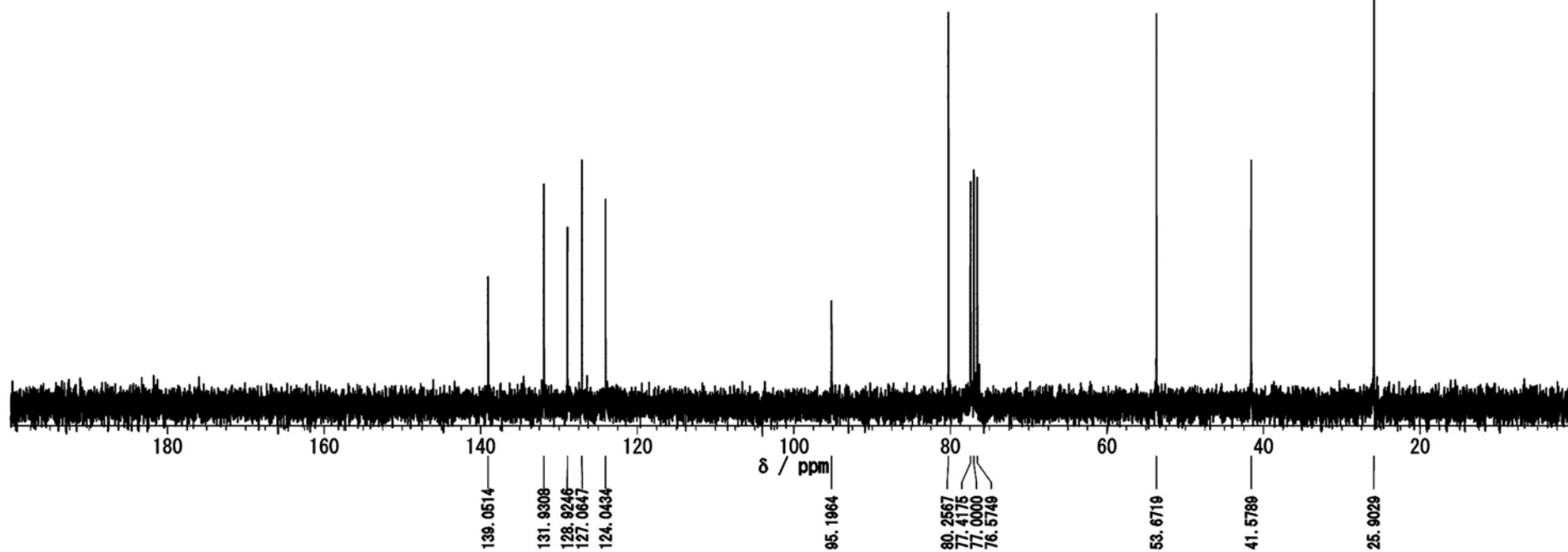
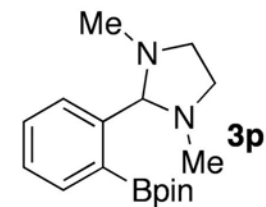


Original File:
 Date Jul 21 11
 Comment
 STANDARD 1H OBSERVE
 ObsNuc ¹H
 ExMode NON
 ObsFreq 299.96 MHz
 ObsSet -1.0 kHz
 ObsPine 995.0047 Hz
 Point 16384
 Frequency (Span) 4500.45 Hz
 Scan 32
 AcqTime 3.4983 s
 PD 1.502 s
 Pulse 6.0 μ s
 Temperature 29.0 $^{\circ}$ C
 Solvent CDCl₃
 Reference 0.0 ppm
 Broad Factor 0.1373 Hz
 RGain 12
 Printed 2011/Sep/03 22:52:39
 Operator

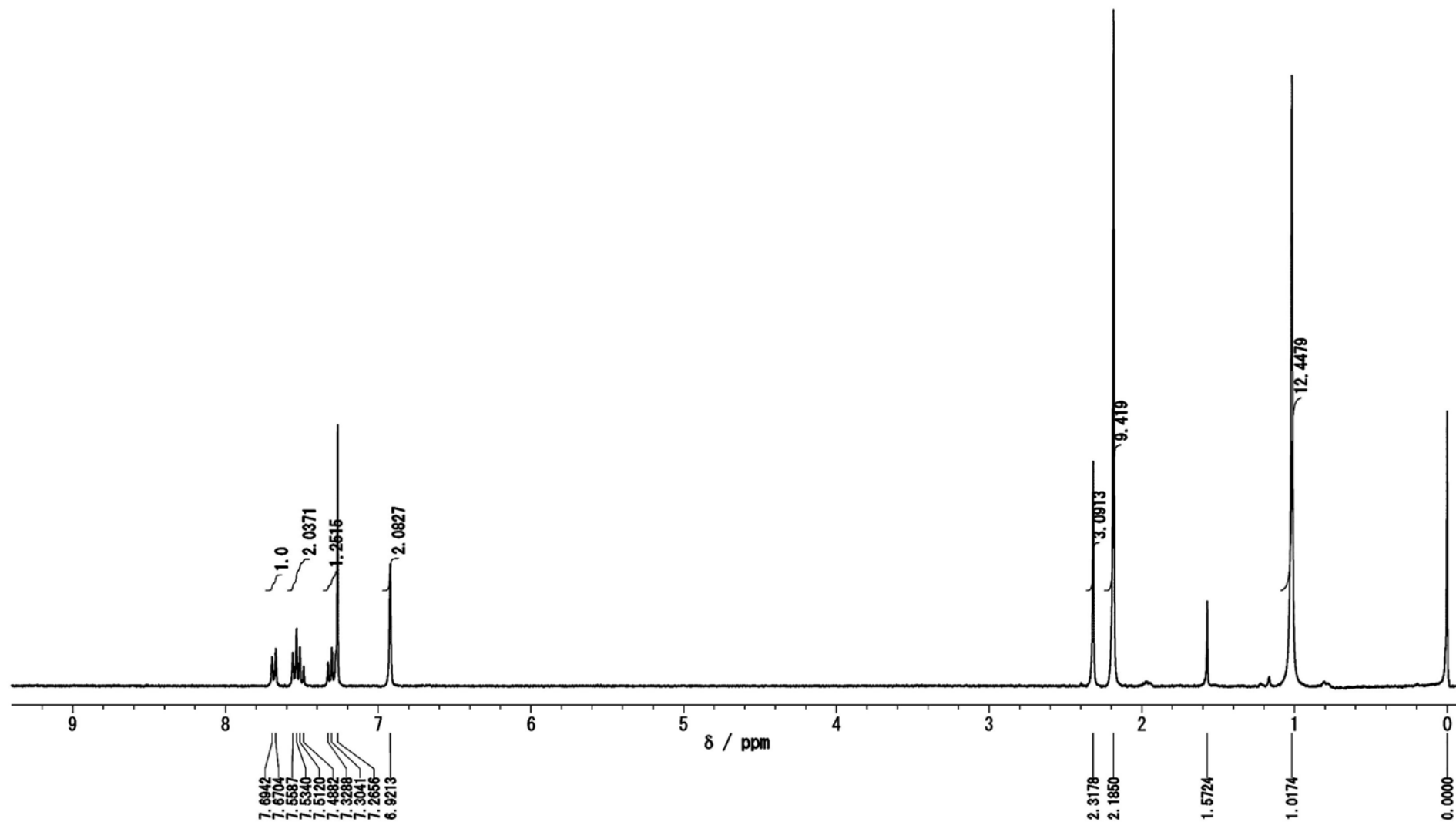
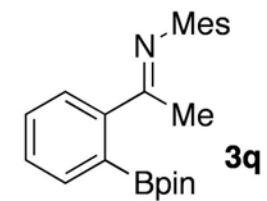


Original File: Jul 21 11
 Date: Jul 21 11
 Comment: C13 Statdard Observe
 Stick=none Tune=6.4 Match=0.4

ObsNuc: ¹³C
 ExMode: NON
 ObsFreq: 75.43 MHz
 ObsSet: -1.0 kHz
 ObsFine: 996.3672 Hz
 Point: 32768
 Frequency (Span): 18761.73 Hz
 Scan: 112
 AcqTime: 1.4992 s
 PD: 1.501 s
 Pulse: 6.0 μs
 Temperature: 29.0 °C
 Solvent: CDCl₃
 Reference: 77.0 ppm
 Broad.Factor: 0.2863 Hz
 RGain: 30
 Printed: 2011/Sep/03 22:54:08
 Operator:

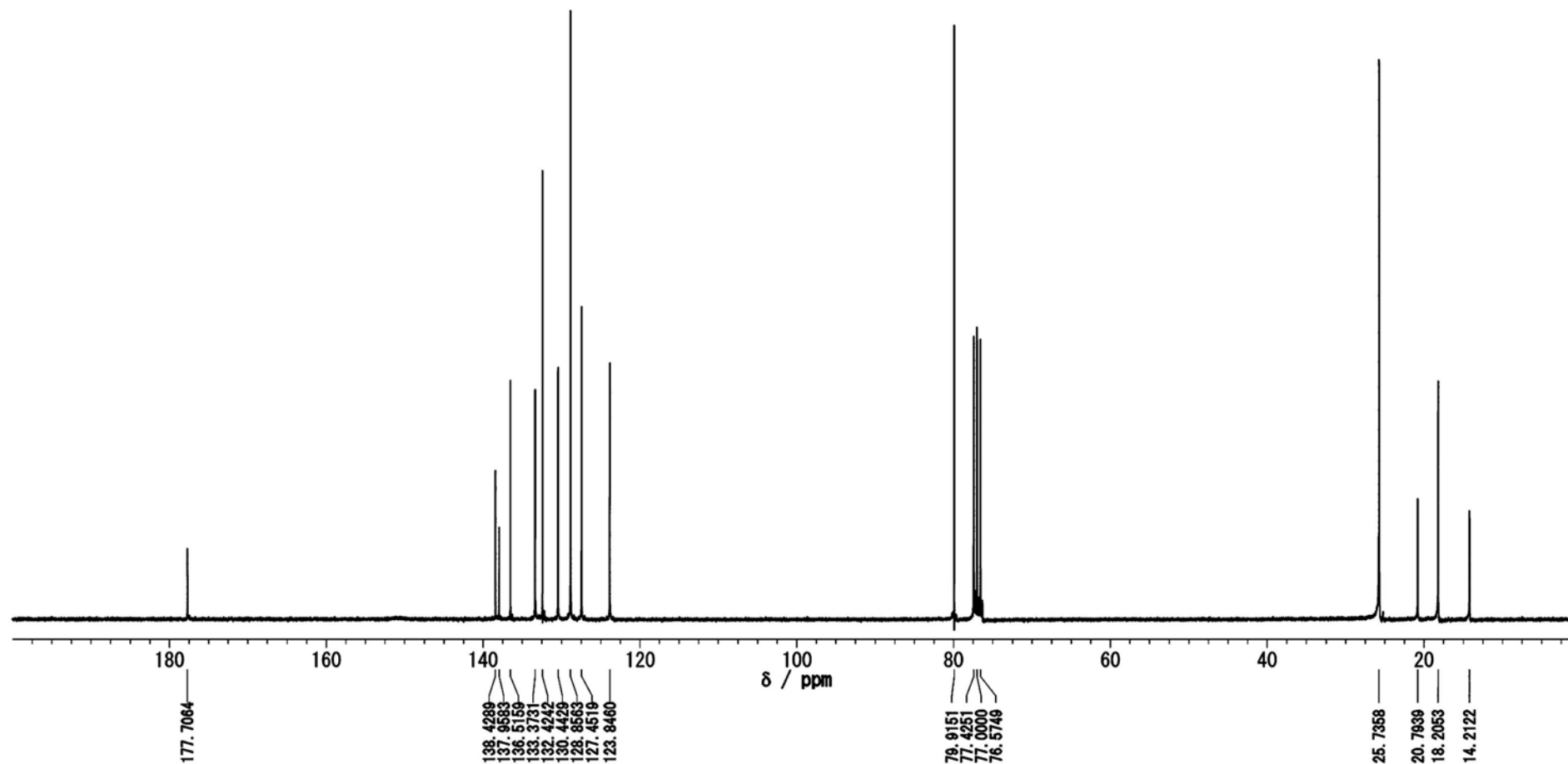
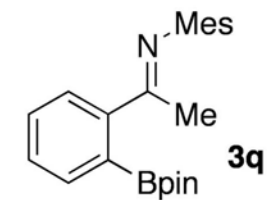


Original File: Jul 25 11
 Date: Jul 25 11
 Comment: STANDARD 1H OBSERVE
 ObsNuc: ¹H
 ExMode: NON
 ObsFreq: 299.96 MHz
 ObsSet: -1.0 kHz
 ObsPine: 995.0047 Hz
 Point: 16384
 Frequency (Span): 4500.45 Hz
 Scan: 16
 AcqTime: 3.4983 s
 PD: 1.502 s
 Pulse1: 6.0 μs
 Temperature: 29.0 °C
 Solvent: CDCl₃
 Reference: 0.0 ppm
 Broad Factor: 0.1373 Hz
 RGain: 29
 Printed: 2011/Jul/28 15:44:44
 Operator:



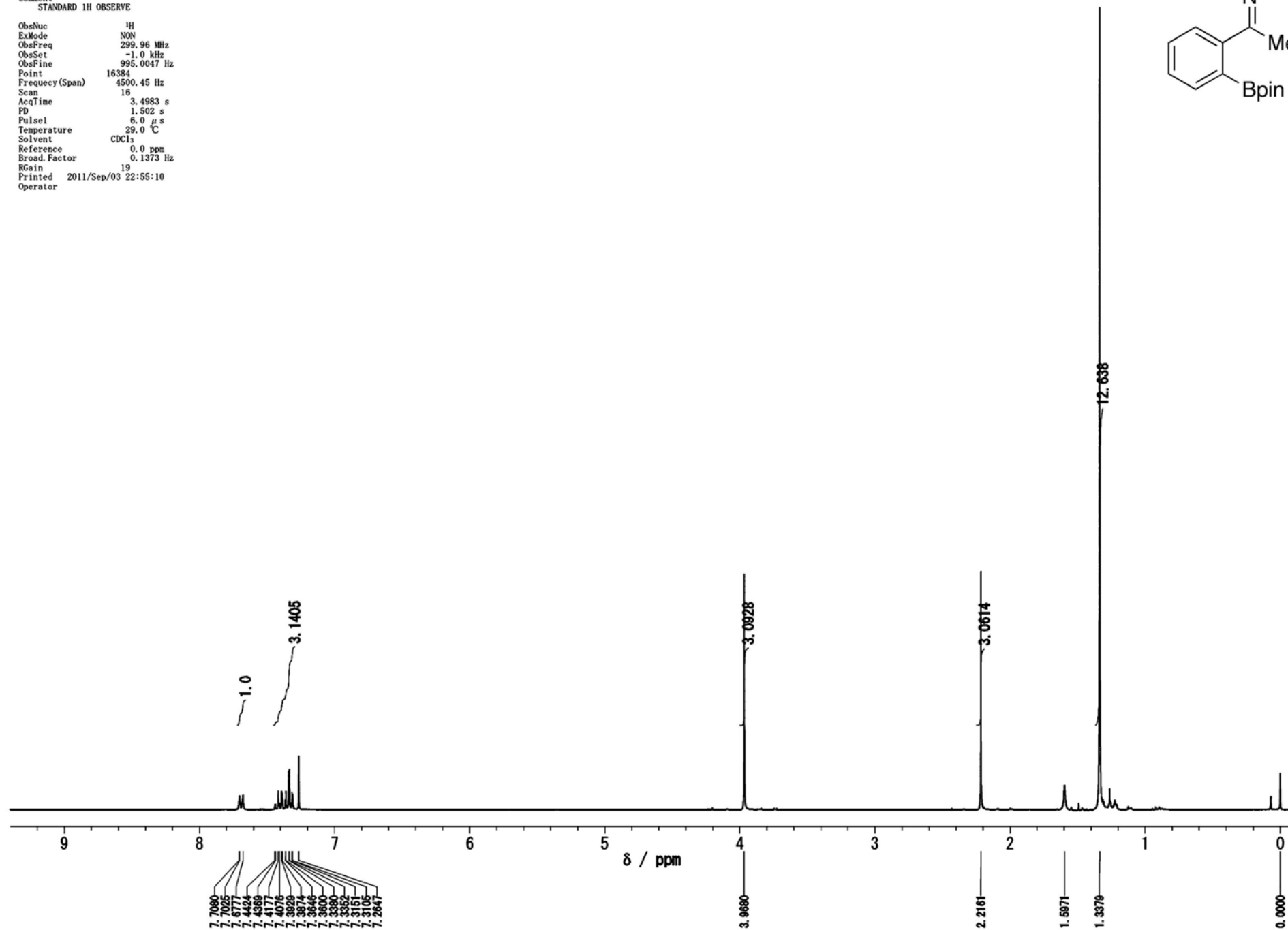
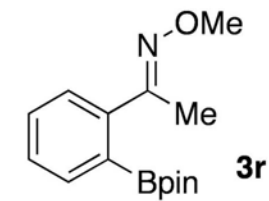
Original File:
Date Jul 25 11
Comment
C13 Statdard Observe
Stick=none Tune=6.4 Match=0.4

ObsNuc ¹³C
ExMode NON
ObsFreq 75.43 MHz
ObsSet -1.0 kHz
ObsFine 996.3672 Hz
Point 32768
Frequency (Span) 18761.73 Hz
Scan 10000
AcqTime 1.4992 s
PD 1.501 s
Pulse 6.0 μ s
Temperature 29.0 $^{\circ}$ C
Solvent CDCl₃
Reference 77.0 ppm
Broad Factor 0.2863 Hz
RGain 30
Printed 2011/Jul/28 15:46:18
Operator



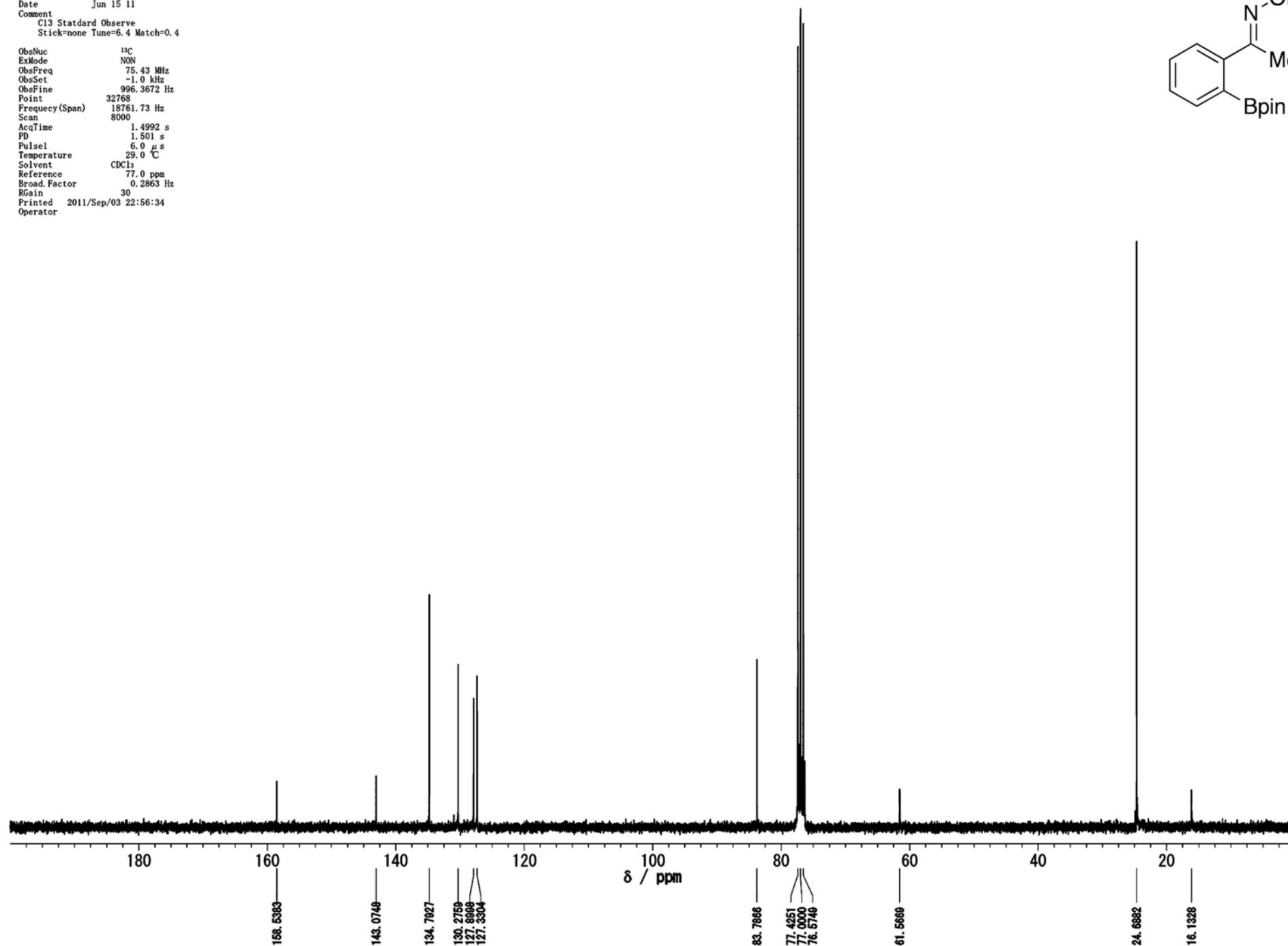
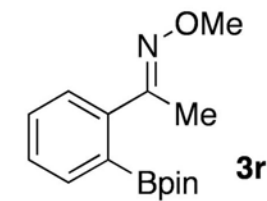
Original File:
 Date Jun 14 11
 Comment
 STANDARD 1H OBSERVE

ObsNuc ¹H
 ExMode NON
 ObsFreq 299.96 MHz
 ObsSet -1.0 kHz
 ObsPine 995.0047 Hz
 Point 16384
 Frequency (Span) 4500.45 Hz
 Scan 16
 AcqTime 3.4983 s
 PD 1.502 s
 Pulse 6.0 μ s
 Temperature 29.0 $^{\circ}$ C
 Solvent CDCl₃
 Reference 0.0 ppm
 Broad Factor 0.1373 Hz
 RGain 19
 Printed 2011/Sep/03 22:55:10
 Operator

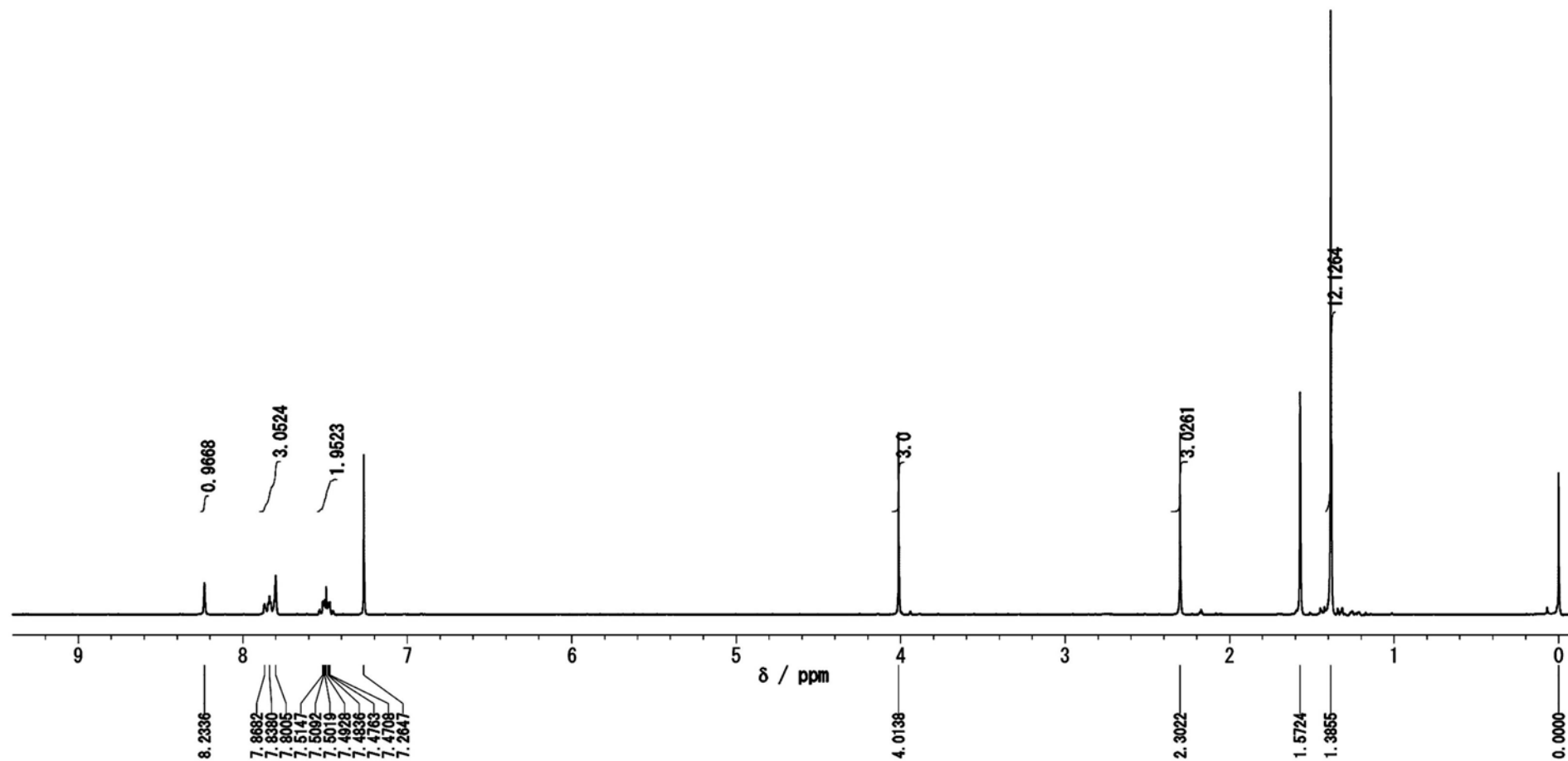
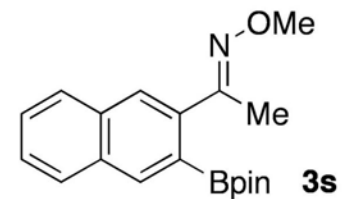


Original File:
 Date Jun 15 11
 Comment
 C13 Statdard Observe
 Stick=none Tune=6.4 Match=0.4

ObsNuc ¹³C
 ExMode NON
 ObsFreq 75.43 MHz
 ObsSet -1.0 kHz
 ObsFine 996.3672 Hz
 Point 32768
 Frequency (Span) 18761.73 Hz
 Scan 8000
 AcqTime 1.4992 s
 PD 1.501 s
 Pulse 6.0 μ s
 Temperature 29.0 $^{\circ}$ C
 Solvent CDCl₃
 Reference 77.0 ppm
 Broad Factor 0.2863 Hz
 RGain 30
 Printed 2011/Sep/03 22:56:34
 Operator

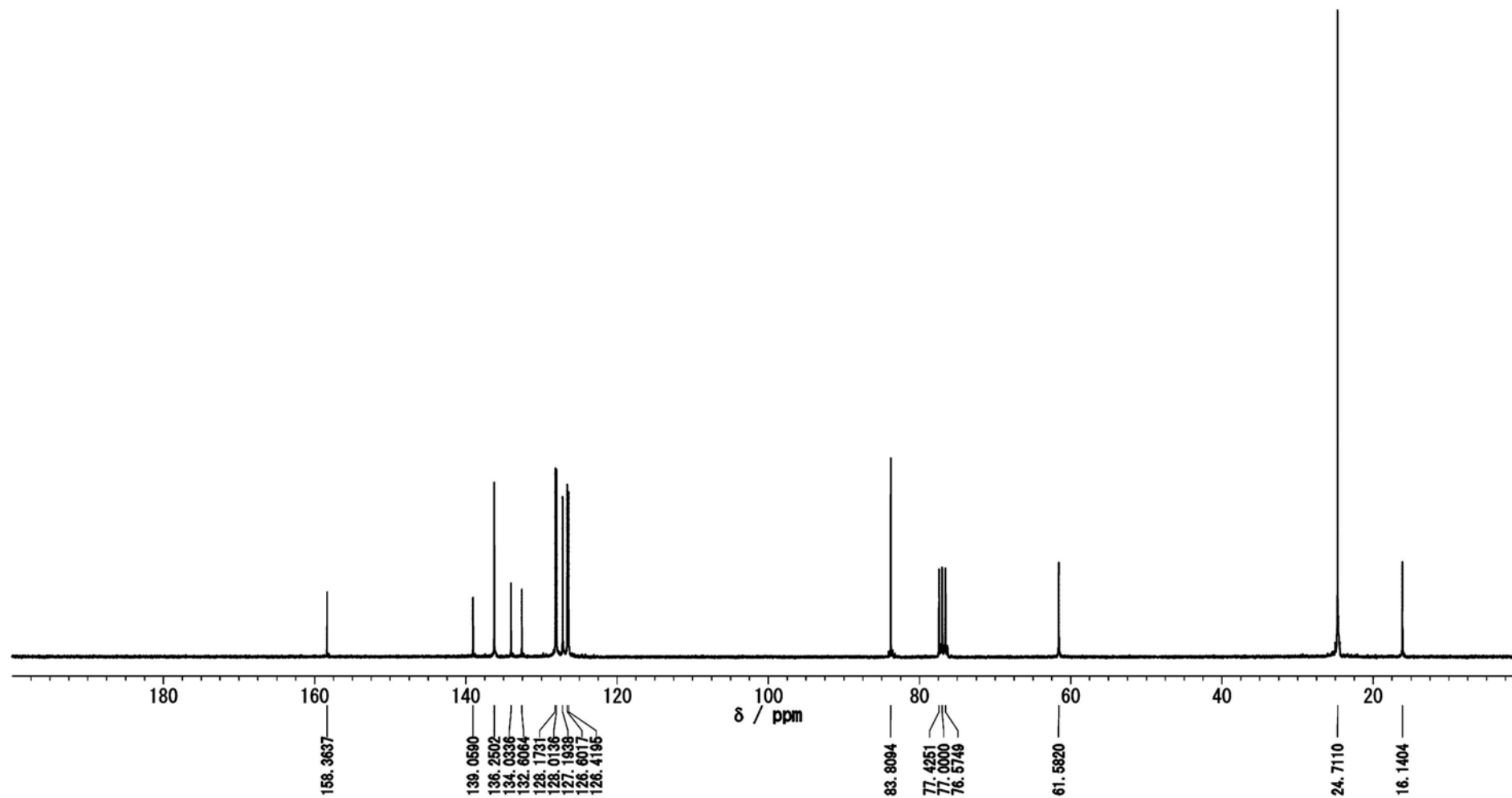
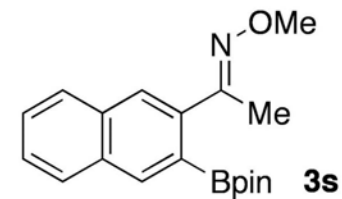


Original File: Jul 21 11
 Date: Jul 21 11
 Comment: STANDARD 1H OBSERVE
 ObsNuc: ¹H
 ExMode: NON
 ObsFreq: 299.96 MHz
 ObsSet: -1.0 kHz
 ObsPine: 995.0047 Hz
 Point: 16384
 Frequency (Span): 4500.45 Hz
 Scan: 16
 AcqTime: 3.4983 s
 PD: 1.502 s
 Pulse: 6.0 μ s
 Temperature: 29.0 $^{\circ}$ C
 Solvent: CDCl₃
 Reference: 0.0 ppm
 Broad. Factor: 0.1373 Hz
 RGain: 26
 Printed: 2011/Jul/28 15:41:39
 Operator:



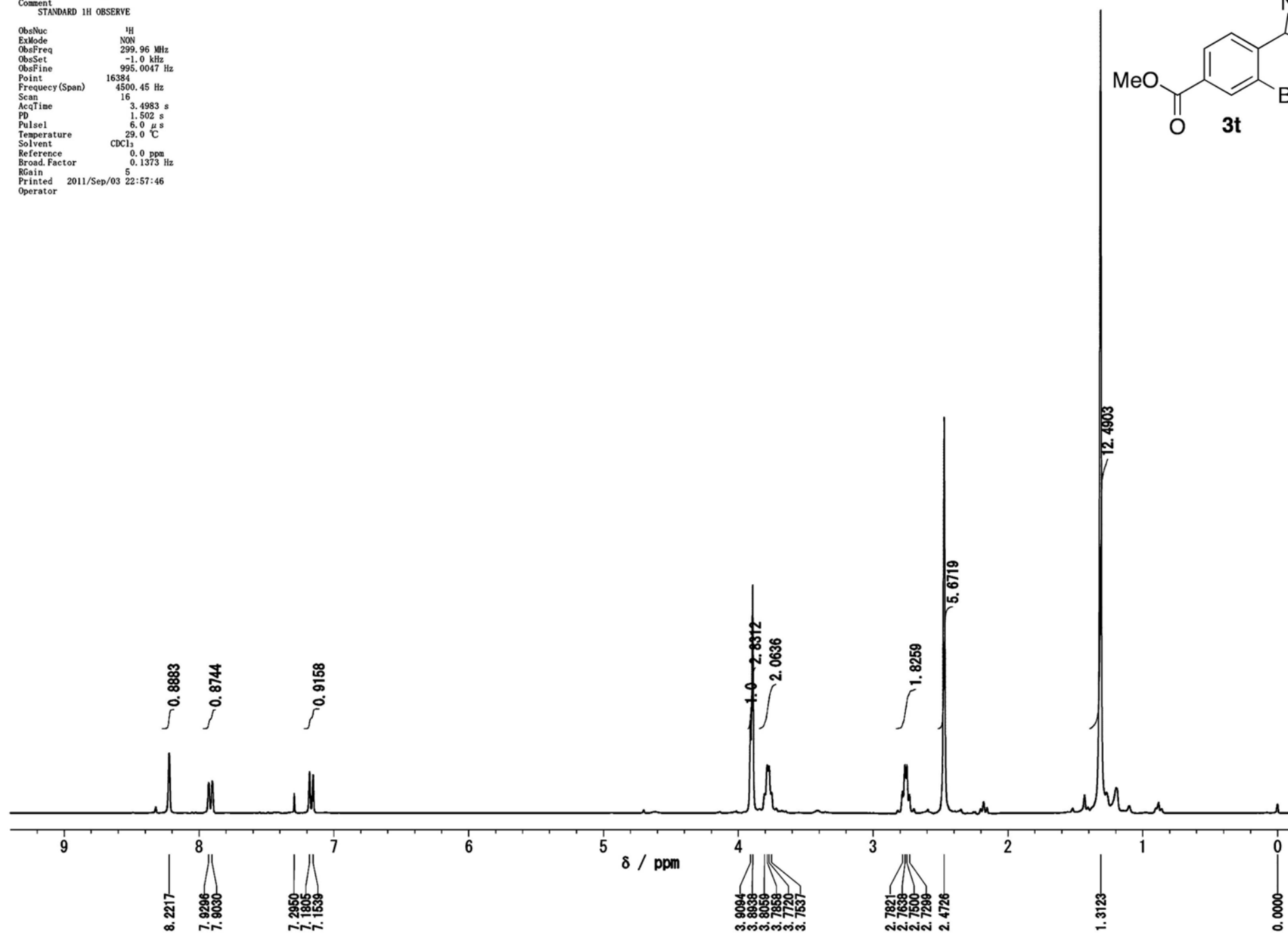
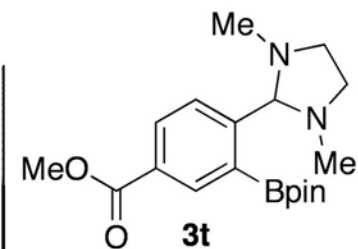
Original File:
Date Jul 21 11
Comment
C13 Statdard Observe
Stick=none Tune=6.4 Match=0.4

ObsNuc ^{13}C
ExMode NON
ObsFreq 75.43 MHz
ObsSet -1.0 kHz
ObsFine 996.3672 Hz
Point 32768
Frequency (Span) 18761.73 Hz
Scan 10000
AcqTime 1.4992 s
PD 1.501 s
Pulse 6.0 μs
Temperature 29.0 $^{\circ}\text{C}$
Solvent CDCl_3
Reference 77.0 ppm
Broad Factor 0.2863 Hz
RGain 30
Printed 2011/Jul/28 15:43:12
Operator



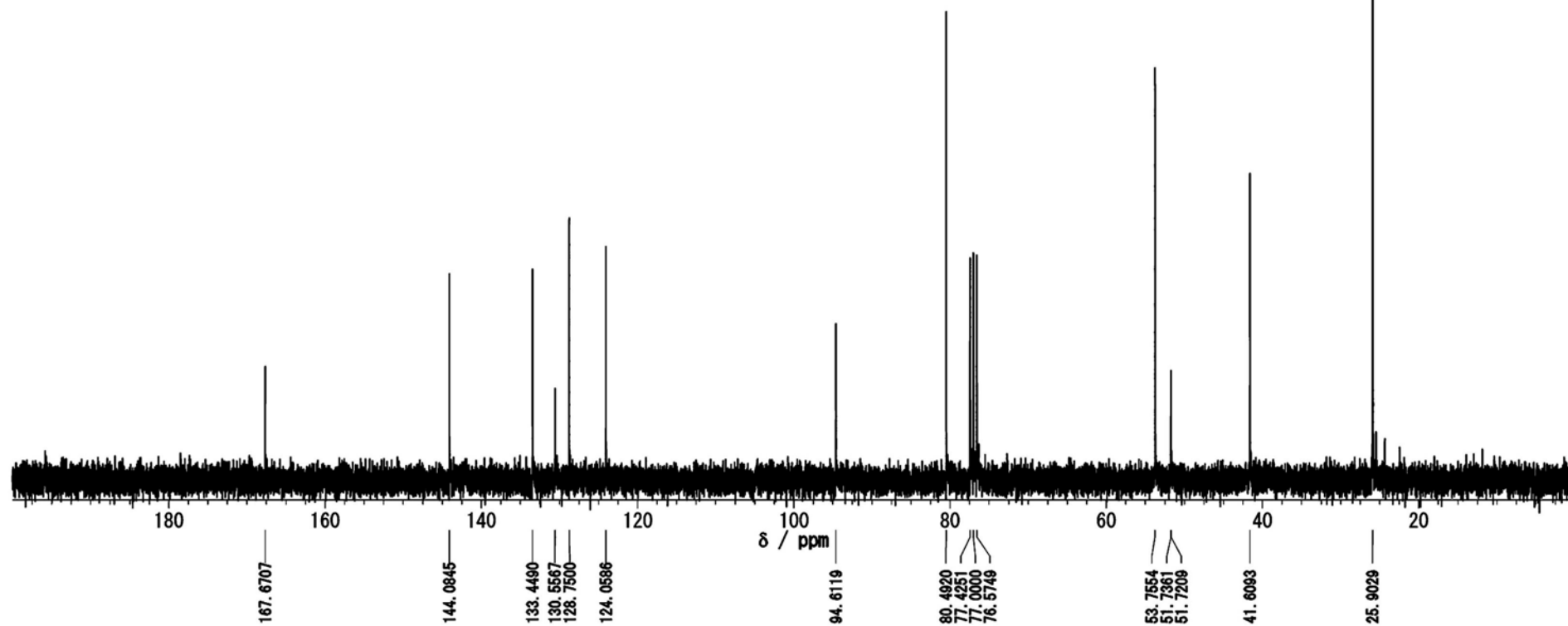
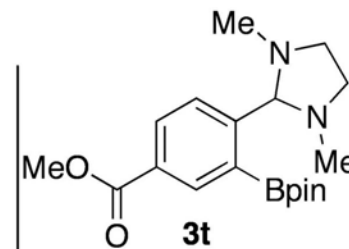
Original File:
 Date Aug 3 11
 Comment
 STANDARD 1H OBSERVE

ObsNuc H
 ExMode NON
 ObsFreq 299.96 MHz
 ObsSet -1.0 kHz
 ObsPine 995.0047 Hz
 Point 16384
 Frequency (Span) 4500.45 Hz
 Scan 16
 AcqTime 3.4983 s
 PD 1.502 s
 Pulse 6.0 μ s
 Temperature 29.0 $^{\circ}$ C
 Solvent CDCl₃
 Reference 0.0 ppm
 Broad Factor 0.1373 Hz
 RGain 5
 Printed 2011/Sep/03 22:57:46
 Operator



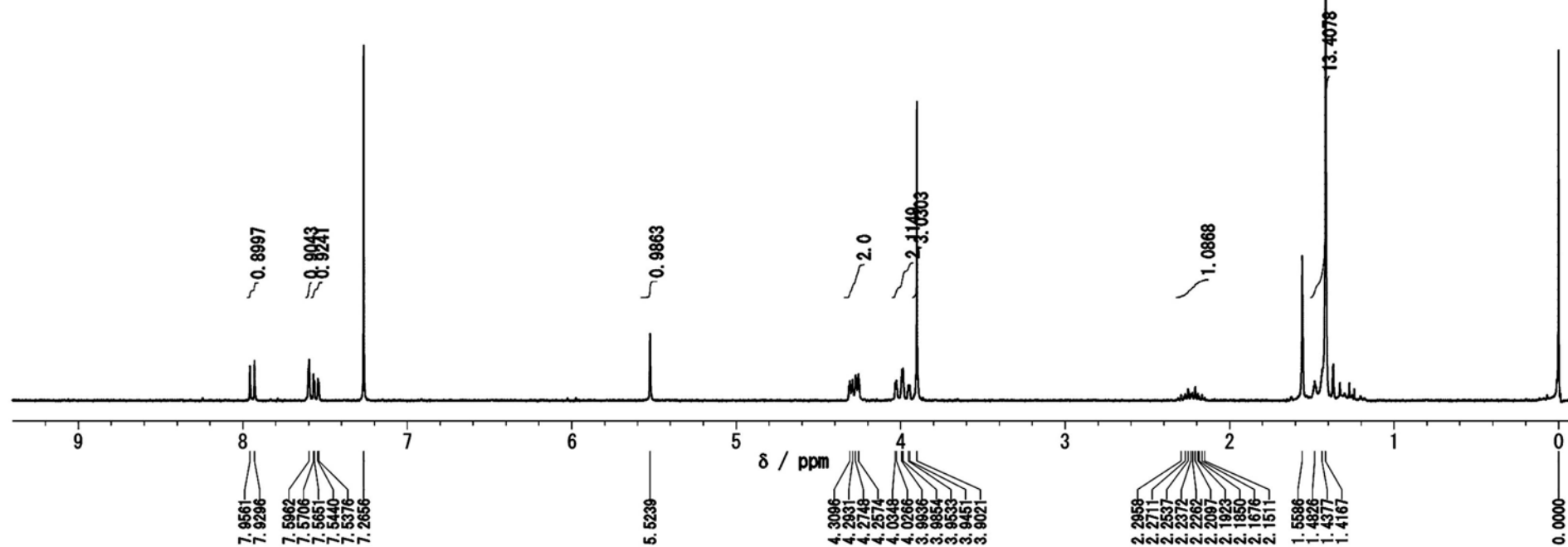
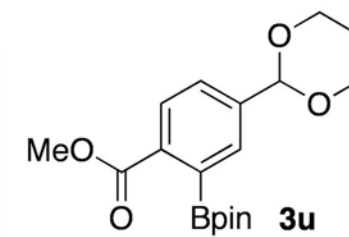
Original File:
 Date Aug 3 11
 Comment
 C13 Statdard Observe
 Stick=none Tune=6.4 Match=0.4

ObsNuc ^{13}C
 ExMode NON
 ObsFreq 75.43 MHz
 ObsSet -1.0 kHz
 ObsFine 996.3672 Hz
 Point 32768
 Frequency (Span) 18761.73 Hz
 Scan 112
 AcqTime 1.4992 s
 PD 1.501 s
 Pulsel 6.0 μs
 Temperature 29.0 $^{\circ}\text{C}$
 Solvent CDCl_3
 Reference 77.0 ppm
 Broad Factor 0.2863 Hz
 RGain 30
 Printed 2011/Sep/03 22:58:48
 Operator



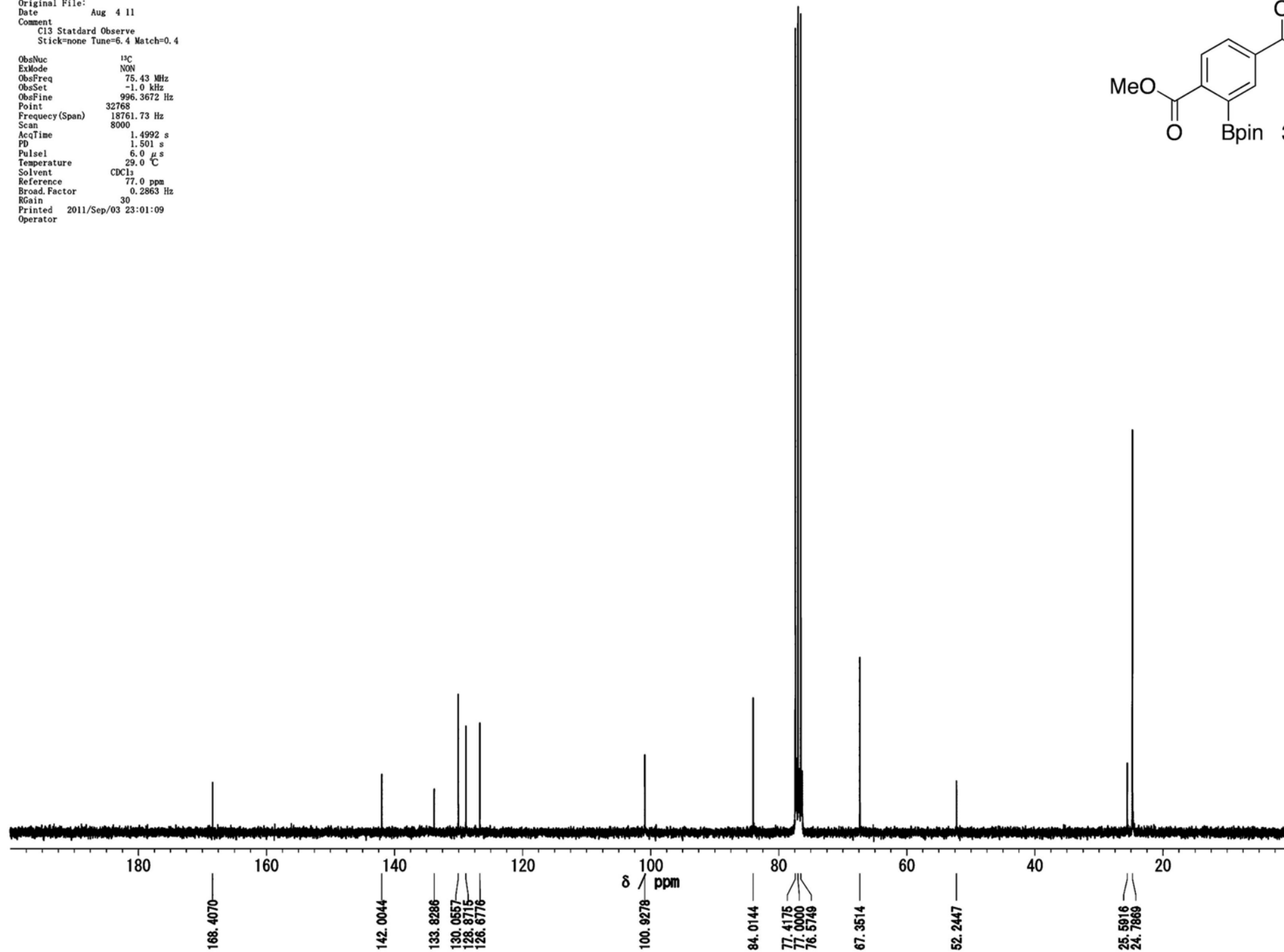
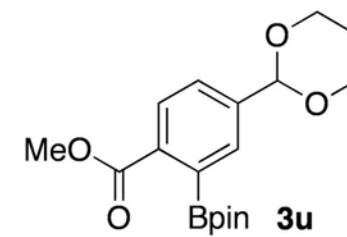
Original File:
 Date Aug 3 11
 Comment
 STANDARD 1H OBSERVE

ObsNuc ¹H
 ExMode NON
 ObsFreq 299.96 MHz
 ObsSet -1.0 kHz
 ObsPine 995.0047 Hz
 Point 16384
 Frequency (Span) 4500.45 Hz
 Scan 16
 AcqTime 3.4983 s
 PD 1.502 s
 Pulse 6.0 μ s
 Temperature 29.0 $^{\circ}$ C
 Solvent CDCl₃
 Reference 0.0 ppm
 Broad Factor 0.1373 Hz
 RGain 30
 Printed 2011/Sep/03 22:59:54
 Operator



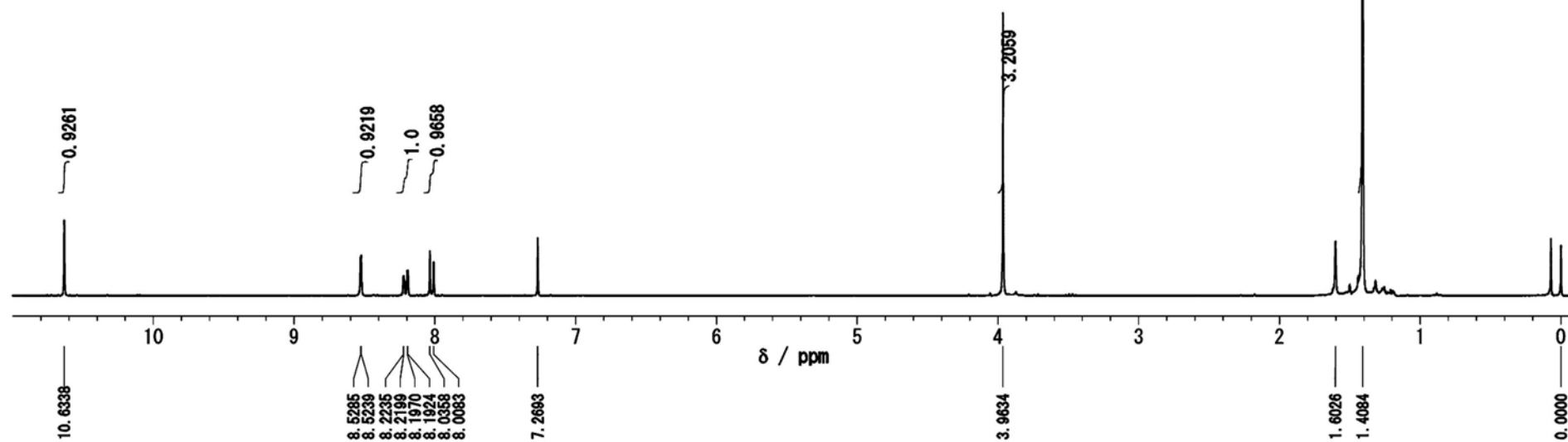
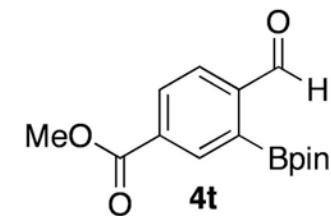
Original File:
 Date Aug 4 11
 Comment
 C13 Statdard Observe
 Stick=none Tune=6.4 Match=0.4

ObsNuc ^{13}C
 ExMode NON
 ObsFreq 75.43 MHz
 ObsSet -1.0 kHz
 ObsFine 996.3672 Hz
 Point 32768
 Frequency (Span) 18761.73 Hz
 Scan 8000
 AcqTime 1.4992 s
 PD 1.501 s
 Pulsel 6.0 μs
 Temperature 29.0 $^{\circ}\text{C}$
 Solvent CDCl_3
 Reference 77.0 ppm
 Broad.Factor 0.2863 Hz
 RGain 30
 Printed 2011/Sep/03 23:01:09
 Operator



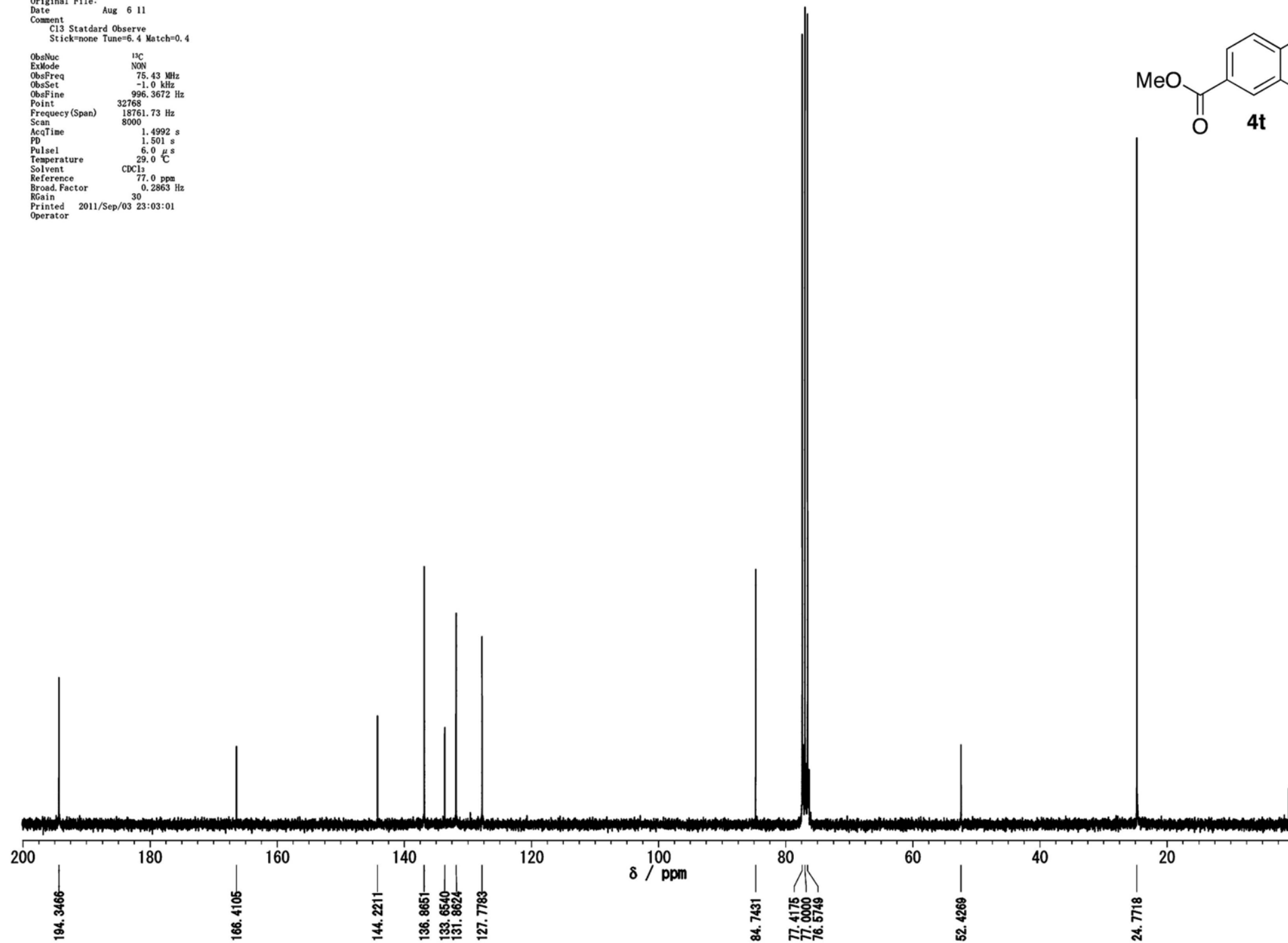
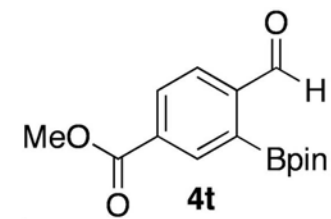
Original File:
 Date Aug 5 11
 Comment
 STANDARD 1H OBSERVE

ObsNuc ¹H
 ExMode NON
 ObsFreq 299.96 MHz
 ObsSet -1.0 kHz
 ObsPine 995.0047 Hz
 Point 16384
 Frequency (Span) 4500.45 Hz
 Scan 16
 AcqTime 3.4983 s
 PD 1.502 s
 Pulse 6.0 μ s
 Temperature 29.0 $^{\circ}$ C
 Solvent CDCl₃
 Reference 0.0 ppm
 Broad. Factor 0.1373 Hz
 RGain 19
 Printed 2011/Sep/03 23:02:08
 Operator



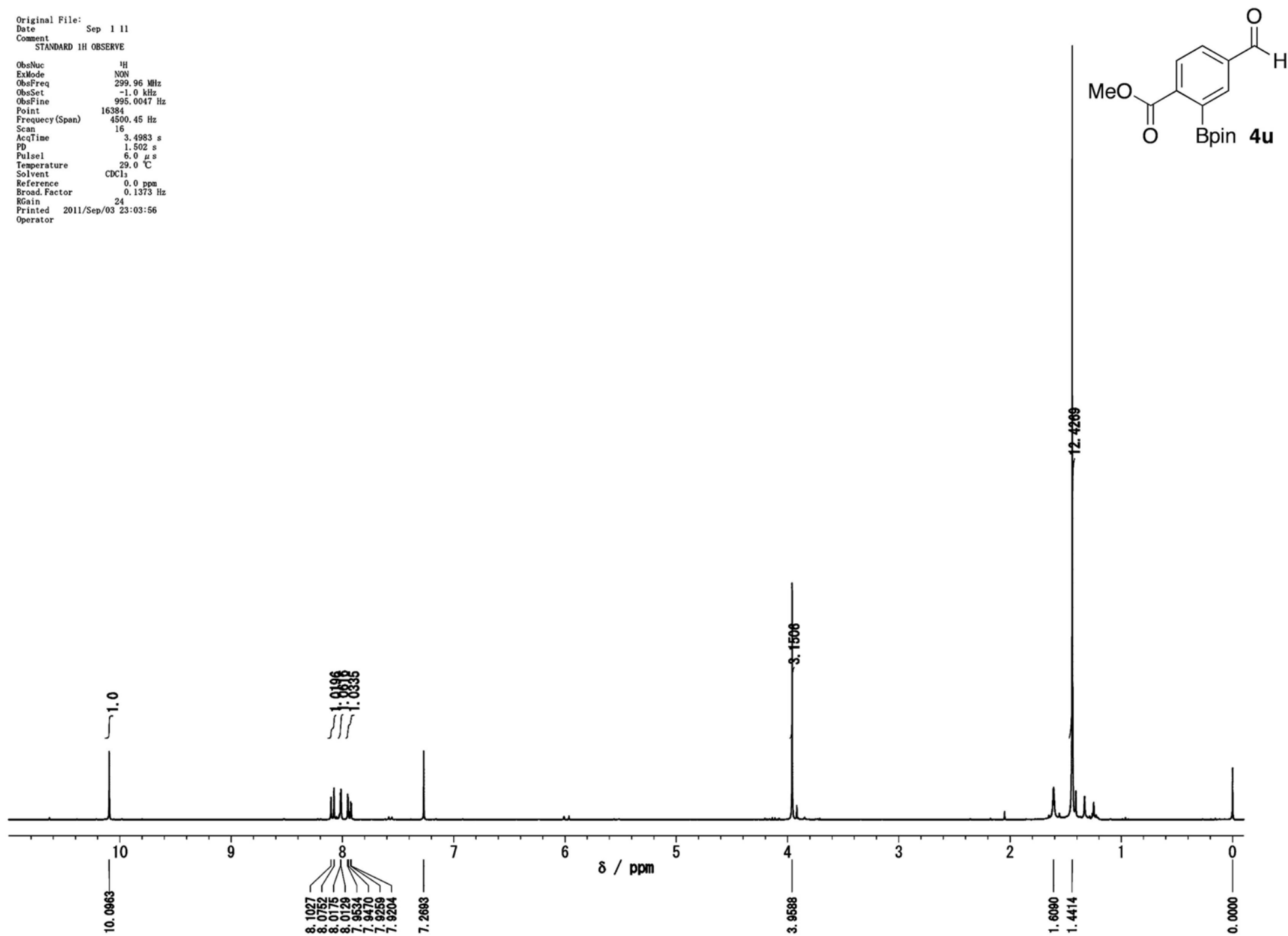
Original File:
 Date Aug 6 11
 Comment
 C13 Statdard Observe
 Stick=none Tune=6.4 Match=0.4

ObsNuc ¹³C
 ExMode NON
 ObsFreq 75.43 MHz
 ObsSet -1.0 kHz
 ObsFine 996.3672 Hz
 Point 32768
 Frequency (Span) 18761.73 Hz
 Scan 8000
 AcqTime 1.4992 s
 PD 1.501 s
 Pulsel 6.0 μ s
 Temperature 29.0 $^{\circ}$ C
 Solvent CDCl₃
 Reference 77.0 ppm
 Broad.Factor 0.2863 Hz
 RGain 30
 Printed 2011/Sep/03 23:03:01
 Operator



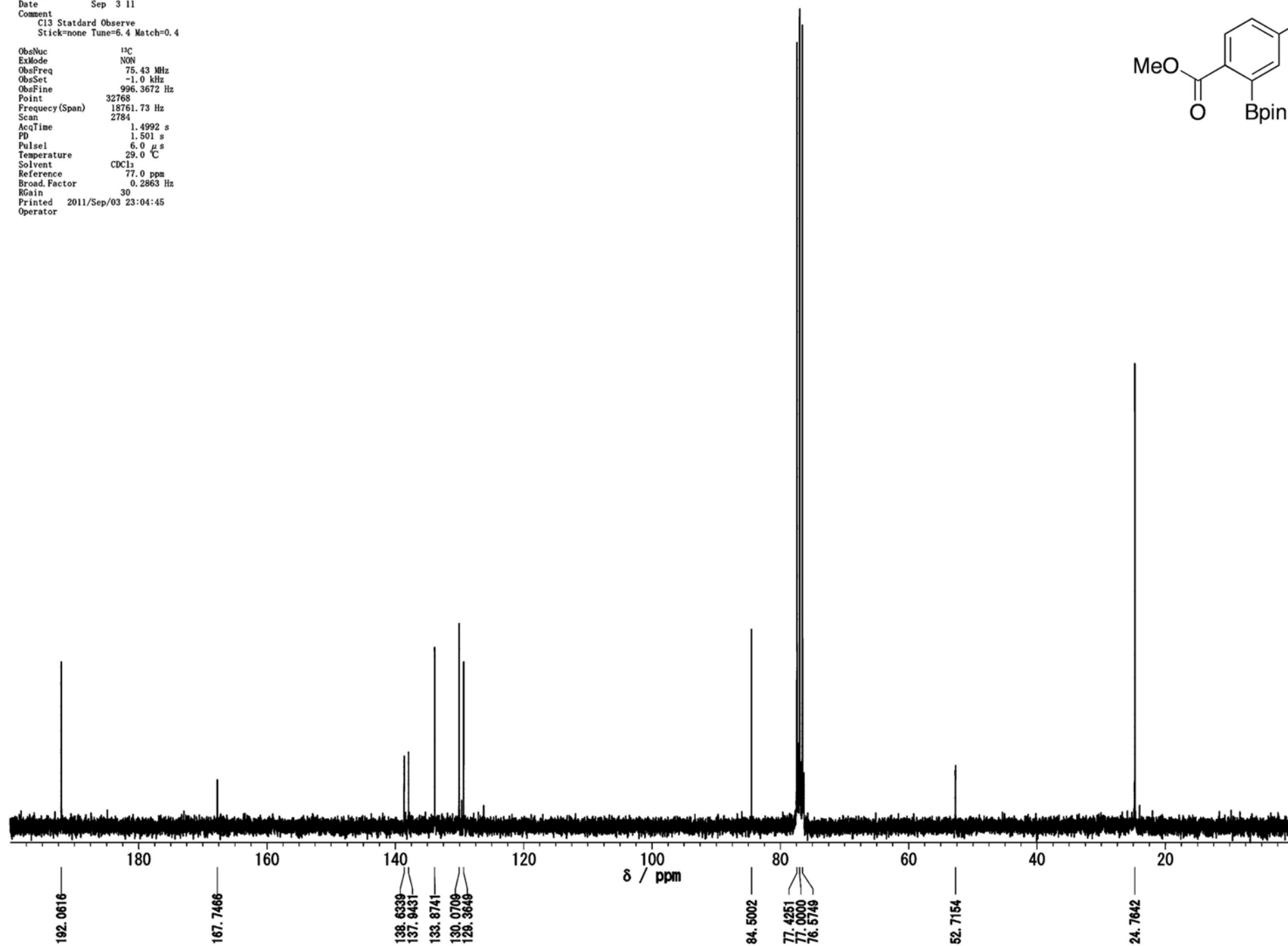
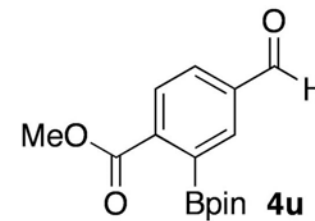
Original File:
 Date Sep 1 11
 Comment
 STANDARD 1H OBSERVE

ObsNuc ¹H
 ExMode NON
 ObsFreq 299.96 MHz
 ObsSet -1.0 kHz
 ObsPine 995.0047 Hz
 Point 16384
 Frequency (Span) 4500.45 Hz
 Scan 16
 AcqTime 3.4983 s
 PD 1.502 s
 Pulse 6.0 μ s
 Temperature 29.0 $^{\circ}$ C
 Solvent CDCl₃
 Reference 0.0 ppm
 Broad. Factor 0.1373 Hz
 RGain 24
 Printed 2011/Sep/03 23:03:56
 Operator



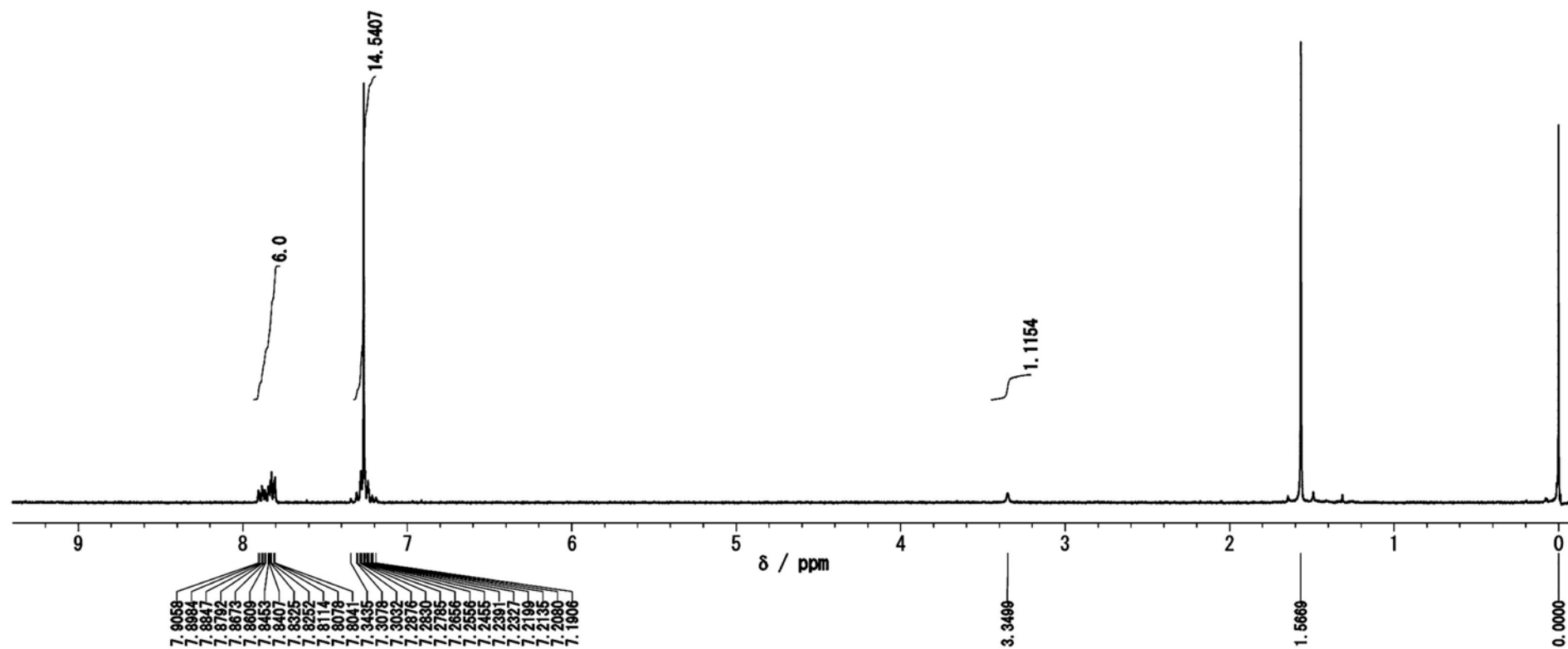
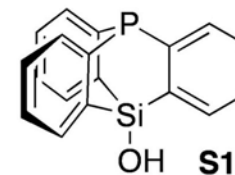
Original File:
 Date Sep 3 11
 Comment
 C13 Statdard Observe
 Stick=none Tune=6.4 Match=0.4

ObsNuc ¹³C
 ExMode NON
 ObsFreq 75.43 MHz
 ObsSet -1.0 kHz
 ObsFine 996.3672 Hz
 Point 32768
 Frequency (Span) 18761.73 Hz
 Scan 2784
 AcqTime 1.4992 s
 PD 1.501 s
 Pulsel 6.0 μ s
 Temperature 29.0 $^{\circ}$ C
 Solvent CDCl₃
 Reference 77.0 ppm
 Broad.Factor 0.2863 Hz
 RGain 30
 Printed 2011/Sep/03 23:04:45
 Operator



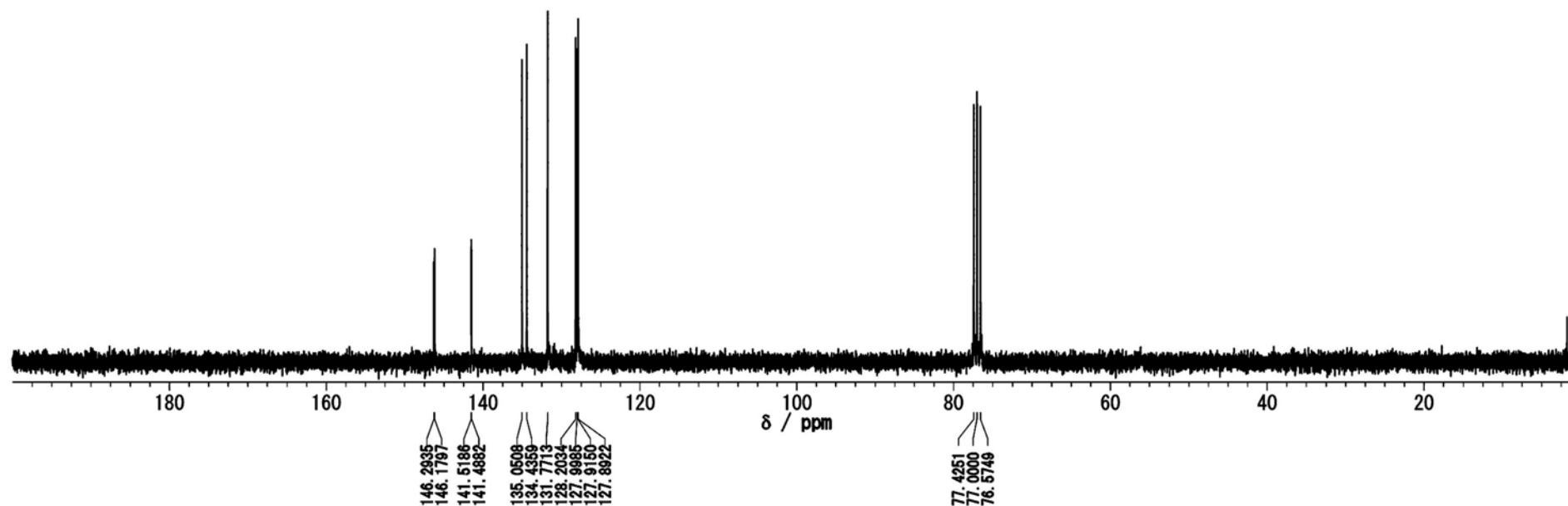
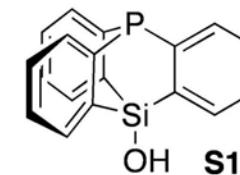
Original File:
Date Aug 25 11
Comment
STANDARD 1H OBSERVE

ObsNuc H
ExMode NON
ObsFreq 299.96 MHz
ObsSet -1.0 kHz
ObsFine 995.0047 Hz
Point 16384
Frequency (Span) 4500.45 Hz
Scan 16
AcqTime 3.4983 s
PD 1.502 s
Pulse 6.0 μ s
Temperature 29.0 $^{\circ}$ C
Solvent CDCl₃
Reference 0.0 ppm
Broad Factor 0.1373 Hz
RGain 30
Printed 2011/Sep/01 20:51:19
Operator



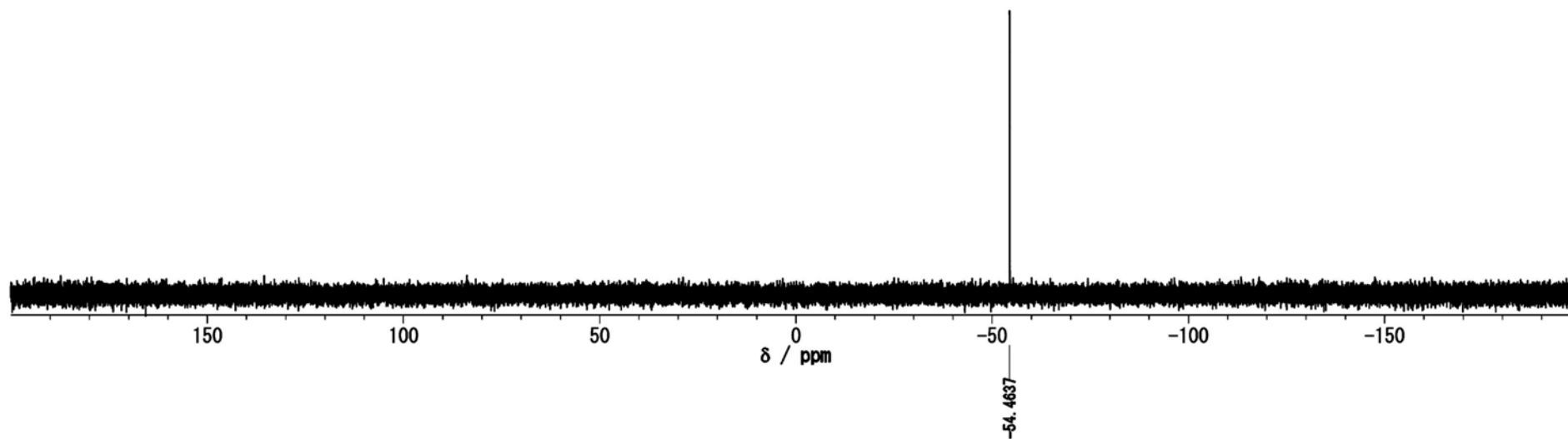
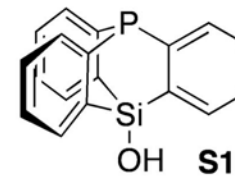
Original File:
 Date Aug 30 10
 Comment
 C13 Statdard Observe
 Stick=none Tune=6.4 Match=0.4

ObsNuc ^{13}C
 ExMode NON
 ObsFreq 75.43 MHz
 ObsSet -1.0 kHz
 ObsFine 996.3672 Hz
 Point 32768
 Frequency (Span) 18761.73 Hz
 Scan 256
 AcqTime 1.4992 s
 PD 1.501 s
 Pulse 6.0 μs
 Temperature 29.0 $^{\circ}\text{C}$
 Solvent CDCl_3
 Reference 77.0 ppm
 Broad Factor 0.2863 Hz
 RGain 30
 Printed 2011/Sep/01 20:45:59
 Operator



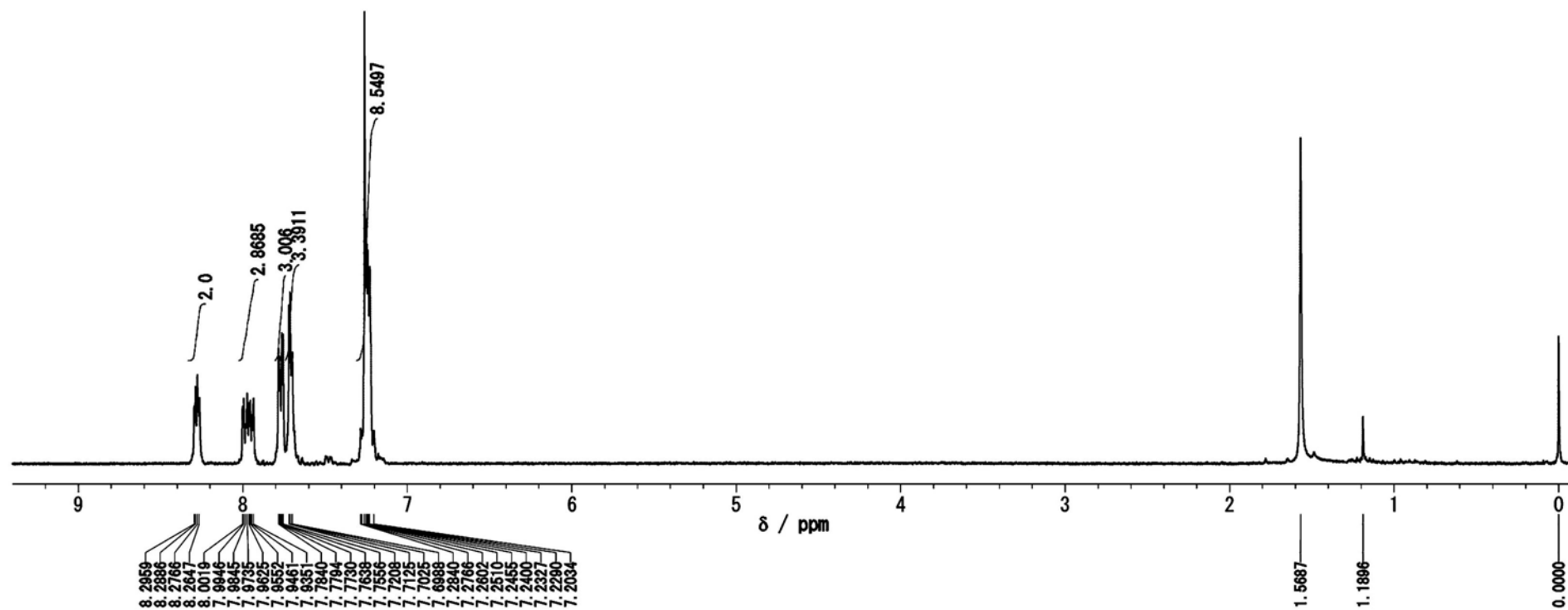
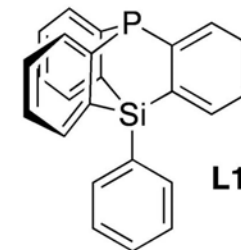
Original File:
Date Dec 17 10
Comment
P-31 STANDARD PARAMETERS
PHOSPHATE REGION

ObsNuc ³¹P
ExMode NON
ObsFreq 121.42 MHz
ObsSet 0.0 kHz
ObsFine 0.7799 Hz
Point 65536
Frequency (Span) 50000.0 Hz
Scan 64
AcqTime 1.28 s
PD 1.4 s
Pulse 6.8 μ s
Temperature 29.0 $^{\circ}$ C
Solvent CDCl₃
Reference 0.0 ppm
Broad Factor 0.3815 Hz
RGain 30
Printed 2011/Sep/05 12:02:19
Operator



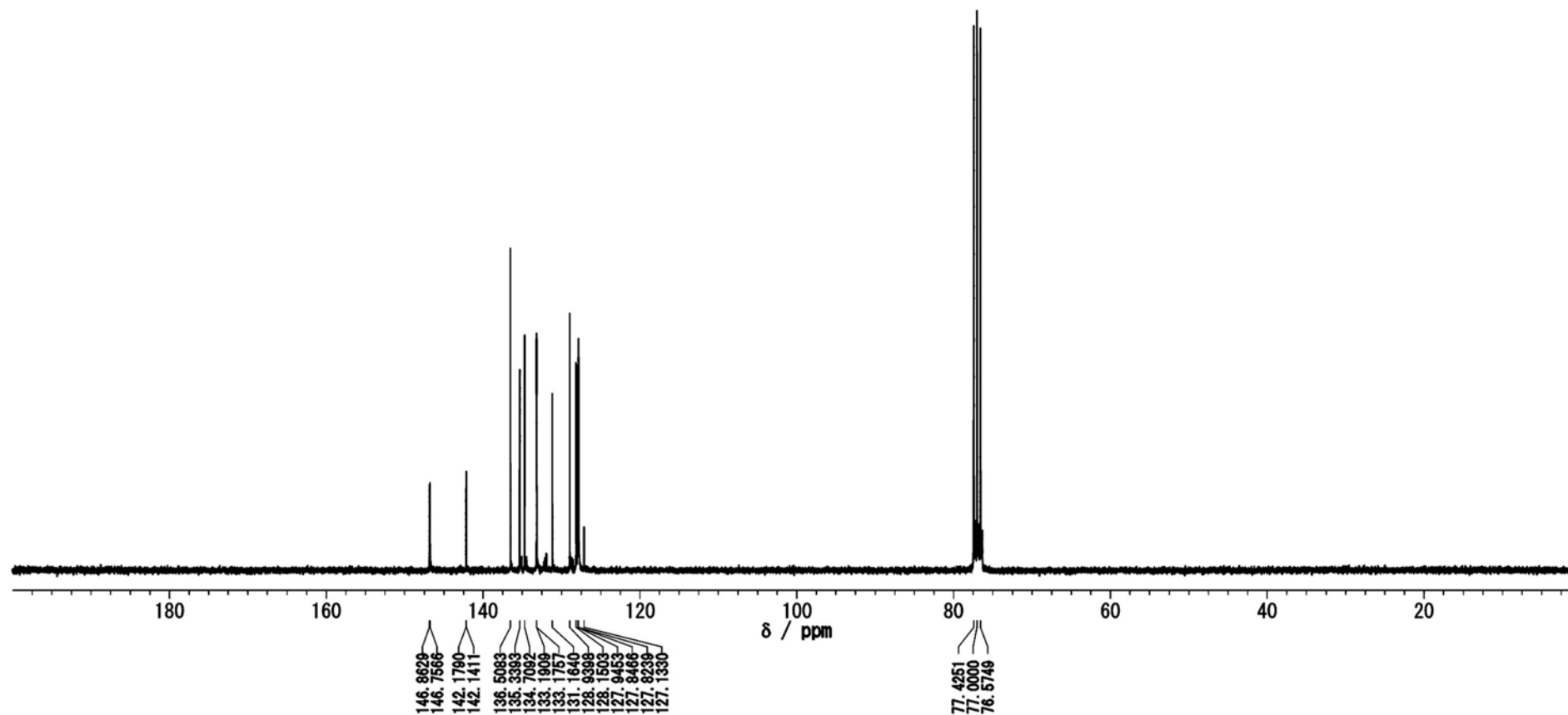
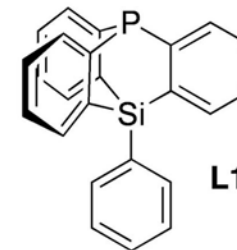
Original File:
Date May 27 11
Comment
STANDARD 1H OBSERVE

ObsNuc ¹H
ExMode NON
ObsFreq 299.96 MHz
ObsSet -1.0 kHz
ObsFine 995.0047 Hz
Point 16384
Frequency (Span) 4500.45 Hz
Scan 16
AcqTime 3.4983 s
PD 1.502 s
Pulse1 6.0 μ s
Temperature 29.0 $^{\circ}$ C
Solvent CDCl₃
Reference 0.0 ppm
Broad Factor 0.1373 Hz
RGain 24
Printed 2011/Sep/01 20:53:24
Operator

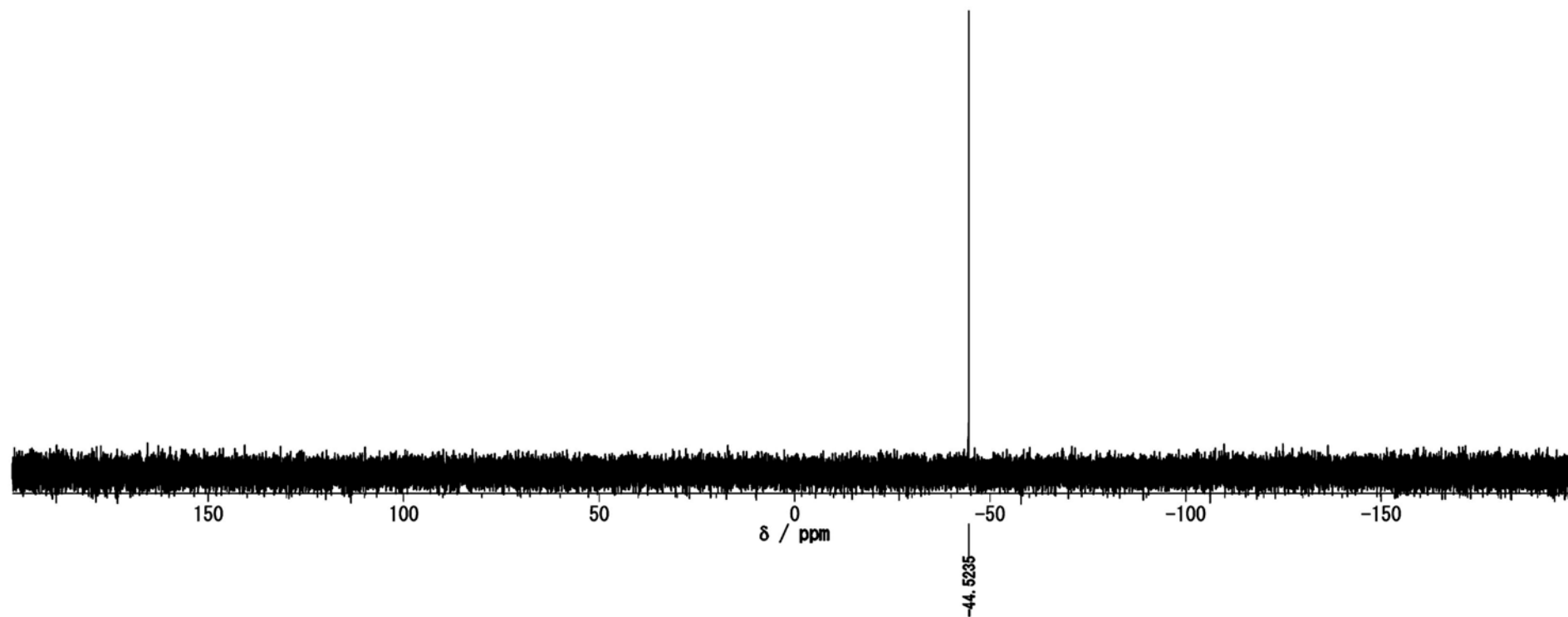
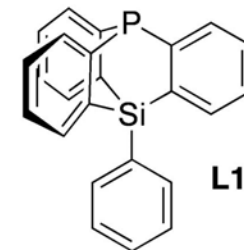


Original File:
 Date Aug 23 11
 Comment
 C13 Statdard Observe
 Stick=none Tune=6.4 Match=0.4

ObsNuc ¹³C
 ExMode NON
 ObsFreq 75.43 MHz
 ObsSet -1.0 kHz
 ObsFine 996.3672 Hz
 Point 32768
 Frequency (Span) 18761.73 Hz
 Scan 10128
 AcqTime 1.4992 s
 PD 1.501 s
 Pulsel 6.0 μ s
 Temperature 29.0 $^{\circ}$ C
 Solvent CDCl₃
 Reference 77.0 ppm
 Broad.Factor 0.2863 Hz
 RGain 30
 Printed 2011/Sep/01 20:43:15
 Operator

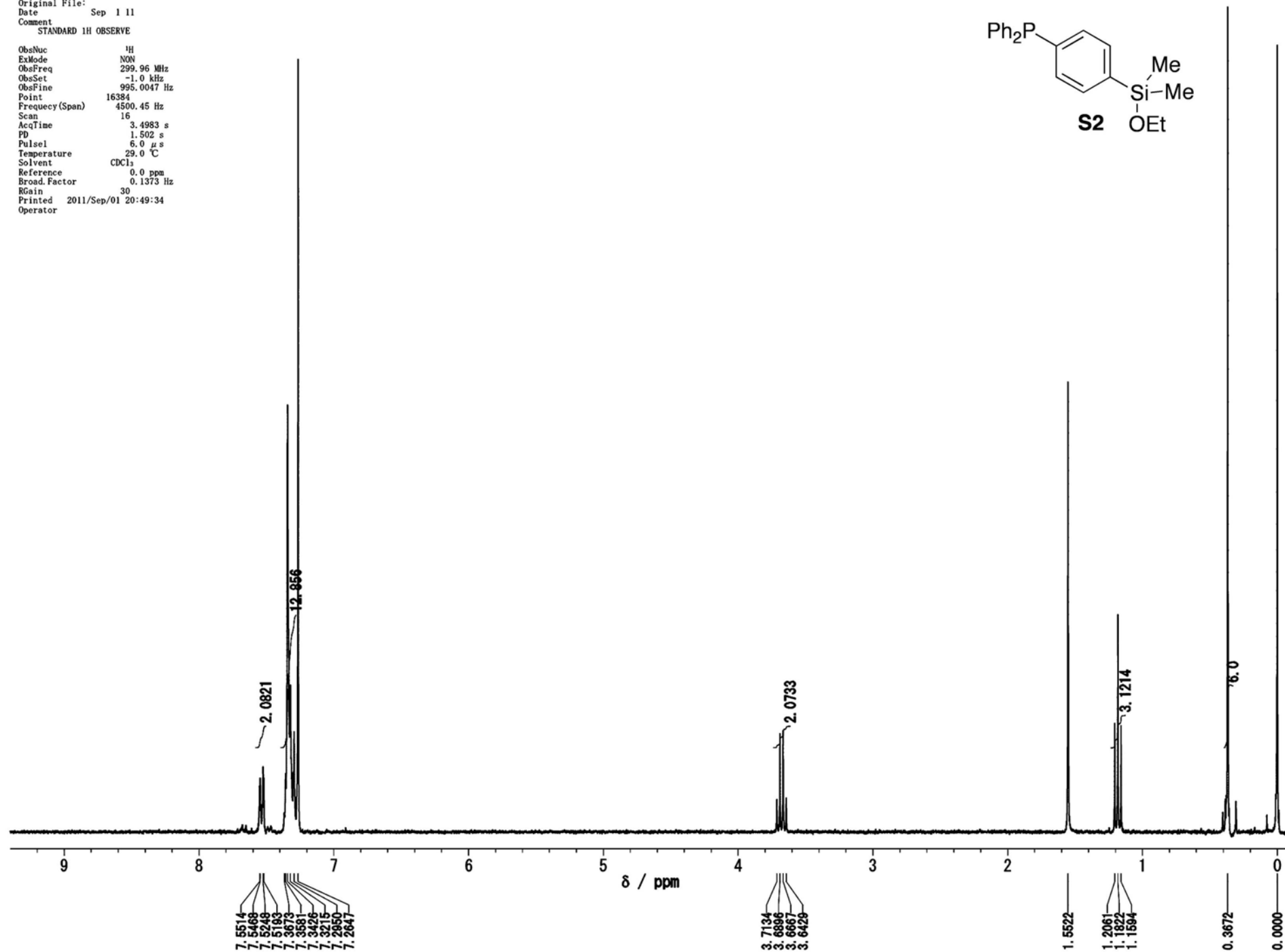
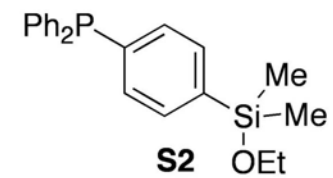


Date May 27 11
 Comment P-31 STANDARD PARAMETERS
 PHOSPHATE REGION
 ObsNuc ³¹P
 ExMode
 ObsFreq 121.42 MHz
 ObsSet 0.0 kHz
 ObsFine 0.7799 Hz
 Point 65536
 Frequency (Span) 50000.0 Hz
 Scan 64
 AcqTime 1.28 s
 PD 1.4 s
 Pulse1 6.8 μ s
 Temperature 29.0 $^{\circ}$ C
 Solvent CDCl₃
 Reference 0.0 ppm
 Broad. Factor 0.25 Hz
 RGain 30
 Printed 2011/Sep/05 12:04:18
 Operator



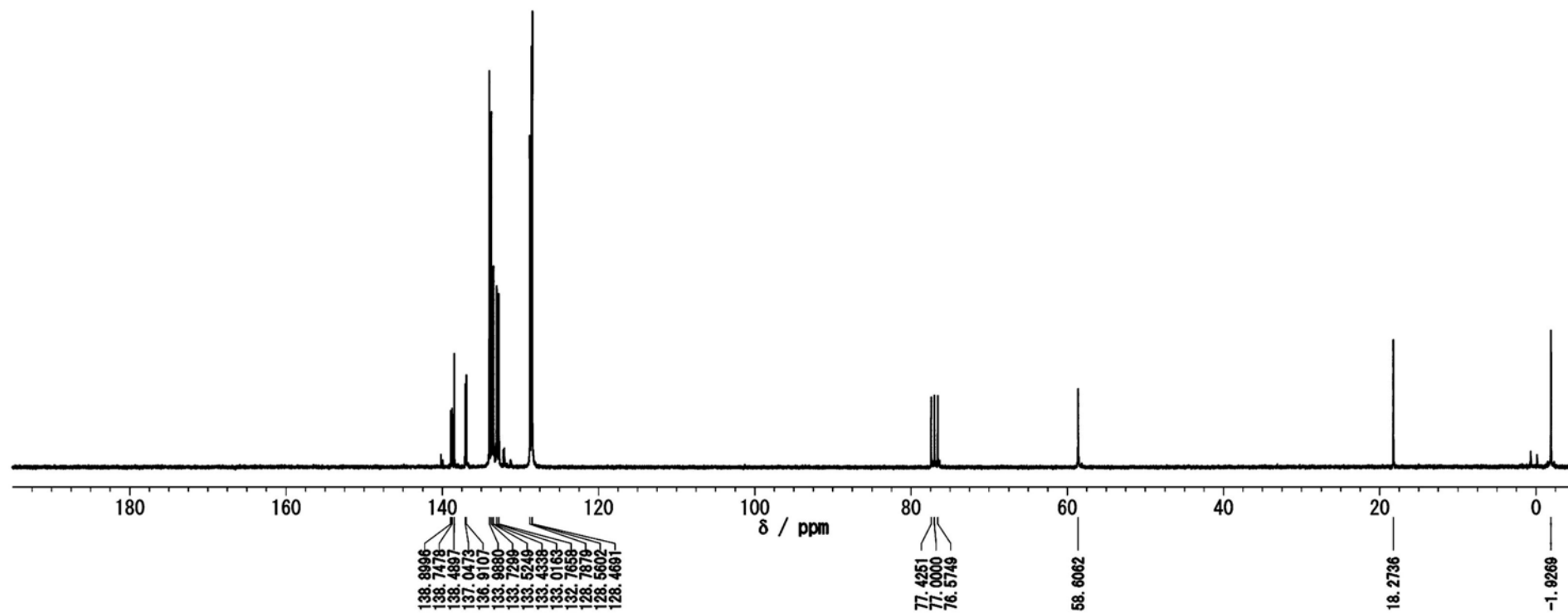
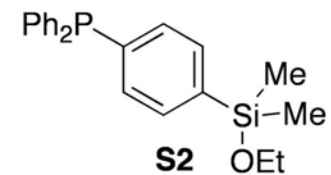
Original File:
 Date Sep 1 11
 Comment
 STANDARD 1H OBSERVE

ObsNuc H
 ExMode NON
 ObsFreq 299.96 MHz
 ObsSet -1.0 kHz
 ObsPine 995.0047 Hz
 Point 16384
 Frequency (Span) 4500.45 Hz
 Scan 16
 AcqTime 3.4983 s
 PD 1.502 s
 Pulse 6.0 μ s
 Temperature 29.0 $^{\circ}$ C
 Solvent CDCl₃
 Reference 0.0 ppm
 Broad Factor 0.1373 Hz
 RGain 30
 Printed 2011/Sep/01 20:49:34
 Operator

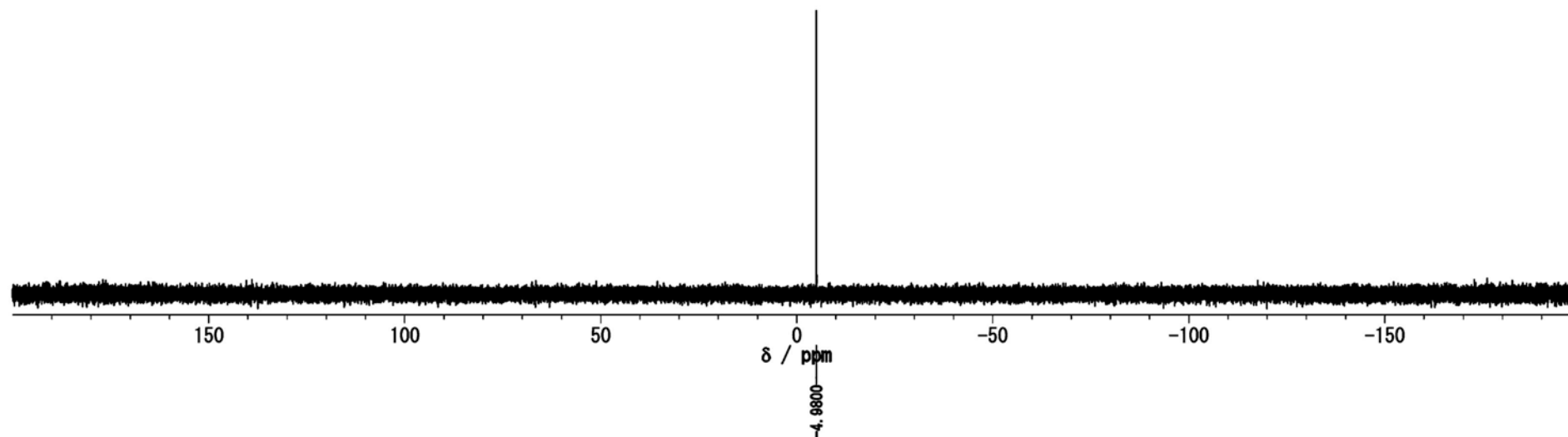
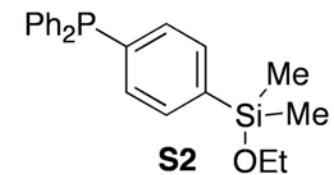


Original File:
 Date Sep 1 11
 Comment
 C13 Statdard Observe
 Stick=none Tune=6.4 Match=0.4

ObsNuc ¹³C
 ExMode NON
 ObsFreq 75.43 MHz
 ObsSet -1.0 kHz
 ObsFine 996.3672 Hz
 Point 32768
 Frequency (Span) 18761.73 Hz
 Scan 1152
 AcqTime 1.4992 s
 PD 1.501 s
 Pulsel 6.0 μ s
 Temperature 29.0 $^{\circ}$ C
 Solvent CDCl₃
 Reference 77.0 ppm
 Broad.Factor 0.2863 Hz
 RGain 30
 Printed 2011/Sep/01 20:48:07
 Operator



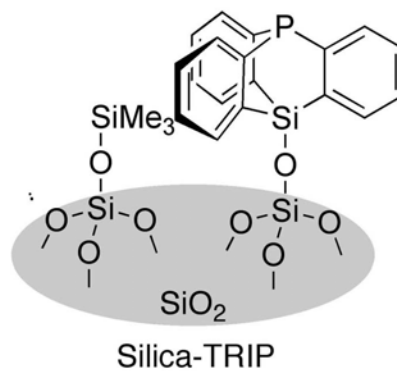
Date Sep 5 11
 Comment P-31 STANDARD PARAMETERS
 PHOSPHATE REGION
 ObsNuc ³¹P
 ExMode
 ObsFreq 121.42 MHz
 ObsSet 0.0 kHz
 ObsFine 0.7799 Hz
 Point 65536
 Frequency (Span) 50000.0 Hz
 Scan 64
 AcqTime 1.28 s
 PD 1.4 s
 Pulse 6.8 μ s
 Temperature 29.0 $^{\circ}$ C
 Solvent CDCl₃
 Reference -4.98 ppm
 Broad Factor 0.25 Hz
 RGain 30
 Printed 2011/Sep/05 12:27:44
 Operator



31P CP/MAS TMZ-041

PPM

-51.8697



MIYA31P1.614
PPG:
CPCYCL.PC
DATE 17-3-7

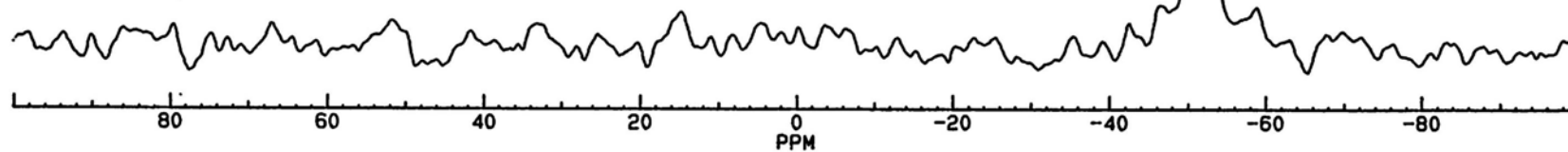
SF 121.496
O1 673.061
SI 8192
TD 1870
SW 35714.286
HZ/PT 8.719

RG 10
NE 1
NS 1300
TE 295

DW 14.0
FW 42900
O2 4980.000
DP 10H B0

D0 10.000S
D1 4.000U
D2 26.000U
D3 16.000U
D4 1.000U
D5 1000.000U
D6 1.000U
D7 26.000M
D11 4.000L
D12 1.000L

LB 150.000
GB 0.0
NC -1
CX 23.50
CY 12.00
SR 681.78





MIYA31P1.609
PPG:
CPCYCL.PC
DATE 9-6-11

SF 121.496
O1 673.061
SI 8192
TD 1870
SW 35714.286
HZ/PT 8.719

RG 5
NE 1
NS 6811
TE 295

DW 14.0
FW 42900
O2 4980.000
DP 10H D0

D0 10.000S
D1 4.000U
D2 26.000U
D3 16.000U
D4 1.000U
D5 1000.000U
D6 1.000U
D7 25.000M
D11 4.400U
D12 1.000U

LB 80.000
GB 0.0
NC -2
CX 23.00
CY 11.00
SR 699.22

