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Graphical Abstract

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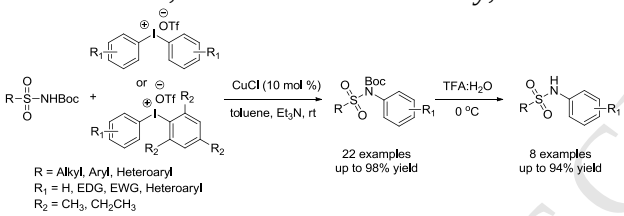

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ABSTRACT

A new and mild synthetic approach for the synthesis of *N*-arylsulfonamides under copper-catalyzed conditions at room temperature has been developed. The reaction employs various *tert*-butyl *N*-sulfonylcarbamates and diaryliodonium salts to avoid potential genotoxic impurities. A one-pot coupling/Boc deprotection sequence is also reported to provide mono *N*-arylsulfonamides in good to excellent yields.

1. Introduction

N-Arylsulfonamides are a ubiquitous pharmacophore in many important medicinal compounds (Figure 1).[1] Due to their anticancer, antibacterial, anticonvulsant and HIV protease inhibitory activities, these arylsulfonamide units have been readily used in many medicinal chemistry endeavors.[1a, 2] These functional groups are normally synthesized from the straightforward reaction of an aromatic amine and a sulfonyl chloride in the presence of base (Scheme 1, eq 1). Unfortunately, the reactants used in this simple approach are mutagenic or genotoxic which can induce genetic mutation or act as carcinogenic compounds.[3] Thus, alternative synthetic methods utilizing the metal-catalyzed cross-coupling of sulfonamides with an appropriate aryl coupling partner have been developed to avoid potential genotoxic intermediates (Scheme 1. Eq 2)[4].

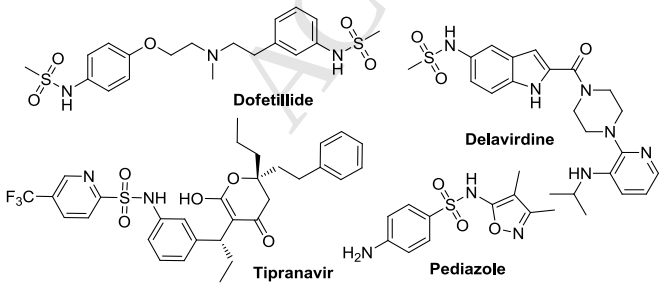
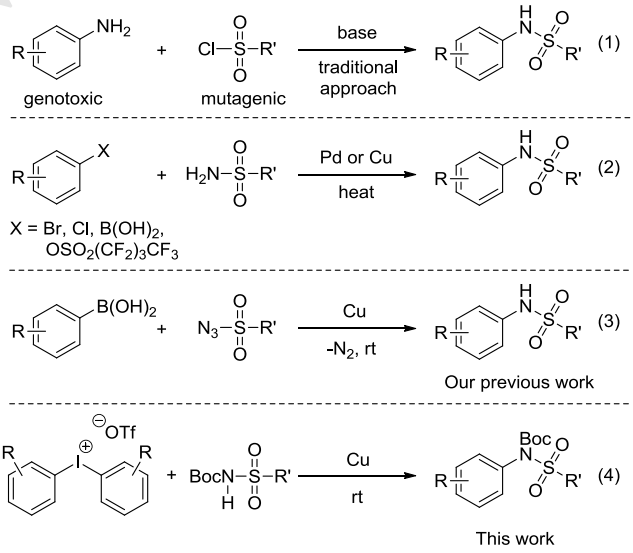


Figure 1. *N*-Arylsulfonamide-containing drugs



Scheme 1. Synthesis of *N*-Arylsulfonamides

In our efforts to improve upon the current methods for the preparation of *N*-arylsulfonamides, we investigated the use of sulfonyl azides in the Chan-Lam reaction (Scheme 1, eq 3)[5]. This cross-coupling reaction successfully proceeded to completion in 2 h at room temperature in an open flask without the use of any base or ligand due to the reactivity of the azide and resultant loss of N_2 driving the reaction. As an extension of this work, we continued our focus on non-mutagenic, stable and non-toxic

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reactants as coupling partners to prepare *N*-arylsulfonamides using mild conditions.

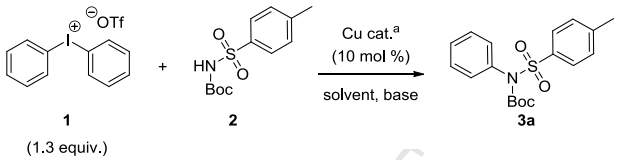
Easy to prepare, stable and non-toxic, diaryliodonium salts have been used successfully as arylating agents since their discovery.[6] In addition, the use of a Cu(I) catalyst can improve the electrophilicity of diaryliodonium salts via the generation of a reactive aryl-Cu(III) intermediate.[7] Recently, diaryliodonium salts were used to arylate various heteroatomic compounds.[8] Thus, we hypothesized that the use of diaryliodonium salts would allow for the facile production of *N*-arylsulfonamides at room temperature in the presence of Cu(I) catalyst even though sulfonamides are relatively unreactive as nucleophiles. Initial studies with catalytic Cu(I) generated a mixture of mono- and di-arylated sulfonamides without the reaction going to completion.[9] To prevent the unwanted diarylsulfonamides, we decided to use the Boc protecting group, which can be easily removed under either acidic or basic conditions after cross-coupling. While this work was underway, Wang *et al*. reported the use of diaryliodonium salts and 20 mol % of CuCl for the *N*-arylation of *N*-arylsulfonamides, which had been prepared from the corresponding arylamines and sulfonyl chlorides, to yield *N,N'*-diarylsulfonamides.[8a] While they demonstrated the possibility for *N*-arylation employing various diaryliodonium salts, this reaction provided *N,N'*-diarylsulfonamides. In comparison, our method generates singly *N*-arylated sulfonamides similar to those found in pharmaceutically active sulfonamide compounds[1] and allow for further modification of the sulfonamide moiety as needed. With these consideration in hand, we report herein a mild and efficient method for the generation of *N*-arylsulfonamides from diaryliodonium salts and *tert*-butyl *N*-sulfonylcarbamates at room temperature.

2. Results and discussion

Initial optimization was attempted using toluene at 50 °C with triethylamine base in the absence of a Cu(I) catalyst. After 24 h at these conditions, no cross-coupling was observed between diphenyliodonium triflate **1** and *tert*-butyl *N*-tosylcarbamate **2** (Table 1, entry 1). When the conditions were repeated in the presence of 10 mol % CuI, the reaction went to completion in 30 min yielding *N*-phenyl *tert*-butyl *N*-tosylcarbamate **3a** in 96% yield (Table 1, entry 2). Utilizing CuCl gave a slightly higher 97% yield (Table 1, entry 3). Both catalytic CuBr and CuBr-dimethyl sulfide complex gave slightly lower yields of the desired product **3a** (Table 1, entries 4-5). Using catalytic Cu(II) salts resulted in prolonged reaction times and lower yields (Table 1, entries 6-10). Decreasing the temperature from 50 °C to 25 °C and using CuI gave the desired product in 94% in only 1 h (Table 1, entry 11). We next explored the effects of various solvents. Utilizing CuCl in CH_2Cl_2, THF and toluene at room temperature provided the desired product in 1 h in 92%, 91% and 97% yield, respectively (Table 1, entries 12-14). Additionally, utilizing only 5 mol % of the copper catalyst at room temperature resulted in a prolonged reaction time with a significantly reduced yield (Table 1, entry 15). When an excess of *tert*-butyl *N*-tosylcarbamate **2** was utilized, the desired product **3a** was isolated in a slightly lower 92% yield in 2 h (Table 1, entry 16). Using DIPEA in toluene at 50 °C (Table 1, entry 17) matched the 97% yield that was obtained with Et_3N. However, when either pyridine or Cs_2CO_3 was used no product was observed even after 24 h (Table 1, entries 18-19). When solid bases like K_2CO_3 and K_3PO_4 were used, the product **3a** was obtained in a slightly lower yield in 18 h (entires 20-21). Thus, for the model reaction of diphenyliodonium triflate **1** with *tert*-butyl *N*-tosylcarbamate **2**, Et_3N in toluene with 10 mol % CuCl at room temperature was found to be the optimal conditions (Table 1, entry 14).

Table 1

Optimization of the *N*-Arylation of Diphenyliodonium Triflate **1** with *tert*-Butyl *N*-Tosylcarbamate **2**



1 (1.3 equiv.) **2** **3a**

entry	Cu cat.	base	solvent	temp. (°C)	time	yield (%)[b]
1	no catalyst	Et_3N	Toluene	50	24 h	0
2	CuI	Et_3N	Toluene	50	30 min	96
3	CuCl	Et_3N	Toluene	50	30 min	97
4	CuBr	Et_3N	Toluene	50	30 min	91
5	$CuBr{\cdot}SMe_2$	Et_3N	Toluene	50	30 min	94
6	$CuCl_2$	Et_3N	Toluene	50	24 h	82
7	$CuBr_2$	Et_3N	Toluene	50	6 h	87
8	CuO	Et_3N	Toluene	50	24 h	0
9	$CuSO_4$	Et_3N	Toluene	50	24 h	31
10	$Cu(OAc)_2$	Et_3N	Toluene	50	24 h	72
11	CuI	Et_3N	Toluene	25	1 h	94
12	CuCl	Et_3N	THF	25	1 h	91
13	CuCl	Et_3N	CH_2Cl_2	25	1 h	92
14	CuCl	Et_3N	Toluene	25	1 h	97
15[c]	CuCl	Et_3N	Toluene	25	24 h	53
16[d]	CuCl	Et_3N	Toluene	25	2 h	92
17	CuCl	DIPEA	Toluene	50	30 min	97
18	CuCl	Pyridine	Toluene	50	24 h	0
19	CuCl	Cs_2CO_3	Toluene	25	24 h	0
20	CuCl	K_2CO_3	Toluene	25	18 h	94
21	CuCl	K_3PO_4	Toluene	25	18 h	95

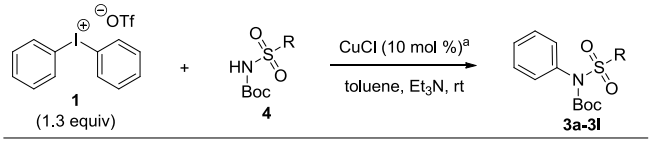
[a]Reaction conditions: 0.325 mmol of diphenyliodonium triflate, 0.25 mmol of *tert*-butyl *N*-tosylcarbamate, 0.50 mmol of base, solvent (0.12 M). [b]isolated yield. [c]5 mol % of CuCl. [d]0.325 mmol of *tert*-butyl *N*-tosylcarbamate, 0.25 mmol of diphenyliodonium triflate.

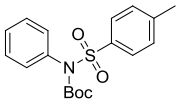
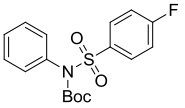
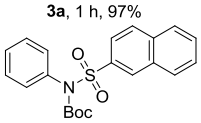
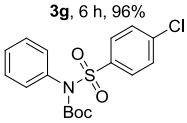
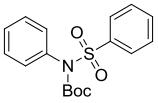
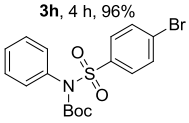
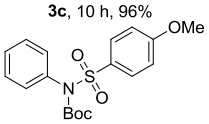
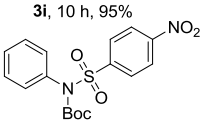
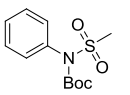
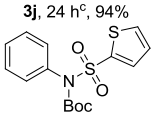
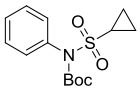
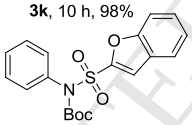
With the optimized conditions in hand, we set out to determine the scope of this cross-coupling reaction with respect to the *tert*-butyl *N*-sulfonylcarbamate coupling partner (Table 2). In general, *tert*-butyl *N*-arylsulfonylcarbamates furnished the desired *N*-arylsulfonamide in excellent yield in a moderate amount of time. *tert*-Butyl *N*-phenyl, *p*-methoxyphenyl, *p*-bromophenyl and *p*-nitrophenyl sulfonylcarbamates, however, required more than 10 h reaction time (Table 2, entries 3, 4, 9 and 10) and *tert*-butyl *N*-*p*-methoxyphenyl and *p*-nitrophenyl sulfonylcarbamate needed 1.5 equiv. of the diphenyliodonium triflate **1** in order to reach completion. When *tert*-butyl *N*-alkylsulfonylcarbamates were used, the coupled products **3e** and **3f** were obtained in excellent yield within 8-12 h (Table 2, entries 5-6). Furthermore, *tert*-butyl *N*-heteroarylsulfonylcarbamates

underwent *N*-arylation in excellent yield in 10-24 h (Table 2, entries 11-12).

Table 2.

Cu-Catalyzed *N*-Arylation of Diphenyliodonium Triflate **1** with Various *tert*-Butyl *N*-Sulfonylcarbamates **4**



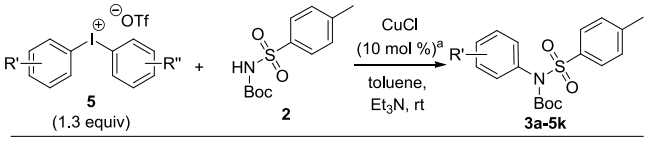
entry	product time, yield[b]	entry	product time, yield[b]
1	 **3a**, 1 h, 97%	7	 **3g**, 6 h, 96%
2	 **3b**, 4 h, 96%	8	 **3h**, 4 h, 96%
3	 **3c**, 10 h, 96%	9	 **3i**, 10 h, 95%
4	 **3d**, 10 h[c], 97%	10	 **3j**, 24 h[c], 94%
5	 **3e**, 8 h, 91%	11	 **3k**, 10 h, 98%
6	 **3f**, 12 h, 96%	12	 **3l**, 24 h[d], 91%

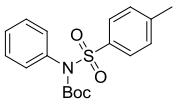
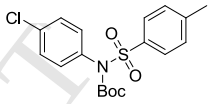
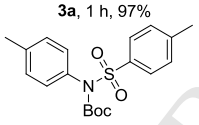
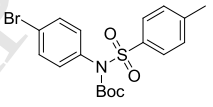
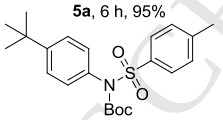
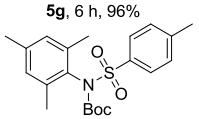
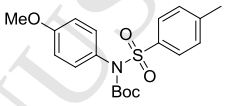
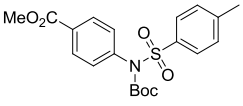
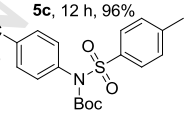
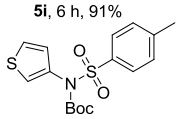
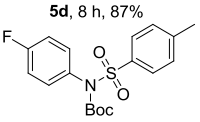
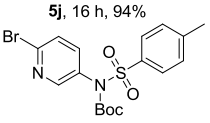
[a]Reaction conditions: 0.325 mmol of diphenyliodonium triflate, 0.25 mmol of *tert*-butyl *N*-sulfonylcarbamate, 10 mol % CuCl, 0.50 mmol of Et_3N, toluene (0.12 M). [b]isolated yield. [c]1.5 equiv. of diphenyliodonium triflate. [d]2.5 equiv. of diphenyliodonium triflate.

Next, we investigated the use of various diaryliodonium triflates. Symmetric diaryliodonium triflates gave rise to their coupled products in yields ranging from 87-97% (Table 3, entries 1-9). The bulky mesityl or 2,4,6-triethylphenyl groups, used to selectively transfer the other aryl group in unsymmetric iodonium salts, were also utilized.[10] The symmetrical mesityl iodonium triflate was too bulky to undergo aryl transfer resulting in no product observed after 24 h (Table 3, entry 9). Unsymmetric mesityl or 2,4,6-triethylphenyl iodonium triflates were used to successfully transfer aryl and heteroaryl groups to *tert*-butyl *N*-tosylcarbamate **2** (Table 3, entries 10-12). Particularly, the 3-thienyl and 2-bromopyridine moieties were coupled with *tert*-butyl-*N*-tosylcarbamate **2** at room temperature in 94% and 61% yield and in 16 and 12 h, respectively (Table 3, entries 11-12). To further demonstrate the flexibility of copper catalyzed *N*-arylation, the cross-coupling reactions were then conducted on a large scale (5.0 mmol), furnishing the desired products in excellent yields (Scheme 2).

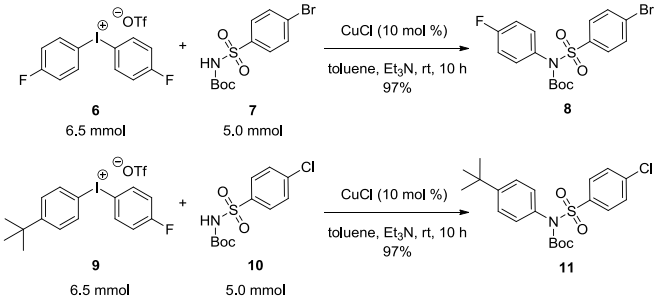
Table 3.

Cu-Catalyzed *N*-Arylation of *tert*-Butyl *N*-Tosylcarbamate **2** with Various Diaryliodonium Triflates **5**



entry	product time, yield[b]	entry	product time, yield[b]
1	 **3a**, 1 h, 97%	7	 **5f**, 10 h, 92%
2	 **5a**, 6 h, 95%	8	 **5g**, 6 h, 96%
3	 **5b**, 4 h, 88%	9	 **5h**, 24 h, 0%
4	 **5c**, 12 h, 96%	10	 **5i**, 6 h, 91%
5	 **5d**, 8 h, 87%	11	 **5j**, 16 h, 94%
6	 **5e**, 6 h, 96%	12	 **5k**,12 h, 61%

[a]Reaction conditions: 0.325 mmol of diphenyliodonium triflate, 0.25 mmol of *tert*-butyl *N*-tosylcarbamate, 10 mol % CuCl, 0.50 mmol of Et_3N, toluene (0.12 M). [b]isolated yield.



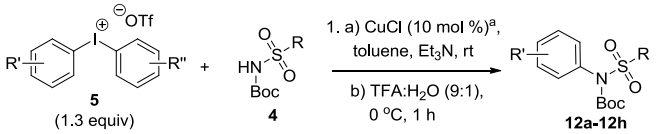
Scheme 2. Large Scale Synthesis

Encouraged by these cross-coupling results, our attention finally turned to a one-pot coupling/Boc-deprotection sequence utilizing aqueous trifluoroacetic acid. As described in Table 4, diaryliodonium triflates were reacted with *tert*-butyl *N*-sulfonylcarbamates, and, upon completion of the cross-coupling reaction, water and TFA were added at 0 °C to synthesize mono *N*-arylsulfonamides. After 1 h under the deprotection conditions, the singly arylated *N*-phenyl tosylsulfonamide **12a** was isolated in 85% yield (Table 4, entry 1). In a similar manner, *tert*-butyl *N*-sulfonylcarbamates containing an aryl, alkyl or a heteroaryl group were reacted with diaryliodonium triflates giving rise to mono *N*-arylsulfonamides in yields ranging from 83-94% (Table

4, entries 2-8).

Table 4.

One-Pot *N*-Arylation and Boc Deprotection of Various *tert*-Butyl *N*-Sulfonylcarbamates **4** with Various Diaryliodonium Triflates **5**



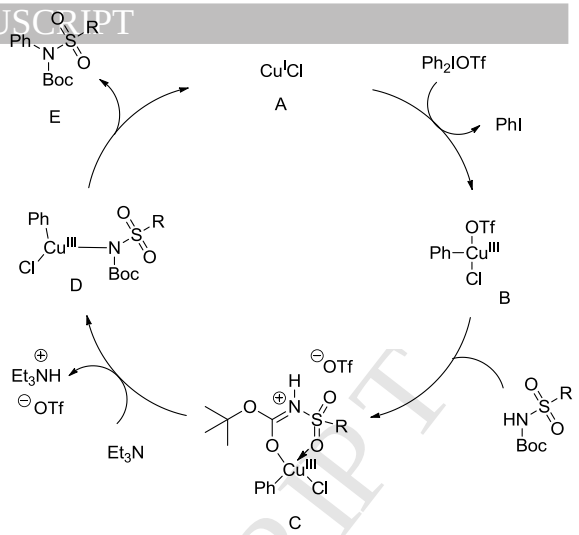
entry	product yield[b]	entry	product yield[b]
1	**12a**, 85%	5	**12e**, 94%
2	**12b**, 93%	6	**12f**, 91%
3	**12c**, 89%	7	**12g**, 94%
4	**12d**, 83%	8	**12h**, 90%

[a]Reaction conditions: 1.30 mmol of diaryliodonium triflate, 1.00 mmol of *tert*-butyl *N*-sulfonylcarbamate, 10 mol % CuCl, 0.50 mmol of Et_3N, toluene (0.12 M). [b]isolated yield.

We propose the following mechanism, similar to other reported copper catalyzed arylations with diaryliodonium salts, for this cross-coupling procedure (Scheme 3).[7b, 8b] Oxidation of Cu(I) salt A with iodonium triflate would give rise to the aryl-Cu(III) species B. Next, coordination of the oxygen lone pairs of *tert*-butyl-*N*-sulfonylcarbamate with this active copper species would form a tetracoordinate Cu(III)-complex C. After the extraction of a proton from C by the base utilized (*cf.* Et_3N, DIPEA), rearrangement would afford Cu(III)-complex D. Finally, reductive elimination would yield the coupled product E and regenerate the Cu(I) catalyst allowing the catalytic cycle to continue.

3. Conclusions

In conclusion, we have described a new synthetic method for the preparation of *N*-arylsulfonamides employing *tert*-butyl *N*-sulfonylcarbamates and diaryliodonium salts. The reaction requires 10 mol % of CuCl catalyst and Et_3N as a base at room temperature. The scope of this method has been explored employing different *tert*-butyl *N*-sulfonylcarbamates and diaryliodonium salts. Furthermore, a one-pot coupling/Boc-deprotection sequence utilizing aqueous trifluoroacetic acid was investigated to successfully furnish mono *N*-arylsulfonamides in good to excellent yields.



Scheme 3. Plausible Mechanism

4. Experimental

4.1. General information

Unless otherwise indicated, all chemical reagents were purchased from Sigma-Aldrich, Alfa Aesar, and TCI and were used without further purification. All diarylisodonium salts[11] and *tert*-butyl *N*-sulfonylcarbamates[12] were prepared by the reported procedures. All reactions were carried out in oven-dried glassware equipped with a magnetic stir bar. Reactions were monitored by thin layer chromatography (TLC) with 0.25-mm E. Merck pre-coated silica gel plates (Kieselgel 60F_{254}, Merck). Products were detected by viewing under a UV light, by staining with an anisaldehyde solution composed of acetic acid, sulfuric acid, and MeOH, or by staining with a $KMnO_4$ solution composed of potassium carbonate, sodium hydroxide, and water. Flash column chromatography was performed on Merck 60 silica gel (70-230 mesh). Yields refer to chromatographically and spectroscopically pure compounds unless otherwise noted. ^1H and ^{13}C spectra were recorded on a Bruker AM-300 or Varian Unity-Inova 500 MHz spectrometer. Chemical shifts are reported as δ values relative to internal $SiMe_4$ or chloroform (δ 0.00 for ^1H and δ 77.0 for ^{13}C) or benzene (δ 128.0 for ^{13}C). IR spectra were measured as neat oils on a Varian Scimitar 800 FT-IR spectrometer. High resolution spectra were obtained at Sogang University's Organic Chemistry Research Center or the Korea Basic Science Institute Mass Spectrometry Service Center.

4.2. General procedure I for copper-catalyzed *N*-arylation (Tables 1-3, Scheme 2)

A 10 mL round bottom flask was charged with a diaryliodonium salt (0.325 mmol), CuCl (10 mol %), triethylamine (0.50 mmol) and the corresponding *tert*-butyl *N*-sulfonylcarbamate (0.25 mmol). Toluene (2 mL) was then added to the flask. The reaction mixture was stirred at room temperature. After completion of the reaction, as monitored by TLC analysis, the solvent was removed *in vacuo* and the residue was purified by flash column chromatography on silica gel to obtain the desired product.

4.2.1. *tert-butyl phenyl(tosyl)carbamate (**3a**).*

General procedure I was used employing *tert*-butyl tosylcarbamate (0.068 g, 0.25 mmol) and diphenyliodonium triflate (0.140 g, 0.325 mmol), and the reaction was complete in 1 h. Flash chromatography on silica gel using hexane/ethyl acetate (7:1) provided pure **3a** (0.084 g, 0.24 mmol, 97%) as a white solid. R_f 0.47 (hexane/ethyl acetate = 3:1); ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.87 (d, *J* = 8.1 Hz, 2H), 7.48-7.37 (m, 3H), 7.34 (d, *J* = 8.1 Hz, 2H), 7.29-7.20 (m, 2H), 2.46 (s, 3H), 1.34 (s, 9H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 150.9, 144.4, 136.7, 136.4, 129.6, 129.3, 129.0, 128.9, 128.6, 84.3, 27.8, 21.6; Data is consistent with that reported in the literature.[13]

4.2.2 *tert-butyl naphthalene-2-ylsulfonyl(phenyl)carbamate (**3b**).*

General procedure I was used employing *tert*-butyl naphthalen-2-ylsulfonylcarbamate (0.077 g, 0.25 mmol) and diphenyliodonium triflate (0.140 g, 0.325 mmol), and the reaction was complete in 4 h. Flash chromatography on silica gel using hexane/ethyl acetate (7:1) provided pure **3b** (0.092 g, 0.24 mmol, 96%) as a white solid. mp: 126-128 ^{o}C; R_f 0.44 (hexane/ethyl acetate = 3:1); IR (neat) 3059, 2977, 1734, 1352, 1292, 1150, 1073, 814, 695 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.59 (s, 1H), 8.02-7.88 (m, 4H), 7.72-7.57 (m, 2H), 7.45-7.38 (m, 3H), 7.36-7.26 (m, 2H), 1.31 (s, 9H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 150.8, 136.4, 136.3, 135.1, 131.7, 130.5, 129.7, 129.3, 129.2, 129.1, 129.0, 128.9, 127.9, 127.6, 123.1, 84.5, 27.7; HRMS-FAB: *m/z* 406.1084 [(M+Na)$^+$; calcd for $C_{21}H_{21}NNaO_4S^+$: 406.1083].

4.2.3. *tert-butyl phenyl(phenylsulfonyl)carbamate (**3c**).*

General procedure I was used employing *tert*-butyl phenylsulfonylcarbamate (0.064 g, 0.25 mmol) and diphenyliodonium triflate (0.140 g, 0.325 mmol), and the reaction was complete in 10 h. Flash chromatography on silica gel using hexane/ethyl acetate (7:1) provided pure **3c** (0.080 g, 0.24 mmol, 96%) as a white solid. mp: 67-68 ^{o}C; R_f 0.43 (hexane/ethyl acetate = 3:1); IR (neat) 3065, 2981, 1734, 1598, 1790, 1368, 1151, 1091, 970, 812, 696, 580 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.00 (d, *J* = 7.5 Hz, 2H), 7.68-7.60 (m, 1H), 7.58-7.50 (m, 2H), 7.46-7.38 (m, 3H), 7.29-7.25 (m, 2H), 1.32 (s, 9H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 150.7, 139.6, 136.2, 133.4, 129.6, 129.0, 128.97, 128.6, 128.4, 84.5, 27.7; HRMS-FAB: *m/z* 356.0927 [(M+Na)$^+$; calcd for $C_{17}H_{19}NNaO_4S^+$: 356.0927].

4.2.4. *tert-butyl (4-methoxyphenyl)sulfonyl(phenyl)carbamate (**3d**).*

General procedure I was used employing *tert*-butyl (4-methoxyphenyl)sulfonylcarbamate (0.072 g, 0.25 mmol) and diphenyliodonium triflate (0.161 g, 0.375 mmol), and the reaction was complete in 10 h. Flash chromatography on silica gel using hexane/ethyl acetate (7:1) provided pure **3d** (0.088 g, 0.24 mmol, 97%) as a colorless oil.; R_f 0.30 (hexane/ethyl acetate = 3:1); IR (neat) 2981, 1733, 1596, 1498, 1368, 1263, 1150, 1090, 839, 677, 581 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.92 (d, *J* = 8.7 Hz, 2H), 7.46-7.38 (m, 3H), 7.27-7.21 (m, 2H), 7.00 (d, *J* = 9.0 Hz, 2H), 3.89 (s, 3H), 1.35 (s, 9H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 163.5, 151.0, 136.6, 131.1, 130.9, 129.6, 129.0, 128.9, 113.8, 84.2, 55.6, 27.8; HRMS-FAB: *m/z 364.1213* [(M+Na)$^+$; calcd for $C_{18}H_{22}NO_5S^+$: 364.1213].

*4.2.5. tert-butyl methylsulfonyl(phenyl)carbamate (**3e**).*

General procedure I was used employing *tert*-butyl methylsulfonylcarbamate (0.049 g, 0.25 mmol) and diphenyliodonium triflate (0.140 g, 0.325 mmol), and the reaction was complete in 8 h. Flash chromatography on silica gel using hexane/ethyl acetate (7:1) provided pure **3e** (0.062 g, 0.23 mmol, 91%) as a white solid. mp: 83-85 ^{o}C; R_f 0.27 (hexane/ethyl acetate = 3:1); IR (neat) 3015, 2982, 2939, 1732, 1596, 1492, 1356, 1148, 967, 838, 694, 547 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.45-7.37 (m, 3H), 7.27-7.21 (m, 2H), 3.42 (s, 3H), 1.46 (s, 9H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 151.5, 135.6, 129.3, 129.1, 129.05, 84.7, 41.6, 27.8; HRMS-FAB: *m/z* 294.0772 [(M+Na)$^+$; calcd for $C_{12}H_{17}NNaO_4S^+$: 294.0770].

4.2.6. *tert-butyl cyclopropylsulfonyl(phenyl)carbamate (**3f**).*

General procedure I was used employing *tert*-butyl cyclopropylsulfonylcarbamate (0.055 g, 0.25 mmol) and diphenyliodonium triflate (0.140 g, 0.325 mmol), and the reaction was complete in 12 h. Flash chromatography on silica gel using hexane/ethyl acetate (7:1) provided pure **3f** (0.071 g, 0.24 mmol, 96%) as a white solid. mp: 75-76 ^{o}C; R_f 0.34 (hexane/ethyl acetate = 3:1); IR (neat) 2982, 1733, 1490, 1361, 1293, 1145, 1074, 970, 885, 711, 586 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.46-7.33 (m, 3H), 7.28-7.22 (m, 2H), 3.36-3.25 (m, 1H), 1.47 (s, 9H), 1.36-1.29 (m, 2H), 1.18-1.09 (m, 2H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 151.6, 136.2, 129.4, 129.0, 128.8, 84.3, 31.7, 27.8, 6.0; HRMS-FAB: *m/z* 298.1107 [(M+Na)$^+$; calcd for $C_{14}H_{20}NO_4S^+$: 298.1108].

4.2.7. *tert-butyl (4-fluorophenyl)sulfonyl(phenyl)carbamate (**3g**).*

General procedure I was used employing *tert*-butyl (4-fluorophenyl)sulfonylcarbamate (0.069 g, 0.25 mmol) and diphenyliodonium triflate (0.140 g, 0.325 mmol), and the reaction was complete in 6 h. Flash chromatography on silica gel using hexane/ethyl acetate (7:1) provided pure **3g** (0.084 g, 0.24 mmol, 96%) as a white solid. mp: 80-82 ^{o}C; R_f 0.55 (hexane/ethyl acetate = 3:1); IR (neat) 2982, 1734, 1591, 1494, 1370, 1151, 968, 841, 695, 579 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.09-7.96 (m, 2H), 7.47-7.37 (m, 3H), 7.29-7.20 (m, 4H), 1.34 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 165.5 (d, *J* = 104 Hz), 150.7, 136.2, 135.5, 131.4 (d, *J* = 7.6 Hz), 129.5, 129.1, 129.06, 115.9 (d, *J* = 17.8 Hz), 84.6, 27.7; HRMS-FAB: *m/z* 374.0833 [(M+Na)$^+$; calcd for $C_{17}H_{18}FNNaO_4S^+$: 374.0833].

4.2.8. *tert-butyl (4-chlorophenyl)sulfonyl(phenyl)carbamate (**3h**).*

General procedure I was used employing *tert*-butyl (4-chlorophenyl)sulfonylcarbamate (0.073 g, 0.25 mmol) and diphenyliodonium triflate (0.140 g, 0.325 mmol), and the reaction was complete in 4 h. Flash chromatography on silica gel using hexane/ethyl acetate (7:1) provided pure **3h** (0.088 g, 0.24 mmol, 96%) as a white solid. mp: 102-103 ^{o}C; R_f 0.61 (hexane/ethyl acetate = 3:1); IR (neat) 2982, 1733, 1584, 1491, 1371, 1150, 1092, 839, 759, 694, 576 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.93 (d, *J* = 8.8 Hz, 2H), 7.52 (d, *J* = 8.7 Hz, 2H), 7.46-7.38 (m, 3H), 7.26-7.18 (m, 2H), 1.34 (s, 9H); ^{13}C NMR (75 MHz, C_6D_6) δ 151.1, 139.8, 138.9, 137.1, 130.7, 130.1, 129.3, 129.1, 129.0, 84.0, 27.7; HRMS-FAB: *m/z* 390.0536 [(M+Na)$^+$; calcd for $C_{17}H_{18}ClNNaO_4S^+$: 390.0537].

4.2.9. *tert-butyl (4-bromophenyl)sulfonyl(phenyl)carbamate (**3i**).*

General procedure I was used employing *tert*-butyl (4-bromophenyl)sulfonylcarbamate (0.084 g, 0.25 mmol) and diphenyliodonium triflate (0.140 g, 0.325 mmol), and the reaction was complete in 10 h. Flash chromatography on silica gel using hexane/ethyl acetate (7:1) provided pure **3i** (0.098 g, 0.24 mmol, 95%) as a white solid. mp: 108-110 ^{o}C; R_f 0.61 (hexane/ethyl acetate = 3:1); IR (neat) 2981, 1736, 1574, 1491, 1391, 1370, 1290, 1148, 745, 602 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.86 (d, *J* = 8.4 Hz, 2H), 7.69 (d, *J* = 8.4 Hz, 2H), 7.47-7.38 (m, 3H), 7.25-7.18 (m, 2H), 1.34 (s, 9H); ^{13}C NMR (75 MHz, C_6D_6) δ 151.1, 139.4, 137.2, 132.0, 130.7, 130.1, 129.2, 129.0, 128.5, 84.0, 27.7; HRMS-FAB: *m/z* 412.0212 [(M+Na)$^+$; calcd for $C_{17}H_{19}BrNO_4S^+$: 412.0213].

4.2.10. *tert-butyl (4-nitrophenyl)sulfonyl(phenyl)carbamate (**3j**).*

General procedure I was used employing *tert*-butyl (4-nitrophenyl)sulfonylcarbamate (0.076 g, 0.25 mmol) and diphenyliodonium triflate (0.161 g, 0.375 mmol), and the reaction was complete in 24 h. Flash chromatography on silica gel using hexane/ethyl acetate (7:1) provided pure **3j** (0.089 g, 0.235 mmol, 94%) as a white solid. mp: 146-148 ^{o}C; R_f 0.46 (hexane/ethyl acetate = 3:1); IR (neat) 3117, 2975, 1734, 1611, 1491, 1361, 1286, 1151, 1090, 747, 695, 568 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.40 (d, *J* = 9.3 Hz, 2H), 8.20 (d, *J* = 8.7 Hz, 2H), 7.50-7.41 (m, 3H), 7.25-7.17 (m, 2H), 1.34 (s, 9H); ^{13}C NMR (75 MHz, C_6D_6) δ 151.0, 150.4, 145.1, 136.7, 130.1, 130.0, 129.4, 129.3, 123.8, 84.5, 27.6; HRMS-FAB: *m/z* 401.0778 [(M+Na)$^+$; calcd for $C_{17}H_{18}N_2NaO_6S^+$: 401.0778].

4.2.11. *tert-butyl phenyl(thiophen-2-ylsulfonyl)carbamate (**3k**).*

General procedure I was used employing *tert*-butyl thiophen-2-ylsulfonylcarbamate (0.066 g, 0.25 mmol) and diphenyliodonium triflate (0.140 g, 0.325 mmol), and the reaction was complete in 10 h. Flash chromatography on silica gel using hexane/ethyl acetate (7:1) provided pure **3k** (0.083 g, 0.245 mmol, 98%) as a white oil.; R_f 0.50 (hexane/ethyl acetate = 3:1); IR (neat) 3105, 2924, 2854, 1733, 1596, 1372, 1290, 1148, 914, 742, 593 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.80 (dd, *J* = 3.8 and 1.3 Hz, 1H), 7.71 (dd, *J* = 5.0 and 1.3 Hz, 1H), 7.46-7.38 (m, 3H), 7.27-7.20 (m, 2H), 7.14 (dd, *J* = 6.3 and 4.9 Hz, 1H), 1.40 (s, 9H); ^{13}C NMR (75 MHz, C_6D_6) δ 150.9, 140.2, 137.0, 134.9, 132.8, 129.8, 128.8, 128.6, 126.4, 83.6, 27.4; HRMS-FAB: *m/z* 340.0672 [(M+Na)$^+$; calcd for $C_{15}H_{18}NO_4S_2^+$: 340.0672].

4.2.12. *tert-butyl benzofuran-2-ylsulfonyl(phenyl)carbamate (**3l**).*

General procedure I was used employing *tert*-butyl benzofuran-2-ylsulfonylcarbamate (0.074 g, 0.25 mmol) and diphenyliodonium triflate (0.269 g, 0.625 mmol), and the reaction was complete in 24 h. Flash chromatography on silica gel using hexane/dichloromathane (1:1) provided pure **3l** (0.085 g, 0.23 mmol, 91%) as a white solid. mp: 116-118 ^{o}C; R_f 0.26 (hexane/ethyl acetate = 3:1); IR (neat) 3120, 2984, 2931, 1738, 1548, 1490, 1374, 1291, 1144, 974, 838, 756, 565 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.74 (d, *J* = 7.8 Hz, 1H), 7.66-7.36 (m, 9H), 1.35 (s, 9H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 155.6, 150.3, 149.1, 135.6, 129.9, 129.3, 129.2, 128.2, 125.7, 124.4, 123.1, 114.8, 112.3, 85.2, 27.7; HRMS-FAB: *m/z* 396.0877 [(M+Na)$^+$; calcd for $C_{19}H_{19}NNaO_5S^+$: 396.0876].

4.2.13. *tert-butyl p-tolyl(tosyl)carbamate (**5a**).*

General procedure I was used employing *tert*-butyl tosylcarbamate (0.068 g, 0.25 mmol) and di-*p*-tolyliodonium triflate (0.149 g, 0.325 mmol), and the reaction was complete in 6 h. Flash chromatography on silica gel using hexane/ethyl acetate (7:1) provided pure **5a** (0.086 g, 0.24 mmol, 95%) as a white solid. mp: 132-134 ^{o}C; R_f 0.56 (hexane/ethyl acetate = 3:1); IR (neat) 2982, 2929, 1733, 1511, 1368, 1290, 1151, 971, 841, 669 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.86 (d, *J* = 8.3 Hz, 2H), 7.33 (d, *J* = 7.7 Hz, 2H), 7.22 (d, *J* = 7.7 Hz, 2H), 7.13 (d, *J* = 8.3 Hz, 2H), 2.46 (s, 3H), 2.38 (s, 3H), 1.34 (s, 9H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 151.0, 144.3, 139.0, 136.8, 133.8, 129.8, 129.3, 129.25, 128.5, 84.3, 27.8, 21.6, 21.2; HRMS-FAB: *m/z* 384.1241 [(M+Na)$^+$; calcd for $C_{19}H_{23}NNaO_4S^+$: 384.1240].

4.2.14. *tert-butyl (4-(tert-butyl)phenyl)(tosyl)carbamate (**5b**).*

General procedure I was used employing *tert*-butyl tosylcarbamate (0.068 g, 0.25 mmol) and bis(4-(*tert*-butyl)phenyl)iodonium triflate (0.176 g, 0.325 mmol), and the reaction was complete in 4 h. Flash chromatography on silica gel using hexane/ethyl acetate (7:1) provided pure **5b** (0.089 g, 0.22 mmol, 88%) as a white solid. mp: 156-158 ^{o}C; R_f 0.63 (hexane/ethyl acetate = 3:1); IR (neat) 2966, 1734, 1368, 1151, 1091, 665, 591 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.87 (d, *J* = 8.1 Hz, 2H), 7.43 (d, *J* = 8.4 Hz, 2H), 7.31 (d, *J* = 8.1 Hz, 2H), 7.18 (d, *J* = 8.4 Hz, 2H), 2.47 (s, 3H), 1.35 (s, 9H), 1.34 (s, 9H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 151.9, 151.1, 144.3, 137.0, 133.6, 129.3. 129.0, 128.5, 126.1, 84.4, 34.7, 31.3, 27.8, 21.6; HRMS-FAB: *m/z* 426.1709 [(M+Na)$^+$; calcd for $C_{22}H_{29}NNaO_4S^+$: 426.1710].

4.2.15. *tert-butyl (4-methoxyphenyl)(tosyl)carbamate (**5c**).*

General procedure I was used employing *tert*-butyl tosylcarbamate (0.068 g, 0.25 mmol) and bis(4-methoxyphenyl)iodonium triflate (0.159 g, 0.325 mmol), and the reaction was complete in 12 h. Flash chromatography on silica gel using hexane/ethyl acetate (7:1) provided pure **5c** (0.091 g, 0.24 mmol, 96%) as a white solid. R_f 0.43 (hexane/ethyl acetate = 3:1); ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.86 (d, *J* = 8.4 Hz, 2H), 7.33 (d, *J* = 8.1 Hz, 2H), 7.16 (dd, *J* = 6.9 and 2.1 Hz, 2H), 6.92 (d, *J* = 8.7 Hz, 2H), 3.81 (s, 3H), 2.45 (s, 3H), 1.34 (s, 9H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 159.7, 151.0, 144.3, 136.7, 130.6, 129.2, 128.9, 128.4, 114.2, 84.2, 55.3, 27.7, 21.5; Data is consistent with that reported in the literature.[14]

4.2.16. *tert-butyl tosyl(4-trifluoromethyl)phenyl)carbamate (**5d**).*

General procedure I was used employing *tert*-butyl tosylcarbamate (0.068 g, 0.25 mmol) and bis(4-(trifluoromethyl)phenyl)iodonium triflate (0.184 g, 0.325 mmol), and the reaction was complete in 8 h. Flash chromatography on silica gel using hexane/ethyl acetate (7:1) provided pure **5d** (0.090 g, 0.22 mmol, 87%) as a white solid. mp: 136-138 ^{o}C; R_f 0.59 (hexane/ethyl acetate = 3:1); IR (neat) 2984, 2931, 1738, 1612, 1326, 1169, 1020, 812, 766, 579 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.86 (d, *J* = 8.3 Hz, 2H), 7.69 (d, *J* = 8.3 Hz, 2H), 7.37 (t, *J* = 8.3 Hz, 4H), 2.47 (s, 3H), 1.35 (s, 9H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 150.4, 144.9, 139.6, 139.59, 136.3, 130.9 (q, *J* = 33 Hz), 130.3, 129.5, 128.5, 126.2 (q, *J* = 3.8 Hz), 85.1, 27.7, 21.6; HRMS-FAB: *m/z* 438.0957 [(M+Na)$^+$; calcd for $C_{19}H_{20}F_3NNaO_4^+$: 438.0957].

4.2.17. *tert-butyl (4-fluorophenyl)(tosyl)carbamate (**5e**).*

General procedure I was used employing *tert*-butyl tosylcarbamate (0.068 g, 0.25 mmol) and bis(4-fluorophenyl)iodonium triflate (0.152 g, 0.325 mmol), and the reaction was complete in 6 h. Flash chromatography on silica gel using hexane/ethyl acetate (7:1) provided pure **5e** (0.088 g, 0.24 mmol, 96%) as a white solid. mp: 104-106 ^{o}C; R_f 0.56 (hexane/ethyl acetate = 3:1); IR (neat) 3449, 2986, 1733, 1598, 1505, 1373, 1093, 975, 842, 671 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.85 (d, *J* = 8.4 Hz, 2H), 7.35 (d, *J* = 8.0 Hz, 2H), 7.27-7.19 (m, 2H), 7.15-7.06 (s, 2H), 2.46 (s, 3H), 1.35 (s, 9H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 162.6 (d, *J* = 248 Hz), 150.8, 144.6, 136.5, 132.4 (d, *J* = 3.6 Hz), 131.4 (d, *J* = 8.8 Hz), 129.4, 128.5, 116.0 (d, *J* = 23 Hz), 84.6, 27.8, 21.6; HRMS-FAB: *m/z* 388.0989 [(M+Na)$^+$; calcd for $C_{18}H_{20}FNNaO_4S^+$: 388.0989].

4.2.18. *tert-butyl (4-chlorophenyl)(tosyl)carbamate (**5f**).*

General procedure I was used employing *tert*-butyl tosylcarbamate (0.068 g, 0.25 mmol) and bis(4-chlorophenyl)iodonium triflate (0.162 g, 0.325 mmol), and the reaction was complete in 10 h. Flash chromatography on silica gel using hexane/ethyl acetate (7:1) provided pure **5f** (0.088 g, 0.23 mmol, 92%) as a white solid. mp: 132-133 ^{o}C; R_f 0.59 (hexane/ethyl acetate = 3:1); IR (neat) 3455, 2980, 1733, 1597, 1489, 1373, 1250, 1091, 973, 813, 665 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.85 (d, *J* = 8.0 Hz, 2H), 7.43-7.30 (m, 4H), 7.18 (d, *J* = 8.4 Hz, 2H), 2.46 (s, 3H), 1.35 (s, 9H); ^{13}C NMR (75 MHz, C_6D_6) δ 151.0, 144.3, 137.6, 136.0, 135.0, 131.6, 129.4 (2C), 129.2, 83.9, 27.7, 21.2; HRMS-FAB: *m/z* 404.0694 [(M+Na)$^+$; calcd for $C_{18}H_{20}ClNNaO_4S^+$: 404.0694].

4.2.19. *tert-butyl (4-bromophenyl)(tosyl)carbamate (**5g**).*

General procedure I was used employing *tert*-butyl tosylcarbamate (0.068 g, 0.25 mmol) and bis(4-bromophenyl)iodonium triflate (0.191 g, 0.325 mmol), and the reaction was complete in 6 h. Flash chromatography on silica gel using hexane/ethyl acetate (7:1) provided pure **5g** (0.102 g, 0.24 mmol, 96%) as a white solid. mp: 148-150 ^{o}C; R_f 0.59 (hexane/ethyl acetate = 3:1); IR (neat) 2987, 2931, 1739, 1595, 1487, 1284, 1173, 1071, 838, 730, 664, 580 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.84 (d, *J* = 8.0 Hz, 2H), 7.55 (d, *J* = 8.9 Hz, 2H), 7.34 (d, *J* = 8.5 Hz, 2H), 7.12 (d, *J* = 8.5 Hz, 2H), 2.47 (s, 3H), 1.35 (s, 9H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 150.5, 144.7, 136.4, 135.5, 132.3, 131.3, 129.4, 128.6, 123.1, 84.8, 27.8, 21.7; HRMS-FAB: *m/z* 448.0189 [(M+Na)$^+$; calcd for $C_{18}H_{20}BrNNaO_4S^+$: 448.0189].

4.2.20. *methyl 4-(N-tert-butoxycarbonyl)-4-methylphenylsulfonamido)benzoate (**5i**).*

General procedure I was used employing *tert*-butyl tosylcarbamate (0.068 g, 0.25 mmol) and mesityl(4-(methoxycarbonyl)phenyl)iodonium triflate (0.172 g, 0.325 mmol), and the reaction was complete in 6 h. Flash chromatography on silica gel using hexane/ethyl acetate (7:1) provided pure **5i** (0.092 g, 0.23 mmol, 91%) as a white solid. mp: 140-142 ^{o}C; R_f 0.41 (hexane/ethyl acetate = 3:1); IR (neat) 2983, 1729, 1605, 1370, 1281, 1150, 702, 661, 548 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.10 (d, *J* = 8.4 Hz, 2H), 7.86 (d, *J* = 8.4 Hz, 2H), 7.39-7.26 (m, 4H), 3.94 (s, 3H), 2.47 (s, 3H), 1.34 (s, 9H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 166.2, 150.4, 144.8, 140.5, 136.4, 130.5, 135.4, 129.7, 129.4, 128.6, 84.8, 52.3, 27.8, 21.7; HRMS-FAB: *m/z* 428.1138 [(M+Na)$^+$; calcd for $C_{20}H_{23}NNaO_6S^+$: 428.1138].

4.2.21. *tert-butyl thiophen-3-yl(tosyl)carbamate (**5j**).*

General procedure I was used employing *tert*-butyl tosylcarbamate (0.068 g, 0.25 mmol) and mesityl(thiophen-3-yl)iodonium triflate (0.155 g, 0.325 mmol), and the reaction was complete in 16 h. Flash chromatography on silica gel using hexane/ethyl acetate (7:1) provided pure **5j** (0.083 g, 0.235 mmol, 94%) as a white solid. mp: 115-117 ^{o}C; R_f 0.52 (hexane/ethyl acetate = 3:1); IR (neat) 3108, 2976, 2360, 1734, 1527, 1369, 1148, 812, 636 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.85 (d, *J* = 8.0 Hz, 2H), 7.36-7.24 (m, 4H), 6.96 (dd, *J* = 5.0 and 1.1 Hz, 1H), 2.45 (s, 3H), 1.35 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 150.6, 144.5, 136.5, 133.3, 129.3, 128.4, 127.2, 124.8, 124.3, 84.5, 27.8, 21.6; HRMS-FAB: *m/z* 376.0647 [(M+Na)$^+$; calcd for $C_{16}H_{19}NNaO_4S_2^+$: 376.0648].

4.2.22. *tert-butyl (6-bromopyridin-3-yl)(tosyl)carbamate (**5k**).*

General procedure I was used employing *tert*-butyl tosylcarbamate (0.068 g, 0.25 mmol) and (6-bromopyridin-3-yl)(2,4,6-triethylphenyl)iodonium triflate (0.193 g, 0.325 mmol), and the reaction was complete in 12 h. Flash chromatography on silica gel using hexane/ethyl acetate (7:1) provided pure **5k** (0.065 g, 0.15 mmol, 61%) as a white solid. mp: 166-168 ^{o}C; R_f 0.48 (hexane/ethyl acetate = 3:1); IR (neat) 2976, 2360, 1735, 1370, 1285, 1149, 1090, 664, 582 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.16 (d, *J* = 2.2 Hz, 1H), 7.83 (d, *J* = 8.1 Hz, 2H), 7.58 (d, *J* = 8.7 Hz, 1H), 7.50 (dd, *J* = 8.7 and 2.7 Hz, 1H), 7.37 (d, *J* = 8.1 Hz, 2H), 2.48 (s, 3H), 1.37 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 150.2, 150.0, 145.3, 142.0, 139.8, 135.8, 133.1, 129.6, 128.6, 128.5, 85.6, 27.8, 21.7; HRMS-FAB: *m/z* 449.0141 [(M+Na)$^+$; calcd for $C_{17}H_{19}BrN_2NaO_4S^+$: 449.0141].

4.2.23. *tert-butyl (4-bromophenyl)sulfonyl(4-fluorophenyl)carbamate (**8**).*

General procedure I was used employing *tert*-butyl (4-bromophenyl)sulfonylcarbamate (1.681 g, 5.00 mmol) and *bis*(4-fluorophenyl)iodonium triflate (3.030 g, 6.50 mmol), and the reaction was complete in 10 h. Flash chromatography on silica gel using hexane/ethyl acetate (10:1) provided pure **8** (2.086 g, 4.85 mmol, 97%) as a white solid. mp: 128-129 ^{o}C; R_f 0.62 (hexane/ethyl acetate = 4:1); IR (neat) 3092, 2981, 2939, 1736, 1574, 1506, 1471, 1370, 1147, 1087, 744, 619, 569 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.84 (d, *J* = 8.7 Hz, 2H), 7.70 (d, *J* = 8.4 Hz, 2H), 7.25-7.03 (m, 4H), 1.35 (s, 9H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 162.7 (d, *J* = 248 Hz), 150.5, 138.3, 132.1, 132.0, 131.3 (d, *J* = 8.8 Hz), 130.1, 128.9, 116.2 (d, *J* = 23 Hz), 85.0, 27.8; HRMS-FAB: m/z 430.0123 [(M+H)$^+$; calcd for $C_{17}H_{18}BrFNO_4S^+$: 430.0124].

4.2.24. *tert-butyl (4-chlorophenyl)sulfonyl(4-(tert-butyl)phenyl)carbamate (**11**).*

General procedure I was used employing *tert*-butyl (4-chlorophenyl)sulfonylcarbamate (1.459 g, 5.00 mmol) and *bis*(4-(*tert*-butyl)phenyl)iodonium triflate (3.525 g, 6.50 mmol), and the reaction was complete in 4 h. Flash chromatography on silica gel using hexane/ethyl acetate (10:1) provided pure **11** (2.077 g,

4.90 mmol, 98%) as a white solid. mp: 156-158 °C; R_f 0.73 (hexane/ethyl acetate = 4:1); IR (neat) 2967, 2871, 1734, 1476, 1367, 1171, 1090, 973, 757, 626, 582 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 7.93 (d, *J* = 8.4 Hz, 2H), 7.52 (d, *J* = 8.4 Hz, 2H), 7.43 (d, *J* = 8.4 Hz, 2H), 7.16 (d, *J* = 8.7 Hz, 2H),1.35 (s, 9H), 1.33 (s, 9H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 152.1, 150.8, 139.9, 138.2, 133.2, 129.9, 128.9, 128.8, 126.1, 84.6, 34.6, 31.1, 27.7; HRMS-FAB: m/z 446.1168 [$(M+Na)^+$; calcd for $C_{21}H_{26}ClNNaO_4S^+$: 446.1169].

4.3. One-pot procedure for the preparation of mono *N*-arylsulfonamide (Table 4)

Upon completion of General procedure I, as determined by TLC, the reaction mixture (1.00 mmol scale) was cooled to 0 °C. Cold trifluoroacetic acid/water (9/1 mL) was slowly added. The reaction was stirred 1 h at 0 °C at which time the reaction was diluted with toluene. All volatiles were removed with a rotary evaporator, and the product isolated by silica gel chromatography using the solvent system indicated.

4.3.1. *4-methyl-N-phenylbenzenesulfonamide (**12a**).*

The one-pot procedure was used employing *tert*-butyl tosylcarbamate (0.271 g, 1.00 mmol) and diphenyliodonium triflate (0.559 g, 1.30 mmol). Flash chromatography on silica gel using hexane/ethyl acetate (5:1) provided pure **12a** (0.210 g, 0.85 mmol, 85%) as a white solid. R_f 0.59 (hexane/ethyl acetate = 2:1); ^1H NMR (300 MHz, $CDCl_3$) δ 7.67 (d, *J* = 8.4 Hz, 2H), 7.29-7.21 (m, 4H), 7.12-7.06 (m, 3H), 6.91 (br, 1H), 2.37 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 143.9, 136.5, 136.0, 129.6, 129.3, 127.2, 125.3, 121.5, 21.5; Data is consistent with that reported in the literature.[5]

4.3.2. *4-methoxy-N-phenylbenzenesulfonamide (**12b**).*

The one-pot procedure was used employing *tert*-butyl (4-methoxyphenyl)sulfonylcarbamate (0.287 g, 1.00 mmol) and diphenyliodonium triflate (0.645 g, 1.50 mmol). Flash chromatography on silica gel using hexane/ethyl acetate (5:1) provided pure **12b** (0.245 g, 0.93 mmol, 93%) as a white solid. R_f 0.61 (dichloromethane); ^1H NMR (300 MHz, $CDCl_3$) δ 7.76 (d, *J* = 9.0 Hz, 2H), 7.61 (br, 1H), 7.25-7.01 (m, 5H), 6.84 (d, *J* = 9.0 Hz, 2H), 3.76 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 163.0, 136.7, 130.3, 129.4, 129.2, 124.9, 121.2, 114.1, 55.4; Data is consistent with that reported in the literature.[5]

4.3.3. *4-chloro-N-phenylbenzenesulfonamide (**12c**).*

The one-pot procedure was used employing *tert*-butyl (4-chlorophenyl)sulfonylcarbamate (0.292 g, 1.00 mmol) and diphenyliodonium triflate (0.559 g, 1.30 mmol). Flash chromatography on silica gel using hexane/ethyl acetate (5:1) provided pure **12c** (0.249 g, 0.89 mmol, 89%) as a white solid.; R_f 0.62 (dichloromethane); ^1H NMR (300 MHz, $CDCl_3$) δ 7.72 (d, *J* = 8.7 Hz, 2H), 7.39 (d, *J* = 8.7 Hz, 2H), 7.28-7.20 (m, 2H), 7.18-7.04 (m, 4H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 139.6, 137.3, 136.0, 129.4, 129.3, 128.7, 125.8, 121.8; Data is consistent with that reported in the literature.[5]

4.3.4. *4-bromo-N-phenylbenzenesulfonamide (**12d**).*

The one-pot procedure was used employing *tert*-butyl (4-bromophenyl)sulfonylcarbamate (0.292 g, 1.00 mmol) and diphenyliodonium triflate (0.559 g, 1.30 mmol). Flash chromatography on silica gel using hexane/ethyl acetate (5:1) provided pure **12d** (0.088 g, 0.83 mmol, 83%) as a white solid.; R_f 0.62 (dichloromethane); ^1H NMR (300 MHz, $CDCl_3$) δ 7.65 (d, *J* = 8.6 Hz, 2H), 7.53 (d, *J* = 8.7 Hz, 2H), 7.49 (br, 1H), 7.28-7.19 (m, 2H), 7.15-7.07 (m, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 137.7, 136.0, 132.3, 129.4, 128.7, 128.1, 125.6, 121.7; Data is consistent with that reported in the literature. [14]

4.3.5. *N-(4-fluorophenyl)-4-methylbenzenesulfonamide (**12e**).*

The one-pot procedure was used employing *tert*-butyl tosylcarbamate (0.271 g, 1.00 mmol) and bis(4-fluorophenyl)iodonium triflate (0.606 g, 1.30 mmol). Flash chromatography on silica gel using hexane/ethyl acetate (5:1) provided pure **12e** (0.249 g, 0.94 mmol, 94%) as a white solid.; R_f 0.45 (hexane/ethyl acetate = 2:1); ^1H NMR (300 MHz, $CDCl_3$) δ 7.60 (d, *J* = 8.3 Hz, 2H), 7.23 (d, *J* = 7.9 Hz, 2H), 7.06-6.98 (m, 2H), 6.97-6.88 (m, 2H), 6.51 (br, 1H), 2.39 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) 160.6 (d, *J* = 244 Hz), 144.0, 135.8, 132.2 (d, *J* = 1.5 Hz), 129.7, 127.3, 124.9 (d, *J* = 8.3 Hz), 116.1 (d, *J* = 22 Hz), 21.6; Data is consistent with that reported in the literature.[15]

4.3.6. *N-phenylmethanesulfonamide (**12f**).*

The one-pot procedure was used employing *tert*-butyl methylcarbamate (0.195 g, 1.00 mmol) and diphenyliodonium triflate (0.559 g, 1.30 mmol). Flash chromatography on silica gel using hexane/ethyl acetate (5:1) provided pure **12f** (0.156 g, 0.91 mmol, 91%) as a white solid. R_f 0.20 (dichloromethane); ^1H NMR (300 MHz, $CDCl_3$) δ 7.39-7.33 (m, 2H), 7.26-7.16 (m, 3H), 6.96 (s, 1H), 3.02 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 136.7, 129.7, 125.4, 120.8, 39.2; Data is consistent with that reported in the literature.[5]

4.3.7. *N-phenylthiophene-2-sulfonamide (**12g**).*

The one-pot procedure was used employing *tert*-butyl thiophen-2-ylsulfonylcarbamate (0.263 g, 1.00 mmol) and diphenyliodonium triflate (0.559 g, 1.30 mmol). Flash chromatography on silica gel using hexane/ethyl acetate (5:1) provided pure **12g** (0.225 g, 0.94 mmol, 94%) as a colorless oil. R_f 0.43 (hexane/ethyl acetate = 2:1); IR (neat) 3241, 3091, 2825, 1599, 1498, 1404, 1335, 1229, 1153, 1092, 928, 589 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 7.53 (dd, *J* = 5.1 and 1.2 Hz, 1H), 7.49 (dd, *J* = 3.7 and 1.2 Hz, 1H), 7.33-7.25 (m, 2H), 7.20-7.10 (m, 3H), 7.03-6.97 (m, 1H), 6.77 (br, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) 139.3, 136.0, 132.9, 132.4, 129.4, 127.3, 125.9, 122.0; HRMS-FAB: *m/z* 261.9966 [$(M+Na)^+$; calcd for $C_{10}H_9NNaO_2S_2^+$: 261.9967].

4.3.8. *4-methyl-N-(thiophen-3-yl)benzenesulfonamide (**12h**).*

The one-pot procedure was used employing *tert*-butyl tosylcarbamate (0.271 g, 1.00 mmol) and mesityl(thiophen-3-yl)iodonium triflate (0.622 g, 1.30 mmol). Flash chromatography on silica gel using hexane/ethyl acetate (5:1) provided pure **12h** (0.228 g, 0.90 mmol, 90%) as a white solid. R_f 0.45 (hexane/ethyl acetate = 2:1); ^1H NMR (300 MHz, $CDCl_3$) δ 7.67 (d, *J* = 8.0 Hz, 2H), 7.23 (d, *J* = 8.0 Hz, 2H), 7.20-7.13 (m, 1H), 7.04 (br, 1H), 6.90-6.81 (m, 2H), 2.39 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$)

143.9, 135.9, 134.2, 129.6, 127.2, 125.6, 123.3, 113.9, 21.5; Data is consistent with that reported in the literature.[5]

4.4. *N,N-diphenyl-p-toluenesulfonamide (**12aa**).*

A 10 mL round bottom flask was charged with a diphenyliodonium triflate (0.140 g, 0.325 mmol), CuI (10 mol %), triethylamine (0.50 mmol) and the corresponding *p*-toluenesulfonamide (0.043 g, 0.25 mmol). Toluene (2 mL) was then added to the flask. The reaction mixture was stirred at 50 ^{o}C. After 24 h complete consumption of diphenyliodonium triflate was observed by TLC analysis. The solvent was removed *in vacuo* and the residue was purified by flash chromatography on silica gel using hexane/ethyl acetate (15:1) provided pure **12aa** (0.041 g, 0.13 mmol, 51%) as a white solid. R_f 0.65 (hexane/ethyl acetate = 2:1); ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.58 (d, *J* = 8.1 Hz, 2H), 7.35-7.21 (m, 12H), 2.42 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 143.5, 141.5, 137.5, 129.5, 129.2, 128.3, 127.7, 127.3, 21.5; Data is consistent with that reported in the literature.[17] **12a** (0.012 g, 0.05 mmol, 19%) as a white solid. R_f 0.59 (hexane/ethyl acetate = 2:1); ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.67 (d, *J* = 8.4 Hz, 2H), 7.29-7.21 (m, 4H), 7.12-7.06 (m, 3H), 6.91 (br, 1H), 2.37 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 143.9, 136.5, 136.0, 129.6, 129.3, 127.2, 125.3, 121.5, 21.5; Data is consistent with that reported in the literature.[5]

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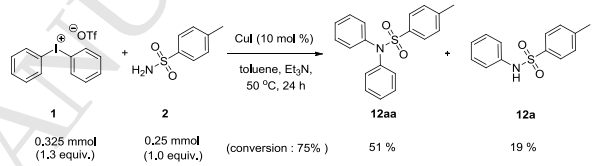

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Supporting Information

Copper-Catalyzed *N*-Arylation of *tert*-Butyl *N*-Sulfonylcarbamates with Diaryliodonium Salts at Room Temperature

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Experimental

I. Materials and Methods

Unless otherwise indicated, all chemical reagents were purchased from Sigma-Aldrich, Alfa Aesar, and TCI and were used without further purification. All diaryliodonium salts[1] and *tert*-butyl *N*-sulfonylcarbamates[2] were prepared by the reported procedures. All reactions were carried out in oven-dried glassware equipped with a magnetic stir bar. Reactions were monitored by thin layer chromatography (TLC) with 0.25-mm E. Merck pre-coated silica gel plates (Kieselgel $60F_{254}$, Merck). Products were detected by viewing under a UV light, by staining with an anisaldehyde solution composed of acetic acid, sulfuric acid, and MeOH, or by staining with a $KMnO_4$ solution composed of potassium carbonate, sodium hydroxide, and water. Flash column chromatography was performed on Merck 60 silica gel (70-230 mesh). Yields refer to chromatographically and spectroscopically pure compounds unless otherwise noted. ^1H and ^{13}C spectra were recorded on a Bruker AM-300 or Varian Unity-Inova 500 MHz spectrometer. Chemical shifts are reported as δ values relative to internal $SiMe_4$ or chloroform (δ 0.00 for ^1H and δ 77.0 for ^{13}C) or benzene (δ 128.0 for ^{13}C). IR spectra were measured as neat oils on a Varian Scimitar 800 FT-IR spectrometer. High resolution spectra were obtained at Sogang University's Organic Chemistry Research Center or the Korea Basic Science Institute Mass Spectrometry Service Center.

II. Experimental Section

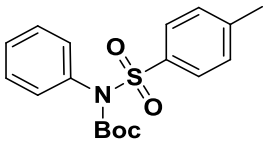
General procedure I for copper-catalyzed *N*-arylation (Table 1-3, scheme 2)

A 10 mL round bottom flask was charged with a diaryliodonium salt (0.325 mmol), CuCl (10 mol %), triethylamine (0.50 mmol) and the corresponding *tert*-butyl *N*-sulfonylcarbamate (0.25 mmol). Toluene (0.12 M) was then added to the flask. The reaction mixture was stirred at room temperature. After completion of the reaction, as monitored by TLC analysis, the

solvent was removed *in vacuo* and the residue was purified by flash column chromatography on silica gel to obtain the desired product.

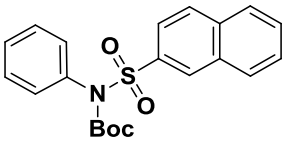
One-pot procedure for the production of mono *N*-arylsulfonamides

Upon completion of **General procedure I**, as determined by TLC, the reaction mixture (1.00 mmol scale) was cooled to 0 ^{o}C. Cold trifluoroacetic acid/water (9/1 mL) was slowly added. The reaction was stirred 1 h at 0 ^{o}C at which time the reaction was diluted with toluene. All volatiles were removed with a rotary evaporator, and the product isolated by silica gel chromatography using the solvent system indicated.



3a

tert-butyl phenyl(tosyl)carbamate (3a). General procedure **I** was used employing *tert*-butyl tosylcarbamate (0.068 g, 0.25 mmol) and diphenyliodonium triflate (0.140 g, 0.325 mmol), and the reaction was complete in 1 h. Flash chromatography on silica gel using hexane/ethyl acetate (7:1) provided pure **3a** (0.084 g, 0.24 mmol, 97%) as a white solid. R_f 0.47 (hexane/ethyl acetate = 3:1); ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.87 (d, *J* = 8.1 Hz, 2H), 7.48-7.37 (m, 3H), 7.34 (d, *J* = 8.1 Hz, 2H), 7.29-7.20 (m, 2H), 2.46 (s, 3H), 1.34 (s, 9H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 150.9, 144.4, 136.7, 136.4, 129.6, 129.3, 129.0, 128.9, 128.6, 84.3, 27.8, 21.6; Data is consistent with that reported in the literature.[1]

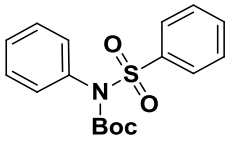


3b

tert-butyl naphthalene-2-ylsulfonyl(phenyl)carbamate (3b). General procedure **I** was used employing *tert*-butyl naphthalen-2-ylsulfonylcarbamate (0.077 g, 0.25 mmol) and diphenyliodonium triflate (0.140 g, 0.325 mmol), and the reaction was complete in 4 h. Flash chromatography on silica gel using hexane/ethyl acetate (7:1) provided pure **3b** (0.092 g, 0.24

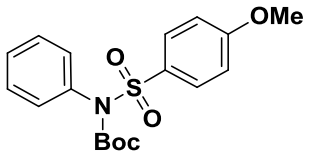
[1] Vellemäe, E.; Lebedev, O.; Mäeorg, U. *Tetrahedron Lett*. **2008**, *49*, 1373-1375.

mmol, 96%) as a white solid. mp: 126-128 ^{o}C; R_f 0.44 (hexane/ethyl acetate = 3:1); IR (neat) 3059, 2977, 1734, 1352, 1292, 1150, 1073, 814, 695 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.59 (s, 1H), 8.02-7.88 (m, 4H), 7.72-7.57 (m, 2H), 7.45-7.38 (m, 3H), 7.36-7.26 (m, 2H), 1.31 (s, 9H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 150.8, 136.4, 136.3, 135.1, 131.7, 130.5, 129.7, 129.3, 129.2, 129.1, 129.0, 128.9, 127.9, 127.6, 123.1, 84.5, 27.7; HRMS-FAB: *m/z* 406.1084 [$(M+Na)^+$; calcd for $C_{21}H_{21}NNaO_4S^+$: 406.1083].



3c

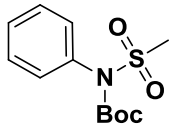
tert-butyl phenyl(phenylsulfonyl)carbamate (3c). General procedure **I** was used employing *tert*-butyl phenylsulfonylcarbamate (0.064 g, 0.25 mmol) and diphenyliodonium triflate (0.140 g, 0.325 mmol), and the reaction was complete in 10 h. Flash chromatography on silica gel using hexane/ethyl acetate (7:1) provided pure **3c** (0.080 g, 0.24 mmol, 96%) as a white solid. mp: 67-68 ^{o}C; R_f 0.43 (hexane/ethyl acetate = 3:1); IR (neat) 3065, 2981, 1734, 1598, 1790, 1368, 1151, 1091, 970, 812, 696, 580 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.00 (d, *J* = 7.5 Hz, 2H), 7.68-7.60 (m, 1H), 7.58-7.50 (m, 2H), 7.46-7.38 (m, 3H), 7.29-7.25 (m, 2H), 1.32 (s, 9H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 150.7, 139.6, 136.2, 133.4, 129.6, 129.0, 128.97, 128.6, 128.4, 84.5, 27.7; HRMS-FAB: *m/z* 356.0927 [$(M+Na)^+$; calcd for $C_{17}H_{19}NNaO_4S^+$: 356.0927].



3d

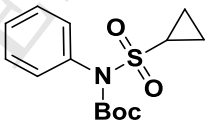
tert-butyl (4-methoxyphenyl)sulfonyl(phenyl)carbamate (3d). General procedure **I** was used employing *tert*-butyl (4-methoxyphenyl)sulfonylcarbamate (0.072 g, 0.25 mmol) and diphenyliodonium triflate (0.161 g, 0.375 mmol), and the reaction was complete in 10 h. Flash chromatography on silica gel using hexane/ethyl acetate (7:1) provided pure **3d** (0.088 g, 0.24 mmol, 97%) as a colorless oil.; R_f 0.30 (hexane/ethyl acetate = 3:1); IR (neat) 2981, 1733, 1596, 1498, 1368, 1263, 1150, 1090, 839, 677, 581 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$)

δ 7.92 (d, *J* = 8.7 Hz, 2H), 7.46-7.38 (m, 3H), 7.27-7.21 (m, 2H), 7.00 (d, *J* = 9.0 Hz, 2H), 3.89 (s, 3H), 1.35 (s, 9H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 163.5, 151.0, 136.6, 131.1, 130.9, 129.6, 129.0, 128.9, 113.8, 84.2, 55.6, 27.8; HRMS-FAB: *m/z 364.1213* [$(M+Na)^+$; calcd for $C_{18}H_{22}NO_5S^+$: 364.1213].



3e

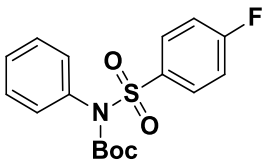
tert-butyl methylsulfonyl(phenyl)carbamate (3e). General procedure **I** was used employing *tert*-butyl methylsulfonylcarbamate (0.049 g, 0.25 mmol) and diphenyliodonium triflate (0.140 g, 0.325 mmol), and the reaction was complete in 8 h. Flash chromatography on silica gel using hexane/ethyl acetate (7:1) provided pure **3e** (0.062 g, 0.23 mmol, 91%) as a white solid. mp: 83-85 ^{o}C; R_f 0.27 (hexane/ethyl acetate = 3:1); IR (neat) 3015, 2982, 2939, 1732, 1596, 1492, 1356, 1148, 967, 838, 694, 547 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.45-7.37 (m, 3H), 7.27-7.21 (m, 2H), 3.42 (s, 3H), 1.46 (s, 9H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 151.5, 135.6, 129.3, 129.1, 129.05, 84.7, 41.6, 27.8; HRMS-FAB: *m/z* 294.0772 [$(M+Na)^+$; calcd for $C_{12}H_{17}NNaO_4S^+$: 294.0770].



3f

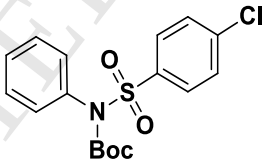
tert-butyl cyclopropylsulfonyl(phenyl)carbamate (3f). General procedure **I** was used employing *tert*-butyl cyclopropylsulfonylcarbamate (0.055 g, 0.25 mmol) and diphenyliodonium triflate (0.140 g, 0.325 mmol), and the reaction was complete in 12 h. Flash chromatography on silica gel using hexane/ethyl acetate (7:1) provided pure **3f** (0.071 g, 0.24 mmol, 96%) as a white solid. mp: 75-76 ^{o}C; R_f 0.34 (hexane/ethyl acetate = 3:1); IR (neat) 2982, 1733, 1490, 1361, 1293, 1145, 1074, 970, 885, 711, 586 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.46-7.33 (m, 3H), 7.28-7.22 (m, 2H), 3.36-3.25 (m, 1H), 1.47 (s, 9H), 1.36-1.29 (m, 2H), 1.18-1.09 (m, 2H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 151.6, 136.2, 129.4, 129.0,

128.8, 84.3, 31.7, 27.8, 6.0; HRMS-FAB: *m/z* 298.1107 [$(M+Na)^+$; calcd for $C_{14}H_{20}NO_4S^+$: 298.1108].



3g

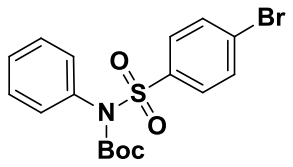
tert-butyl (4-fluorophenyl)sulfonyl(phenyl)carbamate (3g). General procedure **I** was used employing *tert*-butyl (4-fluorophenyl)sulfonylcarbamate (0.069 g, 0.25 mmol) and diphenyliodonium triflate (0.140 g, 0.325 mmol), and the reaction was complete in 6 h. Flash chromatography on silica gel using hexane/ethyl acetate (7:1) provided pure **3g** (0.084 g, 0.24 mmol, 96%) as a white solid. mp: 80-82 ^oC; R_f 0.55 (hexane/ethyl acetate = 3:1); IR (neat) 2982, 1734, 1591, 1494, 1370, 1151, 968, 841, 695, 579 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 8.09-7.96 (m, 2H), 7.47-7.37 (m, 3H), 7.29-7.20 (m, 4H), 1.34 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 165.5 (d, *J* = 104 Hz), 150.7, 136.2, 135.5, 131.4 (d, *J* = 7.6 Hz), 129.5, 129.1, 129.06, 115.9 (d, *J* = 17.8 Hz), 84.6, 27.7; HRMS-FAB: *m/z* 374.0833 [$(M+Na)^+$; calcd for $C_{17}H_{18}FNNaO_4S^+$: 374.0833].



3h

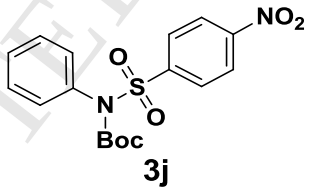
tert-butyl (4-chlorophenyl)sulfonyl(phenyl)carbamate (3h). General procedure **I** was used employing *tert*-butyl (4-chlorophenyl)sulfonylcarbamate (0.073 g, 0.25 mmol) and diphenyliodonium triflate (0.140 g, 0.325 mmol), and the reaction was complete in 4 h. Flash chromatography on silica gel using hexane/ethyl acetate (7:1) provided pure **3h** (0.088 g, 0.24 mmol, 96%) as a white solid. mp: 102-103 ^oC; R_f 0.61 (hexane/ethyl acetate = 3:1); IR (neat) 2982, 1733, 1584, 1491, 1371, 1150, 1092, 839, 759, 694, 576 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 7.93 (d, *J* = 8.8 Hz, 2H), 7.52 (d, *J* = 8.7 Hz, 2H), 7.46-7.38 (m, 3H), 7.26-7.18 (m, 2H), 1.34 (s, 9H); ^{13}C NMR (75 MHz, C_6D_6) δ 151.1, 139.8, 138.9, 137.1, 130.7, 130.1,

129.3, 129.1, 129.0, 84.0, 27.7; HRMS-FAB: *m/z* 390.0536 [$(M+Na)^+$; calcd for $C_{17}H_{18}ClNNaO_4S^+$: 390.0537].



3i

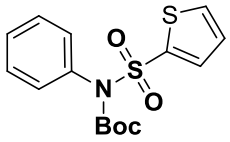
tert-butyl (4-bromophenyl)sulfonyl(phenyl)carbamate (3i). General procedure **I** was used employing *tert*-butyl (4-bromophenyl)sulfonylcarbamate (0.084 g, 0.25 mmol) and diphenyliodonium triflate (0.140 g, 0.325 mmol), and the reaction was complete in 10 h. Flash chromatography on silica gel using hexane/ethyl acetate (7:1) provided pure **3i** (0.098 g, 0.24 mmol, 95%) as a white solid. mp: 108-110 ^oC; R_f 0.61 (hexane/ethyl acetate = 3:1); IR (neat) 2981, 1736, 1574, 1491, 1391, 1370, 1290, 1148, 745, 602 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 7.86 (d, *J* = 8.4 Hz, 2H), 7.69 (d, *J* = 8.4 Hz, 2H), 7.47-7.38 (m, 3H), 7.25-7.18 (m, 2H), 1.34 (s, 9H); ^{13}C NMR (75 MHz, C_6D_6) δ 151.1, 139.4, 137.2, 132.0, 130.7, 130.1, 129.2, 129.0, 128.5, 84.0, 27.7; HRMS-FAB: *m/z* 412.0212 [$(M+Na)^+$; calcd for $C_{17}H_{19}BrNO_4S^+$: 412.0213].



3j

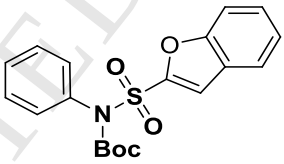
tert-butyl (4-nitrophenyl)sulfonyl(phenyl)carbamate (3j). General procedure **I** was used employing *tert*-butyl (4-nitrophenyl)sulfonylcarbamate (0.076 g, 0.25 mmol) and diphenyliodonium triflate (0.161 g, 0.375 mmol), and the reaction was complete in 24 h. Flash chromatography on silica gel using hexane/ethyl acetate (7:1) provided pure **3j** (0.089 g, 0.235 mmol, 94%) as a white solid. mp: 146-148 ^oC; R_f 0.46 (hexane/ethyl acetate = 3:1); IR (neat) 3117, 2975, 1734, 1611, 1491, 1361, 1286, 1151, 1090, 747, 695, 568 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 8.40 (d, *J* = 9.3 Hz, 2H), 8.20 (d, *J* = 8.7 Hz, 2H), 7.50-7.41 (m, 3H), 7.25-7.17 (m, 2H), 1.34 (s, 9H); ^{13}C NMR (75 MHz, C_6D_6) δ 151.0, 150.4, 145.1, 136.7,

130.1, 130.0, 129.4, 129.3, 123.8, 84.5, 27.6; HRMS-FAB : *m/z* 401.0778 [$(M+Na)^+$; calcd for $C_{17}H_{18}N_2NaO_6S^+$: 401.0778].



3k

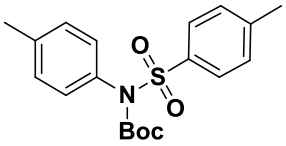
tert-butyl phenyl(thiophen-2-ylsulfonyl)carbamate (3k). General procedure **I** was used employing *tert*-butyl thiophen-2-ylsulfonylcarbamate (0.066 g, 0.25 mmol) and diphenyliodonium triflate (0.140 g, 0.325 mmol), and the reaction was complete in 10 h. Flash chromatography on silica gel using hexane/ethyl acetate (7:1) provided pure **3k** (0.083 g, 0.245 mmol, 98%) as a white oil.; R_f 0.50 (hexane/ethyl acetate = 3:1); IR (neat) 3105, 2924, 2854, 1733, 1596, 1372, 1290, 1148, 914, 742, 593 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 7.80 (dd, *J* = 3.8 and 1.3 Hz, 1H), 7.71 (dd, *J* = 5.0 and 1.3 Hz, 1H), 7.46-7.38 (m, 3H), 7.27-7.20 (m, 2H), 7.14 (dd, *J* = 6.3 and 4.9 Hz, 1H), 1.40 (s, 9H); ^{13}C NMR (75 MHz, C_6D_6) δ 150.9, 140.2, 137.0, 134.9, 132.8, 129.8, 128.8, 128.6, 126.4, 83.6, 27.4; HRMS-FAB: *m/z* 340.0672 [$(M+Na)^+$; calcd for $C_{15}H_{18}NO_4S_2^+$: 340.0672].



3l

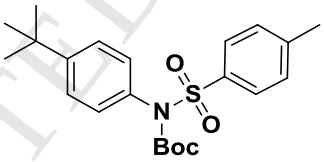
tert-butyl benzofuran-2-ylsulfonyl(phenyl)carbamate (3l). General procedure **I** was used employing *tert*-butyl benzofuran-2-ylsulfonylcarbamate (0.074 g, 0.25 mmol) and diphenyliodonium triflate (0.269 g, 0.625 mmol), and the reaction was complete in 24 h. Flash chromatography on silica gel using hexane/dichloromathane (1:1) provided pure **3l** (0.085 g, 0.23 mmol, 91%) as a white solid. mp: 116-118 ^oC; R_f 0.26 (hexane/ethyl acetate = 3:1); IR (neat) 3120, 2984, 2931, 1738, 1548, 1490, 1374, 1291, 1144, 974, 838, 756, 565 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 7.74 (d, *J* = 7.8 Hz, 1H), 7.66-7.36 (m, 9H), 1.35 (s, 9H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 155.6, 150.3, 149.1, 135.6, 129.9, 129.3, 129.2, 128.2,

125.7, 124.4, 123.1, 114.8, 112.3, 85.2, 27.7; HRMS-FAB: *m/z* 396.0877 [$(M+Na)^+$; calcd for $C_{19}H_{19}NNaO_5S^+$: 396.0876].



5a

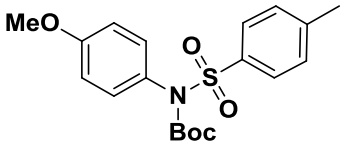
tert-butyl p-tolyl(tosyl)carbamate (5a). General procedure **I** was used employing *tert*-butyl tosylcarbamate (0.068 g, 0.25 mmol) and di-*p*-tolyliodonium triflate (0.149 g, 0.325 mmol), and the reaction was complete in 6 h. Flash chromatography on silica gel using hexane/ethyl acetate (7:1) provided pure **5a** (0.086 g, 0.24 mmol, 95%) as a white solid. mp: 132-134 ^oC; R_f 0.56 (hexane/ethyl acetate = 3:1); IR (neat) 2982, 2929, 1733, 1511, 1368, 1290, 1151, 971, 841, 669 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 7.86 (d, *J* = 8.3 Hz, 2H), 7.33 (d, *J* = 7.7 Hz, 2H), 7.22 (d, *J* = 7.7 Hz, 2H), 7.13 (d, *J* = 8.3 Hz, 2H), 2.46 (s, 3H), 2.38 (s, 3H), 1.34 (s, 9H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 151.0, 144.3, 139.0, 136.8, 133.8, 129.8, 129.3, 129.25, 128.5, 84.3, 27.8, 21.6, 21.2; HRMS-FAB: *m/z* 384.1241 [$(M+Na)^+$; calcd for $C_{19}H_{23}NNaO_4S^+$: 384.1240].



5b

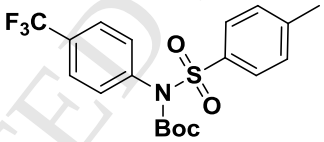
tert-butyl (4-(tert-butyl)phenyl)(tosyl)carbamate (5b). General procedure **I** was used employing *tert*-butyl tosylcarbamate (0.068 g, 0.25 mmol) and bis(4-(*tert*-butyl)phenyl)iodonium triflate (0.176 g, 0.325 mmol), and the reaction was complete in 4 h. Flash chromatography on silica gel using hexane/ethyl acetate (7:1) provided pure **5b** (0.089 g, 0.22 mmol, 88%) as a white solid. mp: 156-158 ^oC; R_f 0.63 (hexane/ethyl acetate = 3:1); IR (neat) 2966, 1734, 1368, 1151, 1091, 665, 591 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 7.87 (d, *J* = 8.1 Hz, 2H), 7.43 (d, *J* = 8.4 Hz, 2H), 7.31 (d, *J* = 8.1 Hz, 2H), 7.18 (d, *J* = 8.4 Hz, 2H), 2.47 (s, 3H), 1.35 (s, 9H), 1.34 (s, 9H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 151.9, 151.1,

144.3, 137.0, 133.6, 129.3. 129.0, 128.5, 126.1, 84.4, 34.7, 31.3, 27.8, 21.6; HRMS-FAB: *m/z* 426.1709 [$(M+Na)^+$; calcd for $C_{22}H_{29}NNaO_4S^+$: 426.1710].



5c

tert-butyl (4-methoxyphenyl)(tosyl)carbamate (5c). General procedure **I** was used employing *tert*-butyl tosylcarbamate (0.068 g, 0.25 mmol) and bis(4-methoxyphenyl)iodonium triflate (0.159 g, 0.325 mmol), and the reaction was complete in 12 h. Flash chromatography on silica gel using hexane/ethyl acetate (7:1) provided pure **5c** (0.091 g, 0.24 mmol, 96%) as a white solid. R_f 0.43 (hexane/ethyl acetate = 3:1); ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.86 (d, *J* = 8.4 Hz, 2H), 7.33 (d, *J* = 8.1 Hz, 2H), 7.16 (dd, *J* = 6.9 and 2.1 Hz, 2H), 6.92 (d, *J* = 8.7 Hz, 2H), 3.81 (s, 3H), 2.45 (s, 3H), 1.34 (s, 9H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 159.7, 151.0, 144.3, 136.7, 130.6, 129.2, 128.9, 128.4, 114.2, 84.2, 55.3, 27.7, 21.5; Data is consistent with that reported in the literature.[2]

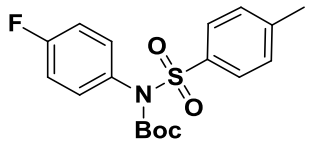


5d

tert-butyl tosyl(4-trifluoromethyl)phenyl)carbamate (5d). General procedure **I** was used employing *tert*-butyl tosylcarbamate (0.068 g, 0.25 mmol) and bis(4-(trifluoromethyl)phenyl)iodonium triflate (0.184 g, 0.325 mmol), and the reaction was complete in 8 h. Flash chromatography on silica gel using hexane/ethyl acetate (7:1) provided pure **5d** (0.090 g, 0.22 mmol, 87%) as a white solid. mp: 136-138 ^{o}C; R_f 0.59 (hexane/ethyl acetate = 3:1); IR (neat) 2984, 2931, 1738, 1612, 1326, 1169, 1020, 812, 766, 579 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.86 (d, *J* = 8.3 Hz, 2H), 7.69 (d, *J* = 8.3 Hz, 2H), 7.37 (t, *J* = 8.3 Hz, 4H), 2.47 (s, 3H), 1.35 (s, 9H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 150.4, 144.9, 139.6,

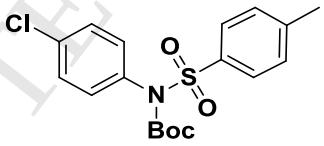
[2] Ankner, T.; Hilmersson, G. *Org. Lett.* **2009**, *11*, 503-506

139.59, 136.3, 130.9 (q, *J* = 33 Hz), 130.3, 129.5, 128.5, 126.2 (q, *J* = 3.8 Hz), 85.1, 27.7, 21.6; HRMS-FAB: *m/z* 438.0957 [$(M+Na)^+$; calcd for $C_{19}H_{20}F_3NNaO_4^+$: 438.0957].



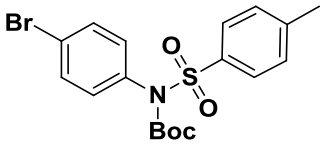
5e

tert-butyl (4-fluorophenyl)(tosyl)carbamate (5e). General procedure **I** was used employing *tert*-butyl tosylcarbamate (0.068 g, 0.25 mmol) and bis(4-fluorophenyl)iodonium triflate (0.152 g, 0.325 mmol), and the reaction was complete in 6 h. Flash chromatography on silica gel using hexane/ethyl acetate (7:1) provided pure **5e** (0.088 g, 0.24 mmol, 96%) as a white solid. mp: 104-106 ^oC; R_f 0.56 (hexane/ethyl acetate = 3:1); IR (neat) 3449, 2986, 1733, 1598, 1505, 1373, 1093, 975, 842, 671 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 7.85 (d, *J* = 8.4 Hz, 2H), 7.35 (d, *J* = 8.0 Hz, 2H), 7.27-7.19 (m, 2H), 7.15-7.06 (s, 2H), 2.46 (s, 3H), 1.35 (s, 9H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 162.6 (d, *J* = 248 Hz), 150.8, 144.6, 136.5, 132.4 (d, *J* = 3.6 Hz), 131.4 (d, *J* = 8.8 Hz), 129.4, 128.5, 116.0 (d, *J* = 23 Hz), 84.6, 27.8, 21.6; HRMS-FAB: *m/z* 388.0989 [$(M+Na)^+$; calcd for $C_{18}H_{20}FNNaO_4S^+$: 388.0989].



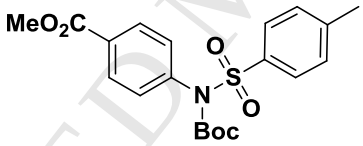
5f

tert-butyl (4-chlorophenyl)(tosyl)carbamate (5f). General procedure **I** was used employing *tert*-butyl tosylcarbamate (0.068 g, 0.25 mmol) and bis(4-chlorophenyl)iodonium triflate (0.162 g, 0.325 mmol), and the reaction was complete in 10 h. Flash chromatography on silica gel using hexane/ethyl acetate (7:1) provided pure **5f** (0.088 g, 0.23 mmol, 92%) as a white solid. mp: 132-133 ^oC; R_f 0.59 (hexane/ethyl acetate = 3:1); IR (neat) 3455, 2980, 1733, 1597, 1489, 1373, 1250, 1091, 973, 813, 665 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 7.85 (d, *J* = 8.0 Hz, 2H), 7.43-7.30 (m, 4H), 7.18 (d, *J* = 8.4 Hz, 2H), 2.46 (s, 3H), 1.35 (s, 9H); ^{13}C NMR (75 MHz, C_6D_6) δ 151.0, 144.3, 137.6, 136.0, 135.0, 131.6, 129.4 (2C), 129.2, 83.9, 27.7, 21.2; HRMS-FAB: *m/z* 404.0694 [$(M+Na)^+$; calcd for $C_{18}H_{20}ClNNaO_4S^+$: 404.0694].



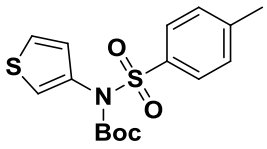
5g

tert-butyl (4-bromophenyl)(tosyl)carbamate (5g). General procedure **I** was used employing *tert*-butyl tosylcarbamate (0.068 g, 0.25 mmol) and bis(4-bromophenyl)iodonium triflate (0.191 g, 0.325 mmol), and the reaction was complete in 6 h. Flash chromatography on silica gel using hexane/ethyl acetate (7:1) provided pure **5g** (0.102 g, 0.24 mmol, 96%) as a white solid. mp: 148-150 ^{o}C; R_f 0.59 (hexane/ethyl acetate = 3:1); IR (neat) 2987, 2931, 1739, 1595, 1487, 1284, 1173, 1071, 838, 730, 664, 580 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.84 (d, *J* = 8.0 Hz, 2H), 7.55 (d, *J* = 8.9 Hz, 2H), 7.34 (d, *J* = 8.5 Hz, 2H), 7.12 (d, *J* = 8.5 Hz, 2H), 2.47 (s, 3H), 1.35 (s, 9H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 150.5, 144.7, 136.4, 135.5, 132.3, 131.3, 129.4, 128.6, 123.1, 84.8, 27.8, 21.7; HRMS-FAB: *m/z* 448.0189 [$(M+Na)^+$; calcd for $C_{18}H_{20}BrNNaO_4S^+$: 448.0189].



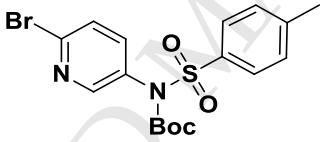
5i

methyl 4-(N-tert-butoxycarbonyl)-4-methylphenylsulfonamido)benzoate (5i). General procedure **I** was used employing *tert*-butyl tosylcarbamate (0.068 g, 0.25 mmol) and mesityl(4-(methoxycarbonyl)phenyl)iodonium triflate (0.172 g, 0.325 mmol), and the reaction was complete in 6 h. Flash chromatography on silica gel using hexane/ethyl acetate (7:1) provided pure **5i** (0.092 g, 0.23 mmol, 91%) as a white solid. mp: 140-142 ^{o}C; R_f 0.41 (hexane/ethyl acetate = 3:1); IR (neat) 2983, 1729, 1605, 1370, 1281, 1150, 702, 661, 548 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.10 (d, *J* = 8.4 Hz, 2H), 7.86 (d, *J* = 8.4 Hz, 2H), 7.39-7.26 (m, 4H), 3.94 (s, 3H), 2.47 (s, 3H), 1.34 (s, 9H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 166.2, 150.4, 144.8, 140.5, 136.4, 130.5, 135.4, 129.7, 129.4, 128.6, 84.8, 52.3, 27.8, 21.7; HRMS-FAB: *m/z* 428.1138 [$(M+Na)^+$; calcd for $C_{20}H_{23}NNaO_6S^+$: 428.1138].



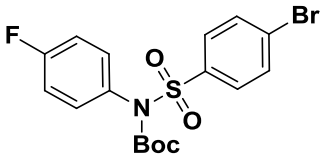
5j

tert-butyl thiophen-3-yl(tosyl)carbamate (5j). General procedure **I** was used employing *tert*-butyl tosylcarbamate (0.068 g, 0.25 mmol) and mesityl(thiophen-3-yl)iodonium triflate (0.155 g, 0.325 mmol), and the reaction was complete in 16 h. Flash chromatography on silica gel using hexane/ethyl acetate (7:1) provided pure **5j** (0.083 g, 0.235 mmol, 94%) as a white solid. mp: 115-117 ^{o}C; R_f 0.52 (hexane/ethyl acetate = 3:1); IR (neat) 3108, 2976, 2360, 1734, 1527, 1369, 1148, 812, 636 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.85 (d, *J* = 8.0 Hz, 2H), 7.36-7.24 (m, 4H), 6.96 (dd, *J* = 5.0 and 1.1 Hz, 1H), 2.45 (s, 3H), 1.35 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 150.6, 144.5, 136.5, 133.3, 129.3, 128.4, 127.2, 124.8, 124.3, 84.5, 27.8, 21.6; HRMS-FAB: *m/z* 376.0647 [$(M+Na)^+$; calcd for $C_{16}H_{19}NNaO_4S_2^+$: 376.0648].



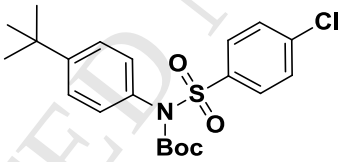
5k

tert-butyl (6-bromopyridin-3-yl)(tosyl)carbamate (5k). General procedure **I** was used employing *tert*-butyl tosylcarbamate (0.068 g, 0.25 mmol) and (6-bromopyridin-3-yl)(2,4,6-triethylphenyl)iodonium triflate (0.193 g, 0.325 mmol), and the reaction was complete in 12 h. Flash chromatography on silica gel using hexane/ethyl acetate (7:1) provided pure **5k** (0.065 g, 0.15 mmol, 61%) as a white solid. mp: 166-168 ^{o}C; R_f 0.48 (hexane/ethyl acetate = 3:1); IR (neat) 2976, 2360, 1735, 1370, 1285, 1149, 1090, 664, 582 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.16 (d, *J* = 2.2 Hz, 1H), 7.83 (d, *J* = 8.1 Hz, 2H), 7.58 (d, *J* = 8.7 Hz, 1H), 7.50 (dd, *J* = 8.7 and 2.7 Hz, 1H), 7.37 (d, *J* = 8.1 Hz, 2H), 2.48 (s, 3H), 1.37 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 150.2, 150.0, 145.3, 142.0, 139.8, 135.8, 133.1, 129.6, 128.6, 128.5, 85.6, 27.8, 21.7; HRMS-FAB: *m/z* 449.0141 [$(M+Na)^+$; calcd for $C_{17}H_{19}BrN_2NaO_4S^+$: 449.0141].



8

tert-butyl (4-bromophenyl)sulfonyl(4-fluorophenyl)carbamate (8). General procedure **I** was used employing *tert*-butyl (4-bromophenyl)sulfonylcarbamate (1.681 g, 5.00 mmol) and *bis*(4-fluorophenyl)iodonium triflate (3.030 g, 6.50 mmol), and the reaction was complete in 10 h. Flash chromatography on silica gel using hexane/ethyl acetate (10:1) provided pure **8** (2.086 g, 4.85 mmol, 97%) as a white solid. mp: 128-129 ^{o}C; R_f 0.62 (hexane/ethyl acetate = 4:1); IR (neat) 3092, 2981, 2939, 1736, 1574, 1506, 1471, 1370, 1147, 1087, 744, 619, 569 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.84 (d, *J* = 8.7 Hz, 2H), 7.70 (d, *J* = 8.4 Hz, 2H), 7.25-7.03 (m, 4H), 1.35 (s, 9H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 162.7 (d, *J* = 248 Hz), 150.5, 138.3, 132.1, 132.0, 131.3 (d, *J* = 8.8 Hz), 130.1, 128.9, 116.2 (d, *J* = 23 Hz), 85.0, 27.8; HRMS-FAB: m/z 430.0123 [$(M+H)^+$; calcd for $C_{17}H_{18}BrFNO_4S^+$: 430.0124].

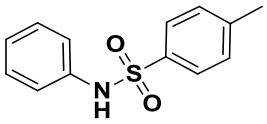


11

tert-butyl (4-chlorophenyl)sulfonyl(4-(tert-butyl)phenyl)carbamate (11). General procedure **I** was used employing *tert*-butyl (4-chlorophenyl)sulfonylcarbamate (1.459 g, 5.00 mmol) and *bis*(4-(*tert*-butyl)phenyl)iodonium triflate (3.525 g, 6.50 mmol), and the reaction was complete in 4 h. Flash chromatography on silica gel using hexane/ethyl acetate (10:1) provided pure **11** (2.077 g, 4.90 mmol, 98%) as a white solid. mp: 156-158 ^{o}C; R_f 0.73 (hexane/ethyl acetate = 4:1); IR (neat) 2967, 2871, 1734, 1476, 1367, 1171, 1090, 973, 757, 626, 582 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.93 (d, *J* = 8.4 Hz, 2H), 7.52 (d, *J* = 8.4 Hz, 2H), 7.43 (d, *J* = 8.4 Hz, 2H), 7.16 (d, *J* = 8.7 Hz, 2H), 1.35 (s, 9H), 1.33 (s, 9H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 152.1, 150.8, 139.9, 138.2, 133.2, 129.9, 128.9, 128.8, 126.1, 84.6, 34.6, 31.1, 27.7; HRMS-FAB: m/z 446.1168 [$(M+Na)^+$; calcd for $C_{21}H_{26}ClNNaO_4S^+$: 446.1169].

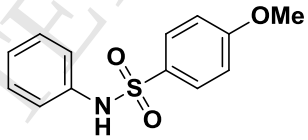
One-pot procedure for the production of mono *N*-arylsulfonamide (Table 4)

Upon completion of **General procedure I**, as determined by TLC, the reaction mixture (1.00 mmol scale) was cooled to 0 ^{o}C. Cold trifluoroacetic acid/water (9/1 mL) was slowly added. The reaction was stirred 1 h at 0 ^{o}C at which time the reaction was diluted with toluene. All volatiles were removed with a rotary evaporator, and the product isolated by silica gel chromatography using the solvent system indicated.



12a

4-methyl-N-phenylbenzenesulfonamide (12a). The one-pot procedure was used employing *tert*-butyl tosylcarbamate (0.271 g, 1.00 mmol) and diphenyliodonium triflate (0.559 g, 1.30 mmol). Flash chromatography on silica gel using hexane/ethyl acetate (5:1) provided pure **12a** (0.210 g, 0.85 mmol, 85%) as a white solid. R_f 0.59 (hexane/ethyl acetate = 2:1); ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.67 (d, *J* = 8.4 Hz, 2H), 7.29-7.21 (m, 4H), 7.12-7.06 (m, 3H), 6.91 (br, 1H), 2.37 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 143.9, 136.5, 136.0, 129.6, 129.3, 127.2, 125.3, 121.5, 21.5; Data is consistent with that reported in the literature.[3]

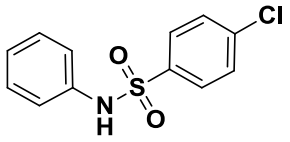


12b

4-methoxy-N-phenylbenzenesulfonamide (12b). The one-pot procedure was used employing *tert*-butyl (4-methoxyphenyl)sulfonylcarbamate (0.287 g, 1.00 mmol) and diphenyliodonium triflate (0.645 g, 1.50 mmol). Flash chromatography on silica gel using hexane/ethyl acetate (5:1) provided pure **12b** (0.245 g, 0.93 mmol, 93%) as a white solid. R_f 0.61 (dichloromethane); ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.76 (d, *J* = 9.0 Hz, 2H), 7.61 (br, 1H), 7.25-7.01 (m, 5H), 6.84 (d, *J* = 9.0 Hz, 2H), 3.76 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ

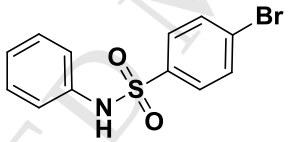
[3] Moon, S. –Y.; Nam, J.; Rathwell, K.; Kim, W. –S. *Org. Lett.* **2014**, *16*, 338-341

163.0, 136.7, 130.3, 129.4, 129.2, 124.9, 121.2, 114.1, 55.4; Data is consistent with that reported in the literature.[3]



12c

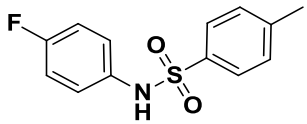
4-chloro-N-phenylbenzenesulfonamide (12c). The one-pot procedure was used employing *tert*-butyl (4-chlorophenyl)sulfonylcarbamate (0.292 g, 1.00 mmol) and diphenyliodonium triflate (0.559 g, 1.30 mmol). Flash chromatography on silica gel using hexane/ethyl acetate (5:1) provided pure **12c** (0.249 g, 0.89 mmol, 89%) as a white solid.; R_f 0.62 (dichloromethane); ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.72 (d, *J* = 8.7 Hz, 2H), 7.39 (d, *J* = 8.7 Hz, 2H), 7.28-7.20 (m, 2H), 7.18-7.04 (m, 4H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 139.6, 137.3, 136.0, 129.4, 129.3, 128.7, 125.8, 121.8; Data is consistent with that reported in the literature.[3]



12d

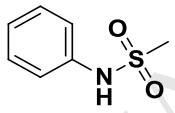
4-bromo-N-phenylbenzenesulfonamide (12d). The one-pot procedure was used employing *tert*-butyl (4-bromophenyl)sulfonylcarbamate (0.292 g, 1.00 mmol) and diphenyliodonium triflate (0.559 g, 1.30 mmol). Flash chromatography on silica gel using hexane/ethyl acetate (5:1) provided pure **12d** (0.088 g, 0.83 mmol, 83%) as a white solid.; R_f 0.62 (dichloromethane); ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.65 (d, *J* = 8.6 Hz, 2H), 7.53 (d, *J* = 8.7 Hz, 2H), 7.49 (br, 1H), 7.28-7.19 (m, 2H), 7.15-7.07 (m, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 137.7, 136.0, 132.3, 129.4, 128.7, 128.1, 125.6, 121.7; Data is consistent with that reported in the literature.[4]

[4] Gowda, B.T.; Shetty, M; Jayalakshmi, K. L. *A: Physical Science*, **2004**, 59, 239-249.



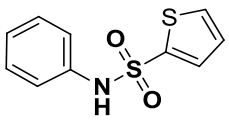
12e

N-(4-fluorophenyl)-4-methylbenzenesulfonamide (12e). The one-pot procedure was used employing *tert*-butyl tosylcarbamate (0.271 g, 1.00 mmol) and bis(4-fluorophenyl)iodonium triflate (0.606 g, 1.30 mmol). Flash chromatography on silica gel using hexane/ethyl acetate (5:1) provided pure **12e** (0.249 g, 0.94 mmol, 94%) as a white solid.; R_f 0.45 (hexane/ethyl acetate = 2:1); ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.60 (d, *J* = 8.3 Hz, 2H), 7.23 (d, *J* = 7.9 Hz, 2H), 7.06-6.98 (m, 2H), 6.97-6.88 (m, 2H), 6.51 (br, 1H), 2.39 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) 160.6 (d, *J* = 244 Hz), 144.0, 135.8, 132.2 (d, *J* = 1.5 Hz), 129.7, 127.3, 124.9 (d, *J* = 8.3 Hz), 116.1 (d, *J* = 22 Hz), 21.6; Data is consistent with that reported in the literature.[5]



12f

N-phenylmethanesulfonamide (12f). The one-pot procedure was used employing *tert*-butyl methylcarbamate (0.195 g, 1.00 mmol) and diphenyliodonium triflate (0.559 g, 1.30 mmol). Flash chromatography on silica gel using hexane/ethyl acetate (5:1) provided pure **12f** (0.156 g, 0.91 mmol, 91%) as a white solid. R_f 0.20 (dichloromethane); ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.39-7.33 (m, 2H), 7.26-7.16 (m, 3H), 6.96 (s, 1H), 3.02 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 136.7, 129.7, 125.4, 120.8, 39.2; Data is consistent with that reported in the literature. [3]

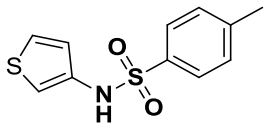


12g

N-phenylthiophene-2-sulfonamide (12g). The one-pot procedure was used employing *tert*-butyl thiophen-2-ylsulfonylcarbamate (0.263 g, 1.00 mmol) and diphenyliodonium triflate

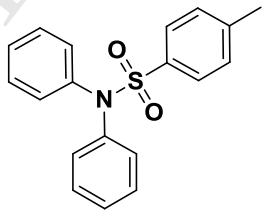
[5] Teo, Y. –C.; Yong, F. –F. *Synlett*, **2011**, 6, 837-843.

(0.559 g, 1.30 mmol). Flash chromatography on silica gel using hexane/ethyl acetate (5:1) provided pure **12g** (0.225 g, 0.94 mmol, 94%) as a colorless oil. R_f 0.43 (hexane/ethyl acetate = 2:1); IR (neat) 3241, 3091, 2825, 1599, 1498, 1404, 1335, 1229, 1153, 1092, 928, 589 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 7.53 (dd, *J* = 5.1 and 1.2 Hz, 1H), 7.49 (dd, *J* = 3.7 and 1.2 Hz, 1H), 7.33-7.25 (m, 2H), 7.20-7.10 (m, 3H), 7.03-6.97 (m, 1H), 6.77 (br, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) 139.3, 136.0, 132.9, 132.4, 129.4, 127.3, 125.9, 122.0; HRMS-FAB: *m/z* 261.9966 [$(M+Na)^+$; calcd for $C_{10}H_9NNaO_2S_2^+$: 261.9967].



12

4-methyl-N-(thiophen-3-yl)benzenesulfonamide (12h). The one-pot procedure was used employing *tert*-butyl tosylcarbamate (0.271 g, 1.00 mmol) and mesityl(thiophen-3-yl)iodonium triflate (0.622 g, 1.30 mmol). Flash chromatography on silica gel using hexane/ethyl acetate (5:1) provided pure **12h** (0.228 g, 0.90 mmol, 90%) as a white solid. R_f 0.45 (hexane/ethyl acetate = 2:1); ^1H NMR (300 MHz, $CDCl_3$) δ 7.67 (d, *J* = 8.0 Hz, 2H), 7.23 (d, *J* = 8.0 Hz, 2H), 7.20-7.13 (m, 1H), 7.04 (br, 1H), 6.90-6.81 (m, 2H), 2.39 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) 143.9, 135.9, 134.2, 129.6, 127.2, 125.6, 123.3, 113.9, 21.5; Data is consistent with that reported in the literature. [3]

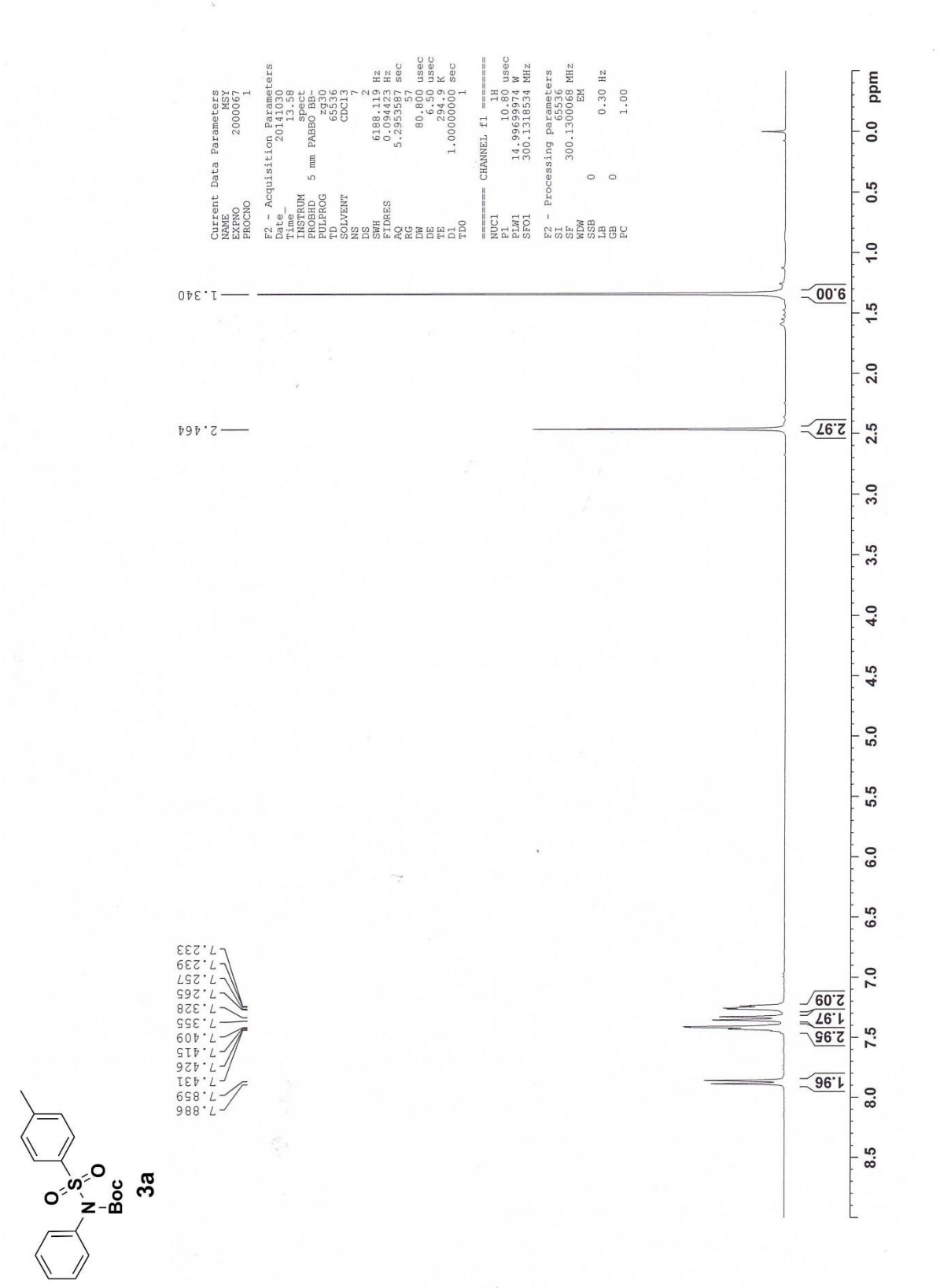


12aa

N,N-diphenyl-p-toluenesulfonamide (12aa). A 10 mL round bottom flask was charged with a diphenyliodonium triflate (0.140 g, 0.325 mmol), CuI (10 mol %), triethylamine (0.50 mmol) and the corresponding *p*-toluenesulfonamide (0.043 g, 0.25 mmol). Toluene (2 mL) was then added to the flask. The reaction mixture was stirred at 50 ^oC. After 24 h complete consumption of diphenyliodonium triflate was observed by TLC analysis. The solvent was removed *in vacuo* and the residue was purified by flash chromatography on silica gel using

hexane/ethyl acetate (15:1) provided pure **12aa** (0.041 g, 0.13 mmol, 51%) as a white solid. R_f 0.65 (hexane/ethyl acetate = 2:1); ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.58 (d, *J* = 8.1 Hz, 2H), 7.35-7.21 (m, 12H), 2.42 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 143.5, 141.5, 137.5, 129.5, 129.2, 128.3, 127.7, 127.3, 21.5; Data is consistent with that reported in the literature.[6] **12a** (0.012 g, 0.05 mmol, 19%) as a white solid. R_f 0.59 (hexane/ethyl acetate = 2:1); ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.67 (d, *J* = 8.4 Hz, 2H), 7.29-7.21 (m, 4H), 7.12-7.06 (m, 3H), 6.91 (br, 1H), 2.37 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 143.9, 136.5, 136.0, 129.6, 129.3, 127.2, 125.3, 121.5, 21.5; Data is consistent with that reported in the literature.[3]

6 Nandi, P.; Redko, M. Y.; Petersen, K.; Dye, J. L.; Lefenfeld, M.; Vogt, P. F.; Jackson, J. E. *Org. Lett.* **2008**, *10*, 5441-5444.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DE 6.50 usec
TE 294.0 K
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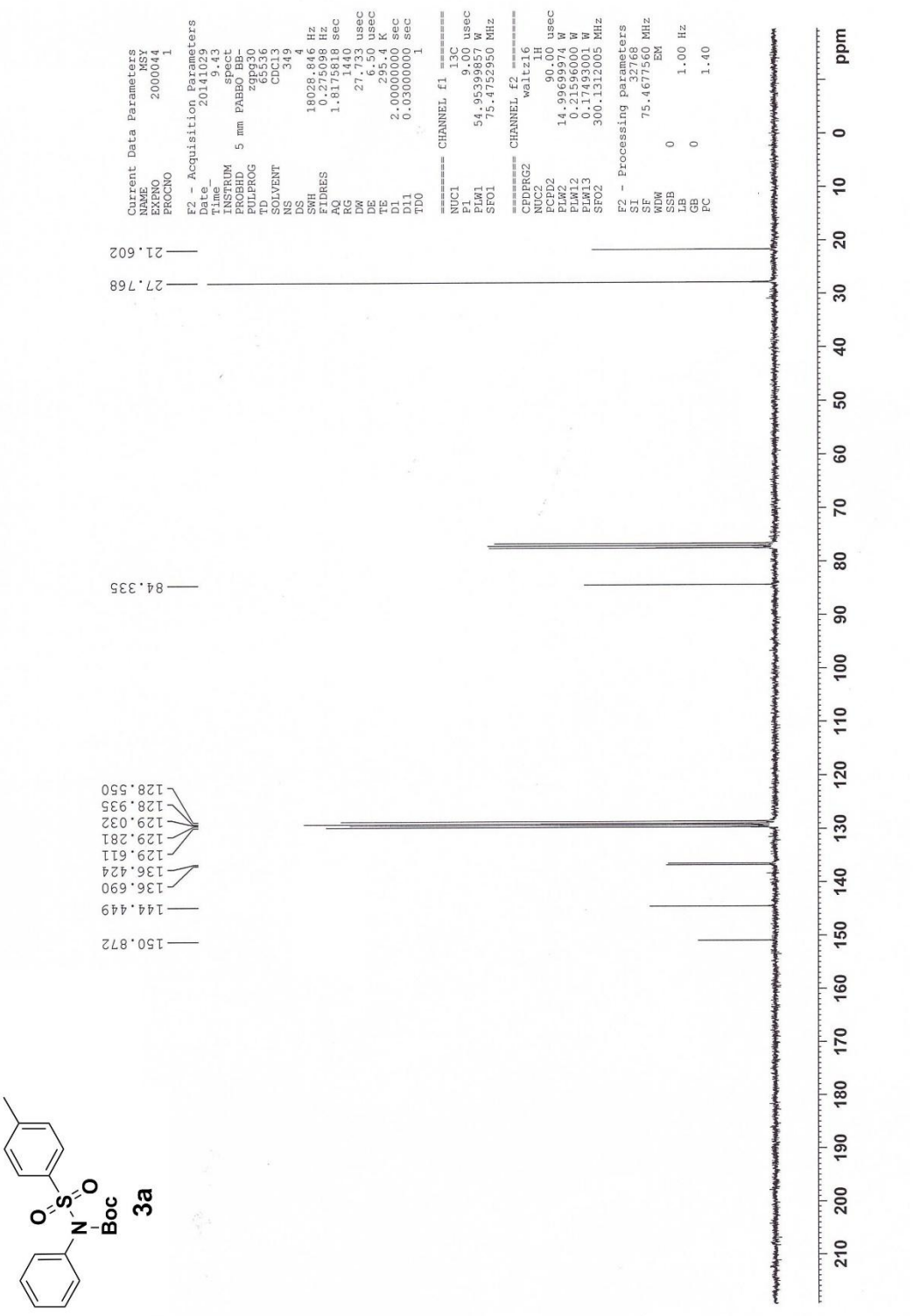
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NUC2 1H
PCPD2 90.00 usec
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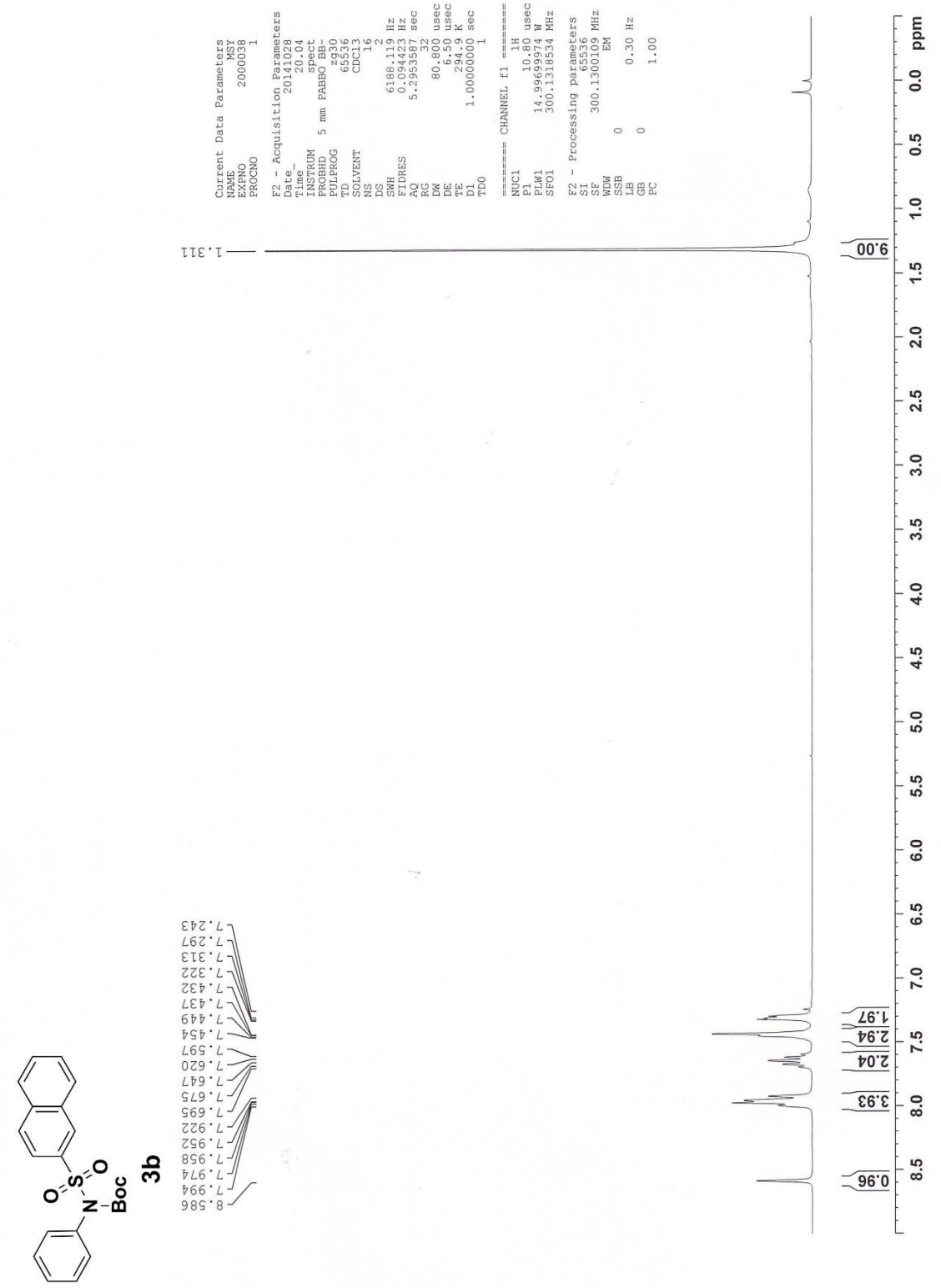
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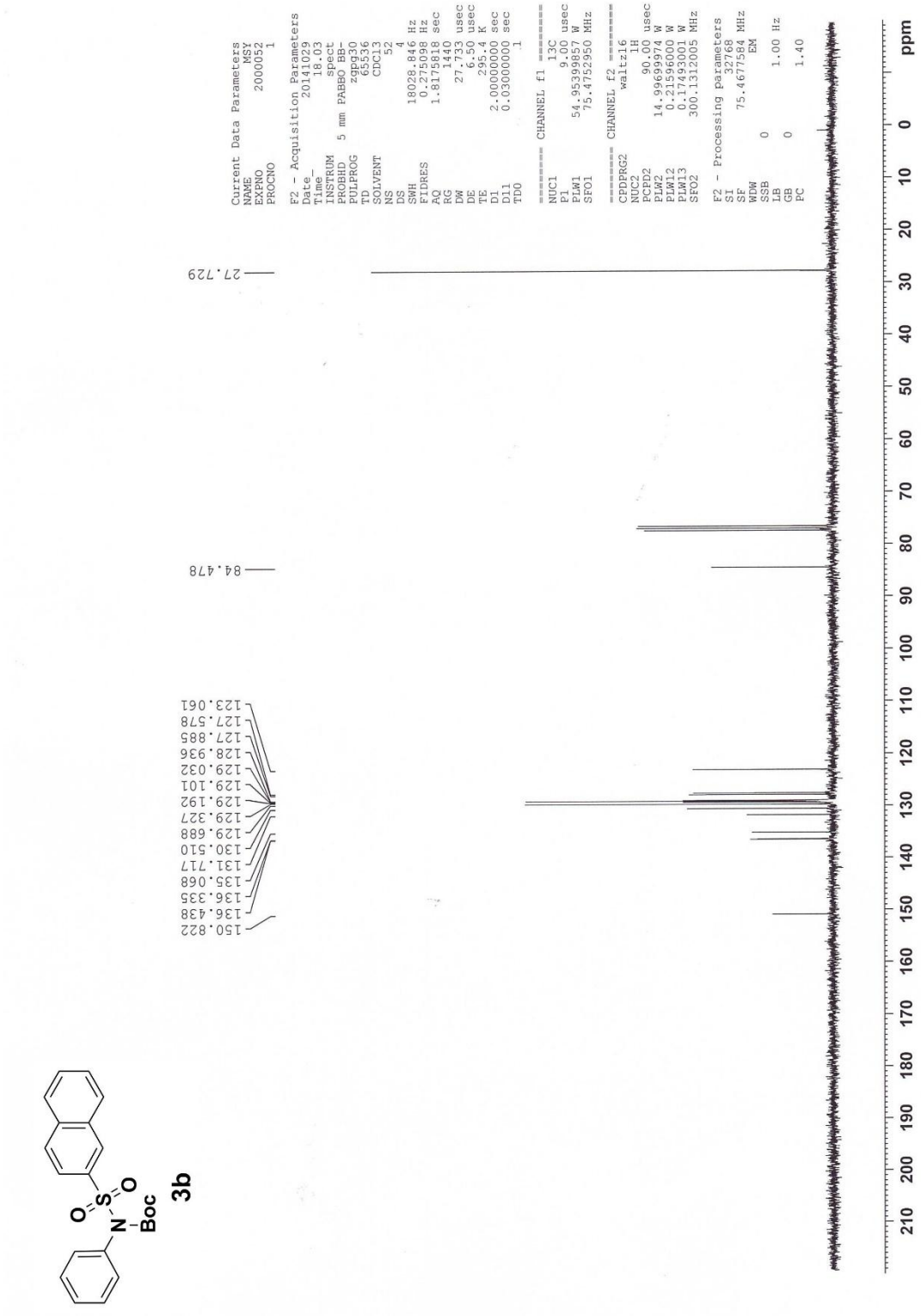
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PCPD2 90.00 usec
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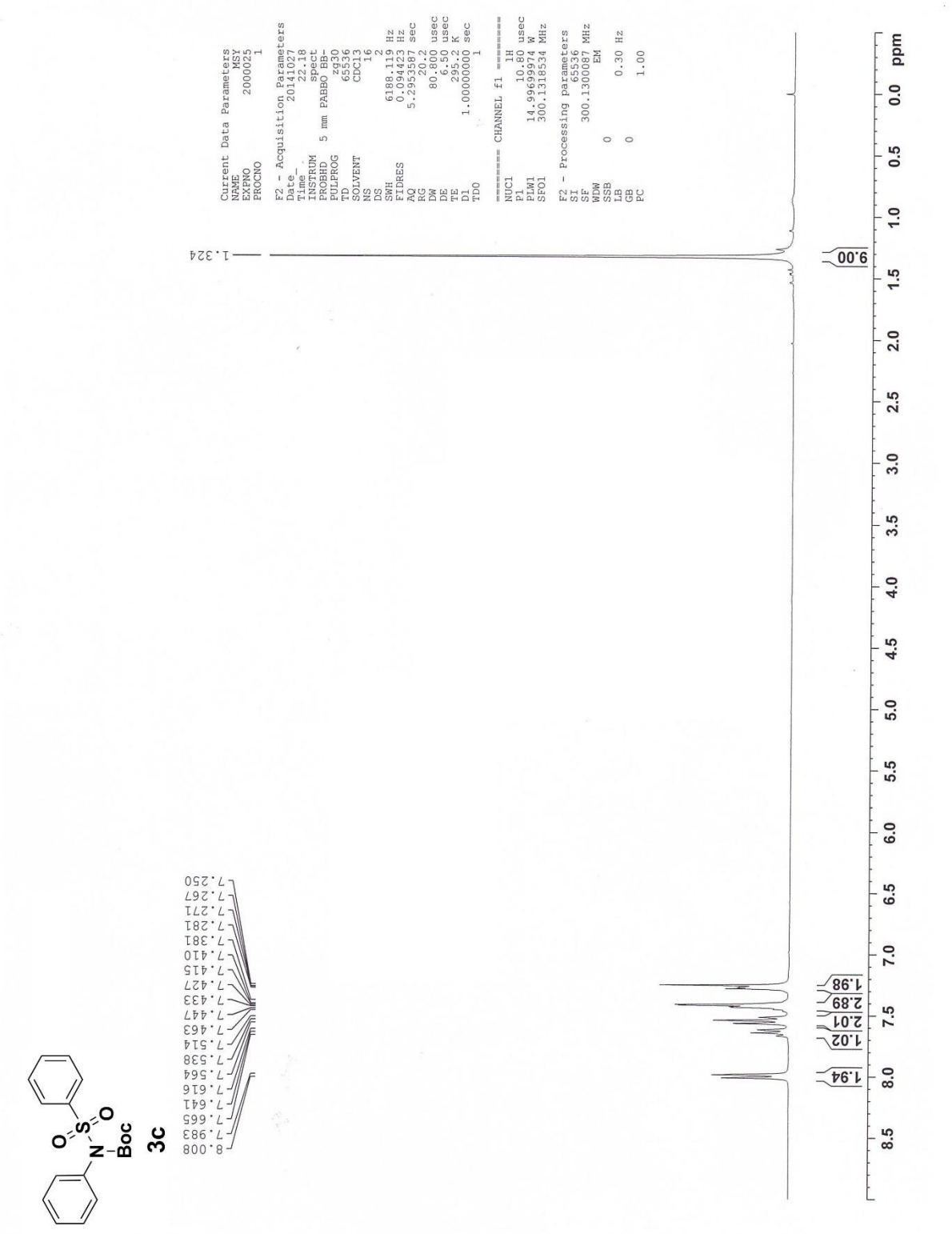
Boc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FIDRES 0.094423 Hz
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TE 295.2 K
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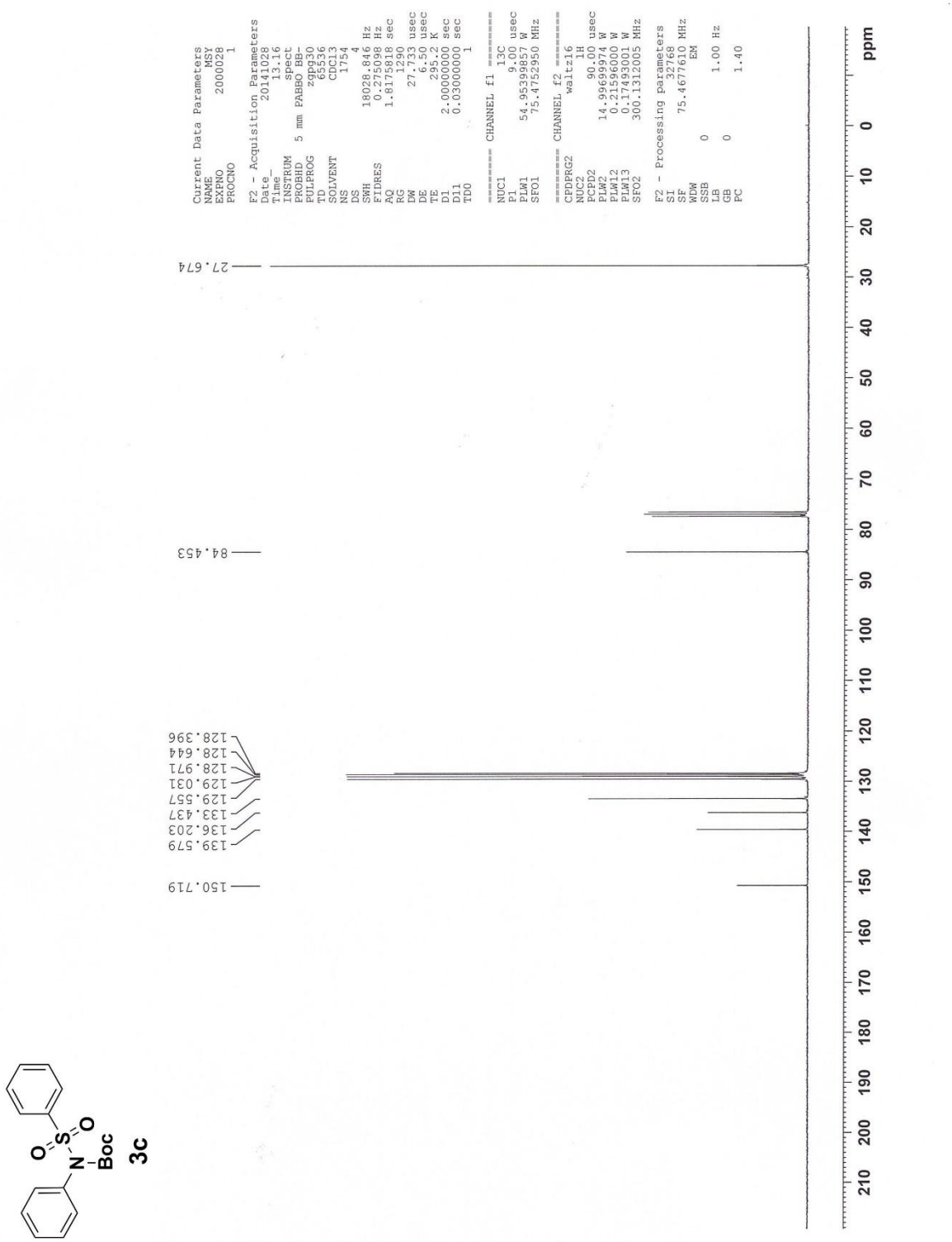
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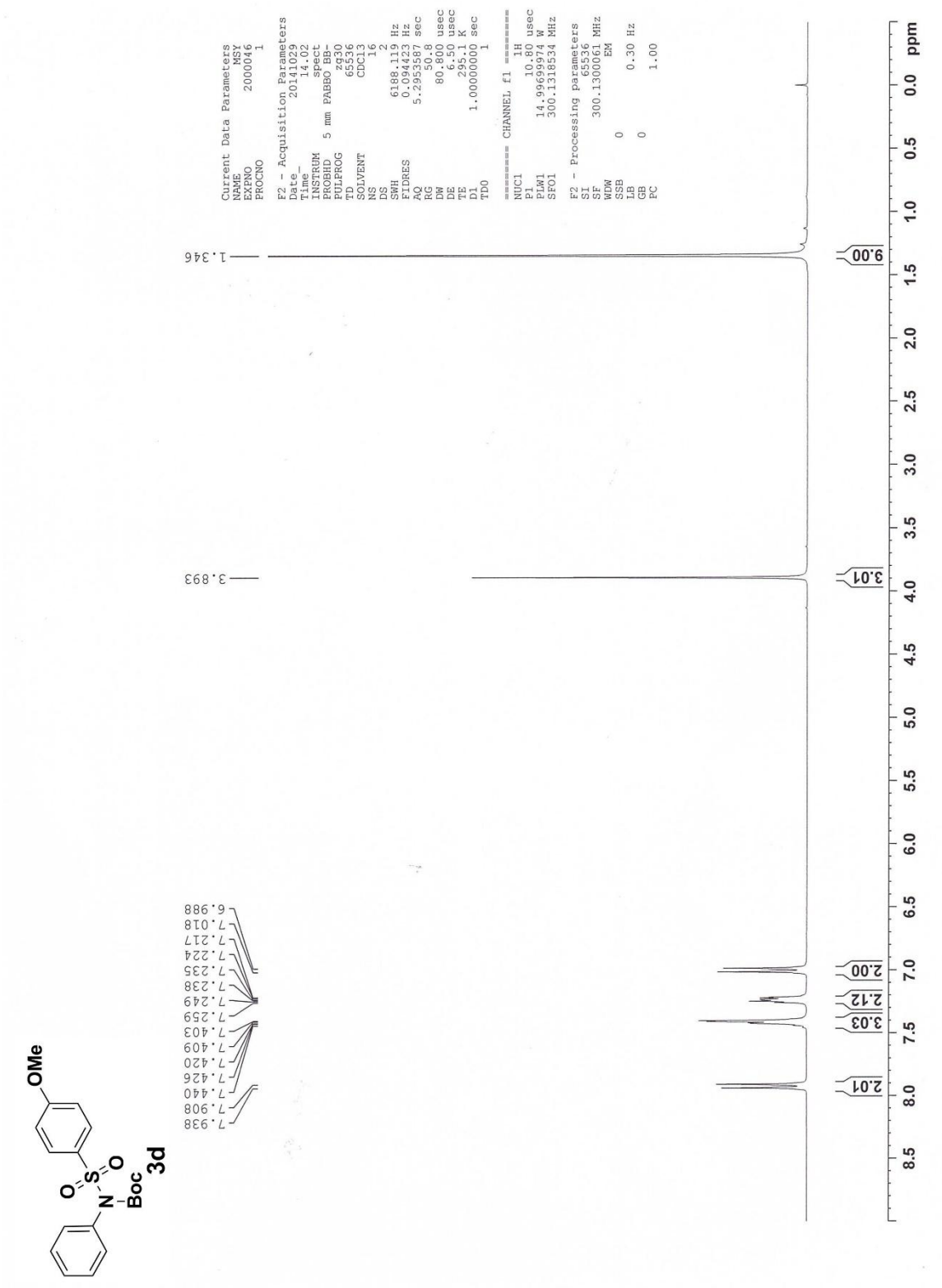
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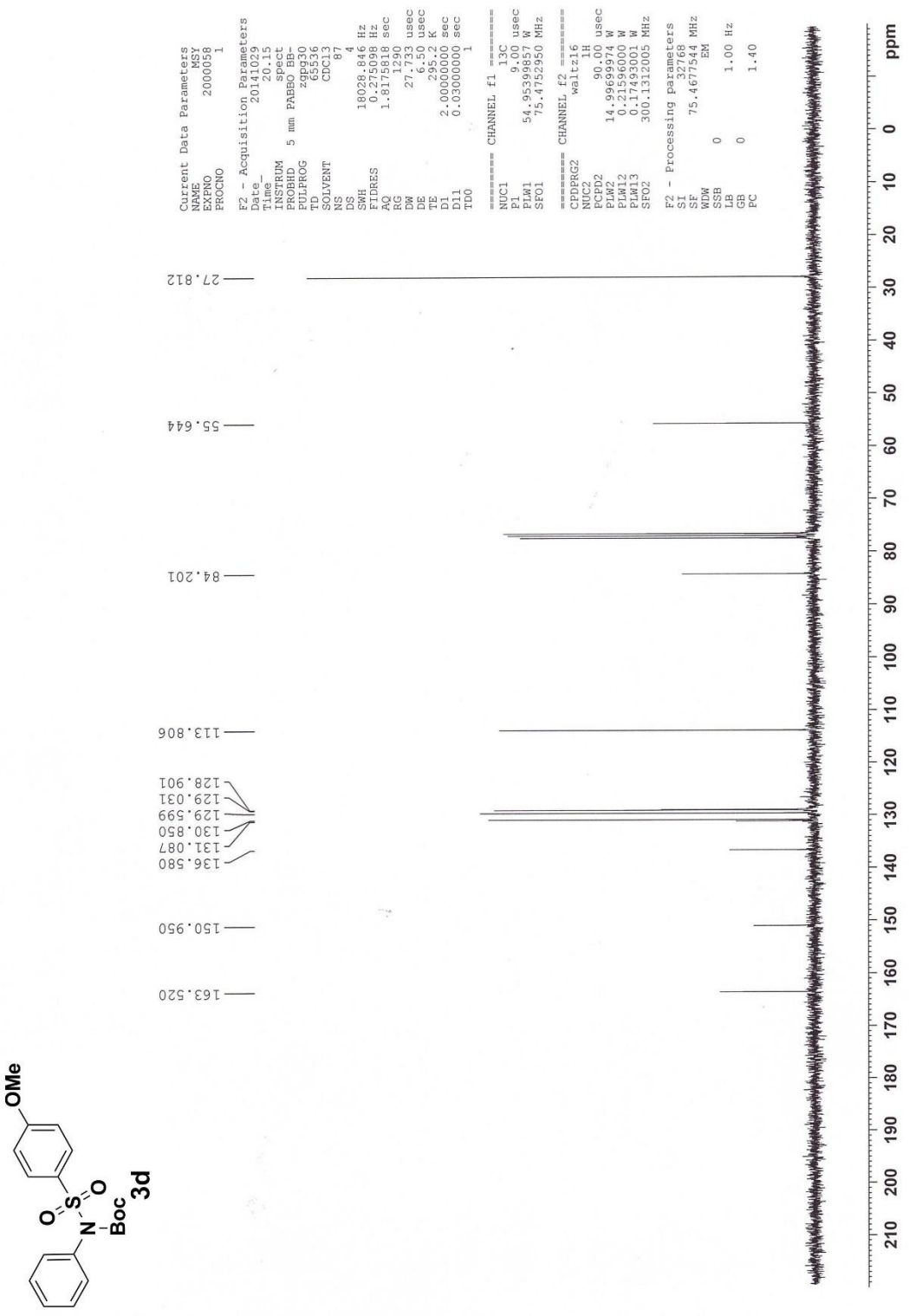
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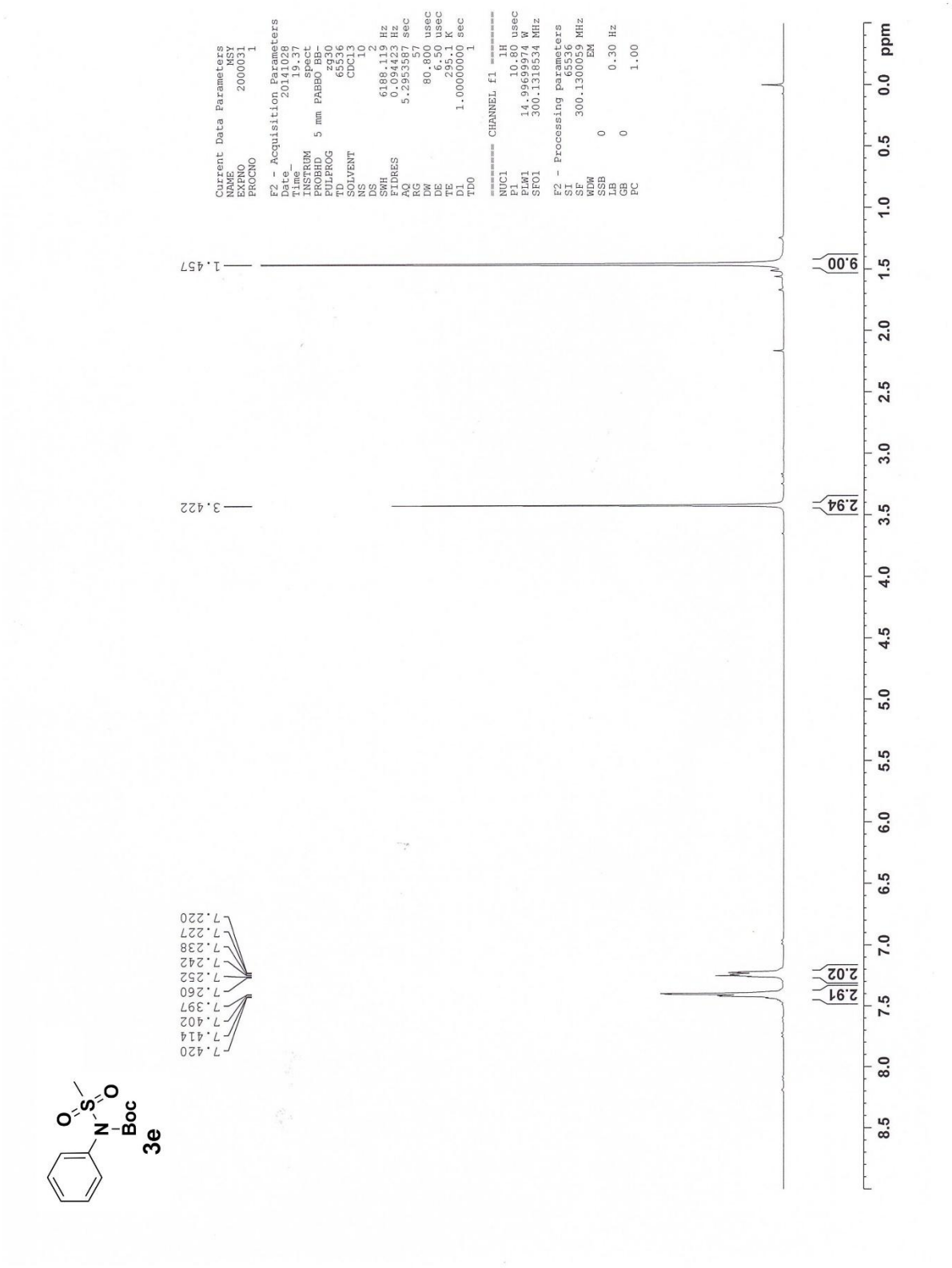
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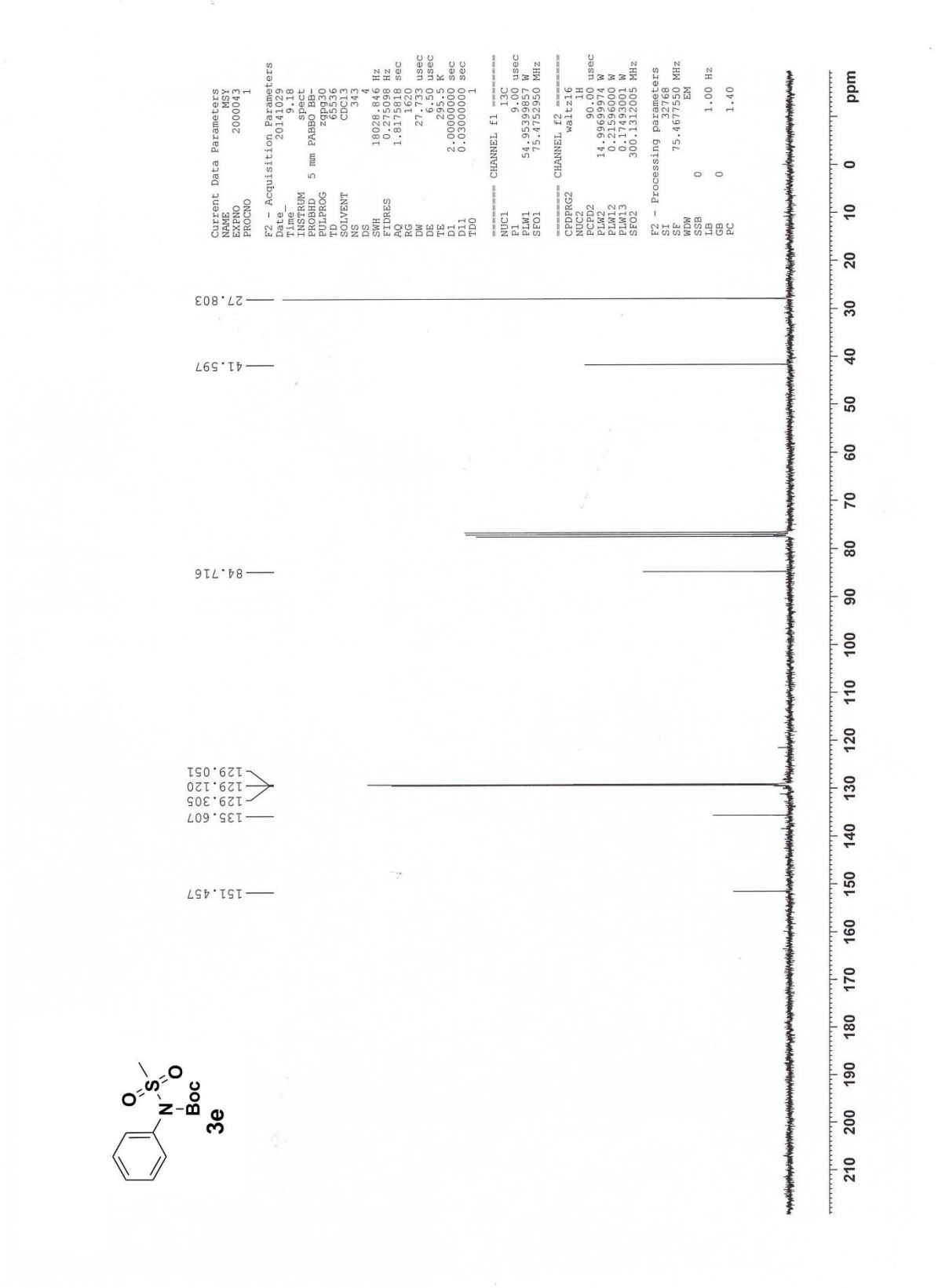
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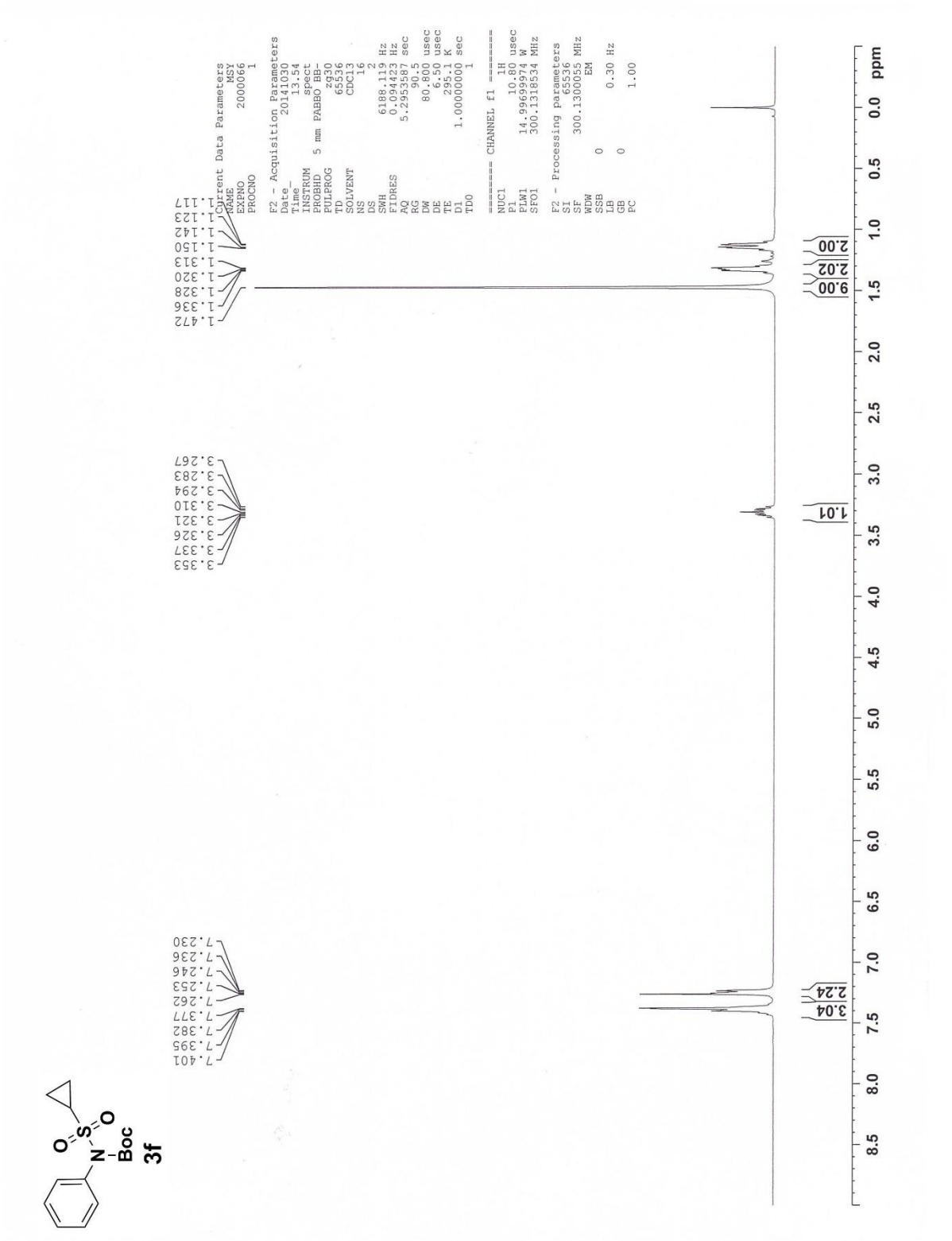
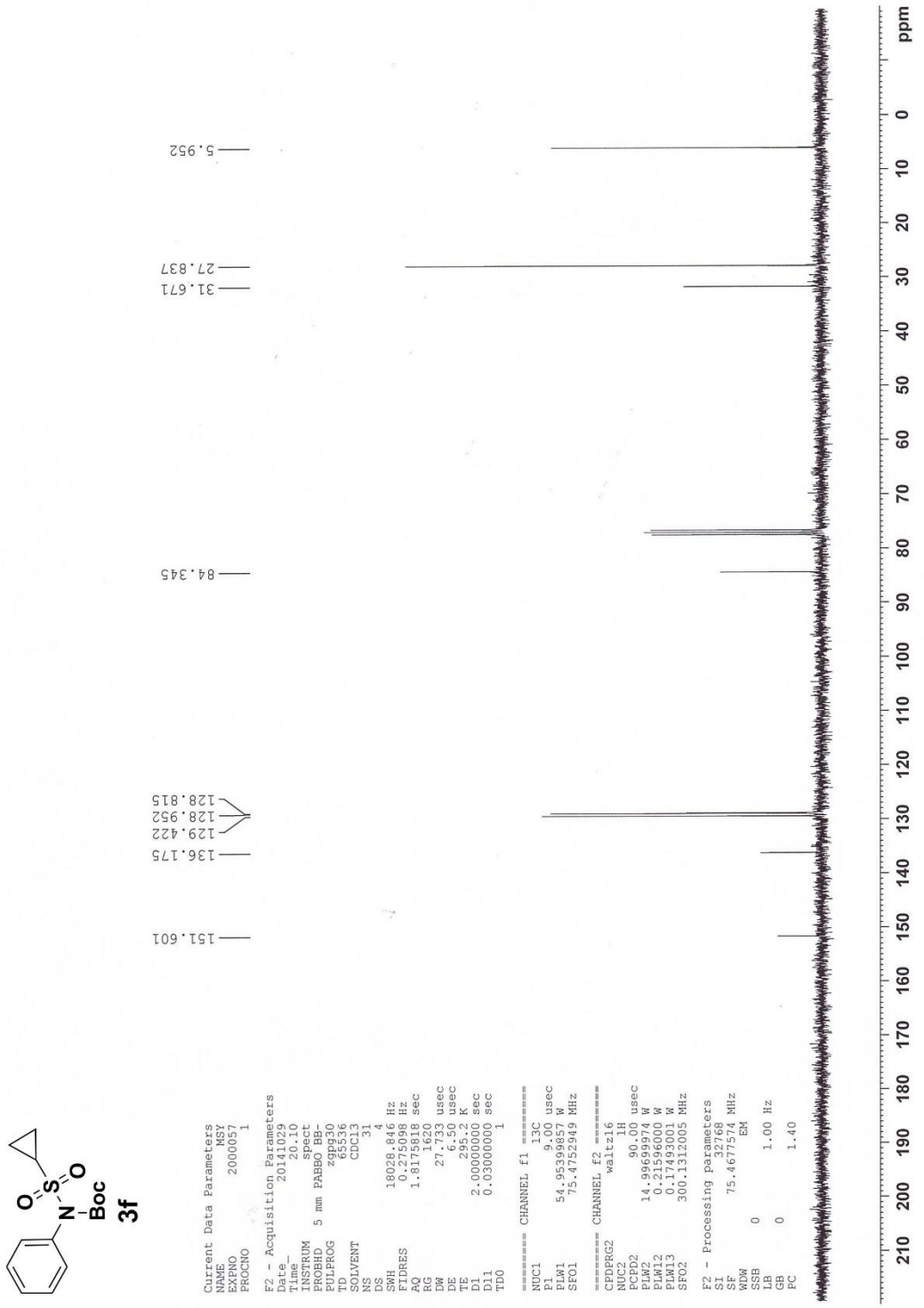
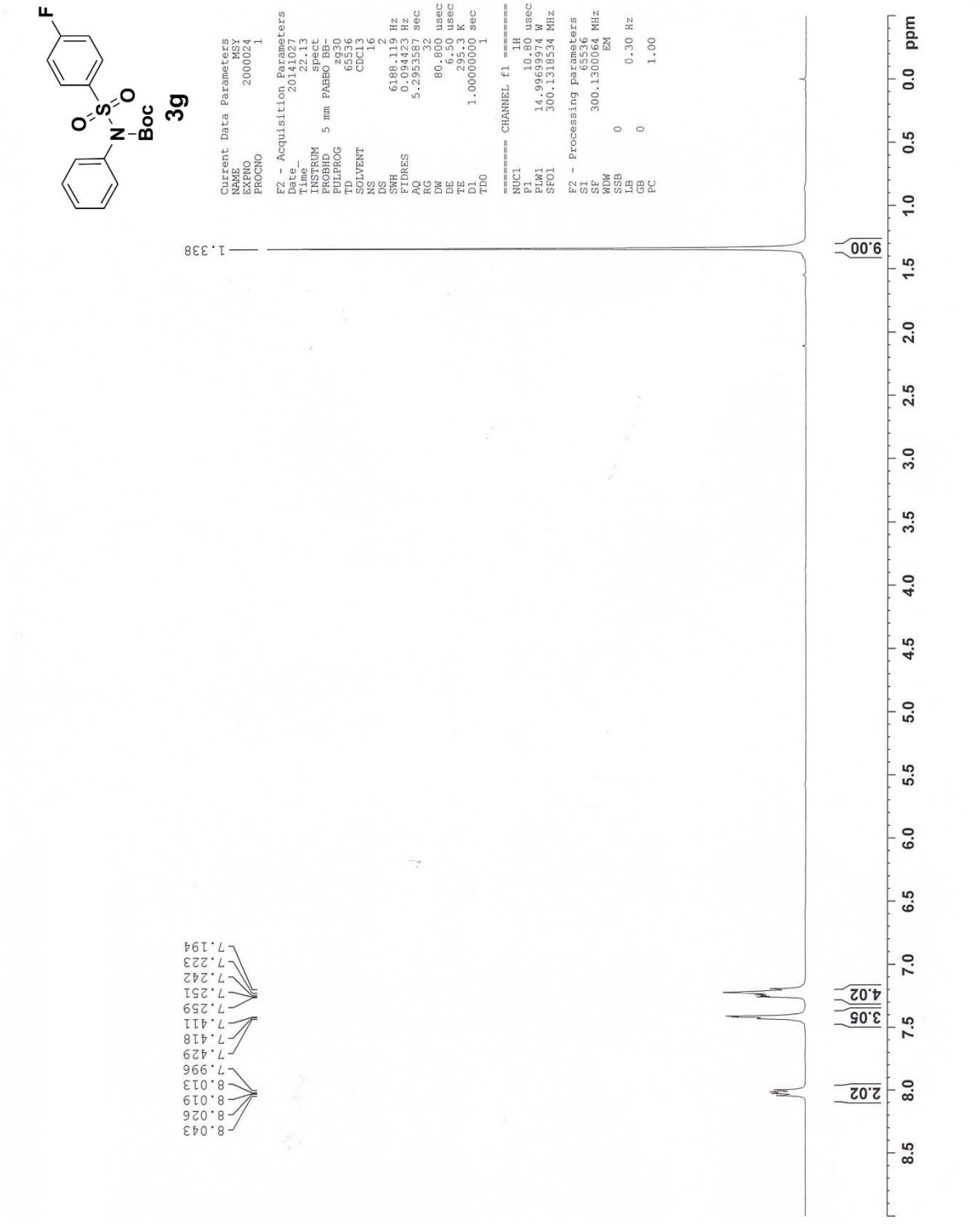
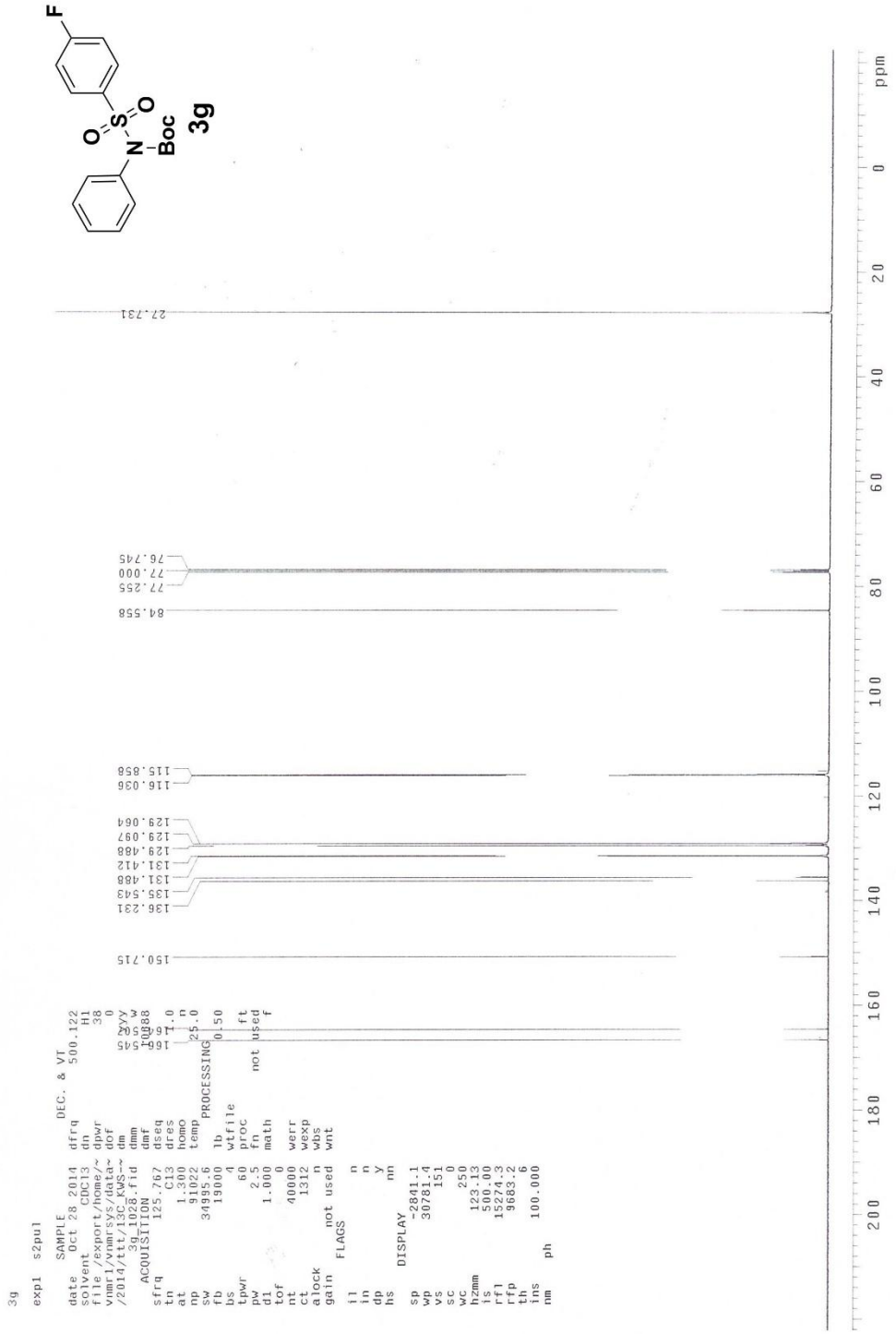
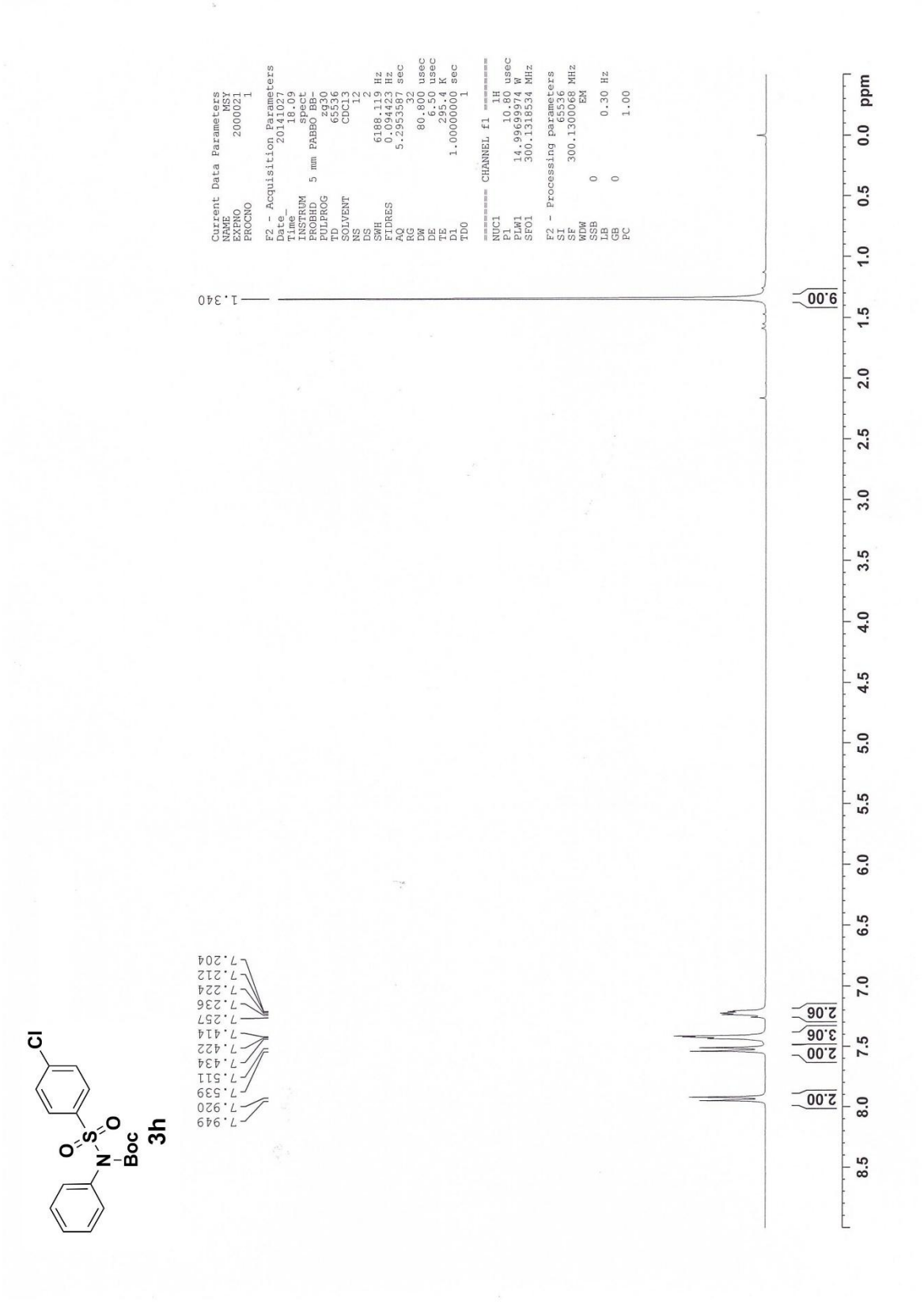
NUC2 1H
PCPD2 90.00 usec
PLW2 14.99699974 W
PLW12 0.21596000 W
PLW13 0.17493001 W
SFO2 300.1312005 MHz
F2 - Processing parameters
SI 32768
SF 75.4677550 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
27.803
41.597
84.716
129.051
129.120
129.305
135.607
151.457
ppm
3e

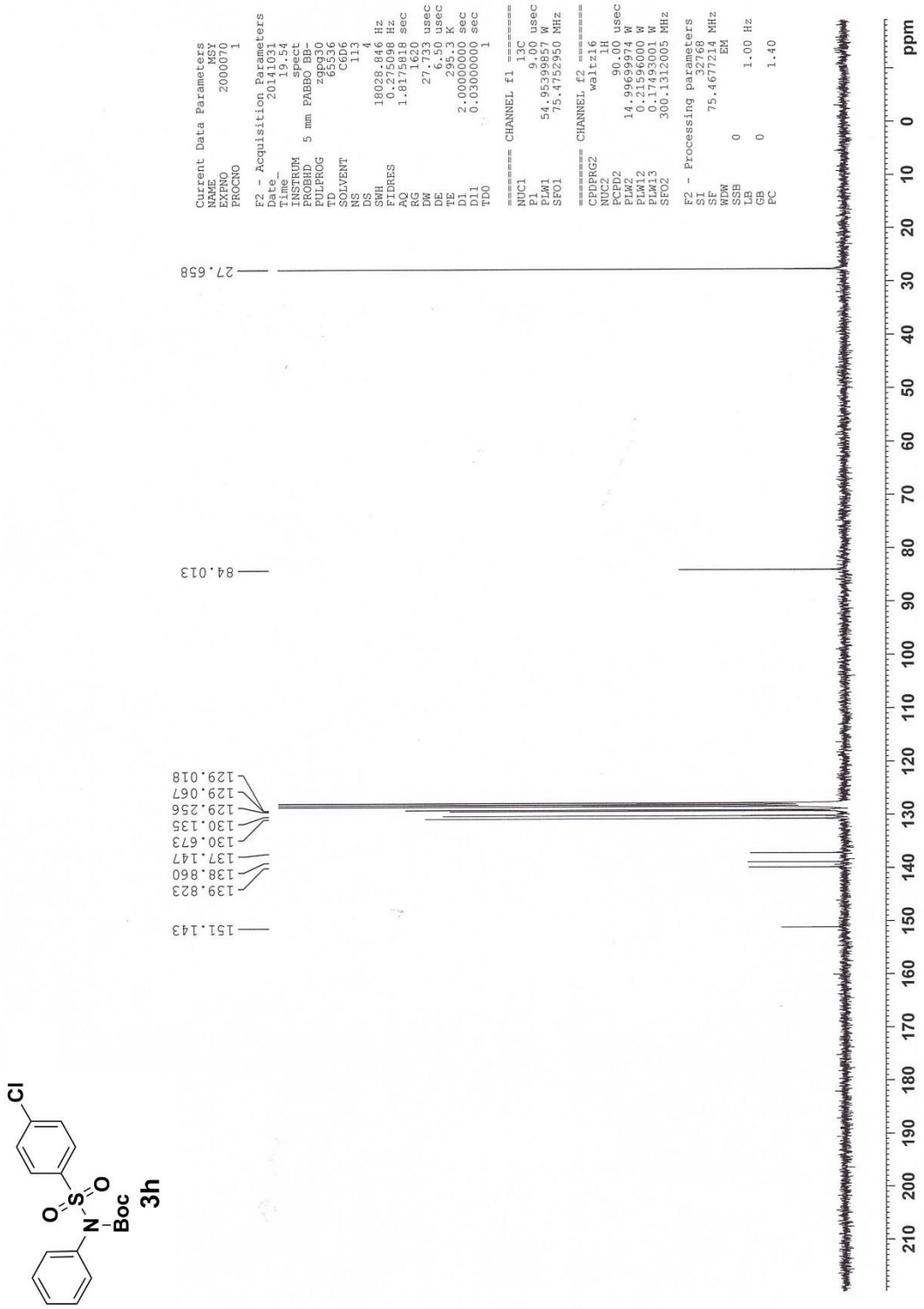

3f
7.401
7.395
7.382
7.377
7.262
7.253
7.246
7.236
7.230
3.353
3.337
3.326
3.321
3.310
3.294
3.283
3.267
1.472
1.336
1.328
1.320
1.313
1.150
1.142
1.123
1.117
3.04
2.24
1.01
9.00
2.02
2.00
Current Data Parameters
NAME MSY
EXPNO 2000066
PROCNO 1
F2 - Acquisition Parameters
Date 20141030
Time 13.54
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 6188.119 Hz
FIDRES 0.094423 Hz
AQ 5.2953587 sec
RG 90.5
DW 80.800 usec
DE 6.50 usec
TE 295.1 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 10.80 usec
PLW1 14.99699974 W
SFO1 300.1318534 MHz
F2 - Processing parameters
SI 65536
SF 300.1300055 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm

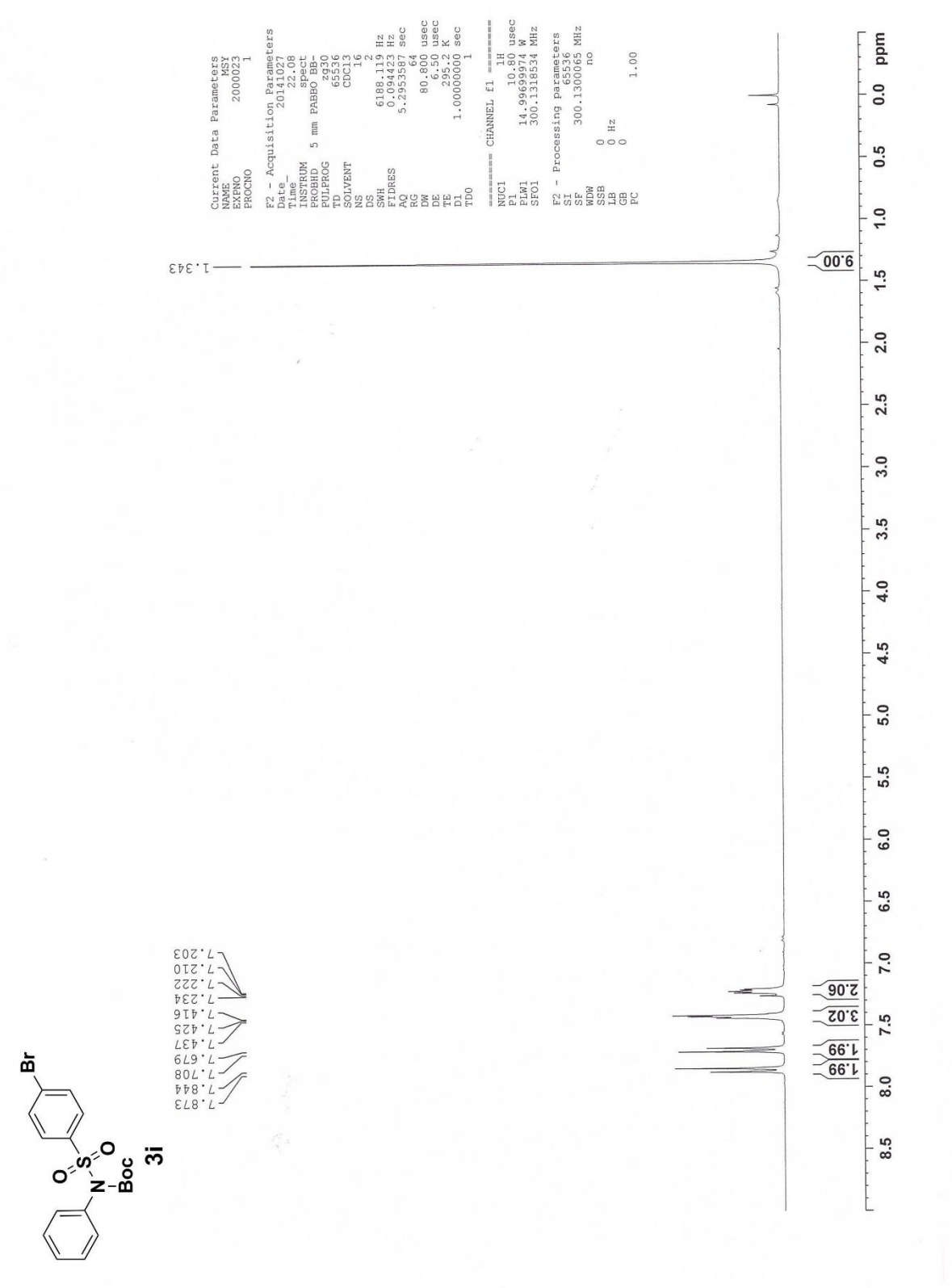

3f
Boc
Current Data Parameters
NAME MSY
EXPNO 2000057
PROCNO 1
F2 - Acquisition Parameters
Date_ 20141029
Time 20.10
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 31
DS 4
SWH 18028.846 Hz
FIDRES 0.275098 Hz
AQ 1.8175818 sec
RG 1620
DW 27.733 usec
DE 6.50 usec
TE 295.2 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.00 usec
PLW1 54.95399857 W
SFO1 75.4752949 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PLW2 14.99699974 W
PLW12 0.21596000 W
PLW13 0.17493001 W
SFO2 300.1312005 MHz
F2 - Processing parameters
SI 32768
SF 75.4677574 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
151.601
136.175
129.422
128.952
128.815
84.345
31.671
27.837
5.952
ppm

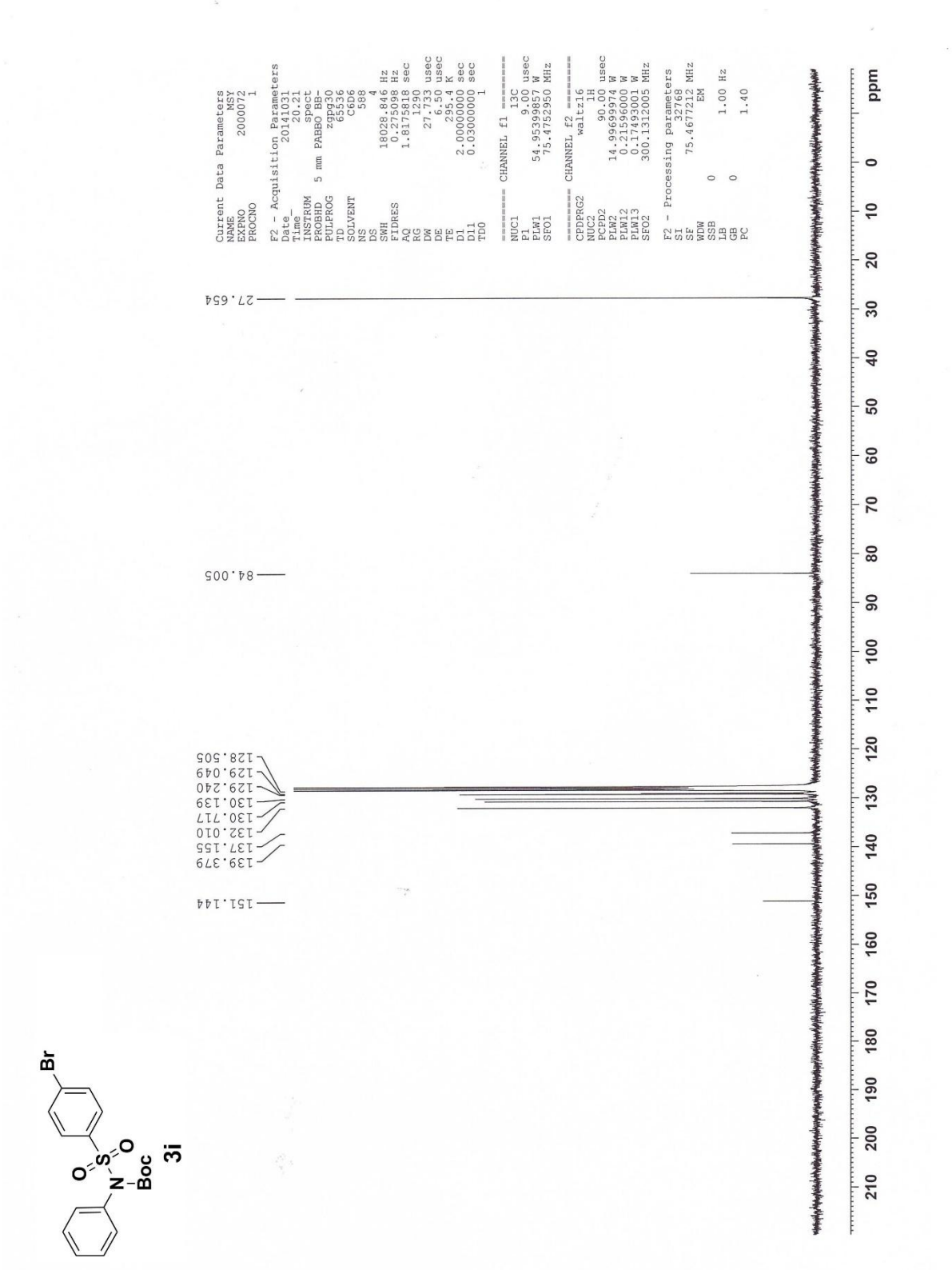

F
O
S
O
N
Boc
3g
Current Data Parameters
NAME MSY
EXPNO 2000024
PROCNO 1
F2 - Acquisition Parameters
Date_ 20141027
Time 22.13
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 6188.119 Hz
FIDRES 0.094423 Hz
AQ 5.2953587 sec
RG 32
DW 80.900 usec
DE 6.50 usec
TE 295.3 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 10.80 usec
PLW1 14.99699974 W
SFO1 300.1318534 MHz
F2 - Processing parameters
SI 65536
SF 300.1300064 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
1.338
7.194
7.223
7.242
7.251
7.259
7.411
7.418
7.429
7.996
8.013
8.019
8.026
8.043
9.00
4.02
3.06
2.02
ppm

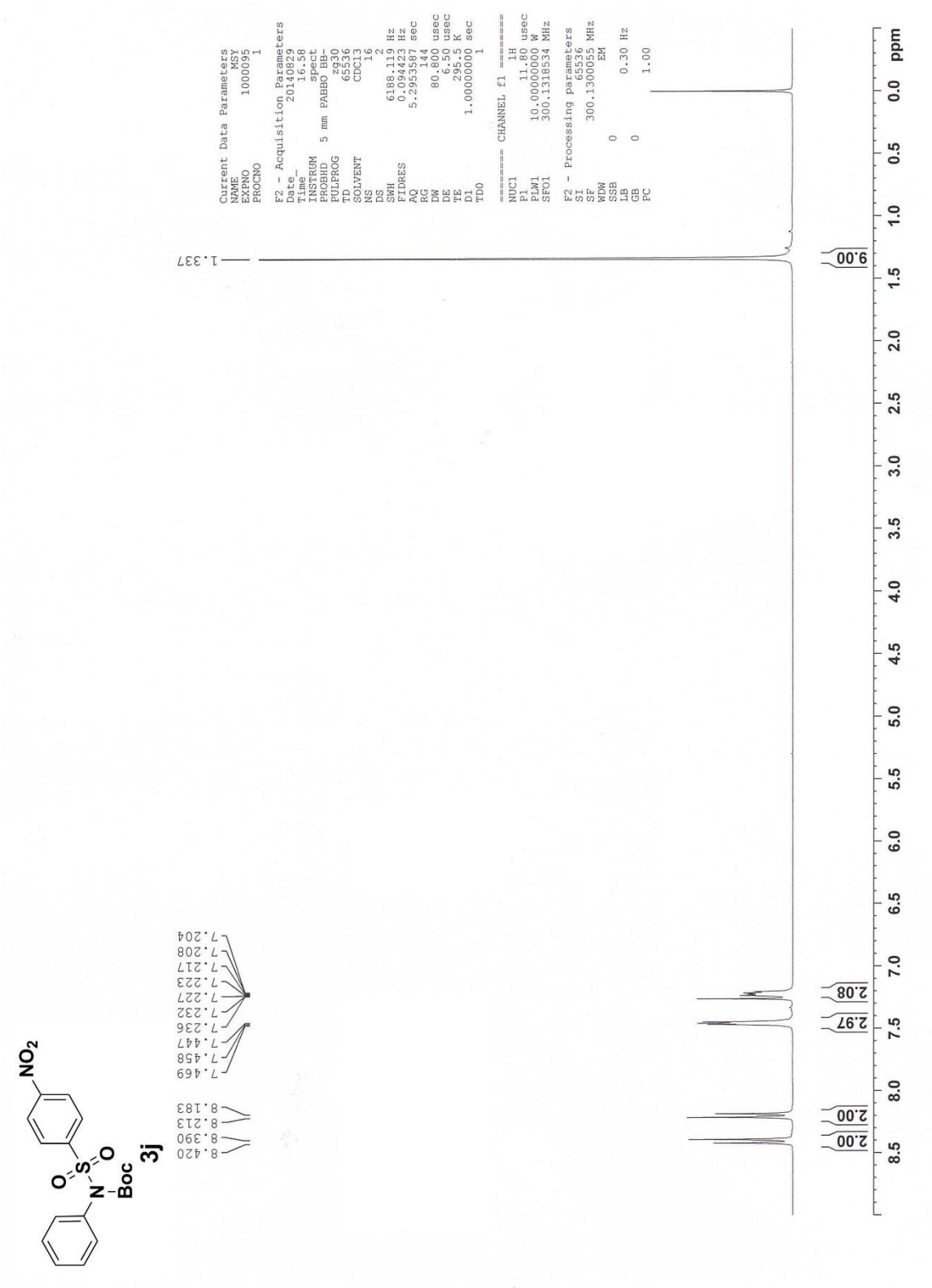

F
O
S
O
N
Boc
3g
27.731
76.745
77.000
77.255
84.558
115.858
116.036
129.064
129.097
129.488
131.412
131.488
135.543
136.231
150.715
200
180
160
140
120
100
80
60
40
20
0
ppm

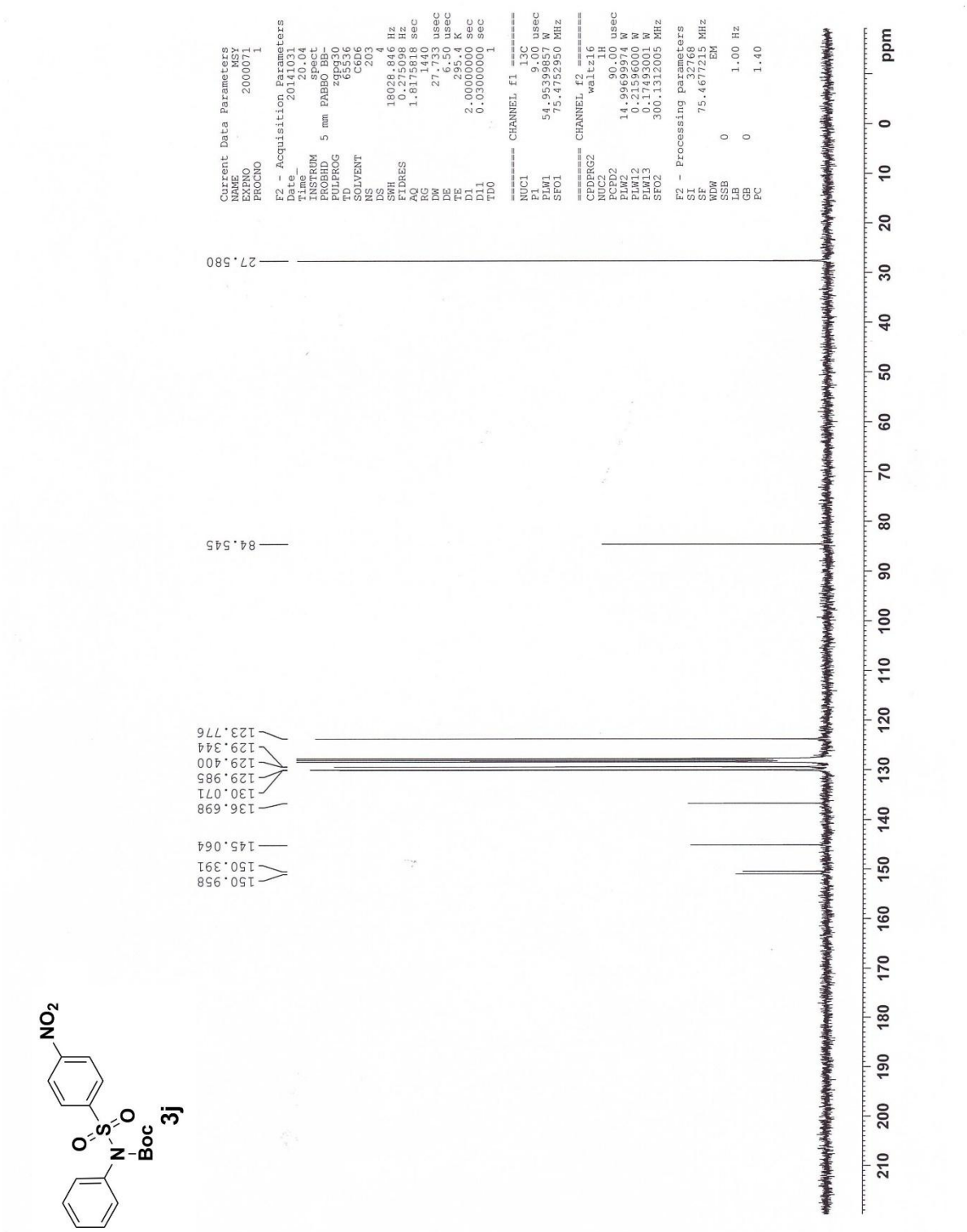

Cl
O
S
O
N
Boc
3h
7.204
7.212
7.224
7.236
7.257
7.414
7.422
7.434
7.511
7.539
7.920
7.949
1.340
9.00
2.06
3.06
2.00
2.00
Current Data Parameters
NAME MSY
EXPNO 2000021
PROCNO 1
F2 - Acquisition Parameters
Date_ 20141027
Time 18.09
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 12
DS 2
SWH 6188.119 Hz
FIDRES 0.094423 Hz
AQ 5.2953587 sec
RG 32
DW 80.900 usec
DE 6.50 usec
TE 295.4 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 10.80 usec
PLW1 14.99699974 W
SFO1 300.1318534 MHz
F2 - Processing parameters
SI 65536
SF 300.1300068 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm

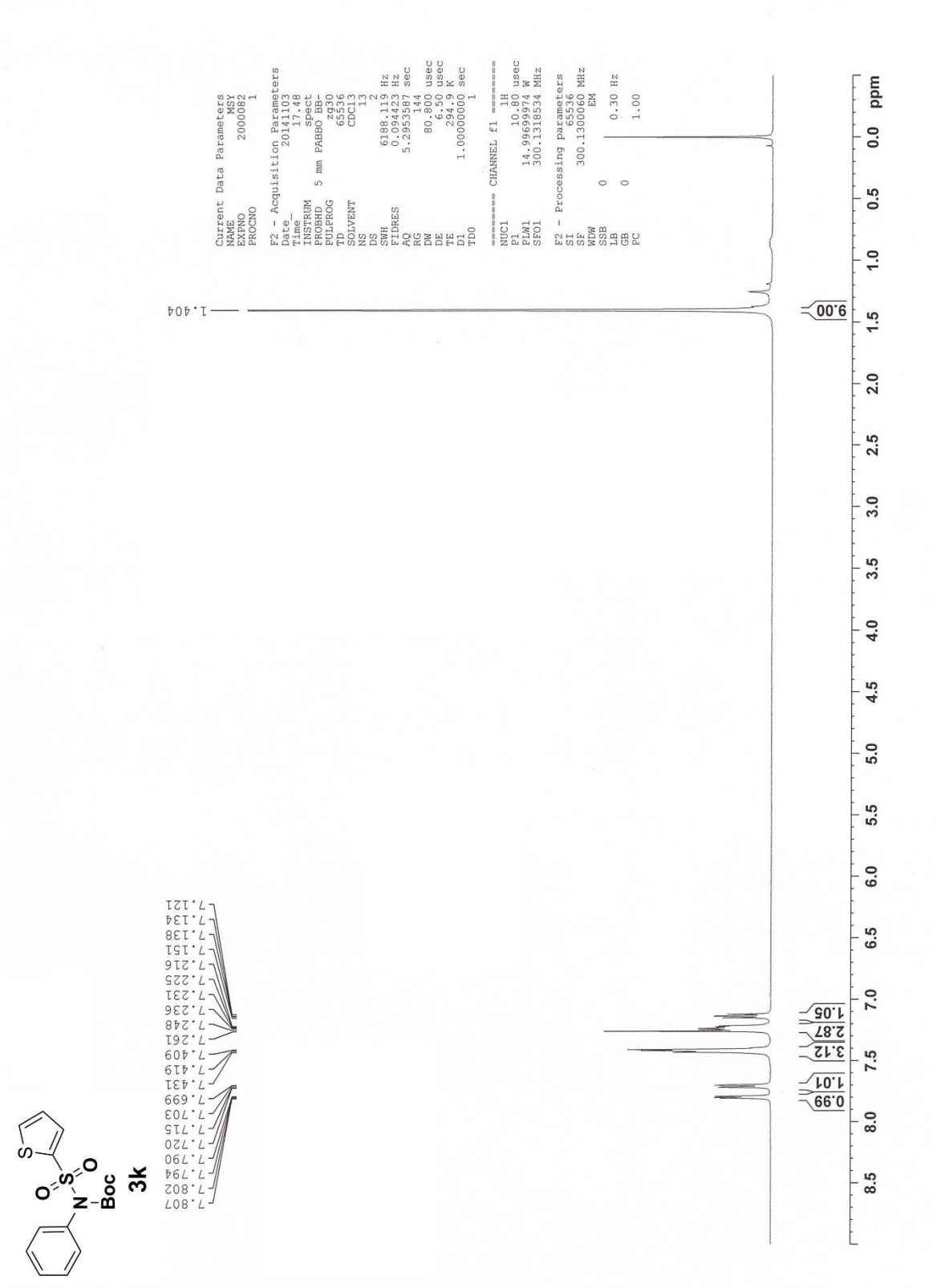

Current Data Parameters
NAME MSY
EXPNO 2000070
PROCNO 1
F2 - Acquisition Parameters
Date_ 20141031
Time_ 19.54
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT C6D6
NS 113
DS 4
SWH 18028.846 Hz
FIDRES 0.275098 Hz
AQ 1.81758818 sec
RG 1620
DW 27.733 usec
DE 6.50 usec
TE 295.3 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.00 usec
PLW1 54.95399857 W
SFO1 75.4752950 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PLW2 14.99699974 W
PLW12 0.21596000 W
PLW13 0.17493001 W
SFO2 300.1312005 MHz
F2 - Processing parameters
SI 32768
SF 75.4677214 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
27.658
84.013
129.018
129.067
129.256
130.135
130.673
137.147
138.860
139.823
151.143
ppm
Cl
O
S
O
N
Boc
3h

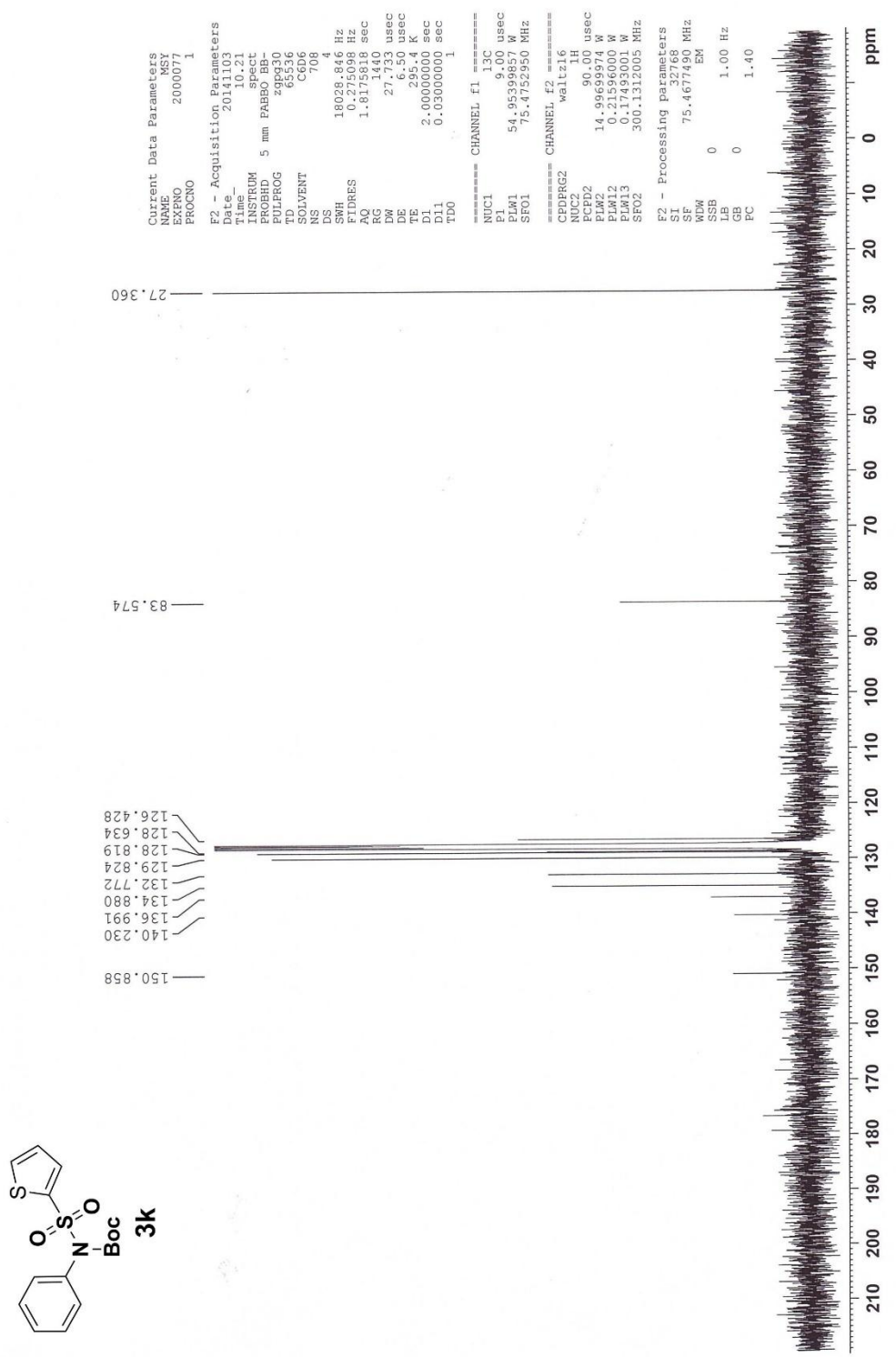

Br
O
S
O
N
Boc
3i
7.873
7.844
7.708
7.679
7.437
7.425
7.416
7.234
7.222
7.210
7.203
1.343
9.00
2.06
3.02
1.99
1.99
Current Data Parameters
NAME MSY
EXPNO 2000023
PROCNO 1
F2 - Acquisition Parameters
Date_ 20141027
Time 22.08
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 6188.119 Hz
FIDRES 0.094423 Hz
AQ 5.2953587 sec
RG 64
DW 80.800 usec
DE 6.50 usec
TE 295.2 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 10.80 usec
PLW1 14.99699974 W
SFO1 300.1318534 MHz
F2 - Processing parameters
SI 65536
SF 300.1300065 MHz
WDW no
SSB 0
LB 0 Hz
GB 0
PC 1.00
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm

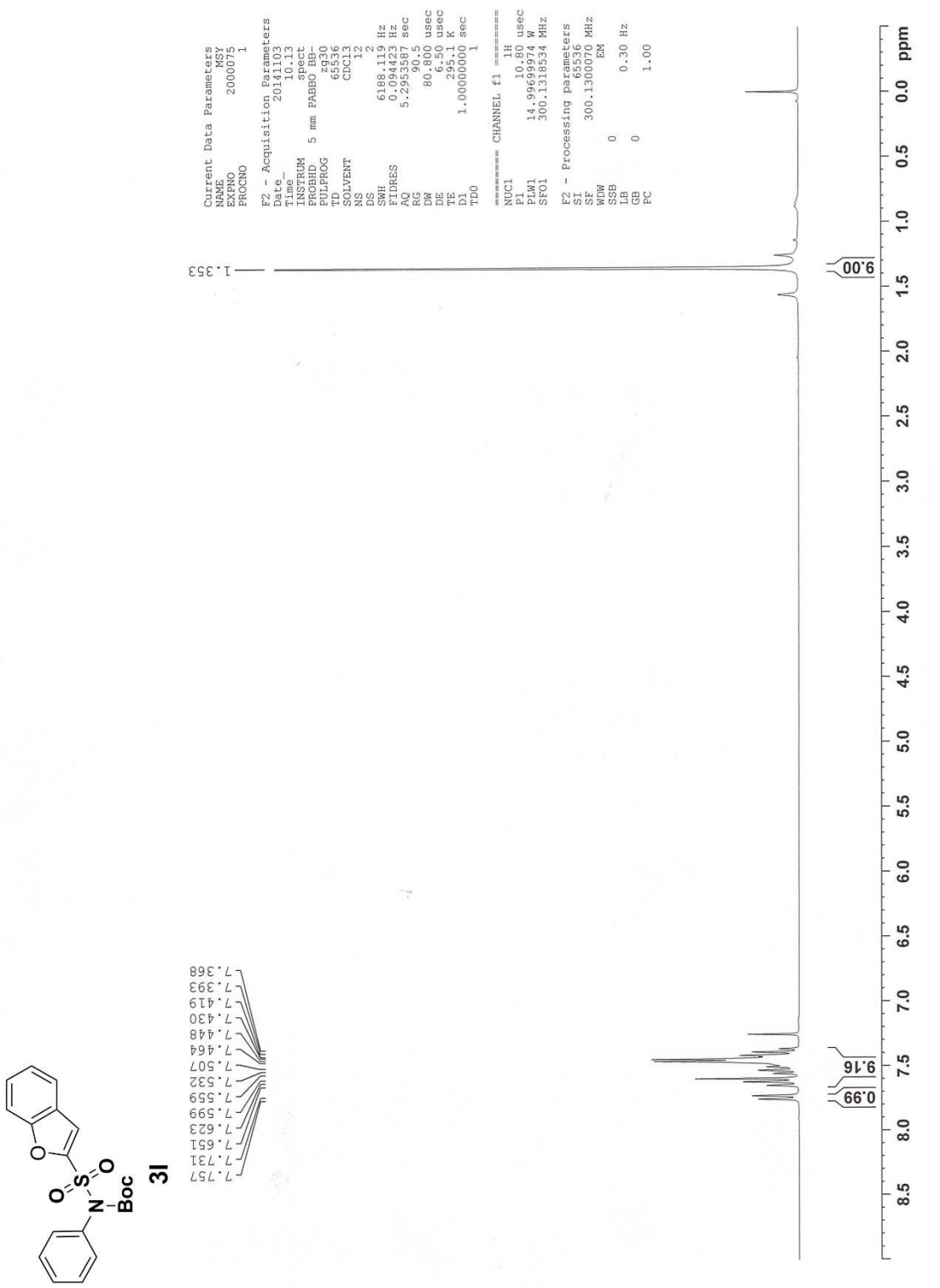

Br
O
S
O
N
Boc
3i
Current Data Parameters
NAME MSY
EXPNO 2000072
PROCNO 1
F2 - Acquisition Parameters
Date_ 20141031
Time 20.21
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT C6D6
NS 588
DS 4
SWH 18028.846 Hz
FIDRES 0.275098 Hz
AQ 1.8175818 sec
RG 1290
DW 27.733 usec
DE 6.50 usec
TE 295.4 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.00 usec
PLW1 54.95399857 W
SFO1 75.4752950 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PLW2 14.99699974 W
PLW12 0.21596000 W
PLW13 0.17493001 W
SFO2 300.1312005 MHz
F2 - Processing parameters
SI 32768
SF 75.4677212 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
27.654
84.005
128.505
129.049
129.240
130.139
130.717
132.010
137.155
139.379
151.144
ppm

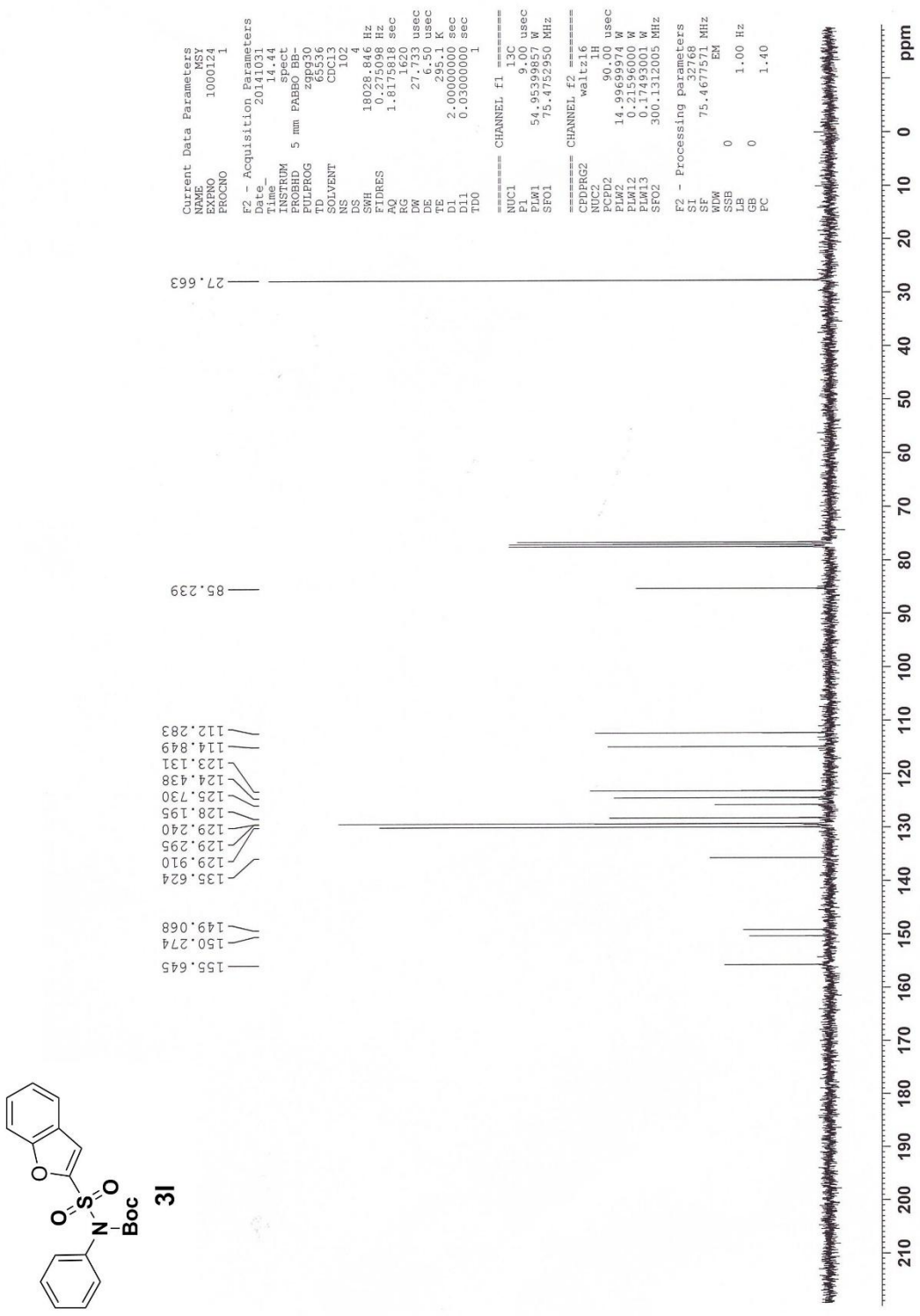

NO2
O
S
O
N
Boc
3j
8.420
8.390
8.213
8.183
7.469
7.458
7.447
7.236
7.232
7.227
7.223
7.217
7.208
7.204
1.337
9.00
2.08
2.97
2.00
2.00
Current Data Parameters
NAME MSY
EXPNO 1000095
PROCNO 1
F2 - Acquisition Parameters
Date_ 20140829
Time 16.58
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 6188.119 Hz
FIDRES 0.094423 Hz
AQ 5.2953587 sec
RG 144
DW 80.800 usec
DE 6.50 usec
TE 295.5 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 11.80 usec
PLW1 10.00000000 W
SFO1 300.1318534 MHz
F2 - Processing parameters
SI 65536
SF 300.1300055 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
ppm

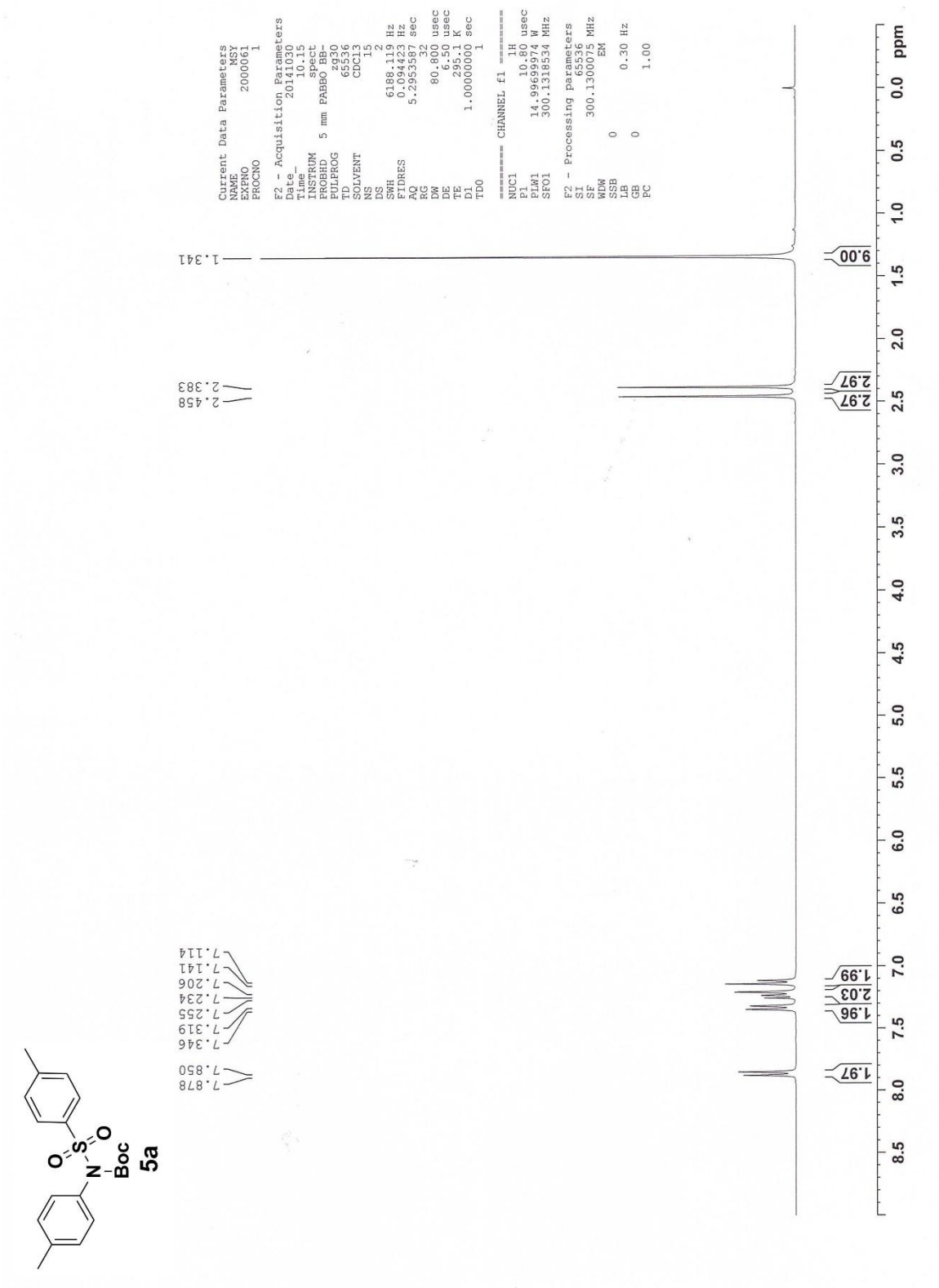

Current Data Parameters
NAME MSY
EXPNO 2000071
PROCNO 1
F2 - Acquisition Parameters
Date_ 20141031
Time_ 20.04
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT C6D6
NS 203
DS 4
SWH 18028.846 Hz
FIDRES 0.275098 Hz
AQ 1.8175818 sec
RG 1440
DW 27.733 usec
DE 6.50 usec
TE 295.4 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.00 usec
PLW1 54.95399857 W
SFO1 75.4752950 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PLW2 14.99699974 W
PLW12 0.21596000 W
PLW13 0.17493001 W
SFO2 300.1312005 MHz
F2 - Processing parameters
SI 32768
SF 75.4677215 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
27.580
84.545
123.776
129.344
129.400
129.985
130.071
136.698
145.064
150.391
150.958
ppm
NO2
O
S
O
N
Boc
3j

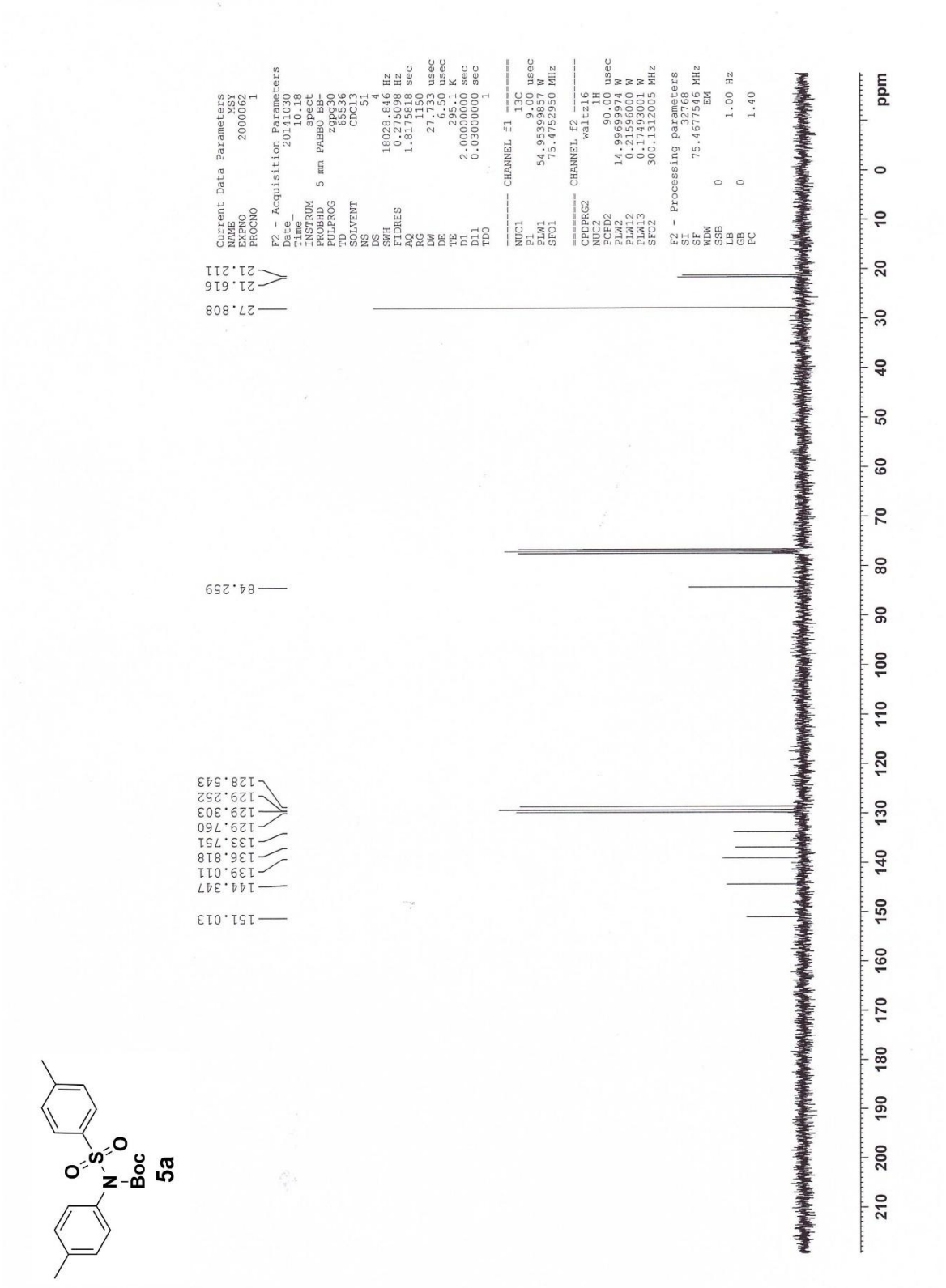

3k
Boc
Current Data Parameters
NAME MSY
EXPNO 2000082
PROCNO 1
F2 - Acquisition Parameters
Date_ 20141103
Time 17.48
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 13
DS 2
SWH 6188.119 Hz
FIDRES 0.094423 Hz
AQ 5.2953587 sec
RG 144
DW 80.800 usec
DE 6.50 usec
TE 294.9 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 10.80 usec
PLW1 14.99699974 W
SFO1 300.1318534 MHz
F2 - Processing parameters
SI 65536
SF 300.1300060 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
1.404
7.807
7.802
7.794
7.790
7.720
7.715
7.703
7.699
7.431
7.419
7.409
7.261
7.248
7.236
7.231
7.225
7.216
7.151
7.138
7.134
7.121
9.00
1.05
2.87
3.12
1.01
0.99
ppm


Current Data Parameters
NAME MSY
EXPNO 2000077
PROCNO 1
F2 - Acquisition Parameters
Date_ 20141103
Time_ 10.21
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT C6D6
NS 708
DS 4
SWH 18028.846 Hz
FIDRES 0.275098 Hz
AQ 1.8175018 sec
RG 143
DW 27.733 usec
DE 6.50 usec
TE 295.4 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.00 usec
PLW1 54.95399857 W
SFO1 75.4752950 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PLW2 14.99699974 W
PLW12 0.21596000 W
PLW13 0.17493001 W
SFO2 300.1312005 MHz
F2 - Processing parameters
SI 32768
SF 75.4677490 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
27.360
83.574
126.428
128.634
128.819
129.824
132.772
134.880
136.991
140.230
150.858
ppm
S
O
O
S
O
N
Boc
3k


3l
Boc
7.757
7.731
7.651
7.623
7.599
7.559
7.532
7.507
7.464
7.448
7.430
7.419
7.393
7.368
1.353
9.00
9.16
0.99
ppm

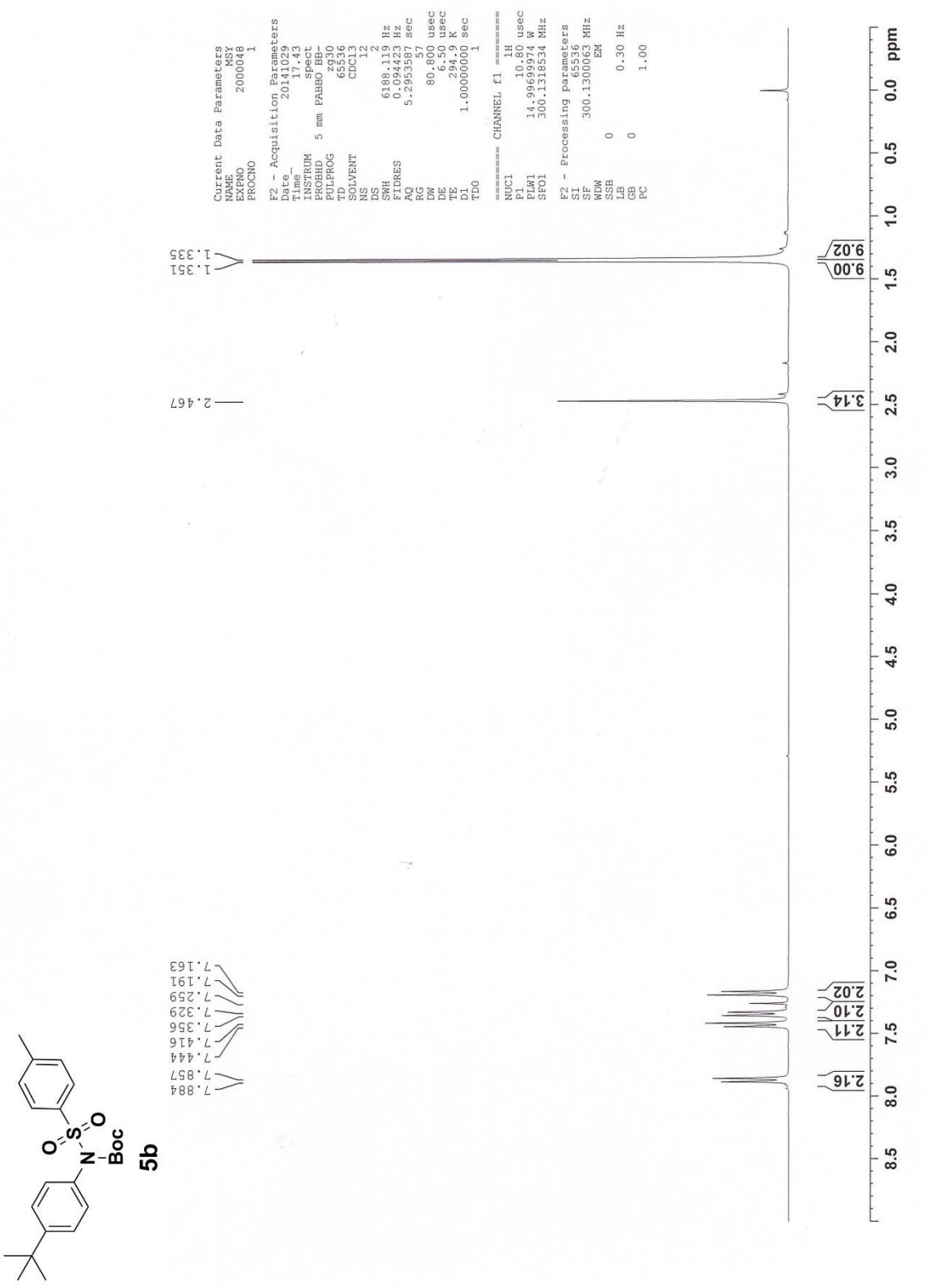

Current Data Parameters
NAME MSY
EXPNO 1000124
PROCNO 1
F2 - Acquisition Parameters
Date_ 20141031
Time 14.44
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 102
DS 4
SWH 18028.846 Hz
FIDRES 0.275098 Hz
AQ 1.8175618 sec
RG 1793
DW 27.750 usec
DE 6.50 usec
TE 295.1 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.00 usec
PLW1 54.95399857 W
SFO1 75.4752950 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PLW2 14.99699974 W
PLW12 0.21596000 W
PLW13 0.17493001 W
SFO2 300.1312005 MHz
F2 - Processing parameters
SI 32768
SF 75.4677571 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
27.663
85.239
112.283
114.849
123.131
124.438
125.730
128.195
129.240
129.295
129.910
135.624
149.068
150.274
155.645
ppm
O
S
O
O
N
Boc
3l

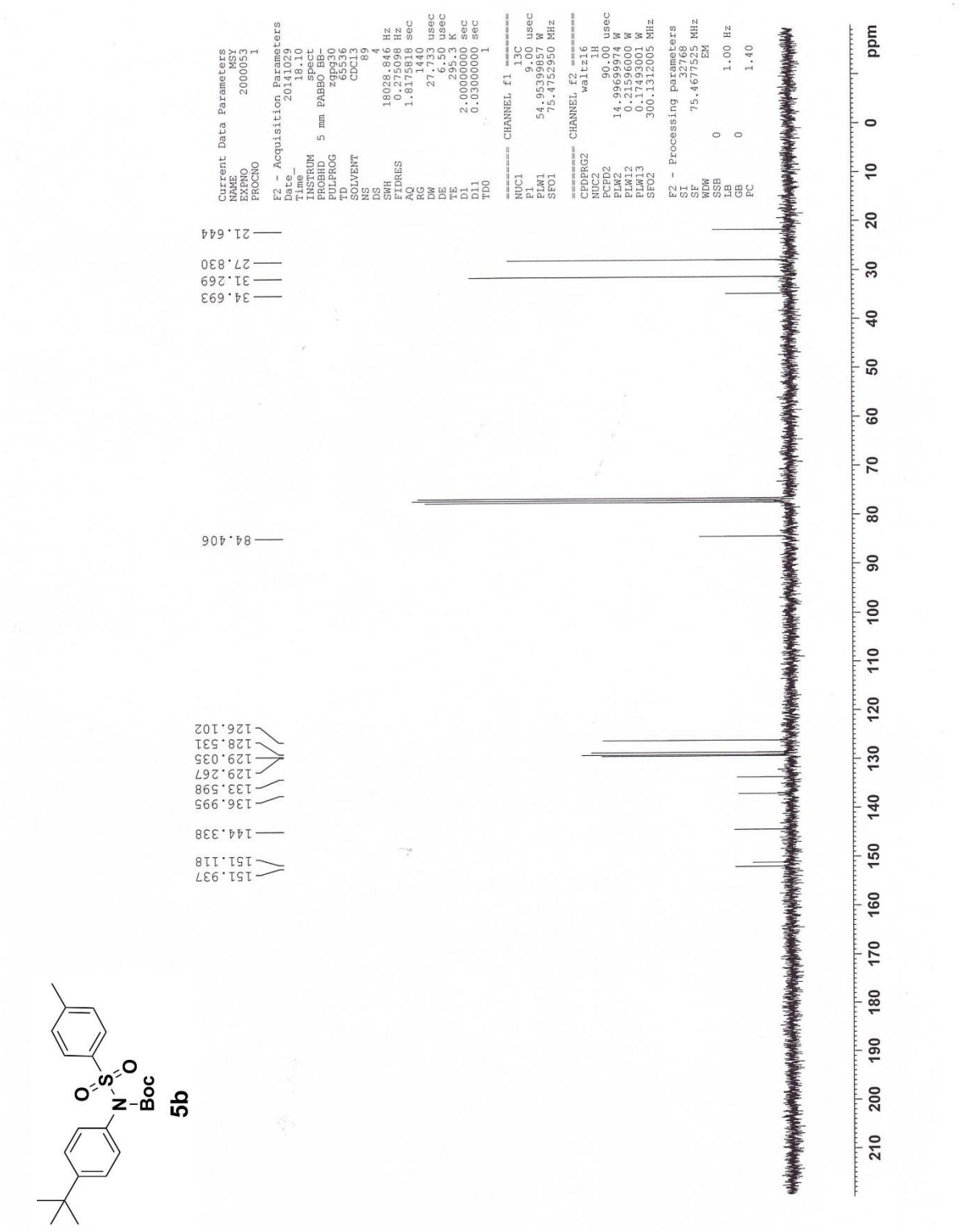

Boc
5a
7.878
7.850
7.346
7.319
7.255
7.234
7.206
7.141
7.114
2.458
2.383
1.341
9.00
2.97
2.97
1.99
2.03
1.96
1.97
ppm

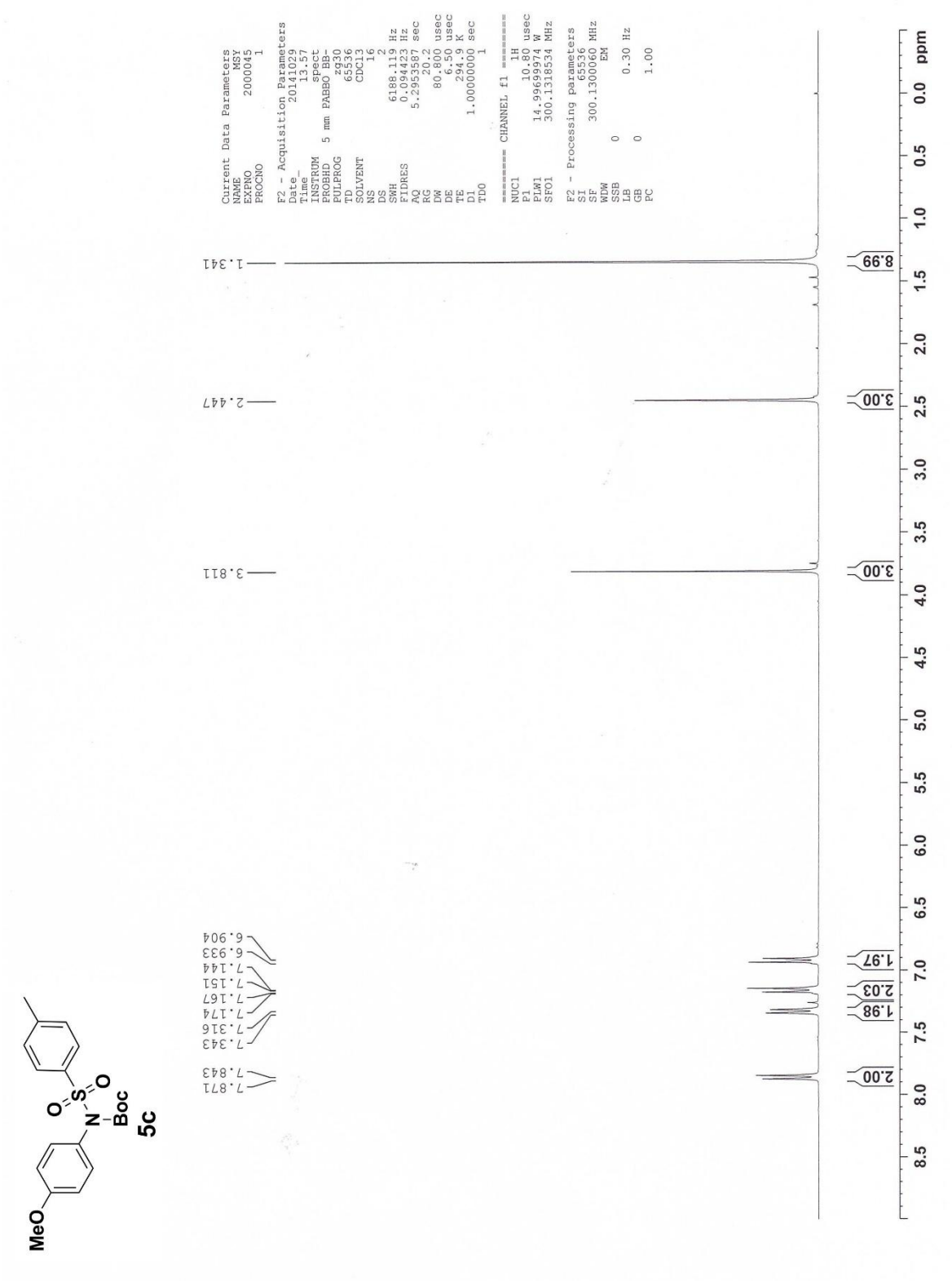

Current Data Parameters
NAME MSY
EXPNO 2000062
PROCNO 1
F2 - Acquisition Parameters
Date_ 20141030
Time 10.18
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 51
DS 4
SWH 18028.846 Hz
FIDRES 0.275098 Hz
AQ 1.8171501 sec
RG 1150
DW 27.733 usec
DE 6.50 usec
TE 295.1 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.00 usec
PLW1 54.95399857 W
SFO1 75.4752950 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PLW2 14.99699974 W
PLW12 0.21596000 W
PLW13 0.17493001 W
SFO2 300.1312005 MHz
F2 - Processing parameters
SI 32768
SF 75.4677546 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
21.211
21.616
27.808
84.259
128.543
129.252
129.303
129.760
133.751
136.818
139.011
144.347
151.013
ppm

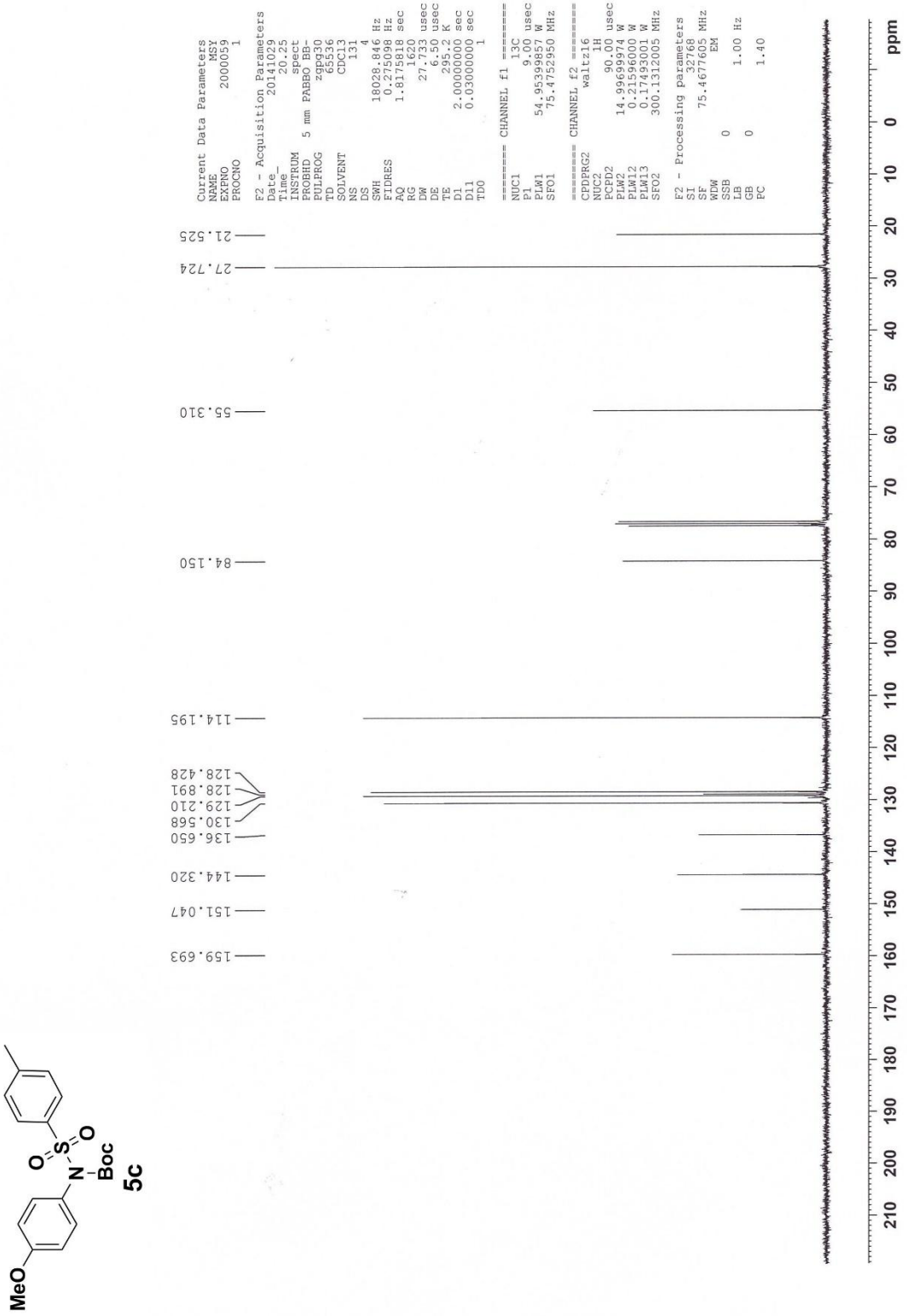
O
S
O
N
Boc

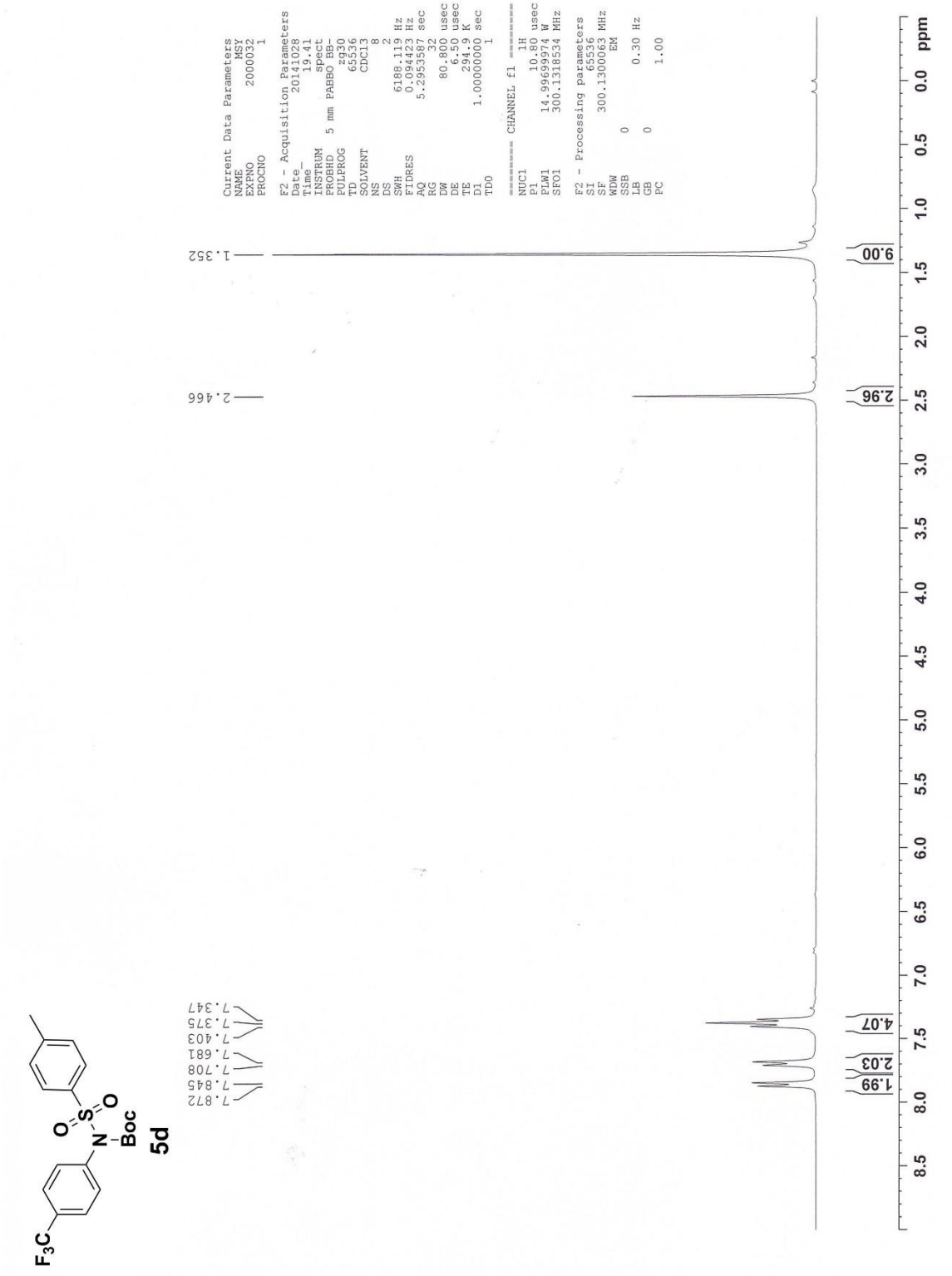
5a

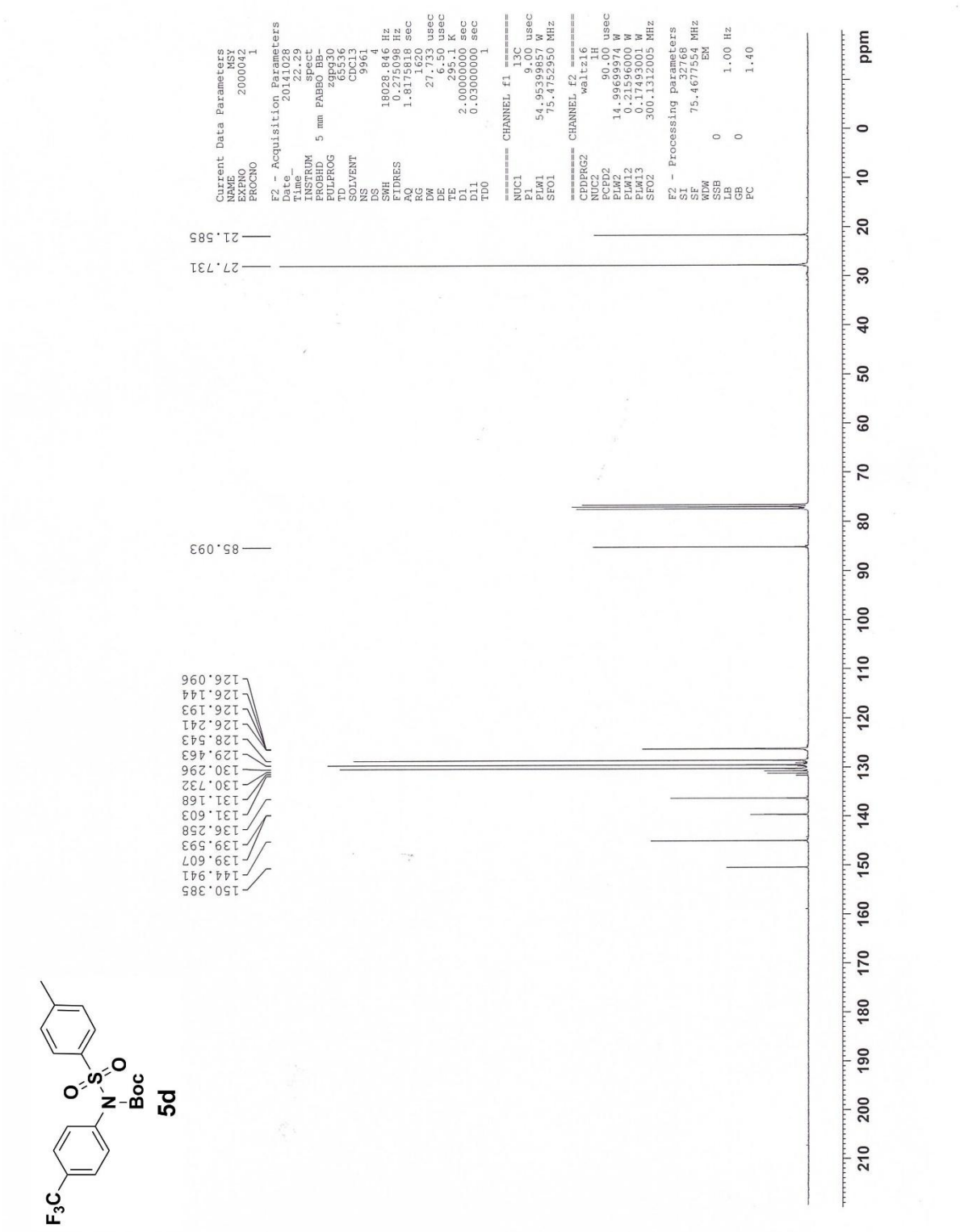

5b
Boc
N
S
O
O
7.884
7.857
7.444
7.416
7.356
7.329
7.259
7.191
7.163
2.467
1.351
1.335
2.16
2.11
2.10
2.02
3.14
9.00
9.02
ppm

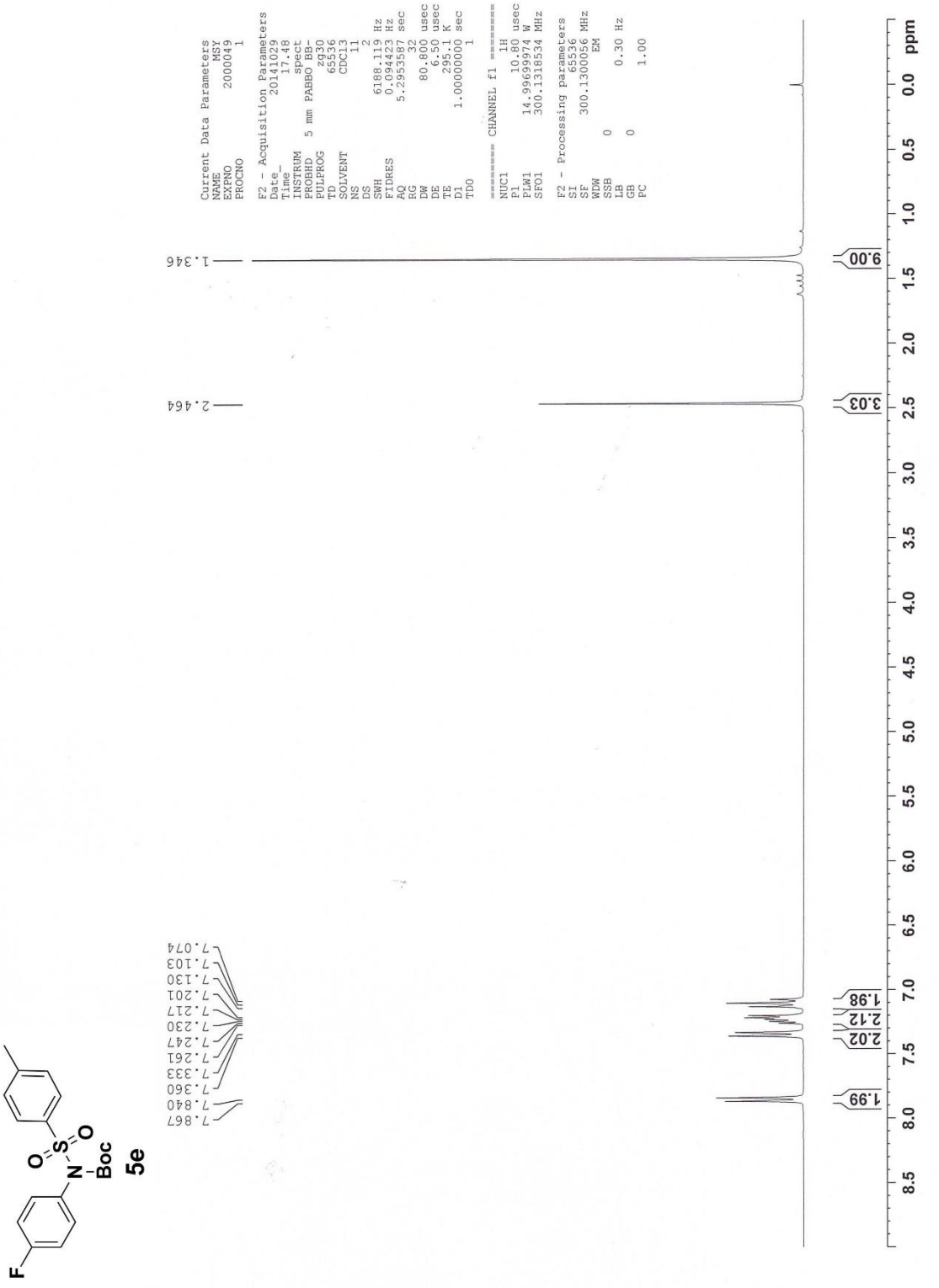

Current Data Parameters
NAME MSY
EXPNO 2000053
PROCNO 1
F2 - Acquisition Parameters
Date_ 20141029
Time_ 18.10
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 89
DS 4
SWH 18028.846 Hz
FIDRES 0.275098 Hz
AQ 1.8175818 sec
RG 1440
DW 27.733 usec
DE 6.50 usec
TE 295.3 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.00 usec
PLW1 54.95399857 W
SFO1 75.4752950 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PLW2 14.99699974 W
PLW12 0.21596000 W
PLW13 0.17493001 W
SFO2 300.1312005 MHz
F2 - Processing parameters
SI 32768
SF 75.4677525 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
21.644
27.830
31.269
34.693
84.406
126.102
128.531
129.035
129.267
133.598
136.995
144.338
151.118
151.937
ppm
Boc
5b

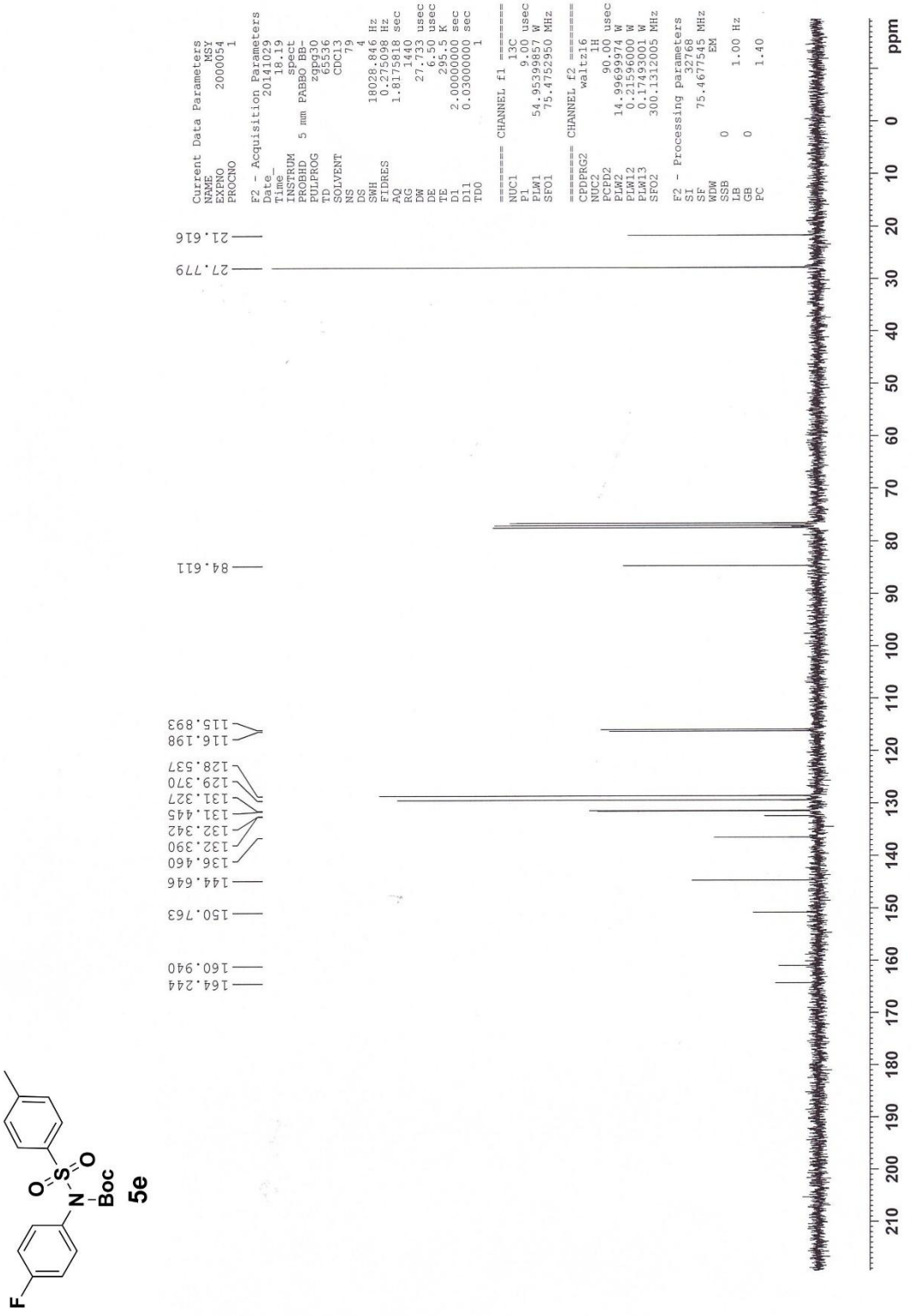

MeO
N
Boc
5c
1.341
2.447
3.811
6.904
6.933
7.144
7.151
7.167
7.174
7.316
7.343
7.843
7.871
8.99
3.00
3.00
1.97
2.03
1.98
2.00
ppm

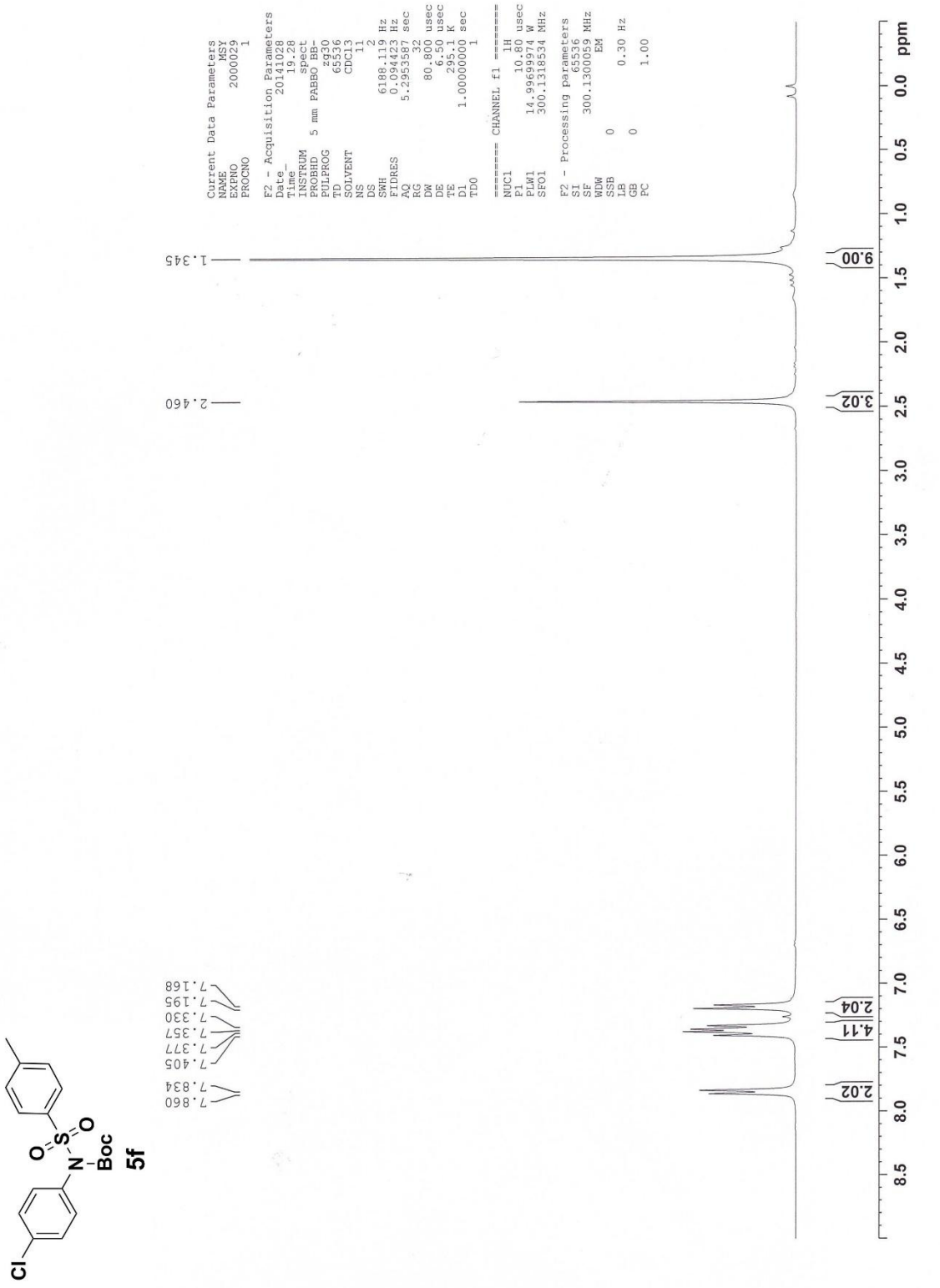

Current Data Parameters
NAME MSY
EXPNO 2000059
PROCNO 1
F2 - Acquisition Parameters
Date_ 20141029
Time_ 20.25
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 131
DS 4
SWH 18028.846 Hz
FIDRES 0.275098 Hz
AQ 1.8175818 sec
RG 1620
DW 27.733 usec
DE 6.50 usec
TE 295.2 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.00 usec
PLW1 54.95399857 W
SFO1 75.4752950 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PLW2 14.99699974 W
PLW12 0.21596000 W
PLW13 0.17493001 W
SFO2 300.1312005 MHz
F2 - Processing parameters
SI 32768
SF 75.4677605 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
21.525
27.724
55.310
84.150
114.195
128.428
128.891
129.210
130.568
136.650
144.320
151.047
159.693
ppm
MeO
N
Boc
S
O
O
5c

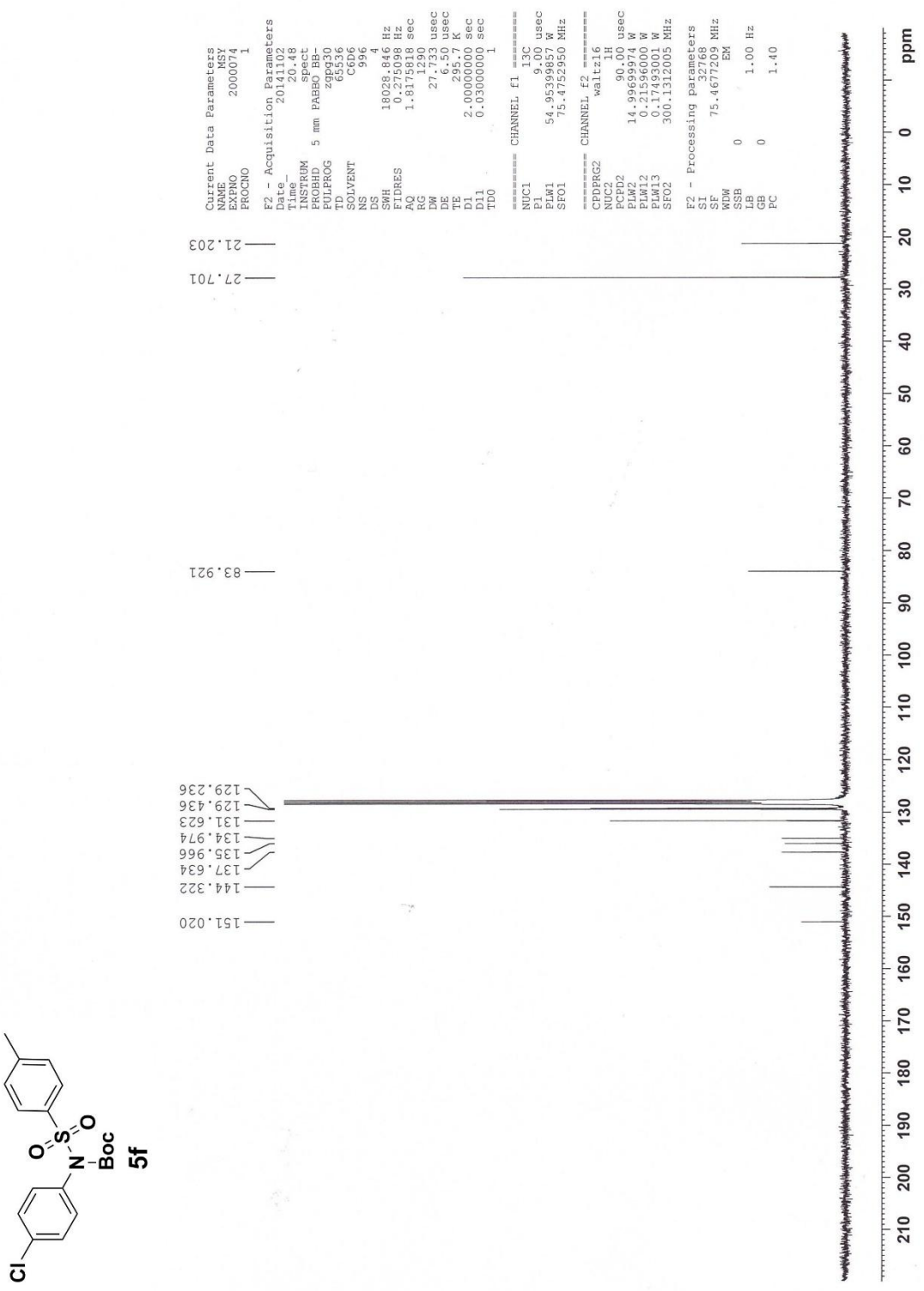

5d
F3C
O
S
N
Boc
7.872
7.845
7.708
7.681
7.403
7.375
7.347
2.466
1.352
9.00
2.96
4.07
2.03
1.99
ppm

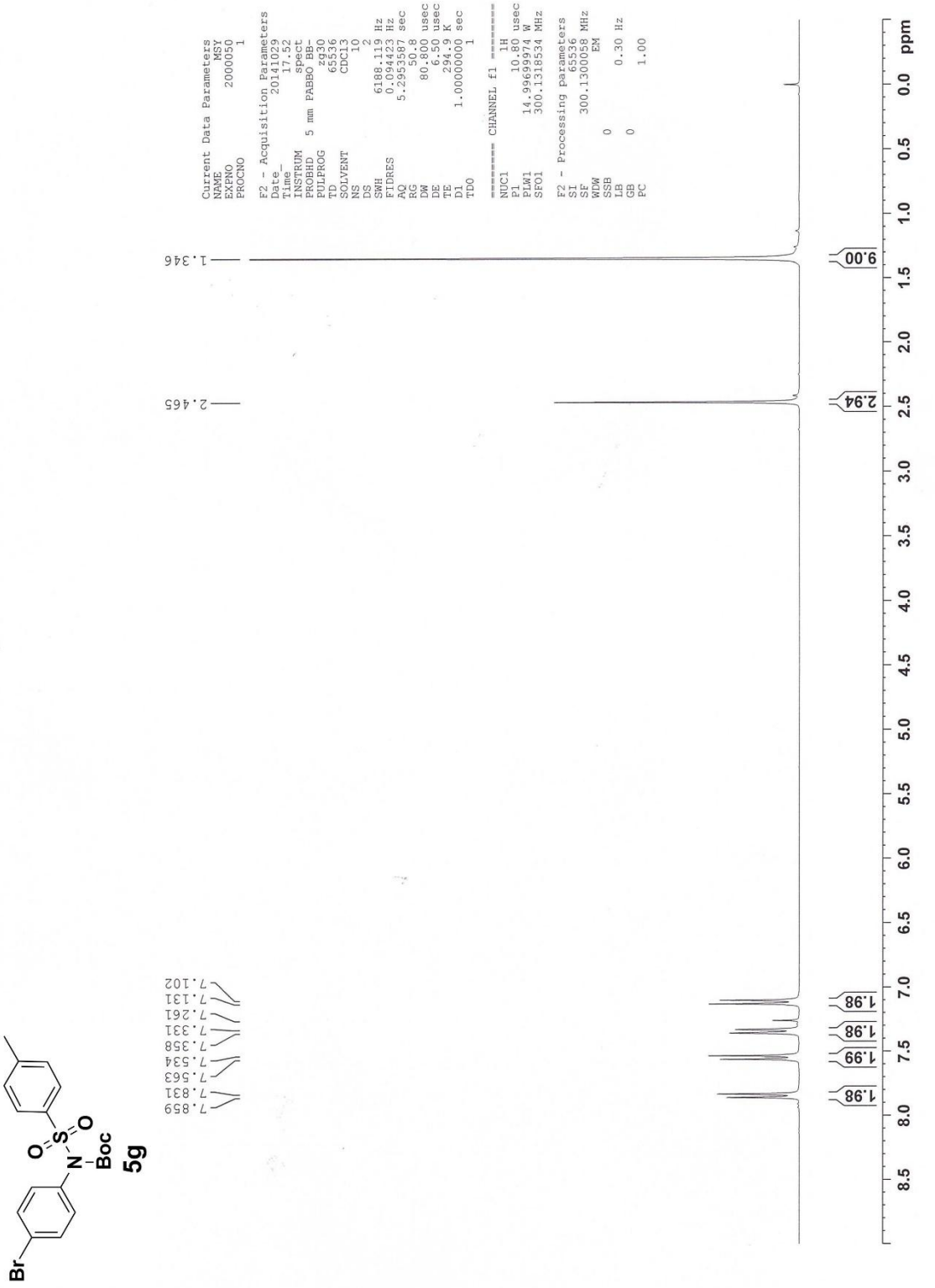

Current Data Parameters
NAME MSY
EXPNO 2000042
PROCNO 1
F2 - Acquisition Parameters
Date_ 20141028
Time 22.29
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 9961
DS 4
SWH 18028.846 Hz
FIDRES 0.275098 Hz
AQ 1.8175618 sec
RG 1710
DW 27.733 usec
DE 6.50 usec
TE 295.1 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.00 usec
PLW1 54.95399857 W
SFO1 75.4752950 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PLW2 14.99699974 W
PLW12 0.21596000 W
PLW13 0.17493001 W
SFO2 300.1312005 MHz
F2 - Processing parameters
SI 32768
SF 75.4677554 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
21.585
27.731
85.093
126.096
126.144
126.193
126.241
128.543
129.463
130.296
130.732
131.168
131.603
136.258
139.593
139.607
144.941
150.385
ppm
5d
Boc
N
S
O
O
F3C

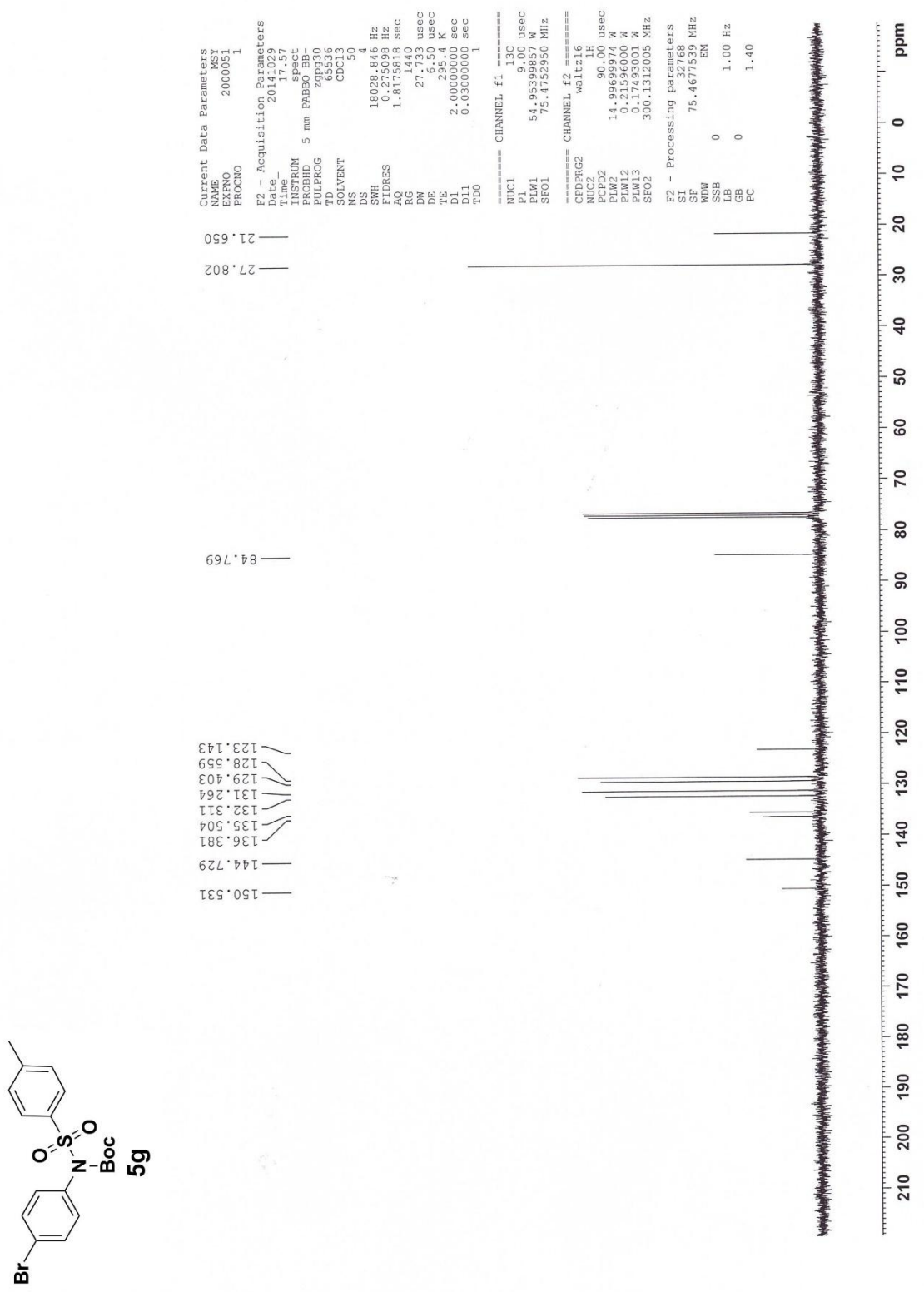

5e
Boc
F
Current Data Parameters
NAME MSY
EXPNO 2000049
PROCNO 1
F2 - Acquisition Parameters
Date_ 20141029
Time 17.48
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 11
DS 2
SWH 6188.119 Hz
FIDRES 0.094423 Hz
AQ 5.2953587 sec
RG 32
DW 80.900 usec
DE 6.50 usec
TE 295.1 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 10.80 usec
PLW1 14.99699974 W
SFO1 300.1318534 MHz
F2 - Processing parameters
SI 65536
SF 300.1300056 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
1.346
2.464
7.867
7.840
7.360
7.333
7.261
7.247
7.230
7.217
7.201
7.130
7.103
7.074
9.00
3.03
1.98
2.12
2.02
1.99
ppm

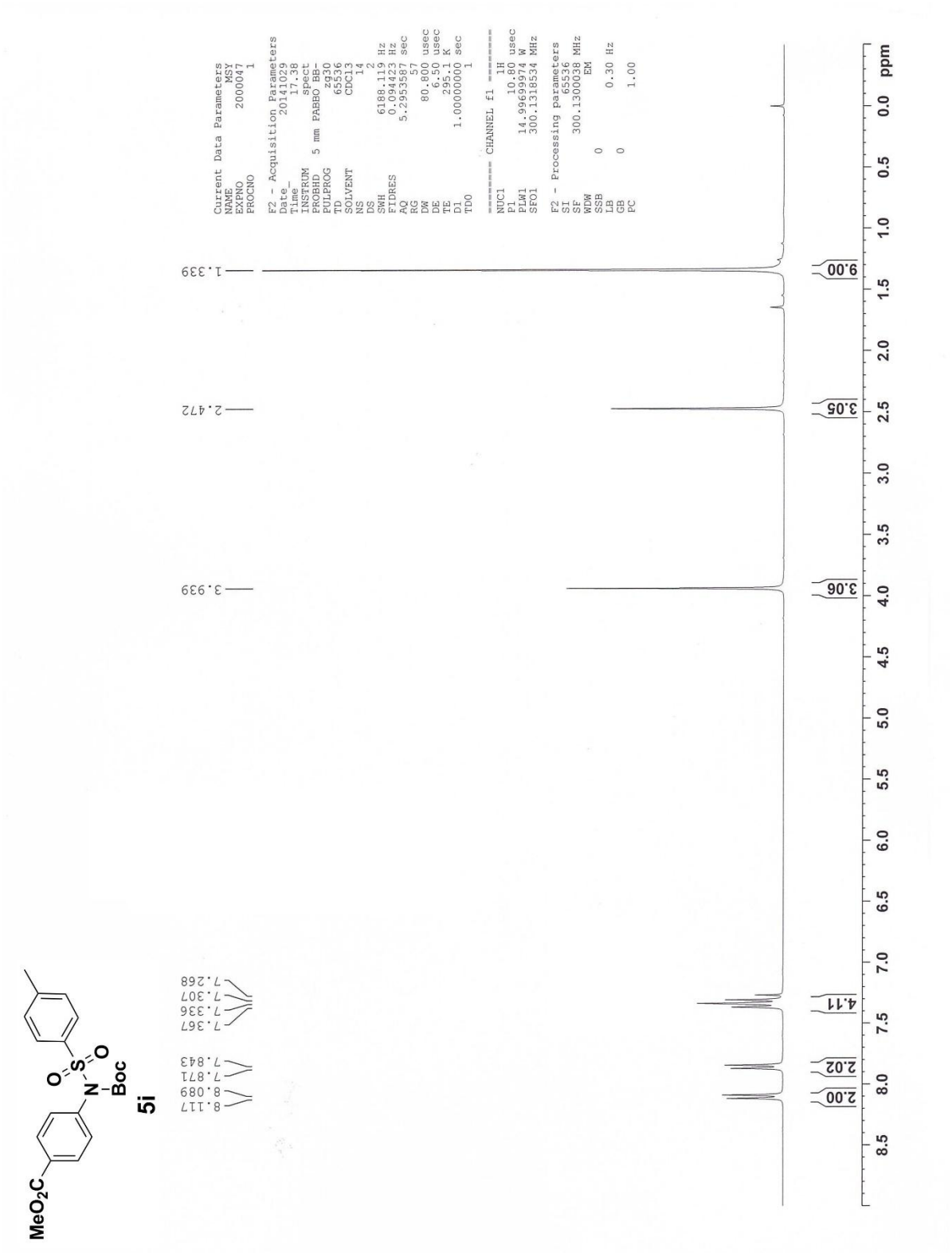

Current Data Parameters
NAME MSY
EXPNO 2000054
PROCNO 1
F2 - Acquisition Parameters
Date_ 20141029
Time 18.19
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 79
DS 4
SWH 18028.846 Hz
FIDRES 0.275098 Hz
AQ 1.8175440 sec
RG 143
DW 27.733 usec
DE 6.50 usec
TE 295.5 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.00 usec
PLW1 54.95399857 W
SFO1 75.4752950 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PLW2 14.99699974 W
PLW12 0.21596000 W
PLW13 0.17493001 W
SFO2 300.1312005 MHz
F2 - Processing parameters
SI 32768
SF 75.4677545 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
21.616
27.779
84.611
115.893
116.198
128.537
129.370
131.327
131.445
132.342
132.390
136.460
144.646
150.763
160.940
164.244
ppm
5e
Boc
N
S
O
O
F

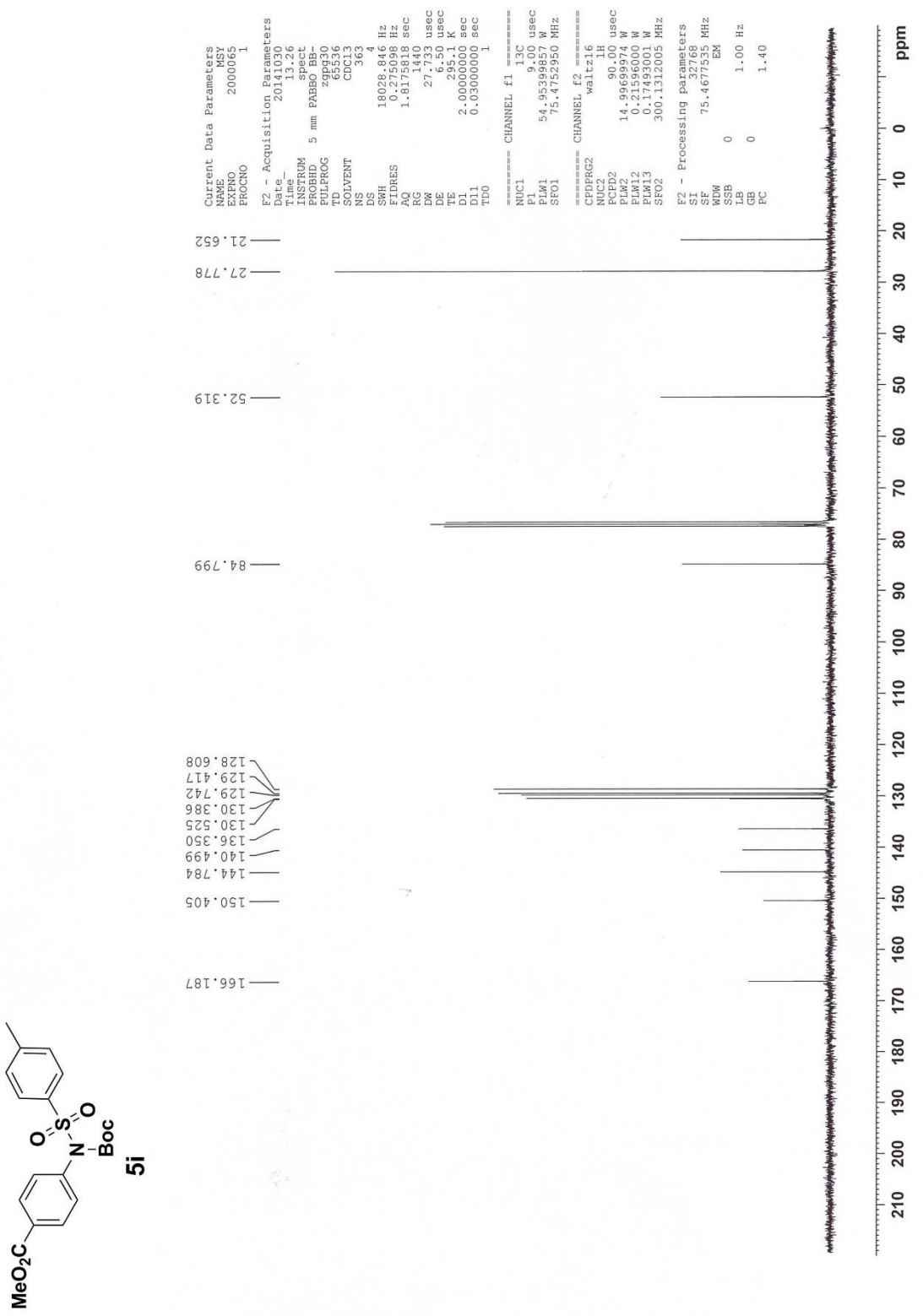

Current Data Parameters
NAME MSY
EXPNO 2000029
PROCNO 1
F2 - Acquisition Parameters
Date_ 20141028
Time 19.28
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 11
DS 2
SWH 6188.119 Hz
FIDRES 0.094423 Hz
AQ 5.295367 sec
RG 32
DW 80.800 usec
DE 6.50 usec
TE 295.1 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 10.80 usec
PLW1 14.99699974 W
SFO1 300.1318534 MHz
F2 - Processing parameters
SI 65536
SF 300.1300059 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
1.345
2.460
7.168
7.195
7.330
7.357
7.377
7.405
7.834
7.860
9.00
3.02
2.04
4.11
2.02
ppm
5f

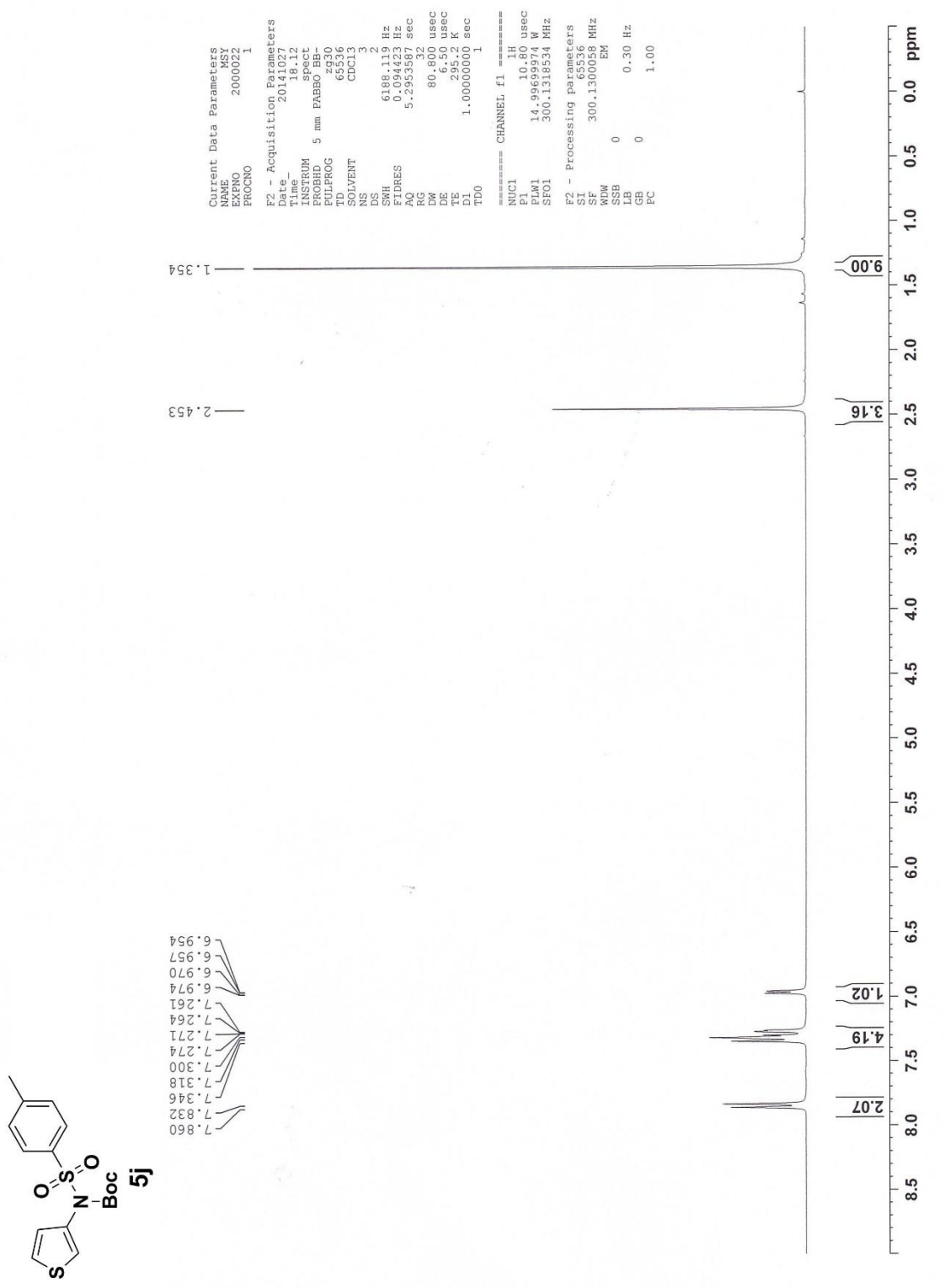

Current Data Parameters
NAME MSY
EXPNO 2000074
PROCNO 1
F2 - Acquisition Parameters
Date_ 20141102
Time 20.48
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT C6D6
NS 996
DS 4
SWH 18028.846 Hz
FIDRES 0.275098 Hz
AQ 1.8175818 sec
RG 1290
DW 27.733 usec
DE 6.50 usec
TE 295.7 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.00 usec
PLW1 54.95399857 W
SFO1 75.4752950 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PLW2 14.99699974 W
PLW12 0.21596000 W
PLW13 0.17493001 W
SFO2 300.1312005 MHz
F2 - Processing parameters
SI 32768
SF 75.4677209 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
21.203
27.701
83.921
129.236
129.436
131.623
134.974
135.966
137.634
144.322
151.020
ppm
0
10
20
30
40
50
60
70
80
90
100
110
120
130
140
150
160
170
180
190
200
210
O
S
O
N
Boc
Cl
5f

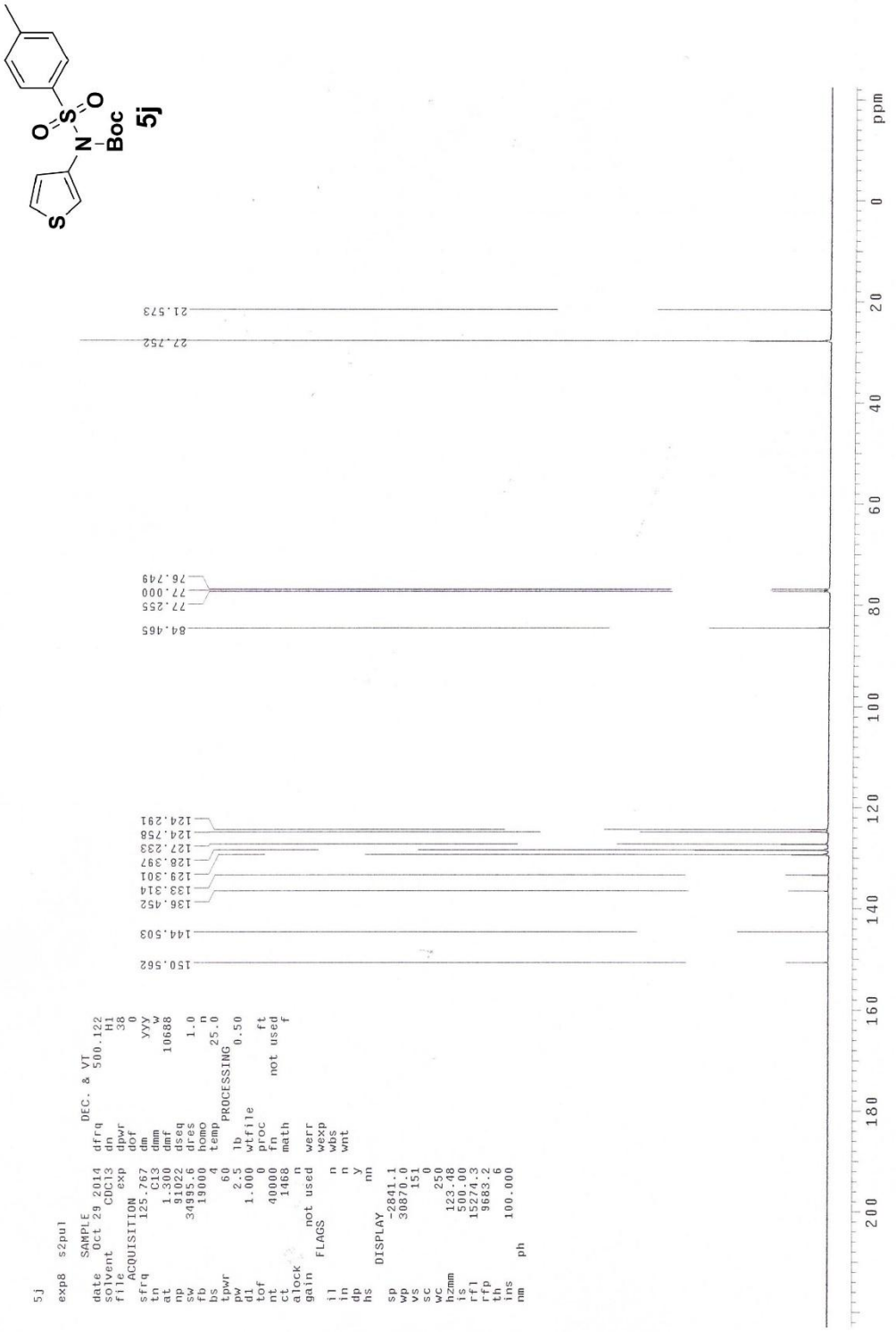

Br
O
S
O
N
Boc
5g
7.859
7.831
7.563
7.534
7.358
7.331
7.261
7.131
7.102
1.346
2.465
1.98
1.98
1.99
1.98
9.00
2.94
ppm

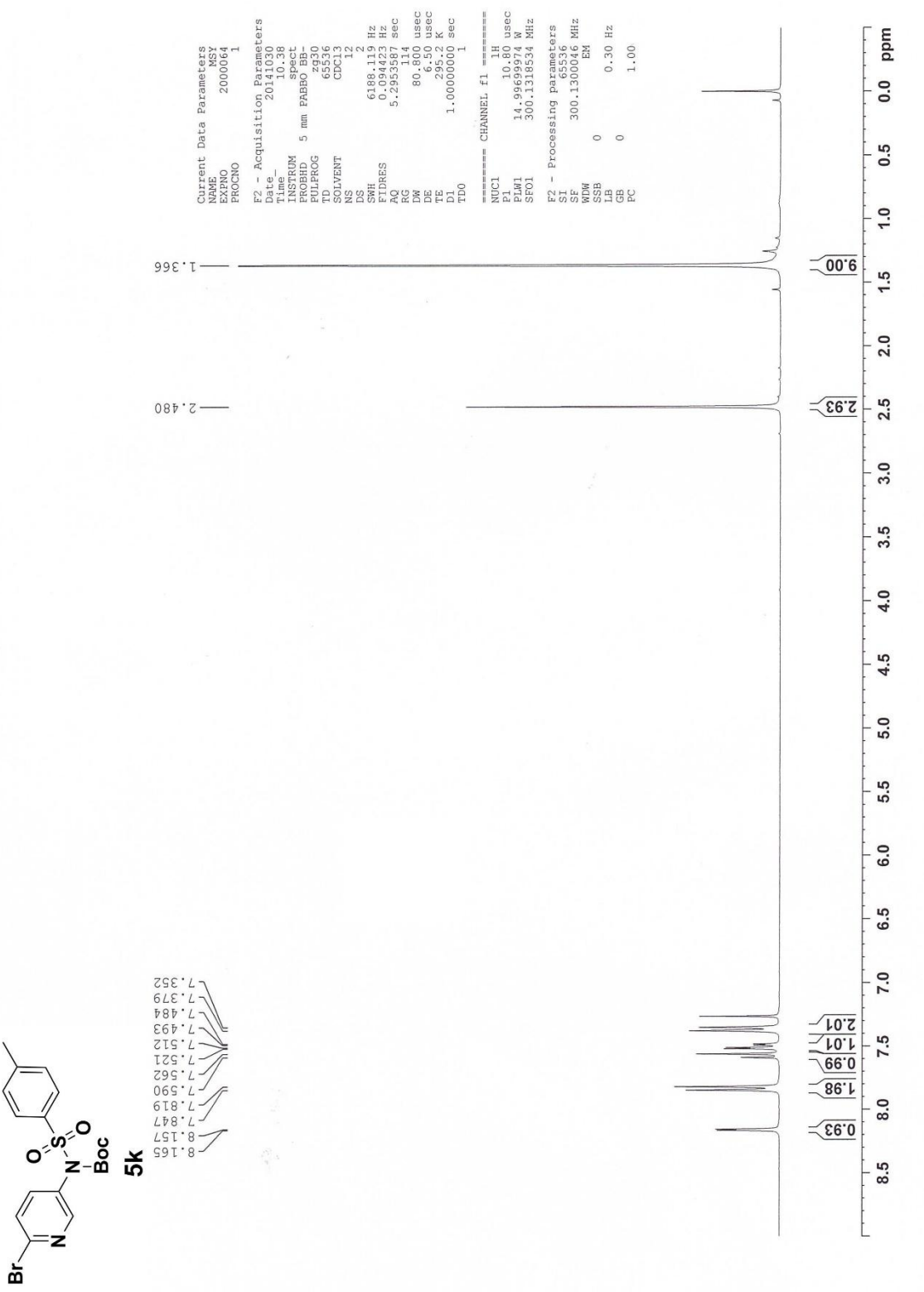

Br
O
S
O
N
Boc
5g
21.650
27.802
84.769
123.143
128.559
129.403
131.264
132.311
135.504
136.381
144.729
150.531
ppm

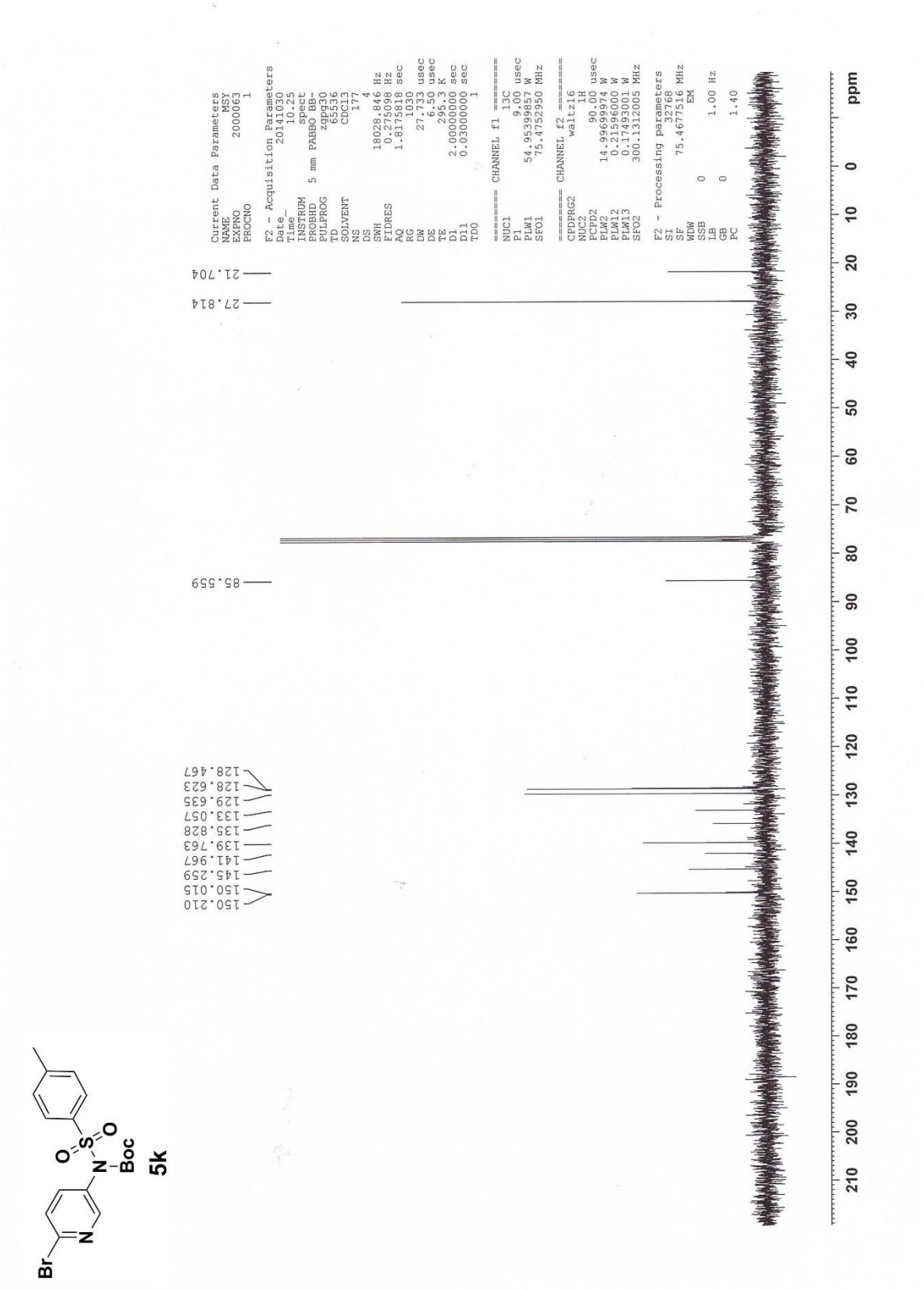

MeO2C
O
S
O
N
Boc
5i
8.117
8.089
7.871
7.843
7.367
7.336
7.307
7.268
1.339
2.472
3.939
9.00
3.05
3.06
4.11
2.02
2.00
Current Data Parameters
NAME MSY
EXPNO 2000047
PROCNO 1
F2 - Acquisition Parameters
Date_ 20141029
Time 17.38
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 14
DS 2
SWH 6188.119 Hz
FIDRES 0.094423 Hz
AQ 5.2953587 sec
RG 57
DW 80.800 usec
DE 6.50 usec
TE 295.1 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 10.80 usec
PLW1 14.99699974 W
SFO1 300.1318534 MHz
F2 - Processing parameters
SI 65536
SF 300.1300038 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm

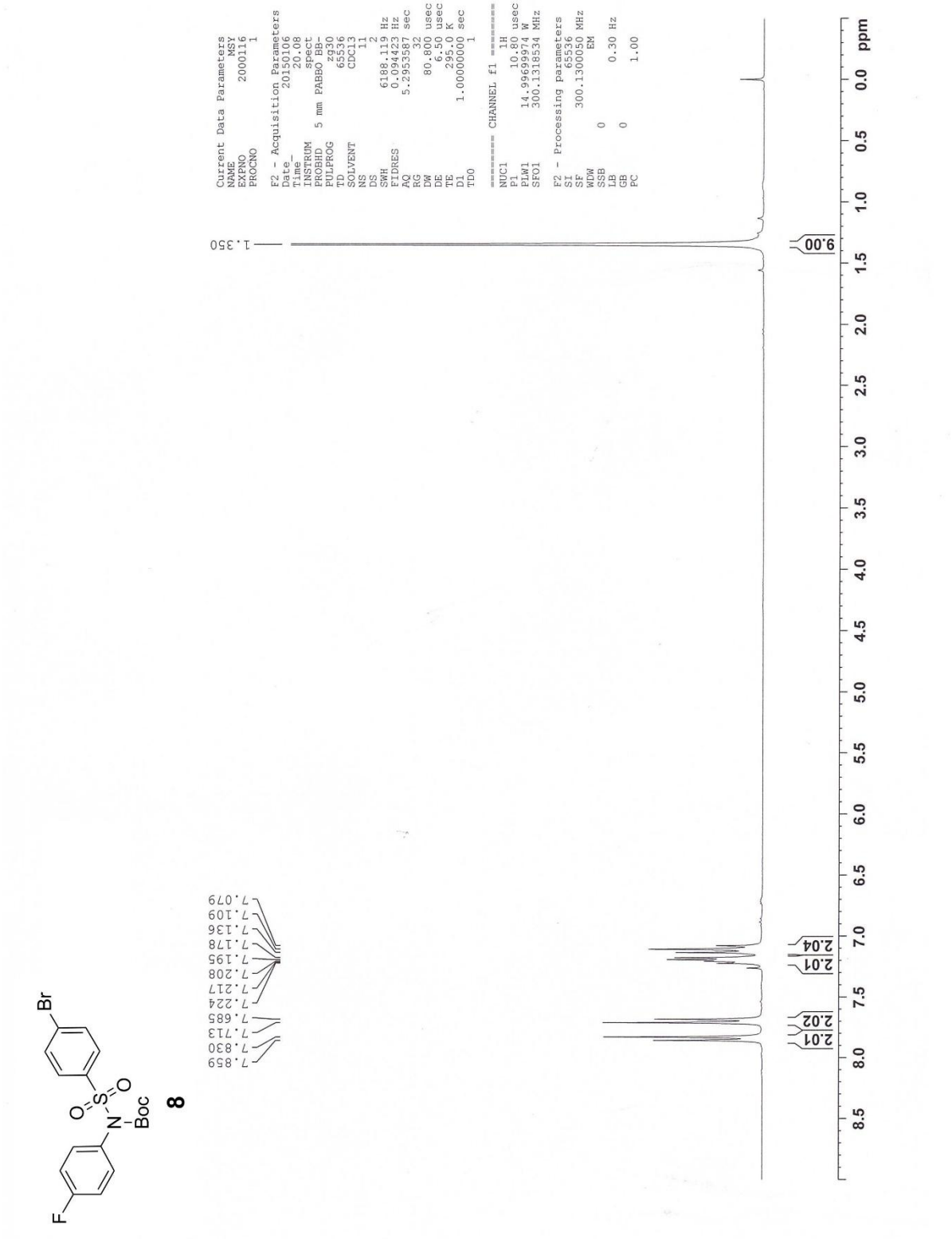

Current Data Parameters
NAME MSY
EXPNO 2000065
PROCNO 1
F2 - Acquisition Parameters
Date_ 20141030
Time 13.26
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 363
DS 4
SWH 18028.846 Hz
FIDRES 0.275098 Hz
AQ 1.8175418 sec
RG 1440
DW 27.733 usec
DE 6.50 usec
TE 295.1 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
CHANNEL f1
NUC1 13C
P1 9.00 usec
PLW1 54.95399857 W
SFO1 75.4752950 MHz
CHANNEL f2
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PLW2 14.99699974 W
PLW12 0.21596000 W
PLW13 0.17493001 W
SFO2 300.1312005 MHz
F2 - Processing parameters
SI 32768
SF 75.4677535 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
21.652
27.778
52.319
84.799
128.608
129.417
129.742
130.386
130.525
136.350
140.499
144.784
150.405
166.187
ppm
MeO2C
O
S
O
N
Boc
5i

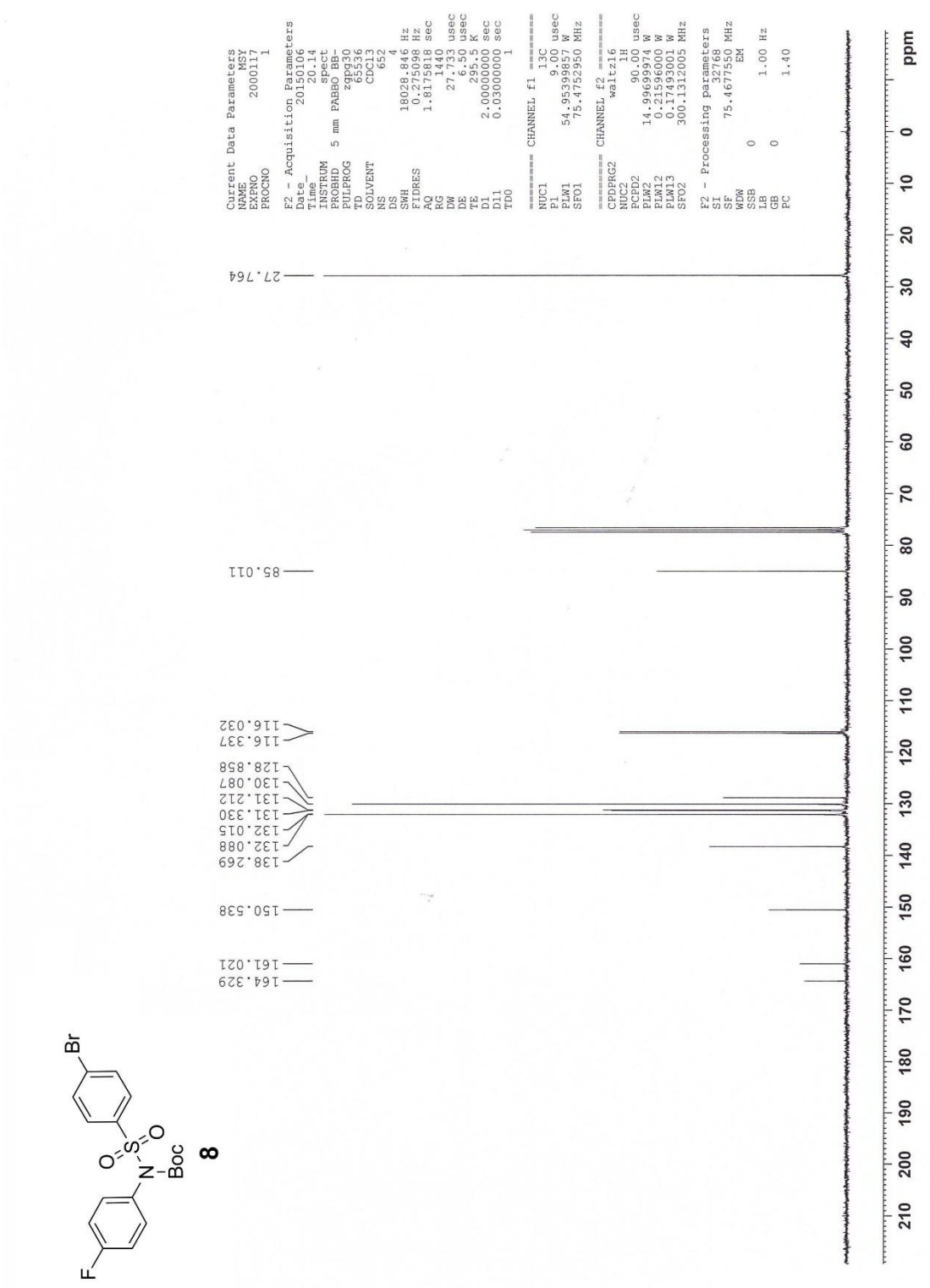

Current Data Parameters
NAME MSY
EXPNO 2000022
PROCNO 1
F2 - Acquisition Parameters
Date_ 20141027
Time 18.12
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 3
DS 2
SWH 6188.119 Hz
FIDRES 0.094423 Hz
AQ 5.295367 sec
RG 32
DW 80.800 usec
DE 6.50 usec
TE 295.2 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 10.80 usec
PLW1 14.99699974 W
SFO1 300.1318534 MHz
F2 - Processing parameters
SI 65536
SF 300.1300058 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
1.354
2.453
6.954
6.957
6.970
6.974
7.261
7.264
7.271
7.274
7.300
7.318
7.346
7.832
7.860
9.00
3.16
1.02
4.19
2.07
ppm
Boc
5j

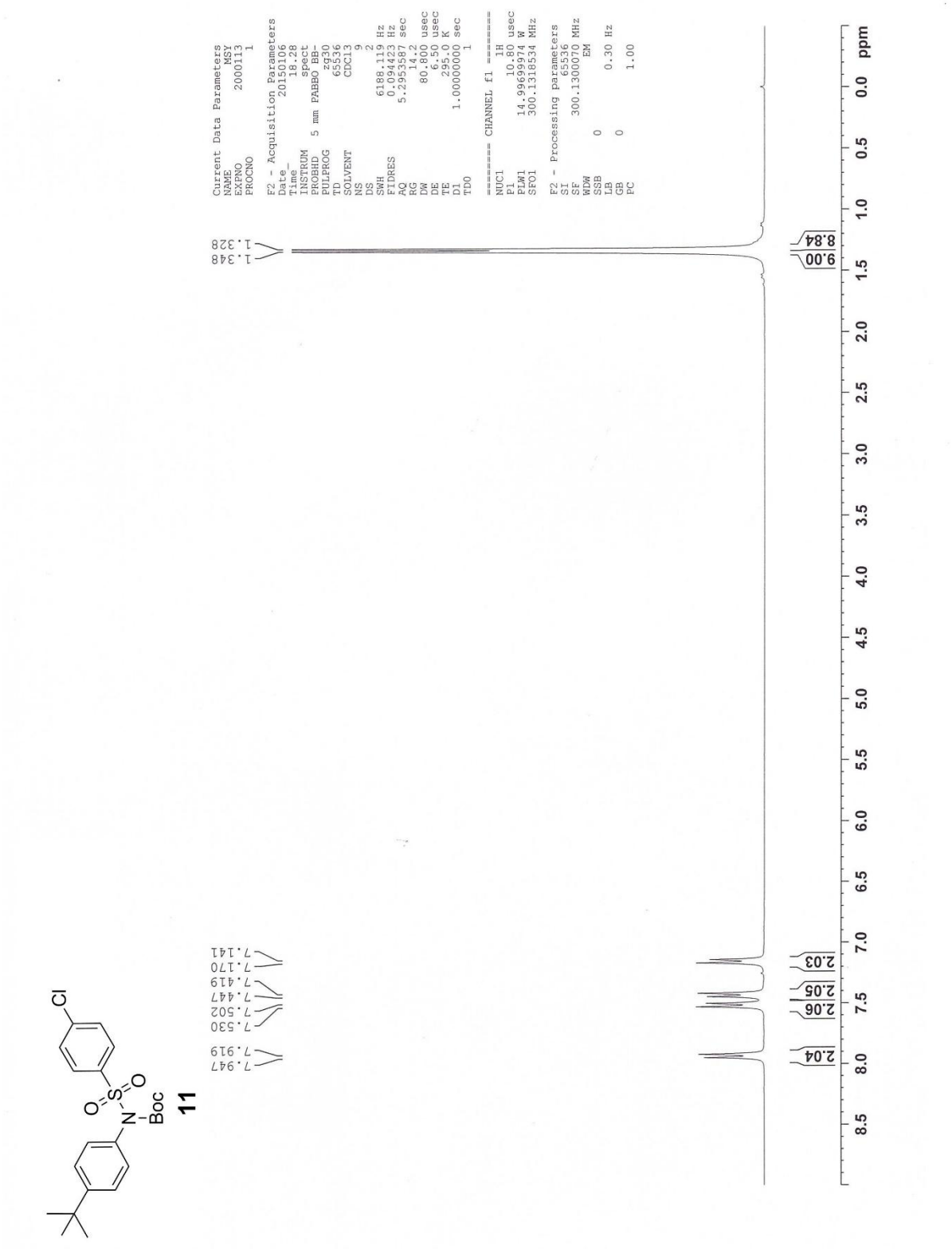

5j
Boc
21.573
27.752
76.749
77.000
77.255
84.465
124.291
124.758
127.233
128.397
129.301
133.314
136.452
144.503
150.562
ppm

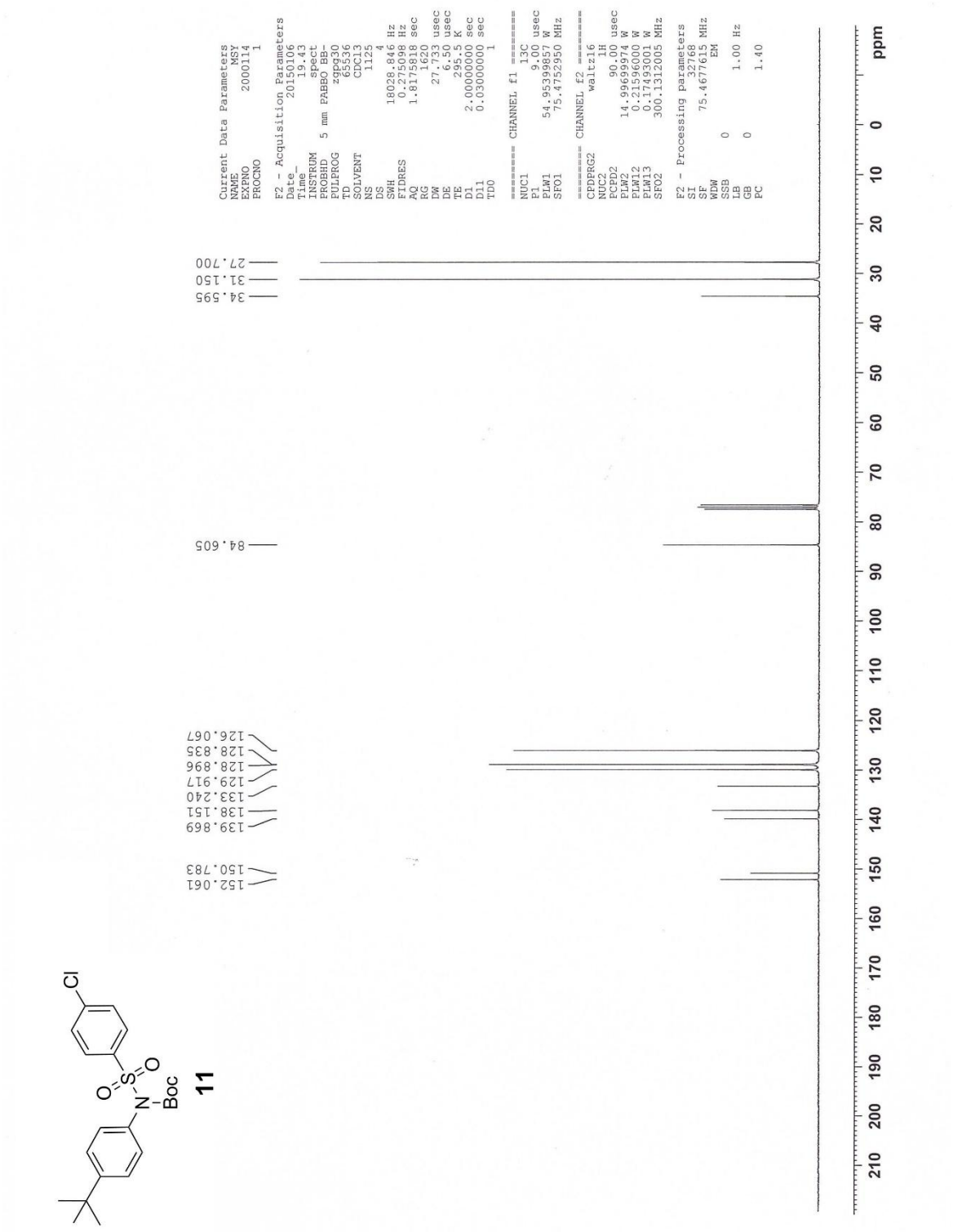

5k
Br
N
Boc
O
S
O
8.165
8.157
7.847
7.819
7.590
7.562
7.521
7.512
7.493
7.484
7.379
7.352
2.480
1.366
0.93
1.98
0.99
1.01
2.01
2.93
9.00
ppm

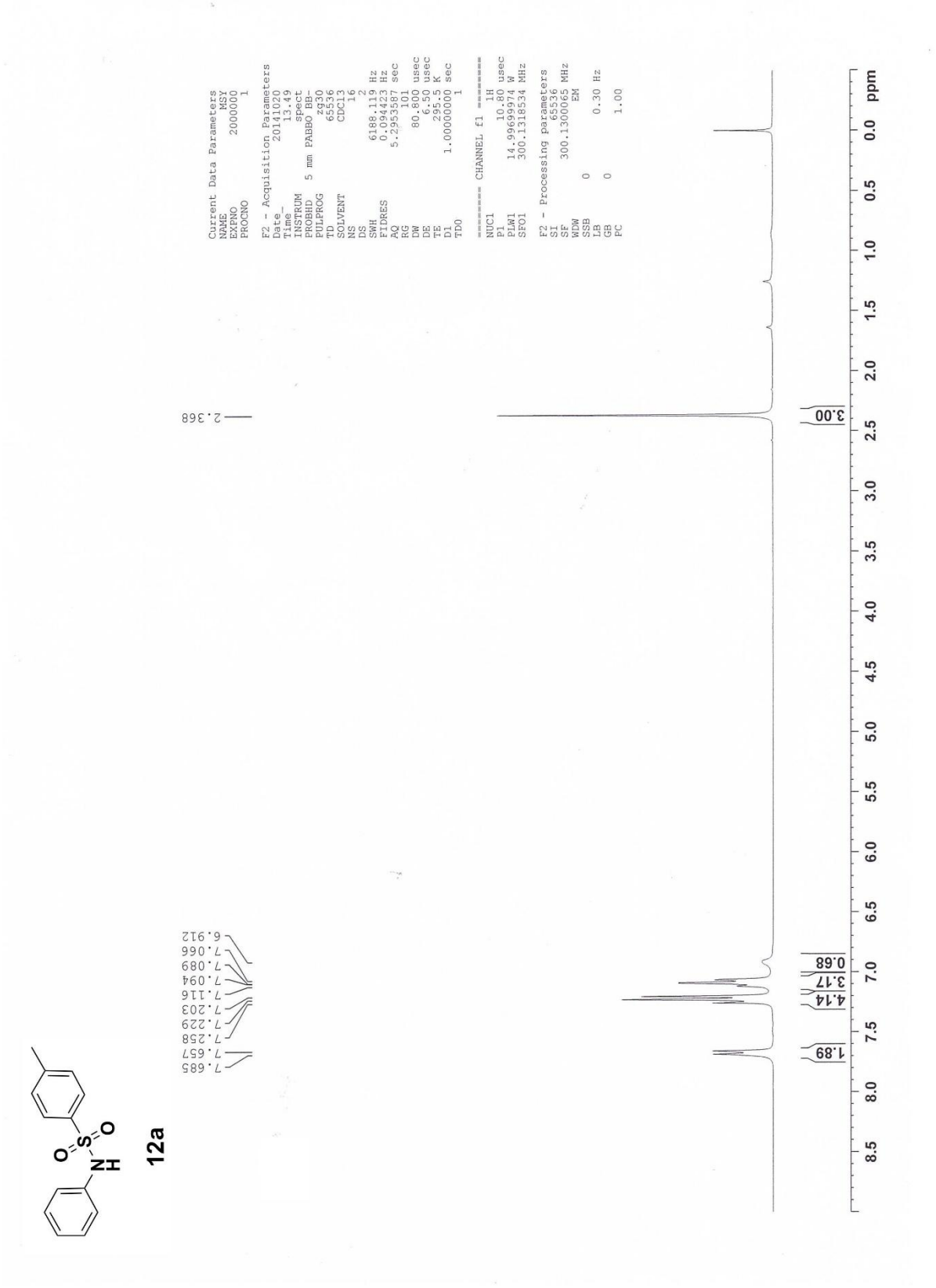

Current Data Parameters
NAME MSY
EXPNO 2000063
PROCNO 1
F2 - Acquisition Parameters
Date_ 20141030
Time 10.25
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 177
DS 4
SWH 18028.846 Hz
FIDRES 0.275098 Hz
AQ 1.817581 sec
RG 1030
DW 27.733 usec
DE 6.50 usec
TE 295.3 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.00 usec
PLW1 54.95399857 W
SFO1 75.4752950 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PLW2 14.99699974 W
PLW12 0.21596000 W
PLW13 0.17493001 W
SFO2 300.1312005 MHz
F2 - Processing parameters
SI 32768
SF 75.4677516 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
21.704
27.814
85.559
128.467
128.623
129.635
133.057
135.828
139.763
141.967
145.259
150.015
150.210
ppm
Br
N
O
S
O
N
Boc
5k

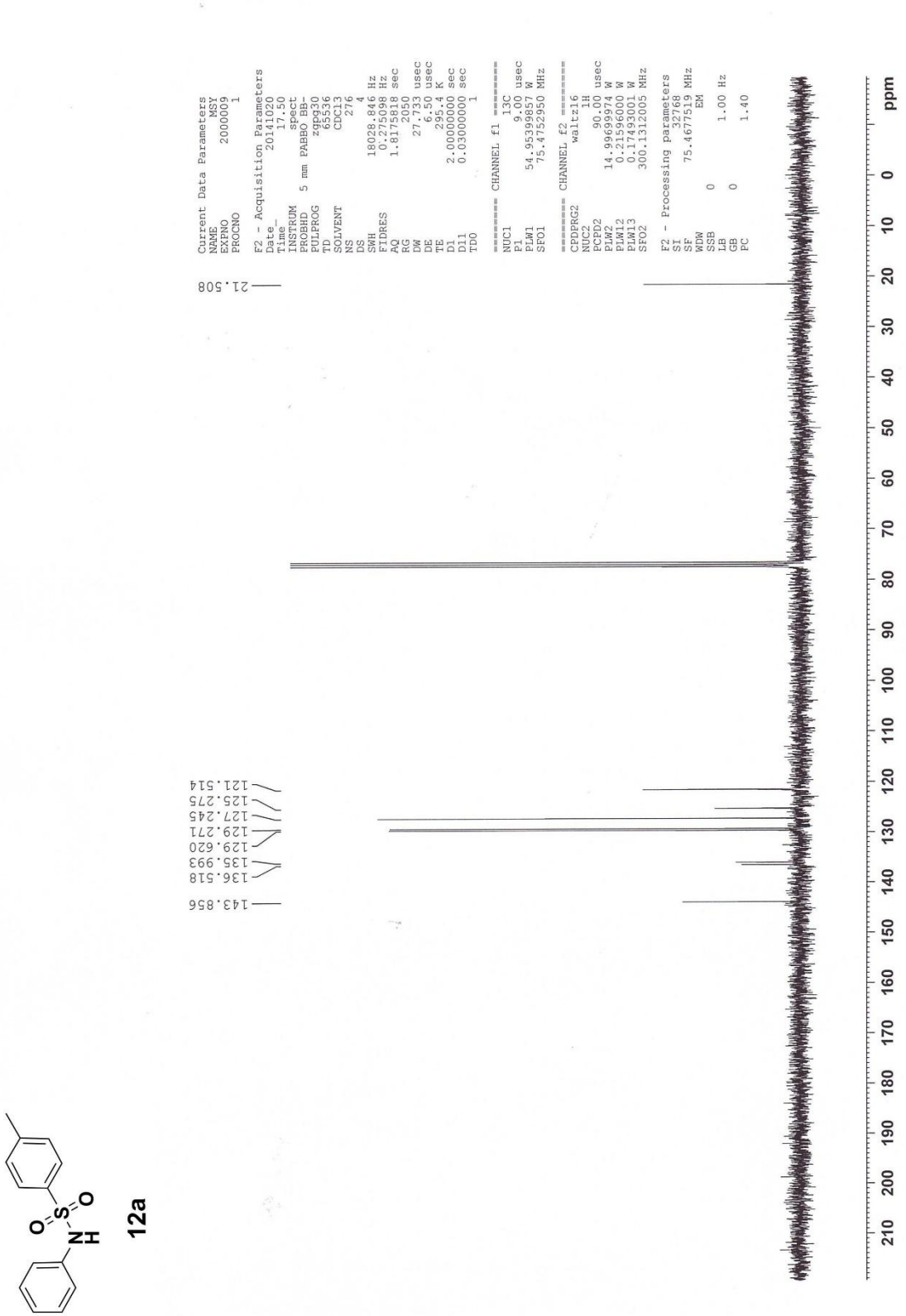

Br
O
S
O
N
Boc
F
8
7.859
7.830
7.713
7.685
7.224
7.217
7.208
7.195
7.178
7.136
7.109
7.079
1.350
9.00
2.04
2.01
2.02
2.01
Current Data Parameters
NAME MSY
EXPNO 2000116
PROCNO 1
F2 - Acquisition Parameters
Date_ 20150106
Time_ 20.08
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 11
DS 2
SWH 6188.119 Hz
FIDRES 0.094423 Hz
AQ 5.2953587 sec
RG 32
DW 80.800 usec
DE 6.50 usec
TE 295.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 10.80 usec
PLW1 14.99699974 W
SFO1 300.1318534 MHz
F2 - Processing parameters
SI 65536
SF 300.1300050 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm


Br
O
S
O
N
Boc
F
8
27.764
85.011
116.032
116.337
128.858
130.087
131.212
131.330
132.015
132.088
138.269
150.538
161.021
164.329
ppm

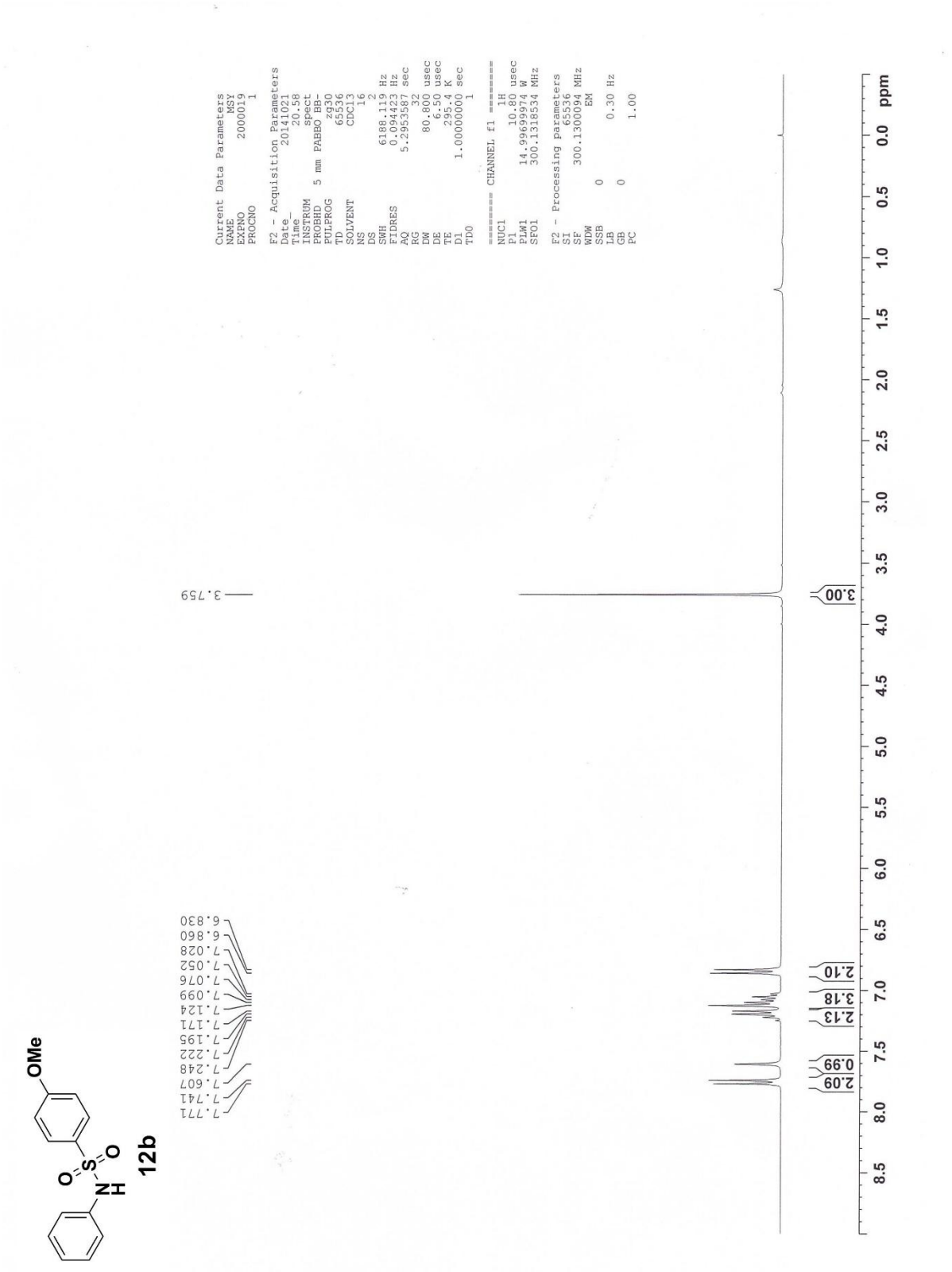

Current Data Parameters
NAME MSY
EXPNO 2000113
PROCNO 1
F2 - Acquisition Parameters
Date_ 20150106
Time 18.28
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 9
DS 2
SWH 6188.119 Hz
FIDRES 0.094423 Hz
AQ 5.2953587 sec
RG 14
DW 80.800 usec
DE 6.50 usec
TE 295.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 10.80 usec
PLW1 14.99699974 W
SFO1 300.1318534 MHz
F2 - Processing parameters
SI 65536
SF 300.1300070 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
1.328
1.348
7.141
7.170
7.419
7.447
7.502
7.530
7.919
7.947
8.84
9.00
2.03
2.05
2.06
2.04
ppm
Cl
O
S
O
N
Boc
11

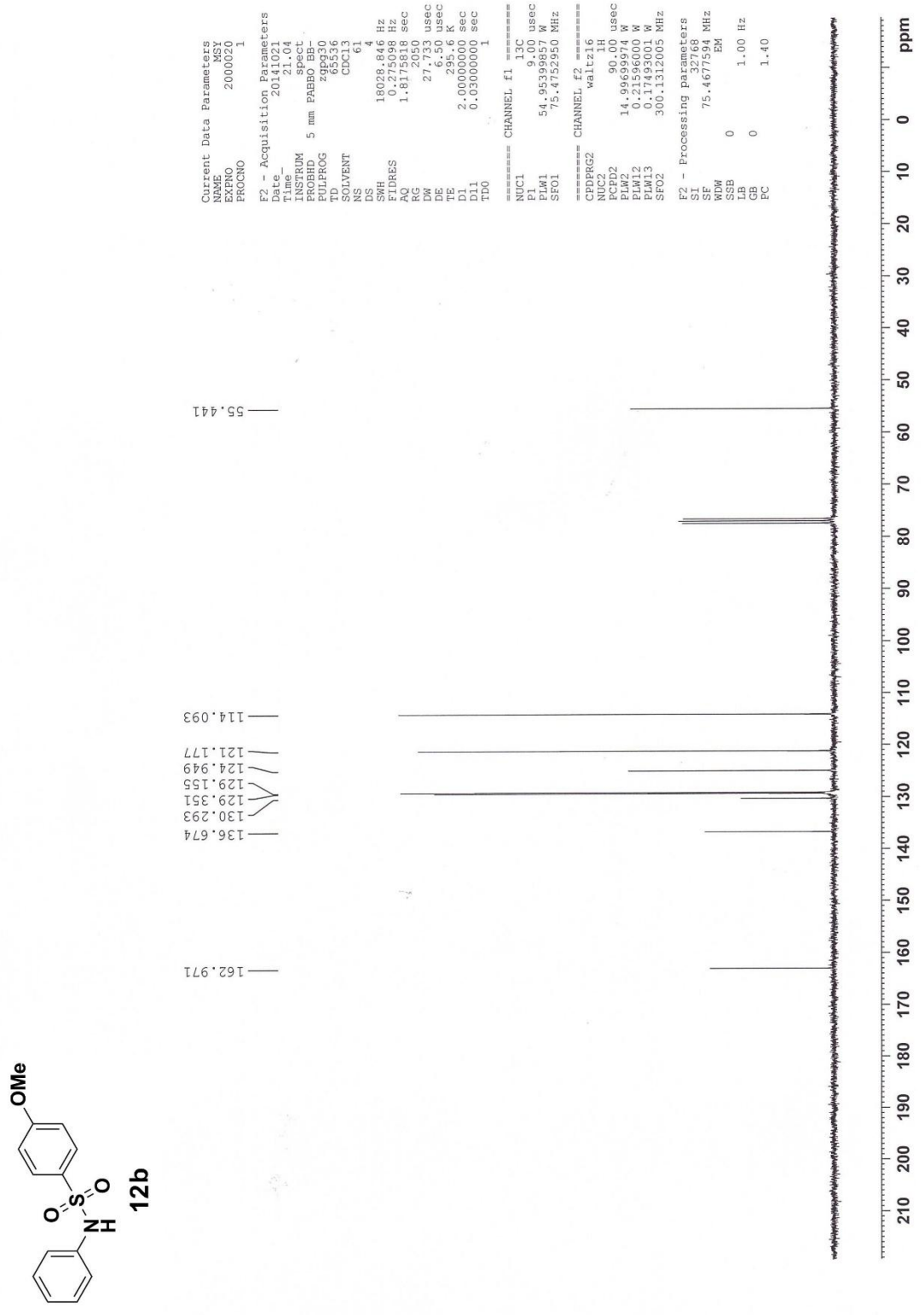

Current Data Parameters
NAME MSY
EXPNO 2000114
PROCNO 1
F2 - Acquisition Parameters
Date_ 20150106
Time 19.43
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1125
DS 4
SWH 18028.846 Hz
FIDRES 0.275098 Hz
AQ 1.8175818 sec
RG 1620
DW 27.733 usec
DE 6.50 usec
TE 295.5 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.00 usec
PLW1 54.95399857 W
SFO1 75.4752950 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PLW2 14.99699974 W
PLW12 0.21596000 W
PLW13 0.17493001 W
SFO2 300.1312005 MHz
F2 - Processing parameters
SI 32768
SF 75.4677615 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
27.700
31.150
34.595
84.605
126.067
128.835
128.896
129.917
133.240
138.151
139.869
150.783
152.061
ppm
11
Cl
O
S
O
N
Boc

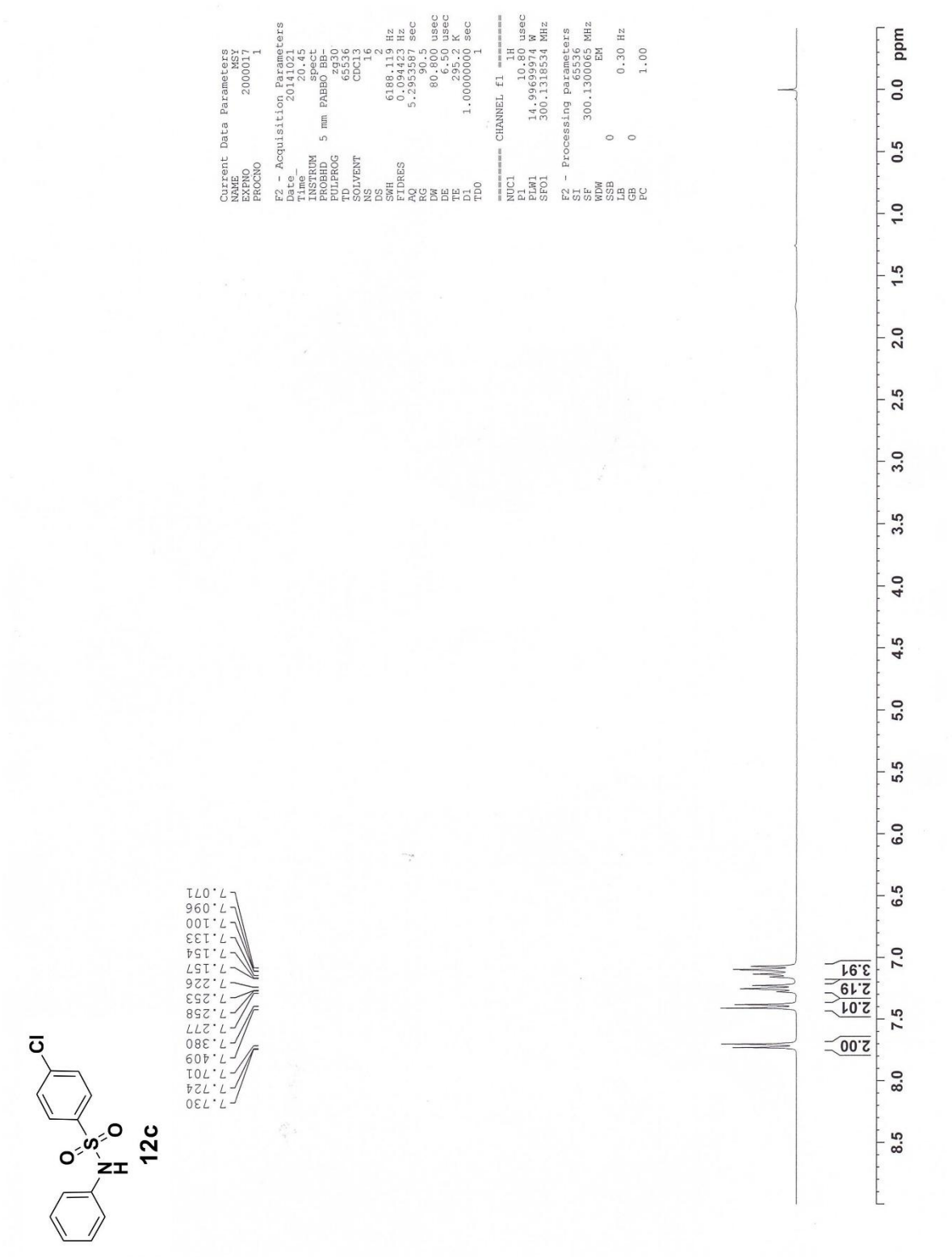

12a
7.685
7.657
7.258
7.229
7.203
7.116
7.094
7.089
7.066
6.912
2.368
3.00
1.89
4.14
3.17
0.68
ppm

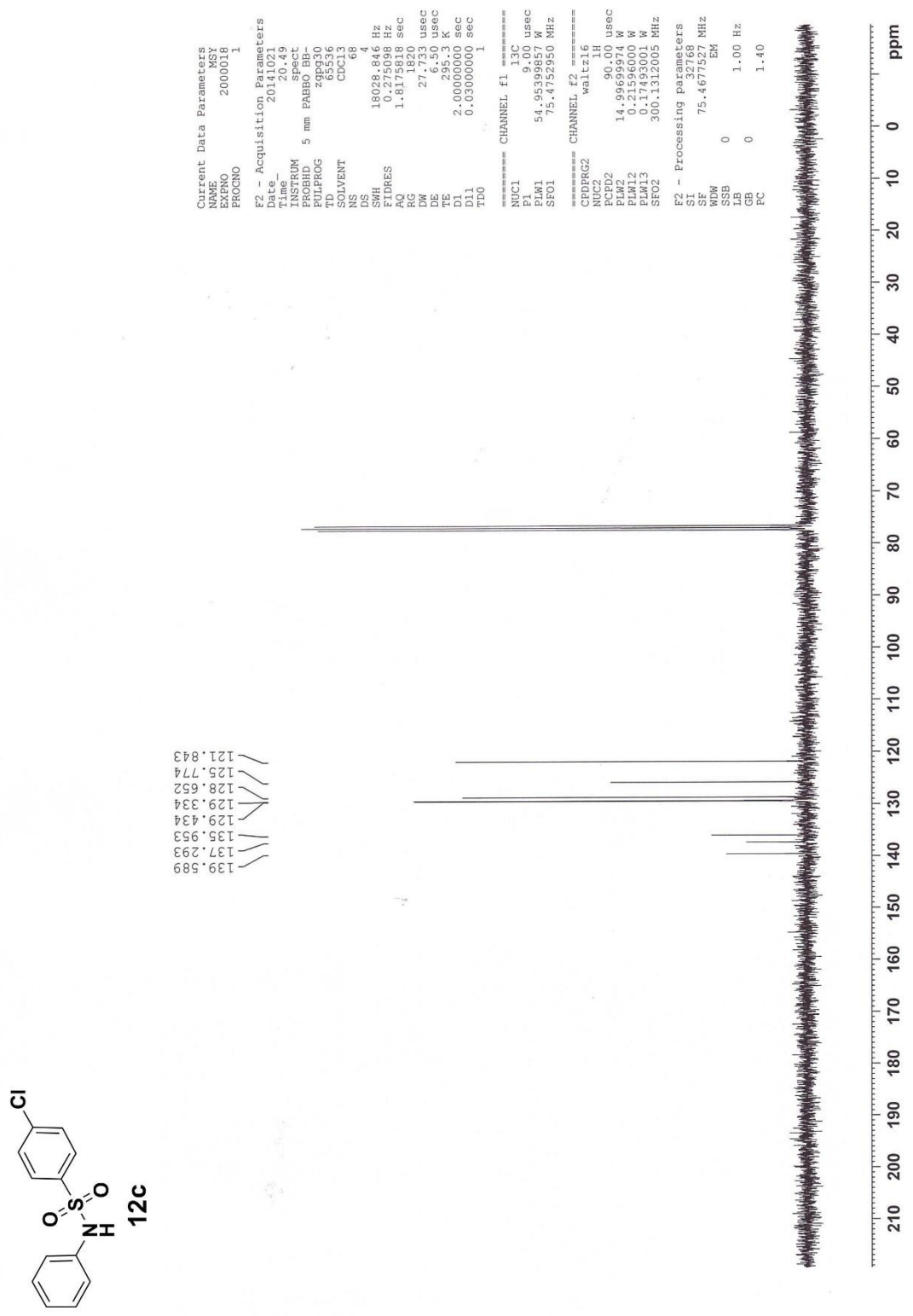

Current Data Parameters
NAME MSY
EXPNO 2000009
PROCNO 1
F2 - Acquisition Parameters
Date_ 20141020
Time 17.50
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 276
DS 4
SWH 18028.846 Hz
FIDRES 0.275098 Hz
AQ 1.8171205 sec
RG 27.733
DW 27.733 usec
DE 6.50 usec
TE 295.4 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.00 usec
PLW1 54.95399857 W
SFO1 75.4752950 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PLW2 14.99699974 W
PLW12 0.21596000 W
PLW13 0.17493001 W
SFO2 300.1312005 MHz
F2 - Processing parameters
SI 32768
SF 75.4677519 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
21.508
121.514
125.275
127.245
129.271
129.620
135.993
136.518
143.856
ppm

12a

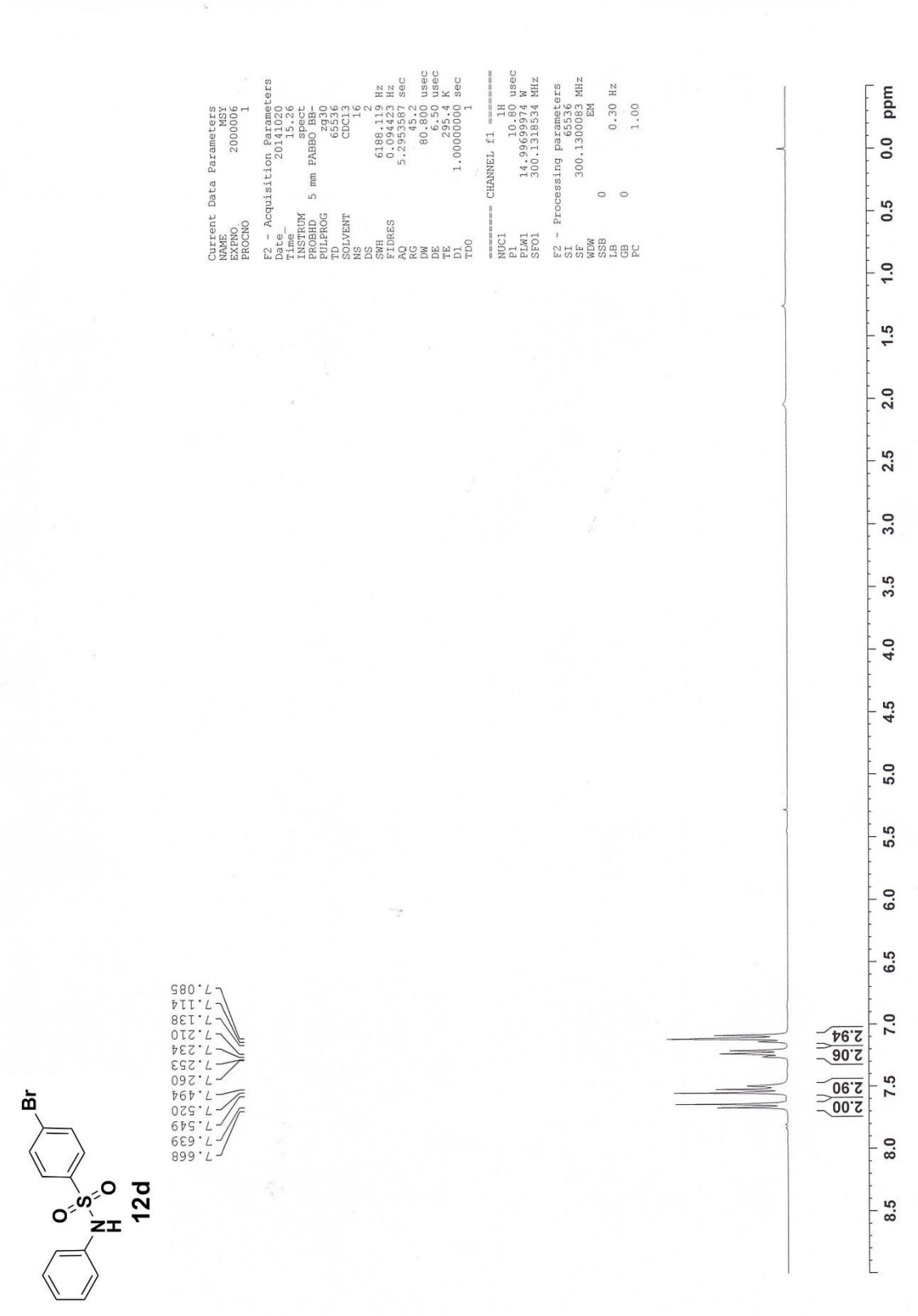

OMe
12b
7.771
7.741
7.607
7.248
7.222
7.195
7.171
7.124
7.099
7.076
7.052
7.028
6.860
6.830
3.759
3.00
2.10
3.18
2.13
0.99
2.09
ppm

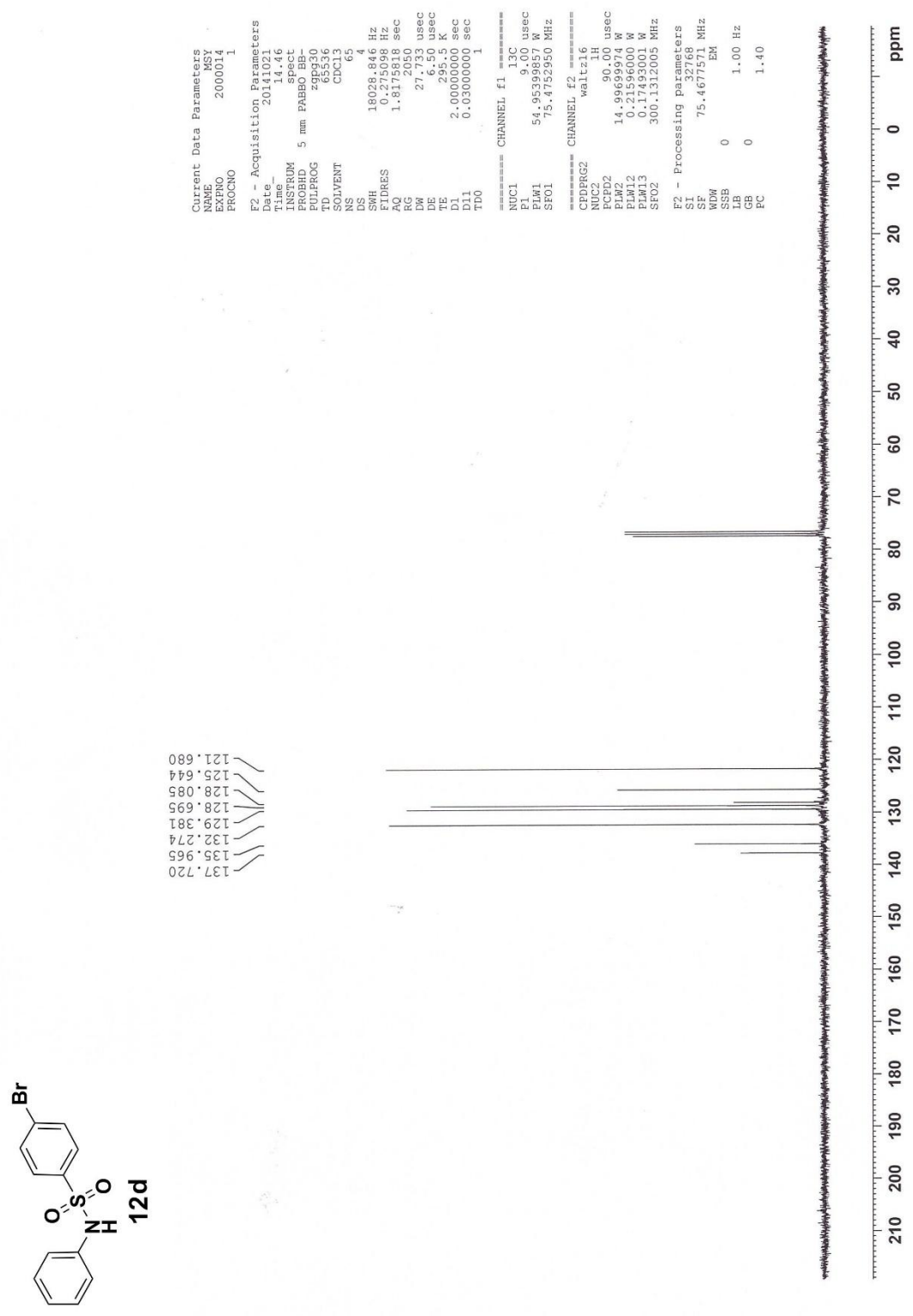

OMe
O
S
O
N
H
12b
Current Data Parameters
NAME MSY
EXPNO 2000020
PROCNO 1
F2 - Acquisition Parameters
Date_ 20141021
Time 21.04
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 61
DS 4
SWH 18028.846 Hz
FIDRES 0.275098 Hz
AQ 1.8175818 sec
RG 2050
DW 27.733 usec
DE 6.50 usec
TE 295.6 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.00 usec
PLW1 54.95399857 W
SFO1 75.4752950 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PLW2 14.99699974 W
PLW12 0.21596000 W
PLW13 0.17493001 W
SFO2 300.1312005 MHz
F2 - Processing parameters
SI 32768
SF 75.4677594 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
55.441
114.093
121.177
124.949
129.155
129.351
130.293
136.674
162.971
ppm

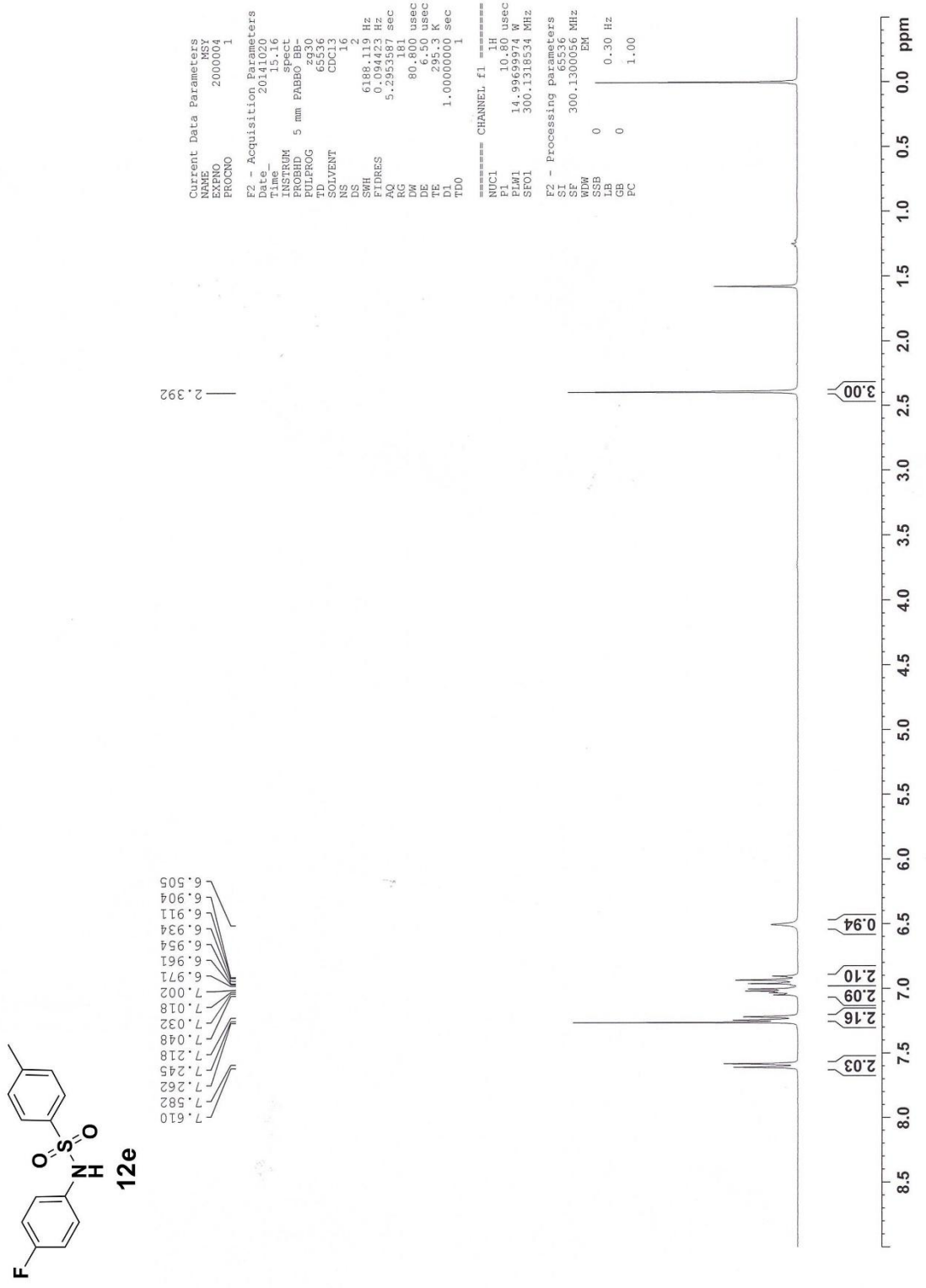

Current Data Parameters
NAME MSY
EXPNO 2000017
PROCNO 1
F2 - Acquisition Parameters
Date_ 20141021
Time_ 20.45
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 6188.119 Hz
FIDRES 0.094423 Hz
AQ 5.2953587 sec
RG 90.5
DW 80.800 usec
DE 6.50 usec
TE 295.2 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 10.80 usec
PLW1 14.99699974 W
SFO1 300.1318534 MHz
F2 - Processing parameters
SI 65536
SF 300.1300065 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
7.730
7.724
7.701
7.409
7.380
7.277
7.258
7.253
7.226
7.157
7.154
7.133
7.100
7.096
7.071
2.00
2.01
2.19
3.91
ppm
12c

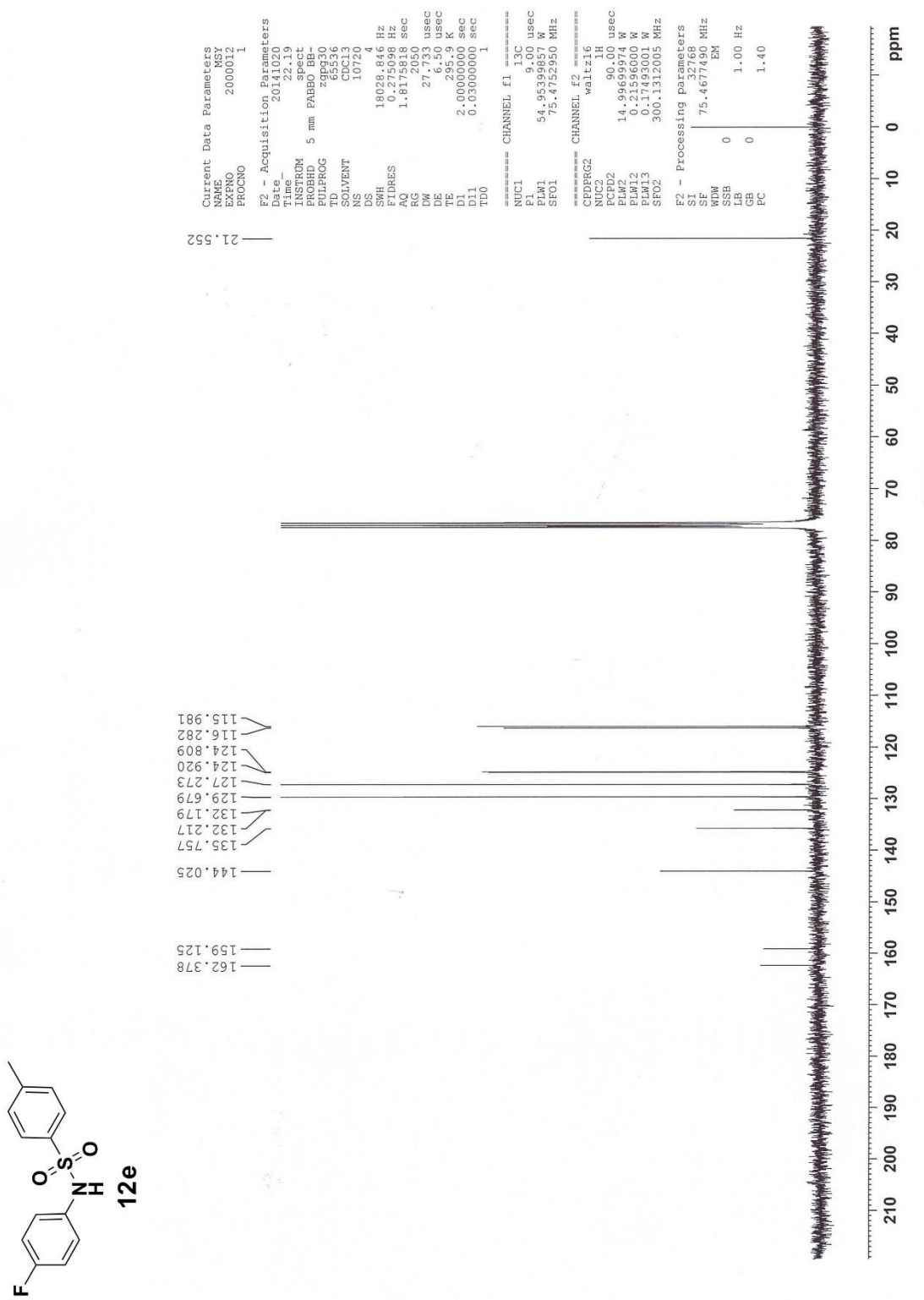

Current Data Parameters
NAME MSY
EXPNO 2000018
PROCNO 1
F2 - Acquisition Parameters
Date_ 20141021
Time 20.49
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 68
DS 4
SWH 18028.846 Hz
FIDRES 0.275098 Hz
AQ 1.8175818 sec
RG 1820
DW 27.733 usec
DE 6.50 usec
TE 295.3 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.00 usec
PLW1 54.95399857 W
SFO1 75.4752950 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PLW2 14.99699974 W
PLW12 0.21596000 W
PLW13 0.17493001 W
SFO2 300.1312005 MHz
F2 - Processing parameters
SI 32768
SF 75.4677527 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
139.589
137.293
135.953
129.434
129.334
128.652
125.774
121.843
ppm

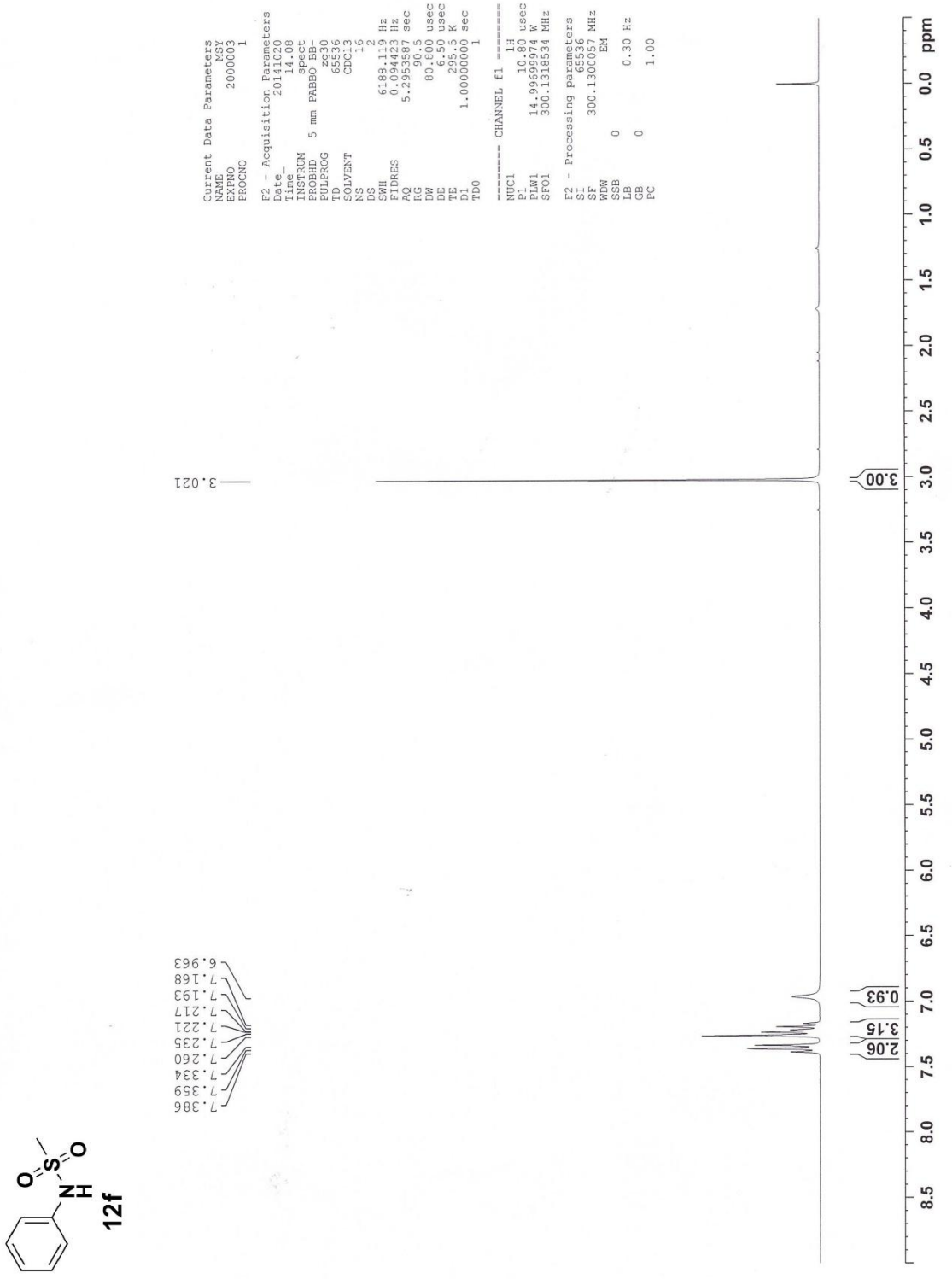
Cl
O
S
O
N
H
12c

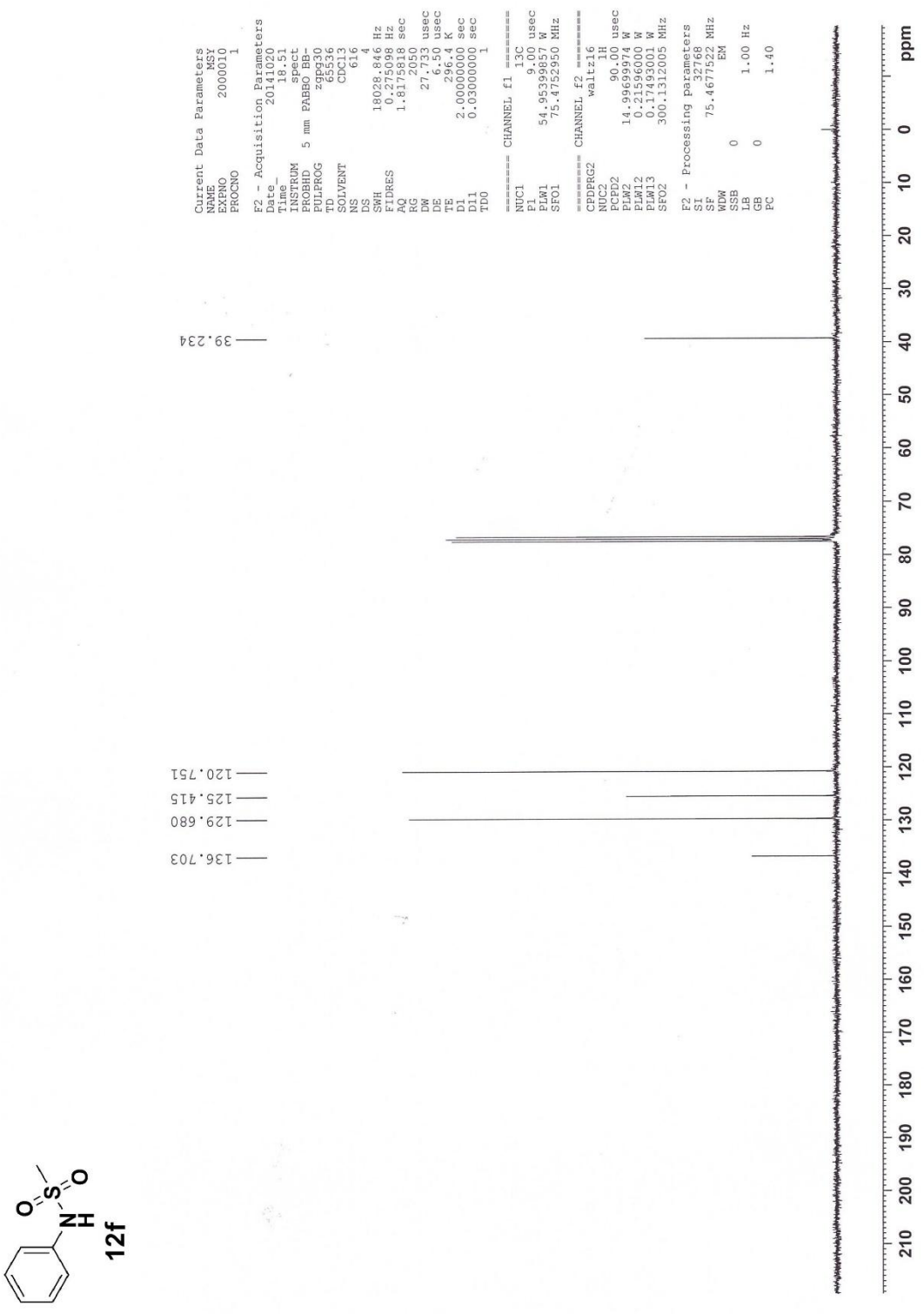

Current Data Parameters
NAME MSY
EXPNO 2000006
PROCNO 1
F2 - Acquisition Parameters
Date_ 20141020
Time_ 15.26
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 6188.119 Hz
FIDRES 0.094423 Hz
AQ 5.2953587 sec
RG 45.2
DW 80.800 usec
DE 6.50 usec
TE 295.4 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 10.80 usec
PLW1 14.99699974 W
SFO1 300.1318534 MHz
F2 - Processing parameters
SI 65536
SF 300.1300083 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
7.668
7.639
7.549
7.520
7.494
7.260
7.253
7.234
7.210
7.138
7.114
7.085
2.00
2.90
2.06
2.94
Br
O
S
O
N
H
O
12d
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm


Br
O
S
O
N
H
O
12d
137.720
135.965
132.274
129.381
128.695
128.085
125.644
121.680
Current Data Parameters
NAME MSY
EXPNO 2000014
PROCNO 1
F2 - Acquisition Parameters
Date_ 20141021
Time_ 14.46
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 65
DS 4
SWH 18028.846 Hz
FIDRES 0.275048 Hz
AQ 1.8171205 sec
RG 205
DW 27.733 usec
DE 6.50 usec
TE 295.5 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.00 usec
PLW1 54.95399857 W
SFO1 75.4752950 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PLW2 14.99699974 W
PLW12 0.21596000 W
PLW13 0.17493001 W
SFO2 300.1312005 MHz
F2 - Processing parameters
SI 32768
SF 75.4677571 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
ppm

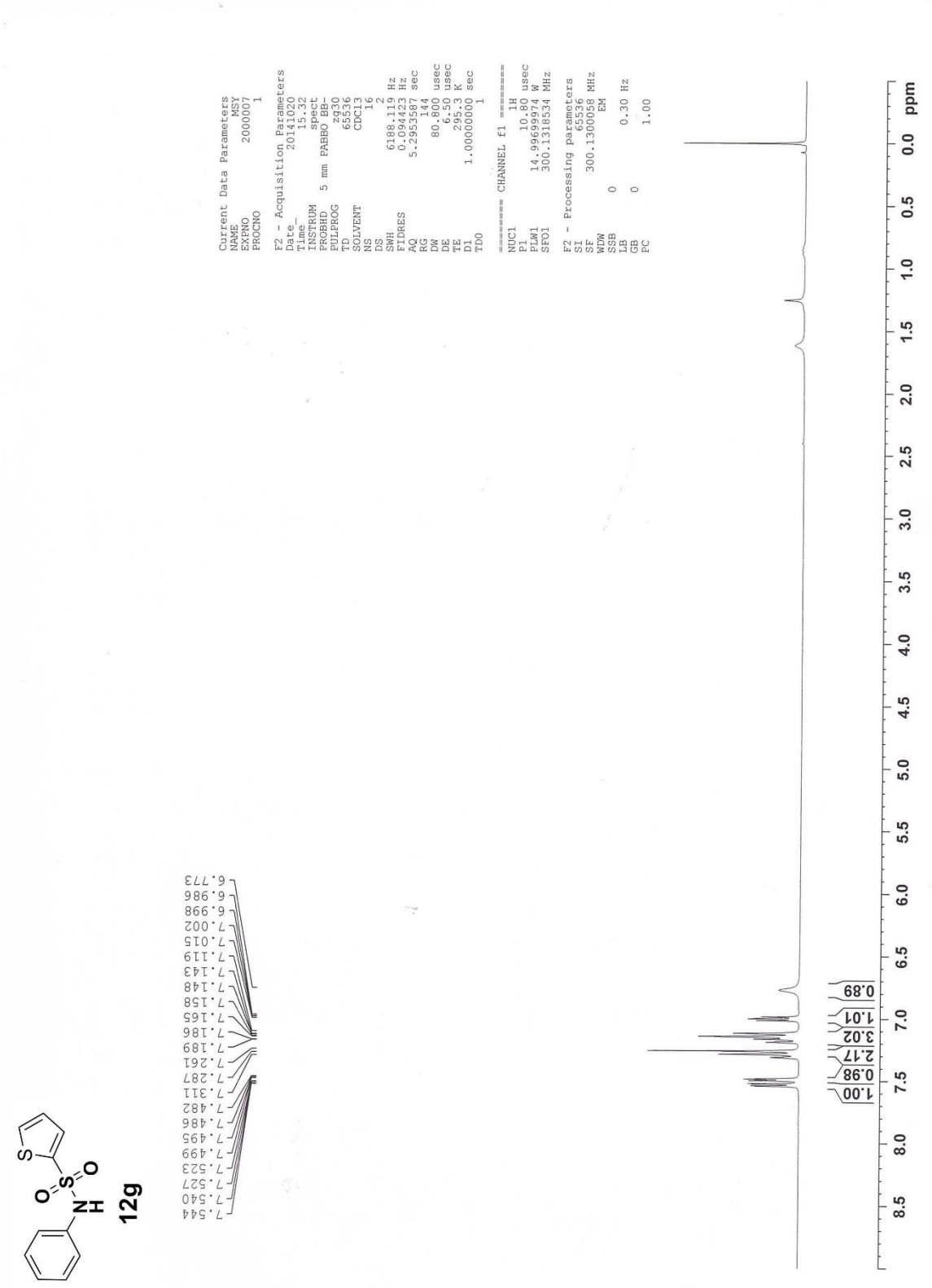

12e
F
O
S
O
N
H
O
7.610
7.582
7.262
7.245
7.218
7.048
7.032
7.018
7.002
6.971
6.961
6.954
6.934
6.911
6.904
6.505
2.392
3.00
0.94
2.10
2.09
2.16
2.03
Current Data Parameters
NAME MSY
EXPNO 2000004
PROCNO 1
F2 - Acquisition Parameters
Date_ 20141020
Time 15.16
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 6188.119 Hz
FIDRES 0.094423 Hz
AQ 5.2953587 sec
RG 181
DW 80.800 usec
DE 6.50 usec
TE 295.3 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 10.80 usec
PLW1 14.99699974 W
SFO1 300.1318534 MHz
F2 - Processing parameters
SI 65536
SF 300.1300056 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
ppm

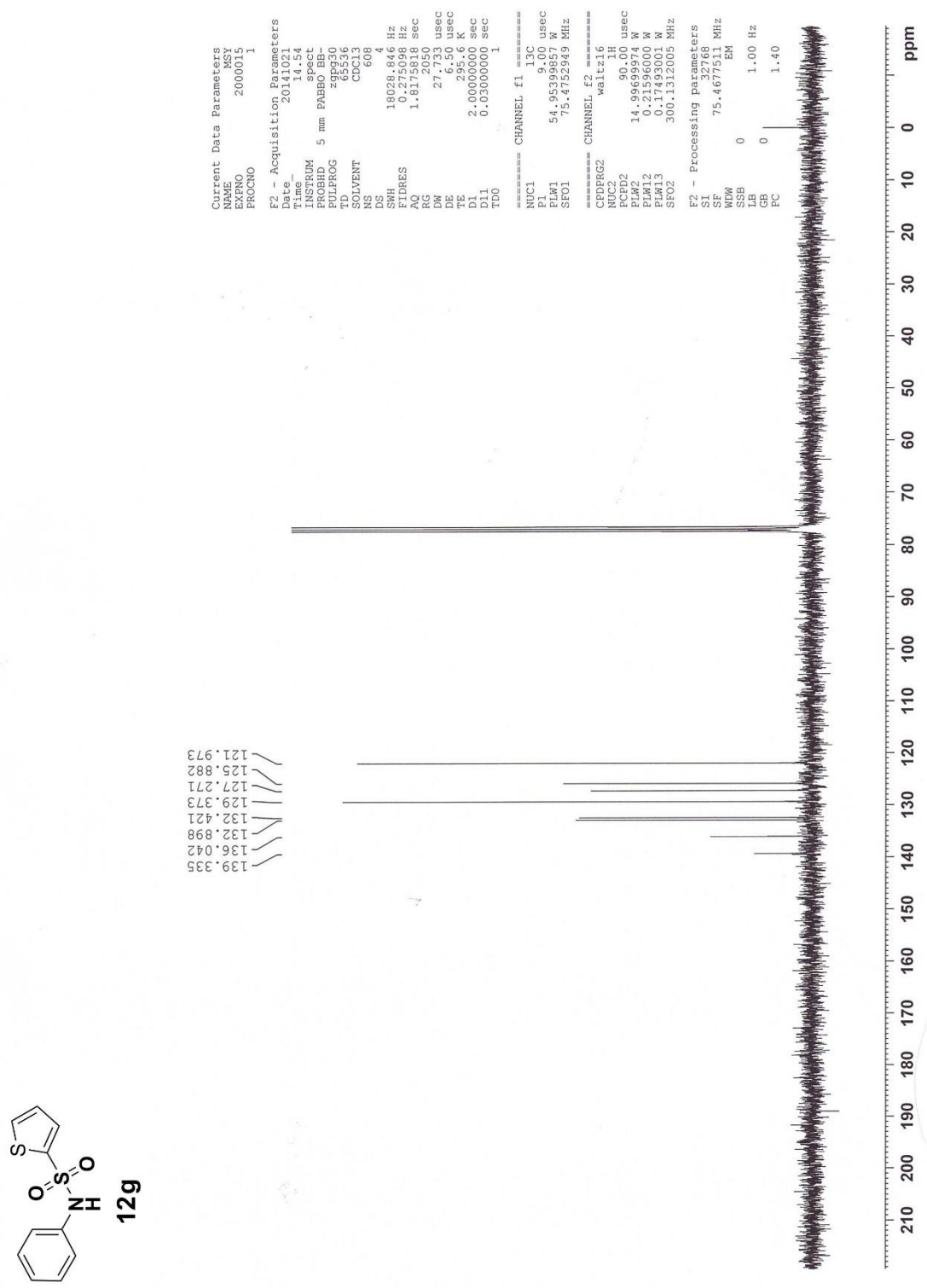

Current Data Parameters
NAME MSY
EXPNO 2000012
PROCNO 1
F2 - Acquisition Parameters
Date_ 20141020
Time_ 22.19
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 10720
DS 4
SWH 18028.846 Hz
FIDRES 0.275098 Hz
AQ 1.8175818 sec
RG 2050
DW 27.733 usec
DE 6.50 usec
TE 295.9 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.00 usec
PLW1 54.95399857 W
SFO1 75.4752950 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PLW2 14.99699974 W
PLW12 0.21596001 W
PLW13 0.17499301 W
SFO2 300.1312005 MHz
F2 - Processing parameters
SI 32768
SF 75.4677490 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
21.552
115.981
116.282
124.809
124.920
127.273
129.679
132.179
132.217
135.757
144.025
159.125
162.378
ppm
12e

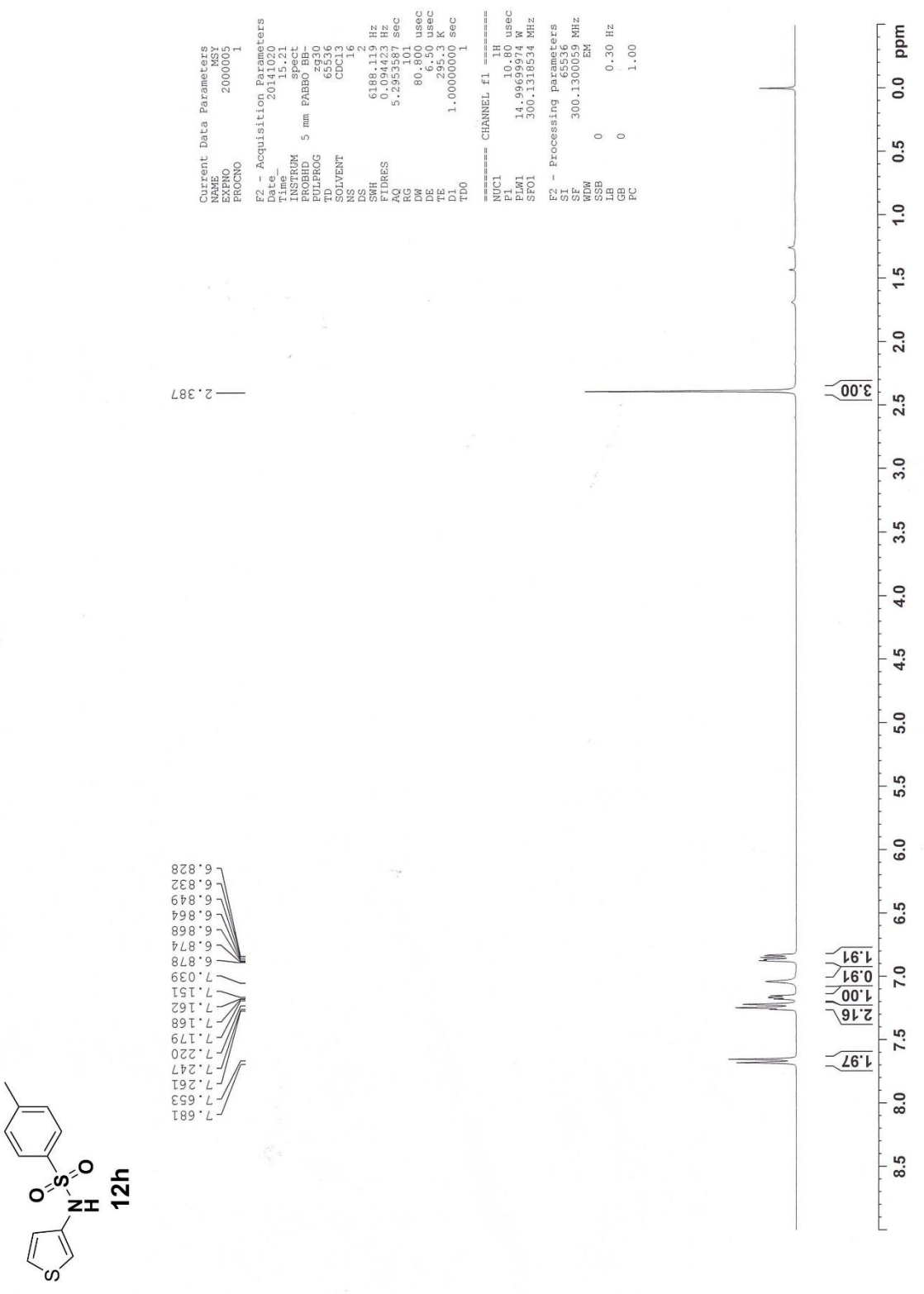

12f
7.386
7.359
7.334
7.260
7.235
7.221
7.217
7.193
7.168
6.963
3.021
3.00
0.93
3.15
2.06
ppm

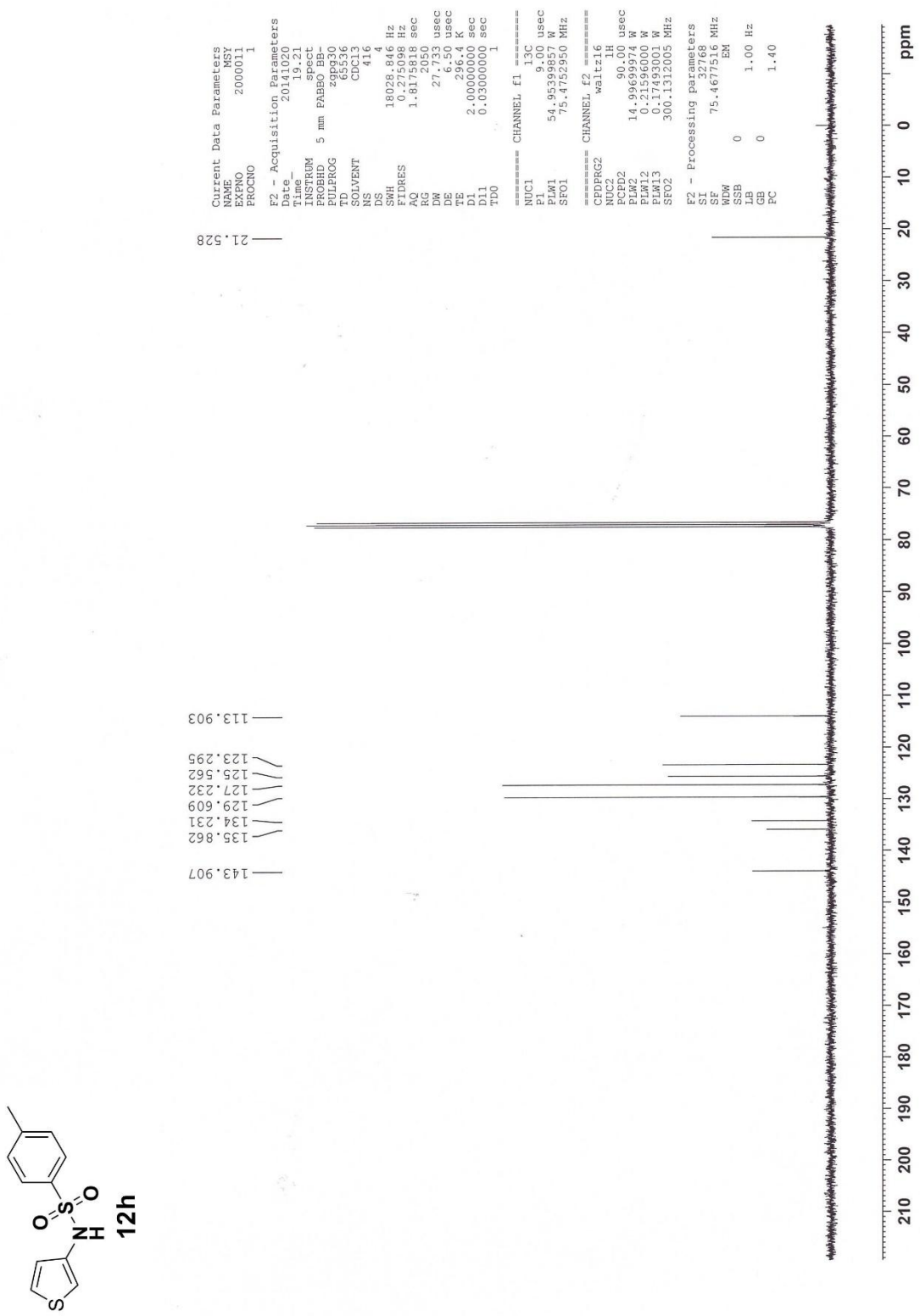

Current Data Parameters
NAME MSY
EXPNO 2000010
PROCNO 1
F2 - Acquisition Parameters
Date_ 20141020
Time 18.51
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 616
DS 4
SWH 18028.846 Hz
FIDRES 0.275098 Hz
AQ 1.8175518 sec
RG 203
DW 27.733 usec
DE 6.50 usec
TE 296.4 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.00 usec
PLW1 54.95399857 W
SFO1 75.4752950 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PLW2 14.99699974 W
PLW12 0.21596000 W
PLW13 0.17493001 W
SFO2 300.1312005 MHz
F2 - Processing parameters
SI 32768
SF 75.4677522 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
39.234
120.751
125.415
129.680
136.703
ppm

12f


Current Data Parameters
NAME MSY
EXPNO 2000007
PROCNO 1
F2 - Acquisition Parameters
Date_ 20141020
Time 15.32
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 6188.119 Hz
FIDRES 0.094423 Hz
AQ 5.2953587 sec
RG 144
DW 80.800 usec
DE 6.50 usec
TE 295.3 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 10.80 usec
PLW1 14.99699974 W
SFO1 300.1318534 MHz
F2 - Processing parameters
SI 65536
SF 300.1300058 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
7.544
7.540
7.527
7.523
7.499
7.495
7.486
7.482
7.311
7.287
7.261
7.189
7.186
7.165
7.158
7.148
7.143
7.119
7.015
7.002
6.998
6.986
6.773
1.00
0.98
2.17
3.02
1.01
0.89
ppm
12g


12g
139.335
136.042
132.898
132.421
129.373
127.271
125.882
121.973
Current Data Parameters
NAME MSY
EXPNO 2000015
PROCNO 1
F2 - Acquisition Parameters
Date_ 20141021
Time 14.54
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 608
DS 4
SWH 18028.846 Hz
FIDRES 0.275098 Hz
AQ 1.8175055 sec
RG 27.733
DW 27.733 usec
DE 6.50 usec
TE 295.6 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.00 usec
PLW1 54.95399857 W
SFO1 75.4752949 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PLW2 14.99699974 W
PLW12 0.21596000 W
PLW13 0.17493001 W
SFO2 300.1312005 MHz
F2 - Processing parameters
SI 32768
SF 75.4677511 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
ppm


12h
7.681
7.653
7.261
7.247
7.220
7.179
7.168
7.162
7.151
7.039
6.878
6.874
6.868
6.864
6.849
6.832
6.828
2.387
3.00
1.97
2.16
1.00
0.91
1.91
ppm
Current Data Parameters
NAME MSY
EXPNO 2000005
PROCNO 1
F2 - Acquisition Parameters
Date_ 20141020
Time 15.21
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 6188.119 Hz
FIDRES 0.094423 Hz
AQ 5.2953367 sec
RG 101
DW 80.600 usec
DE 6.50 usec
TE 295.3 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 10.80 usec
PLW1 14.99699974 W
SFO1 300.1318534 MHz
F2 - Processing parameters
SI 65536
SF 300.1300059 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00


Current Data Parameters
NAME MSY
EXPNO 2000011
PROCNO 1
F2 - Acquisition Parameters
Date_ 20141020
Time 19.21
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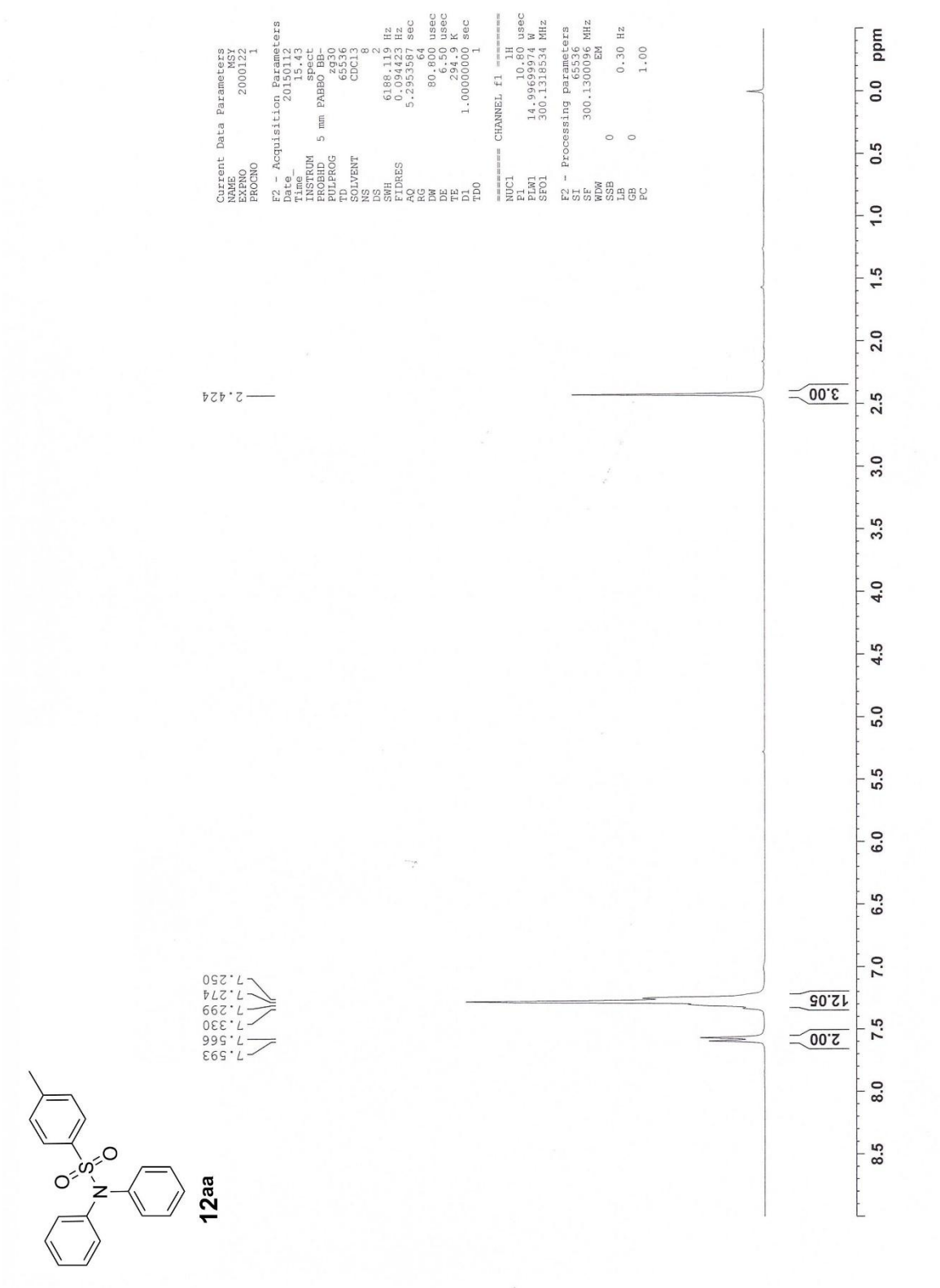
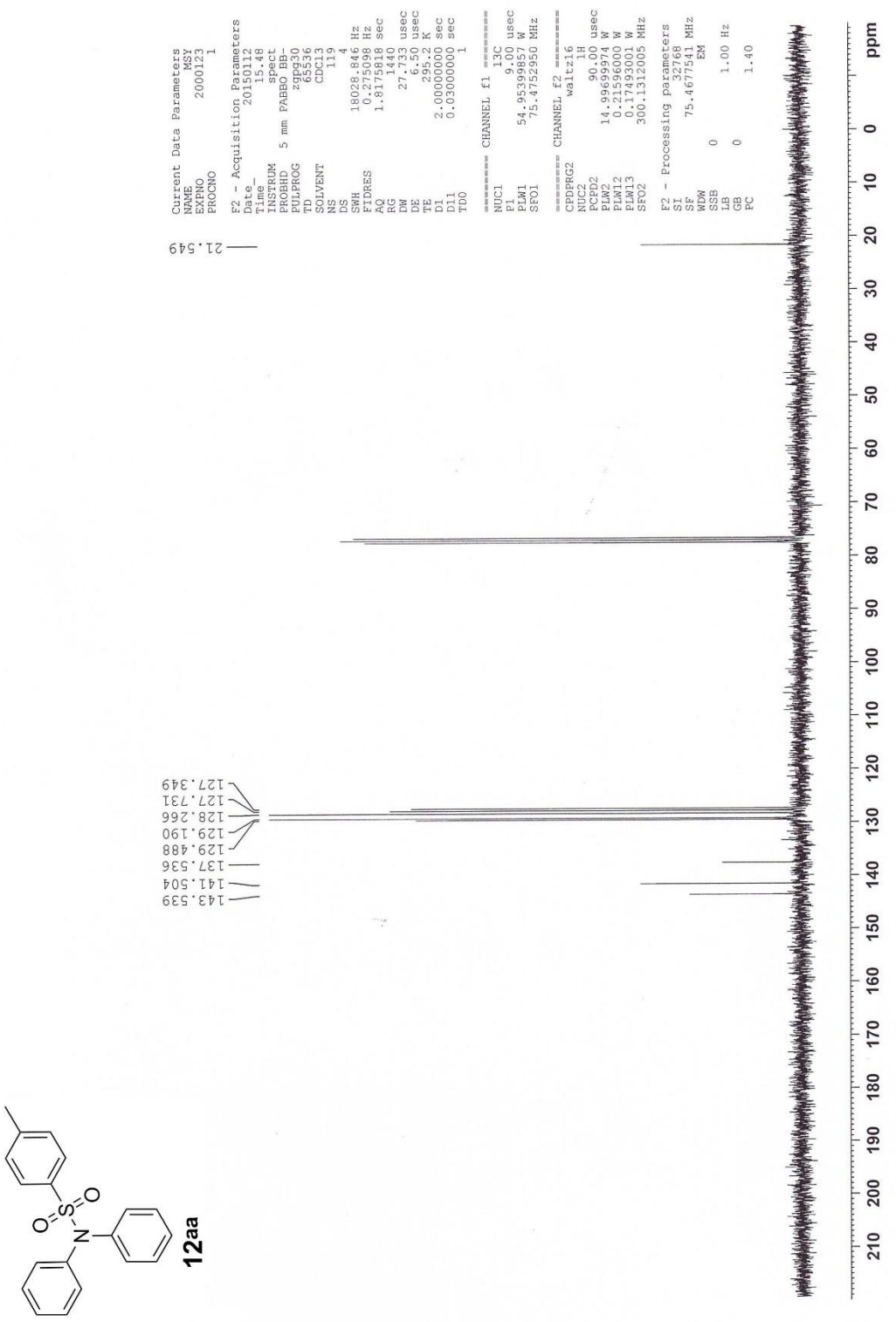
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 416
DS 4
SWH 18028.846 Hz
FIDRES 0.275098 Hz
AQ 1.8175818 sec
RG 2050
DW 27.733 usec
DE 6.50 usec
TE 296.4 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.00 usec
PLW1 54.95399857 W
SFO1 75.4752950 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PLW2 14.99699974 W
PLW12 0.21596000 W
PLW13 0.17493001 W
SFO2 300.1312005 MHz
F2 - Processing parameters
SI 32768
SF 75.4677516 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
21.528
113.903
123.295
125.562
127.232
129.609
134.231
135.862
143.907
ppm
12h


12aa
7.593
7.566
7.330
7.299
7.274
7.250
2.424
3.00
12.05
2.00
ppm


Current Data Parameters
NAME MSY
EXPNO 2000123
PROCNO 1
F2 - Acquisition Parameters
Date_ 20150112
Time 15.48
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 119
DS 4
SWH 18028.846 Hz
FIDRES 0.275098 Hz
AQ 1.8175818 sec
RG 1440
DW 27.733 usec
DE 6.50 usec
TE 295.2 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
CHANNEL f1
NUC1 13C
P1 9.00 usec
PLW1 54.95399857 W
SFO1 75.4752950 MHz
CHANNEL f2
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PLW2 14.99699974 W
PLW12 0.21596000 W
PLW13 0.17493001 W
SFO2 300.1312005 MHz
F2 - Processing parameters
SI 32768
SF 75.4677541 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
21.549
127.349
127.731
128.266
129.190
129.488
137.536
141.504
143.539
ppm
12aa