



A Publication  
of Reliable Methods  
for the Preparation  
of Organic Compounds

## Working with Hazardous Chemicals

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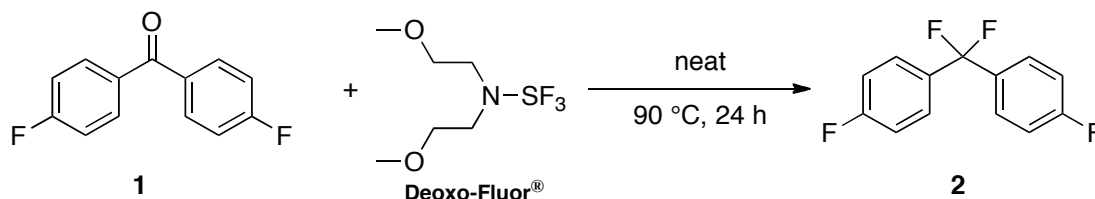
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*September 2014: The paragraphs above replace the section "Handling and Disposal of Hazardous Chemicals" in the originally published version of this article. The statements above do not supersede any specific hazard caution notes and safety instructions included in the procedure.*

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**DIRECT FLUORINATION OF THE CARBONYL GROUP OF  
BENZOPHENONES USING DEOXO-FLUOR<sup>®</sup>: PREPARATION OF  
BIS(4-FLUOROPHENYL)DIFLUOROMETHANE**



Submitted by Ying Chang, Hyelee Lee, Chulsung Bae.<sup>1</sup>

Checked by David Hughes.

### 1. Procedure

*Caution! Deoxo-Fluor<sup>®</sup> reacts rapidly and exothermally with water, generating HF. It is volatile and a respiratory hazard and must be handled in a well-ventilated hood.*

*Bis(4-fluorophenyl)difluoromethane.* An oven-dried 50-mL round-bottomed flask equipped with an oval Teflon-coated magnetic stirring bar (2 cm) is charged with 4,4'-difluorobenzophenone (**1**) (5.11 g, 23.4 mmol, 1.0 equiv) (Note 1). Deoxo-Fluor<sup>®</sup> (Note 2) (13 mL, 15.7 g, 71 mmol, 3 equiv) is added via a disposable graduated pipette. The flask is fitted with a reflux condenser equipped with a gas inlet adapter connected to a nitrogen line and a gas bubbler. The reaction solution is stirred in a preheated 90 °C oil bath (Notes 3, 4, and 5) for 24 h under nitrogen to give a dark red mixture with yellow solids. The flask is removed from the oil bath and cooled to room temperature. Dichloromethane (30 mL) is added and the mixture is transferred to a 250-mL separatory funnel, followed by additional dichloromethane (2 × 30 mL) to rinse the flask. The organic solution is washed with water (2 × 50 mL) (Note 6), then saturated aqueous NaHCO<sub>3</sub> solution (50 mL) (**caution:** Due to the generation of CO<sub>2</sub>, the separatory funnel should be shaken carefully and the pressure released frequently). A final wash is carried out with saturated aqueous NaCl solution (50 mL), then the organic layer is vacuum-filtered through a bed of Na<sub>2</sub>SO<sub>4</sub> (20 g) in a 150-mL medium-porosity sintered-glass funnel. The cake is washed with dichloromethane (3 × 25 mL) until colorless. The filtrate is concentrated by

rotary evaporation (40 °C bath, 100 mmHg initial vacuum, lowered to 20 mmHg) to afford the crude product as a red oil (6.9 g). Purification using column chromatography (Note 7) on SiO<sub>2</sub> affords 4.42–4.87 g of bis(4-fluorophenyl)difluoromethane (**2**) as a colorless oil (78–86% yield) (Notes 8 and 9).

## 2. Notes

1. Reagents and solvents were used as received and sourced as follows: 4,4'-difluorobenzophenone (Acros, 99%), bis(2-methoxyethyl)aminosulfur trifluoride (Deoxo-Fluor<sup>®</sup>) (Sigma-Aldrich), dichloromethane (Sigma-Aldrich, ACS reagent, 99.5%), silica gel (Sigma-Aldrich, 230-400 mesh, 60 Å), ethyl acetate (Sigma-Aldrich, ACS reagent, >99.5%), hexanes (Sigma-Aldrich, ACS reagent, >98.5%), and sodium sulfate (Fisher Scientific).

2. Deoxo-Fluor<sup>®</sup> is volatile and a respiratory hazard and must be handled in a well-ventilated hood. The checker pipetted Deoxo-Fluor<sup>®</sup> in the hood and weighed the stoppered flask before and after addition.

3. Deoxo-Fluor<sup>®</sup> is known to decompose initiating at 140 °C.<sup>2</sup> Thus, the oil bath temperature must be kept below 110 °C for safety concerns. A 90 °C oil bath temperature provides efficient fluorination; raising the oil bath temperature to 100 °C does not improve the yield. For substrates with high molecular weight, such as 4,4'-dibromobenzophenone, the stirring may be difficult at the beginning, but this does not affect the reaction yield as the stirring gradually becomes more efficient during the course of the reaction.

4. In a separate experiment, a 2.5 g reaction was carried out in 2-necked, 50-mL flask with a thermocouple thermometer inserted through a septum. The internal temperature was monitored (84 °C) using a J-Kem Gemini digital thermometer with a Teflon-coated T-Type thermocouple probe (12-inch length, 1/8 inch outer diameter, temperature range –200 to +250 °C). This reaction proceeded to 84% conversion with a 78% isolated yield.

5. The reaction was monitored by <sup>1</sup>H NMR as follows. A drop of the reaction mixture was added to 1 mL of CDCl<sub>3</sub> and 1 mL of sat. NaHCO<sub>3</sub>. The layers were mixed, then the bottom layer was filtered into an NMR tube through Na<sub>2</sub>SO<sub>4</sub> and a cotton plug. Multiplets at 7.20 and 7.85 ppm from the starting material were integrated relative to the product resonances at 7.1 and 7.5 ppm to assess conversion. The reaction proceeded to 84–90% conversion

for experiments carried out at the 2.5-5 g scale. Additional reaction time did not result in increased conversion. Addition of one equiv of Deoxo-Fluor<sup>®</sup> at the end of the reaction and heating for a further 24 h only increased conversion by 2-3%.

6. Slight foams are produced due to the remaining Deoxo-Fluor<sup>®</sup> or its decomposition products.

7. A 5-cm glass column is wet-packed (2.5% hexanes/EtOAc) with SiO<sub>2</sub> (200 g) topped with 0.5 cm sand. The crude reaction product is loaded neat onto the column and eluted as follows: 2.5% EtOAc/hexanes (600 mL), 3% EtOAc/hexanes (750 mL), collecting 50-mL fractions. TLC (UV visualization) is used to follow the chromatography. The R<sub>f</sub> value of the title compound is 0.5 (2.5% EtOAc/hexanes), the starting material has an R<sub>f</sub> of 0.05. Fractions 8-21 are concentrated by rotary evaporation (40 °C bath, 20 mmHg), then vacuum dried (20 mmHg) at 22 °C for 2 h to constant weight (4.42–4.87 g, 78–86% yield). The product contains <0.1 wt % EtOAc and hexanes by <sup>1</sup>H NMR analysis. Fractions 25-29 are combined and concentrated to afford 0.50–0.8 g (10–16%) of unreacted starting material **1**.

8. The checker obtained the following yields from three experiments:

Scale ( <b>1</b> )	Isolated product <b>2</b> (%)	Unreacted starting material ( <b>1</b> ) (%)
5.11 g	4.86 g (86%)	0.50 g (10%)
3.88 g	3.56 g (83%)	0.49 (13%)
2.56 g	2.19 g (78%)	0.41 (16%)

9. *Bis(4-fluorophenyl)difluoromethane* (**2**) has the following physical and spectroscopic data: <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ: 7.09-7.14 (m, 4 H), 7.46-7.51 (m, 4 H); <sup>19</sup>F NMR (376 MHz, CDCl<sub>3</sub>) δ: -110.7 (t, <sup>6</sup>J<sub>FF</sub> = 3 Hz, 2 F, *Ar-F*), -86.3 (t, <sup>6</sup>J<sub>FF</sub> = 3 Hz, 2 F, *CF*<sub>2</sub>); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ: 115.5 (d, <sup>2</sup>J<sub>CF</sub> = 22 Hz), 120.1 (t, <sup>1</sup>J<sub>CF</sub> = 242 Hz), 128.1 (dt, <sup>3</sup>J<sub>CF</sub> = 9.0 Hz, <sup>3</sup>J<sub>CF</sub> = 5.2 Hz), 133.5 (td, <sup>2</sup>J<sub>CF</sub> = 29 Hz, <sup>4</sup>J<sub>CF</sub> = 3 Hz), 163.6 (dt, <sup>1</sup>J<sub>CF</sub> = 249 Hz, <sup>5</sup>J<sub>CF</sub> = 2 Hz); MS (EI) *m/z* 240 (M<sup>+</sup>, 48), 221 (16), 145 (100), 126 (16), 95 (18), 75 (17). GC/MS (Shimadzu QP2010S equipped with a 30 m × 0.25 mm SHR-XLB GC column and an EI ionization MS detector) indicated product purity >99%. An analytical sample was prepared by dissolving 100 mg of product in 3 mL of hexanes, filtering through a 0.45 micron PTFE syringe filter, and vacuum concentration for 3 h at ambient temperature. Anal. calcd. for C<sub>13</sub>H<sub>8</sub>F<sub>4</sub>: C, 65.01; H, 3.36; found: C, 64.77; H, 3.32.

## Safety and Waste Disposal Information

All hazardous materials should be handled and disposed of in accordance with "Prudent Practices in the Laboratory"; National Academy Press; Washington, DC, 1995.

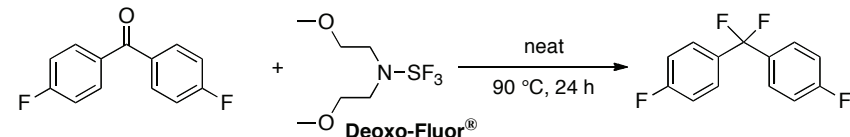
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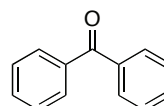
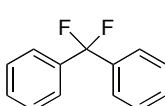
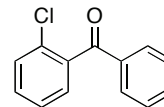
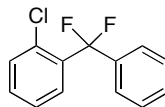
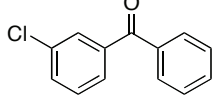
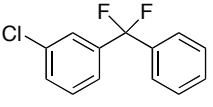
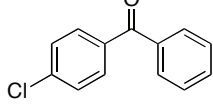
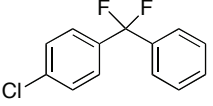
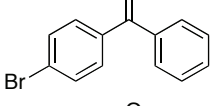
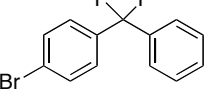
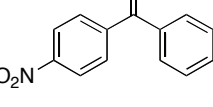
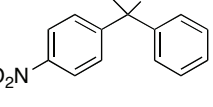
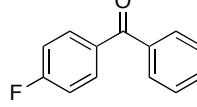
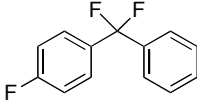
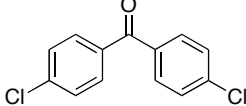
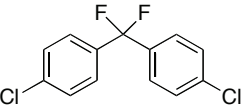
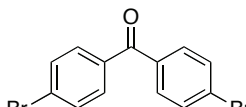
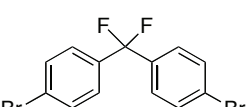
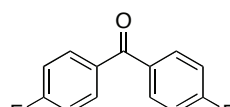
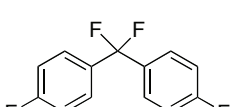
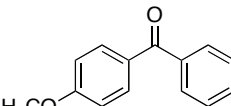
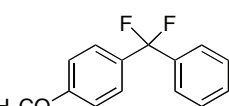
Selective fluorination of carbonyl groups to the *gem*-difluorides is a useful transformation that has been traditionally achieved by the use of gaseous sulfur tetrafluoride<sup>3</sup> and diethylaminosulfur trifluoride (DAST)<sup>4</sup>; however, the harsh reaction conditions and the toxicity of sulfur tetrafluoride and the thermal instability of DAST have limited their use in large-scale reactions. Deoxo-Fluor<sup>®</sup> is known to be more thermally stable than DAST.<sup>2,5</sup>

Compared with the well-studied fluorination of the carbonyl group of aldehydes and alkyl ketones, the carbonyl group of diaryl ketones is much less reactive under the general fluorination conditions and requires harsh conditions, which causes safety concerns.<sup>6</sup> Thus, two-step procedures which involve conversion of the carbonyl group of benzophenones to more reactive thioketones or thiolanes were developed for the *gem*-difluorination of benzophenones.<sup>6-7</sup> The direct fluorination procedure of benzophenones with Deoxo-Fluor<sup>®</sup> described here is a modified procedure of our previously reported method.<sup>8</sup> In this work, the fluorination reactions are conducted in a flask equipped with a reflux condenser under a nitrogen atmosphere rather than in a closed system, such as a pressure tube and screw-capped vial. The implementation of this more general experimental procedure allows a more convenient, safer, and scalable preparation of *gem*-difluoride compounds from the corresponding benzophenones.

The fluorination of 4,4'-difluorobenzophenone (1 g scale) with Deoxo-Fluor<sup>®</sup> was monitored by GC/MS and the conversion was improved from 56% to 75% by increasing the reaction time from 4 to 24 h. Considering the high cost of Deoxo-Fluor<sup>®</sup> and the limited solubility of some of the substrates in Deoxo-Fluor<sup>®</sup> under neat conditions, we choose 24 h as the standard reaction time. The effect of the fluorinating reagent ratio on the conversion was investigated using benzophenone (1 g scale) as substrate. When 1.4 equiv of Deoxo-Fluor<sup>®</sup> was used, the corresponding *gem*-difluoride was isolated in 27% yield. Increasing the amount of Deoxo-Fluor<sup>®</sup>

**Table 1.** Conversion of Diaryl Ketones to Diaryldifluoromethanes<sup>a</sup>



Entry	Substrate	Product	Yield <sup>b</sup>
1			63
2			20
3			75
4			83
5			77
6			95
7			68
8			85
9			61
10			74, 86 <sup>c</sup>
11			13

<sup>a</sup> Reactions were conducted in an oven-dried 10 mL one-necked round-bottomed flask using 1.0 g of benzophenone substrate and 3 equiv of Deoxo-Fluor®. <sup>b</sup> Isolated yield; remainder of mass balance is unreacted starting material. <sup>c</sup> Yield from a 5.0 g scale reaction.

to 3 equiv improved the yield to 63%. No significant additional improvement was obtained by a further increase of Deoxo-Fluor<sup>®</sup> to 4 equiv.

The scope of the direct conversion of the carbonyl groups of benzophenones to the *gem*-difluorides was examined with substrates containing halogen, alkoxy, and nitro substituents on one or both aromatic rings (Table 1). The effect of steric hindrance was observed in the *gem*-difluorination of 2-chlorobenzophenone, as only a 20% yield was obtained (entry 2). The electronic effect also plays an important role in the reaction yields: substrates bearing an electron-withdrawing group formed the *gem*-difluorinated products in good to high yields (61–95%, entries 1 and 3–10) while low yields were obtained for benzophenones bearing an electron-donating group such as methoxy (13%, entry 11).

1. Department of Chemistry, University of Nevada Las Vegas, 4505 Maryland Parkway, Box 454003, Las Vegas, Nevada 89154-4003.
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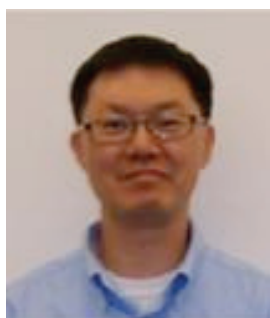
## Appendix

### Chemical Abstracts Nomenclature (Registry Number)

Deoxo-Fluor: Bis(2-methoxyethyl)aminosulfur trifluoride: Ethanamine, 2-ethoxy-N-(2-ethoxyethyl)-N-(trifluorothio)-; (202289-38-1)

Benzophenone: Diphenylmethanone: Benzoylbenzene; (119-61-9)

Bis(4-fluorophenyl) ketone: 4,4'-Difluorobenzophenone; (345-92-6)



Chulsung Bae received his B.S. degree from Inha University and M.S. degrees from Pohang University of Science & Technology in Korea and University of Massachusetts Lowell. He received a Ph.D. in chemistry from University of Southern California in 2002 under the guidance of G. K. Surya Prakash and Nobel Laureate George A. Olah. After postdoctoral research at Yale University with John F. Hartwig in 2002–2004, he joined the faculty in the Chemistry Department at University of Nevada Las Vegas. His current research interests include organic chemistry, organofluorine chemistry, catalysis, green chemistry, polymer science for renewable energy applications.





Ying Chang was born in Anhui Province, China in 1981. He obtained his Ph.D. degree in Applied Chemistry from Nanjing University of Science and Technology, China, in 2006 under the supervision of Professor Chun Cai. After carrying out a postdoctoral research work with Professor William R. Dolbier, Jr. at the University of Florida (2006-2007) on synthetic organofluorine chemistry, he joined Professor Chulsung Bae's group at the University of Nevada Las Vegas as a postdoctoral scholar and now works in the fields of synthetic organofluorine chemistry, functional polymers synthesis for renewable energy applications.



Hyelee Lee was born in Incheon, South Korea in 1984. She received her B.S. degree in Chemistry from the Chung-Ang University in 2008. After working as a research assistant in the Drug Evaluation Department, Korea Food & Drug Administration until summer of 2009, she joined Professor Chulsung Bae's group at University of Nevada Las Vegas as a Ph.D. student. Her current interest is development of new organic synthetic method for the applications in proton exchange membrane fuel cells.

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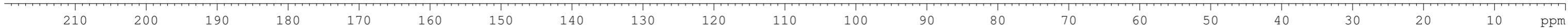
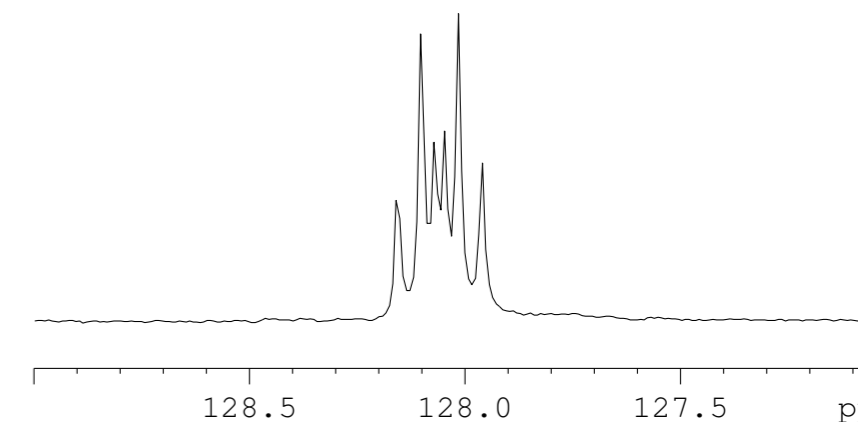
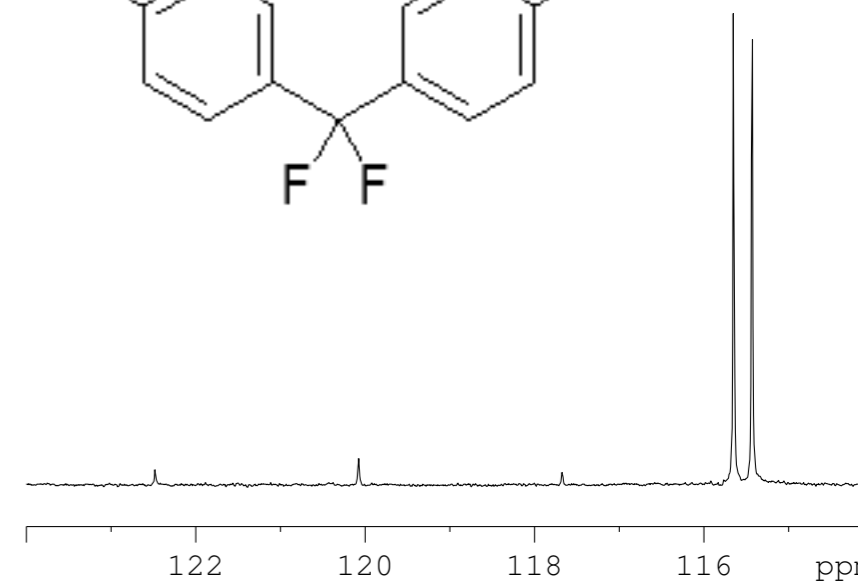
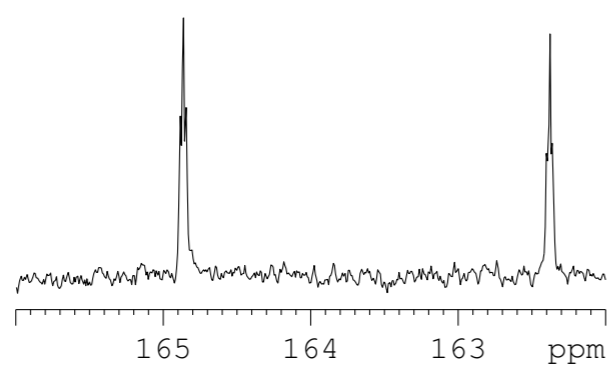
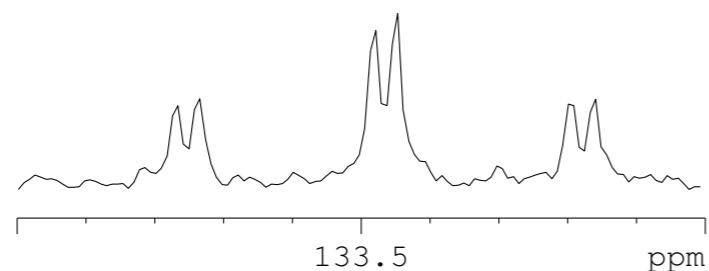
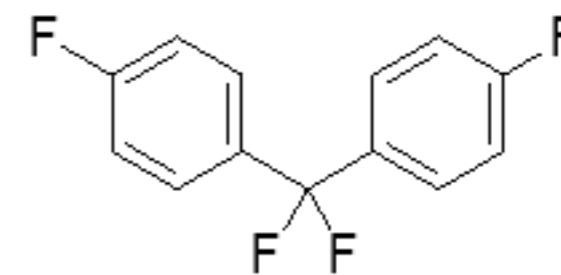
F2 - Processing parameters  
SI 32768  
SF 100.5473910 MHz  
WDW EM  
SSB 0  
LB 1.00 Hz  
GB 0  
PC 1.40

164.89  
164.87  
164.85  
162.38

133.48  
133.45  
128.16  
128.11  
128.07  
128.05  
128.02  
127.96  
120.08  
115.43

77.34  
77.22  
77.02  
76.70

Peak	?(F1) [ppm]	?(F1) [Hz]	Intensity
1	164.8861	16578.8672	0.80
2	164.8659	16576.8361	1.28
3	164.8497	16575.2072	0.83
4	162.3807	16326.9557	1.18
5	133.7723	13450.4558	0.33
6	133.7404	13447.2483	0.36
7	133.4847	13421.5383	0.67
8	133.4525	13418.3007	0.74
9	133.1991	13392.8220	0.35
10	133.1652	13389.4134	0.36
11	128.1604	12886.1938	3.30
12	128.1057	12880.6939	7.61
13	128.0742	12877.5267	4.71
14	128.0511	12875.2040	4.97
15	128.0191	12871.9865	8.05
16	127.9636	12866.4061	4.14
17	122.4828	12315.3260	0.48
18	120.0791	12073.6402	0.84
19	117.6760	11832.0148	0.40
20	115.6537	11628.6778	15.00
21	115.4329	11606.4769	14.27



Current Data Parameters  
NAME 2010-109  
EXPNO 6  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20100227  
Time 10.54  
INSTRUM spect  
PROBHD 5 mm QNP 1H/1  
PULPROG zgfhigqn  
TD 131072  
SOLVENT CDCl3  
NS 214  
DS 4  
SWH 112994.352 Hz  
FIDRES 0.862078 Hz  
AQ 0.5800436 sec  
RG 456.1  
DW 4.425 usec  
DE 7.00 usec  
TE 300.0 K  
D1 1.00000000 sec  
d11 0.03000000 sec  
d12 0.00002000 sec  
TD0 1

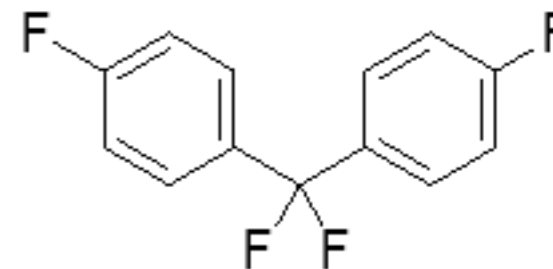
==== CHANNEL f1 =====  
NUC1 19F  
P1 3.00 usec  
PL1 -6.00 dB  
SFO1 376.1972840 MHz

==== CHANNEL f2 =====  
CPDPRG2 waltz16  
NUC2 1H  
PCPD2 100.00 usec  
PL2 6.00 dB  
PL12 24.00 dB  
SFO2 399.8719990 MHz

F2 - Processing parameters  
SI 65536  
SF 376.2537220 MHz  
WDW no  
SSB 0  
LB 0.00 Hz  
GB 0  
PC 1.00

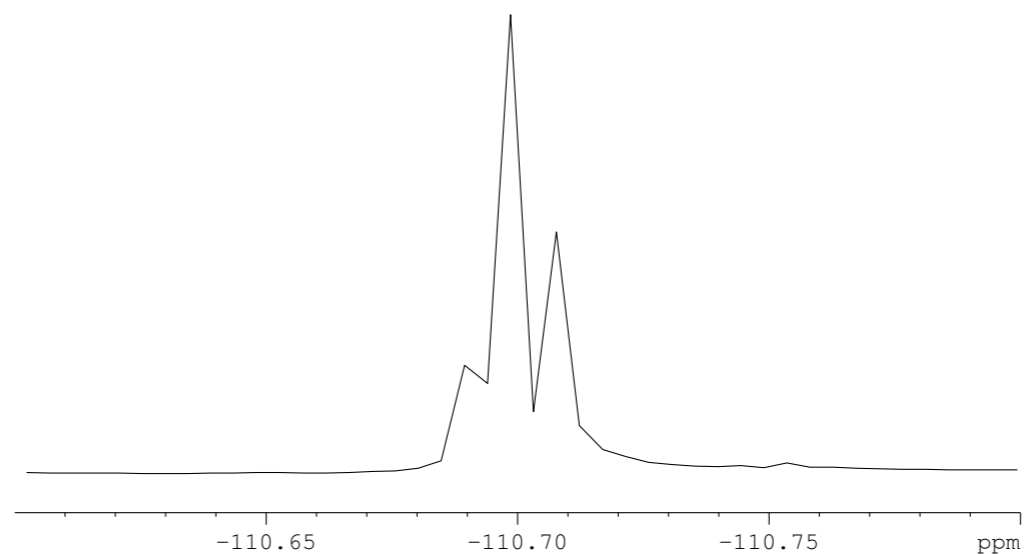
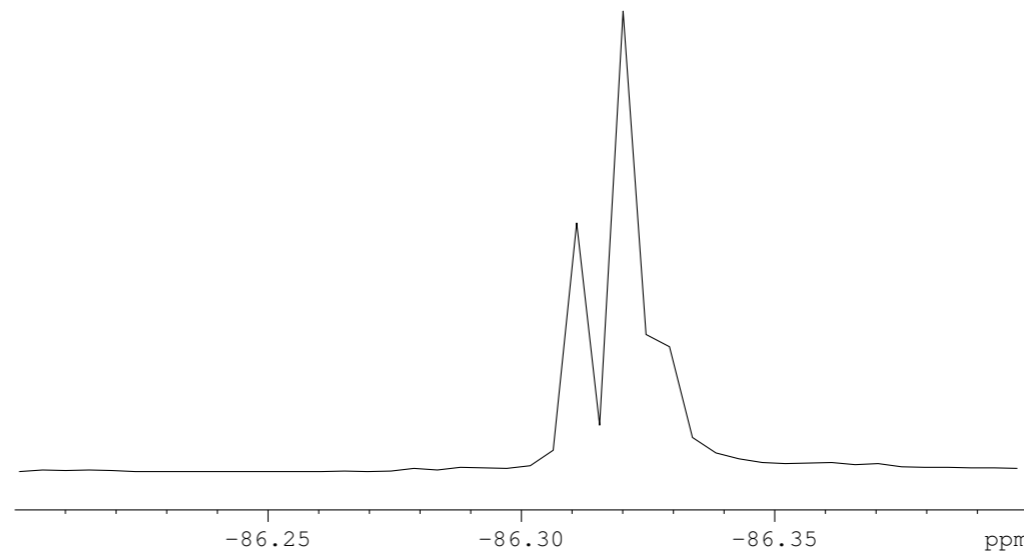
Peak	?(F1) [ppm]	?(F1) [Hz]	Intensity
1	-86.3085	-32473.8944	8.08
2	-86.3178	-32477.3935	15.00
3	-110.6884	-41646.9225	3.48
4	-110.6959	-41649.7444	14.06
5	-110.7051	-41653.2059	7.38

2010-109  
Fr 5-11  
nmr400b f-19



-86.31  
-86.32

-110.69  
-110.70  
-110.71



-10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 -210 -220 -230 -240 -250 -260 -270 -280 -290 ppm

Current Data Parameters  
NAME 2010-109  
EXPNO 5  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20100227  
Time 10.48  
INSTRUM spect  
PROBHD 5 mm QNP 1H/1  
PULPROG zg30  
TD 32768  
SOLVENT CDC13  
NS 32  
DS 2  
SWH 6578.947 Hz  
FIDRES 0.200774 Hz  
AQ 2.4904180 sec  
RG 456.1  
DW 76.000 usec  
DE 7.00 usec  
TE 300.0 K  
D1 0.10000000 sec  
TD0 1

==== CHANNEL f1 =====  
NUC1 1H  
P1 11.20 usec  
PL1 6.00 dB  
SFO1 399.8724694 MHz

F2 - Processing parameters  
SI 16384  
SF 399.8700089 MHz  
WDW no  
SSB 0  
LB 0.00 Hz  
GB 0  
PC 1.00

Peak	?(F1) [ppm]	?(F1) [Hz]	Intensity
1	7.5087	3002.5039	1.51
2	7.5015	2999.6249	10.31
3	7.4959	2997.3856	5.54
4	7.4941	2996.6658	3.85
5	7.4902	2995.1063	7.24
6	7.4885	2994.4266	11.47
7	7.4869	2993.7868	9.85
8	7.4851	2993.0670	8.06
9	7.4788	2990.5478	13.04
10	7.4735	2988.4285	4.30
11	7.4716	2987.6688	5.66
12	7.4658	2985.3495	11.61
13	7.4588	2982.5504	1.84
14	7.2702	2907.1349	4.73
15	7.1448	2856.9912	1.49
16	7.1372	2853.9522	11.89
17	7.1324	2852.0329	4.83
18	7.1158	2845.3950	20.00
19	7.0989	2838.6372	3.91
20	7.0941	2836.7178	10.09

2010-109  
Fr 5-11  
fluorinated benzophenone  
nmr400b h-1

