Supporting Information

for

Hydrazone-Pd-catalyzed Direct Intermolecular Reaction of *o*-Alkynylphenols with Allylic Acetates

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Procedure of Preparation of starting material 1, 2 and 5a

Preparation of *o*-alkynylphenols 1 and *o*-allyloxyethynylbenzene 5a

All of *o*-alkynylphenol derivatives **1** and 1-cinnmayloxy-2-(phenylethynyl)benzene (**5a**) were prepared accrding to our previous reported literture.[S1]

Preparation of allylic acetate 2

Cinnamyl acetate (**2a**) and allyl acetate (**2f**) were commercialy available.

Allylic acetates **2b**-**e** were prepared *via* two-step ractions *via* cinnamyl alcohols.

As first step, cinnamyl alcohols such as (*E*)-3-(*p*-tolyl)-2-propene-1-ol, (*E*)-3-(4-methoxyphenyl)-2-propene-1-ol, (*E*)-3-(4-chlorophenyl)-2-propene-1-ol and (*E*)-3-(4-(trifluoromethyl)phenyl)-2-propene-1-ol were prepared according to the literture reported by Schmalz's group[S2] using the corresponding cinnamic acids such as 4-methylcinnamic acid, 4-methoxycinnamic acid, 4-chlorocinnamic acid and 4-(trifluoromethyl)cinnamic acid, respectively.

Preparation of (*E*)-3-(*p*-tolyl)allyl acetate (2b)

Acetic anhydrous (0.76 mL, 8.0 mmol) and Et_3N (1.3 mL, 9.0 mmol) were added to the mixture of (*E*)-3-(*p*-tolyl)-2-propene-1-ol (0.8882 g, 6.0 mmol), *N*,*N*-dimethyl-4-aminopyridine (0.4892 g, 4.0 mmol) and THF (8.0 mL) under an Ar atmosphere. The mixture was stirred at r.t. under an Ar atmosphere. After 24 h, the reaction was quenched with 2N HCl aq. The solution was extracted, washed with satutrated $NaHCO_3$ aq., dried over anhydrous Na_2SO_4, filtered, and concentrated under reduced pressure. The residue was purified by silica gel column chromatography (hexane/ethyl acetate (v/v = 50/1)) to afford **2b** in 79% yield (0.8991 g, 4.73 mmol).

(*E*)-3-(*p*-Tolyl)allyl acetate (2b)[3]: Colorless oil; ^{1}H NMR ($CDCl_3$) δ: 2.09 (s, 3H), 2.33 (s, 3H), 4.71 (dd, *J* = 6.6 and 0.9 Hz, 2H), 6.23 (dt, *J* = 15.9 and 6.6 Hz, 1H), 6.62 (d, *J* = 15.9 Hz, 1H), 7.13 (d, *J* = 8.1 Hz, 2H), 7.28 (d, *J* = 8.1 Hz, 2H); ^{13}C NMR ($CDCl_3$) δ: 21.0, 21.2, 65.2, 122.0, 126.5, 129.3, 133.3, 134.2, 138.0, 170.8; EI-MS *m/z* (rel intensity): 190 (M^+, 90).

Preparation of (*E*)-3-(4-methoxyphenyl)allyl acetate (2c)

Acetic anhydrous (0.38 mL, 4.0 mmol) and Et_3N (0.65 mL, 4.5 mmol) were added to the mixture of (*E*)-3-(4-methoxyphenyl)-2-propene-1-ol (0.4920 g, 3.0 mmol), *N*,*N*-dimethyl-4-aminopyridine (0.2442 g, 4.0 mmol) and THF (4.0 mL) under an Ar atmosphere. The mixture was stirred at r.t. under an Ar atmosphere. After 24 h, the reaction was quenched with 2N HCl aq. The solution was extracted, washed with satutrated $NaHCO_3$ aq., dried over anhydrous

Na_2SO_4, filtered, and concentrated under reduced pressure. The residue was purified by silica gel column chromatography (hexane/ethyl acetate (v/v = 10/1)) to afford **2c** in 81% yield (0.4984 g, 2.42 mmol).

(*E*)-3-(4-Methoxyphenyl)allyl acetate (2c)[3]: Colorless oil; ^{1}H NMR ($CDCl_3$) δ: 2.10 (s, 3H), 3.81 (s, 3H), 4.70 (dd, *J* = 6.6 and 0.9 Hz, 2H), 6.15 (dt, *J* = 15.9 and 6.6 Hz, 1H), 6.60 (d, *J* = 15.9 Hz, 1H), 6.86 (dt, *J* = 8.6 and 3.0 Hz, 2H), 7.33 (dt, *J* = 8.6 and 3.0 Hz, 2H); ^{13}C NMR ($CDCl_3$) δ: 21.0, 55.2, 65.3, 113.9, 120.8, 127.8, 128.9, 134.0, 159.5, 170.8; EI-MS *m/z* (rel intensity): 206 (M^+, 100).

Preparation of (*E*)-3-(4-chlorophenyl)allyl acetate (2d)

Acetic anhydrous (0.76 mL, 8.0 mmol) and triethylamine (1.3 mL, 9.0 mmol) were added to the mixture of (*E*)-3-(4-chlorophenyl)-2-propene-1-ol (1.0085 g, 6.0 mmol), *N*,*N*-dimethyl-4-aminopyridine (0.4891 g, 4.0 mmol) and THF (8.0 mL) under an Ar atmosphere. The mixture was stirred at r.t. under an Ar atmosphere. After 24 h, the reaction was quenched with 2N HCl aq. The solution was extracted, washed with satutrated $NaHCO_3$ aq., dried over anhydrous Na_2SO_4, filtered, and concentrated under reduced pressure. The residue was purified by silica gel column chromatography (hexane/ethyl acetate (v/v = 10/1)) to afford **2c** in 91% yield (1.1452 g, 5.46 mmol).

(*E*)-3-(4-Chlorophenyl)allyl acetate (2d)[3]: Colorless oil; ^{1}H NMR ($CDCl_3$) δ: 2.11 (s, 3H), 4.72 (dd, *J* = 6.3 and 1.2 Hz, 2H), 6.26 (dt, *J* = 15.9 and 6.3 Hz, 1H), 6.60 (d, *J* = 15.9 Hz, 1H), 7.24-7.33 (m, 4H); ^{13}C NMR ($CDCl_3$) δ: 20.9, 64.8, 123.8, 127.7, 128.7, 132.8, 133.7, 134.6, 170.7; EI-MS *m/z* (rel intensity): 210 (M^+, 26).

Preparation of (*E*)-3-(4-(trifluoromethyl)phenyl)allyl acetate (2e)

Acetic anhydrous (0.38 mL, 4.0 mmol) and triethylamine (0.65 mL, 9.0 mmol) were added to the mixture of (*E*)-3-(4-(trifluoromethyl)phenyl)-2-propene-1-ol (0.6062 g, 3.0 mmol), *N*,*N*-dimethyl-4-aminopyridine (0.2443 g, 2.0 mmol) and THF (4.0 mL) under an Ar atmosphere. The mixture was stirred at r.t. under an Ar atmosphere. After 24 h, the reaction was quenched with 2N HCl aq. The solution was extracted, washed with satutrated $NaHCO_3$ aq., dried over anhydrous Na_2SO_4, filtered, and concentrated under reduced pressure. The residue was purified by silica gel column chromatography (hexane/ethyl acetate (v/v = 10/1)) to afford **2e** in 81% yield (0.5913 g, 2.42 mmol).

(*E*)-3-(4-(Trifluoromethyl)phenyl)allyl acetate (2e)[3]: Colorless oil; ^{1}H NMR ($CDCl_3$) δ: 2.13 (s, 3H), 4.76 (dd, *J* = 6.0 and 1.2 Hz, 2H), 6.38 (dt, *J* = 15.9 and 6.0 Hz, 1H), 6.68 (d, *J* = 15.9 Hz, 1H), 7.49 (d, *J* = 8.4 Hz, 2H), 7.58 (d, *J* = 8.4 Hz, 2H); ^{13}C NMR ($CDCl_3$) δ: 20.9, 64.6, 124.1 (q, *J* = 271.9 Hz), 125.5 (q, *J* = 3.9 Hz), 126.0, 126.7, 129.8 (q, *J* = 32.6 Hz), 132.3, 139.6, 170.7; EI-MS *m/z*

(rel intensity): 244 (M^+, 13).

References

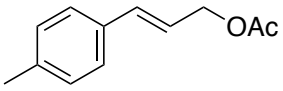
S1. K. Watanabe, T. Mino, T. Ikematsu, C. Hatta, Y. Yoshida, M. Sakamoto, *Org. Chem. Front.*, 2016, **3**, 979.

S2. W. Lölsberg, S. Ye, H.-S. Schmalza, *Adv. Synth. Catal.*, 2010, **352**, 2023.

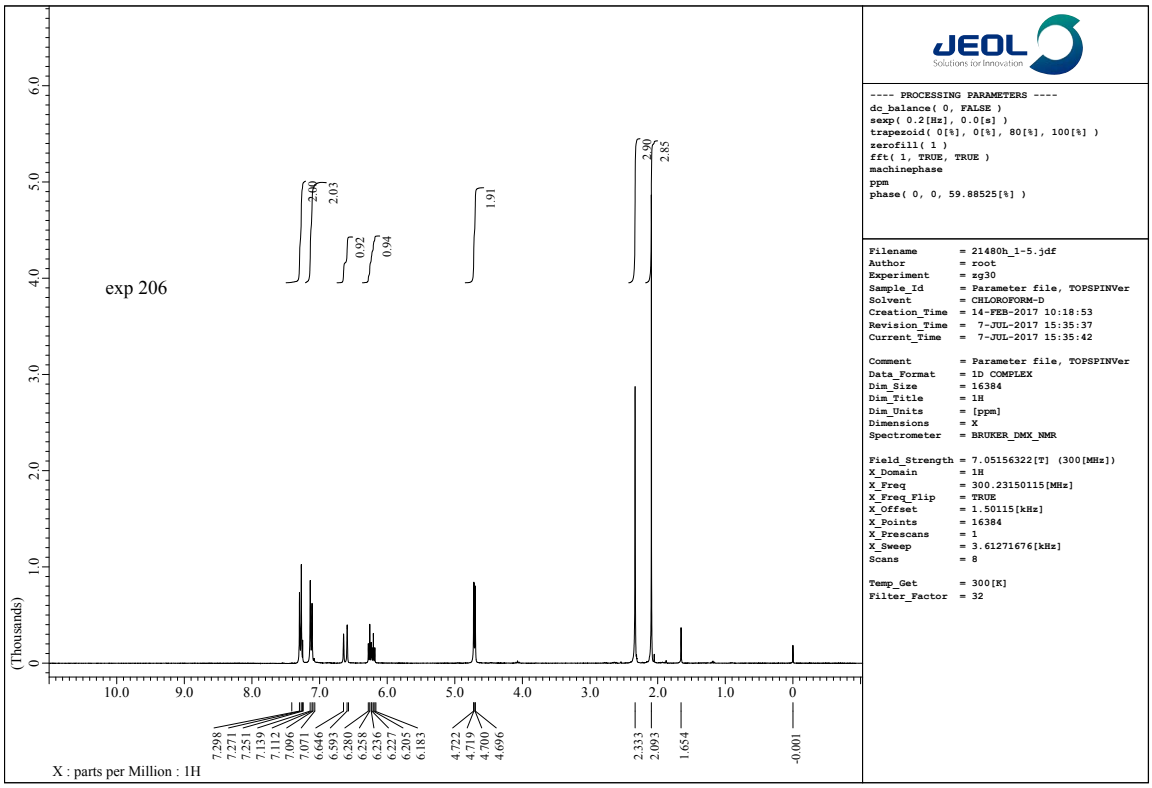
S3. Y. Su, N, Jiao, *Org. Lett.*, 2009, **11**, 2980.

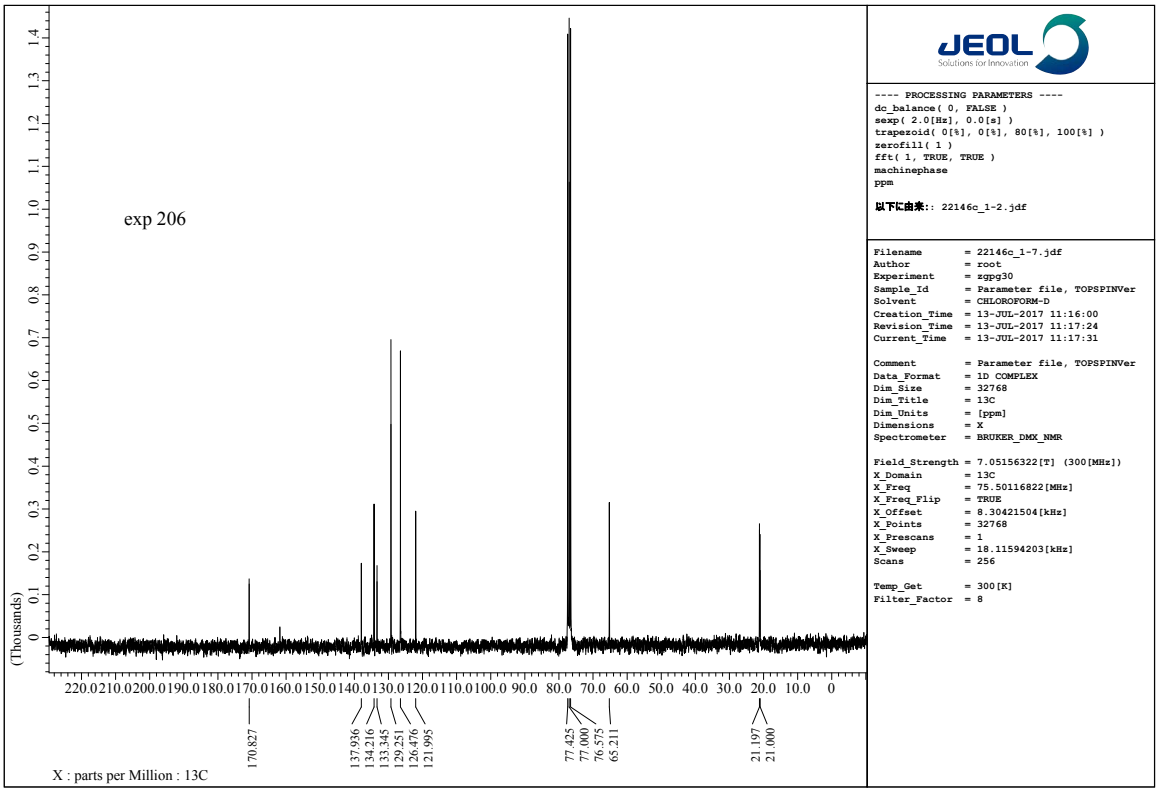
H and ^{13}C NMR

(*E*)-3-(*p*-Tolyl)allyl acetate (2b)



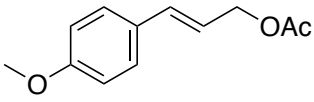
2b





^{1}H and ^{13}C NMR

(*E*)-3-(4-Methoxyphenyl)allyl acetate (2c)



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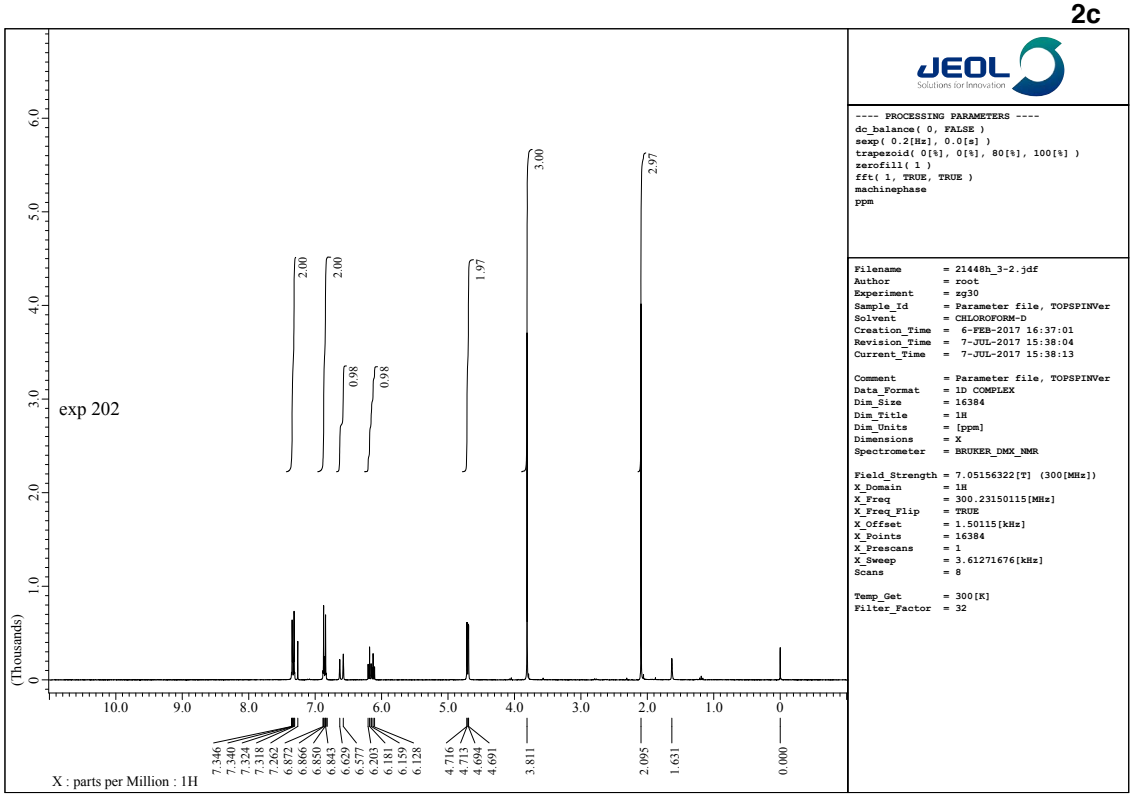

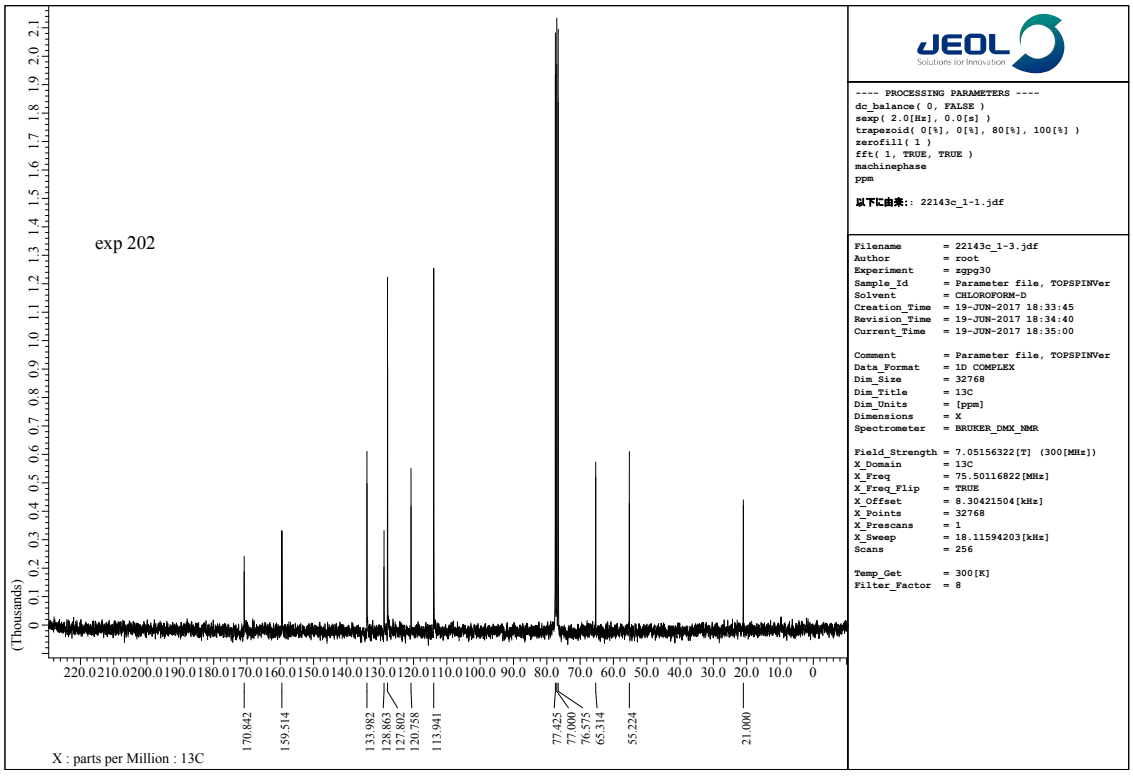

^{1}H and ^{13}C NMR

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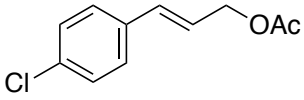

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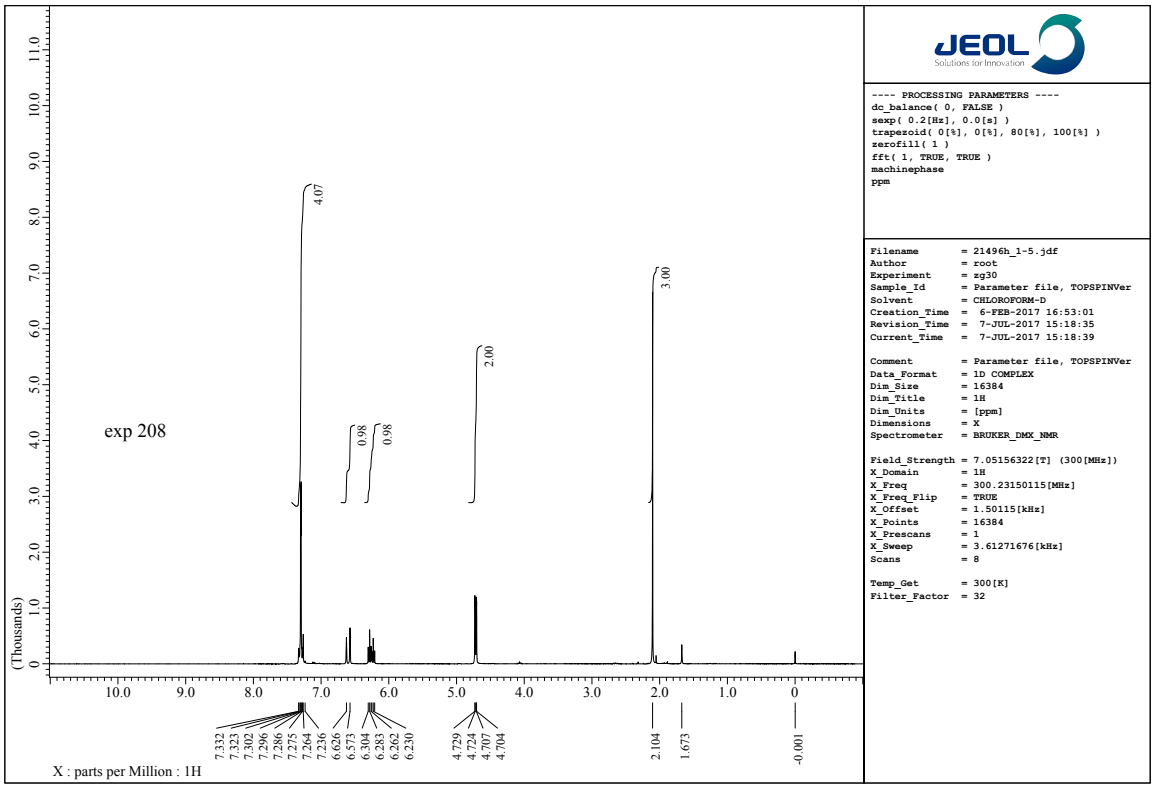
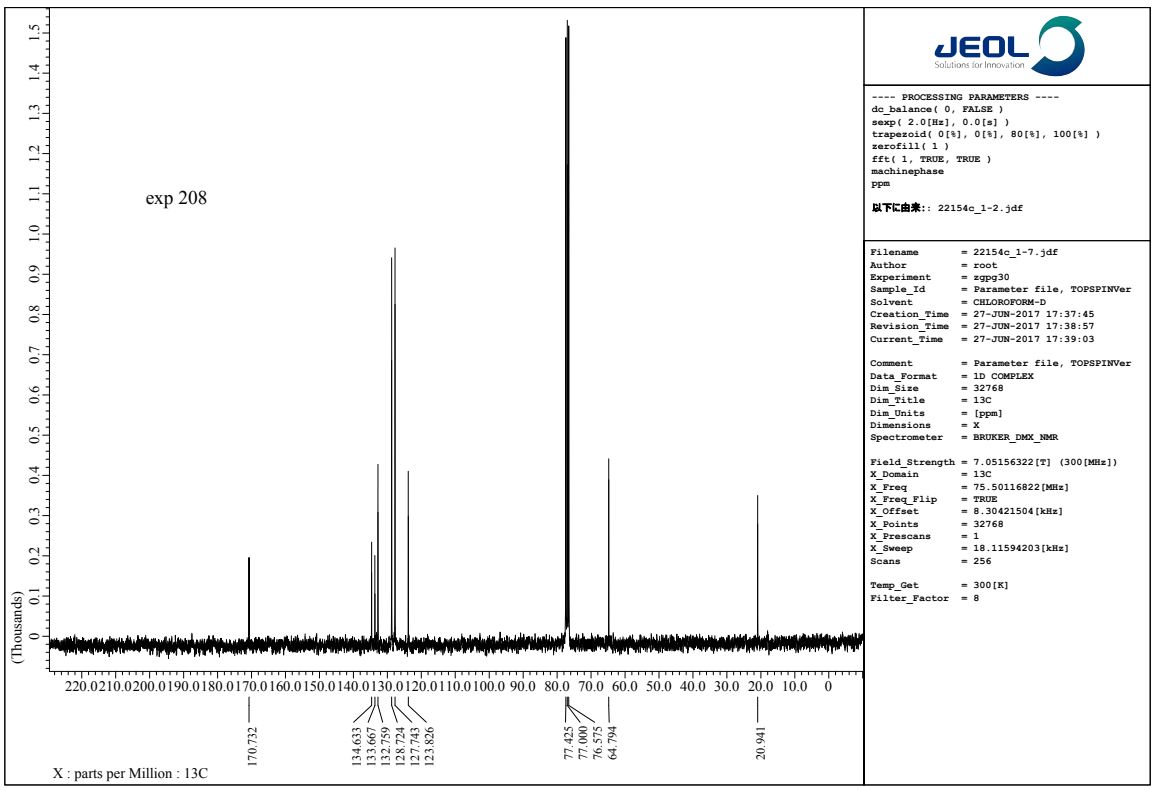
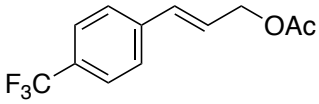


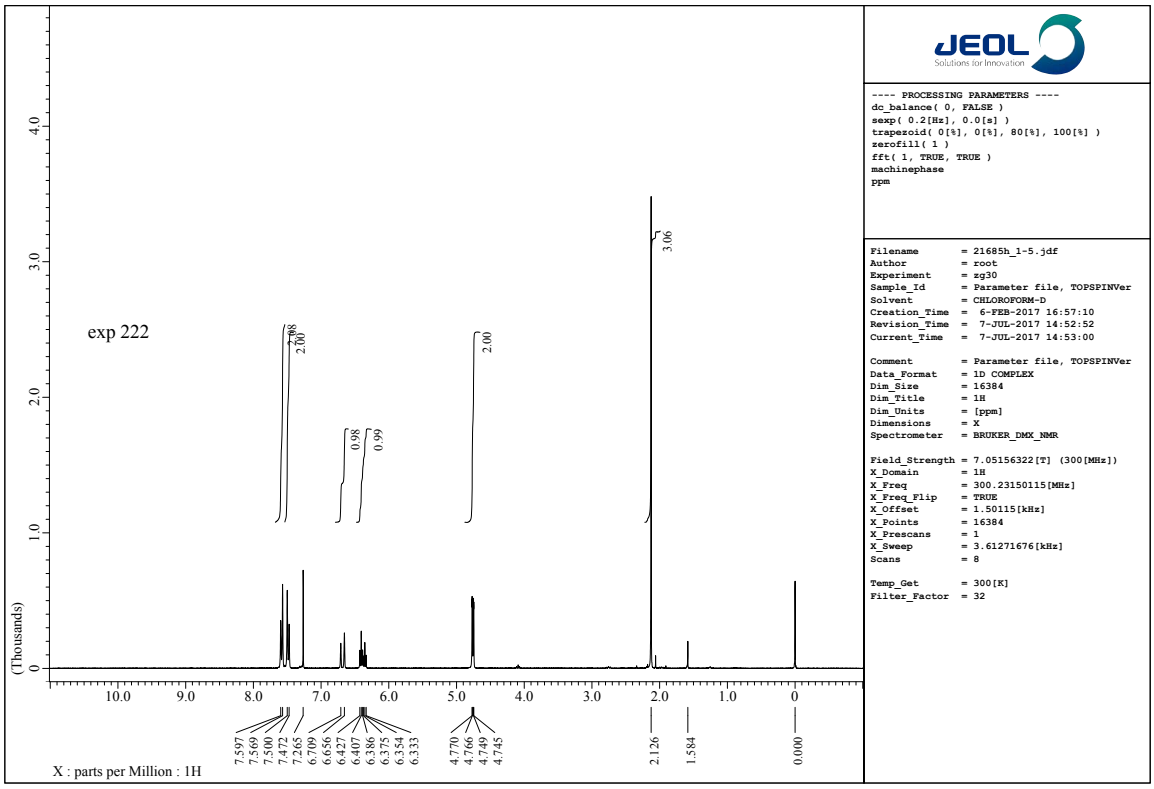


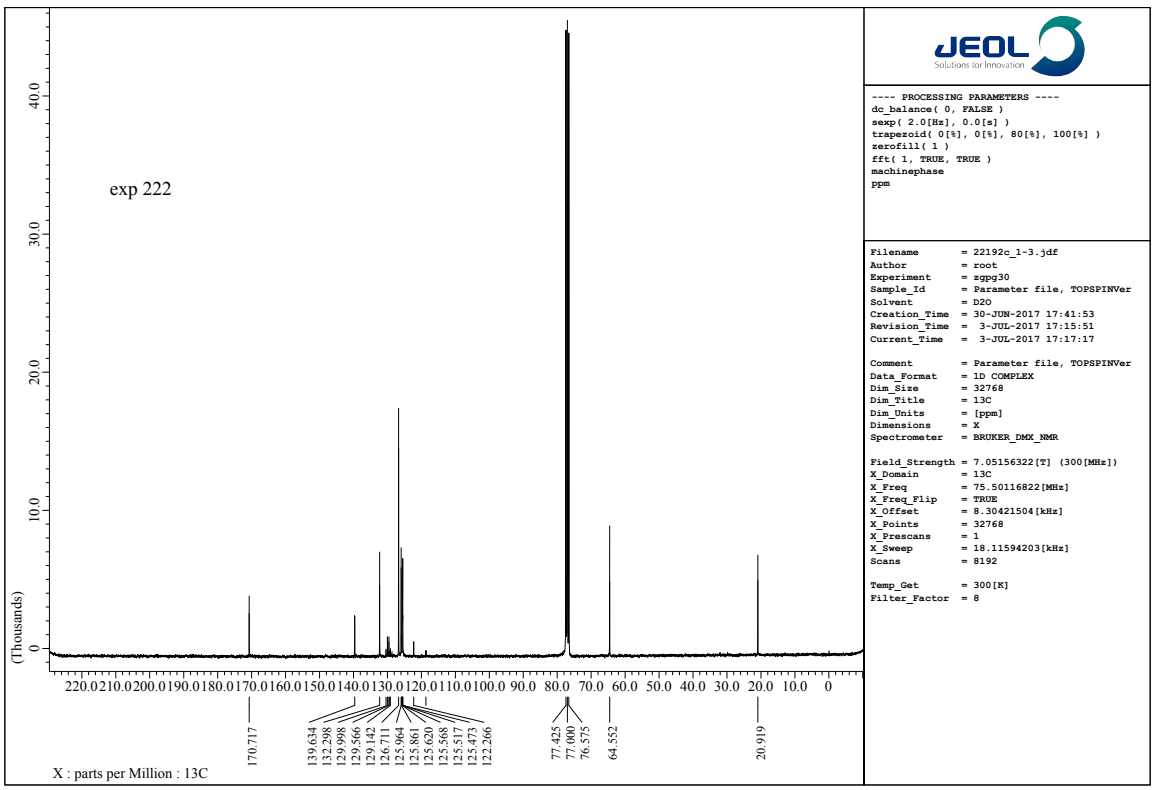
^{1}H and ^{13}C NMR

(*E*)-3-(4-(Trifluoromethyl)phenyl)allyl acetate (2e)



2e





^{1}H and ^{13}C NMR

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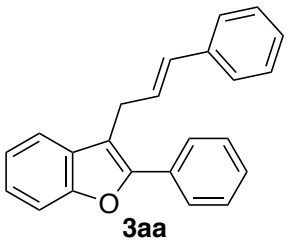

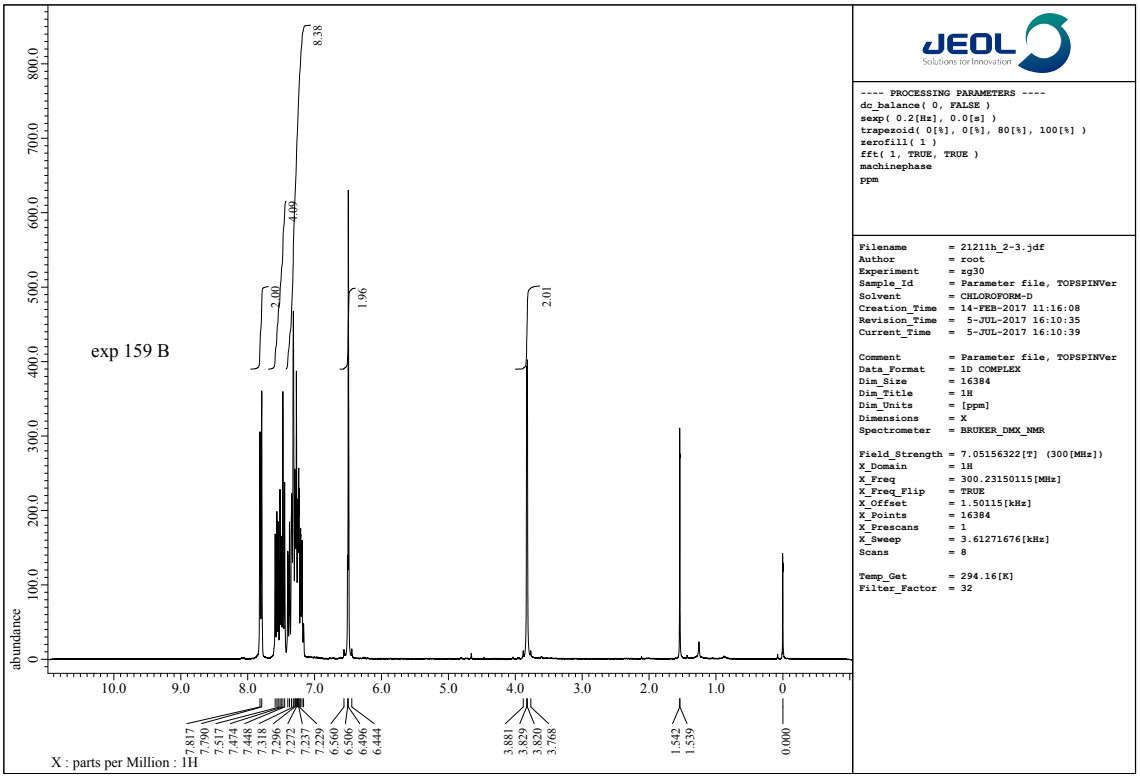

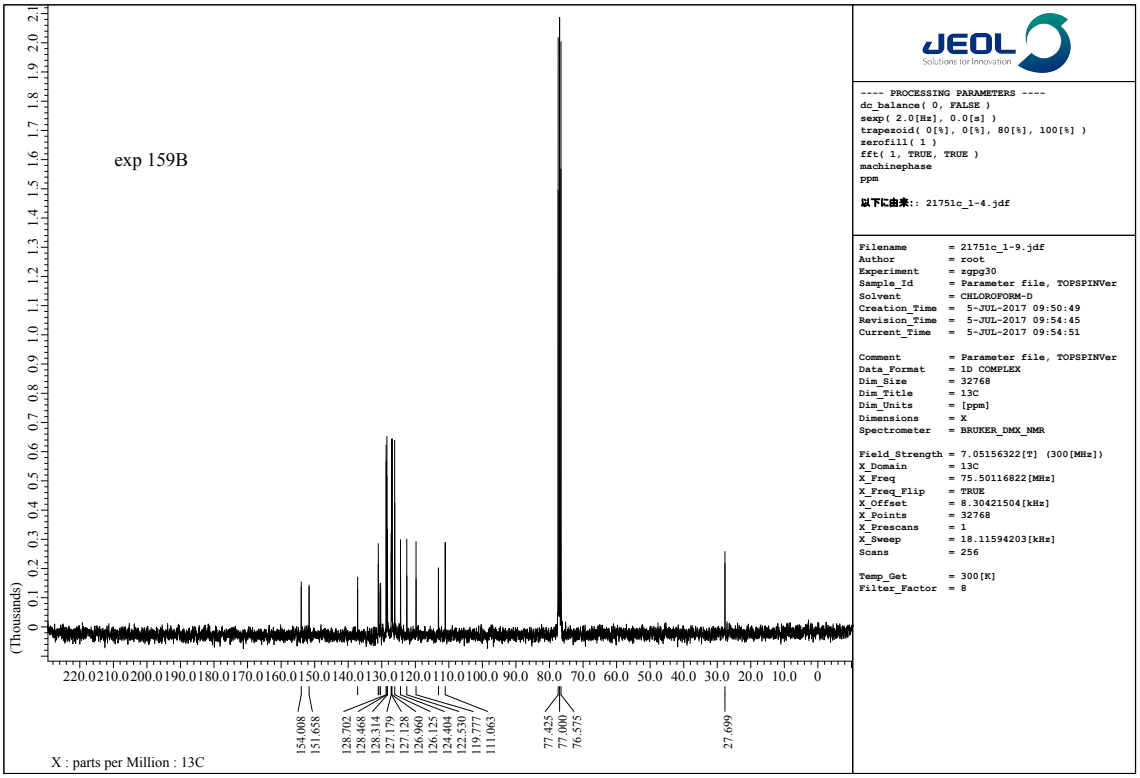

^{1}H and ^{13}C NMR

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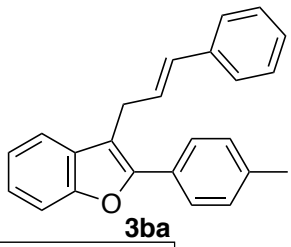

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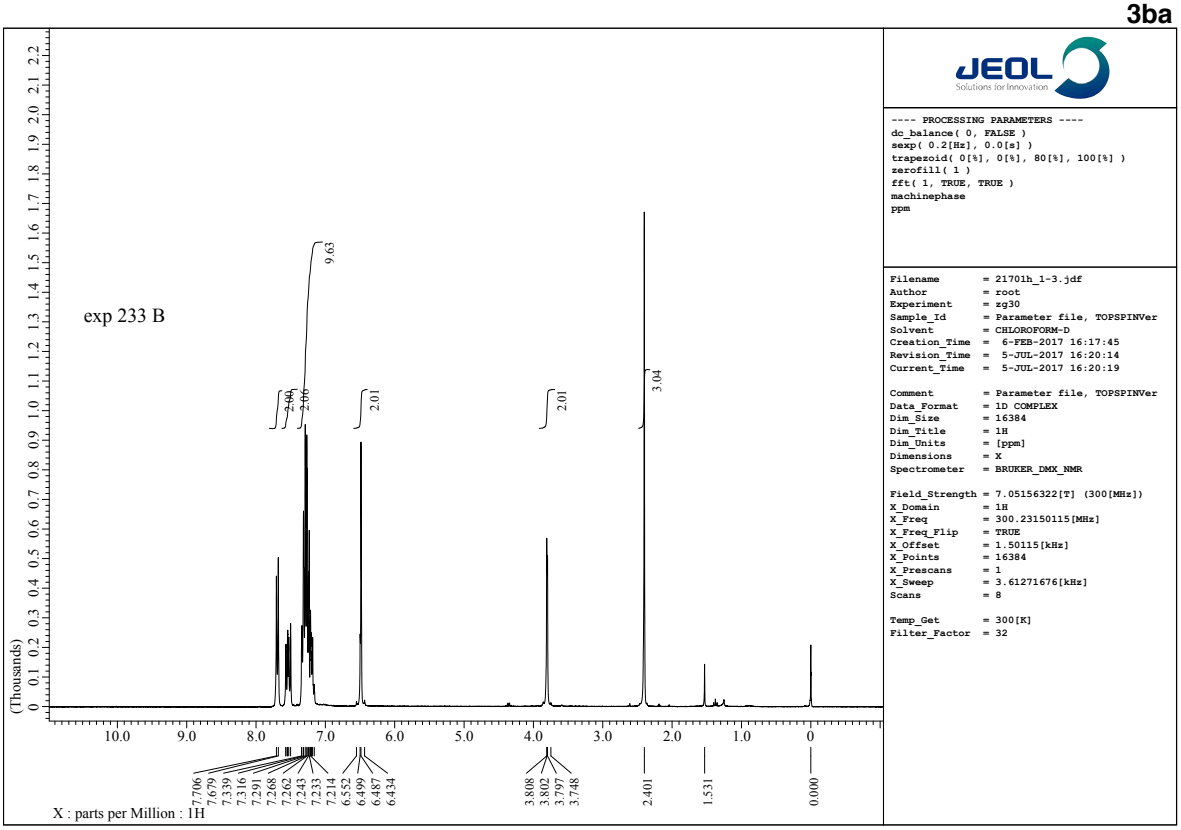

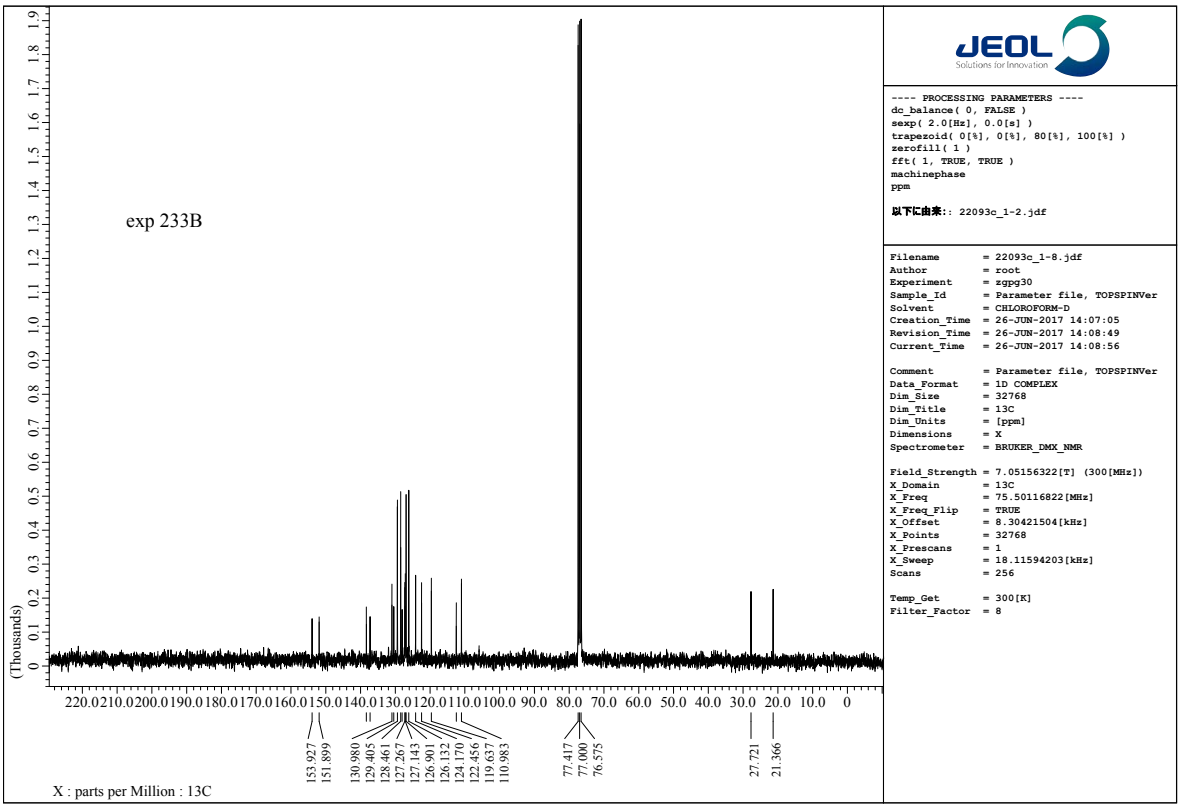

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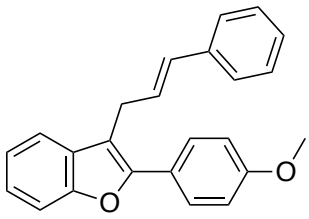

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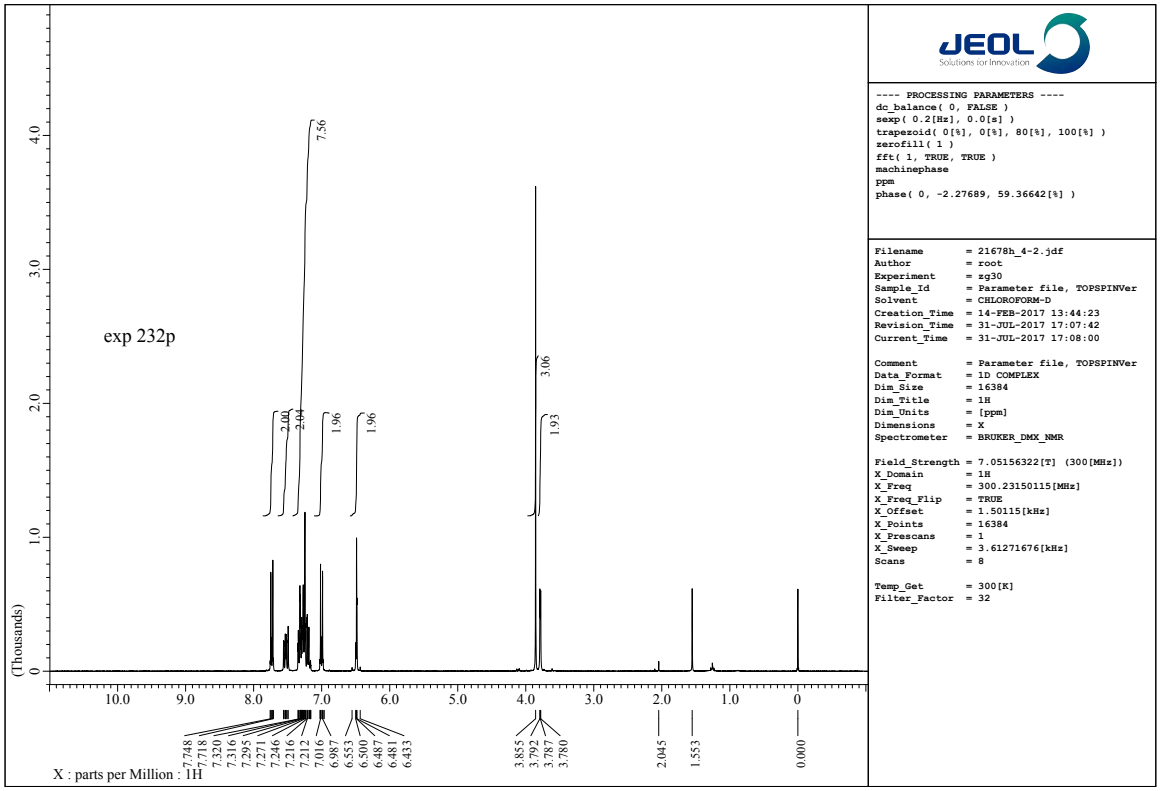

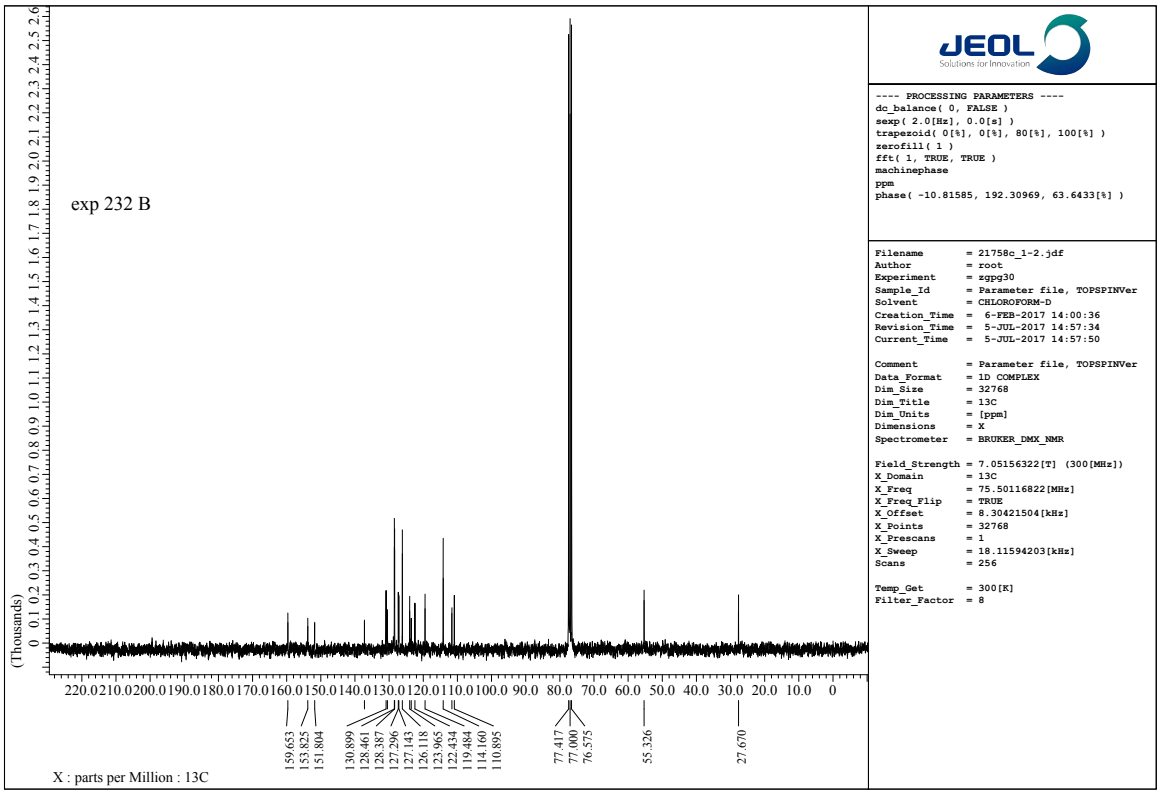

^{1}H and ^{13}C NMR

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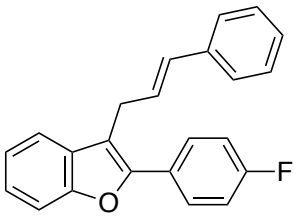

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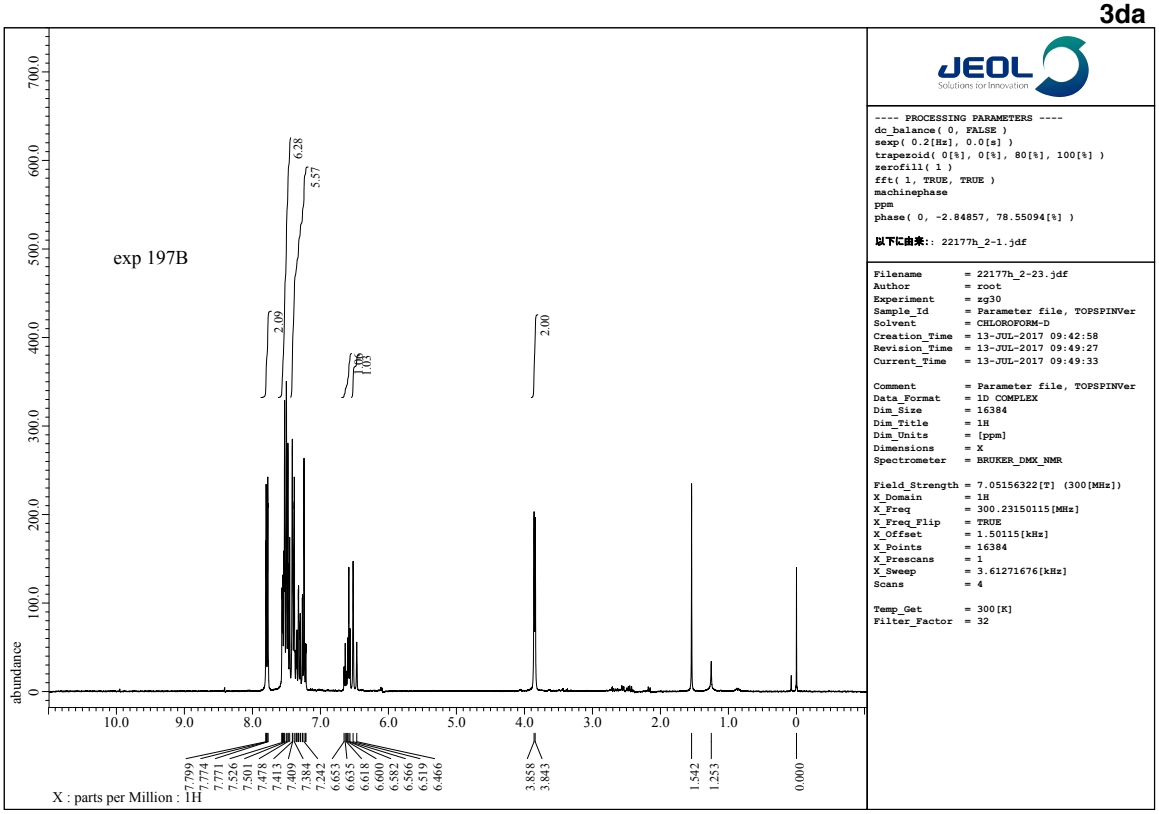

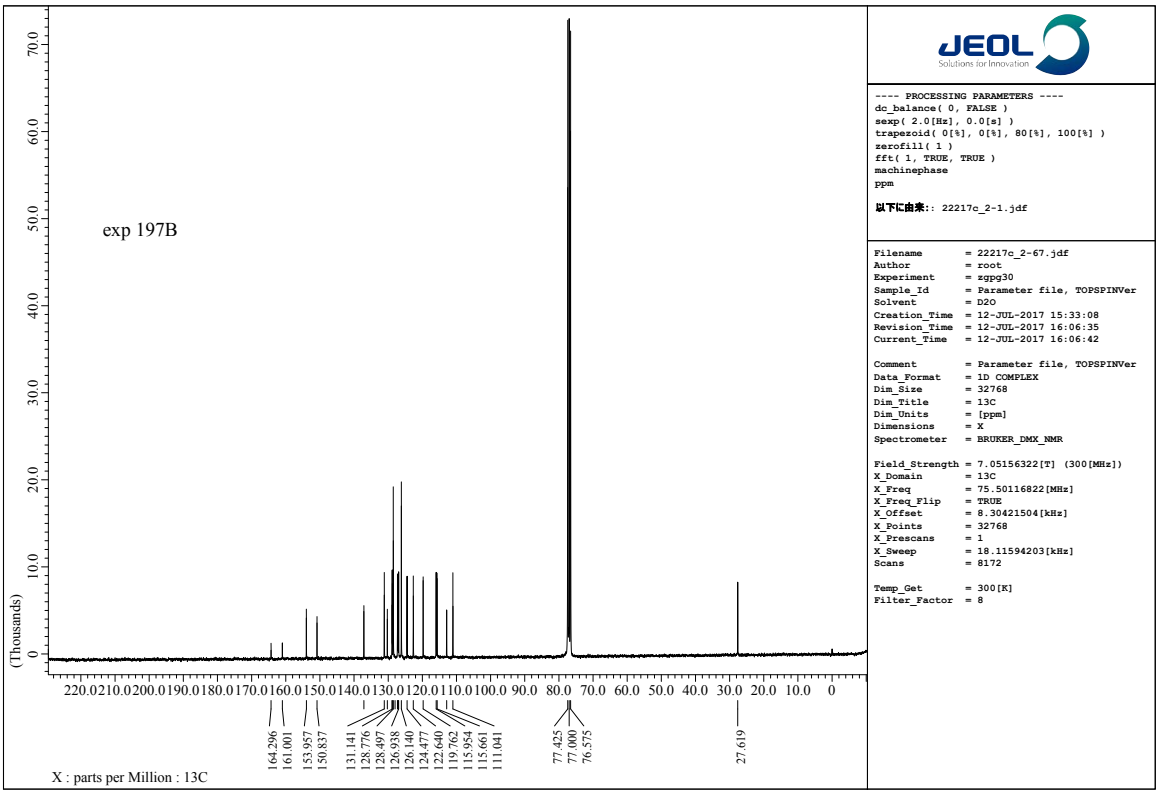

^{1}H and ^{13}C NMR

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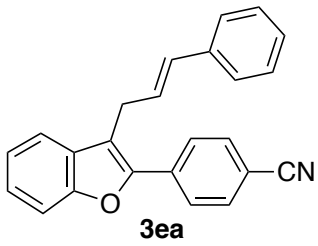

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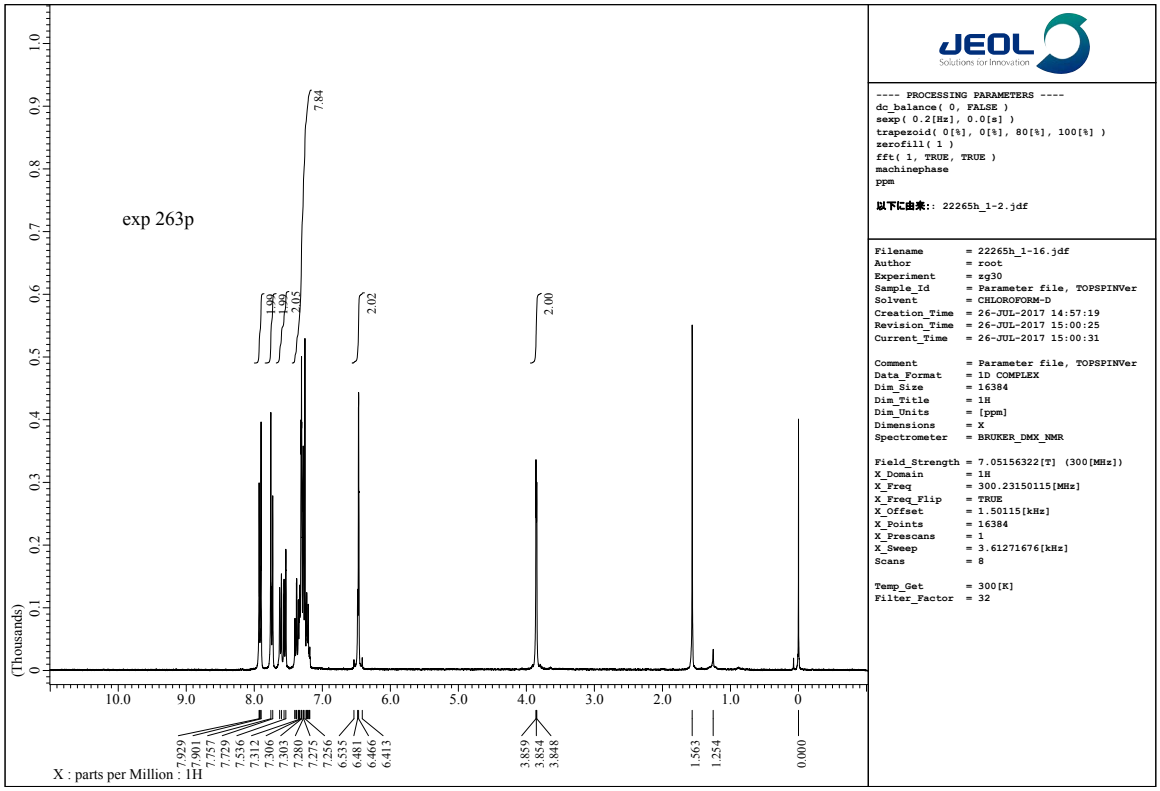

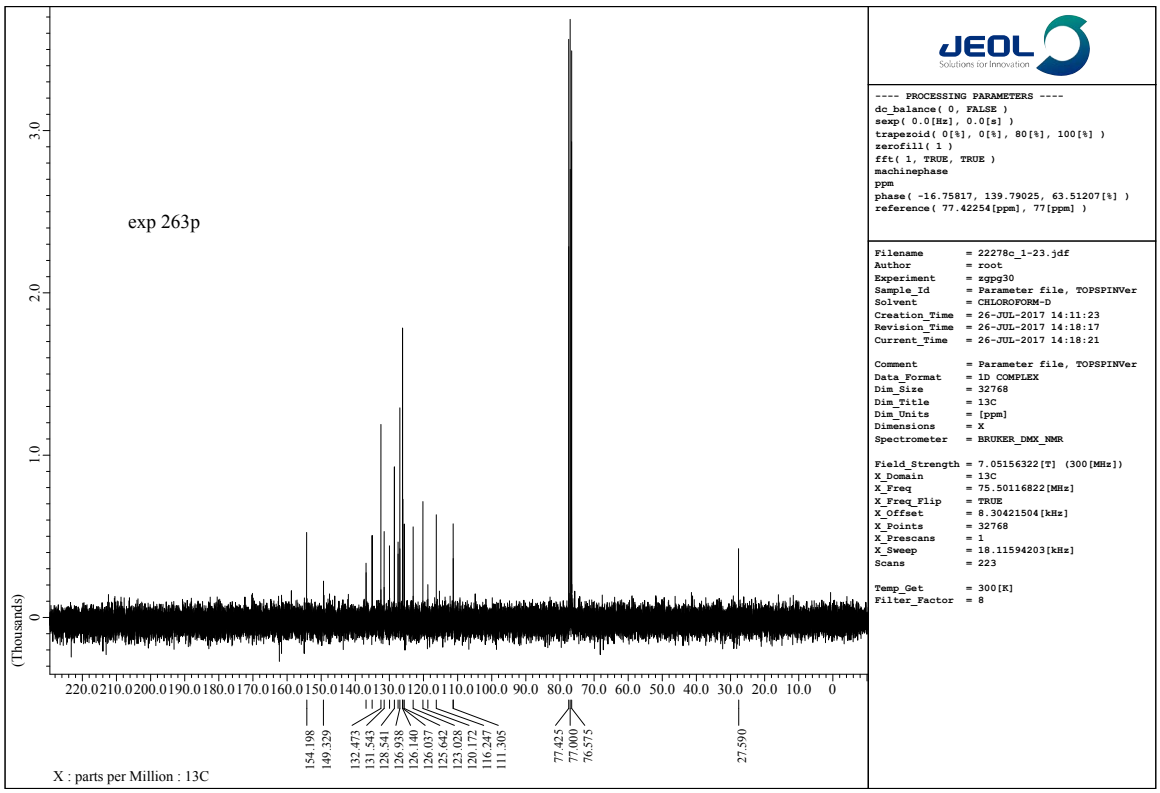

^{1}H and ^{13}C NMR

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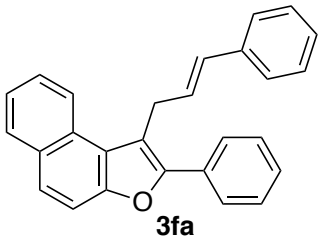

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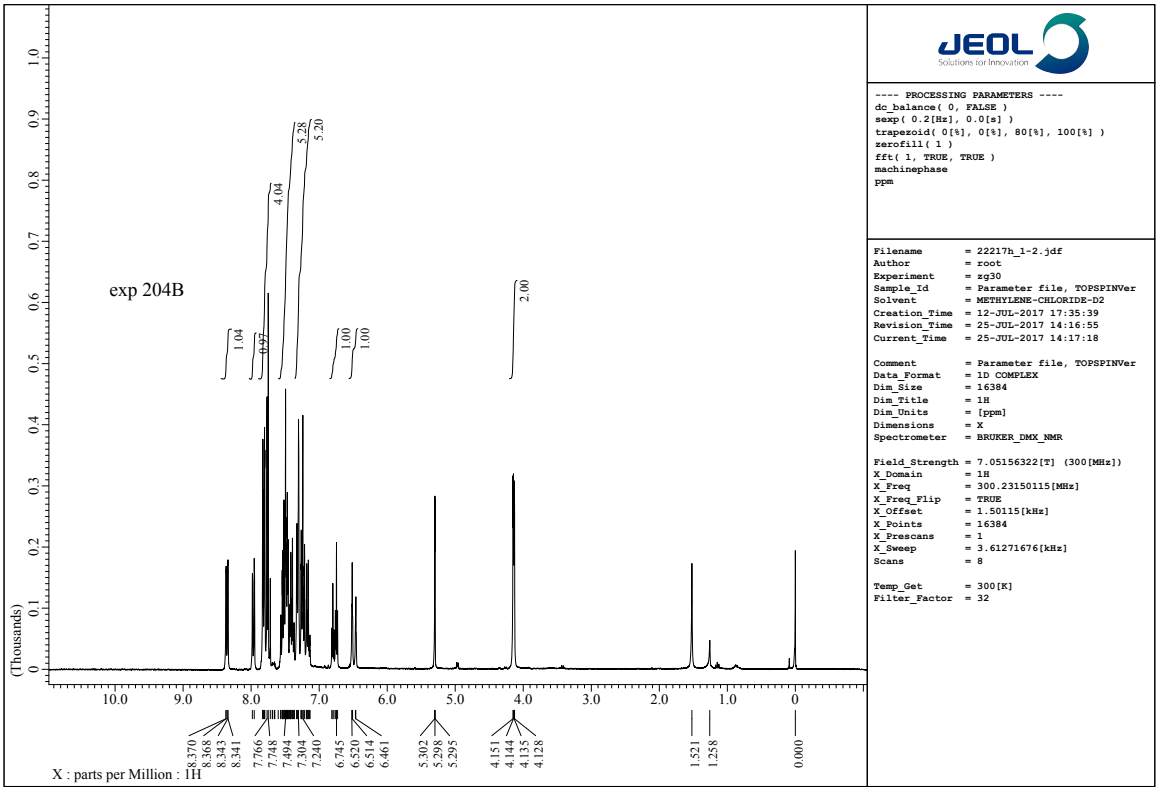
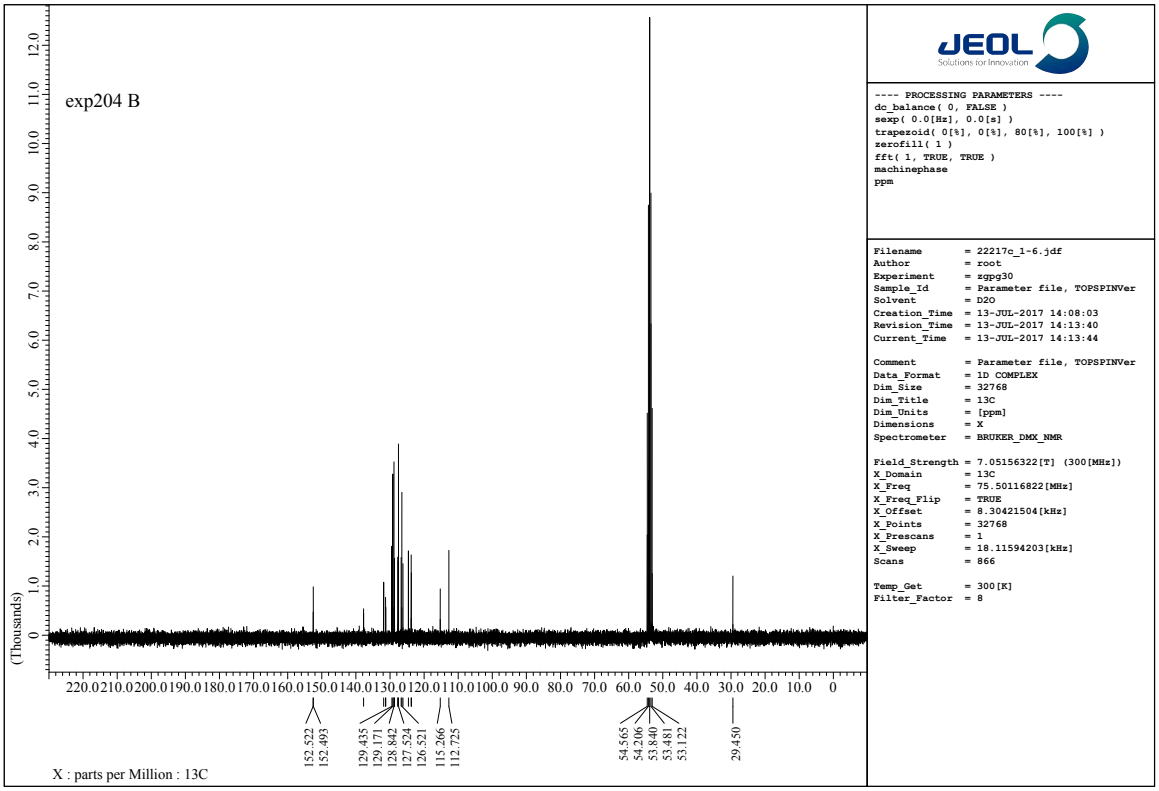
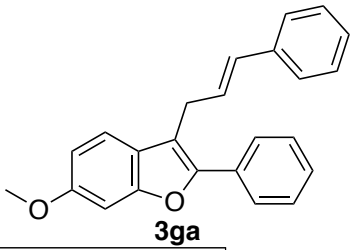


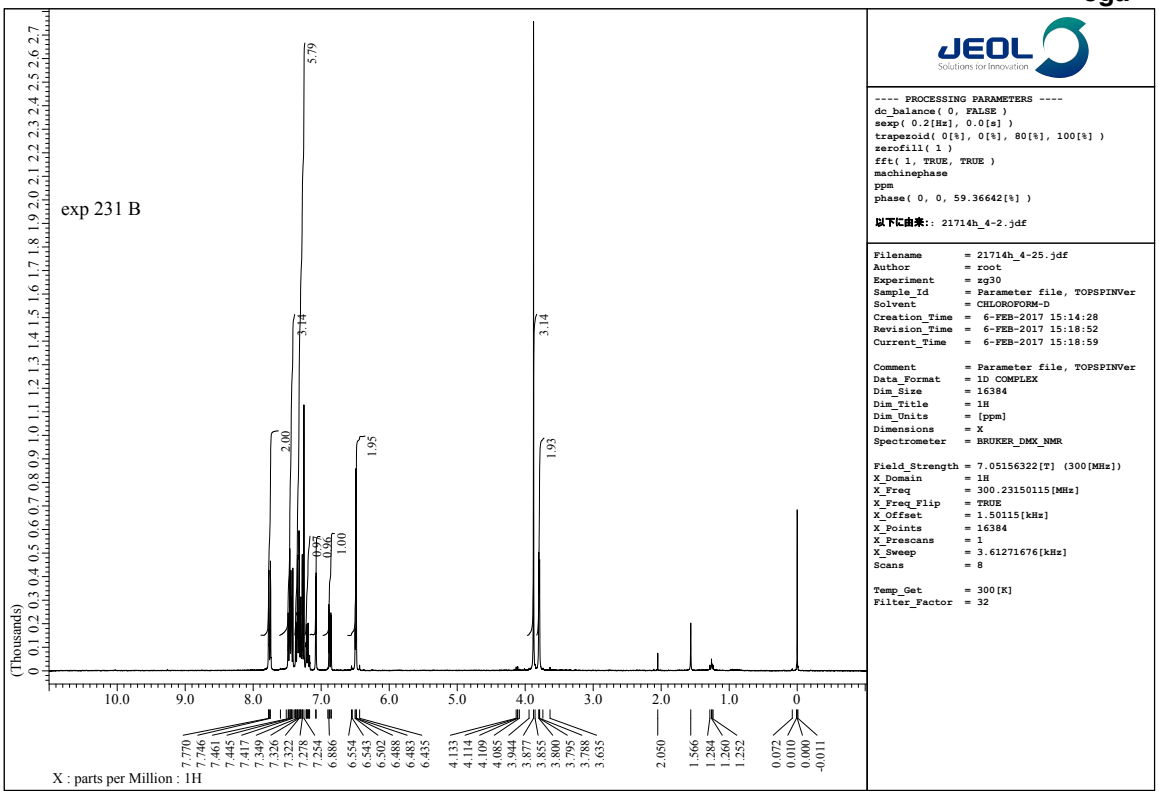


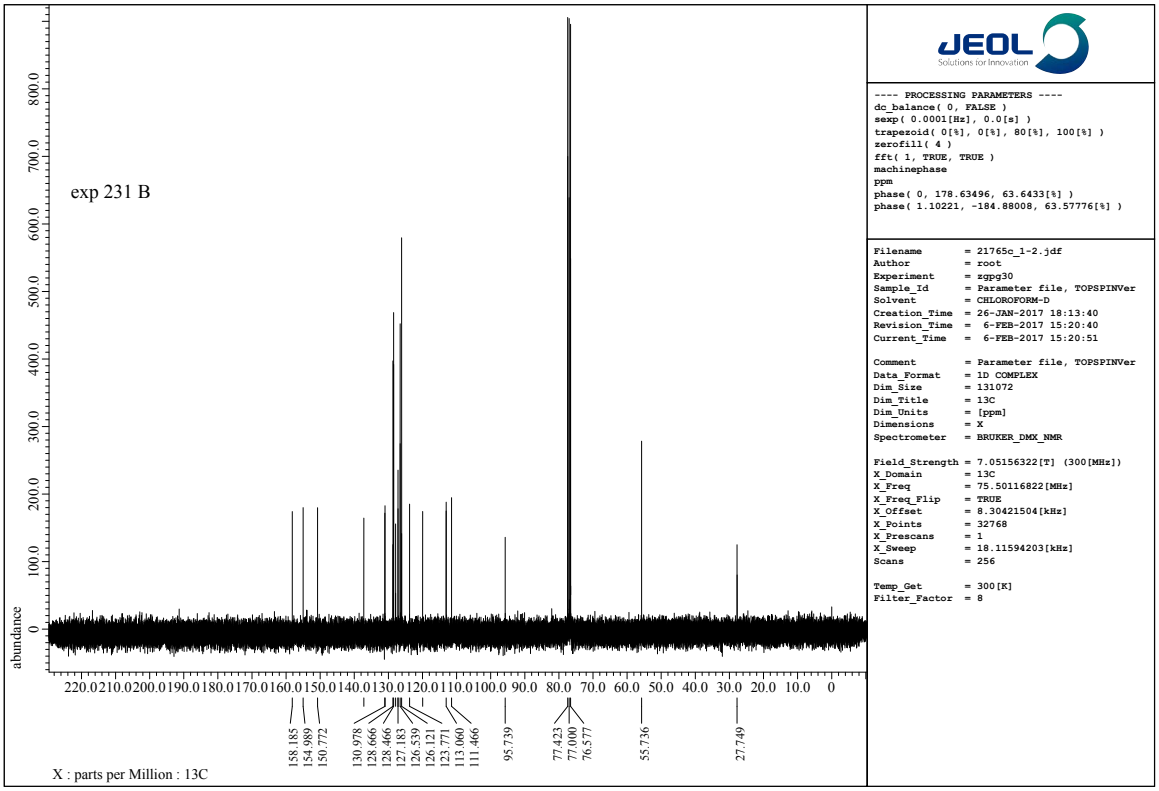
^{1}H and ^{13}C NMR

3-Cinnamyl-6-methoxy-2-phenylbenzofuran (3ga)



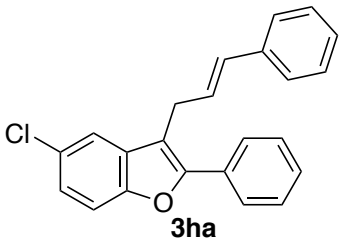
3ga





^{1}H and ^{13}C NMR

5-Chloro-3-cinnamyl-2-phenylbenzofuran (3ha)



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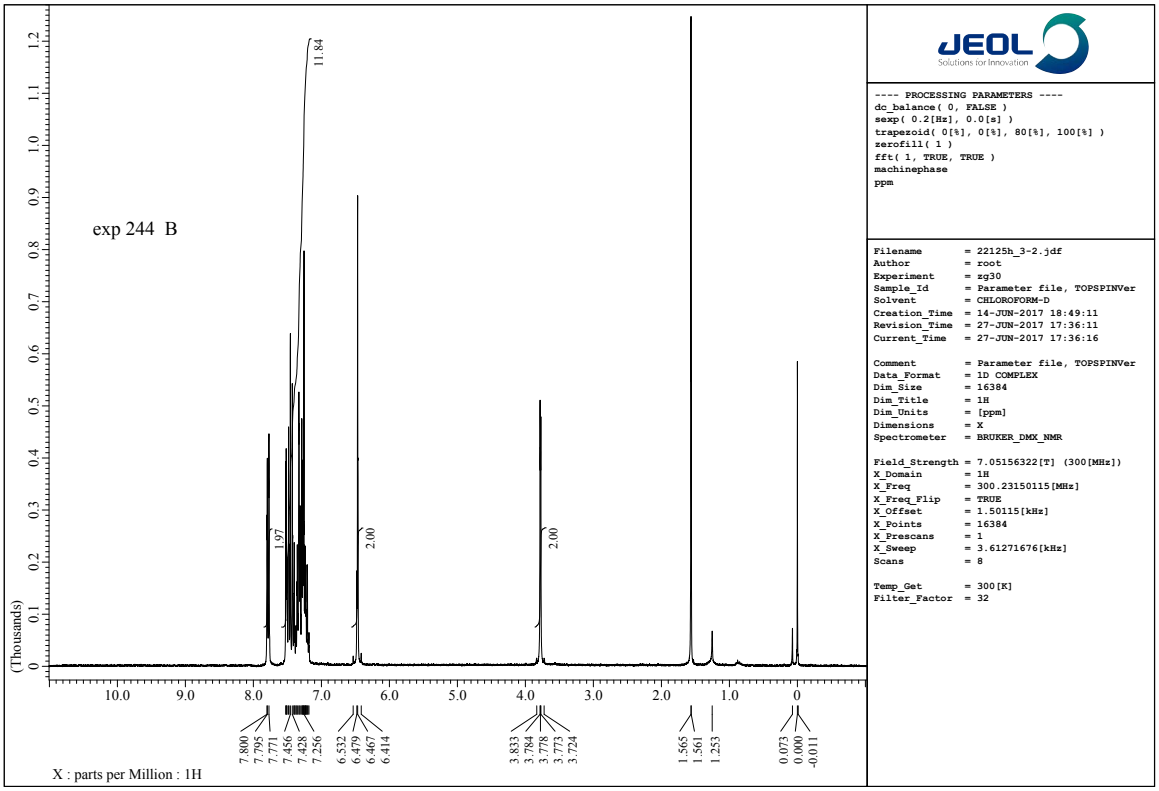


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Filter_Factor   = 32
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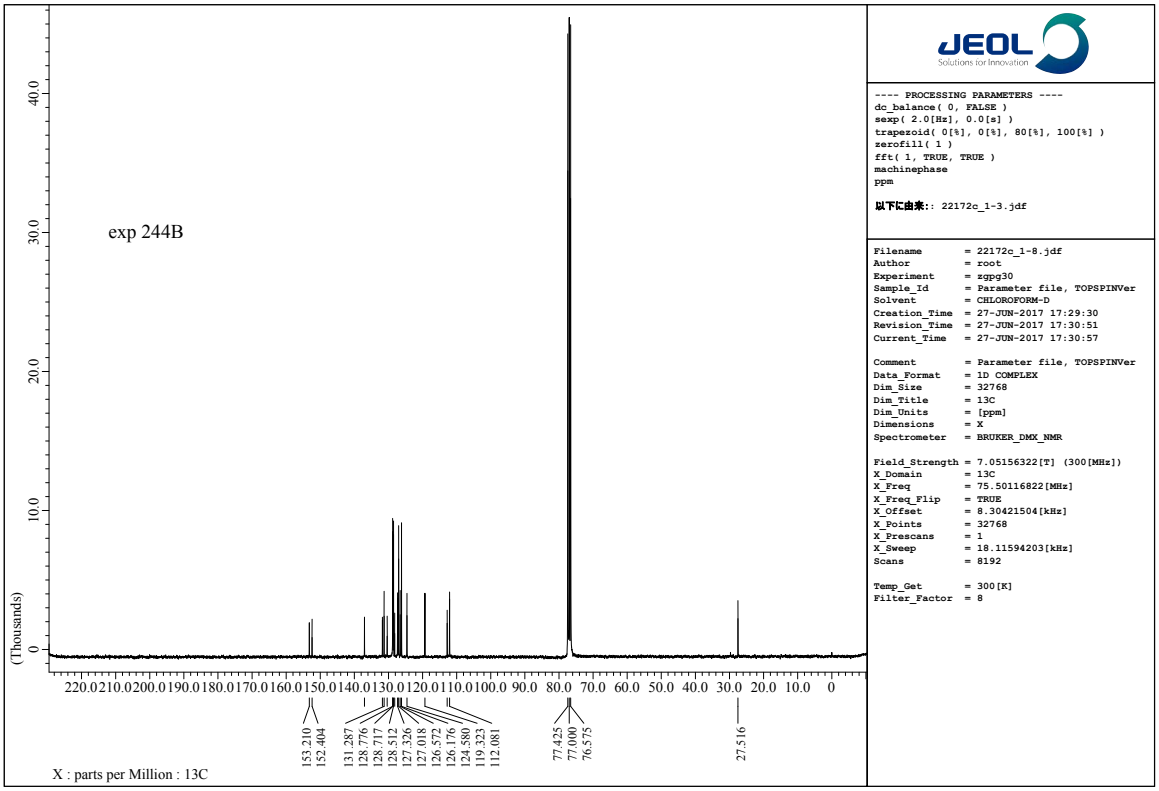
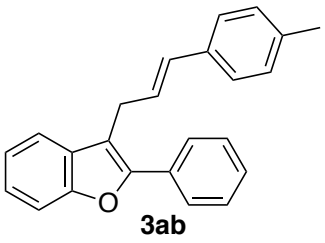


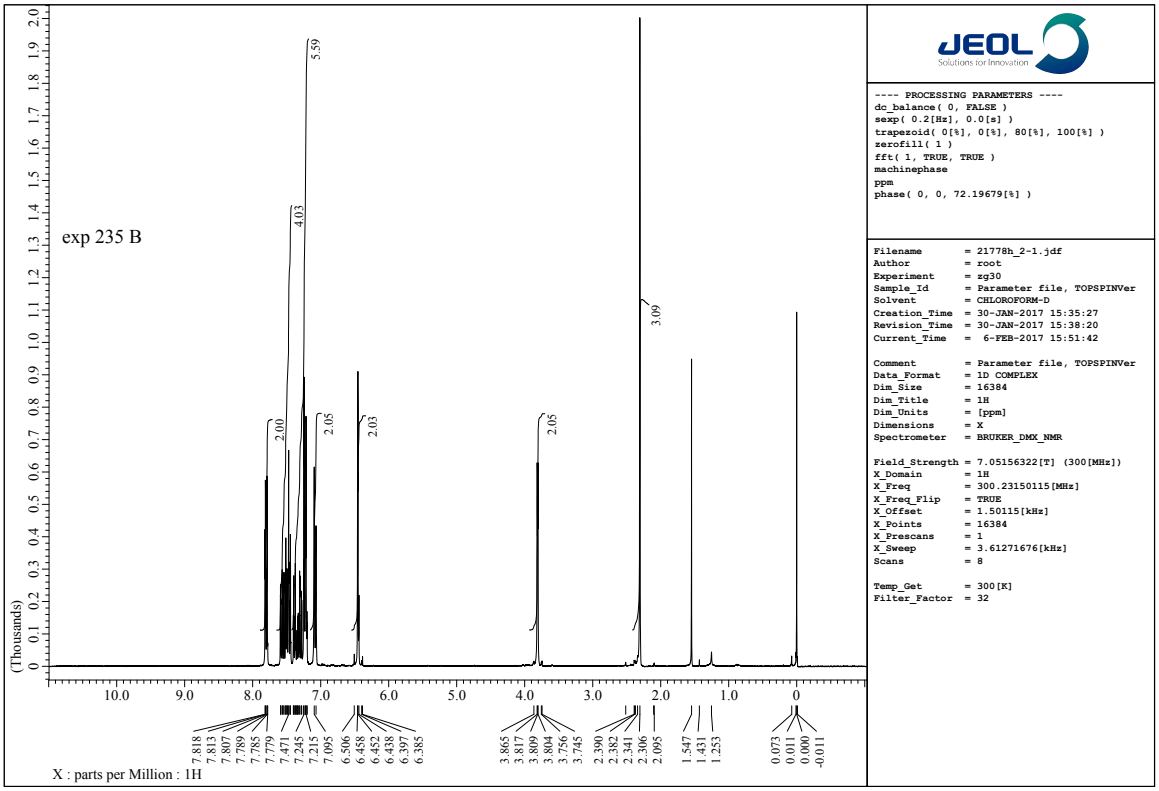

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

以下に由来:: 22172c_1-3.jdf

Filename        = 22172c_1-8.jdf
Author          = root
Experiment      = zgpg30
Sample_Id       = Parameter file, TOPSPINVer
Solvent         = CHLOROFORM-D
Creation_Time   = 27-JUN-2017 17:29:30
Revision_Time   = 27-JUN-2017 17:30:51
Current_Time    = 27-JUN-2017 17:30:57

Comment         = Parameter file, TOPSPINVer
Data_Format     = 1D COMPLEX
Dim_Size        = 32768
Dim_Title       = 13C
Dim_Units       = [ppm]
Dimensions      = X
Spectrometer    = BRUKER_DMX_NMR

Field_Strength = 7.05156322[T] (300[MHz])
X_Domain        = 13C
X_Freq          = 75.50116822[MHz]
X_Freq_Flip     = TRUE
X_Offset        = 8.30421504[kHz]
X_Points        = 32768
X_Prescans      = 1
X_Sweep         = 18.11594203[kHz]
Scans           = 8192

Temp_Get        = 300[K]
Filter_Factor   = 8
```

^{1}H and ^{13}C NMR

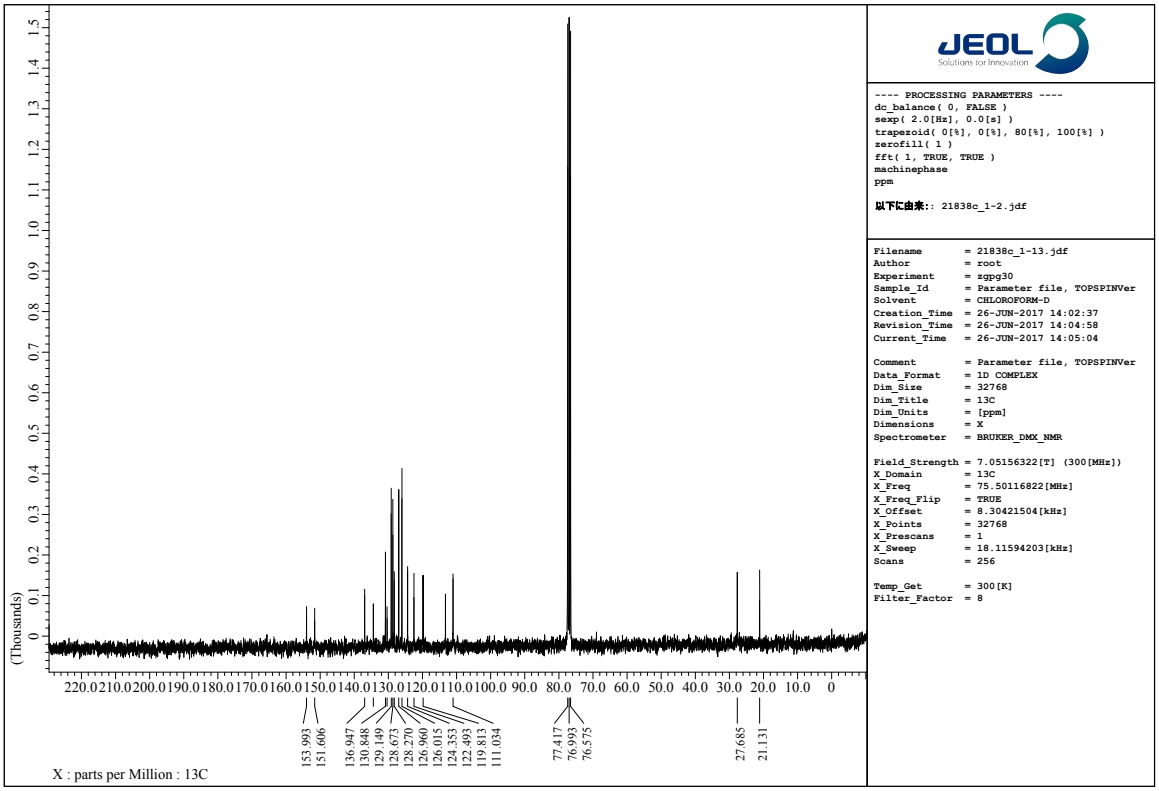
(*E*)-3-(3-(*p*-Tolyl)allyl)-2-phenylbenzofuran (3ab)



3ab



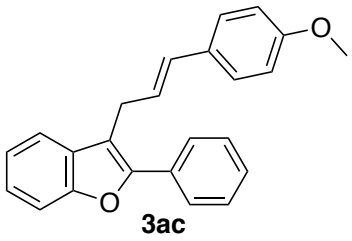

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm
phase( 0, 0, 72.19679[%] )

Filename        = 21778h_2-1.jdf
Author          = root
Experiment      = zg30
Sample_Id       = Parameter file, TOPSPINVer
Solvent         = CHLOROFORM-D
Creation_Time   = 30-JAN-2017 15:35:27
Revision_Time   = 30-JAN-2017 15:38:20
Current_Time    =  6-FEB-2017 15:51:42

Comment         = Parameter file, TOPSPINVer
Data_Format     = 1D COMPLEX
Dim_Size        = 16384
Dim_Title       = 1H
Dim_Units       = [ppm]
Dimensions      = X
Spectrometer    = BRUKER_DMX_NMR

Field_Strength = 7.05156322[T] (300[MHz])
X_Domain        = 1H
X_Freq          = 300.23150115[MHz]
X_Freq_Flip     = TRUE
X_Offset        = 1.50115[kHz]
X_Points        = 16384
X_Prescans      = 1
X_Sweep         = 3.61271676[kHz]
Scans           = 8

Temp_Get        = 300[K]
Filter_Factor   = 32
```



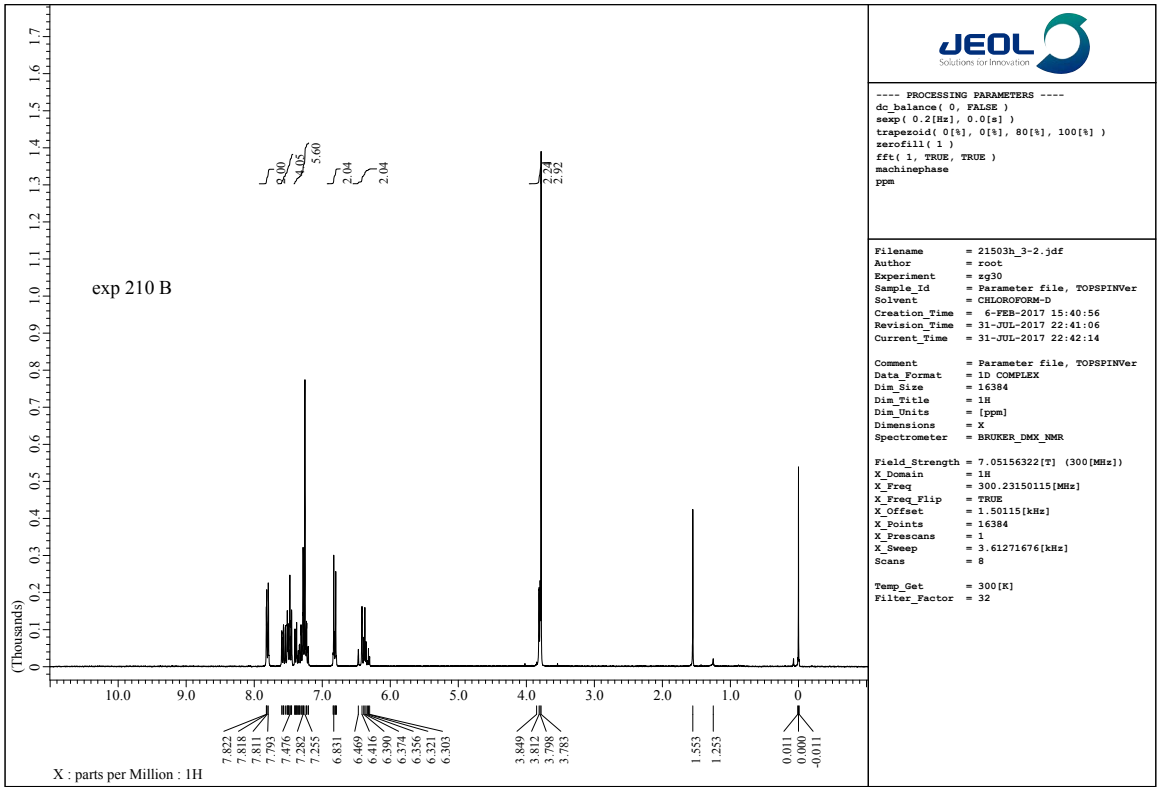

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

以下に由来:: 21838c_1-2.jdf

Filename        = 21838c_1-13.jdf
Author          = root
Experiment      = zgpg30
Sample_Id       = Parameter file, TOPSPINVer
Solvent         = CHLOROFORM-D
Creation_Time   = 26-JUN-2017 14:02:37
Revision_Time   = 26-JUN-2017 14:04:58
Current_Time    = 26-JUN-2017 14:05:04

Comment         = Parameter file, TOPSPINVer
Data_Format     = 1D COMPLEX
Dim_Size        = 32768
Dim_Title       = 13C
Dim_Units       = [ppm]
Dimensions      = X
Spectrometer    = BRUKER_DMX_NMR

Field_Strength = 7.05156322[T] (300[MHz])
X_Domain        = 13C
X_Freq          = 75.50116822[MHz]
X_Freq_Flip     = TRUE
X_Offset        = 8.30421504[kHz]
X_Points        = 32768
X_Prescans      = 1
X_Sweep         = 18.11594203[kHz]
Scans           = 256

Temp_Get        = 300[K]
Filter_Factor   = 8
```

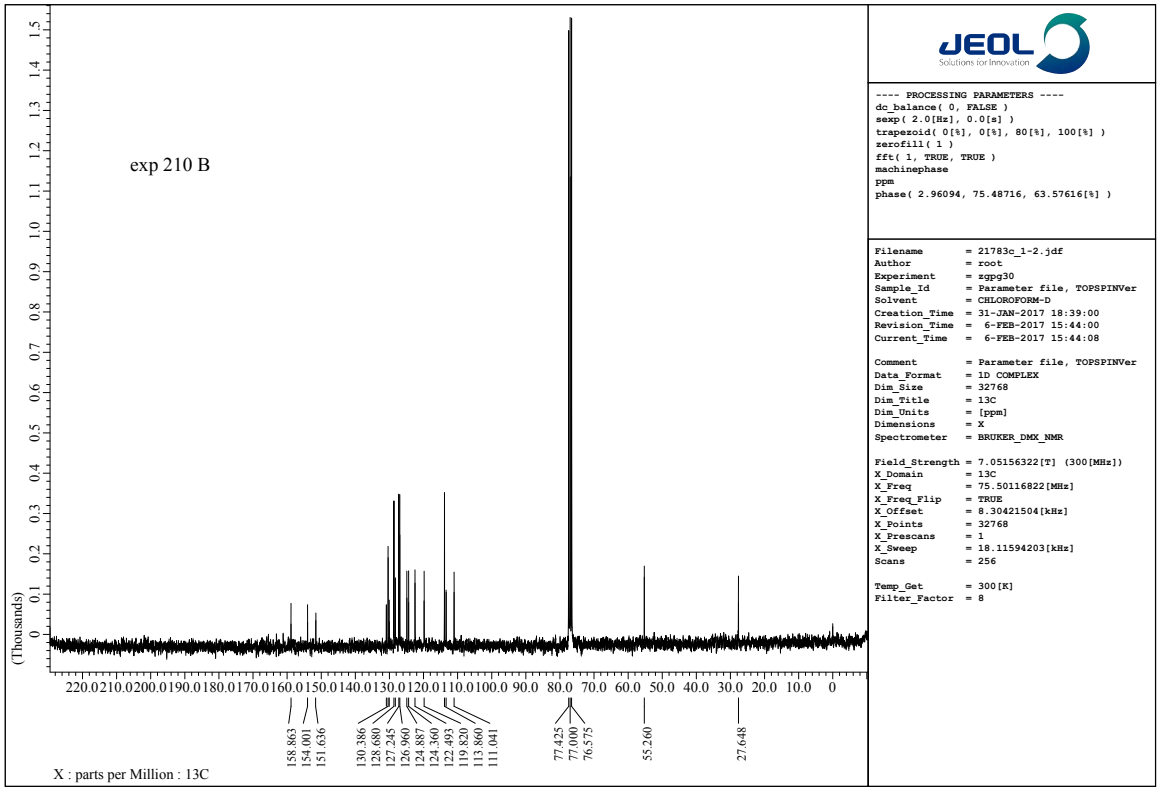
^{1}H and ^{13}C NMR

(*E*)-3-(3-(4-Methoxyphenyl)allyl)-2-phenylbenzofuran (3ac)



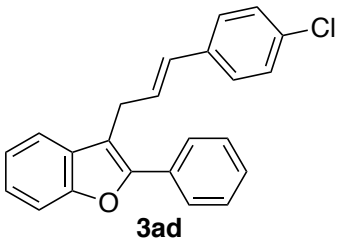
3ac



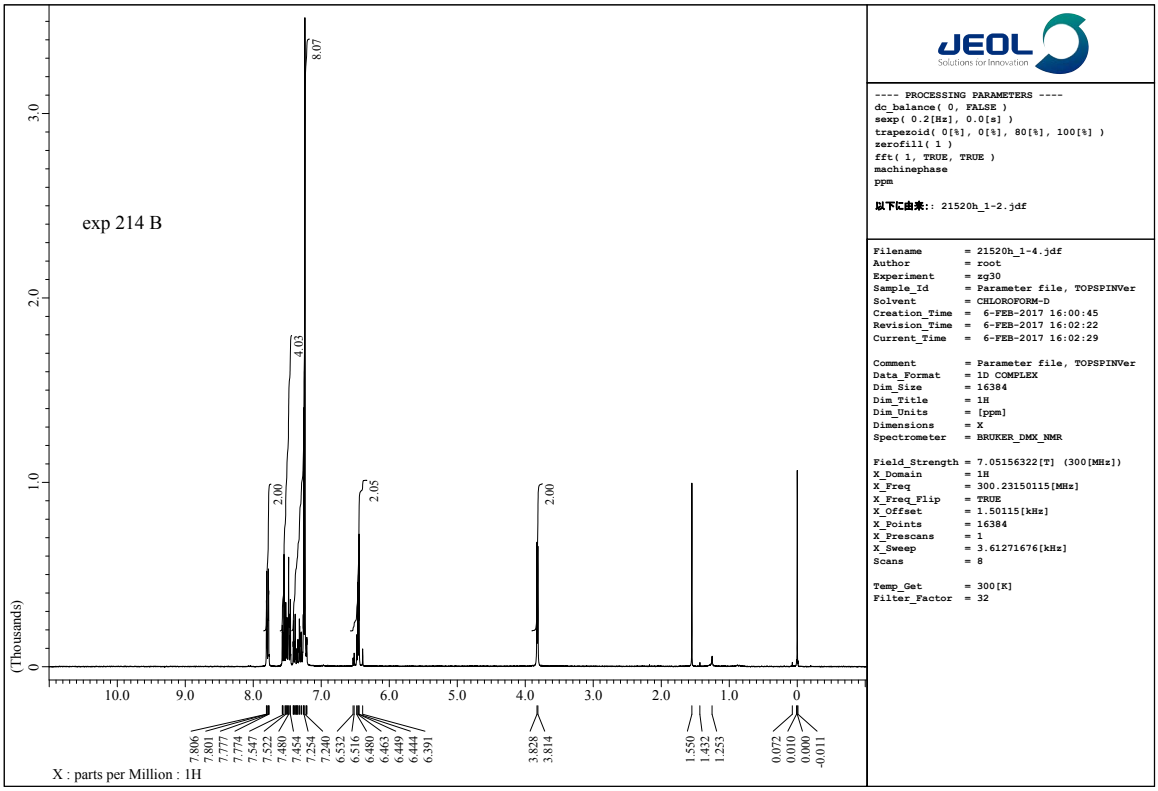


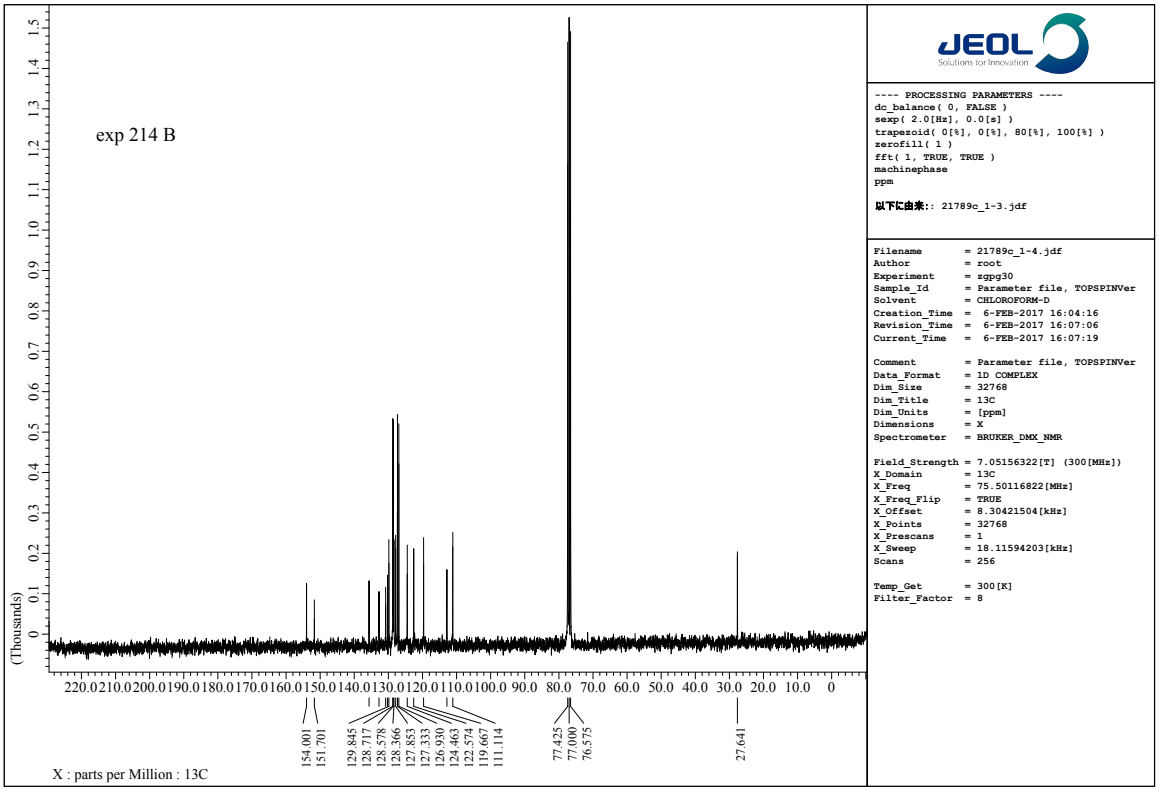
^{1}H and ^{13}C NMR

(*E*)-3-(3-(4-Chlorophenyl)allyl)-2-phenylbenzofuran (3ad)



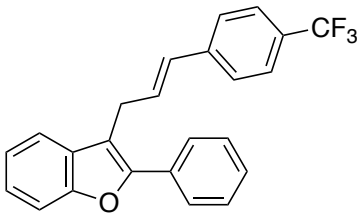
3ad



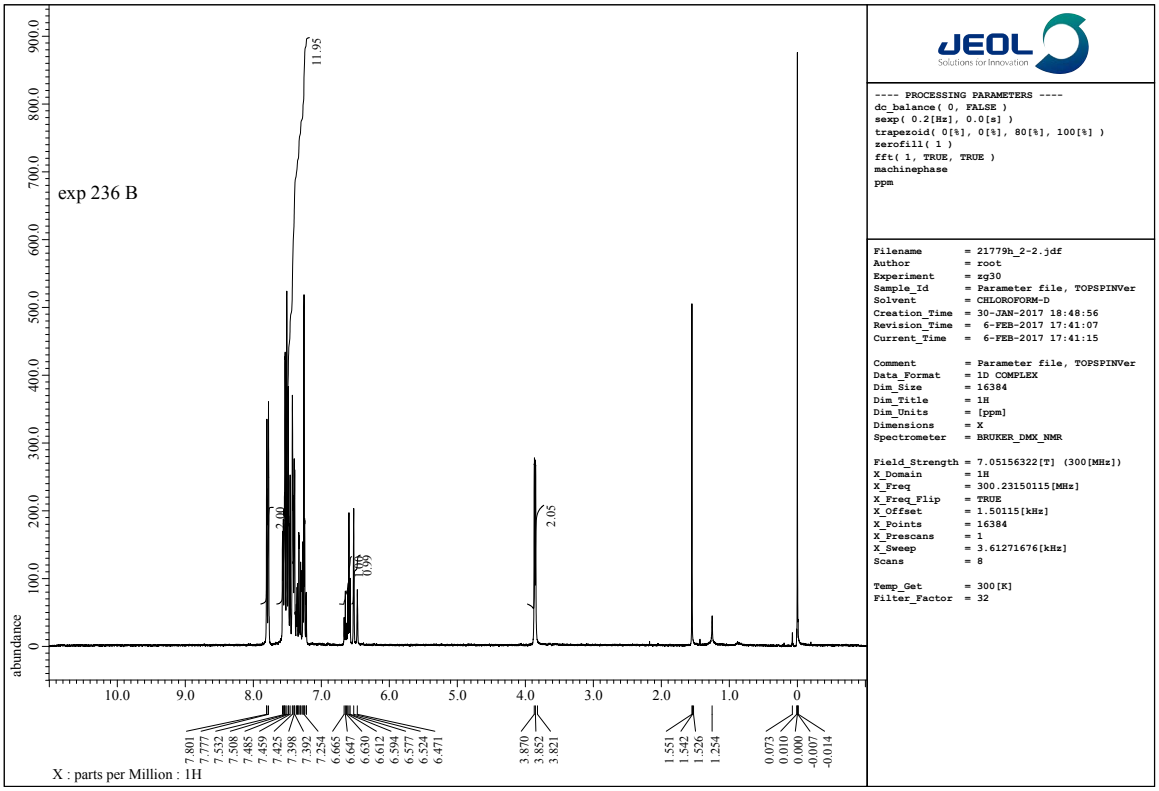


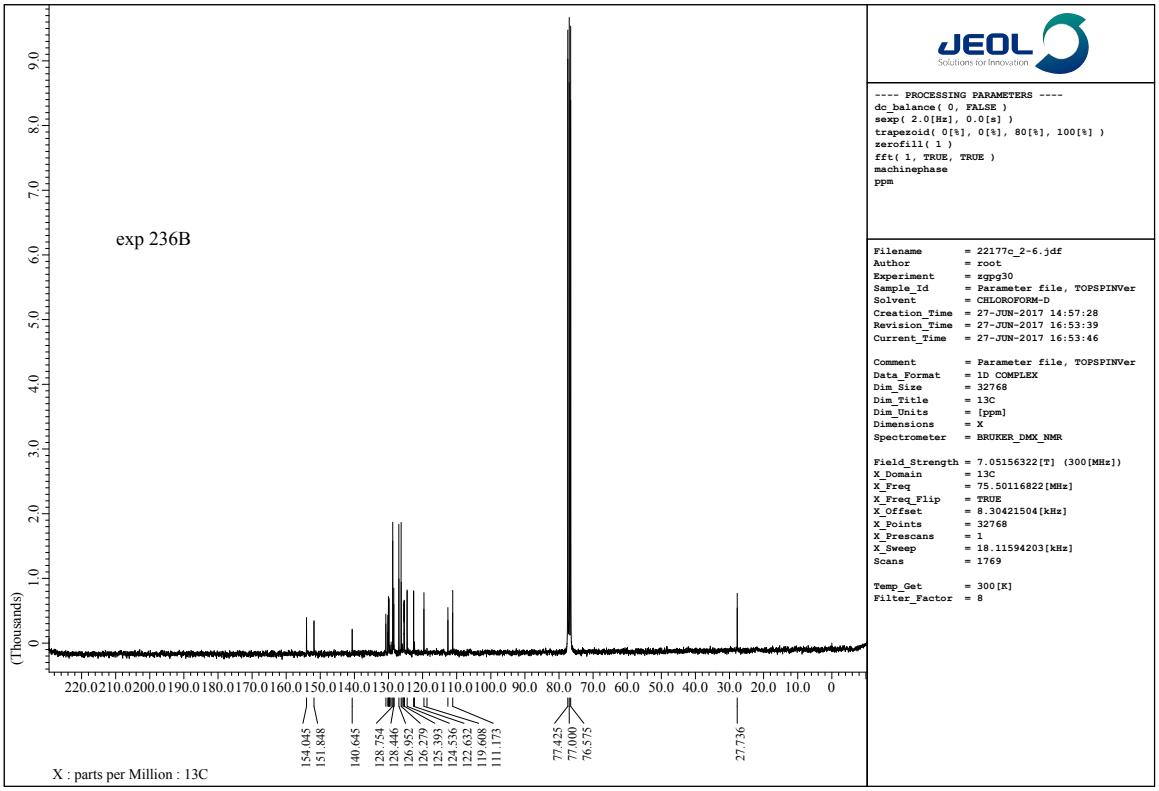
^{1}H and ^{13}C NMR

(*E*)-3-(3-(4-(Trifluoromethyl)phenyl)allyl)-2-phenylbenzofuran (3ae)



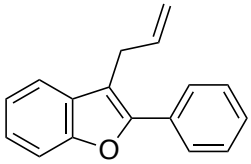
3ae



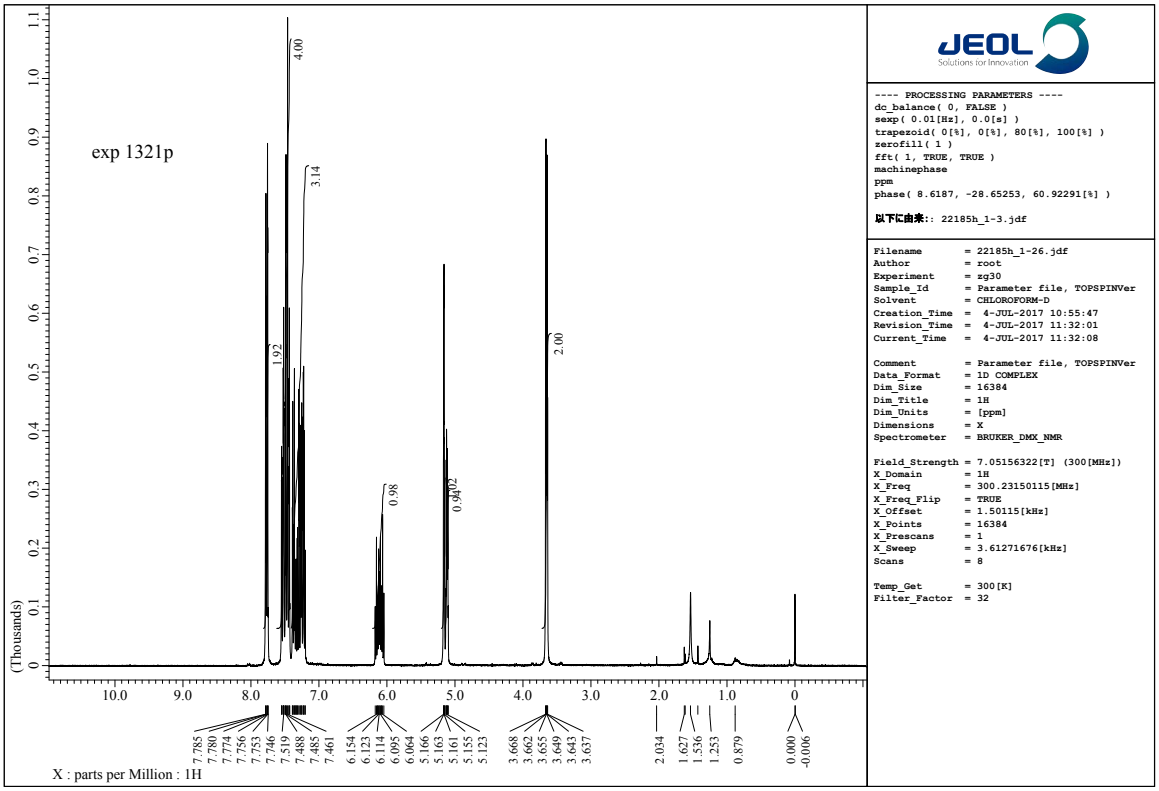


^{1}H and ^{13}C NMR

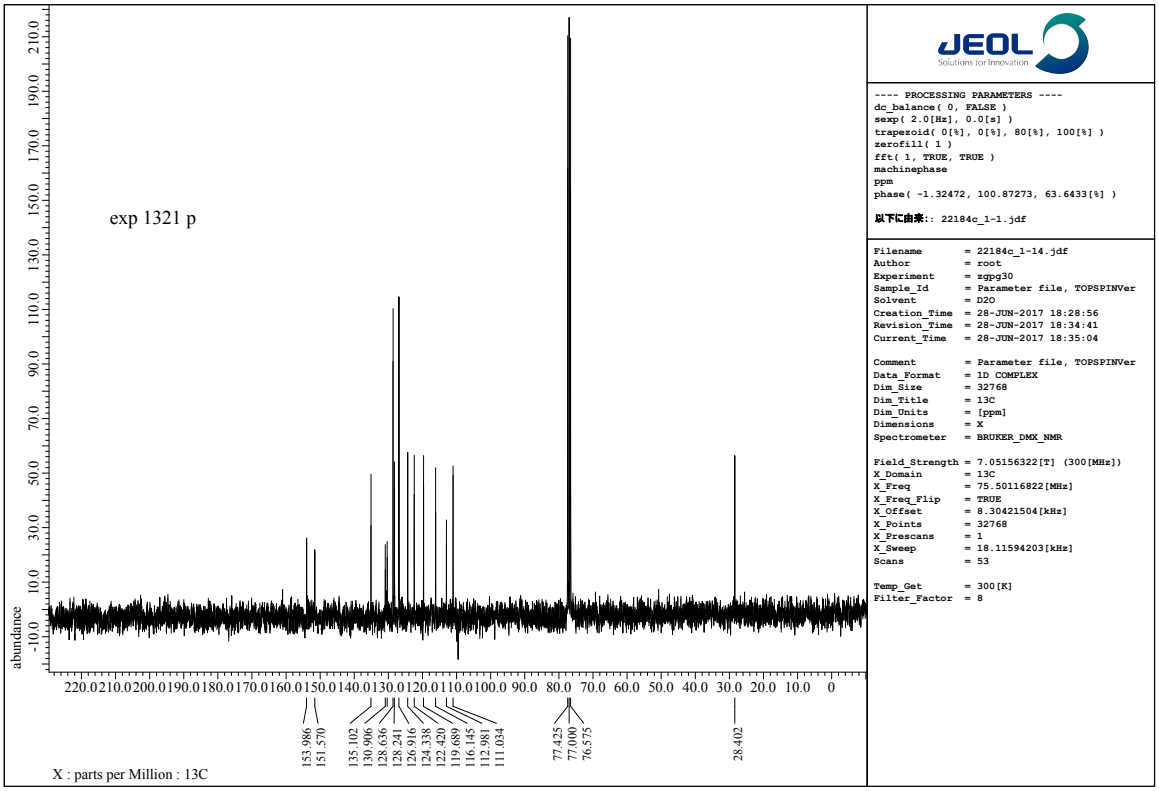
3-Allyl-2-phenylbenzofuran (3af)



3af



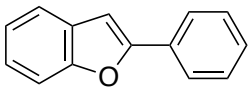

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.01[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm
phase( 8.6187, -28.65253, 60.92291[%] )

以下に由来:: 22185h_1-3.jdf

Filename        = 22185h_1-26.jdf
Author          = root
Experiment      = zg30
Sample_Id       = Parameter file, TOPSPINVer
Solvent         = CHLOROFORM-D
Creation_Time   =  4-JUL-2017 10:55:47
Revision_Time   =  4-JUL-2017 11:32:01
Current_Time    =  4-JUL-2017 11:32:08

Comment         = Parameter file, TOPSPINVer
Data_Format     = 1D COMPLEX
Dim_Size        = 16384
Dim_Title       = 1H
Dim_Units       = [ppm]
Dimensions      = X
Spectrometer    = BRUKER_DMX_NMR

Field_Strength = 7.05156322[T] (300[MHz])
X_Domain        = 1H
X_Freq          = 300.23150115[MHz]
X_Freq_Flip     = TRUE
X_Offset        = 1.50115[kHz]
X_Points        = 16384
X_Prescans      = 1
X_Sweep         = 3.61271676[kHz]
Scans           = 8

Temp_Get        = 300[K]
Filter_Factor   = 32
```



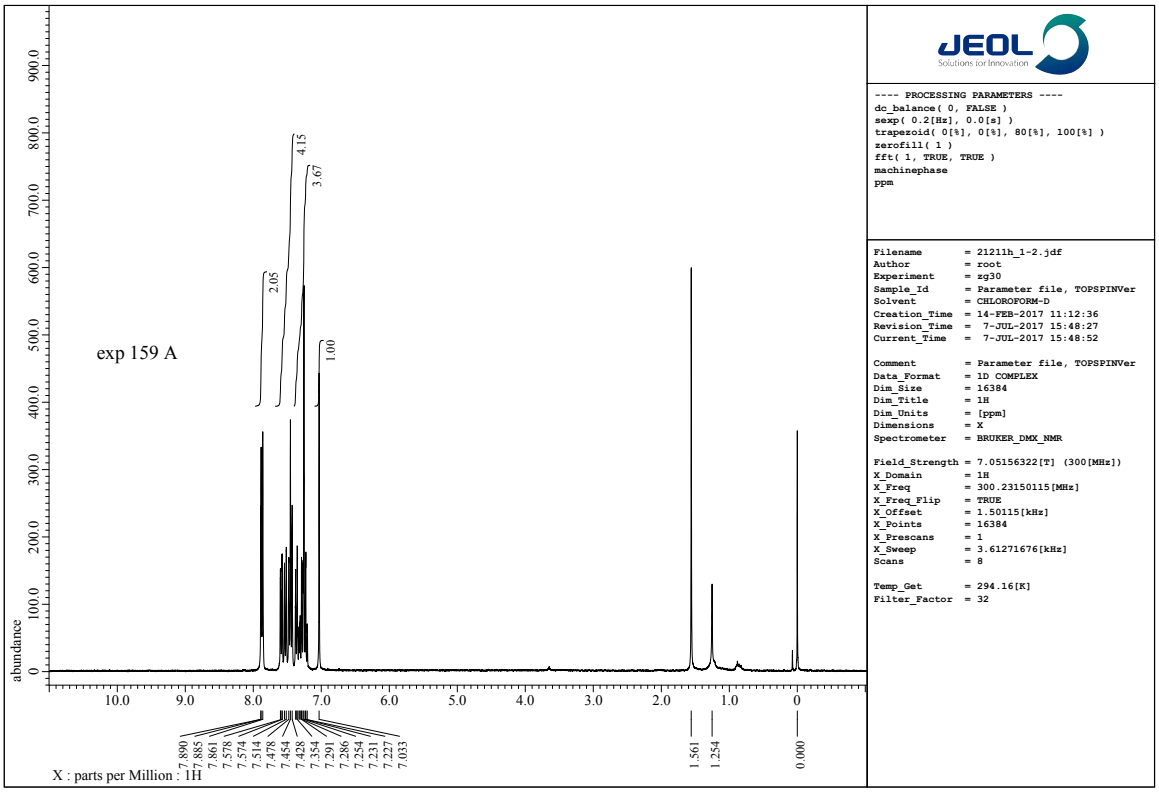

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm
phase( -1.32472, 100.87273, 63.6433[%] )

以下に由来:: 22184c_1-1.jdf

Filename        = 22184c_1-14.jdf
Author          = root
Experiment      = zgpg30
Sample_Id       = Parameter file, TOPSPINVer
Solvent         = D2O
Creation_Time   = 28-JUN-2017 18:28:56
Revision_Time   = 28-JUN-2017 18:34:41
Current_Time    = 28-JUN-2017 18:35:04

Comment         = Parameter file, TOPSPINVer
Data_Format     = 1D COMPLEX
Dim_Size        = 32768
Dim_Title       = 13C
Dim_Units       = [ppm]
Dimensions      = X
Spectrometer    = BRUKER_DMX_NMR

Field_Strength = 7.05156322[T] (300[MHz])
X_Domain        = 13C
X_Freq          = 75.50116822[MHz]
X_Freq_Flip     = TRUE
X_Offset        = 8.30421504[kHz]
X_Points        = 32768
X_Prescans      = 1
X_Sweep         = 18.11594203[kHz]
Scans           = 53

Temp_Get        = 300[K]
Filter_Factor   = 8
```

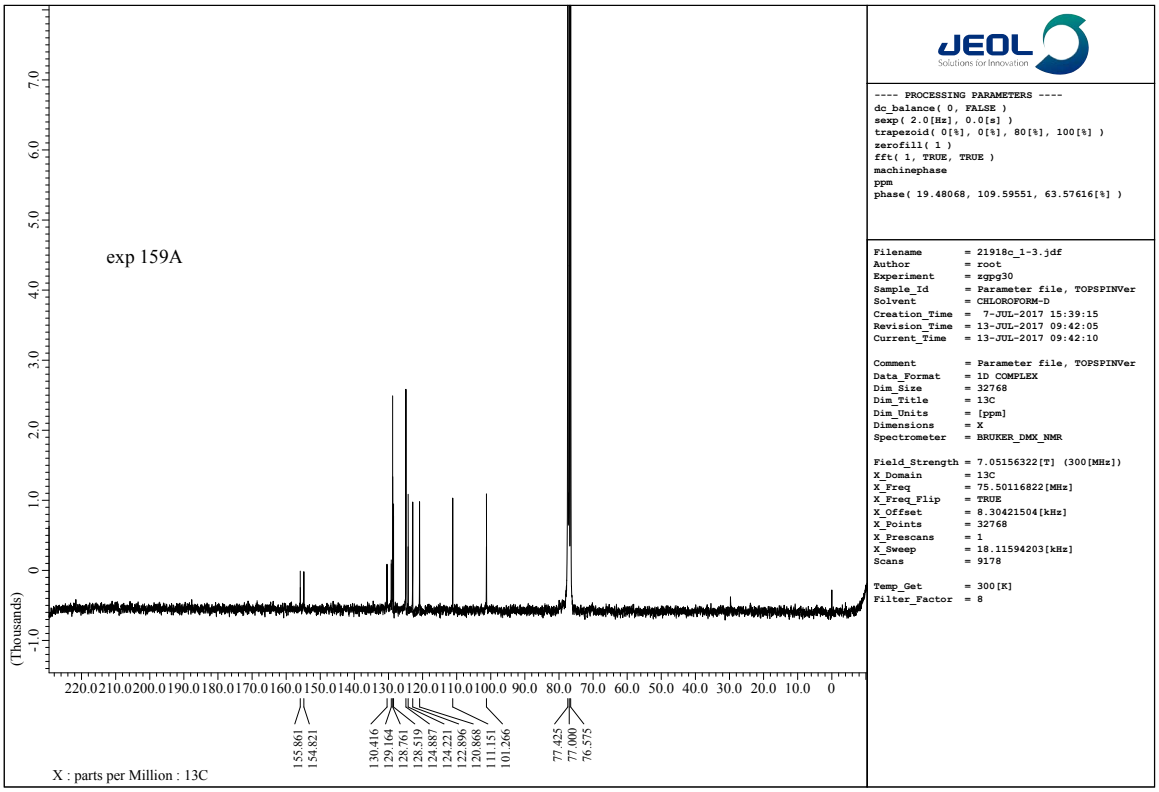
^{1}H and ^{13}C NMR

2-Phenylbenzofuran (4a)



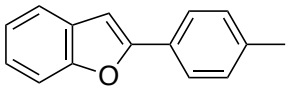
4a



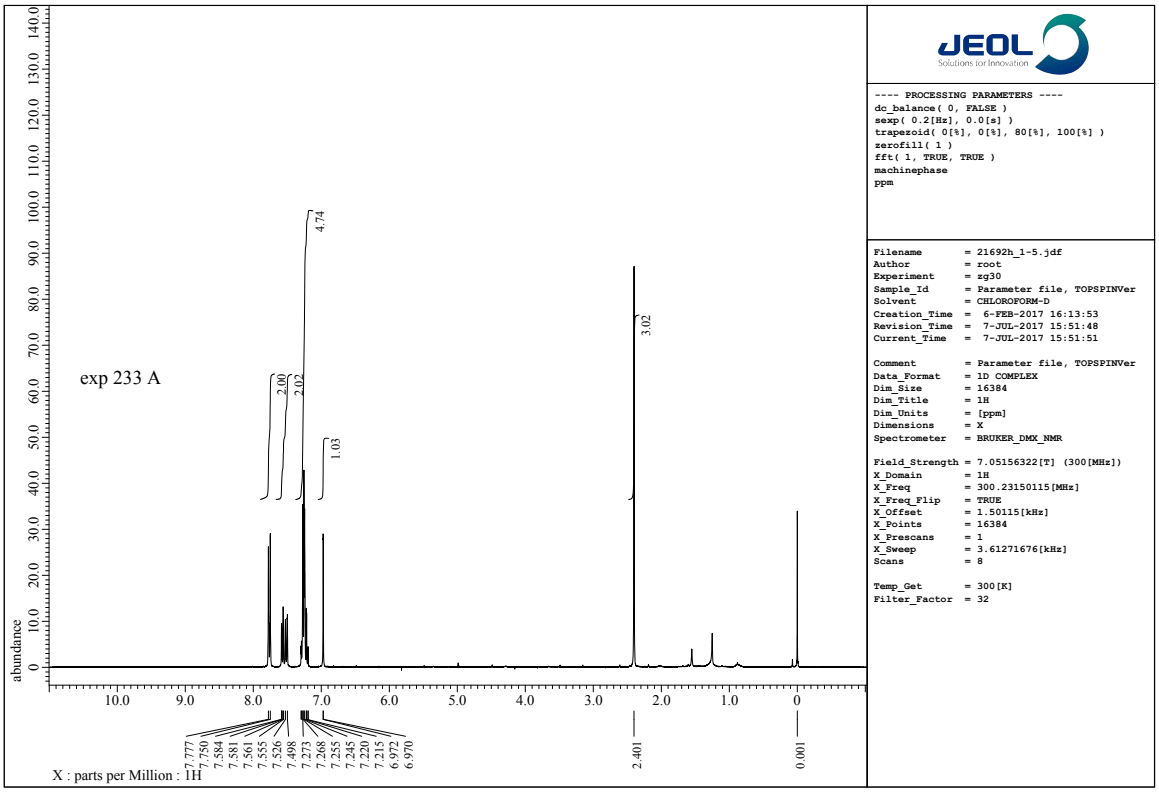


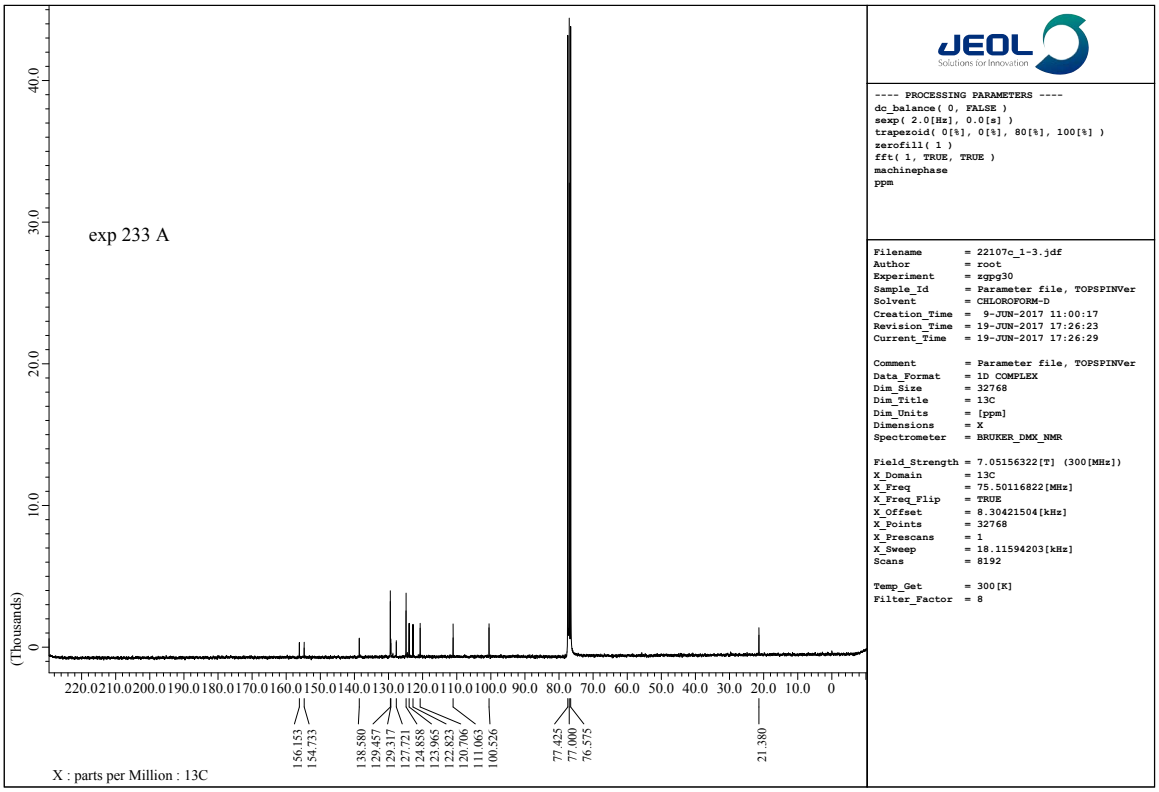
^{1}H and ^{13}C NMR

2-(*p*-Tolyl)benzofuran (4b)



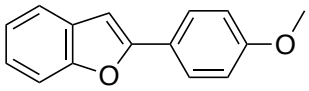
4b



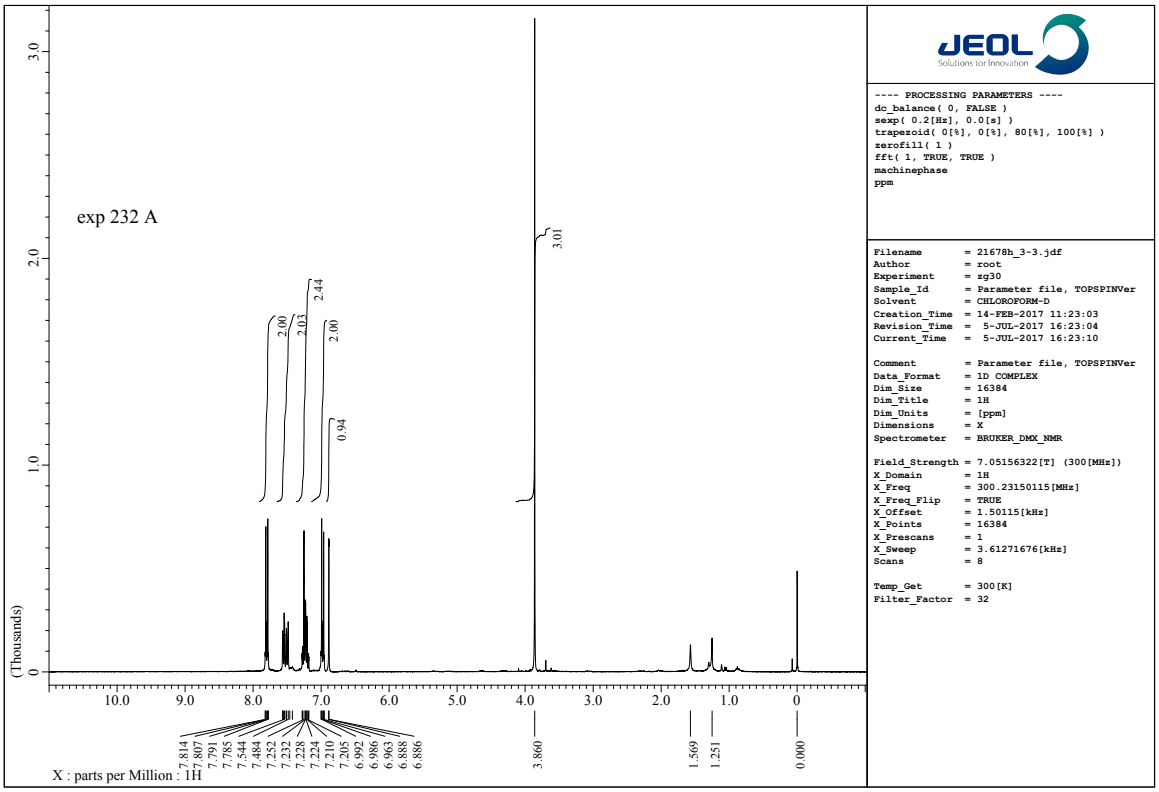


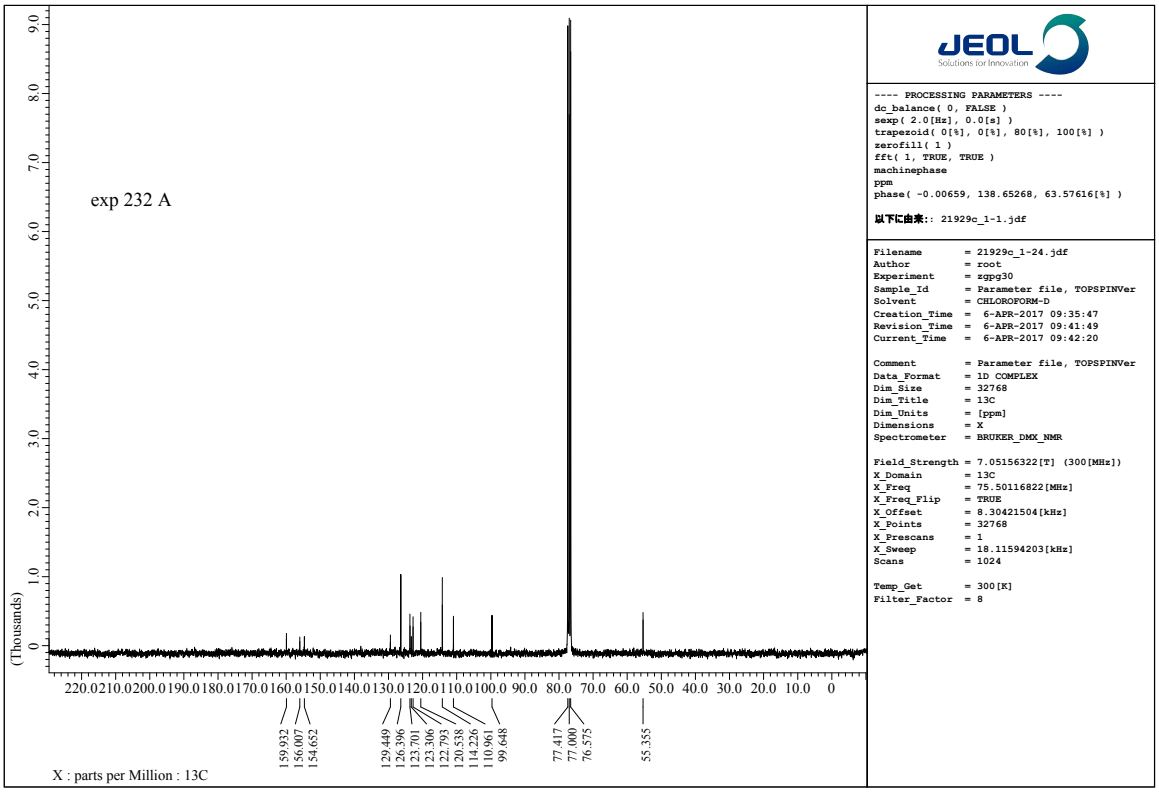
^{1}H and ^{13}C NMR

2-(4-Methoxyphenyl)benzofuran (4c)



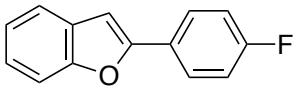
4c



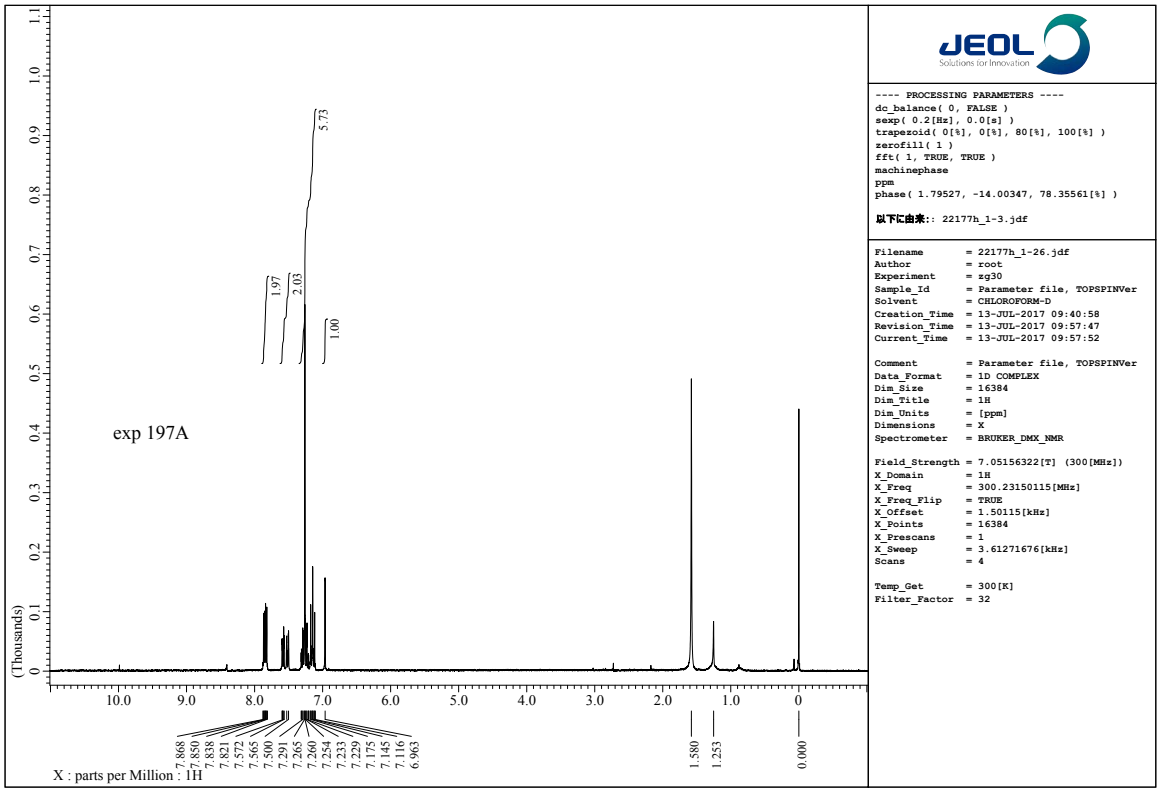


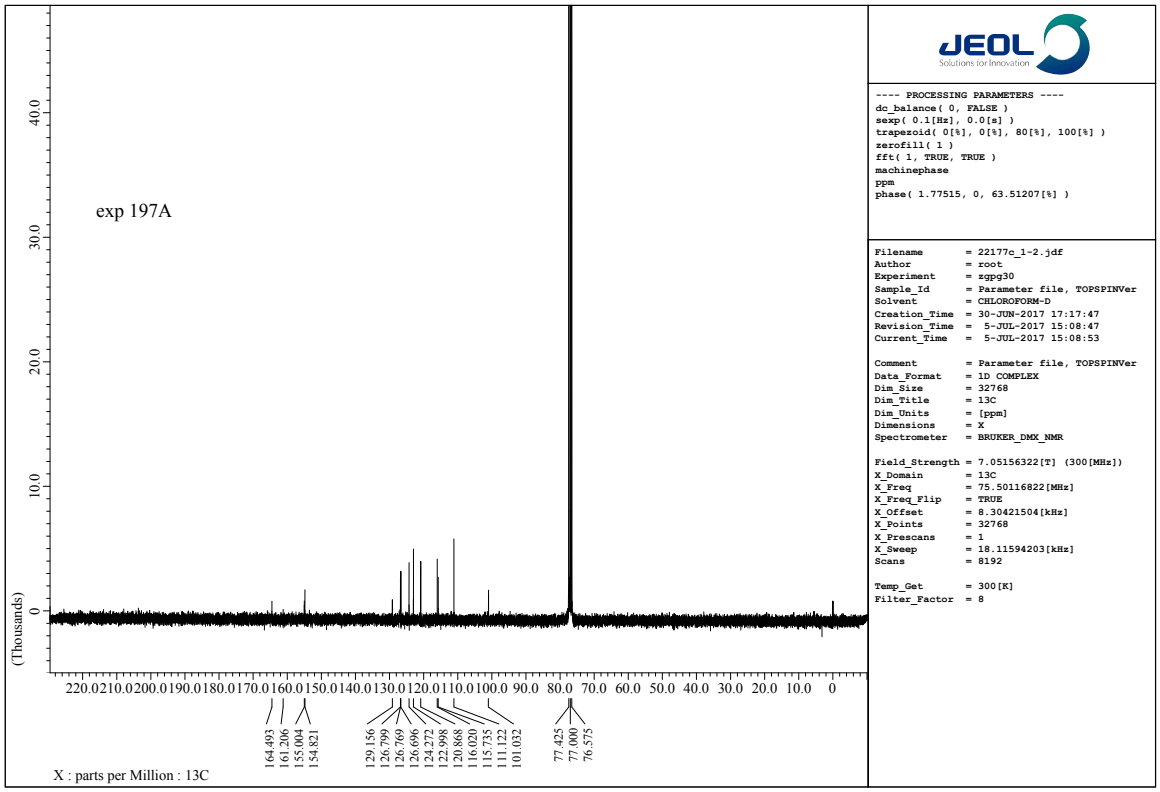
^{1}H and ^{13}C NMR

2-(4-Fluorophenyl)benzofuran (4d)



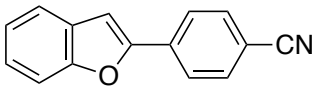
4d



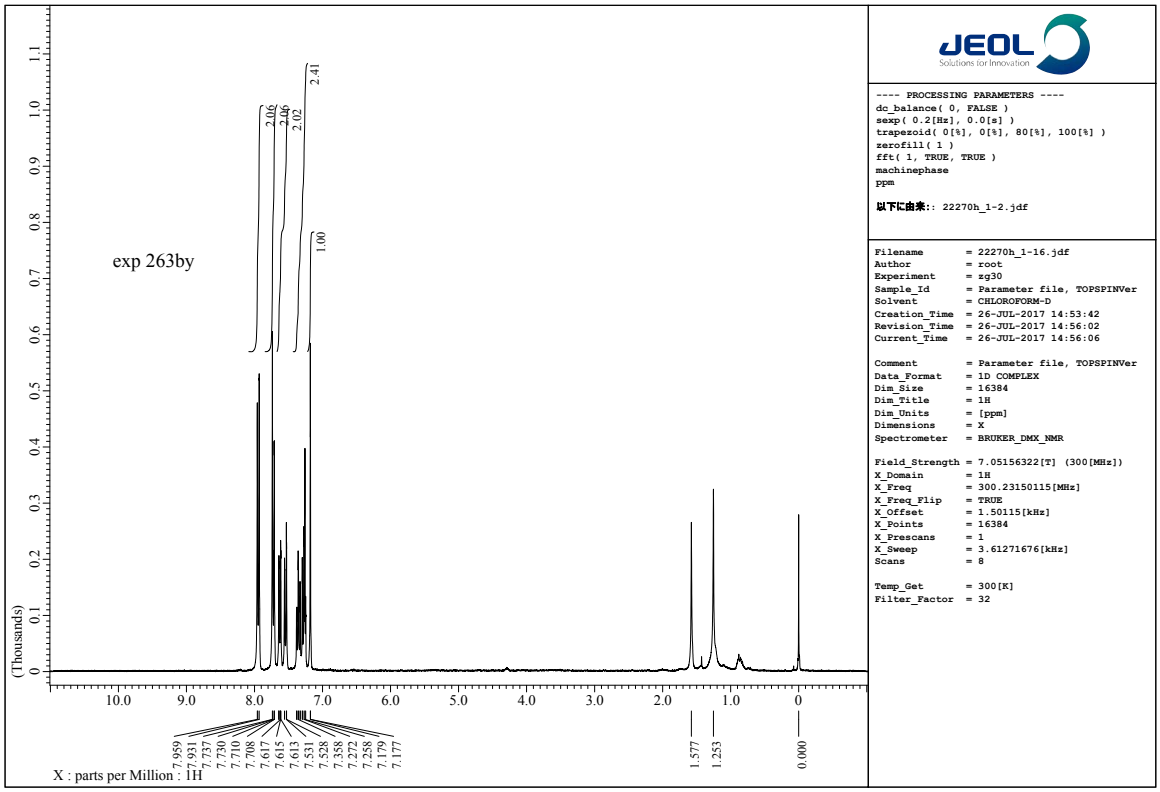


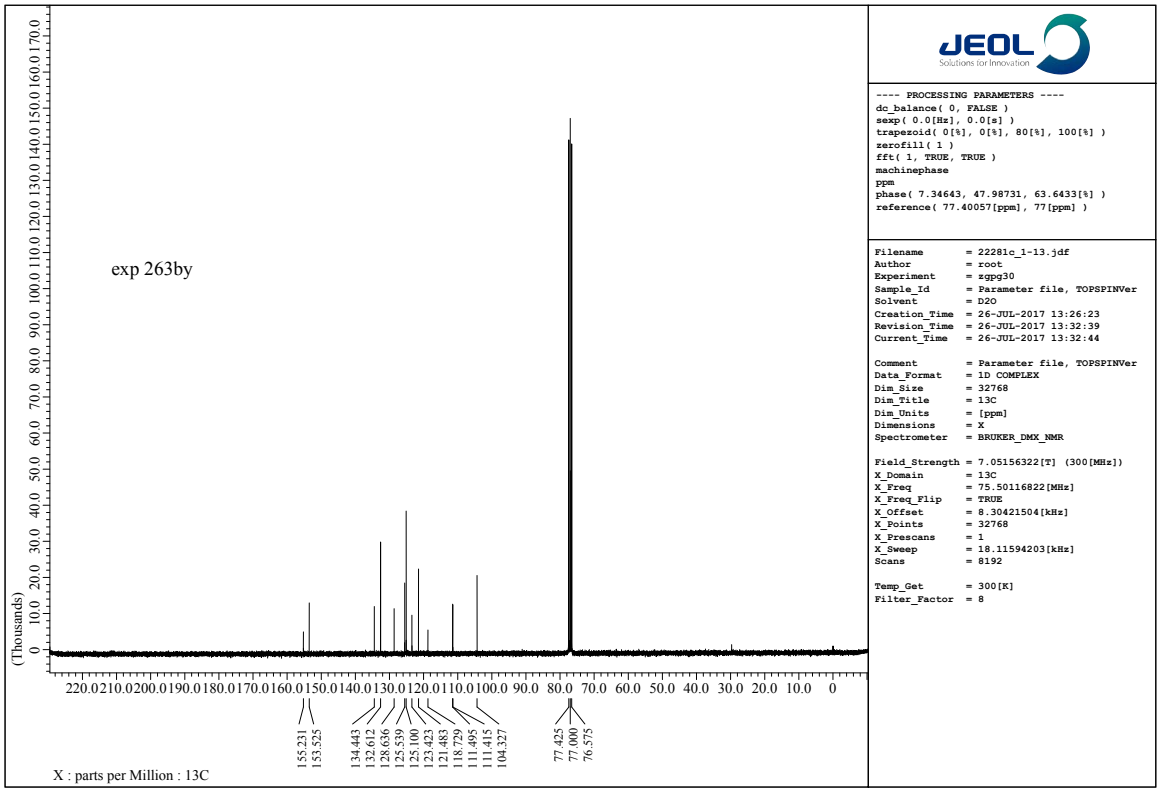
^{1}H and ^{13}C NMR

3-Cinnamyl-2-(4-cyanophenyl)benzofuran (4e)



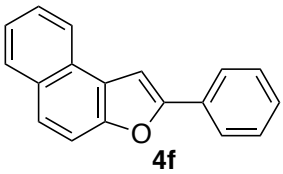
4e

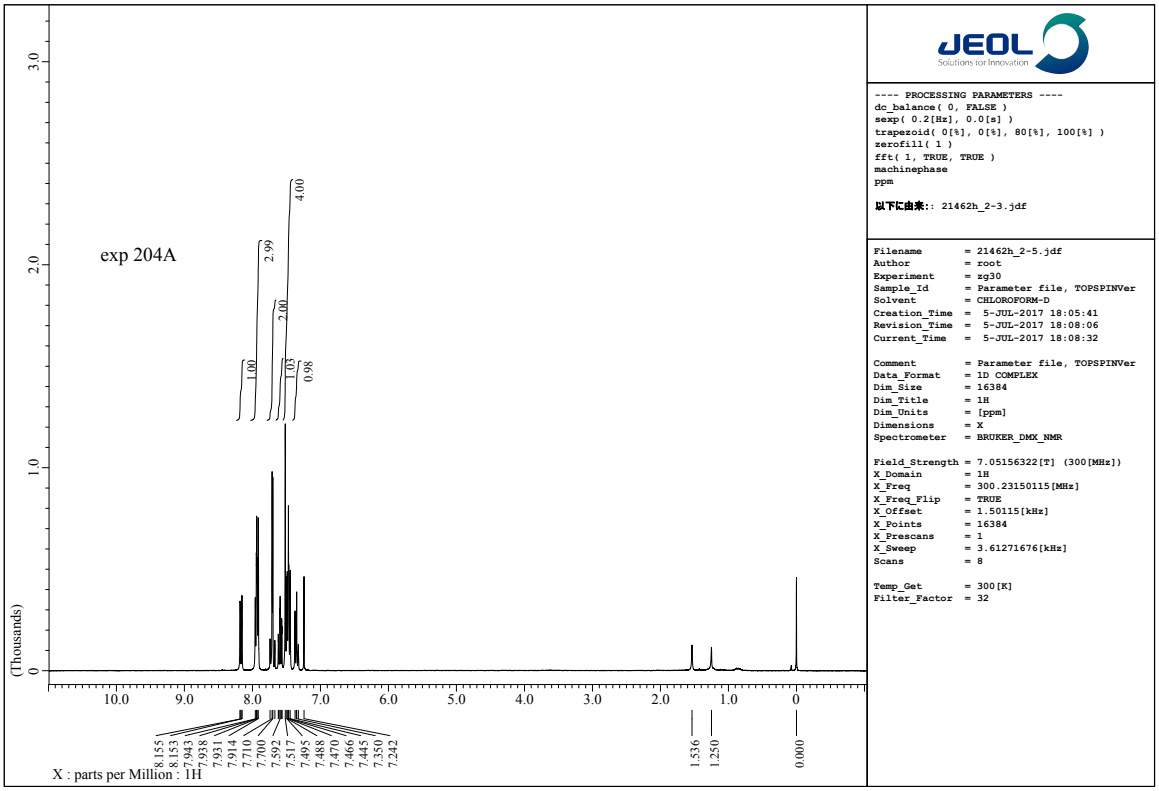


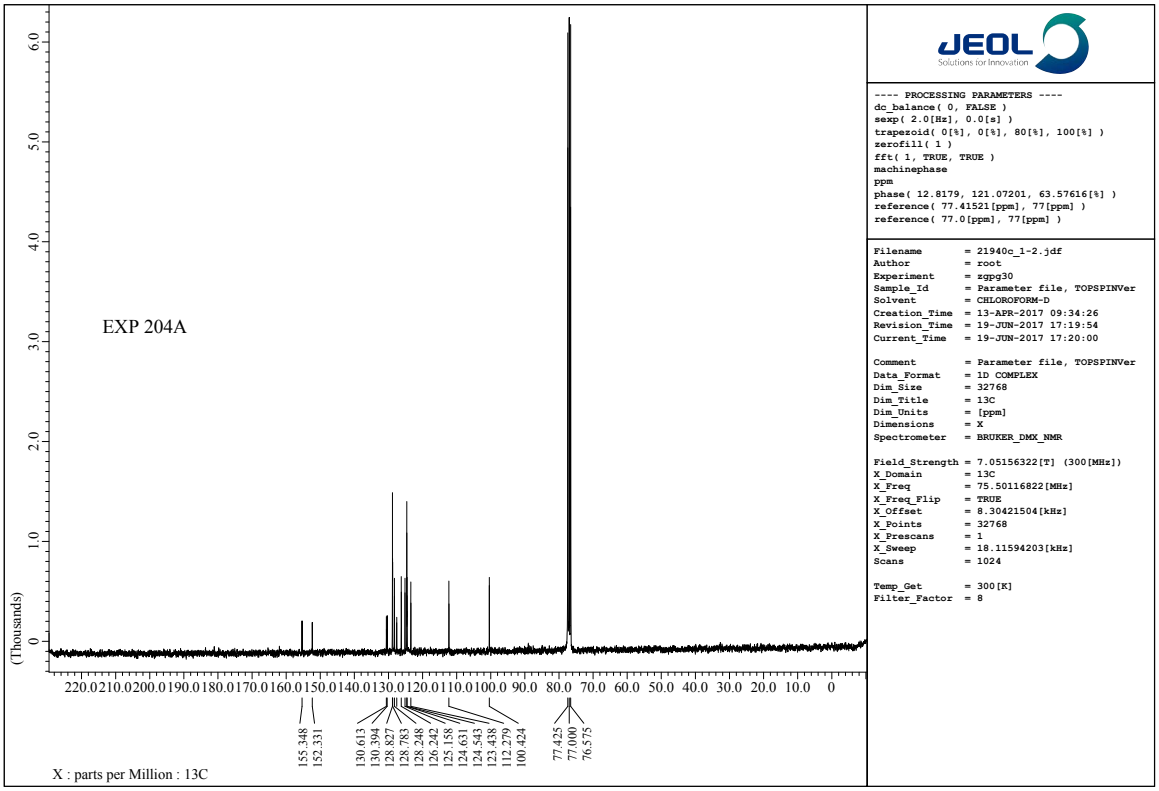


^{1}H and ^{13}C NMR

2-Phenylnaphtho[2,1-*b*]furan (4f)

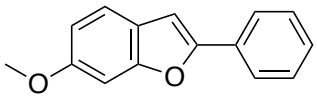




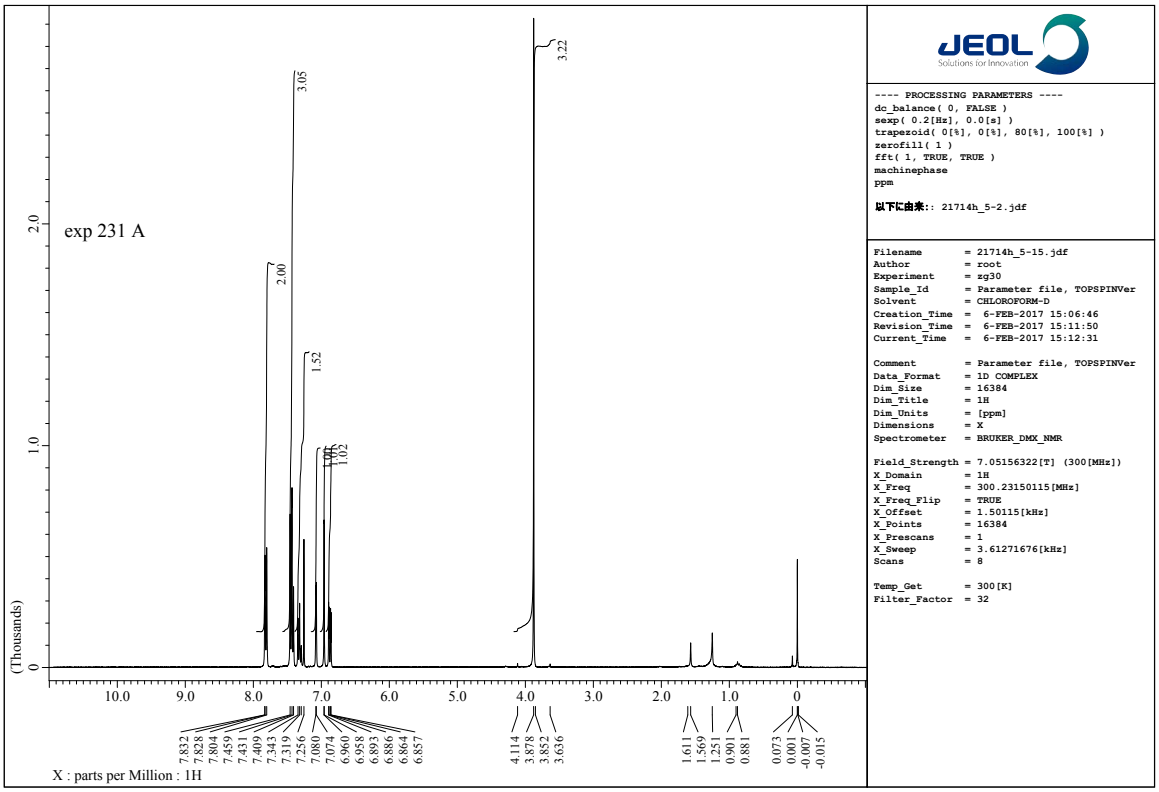


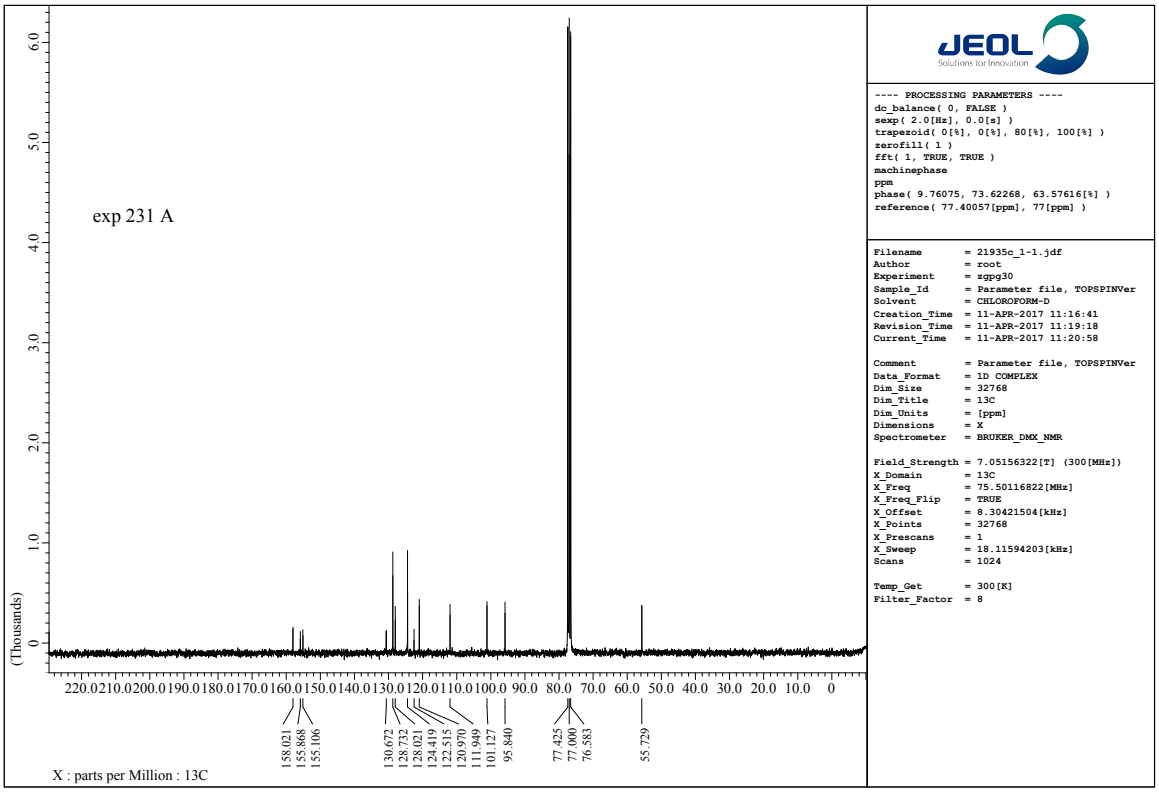
^{1}H and ^{13}C NMR

6-Methoxy-2-phenylbenzofuran (4g)



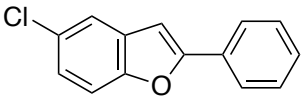
4g



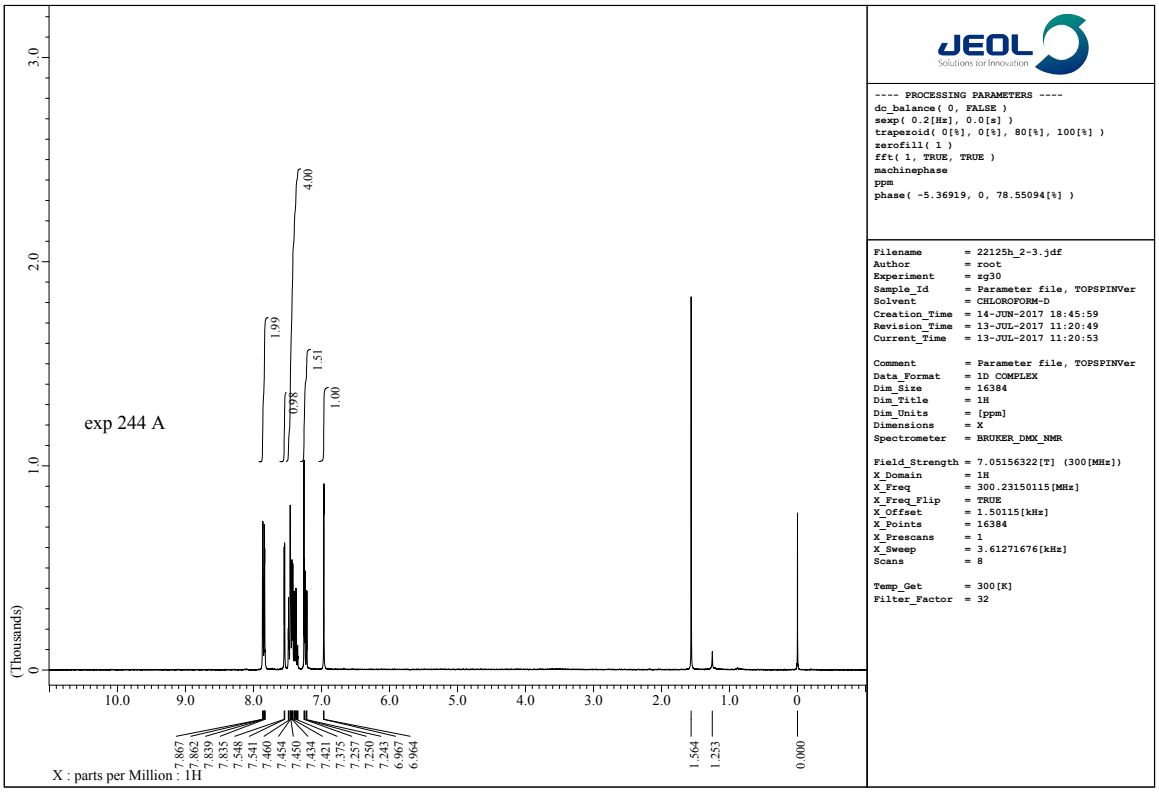


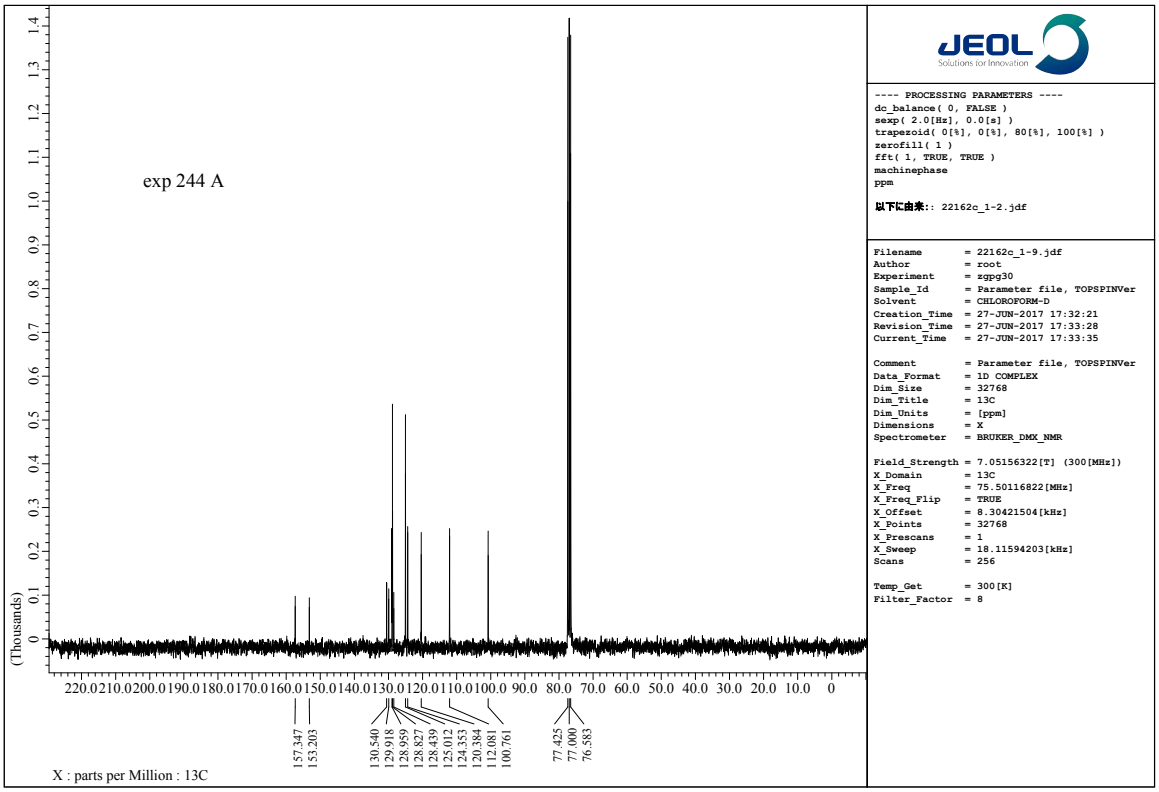
^{1}H and ^{13}C NMR

5-Chloro-2-phenylbenzofuran (4h)



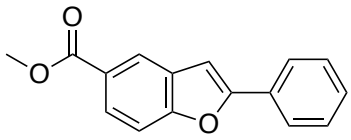
4h



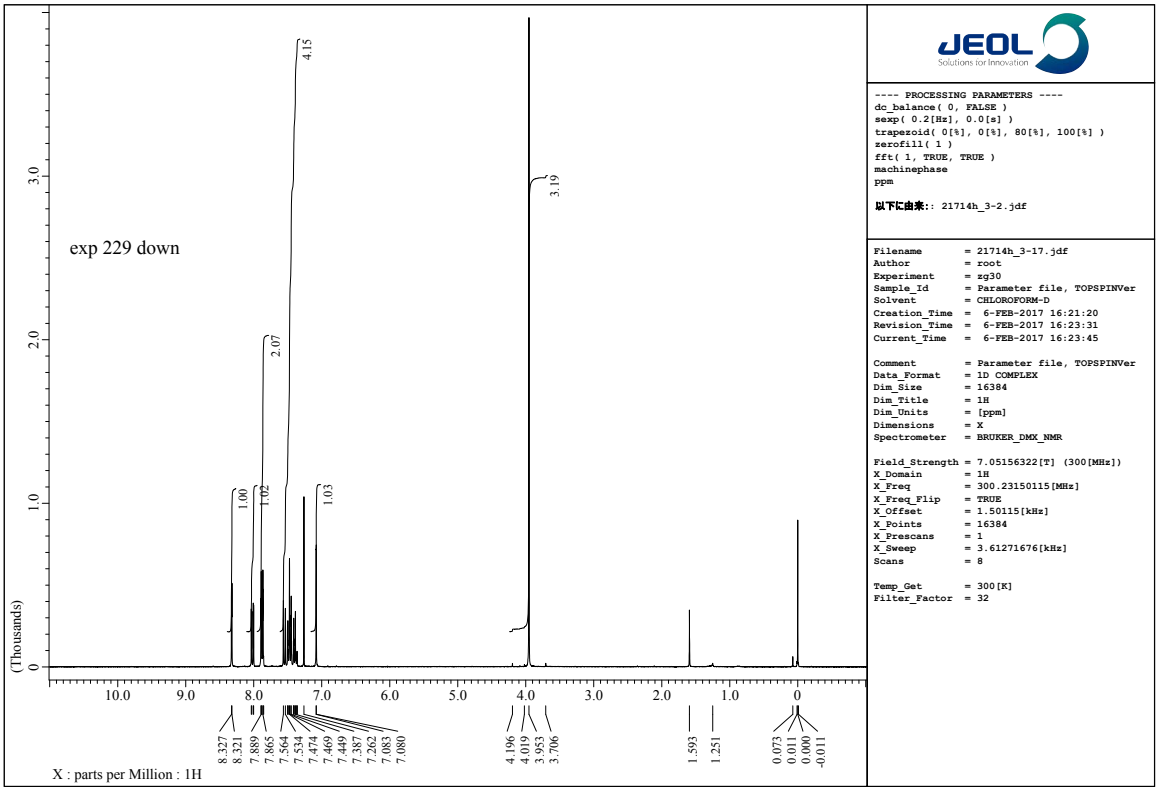


^{1}H and ^{13}C NMR

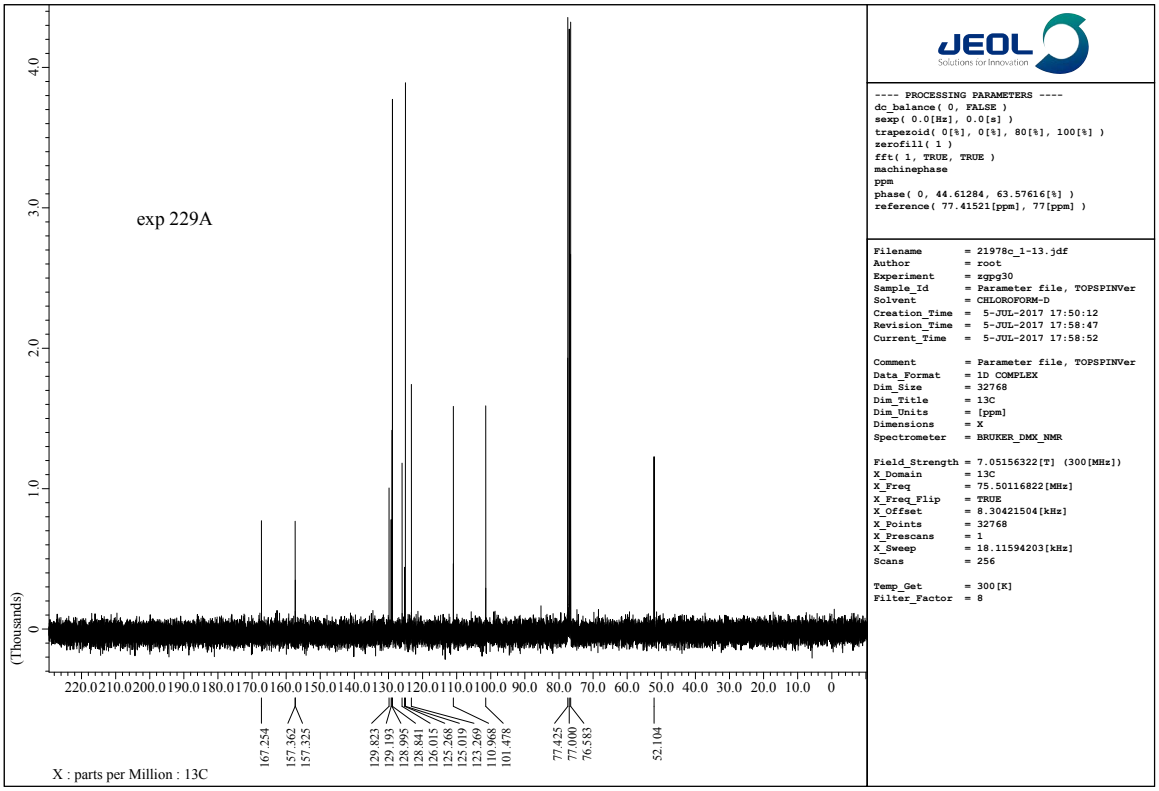
Methyl 2-phenylbenzofuran-5-carboxylate (4i)



4i



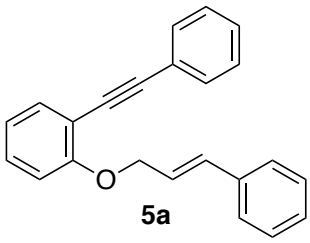

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

以下に由来:: 21714h_3-2.jdf

Filename        = 21714h_3-17.jdf
Author          = root
Experiment      = zg30
Sample_Id       = Parameter file, TOPSPINVer
Solvent         = CHLOROFORM-D
Creation_Time   =  6-FEB-2017 16:21:20
Revision_Time   =  6-FEB-2017 16:23:31
Current_Time    =  6-FEB-2017 16:23:45

Comment         = Parameter file, TOPSPINVer
Data_Format     = 1D COMPLEX
Dim_Size        = 16384
Dim_Title       = 1H
Dim_Units       = [ppm]
Dimensions      = X
Spectrometer    = BRUKER_DMX_NMR

Field_Strength = 7.05156322[T] (300[MHz])
X_Domain        = 1H
X_Freq          = 300.23150115[MHz]
X_Freq_Flip     = TRUE
X_Offset        = 1.50115[kHz]
X_Points        = 16384
X_Prescans      = 1
X_Sweep         = 3.61271676[kHz]
Scans           = 8

Temp_Get        = 300[K]
Filter_Factor   = 32
```




```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm
phase( 0, 44.61284, 63.57616[%] )
reference( 77.41521[ppm], 77[ppm] )

Filename        = 21978c_1-13.jdf
Author          = root
Experiment      = zgpg30
Sample_Id       = Parameter file, TOPSPINVer
Solvent         = CHLOROFORM-D
Creation_Time   =  5-JUL-2017 17:50:12
Revision_Time   =  5-JUL-2017 17:58:47
Current_Time    =  5-JUL-2017 17:58:52

Comment         = Parameter file, TOPSPINVer
Data_Format     = 1D COMPLEX
Dim_Size        = 32768
Dim_Title       = 13C
Dim_Units       = [ppm]
Dimensions      = X
Spectrometer    = BRUKER_DMX_NMR

Field_Strength = 7.05156322[T] (300[MHz])
X_Domain        = 13C
X_Freq          = 75.50116822[MHz]
X_Freq_Flip     = TRUE
X_Offset        = 8.30421504[kHz]
X_Points        = 32768
X_Prescans      = 1
X_Sweep         = 18.11594203[kHz]
Scans           = 256

Temp_Get        = 300[K]
Filter_Factor   = 8
```

^{1}H and ^{13}C NMR

1-Cinnamyloxy-2-(phenylethynyl)benzene (5a)



5a