Supporting Information

Fluoro-Julia Olefination as a Mild, High-Yielding Route to α-Fluoro Acrylonitriles

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GENERAL EXPERIMENTAL METHODS

THF was distilled over $LiAlH_4$ and then over sodium, and CH_2Cl_2 was distilled over $CaCl_2$. DMF and CH_3CN were obtained from commercial sources and were used without further purification. For reactions that were performed under a nitrogen atmosphere, glassware was flame dried under vacuum. Dry sodium hydride (95%) was used for the reactions, fluorinating reagent NFSI was obtained from Honeywell and fluorinating reagent Selectfluor was obtained from Air Products (both, NFSI and Selectfluor are commercially available). Ethyl (1,3-benzothiazol-2-ylsulfanyl)acetate (**10**),[1] (1-phenyl-1*H*-tetrazol-5-ylsulfanyl)acetonitrile (**2b**),[2] (1-Boc-imidazole-4-carboxaldehyde)[3] and 1-Boc-indole-3-carboxaldehyde[3,4] are known compounds and were synthesized as reported. Our syntheses of (1,3-benzothiazol-2-ylsulfanyl)acetonitrile (**2a**)[5] and ethyl (1,3-benzothiazol-2-ylsulfanyl)fluoroacetate (**8**)[6] described herein are minor modifications of reported literature procedures. All other reagents were obtained from commercial sources and used without further purification. Thin layer chromatography was performed on 250 μm silica plates and column chromatographic purifications were performed on 200-300 mesh silica gel. For products **25**–**27**, $KMnO_4$ stain[7] was used for TLC detection. ^{1}H NMR spectra were recorded at 500 MHz in $CDCl_3$ and were referenced to residual $CHCl_3$ or to tetramethylsilane (TMS). ^{13}C NMR spectrum of **4** was recorded at 125 MHz and was referenced to $CDCl_3$ (see page S15). ^{19}F NMR spectra were recorded at 282 MHz using $CFCl_3$ as internal standard. Chemical shifts (δ) are reported in parts per million and coupling constants (*J*) are in hertz.

(1,3-Benzothiazol-2-ylsulfanyl)acetonitrile (2a).[5]

To a solution of bromoacetonitrile (**1**, 1.00 g, 8.34 mmol, 1 molar equiv) in DMF (17.5 mL) at rt, the sodium salt of 2-mercapto-1,3-benzothiazole (2.05 g, 10.8 mmol, 1.3 molar equiv) was added and the reaction mixture was allowed to stir for 3 h (TLC showed complete consumption of the starting material). The reaction mixture was diluted with water and extracted with EtOAc (3x). The combined organic layer was thoroughly washed with water and brine, dried over anhydrous Na_2SO_4 and the solvent was evaporated under reduced pressure. The crude product was purified by column chromatography on silica gel using CH_2Cl_2 to yield **2a** as white solid (1.52 g, 89%). Mp (recrystallized from 10% EtOAc in hexanes) 78-79 °C. ^{1}H NMR (500 MHz): δ 7.94 (d, 1H, Ar-H, *J* = 8.2), 7.80 (d, 1H, Ar-H, *J* = 7.9), 7.47 (t, 1H, Ar-H, *J* = 7.6), 7.36 (t, 1H, Ar-H, *J* = 7.6), 4.20 (s, 2H, CH_2CN). HRMS (positive ion ESI): calcd. for $C_9H_6N_2S_2Na^+$ (M^+ + Na) 228.986461, found 228.985874.

Ethyl (1,3-Benzothiazol-2-ylsulfanyl)fluoroacetate (8) from Commercial Fluorinated Precursor 7.[6]

To a solution of ethyl bromofluoroacetate (**7**, 3.00 g, 16.2 mmol, 1 molar equiv) in DMF (40.0 mL) at rt, the sodium salt of 2-mercapto-1,3-benzothiazole (3.15 g, 16.6 mmol, 1.03 molar equiv) was added and the reaction mixture was allowed to stir for 1 h (TLC showed complete consumption of the starting material). The reaction mixture was diluted with water and extracted with EtOAc (3x). The combined organic layer was thoroughly washed with water and brine, dried over anhydrous Na_2SO_4 and the solvent was evaporated under reduced pressure. The crude product was purified by column chromatography on silica gel using CH_2Cl_2 to yield **8** as a clear thick pale yellow liquid (3.89 g, 88%). For characterization data please refer to synthesis of **8** via fluorination of **10** in the Experimental Section of the paper.

(1,3-Benzothiazol-2-ylsulfanyl)fluoroacetonitrile (6a).[5]

For analytical purposes, a small sample of crude **6a** was purified by column chromatography (SiO_2, 50% ethyl acetate in hexanes). White solid, mp (recrystallized from 3% EtOAc in hexanes) 48-49 °C. ^{1}H NMR (500 MHz): δ 8.01 (d, 1H, Ar-H, J = 7.9), 7.85 (d, 1H, Ar-H, J = 7.9), 7.52 (t, 1H, Ar-H, J = 7.6), 7.43 (t, 1H, Ar-H, J = 7.6), 7.31 (d, 1H, CHF, $^2J_{FH}$ = 49.4). ^{19}F NMR (282 MHz): δ -156.9 (d, $^2J_{FH}$ = 48.8). HRMS (positive ion ESI): calcd. for $C_9H_5FN_2S_2H^+$ (M^+ + H) 224.995094, found 224.994774.

(1-Phenyl-1*H*-tetrazol-5-ylsulfanyl)fluoroacetonitrile (6b).[2]

A stirred solution of (1-phenyl-1*H*-tetrazol-5-ylsulfanyl)acetonitrile (**2b**, 0.600 g, 2.76 mmol,1 molar equiv) in dry toluene (24 mL), was cooled to –78 ºC (dry-ice/*iso*-PrOH) and under nitrogen, *t*-BuLi (1.79 mL of a 1.7 M solution in pentane, 3.04 mmol, 1.1 molar equiv) was added to the reaction mixture. After 12 min solid NFSI (1.05 g, 3.32 mmol, 1.2 molar equiv) was added. The mixture was allowed to stir at –78 ºC for 50 min, then warmed to rt and stirring was continued for an additional 1 h. Sat aq NH_4Cl was added to the mixture and the layers were separated. The aqueous layer was extracted with EtOAc (3x), and the combined organic layer was washed with sat aq $NaHCO_3$ and brine. The organic layer was dried over Na_2SO_4 and the solvent was evaporated under reduced pressure. The ^{1}H NMR of the crude reaction mixture showed the presence of starting **2b** and monofluoro derivative **6b**, and ^{19}F NMR showed monofluoro **6b** and difluoro derivative. Purification of the crude reaction mixture by column chromatography (silica gel, eluted by CH_2Cl_2) afforded **6b** as a thick, pale-reddish liquid (0.294

g, 45%). ^{1}H NMR (500 MHz): δ 7.64-7.62 (m, 3H, Ar-H), 7.52-7.50 (m, 2H, Ar-H), 7.27 (d, 1H, CHF, $^2J_{FH}$ = 48.8). ^{19}F NMR (282 MHz): δ -157.7 (d, $^2J_{FH}$ = 47.0).

Table 1. ^{19}F NMR data and HRMS data (or lit. reference for known compounds) of **12**–**27**.

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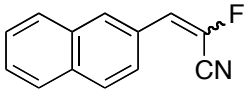
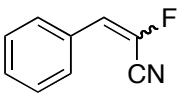
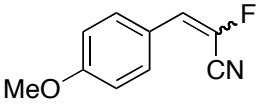
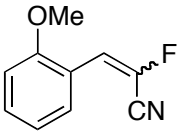
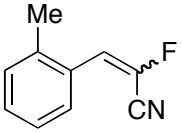
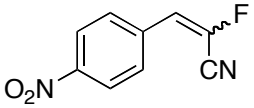
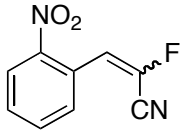
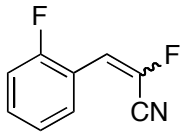
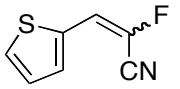
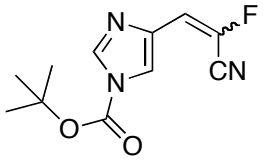
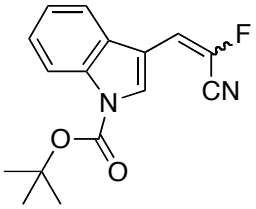
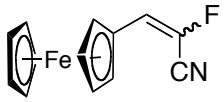
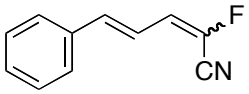
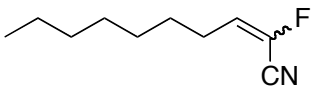
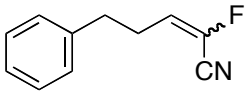
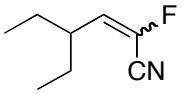
Compound	^{19}F NMR data ($CDCl_3$)	Ionization method and HRMS data or lit. reference
***E*- and *Z*-12.** 	282 MHz: δ -122.5 (d, $^3J_{FH}$ =15.3, *E* isomer), δ -122.1 (d, $^3J_{FH}$ = 36.6, *Z* isomer)	HRMS (positive ion APPI): calcd. for $C_{13}H_8FN^+$ (M^+) 197.063529, found 197.063385
***E*- and *Z*-13.** 	282 MHz: δ -121.9 (d, $^3J_{FH}$ = 35.2, *Z* isomer), δ -122.6 (d, $^3J_{FH}$ = 17.6, *E* isomer)	Lit. ref.: 8, 9
***E*- and *Z*-14.** 	282 MHz: δ -126.2 (d, $^3J_{FH}$ = 36.6, *Z* isomer), δ -127.1 (d, $^3J_{FH}$ = 18.3, *E* isomer)	Lit. ref.: 9, 10
***E*- and *Z*-15.** 	282 MHz: δ -122.3 (d, $^3J_{FH}$ = 15.3, *E* isomer), δ -124.11 (d, $^3J_{FH}$ = 36.6, *Z* isomer)	HRMS (positive ion ESI): Exact mass of sodiated dimer calcd. for $(C_{10}H_8FNO)_2Na^+$ (M_2^+ + Na) 377.107205, found 377.107102
***E*- and *Z*-16.** 	282 MHz: δ -120.4 (d, $^3J_{FH}$ = 15.3, *E* isomer), δ -123.0 (d, $^3J_{FH}$ = 36.6, *Z* isomer)	HRMS (positive ion APPI): calcd. for $C_{10}H_8FN^+$ (M^+) 161.063529, found 161.063284
***E*- and *Z*-17.** 	282 MHz: δ -115.3 (d, $^3J_{FH}$ = 15.3, *E* isomer), δ -115.7 (d, $^3J_{FH}$ = 33.6, *Z* isomer)	Lit. ref.: 10
***E*- and *Z*-18.**	282 MHz: δ -117.0 (d, $^3J_{FH}$ = 12.2, *E* isomer), δ -120.1 (d, $^3J_{FH}$ = 30.5, *Z* isomer)	Lit. ref.: 10

		
***E*- and *Z*-19.** 	282 MHz: δ -118.8 (d, $^3J_{FH}$ = 15.3, *E* isomer), δ -114.3 (s, Ar-F, *E* isomer), δ -119.8 (d, $^3J_{FH}$ = 36.6, *Z* isomer), δ -114.7 (s, Ar-F, *Z* isomer)	HRMS (EI): calcd. for $C_9H_5F_2N^+$ (M^+) 165.0390, found 165.0402
***E*- and *Z*-20.** 	282 MHz: δ -122.8 (d, $^3J_{FH}$ = 29.4, *Z* isomer), δ -129.0 (d, $^3J_{FH}$ = 11.7, *E* isomer)	HRMS (positive ion APPI): calcd. for $C_7H_4FNS^+$ (M^+) 153.004299, found 153.004145
***E*- and *Z*-21.** 	282 MHz: δ -117.9 (d, $^3J_{FH}$ = 33.6, *Z* isomer), δ -125.3 (d, $^3J_{FH}$ = 15.3, *E* isomer)	HRMS (positive ion ESI): calcd. for $C_{11}H_{12}FN_3O_2Na^+$ (M^+ + Na) 260.080576, found 260.080458
***E*- and *Z*-22.** 	282 MHz: δ -119.2 (d, $^3J_{FH}$ = 33.6, *Z* isomer), δ -125.9 (d, $^3J_{FH}$ = 12.2, *E* isomer)	HRMS (positive ion ESI): calcd. for $C_{16}H_{15}FN_2O_2Na^+$ (M^+ + Na) 309.100977, found 309.100939
***E*- and *Z*-23.** 	282 MHz: δ -128.5 (d, $^3J_{FH}$ = 35.2, *Z* isomer), δ -129.6 (d, $^3J_{FH}$ = 17.6, *E* isomer)	HRMS (positive ion ESI): calcd. for $C_{13}H_{10}FFeN^+$ (M^+) 255.014121, found 255.014173
***E*- and *Z*-24.** 	282 MHz: δ -126.6 (d, $^3J_{FH}$ = 30.5, *Z* isomer), δ -127.7 (d, $^3J_{FH}$ = 12.2, *E* isomer)	Lit. ref.: 8, 9
***E*- and *Z*-25.** 	282 MHz: δ -123.9 (d, $^3J_{FH}$ = 11.8, *E* isomer), δ -125.8 (d, $^3J_{FH}$ = 35.2, *Z* isomer)	HRMS (positive ion APPI): calcd. for $C_{10}H_{15}FN^+$ (M^+) 168.118304, found 168.118068

***E*- and *Z*-26.** 	282 MHz: δ -122.6 (d, $^3J_{FH}$ = 14.7, *E* isomer), δ -124.2 (d, $^3J_{FH}$ = 32.3, *Z* isomer)	Lit. ref.: 9
***E*- and *Z*-27.** 	282 MHz: δ -122.8 (d, $^3J_{FH}$ = 15.3, *E* isomer), δ -125.6 (d, $^3J_{FH}$ = 30.5, *Z* isomer)	HRMS (EI): calcd. for $C_8H_{12}FN^+$ (M^+) 141.0954, found 141.0955

TLC Separation of *E*/*Z* Mixtures 12, 14, 16, 17, 21 and 26

(*E*/*Z*)-**12**: SiO_2, 10% EtOAc in hexanes: R_f = 0.42; R_f = 0.49

(*E*/*Z*)-**14**: SiO_2, 15% EtOAc in hexanes: R_f = 0.38; R_f = 0.42

(*E*/*Z*)-**16**: SiO_2, hexanes: R_f = 0.17; R_f = 0.26

(*E*/*Z*)-**17**: SiO_2, 25% EtOAc in hexanes: R_f = 0.64; R_f = 0.68

(*E*/*Z*)-**21**: SiO_2, 25% EtOAc in hexanes: R_f = 0.23; R_f = 0.28

(*E*/*Z*)-**26**: SiO_2, 10% EtOAc in hexanes: R_f = 0.44; R_f = 0.67

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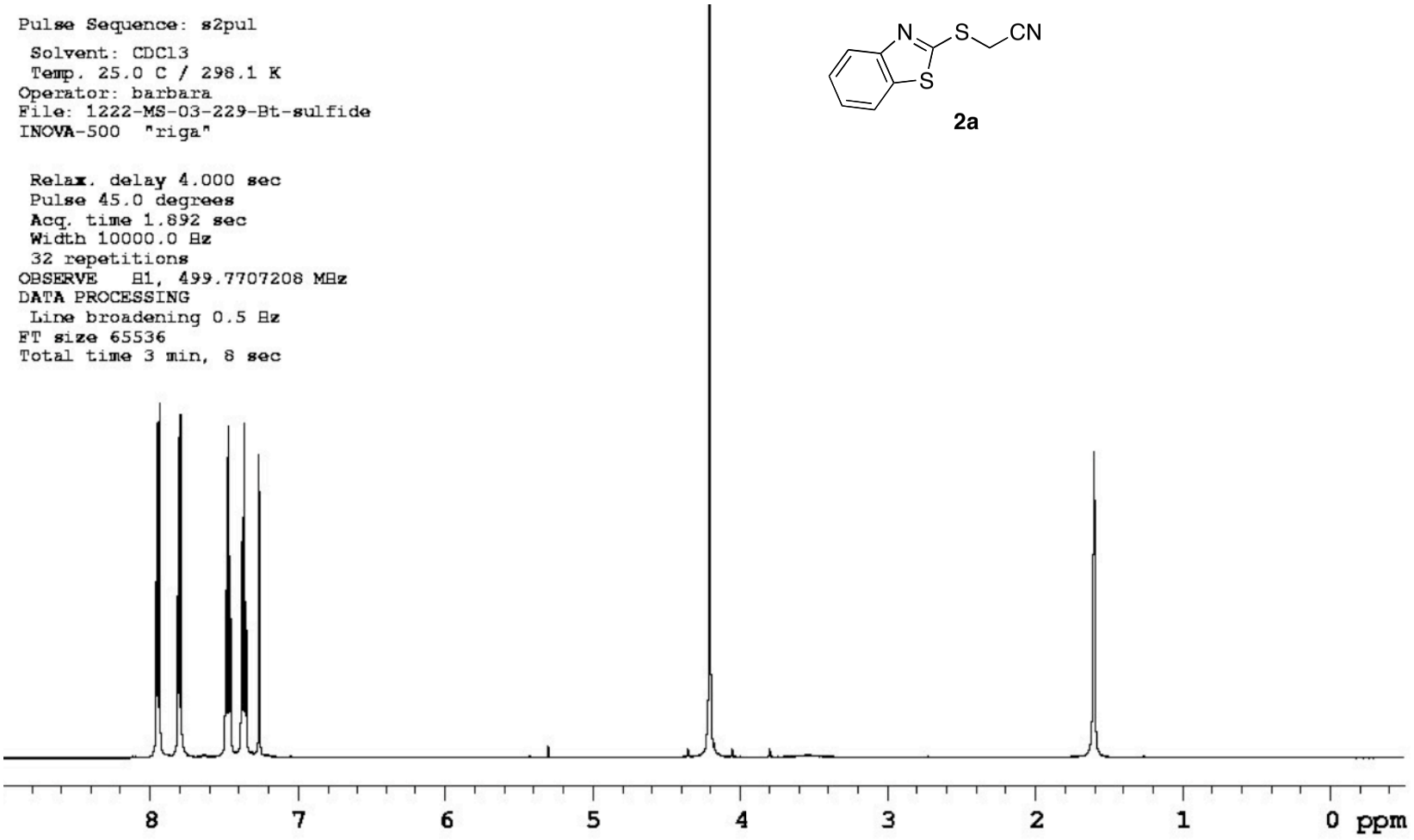
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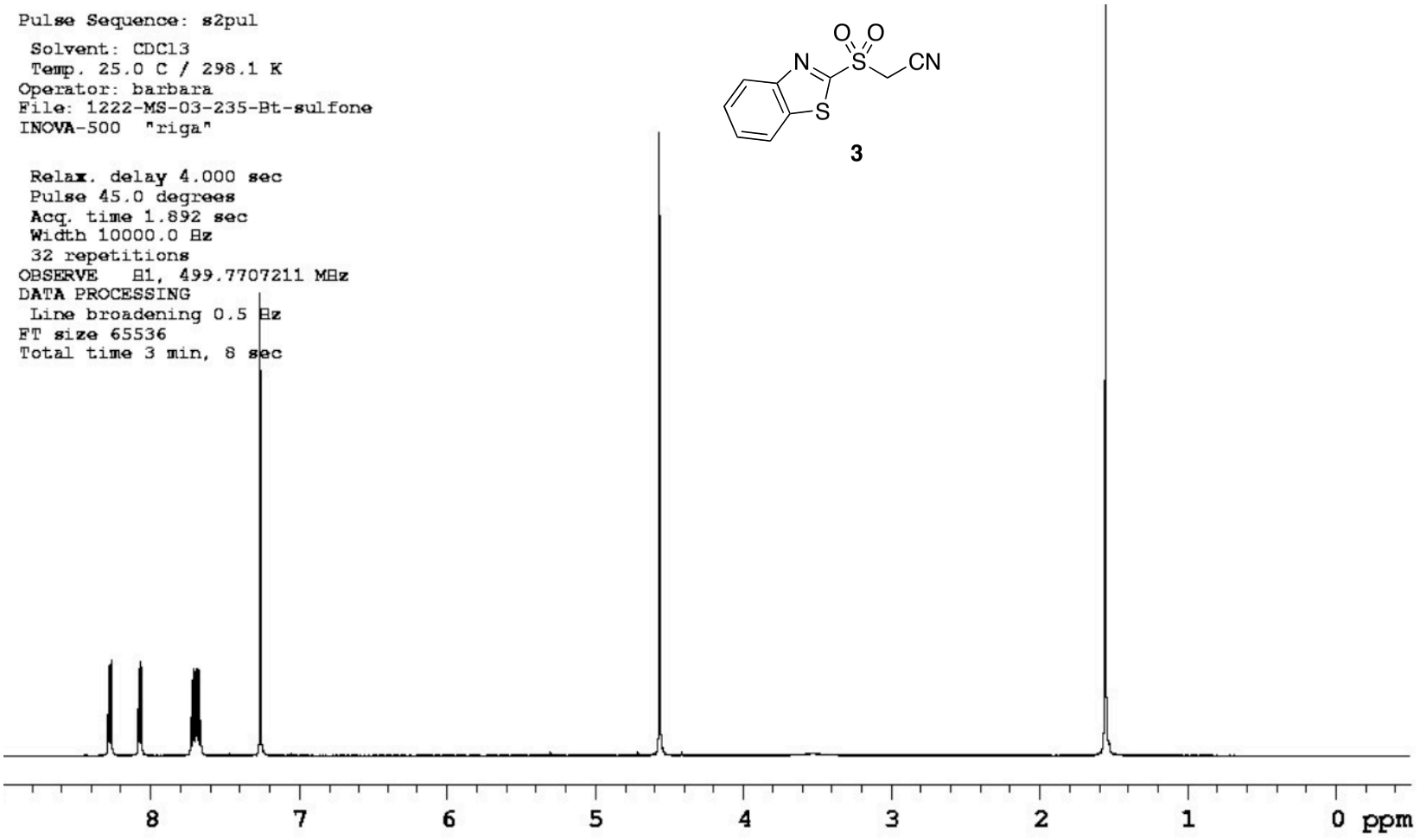
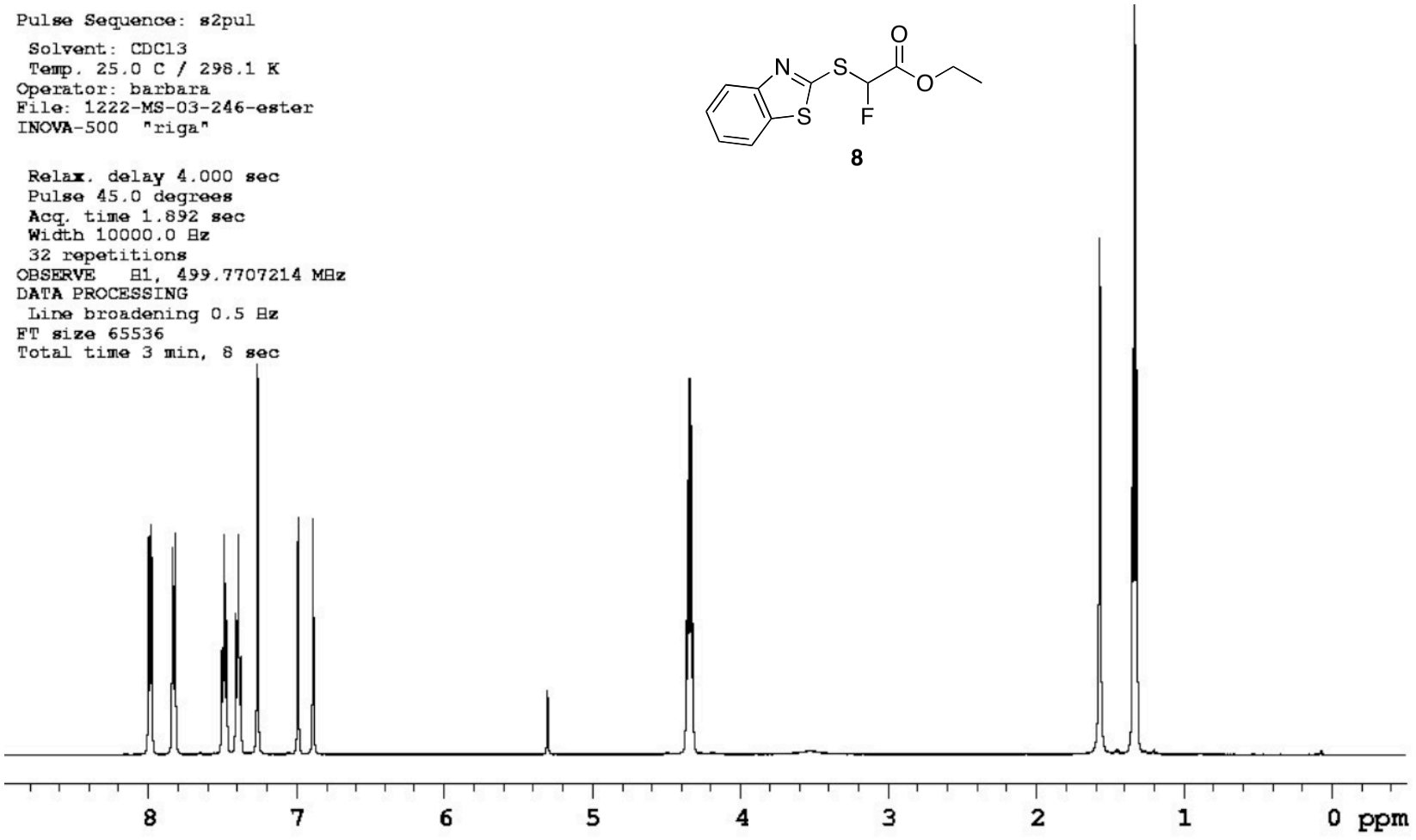
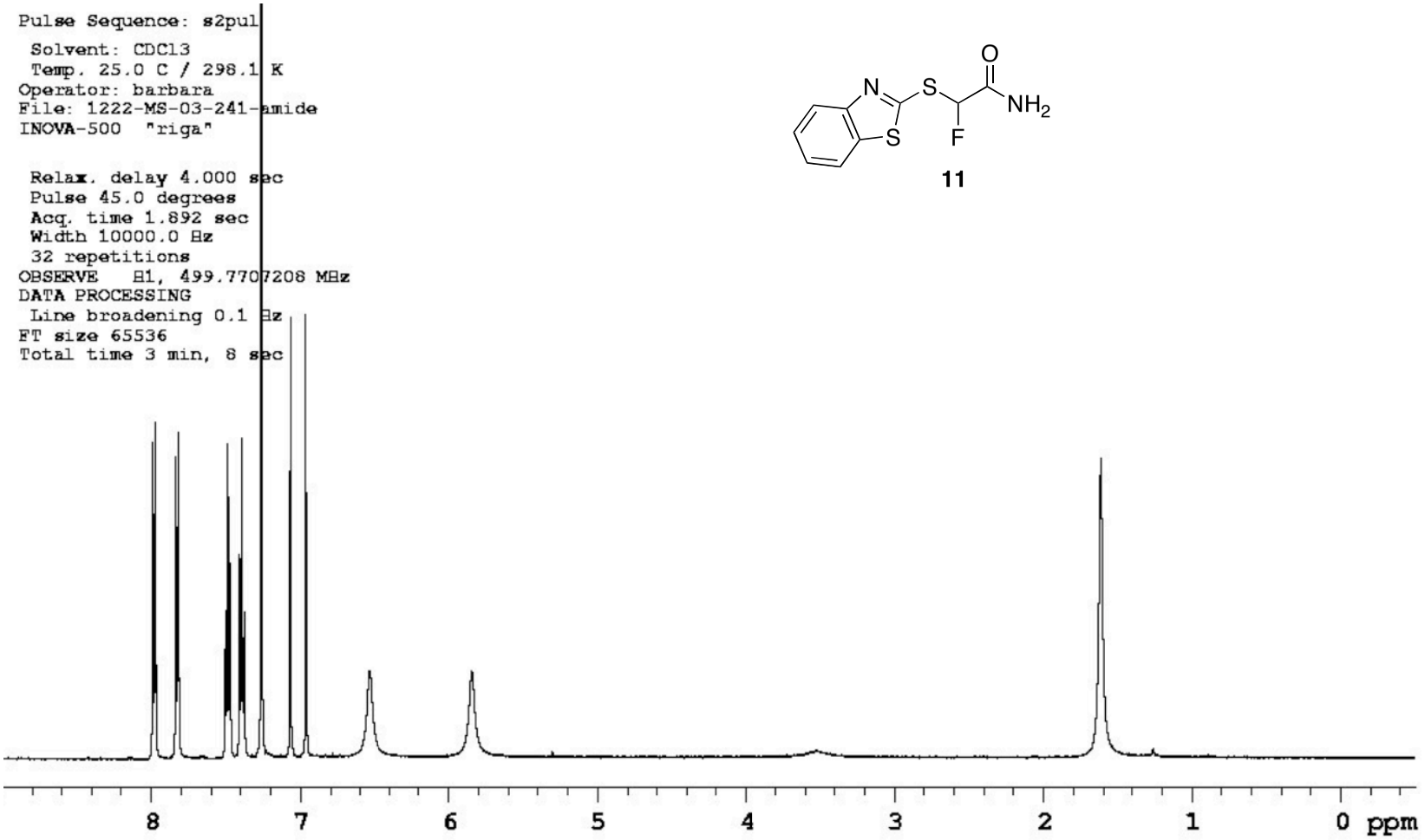
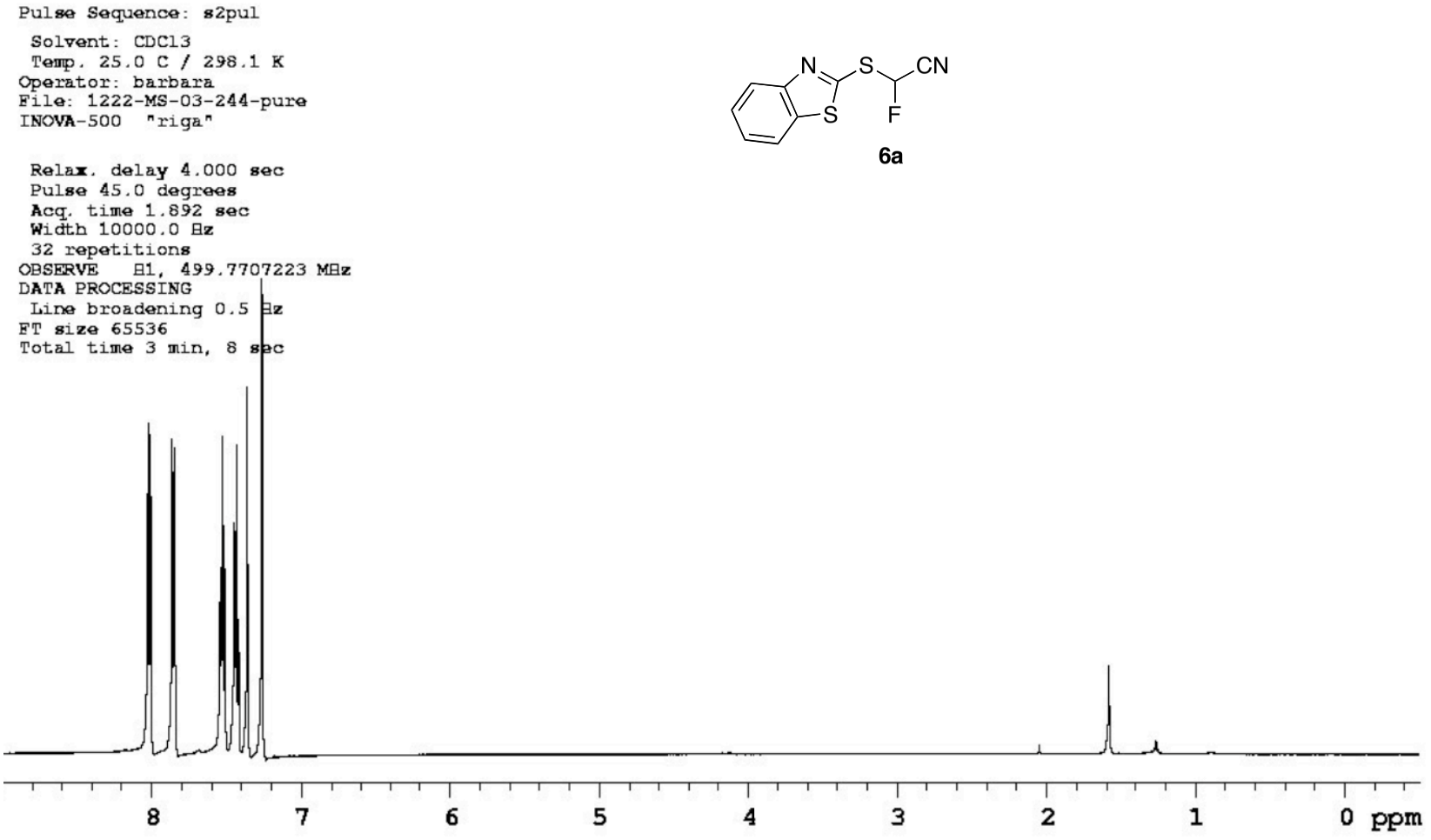
Solvent: CDCl3
Temp. 25.0 C / 298.1 K
Operator: barbara
File: 1222-MS-03-229-Bt-sulfide
INOVA-500 "riga"
Relax. delay 4.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 10000.0 Hz
32 repetitions
OBSERVE H1, 499.7707208 MHz
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 3 min, 8 sec
N
S
CN
S
2a
8
7
6
5
4
3
2
1
0 ppm

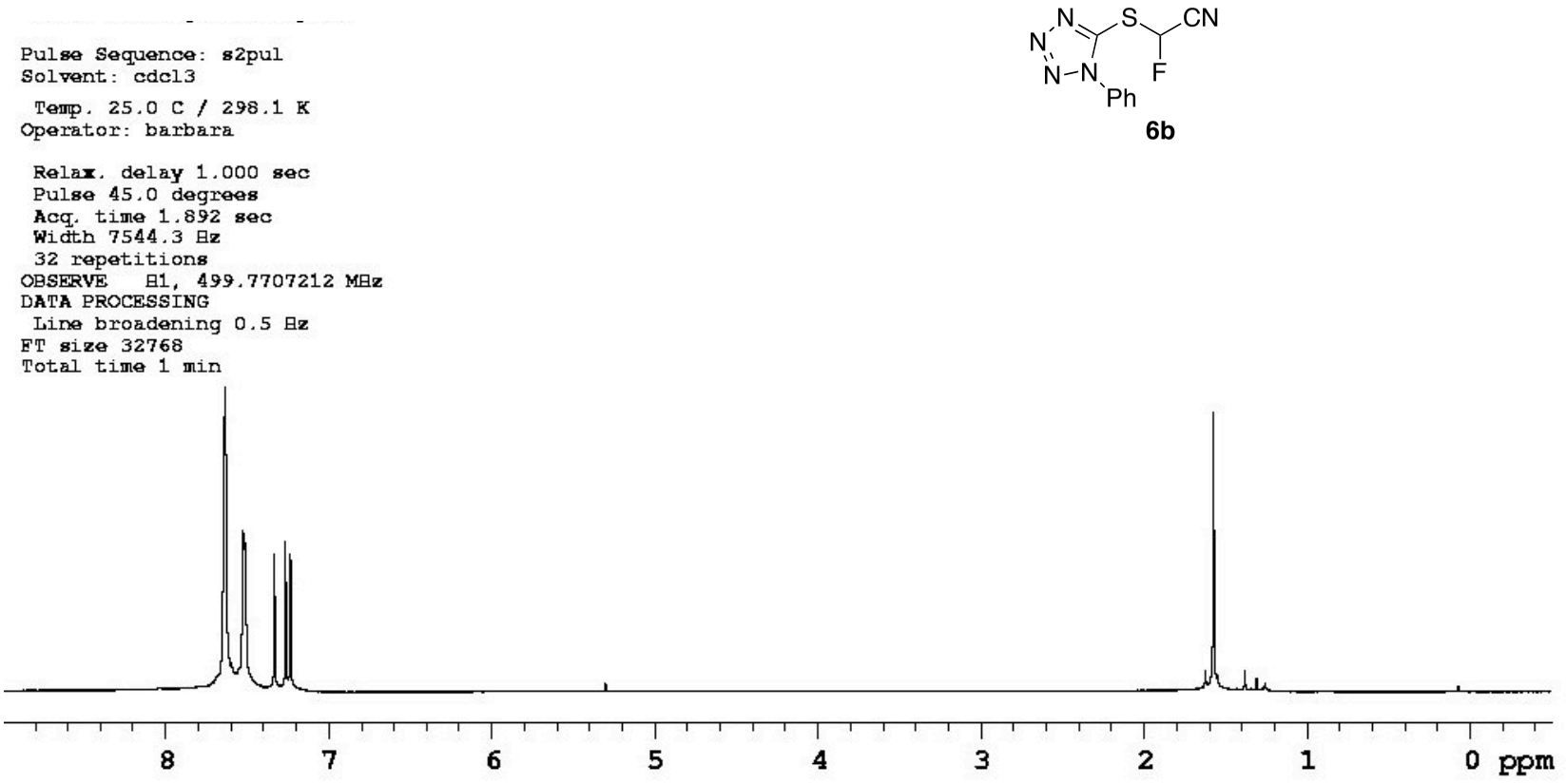

Pulse Sequence: s2pul
Solvent: CDCl3
Temp. 25.0 C / 298.1 K
Operator: barbara
File: 1222-MS-03-235-Bt-sulfone
INOVA-500 "riga"
Relax. delay 4.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 10000.0 Hz
32 repetitions
OBSERVE H1, 499.7707211 MHz
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 3 min, 8 sec
O
O
N
S
CN
S
3
8
7
6
5
4
3
2
1
0 ppm


Pulse Sequence: s2pul
Solvent: CDCl3
Temp. 25.0 C / 298.1 K
Operator: barbara
File: 1222-MS-03-246-ester
INOVA-500 "riga"
Relax. delay 4.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 10000.0 Hz
32 repetitions
OBSERVE H1, 499.7707214 MHz
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 3 min, 8 sec
N
S
S
O
O
F
8
8
7
6
5
4
3
2
1
0 ppm


Pulse Sequence: s2pul
Solvent: CDCl3
Temp. 25.0 C / 298.1 K
Operator: barbara
File: 1222-MS-03-241-amide
INOVA-500 "riga"
Relax. delay 4.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 10000.0 Hz
32 repetitions
OBSERVE H1, 499.7707208 MHz
DATA PROCESSING
Line broadening 0.1 Hz
FT size 65536
Total time 3 min, 8 sec
N
S
O
NH2
S
F
11
8
7
6
5
4
3
2
1
0 ppm

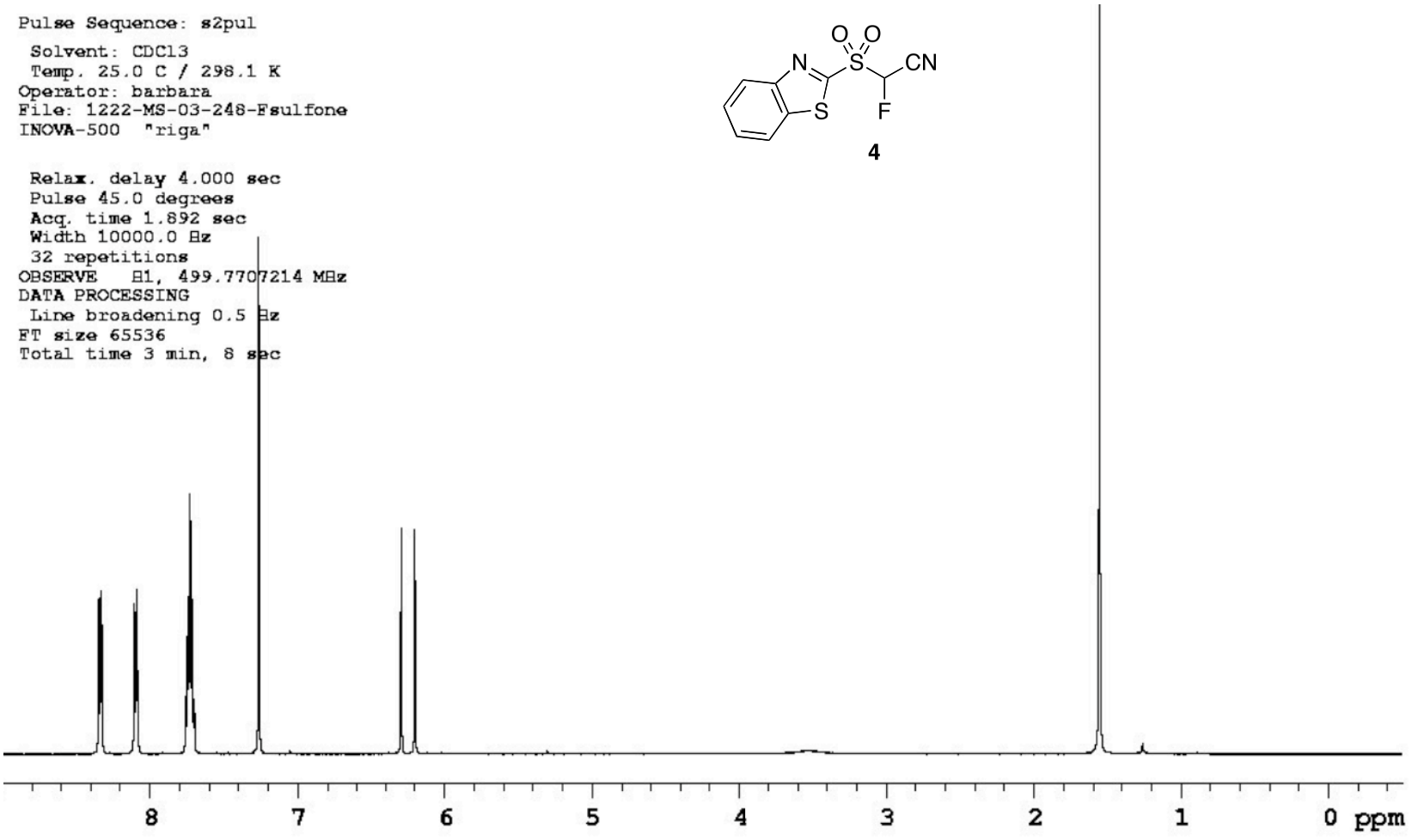

Pulse Sequence: s2pul
Solvent: CDCl3
Temp. 25.0 C / 298.1 K
Operator: barbara
File: 1222-MS-03-244-pure
INOVA-500 "riga"
Relax. delay 4.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 10000.0 Hz
32 repetitions
OBSERVE H1, 499.7707223 MHz
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 3 min, 8 sec
N
S
CN
S
F
8
7
6
5
4
3
2
1
0 ppm

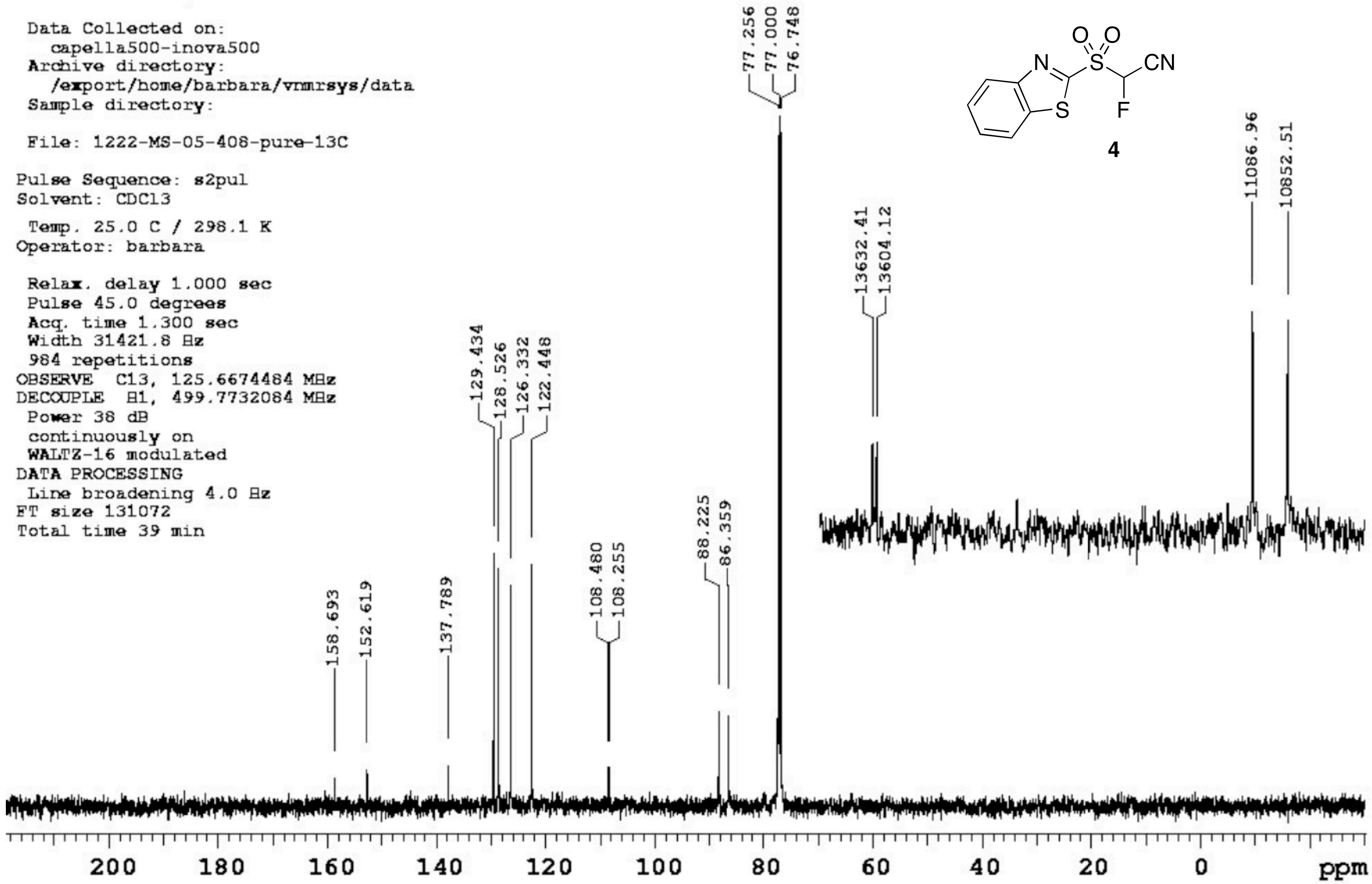
6a


Pulse Sequence: s2pul
Solvent: cdcl3
Temp. 25.0 C / 298.1 K
Operator: barbara
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 7544.3 Hz
32 repetitions
OBSERVE H1, 499.7707212 MHz
DATA PROCESSING
Line broadening 0.5 Hz
FT size 32768
Total time 1 min
8
7
6
5
4
3
2
1
0 ppm

N
N
N
N
S
CN
F
Ph

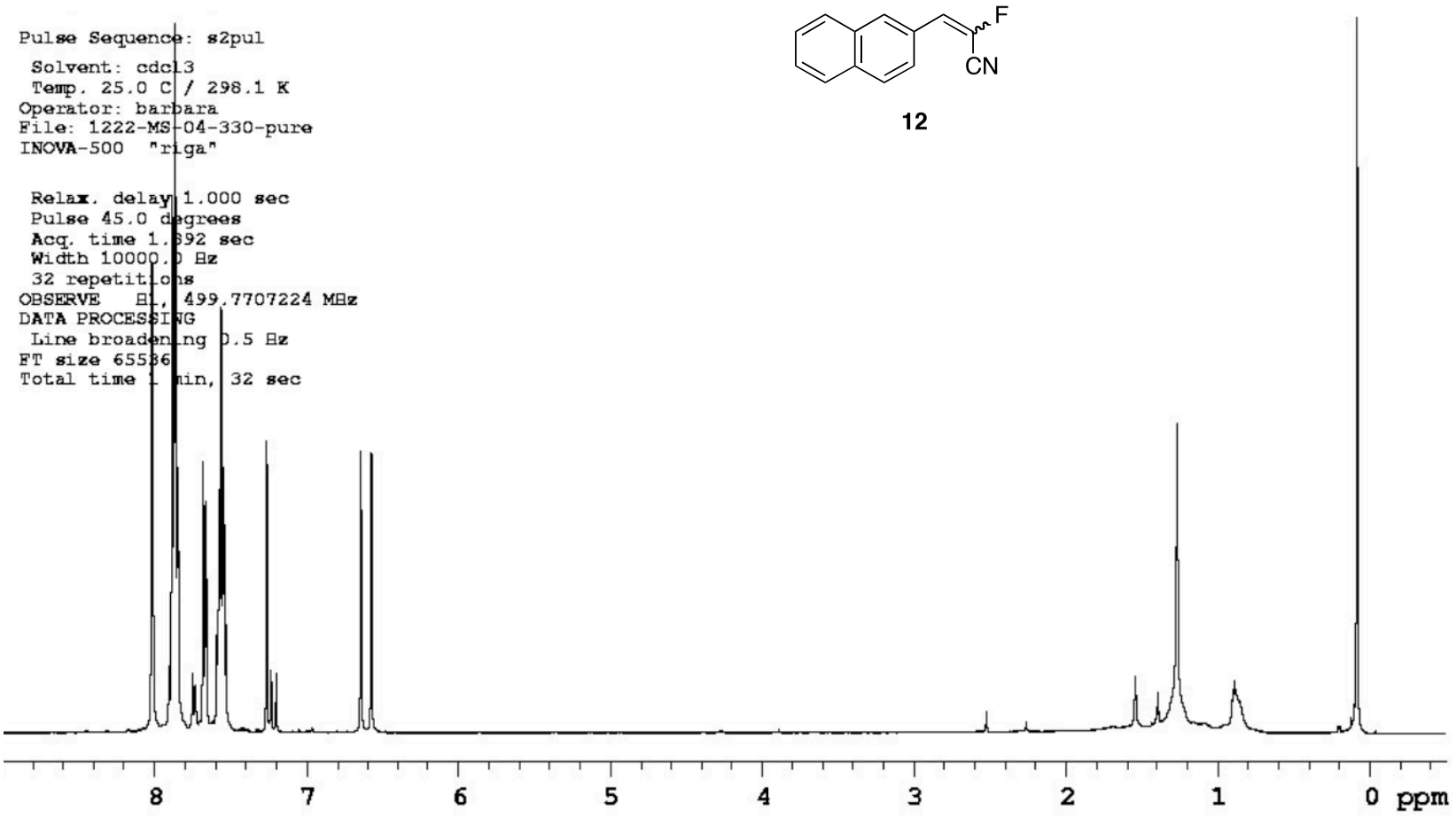
6b

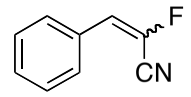

Pulse Sequence: s2pul
Solvent: CDCl3
Temp. 25.0 C / 298.1 K
Operator: barbara
File: 1222-MS-03-248-Fsulfone
INOVA-500 "riga"
Relax. delay 4.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 10000.0 Hz
32 repetitions
OBSERVE H1, 499.7707214 MHz
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 3 min, 8 sec
O
O
N
S
CN
S
F
4
8
7
6
5
4
3
2
1
0 ppm



4

Data Collected on:
capella500-inova500
Archive directory:
/export/home/barbara/vnmrsys/data
Sample directory:
File: 1222-MS-05-408-pure-13C
Pulse Sequence: s2pul
Solvent: CDCl3
Temp. 25.0 C / 298.1 K
Operator: barbara
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
984 repetitions
OBSERVE C13, 125.6674484 MHz
DECOUPLE H1, 499.7732084 MHz
Power 38 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 4.0 Hz
FT size 131072
Total time 39 min
158.693
152.619
137.789
129.434
128.526
126.332
122.448
108.480
108.255
88.225
86.359
77.256
77.000
76.748
13632.41
13604.12
11086.96
10852.51
200
180
160
140
120
100
80
60
40
20
0
ppm


Pulse Sequence: s2pul
Solvent: cdcl3
Temp. 25.0 C / 298.1 K
Operator: barbara
File: 1222-MS-04-330-pure
INOVA-500 "riga"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 10000.0 Hz
32 repetitions
OBSERVE H1, 499.7707224 MHz
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 1 min, 32 sec
F
CN
12
8
7
6
5
4
3
2
1
0 ppm



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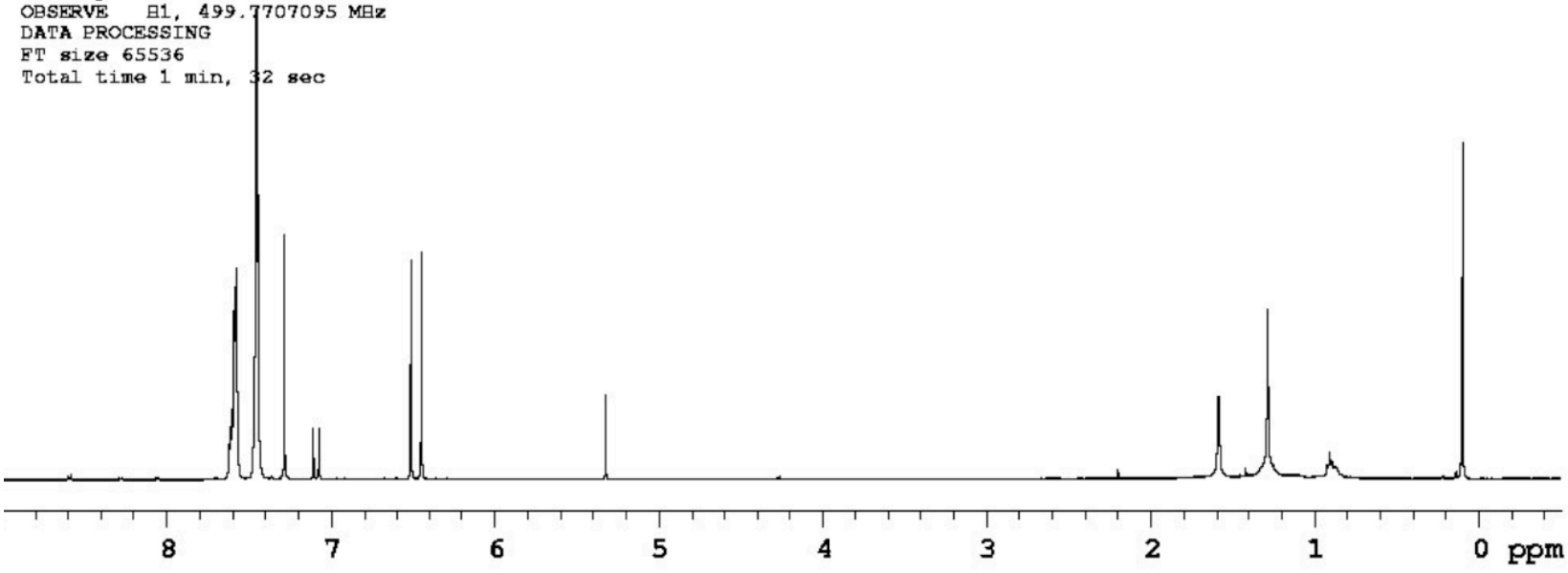
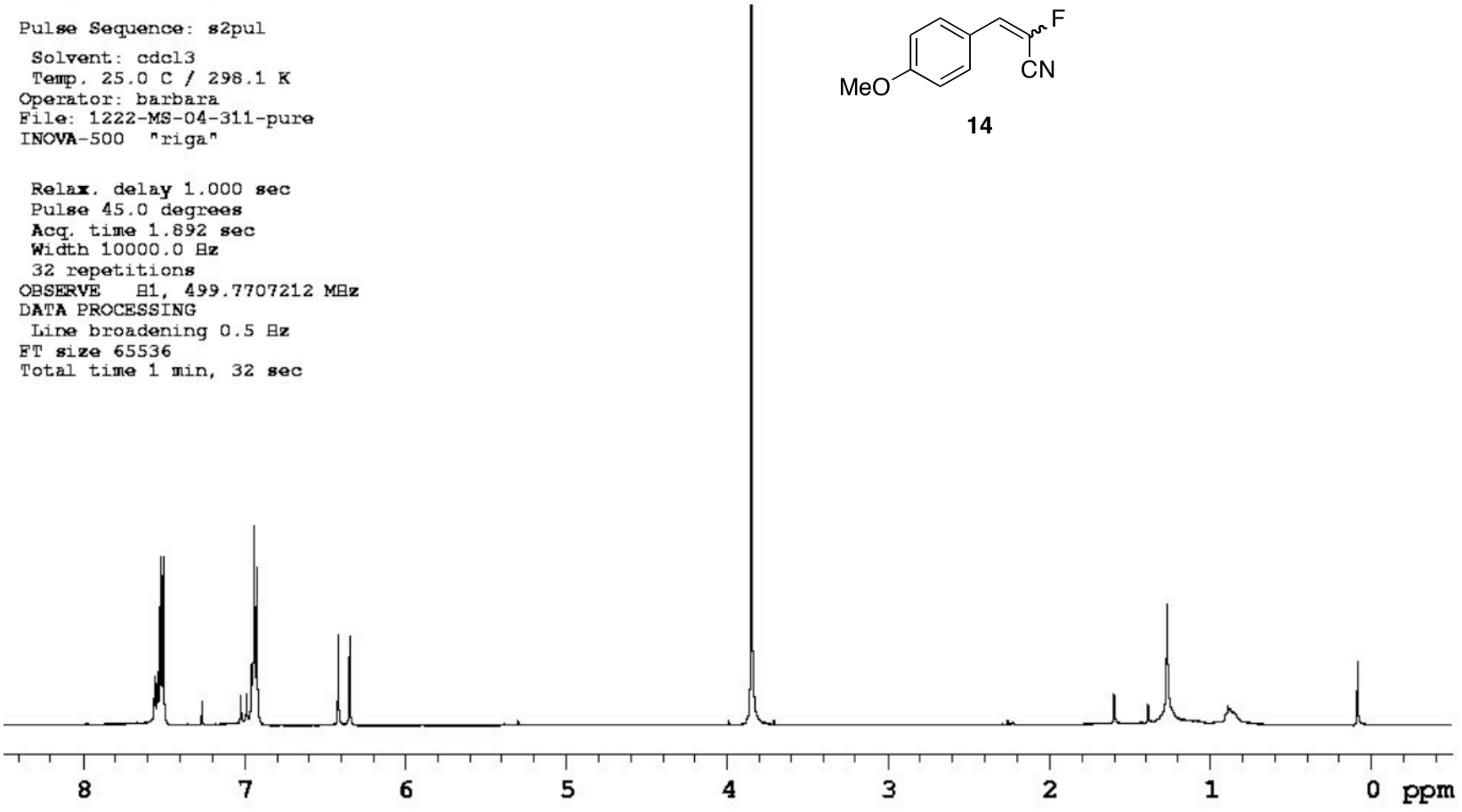
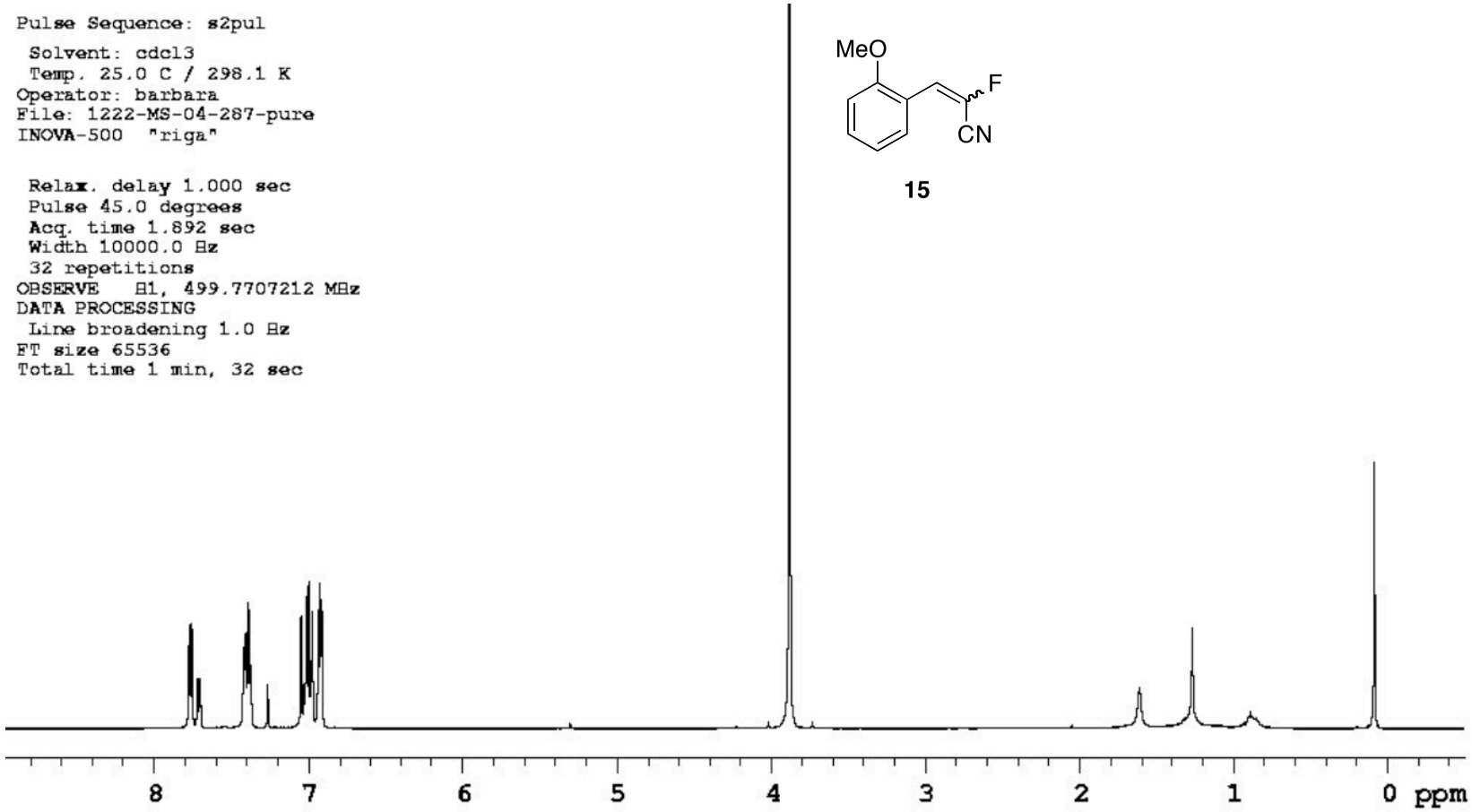
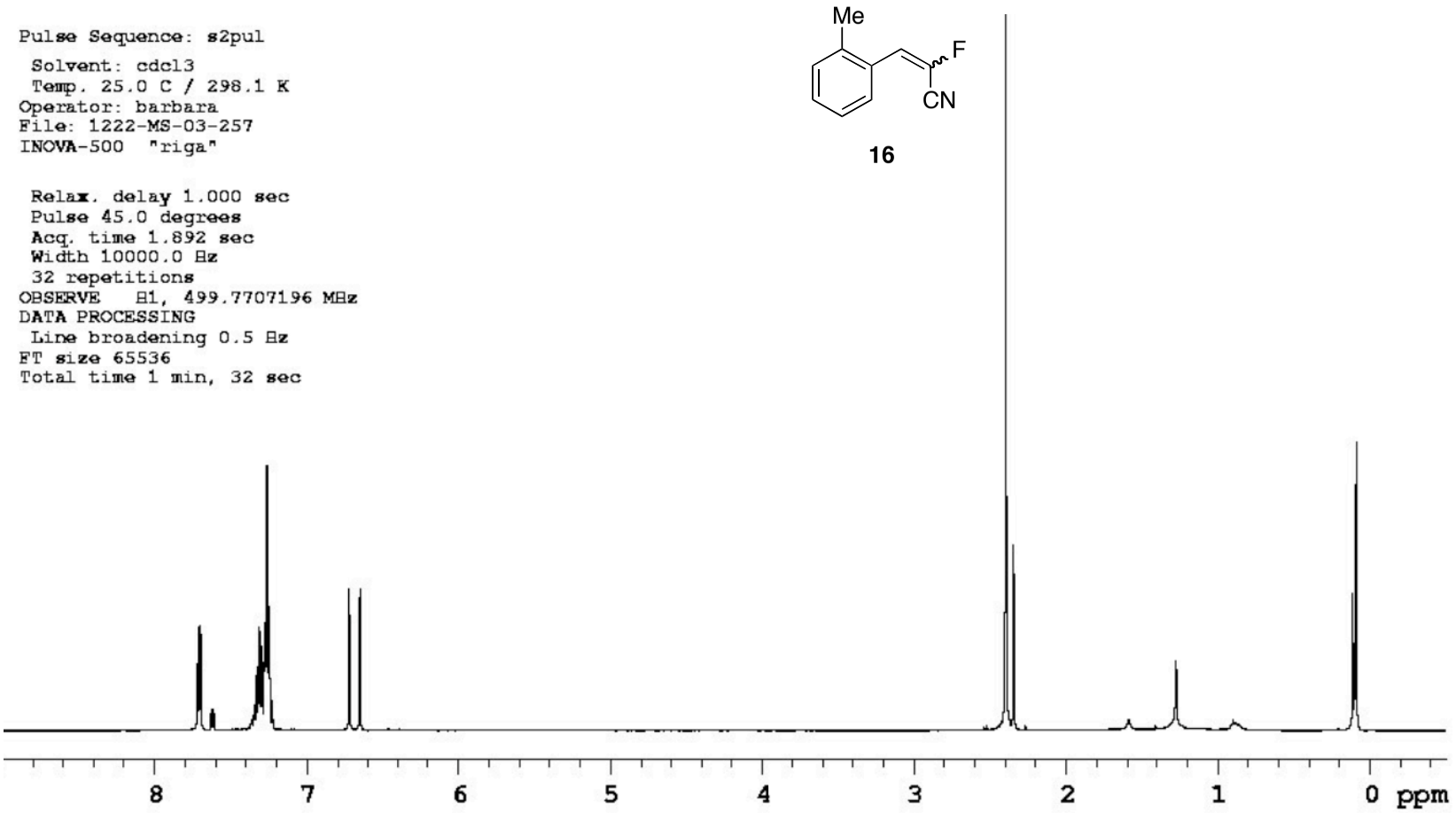
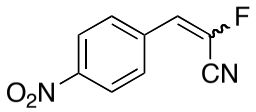
```
Pulse Sequence: s2pul
 Solvent: cdcl3
 Temp. 25.0 C / 298.1 K
Operator: barbara
File: 1222-MS-04-319-pure
INOVA-500  "riga"

 Relax. delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 1.892 sec
 Width 10000.0 Hz
 28 repetitions
OBSERVE   H1, 499.7707095 MHz
DATA PROCESSING
FT size 65536
Total time 1 min, 32 sec
```



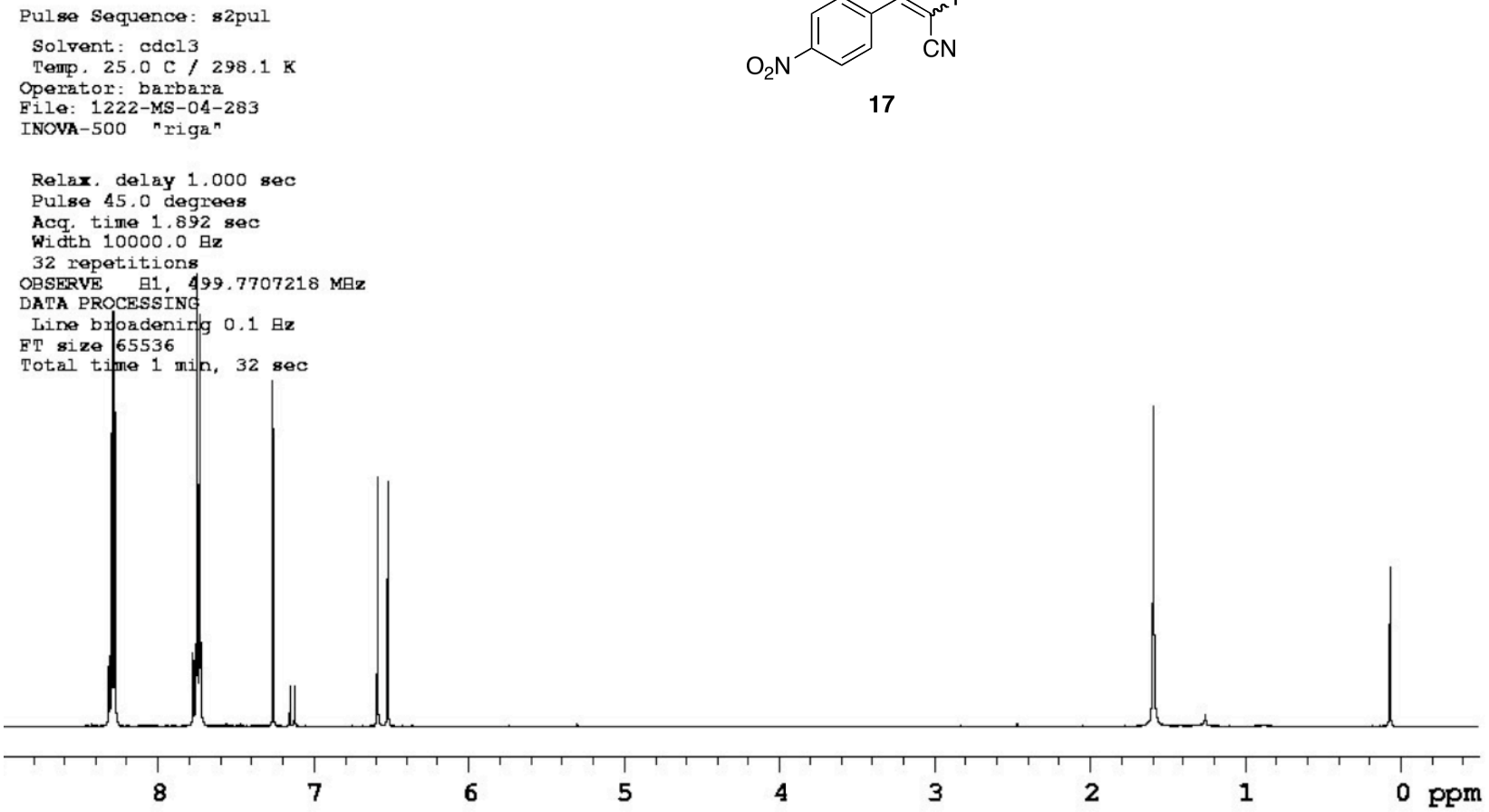

Pulse Sequence: s2pul
Solvent: cdcl3
Temp. 25.0 C / 298.1 K
Operator: barbara
File: 1222-MS-04-311-pure
INOVA-500 "riga"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 10000.0 Hz
32 repetitions
OBSERVE H1, 499.7707212 MHz
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 1 min, 32 sec
MeO
F
CN
14
8
7
6
5
4
3
2
1
0
ppm

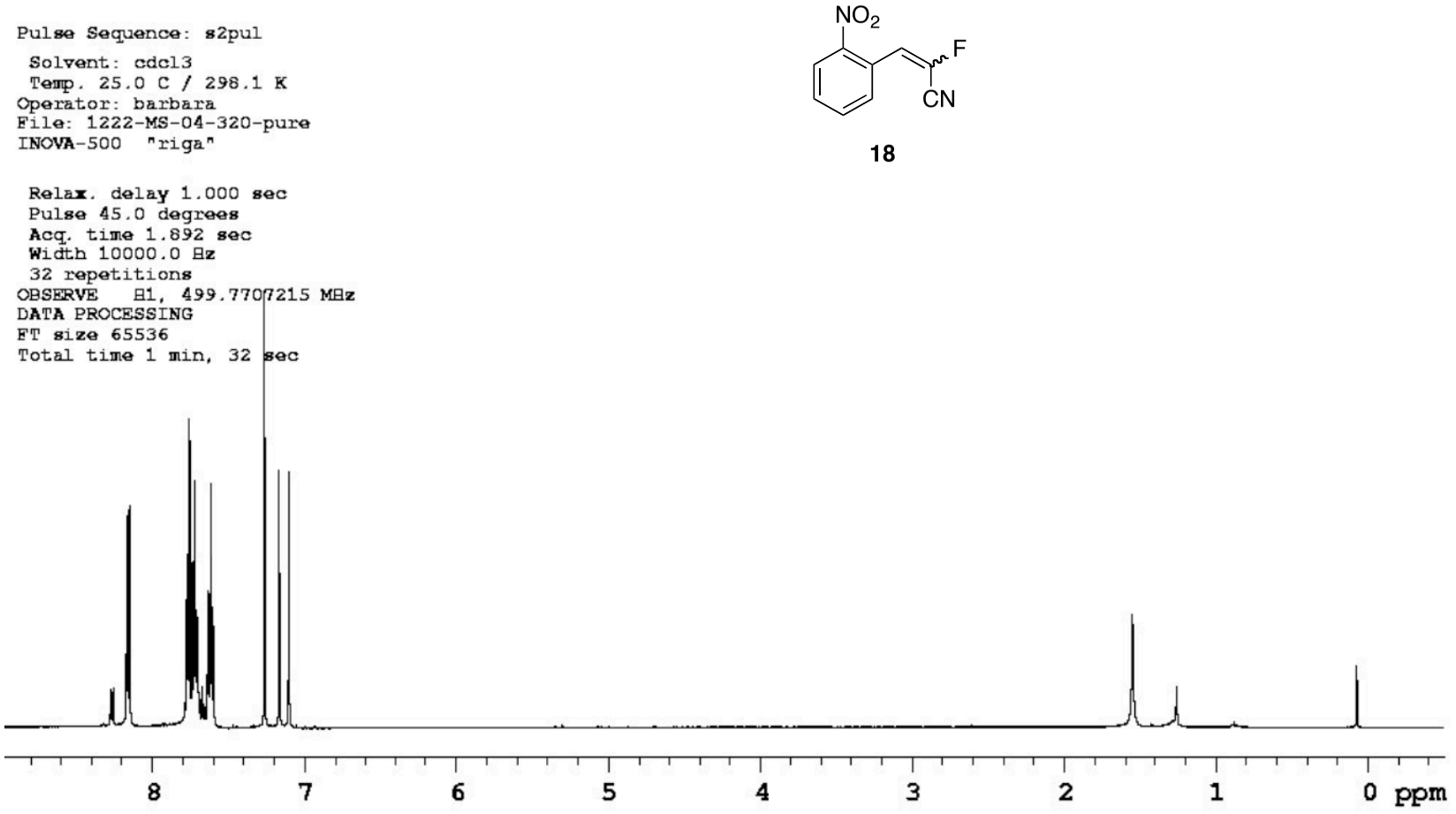

Pulse Sequence: s2pul
Solvent: cdcl3
Temp. 25.0 C / 298.1 K
Operator: barbara
File: 1222-MS-04-287-pure
INOVA-500 "riga"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 10000.0 Hz
32 repetitions
OBSERVE H1, 499.7707212 MHz
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 1 min, 32 sec
MeO
F
CN
15
8
7
6
5
4
3
2
1
0 ppm


Pulse Sequence: s2pul
Solvent: cdcl3
Temp. 25.0 C / 298.1 K
Operator: barbara
File: 1222-MS-03-257
INOVA-500 "riga"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 10000.0 Hz
32 repetitions
OBSERVE H1, 499.7707196 MHz
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 1 min, 32 sec
Me
F
CN
16
8
7
6
5
4
3
2
1
0 ppm


F
CN
O2N

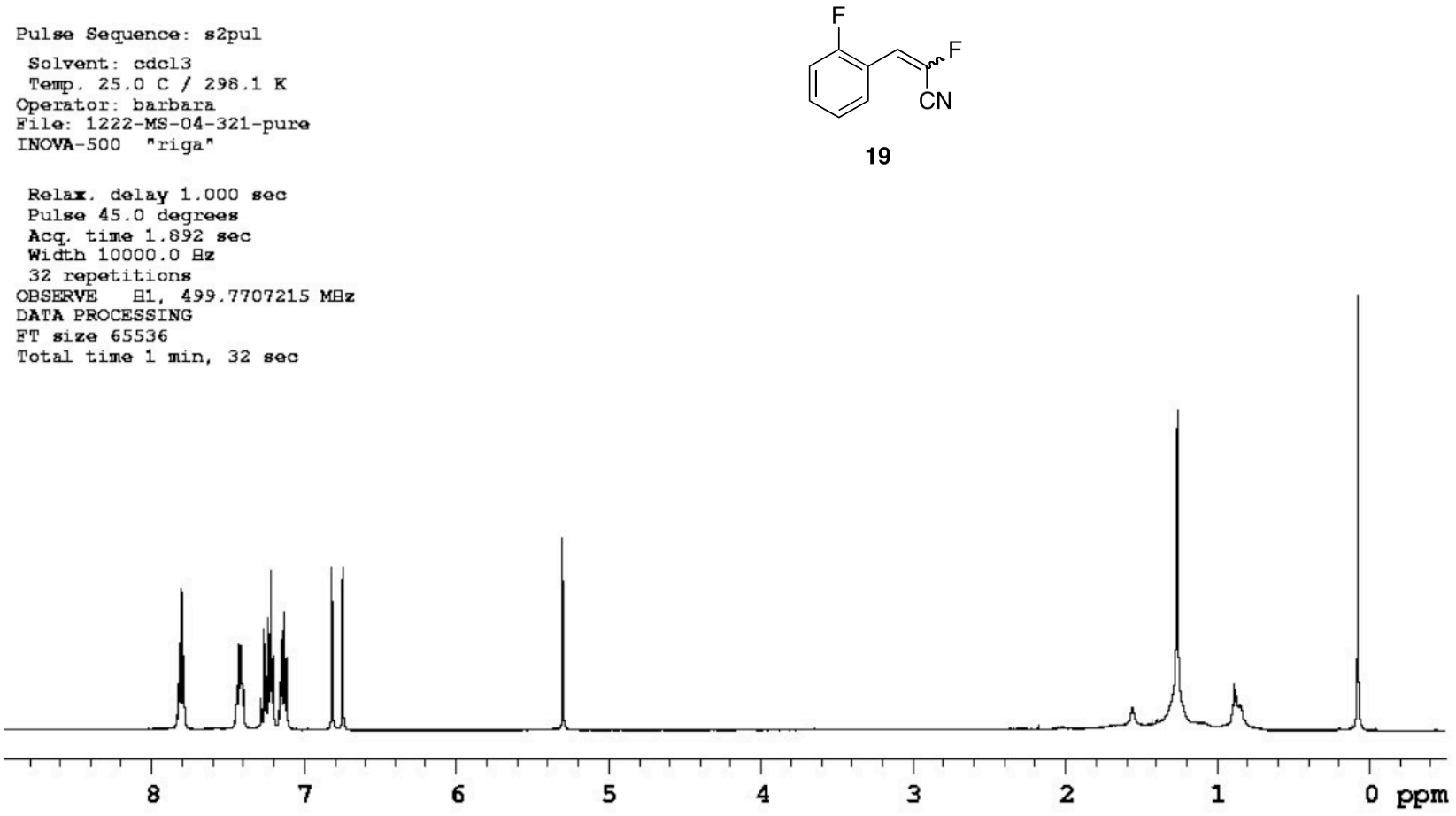
17


Pulse Sequence: s2pul
Solvent: cdcl3
Temp. 25.0 C / 298.1 K
Operator: barbara
File: 1222-MS-04-283
INOVA-500 "riga"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 10000.0 Hz
32 repetitions
OBSERVE H1, 499.7707218 MHz
DATA PROCESSING
Line broadening 0.1 Hz
FT size 65536
Total time 1 min, 32 sec
8
7
6
5
4
3
2
1
0 ppm

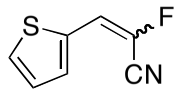

Pulse Sequence: s2pul
Solvent: cdcl3
Temp. 25.0 C / 298.1 K
Operator: barbara
File: 1222-MS-04-320-pure
INOVA-500 "riga"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 10000.0 Hz
32 repetitions
OBSERVE H1, 499.7707215 MHz
DATA PROCESSING
FT size 65536
Total time 1 min, 32 sec
8
7
6
5
4
3
2
1
0 ppm

NO2
F
CN

18


Pulse Sequence: s2pul
Solvent: cdcl3
Temp. 25.0 C / 298.1 K
Operator: barbara
File: 1222-MS-04-321-pure
INOVA-500 "riga"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 10000.0 Hz
32 repetitions
OBSERVE H1, 499.7707215 MHz
DATA PROCESSING
FT size 65536
Total time 1 min, 32 sec
F
F
CN
8
7
6
5
4
3
2
1
0 ppm

19



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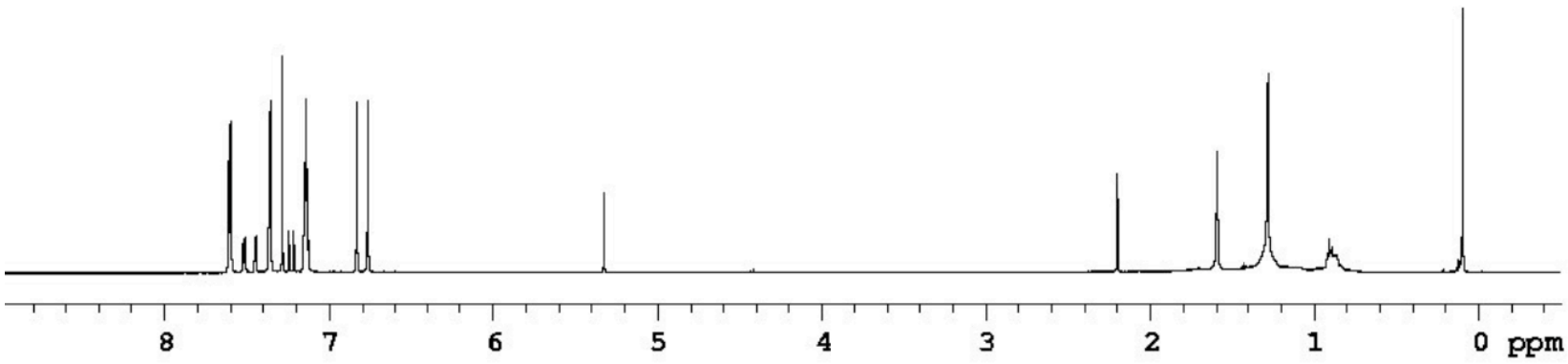
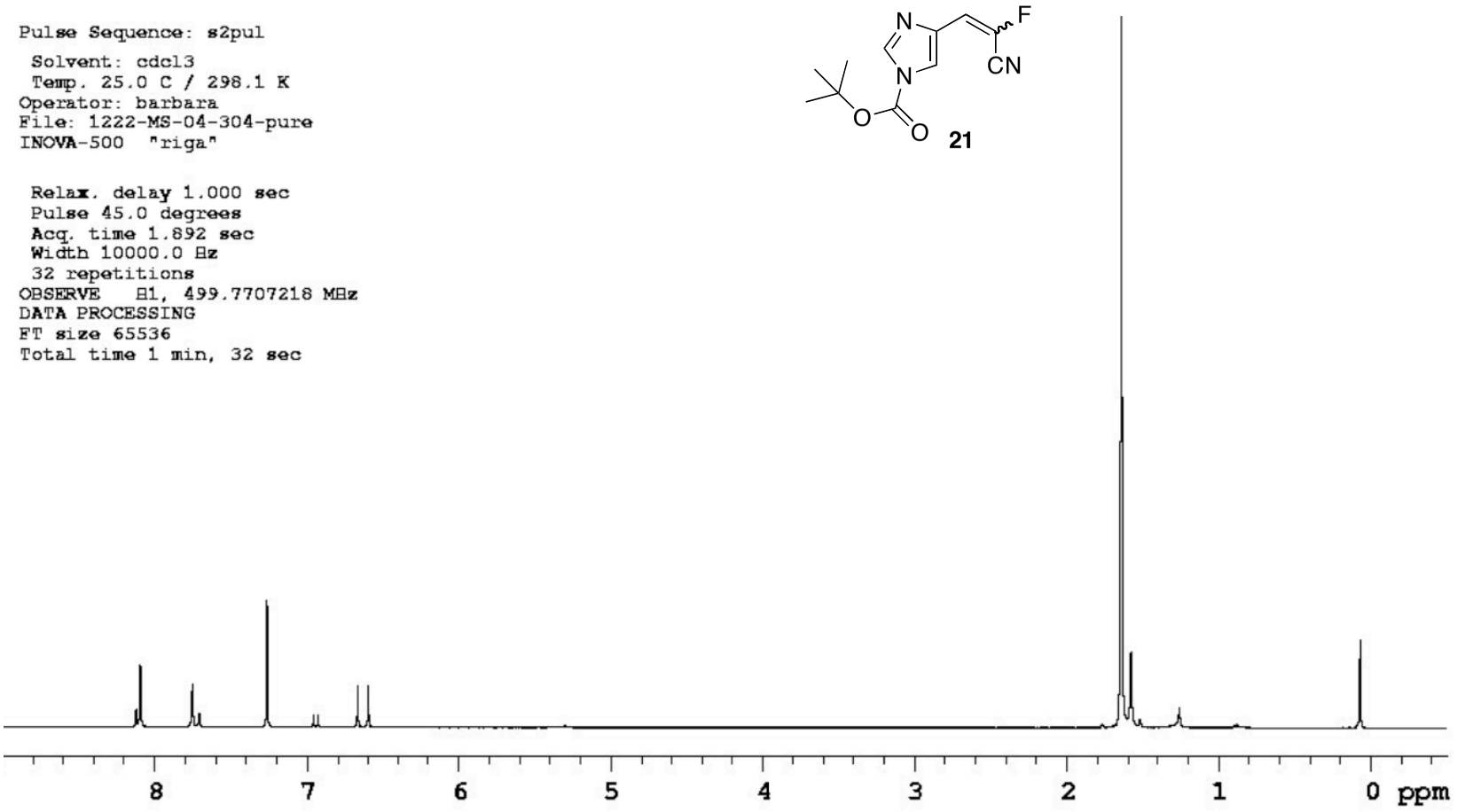
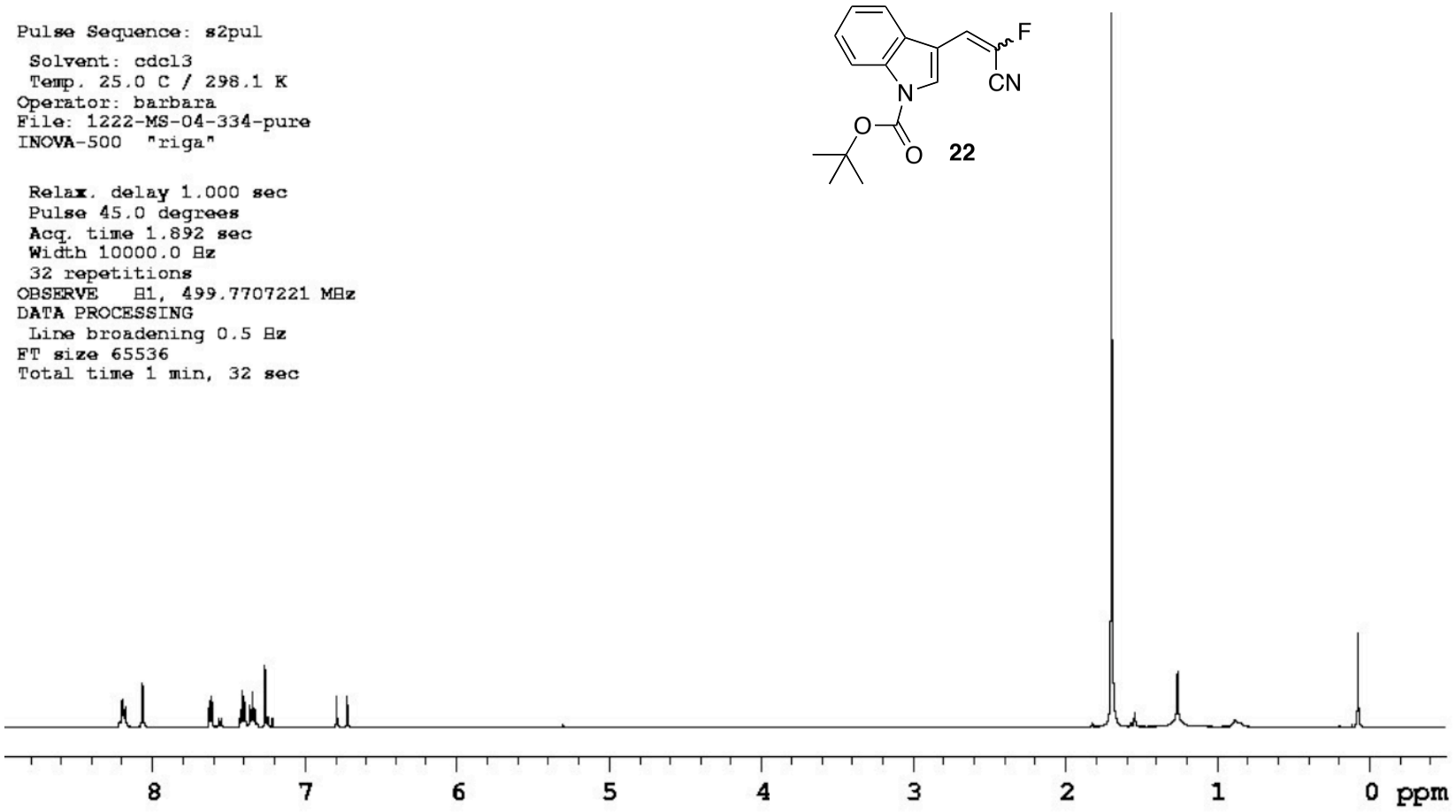
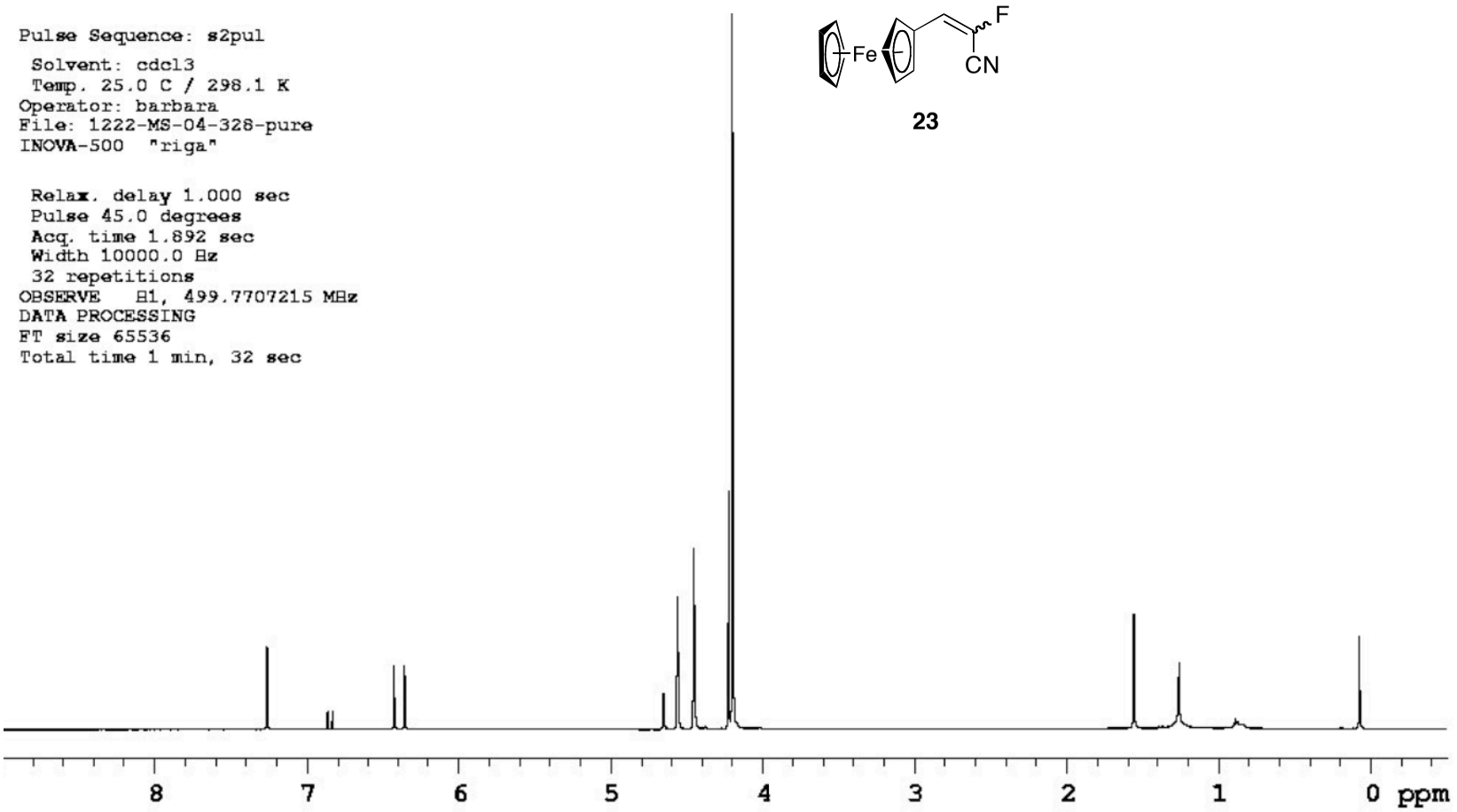
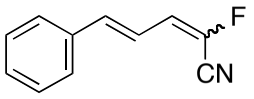
```
Pulse Sequence: s2pul
 Solvent: cdcl3
 Temp. 25.0 C / 298.1 K
Operator: barbara
File: 1222-MS-04-327-pure
INOVA-500  "riga"

 Relax. delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 1.892 sec
 Width 10000.0 Hz
 32 repetitions
OBSERVE   H1, 499.7707095 MHz
DATA PROCESSING
FT size 65536
Total time 1 min, 32 sec
```




Pulse Sequence: s2pul
Solvent: cdcl3
Temp. 25.0 C / 298.1 K
Operator: barbara
File: 1222-MS-04-304-pure
INOVA-500 "riga"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 10000.0 Hz
32 repetitions
OBSERVE H1, 499.7707218 MHz
DATA PROCESSING
FT size 65536
Total time 1 min, 32 sec
N
F
CN
N
O
O
21
8
7
6
5
4
3
2
1
0 ppm


Pulse Sequence: s2pul
Solvent: cdcl3
Temp. 25.0 C / 298.1 K
Operator: barbara
File: 1222-MS-04-334-pure
INOVA-500 "riga"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 10000.0 Hz
32 repetitions
OBSERVE H1, 499.7707221 MHz
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 1 min, 32 sec
F
CN
N
O
O
22
8
7
6
5
4
3
2
1
0 ppm


Pulse Sequence: s2pul
Solvent: cdcl3
Temp. 25.0 C / 298.1 K
Operator: barbara
File: 1222-MS-04-328-pure
INOVA-500 "riga"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 10000.0 Hz
32 repetitions
OBSERVE H1, 499.7707215 MHz
DATA PROCESSING
FT size 65536
Total time 1 min, 32 sec
Fe
F
CN
23
8
7
6
5
4
3
2
1
0 ppm



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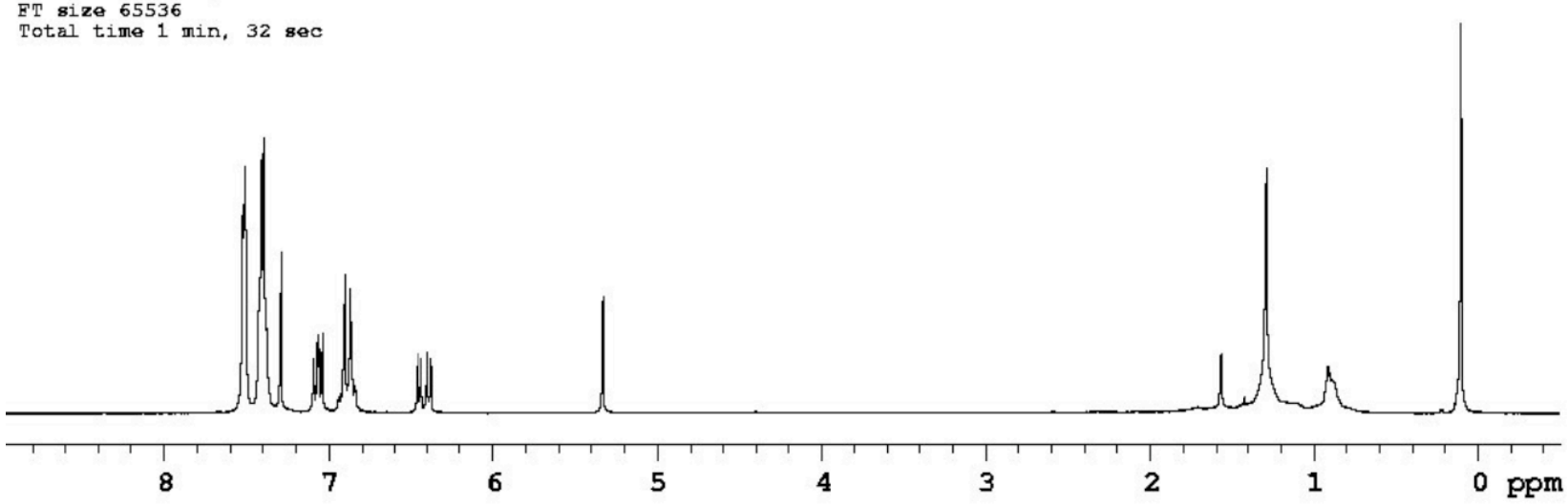
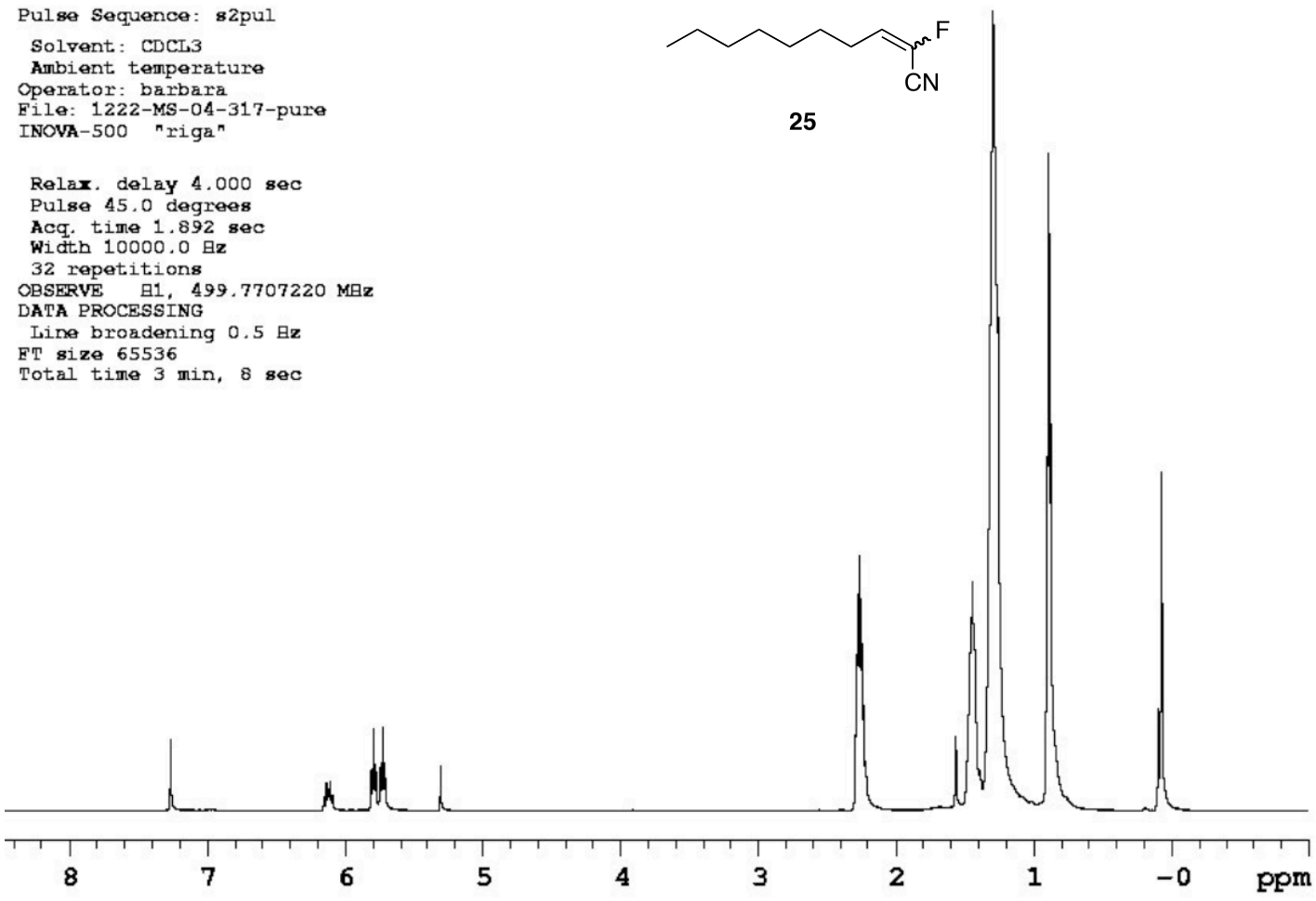
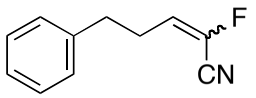
```
Pulse Sequence: s2pul
 Solvent: cdcl3
 Temp. 25.0 C / 298.1 K
Operator: barbara
File: 1222-MS-04-329-pure
INOVA-500  "riga"

 Relax. delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 1.892 sec
 Width 10000.0 Hz
 32 repetitions
OBSERVE   H1, 499.7707095 MHz
DATA PROCESSING
 Line broadening 0.1 Hz
FT size 65536
Total time 1 min, 32 sec
```




Pulse Sequence: s2pul
Solvent: CDCL3
Ambient temperature
Operator: barbara
File: 1222-MS-04-317-pure
INOVA-500 "riga"
Relax. delay 4.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 10000.0 Hz
32 repetitions
OBSERVE H1, 499.7707220 MHz
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 3 min, 8 sec
F
CN
25
8
7
6
5
4
3
2
1
-0
ppm

Pulse Sequence: s2pul
Solvent: CDCL3
Ambient temperature
Operator: barbara
File: 1222-MS-04-318-pure
INOVA-500 "riga"

Relax. delay 4.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 10000.0 Hz
12 repetitions
OBSERVE H1, 499.7707226 MHz
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 3 min, 8 sec



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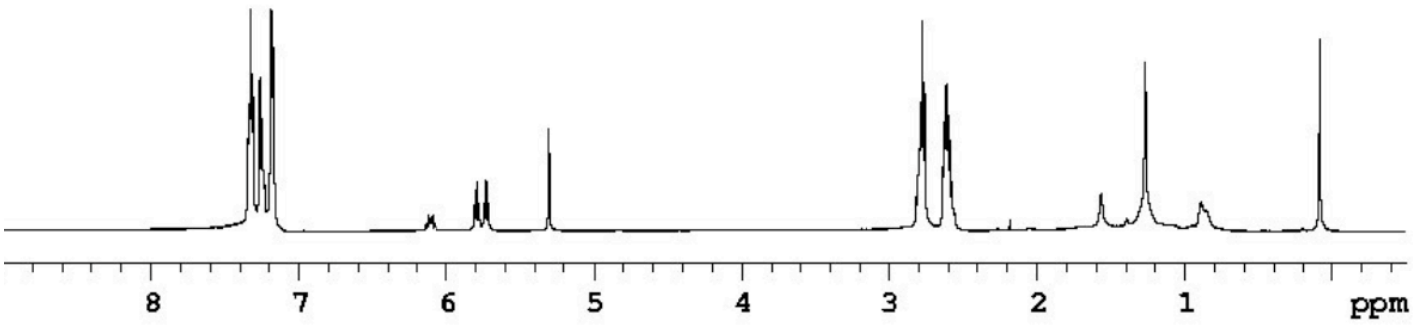

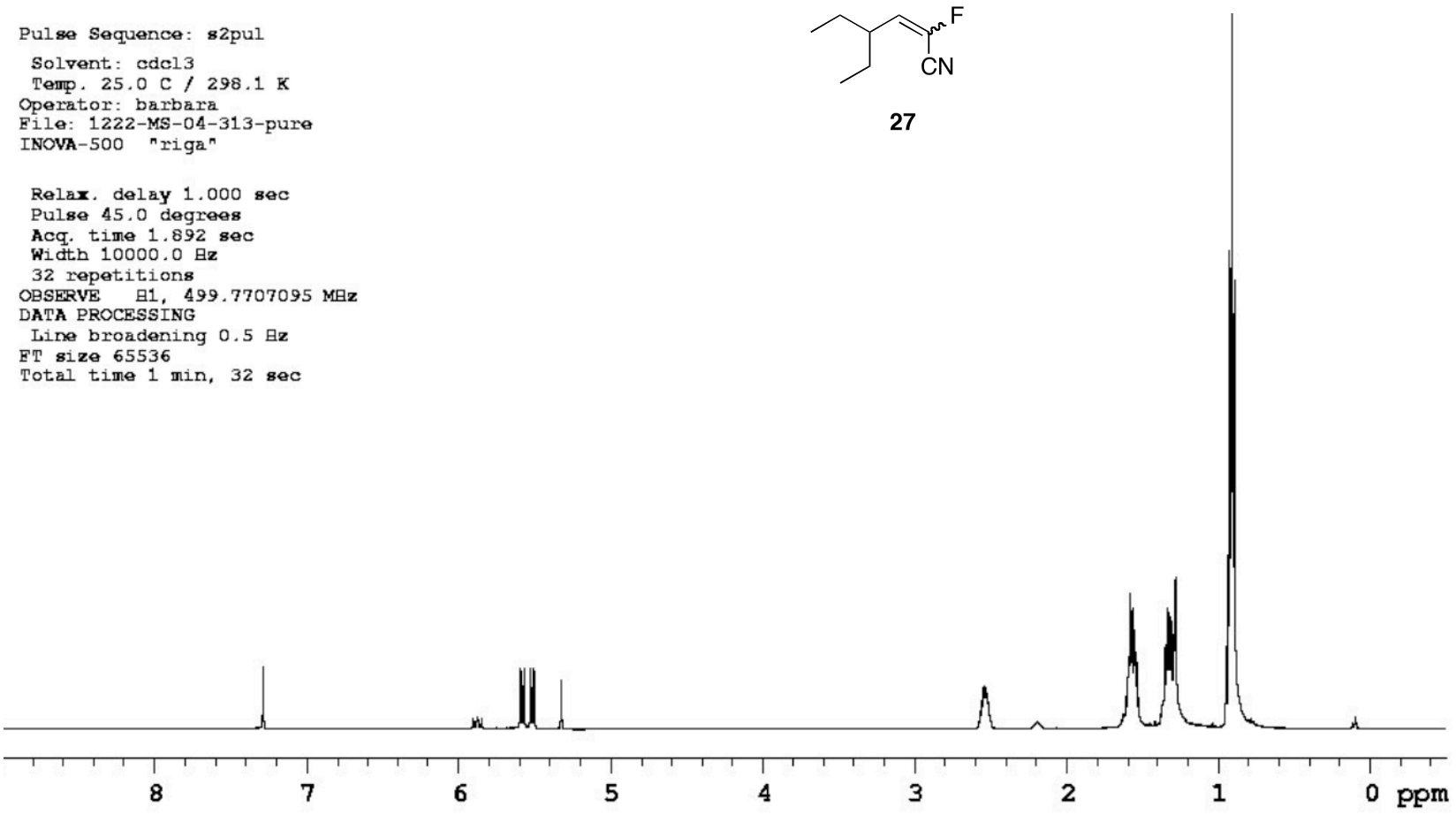
Pulse Sequence: s2pul
Solvent: cdcl3
Temp. 25.0 C / 298.1 K
Operator: barbara
File: 1222-MS-04-313-pure
INOVA-500 "riga"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 10000.0 Hz
32 repetitions
OBSERVE H1, 499.7707095 MHz
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 1 min, 32 sec
F
CN
27
8
7
6
5
4
3
2
1
0 ppm