Supplementary material

3-Functional substituted 4-trifluoromethyl tetrahydrothiophenes via [3+2]-cycloaddition reactions

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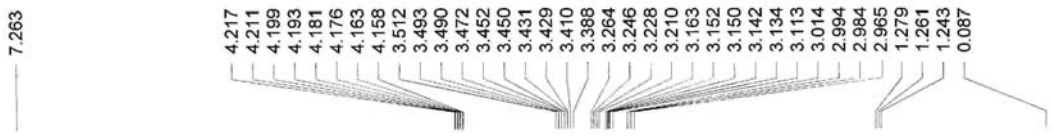
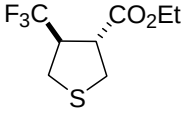
General. ^{1}H, ^{13}C, ^{13}C APT, COSY, HSQC, ^{19}F and ^{31}P NMR spectra were recorded on a Bruker Avance-400 or a Bruker Avance-500 (400 MHz for ^{1}H, 125 MHz for ^{13}C, 376 MHz for ^{19}F, 202 MHz for ^{31}P, and 500 MHz for COSY and HSQC) spectrometers in $CDCl_3$ or DMSO-d_6 solutions. The solvent residual peak was used as reference for ^{1}H and ^{13}C spectra ($CDCl_3$ δ_H = 7.26 ppm, δ_C = 77.16 ppm and DMSO-d_6 δ_H = 2.50 ppm, δ_C = 39.52 ppm) and C_6F_6 (δ_F = –162.9 ppm relative to $CFCl_3$) and H_3PO_4 (δ_P = 0.00 ppm) were used as internal standards for ^{19}F and ^{31}P NMR spectra respectively.

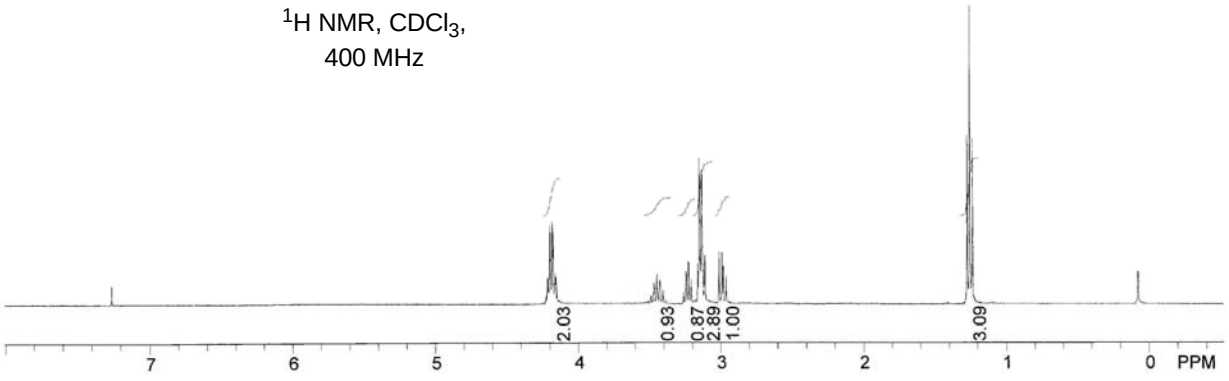
NMR spectra of all synthesized compounds

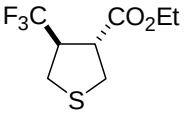




4a

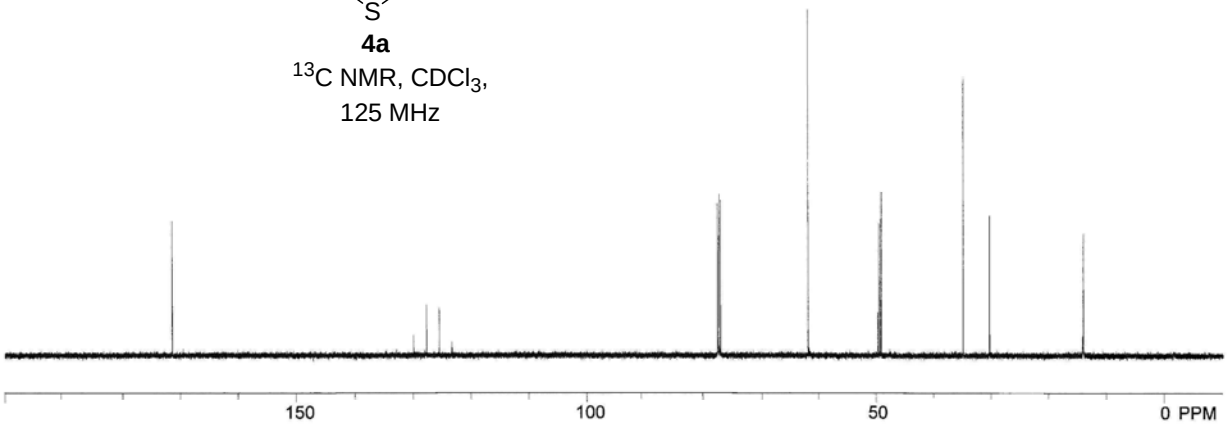
^{1}H NMR, $CDCl_3$,
400 MHz

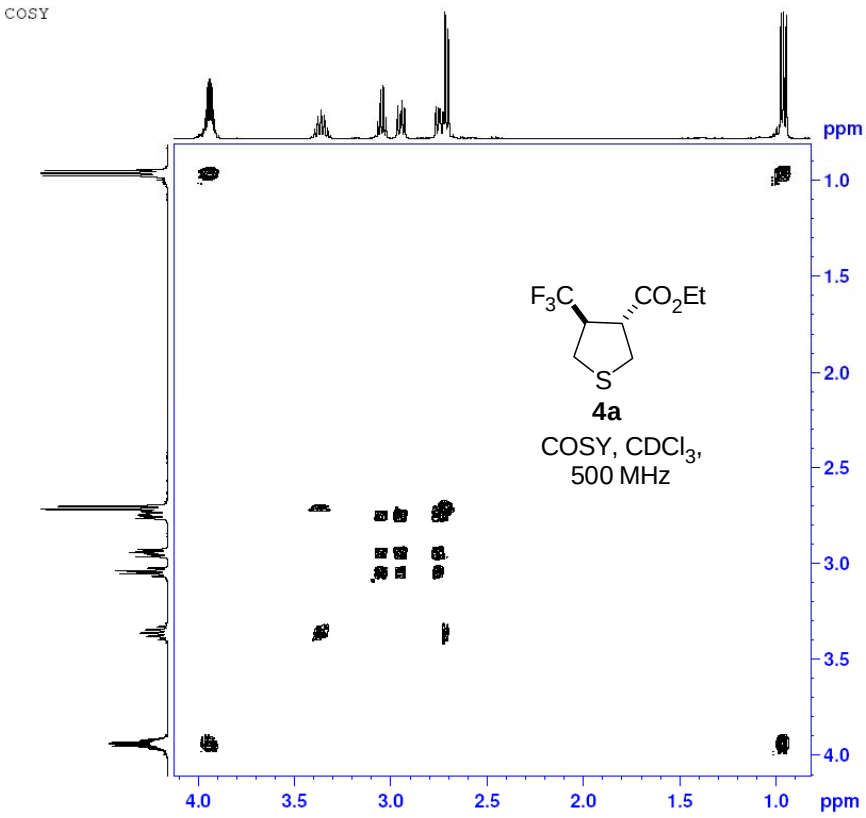




4a

^{13}C NMR, $CDCl_3$,
125 MHz





4a

COSY, $CDCl_3$, 500 MHz

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PROCNO                     1
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Time                    0.32
INSTRUM                spect
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PULPROG             cosygpqf
TD                      2048
SOLVENT                 C6D6
NS                         1
DS                        16
SWH                2003.205 Hz
FIDRES             0.978127 Hz
AQ                 0.5114804 sec
RG                     161.3
DW                   249.600 usec
DE                     6.00 usec
TE                    683.2 K
d0                 0.00000300 sec
D1                 2.00000000 sec
d13                0.00000400 sec
D16                0.00050000 sec
IN0                0.00050005 sec

======== CHANNEL f1 ========
NUC1                       1H
P0                       9.50 usec
P1                       9.50 usec
PL1                      0.00 dB
SFO1               499.9512499 MHz

====== GRADIENT CHANNEL =====
GPNAM1               SINE.100
GPNAM2               SINE.100
GPZ1                    10.00 %
GPZ2                    10.00 %
P16                   1000.00 usec
ND0                        1
TD                       128
SFO1                499.9512 MHz
FIDRES             15.623438 Hz
SW                      4.000 ppm
FnMODE                    QF
SI                      2048
SF                 499.9500000 MHz
WDW                     SINE
SSB                        0
LB                      0.00 Hz
GB                         0
PC                      1.40
SI                      2048
MC2                       QF
SF                 499.9500000 MHz
WDW                     SINE
SSB                        0
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GB                         0
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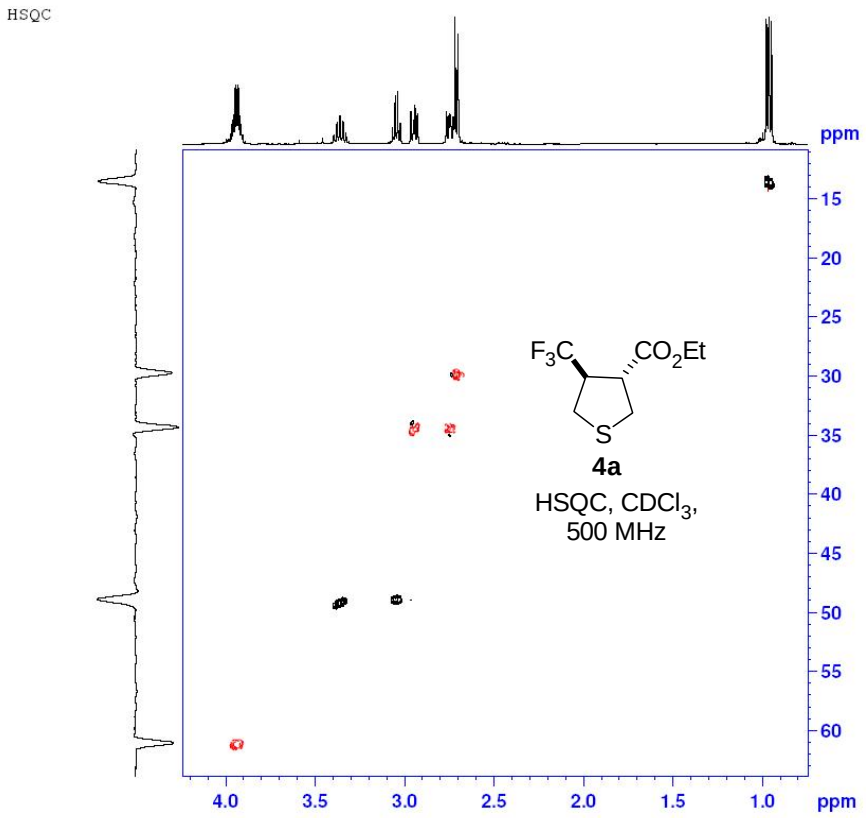

4a

HSQC, $CDCl_3$, 500 MHz

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PROCNO                     1
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INSTRUM                spect
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TD                      1024
SOLVENT                 C6D6
NS                         2
DS                        16
SWH                2003.205 Hz
FIDRES             1.956255 Hz
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RG                     49152
DW                   249.600 usec
DE                     6.00 usec
TE                    683.2 K
CNST2              145.0000000
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D1                 1.75000000 sec
d4                 0.00172414 sec
d11                0.03000000 sec
d13                0.00000400 sec
D16                0.00020000 sec
D21                0.00344830 sec
DELTA              0.00222270 sec
DELTA1             0.00071614 sec
IN0                0.00006120 sec
STSCNT                     0
ZGOPTNS

-------- CHANNEL f1 --------
NUC1                       1H
P1                       9.80 usec
p2                      19.60 usec
P28                   1000.00 usec
PL1                      0.00 dB
SFO1               499.9512499 MHz

-------- CHANNEL f2 --------
CPDPRG2                 garp
NUC2                       13C
P3                       4.30 usec
p4                       8.60 usec
PCPD2                   60.00 usec
PL2                      10.00 dB
PL12                     40.00 dB
SFO2               125.7172412 MHz

------ GRADIENT CHANNEL -----
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GPNAM2               SINE.100
GPZ1                    80.00 %
GPZ2                    20.10 %
P16                   1000.00 usec
ND0                        2
TD                       100
SFO1                125.7172 MHz
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WDW                     QSINE
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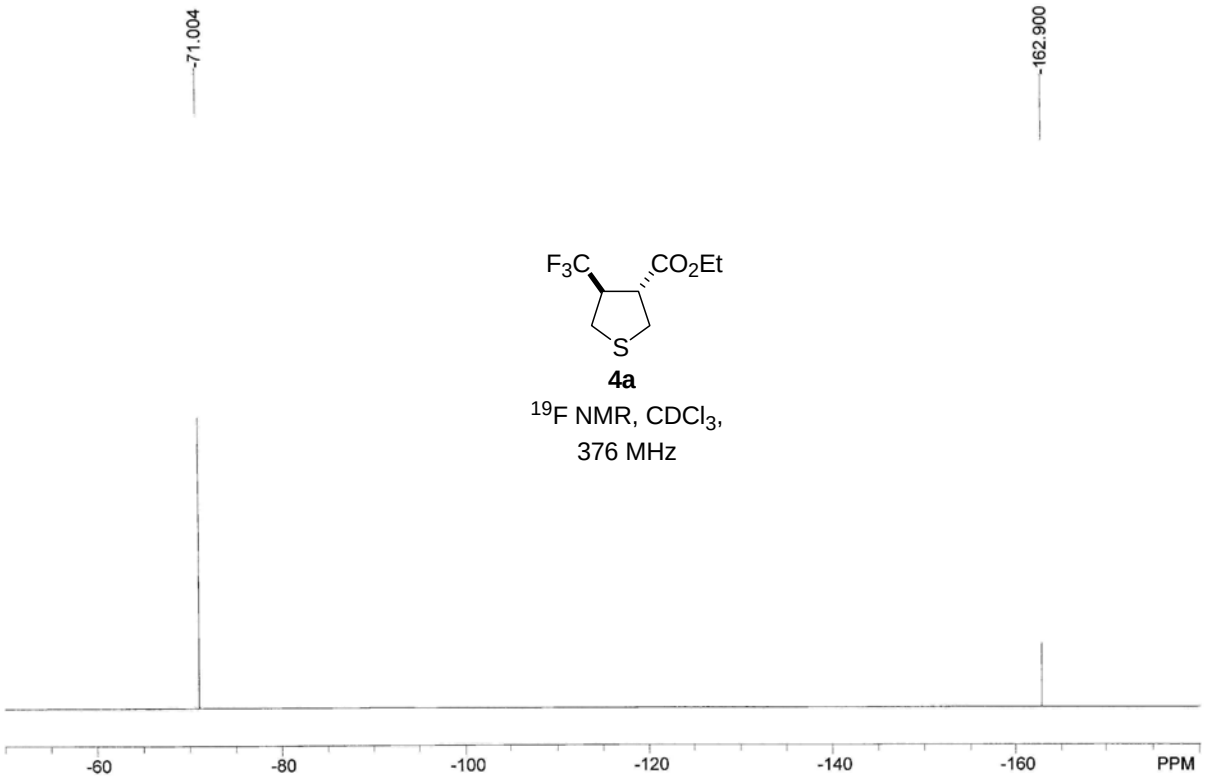
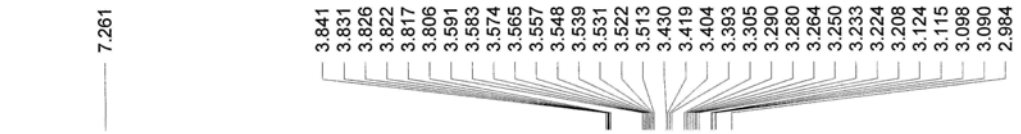
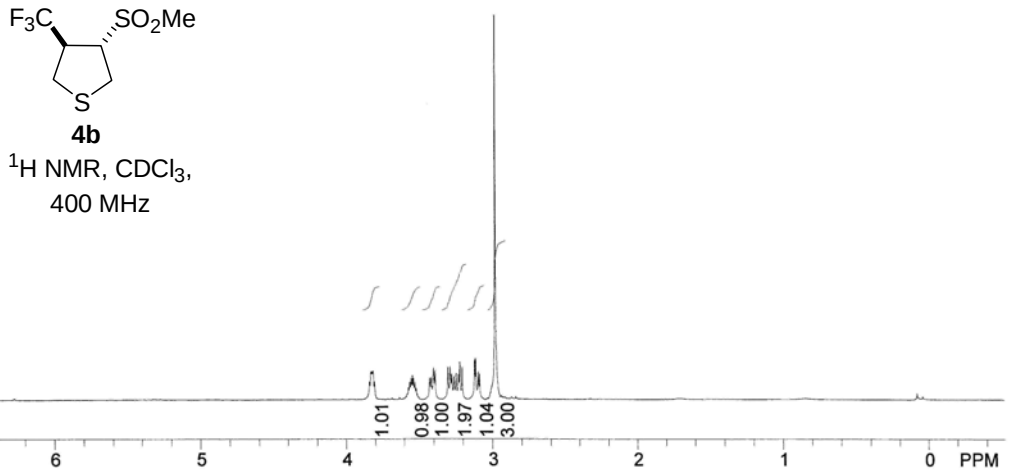


4a

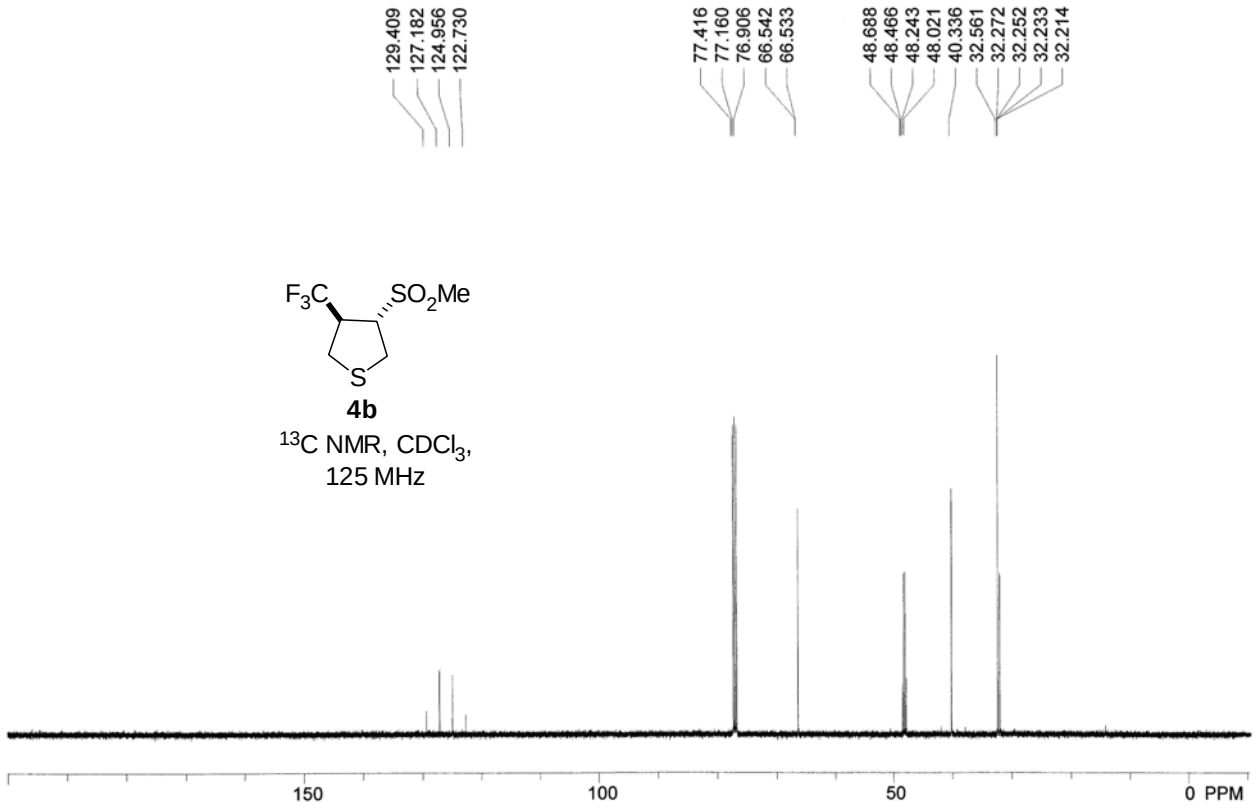
^{19}F NMR, $CDCl_3$,
376 MHz





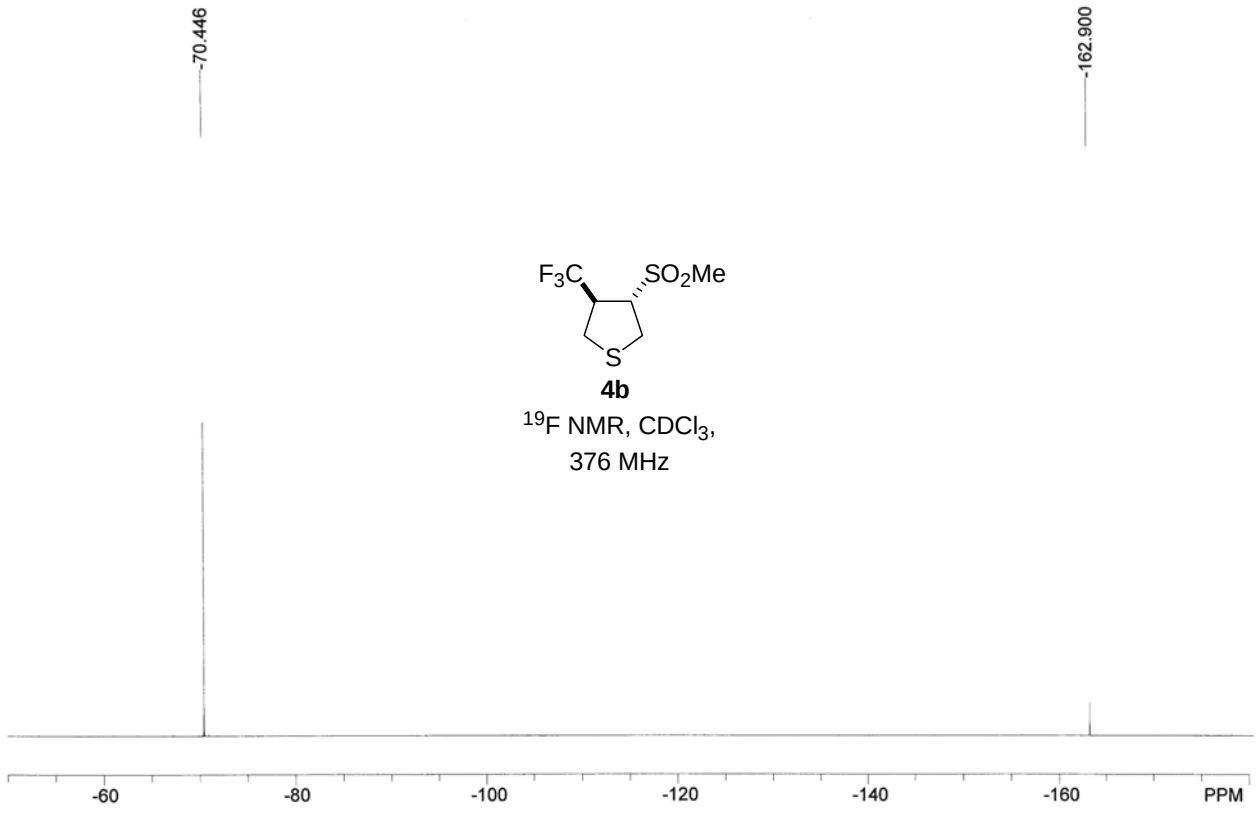
4b

^{1}H NMR, $CDCl_3$,
400 MHz



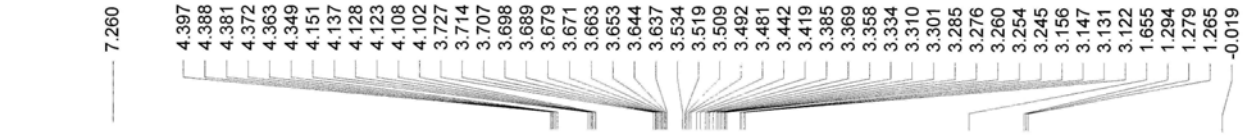
4b

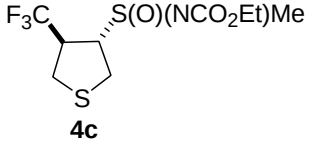
^{13}C NMR, $CDCl_3$, 125 MHz



4b

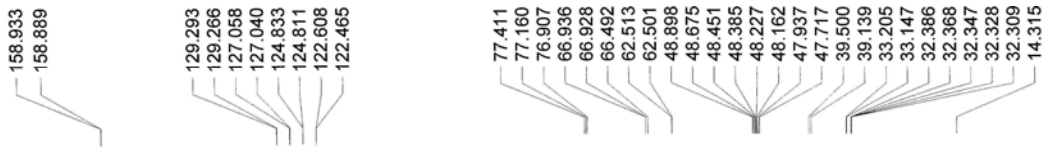
^{19}F NMR, $CDCl_3$, 376 MHz

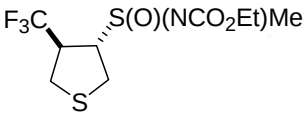




4c

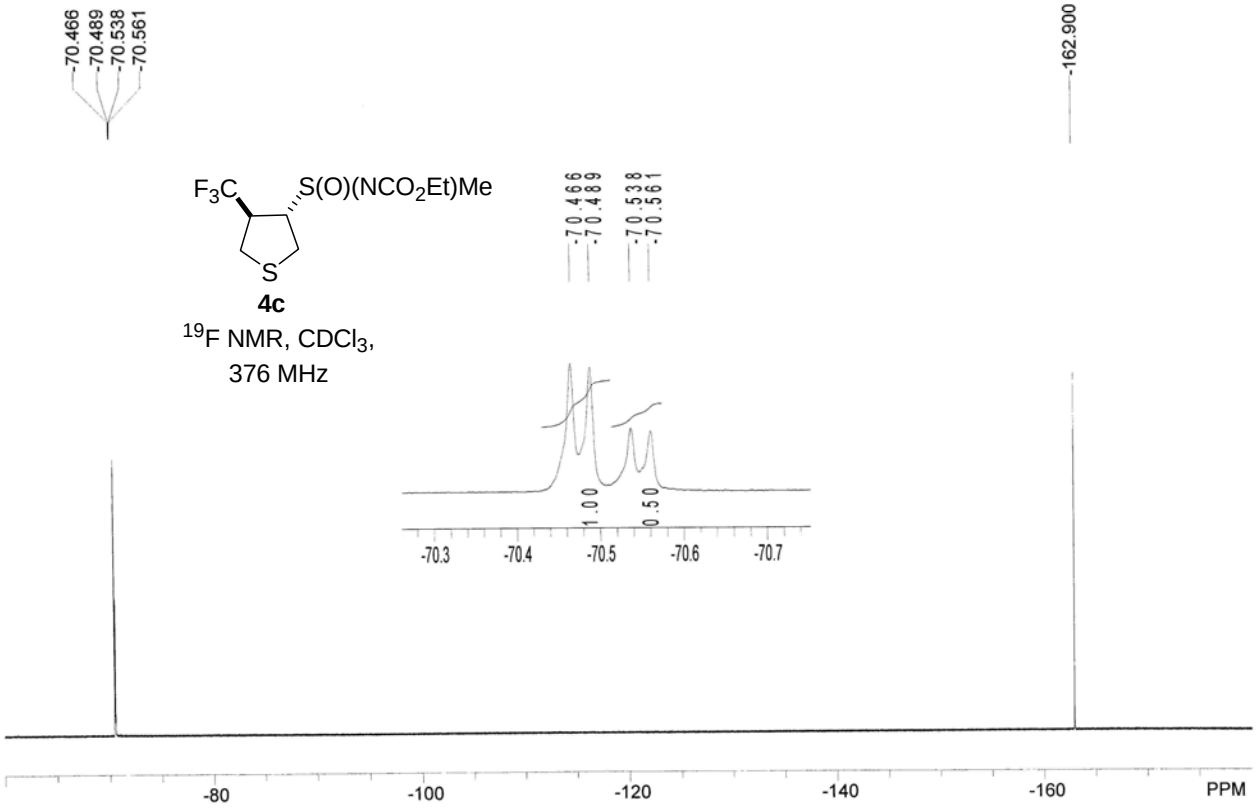
^{1}H NMR, $CDCl_3$,
400 MHz

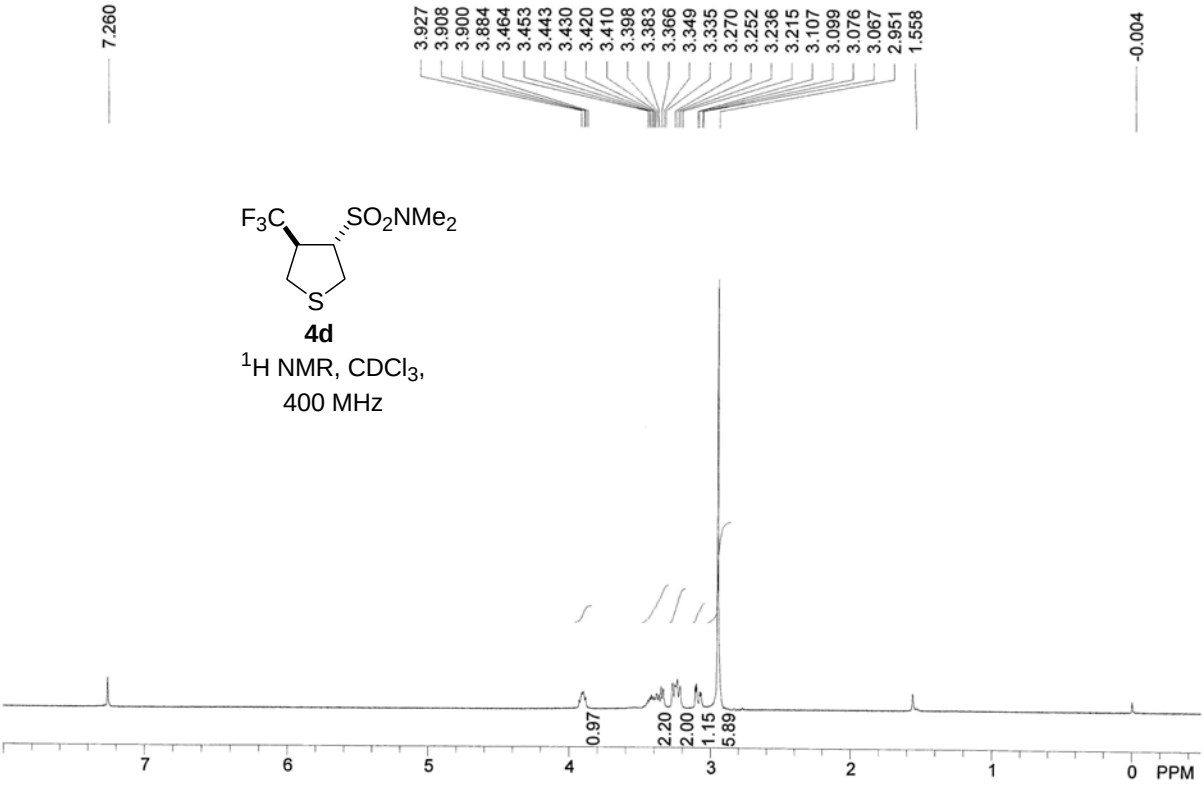




4c

^{13}C NMR, $CDCl_3$,
125 MHz


-70.466
-70.489
-70.538
-70.561
-162.900
F3C
S(O)(NCO2Et)Me
S
4c
19F NMR, CDCl3,
376 MHz
1.00
0.50
-70.3
-70.4
-70.5
-70.6
-70.7
-80
-100
-120
-140
-160
PPM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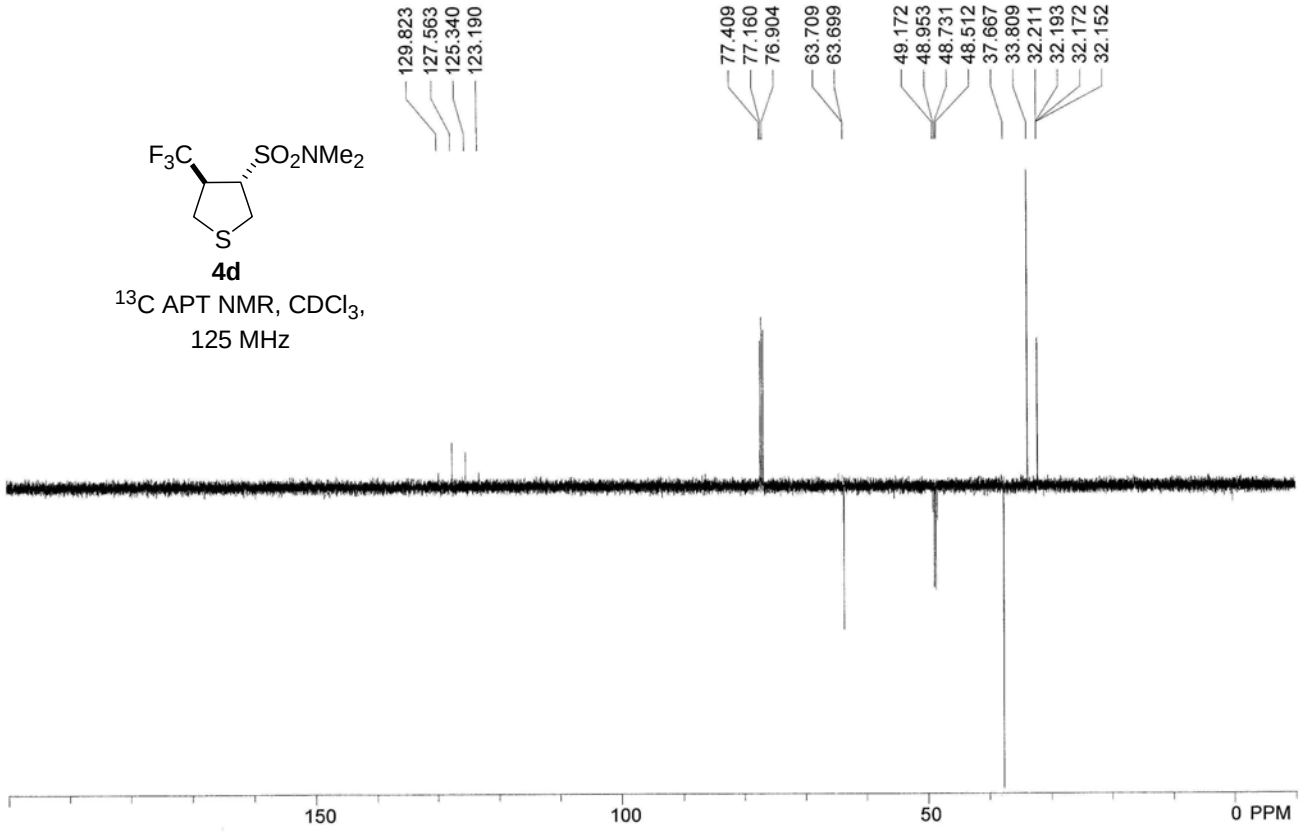
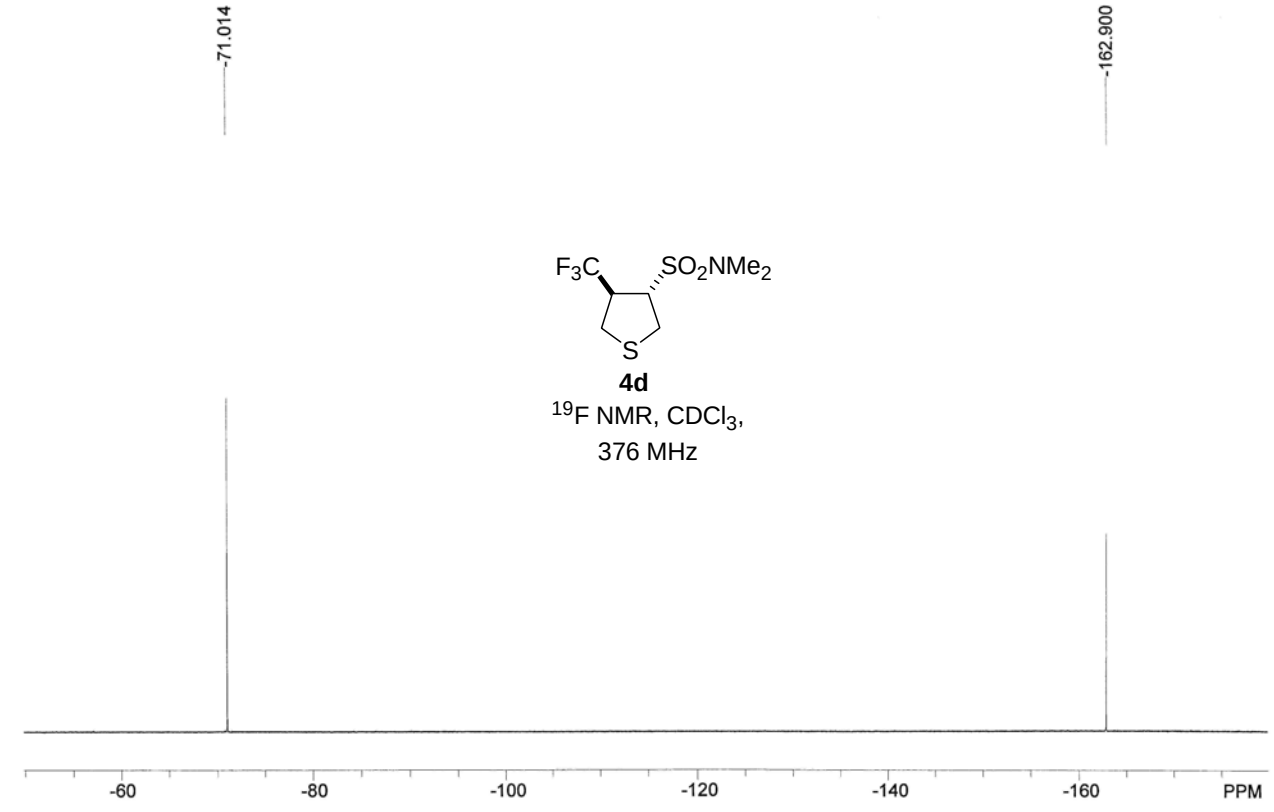
3.927
3.908
3.900
3.884
3.464
3.453
3.443
3.430
3.420
3.410
3.398
3.383
3.366
3.349
3.335
3.270
3.252
3.236
3.215
3.107
3.099
3.076
3.067
2.951
1.558
-0.004
F3C
SO2NMe2
S
4d
1H NMR, CDCl3,
400 MHz
0.97
2.20
2.00
1.15
5.89
7
6
5
4
3
2
1
0
PPM



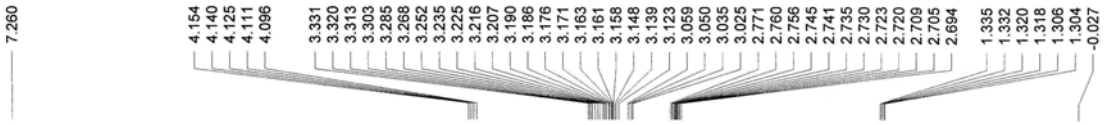
4d

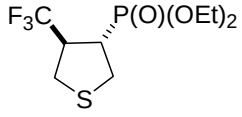
^{13}C APT NMR, $CDCl_3$, 125 MHz



4d

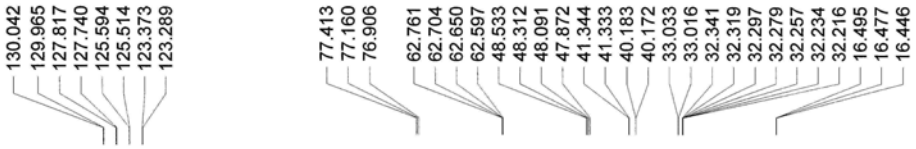
^{19}F NMR, $CDCl_3$, 376 MHz

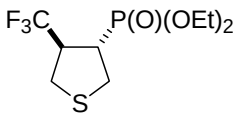




4e

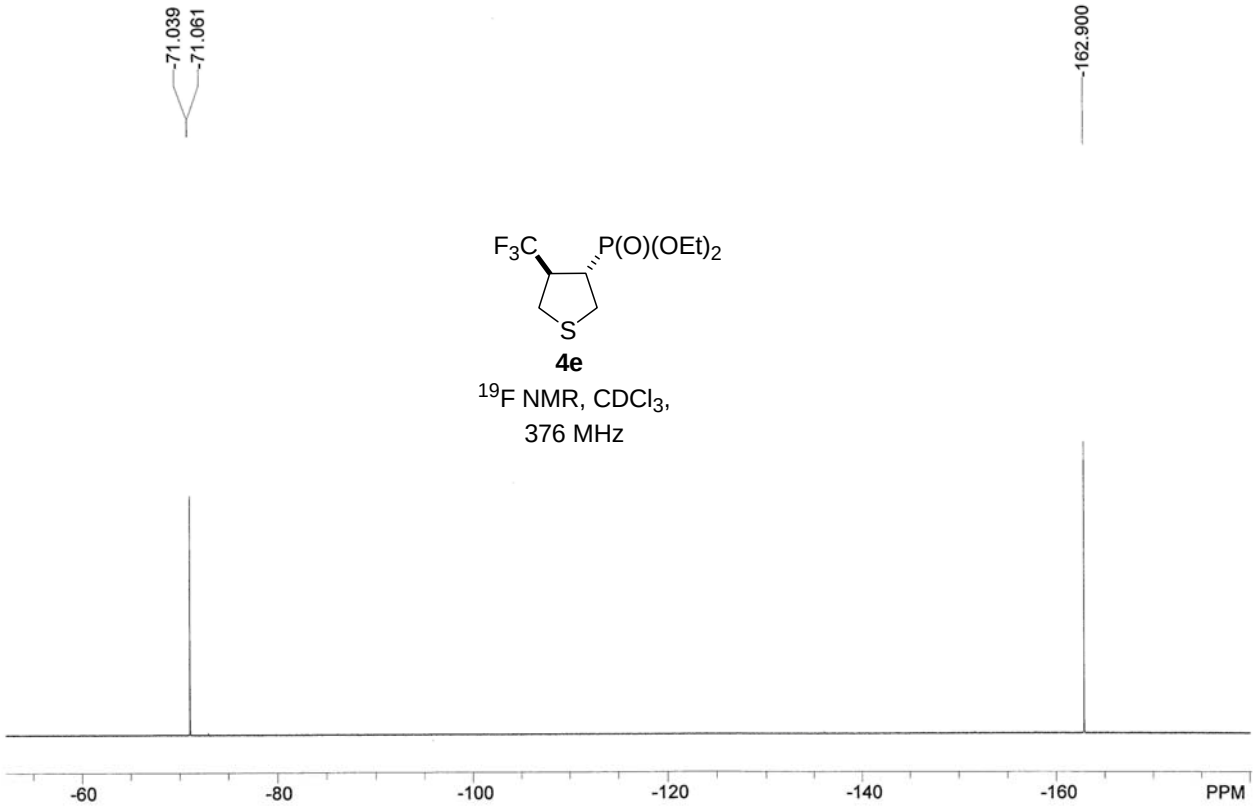
^{1}H NMR, $CDCl_3$,
400 MHz





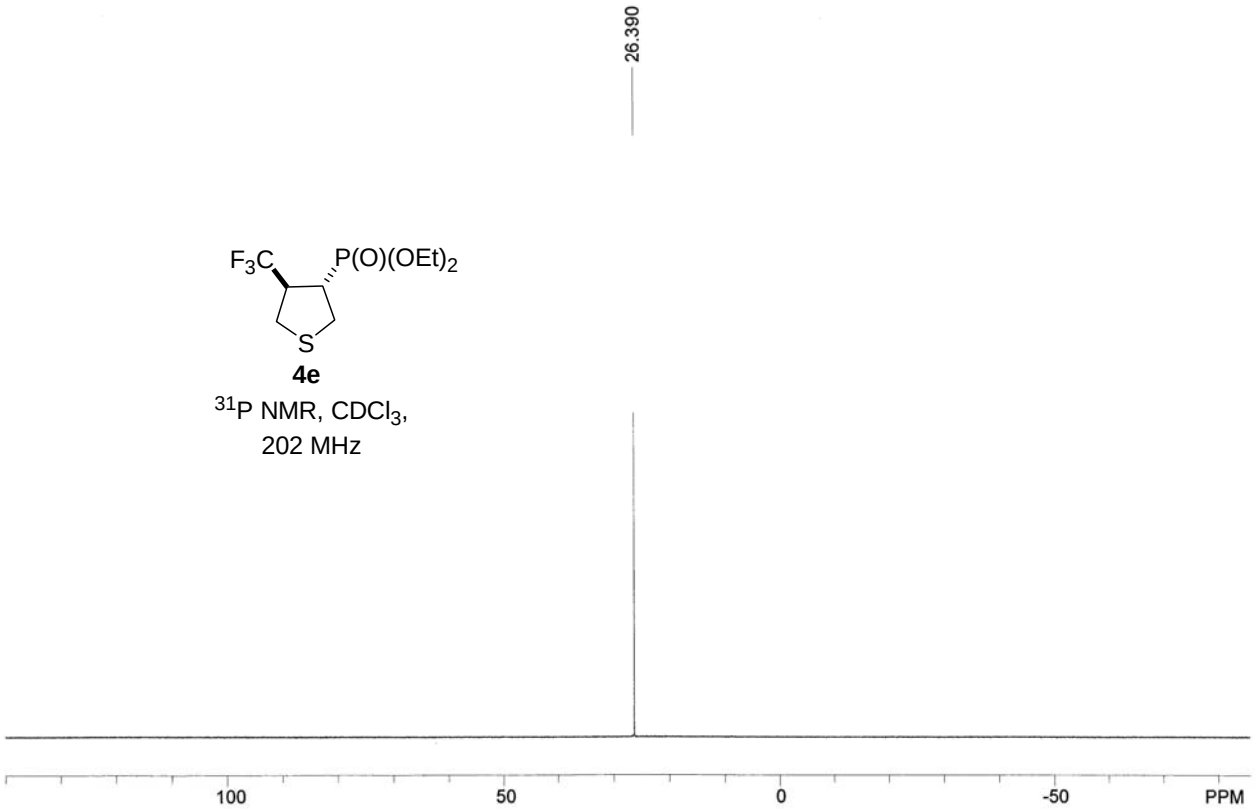
4e

^{13}C NMR, $CDCl_3$,
125 MHz



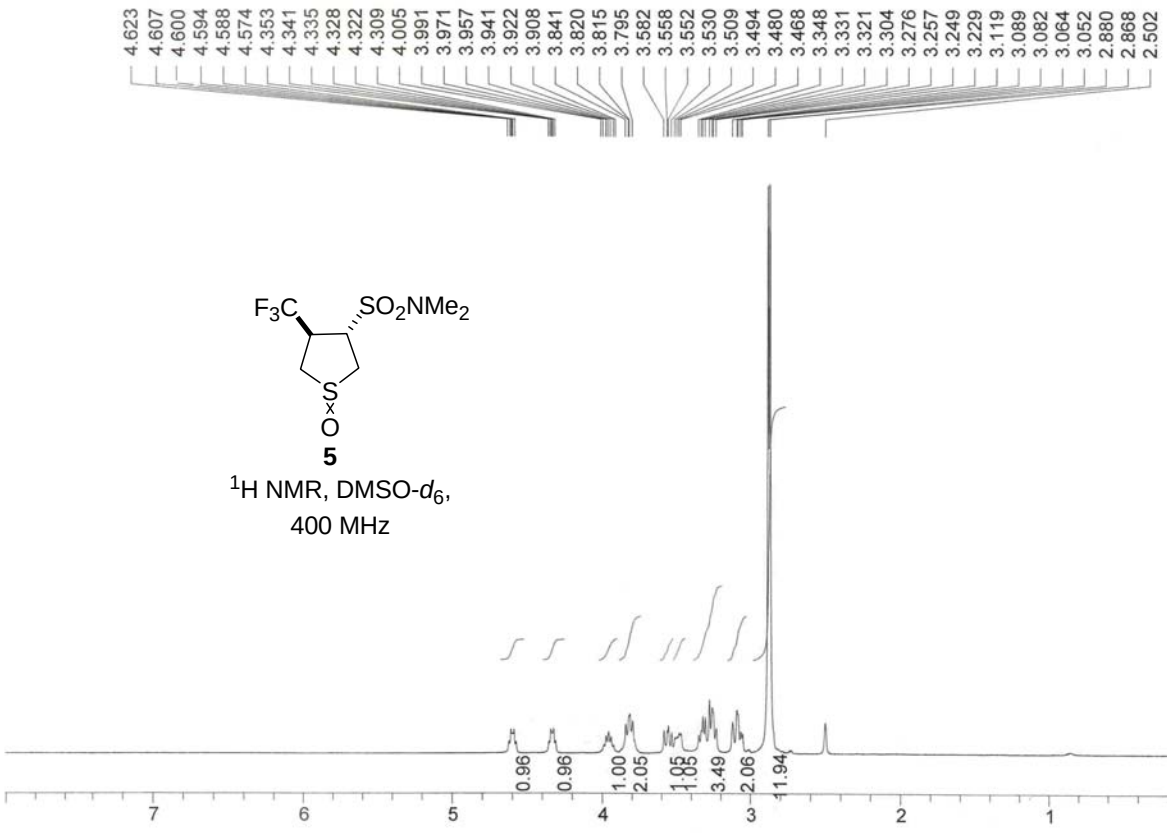
4e

^{19}F NMR, $CDCl_3$,
376 MHz



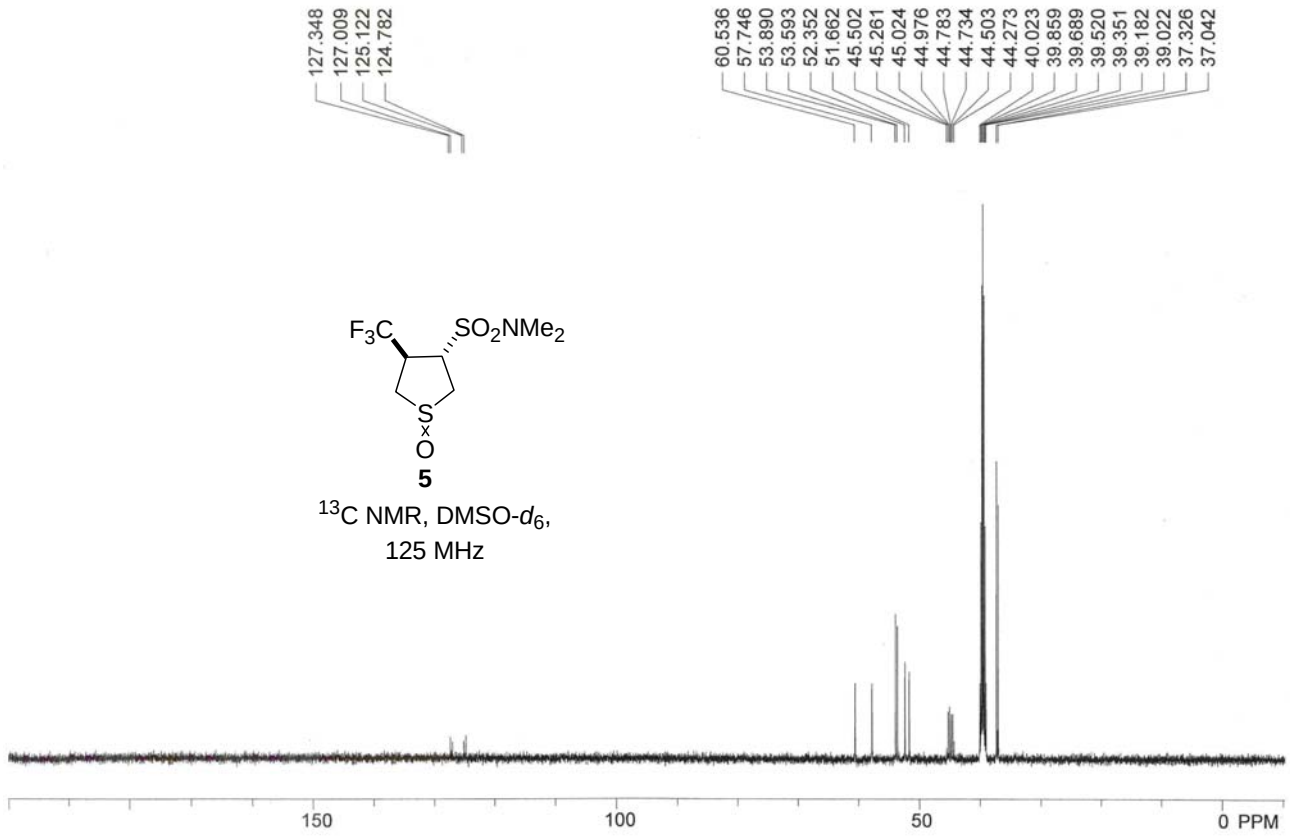
4e

^{31}P NMR, $CDCl_3$,
202 MHz



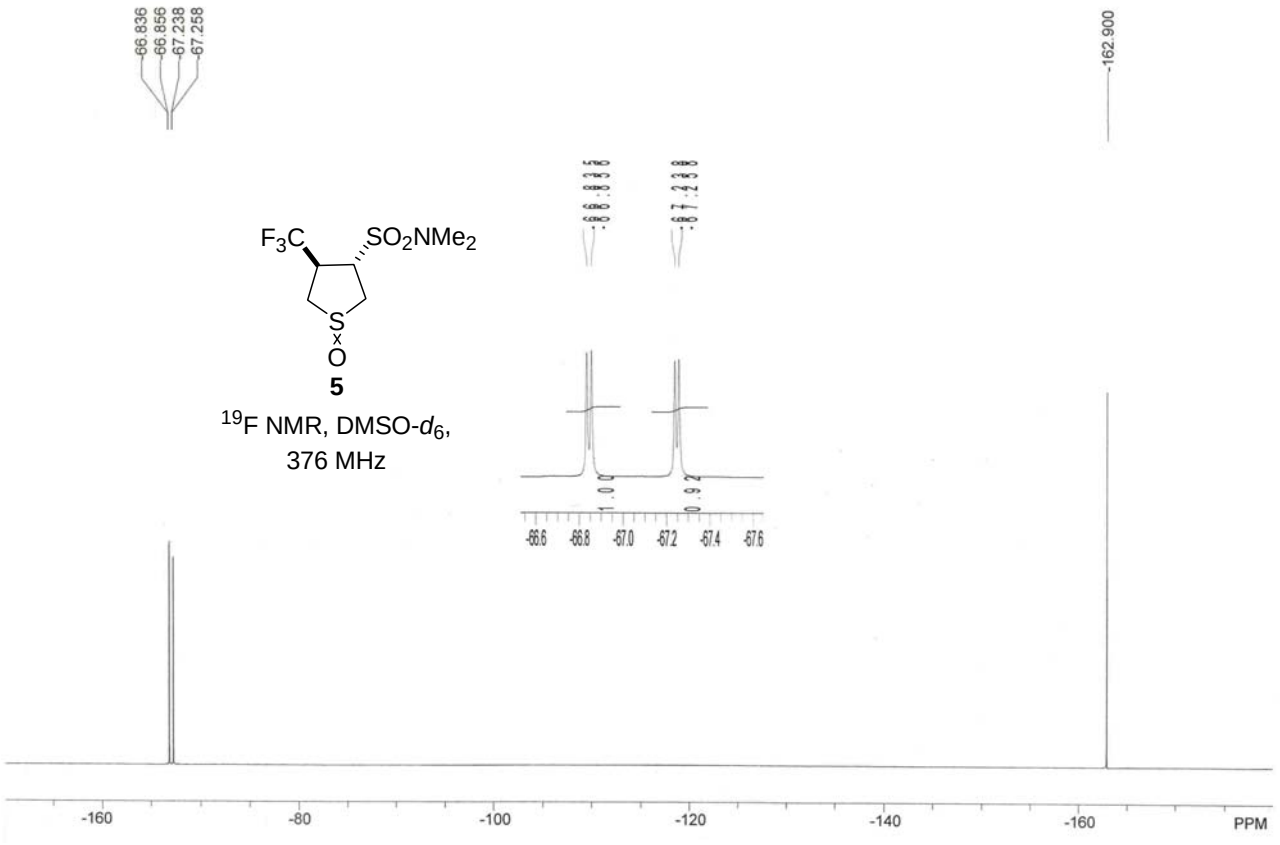
5

^{1}H NMR, DMSO-d_6,
400 MHz



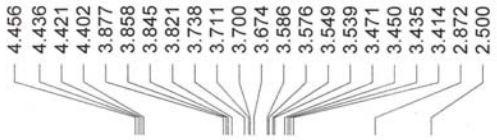
5

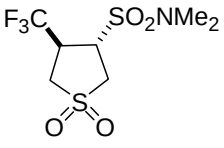
^{13}C NMR, DMSO-d_6,
125 MHz



5

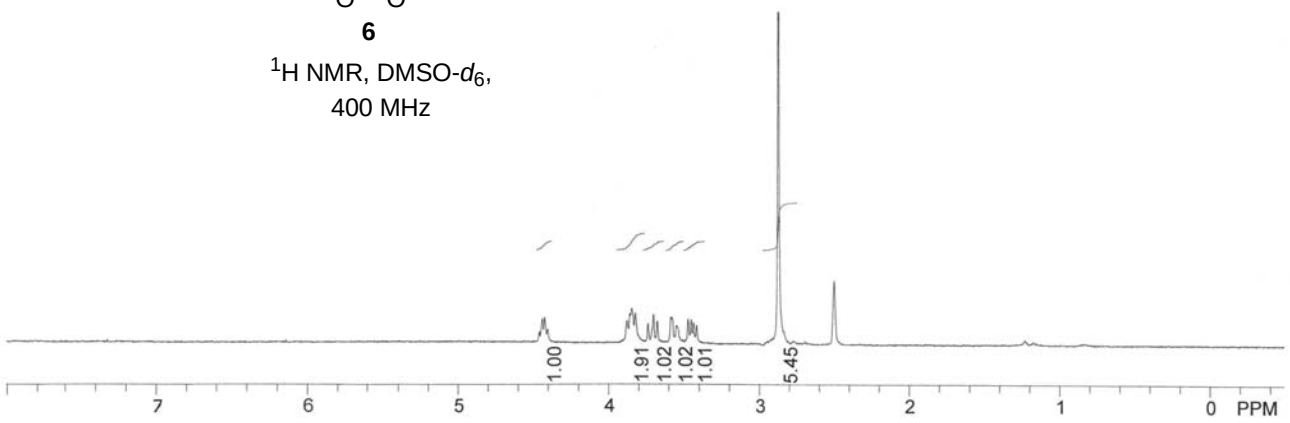
^{19}F NMR, DMSO-d_6,
376 MHz

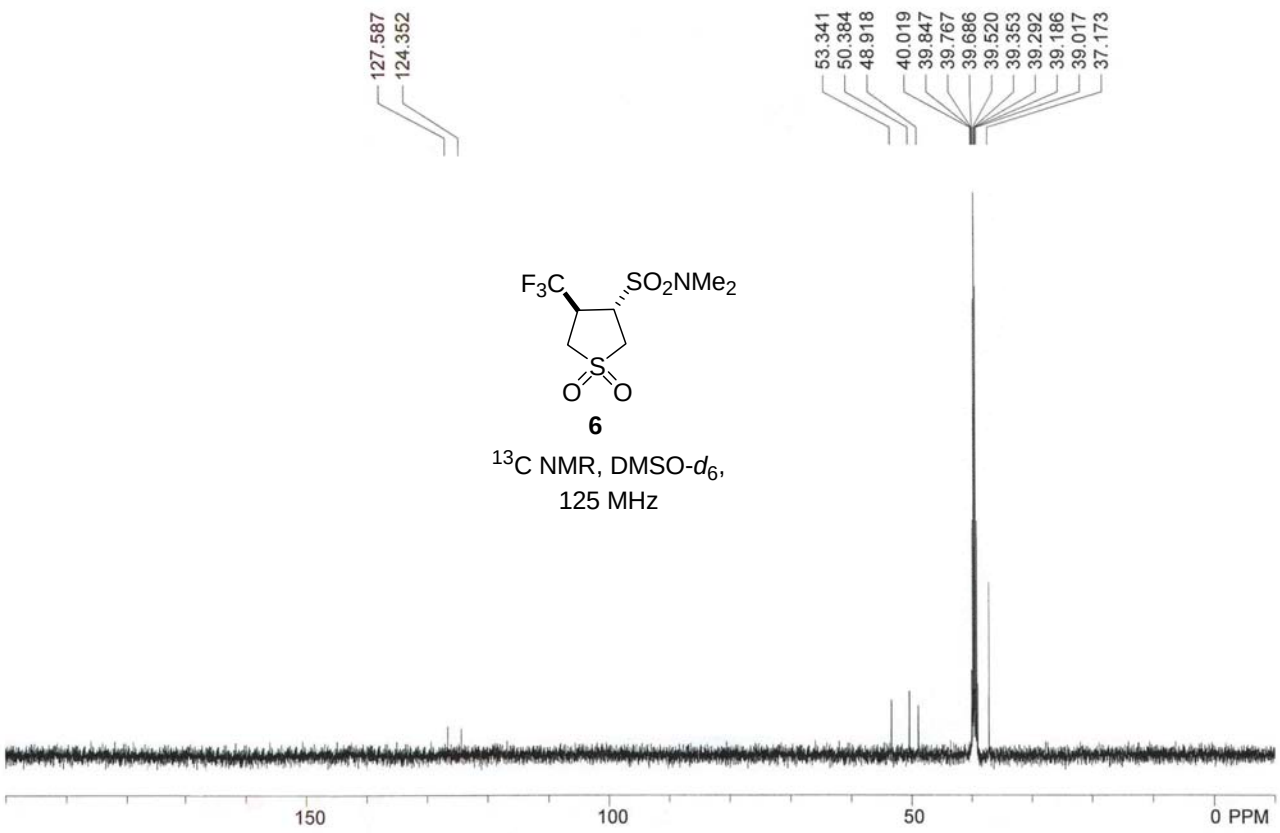




6

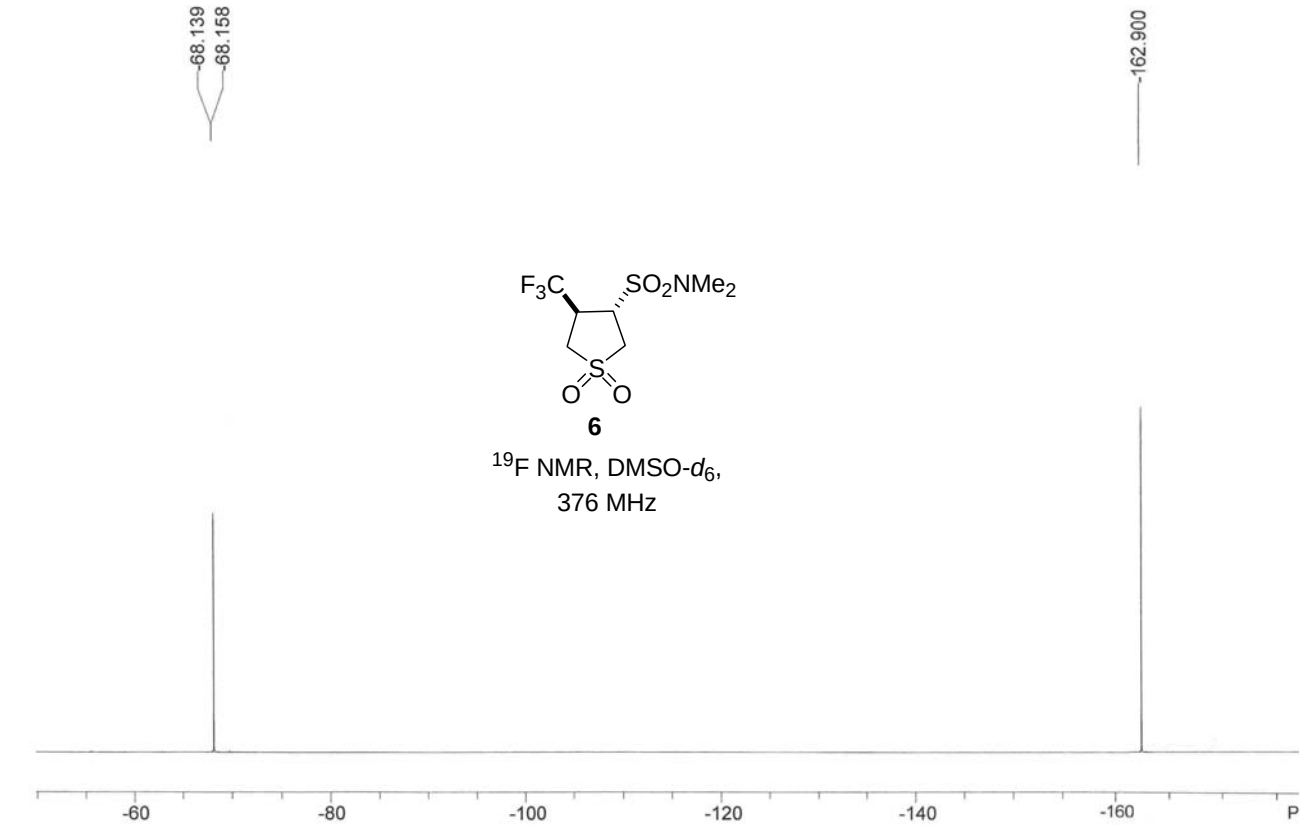
^{1}H NMR, DMSO-d_6,
400 MHz





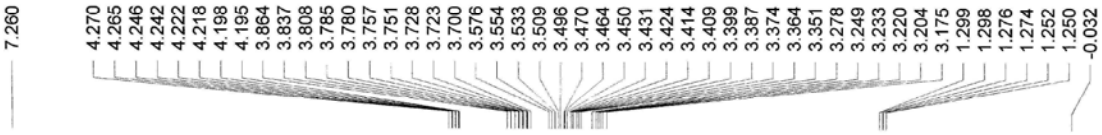
6

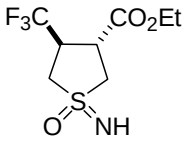
^{13}C NMR, DMSO-d_6,
125 MHz



6

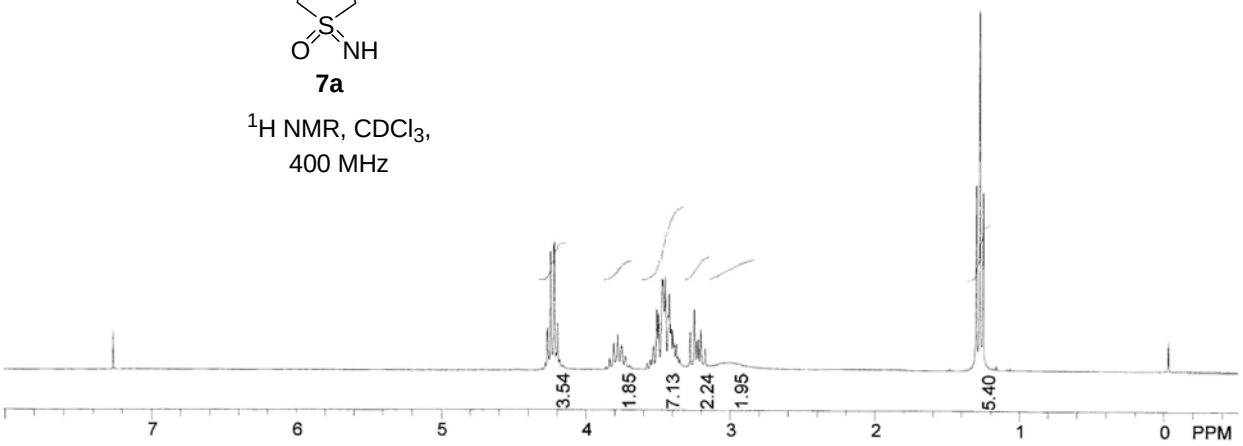
^{19}F NMR, DMSO-d_6,
376 MHz

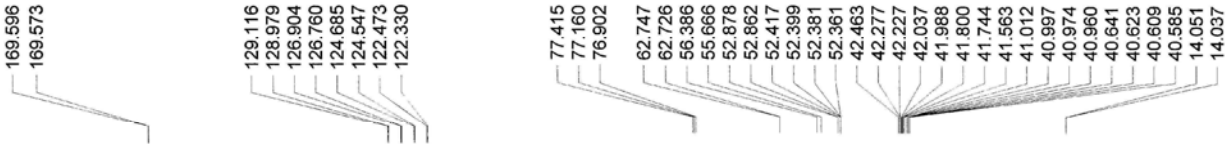


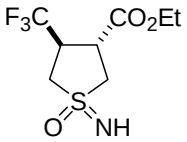


7a

^{1}H NMR, $CDCl_3$,
400 MHz

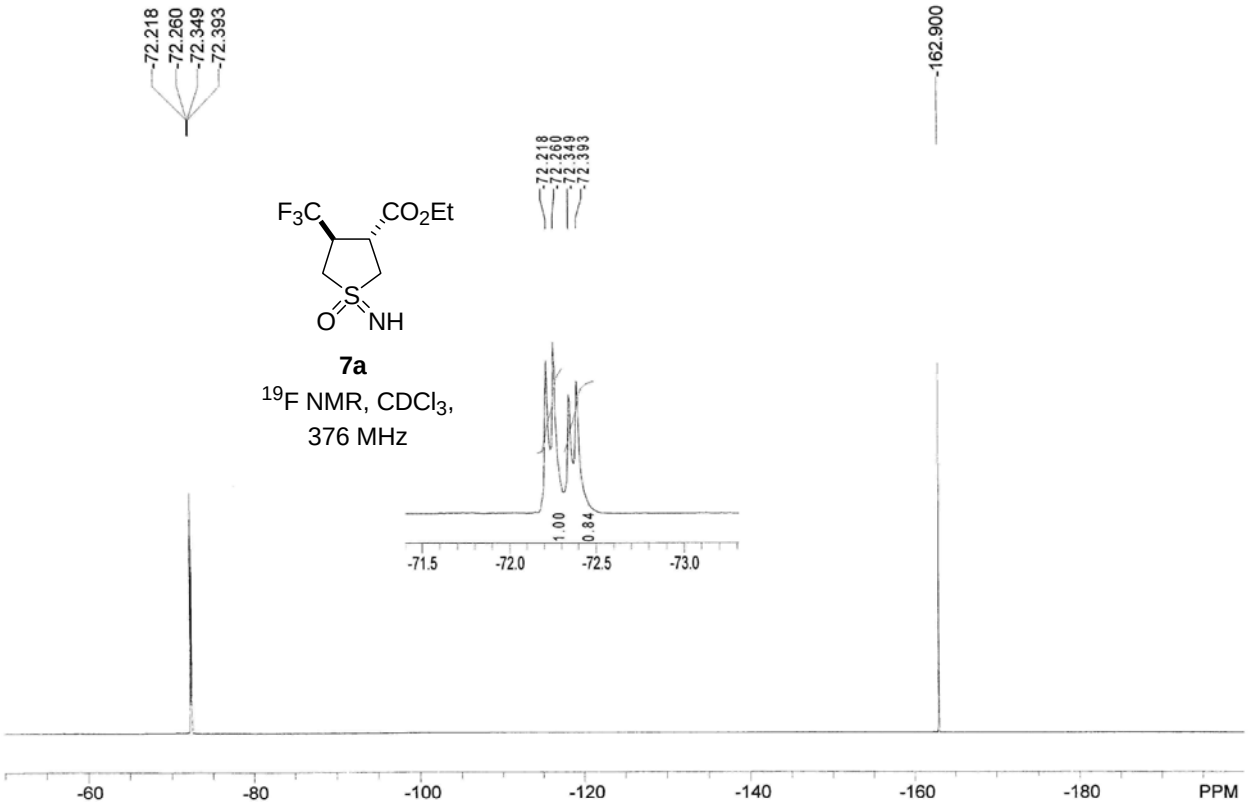






7a

^{13}C NMR, $CDCl_3$,
125 MHz


-72.218
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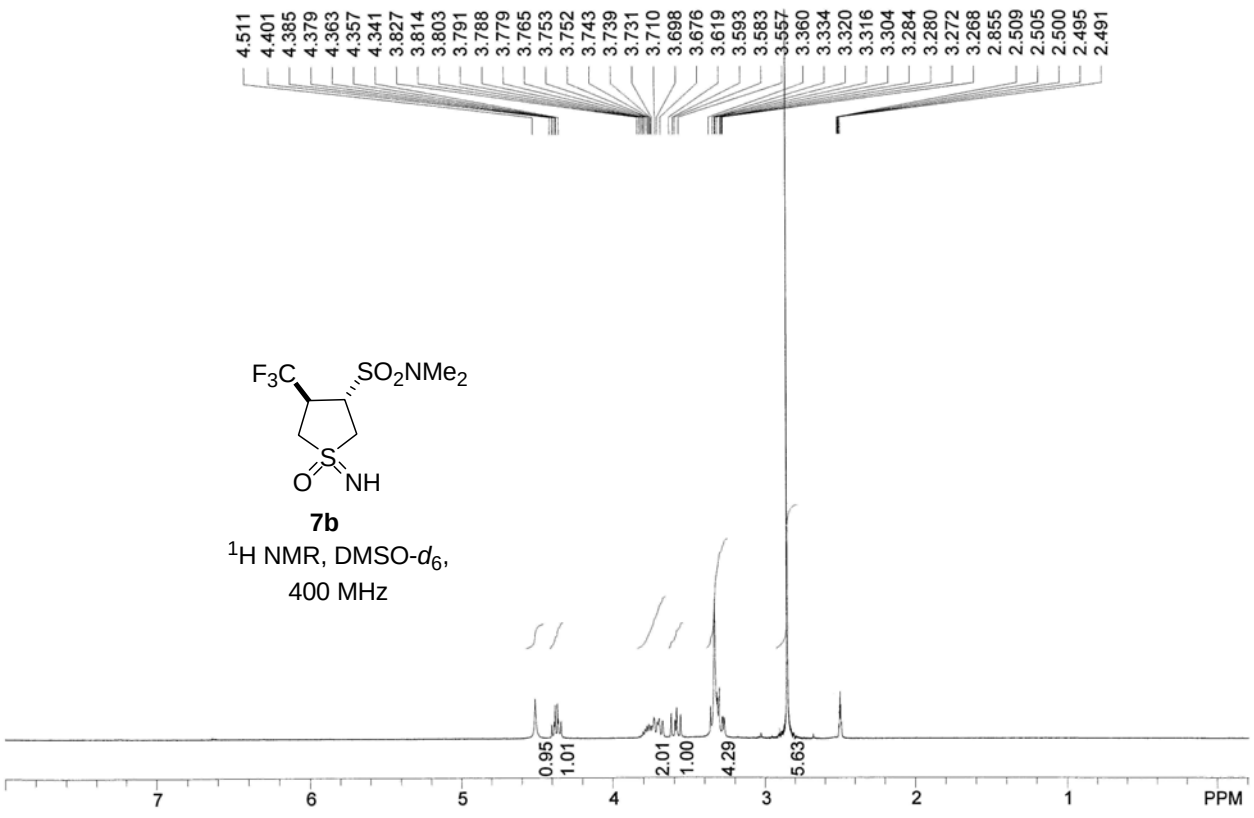
-72.349
-72.393
-162.900
F3C
CO2Et
S
O
NH
7a
19F NMR, CDCl3,
376 MHz
1.00
0.84
-71.5
-72.0
-72.5
-73.0
-60
-80
-100
-120
-140
-160
-180
PPM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3.803
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3.593
3.583
3.557
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3.320
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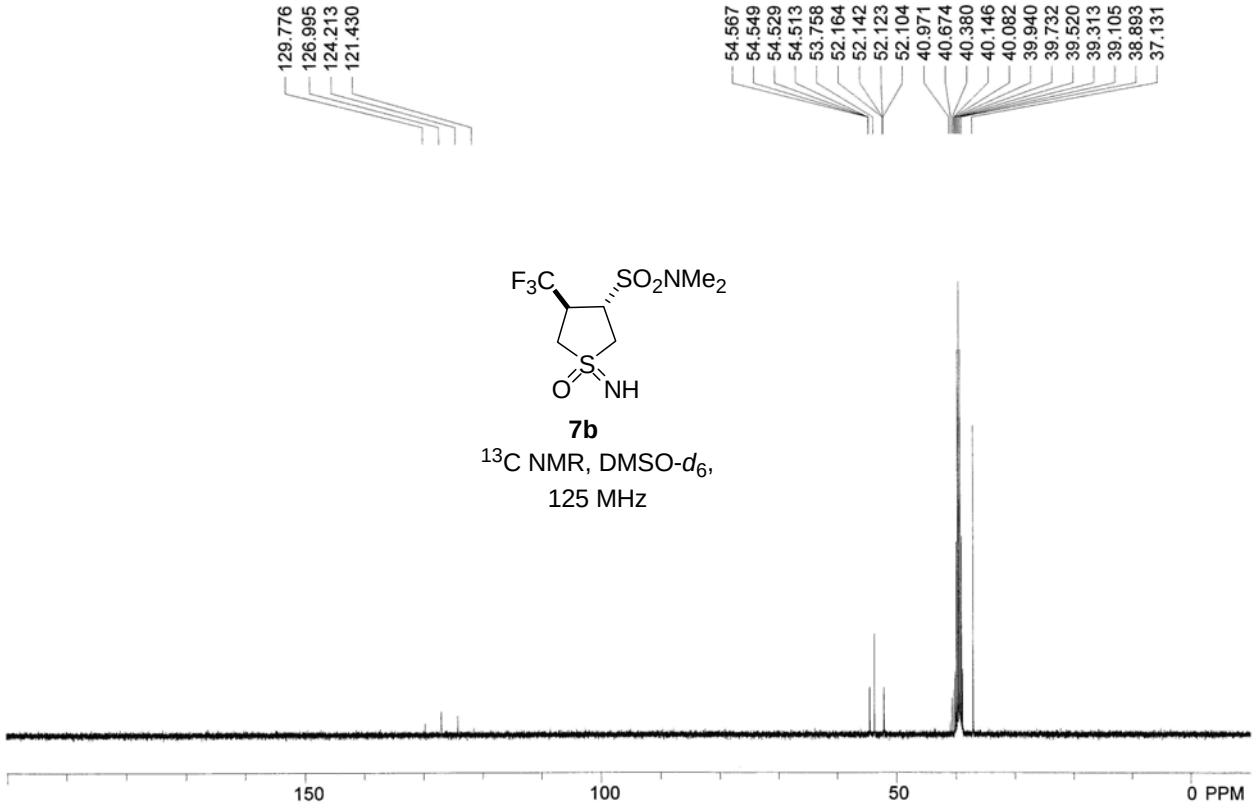
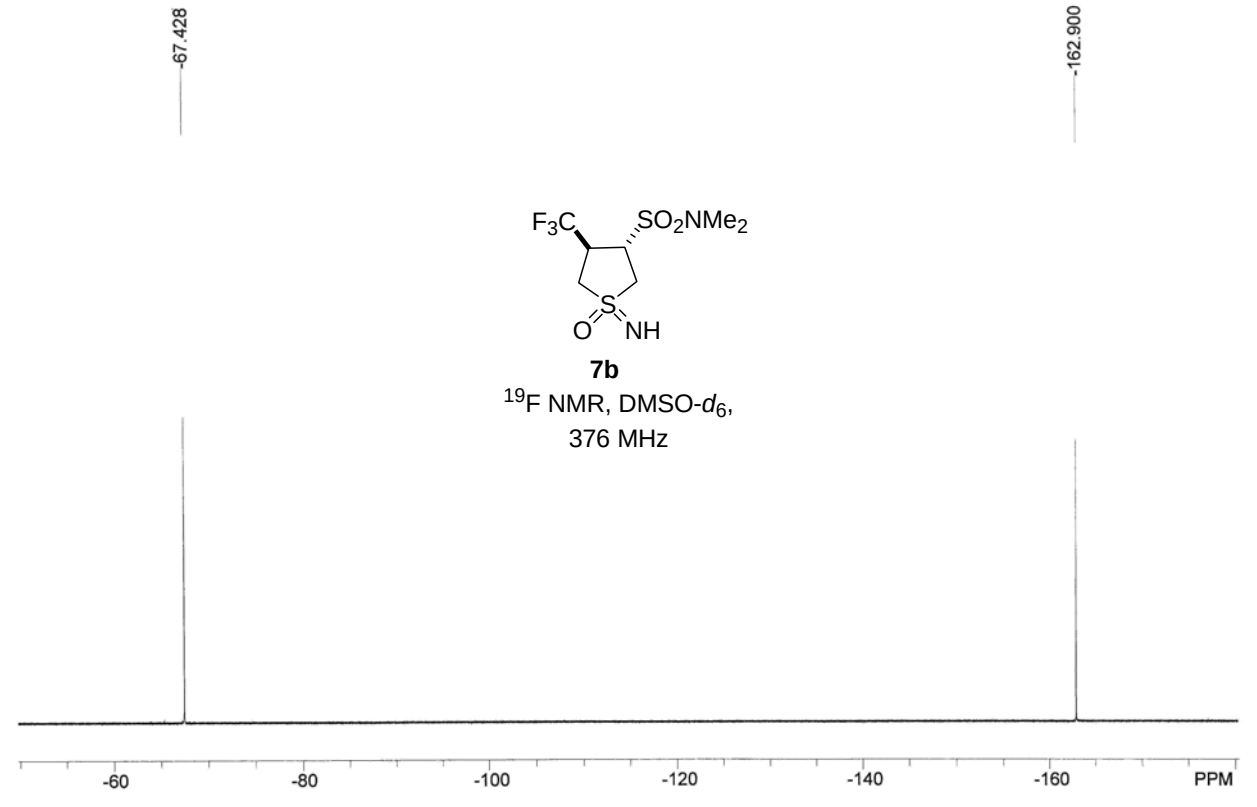
3.284
3.280
3.272
3.268
2.855
2.509
2.505
2.500
2.495
2.491
F3C
SO2NMe2
S
O
NH
7b
1H NMR, DMSO-d6,
400 MHz
0.95
1.01
2.01
1.00
4.29
5.63
7
6
5
4
3
2
1
PPM



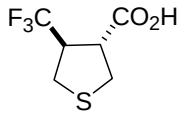
7b

^{13}C NMR, DMSO-d_6,
125 MHz



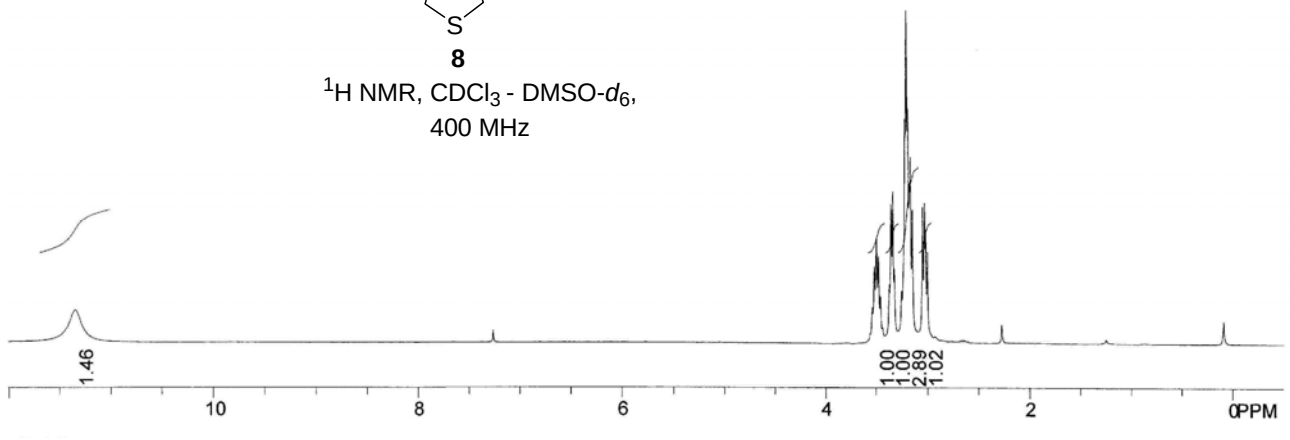
7b

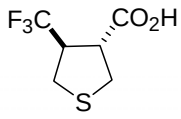
^{19}F NMR, DMSO-d_6,
376 MHz



8

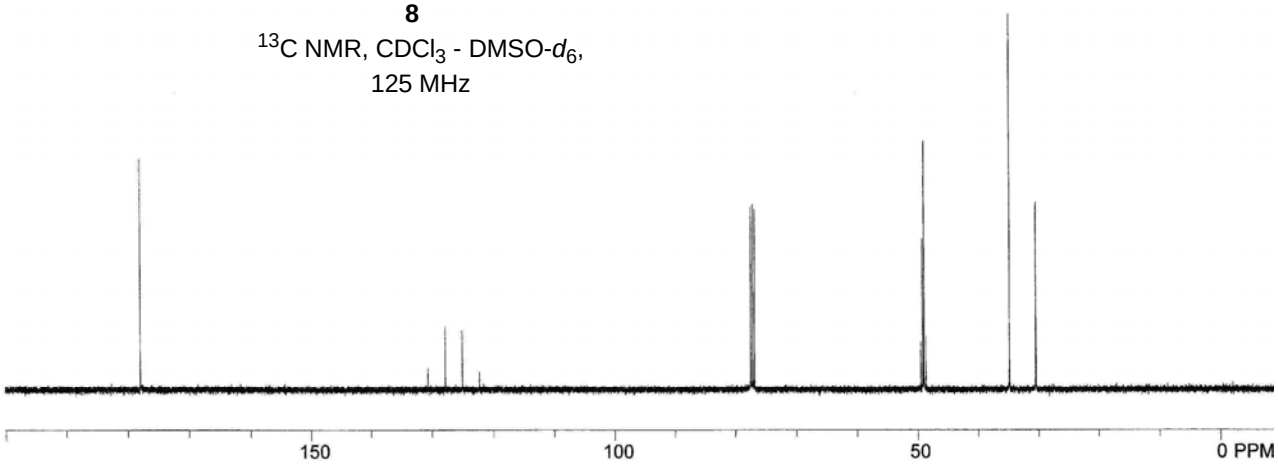
^{1}H NMR, $CDCl_3$ - DMSO-d_6,
400 MHz

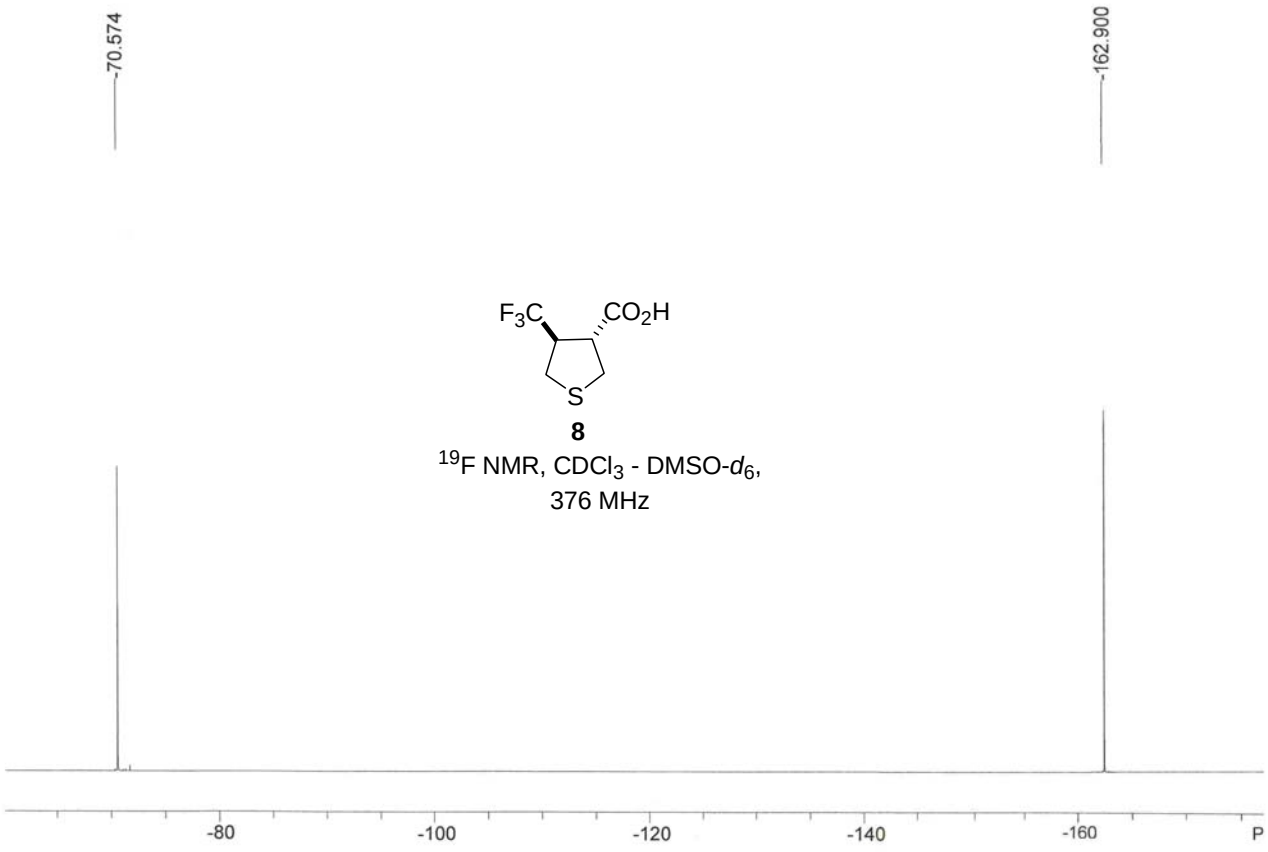




8

^{13}C NMR, $CDCl_3$ - DMSO-d_6,
125 MHz



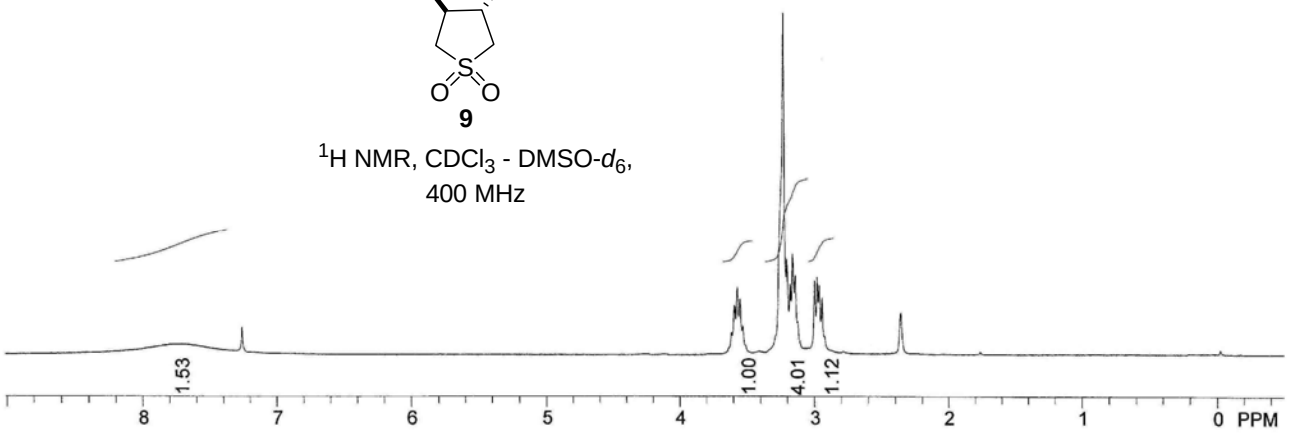


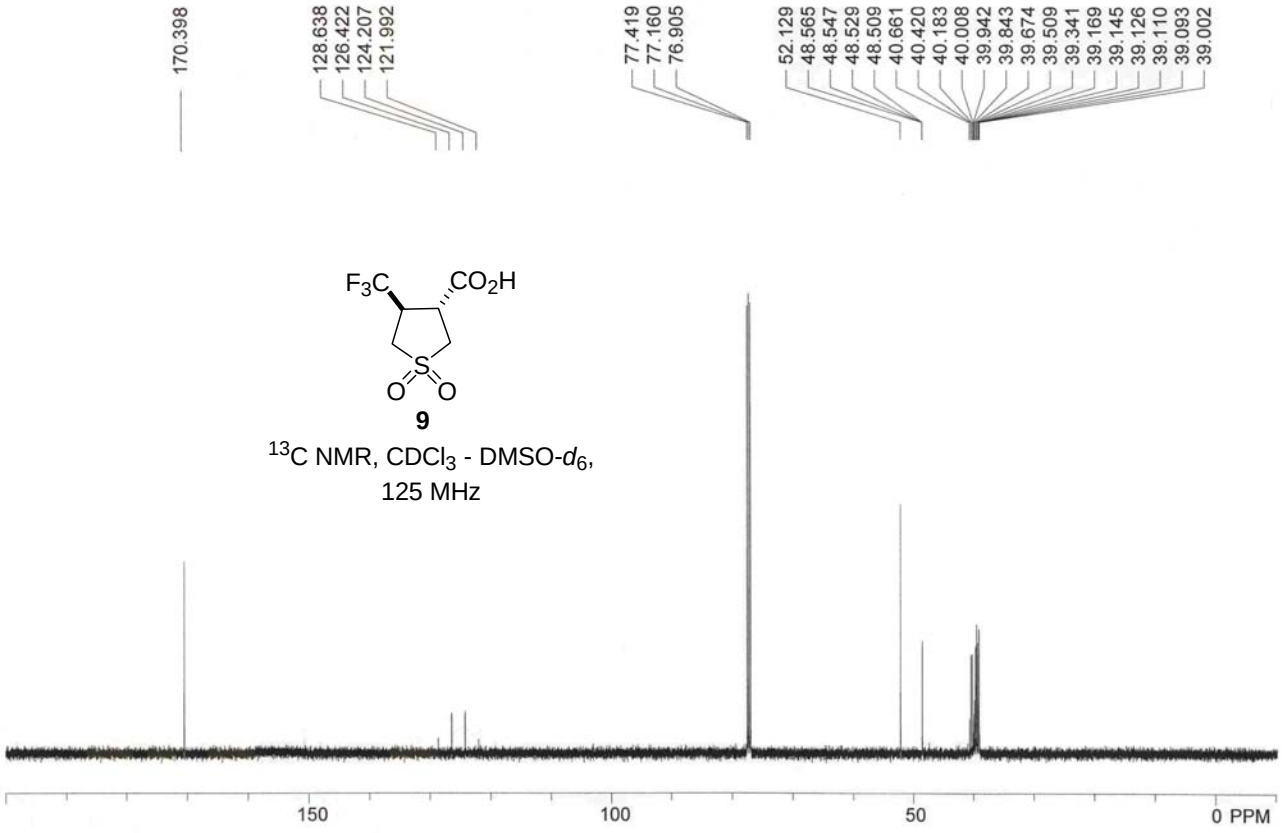
8

^{19}F NMR, $CDCl_3$ - DMSO-d_6,
376 MHz

9

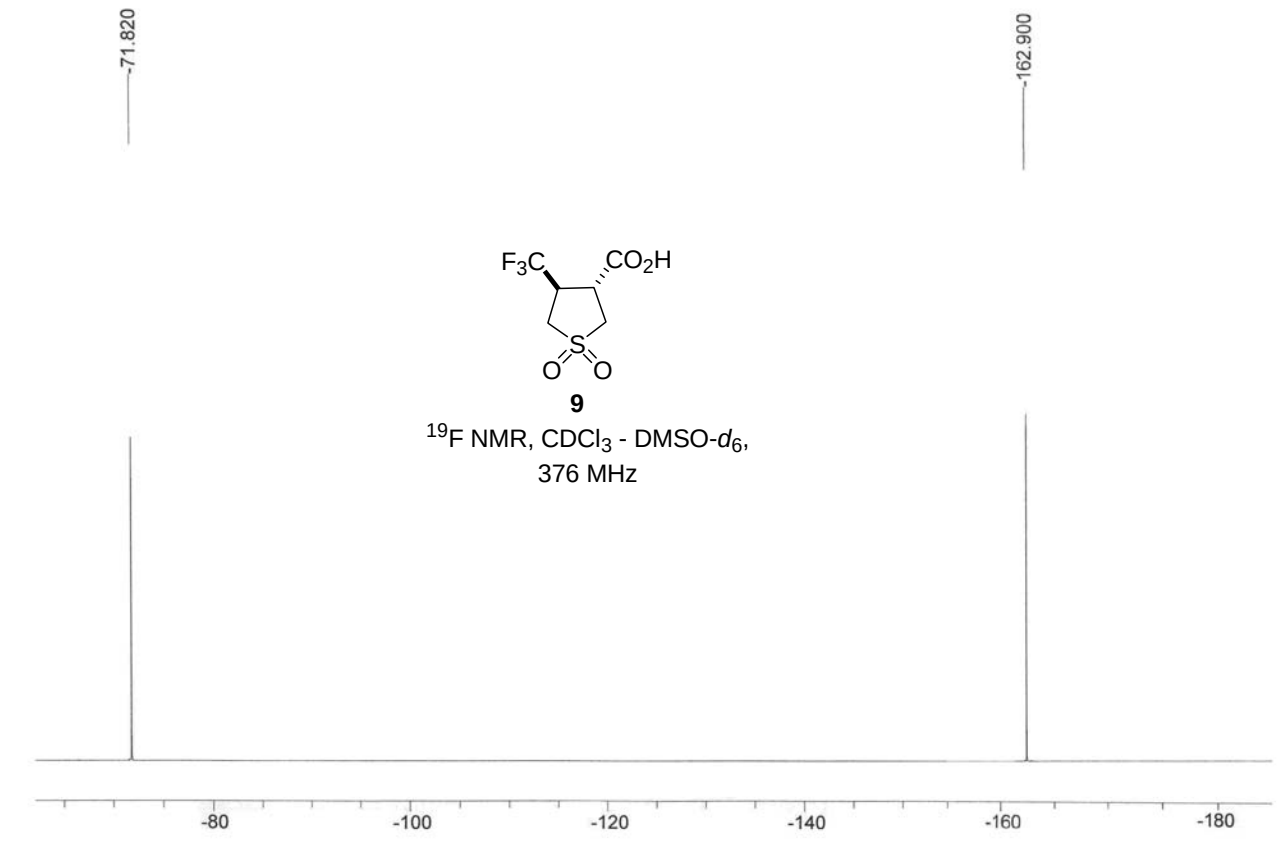
^{1}H NMR, $CDCl_3$ - DMSO-d_6,
400 MHz





9

^{13}C NMR, $CDCl_3$ - DMSO-d_6,
125 MHz



9

^{19}F NMR, $CDCl_3$ - DMSO-d_6,
376 MHz