Supporting Information for Polyfluorination of Aryl Alkyl Sulfides by IF₅ with Concomitant Migration of the Arylthio Group

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IF₅-CH₂Cl₂: As IF₅ is highly hygroscopic, it was used as a solution of CH₂Cl₂. From a cylinder, IF₅ was transferred through a Teflon™ tube into a bottle made of Teflon™ PFA under an N₂ atmosphere. After measuring the amount of IF₅ in the bottle, a 5 molar amount of CH₂Cl₂ was added at room temperature. It can be used in the open air and kept in a Teflon™ PFA bottle for a few months without any change. However, it should be carefully handled with equipment made of Teflon™ in a bench hood.

General Procedure for the Fluorination of Sulfides 1 with IF₅: IF₅ in CH₂Cl₂ (0.776-2.328 g, 1.2-3.6 mmol), hexane (4 mL), and sulfide 1 (1 mmol) were introduced into a reaction vessel made of Teflon™ PFA with a tight screw cap and stirred at 40 °C for 6-96 h. After consumption of the starting material was confirmed by GC, the reaction mixture was poured into ice-water and the product was extracted three times with ether. The combined ethereal phases were washed with aqueous Na₂S₂O₃, NaHCO₃, and brine and dried over MgSO₄. After concentration under reduced pressure, the product was isolated by column chromatography (silica gel / hexane-ether as eluent).

Characterization of polyfluorosulfides 2.

p-Chlorophenyl 1,2,2-trifluoroethyl sulfide 2a: liquid; IR (liquid film) 1571, 1477, 1378, 1151, 1095, 1013, 826 cm⁻¹; ¹H NMR (400 MHz, CDCl₃) δ 5.67 (ddt, *J* = 25.2, 9.0, 3.9 Hz, 1H), 5.80 (tt, *J* = 54.8, 3.7 Hz, 1H), 7.36 (d, *J* = 8.1 Hz, 2H), 7.51 (d, *J* = 8.1 Hz, 2H); ¹⁹F NMR (376 MHz, CDCl₃) δ -168.00 (ddd, *J* = 50.7, 19.2, 3.7 Hz, 1F), -127.22 (dddd, *J* = 293.0, 54.9, 18.9, 9.8 Hz, 1F), -126.44 (dddd, *J* = 292.3, 54.3, 23.8, 9.2 Hz, 1F); ¹³C NMR (100 MHz, CDCl₃) δ 97.63 (dt, *J* = 226.6, 28.1 Hz), 111.48 (dt, *J* = 248.9, 34.7 Hz), 127.62 (s), 129.70 (s), 135.32 (s), 136.06 (s); MS 228 (M⁺+2, 37), 226 (M⁺, 100), 177 (33), 175 (90), 143 (47), 108(29); HRMS (EI) Calculated for C₈H₆ClF₃S : (M⁺) 225.9831. Found : 225.9829.

***p*-Chlorophenyl 1,2,2,3,3-pentafluoropropyl sulfide 2b:** liquid; IR (liquid film) 2988, 1776, 1575, 1478, 1450, 1391, 1375, 1317, 1215, 1173, 1139, 1096, 1013, 829, 744 cm^{-1} ; ^1H NMR (400 MHz, CDCl_3) δ 5.87 (ddd, $J = 66.8, 16.1, 6.1$ Hz, 1H), 6.02 (dt, $J = 52.7, 6.4$ Hz, 1H), 7.37 (d, $J = 8.5$ Hz, 2H), 7.52 (d, $J = 8.5$ Hz, 2H); ^{19}F NMR (376 MHz, CDCl_3) δ -167.19 (ddt, $J = 50.7, 18.9, 9.2$ Hz, 1F), -140.65 (dddd, $J = 305.2, 53.1, 8.9, 5.8$ Hz, 1F), -137.08 (ddt, $J = 304.9, 52.8, 9.5$ Hz, 1F), -128.50 (dm, $J = 276.5$ Hz, 1F), -125.77 (dm, $J = 276.5$ Hz, 1F); ^{13}C NMR (100 MHz, CDCl_3) δ 98.60 (ddd, $J = 227.4, 33.1, 25.6$ Hz), 108.70 (tm, $J = 251.5$ Hz), 112.74 (tm, $J = 255.4$ Hz), 128.35 (s), 129.79 (s), 134.81 (s), 136.18 (s); MS 278 ($\text{M}^+ + 2$, 37), 276 (M^+ , 100), 143 (48), 108 (27); HRMS (EI) Calculated for $\text{C}_9\text{H}_6\text{ClF}_5\text{S}$: (M^+) 275.9799. Found: 275.9797.

***p*-Chlorophenyl 1,2,2,3,3,4,4-heptafluorobutyl sulfide 2c:** liquid; IR (liquid film) 2971, 1478, 1212, 1154, 1096, 1014, 932 cm^{-1} ; ^1H NMR (400 MHz, CDCl_3) δ 5.94 (ddd, $J = 50.3, 17.1, 4.6$ Hz, 1H), 6.04 (tt, $J = 52.1, 5.4$ Hz, 1H), 7.38 (d, $J = 8.5$ Hz, 2H), 7.53 (d, $J = 8.5$ Hz, 2H); ^{19}F NMR (376 MHz, CDCl_3) δ -166.17 - -165.86 (m, 1F), -137.59 (dm, $J = 55.6$ Hz, 2F), -130.43 (dd, $J = 504.9, 303.8$ Hz, 2F), -125.17 (dm, $J = 306.0$ Hz, 1F), -117.29 (dm, $J = 306.0$ Hz, 1F); ^{13}C NMR (100 MHz, CDCl_3) δ 97.97 (ddd, $J = 229.9, 33.9, 24.8$ Hz), 107.86 (tt, $J = 253.9, 31.4$ Hz), 110.35 (tm, $J = 262.5$ Hz), 111.60 (dq, $J = 256.4, 30.6$ Hz), 114.22 (dq, $J = 256.4, 30.6$ Hz), 127.93 (s), 129.82 (s), 135.35 (s), 136.40 (s); MS 328 ($\text{M}^+ + 2$, 37), 326 (M^+ , 100), 175 (92), 143 (49), 108 (24); HRMS (EI) Calculated for $\text{C}_{10}\text{H}_6\text{F}_7\text{ClS}$: (M^+) 325.9767. Found: 325.9766.

***p*-Chlorophenyl 1,2,2-trifluoropropyl sulfide 2d:** liquid; IR (liquid film) 2954, 1478, 1392, 1133, 1096, 1013, 939, 826 cm^{-1} ; ^1H NMR (400 MHz, CDCl_3) δ 1.77 (dt, $J = 18.4, 1.8$ Hz, 3H), 5.68 (ddd, $J = 26.4, 9.3, 7.8$ Hz, 1H), 7.35 (d, $J = 8.4$ Hz, 2H), 7.50 (d, $J = 8.4$ Hz, 2H); ^{19}F NMR (376 MHz, CDCl_3) δ -161.67 (ddt, $J = 52.8, 17.1, 1.8$ Hz, 1F), -101.53 (dm, $J = 254.5$ Hz, 1F), -98.10 (dm, $J = 254.5$ Hz, 1F); ^{13}C NMR (100 MHz, CDCl_3) δ 19.74 (t, $J = 25.6$ Hz), 101.53 (dt, $J = 228.3, 33.9$ Hz), 119.80 (dt, $J = 224.8, 28.1$ Hz), 129.51 (s), 129.59 (s), 134.38 (s), 135.43 (s); MS 242 ($\text{M}^+ + 2$, 22), 240 (M^+ , 65), 177 (32), 175 (100), 143 (20), 108 (22), 75 (10); HRMS (EI) Calculated for $\text{C}_9\text{H}_8\text{ClF}_3\text{S}$: (M^+) 239.9987. Found: 239.9977.

Methyl 3-(*p*-chlorophenylthio)-2,2,3-trifluoropropanate 2e: liquid; IR (liquid film) 2961, 1785, 1575, 1478, 1442, 1391, 1312, 1220, 1096, 1074, 1014 cm^{-1} ; ^1H NMR (400 MHz, CDCl_3) δ 3.95 (s, 3H), 6.00 (ddd, $J = 50.0, 12.2, 9.3$ Hz, 1H), 7.36 (d, $J = 8.5$ Hz, 2H), 7.49 (d, $J = 8.5$ Hz, 2H); ^{19}F NMR (376 MHz, CDCl_3) δ -167.38 (ddd, $J = 50.0, 22.0, 20.1$ Hz, 1F), -115.63 (ddd, $J = 268.6, 20.1, 12.2$ Hz, 1F), -114.27 (ddd, $J = 268.6, 22.0, 9.3$ Hz, 1F); ^{13}C NMR (100 MHz, CDCl_3) δ 53.91 (s), 99.23 (ddd, $J = 230.5, 29.7, 27.3$

Hz), 111.13 (dt, J = 259.3, 30.6 Hz), 128.47 (s), 129.61 (s), 134.74 (s), 135.85 (s), 161.78 (t, J = 31.0 Hz); MS 286 (M^+ +2, 23), 284 (M^+ , 57), 177 (37), 175 (100), 145 (20), 143 (53), 108 (46); HRMS (EI) Calculated for $C_{10}H_8F_3O_2ClS$: (M^+) 283.9885. Found: 283.9880.

Ethyl 4-(*p*-chlorophenylthio)-2,2,3,3,4-pentafluorobutanoate 2f: liquid; IR (liquid film) 2987, 1776, 1478, 1375, 1317, 1215, 1173, 1139, 1096, 1013, 829, 744 cm^{-1} ; 1H NMR (400 MHz, $CDCl_3$) δ 1.38 (t, J = 7.1 Hz, 3H), 4.41 (q, J = 7.1 Hz, 2H), 5.98 (ddd, J = 50.3, 16.6, 5.1 Hz, 1H), 7.37 (d, J = 8.7 Hz, 2H), 7.52 (d, J = 8.7 Hz, 2H); ^{19}F NMR (376 MHz, $CDCl_3$) δ -165.48 - -165.19 (m, 1F), -124.84 (dt, J = 279.5, 17.1 Hz, 1F), -119.87 (dd, J = 7.3, 3.7 Hz, 1F), -119.76 (d, J = 8.5 Hz, 1F), -117.42 (ddm, J = 279.8, 21.2 Hz, 1F); ^{13}C NMR (100 MHz, $CDCl_3$) δ 13.73 (s), 64.25 (s), 98.10 (ddd, J = 229.9, 33.9, 24.8 Hz), 108.49 (tt, J = 265.1, 33.1 Hz), 111.39 (dq, J = 257.2, 30.6 Hz), 114.02 (dq, J = 257.2, 30.6 Hz), 128.15 (s), 129.75 (s), 135.21 (s), 136.21 (s), 159.51 (t, J = 29.8 Hz); MS 350 (M^+ +2, 38), 348 (M^+ , 100), 328 (24), 255 (27), 177 (78), 175 (66), 143 (34), 108 (29); HRMS (EI) Calculated for $C_{12}H_{10}F_5O_2ClS$: (M^+) 348.0010. Found: 348.0006.

Ethyl 2-(*p*-chlorophenylthio)-2,3,3-trifluoropropanate 2g: liquid; IR (neat) 2987, 1768, 1744, 1477, 1256, 1171, 1094, 1039 cm^{-1} ; 1H NMR (400 MHz, $CDCl_3$) δ 1.11 (t, J = 7.2 Hz, 3H), 3.97-4.11 (m, 2H), 6.20 (dt, J = 53.5, 9.8 Hz, 1H), 7.36 (d, J = 8.4 Hz, 2H), 7.52 (d, J = 8.4 Hz, 2H); ^{19}F NMR (376 MHz, $CDCl_3$) δ -156.52 (dt, J = 19.5, 9.8 Hz, 1F), -133.60 (ddd, J = 285.0, 53.7, 19.5 Hz, 1F), -124.87 (ddd, J = 285.0, 53.7, 19.5 Hz, 1F); ^{13}C NMR (100 MHz, $CDCl_3$) δ 13.59 (s), 63.20 (s), 100.72 (dt, J = 248.1, 22.7 Hz), 112.04 (dt, J = 253.1, 24.0 Hz), 124.19 (s), 129.46 (s), 137.30 (s), 137.55 (s), 162.65 (dd, J = 28.5, 5.8 Hz); MS 300 (M^+ +2, 85), 298 (M^+ , 100), 227 (78), 226 (42), 225 (95), 206 (46), 205 (37), 170 (44), 143 (74), 108 (60), 99 (17), 75 (33); HRMS (EI) Calculated for $C_{11}H_{10}O_2ClF_3S$: (M^+) 298.0042. Found: 298.0015.

***N,N*-Dimethyl 2-(*p*-chlorophenylthio)-2,2,3-trifluoropropanamide 2h:** white solid; mp 37-39 °C; IR (KBr) 3056, 2942, 1651, 1476, 1402, 1153, 1085, 1015, 926, 825 cm^{-1} ; 1H NMR (400 MHz, $CDCl_3$) δ 2.81 (s, 3H), 2.85 (d, J = 7.1 Hz, 3H), 6.43 (ddd, J = 54.6, 53.4, 11.0 Hz, 1H), 7.37 (d, J = 8.3 Hz, 2H), 7.56 (d, J = 8.3 Hz, 2H); ^{19}F NMR (376 MHz, $CDCl_3$) δ -152.70 - -152.54 (m, 1F), -135.99 (ddd, J = 223.4, 54.9, 17.7 Hz, 1F), -125.32 (ddd, J = 225.2, 53.1, 20.1 Hz, 1F); ^{13}C NMR (100 MHz, $CDCl_3$) δ 37.12 (d, J = 15.7 Hz), 37.30 (s), 103.29 (dt, J = 256.4, 22.3 Hz), 113.48 (ddd, J = 254.4, 249.8, 24.0 Hz), 124.69 (s), 129.23 (s), 137.29 (s), 138.14 (s), 162.24 (dd, J = 20.7, 4.1 Hz); MS 299 (M^+ +2, 10), 297 (M^+ , 27), 72 (100); HRMS (EI) Calculated for $C_{11}H_{11}F_3ONClS$: (M^+) 297.0202. Found: 297.0202.

OFFSET CHECK

Date : Sat Dec 29 03 01:43 2001

Filename : Load:refid.madeata
Comment : OFFSET CHECK

Slit:history
EXMODE : nmr

POINT : 32768 points
SAMPD : 32768 points
FREQ : 7993.6 Hz

FILTR : 4000 Hz

DELT : 50.0 usec

DEAD : 72.3 usec

INVL : 125.1 usec

TIME : 16 times

NUMY : 0 times

PD : 2.9007 sec

ACQIM : 4099.2769 msec

PREC : 10.00000 msec

INIM : 10.0000 msec

RESOL : 0.24 Hz

PM : 5.40 usec

DBNUC : 399.65 MHz

DBFRO : 134300.00 Hz

DBSET : 22

RGAIN : 16 times

SCANS : 16 times

SLVNT : CDCl3

SPINNING : 11 Hz

TEMP : 25.4 C

¹H-NMR
2a
p-ClPhSCH₂CH₂F₂

OFFSET CHECK

Date : Sat Dec 29 22:01:04 2001
 File Name : Load:mgf10.nmdata
 Comment : OFFSET CHECK
 Slice History :
 EXMODE : non

COIN1 : 131072 points
 SAMPD : 131072 points
 FREQD : 80000.0 Hz
 FILLR : 40000 Hz
 DELAY : 5.0 usec
 DEADT : 10.0 usec
 INI.VL : 12.5 usec
 TIMES : 16 times
 DUMMY : 0 times
 PD : 2.9607 sec
 ACQTM : 1638.399 msec
 PRECI : 10.0000 msec
 INI.W : 10.0000 msec
 RESUL : 0.61 Hz
 PH1 : 5.40 usec
 DBNUC : 19F
 OBSFQ : 376.00 MHz
 OBSCT : 110000.00 Hz
 RGAIN : 13

SCANS : 16 times

SLVNT : CDCl3
 SPINNING : 12 Hz
 TEMP : 24.7 C

19F-NMR
 2a

p-CIPhSCH₂CH₂F₂

OFFSET CHECK

0.0000
-0.0024

Date : Nov 21 03 23 28 2003

Filename : LoadingFile.mnista
 Comment : OFFSET CHECK
 Slice: 1 of 1
 EPMODE : none

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 SAMPLE : 32768 points
 FREQ : 7993.6 Hz
 FILTH : 4000 Hz
 DELAY : 50.0 usec
 DEAD1 : 72.3 usec
 INVL : 125.1 usec
 TIMES : 16 times
 DUMMY : 0 times
 PU : 2.9007 sec
 ACQIM : 4099.2769 msec
 PRECL : 10.00000 msec
 INIWI : 10.0000 msec
 RESOL : 0.24 Hz
 PWT : 5.40 usec
 OBNUL : 1H
 OBRFO : 399.65 MHz
 OBSET : 134500.00 Hz
 RGAIN : 21

SCANS : 16 times

SOLVENT : CDCl3

SPINNING : 13 Hz

TEMP : 23.6 C

1H-NMR

2b

p-CIPhSCHFCF₂CHF₂

19F-NMR
2b
p-CIPhSCHFCF2CHF2

Date : Wed Jan 16 05:06:12 2002

File Name	Load Info	Offset Check
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FREQ: 400000 Hz	800000 Hz	
DELTA: 5.00 usec	10.00 usec	
DEAD: 12.5 usec	16.00 usec	
INITIAL: 0 times	0 times	
PD: 2.9007 sec	1638.3959 msec	
ACQ: 10.00000 msec	10.0000 msec	
PRD: 10.0000 msec	0.61 Hz	
RES: 0.61 Hz	5.40 usec	
PH: 19F	376.00 MHz	
OB: 110000.00 Hz	13	
RGAIN: 13	16 times	
SCANS: 16 times	13 Hz	
SLV: 13 Hz	23.3 C	
TEMP: 23.3 C		

OFFSET CHECK

7.5493
7.5426
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7.5268
7.5213
7.5152
7.3962
7.3825
7.3646
7.3730
7.3681
7.3614
7.2632
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6.1715
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6.0550
6.0415
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Date : Sat Jun 22 04:30:29 2002
Filename : I034912f01.ms
Comment : OFFSET CHECK
S1:CH3SLV1
EXMODE : none

POINT : 32768 points
SAMPD : 32768 points
FREQ : 799.36 MHz
FILT : 4000 Hz
DELTA : 50.0 usec
DELT : 72.3 usec
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TIMES : 16 times
DUMY : 0 times
PD : 2.9007 sec
ACQ : 4099.2769 msec
PROC : 10.0000 msec
INJ : 10.0000 msec
RES : 0.24 Hz
PR1 : 5.40 usec
ORAC : 399.65 MHz
ORFO : 134500.00 Hz
RGAIN : 26

SCANS : 16 times
SLV : CDCl3
SPIN : 11 Hz
TEMP : 21.4 C

¹H-NMR
2c
p-CIPhSCHF(CF₂)₂CHF₂

p-CIPhSCHF(CF₂)₂CHF₂

19F-NMR
2c

Date: Sat Jun 22 04:37:06 2002

File Name: 131072 points
Comment: Offset Check
S1: 10000.00 Hz
F2: 10000.00 Hz
F1: 10000.00 Hz
F0: 10000.00 Hz
F-1: 10000.00 Hz
F-2: 10000.00 Hz
F-3: 10000.00 Hz
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F-96: 10000.00 Hz
F-97: 10000.00 Hz
F-98: 10000.00 Hz
F-99: 10000.00 Hz
F-100: 10000.00 Hz

2d

OFFSET CHECK

0.0000

Date: Sat Feb 22 10:55:12 2003

Filename: LoadingID.mwdata
 Comment: OFFSET CHECK
 SliceHistory: none

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FREQU	79913.6 Hz
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DEAD	72.3 usec
TRVL	125.1 usec
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DUMMY	0 times
PD	2.9007 sec
ACQIM	4099.2769 msec
PRCL	10.00000 msec
INMT	10.0000 msec
RESOL	0.24 Hz
PHI	5.40 usec
OBNUC	¹ H 399.65 MHz
ORFID	134500.00 Hz
DBSET	23
RGAIN	16 times
SCANS	
SOLVT	CDCL3
SPINNING	14 Hz
TEMP	25.0 C

OFFSET CHECK

Date : Sat Feb 22 10:49:55 2003
 Filename : LoadingFile.amca
 Comment : OFFSET CHECK
 Solvent :
 EXMODE :
 num :

POINTS : 131072
 SAMPD : 131072 points
 FREQ : 400.00 MHz
 F1 F2 :
 DELAY : 5.00 usec
 DEAD : 10.00 usec
 INTR : 12.50 usec
 TIMES : 16 times
 DUMPR : 0 times
 PD : 2.9007 sec
 ACQIM : 1639.3999 msec
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 INTR : 10.0000 msec
 RESOL : 0.61 Hz
 PWT : 5.40 usec
 OBRAC :
 DBFBD :
 DBSET : 110000.00 Hz
 RGAIN : 13

SCANS : 16 times
 SOLVNT : CDCl3
 SPINNING : 13 Hz
 TEMP : 24.9 C

OFFSET CHECK

Date : Sat Feb 22 10:40:23 2003

File Name : LoadingID.mdata

Comment : OFFSET CHECK

Slit (cm⁻¹)

num

POINT	SAMP	TIME	PROG	FILE	IR	DELT	INVL	TIME	PU	ACQ	PREL	INVL	RESOL	PHI	ORNUC	ORFRO	OBSET	RGAIN	SCANS	SOLVT	SPINNING	TEMP	CDL3																			
13072	points	13072	points	80000	0	Hz	5.0	usec	30.0	usec	12.5	usec	16	times	2.0007	sec	1638	3909	msc	10.00000	msc	10.00000	msc	0.61	Hz	5.40	usec	19F	376.00	MHz	110000	00	Hz	13	16	times	CDL3	0	Hz	24.8	C	

19F-NMR

2f

OFFSET CHECK

Date: Tue Jul 30 04:26:51 2002

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SCANS	16 times	
ORBIT	399.65 MHz	
ORBIT	134500.00 Hz	
RGAIN	23	
ACQIN	2.9002 sec	
PHASE	0.0000 msec	
INSTR	10.0000 msec	
RESOL	0.24 Hz	
PR1	5.40 usec	
ORBIT	399.65 MHz	
ORBIT	134500.00 Hz	
RGAIN	23	
SCANS	16 times	
SLVNT	CDCl3	
SPINNING	11 Hz	
TEMP	22.4 C

OFFSET CHECK

Date : Tue Jul 30 04:21:06 2002

Filename : Loading10.amdata
 Comment : OFFSET CHECK
 Site history :
 EXMODE : run

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 F2 : 101.625 MHz
 F1 : 101.625 MHz
 DELTA : 5.00 usec
 DECT : 10.0 usec
 INTR : 12.5 usec
 TIMES : 16 times
 DUMY : 0 times
 PD : 2.000 sec
 ACQ : 1638.399 msec
 PREP : 10.0000 msec
 INTR : 10.0000 msec
 RESOL : 0.61 Hz
 PM1 : 5.40 usec
 OBMC : 19F
 OBFO : 376.00 MHz
 OBSE : 110000.00 Hz
 RGAIN : 13

SCANS : 16 times
 SOLVENT : CDCl3
 SPINNING : 11 Hz
 TEMP : 22.9 C

$$\begin{array}{r} 2.8606 \\ - 2.8484 \\ \hline 2.8093 \end{array}$$

0 0000

```
Filename      : LoadingID.mdata
Comment       : OFFSET CHECK
StackHistory  :
EXMODE        : non
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ကုမ္ပဏီ

327bb points

1993. b HZ
1990 HZ

50 G user
723 user

125.1 user

0 times
10 times

2.9107 sec
4099 2769 msec

10.00000 msec

0.24 Hz

540 usec
1H

399.65 MHz
134500.00 Hz

21

16 times

Figure 3

12 Hz
75.4 °C

2016

1000

¹H-NMR

2h

•

CONMe₂

1

67

1H-NMR
2h

OFFSET CHECK

Date : Tue Jan 15 11:13:11 2002

Filename : Loading ID, no date

Comment : OFFSET CHECK

Spectrum history

EXMODE

run

POINT 131072 points
 SAMPLE 131072 points
 FREQ 80000.0 Hz
 H1 1H
 DELAY 5.00 usec
 DEAD 10.00 usec
 INIT 12.50 usec
 TIMES 16 times
 DUMY 0 times
 PU 2.9007 sec
 ACQ 1638.3999 msec
 PRE 10.0000 msec
 INI 10.0000 msec
 RES 0.61 Hz
 PW 5.40 usec
 19F 376.00 MHz
 OBS 110000.00 Hz
 DB 13
 RGAIN 16 times
 SCANS 16 times
 SLYNT CDCL3 12 Hz
 SPINNING 22.1 C
 TEMP

19F-NMR

2h