Aziridines *versus* Vinyl Carbamates from the Direct Amination of Electron Withdrawing Group-Substituted Trifluoromethyl Enoates

*Stefania Fioravanti, * Daniele Colantoni, Lucio Pellacani, * and Paolo A. Tardella**

Dipartimento di Chimica, Università degli Studi "La Sapienza", P.le Aldo Moro 2, I-00185 Roma, Italy

lucio.pellacani@uniroma1.it. Fax: +39 06 490631. Phone: +39 06 49913673

Table of Contents

General	S2
Spectroscopic data for substrates 2 and 3	S2
Spectroscopic data for aziridines 4b-d	S2
Spectroscopic data for aziridines 5b-d	S3
Spectroscopic data for aziridines 6b-d	S4
Spectroscopic data for vinyl carbamates 7c-d and 8d	S4
Spectroscopic data for vinyl carbamates 11b-d	S4
¹ H NMR spectrum of compound 1	S6
¹ H NMR spectrum of compound 2	S7
¹³ C NMR spectrum of compound 3	S8
¹ H NMR spectrum of compound 4a	S9
¹³ C NMR spectrum of compound 4b	S10
¹ H NMR spectrum of compound 4c	S11
¹³ C NMR spectrum of compound 4d	S12
¹ H NMR spectrum of compound 5a	S13
¹ H NMR spectrum of compound 5b	S14
¹ H NMR spectrum of compound 5c	S15
¹ H NMR spectrum of compound 5d	S16
¹ H NMR spectrum of compound 6a	S17
¹ H NMR spectrum of compound 6b	S18
¹ H NMR spectrum of compound 6c	S19
¹ H NMR spectrum of compound 6d	S20
¹ H NMR spectrum of compound 7a	S21
¹³ C NMR spectrum of compound 7a	S22
¹⁹ F NMR spectrum of compound 7c	S23
¹⁹ F NMR spectrum of compound 7d	S24
¹⁹ F NMR spectrum of compound 8a	S25
¹⁹ F NMR spectrum of compound 8d	S26
¹³ C NMR spectrum of compound 11a	S27
¹ H NMR spectrum of compound 11b	S28
¹³ C NMR spectrum of compound 11c	S29
¹ H NMR spectrum of compound 11d	S30

General. GC analyses were performed on a capillary column (methyl silicone, 12.5 m $\times$ 0.2 mm). GC MS were performed using a quadrupole mass spectrometer on a capillary column (phenyl methyl silicone, 30 m $\times$ 0.25 mm). IR spectra were recorded on a FTIR spectrophotometer in CHCl₃ as the solvent. ¹H NMR and ¹³C NMR spectra were recorded at 200 or 300 and 50 or 75 MHz, respectively. CDCl₃ was used as the solvent and CHCl₃ as the internal standard. ¹⁹F NMR spectra were recorded at 282.2 MHz, using CDCl₃ as the solvent and C₆F₆ as the internal standard; chemical shifts are given in ppm relative to CFC1₃. ESI MS analyses were performed using a quadrupole-time of flight (TOF) mass spectrometer equipped with an ESI source and a syringe pump. Melting points were determined with a Büchi apparatus and are uncorrected. Analytical thin-layer chromatography (TLC) was carried out on precoated (0.25 mm) silica gel plates using a mixture of EtOAc/hexane 20/80 as eluent.

The experiments were conducted in the positive ion mode. NaH (55-65% suspension in mineral oil) was washed twice with pentane and dried under nitrogen. Anhydrous THF and toluene were used as such.

2: pale yellow liquid; IR: 1735, 1662 cm⁻¹; ¹H NMR: δ 1.35 (t, J = 7.2 Hz, 3 H), 1.37 (t, J = 7.2 Hz, 3 H), 4.31 (q, J = 7.2 Hz, 2 H), 4.37 (q, J = 7.2 Hz, 2 H), 6.69 (q, J = 1.2 Hz, 1 H); ¹³C NMR: δ 13.1, 14.6, 62.0, 62.6, 120.7 (q, J = 273.3 Hz), 129.7 (q, J = 5.4 Hz), 133.3 (q, J = 32.9 Hz), 160.8, 162.9; ¹⁹F NMR: δ -67.13; GC MS m/z 240 (M⁺, 0.05), 195 (18), 168 (13), 167 (100), 150 (10), 148 (14), 147 (16), 103 (12), 75 (14), 53 (11); HRMS-ESI (m/z) [M+Na]⁺ calcd for C₉H₁₁F₃O₄: 263.0507; found: 263.0514.

3: pale yellow liquid; IR: 2236 (vw), 1741, 1644 cm⁻¹; ¹H NMR: δ 1.41 (t, J = 7.2 Hz, 3 H), 4.43 (q, J = 7.2 Hz, 2 H), 6.49 (q, J = 1.5 Hz, 1 H); ¹³C NMR: δ 13.5, 63.5, 112.4, 113.0 (q, J = 6.6 Hz), 120.1 (q, J = 274.4 Hz), 139.3 (q, J = 32.6 Hz), 158.4; ¹⁹F NMR: δ -67.10; GC MS m/z 193 (M⁺, 0.05), 166 (11), 165 (13), 148 (100), 121 (11), 69 (29); HRMS-ESI (m/z) [M+Na]⁺ calcd for C₇H₆F₃NO₂: 216.0248; found: 216.0242.

4b: yellow oil, R_f : 0.49; IR: 1747 cm^{-1} ; ^1H NMR: δ 1.27 (t, $J = 7.2$ Hz, 3 H), 1.46 (s, 9 H), 2.32 (s, 3 H), 3.47 (s, 1 H), 4.22-4.30 (m, 2 H); ^{13}C NMR: δ 13.8, 27.6, 28.3, 46.1, 48.8 (q, $J = 34.9$ Hz), 63.4, 84.3, 121.3 (q, $J = 279.9$ Hz), 154.7, 161.2, 198.8; ^{19}F NMR: δ -68.85; GC MS m/z 325 (M^+ , 1), 152 (100), 57 (92), 43 (10), 41 (15); HRMS-ESI (m/z) [$\text{M}+\text{Na}$] $^+$ calcd for $\text{C}_{13}\text{H}_{18}\text{F}_3\text{NO}_5$: 348.1035; found: 348.1031.

4c: pale yellow oil, R_f : 0.39; IR: 1750 cm^{-1} ; ^1H NMR: δ 1.27 (t, $J = 7.2$ Hz, 3 H), 2.34 (s, 3 H), 3.56 (s, 1 H), 4.20-4.29 (m, 2 H); 5.18-5.19 (m, 2 H), 7.36 (s, 5 H); ^{13}C NMR: δ 13.7, 28.4, 46.1, 48.7 (q, $J = 35.7$ Hz), 63.5, 69.6, 121.0 (q, $J = 279.6$ Hz), 128.4, 128.5, 128.6, 134.1, 156.1, 160.8, 198.0; ^{19}F NMR: δ -69.31; GC MS m/z 359 (M^+ , 1), 91 (100); HRMS-ESI (m/z) [$\text{M}+\text{Na}$] $^+$ calcd for $\text{C}_{16}\text{H}_{16}\text{F}_3\text{NO}_5$: 382.0878; found: 382.0869.

4d: white low-melting solid; IR: 1751 cm^{-1} ; ^1H NMR: δ 1.35 (t, $J = 7.2$ Hz, 3 H), 2.33 (s, 3 H), 3.52 (s, 1 H), 4.23-4.30 (m, 1 H), 4.34 (q, $J = 7.2$ Hz, 2 H), 4.51-4.55 (m, 2 H), 7.30-7.44 (m, 4 H), 7.58-7.65 (m, 2 H), 7.75-7.78 (m, 2 H); ^{13}C NMR: δ 13.8, 28.4, 45.9, 46.6, 48.9 (q, $J = 35.8$ Hz), 63.6, 69.6, 119.9, 121.0 (q, $J = 279.9$ Hz), 124.8, 124.9, 127.1, 127.8, 141.1, 142.7, 143.0, 156.0, 160.8, 197.9; ^{19}F NMR: δ -69.40; HRMS-ESI (m/z) [$\text{M}+\text{Na}$] $^+$ calcd for $\text{C}_{23}\text{H}_{20}\text{F}_3\text{NO}_5$: 470.1191; found: 470.1179.

5b: pale yellow oil, R_f : 0.45; IR: 1749 cm^{-1} ; ^1H NMR: δ 1.27 (t, $J = 7.2$ Hz, 3 H), 1.29 (t, $J = 7.2$ Hz, 3 H), 1.45 (s, 9 H), 3.53 (s, 1 H), 4.22 (q, $J = 7.2$ Hz, 2 H), 4.29 (q, $J = 7.2$ Hz, 2 H); ^{13}C NMR: δ 13.9, 14.0, 27.6, 40.9, 48.6 (q, $J = 35.5$ Hz), 62.5, 63.2, 84.2, 121.3 (q, $J = 280.5$ Hz), 154.7, 160.7, 163.8; ^{19}F NMR: δ -69.06; GC MS m/z 340 (M^+ -15, <1), 296 (11), 182 (14), 154 (10), 57 (100), 41 (11); HRMS-ESI (m/z) [$\text{M}+\text{Na}$] $^+$ calcd for $\text{C}_{14}\text{H}_{20}\text{F}_3\text{NO}_6$: 378.1140; found: 378.1133.

5c: yellow oil, R_f : 0.39; IR: 1748 cm^{-1} ; ^1H NMR: δ 1.31 (t, $J = 7.2$ Hz, 3 H), 1.32 (t, $J = 7.2$ Hz, 3 H), 3.63 (s, 1 H), 4.27 (q, $J=7.2$ Hz, 2 H), 4.32 (q, $J=7.2$ Hz, 2 H), 5.21-5.22 (m, 2 H), 7.37 (s, 5 H); ^{13}C NMR: δ 13.7, 13.8, 40.9, 48.5 (q, $J = 36.0$ Hz), 62.6, 63.3, 69.5, 121.1 (q, $J = 280.3$ Hz), 128.3, 128.5,

134.2, 155.8, 160.3, 163.3; ^{19}F NMR: δ -69.66; GC MS m/z 389 (M^+ , 0.01), 210 (60), 182 (30), 107 (46), 91 (100); HRMS-ESI (m/z) [$\text{M}+\text{Na}$] $^+$ calcd for $\text{C}_{17}\text{H}_{18}\text{F}_3\text{NO}_6$: 412.0984; found: 412.0973.

5d: white solid, mp: 66-68 °C (pentane/ CH_2Cl_2); IR: 1748 cm^{-1} ; ^1H NMR: δ 1.35 (t, $J = 7.2$ Hz, 3 H), 1.39 (t, $J = 7.2$ Hz, 3 H), 3.66 (s, 1 H), 4.24-4.33 (m, 3 H), 4.35-4.43 (m, 2 H), 4.45-4.51 (m, 2 H), 7.32-7.45 (m, 4 H), 7.62-7.69 (m, 2 H), 7.77-7.80 (m, 2 H); ^{13}C NMR: δ 13.9, 14.0, 40.8, 46.5, 48.7 (q, $J = 35.8$ Hz), 62.8, 63.4, 69.7, 119.9, 121.1 (q, $J = 280.5$ Hz), 125.0, 125.1, 127.0, 127.8, 141.1, 142.8, 143.0, 155.7, 160.3, 163.4; ^{19}F NMR: δ -69.81; HRMS-ESI (m/z) [$\text{M}+\text{Na}$] $^+$ calcd for $\text{C}_{24}\text{H}_{22}\text{F}_3\text{NO}_6$: 500.1297; found: 500.1287.

6b: pale yellow oil, R_f : 0.55; IR: 2253 (vw), 1758 cm^{-1} ; ^1H NMR: δ 1.38 (t, $J = 7.2$ Hz, 3 H), 1.51 (s, 9 H), 3.49 (s, 1 H), 4.33-4.50 (m, 2 H); ^{13}C NMR: δ 13.9, 27.7, 29.5, 47.0 (q, $J = 36.6$ Hz), 64.7, 85.6, 110.6, 120.2 (q, $J = 278.3$ Hz), 152.0, 159.5; ^{19}F NMR: δ -72.06; GC MS m/z 308 (M^+ , 0.01), 207 (20), 57 (100), 41 (16); HRMS-ESI (m/z) [$\text{M}+\text{Na}$] $^+$ calcd for $\text{C}_{12}\text{H}_{15}\text{F}_3\text{N}_2\text{O}_4$: 331.0882; found: 331.0890.

6c: pale yellow oil, R_f : 0.44; IR: 2254 (vw), 1760 cm^{-1} ; ^1H NMR: δ 1.29 (t, $J = 7.2$ Hz, 3 H), 3.49 (s, 1 H), 4.21-4.41 (m, 2 H), 5.26 (s, 2 H), 7.37 (s, 5 H); ^{13}C NMR: δ 13.8, 29.3, 47.6 (q, $J = 36.7$ Hz), 64.7, 70.2, 110.2, 120.0 (q, $J = 278.3$ Hz), 128.5, 128.6, 128.8, 133.8, 153.7, 159.2; ^{19}F NMR: δ -72.33; GC MS m/z 342 (M^+ , 0.07), 225 (22), 107 (72), 91 (100), 90 (10), 79 (19), 65 (12); HRMS-ESI (m/z) [$\text{M}+\text{Na}$] $^+$ calcd for $\text{C}_{15}\text{H}_{13}\text{F}_3\text{N}_2\text{O}_4$: 365.0725; found: 365.0717.

6d: white solid, mp: 174-176 °C (pentane/ CH_2Cl_2); IR: 2254 (vw), 1761 cm^{-1} ; ^1H NMR: δ 1.32 (t, $J = 7.2$ Hz, 3 H), 3.60 (s, 1 H), 4.27-4.41 (m, 3 H), 4.61-4.67 (m, 2 H), 7.31-7.37 (m, 2 H), 7.41-7.46 (m, 2 H), 7.56-7.61 (m, 2 H), 7.72-7.78 (m, 2 H); ^{13}C NMR: δ 13.8, 29.2, 46.4, 47.1 (q, $J = 37.8$ Hz), 64.9, 70.3, 110.1, 120.0 (q, $J = 278.3$ Hz), 120.1, 124.8, 125.0, 127.1, 127.9, 141.2, 142.5, 142.9, 153.7,

159.3; ^{19}F NMR: δ -72.46; HRMS-ESI (m/z) $[\text{M}+\text{Na}]^+$ calcd for $\text{C}_{22}\text{H}_{17}\text{F}_3\text{N}_2\text{O}_4$: 453.1038; found: 453.1036.

7c: ^{19}F NMR: δ -55.84; GC MS m/z 359 (M^+ , 0.5), 91 (100).

7d: ^{19}F NMR: δ -55.88.

8d: ^{19}F NMR: δ -58.23.

11b: colorless oil, R_f : 0.52; IR: 1752 cm^{-1} ; ^1H NMR: δ 1.28-1.38 (m, 6 H), 1.46 (s, 9 H), 3.70 (q, $J = 5.1\text{ Hz}$, 1 H), 4.27-4.36 (m, 4 H); ^{13}C NMR: δ 13.9, 14.0, 27.8, 44.6 (q, $J = 41.5\text{ Hz}$), 48.5, 62.6, 63.9, 84.1, 121.2 (q, $J = 274.9\text{ Hz}$), 155.1, 162.0, 163.2; ^{19}F NMR: δ -71.92 (d, $J = 4.8\text{ Hz}$); GC MS m/z 340 (M^+ -15, <1), 296 (7), 182 (8), 57 (100), 41 (12); HRMS-ESI (m/z) $[\text{M}+\text{Na}]^+$ calcd for $\text{C}_{14}\text{H}_{20}\text{F}_3\text{NO}_6$: 378.1140; found: 378.1144.

11c: colorless oil, R_f : 0.44; IR: 1754 cm^{-1} ; ^1H NMR δ 1.19 (t, $J = 7.2\text{ Hz}$, 3 H), 1.30 (t, $J = 7.2\text{ Hz}$, 3 H), 3.77 (q, $J = 4.8\text{ Hz}$, 1 H), 4.11 (q, $J = 7.2\text{ Hz}$, 2 H), 4.32 (q, $J = 7.2\text{ Hz}$, 2 H), 5.13-5.25 (m, 2 H), 7.35 (s, 5 H); ^{13}C NMR: δ 13.6, 44.4 (q, $J = 41.8\text{ Hz}$), 48.3, 62.7, 64.0, 69.4, 121.1 (q, $J = 275.3\text{ Hz}$), 128.4, 128.5, 128.6, 134.5, 156.7, 161.8, 163.0; ^{19}F NMR: δ -71.47; GC MS m/z 298 (M^+ -91, <1), 214 (51), 168 (32), 107 (25), 91 (100); HRMS-ESI (m/z) $[\text{M}+\text{Na}]^+$ calcd for $\text{C}_{17}\text{H}_{18}\text{F}_3\text{NO}_6$: 412.0984; found: 412.0977.

11d: yellow viscous oil, R_f : 0.41; IR: 1753 cm^{-1} ; ^1H NMR: δ 1.27 (t, $J = 7.2\text{ Hz}$, 3 H), 1.39 (t, $J = 7.2\text{ Hz}$, 3 H), 3.82 (q, $J = 4.8\text{ Hz}$, 1 H), 4.20-4.34 (m, 3 H), 4.38-4.46 (m, 2 H), 4.50-4.55 (m, 2 H), 7.33-7.36 (m, 2 H), 7.38-7.45 (m, 2 H), 7.62-7.71 (m, 2 H), 7.73-7.79 (m, 2 H); ^{13}C NMR: δ 13.8, 13.9, 44.6 (q, $J = 41.2\text{ Hz}$), 46.5, 48.3, 62.8, 64.2, 69.7, 119.9, 121.1 (q, $J = 274.8\text{ Hz}$), 125.0, 125.1, 127.0, 127.1, 127.8, 127.9, 141.1, 142.8, 143.0, 156.5, 161.3, 163.1; ^{19}F NMR: δ -71.43 (d, $J = 6.2\text{ Hz}$); HRMS-ESI (m/z) $[\text{M}+\text{Na}]^+$ calcd for $\text{C}_{24}\text{H}_{22}\text{F}_3\text{NO}_6$: 500.1297; found: 500.1304.

STANDARD 1H OBSERVE

```

exp1 stdlh
SAMPLE
date Feb 3 2004 dfrq 299.942
solvent CDCl3 dm H1
file exp 30 dpr 30
ACQUISITION dof 0
sfrq 299.942 dm mmn
tn H1 dm c
at 1.998 dmf 200
np 18008
sw 4506.5 wfile PROCESSING
fb 2600 proc ft
bs 16 fn not used
tpwr 55
pw 7.0 werr
dl 0 wexp
tof 400.0 wbs
nt 320 wnt
ct 320
alock n
gain not used
FLAGS
il n
in n
dp y
DISPLAY
sp -317.6
wp 4506.5
vs 213
sc 0
wc 265
hzmm 1.73
is 390.45
rfl 317.6
rfp 0
th 20
ins 100.000
na ph

```

STANDARD 1H OBSERVE

exp2 stdlh

SAMPLE		DEC. & VT	
date	May 3 2004	dfreq	299.942
solvent	CDCl3	dn	H1
file	exp	dpwr	30
ACQUISITION		dof	0
sfrq	299.942	dm	nmn
tn	H1	dmm	c
at	1.998	dmf	200
np	18008	PROCESSING	
sw	4506.5	wtfile	
fb	2600	proc	ft
bs	16	fn	not used
tpwr	55		
pw	7.0	werr	
dl	0	wexp	
tof	400.0	wbs	
nt	320	wnt	
ct	320		
alock	n		
gain	not used		
FLAGS			
il	n		
in	n		
dp	y		
DISPLAY			
sp	-318.2		
wp	4506.5		
vs	291		
sc	0		
vc	265		
hzmm	17.01		
is	2150.18		
rfl	2495.7		
rfp	2177.6		
th	34		
ins	100.000		
nm	ph

13C OBSERVE

exp5 std13c

SAMPLE		DEC. & VT	
date	Nov 5 2004	dfrq	299.942
solvent	CDCl3	dn	H1
file	exp	dpwr	43
ACQUISITION		dof	0
sfrq	75.428	dm	Y77
tn	C13	dmm	w
at	1.815	dmf	9900
mp	68106	PROCESSING	
sw	18761.7	lb	1.00
fb	10400	wtfile	
bs	16	proc	ft
tpwr	55	fn	not used
pw	5.5		
di	1.000	werr	
tof	0	wexp	
nt	10000	wbs	
ct	7008	wnt	
alock	n		
gain	28		
FLAGS			
il	n		
in	n		
op	y		
DISPLAY			
sp	-1834.1		
wp	18761.4		
vs	167		
sc	0		
wc	265		
hramm	1.98		
is	500.00		
rfl	7641.7		
rflp	5807.3		
th	19		
ins	100.000		
na	ph

STANDARD 1H OBSERVE

```

exp3 stdih
SAMPLE
date Feb 5 2004 dfrq 299.942
solvent CDCl3 dn H1
file exp dpr 30
ACQUISITION
sfrq 299.942 dm nnn
tn H1 dnm c
at 1.998 dmf 200
np 18008
sw 4506.5 wfile
fb 2600 proc ft
bs 16 fn not used
tpwr 55
pw 7.0 weiz
dl 0 wezp
tof 400.0 wbs
nt 320 wnt
ct 320
alock n
gain not used
FLAGS
il n
in n
dp y
DISPLAY
sp -318.2
wp 4506.5
va 173
sc 0
wc 265
hzmm 4.45
ls 393.71
rf1 318.2
rfp 0
th 20
ins 100.000
nm pb

```

13C OBSERVE

exp2 std13c

SAMPLE DEC. & VT
 date May 10 2004 dfrq 299.942
 solvent CDCl3 dn H1
 file exp dpr 44
 ACQUISITION dof 0
 sfrq 75.428 dm yyy
 tn C13 dnm w
 at 1.815 dmf 2800
 np 68106 PROCESSING
 sw 18761.7 lb 1.00
 fb 10400 wfile
 bs 16 proc ft
 tpwr 55 fn not used
 pw 8.7
 dl 0.500 werr
 tof 400.0 wep
 nt 3000 whs
 ct 2528 wnt

FLAGS

il n
 in n
 dp y

DISPLAY

sd -1436.5
 wp 18761.0
 vs 350
 sc 265
 wc 265
 hnm 2.35
 is 500.00
 rfi 7243.9
 rfp 5807.5
 th 13
 ins 100.000
 nm ph

4b

STANDARD 1H OBSERVE

exp1 std1h

SAMPLE		DEC. & VT	
date	May 14 2004	dfrq	299.942
solvent	CDCl3	dn	H1
file	exp	dpwr	30
ACQUISITION		dof	0
sfrq	299.942	dm	nan
tn	H1	dmm	C
at	1.998	daf	200
np	18008	PROCESSING	
sw	4506.5	wtfile	ft
fb	2600	proc	not used
bs	4	fn	not used
tpwr	55		
pw	7.0	werr	
dl	0	wexp	wft
tof	400.0	whs	
nt	320	wnt	
ct	284		
alock	n		
gain	not used		
FLAGS			
il	n		
in	n		
dp	y		
DISPLAY			
sp	-348.4		
wp	4506.5		
vs	134		
sc	0		
wc	265		
hmm	4.47		
is	392.15		
rfl	348.4		
rfp	0		
th	34		
ins	100.000		
ai	ph

13C OBSERVE

exp2 std13c

SAMPLE DEC. & VT

date Apr 21 2004 dfrq 299.942

solvent CDCl₃ dn H1

file exp 44

ACQUISITION exp 0

sfrq 75.428 dn yyy

tn C13 dnm w

at 1.815 dmf 2800

np 68106 PROCESSING

sw 18761.7 lb 1.00

fb 10400 wfile

bs 16 proc ft

tpwr 55 fn not used

pw 8.7

dl 1.000 verr

tof 400.0 wexp

nt 10000 wbs

ct 5040 wnt

alock n

gain 28

FLAGS

il n

in n

dp y

DISPLAY

sp -1437.1

wp 18761.7

vs 170

sc 0

wc 265

hnm 22.78

ls 500.00

xf1 7244.5

rfp 5807.3

th 34

ins 100.000

nm ph

STANDARD 1H OBSERVE

```

exp1 stdlh
SAMPLE
date May 17 2004 dfrq 299.942
solvent CDCl3 dn H1
file exp dpr 30
ACQUISITION
sfrq 299.942 dn H1
tn 1.998 dmf 200
at 18008
mp 4506.5 lb 0.50
wf 2600 wfile
fb 4
bs 4 proc ft
tpwr 55 fn not used
pw 7.0
d1 0 werr
tof 400.0 wexp wft
nt 320 wbs
ct 320 wnt
alock n
gain not used
FLAGS
il n
in n
dp y
DISPLAY
sp -317.9
wp 4506.5
vs 231
sc 0
wc 265
hmm 17.01
ls 324.04
rf1 317.9
rfp 0
th 68
ins 100.000
ai ph
  
```

5a

¹H NMR Spectrum recorded at 300 MHz instrument.

Solvent: CDCl₃

5b

STANDARD 1H OBSERVE

exp3 stdlh

SAMPLE		DEC. & VT	
date	Jul 16 2004	dfrq	299.942
solvent	CDCl3	dn	H1
file	exp	qpr	30
ACQUISITION		dof	0
sfrq	299.942	dm	mm
tn	H1	dm	c
at	1.998	dmf	200
PROCESSING			
up	18008	lb	0.50
sw	4506.5	wtfile	
fb	2600	proc	ft
bs	16	fn	not used
tpwr	55		
pw	7.0		
dl	0	werr	
tof	400.0	wexp	
nt	320	whs	
ct	320	wnt	
alock	n		
gain	not used		
FLAGS			
il	n		
in	n		
qp	y		
DISPLAY			
sp	-344.6		
wp	4506.5		
vs	136		
sc	0		
wc	265		
hzm	0.36		
is	299.43		
rfl	2522.2		
rfp	2177.6		
th	51		
ins	100.000		
nm	ph

STANDARD 1H OBSERVE

exp4 std1h

SAMPLE		DEC. & VT	
date	Sep 2 2004	dfrq	299.942
solvent	CDCl3	dn	H1
file	exp	dpr	30
ACQUISITION		dof	0
sfrq	299.942	dm	nan
tn	H1	dmm	c
at	1.998	dmf	200
np	18008	PROCESSING	
sw	4506.5	wtfile	ft
fb	2600	proc	not used
bs	16	fn	
tpwr	55		
pw	7.0	werr	
dl	0	wexp	
tof	400.0	wbs	
nt	320	wnt	
ct	320		
alock	n		
gain	not used		
FLAGS			
il	n		
in	n		
dp	y		
DISPLAY			
sp	-318.2		
wp	4506.5		
vs	292		
sc	0		
wc	265		
hmm	0.47		
is	1000.00		
rfl	318.2		
rfl	0		
th	71		
ins	100.000		
nm	ph		

5d

6a

STANDARD IN OBSERVE

exp4 stdlh

SAMPLE		DEC. & VT	
date	May 19 2004	dfrq	299.942
solvent	CDCl3	dn	H1
file	exp	dpwr	30
ACQUISITION		dof	0
sfrq	299.942	dm	mm
tn	H1	dm	C
at	1.998	dmf	200
np	18008	PROCESSING	
sw	4506.5	wtfile	
fb	2600	proc	ft
bs	16	fn	not used
tpwr	55		
pw	7.0	weir	
d1	0	wexp	
tof	400.0	whs	
nt	320	wat	
ct	320		
alock	n		
gain	not used		
FLAGS			
il	n		
in	n		
dp	y		
DISPLAY			
sp	-317.9		
wp	4506.5		
vs	189		
sc	0		
wc	265		
hmm	0.26		
is	430.11		
rfl	317.9		
rfp	0		
th	47		
ins	100.000		
na	ph

¹H NMR Spectrum recorded at 300 MHz instrument.

Solvent: CDCl₃

6b

¹H NMR Spectrum recorded at 300 MHz instrument.

Solvent: CDCl₃

¹H NMR Spectrum recorded at 300 MHz instrument.

Solvent: CDCl₃

6d

exp8 std1h

CCOC(=O)N(C(=O)OCC)C(=C)C(F)(F)FC(=O)OCC

7a

7a

13C OBSERVE

exp7 std13c

SAMPLE DEC. & VT

date Jun 9 2004 dfrq 299.942

solvent CDCl3 dn H1

file exp dpr 44

ACQUISITION dof 0

sfrq 75.428 dn yyy

tn Cl3 dnm w

at 1.815 dmf 2800

np 68106 PROCESSING

sw 18761.7 lb 1.00

fb 10400 wtfile

bs 16 proc ft

tpwr 55 fn not used

pw 8.7

dl 1.000

tof 400.0 yexp

nt 7000 qbs

ct 5648 wnt

alock n

gain 28

FLAGS

il n

in n

dp y

DISPLAY

sp -1434.2

wp 18761.7

vs 345

sc 0

wc 265

hzmm 18.91

is 500.00

rfl 7241.6

rfp 5807.3

th 42

ins 100.000

nm ph

55.842

19F OBSERVE
STANDARD PARAMETERS

exp6 s2pul

SAMPLE		DEC. & VT	
date	Jun 9 2004	dfrq	299.942
solvent	CDC13	dn	30
file	exp	dpwr	0
ACQUISITION		dof	0
sfrq	282.197	dm	nan
tn	F19	dmm	c
at	0.300	dmf	200
np	29952	PROCESSING	
sw	50000.0	lb	0.30
fb	27600	wtfile	
bs	16	proc	ft
tpwr	55	fn	not used
pw	5.0		
d1	4.000	werr	
tof	400.0	wexp	
nt	320	wbs	
ct	164	wnt	
alock	n		
gain	not used		
FLAGS			
il	n		
in	n		
qp	y		
DISPLAY			
sp	5597.4		
wp	50000.0		
vs	200		
sc	0		
wc	265		
hnm	5.56		
is	713.54		
xf1	-5597.4		
rfp	0		
th	43		
ins	100.000		
nm	ph

7c

-162.900

-30 -50 -70 -90 -110 -130 -150 -170 ppm

19F OBSERVE

STANDARD PARAMETERS

exp3 s2pul

SAMPLE		DEC. & VT	
date	Apr 21 2004	dfrq	299.942
solvent	CDCl3	dn	H1
file	exp	dpwr	30
ACQUISITION		dof	0
sfrq	282.197	dm	mm
tn	F19	dmm	c
at	0.300	daf	200
np	29952	PROCESSING	
sw	50000.0	lb	0.30
fb	27600	wtfile	
bs	16	proc	ft
tpwr	55	fn	not used
pw	5.0		
d1	4.000	werr	
tof	400.0	wexp	
nt	320	wbs	
ct	320	wnt	
alock	n		
gain	not used		
FLAGS			
il	n		
in	n		
dp	y		
DISPLAY			
sp	5588.2		
wp	50000.0		
vs	166		
sc	0		
wc	265		
hzmm	3.12		
is	944.28		
rfl	8209.7		
rfd	13797.9		
th	13		
ins	100.000		
nm	ph

7d

-162.900

ppm

-170

-150

-130

-110

-90

-70

-50

-30

19F OBSERVE
STANDARD PARAMETERS

```

exp3  s2pul
SAMPLE
date   Oct 29 2004   dfrq   299.942
solvent CDC13      dn      H1
file    exp      30
ACQUISITION
sfrq    282.197    dm      ann
tn       F19      dm      c
at       0.300    dm      200
np      29952
sw      50000.0    lb      0.30
fb      27600    wfile
bs       8      proc      ft
tpwr     60    fn      not used
pw       5.0
d1      4.000    werr
tof     400.0    wexp
nt      320    wbs
ct      192    wnt
alock    n
gain     not used
FLAGS
il       n
in       n
cp       y
DISPLAY
sp      5643.1
wp      50000.0
vs      816
sc       0
wc      265
hzmm    188.68
is      501.27
rfl     8154.8
rfp     13797.9
th       52
ins     100.000
nm      ph
  
```

17F OBSERVE
STANDARD PARAMETERS

exp2 s2pul

SAMPLE		date Jun 14 2004		dfreq	299.942
solvent	CDC13	dn	H1		
file	exp	dpwr	30		
ACQUISITION		dof	0		
sfrq	282.197	dm	mm		
tn	F19	dmm	c		
at	0.300	dmf	200		
np	29952	PROCESSING			
sw	50000.0	lb	0.30		
fb	27600	wtfile			
bs	4	proc	ft		
tpwr	55	fn	not used		
pw	5.0				
d1	4.000	werr			
tof	400.0	wexp			
nt	320	wbs			
ct	320	wnt			
alock	n				
gain	not used				
FLAGS					
il	n				
in	n				
dp	y				
DISPLAY					
sp	5548.5				
wp	50000.0				
vs	167				
sc	0				
wc	265				
hzmm	19.99				
is	500.00				
rfl	-5548.5				
rfp	0				
th	20				
ins	100.000				
nm	no	ph

8d

-162.900

-58.234

ppm

-170

-150

-130

-110

-90

-70

-50

-30

13C OBSERVE

exp2 std13c

SAMPLE		DEC. & VT	
date	Oct 23 2002	dfrq	299.942
solvent	CDCl3	dn	H1
file		exp	44
ACQUISITION		dof	0
sfrq	75.428	dm	YTY
tn	C13	dm	w
at	1.815	dmf	2800
np	68106	PROCESSING	
sw	18761.7	lb	2.00
fb	10400	wtfile	
bs	16	proc	ft
typr	55	fn	not used
pw	8.7		
dl	0	werr	
tof	400.0	wexp	
nt	3000	wbs	
ct	2160	wnt	
alock	n		
gain	28	FLAGS	
il	n		
in	n		
dp	y	DISPLAY	
sp	-1436.8		
wp	18761.7		
vs	200		
sc	0		
wc	265		
hnm	1.23		
is	500.00		
rfl	7244.2		
rfd	5807.3		
th	26		
ins	100.000		
na	ph

11a

STANDARD 1H OBSERVE

```

exp1 std1h
SAMPLE
date Mar 10 2003 dfrq 299.942
solvent CDCl3 dn H1
file exp dpwr 30
ACQUISITION dof 0
sfrq 299.942 dm nnn
tn H1 dnm c
at 1.998 dmf 200
np 18008
sw 4506.5 wfile
fb 2600 proc ft
bs 16 fn not used
tpwr 55
pw 7.0 werr
d1 0 wexp
tof 400.0 wbs
nt 320 wnt
ct 320
alock n
gain not used
FLAGS
il n
in n
cp y
DISPLAY
sp -317.9
wp 4506.5
vs 536
sc 0
wc 265
hzmm 0.18
fs 262.61
xfl 317.9
rfp 0
th 57
ins 100.000
na ph

```

11c

13C OBSERVE

exp5 std13c

SAMPLE DEC. & VT
 date Nov 10 2004 dfrq 299.942
 solvent CDCl3 dn H1
 file exp 43 dpwr 43
 ACQUISITION dof 0
 sfrq 75.428 dm YYY
 tn C13 dnm w
 at 1.815 dmf 9900
 np 68106
 sw 18761.7 lb 1.00
 fb 10400 wfile
 bs 15 proc ft
 tpwr 55 fn not used
 pw 5.5
 dl 1.000 wevr
 tof 400.0 wevp
 nt 2500 wbs
 ct 2500 vnt
 alock n
 gain 28
 FLAGS
 il n
 in n
 dp y
 DISPLAY
 sp -1643.7
 wp 18761.4
 vs 206
 sc 0
 wc 265
 hzmm 8.59
 is 500.00
 rf1 7451.3
 rfp 5807.4
 th 11
 ins 100.000
 nm ph

exp3 std1h

11d