Amination of CF₃-Enones with Nosyloxycarbamates

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General Experimental Methods. 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 CHCl_3 as the solvent. ^1H NMR and ^{13}C NMR spectra were recorded at 300 and 75 MHz, respectively. CDCl_3 was used as the solvent and CHCl_3 as the internal standard. ^{19}F NMR spectra were recorded at 282.2 MHz, using CDCl_3 as the solvent and C_6F_6 as the internal standard; chemical shifts are given in ppm relative to CFCl_3 . ESI MS analyses were performed using a quadrupole-time of flight (TOF) mass spectrometer equipped with an ESI source and a syringe pump. 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 was used as such. The synthesis of substrates **10-12**,¹ **13**,² and of NsONH-R^{3,4} were performed following reported procedures.

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- (2) Andrew, R. J.; Mellor, J. M. *Tetrahedron* **2000**, 56, 7261-7266.
- (3) Lwowski, W.; Maricich, T. J. *J. Am. Chem. Soc.* **1965**, 87, 3630-3637.
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Analytical and spectroscopic data for compounds 6-21.

Ethyl 2-phenyl-5-(trifluoromethyl)-1,3-oxazole-3(2H)-carboxylate (6): pale yellow oil; IR (CHCl₃) 1728, 1684 cm⁻¹; ¹H NMR (CDCl₃) δ 1.27 (br, 3 H), 4.17 (br, 2 H), 6.82 (br, 1 H), 6.92 (s, 1 H), 7.43 (s, 3 H), 7.49 (s, 2 H); ¹³C NMR (CDCl₃) δ 14.3, 62.6, 94.2, 110.9, 119.0 (q, ¹J_{CF} = 266.7 Hz), 126.2, 128.7, 130.0, 132.4 (q, ²J_{CF} = 41.8 Hz), 136.8, 151.0; ¹⁹F NMR (CDCl₃) δ -69.14; HRMS-ESI (*m/z*) [M+Na]⁺ calcd for C₁₃H₁₂F₃NNaO₃: 310.0661; found: 310.0665.

tert-Butyl 2-phenyl-5-(trifluoromethyl)-1,3-oxazole-3(2H)-carboxylate (7): pale yellow oil; IR (CHCl₃) 1731, 1687 cm⁻¹; ¹H NMR (CDCl₃) δ 1.27/1.48 (s, 9 H, due to the appearance of rotamers the signal is doubled), 6.74 (br, 1 H), 6.92 (s, 1 H), 7.38-7.51 (m, 5 H); ¹³C NMR (CDCl₃) δ 28.0, 82.6, 94.3, 111.5, 119.1 (q, ¹J_{CF} = 266.5 Hz), 126.2, 128.6, 129.9, 131.9 (q, ²J_{CF} = 41.9 Hz), 137.3, 150.1; ¹⁹F NMR (CDCl₃) δ -69.09; HRMS-ESI (*m/z*) [M+Na]⁺ calcd for C₁₅H₁₆F₃NNaO₃: 338.0974; found: 338.0968.

Ethyl 2-ethoxy-5-(trifluoromethyl)-1,3-oxazole-3(2H)-carboxylate (8): colorless oil; IR (CHCl₃) 1728, 1683 cm⁻¹; ¹H NMR (CDCl₃) δ 1.24 (t, *J* = 7.2 Hz, 3 H), 1.29 (t, *J* = 7.2 Hz, 3 H), 3.71 (q, *J* = 7.2 Hz, 2 H), 4.24 (q, *J* = 7.2 Hz, 2 H), 6.69 (s, 1 H), 6.77 (br, 1 H); ¹³C NMR (CDCl₃) δ 14.2, 14.7, 53.3, 62.8, 107.6, 109.9, 118.9 (q, ¹J_{CF} = 266.5 Hz), 130.6 (q, ²J_{CF} = 42.3 Hz), 150.5; ¹⁹F NMR (CDCl₃) δ -68.98; HRMS-ESI (*m/z*) [M+Na]⁺ calcd for C₉H₁₂F₃NNaO₄: 278.0611; found: 278.0618.

Ethyl trans-2-acetyl-3-phenylaziridine-1-carboxylate (9): pale yellow oil; IR (CHCl₃) 1736, 1721 cm⁻¹; ¹H NMR (CDCl₃) δ 1.23 (t, *J* = 7.2 Hz, 3 H), 2.35 (s, 3 H), 3.33 (d, *J* = 2.4 Hz, 1 H), 3.66 (d, *J* = 2.4 Hz, 1 H), 4.11-4.23 (m, 2 H), 7.27-7.35 (m, 5 H); ¹³C NMR (CDCl₃) δ 14.1, 30.0, 46.7, 49.5, 62.6, 126.2, 128.3, 128.5, 135.3, 159.9, 200.7; HRMS-ESI (*m/z*) [M+Na]⁺ calcd for C₁₃H₁₅NNaO₃: 256.0944; found: 256.0936.

Ethyl [(E)-2-(2-thienyl)vinyl]carbamate (*trans*-14): white solid, mp 84-85 °C (CHCl₃/pentane); IR (CHCl₃) 3435, 1727, 1659 cm⁻¹; ¹H NMR (CDCl₃) δ 1.28 (t, *J* = 6.9 Hz, 3 H), 4.20 (q, *J* = 6.9 Hz, 2 H), 6.15 (d, *J* = 14.4 Hz, 1 H), 6.82 (d, *J* = 3.3 Hz, 1 H), 6.91 (dd, *J* = 5.1 Hz, *J* = 3.3 Hz, 1 H), 7.04 (d, *J* = 5.1 Hz, 1 H), 7.07-7.16 (m, 2 H); ¹³C NMR (CDCl₃) δ 14.4, 61.6, 104.5, 122.3, 123.4, 123.9, 127.2, 140.6, 153.6; HRMS-ESI (*m/z*) [M+Na]⁺ calcd for C₉H₁₁NNaO₂S: 220.0403; found: 220.0407.

Ethyl [(Z)-2-(2-thienyl)vinyl]carbamate (*cis*-14): pale yellow oil; IR (CHCl₃) 3417, 1732, 1654 cm⁻¹; ¹H NMR (CDCl₃) δ 1.30 (t, *J* = 7.2 Hz, 3 H), 4.23 (q, *J* = 7.2 Hz, 2 H), 5.78 (d, *J* = 9.6 Hz, 1 H), 6.68 (dd, *J* = 10.1 Hz, *J* = 9.6 Hz, 1 H), 6.94 (d, *J* = 3.6 Hz, 1 H), 7.02-7.05 (m, 2 H), 7.23 (d, *J* = 4.8 Hz, 1 H); ¹³C NMR (CDCl₃) δ 14.5, 61.9, 101.1, 122.6, 123.8, 125.4, 127.5, 138.3, 153.7; HRMS-ESI (*m/z*) [M+Na]⁺ calcd for C₉H₁₁NNaO₂S: 220.0403; found: 220.0399.

Ethyl [(E)-2-(3-thienyl)vinyl]carbamate (*trans*-15): white solid, mp 81-83°C (CHCl₃/pentane); IR (CHCl₃) 3435, 1725, 1665 cm⁻¹; ¹H NMR (CDCl₃) δ 1.28 (t, *J* = 6.9 Hz, 3 H), 4.20 (q, *J* = 6.9 Hz, 2 H), 6.00 (d, *J* = 14.4 Hz, 1 H), 6.65 (br, 1 H), 6.96 (s, 1 H), 7.03-7.07 (m, 1 H), 7.13 (d, *J* = 5.1 Hz, 1 H), 7.20-7.24 (m, 1 H); ¹³C NMR (CDCl₃) δ 14.5, 61.6, 105.5, 119.1, 124.2, 124.4, 126.0, 137.9, 153.7; HRMS-ESI (*m/z*) [M+Na]⁺ calcd for C₉H₁₁NNaO₂S: 220.0403; found: 220.0397.

Ethyl [(Z)-2-(3-thienyl)vinyl]carbamate (*cis*-15): pale yellow oil; IR (CHCl₃) 3429, 1730, 1659 cm⁻¹; ¹H NMR (CDCl₃) δ 1.26 (t, *J* = 6.9 Hz, 3 H), 4.20 (q, *J* = 6.9 Hz, 2 H), 5.61 (d, *J* = 9.6 Hz, 1 H), 6.68 (dd, *J* = 10.1 Hz, *J* = 9.6 Hz, 1 H), 7.03-7.14 (m, 1 H), 7.19 (br, 1 H), 7.29-7.37 (m, 2 H); ¹³C NMR (CDCl₃) δ 14.3, 61.4, 103.8, 121.3, 123.2, 124.4, 126.3, 138.2, 153.6; HRMS-ESI (*m/z*) [M+Na]⁺ calcd for C₉H₁₁NNaO₂S: 220.0403; found: 220.0409.

Ethyl [(E)-2-(2-furyl)vinyl]carbamate (*trans*-16): pale yellow solid, mp 75-77 °C (CHCl₃/pentane); IR (CHCl₃) 3435, 1725, 1668 cm⁻¹; ¹H NMR (CDCl₃) δ 1.26 (t, *J* = 6.9 Hz, 3 H), 4.19 (q, *J* = 6.9 Hz, 2 H), 5.86 (d, *J* = 14.4 Hz, 1 H), 6.04 (d, *J* = 3.0 Hz, 1 H), 6.31 (d, *J* = 1.8 Hz, 1

H), 6.86 (br, 1 H), 7.12-7.24 (m, 2 H); ^{13}C NMR (CDCl_3) δ 14.4, 61.6, 100.1, 105.0, 111.1, 123.6, 140.7, 151.7, 153.7; HRMS-ESI (m/z) $[\text{M}+\text{Na}]^+$ calcd for $\text{C}_9\text{H}_{11}\text{NNaO}_3$: 204.0631; found: 204.0638.

Ethyl [(Z)-2-(2-furyl)vinyl]carbamate (cis-16): pale yellow oil; IR (CHCl_3) 3430, 1734, 1654 cm^{-1} ; ^1H NMR (CDCl_3) δ 1.31 (t, $J = 7.2$ Hz, 3 H), 4.23 (q, $J = 7.2$ Hz, 2 H), 5.40 (d, $J = 9.6$ Hz, 1 H), 6.13 (d, $J = 3.3$ Hz, 1 H), 6.38-6.40 (m, 1 H), 6.62 (dd, $J = 10.2$ Hz, $J = 9.6$ Hz, 1 H), 7.42 (s, 1 H), 8.09 (br, 1 H); ^{13}C NMR (CDCl_3) δ 14.5, 61.7, 96.0, 107.0, 111.3, 121.7, 141.0, 152.8, 153.4; HRMS-ESI (m/z) $[\text{M}+\text{Na}]^+$ calcd for $\text{C}_9\text{H}_{11}\text{NNaO}_3$: 204.0631; found: 204.0635.

Ethyl [(E)-2-(4-methoxyphenyl)vinyl]carbamate (trans-17): white solid, mp 72-74°C (CHCl_3 /pentane); IR (CHCl_3) 3437, 1726, 1663 cm^{-1} ; ^1H NMR (CDCl_3) δ 1.27 (t, $J = 7.2$ Hz, 3 H), 3.78 (s, 3 H), 4.19 (q, $J = 7.2$ Hz, 2 H), 5.92 (d, $J = 14.4$ Hz, 1 H), 6.67-6.71 (m, 1 H), 6.82 (d, $J = 8.7$ Hz, 2 H), 7.06-7.18 (m, 1 H), 7.20 (d, $J = 8.7$ Hz, 2 H); ^{13}C NMR (CDCl_3) δ 14.5, 55.2, 61.5, 110.2, 114.1, 122.5, 126.3, 128.9, 153.8, 158.2; HRMS-ESI (m/z) $[\text{M}+\text{Na}]^+$ calcd for $\text{C}_{12}\text{H}_{15}\text{NNaO}_3$: 244.0944; found: 244.0951.

Ethyl 2-(2-thienyl)-5-(trifluoromethyl)-1,3-oxazole-3(2H)-carboxylate (18): pale yellow oil, IR (CHCl_3) 1714, 1686 cm^{-1} ; ^1H NMR (CDCl_3) δ 1.25 (t, $J = 6.9$ Hz, 3 H), 4.20 (d, $J = 6.9$ Hz, 2 H), 6.81 (br, 1 H), 7.01 (dd, $J = 4.8$ Hz, $J = 3.3$ Hz, 1 H), 7.16 (br, 1 H), 7.37 (d, $J = 4.8$ Hz, 1 H), 7.78 (d, $J = 3.3$ Hz, 1 H); ^{13}C NMR (CDCl_3) δ 14.3, 62.8, 90.2, 110.5, 118.9 (q, $^1J_{\text{CF}} = 266.7$ Hz), 126.8, 127.1, 128.3, 131.9 (q, $^2J_{\text{CF}} = 42.1$ Hz), 135.1, 150.7; ^{19}F NMR (CDCl_3) δ -69.18; HRMS-ESI (m/z) $[\text{M}+\text{Na}]^+$ calcd for $\text{C}_{11}\text{H}_{10}\text{F}_3\text{NNaO}_3\text{S}$: 316.0226; found: 316.0232.

Ethyl 2-(2-furyl)-5-(trifluoromethyl)-1,3-oxazole-3(2H)-carboxylate (19): pale yellow oil, IR (CHCl_3) 1720, 1686 cm^{-1} ; ^1H NMR (CDCl_3) δ 1.26 (t, $J = 6.9$ Hz, 3 H), 4.18 (d, $J = 6.9$ Hz, 2 H), 6.38 (dd, $J = 3.3$ Hz, $J = 1.8$ Hz, 1 H), 6.53 (s, 1 H), 6.86 (br, 2 H), 7.44 (s, 1 H); ^{13}C NMR (CDCl_3) δ 14.3, 62.7, 87.4, 96.2, 110.1, 110.5 (two), 119.0 (q, $^1J_{\text{CF}} = 266.4$ Hz), 132.3 (q, $^2J_{\text{CF}} = 42.1$ Hz),

143.8, 150.5; ^{19}F NMR (CDCl_3) δ -69.11 ; HRMS-ESI (m/z) $[\text{M}+\text{Na}]^+$ calcd for $\text{C}_{11}\text{H}_{10}\text{F}_3\text{NNaO}_4$: 300.0454; found: 300.0457.

Ethyl [(1Z)-3,3,3-trifluoro-2-oxo-1-(2-thienylmethylene)propyl]carbamate (20): white solid, mp 89-91 °C (CHCl_3 /pentane); IR (CHCl_3) 3402, 1731, 1609 cm^{-1} ; ^1H NMR (CDCl_3) δ 1.25 (t, $J = 7.2$ Hz, 3 H), 4.21 (q, $J = 7.2$ Hz, 2 H), 6.02 (br, 1 H), 7.18 (dd, $J = 5.1$ Hz, $J = 3.6$ Hz, 1 H), 7.54 (d, $J = 3.6$ Hz, 1 H), 7.71 (d, $J = 5.1$ Hz, 1 H), 7.86 (s, 1 H); ^{13}C NMR (CDCl_3) δ 14.4, 62.4, 116.6 (q, $^1J_{\text{CF}} = 290.4$ Hz), 123.9, 127.9, 134.7, 135.7, 135.8, 136.7, 154.4, 178.1 (q, $^2J_{\text{CF}} = 34.6$ Hz); ^{19}F NMR (CDCl_3) δ -71.74 ; HRMS-ESI (m/z) $[\text{M}+\text{Na}]^+$ calcd for $\text{C}_{11}\text{H}_{10}\text{F}_3\text{NNaO}_3\text{S}$: 316.0226; found: 316.0221.

Ethyl [(1Z)-3,3,3-trifluoro-1-(2-furylmethylene)-2-oxopropyl]carbamate (21): white solid, mp 83-84 °C (CHCl_3 /pentane); IR (CHCl_3) 3423, 1724, 1629 cm^{-1} ; ^1H NMR (CDCl_3) δ 1.27 (t, $J = 7.2$ Hz, 3 H), 4.20 (q, $J = 7.2$ Hz, 2 H), 6.57 (s, 1 H), 6.82 (d, $J = 3.0$ Hz, 1 H), 6.88 (s, 1 H), 7.03 (br, 1 H), 7.63 (s, 1 H); ^{13}C NMR (CDCl_3) δ 14.3, 62.5, 113.0, 116.3 (q, $^1J_{\text{CF}} = 290.5$ Hz), 118.4, 118.7, 125.4, 146.1, 149.2, 154.1, 178.9 (q, $^2J_{\text{CF}} = 35.4$ Hz); ^{19}F NMR (CDCl_3) δ -73.14 ; HRMS-ESI (m/z) $[\text{M}+\text{Na}]^+$ calcd for $\text{C}_{11}\text{H}_{10}\text{F}_3\text{NNaO}_4$: 300.0454; found: 300.0458.

STANDARD 1H OBSERVE

```

exp1 stdlh
SAMPLE
date Apr 14 2005 dfrq 299.942
solvent CDCl3 dn H1
file exp dpr 30
ACQUISITION
sfrq 299.942 dm nun
tn H1 dm c
at 1.998 dmf 200
np 18008 PROCESSING
sw 4506.5 lb 0.50
fb not used wfile
bs 16 proc ft
tpwr 58 fn not used
pw 7.0
d1 0 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 -318.6
wp 4506.3
vs 163
sc 0
wc 265
hzmm 17.00
is 683.48
rfl 318.9
rfp 0
th 20
ins 100.000
nm ph

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STANDARD 1H OBSERVE

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 solvent CDCl3 dn H1
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 ACQUISITION dof 0
 sfrq 299.942 dm nm
 tn H1 dm c
 at 1.998 dmf 200
 np 18008 PROCESSING
 sw 4506.5 wfile ft
 fb 2600 proc
 bs 16 fn not used
 tpwr 58
 pw 7.0 weir
 d1 0 wexp
 tof 600.0 wbs
 nt 320 wnt
 ct 320
 alook n
 gain not used
 FLAGS
 il n
 in n
 dp y
 DISPLAY
 sp -117.8
 wp 4506.3
 vs 232
 sc 0
 wc 265
 hzmm 1.33
 is 236.32
 rfl 118.1
 rfp 0
 th 22
 ins 100.000
 nm ph

19F OBSERVE

STANDARD PARAMETERS

exp3 s2pul

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date	Mar 29 2006	dfrq	299.942
solvent	CDCl3	dn	H1
file	exp	dpwr	30
ACQUISITION		dof	0
sfrq	282.191	dm	mm
tn	F19	dmm	c
at	0.300	dmf	200
np	29952	PROCESSING	
sw	50000.0	wtfile	
fb	27600	proc	ft
bs	4	fn	not used
tpwr	60		
pw	10.0	werr	
dl	4.000	wexp	
tof	-5500.0	wbs	
nt	320	wnt	
ct	224		
alock	n		
gain	2		
FLAGS			
il	n		
in	n		
dp	y		
DISPLAY			
sp	-270.7		
vp	49996.9		
vs	277		
sc	0		
wc	265		
hamm	4.80		
is	3000.00		
rf1	273.7		
rfl	0		
rflp	15		
th	15		
ins	100.000		
nm	ph

7

ppm

-190

-170

-150

-130

-110

-90

-70

-50

exp2 std1h

SAMPLE		date		DEC. & VT	
solvent	CDCl3	Apr 7 2006	dfrq	299.942	
file	exp		dn		H1
ACQUISITION			dpr		30
sfreq	299.942		dof		0
tn	H1		dm		nm
at	1.998		dmc		c
np	1808				200
sw	4506.5		PROCESSING		
fb	2600		lb		0.30
bs	16		wfile		ft
tpwr	58		fn		not used
pw	7.0				
dl	0		werr		
toft	600.0		wexp		
nt	320		wba		
ct	320		wnt		
alalock	n				
gain	not used				
FLAGS					
il	n				
in	n				
dp	y				
DISPLAY					
sp	-117.5				
wp	4506.3				
vs	599				
sc	0				
wc	265				
hmm	0.51				
is	389.48				
rfl	2295.4				
rfp	2177.6				
th	31				
ins	100.000				
nm					
ph

13C OBSERVE

exp3 std13c

SAMPLE		DEC. & VT	
date	Apr 7 2006	dfrq	299.942
solvent	CDCl3	dn	H1
file	exp	dpwr	43
ACQUISITION		dof	0
sfrq	75.428	dm	YYY
tn	CL3	dmm	w
at	1.815	dmf	10620
np	68106	PROCESSING	
sw	18761.7	lb	1.00
fb	10400	wtfile	
bs	16	proc	ft
tpwr	54	fn	not used
pw	5.5		
d1	0.500	werr	
tof	600.0	wexp	
nt	5000	wbs	
ct	4000	wnt	
alock	n		
gain	28		
FLAGS			
il	n		
in	n		
dp	y		
DISPLAY			
sp	-1231.5		
wp	18761.4		
vs	179		
sc	0		
wc	265		
hnum	13.66		
is	500.00		
rfl	7039.1		
rfd	5807.3		
th	12		
ins	100.000		
nm	no	ph

8

13F OBSERVE
STANDARD PARAMETERS

exp2 s2pul

SAMPLE		DEC. & VT	
date	Oct 14 2005	dfrq	299.942
solvent	CDCl3	dn	M1
file	exp	dpr	30
ACQUISITION		dof	0
sfrq	282.197	dn	nm
tn	F19	dm	c
at	0.300	daf	200
xp	29952	PROCESSING	
sw	50000.0	lb	0.30
fb	not used	wtfile	
bs	4	proc	ft
tpwr	58	fn	not used
pw	5.0		
dl	4.000	werr	
tof	400.0	wexp	
nt	320	wbs	
ct	148	wnt	
alock	n		
gain	2		
FLAGS			
il	n		
in	n		
dp	y		
DISPLAY			
sp	5602.5		
wp	49996.9		
vs	278		
sc	0		
wc	265		
hmm	3.32		
is	500.00		
rfl	8142.1		
rfp	13741.5		
th	18		
ins	100.000		
nm	ph

8

STANDARD 1H OBSERVE

exp6 stdlib

SAMPLE		DEC. & VT	
date	Jul 15 2005	dfreq	299.942
solvent	CDCl3	dn	H1
file	exp	dpwr	30
ACQUISITION		dof	0
sfreq	299.942	dm	nan
tn	H1	dmm	c
at	1.998	daf	200
np	18008	PROCESSING	
sw	4506.5	wtfile	
fb	not used	proc	ft
bs	16	fn	not used
tpwr	58		
pw	7.0	werr	
dl	0	wexp	
tof	400.0	wbs	
nt	320	wat	
ct	320		
alock	n		
gain	not used		
FLAGS			
il	n		
in	n		
dp	y		
DISPLAY			
sp	-318.3		
wp	4506.3		
vs	200		
sc	0		
wc	265		
hmm	0.50		
ls	500.00		
rfl	2496.1		
xfp	2177.6		
th	20		
ins	100.000		
nm	ph

9

STANDARD IN OBSERVE

exp2 stdih

SAMPLE		DEC. & VT	
date	Feb 21 2006	dfrq	299.942
solvent	CDCl3	dn	H1
file	exp	dpwr	30
ACQUISITION		dof	0
sfrq	299.942	dm	mmn
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	58		
pw	7.0	werr	
d1	0	wexp	
tof	600.0	wbs	
nt	320	wnt	
ct	320		
alock	n		
gain	not used		
FLAGS			
il	n		
in	n		
dp	y		
DISPLAY			
sp	-117.8		
wp	4506.3		
vs	209		
sc	0		
wc	265		
hzmm	2.83		
ls	358.00		
rfl	118.1		
rfp	0		
th	71		
ins	100.000		
nm	ph

13C OBSERVE

exp3 std13c

SAMPLE DEC. & VT

date Feb 21 2006 dfrq 299.942

solvent CDCl3 dn H1

file exp dpr 43

ACQUISITION exp dof 0

sfreq 75.428 dm YYY

tn C13 dm W

at 1.815 dmf 10620

np 68106

sw 18761.7 lb PROCESSING

fb 10400 wtfile 1.00

bs 16 proc ft

tpwr 54 fn not used

pw 5.5

d1 1.000 werr

tof 600.0 wexp

nt 10000 wbs

ct 3600 wnt

alock n

gain 28

FLAGS

il n

in n

dp y

DISPLAY

sp -1235.5

wp 18761.4

vs 157

sc 0

sc 265

wc 1.30

hzmm 500.00

is 7043.1

rfl 5807.3

rfd 11

th 100.000

ins

nm ph

STANDARD 1H OBSERVE

exp2 stdlh

SAMPLE		DEC. & VT	
date	Feb 21 2006	dfrq	299.942
solvent	CDCl3	dn	H1
file	exp	dpr	30
ACQUISITION		dof	0
sfrq	299.942	dm	nm
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	58		
pw	7.0	weir	
d1	0	wexp	
tof	600.0	wbs	
nt	320	wnt	
ct	320		
alock	n		
gain	not used		
FLAGS			
il	n		
in	n		
dp	Y		
DISPLAY			
sp	-118.1		
wp	4506.3		
vs	210		
sc	0		
wc	265		
hzmm	0.96		
is	484.63		
rfl	118.3		
rfp	0		
th	55		
ins	100.000		
nm	ph

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13C OBSERVE

exp6 std13c

SAMPLE		DEC. & VT	
date	Mar 27 2006	dfrq	299.942
solvent	CDCl3	dn	H1
file	exp	dpr	43
ACQUISITION		dof	0
sfrq	75.428	dm	YYY
tn	C13	dmm	w
at	1.815	dmf	10620
np	68106	PROCESSING	
sw	18761.7	lb	1.00
fb	10400	wtfile	
bs	16	proc	ft
tpwr	54	fn	not used
pw	5.5		
d1	1.000	werr	
tof	600.0	wexp	
nt	40000	whs	
ct	22000	wnt	
alock	n		
gain	28		
FLAGS			
il	n		
in	n		
dp	y		
DISPLAY			
sp	-1231.2		
wp	18761.4		
vs	214		
sc	0		
vc	265		
hmm	3.71		
is	500.00		
rf1	7038.8		
rfp	5807.3		
th	17		
ins	100.000		
nm			
ph

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STANDARD 1H OBSERVE

exp5 std1h

SAMPLE		DEC. & VT	
date	Mar 15 2006	dfrq	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	ft
fb	2600	proc	not used
bs	16	fn	
tpwr	58		
pw	7.0	verr	
dl	0	wexp	
tof	600.0	whs	
nt	320	wnt	
ct	320		
alock	n		
gain	not used		
FLAGS			
il	n		
in	n		
qp	y		
DISPLAY			
sp	-118.1		
vp	4506.3		
vs	631		
sc	0		
wc	265		
hmm	2.89		
is	232.09		
rfl	2295.9		
rfp	2177.6		
th	39		
ins	100.000		
nm	ph

13C OBSERVE

exp6 std13c

SAMPLE DEC. & VT
date Mar 15 2006 dfrq 299.942
solvent CDCl3 dn H1
file exp 43
ACQUISITION dof 0
sfreq 75.428 dm YYY
tn 1.815 dnm w
at 1.815 dmf 10620
np 68106
sw 18761.7 lb PROCESSING 1.00
fb 10400 wfile
bs 16 proc
tpwr 54 fn not used
pw 5.5
d1 0.500 weir
tof 600.0 wexp wft
nt 5000 wbs wft
ct 4512 wnt
alock n
gain 28
FLAGS
il n
in n
dp y
DISPLAY
sp -1234.6
wp 18761.4
vs 157
sc 0
wc 265
hzmm 22.92
is 500.00
xf1 7042.2
rfd 5807.3
th 12
ins 100.000
nm ph

STANDARD 1H OBSERVE

exp1 std1h

SAMPLE DEC. & VT
 date Mar 31 2006 dfreq 299.942
 solvent CDCl3 dn H1
 file exp 30
 ACQUISITION dpwr 0
 dof 0
 sfreq 299.942 dm nnn
 tn H1 dnm c
 at 1.998 dmf 200
 np 18008
 sw 4506.5 lb PROCESSING 0.30
 fb 2600 wfile
 bs 16 proc ft
 tpwr 58 fn not used
 pw 7.0
 dl 0 weir
 tof 600.0 wexp
 nt 320 wbs
 ct 320 wnt
 alock n
 gain not used
 flags
 il n
 in n
 dp Y
 DISPLAY
 sp -118.3
 wp 4506.3
 vs 1558
 sc 0
 wc 265
 hzmm 4.24
 is 435.91
 rfl 2296.2
 rfp 2177.6
 th 32
 ins 100.000
 nm ph

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STANDARD 1H OBSERVE

exp2 stdlh

SAMPLE		DEC. & VT	
date	Feb 8 2006	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	dmf	200
up	18008	PROCESSING	
sw	4506.5	lb	0.30
fb	2600	wtfile	
bs	16	proc	ft
tpwr	58	fn	not used
pw	7.0	werr	0
dl	600.0	wexp	
nt	320	wbs	
ct	320	wnt	
alock	n		
gain	not used		
FLAGS			
il	n		
in	n		
dp	y		
DISPLAY			
sp	-121.6		
vp	4506.3		
vs	163		
sc	0		
wc	265		
hzmm	7.75		
ls	234.89		
xf1	121.9		
xfp	0		
th	19		
ins	100.000		
nm	ph		

13C OBSERVE

exp3 std13c

SAMPLE		DEC. & VT	
date	Feb 8 2006	dirq	299.942
solvent	CDCl3	dn	M1
file	exp	dpwr	43
ACQUISITION		dof	0
sfreq	75.428	dm	YVY
tn	C13	dmm	W
at	1.815	dmf	10620
np	68106	PROCESSING	
sw	18761.7	lb	1.00
fb	10400	wfile	
bs	16	proc	ft
tpwr	54	fn	not used
pw	5.5		
dl	0.500	werr	
tof	600.0	wexp	
nt	10000	wbs	
ct	3008	wnt	
alock	n		
gain	28		
FLAGS			
il	n		
in	n		
dp	Y		
DISPLAY			
sp	-1234.9		
wp	18761.4		
vs	163		
sc	0		
wc	265		
hzmm	1.67		
is	500.00		
rfl	7042.5		
rfd	5807.3		
th	11		
ins	100.000		
nm			
ph

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STANDARD 1H OBSERVE

```

exp5 stdih
SAMPLE
date Mar 23 2006 dfrq 299.942
solvent CDCl3 dn H1
file exp 30 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 58
pw 7.0 werr
dl 0 wexp
tof 600.0 vba
nt 320 wnt
ct 320
alock n
gain not used
FLAGS
il n
in n
dp y
DISPLAY
sp -119.4
vp 4506.3
vs 243
sc 0
wc 265
hzmm 0.47
is 280.16
rf1 2297.3
rfp 2177.6
th 36
ins 100.000
nm ph
  
```

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STANDARD 1H OBSERVE

```

exp1 stdlh
SAMPLE DEC. & VT
date Feb 3 2006 dfrq 299.942
solvent CDCl3 dn H1
file exp 30
ACQUISITION dbr 0
sfrq 299.942 dm nm
tn H1 dm c
at 1.998 dmf 200
np 18008 PROCESSING
sw 4506.5 wfile
fb 2600 proc ft
bs 16 fn not used
tpwr 58
pw 7.0 werr
d1 0 wexp
tof 600.0 wbs
nt 320 wnt
ct 320
alock n
gain not used
FLAGS
il n
in n
dp y
DISPLAY
sp -117.5
wp 4506.3
vs 354
sc 0
wc 265
hzmm 3.22
is 343.86
rf1 117.8
rfp 0
th 39
ins 100.000
nm ph
  
```

413C OBSERVE

exp2 std13c

SAMPLE		DEC. & V _F	
date	Mar 13 2006	dfrq	299.942
solvent	CDCl ₃	dn	H1
file	exp	dpwr	43
ACQUISITION		dof	0
sfrq	75.428	dm	YNY
tn	C13	dmm	W
at	1.815	daf	10620
np	68106	PROCESSING	
sw	18761.7	lb	1.00
fb	10400	wtfile	
bs	16	proc	ft
tpwr	54	fn	not used
pw	5.5		
dl	1.000	werr	
tof	600.0	wexp	wft
nt	5000	wba	wft
ct	5000	wnt	
alock	n		
Gain	28		
FLAGS			
il	n		
in	n		
dp	Y		
DISPLAY			
sp	-1234.3		
wp	18761.4		
vs	169		
sc	0		
wc	265		
hzmm	17.42		
is	500.00		
rfl	7041.9		
xfp	5807.3		
th	23		
ins	100.000		
rm	ph

STANDARD 1H OBSERVE

```

exp1 stdlh
SAMPLE DEC. & VT
date Mar 24 2006 dfrq 299.942
solvent CDCl3 dn H1
file exp dpr 30
ACQUISITION exp dpr 0
sfreq 299.942 dm nnn
tn H1 dnm c
at 1.998 dmf 200
np 18008
sw 4506.5 lb 0.30
fb 2600 wtfile
bs 16 proc ft
tpwr 58 fn not used
pw 7.0
d1 0 werr
tof 600.0 wexp
nt 320 wbs
ct 320 wnt
alock n
gain not used
FLAGS
il n
in n
cp K
DISPLAY
sp -118.9
wp 4506.3
vs 183
sc 0
wc 265
hzmm 0.71
is 402.65
rfl 2296.7
rfd 2177.6
th 38
ins 100.000
nm ph
  
```

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18

```

13C OBSERVE
exp2 std13c
SAMPLE DEC. & VT
date Mar 24 2006 dfrq 299.942
solvent CDCl3 dn H1
file exp dpr 43
ACQUISITION dof 0
sfrq 75.428 dm YYY
tn C13 dnm w
at 1.815 dmf 10620
np 68106
sw 18761.7 lb PROCESSING
fb 10400 wtfile 1.00
bs 16 proc ft
tpwr 54 fn not used
pw 5.5
d1 0.500 werr
tof 600.0 wexp
nt 4000 wbs
ct 4000 wnt
alock n
gain 28
FLAGS
il n
in n
dp Y
DISPLAY
sp -1232.0
wp 18761.4
vs 192
sc 0
wc 265
hzmm 1.85
is 500.00
rf1 7039.7
rfp 5807.3
th 29
ins 100.000
nm ph

```

19

19

19F OBSERVE
STANDARD PARAMETERS

exp3 s2pul

SAMPLE		DEC. & VT	
date	Mar 29 2006	dfrq	299.942
solvent	CDCl3	dn	H1
file	exp	dpwr	30
ACQUISITION		dof	0
sfrq	282.191	dm	nmn
tn	F19	dmm	c
at	0.300	dmf	200
np	29952	PROCESSING	
sw	50000.0	wtfile	ft
fb	27600	proc	not used
bs	4	fn	
tpwr	60		
pw	10.0	werr	
d1	4.000	wexp	
tof	-5500.0	whs	
nt	320	wnt	
ct	224		
alock	n		
gain	2		
FLAGS			
il	n		
in	n		
dp	y		
DISPLAY			
sp	-287.6		
wp	49996.9		
vs	444		
sc	0		
wc	265		
hzmm	3.36		
is	57721.13		
zfl	14068.6		
rfd	13797.9		
th	41		
ins	100.000		
nm	ph

19

-50 -70 -90 -110 -130 -150 -170 -190 ppm

STANDARD 1H OBSERVE

exp2 std1h

SAMPLE DEC. & VT
date Feb 9 2006 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 PROCESSING
sw 4506.5 wfile
fb 2600 proc ft
bs 16 fn not used
tpwr 58
pw 7.0 weir
d1 0 wexp
tof 600.0 wbs
nt 320 wnt
ct 320
alock n
Gain not used
FLAGS
il n
in n
dp y
DISPLAY
sp -117.8
wp 4506.3
vs 159
sc 0
wc 265
hazmm 0.21
is 433.19
rfl 118.1
rfd 0
th 46
ins 100.000
nm ph

20

20

13C OBSERVE

exp3 std13c

SAMPLE		DEC. & VT	
date	Feb 9 2006	dfrq	299.942
solvent	CDCl ₃	dn	H1
file	exp	dpwr	43
ACQUISITION		dof	0
sfrq	75.428	dm	YYY
tn	CL3	dmm	W
at	1.815	dmf	10620
up	68106	PROCESSING	
sw	18761.7	lb	1.00
fb	10400	wtfile	
bs	16	proc	ft
tpwr	54	fn	not used
pw	5.5		
dl	0.500	werr	
tof	600.0	wexp	
nt	10000	whs	
ct	7200	wnt	
alock	n		
gain	28		
FLAGS			
il	n		
in	n		
dp	Y		
DISPLAY			
sp	-1230.0		
vp	18761.4		
vs	162		
sc	0		
wc	265		
harm	3.33		
is	500.00		
rf1	7037.7		
rfp	5807.3		
th	15		
ins	100.000		
nm	ph		

19F OBSERVE
STANDARD PARAMETERS

exp3 s2pul

SAMPLE		DEC. & VT	
date	Mar 29 2006	dfrq	299.942
solvent	CDCl3	dn	H1
file	exp	dpwr	30
ACQUISITION		dof	0
sfrq	282.191	dm	mm
tn	F19	dm	c
at	0.300	dmf	200
np	29952	PROCESSING	
sw	50000.0	lb	2.00
fb	27600	wtfile	
bs	4	proc	ft
tpwr	60	fn	not used
pw	10.0		
d1	4.000	verr	
tof	-5500.0	vexp	
nt	320	whs	
ct	224	wnt	
alock	n		
gain	2		
FLAGS			
il	n		
in	n		
qp	y		
DISPLAY			
sp	-240.1		
wp	49996.9		
vs	1097		
sc	0		
wc	265		
harm	4.08		
is	3000.00		
xf1	14041.1		
xfp	13797.9		
th	85		
ins	100.000		
nm	ph

20

-50 -70 -90 -110 -130 -150 -170 -190 ppm

STANDARD 1H OBSERVE

```

exp1 std1h
SAMPLE DEC. & VT
date Mar 10 2006 dfreq 299.942
solvent CDCl3 dn H1
file exp 30
ACQUISITION dof 0
nuc1 299.942 dm nnn
tn H1 dnm c
at 1.998 dmf 200
nd 18008
sw 4506.5 wfile
fb 2600 proc ft
bs 16 fn not used
tpwr 58
pw 7.0 werr
dl 0 wexp
tof 600.0 vbs
nt 320 wnt
ct 320
alock n
gain not used
FLAGS
il n
in n
cp y
DISPLAY
sp -116.7
wp 4506.3
vs 776
sc 0
wc 265
hzmm 1.68
is 275.29
rf1 117.0
rfp 0
th 35
ins 100.000
nm ph

```

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13C OBSERVE

exp2 std13c

SAMPLE		DEC. & VT	
date	Mar 10 2006	dfrq	299.942
solvent	CDCl3	dn	H1
file	exp	dpr	43
ACQUISITION		dof	0
sfrq	75.428	dm	YYY
tn	Cl3	dmm	
at	1.815	daf	10620
np	68106	PROCESSING	
sw	18761.7	lb	1.00
fb	10400	wfile	
bs	16	proc	ft
tpwr	54	fn	not used
pw	5.5		
dl	0.500	werr	
tof	600.0	wexp	
nt	5000	whs	
ct	4016	wnt	
alock	n		
gain	28		
FLAGS			
il	n		
in	n		
dp	y		
DISPLAY			
sp	-1230.9		
wp	18761.4		
vs	484		
sc	0		
wc	265		
hmm	22.41		
is	500.00		
rfl	7038.5		
rfp	5807.3		
th	23		
ins	100.000		
nm	ph