Different C-glycosidation products of glucal with alkynyl or propargyl silanes under acidic conditions

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Supporting Information

Experimental Section

entry	Lewis acid	solvent	time	(%)yield of 5+6	5/6
1	BF ₃ ·OEt ₂	CH ₂ Cl ₂	1.0 h	74	1 : 2.5
2	BF ₃ ·OEt ₂	CH ₂ Cl ₂	1.5 h	87	1 : 2.5
3	SnCl ₄	CH ₂ Cl ₂	15 min	83	1 : 2
4	SnCl ₄	CH ₃ CN	15 min	60	4 : 1

To a solution of **1** (0.05 g, 0.18 mmol) and 1,3-bis(trimethylsilyl)propyne **2** (0.08 g, 0.37 mmol) in 35 ml. CH₂Cl₂ was added dropwise the solution of SnCl₄ (0.02 ml, 0.18 mmol) in 5 ml CH₂Cl₂ at –20°C under argon atmosphere. The mixture was stirred for 15 min. and then poured into the mixture of saturated sodium hydrogen carbonate and saturated potassium sodium tartrate (1vol./1 vol.). After stirring at 0°C for 30 min, the organic layer was separated and the water layer was extracted with CH₂Cl₂. The combined organic layer was washed with water, brine, dried over anhydrous sodium sulfate and concentrated in vacuo. The residue was purified by silica gel column chromatography (10% EtOAc/hexane) to afford a mixture of compound **3** and **4**.

[RNS-59-2, 49-2-2]

Compound 3. $[\alpha]_D^{25}$ 62.8° ($c = 0.06$, CHCl_3). IR (KBr) : 2955, 2926, 2854, 1933, 1744, 1370, 1233, 1047, 844 cm^{-1} . $^1\text{H-NMR}$ (500 MHz, CDCl_3) : δ 0.11 (9H, s, SiMe_3), 2.05 (3H, s, Ac), 2.06 (3H, s, Ac), 3.74 (1H, ddd, $J = 9.0, 6.0, 2.5$ Hz, H-5), 4.12 (1H, dd, $J = 12.0, 6$ Hz, H-6a), 4.22 (1H, dd, $J = 12.0, 2.5$ Hz, H-6b), 4.46 (1H, dd, $J = 12.0, 2.5$ Hz, H-3'a), 4.50 (1H, dd, $J = 12.0, 2.5$ Hz, H-3'b), 4.85 (1H, m, H-1), 5.29 (1H, ddd, $J = 9.0, 4.0, 2.0$ Hz, H-4), 5.71 (1H, dt, $J = 10.0, 2.0$ Hz, H-2), 5.93 (1H, dt, $J = 10.0, 2.0$ Hz, H-3). $^{13}\text{C-NMR}$ (100 MHz, CDCl_3) : δ -0.64, 20.8, 63.5, 65.5, 70.9, 74.4, 75.4, 97.0, 124.5, 133.1, 170.3, 170.9, 208.9. Anal. Calcd. For $\text{C}_{16}\text{H}_{24}\text{O}_5\text{Si}$: C, 59.23; H, 7.46 Found: C, 59.39; H, 7.50.

[RA-20-3 = RNS-21]

Compound 4. $[\alpha]_D^{26}$ -64.0° ($c = 0.84$, CHCl_3). IR (KBr) : 2956, 2891, 2211, 1743, 1370, 1235, 1051, 853, 699 cm^{-1} . $^1\text{H-NMR}$ (500 MHz, CDCl_3) : δ 0.09 (9H, s, SiMe_3), 1.49 (2H, d, $J = 2.0$ Hz, H-3'), 2.06 (3H, s, Ac), 2.07 (3H, s, Ac), 4.09 (1H, dt, $J = 9.0, 4.0$ Hz, H-5), 4.18 (2H, d, $J = 4.0$ Hz, H-6), 4.92 (1H, brs, H-1), 5.24 (1H, ddd, $J = 9.0, 4.0, 2.0$ Hz, H-4), 5.68 (1H, dt, $J = 10.0, 2.0$ Hz, H-2), 5.84 (1H, ddd, $J = 10.0, 4.0, 2.0$ Hz, H-3). $^{13}\text{C-NMR}$ (125 MHz, CDCl_3) : δ -2.1, 7.2, 20.8, 20.9, 63.3, 64.5, 65.0, 69.5, 74.6, 85.9, 124.4, 130.4, 170.3, 170.9. Anal. Calcd. For $\text{C}_{16}\text{H}_{24}\text{O}_5\text{Si}$: C, 59.23; H, 7.46 Found: C, 59.38; H, 7.16.

To a solution of **1** (0.05 g, 0.18 mmol) and 1-trimethylsilyl-3-triisopropylsilylpropyne **8** (0.06 g, 0.22 mmol) in 35 ml. CH_2Cl_2 was added dropwise the solution of SnCl_4 (0.02 ml, 0.18 mmol) in 5 ml CH_2Cl_2 at -25°C under argon atmosphere. The mixture was stirred for 15 min. and then poured into the mixture of saturated sodium hydrogen carbonate and saturated potassium sodium tartrate (1vol./1 vol.). After stirring at 0°C for 30 min, the organic layer was separated and the water layer was extracted with CH_2Cl_2 . The combined organic layer was washed with water, brine, dried over anhydrous sodium sulfate and concentrated in vacuo. The residue was purified by silica gel column chromatography (10% EtOAc/hexane) to afford the product **9**.

[RNS-28]

Compound 9. $[\alpha]_D^{26} -53.0^\circ$ ($c = 0.59$, CHCl_3). IR (KBr): 2943, 2867, 2212, 1747, 1465, 1369, 1240, 1054, 883 cm^{-1} . $^1\text{H-NMR}$ (500 MHz, CDCl_3): δ 1.06, 1.07 (18H, s, $3\times\text{SiCH}(\text{CH}_3)_2$), 1.12 (3H, m, $3\times\text{SiCHMe}_2$), 1.56 (2H, d, $J = 2.5$ Hz, H-3'), 2.06 (3H, s, Ac), 2.07 (3H, s, Ac), 4.08 (1H, ddd, $J = 9.0, 5.0, 2.5$ Hz, H-5), 4.14 (1H, dd, $J = 12.0, 2.5$ Hz, H-6a), 4.23 (1H, dd, $J = 12.0, 5.0$ Hz, H-6b), 4.90 (1H, brs, H-1), 5.26 (1H, ddd, $J = 9.0, 4.0, 2.0$ Hz, H-4), 5.68 (1H, dt, $J = 10.0, 2.0$ Hz, H-2), 5.83 (1H, ddd, $J = 10.0, 4.0, 2.0$ Hz, H-3). $^{13}\text{C-NMR}$ (75 MHz, CDCl_3): δ -0.9, 10.9, 18.4, 20.7, 20.9, 63.2, 64.5, 64.9, 69.4, 74.4, 86.7, 124.3, 130.4, 170.5, 171.1. Anal. Calcd. For $\text{C}_{22}\text{H}_{36}\text{O}_5\text{Si}$: C, 64.67; H, 8.88 Found: C, 64.67; H, 8.91

entry	equiv. of 4	Lewis acid	solvent	time	(%) yield of 8+9	8/9
1	2	SnCl ₄	CH ₂ Cl ₂	15 min	74	1 : 2.5
2	1.1	TiCl ₄	CH ₂ Cl ₂	15 min	87	1 : 2.5
3	1.1	SnCl ₄	CH ₂ Cl ₂	45 min	69	1 : 5
4	1.1	SnCl ₄	CH ₃ CN	1 h	58	1 : 9
5	1.1	SnCl ₄	CH ₃ CN	15 min	60	1 : 9
6	0.5	SnCl ₄	CH ₃ CN	1.5 h	30	1 : 80

To a solution of **1** (0.05 g, 0.18 mmol) and 1-triisopropylsilyl-3-trimethylsilylpropyne **5** (0.10 g, 0.37 mmol) in 35 ml. CH₂Cl₂ was added dropwise the solution of SnCl₄ (0.02 ml, 0.18 mmol) in 5 ml CH₂Cl₂ at –20°C under argon atmosphere. The mixture was stirred for 15 min. and then poured into the mixture of saturated sodium hydrogen carbonate and saturated potassium sodium tartrate (1vol./1 vol.). After stirring at 0°C for 30 min, the organic layer was separated and the water layer was extracted with CH₂Cl₂. The combined organic layer was washed with water, brine, dried over anhydrous sodium sulfate and concentrated in vacuo. The residue was purified by silica gel column chromatography (10% EtOAc/hexane) to afford a mixture of compound **6** and **7**.

[RNS-52-1-2]

Compound 6. $[\alpha]_D^{27} -22.9^\circ$ (c = 0.24, CHCl₃). IR (KBr) : 2943, 2866, 1922, 1747, 1237, 1049, 804 cm⁻¹. ¹H-NMR (500 MHz, CDCl₃) : δ 1.06, 1.07, 1.08, 1.09 (18H, s, 3xSiCH(CH₃)₂), 1.25 (3H, m, 3xSiCHMe₂), 2.04 (3H, s, Ac), 2.06 (3H, s, Ac), 3.96 (1H, ddd, *J* = 8.5, 5.5, 2.5 Hz, H-5), 4.10 (1H, dd, *J* = 12.0, 2.5 Hz, H-6a), 4.15 (1H, dd, *J* = 12.0, 5.5 Hz, H-6b), 4.50 (2H, m, H-3'), 4.77 (1H, m, H-1), 5.20 (1H, ddd, *J* = 10.0, 3.0, 1.5 Hz, H-4), 5.79 (1H, dt, *J* = 10.5, 1.5 Hz, H-2), 5.85 (1H, ddd, *J* = 10.5, 3.0, 1.5 Hz, H-3).

¹³C-NMR (75 MHz, CDCl₃): δ 11.9, 12.1, 18.5, 18.6, 20.8, 21.1, 29.7, 63.4, 65.4, 67.8, 70.6,

71.2, 74.5, 90.1, 124.0, 131.6, 170.4, 171.0, 213.4.

Anal. Calcd. For $C_{22}H_{36}O_5Si$: C, 64.67; H, 8.88 Found: C, 64.67; H, 8.93

[RNS-57-P]

Compound 7. $[\alpha]_D^{27} -15.0^\circ$ ($c = 1.45$, $CHCl_3$). IR (KBr): 2942, 2865, 2175, 1747, 1464, 1369, 1233, 1022, 883, 678 cm^{-1} . 1H -NMR (500 MHz, $CDCl_3$): δ 1.02, 1.03 (18H, s, $3 \times SiCH(CH_3)_2$), 1.03 (3H, m, $3 \times SiCHMe_2$), 2.05 (3H, s, Ac), 2.07 (3H, s, Ac), 2.57 (1H, dd, $J = 16.5, 8.0$ Hz, H-1'a), 2.70 (1H, dd, $J = 16.5, 5.0$ Hz, H-1'b), 4.07 (1H, td, $J = 6.0, 3.5$ Hz, H-5), 4.14 (1H, dd, $J = 12.0, 3.5$ Hz, H-6a), 4.21 (1H, dd, $J = 12.0, 6.0$ Hz, H-6b), 4.36 (1H, m, H-1), 5.12 (1H, ddd, $J = 6.0, 4.5, 2.5$ Hz, H-4), 5.85 (1H, dt, $J = 10.5, 2.5$ Hz, H-2), 6.10 (1H, ddd, $J = 10.5, 2.5, 2.0$ Hz, H-3). ^{13}C -NMR (75 MHz, $CDCl_3$): δ 11.2, 18.6, 20.8, 21.0, 62.7, 64.8, 70.5, 83.7, 103.8, 124.6, 131.7, 170.4, 170.8

Anal. Calcd. For $C_{22}H_{36}O_5Si$: C, 64.67; H, 8.88 Found: C, 64.68; H, 8.94

To a solution of **1** (0.10 g, 0.37 mmol) and 1,3-bis(trimethylsilyl)propyne **2** (0.04 g, 0.19 mmol) in 35 ml. CH₂Cl₂ was added dropwise the solution of SnCl₄ (0.04 ml, 0.37 mmol) in 5 ml CH₂Cl₂ at -20°C under argon atmosphere. The mixture was stirred for 40 min. and then poured into the mixture of saturated sodium hydrogen carbonate and saturated potassium sodium tartrate (1vol./1 vol.). After stirring at 0°C for 30 min, the organic layer was separated and the water layer was extracted with CH₂Cl₂. The combined organic layer was washed with water, brine, dried over anhydrous sodium sulfate and concentrated in vacuo. The residue was purified by silica gel column chromatography (30-50% EtO₂/hexane) to afford a mixture of compound **10**, **11**, **12** and **13**.

[RNS-55-1, RNS-59-1]

Compound 10. IR (KBr) : 2957, 1743, 1606, 1370, 1236, 843 cm⁻¹. ¹H-NMR (500 MHz, CDCl₃) : δ 0.14 (9H, s, SiMe₃), 2.06 (3H, s, Ac), 2.07 (3H, s, Ac), 2.61 (1H, dd, *J* = 14.0, 7.0 Hz, H-1'a), 2.75 (1H, dd, *J* = 16.5, 7.5 Hz, H-1'b), 3.95 (1H, m, H-5), 4.12 (1H, dd, *J* = 12.0, 4.0 Hz, H-6a), 4.28 (1H, dd, *J* = 12.0, 6.5 Hz, H-6b), 4.61 (1H, m, H-1), 5.10 (1H, m, H-4), 5.85 (1H, ddd, *J* = 10.0, 3.5, 2.0 Hz, H-2), 5.96 (1H, ddd, *J* = 10.0, 2.0, 1.5 Hz, H-3).

[RNS-49-3, RNS-55-2]

Compound 11. [α]_D²⁵ +29.4° (c = 0.62, CHCl₃). IR (KBr) : 3284, 2923, 1740, 1370, 1234, 1046 cm⁻¹. ¹H-NMR (500 MHz, CDCl₃) : δ 2.04 (1H, t, *J* = 2.5 Hz, H-3'), 2.06 (3H, s, Ac), 2.07 (3H, s, Ac), 2.47 (1H, ddd, *J* = 16.5, 7.5, 2.5 Hz, H-1'a), 2.58 (1H, ddd, *J* = 16.5, 6.5, 2.5 Hz, H-1'b), 3.98

(1H, ddd, $J = 9.0, 6.5, 3.5$ Hz, H-5), 4.14 (1H, dd, $J = 12.0, 3.5$ Hz, H-6a), 4.24 (1H, dd, $J = 12.0, 6.5$ Hz, H-6b), 4.38 (1H, m, H-1), 5.12 (1H, m, H-4), 5.86 (1H, ddd, $J = 10.0, 3.0, 2.0$ Hz, H-2), 6.07 (1H, ddd, $J = 10.0, 2.5, 1.5$ Hz, H-3). ^{13}C -NMR (75 MHz, CDCl_3): δ 20.8, 21.1, 23.9, 62.6, 64.7, 69.9, 70.4, 70.7, 79.8, 124.6, 131.6, 170.4, 170.8. Anal. Calcd. For $\text{C}_{13}\text{H}_{16}\text{O}_5$: C, 61.90; H, 6.39 Found: C, 61.93; H, 6.36

12

[RNS-55-3]

Compound 12. $[\alpha]_{\text{D}}^{27} -145.0^\circ$ ($c = 1.1$, CHCl_3). IR (KBr) : 2952, 1953, 1740, 1371, 1235, 1049 cm^{-1} . ^1H -NMR (500 MHz, CDCl_3) : δ 2.06 (3H, s, Ac), 2.07 (3H, s, Ac), 3.80 (1H, ddd, $J = 9.0, 5.0, 4.0$ Hz, H-5), 4.14 (2H, m, H-6), 4.95 (1H, m, H-1), 5.24 (1H, ddd, $J = 9.0, 3.0, 2.0$ Hz, H-4), 5.79 (1H, dt, $J = 10.5, 2.0$ Hz, H-2), 5.88 (1H, ddd, $J = 10.5, 3.0, 2.0$ Hz, H-3). ^{13}C -NMR (75 MHz, CDCl_3): δ 20.7, 20.9, 63.3, 65.5, 68.5, 70.3, 78.8, 101.1, 125.6, 130.1, 170.3, 170.7, 208.6.

Anal. Calcd. For $\text{C}_{23}\text{H}_{28}\text{O}_{10}$: C, 59.48; H, 6.08 Found: C, 59.49; H, 6.16

13

[RNS-55-4]

Compound 13. $[\alpha]_{\text{D}}^{27} +20.2^\circ$ ($c = 0.35$, CHCl_3). IR (KBr) : 2922, 1740, 1372, 1234, 1049 cm^{-1} . ^1H -NMR (500 MHz, CDCl_3) : δ 2.05 (3H, s, Ac), 2.06 (3H, s, Ac), 2.07 (3H, s, Ac), 2.08 (3H, s, Ac), 2.52 (1H, ddd, $J = 17.0, 8.0, 2.0$ Hz, H-9a), 2.63 (1H, ddd, $J = 17.0, 6.0, 2.0$ Hz, H-9b), 3.95

(1H, m, H-14), 4.05 (1H, ddd, $J = 9.0, 4.5, 3.0$ Hz, H-2), 4.13 (1H, dd, $J = 12.0, 3.5$ Hz, H-15a), 4.14 (1H, m, H-1a), 4.22 (1H, m, H-1b), 4.24 (1H, dd, $J = 12.0, 6.0$ Hz, H-15b), 4.36 (1H, ddt, $J = 8.0, 6.0, 2.0$ Hz, H-10), 4.96 (1H, m, H-6), 5.12 (1H, m, H-13), 5.27 (1H, ddd, $J = 9.0, 4.0, 2.0$ Hz, H-3), 5.74 (1H, dt, $J = 10.0, 2.0$ Hz, H-4), 5.85 (1H, ddd, $J = 10.0, 3.5, 2.0$ Hz, H-5), 5.86 (1H, ddd, $J = 10.0, 5.0, 3.0$ Hz, H-5), 6.06 (1H, ddd, $J = 10.0, 2.5, 1.5$ Hz, H-3).

Anal. Calcd. For $C_{23}H_{28}O_{10}$: C, 59.48; H, 6.08 Found: C, 59.48; H, 6.27

7.2620
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OBNUC : 1H
OBFRQ : 500.00 MHz
OBSET : 162410.00 Hz
RGAIN : 26

SCANS : 32 times

SLVNT : CDCL3
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 AQ: 1.5661750 sec
 RG: 18596.5
 DQ: 20.856 nsec
 DE: 6.00 nsec
 TE: 300.0 K
 D1: 2.00000000 sec
 d11: 0.05000000 sec
 d12: 0.00000000 sec

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 NUC1: 13C
 P1: 9.00 nsec
 PL1: -1.00 dB
 SFO1: 100.6228200 MHz

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 CPDPRG2: waltz16
 NUC2: 1H
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 PL2: -1.00 dB
 PL12: -1.00 dB
 PL13: -1.00 dB
 SFO2: 400.1316000 MHz

F2 - Processing parameters
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 LB: 1.00 Hz
 GB: 0
 PC: 1.00

RA-20-3

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 INTVL : 125.0 usec
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 DUMMY : 1 times
 PD : 4.9520 sec
 ACQTM : 4096.0000 msec
 PREOL : 0.01000 msec
 INIWT : 1000.0000 msec
 RESOL : 0.24 Hz
 PW1 : 5.50 usec

OBNUC : 1H
 OBFRQ : 500.00 MHz
 OBSET : 162410.00 Hz
 RGAIN : 16

SCANS : 8 times

SLVNT : COCL3
 SPINNING : 12 Hz
 TEMP : 26.8 C

RA-20-3

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 RESOL : 0.92 Hz
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 OBFRQ : 125.65 MHz
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 RGAIN : 32
 IRNUC : 1H
 IRFRQ : 500.00 MHz
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 IRRPW : 50.0 usec
 IRRNS : 0

SCANS : 128 times

SLVNT : CDCL3
 SPINNING : 12 Hz
 TEMP : 28.5 C

expt stdih

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sfrq	299.967	dma	0
tn	H1	daf	200
PROCESSING			
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sw	4499.5	proc	not used
fb	2250	fn	
bs	16		
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tof	149.3	ext')	'hl_process_0-
nt	16		135')
ct	16	wbs	
alock	n	wat	
gain	not used		
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in	n		
dp	y		
DISPLAY			
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wp	3119.7		
vs	2035		
sc	0		
wc	400		
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th	2		
ins	1.000		
ai	cdc ph

expi std1h

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sw 4499.5 proc ft
fb 2250 fa not used
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dl 0.252 wamp autolist('ant-
tof 149.3 exV', 'hi_process_J-
nt 16 136')
ct 16 vbs
alock n wnt
gain not used
FLAGS
il n
in n
dp Y
DISPLAY
sp -60.0
wp 3119.7
vs 858
sc 0
wc 400
hnm 7.80
is 13956.52
rf1 598.3
rfp 0
th 3
ins 1.000
al cdc ph

Solvent: CDCl₃
 Ambient temperature
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 Pulse 35.0 degrees
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 DECOUPLE H1, 299.9672195 MHz
 Power 30 dB
 continuously on
 WALTZ-16 modulated
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 Line broadening 2.0 Hz
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 Total time 10 minutes

TIPSacetylene-glucal

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 INTVL : 125.0 usec
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 DUMMY : 1 times
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 ACQTM : 4096.0000 msec
 PREDL : 0.01000 msec
 INIWT : 1000.0000 msec
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 OBNUC : 1H
 OBFRQ : 500.00 MHz
 OBSET : 162410.00 Hz
 RGAIN : 22

SCANS : 8 times

SLVNT : CDCL3
 SPINNING : 13 Hz
 TEMP : 27.9 C

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 ambient temperature
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 acq. time 1.697 sec
 width 18858.5 Hz
 36 repetitions
 SKEW C13, 75.4264040 MHz
 COUPLE H1, 299.9672195 MHz
 over 10 dB
 continuously on
 ALTA-16 modulated
 TA PROCESSING
 line broadening 1.0 Hz
 size 65536
 total time 9 minutes

Date : Thu May 22 01:48:49 2003

FileName : LoadingFID.nmdata
Comment : RNS-55-1
SliceHistory :
EXMODE : non

POINT : 32768 points
SAMPD : 32768 points
FREQU : 8000.0 Hz
FILTR : 4000 Hz
DELAY : 50.0 usec
DEAD : 72.3 usec
INTVL : 125.0 usec
TIMES : 8 times
DUMMY : 1 times
PD : 4.9520 sec
ACQTM : 4096.0000 msec
PREDL : 0.01000 msec
ININT : 1000.0000 msec
RESOL : 0.24 Hz
PW1 : 5.50 usec
OBNUC : 1H
OBFRQ : 500.00 MHz
OBSET : 162410.00 Hz
RGAIN : 22

SCANS : 8 times

SLVNT : CDCL3
SPINNING : 10 Hz
TEMP : 27.3 C

RMS-95-1

1H S/N

Date : Fri May 23 03:23:15 2003

FileName : LoadingFID.nmdata
Comment : 1H S/N
SliceHistory :
EXMODE : bcm

POINT : 32768 points
SAMPD : 32768 points
FREQ : 30030.0 Hz
FILTR : 15000 Hz
DELAY : 13.3 usec
DEADT : 17.3 usec
INTVL : 33.3 usec
TIMES : 768 times
DUMMY : 1 times
PD : 1.9088 sec
ACQTM : 1091.1743 msec
PREDL : 0.01000 msec
INIWT : 1000.0000 msec
RESOL : 0.92 Hz
PW1 : 5.50 usec

OBNUC : 13C
OBFRQ : 125.65 MHz
OBSET : 127958.00 Hz
RGAIN : 31
IRNUC : 1H
IRFRQ : 500.00 MHz
IRSET : 162410.00 Hz
IARPW : 50.0 usec
IARNS : 0

SCANS : 768 times

SLVNT : CDCL3
SPINNING : 13 Hz
TEMP : 29.7 C

Date : Wed Apr 30 07:17:40 2003

FileName : LoadingFID.nmdata
Comment : RNS-49-3
SliceHistory :
EXMODE : non

POINT : 32768 points
SAMPD : 32768 points
FREQ : 8000.0 Hz
FILTR : 4000 Hz
DELAY : 50.0 usec
DEADT : 72.3 usec
INTVL : 125.0 usec
TIMES : 8 times
DUMMY : 1 times
PD : 4.9520 sec
ACQTM : 4096.0000 msec
PREDL : 0.01000 msec
INIWT : 1000.0000 msec
RESOL : 0.24 Hz
PW1 : 5.50 usec
OBNUC : 1H
OBFRQ : 500.00 MHz
OBSET : 162410.00 Hz
RGAIN : 20

SCANS : 8 times

SLVNT : CDCL3
SPINNING : 12 Hz
TEMP : 465.0 C

RNS-49-3

Date : Wed Apr 30 07:50:38 2003

FileName : LoadingFID.nmdata
 Comment : RNS-49-3
 SliceHistory :
 EXMODE : bcm

POINT : 32768 points
 SAMPO : 32768 points
 FREQU : 30030.0 Hz
 FILTR : 15000 Hz
 DELAY : 13.3 usec
 DEADT : 17.3 usec
 INTVL : 33.3 usec
 TIMES : 512 times
 DUMMY : 1 times
 PD : 1.9088 sec
 ACGTM : 1091.1743 msec
 PREDL : 0.01000 msec
 ININT : 1000.0000 msec
 RESOL : 0.92 Hz
 PW1 : 5.50 usec
 OBNUC : 13C
 OBFRQ : 125.65 MHz
 OBSET : 127958.00 Hz
 RGAIN : 31
 IRNUC : 1H
 IRFRQ : 500.00 MHz
 IRSET : 162410.00 Hz
 IRRPW : 50.0 usec
 IRRNS : 0

SCANS : 512 times

SLVNT : CDCL3
 SPINNING : 12 Hz
 TEMP : 465.0 C

1H.NOESY NON RUNA_MAY16 EN1

16-MAY-2003 15:08:27.37

DFILE : \$ALPHA2
SFIL: RUNA_MAY162NOESY_E1
HR2FILE: RUNA_MAY161NON_E1
HR1FILE: RUNA_MAY161NON_E1

COMNT : 1H NOESY NON RUNA_MAY16 EN1
EXMOD : NOESY
IRMOD : NON
POINT : 512
FREQ: 5189.41 Hz
SCANS : 16
DUMMY : 4
AQTM : 0.0493 sec
PD : 2.4507 sec
RGAIN : 13

CLFRQ : 5189.41 Hz
CLPNT : 512
TOSCN : 256
CINWT : 10.00 usec
CINTV : 192.70 usec

PW1 : 12.90 usec
PW2 : 20.00 usec
PI1 : 500.0000 msec
PI2 : 1.0000 msec

OBNUC : 1H
OBFRQ : 600.05 MHz
OBSET : 124505.37 Hz

IRNUC : 1H
IRFRQ : 600.05 MHz
IRSET : 125300.00 Hz
IRATN : 511
IRAPW : 50.0 usec
IRBP1 : 30
IRBP2 : 6
IRANS : 0

ADBIT : 16
CTEMP : 22.3 c
CSPED : 12 Hz
SLVNT : CDCL3

RESOL : 10.14 Hz
CLRSO : 10.14 Hz
TLINE : 10
THTOP : 2.0000
THBTM : 0.0500

operator

Date : Mon May 26 07:19:52 2003

FileName : LoadingFID.nmdata
Comment : RNS-55-3-1
SliceHistory :
EXMODE : non

POINT : 32768 points
SAMP0 : 32768 points
FREGU : 8000.0 Hz
FILTR : 4000 Hz
DELAY : 50.0 usec
DEADT : 72.3 usec
INTVL : 125.0 usec
TIMES : 8 times
DUMMY : 1 times
PD : 4 9520 sec
ACQTM : 4096.0000 msec
PREDL : 0.01000 msec
INIWT : 1000.0000 msec
RESOL : 0.24 Hz
PW1 : 5.50 usec
QBNUC : 1H
QBFRQ : 500.00 MHz
QBSET : 162410.00 Hz
RGAIN : 17

SCANS : 8 times

SLVNT : CDCL3
SPINNING : 11 Hz
TEMP : 27.5 C

RNS-55-3-1

Date : Mon May 26 08:42:19 2003

FileName : LoadingFID.nmdata
 Comment : RNS-55-3-1
 SliceHistory :
 EXMODE : bcm

POINT : 32768 points
 SAMPD : 32768 points
 FREQU : 30030.0 Hz
 FILTR : 15000 Hz
 DELAY : 13.3 usec
 DEADT : 17.3 usec
 INTVL : 33.3 usec
 TIMES : 1024 times
 DUMMY : 1 times
 PD : 1.9088 sec
 ACQTM : 1091.1743 msec
 PREQL : 0.01000 msec
 INIWT : 1000.0000 msec
 RESOL : 0.92 Hz
 PW1 : 5.50 usec
 OBNUC : 13C
 OBFRQ : 125.65 MHz
 OBSET : 127958.00 Hz
 RGAIN : 33
 IRNUC : 1H
 IRFRQ : 500.00 MHz
 IRSET : 162410.00 Hz
 IRRPW : 50.0 usec
 IRRNS : 0

SCANS : 1024 times

SLVNT : CDCL3
 SPINNING : 12 Hz
 TEMP : 29.8 C

$$\begin{array}{ccc} \mathbb{C}^2 & \xrightarrow{\quad \varphi \quad} & \mathbb{C}^2 \\ \downarrow \text{pr}_1 & & \downarrow \text{pr}_1 \\ \mathbb{C} & \xrightarrow{\quad \varphi \quad} & \mathbb{C} \end{array}$$

9.5

210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

non FNS-55-4

Date : Thu May 22 06:49:07 2003

FileName auto_1H.nmdata
 Comment non
 EXMODE non
 POINT 32768 points
 SAMPO 32768 points
 FREQU 10000.0 Hz
 FILTR 5000 Hz
 DELAY 40.0 usec
 QEADT 57.1 usec
 INTVL 100.0 usec
 TIMES 8 times
 DUMMY 0 times
 PD 3.7232 sec
 ACQTM 3276.7998 msec
 PREOL 10.0000 msec
 INIWT 0.5000 msec
 RESOL 0.31 Hz
 PW1 5.70 usec
 OBNUC 1H
 OBFRQ 500.00 MHz
 OBSET 162160.00 Hz
 RGAIN 21
 SCANS 8 times
 SLVNT CDCL3
 SPINNING 12 Hz
 TEMP 28.3 C

cosy

Date : Thu May 22 08:06:16 2003

Filename : auto_HH2D.nmdata
Comment : cosy
SliceHistory :
EXMODE : cosy

POINT 512 points
SAMP 512 points
FREQ 3963.5 Hz
FILTR 2000 Hz
DELAY 100.0 usec
DEADT 144.3 usec
INTVL 252.3 usec
TIMES 16 times
DUMMY 4 times
PD 0.9488 sec
ACQTM 129.1776 msec
PREDL 0.01000 msec
INIWT 1000.0000 msec
RESOL 7.74 Hz
PW1 11.40 usec
OBNUC 1H
OBFRQ 500.00 MHz
OBSET 161754.12 Hz
RGAIN 19
CLFRQ 3963.5 Hz
CLPNT 256 points
TODAT 256 points
CINWT 5.00 usec
CINTV 252.3 usec
PW2 11.40 usec
IRNUC 1H
IRFRQ 500.00 MHz
IRSET 161754.12 Hz
IRATN 511
SCANS 16 times
SLVNT CDCL3
SPINNING 14 Hz
TEMP 27.7 C