Supporting Information for

Chemical Synthesis of CD52 Glycopeptides Containing the Acid-Labile Fucosyl Linkage

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I. Experimental Section

General Methods. The NMR spectra were recorded on a 200, 300 or 600 MHz machine. Proton chemical shifts are reported in ppm (δ) downfield from tetramethylsilane (TMS) if not specified otherwise, and carbon chemical shifts are reported in ppm (δ) in reference to the solvent peak of CDCl_3 (δ 77.16). Coupling constants (J) are reported in hertz (Hz). Thin layer chromatography (TLC) was performed on silica gel GF₂₅₄ plates detected by charring with phosphomolibdic acid/EtOH or 5% H_2SO_4 /EtOH solutions. Molecular sieves were dried under high vacuum at 170-180 °C for 6 to 10 h just before use. Commercial anhydrous solvents and other reagents were used without further purification.

II. Selected NMR and MS spectra

1°

by precipitation only

by precipitation only

Water Suppression Experiment

expt water

ACQUISITION		PROCESSING	
temp	45.0	lb	0.10
solvent	D2O	oversamp	50
date	Nov 18 2002	proc	ft
		fn	32768
freq	599.914		
tn	H1		
di	1.000	AUTOMATION	
at	1.500	werf	
np	24000	wexp wft f full vs~	
sw	8000.0	wnt wft f full sel~	
bs	4	ect(1) vsadj dssh	
ss	-214.0		
tof	2.0		
rofa	4.0	CHANNEL 2	C13
gain	58	dm	nm
nt	64	CHANNEL 3	N15
ct	64	dm2	nm
OPTIONS			
presat	y	dm2	nm
scuba	n	g2lv10	1000
jumpret	n	gt0	0.0020
shaped	n	sspul	n
wet	n	satmode	y
watergate	n	satfrq	-214.0
	n	satpwr	-6
il	n	tpwr	58
in	n	pw	6.1
dp	y	composit	n
hs	n		

19 at 45°C

Pulse Sequence: gcosy
Solvent: D2O
Temp: 25.0 C / 298.1 K
User: george
File: cosyang61a1
INOVA-500 "joe"

Relax. delay 1.000 sec
Mixing 0.000 sec
Acq. time 0.128 sec
Width 3968.0 Hz
2D Width 8000.0 Hz
64 repetitions
512 increments
OBSERVE H1, 599.9108796 MHz
DECOUPLE H1, 599.9136593 MHz
Power 6 dB
off during acquisition
on during delay
single frequency
DATA PROCESSING
Sine bell 0.064 sec
F1 DATA PROCESSING
Sine bell 0.032 sec
F1 size 2048 x 2048

COSY of 19

by precipitation only

After release from resin

After precipitation only

a.i.

INSTNUM	TOP	DATE	TIME	NAME	EXP	FILE	REF
NO		MM	DD	HH:MM:SS	SEC		
001	1	2003	03	11:42:55	2003	0303	
002	1	2003	03	11:42:55	2003	0303	
003	1	2003	03	11:42:55	2003	0303	
004	1	2003	03	11:42:55	2003	0303	
005	1	2003	03	11:42:55	2003	0303	
006	1	2003	03	11:42:55	2003	0303	
007	1	2003	03	11:42:55	2003	0303	
008	1	2003	03	11:42:55	2003	0303	
009	1	2003	03	11:42:55	2003	0303	
010	1	2003	03	11:42:55	2003	0303	
011	1	2003	03	11:42:55	2003	0303	
012	1	2003	03	11:42:55	2003	0303	
013	1	2003	03	11:42:55	2003	0303	
014	1	2003	03	11:42:55	2003	0303	
015	1	2003	03	11:42:55	2003	0303	
016	1	2003	03	11:42:55	2003	0303	
017	1	2003	03	11:42:55	2003	0303	
018	1	2003	03	11:42:55	2003	0303	
019	1	2003	03	11:42:55	2003	0303	
020	1	2003	03	11:42:55	2003	0303	
021	1	2003	03	11:42:55	2003	0303	
022	1	2003	03	11:42:55	2003	0303	
023	1	2003	03	11:42:55	2003	0303	
024	1	2003	03	11:42:55	2003	0303	
025	1	2003	03	11:42:55	2003	0303	
026	1	2003	03	11:42:55	2003	0303	
027	1	2003	03	11:42:55	2003	0303	
028	1	2003	03	11:42:55	2003	0303	
029	1	2003	03	11:42:55	2003	0303	
030	1	2003	03	11:42:55	2003	0303	
031	1	2003	03	11:42:55	2003	0303	
032	1	2003	03	11:42:55	2003	0303	
033	1	2003	03	11:42:55	2003	0303	
034	1	2003	03	11:42:55	2003	0303	
035	1	2003	03	11:42:55	2003	0303	
036	1	2003	03	11:42:55	2003	0303	
037	1	2003	03	11:42:55	2003	0303	
038	1	2003	03	11:42:55	2003	0303	
039	1	2003	03	11:42:55	2003	0303	
040	1	2003	03	11:42:55	2003	0303	
041	1	2003	03	11:42:55	2003	0303	
042	1	2003	03	11:42:55	2003	0303	
043	1	2003	03	11:42:55	2003	0303	
044	1	2003	03	11:42:55	2003	0303	
045	1	2003	03	11:42:55	2003	0303	
046	1	2003	03	11:42:55	2003	0303	
047	1	2003	03	11:42:55	2003	0303	
048	1	2003	03	11:42:55	2003	0303	
049	1	2003	03	11:42:55	2003	0303	
050	1	2003	03

HPLC diagram of the final product of solution-phase synthesis using PfpOH/DCC as the coupling reagent (C18 column: 1.5X25cm, eluent: 2% of isopropyl alcohol in water containing 0.1% TFA, 200 nm).

HPLC diagram of the final product of solid-phase synthesis using HOBt/DCC as the coupling reagent (C18 column: 1.5X25cm, eluent: 2% of isopropyl alcohol in water containing 0.1% TFA, 200 nm).