

Supporting Information

^1H and ^{13}C NMR Spectra

for

Birch Reductive Alkylation of Methyl *m*-(Hydroxymethyl)benzoate Derivatives and the Behavior of *o*- and *p*-(Hydroxymethyl)benzoates under Reductive Alkylation Conditions

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TABLE OF CONTENTS

Experiments Described in Tables 3-4 and Scheme 5

Table 3 Entry 1	S2-4
Table 3 Entry 2	S5-6
Table 3 Entry 3	S7-15
Table 4 Entry 1	S16-18
Table 4 Entry 3	S19-27
Scheme 5	S28-30

Nuclei Refer

• = CH_2 CH_2 27 CH_2 10
 x = CH_2 CH_2 21 CH_2 1
 o = CH_2 CH_2 24 CH_2 2

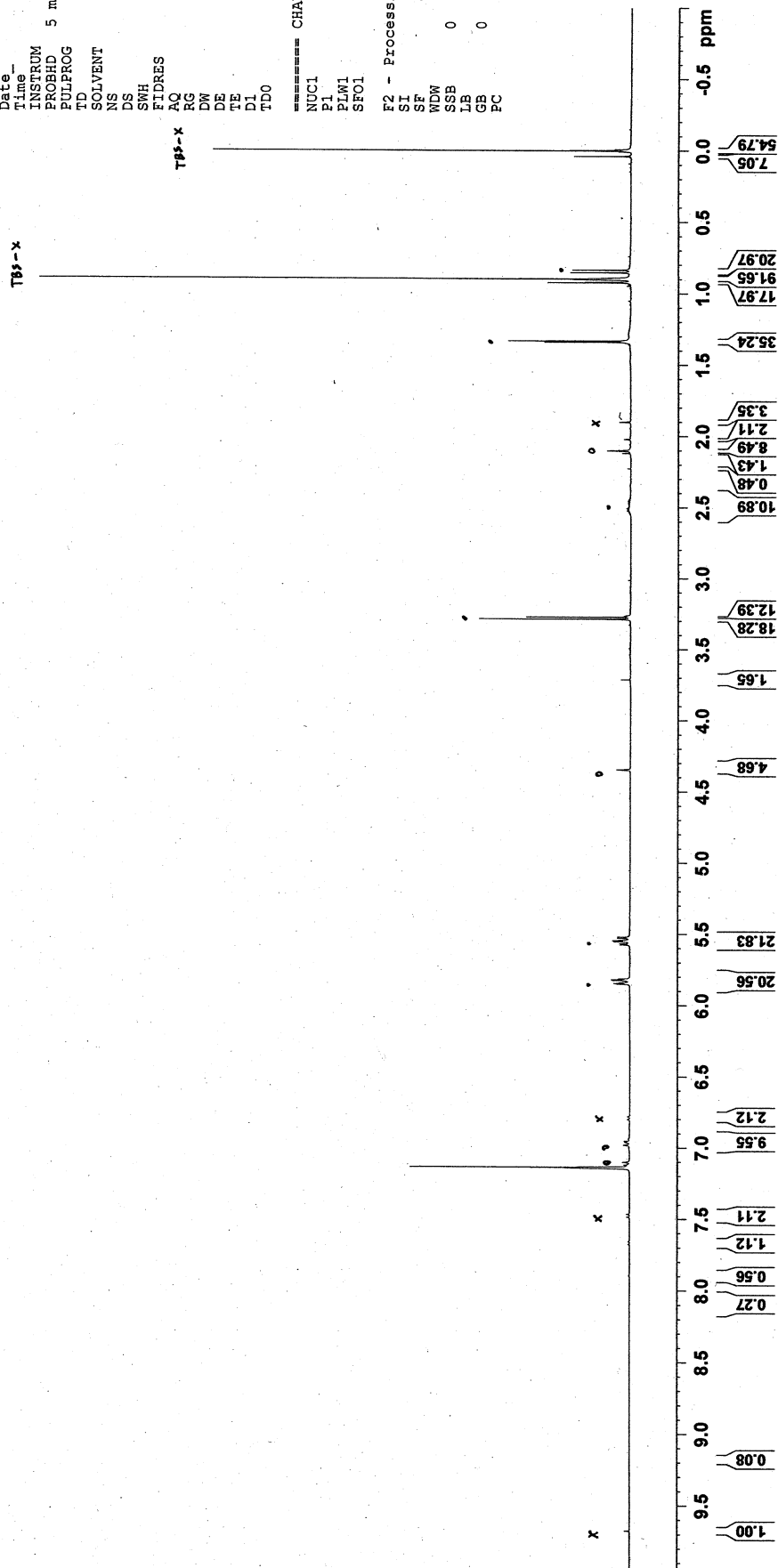
Table 3 Entry 1

crude from CH_2 w/ 2 equiv tBuOH

crude product = 445 mg

and some other stuff
in lesser amounts

Current Data Parameters
 NAME DUH-III-254
 EXPNO 1
 PROCNO 1
 F2 - Acquisition Parameters
 Date_ 20120328
 Time_ 15.06
 INSTRUM spect
 PROBD 5 mm PABBO BB/
 PULPROG zg30
 TD 65536
 SOLVENT C6D6
 NS 16
 DS 2
 SWH 8223.685 Hz
 FIDRES 0.125483 Hz
 AQ 3.9846387 sec
 RG 79.06
 DW 60.800 usec
 DE 6.50 usec
 TE 300.2 K
 D1 1.00000000 sec
 TD0 1
 ===== CHANNEL f1 =====
 NUC1 ^1H
 P1 15.00 usec
 PLW1 12.52000046 W
 SFO1 400.1724712 MHz
 F2 - Processing parameters
 SI 65536
 SF 400.1700027 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00



Fractions 6-8

Table 3 Entry 1

27



from

OTBS w/ 2 equiv tBuSH

Current Data Parameters
NAME DJH-III-255-68
EXENO 1
PROCNO 1

F2 - Acquisition Parameters

Date_ 20120329
Time_ 14.26
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT C6D6
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 120.36
DM 60.800 use
DE 6.50 use
TE 300.2 K
D1 1.00000000 sec
TD0 1

CHANNEL f1
NUC1 1H
P1 15.00 use
PLW1 12.52000046 W
SFO1 400.1724712 MHz

F2 - Processing parameters
SI 65536
SF 400.1699990 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

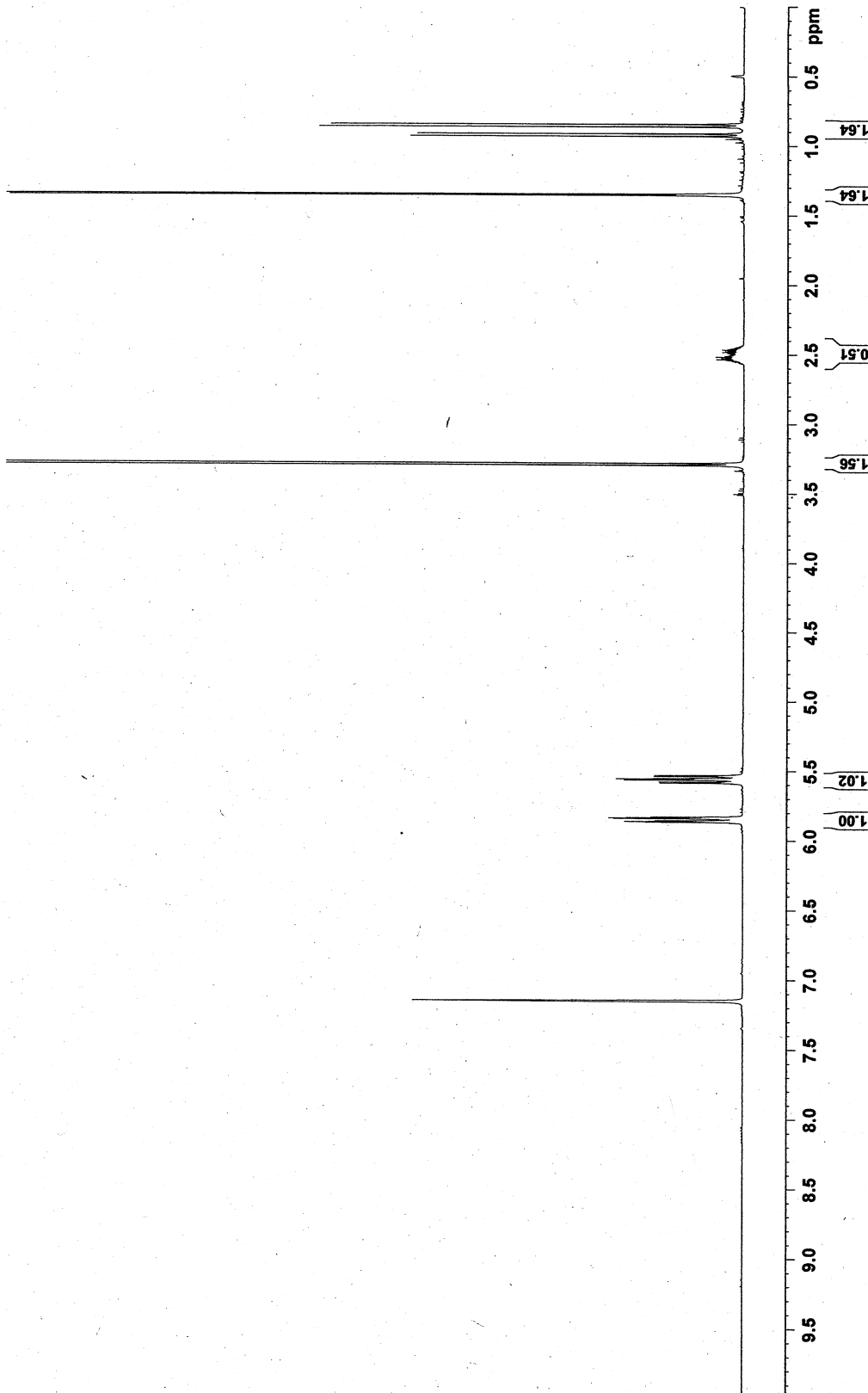
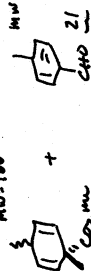


Table 3 Entry 1
 Fractions 9-10

MW 166

MW 120



2.8 : 2
 1.4 : 1

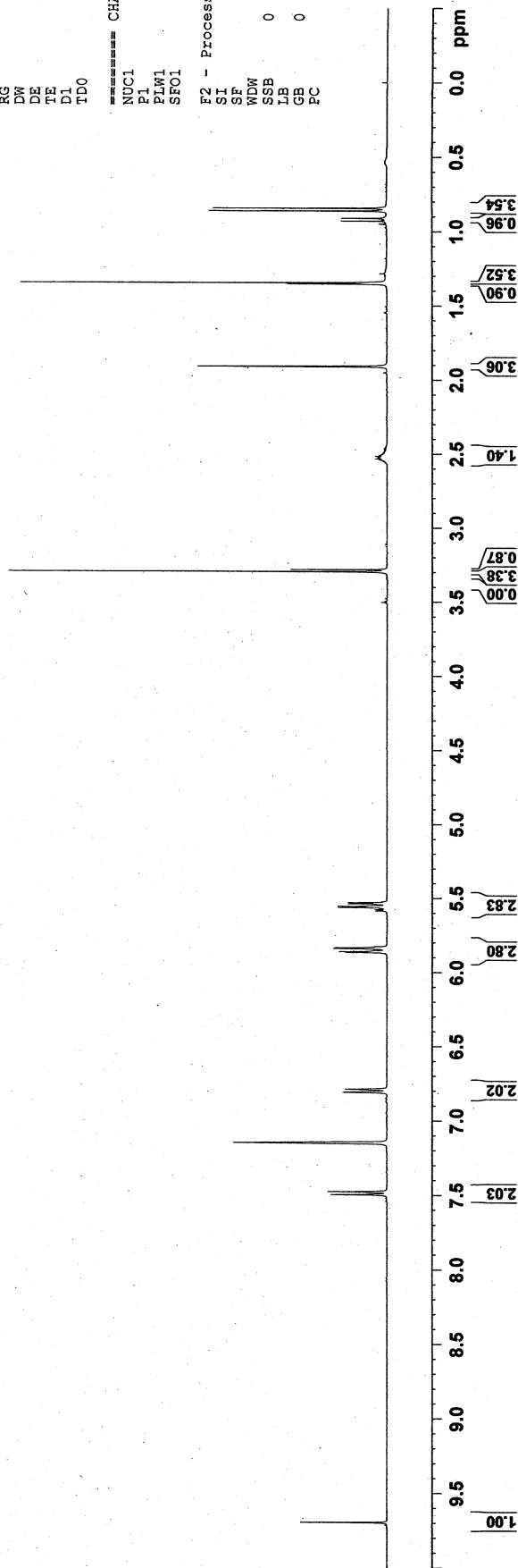
from c1ccc(cc1)C(=O)O w/ Zequiv BuOH

Current Data Parameters
 NAME DJH-III-255-910
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20120329
 Time_ 14.21
 INSTRUM spect
 PROBD 5 mm PABBO BB/
 PULPROG zg30
 TD 65536
 SOLVENT C6D6
 NS 16
 DS 2
 SWH 8223.685 Hz
 FIDRES 0.125483 Hz
 AQ 3.9846387 sec
 RG 112.47
 DW 60.800 usec
 DE 6.50 usec
 TE 300.2 K
 D1 1.00000000 sec
 TD0 1

===== CHANNEL f1 =====
 NUC1 1H
 P1 15.00 usec
 PLW1 12.52000046 W
 SFO1 400.1724712 MHz

F2 - Processing parameters
 SI 65536
 SF 400.1699994 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00



Chromatographed

from $\text{C}_{10}\text{H}_{16}$ w/zevuv + Bush

MW = 166

27



MW = 120

21



MW = 152

18



Table 3 Entry 2

Current Data Parameters
NAME DJH-III-253
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20120328
Time_ 15.02
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT C6D6
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 99.77
DW 60.800 usec
DE 6.50 usec
TE 300.2 K
D1 1.00000000 sec
TD0 1

CHANNEL f1
NUC1 ^1H
P1 15.00 usec
PLW1 12.52000046 W
SFO1 400.1724712 MHz

F2 - Processing parameters
SI 65536
SF 400.1700044 MHz
EM
WDW 0
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

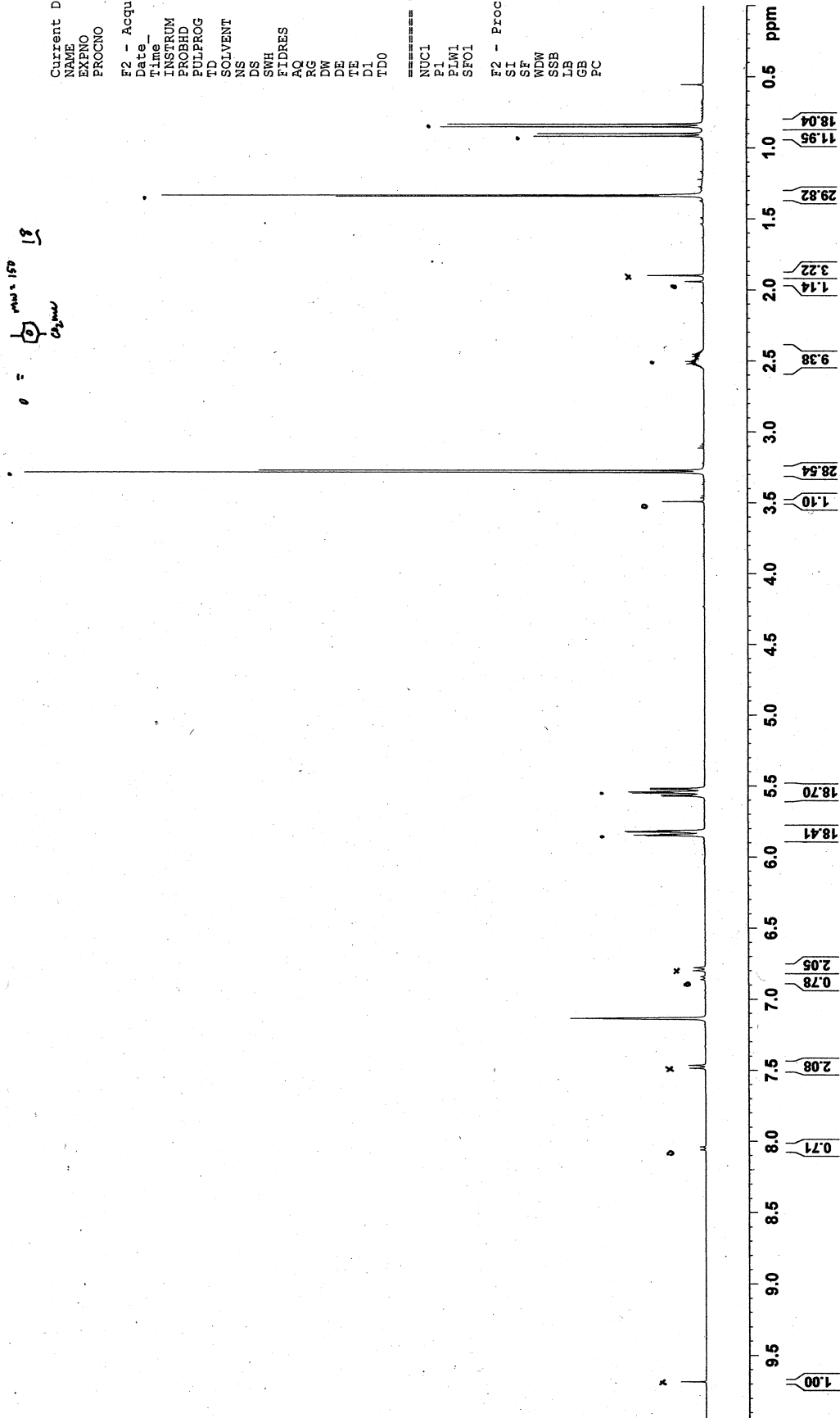
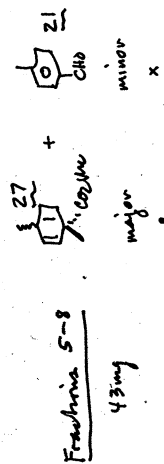


Table 3 Entry 3

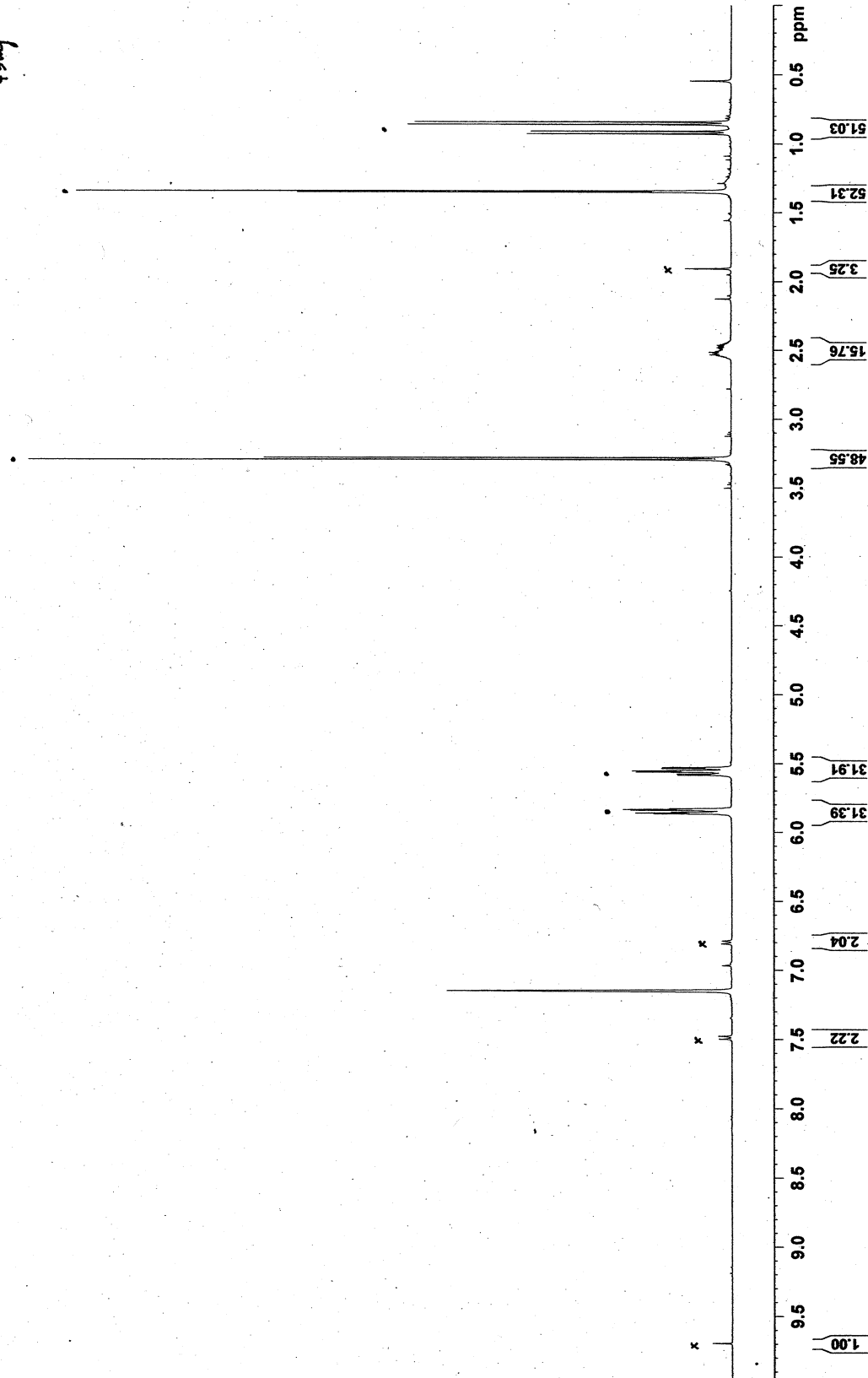


Current Data Parameters
 NAME DuH-III-275a
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20120502
 Time 14.01
 INSTRUM spect
 PROBD 5 mm PABBO BB/
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 8223.685 Hz
 FIDRES 0.125483 Hz
 AQ 3.9846387 sec
 RG 112.47
 DW 60.800 usec
 DE 6.50 usec
 TE 300.2 K
 D1 1.00000000 sec
 TD0 1

===== CHANNEL f1 =====
 NUC1 1H
 P1 15.00 usec
 PLW1 12.52000046 W
 SFO1 400.1724712 MHz

F2 - Processing parameters
 SI 65536
 SF 400.1700000 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00



c1ccc(cc1)C(=O)O
 x = minor
 o = major
 30
 Fraction 24

Table 3 Entry 3

Current Data Parameters
 NAME DJH-III-275d
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20120502
 Time_ 14.14
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zg30
 TD 65536
 SOLVENT C6D6
 NS 16
 DS 2
 SWH 8223.685 Hz
 FIDRES 0.125483 Hz
 AQ 3.9846387 sec
 RG 134.1
 DW 60.800 usec
 DE 6.50 usec
 TE 300.2 K
 D1 1.00000000 sec
 TD0 1

CHANNEL f1
 NUC1 1H
 P1 15.00 usec
 PL1 12.5200046 W
 SFO1 400.1724712 MHz

F2 - Processing parameters
 SI 65536
 SF 400.1700000 MHz
 EQ 0
 LB 0.30 Hz
 GB 0
 PC 1.00

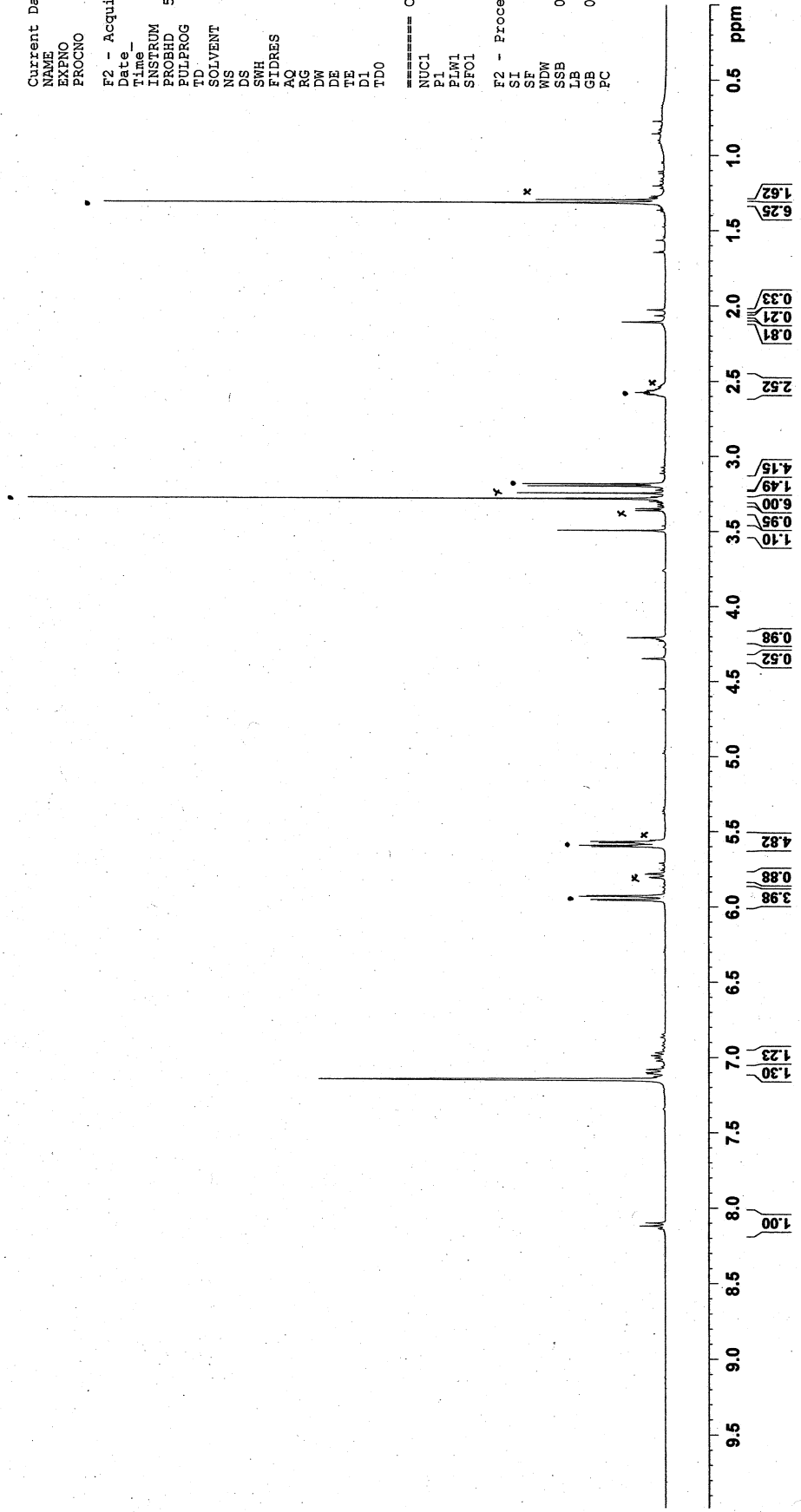
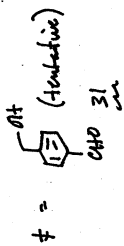
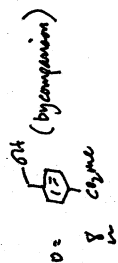


Table 3 Entry 3



Fraction 25-27

• = EtOAc

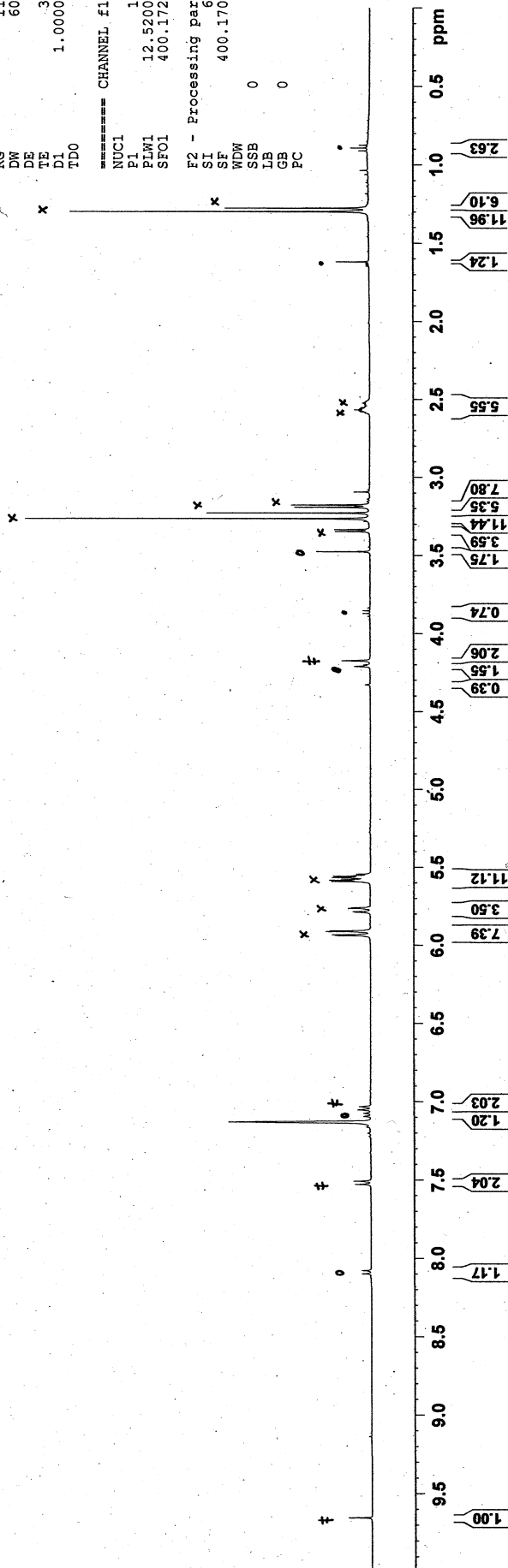
Current Data Parameters
 NAME DJH-III-275c
 EXPNO 1
 PROCNO 1
 F2 - Acquisition Parameters
 Date_ 20120502
 Time 14.03
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zg30
 TD 65536
 SOLVENT C6D6
 NS 16
 DS 2
 SWH 8223.685 Hz
 FIDRES 0.125483 Hz
 AQ 3.9846387 sec
 RG 112.47
 DW 60.800 usec
 DE 6.50 usec
 TE 300.2 K
 D1 1.00000000 sec
 TDO 1

CHANNEL f1
 NUC1 1H
 P1 15.00 usec
 PL1 12.52000046 W
 SF01 400.1724712 MHz

F2 - Processing parameters
 SI 65536
 SF 400.1700072 MHz
 EM
 SSB 0
 LB 0
 GB 0
 PC 1.00

x (major): x (minor): # : o = 8:4:2:1

Reductive ALE: RCHO: SM = 12:2:1



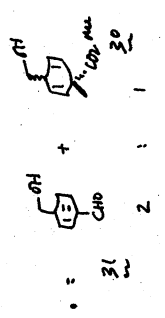


Table 3 Entry 3
6mg

Current Data Parameters
 NAME DJH-III-275e
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20120507
 Time 13.19
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zg30
 TD 65536
 SOLVENT C6D6
 NS 16
 DS 2
 SWH 8223.685 Hz
 FIDRES 0.125483 Hz
 AQ 3.9846397 sec
 RG 188.13
 DW 60.800 usec
 DE 6.50 usec
 TE 300.2 K
 D1 1.00000000 sec
 TDO 1

CHANNEL f1 1H
 NUC1 1H
 P1 15.00 usec
 PLW1 12.52000046 W
 SFO1 400.1724712 MHz

F2 - Processing parameters
 SI 65536
 SF 400.1700000 MHz
 EM
 SSB 0
 LB 0
 GB 0
 PC 1.00

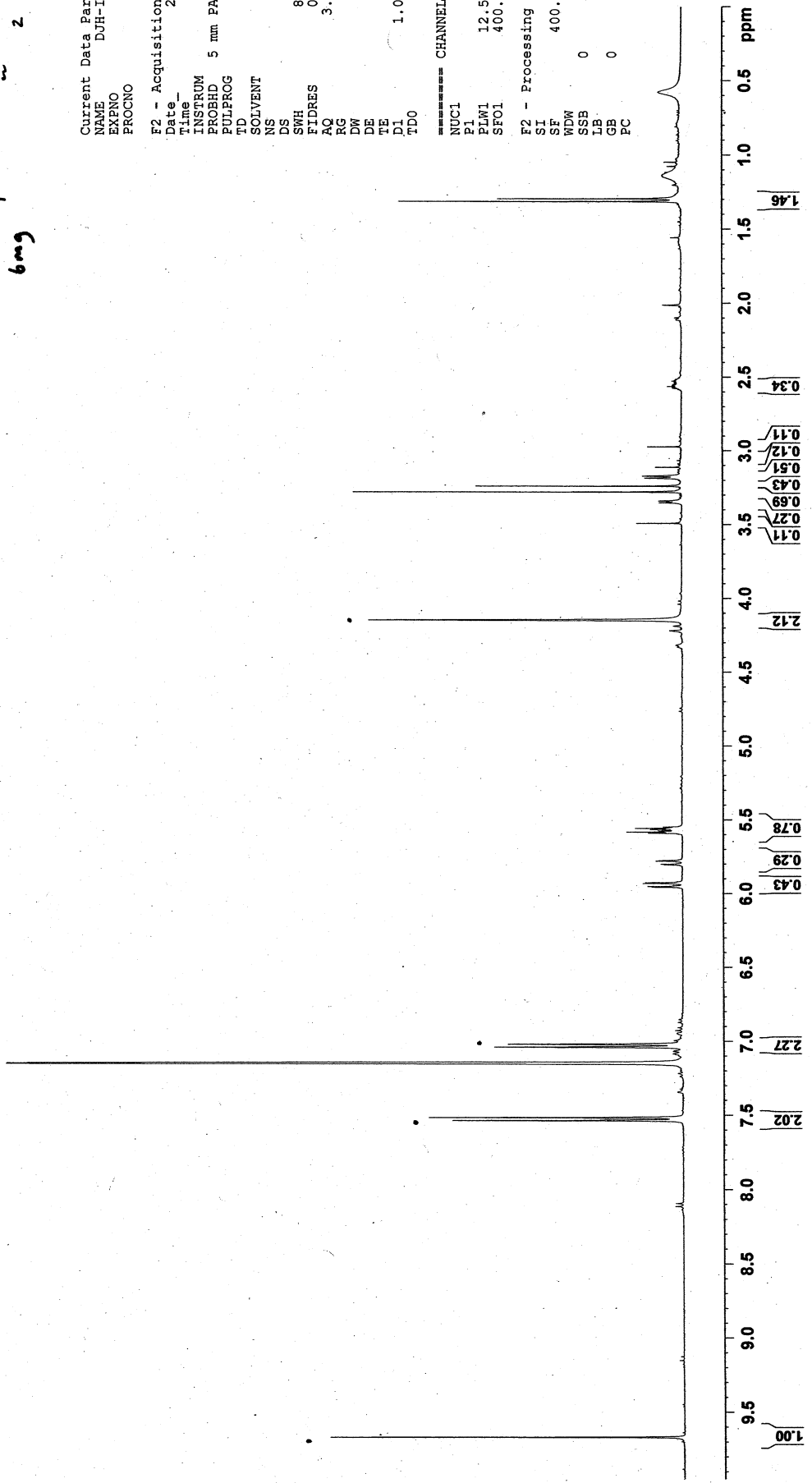
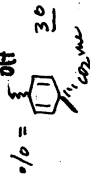
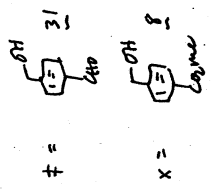


Table 3 Entry 3
 Fractions 24-27 combined



191.24
 174.59
 148.37
 146.71
 130.83
 130.71
 129.87
 129.66
 126.65
 126.60
 126.39
 126.12

65.90
 65.17
 64.13
 64.02
 51.87
 51.58
 51.43
 44.94
 44.62
 39.25
 39.08
 27.98
 26.36



Current Data Parameters
 NAME DJH-III-275
 EXPNO 2
 PROCNO 1
 F2 - Acquisition Parameters
 Date_ 20120703
 Time 0.13
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zgpg30
 TD 65536
 SOLVENT C6D6
 NS 1024
 DS 4
 SWH 24038.461 Hz
 FIDRES 0.366798 Hz
 AQ 1.3631988 sec
 RG 2050
 DW 20.800 usec
 DE 6.50 usec
 TE 300.2 K
 D1 2.00000000 sec
 D11 0.03000000 sec
 TD0 1

CHANNEL f1
 NUC1 13C
 P1 9.50 usec
 PLW1 48.00000000 W
 SFO1 100.6328883 MHz

CHANNEL f2
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 80.00 usec
 PLW2 12.52000046 W
 PLW12 0.44016001 W
 PLW13 0.28169999 W
 SFO2 400.1716007 MHz

F2 - Processing parameters
 SI 32768
 SF 100.6228049 MHz
 EM
 WDW 0
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

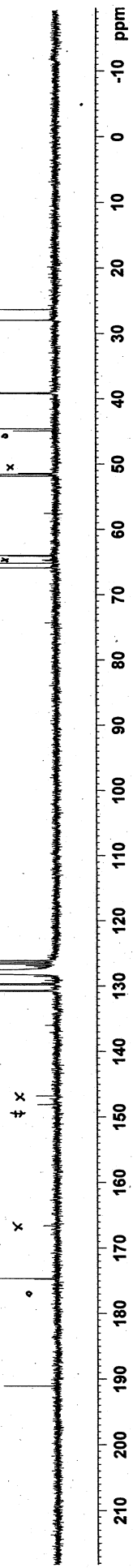




Table 3 Entry 3 Expansion

10 = 10000

Frashers 24-27 combined

Current Data Parameters
NAME DJH-III-275
EXPNO 2
PROCNO 1

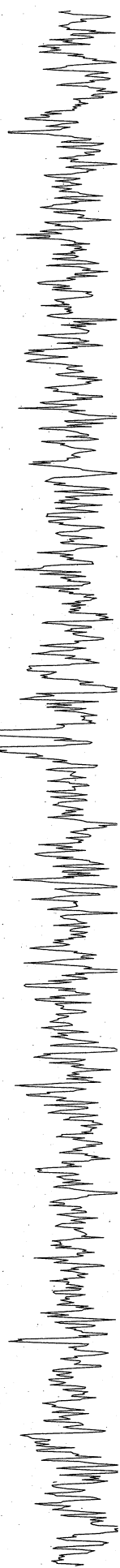
F2 - Acquisition Parameters
Date_ 20120703
Time 0.13
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CS₂
NS 1024
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 2050
DW 20.800 usec
DE 6.50 usec
TE 300.2 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 ¹³C
P1 9.50 usec
PLW1 48.00000000 W
SFO1 100.6328883 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 ¹H
PCPD2 80.00 usec
PLW2 12.52000046 W
PLW12 0.44016001 W
PLW13 0.28169999 W
SFO2 400.1716007 MHz

F2 - Processing parameters
SI 32768
SF 100.6228049 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

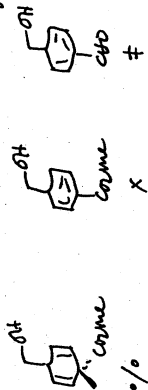
174.74
174.59



179.5 179.0 178.5 178.0 177.5 177.0 176.5 176.0 175.5 175.0 174.5 174.0 173.5 173.0 172.5 172.0 171.5 171.0 170.5 ppm

S13

Table 3 Entry 3 Fraction 24-27 combined



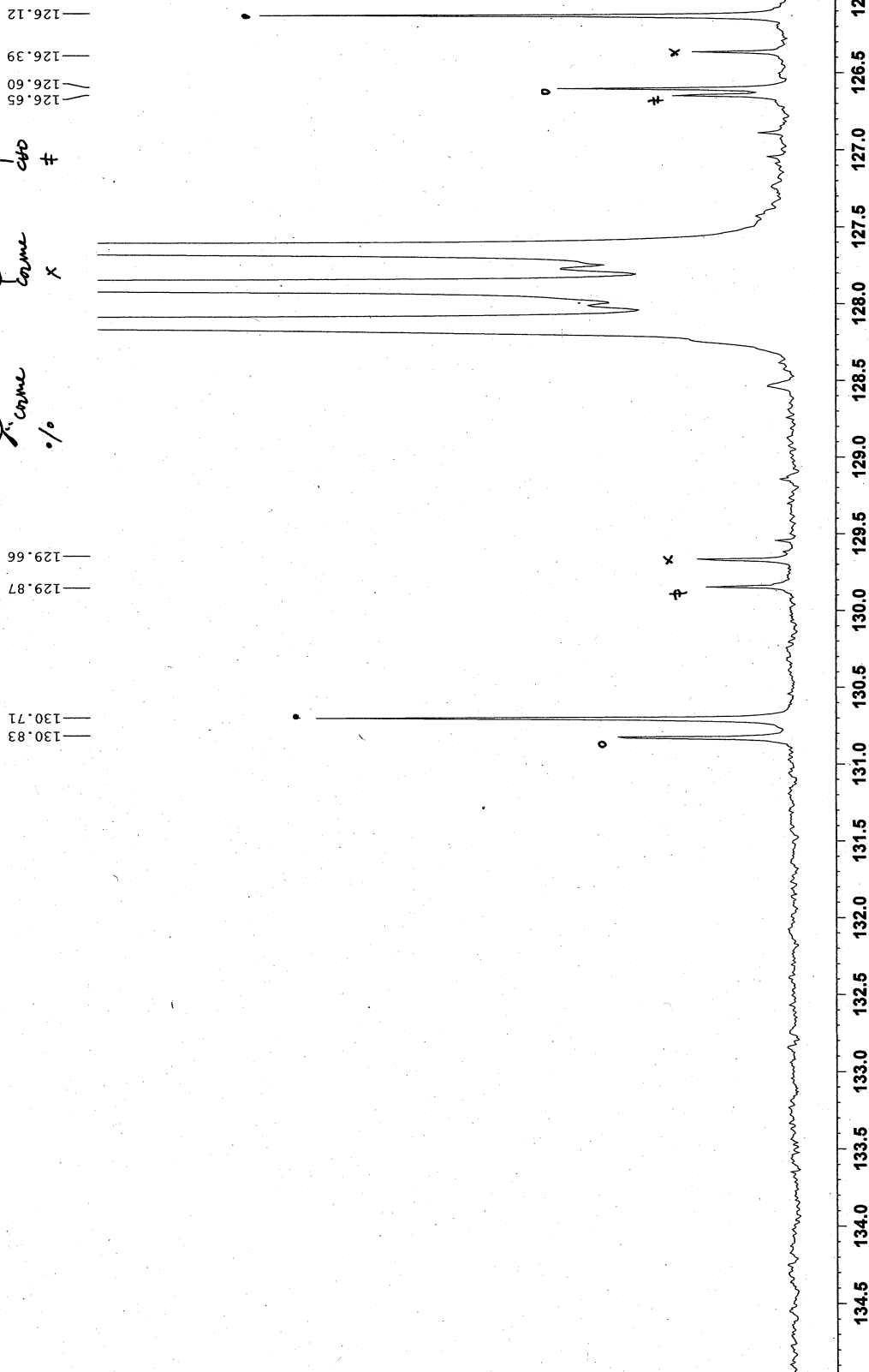
Current Data Parameters
NAME DJH-III-275
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20120703
Time 0.13
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT C6D6
NS 1024
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 2050
DW 20.800 usec
DE 6.50 usec
TE 300.2 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 13C
P1 9.50 usec
PLW1 48.00000000 W
SFO1 100.6328883 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PLW2 12.52000046 W
PLW12 0.44016001 W
PLW13 0.28169999 W
SFO2 400.1716007 MHz

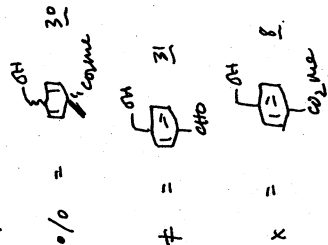
F2 - Processing parameters
SI 32768
SF 100.6228049 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



Fructose 24-27 combined

Table 3 Entry 3

Expansion



39.08
39.25

44.62
44.94

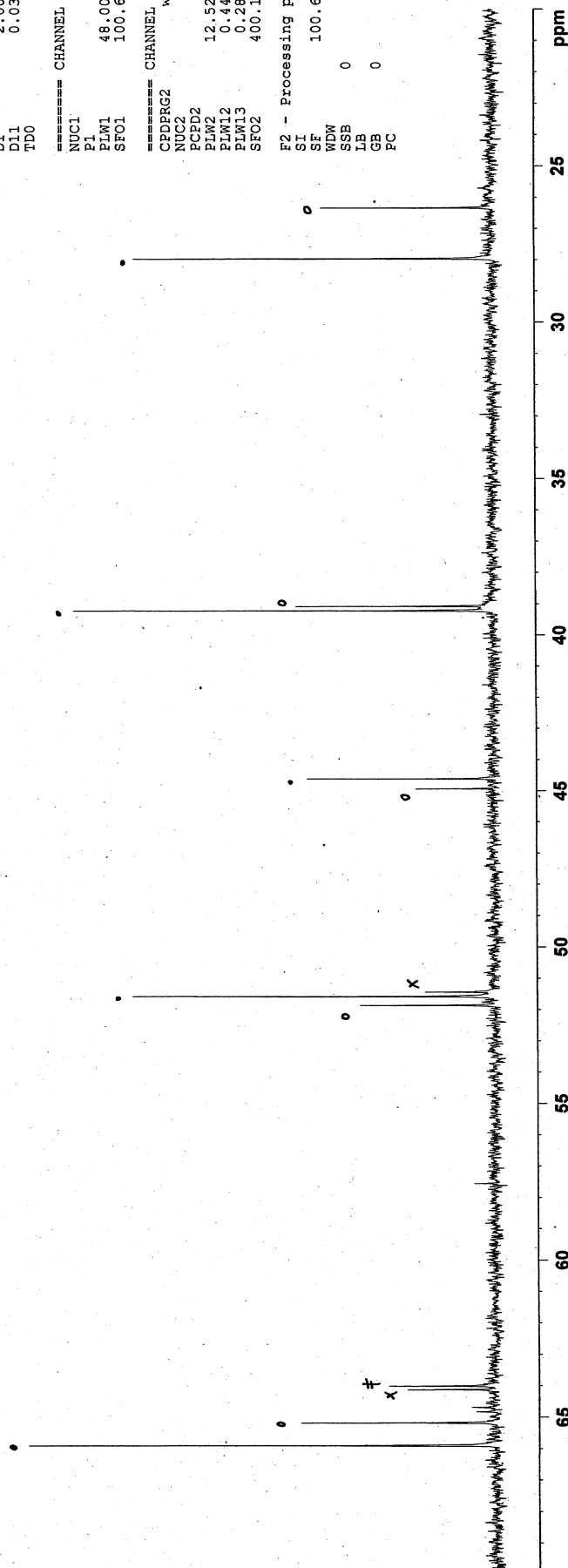
51.87
51.58
51.43

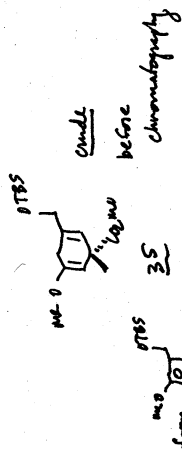
64.02
64.13
65.17
65.90

Current Data Parameters
 NAME DJH-III-275
 EXFNO 2
 PROCNO 1
 F2 - Acquisition Parameters
 Date_ 20120703
 Time_ 0.13
 INSTRUM spect
 PROBHD 5 mm PABBO BH/
 PULPROG zgpg30
 TD 65536
 SOLVENT C6D6
 NS 1024
 DS 4
 SWH 24038.461 Hz
 FIDRES 0.366798 Hz
 AQ 1.3631988 sec
 RG 2050
 DW 20.800 usec
 DE 6.50 usec
 TE 300.2 K
 D1 2.00000000 sec
 D11 0.03000000 sec
 TD0 1

===== CHANNEL f1 =====
 NUC1 13C
 P1 9.50 usec
 PLW1 48.00000000 W
 SFO1 100.6328883 MHz
 ===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 80.00 usec
 PLW2 12.52000046 W
 PLW12 0.44016001 W
 PLW13 0.28169999 W
 SFO2 400.1716007 MHz

F2 - Processing parameters
 SI 32768
 SF 100.6228049 MHz
 WDW EM
 SSB 0
 LB 0
 GB 0
 PC 1.40





Current Data Parameters	
NAME	DJH-III-260
EXPNO	1
PROCNO	1

F2 - Acquisition Parameters	
Date_	20120403
Time	15.57
INSTRUM	spec
PROBHD	5 mm PABBO B57
PULPROG	zg30
TD	65536
SOLVENT	C6D6
NS	16
DS	2
SWH	8223.685 Hz
FIDRES	0.125483 Hz
AQ	3.9846387 sec
RG	134.1
WDW	60.800 use
SS	6.50 use
TE	300.2 K
DE	1.00000000 sec
TD0	1

```

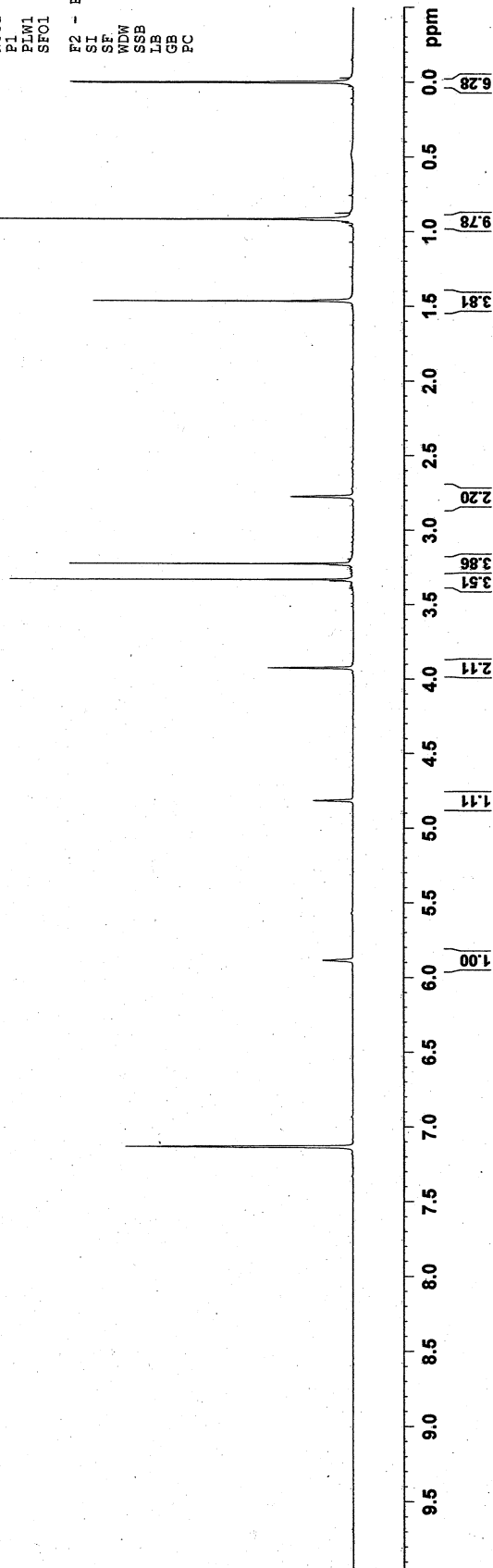
===== CHANNEL f1 =====
NUC1      1H
P1        15.00 use:
PLW1      12.52000046 W
SF01      400.1724712 MHz

```

```

F2 - Processing parameters
SI      65536
SF      400.1700054 MHz
WDW      EM
SSB      0
LB      0.30 Hz
GB      0
PC      1.00

```



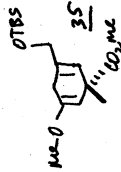


Table 4 Entry 1

Fraction 9-15

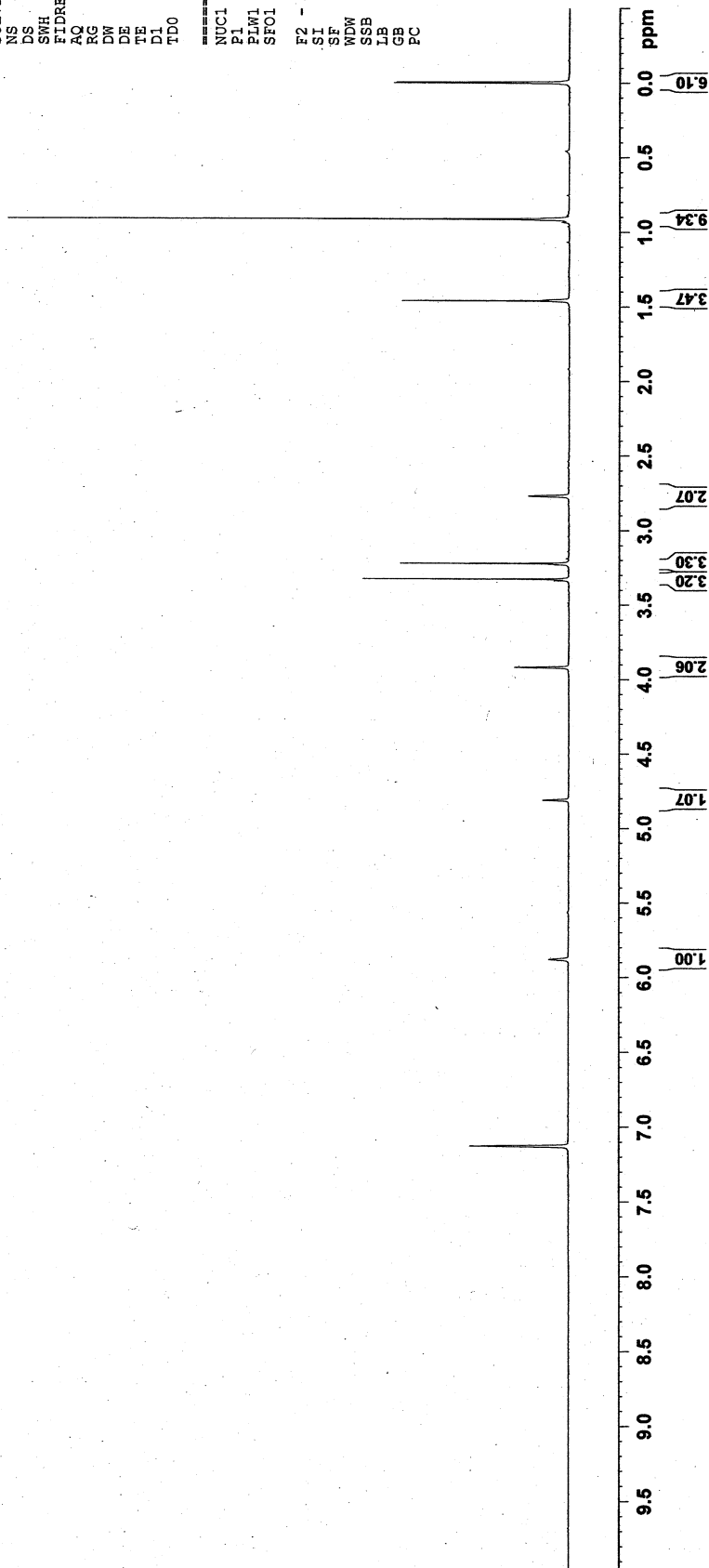
after chromatography

Current Data Parameters
 NAME DUH-III-261-915
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20120403
 Time_ 16.01
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zg30
 TD 65536
 SOLVENT C6D6
 NS 16
 DS 2
 SWH 8223.685 Hz
 FIDRES 0.125483 Hz
 AQ 3.9846387 sec
 RG 120.36
 DW 60.800 usec
 DE 6.50 usec
 TE 300.2 K
 D1 1.00000000 sec
 TD0 1

===== CHANNEL f1 =====
 NUC1 1H
 P1 15.00 usec
 PLW1 12.52000046 W
 SFO1 400.1724712 MHz

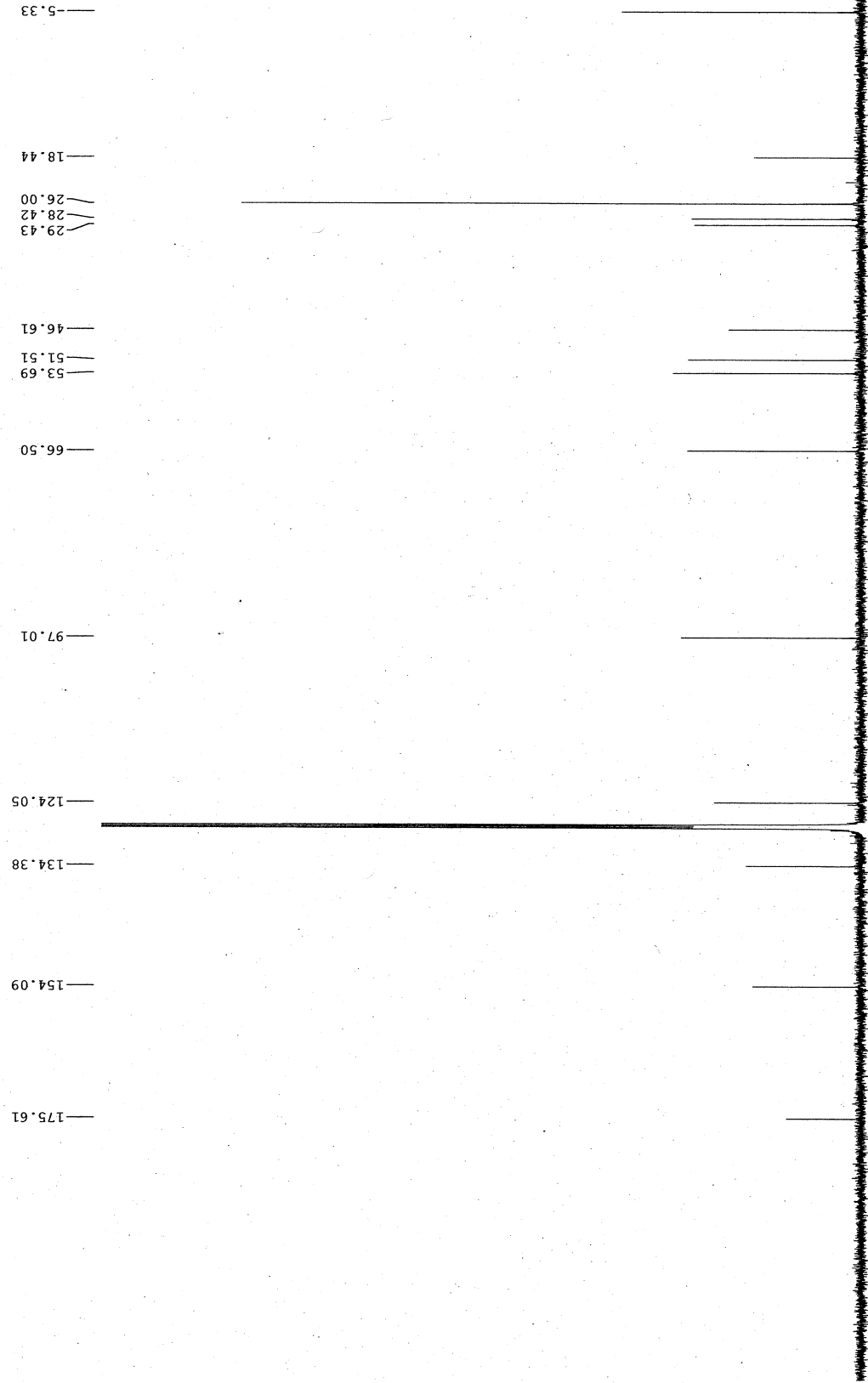
F2 - Processing parameters
 SI 65536
 SF 400.1700076 MHz
 EM
 WDW 0
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00



35 *obs after 24h in water*
tube

Table 4 Entry 1

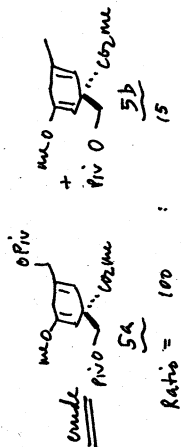
Current Data Parameters
 NAME DJH-III-257-914
 EXPNO 3
 PROCNO 1
 F2 - Acquisition Parameters
 Date_ 20120404
 Time_ 3.00
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zgpg30
 TD 65536
 SOLVENT C6D6
 NS 1024
 DS 4
 SWH 24038.461 Hz
 FIDRES 0.366798 Hz
 AQ 1.3631988 sec
 RG 2050
 DW 20.900 usec
 DE 6.50 usec
 TE 300.2 K
 D1 2.00000000 sec
 D11 0.03000000 sec
 TDO 1
 =====
 CHANNEL f1 =====
 NUC1 13C
 P1 9.50 usec
 PLW1 48.00000000 W
 SFO1 100.6328863 MHz
 =====
 CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 P2 80.00 usec
 PLW2 12.52000046 W
 PLW12 0.44016001 W
 PLW13 0.28169999 W
 SFO2 400.1716007 MHz
 F2 - Processing parameters
 SI 32768
 SF 100.6228049 MHz
 EM
 WDW 0
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40



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

518

Table 4 Entry 3



Current Data Parameters
 NAME DJH-III-291
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20120710
 Time_ 14.25
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zg30
 TD 65536
 SOLVENT C6D6
 NS 16
 DS 2
 SWH 8223.685 Hz
 FIDRES 0.125483 Hz
 AQ 3.9846387 sec
 RG 32.11
 DW 60.800 usec
 DE 6.50 usec
 TE 300.2 K
 D1 1.00000000 sec
 TD0 1

===== CHANNEL f1 =====
 NUC1 1H
 P1 15.00 usec
 PLW1 12.52000046 W
 SFO1 400.1724712 MHz

F2 - Processing parameters
 SI 85536
 SF 400.1700045 MHz
 EM
 WDW 0
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

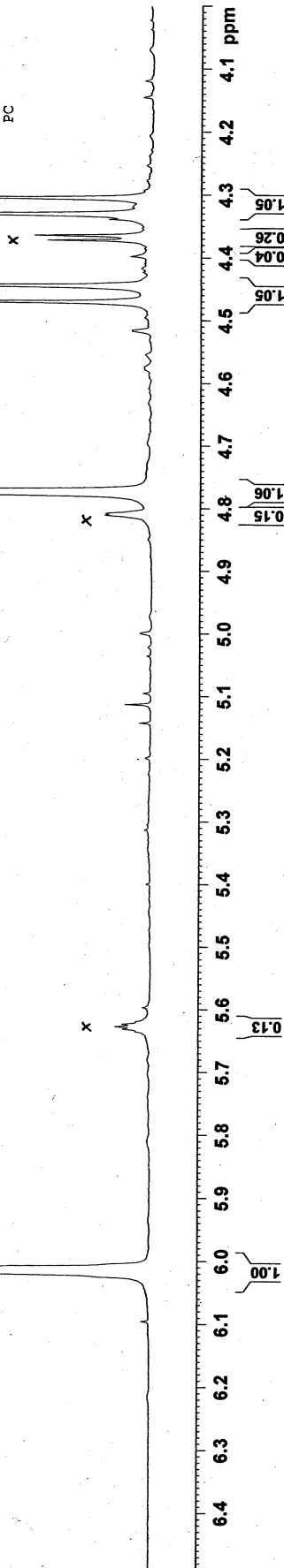
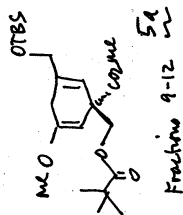


Table 4. Entry 3



```

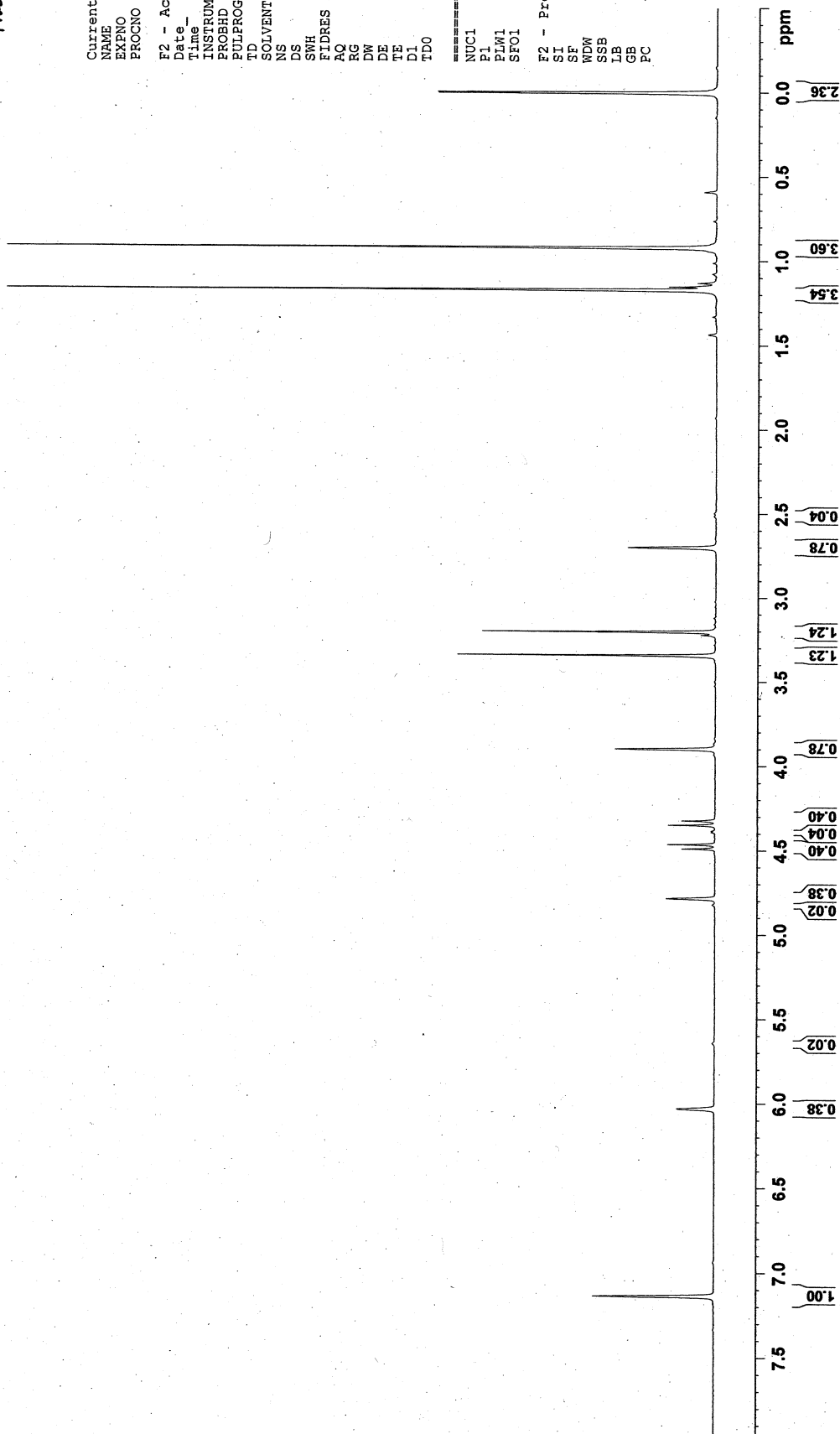
Current Data Parameters
NAME          DUH-III-252
EXPNO         1
PROCNO        1

F2 - Acquisition Parameters
Date_         20120710
Time_         19.20
INSTRUM       spect
PROBHD        5 mm PABBO BB/
PULPROG       zg30
TD            65536
SOLVENT       CDCl3
NS            16
DS            2
SWH           8223.695 Hz
SF            0.125463 Hz
FIDRES        3.9846357 sec
AQ            61.71
RG            60.800 usec
DW            6.50 usec
DE            300.2 K
TE            1.00000000 sec
D1            1
TD0           1

===== CHANNEL f1 =====
NUC1          1H
P1            15.00 usec
PLW1          12.52000046 W
SFO1          400.1724712 MHz

F2 - Processing parameters
SI            65536
SF            400.1700041 MHz
WDW           EM
SSB           0
LB            0
GB            0
PC            1.00

```



CC1=CC=C(C=C1)C(=O)O
 59
 PVO
 Fructose 9-12

Table 4 Entry 3
 purified

Current Data Parameters

NAME	DJH-III-292
EXPNO	2
PROCNO	1

F2 - Acquisition Parameters

Date_	20120711
Time	4.12
INSTRUM	spect
PROBHD	5 mm PABBO BB/
PULPROG	zgpg30
TD	65536
SOLVENT	COD6
NS	1024
DS	4
SWH	24038.461 Hz
FIDRES	0.366798 Hz
AQ	1.3631988 sec
RG	2050
DW	20.800 usec
DE	6.50 usec
TE	300.2 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1

===== CHANNEL f1 =====

NUC1	¹³ C
P1	9.50 usec
PLW1	48.00000000 W
SFO1	100.6328883 MHz

===== CHANNEL f2 =====

CPDPRG2	waltz16
NUC2	¹ H
PCPD2	80.00 usec
PLM2	12.52000046 W
PLW2	0.44016001 W
PLW3	0.28169999 W
SFO2	400.1716007 MHz

F2 - Processing parameters

SI	32768
SF	100.6228049 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

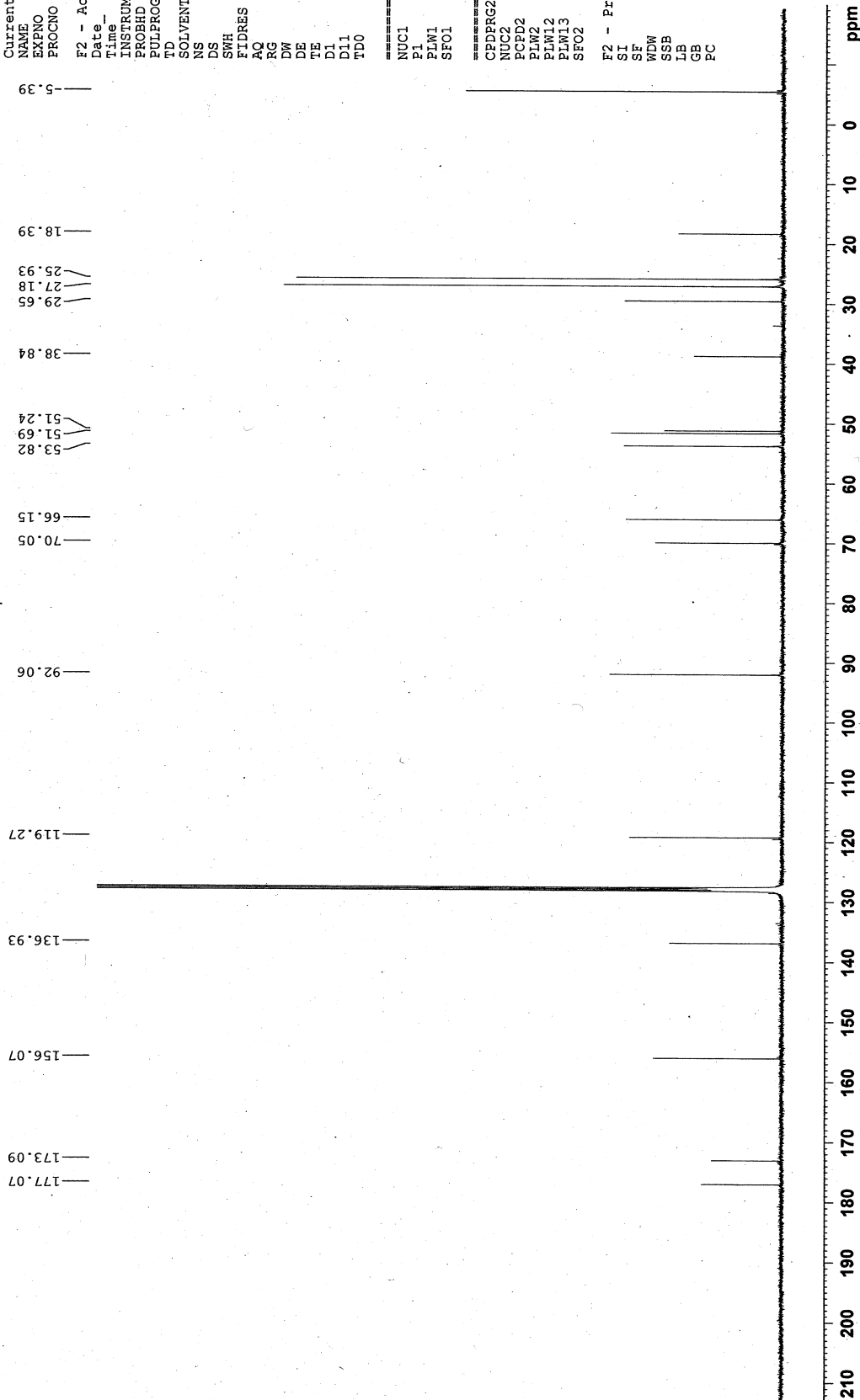


Table 4 Entry 3

CC(C)(C)C1=CC=C(C=C1)C(=O)OC + CC(C)(C)C1=CC=C(C=C1)C(=O)OC

 100 75 = 4:3

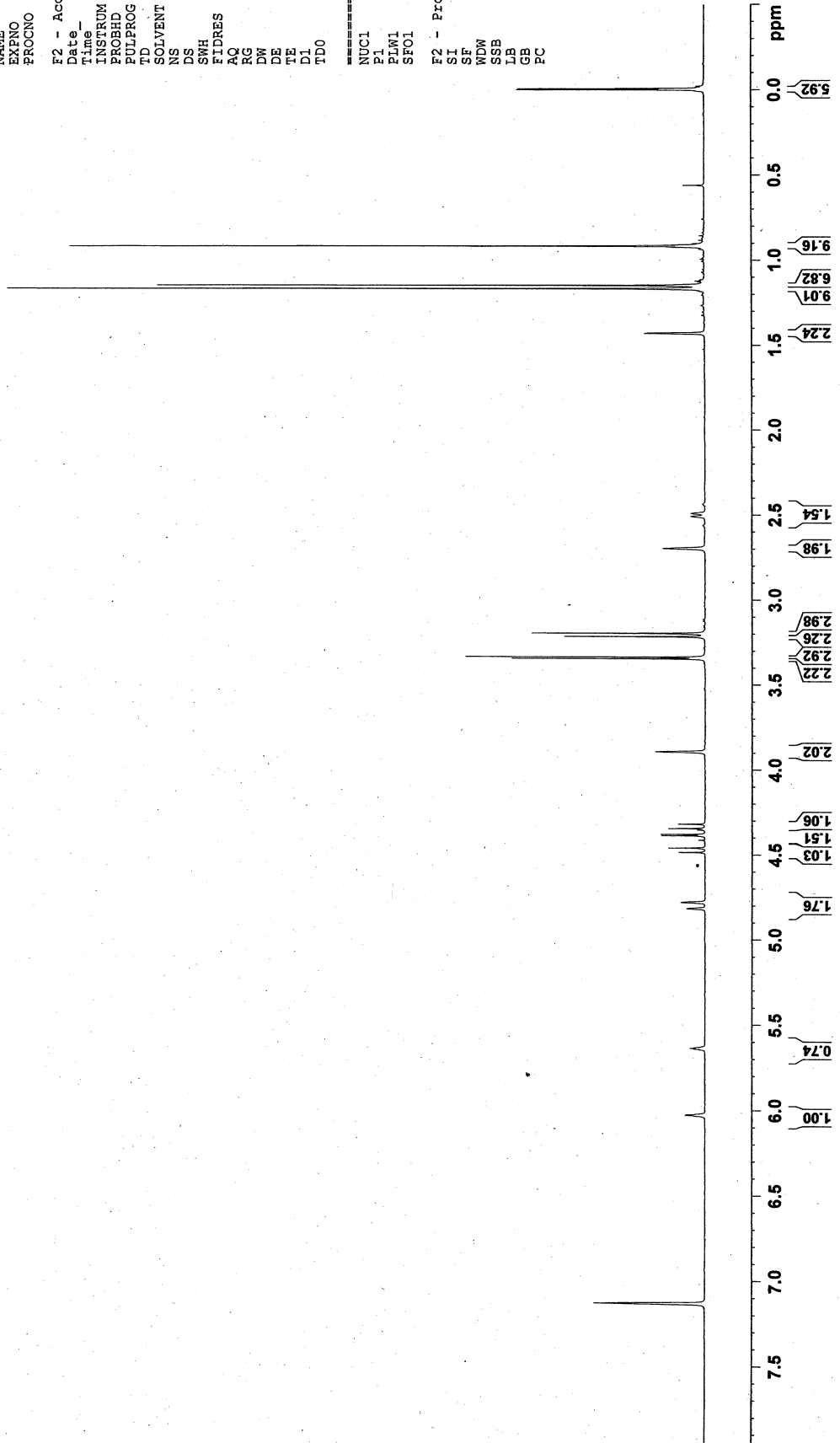
Fraction 13

Current Data Parameters
 NAME DJH-III-292-13
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20120711
 Time 14.21
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zg30
 TD 65536
 SOLVENT C6D6
 NS 16
 DS 2
 SWH 8223.685 Hz
 FIDRES 0.125483 Hz
 AQ 3.9946387 sec
 RG 79.06
 DW 60.800 usec
 DE 6.50 usec
 TE 300.2 K
 D1 1.00000000 sec
 TD0 1

CHANNEL f1
 NUC1 1H
 P1 15.00 usec
 PLW1 12.5200046 W
 SFO1 400.172412 MHz

F2 - Processing parameters
 SI 65536
 SF 400.1700066 MHz
 WDW EM
 SSB 0
 LB 0
 GB 0
 PC 1.00



Fraction 13

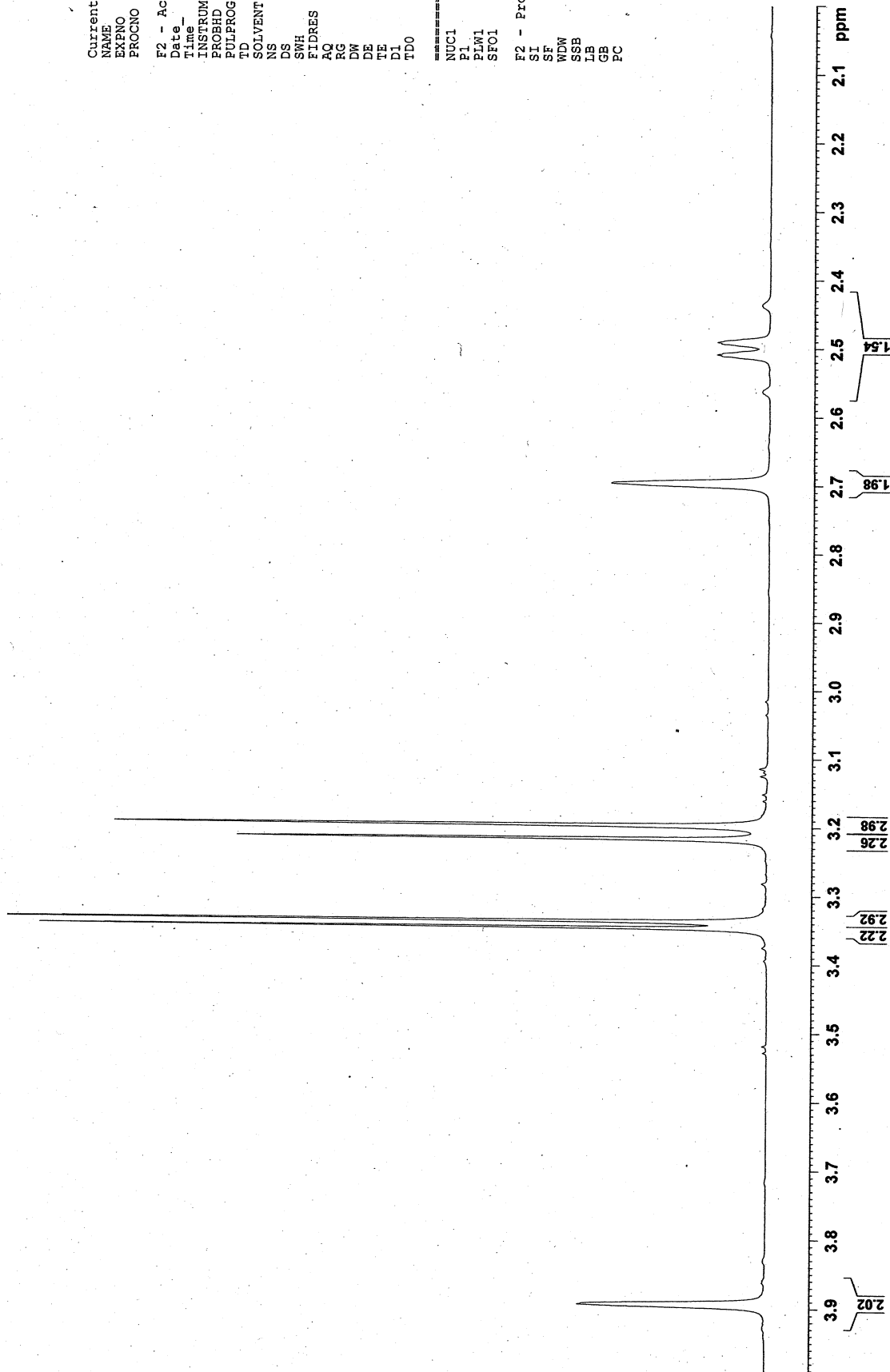
5a + 5b Upfield expansion

Current Data Parameters
NAME DJH-III-292-13
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20120711
Time 14.21
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT C6D6
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125493 Hz
AQ 3.9846387 sec
RG 79.06
DW 60.800 usec
DE 6.50 usec
TE 300.2 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 15.00 usec
PLW1 12.52000046 W
SFO1 400.1724712 MHz

F2 - Processing parameters
SI 65536
SF 400.1700066 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



S24

Table 4 Entry 3
 Fraction 13
 Expanded

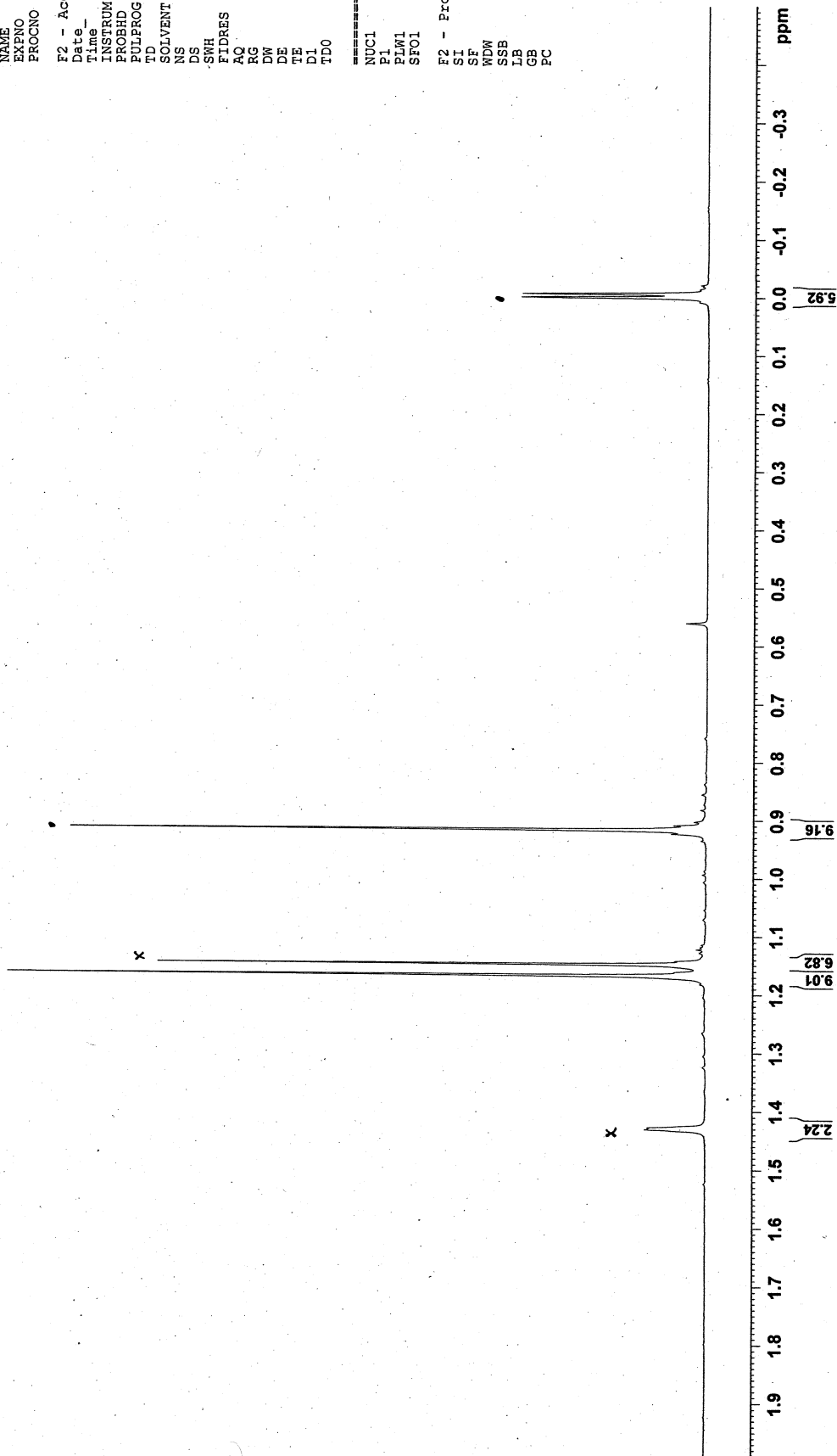
OTBS
 +
 AVO
 AVO
 5b
 5a

Current Data Parameters
 NAME DJH-III-292-13
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20120711
 Time_ 14.21
 INSTRUM spect
 PROBD 5 mm PABBO BB7
 PULPROG zg30
 ID 65536
 SOLVENT C6D6
 NS 16
 DS 2
 SWH 8223.685 Hz
 FIDRES 0.125483 Hz
 AQC 3.9846387 sec
 RG 79.06
 DW 60.800 usec
 DE 6.50 usec
 TE 300.2 K
 D1 1.00000000 sec
 TDO 1

===== CHANNEL f1 =====
 NUC1 1H
 P1 15.00 usec
 PLW1 12.5200046 W
 SFO1 400.1724712 MHz

F2 - Processing parameters
 SI 65536
 SF 400.1700066 MHz
 WDW EM
 SSB 0
 LB 0
 GB 0
 PC 1.00



PivOCH_2I

Material used in synthesis

The Ohio State University
Department of Chemistry
NMR Facility

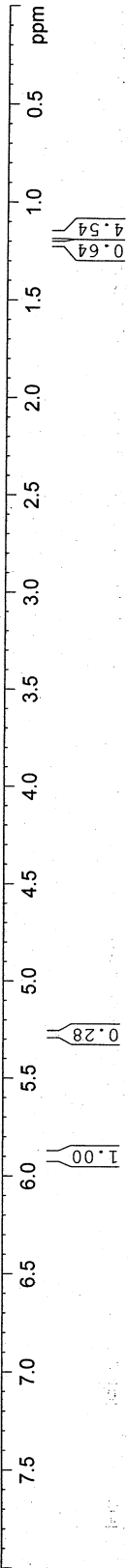
1.047
1.097
1.105
1.110
1.118
1.128
1.168
1.192
1.209
1.229
1.267
1.270
1.290
1.304
1.325
1.366
1.380
1.414
1.425
1.447

PivOCH_2I

PivOCH_2I

CH_2Cl_2

PivOCH_2Cl



Current Data Parameters
NAME DUH-III-93b
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20110208
Time 10.05
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 90.5
DM 60.400 usec
DE 6.00 usec
TE 300.2 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 13.00 usec
PL1 0.00 dB
SFO1 400.1324710 MHz

F2 - Processing parameters
SI 32768
SF 400.1300179 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

2.069
1.952
1.365
1.347
1.307
1.274
1.263
1.260
1.249
1.208
1.163
1.147
1.139
1.107
1.077
1.062
1.046
0.904
0.885
0.856

Piv OH₂eI
(pushed)

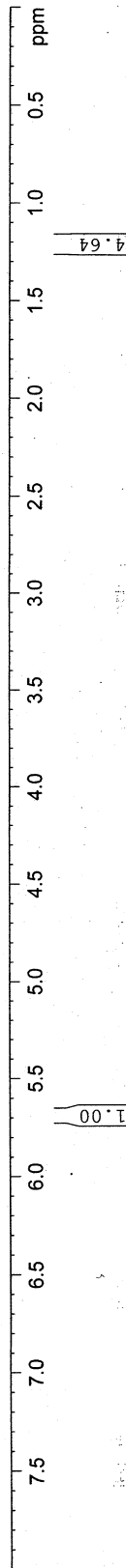
The Ohio State University
Department of Chemistry
NMR Facility

Current Data Parameters
NAME DJH-III-93a
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20110208
Time_ 10.11
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDCl₃
NS 4
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 71.8
DW 60.400 usec
DE 6.00 usec
TE 300.2 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 13.00 usec
PL1 0.00 dB
SFO1 400.1324710 MHz

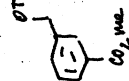
F2 - Processing parameters
SI 32768
SF 400.1300174 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.40



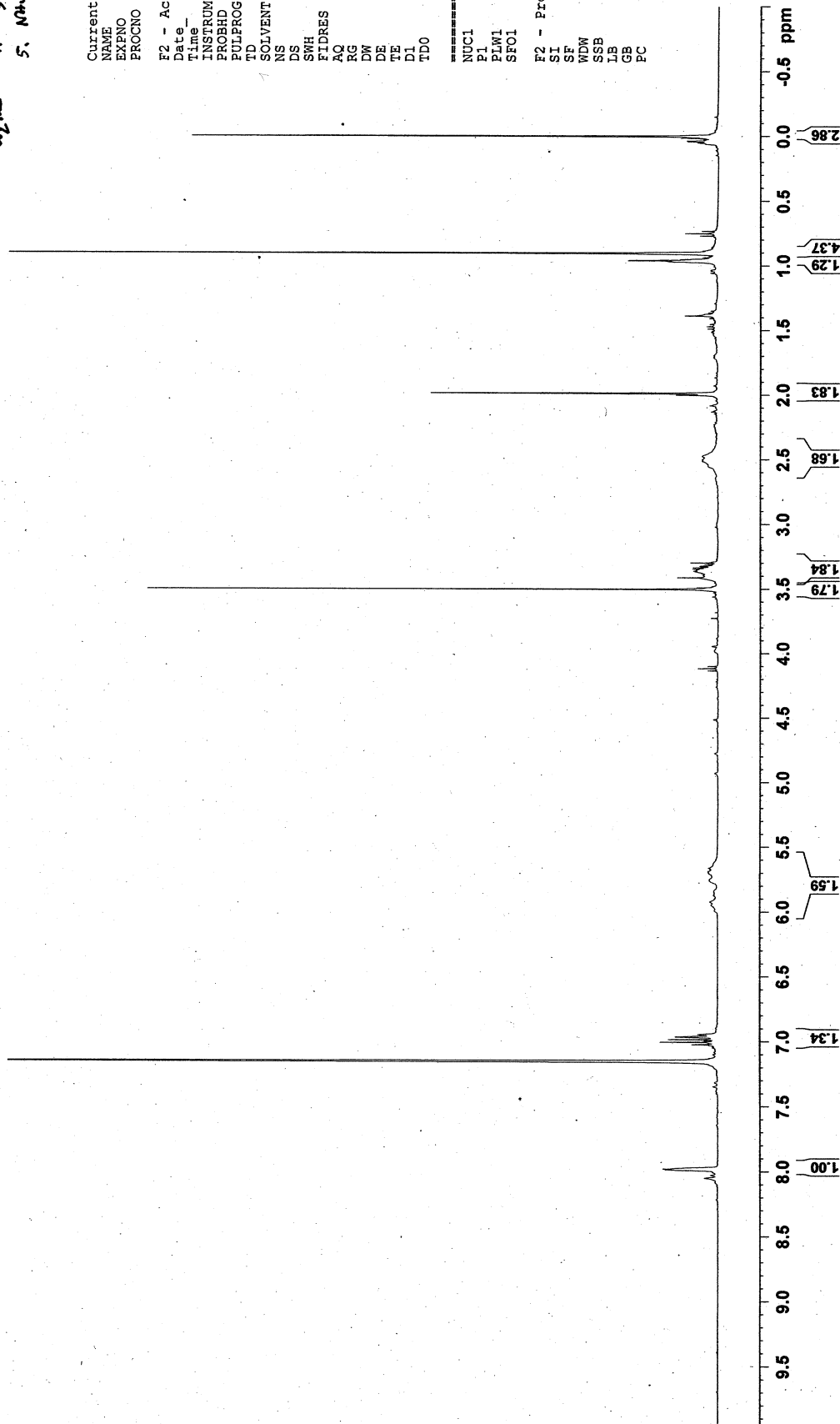
Scheme 5

Mechanistic Experiment

T → -28°C.



1. Li, NMe₃, tBuOH, THF, -65°C
 2. -65°C → -28°C
 3. -28°C → -65°C
 4. CH₃I
 5. NaHCl
- crude product



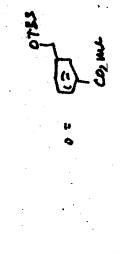
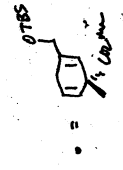
Current Data Parameters
 NAME DJH-III-278
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20120502
 Time_ 16.33
 INSTRUM spect
 PROBD 5 mm PABBO BB7
 PULPROG zg30
 TD 65536
 SOLVENT C6D6
 NS 16
 DS 2
 SWH 8223.685 Hz
 FIDRES 0.125483 Hz
 AQ 3.9846387 sec
 RG 148.14
 DW 60.800 usec
 DE 6.50 usec
 TE 300.2 K
 DI 1.00000000 sec
 TDO 1

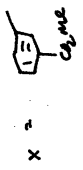
CHANNEL f1
 NUC1 1H
 P1 15.00 usec
 PLW1 12.52000046 W
 SFO1 400.1724712 MHz

F2 - Processing parameters
 SI 65536
 SF 400.1699990 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

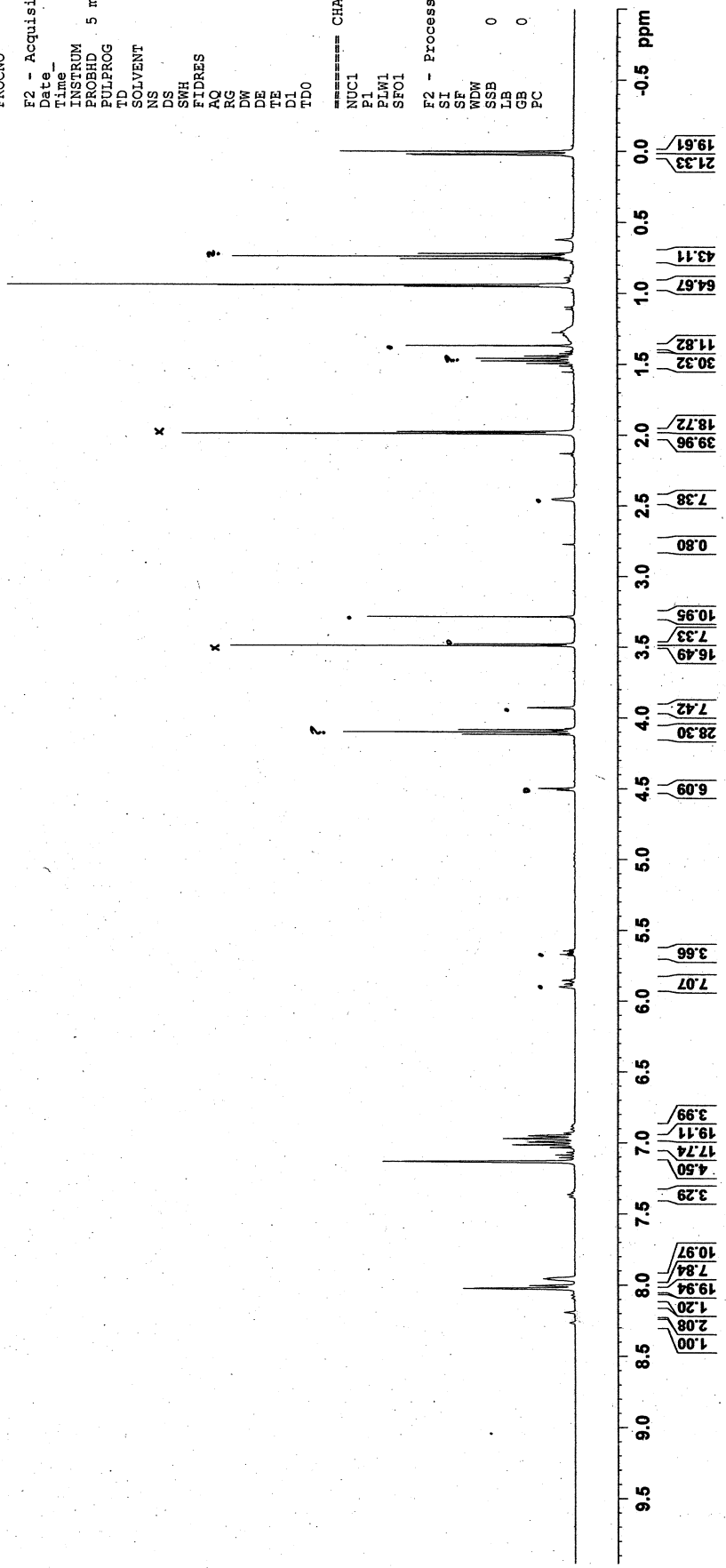
24mg



Mechanistic Experiment
Scheme 5



Current Data Parameters
NAME DUH-III-279-56
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20120507
Time 13.29
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT C6D6
NS 16
DS 2
SWH 823.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 79.06
DE 60.800 use
TE 6.50 use
D1 300.2 K
D1 1.00000000 sec
TD0 1
===== CHANNEL f1 =====
NUC1 1H
P1 15.00 use
PLW1 12.52000046 W
SFO1 400.1724712 MHz
F2 - Processing parameters
SI 65536
SF 400.1700046 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



74 mg

Fractions 7-8

chromatographed

OTBS 1. LiNH_2 , tBuOH , THF

-65°C

2. -65°C → -28°C

3. -28°C → -65°C

4. CH_3I 5. NH_4Cl

Scheme 5

Mechanism

Experiment

major product

Current Data Parameters
 NAME DJH-III-279-78
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20120507
 Time 13.50
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zg30
 TD 65536
 SOLVENT C6D6
 NS 16
 DS 2
 SWH 8223.685 Hz
 FIDRES 0.125483 Hz
 AQ 3.9846387 sec
 RG 32.11
 DW 60.800 usec
 DE 6.50 usec
 TE 300.2 K
 D1 1.00000000 sec
 TD0 1

CHANNEL f1
 NUC1 ^1H
 P1 15.00 usec
 PLW1 12.52000046 W
 SFO1 400.1724712 MHz

F2 - Processing Parameters
 SI 83536
 SF 400.1700032 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

