A Novel Enantioselective Alkylation and Its Application to the Synthesis of an AntiCancer Agent

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Supporting Information

A total of 36 pages of ^1H and ^{13}C NMR spectra for compounds **2a**, **2b**, **2c**, **4a**, **4**, **4** salt, **4e**, **4f**, **4g**, **5a**, **5b**, **5c**, **6a**, **6b**, **6c**, **6d**, **6e**, **8**, and lonafernib and chiral HPLC chromatogram for **4a**.

Preparation of 2b. The zinc method described in the experimental section was used and **2b** was isolated as a white solid. Mp 70-71 °C. ^1H NMR (CDCl_3) δ 8.35 (d, J = 2.1 Hz, 1H), 7.50 (d, J = 2.1 Hz, 1H), 7.17-7.09 (m, 3 H), 4.26 (s, 2H), 3.13-3.06 (m, 4H). ^{13}C NMR (CDCl_3): δ 155.9, 147.8, 141.0, 140.3, 136.1, 135.9, 132.9, 131.1, 129.3, 126.9, 119.0, 42.8, 31.7, 31.6. MS (MH^+): 308. Calcd for $\text{C}_{14}\text{H}_{12}\text{BrClN}$ (MH^+): 308.

Preparation of 2c. The zinc method described in the experimental section was used and **2c** was isolated as a white solid. Mp 138-139 °C. ^1H NMR (CDCl_3) δ 8.35 (d, J = 4.7 Hz, 1H), 7.45 (d, J = 1.9 Hz, 1H), 7.32 (d, J = 7.7 Hz, 1H), 7.15 (s, 1H), 7.07 (dd, J = 7.7, 4.7 Hz, 1H), 4.61 (s, 2H), 3.20-3.10 (m, 4H). ^{13}C NMR (CDCl_3): δ 155.0, 147.2, 143.7, 138.9, 137.2, 133.2, 133.0, 130.8, 129.0, 124.9, 122.5, 41.6, 32.4, 32.1. MS (MH^+): 308. Calcd for $\text{C}_{14}\text{H}_{12}\text{BrClN}$ (MH^+): 308.

INDEX	FREQUENCY	PPM	HEIGHT
1	354.823	8.386	58.2
2	297.330	7.469	52.3
3	297.330	7.444	45.7
4	297.330	7.444	45.7
5	297.330	7.239	14.6
6	297.330	7.138	53.0
7	297.330	4.544	20.0
8	1817.683	3.191	20.0
9	1276.106	3.168	20.0
10	1276.106	3.109	20.0
11	1276.106	3.090	20.0
12	1276.106	3.076	20.0
13	1276.106	3.076	20.0

SZ-008-02-001-1
 PROTON C0C13 D: / jxie2 4

Current Data Parameters
 NAME WAG
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20020628
 Time 9.16
 INSTRUM spect
 PROBHD 5 mm QNP 1H
 PULPROG zg30
 TD 65536
 SOLVENT C0C13
 NS 16

#	ADDRESS	FREQUENCY (Hz)	INTENSITY
1	12889.0	3344.271	8.3580
2	12907.4	3339.618	8.3463
3	14325.3	2981.417	7.4511
4	14332.6	2979.561	7.4465
5	14515.9	2933.263	7.3308
6	14546.3	2925.596	7.3116
7	14628.2	2904.897	7.2599
8	14803.7	2860.557	7.1491
9	14894.1	2837.729	7.0920
10	14912.7	2833.021	7.0803
11	14923.6	2830.279	7.0734
12	14943.0	2825.357	7.0611
13	18827.2	1844.110	4.6088
14	21035.6	1286.201	3.2145
15	21058.7	1280.367	3.1999
16	21063.5	1279.148	3.1968
17	21087.5	1273.089	3.1817
18	21159.9	1254.802	3.1360
19	21187.0	1247.946	3.1189
20	21209.7	1242.213	3.1045

SI 32/00
 SF 400.1300096 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

1D NMR plot parameters
 CX 20.00 cm
 F1P 8.799 ppm
 F1 3520.70 Hz
 F2P 0.913 ppm
 F2 385.39 Hz
 PPMCH 0.39429 ppm/cm
 HZCM 157.76575 Hz/cm

SZ-008-02-001-1
C13CPD CDC13 D: / jxie2 4

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Current Data Parameters
NAME      MAG
EXPNO     2
PROCNO    1

F2 - Acquisition Parameters
Date_     20020628
Time      12 23
INSTRUM   spect
PROBHD    5 mm QNP 1H
PULPROG   zgpg30
TD         65536
SOLVENT   CDCl3
NS         128
DS         4
SWH        25125.629 Hz
FIDRES     0.363387 Hz
AQ         1.3042164 sec
RG         16384
OH         19.900 usec
DE         6.00 usec
TE         300.0 K
D1         2.00000000 sec
d11        0.03000000 sec
d12        0.00002000 sec

===== CHANNEL f1 =====
NUC1       13C
P1         9.00 usec
PL1        -3.00 dB
SF01       100.6237959 MHz

===== CHANNEL f2 =====
CPDPRG2    waltz16
NUC2       1H
PCPD2      60.00 usec
PL2        -3.00 dB
PL12       21.00 dB
PL13       21.00 dB
SF02       400.1316005 MHz

F2 - Processing parameters
SI         32768
SF         100.6127290 MHz
WDW        EM
SSB        0
LB         1.00 Hz
GB         0
PC         1.40

1D NMR plot parameters
CX         20.00 cm
F1P        165.597 ppm
F1         16771.88 Hz
F2P        17.653 ppm
F2         1776.13 Hz
PPMCH      7.45221 ppm/cm
HZCH       749.78772 Hz/cm

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10-des-bromo-J
 PROTON CDC13 D: / jxie2 21

Current Data Parameters
 NAME 75919-17
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20020701
 Time 15.42
 INSTRUM spect
 PROBHD 5 mm QNP 1H
 PULPROG zg30
 TO 65536
 SOLVENT CDC13
 NS 16

#	ADDRESS	FREQUENCY (Hz)	INTENSITY
1	12925.3	3344.713	8.3591
2	12933.8	3342.571	8.3537
3	14273.0	3004.241	7.5082
4	14280.4	3002.376	7.5035
5	14801.6	2870.703	7.1744
6	14833.3	2862.695	7.1344
7	14883.0	2850.143	7.1230
8	14904.1	2844.795	7.1097
9	14912.5	2842.695	7.1044
10	14935.8	2836.802	7.0897
11	14944.1	2834.696	7.0844
12	19421.4	1703.605	4.2576
13	21211.2	1251.437	3.1276
14	21251.4	1241.301	3.1022
15	21269.6	1236.703	3.0908
16	21308.8	1226.791	3.0660

F2 - Processing parameters
 SI 32768
 SF 400.130000 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

1D NMR plot parameters
 CX 20.00 cm
 FIP 8.672 ppm
 F1 3469.73 Hz
 F2P 1.954 ppm
 F2 781.95 Hz
 PPMCH 0.33586 ppm/cm
 HZCH 134.38927 Hz/cm

10-des-bromo-J-C13
C13CPD CDC13 D: / jxie2 21

Current Data Parameters
NAME 75919-17
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20020701
Time 16.36
INSTRUM spect
PROBHD 5 mm GNP 1H
PULPROG zgpg30
TD 65536
SOLVENT CDC13
NS 128

ADDRESS	FREQUENCY (Hz)	PPM	INTENSITY
10362.1	15682.989	155.8748	6.40
11427.2	14866.289	147.7575	10.00
12317.1	14183.937	140.9756	7.51
12399.9	14120.403	140.3441	10.52
12962.9	13688.714	136.0535	7.68
12979.1	13676.349	135.9306	7.28
13367.7	13378.382	132.9691	5.21
13616.0	13187.978	131.0766	12.13
13841.3	13015.241	129.3598	10.66
14155.8	12774.030	126.9624	11.26
15200.9	11972.707	118.9979	6.10
20600.7	7832.315	77.8462	8.55
20642.3	7800.392	77.5289	8.67
20684.0	7768.387	77.2108	8.72
25194.8	4309.476	42.8343	9.48
26650.0	3193.853	31.7440	12.50
26664.2	3182.937	31.6355	11.44

===== CHANNEL f2 =====
CPOPG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -3.00 dB
PL12 21.00 dB
PL13 21.00 dB
SF02 400.1316005 MHz

F2 - Processing parameters
SI 32768
SF 100.6127250 MHz
WDW EN
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
F1P 159.223 ppm
F1 16019.88 Hz
F2P 21.390 ppm
F2 2152.13 Hz
PANCH 5.89165 ppm/cm
HZCN 693.38776 Hz/cm

38407-122
PROTON CCl13 D: / mhuang2 4

5a

Current Data Parameters
NAME 38407-127
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters

Date_ 20020621
Time 10.30
INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG zg30
TD 65536
SOLVENT CDC13

#	ADDRESS	FREQUENCY [Hz]	INTENSITY
1	14487.1	2940.533	7.3489
2	14496.2	2938.253	7.3432
3	14520.1	2932.197	7.3281
4	14537.5	2927.806	7.3171
5	14554.4	2923.527	7.3064
6	14571.6	2919.187	7.2956
7	14591.0	2916.809	7.2897
8	14599.0	2914.804	7.2846
9	14601.4	2911.652	7.2768
10	14617.8	2907.514	7.2664
11	14628.3	2904.877	7.2598
12	14635.0	2903.179	7.2556
13	14651.9	2898.907	7.2449
14	14665.1	2895.580	7.2366
15	14670.7	2894.145	7.2330
16	14686.0	2890.292	7.2234
17	14721.8	2881.238	7.2008
18	14755.6	2872.719	7.1795
19	15191.2	2762.672	6.9044
20	15199.4	2760.581	6.8992
21	15217.3	2756.076	6.8880
22	15225.5	2753.989	6.8827
23	15245.5	2748.952	6.8701
24	15256.9	2746.073	6.8630
25	15264.8	2744.075	6.8580
26	15283.2	2739.419	6.8463
27	15291.1	2737.431	6.8414
28	15302.7	2734.504	6.8340
29	18288.9	1980.104	4.9487
30	18305.8	1975.824	4.9380
31	18809.3	1848.614	4.8200
32	20092.2	1524.538	3.9101
33	20099.0	1522.822	3.9038
34	20124.1	1516.482	3.7900
35	20149.3	1510.088	3.7740
36	20387.2	1450.003	3.6238
37	20421.8	1441.264	3.6020
38	21391.3	1196.331	2.9859
39	21418.3	1194.424	2.9728
40	21436.8	1194.424	2.9612
41	21445.0	1192.767	2.9559
42	21463.7	1178.052	2.9442
43	21480.3	1171.283	2.9273
44	24516.3	886.863	2.2164
45	24595.8	355.833	0.9894
46	24596.9	355.047	0.9723

38407-127-C13
C13CPD CDC13 D: / mhuang2 17

Current Data Parameters
NAME 38407-127C13
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20020624
Time 18.26
INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG zgpg30
TD 65536
SOLVENT CDC13
NS 1024

ADDRESS	FREQUENCY (Hz)	INTENSITY
1	9888.3	16046.271
2	9942.3	16004.805
3	12038.5	14397.583
4	13260.0	13460.952
5	13536.1	13249.233
6	13713.9	13112.912
7	13866.9	12980.208
8	13965.1	12920.276
9	14103.8	12813.960
10	14194.9	12744.083
11	15819.2	11498.641
12	15842.3	11480.917
13	20595.8	7836.025
14	20637.5	7804.046
15	20679.2	7772.063
16	21105.9	7444.909
17	22263.5	6557.263
18	22474.3	6395.655
19	23083.3	5928.697
20	23511.0	5600.732
21	25720.8	3906.356
22	28477.0	1026.205

F2 - Processing parameters
SI 32768
SF 100.6127250 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

10 NMR plot parameters
CX 20.00 cm
FIP 234.844 ppm
F1 23628.35 Hz
F2 -14.882 ppm
F2 -1497.28 Hz
PPMCH 12.48631 ppm/cm
HZCM 1256.28137 Hz/cm

78501-136

C030D

PROTON MeOH D: / wug 44

5c

Current Data Parameters
 NAME 78501-136
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters

Date_ 20020710
 Time 11.13
 INSTRUM spect
 PROBD 5 mm QNP 1H
 PULPROG zg30
 TO 65536
 SOLVENT MeOH
 NS 16

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1	14301.6	2997.015	7.4901	0.33
2	14329.8	2989.897	7.4723	0.53
3	14430.4	2964.479	7.4088	2.19
4	14454.8	2958.323	7.3934	1.42
5	14549.1	2934.477	7.3338	0.58
6	14576.4	2927.595	7.3166	0.79
7	14603.8	2920.666	7.2993	0.32
8	15034.2	2806.873	7.0149	3.41
9	17275.3	2245.770	5.6126	0.34
10	17474.9	2195.355	5.4866	0.60
11	18280.5	1991.833	4.9780	0.18
12	18331.8	1978.876	4.9456	0.20
13	18402.2	1961.089	4.9011	11.75
14	18993.0	1837.100	4.5913	0.31
15	18944.3	1824.142	4.5589	0.39
16	19290.9	1756.803	4.3406	0.41
17	19341.2	1723.868	4.3083	0.33
18	19417.6	1704.557	4.2600	0.21
19	19468.9	1691.602	4.2276	0.19
20	19674.8	1639.576	4.0976	0.16
21	19957.3	1588.211	3.9193	30.00
22	20125.2	1525.813	3.7360	20.34
23	20247.6	1494.890	3.7360	0.17
24	20380.2	1461.377	3.6253	0.32
25	20407.5	1454.493	3.6251	0.53
26	20415.3	1452.506	3.6301	0.42
27	20422.0	1450.822	3.6259	0.46
28	20434.9	1447.570	3.6177	0.40
29	20879.8	1335.177	3.3369	0.50
30	20886.1	1332.569	3.3287	1.02
31	20892.7	1331.903	3.3287	1.45
32	20899.1	1330.295	3.3247	1.04
33	20905.7	1328.632	3.3205	0.53
34	21510.9	1175.743	2.9384	4.75
35	24067.5	529.874	1.3243	0.68
36	24093.3	523.352	1.3080	0.75
37	24168.8	504.267	1.2603	1.35
38	24195.5	497.516	1.2434	1.26
39	24241.1	486.003	1.2146	0.13
40	24269.1	478.944	1.1970	0.20

HZCM 172.05591 Hz/cm

S111

78501-136-C13
CD300
C13CPD MeOH D: / mhuang2 44

Current Data Parameters
NAME 38974-17-2
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters

Date_	Time	Address	F2	Frequency [Hz]	Intensity
20020621					
1	16097.6	3022.826		7.5546	0.24
2	16709.2	3019.896		7.5473	2.24
3	16717.2	3017.871		7.5422	0.76
4	16735.8	3013.185		7.5305	0.80
5	16743.8	3011.167		7.5255	2.47
6	16755.4	3008.233		7.5182	0.31
7	16882.6	2976.084		7.4378	0.25
8	16902.9	2970.972		7.4250	5.75
9	16916.9	2967.433		7.4162	3.16
10	16922.1	2966.125		7.4129	3.24
11	16946.1	2960.042		7.3977	0.22
12	16952.9	2958.336		7.3934	0.27
13	16956.7	2957.367		7.3910	0.32
14	16999.8	2946.477		7.3638	0.11
15	17016.8	2942.200		7.3531	0.40
16	17029.2	2939.056		7.3453	0.50
17	17036.2	2937.294		7.3409	0.43
18	17040.8	2936.136		7.3380	0.46
19	17051.1	2933.528		7.3314	0.61
20	17060.5	2931.161		7.3255	0.34
21	17064.3	2930.204		7.3231	0.32
22	17070.1	2928.726		7.3184	0.27
23	17085.0	2924.974		7.3101	0.15
24	17453.1	2831.982		7.0777	0.28
25	17464.8	2829.010		7.0702	2.67
26	17473.0	2826.945		7.0651	0.83
27	17491.3	2822.323		7.0535	0.80
28	17499.4	2820.281		7.0484	2.42
29	17511.2	2817.303		7.0410	0.28
30	20353.6	2099.229		5.2464	1.34
31	20365.6	2096.201		5.2388	1.36
32	20460.6	1971.136		4.9262	10.04
33	21770.8	1741.203		4.3516	0.16
34	21823.5	1727.849		4.3183	2.78
35	21828.2	1726.697		4.3153	2.76
36	21881.1	1713.322		4.2819	0.17
37	22543.2	1546.061		3.8639	12.50
38	23084.1	1409.405		3.5224	0.18
39	23096.3	1406.328		3.5147	0.19
40	23110.9	1402.644		3.5055	0.60
41	23123.0	1399.577		3.4978	0.59
42	23137.7	1395.864		3.4885	0.61
43	23149.9	1392.784		3.4809	0.60
44	23164.5	1389.116		3.4717	0.20
45	23176.7	1386.036		3.4640	0.18
46	23360.0	1339.718		3.3482	0.79
47	23366.3	1338.116		3.3442	1.53
48	23372.8	1336.473		3.3401	2.16
49	23379.2	1334.870		3.3361	1.53
50	23385.7	1333.234		3.3320	0.80
51	26874.2	451.919		1.1294	4.71
52	26901.1	445.139		1.1125	4.67

38974-17-C13
C13CPD MeOH D: / mhuang2 11

Current Data Parameters
NAME 38974-17C13
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20020624
Time 13.09
INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG zgpg30
TD 65536
SOLVENT MeOH
NS 1024
DS 4

F2 - Processing parameters
SI 32768
SF 100.6125861 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
F1P 236.265 ppm
F1 23771.19 Hz
F2P -13.462 ppm
F2 -1354.44 Hz
PPMCM 12.48633 ppm/cm
HZCM 1256.28149 Hz/cm

#	ADDRESS	FREQUENCY [Hz]	INTENSITY
1	9683.2	16346.356	5.00
2	12343.5	14306.527	5.24
3	13509.9	13412.166	12.17
4	13947.6	13076.577	11.80
5	14038.0	13007.225	5.79
6	14284.5	12818.193	11.77
7	14624.1	12357.849	6.36
8	15781.3	11670.535	12.50
9	21606.5	7203.918	5.83
10	23138.0	6029.619	5.11
11	23603.2	5672.862	4.36
12	24418.3	5047.912	2.23
13	24446.2	5026.505	6.85
14	24458.8	5016.846	4.90
15	24474.1	5005.082	13.58
16	24502.1	4982.617	13.59
17	24530.0	4962.212	15.82
18	24558.0	4940.778	6.88
19	24586.0	4919.340	2.33
20	25986.7	1084.919	6.19

38851-16-02
 PROTON MeOH D: / mhuang2 9

Current Data Parameters
 NAME 38851-16-02
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20020621
 Time 11:53
 INSTRUM spect
 PROBHD 5 mm QNP 1H
 PULPROG zg30
 TD 65536
 SOLVENT MeOH
 NS 16

#	ADDRESS	FREQUENCY (Hz)	PPM	INTENSITY
1	16842.0	2984.338	7.4584	1.53
2	16873.9	2976.277	7.4383	3.65
3	16891.0	2971.948	7.4275	12.30
4	16908.4	2967.552	7.4165	14.48
5	16938.7	2959.894	7.3973	0.44
6	16942.9	2958.849	7.3947	0.45
7	16985.0	2945.670	7.3618	0.97
8	17012.5	2941.263	7.3508	1.56
9	17029.5	2936.955	7.3400	1.57
10	17047.9	2932.319	7.3284	0.94
11	17063.3	2928.427	7.3187	0.36
12	17270.9	2873.967	7.1876	1.51
13	17307.8	2868.632	7.1643	1.39
14	17336.8	2859.326	7.1460	1.08
15	17436.7	2834.091	7.0829	1.59
16	17443.7	2832.330	7.0785	1.52
17	17469.6	2825.772	7.0621	1.43
18	17476.6	2823.854	7.0576	1.36
19	20370.1	2093.021	5.2309	1.46
20	20462.5	1968.884	4.9206	67.98
21	21771.7	1739.959	4.3460	3.71
22	22802.8	1354.252	3.9844	25.85
23	23057.2	1414.202	3.5344	0.58
24	23089.0	1411.211	3.5269	0.77
25	23093.1	1407.643	3.5180	1.00
26	23094.6	1404.745	3.5107	0.98
27	23352.2	1339.655	3.3481	9.32
28	23358.3	1338.129	3.3442	17.38
29	23364.6	1336.535	3.3403	23.70
30	23370.9	1334.952	3.3363	17.18
31	23377.0	1333.397	3.3324	8.93
32	26852.4	455.423	1.1382	10.34
33	26879.2	448.654	1.1213	10.23

F1P 7.766 ppm
 F1 3107.32 Hz
 F2P 0.609 ppm
 F2 243.49 Hz
 PPMCH 0.35786 ppm/cm
 HZCH 143.19153 Hz/cm

39713-65R
PROTON MeOH D: / mhuang2 11

Current Data Parameters
NAME 39713-65R
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20020621
Time 12:21

INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG zg30
TD 65536
SOLVENT MeOH
NS 16

#	ADDRESS	FREQUENCY (Hz)	PPM	INTENSITY
1	16894.9	2971.476	7.4263	32.02
2	16912.4	2967.031	7.4152	39.25
3	16929.7	2962.475	7.4043	2.00
4	16946.9	2945.701	7.3819	2.17
5	17014.1	2941.347	7.3510	3.37
6	17031.1	2937.052	7.3403	3.56
7	17049.5	2932.116	7.3287	1.97
8	17068.9	2927.120	7.3171	11.18
9	17087.3	2922.394	7.3054	7.59
10	17106.0	2917.826	7.2937	4.31
11	17124.4	2913.312	7.2820	7.87
12	17142.8	2908.857	7.2703	4.57
13	17161.2	2904.463	7.2586	4.87
14	17179.6	2900.129	7.2469	3.09
15	17198.0	2895.855	7.2352	159.38
16	17216.4	2891.641	7.2235	11.15
17	17234.8	2887.487	7.2118	119.03
18	17253.2	2883.393	7.2001	2.44
19	17271.6	2879.359	7.1884	2.20
20	17290.0	2875.385	7.1767	2.31
21	17308.4	2871.471	7.1650	1.99
22	17326.8	2867.617	7.1533	20.68
23	17345.2	2863.823	7.1416	39.22
24	17363.6	2859.989	7.1299	54.30
25	17382.0	2856.215	7.1182	39.15
26	17400.4	2852.501	7.1065	20.49
27	17418.8	2848.847	7.0948	23.42
28	17437.2	2845.253	7.0831	23.19

ID NMR plot parameters
CX 20.00 cm
F1P 7.895 ppm
F1 3158.84 Hz
F2P 0.282 ppm
F2 112.83 Hz
PPMCH 0.38063 ppm/cm
HZCH 152.30038 Hz/cm

38713-65R-C13
C13CPD MeOH D: / mhuang2 21

6C

Current Data Parameters
NAME 38713-65R-C13
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20020624
Time 22.48
INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG zgpg30
TD 65536
SOLVENT MeOH
NS 1024

ADDRESS	FREQUENCY (Hz)	INTENSITY
1	9570.1	16433.131
2	12357.9	14295.473
3	12364.1	13585.309
4	12949.1	13075.388
5	14034.8	13009.681
6	14287.6	12815.832
7	16460.1	10996.676
8	17534.2	10326.465
9	21604.1	7205.709
10	23102.2	6057.029
11	23546.8	5882.492
12	24394.9	5065.498
13	24426.5	5041.576
14	24454.4	5020.202
15	24482.3	4998.806
16	24510.2	4977.396
17	24538.2	4955.974
18	24566.1	4934.545
19	24594.1	4913.116
20	29589.0	1083.104

CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -3.00 dB
PL12 21.00 dB
PL13 21.00 dB
SF02 400.1316005 MHz

F2 - Processing parameters
SI 32768
SF 100.6125861 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
FIP 236.285 ppm
F1 23771.19 Hz
F2 -13.462 ppm
PPMCH -12.48633 ppm/cm
HZCM 1256.28149 Hz/cm

INDEX	FREQUENCY	PPM	HEIGHT
1	155.18	154.262	86.0
2	151.45	151.156	35.4
3	147.01	146.439	17.1
4	137.02	136.115	15.0
5	136.33	135.095	171.9
6	132.75	131.034	180.0
7	106.65	106.488	84.4
8	78.93	78.489	89.5
9	78.63	78.189	89.5
10	77.72	77.354	89.5
11	77.44	77.037	59.2
12	62.40	62.087	59.2
13	59.44	59.212	163.2
14	57.63	57.309	73.0
15	53.02	52.849	
16	15.34	15.179

Current Data Parameters
 NAME 38974-13
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20020621
 Time 10.20

INSTRUM spect
 PROBHD 5 mm QNP 1H
 PULPROG zg30
 TO 65536
 SOLVENT CDC13

#	ADDRESS	FREQUENCY (Hz)	INTENSITY
1	13378.8	3220.278	8.0481
2	13412.2	3211.841	8.0270
3	13737.4	3129.675	7.8216
4	13773.8	3120.479	7.7987
5	13809.7	3111.412	7.7760
6	14170.6	3020.253	7.5482
7	14175.7	3018.959	7.5442
8	14197.4	3013.473	7.5312
9	14203.3	3011.981	7.5273
10	14209.8	3010.352	7.5234
11	14231.5	3004.858	7.5097
12	14256.7	3003.539	7.5064
13	1448.7	2949.996	7.3726
14	1448.4	2942.491	7.3538
15	14507.4	2935.158	7.3355
16	14525.4	2930.608	7.3241
17	14535.4	2928.086	7.3178
18	14588.9	2922.148	7.3030
19	14590.0	2916.911	7.2897
20	14595.6	2912.877	7.2798
21	14628.6	2904.342	7.2590
22	14644.6	2900.303	7.2489
23	14652.9	2898.392	7.2436
24	14655.8	2897.677	7.2418
25	14667.7	2894.653	7.2343
26	14671.9	2893.594	7.2316
27	14678.8	2891.848	7.2273
28	14692.3	2889.438	7.2187
29	14702.7	2885.809	7.2122
30	14712.3	2883.399	7.2062
31	18213.5	1998.900	4.9956
32	18228.1	1995.203	4.9864
33	19119.0	1770.142	4.4239
34	19168.4	1757.657	4.3927
35	19207.8	1747.692	4.3678
36	19257.5	1735.141	4.3364
37	19824.9	1591.798	3.9782
38	19860.1	1582.902	3.9560
39	19877.4	1578.548	3.9451
40	21226.6	1237.706	3.0933
41	21241.7	1233.689	3.0837
42	21252.5	1231.148	3.0769
43	21267.3	1227.407	3.0675
44	21278.4	1224.616	3.0605
45	21293.1	1220.883	3.0512
46	21303.9	1218.178	3.0445
47	21318.9	1214.388	3.0350
48	24771.2	342.228	0.8453
49	24797.2	335.650	0.8388
50	24825.8	328.421	0.8288

38974-C13
C13CPD CDC13 D: / mhuang2 20

Current Data Parameters
NAME 38974-13C13
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20020624
Time 21.45
INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG zgpg30
TD 65536
SOLVENT CDC13
NS 1024

ADDRESS	FREQUENCY [MHz]	INTENSITY
1	10408.0	15647.757
2	12169.9	14296.774
3	13283.0	13443.320
4	13787.1	13056.796
5	13807.3	13041.287
6	13875.8	12988.763
7	13954.8	12928.206
8	14085.6	12820.218
9	14105.8	12812.377
10	14213.2	12730.061
11	14384.3	12448.376
12	14631.2	12394.173
13	14944.0	12169.975
14	15931.1	11412.786
15	20822.2	7838.774
16	20834.0	7806.736
17	20873.8	7774.899
18	21204.4	7389.365
19	23149.6	5677.862
20	23356.1	5718.002
21	25423.1	4134.587
22	28603.0	1341.421

F2 - Processing parameters
SI 32768
SF 100.6127290 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
F1P 234.844 ppm
F1 23628.35 Hz
F2P -14.882 ppm
F2 -1497.28 Hz
PPMCH 12.48631 ppm/cm
HZCH 1256.28137 Hz/cm

WT-38267-71 (shorttailBoc)
 PROTON COC13 D: / jx1e2 2

D:/data/jx1e2/nmr/Jun12-2002-jx1/1/pdata/1/screen

WD-D:/, USER-jx1e2, NAME-Jun12-2002-jx1, EXPNO=1, PROCNO=1
 F1=8.943ppm, F2=-0.099ppm, NI=0.34cm, MAXI=1000.00cm, FC=1.000

	ADDRESS	F1 (Hz)	F2 (Hz)	INTENSITY
1	12746.2	3328.718	8.3191	3.47
2	12755.1	3326.470	8.3135	3.53
3	14188.8	2964.283	7.4083	3.10
4	14197.3	2962.127	7.4029	3.09
5	14255.8	2947.365	7.3660	4.38
6	14264.4	2945.182	7.3606	4.49
7	14268.8	2853.111	7.1305	31.80
8	14827.4	2802.956	7.0051	3.81
9	14836.0	2800.790	6.9997	3.72
10	18361.3	1910.188	4.7739	2.89
11	18402.3	1899.833	4.7480	2.95
12	19679.7	1577.113	3.9415	0.69
13	20322.1	1414.827	3.5261	0.44
14	20337.7	1410.884	3.5359	0.51
15	20376.9	1406.984	3.5013	0.88
16	20393.5	1396.781	3.4908	0.86
17	20431.9	1387.091	3.4666	0.60
18	20447.4	1383.187	3.4568	0.57
19	20901.3	1268.501	3.1702	0.52
20	20918.8	1264.079	3.1592	1.03
21	20935.7	1259.808	3.1485	0.58
22	20970.6	1250.992	3.1265	0.72
23	20988.5	1246.470	3.1132	1.37
24	21005.7	1242.139	3.1043	0.66
25	21348.4	1155.567	2.9880	0.58
26	21365.4	1151.296	2.8773	0.71
27	21400.5	1142.383	2.8550	0.60
28	21417.7	1138.043	2.8432	1.08
29	21434.9	1133.692	2.8333	0.58
30	21470.1	1124.815	2.8111	0.43
31	21486.9	1120.339	2.8005	0.47
32	21634.4	1083.296	2.7074	0.80
33	21652.2	1078.819	2.6962	1.49
34	21670.1	1074.293	2.6849	0.74
35	21692.9	1068.538	2.6705	0.78
36	21710.8	1063.993	2.6591	1.39
37	21728.7	1059.470	2.6478	0.60
38	22113.2	962.336	2.4031	0.90
39	22513.5	861.224	2.1524	0.57
40	22551.5	851.617	2.1284	0.58
41	22576.7	845.244	2.1124	0.40
42	23635.7	577.713	1.4438	52.85
43	23773.0	543.030	1.3571	0.46
44	23851.5	523.192	1.3076	76.30
45	23975.3	491.933	1.2284	0.50
46	24023.3	479.809	1.1991	1.84
47	24050.6	472.910	1.1819	1.24
48	24105.9	458.930	1.1470	0.38

1D NMR plot parameters
 CX 20.00 cm
 F1P 8.943 ppm
 F1 3578.47 Hz
 F2P -0.099 ppm
 F2 -39.49 Hz
 PPMCH 0.45210 ppm/cm
 HZCM 180.89832 Hz/cm

File : c:\nouveau\chrom\3893694j
 Method : c:\nouveau\methods\ligand2.met
 Pretreatment : c:\nouveau\testmlx.apg
 Sample ID : 38936-94j
 Volume : 15
 Acquired : Jan 05, 1998 15:22:21
 Printed : Jan 05, 1998 15:39:41
 User : System
 c:\nouveau\chrom\3893694j - Channel A PDA Channel 1

Channel A Results -- PDA Channel 1, 277 nm, 4 nm Band

Peak	Time	Area	Area %
1	3.83	121382	0.491
2	4.35	23563114	95.386
3	5.42	84443	0.342
4	5.97	8102	0.033
5	6.42	130073	0.527
6	6.85	17082	0.069
7	7.42	161524	0.654
8	9.28	598728	2.424
9	10.95	18482	0.075
Totals :			
		24702932	100.000

After ligand removed.

95.386

$$\frac{95.386 - 2.424}{95.386 + 2.424} \cdot 100 = 92.962$$

$$\frac{97.81}{100} = 97.81$$

ZD-39275-21

R?
PROTON CDC13 D / chenx 49

8.27

X
H₂O

Chemical Shift (ppm)	Integration
1.0090	1.0090
1.0592	1.0592
1.0059	1.0059
1.0417	1.0417
1.0516	1.0516
1.0651	1.0651
1.0707	1.0707
2.4369	2.4369
0.7686	0.7686
1.0711	1.0711
2.1128	2.1128
0.1427	0.1427
1.0724	1.0724
2.2565	2.2565
2.3476	2.3476

ppm

ADDRESS	FREQUENCY (Hz)	FREQUENCY (PPM)	INTENSITY
1	12781.4	9.4454	10.47
2	12740.4	9.4398	10.33
3	12700.2	9.4307	9.89
4	12659.0	9.4222	9.59
5	12617.6	9.4141	13.12
6	12576.0	9.4062	13.21
7	12534.2	9.3983	13.84
8	12492.5	9.3904	11.90
9	12450.7	9.3825	11.39
10	12408.9	9.3746	9.42
11	12367.1	9.3667	1.66
12	12325.3	9.3588	1.88
13	12283.5	9.3509	2.72
14	12241.7	9.3430	2.83
15	12199.9	9.3351	2.18
16	12158.1	9.3272	2.07
17	12116.3	9.3193	1.66
18	12074.5	9.3114	1.35
19	12032.7	9.3035	1.72
20	11990.9	9.2956	2.33
21	11949.1	9.2877	4.41
22	11907.3	9.2798	2.12
23	11865.5	9.2719	3.09
24	11823.7	9.2640	3.91
25	11781.9	9.2561	6.96
26	11740.1	9.2482	2.83
27	11698.3	9.2403	4.21
28	11656.5	9.2324	2.36
29	11614.7	9.2245	2.00
30	11572.9	9.2166	3.54
31	11531.1	9.2087	1.88
32	11489.3	9.2008	1.45
33	11447.5	9.1929	1.60
34	11405.7	9.1850	2.70
35	11363.9	9.1771	5.17
36	11322.1	9.1692	2.43
37	11280.3	9.1613	2.63
38	11238.5	9.1534	4.77
39	11196.7	9.1455	2.03
40	11154.9	9.1376	2.29
41	11113.1	9.1297	2.82
42	11071.3	9.1218	3.89
43	11029.5	9.1139	3.06
44	10987.7	9.1060	6.27
45	10945.9	9.0981	4.92
46	10904.1	9.0902	4.35
47	10862.3	9.0823	2.24
48	10820.5	9.0744	2.43
49	10778.7	9.0665	1.64
50	10736.9	9.0586	1.90
51	10695.1	9.0507	2.08
52	10653.3	9.0428	1.95
53	10611.5	9.0349	1.59
54	10569.7	9.0270	1.23
55	10527.9	9.0191	1.14
56	10486.1	9.0112	1.44
57	10444.3	9.0033	1.52
58	10402.5	8.9954	5.32
59	10360.7	8.9875	4.69
60	10318.9	8.9796	1.46
61	10277.1	8.9717	3.23
62	10235.3	8.9638	3.12
63	10193.5	8.9559	1.83
64	10151.7	8.9480	2.07
65	10109.9	8.9401	3.30
66	10068.1	8.9322	4.42
67	10026.3	8.9243	7.69
68	9984.5	8.9164	7.62
69	9942.7	8.9085	3.02
70	9900.9	8.9006	2.02
71	9859.1	8.8927	1.32
72	9817.3	8.8848	1.07

-D1/, USER-jx1e2, NAME-Jun13-2002-jx1, EXPNO=7, PROCNO=1
-160.960ppm, F2=27.338ppm, H1=0.00cm, MAXI=10000.00cm, PC=

8 ADDRESS
9 FREQUENCY
10 INTENSITY

1	10368.9	15677.726	155.8225	8.05
2	11451.7	14847.453	147.5703	10.13
3	12052.1	14387.081	142.9846	10.02
4	12227.6	14252.519	141.6572	10.09
5	12700.8	13869.685	138.0510	8.43
6	13043.0	13627.351	135.4436	10.31
7	13340.8	13398.938	133.1734	8.04
8	13584.9	13211.819	131.3136	10.76
9	13836.2	13019.131	129.3884	12.50
10	13943.9	12956.501	128.5772	0.61
11	14064.2	12844.329	127.4611	8.21
12	15190.0	11981.063	119.0810	9.22
13	20595.6	7835.385	77.8767	10.34
14	20638.3	7803.416	77.5589	10.44
15	20680.1	7771.408	77.2408	10.40
16	23048.3	5955.431	59.1922	10.60
17	24615.9	4732.767	47.2392	9.03
18	24646.1	4730.364	47.0156	9.11
19	25241.2	4274.099	42.4807	9.47
20	28435.9	3358.006	33.3756	8.89
21	28566.2	3258.105	32.3826	10.65
22	28604.0	3229.122	32.0946	10.22
23	28615.9	3219.973	32.0036	9.32

66336-R
C13CPD CDC13 D: / jx1e2 28

Current Data Parameters
NAME Jun13-2002-jx1
EXPNO 7
PROCNO 1

F2 - Acquisition Parameters
Date_ 20020613
Time 16.13

INSTRUM spect
PROBHD 5 mm GNP 1H
PULPROG zgpg30
TD 65536
SOLVENT CDC13
NS 128
DS 4

SWH 25125.620 Hz
FIDRES 0.383387 Hz
AQ 1.3042164 sec
RG 16384
DM 19.900 USEC
DE 6.00 USEC
TE 300.0 K

D1 2.00000000 sec
d11 0.03000000 sec
d12 0.00002000 sec

===== CHANNEL f1 =====
NUC1 13C
P1 9.00 usec
PL1 -3.00 dB
SF01 100.6237959 MHz

===== CHANNEL f2 =====
CPOPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -3.00 dB
PL12 21.00 dB
PL13 21.00 dB
SF02 400.1316005 MHz

F2 - Processing parameters
SI 32768
SF 100.6127290 MHz
WDW EN
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
FIP 160.960 ppm
F1 16194.59 Hz
F2 27.338 ppm
F2 2750.58 Hz
PPMCH 6.68107 ppm/cm
HZCH 672.20087 Hz/cm

3-Des-Bromo-Shorttail-T
 PROTON CCl3 D: / chenx 11

79366-67

49

Current Data Parameters
 NAME 79366-67
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20020626
 Time 14.19

INSTRUM spect
 PROBHD 5 mm QNP 1H
 PULPROG zg30
 TD 65536
 SOLVENT CCl3
 NS 16

#	ADDRESS	FREQUENCY (MHz)	F2 (PPM)	INTENSITY
1	12854.8	331.444	8.3259	5.00
2	12871.8	327.658	8.3164	4.81
3	14323.8	296.841	7.3997	7.43
4	14329.2	295.459	7.3962	7.04
5	14479.2	292.398	7.3011	4.27
6	14509.6	291.896	7.2824	4.62
7	14871.2	282.541	7.0541	7.73
8	14920.0	281.030	7.0233	3.95
9	14936.5	280.538	7.0116	4.30
10	14949.8	280.241	7.0044	4.04
11	14966.3	279.944	6.9927	3.29
12	18342.3	1945.657	4.8626	4.79
13	18383.0	1935.355	4.8368	4.85
14	19708.0	1600.637	4.0003	2.57
15	20313.0	1447.782	3.6183	1.26
16	20327.0	1444.249	3.6095	1.34
17	20368.9	1433.677	3.5830	2.44
18	20379.4	1431.005	3.5763	2.39
19	20421.9	1420.285	3.5495	1.57
20	20436.2	1416.673	3.5405	1.41
21	20520.5	1294.320	3.2347	2.40
22	20536.5	1290.269	3.2246	1.53
23	20573.8	1280.849	3.2011	2.10
24	20589.3	1276.937	3.1913	3.04
25	21393.2	1174.896	2.9363	1.69
26	21407.8	1171.213	2.9271	1.79
27	21460.3	1157.953	2.8939	2.64
28	21671.4	1104.628	2.7607	1.96
29	21688.3	1100.341	2.7500	2.91
30	21706.0	1095.883	2.7398	1.85
31	21729.0	1090.056	2.7243	2.00
32	21746.6	1085.626	2.7132	2.68
33	21763.0	1081.480	2.7028	1.43
34	22134.3	987.674	2.4684	3.32
35	22468.1	903.361	2.2577	2.25
36	22480.2	890.304	2.2500	2.15
37	22517.7	880.819	2.2363	2.02
38	22655.0	856.138	2.1396	1.19
39	22942.7	783.458	1.9980	1.43
40	23415.2	664.082	1.6597	1.25
41	23891.3	543.813	1.3591	92.27
42	24025.5	509.897	1.2743	6.65
43	24144.1	479.954	1.1995	1.15
44	24387.0	423.625	1.0587	2.66

79366-67-C13
C13CPD CDC13 D: / chenx 11

4g

78501134
10-des-shorttail
PROTON CDC13 D: / chenx 3

4f

Current Data Parameters
NAME JUL10-2002
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20020710
Time 15.26

INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG zg30
TD 65536
SOLVENT CDC13
NS 16
DS 2

SHH 8278.146 Hz

F1 FREQS	ADDRESS	F2 FREQS	INTENSITY
1	12845.1	3329.514	8.3211
2	12852.0	3327.568	8.3162
3	14159.4	2997.474	7.4912
4	14628.0	2878.904	7.1949
5	14830.4	2827.976	7.0676
6	14837.4	2826.214	7.0632
7	14865.8	2819.025	7.0453
8	14874.1	2816.936	7.0403
9	14897.9	2810.917	7.0350
10	14906.3	2808.807	7.0319
11	14933.5	2801.939	7.0026
12	14965.7	2793.602	6.9822
13	17739.8	2092.984	5.2308
14	18701.8	1597.306	3.9920
15	18970.8	1528.594	3.8226
16	20010.5	1519.321	3.7971
17	20630.0	1362.821	3.4059
18	20665.3	1353.753	3.3833
19	20841.0	1309.323	3.2727
20	21495.8	1146.827	2.8656
21	21541.9	1132.450	2.8302
22	21583.6	1025.420	2.5627
23	22086.9	994.759	2.4861
24	22439.4	905.728	2.2636
25	22567.8	868.219	2.1698
26	23511.3	634.322	1.9868
27	23616.4	608.363	1.5204
28	23864.1	545.793	1.3640
29	23942.8	525.916	1.3144
30	24093.3	487.887	1.2193
31	24117.5	481.782	1.2041
32	24135.2	477.315	1.1929
33	24187.4	464.112	1.1599
34	24238.2	451.279	1.1278
35	24254.8	447.093	1.1174
36	24287.9	438.730	1.0963
37	24302.7	434.992	1.0871
38	24339.9	425.602	1.0637
39	24350.2	422.996	1.0571
40	24384.3	414.384	1.0356
41	24740.9	324.303	0.8105
42	26024.6	0.002	0.0000

10-des-bromo-shorttail-C13
C13CPD CDC13 D: / chenx 57

#	ADDRESS	FREQUENCY [Hz]	FREQUENCY [PPM]	INTENSITY
1	12916.5	3311.397	8.3758	2.93
2	12930.4	3307.889	8.3670	2.97
3	12943.1	3299.618	8.3464	0.35
4	12969.1	3298.115	8.3426	0.34
5	12982.2	3294.803	8.3343	0.30
6	12988.1	3293.317	8.3306	0.29
7	14384.9	2940.446	7.3487	1.87
8	14415.5	2932.706	7.3294	2.18
9	14449.9	2924.023	7.3077	0.41
10	14628.5	2878.905	7.1949	6.45
11	14716.6	2856.651	7.1393	0.33
12	14748.3	2848.626	7.1193	0.49
13	14838.0	2825.984	7.0427	6.19
14	14868.9	2818.173	7.0348	0.96
15	14882.2	2814.822	7.0348	2.12
16	14896.2	2809.755	7.0196	19.02
17	14910.9	2807.563	7.0166	18.16
18	19259.2	1706.521	4.2649	1.38
19	19710.9	1594.934	3.9860	1.13
20	19842.1	1561.787	3.9032	0.35
21	19916.1	1543.093	3.8565	1.43
22	19956.3	1532.943	3.8311	1.44
23	20604.8	1369.123	3.4217	0.72
24	20770.0	1327.380	3.3174	0.76
25	21152.3	1230.807	3.0760	1.47
26	21536.8	1133.665	2.8332	1.15
27	21963.2	1025.944	2.5640	0.33
28	22095.2	982.588	2.4807	1.29
29	22535.6	881.334	2.2026	0.67
30	22685.1	843.559	2.1082	0.28
31	22694.4	841.229	2.1024	2.86
32	22709.5	837.418	2.0929	0.29
33	23408.4	660.843	1.6516	3.34
34	23619.5	607.521	1.5183	0.51
35	23689.9	589.724	1.4738	1.03
36	23695.8	588.239	1.4701	1.31
37	23768.9	569.783	1.4240	0.30
38	23788.9	564.715	1.4113	0.71
39	23830.7	554.165	1.3850	0.96
40	23867.1	544.975	1.3620	135.67
41	23948.2	524.471	1.3108	0.80
42	23975.8	517.508	1.2833	0.58
43	24075.1	492.422	1.2307	2.03
44	24119.9	481.092	1.2023	1.66
45	24147.2	474.201	1.1851	1.16
46	24198.7	461.187	1.1526	0.65
47	24215.2	457.015	1.1422	0.71
48	24248.1	448.711	1.1214	1.22
49	24262.9	444.973	1.1121	1.76
50	24296.2	436.550	1.0910	1.12
51	24311.9	432.605	1.0812	1.10
52	24344.5	424.365	1.0606	0.50
53	24361.7	420.023	1.0497	0.43

SI 32788
SF 100.6127290 MHz
WDW EN
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

ID NMR plot parameters
CX 20.00 cm
F1P 161.421 ppm
F1 16241.06 Hz
F2P 26.007 ppm
F2 2616.60 Hz
PPMCH 6.77074 ppm/cm
HZCM 681.22296 Hz/cm

78501-132/cyclohexyl-J
PROTON CDC13 D: / chemx 43

4e

* CDCl₃

65	24135.7	476.251	1.1902	0.66
66	24167.2	468.233	1.1704	0.93
67	24178.9	465.340	1.1630	0.75
68	24212.1	456.944	1.1420	0.84
69	24221.0	454.715	1.1364	1.10
70	24231.0	452.176	1.1301	0.90
71	24272.8	441.619	1.1037	1.02
72	24282.6	439.151	1.0975	0.96
73	24324.0	428.674	1.0713	1.46
74	24330.6	427.012	1.0672	1.54
75	24364.4	418.481	1.0459	2.15
76	24387.9	412.342	1.0310	1.60
77	24399.5	409.415	1.0237	1.82
78	24437.1	400.115	1.0000	0.93
79	24450.3	396.771	0.9916	0.67
80	24486.7	387.588	0.9687	0.29
81	24590.7	361.313	0.9030	0.28
82	24616.9	354.693	0.8864	0.31
83	24707.9	331.696	0.8250	0.31
84	24734.5	324.988	0.8122	0.36
85	24762.3	317.943	0.7946	0.36
86	26020.9	-0.001	-0.0000	0.48

ADDRESS	(Hz)	FREQUENCY (MHz)	INTENSITY
1	12766.1	3348.545	3.12
2	12775.1	3346.276	8.3686
3	14226.0	2979.740	3.16
4	14230.2	2978.663	7.4469
5	14234.7	2977.543	2.42
6	14289.1	2963.791	7.4114
7	14297.6	2961.642	7.4071
8	14323.5	2878.054	7.4017
9	14713.0	2856.709	7.1328
10	14866.2	2818.008	7.1395
11	14874.7	2815.843	7.0427
12	14873.7	2810.842	7.0373
13	14844.1	1914.107	5.7752
14	14844.8	1903.845	4.7937
15	20272.0	1452.332	4.7981
16	20289.7	1447.854	3.6297
17	20284.4	1439.107	2.97
18	20329.6	1437.790	3.6185
19	20342.1	1434.626	3.5966
20	20381.4	1424.707	3.5854
21	20399.2	1420.202	3.5494
22	20887.3	1296.895	3.2412
23	20905.4	1292.322	3.2298
24	20923.7	1287.705	3.2182
25	20956.5	1279.402	3.1975
26	20974.7	1274.805	3.1860
27	20992.8	1270.251	3.1746
28	21387.2	1170.600	2.9256
29	21405.8	1165.897	2.9138
30	21438.6	1157.627	2.8931
31	21456.8	1153.013	2.8816
32	21474.9	1148.451	2.8702
33	21507.6	1140.182	2.8495
34	21526.2	1135.482	2.8378
35	21685.3	1095.303	2.7374
36	21703.7	1090.641	2.7257
37	21722.2	1085.972	2.7140
38	21743.8	1080.513	2.7004
39	21762.3	1075.839	2.6887
40	21780.8	1071.178	2.6771
41	22665.6	847.646	2.1184
42	22678.6	844.346	2.1102
43	22696.0	839.874	2.0993
44	22708.0	836.835	2.0917
45	22720.7	833.714	2.0836
46	22737.1	829.581	2.0733
47	22750.0	826.324	2.0651
48	22762.9	823.055	2.0570
49	22782.2	815.461	2.0385
50	23392.7	663.956	1.6593
51	23399.8	662.167	1.6549
52	23411.7	659.162	1.6474
53	23448.8	649.777	1.6239
54	23490.5	639.239	1.5976
55	23521.3	631.456	1.5781
56	23563.9	620.692	1.5512
57	23597.2	612.290	1.5302
58	23602.3	610.996	1.5270
59	23704.4	585.204	1.4625
60	23754.9	572.441	1.4306
61	23871.8	542.829	1.3569
62	23909.6	533.359	1.3330
63	24117.9	480.755	1.2015
64	24131.0	477.429	1.1932

Current Data Parameters
NAME Jun13-2002-jx1
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20020613
Time 10.20

INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG zg30
TD 65536
SOLVENT CDC13
NS 16
DS 2
SHH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 574.7
DW 60.400 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec

43	22402.5	941.134	2.3521
44	22425.3	935.370	2.3377
45	22556.2	902.303	2.2550
46	22582.0	893.269	2.2324
47	22619.9	886.202	2.2148
48	22645.3	879.795	2.1988
49	22666.9	874.345	2.1852
50	22704.9	864.731	2.1611
51	22892.1	817.437	2.0429
52	22948.8	803.120	2.0071
53	22989.8	792.771	1.9813
54	23003.0	785.428	1.9729
55	23017.2	785.829	1.9639
56	23030.9	782.384	1.9553
57	23050.2	787.825	1.9490
58	23066.5	774.849	1.9366
59	23080.7	651.056	1.6271
60	23094.2	632.485	1.6271
61	23094.2	617.873	1.5442
62	23102.1	601.229	1.5026
63	23102.1	598.447	1.4956
64	23102.1	592.358	1.4804
65	23102.1	588.119	1.4696
66	23102.1	580.747	1.4514
67	23102.1	578.017	1.4446
68	23102.1	564.450	1.4117
69	23102.1	554.399	1.3855
70	23102.1	549.182	1.3725
71	23102.1	541.942	1.3544
72	23102.1	538.661	1.3462
73	23102.1	530.871	1.3267
74	23102.1	526.976	1.3170
75	23102.1	516.624	1.2911
76	23102.1	509.518	1.2734
77	23102.1	502.389	1.2556
78	23102.1	495.222	1.2377
79	23102.1	485.839	1.2142
80	23102.1	479.725	1.1989
81	23102.1	446.567	1.1161
82	23102.1	434.479	1.0858
83	23102.1	350.592	0.8762
84	23102.1	24740.1	0.8762

Frequency (Hz)	Intensity
12751.1	0.4457
12757.9	0.4401
12759.9	0.5530
12761.9	0.5530
12763.9	0.5530
12765.9	0.5530
12767.9	0.5530
12769.9	0.5530
12771.9	0.5530
12773.9	0.5530
12775.9	0.5530
12777.9	0.5530
12779.9	0.5530
12781.9	0.5530
12783.9	0.5530
12785.9	0.5530
12787.9	0.5530
12789.9	0.5530
12791.9	0.5530
12793.9	0.5530
12795.9	0.5530
12797.9	0.5530
12799.9	0.5530
12801.9	0.5530
12803.9	0.5530
12805.9	0.5530
12807.9	0.5530
12809.9	0.5530
12811.9	0.5530
12813.9	0.5530
12815.9	0.5530
12817.9	0.5530
12819.9	0.5530
12821.9	0.5530
12823.9	0.5530
12825.9	0.5530
12827.9	0.5530
12829.9	0.5530
12831.9	0.5530
12833.9	0.5530
12835.9	0.5530
12837.9	0.5530
12839.9	0.5530
12841.9	0.5530
12843.9	0.5530
12845.9	0.5530
12847.9	0.5530
12849.9	0.5530
12851.9	0.5530
12853.9	0.5530
12855.9	0.5530
12857.9	0.5530
12859.9	0.5530
12861.9	0.5530
12863.9	0.5530
12865.9	0.5530
12867.9	0.5530
12869.9	0.5530
12871.9	0.5530
12873.9	0.5530
12875.9	0.5530
12877.9	0.5530
12879.9	0.5530
12881.9	0.5530
12883.9	0.5530
12885.9	0.5530
12887.9	0.5530
12889.9	0.5530
12891.9	0.5530
12893.9	0.5530
12895.9	0.5530
12897.9	0.5530
12899.9	0.5530
12901.9	0.5530
12903.9	0.5530
12905.9	0.5530
12907.9	0.5530
12909.9	0.5530
12911.9	0.5530
12913.9	0.5530
12915.9	0.5530
12917.9	0.5530
12919.9	0.5530
12921.9	0.5530
12923.9	0.5530
12925.9	0.5530
12927.9	0.5530
12929.9	0.5530
12931.9	0.5530
12933.9	0.5530
12935.9	0.5530
12937.9	0.5530
12939.9	0.5530
12941.9	0.5530
12943.9	0.5530
12945.9	0.5530
12947.9	0.5530
12949.9	0.5530
12951.9	0.5530
12953.9	0.5530
12955.9	0.5530
12957.9	0.5530
12959.9	0.5530
12961.9	0.5530
12963.9	0.5530
12965.9	0.5530
12967.9	0.5530
12969.9	0.5530
12971.9	0.5530
12973.9	0.5530
12975.9	0.5530
12977.9	0.5530
12979.9	0.5530
12981.9	0.5530
12983.9	0.5530
12985.9	0.5530
12987.9	0.5530
12989.9	0.5530
12991.9	0.5530
12993.9	0.5530
12995.9	0.5530
12997.9	0.5530
12999.9	0.5530
13001.9	0.5530
13003.9	0.5530
13005.9	0.5530
13007.9	0.5530
13009.9	0.5530
13011.9	0.5530
13013.9	0.5530
13015.9	0.5530
13017.9	0.5530
13019.9	0.5530
13021.9	0.5530
13023.9	0.5530
13025.9	0.5530
13027.9	0.5530
13029.9	0.5530
13031.9	0.5530
13033.9	0.5530
13035.9	0.5530
13037.9	0.5530
13039.9	0.5530
13041.9	0.5530
13043.9	0.5530
13045.9	0.5530
13047.9	0.5530
13049.9	0.5530
13051.9	0.5530
13053.9	0.5530
13055.9	0.5530
13057.9	0.5530
13059.9	0.5530
13061.9	0.5530
13063.9	0.5530
13065.9	0.5530
13067.9	0.5530
13069.9	0.5530
13071.9	0.5530
13073.9	0.5530
13075.9	0.5530
13077.9	0.5530
13079.9	0.5530
13081.9	0.5530
13083.9	0.5530
13085.9	0.5530
13087.9	0.5530
13089.9	0.5530
13091.9	0.5530
13093.9	0.5530
13095.9	0.5530
13097.9	0.5530
13099.9	0.5530
13101.9	0.5530
13103.9	0.5530
13105.9	0.5530
13107.9	0.5530
13109.9	0.5530
13111.9	0.5530
13113.9	0.5530
13115.9	0.5530
13117.9	0.5530
13119.9	0.5530
13121.9	0.5530
13123.9	0.5530
13125.9	0.5530
13127.9	0.5530
13129.9	0.5530
13131.9	0.5530
13133.9	0.5530
13135.9	0.5530
13137.9	0.5530
13139.9	0.5530
13141.9	0.5530
13143.9	0.5530
13145.9	0.5530
13147.9	0.5530
13149.9	0.5530
13151.9	0.5530
13153.9	0.5530
13155.9	0.5530
13157.9	0.5530
13159.9	0.5530
13161.9	0.5530
13163.9	0.5530
13165.9	0.5530
13167.9	0.5530
13169.9	0.5530
13171.9	0.5530
13173.9	0.5530
13175.9	0.5530
13177.9	0.5530
13179.9	0.5530
13181.9	0.5530
13183.9	0.5530
13185.9	0.5530
13187.9	0.5530
13189.9	0.5530
13191.9	0.5530
13193.9	0.5530
13195.9	0.5530
13197.9	0.5530
13199.9	0.5530
13201.9	0.5530
13203.9	0.5530
13205.9	0.5530
13207.9	0.5530
13209.9	0.5530
13211.9	0.5530
13213.9	0.5530
13215.9	0.5530
13217.9	0.5530
13219.9	0.5530
13221.9	0.5530
13223.9	0.5530
13225.9	0.5530
13227.9	0.5530
13229.9	0.5530
13231.9	0.5530
13233.9	0.5530
13235.9	0.5530
13237.9	0.5530
13239.9	0.5530
13241.9	0.5530
13243.9	0.5530
13245.9	0.5530
13247.9	0.5530
13249.9	0.5530
13251.9	0.5530
13253.9	0.5530
13255.9	0.5530
13257.9	0.5530
13259.9	0.5530
13261.9	0.5530
13263.9	0.5530
13265.9	0.5530
13267.9	0.5530
13269.9	0.5530
13271.9	0.5530
13273.9	0.5530
13275.9	0.5530
13277.9	0.5530
13279.9	0.5530
13281.9	0.5530
13283.9	0.5530
13285.9	0.5530
13287.9	0.5530
13289.9	0.5530
13291.9	0.5530
13293.9	0.5530
13295.9	0.5530
13297.9	0.5530
13299.9	0.5530
13301.9	0.5530
13303.9	0.5530
13305.9	0.5530
13307.9	0.5530
13309.9	0.5530
13311.9	0.5530
13313.9	0.5530
13315.9	0.5530
13317.9	0.5530
13319.9	0.5530
13321.9	0.5530
13323.9	0.5530
13325.9	0.5530
13327.9	0.5530
13329.9	0.5530
13331.9	0.5530
13333.9	0.5530
13335.9	0.5530
13337.9	0.5530
13339.9	0.5530
13341.9	0.5530
13343.9	0.5530
13345.9	0.5530
13347.9	0.5530
13349.9	0.5530
13351.9	0.5530
13353.9	0.5530
13355.9	0.5530
13357.9	0.5530
13359.9	0.5530
13361.9	0.5530
13363.9	0.5530
13365.9	0.5530
13367.9	0.5530
13369.9	0.5530
13371.9	0.5530
13373.9	0.5530
13375.9	0.5530
13377.9	0.5530
13379.9	0.5530
13381.9	0.5530
13383.9	0.5530
13385.9	0.5530
13387.9	0.5530
13389.9	0.5530
13391.9	0.5530
13393.9	0.5530
13395.9	0.5530
13397.9	0.5530
13399.9	0.5530
13401.9	0.5530
13403.9	0.5530
13405.9	0.5530
13407.9	0.5530
13409.9	0.5530
13411.9	0.5530
13413.9	0.5530
13415.9	0.5530
13417.9	0.5530
13419.9	0.5530
13421.9	0.5530
13423.9	0.5530
13425.9	0.5530
13427.9	0.5530
13429.9	0.5530
13431.9	0.5530
13433.9	0.5530
13435.9	0.5530
13437.9	0.5530
13439.9	0.5530
13441.9	0.5530
13443.9	0.5530
13445.9	0.5530
13447.9	0

Current Data Parameters
NAME Jun13-2002-jxi
EXPNO 3
PROCNO 1

F2 - Acquisition Parameters

Date_ 20020613
Time 14.43
INSTRUM spect
PROBHD 5 mm QNP 1H

#	ADDRESS	FREQUENCY (Hz)	FREQUENCY (PPM)	INTENSITY
1	8501.0	17111.391	170.0718	9.09
2	8503.9	17107.662	170.0348	6.82
3	10456.1	15612.239	155.1716	5.56
4	10464.2	15606.061	155.1102	11.59
5	10469.2	15602.236	155.0722	4.30
6	11439.2	14858.446	147.6796	4.24
7	11449.8	14850.352	147.5991	4.85
8	12035.2	14401.487	143.1378	4.25
9	12047.9	14391.686	143.0404	7.16
10	12206.8	14269.847	141.8294	4.14
11	12223.1	14257.366	141.7054	3.69
12	12752.7	13851.258	137.6690	4.31
13	12784.8	13826.675	137.4247	5.68
14	13011.9	13652.523	135.6938	6.75
15	13030.0	13638.665	135.5561	5.59
16	13306.1	13426.994	133.4522	7.21
17	13580.9	13216.246	131.3576	5.92
18	13585.3	13212.898	131.3243	4.81
19	13810.9	13035.887	129.5047	5.35
20	13817.6	13034.787	129.5541	7.14
21	14084.0	12830.495	127.5256	4.33
22	14097.7	12819.972	127.4190	9.45
23	15169.6	11998.062	119.2500	17.89
24	20381.0	8002.146	79.5341	23.74
25	20378.8	7850.465	78.0266	24.78
26	20620.7	7818.355	77.7074	22.50
27	20662.5	7786.289	77.3887	5.01
28	23142.7	5884.513	58.4868	4.70
29	23150.7	5878.423	58.4262	2.80
30	24744.1	4656.582	46.2822	3.16
31	24762.3	4642.656	46.1438	1.37
32	24964.8	4487.363	44.6004	4.08
33	25259.2	4261.634	42.3568	6.96
34	25273.1	4250.995	42.2511	2.97
35	25297.5	4232.314	42.0654	7.51
36	25585.2	4011.676	39.8725	13.50
37	26413.0	3376.938	33.5637	7.80
38	26544.1	3276.439	32.5649	5.22
39	26562.0	3262.705	32.4284	4.36
40	26570.7	3255.985	32.3616	4.96
41	26600.9	3232.829	32.1314	3.84
42	26674.2	3176.692	31.5735	3.28
43	26807.7	3074.321	30.5560	63.60
44	27035.1	2899.940	28.8228	1.62
45	27437.9	2591.061	25.7528	1.44
46	28910.1	1462.221	14.5332	

10 mm pvt parameters

CX 20.00 cm
FIP 177.592 ppm
F1 17868.00 Hz
F2 6.242 ppm
F3 628.03 Hz
PPMCH 8.56749 ppm/cm
HZCM 861.99884 Hz/cm

01-66336-YYC-405
PROTON CDC13 D: / WUG 4

Lonafarnib

Current Data Parameters
NAME 01-66336-YYC-4
EXPNO 1
PROCNO 1

#	ADDRESS	FREQUENCY (Hz)	F1 (ppm)	INTENSITY
1	12750.2	3353.185	8.3802	1.75
2	12750.0	3350.967	8.3747	1.78
3	14160.5	2996.911	7.4896	0.84
4	14168.7	2994.823	7.4846	0.93
5	14179.6	2992.076	7.4778	0.77
6	14187.8	2990.020	7.4726	0.68
7	14248.6	2974.599	7.4341	1.62
8	14255.6	2972.686	7.4298	1.37
9	14628.4	2876.707	7.1944	12.50
10	14798.7	2835.674	7.0869	0.78
11	14810.2	2832.774	7.0796	1.36
12	14819.5	2830.419	7.0737	0.99
13	18349.2	1936.713	4.8452	0.57
14	18365.7	1934.546	4.8348	0.72
15	18389.9	1928.429	4.8195	0.61
16	18406.9	1924.134	4.8088	0.73
17	18438.9	1815.005	4.5360	0.35
18	18483.9	1803.649	4.5077	0.27
19	19148.6	1736.766	4.3405	1.35
20	19921.2	1541.590	3.8527	0.64
21	19972.1	1528.736	3.8206	0.42
22	20020.3	1516.551	3.7901	0.26
23	20074.4	1502.893	3.7560	0.43
24	20129.5	1488.962	3.7212	0.21
25	20344.9	1434.551	3.5852	0.22
26	20363.4	1429.874	3.5735	0.14
27	20382.8	1424.960	3.5612	0.28
28	20399.3	1420.798	3.5508	0.42
29	20438.6	1410.879	3.5261	0.20
30	20455.2	1406.871	3.5155	0.26
31	20475.0	1401.687	3.5031	0.15
32	20905.0	1293.057	3.2316	0.25
33	20921.8	1288.803	3.2210	0.28
34	20938.6	1284.549	3.2103	0.49
35	20975.0	1275.371	3.1874	0.35
36	20993.3	1271.242	3.1772	0.66
37	21008.0	1267.029	3.1665	0.33
38	21341.3	1182.833	2.8551	0.31
39	21393.3	1169.489	2.8253	0.39
40	21410.3	1165.393	2.8125	0.37
41	21463.7	1151.901	2.8788	0.41
42	21522.5	1129.480	2.8228	0.47
43	21603.0	1118.723	2.7909	1.25
44	21647.2	1105.555	2.7630	1.22
45	21679.0	1097.507	2.7429	0.52
46	21689.1	1094.956	2.7365	0.65
47	22275.2	946.898	2.3645	0.24
48	22290.4	943.046	2.3568	0.23
49	22324.2	934.520	2.3385	0.57
50	22339.5	930.663	2.3259	0.52
51	22376.1	921.404	2.3028	0.51
52	22425.3	908.983	2.2717	0.24
53	22435.6	891.118	2.2021	0.20
54	22469.3	872.596	2.1808	0.51
55	22596.1	865.834	2.1639	0.85
56	22629.3	857.438	2.1429	0.84
57	22652.5	851.569	2.1282	0.48
58	22690.1	842.076	2.1045	0.21
59	22867.5	797.257	1.9925	0.19
60	22889.6	791.672	1.9785	0.38
61	22910.4	786.421	1.9654	0.34
62	22923.5	783.118	1.9572	0.32

SI35

66336-YYC-C13
C13CPD DMSO D: / jx1e2 20

Current Data Parameters		F2 - Acquisition Parameters	
NAME	Jul10-2002	Date_	20020710
EXPNO	1	Time	21.35
PROCNO	1	INSTRUM	spect
		PROBHD	5 mm QNP 1H
		PULPROG	zgpg30
		TD	65536
		SOLVENT	DMSO
		NS	4096

#	ADDRESS	FREQUENCY (Hz)	INTENSITY
1	8514.1	17049.656	169.4582
2	9976.4	15928.372	158.3136
3	10397.1	15603.787	155.1074
4	10402.7	15601.541	155.0652
5	11480.6	14775.002	146.8502
6	11826.4	14509.864	144.2149
7	11835.8	14502.659	144.1433
8	12139.0	14270.183	141.8327
9	12664.6	13867.150	137.8269
10	12677.5	13857.245	137.7285
11	12865.5	13713.145	136.2963
12	12874.4	13706.288	136.2281
13	13374.0	13323.188	132.4204
14	13639.0	13120.015	130.4011
15	13715.7	13061.201	129.8165
16	13721.5	13056.740	129.7722
17	14118.9	12752.017	126.7435
18	14123.3	12748.646	126.7100
19	15162.0	11952.243	118.7845
20	23102.6	5863.602	58.2785
21	23109.0	5858.656	58.2397
22	24789.4	4570.191	45.4236
23	24808.1	4555.820	45.2807
24	24984.5	4420.631	43.9371
25	25320.2	4163.184	41.3783
26	25336.9	4150.365	41.2509
27	25399.5	4102.386	40.7740
28	25438.2	4072.729	40.4792
29	25465.6	4051.728	40.2705
30	25482.9	4030.745	40.0620
31	25520.3	4009.773	39.8535
32	25575.7	3987.784	39.6446
33	25575.0	3987.784	39.4362
34	25602.4	3946.788	39.2275
35	25628.4	3926.895	39.0298
36	25634.5	3922.209	38.9832
37	26401.7	3333.920	33.1362
38	26455.0	3223.042	32.7299
39	26455.1	3223.960	32.4032
40	26586.2	3206.286	31.8676
41	26628.1	3160.318	31.4107
42	26639.2	3151.843	31.3265
43	26701.0	3104.459	30.8555
44	26738.3	3075.861	30.5713

F1	17597.58 Hz
F2	24.911 ppm
PRMCM	2506.37 Hz
HZCM	7.54934 ppm/cm
	759.56018 Hz/cm