Supporting Information

Iridium/Diene Complex Catalyzes Allylation Reactions of Ketones and Imines

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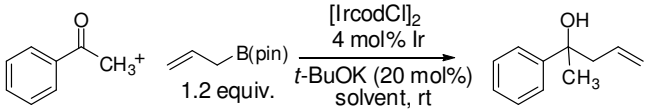
I. General Procedures.

All reactions were carried out in a glovebox under an atmosphere of N_2. All glassware was either oven or flame-dried prior to use. Tetrahydrofuran (THF), diethyl ether (Et_2O), and dichloromethane (CH_2Cl_2) were degassed with argon and then passed through two 4 x 36 inch columns of anhydrous neutral A-2 alumina (8 x 14 mesh; LaRoche Chemicals; activated under a flow of argon at 350 ºC for 12 h) to remove H_2O. All other solvents utilized were purchased "anhydrous" commercially, or purified as described (*vide infra)*. Molarities of organolithium and organomagnesium reagents were determined by titration with menthol/bipyridine.[1] ^{1}H NMR spectra were recorded on Bruker GN-500 (500 MHz ^{1}H, 125.7 MHz ^{13}C), CRYO-500 (500 MHz ^{1}H, 125.7 MHz ^{13}C) or DRX-400 (400 MHz ^{1}H, 100MHz ^{13}C) spectrometers. Proton chemical shifts are reported in ppm (δ) relative to the solvent resonance ($CDCl_3$, δ 7.27) Data are reported as follows: chemical shift (multiplicity [singlet (s), broad singlet (br s), doublet (d), doublet of doublets (dd), triplet (t), doublet of triplets (dt), doublet of doublet of triplets (ddt), triplet of triplets (tt), quartet (q), multiplet (m), apparent doublet (ad), broad doublet (br d) and broad multiplet (br m)], coupling constants [Hz], integration. Carbon chemical shifts are reported in ppm (δ) relative to TMS with the respective solvent resonance as the internal standard ($CDCl_3$, δ 77.23 ppm). Unless otherwise indicated, NMR data were collected at 25 ºC. Infrared spectra were obtained on a Mattson Instruments *Galaxy 5000* spectrometer. Analytical thin-layer chromatography (TLC) was performed using Silica Gel 60 F254 precoated plates (0.25 mm thickness). Visualization was accomplished by irradiation with a UV lamp and/or staining with $KMnO_4$, ceric ammonium molybdate (CAM), or p-anisaldehyde (PAA) solutions. Flash chromatography was performed using Silica Gel 60A (170-400 mesh) from Fisher Scientific. Allylboronic acid pinacol ester was purchased from Frontier Scientific, Inc. and was distilled through a 15 cm Vigreux fractionating column connected to a short-path distillation head (95 ºC, 17 Torr) to remove $B(OH)_3$. $[Ir(cod)Cl]_2$ and $[Ir(cod)OMe]_2$ was purchased from Strem, stored in the glovebox, and used as received. $[Ir(coe)_2Cl]_2$ was synthesized according to a known procedure.[2] Potassium *tert*-butoxide was purchased from Alfa Aesar, stored in a

glovebox and used as received. Deutero-allylboronic acid pinacol ester **9** was prepared according to the procedure reported by Morken et. al.[3] Deuterochloroiodomethane used for the synthesis of **9** was synthesized according to a known procedure.[4] All ketones were purchased commercially and liquids were distilled prior to use. High resolution mass spectrometry was performed by the University of California, Irvine Mass Spectrometry Center.

II. Solvent Screen for Allylation of Ketones.

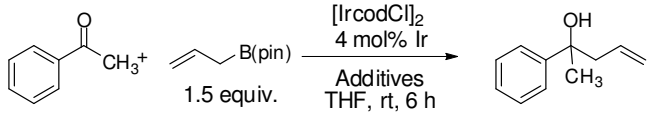


entry	solvent	time (h)	conversion (%)[a]
1	DCM	24	25
2	DMF	24	6
3	THF	24	>95
4	Toluene	24	27

[a] 1,4-bis(trifluoromethyl)benzene was used as an internal standard.

III. $B(OH)_3$ and KO*t*Bu Additives Screen for Allylation of Ketones.

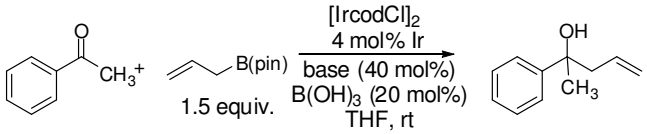
Commercially available allylboronic acid pinacol ester was used in entry 1 and was found to be contaminated with boric acid by ^{13}B NMR. In entries 2-9, the allylboronic acid pinacol ester was distilled and was analytically pure by ^{13}B NMR. Adding 20 mol% of boric acid and 40 mol% KO*t*Bu to reactions with analytically pure allylboronic acid pinacol ester were the optimized conditions that provided reproducible results.



entry	KO*t*Bu (mol%)	$B(OH)_3$ (mol%)	conversion (%)[a]
1	20	none	100
2	20	none	60
3	50	none	82
4	100	none	65
5	50	5	82
6	50	20	94
7	40	20	95
8	30	20	80
9	20	20	15

[a] 1,4-bis(trifluoromethyl)benzene was used as an internal standard.

IV. Base Screen for Allylation of Ketones.



entry	base	time (h)	conversion (%)[a]
1	KO*t*Bu	3	>95
2	NaO*t*Bu	24	<5
3	LiO*t*Bu	24	<5
4	LaO*i*Pr	24	38
5	KOPh	24	0[b]

[a] 1,4-bis(trifluoromethyl)benzene was used as an internal standard.
[b] Under these reaction condition the allylboronic acid pinacol ester was isomerized to the 1-propenylboronic acid pinacol ester.

V. General Procedure for the Allylation of Ketones and Imines.

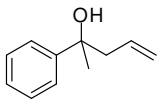
Representative Procedure for Allylation of Acetophenone **1** *with ligands.*
To a flame-dried 5 mL round-bottom flask in a glovebox, $[Ir(cod)Cl]_2$ (7 mg, 0.010 mmol) and 1,2-bis(dicyclohexylphosphino)ethane (11 mg, 0.025 mmol) were stirred in 1 mL of THF for 3 h. After 3 h, potassium *tert*-butoxide (11 mg, 0.10 mmol), acetophenone (58 μL, 0.50 mmol), allylboronic acid pinacol ester (140 μL, 0.75 mmol), and 1,4-bis(trifluoromethyl)benzene (79 μL, 0.50 mmol) (as an internal standard) were added. The reaction mixture was capped with a septa and stirred in the glovebox for 24 h at room temperature. After 24 h, an aliquot was removed and an ^{1}H-NMR was acquired. Conversion and yield of allylated product **3** was then calculated by comparison to the 1,4-bis(trifluoromethyl)benzene standard.

Representative Procedure for Allylation of Acetophenone **5a**.
To a flame-dried 5 mL round-bottom flask in a glovebox, was added $[Ir(cod)Cl]_2$ (7 mg, 0.010 mmol), potassium *tert*-butoxide (22 mg, 0.20 mmol), boric acid (6.2 mg, 0.10 mmol), THF (1 mL), acetophenone (58 μL, 0.50 mmol) and allylboronic acid pinacol ester (140 μL, 0.75 mmol). The reaction mixture was capped with a septa and stirred in the glovebox for 3 h at room temperature. After 3 h, the reaction was removed from the glovebox and quenched with 1 mL of saturated NH_4Cl solution and stirred for 10 min. The aqueous layer was extracted (3 x 5 mL Et_2O) and the combined organic layers were dried over $MgSO_4$, filtered, and concentrated in vacuo. Purification was performed using silica gel chromatography (90:10 pentane/Et_2O affording **6a** as a colorless oil (63 mg, 78%). Compounds **6e**, **6f**, and **6j** were purified using silver nitrate impregnated silica gel.[5]

Representative Procedure for Allylation of N-benzylidene-4-methoxyaniline **5m**.
To a flame-dried 5 mL round-bottom flask in a glovebox, was added $[Ir(cod)Cl]_2$ (6.7 mg, 0.010 mmol), potassium *tert*-butoxide (11 mg, 0.10 mmol), THF (1 mL), *N*-benzylidene-4-methoxyaniline (110 μL, 0.50 mmol) and allylboronic acid pinacol ester (140 μL, 0.75 mmol). The reaction mixture was capped with a septa and stirred in the

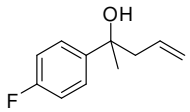
glovebox for 12 h at room temperature. After 12 h, the reaction was removed from the glovebox and quenched with 2 mL of H_2O and stirred for 10 min. The aqueous layer was extracted (3 x 8 mL CH_2Cl_2) and the combined organic layers were dried over Na_2SO_4, filtered, and concentrated in vacuo. Purification was performed using silica gel chromatography (3–5% Et_2O in pentane) affording **6m** as a pale yellow oil (86 mg, 68%).

VI. Characterization Data for Products.



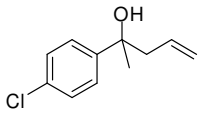
6a

2-Phenyl-4-penten-2-ol (6a): ^{1}H NMR ($CDCl_3$, 400 MHz) δ 7.44 (d, *J* = 7.8 Hz, 2H), 7.34 (t, *J* = 7.4 Hz, 2H), 7.23 (d, *J* = 7.2 Hz, 1H), 5.68-5.56 (m, 1H), 5.14 (d, *J* = 18.8 Hz, 1H), 5.12 (d, *J* = 8.5 Hz, 1H), 2.69 (dd, *J* = 13.4, 6.7 Hz, 1H) 2.50 (dd, *J* = 13.8, 8.2 Hz, 1H), 2.03 (s, 1H), 1.54 (d, *J* = 1.54 Hz, 3H). The spectral values obtained were consistent with those reported in the literature.[6]



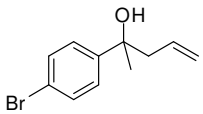
6b

2-(4-Fluorophenyl)-4-penten-2-ol (6b): ^{1}H NMR ($CDCl_3$, 400 MHz) δ 7.43–7.39 (m, 2H), 7.05–7.00 (m, 2H), 5.66–5.58 (m, 1H), 5.17–5.12 (m, 2H), 2.66 (dd, *J* = 13.8, 6.6 Hz, 1H) 2.50 (dd, *J* = 13.7, 8.2 Hz, 1H), 2.02 (s, 1H), 1.55 (s, 3H). The spectral values obtained were consistent with those reported in the literature.[7]



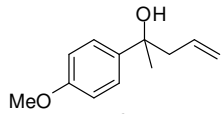
6c

2-(4-Chlorophenyl)-4-penten-2-ol (6c): ^{1}H NMR ($CDCl_3$, 400 MHz) δ 7.38 (dd, *J* = 8.8 Hz, 2H), 7.31 (dd, *J* = 8.8 Hz, 2H), 5.64–5.56 (m, 1H), 5.17–5.12 (m, 2H), 2.66 (dd, *J* = 13.7, 6.5 Hz, 1H) 2.49 (dd, *J* = 13.7, 8.3 Hz, 1H), 2.02 (s, 1H), 1.54 (s, 3H). The spectral values obtained were consistent with those reported in the literature.[5]



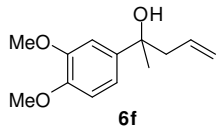
6d

2-(4-Bromophenyl)-4-penten-2-ol (6d): ^{1}H NMR ($CDCl_3$, 400 MHz) δ 7.47 (d, *J* = 8.6 Hz, 2H), 7.32 (d, *J* = 8.6 Hz, 2H), 5.66–5.55 (m, 1H), 5.17–5.13 (m, 2H), 2.66 (dd, *J* = 13.7, 6.5 Hz, 1H) 2.49 (dd, *J* = 13.7, 8.3 Hz, 1H), 2.03 (s, 1H), 1.54 (s, 3H). The spectral values obtained were consistent with those reported in the literature.[6]



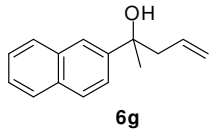
6e

2-(4-Methoxylphenyl)-4-penten-2-ol (6e): ^{1}H NMR ($CDCl_3$, 400 MHz) δ 7.37 (d, *J* = 8.9 Hz, 2H), 6.89 (d, *J* = 8.9 Hz, 1H), 5.70–5.59 (m, 1H), 5.16–5.11 (m, 2H), 3.82 (s, 3H), 2.67 (dd, *J* = 13.7, 6.5 Hz, 1H), 2.50 (dd, *J* = 13.7, 8.2 Hz, 1H), 2.00 (s, 1H), 1.54 (s, 3H). The spectral values obtained were consistent with those reported in the literature.[6]



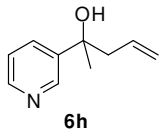
6f

2-(3,4-Dimethoxylphenyl)-4-penten-2-ol (6f): ^{1}H NMR ($CDCl_3$, 400 MHz) δ 7.04 (d, *J* = 2.1 Hz, 1H), 6.94 (dd, *J* = 8.4, 2.1 Hz, 1H), 6.84 (d, *J* = 8.4 Hz, 1H), 5.71–5.60 (m, 1H), 5.15 (d, *J* = 17.5 Hz, 1H), 5.14 (d, *J* = 9.9 Hz, 1H), 3.91 (s, 3H), 3.89 (s, 3H), 2.70 (dd, *J* = 13.7, 6.5 Hz, 1H) 2.50 (dd, *J* = 13.7, 8.2 Hz, 1H), 2.01 (s, 1H), 1.55 (s, 3H); **^{13}C NMR** ($CDCl_3$, 125 MHz) δ 148.8, 147.8, 140.7, 134.0, 119.6, 117.0, 110.9, 108.7, 73.7, 56.1, 48.7, 30.2, 24.8; **IR** (thin film, cm^{-1}) 3482, 3076, 2976, 2934, 1261, 1143, 1028; **HRMS** (TOF MS ES+) *m/z* calculated for $C_{13}H_{18}O_3$ 245.1154 $[M+Na]^+$ found 245.1146; **TLC R_f** = 0.3 (65:35 hexanes/EtOAc).



6g

2-(Naphthalen-2-yl)-4-penten-2-ol (6g): ^{1}H NMR ($CDCl_3$, 500 MHz) δ 7.93 (s, 1H), 7.86–7.83 (m, 3H), 7.55 (dd, *J* = 8.6, 1.7 Hz, 1H), 7.50–7.46 (m, 2H), 5.67–5.59 (m, 1H), 5.18 (d, *J* = 17.1 Hz, 1H), 5.13 (d, *J* = 10.1 Hz, 1H), 2.82 (dd, *J* = 13.8, 6.3 Hz, 1H) 2.60 (dd, *J* = 13.8, 8.4 Hz, 1H), 2.18 (s, 1H), 1.65 (s, 3H) . The spectral values obtained were consistent with those reported in the literature.[5]



6h

2-(Pyridin-3-yl)-4-penten-2-ol (6h): ^{1}H NMR ($CDCl_3$, 400 MHz) δ 8.70 (d, *J* = 1.8 Hz, 1H), 8.50 (d, *J* = 4.6 Hz, 1H), 8.00 (d, *J* = 8.0 Hz, 1H), 7.27 (dd, *J* = 7.7, 4.8 Hz, 1H), 5.68–5.60 (m, 1H), 5.17 (d, *J* = 10.3 Hz, 1H), 5.16 (d, *J* = 17.8 Hz, 1H), 2.68 (dd, *J* = 13.8, 6.7 Hz, 1H) 2.54 (dd, *J* = 13.8, 8.1 Hz, 1H), 2.30 (s, 1H), 1.59 (s, 3H); **^{13}C NMR** ($CDCl_3$, 125 MHz) δ 148.2, 147.1, 143.0, 133.0, 132.9, 123.2, 120.5, 72.7, 48.6, 30.0; **IR** (thin film, cm^{-1}) 3267, 2976, 1639, 1579, 1419; **HRMS** (TOF MS ES+) *m/z* calculated for $C_{10}H_{13}O_1$ 164.1075 $[M+H]^+$ found 164.1074; **TLC R_f** = 0.2 (6:4 hexanes/EtOAc).

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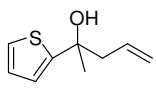
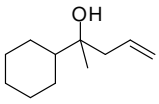


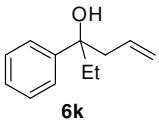
6i

2-(Thiophen-2-yl)-4-penten-2-ol (6i): ^{1}H NMR ($CDCl_3$, 400 MHz) δ 7.21 (dd, *J* = 5.0, 1.2 Hz, 1H), 6.96 (dd, *J* = 5.0, 3.6 Hz, 1H), 5.79–5.70 (m, 1H), 5.20–5.16 (m, 2H), 2.72 (dd, *J* = 13.7, 6.6 Hz, 1H), 2.58 (dd, *J* = 13.6, 8.1 Hz, 1H), 2.27 (s, 1H), 1.63 (s, 3H). The spectral values obtained were consistent with those reported in the literature.[5]



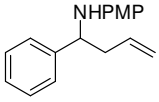
6j

***α*-Methyl-*α*-(2-propenyl)cyclohexanemethanol (6j): ^{1}H NMR** ($CDCl_3$, 500 MHz) δ 5.94–5.86 (m, 1H), 5.16 (d, *J* = 17.1 Hz, 1H), 5.12 (d, *J* = 10.4 Hz, 1H), 2.29–2.19 (m, 2H), 1.88–1.58 (m, 5H), 1.42 (s, 1H) 1.33–0.98 (m, 5H); **^{13}C NMR** ($CDCl_3$, 125 MHz) δ 134.4, 118.8, 74.1, 47.7, 44.4, 27.8, 27.1, 27.0, 26.9, 26.8, 23.9; **IR** (thin film, cm^{-1}) 3431, 3076, 2927, 2854, 1639, 1450; **HRMS** (TOF MS ES+) *m/z* calculated for $C_{11}H_{20}O$ 191.1412 $[M+Na]^+$ found 191.1415; **TLC R_f** = 0.2 (9:1 pentane/Et_2O).



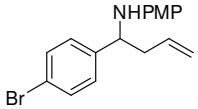
6k

2-Phenyl-4-hexen-2-ol (6k): ^{1}H NMR ($CDCl_3$, 500 MHz) δ 7.40 (d, *J* = 7.2 Hz, 2H), 7.37 (t, *J* = 7.7 Hz, 2H), 7.25 (t, *J* = 7.2 Hz, 1H), 5.63–5.55 (m, 1H), 5.15 (d, *J* = 19.3 Hz, 1H), 5.12 (d, *J* = 10.6 Hz, 1H), 2.74 (dd, *J* = 13.7, 6.0 Hz, 1H) 2.51 (dd, *J* = 13.7, 8.7 Hz, 1H), 2.02 (s, 1H), 1.91-1.81 (m, 2H), 0.78 (t, *J* = 7.4 Hz, 3H). The spectral values obtained were consistent with those reported in the literature.[8]



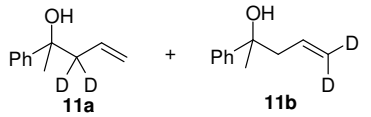
9a

4-methoxy-*N*-(1-phenylbut-3-enyl)aniline (9a): ^{1}H NMR ($CDCl_3$, 500 MHz) δ 7.39–7.17 (m, 5H), 6.67 (ad, *J* = 8.9 Hz, 2H), 6.45 (ad, *J* = 8.9 Hz, 2H), 5.81–5.70 (m, 1H), 5.21–5.09 (m, 2H), 4.30 (dd, *J* = 8.3, 5.1 Hz, 1H), 3.87 (br s, 1H), 3.67 (s, 3H), 2.63–2.53 (m, 1H), 2.51–2.41 (m, 1H). Analytical data is consistent with reported literature values.[9]



9b

***N*-(1-(4-bromophenyl)but-3-enyl)-4-methoxyaniline (9b): ^{1}H NMR** ($CDCl_3$, 500 MHz) δ 7.45 (d, *J* = 8.4 Hz, 2H), 7.25 (d, *J* = 8.4 Hz, 2H), 6.68 (d, *J* = 8.9 Hz, 2H), 6.42 (d, *J* = 8.9 Hz, 2H), 5.80–5.69 (m, 1H), 5.18 (d, *J* = 17.1 Hz, 1H), 5.16 (d, *J* = 9.6 Hz, 1H), 4.26 (m, 1H), 3.90 (s, 1H), 3.70 (s, 1H), 2.60–2.53 (m, 1H), 2.48–2.40 (m, 1H). **^{13}C NMR** ($CDCl_3$, 125 MHz) δ 152.5, 143.0, 141.2, 134.5, 131.9, 128.4, 120.8, 118.9, 115.0, 114.9, 57.8, 55.9, 43.4; **IR** (film, cm^{-1}) 3402, 3076, 2999, 2933, 2908, 2833, 1512, 1240; **HRMS** (TOF MS ES+) (*m/z*): $[M+H]^+$ calculated for $C_{17}H_{18}NOBr$ 332.0650, found 332.0647; **TLC R_f** = 0.5 (5% Et_2O in pentane).



3,3-dideutero-2-phenyl-4-penten-2-ol (11a) and 5,5-dideutero-2-phenyl-4-penten-2-ol (11b): **^{1}H NMR** ($CDCl_3$, 400 MHz) δ 7.44 (d, *J* = 7.8 Hz, 2H), 7.34 (t, *J* = 7.4 Hz, 2H), 7.23 (d, *J* = 7.2 Hz, 1H), 5.68–5.56 (m, 1H), 5.14–5.12 (m, 1H), 2.69–2.50 (m, 1H), 2.03 (s, 1H), 1.54 (d, *J* = 1.54 Hz, 3H); **^{13}C NMR** ($CDCl_3$, 125 MHz) δ 147.8, 133.8, 133.6, 128.4, 126.8, 125.0, 119.8, 73.8, 48.6, 30.13, 30.08; **IR** (thin film, cm^{-1}) 3425, 3061, 2976, 1601, 1446, 1373; **HRMS** (TOF MS APCI+) *m/z* calculated for $C_{11}H_{12}D_2O$ 187.1068 $(M+Na)^+$ found 187.1067; **TLC R_f** = 0.2 (9:1 Pentane/Et_2O).

VII. References.

[1] Black, H.T. Titrating Alkyllithium Reagents. http://www.ux1.eiu.edu/~cfthb/research/handbook/titrating.htm (accessed July 2008).

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[3] Sieber, J.D.; Morken, J.P. *J. Am. Chem. Soc.* **2008**, *130*, 4978-4983.

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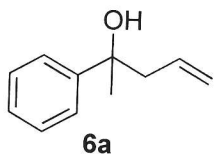

6a

Current Data Parameters
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NAME 1-TJB-171
EXPNO 3
PROCNO 1

F2 - Acquisition Parameters
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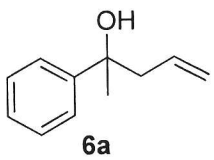
Time 17.32
INSTRUM drx400
PROBHD 5 mm QNP H/F/P
PULPROG zg30
TD 65536
SOLVENT CDCl3T
NS 8
DS 2
SWH 6410.256 Hz
FIDRES 0.097813 Hz
AQ 5.1118579 sec
RG 1149.4
DW 78.000 usec
DE 4.50 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======== CHANNEL f1 ========
NUC1 1H
P1 12.00 usec
PL1 0.00 dB
SFO1 400.1328009 MHz

F2 - Processing parameters
SI 65536
SF 400.1300221 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 2.00

1D NMR plot parameters
CX 22.80 cm
CY 10.00 cm
F1P 11.000 ppm
F1 4401.43 Hz
F2P -1.000 ppm
F2 -400.13 Hz
PPMCM 0.52632 ppm/cm
HZCM 210.59476 Hz/cm



6a

Current Data Parameters

USER	tjbark
NAME	1-TJB-278
EXPNO	5
PROCNO	1

F2 - Acquisition Parameters

Date_	20080503
Time	10.08
INSTRUM	cryo500
PROBHD	5 mm CPTCI 1H-
PULPROG	zgdc30
TD	65418
SOLVENT	CDCl3T
NS	513
DS	4
SWH	30303.031 Hz
FIDRES	0.463222 Hz
AQ	1.0794470 sec
RG	14596.5
DW	16.500 usec
DE	6.00 usec
TE	298.0 K
D1	0.25000000 sec
d11	0.03000000 sec
MCREST	0.00000000 sec
MCWRK	0.01500000 sec

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

NUC1	13C
P1	14.75 usec
PL1	-1.00 dB
SFO1	125.7942548 MHz

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

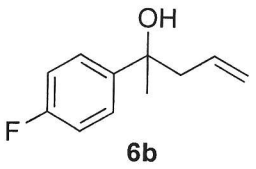
CPDPRG2	waltz16
NUC2	1H
PCPD2	100.00 usec
PL2	1.60 dB
PL12	24.80 dB
SFO2	500.2225011 MHz

F2 - Processing parameters

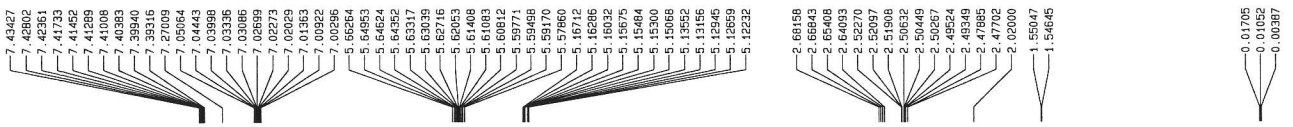
SI	65536
SF	125.7803983 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	2.00

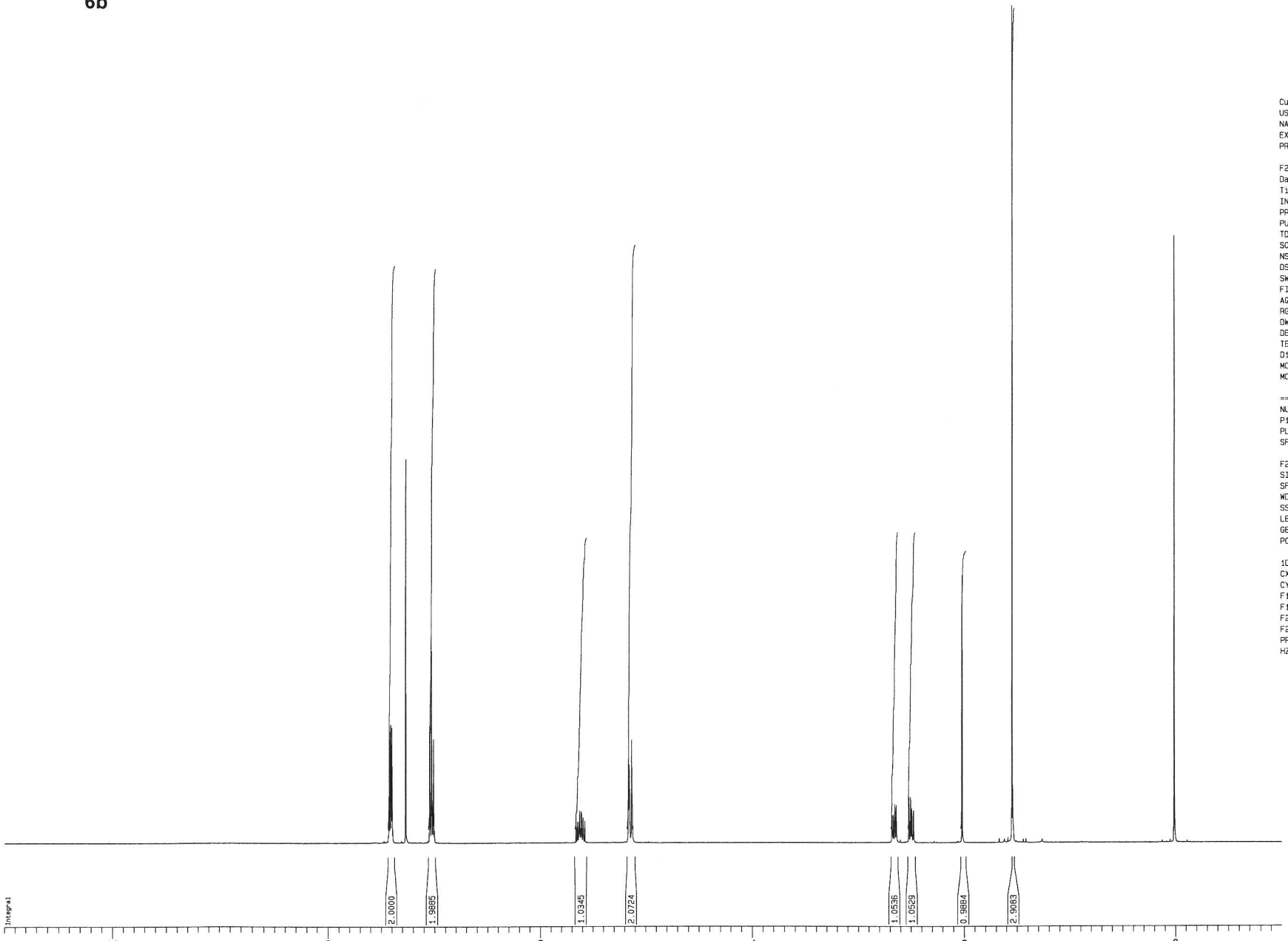
1D NMR plot parameters

CX	22.80 cm
CY	50.00 cm
F1P	230.625 ppm
F1	29008.05 Hz
F2P	-10.296 ppm
F2	-1294.98 Hz
PPMCM	10.56667 ppm/cm
HZCM	1329.08032 Hz/cm



6b





Current Data Parameters

USER	tjbark
NAME	2-TJB-15
EXPNO	6
PROCNO	1

F2 - Acquisition Parameters

Date_	20080614
Time	14.54
INSTRUM	gn500
PROBHD	5 mm broadband
PULPROG	zg30
TD	81728
SOLVENT	CDCl3T
NS	8
DS	2
SWH	8012.820 Hz
FIDRES	0.098043 Hz
AQ	5.0998774 sec
RG	1824.6
DW	62.400 usec
DE	6.00 usec
TE	298.0 K
D1	0.10000000 sec
MCREST	0.00000000 sec
MCWRK	0.01500000 sec

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

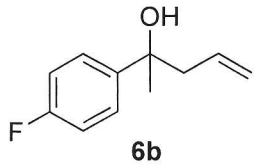
NUC1	1H
P1	12.00 usec
PL1	-3.00 dB
SFO1	499.8284988 MHz

F2 - Processing parameters

SI	65536
SF	499.8250256 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	4.00

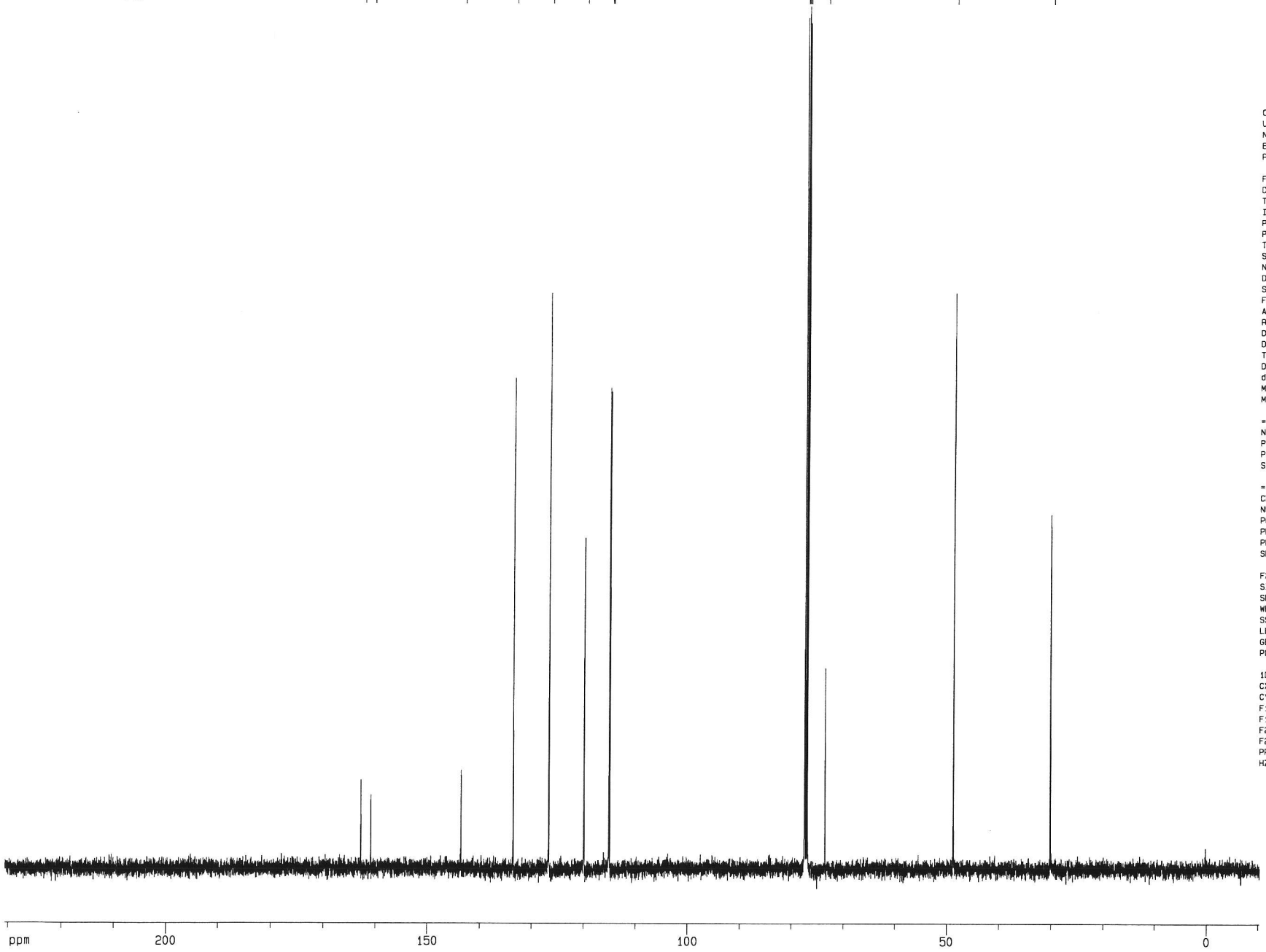
1D NMR plot parameters

CX	22.80 cm
CY	15.00 cm
F1P	11.000 ppm
F1	5498.08 Hz
F2P	-1.000 ppm
F2	-499.83 Hz
PPMCM	0.52632 ppm/cm
HZCM	263.06580 Hz/cm



6b

162.79
160.84
143.57
143.54
133.59
126.74
126.67
119.94
115.13
114.96
77.48
77.23
76.98
73.57
48.74
30.20

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Current Data Parameters
USER                tjbark
NAME             2-TJB-15
EXPNO                   5
PROCNO                  1

F2 - Acquisition Parameters
Date_            20080617
Time                8.34
INSTRUM          cryo500
PROBHD   5 mm CPTCI 1H-
PULPROG           zgdc30
TD                 65418
SOLVENT            CDCl3T
NS                   129
DS                     4
SWH           30303.031 Hz
FIDRES         0.463222 Hz
AQ             1.0794470 sec
RG                 13004
DW                16.500 usec
DE                 6.00 usec
TE                298.0 K
D1           0.25000000 sec
d11          0.03000000 sec
MCREST       0.00000000 sec
MCWRK        0.01500000 sec

======== CHANNEL f1 ========
NUC1                13C
P1                14.75 usec
PL1               -1.00 dB
SFO1         125.7942548 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                1H
PCPD2            100.00 usec
PL2                1.60 dB
PL12               24.80 dB
SFO2         500.2225011 MHz

F2 - Processing parameters
SI                 65536
SF           125.7804006 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  2.00

1D NMR plot parameters
CX                 22.80 cm
CY                 15.65 cm
F1P              230.637 ppm
F1               29009.68 Hz
F2P               -10.287 ppm
F2               -1293.96 Hz
PPMCM           10.56688 ppm/cm
HZCM          1329.10693 Hz/cm
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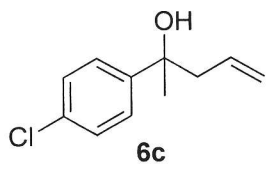


6c

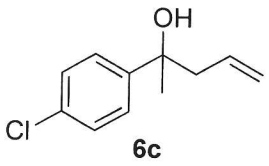
Current Data Parameters
USER tjbark
NAME 2-TJB-10
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20080607
Time 17.32
INSTRUM drx400
PROBHD 5 mm GNP H/F/P
PULPROG zg30
TD 65536
SOLVENT CDCl3T
NS 8
DS 2
SWH 6410.256 Hz
FIDRES 0.097813 Hz
AQ 5.1118579 sec
RG 1149.4
DW 78.000 usec
DE 4.50 usec
TE 297.9 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======== CHANNEL f1 ========
NUC1 1H
P1 12.00 usec
PL1 0.00 dB
SFO1 400.1328009 MHz

F2 - Processing parameters
SI 65536
SF 400.1300179 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 2.00

1D NMR plot parameters
CX 22.80 cm
CY 25.00 cm
F1P 11.000 ppm
F1 4401.43 Hz
F2P -1.000 ppm
F2 -400.13 Hz
PPMCM 0.52632 ppm/cm
HZCM 210.59474 Hz/cm



6c

Current Data Parameters

USER	tjbark
NAME	2-TJB-10
EXPNO	4
PROCNO	1

F2 - Acquisition Parameters

Date_	20080609
Time	11.02
INSTRUM	cryo500
PROBHD	5 mm CPTCI 1H-
PULPROG	zgdc30
TD	65418
SOLVENT	CDCl3T
NS	256
DS	4
SWH	30303.031 Hz
FIDRES	0.463222 Hz
AQ	1.0794470 sec
RG	9195.2
DW	16.500 usec
DE	6.00 usec
TE	298.0 K
D1	0.25000000 sec
d11	0.03000000 sec
MCREST	0.00000000 sec
MCWRK	0.01500000 sec

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

NUC1	13C
P1	14.75 usec
PL1	-1.00 dB
SFO1	125.7942548 MHz

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

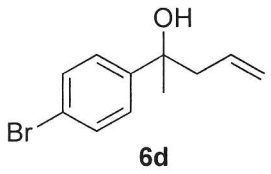
CPDPRG2	waltz16
NUC2	1H
PCPD2	100.00 usec
PL2	1.60 dB
PL12	24.60 dB
SFO2	500.2225011 MHz

F2 - Processing parameters

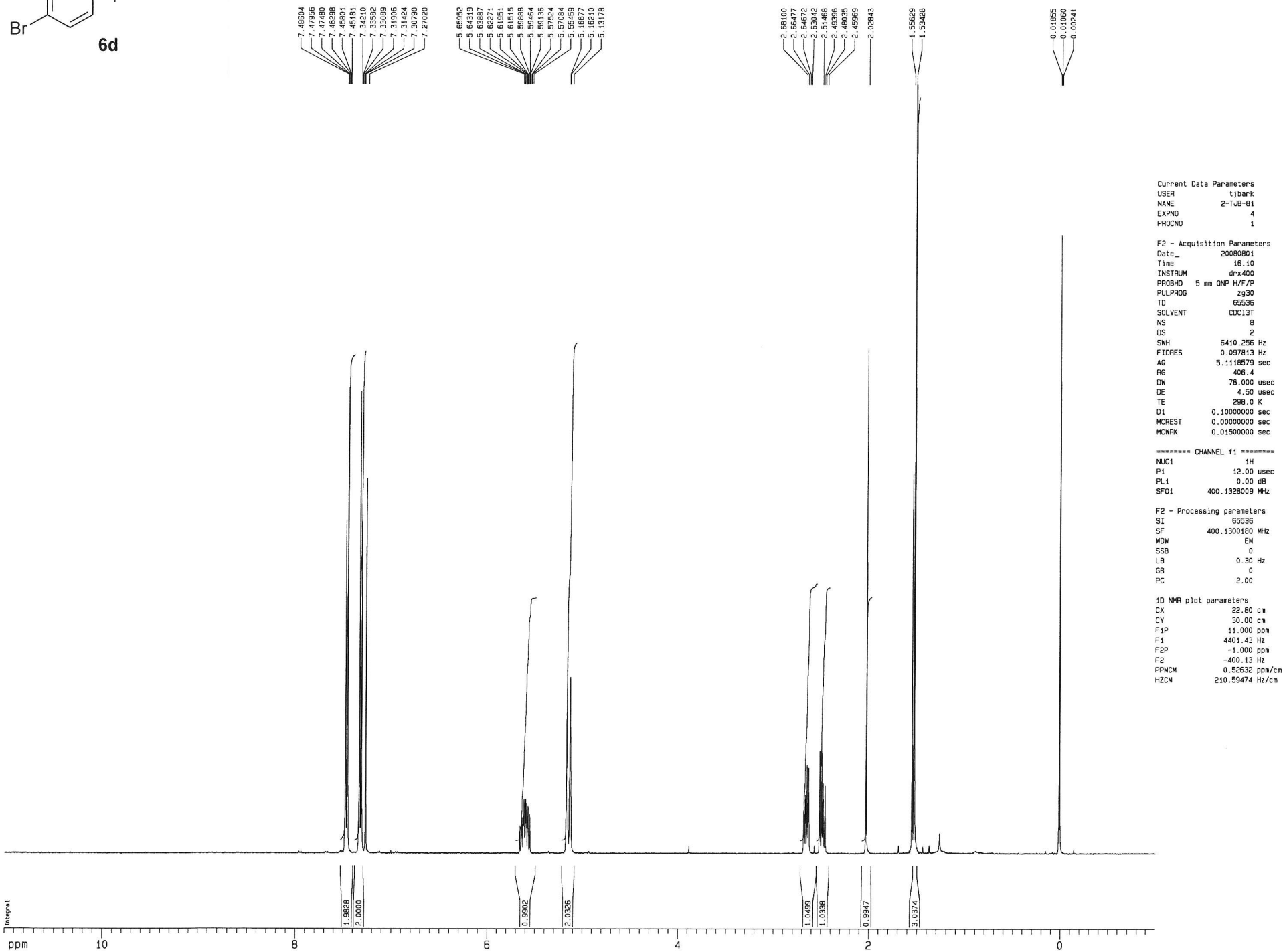
SI	65536
SF	125.7803983 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	2.00

1D NMR plot parameters

CX	22.80 cm
CY	30.00 cm
F1P	230.637 ppm
F1	29009.67 Hz
F2P	-10.287 ppm
F2	-1293.96 Hz
PPMCM	10.56688 ppm/cm
HZCM	1329.10681 Hz/cm



6d



Current Data Parameters

USER	tjbark
NAME	2-TJB-81
EXPNO	4
PROCNO	1

F2 - Acquisition Parameters

Date_	20080801
Time	16.10
INSTRUM	drx400
PROBHD	5 mm QNP H/F/P
PULPROG	zg30
TD	65536
SOLVENT	CDCl3T
NS	8
DS	2
SWH	6410.256 Hz
FIDRES	0.097813 Hz
AQ	5.1118579 sec
RG	406.4
DW	78.000 usec
DE	4.50 usec
TE	298.0 K
D1	0.10000000 sec
MCREST	0.00000000 sec
MCWRK	0.01500000 sec

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

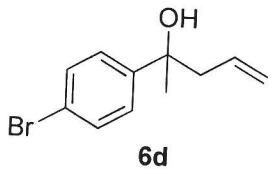
NUC1	1H
P1	12.00 usec
PL1	0.00 dB
SFO1	400.1328009 MHz

F2 - Processing parameters

SI	65536
SF	400.1300180 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	2.00

1D NMR plot parameters

CX	22.80 cm
CY	30.00 cm
F1P	11.000 ppm
F1	4401.43 Hz
F2P	-1.000 ppm
F2	-400.13 Hz
PPMCM	0.52632 ppm/cm
HZCM	210.59474 Hz/cm



6d


```
Current Data Parameters
USER                tjbark
NAME              2-TJB-17
EXPNO                    2
PROCNO                   1

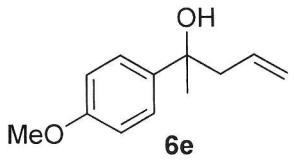
F2 - Acquisition Parameters
Date_            20080614
Time                14.28
INSTRUM          cryo500
PROBHD   5 mm CPTCI 1H-
PULPROG            zgdc30
TD                  65536
SOLVENT             CDCl3T
NS                    145
DS                      4
SWH             30303.031 Hz
FIDRES          0.462388 Hz
AQ              1.0813940 sec
RG                  13004
DW                16.500 usec
DE                 6.00 usec
TE                298.0 K
D1            0.25000000 sec
d11           0.03000000 sec
MCREST        0.00000000 sec
MCWRK         0.01500000 sec

======== CHANNEL f1 ========
NUC1                  13C
P1                 14.75 usec
PL1                -1.00 dB
SFO1         125.7942548 MHz

======== CHANNEL f2 ========
CPDPRG2           waltz16
NUC2                  1H
PCPD2             100.00 usec
PL2                1.60 dB
PL12               24.80 dB
SFO2         500.2225011 MHz

F2 - Processing parameters
SI                  65536
SF           125.7803988 MHz
WDW                    EM
SSB                     0
LB                 1.00 Hz
GB                      0
PC                   2.00

1D NMR plot parameters
CX                 22.80 cm
CY                 30.00 cm
F1P               230.637 ppm
F1               29009.67 Hz
F2P               -10.287 ppm
F2               -1293.96 Hz
PPMCM             10.56688 ppm/cm
HZCM          1329.10681 Hz/cm
```



6e


```
Current Data Parameters
USER                tjbark
NAME              2-TJB-77
EXPNO                    2
PROCNO                   1

F2 - Acquisition Parameters
Date_              20080729
Time                 13.34
INSTRUM              drx400
PROBHD   5 mm QNP H/F/P
PULPROG               zg30
TD                   65536
SOLVENT              CDCl3T
NS                       8
DS                       2
SWH               6410.256 Hz
FIDRES           0.097813 Hz
AQ               5.1118579 sec
RG                   322.5
DW                78.000 usec
DE                 4.50 usec
TE                  298.0 K
D1              0.10000000 sec
MCREST          0.00000000 sec
MCWRK           0.01500000 sec

======== CHANNEL f1 ========
NUC1                    1H
P1                  12.00 usec
PL1                  0.00 dB
SFO1          400.1328009 MHz

F2 - Processing parameters
SI                   65536
SF          400.1300179 MHz
WDW                     EM
SSB                      0
LB                  0.30 Hz
GB                       0
PC                    2.00

1D NMR plot parameters
CX                  22.80 cm
CY                  40.00 cm
F1P                11.000 ppm
F1                4401.43 Hz
F2P                -1.000 ppm
F2                -400.13 Hz
PPMCM            0.52632 ppm/cm
HZCM           210.59474 Hz/cm
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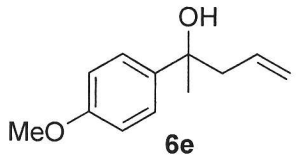


6e

158.46
139.97
134.01
126.15
119.13
113.66
77.49
77.23
76.98
73.60
55.44
48.72
30.17

```
Current Data Parameters
USER              tjbark
NAME            2-TJB-76
EXPNO                  6
PROCNO                 1

F2 - Acquisition Parameters
Date_          20080726
Time              14.47
INSTRUM         cryo500
PROBHD   5 mm CPTCI 1H-
PULPROG          zgdc30
TD                65418
SOLVENT           CDCl3
NS                  128
DS                    4
SWH           30303.031 Hz
FIDRES         0.463222 Hz
AQ             1.0794470 sec
RG                13004
DW               16.500 usec
DE                6.00 usec
TE               298.0 K
D1           0.25000000 sec
d11          0.03000000 sec
MCREST       0.00000000 sec
MCWRK        0.01500000 sec

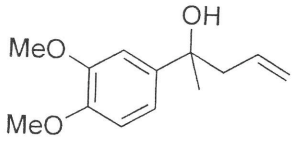
======== CHANNEL f1 ========
NUC1                13C
P1                14.75 usec
PL1               -1.00 dB
SFO1         125.7942548 MHz

======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2                1H
PCPD2           100.00 usec
PL2               1.60 dB
PL12             24.80 dB
SFO2         500.2225011 MHz

F2 - Processing parameters
SI                 65536
SF           125.7804006 MHz
WDW                   EM
SSB                    0
LB                1.00 Hz
GB                     0
PC                  2.00

1D NMR plot parameters
CX                22.80 cm
CY                15.65 cm
F1P             230.637 ppm
F1              29009.68 Hz
F2P             -10.287 ppm
F2              -1293.96 Hz
PPMCM          10.56688 ppm/cm
HZCM         1329.10693 Hz/cm
```

ppm 200 150 100 50 0



6f

7.27022
7.04659
7.04137
6.95465
6.93377
6.92844
6.85427
6.83336

5.17212
5.15317
5.12846

3.91028
3.80622

2.00803

1.56295
1.54753

0.00911

Current Data Parameters
USER tjbark
NAME 2-TJB-46
EXPNO 5
PROCNO 1

F2 - Acquisition Parameters
Date_ 20080705
Time 13.20
INSTRUM drx400
PROBHD 5 mm QNP H/F/P
PULPROG zg30
TD 65536
SOLVENT CDCl3T
NS 8
DS 2
SWH 6410.256 Hz
FIDRES 0.097813 Hz
AQ 5.1118579 sec
RG 645.1
DW 78.000 usec
DE 4.50 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

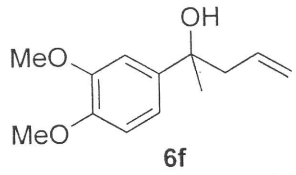
======== CHANNEL f1 ========
NUC1 1H
P1 12.00 usec
PL1 0.00 dB
SFO1 400.1328009 MHz

F2 - Processing parameters
SI 65536
SF 400.1300179 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 2.00

1D NMR plot parameters
CX 22.80 cm
CY 7.00 cm
F1P 11.000 ppm
F1 4401.43 Hz
F2P -1.000 ppm
F2 -400.13 Hz
PPMCM 0.52632 ppm/cm
HZCM 210.59474 Hz/cm

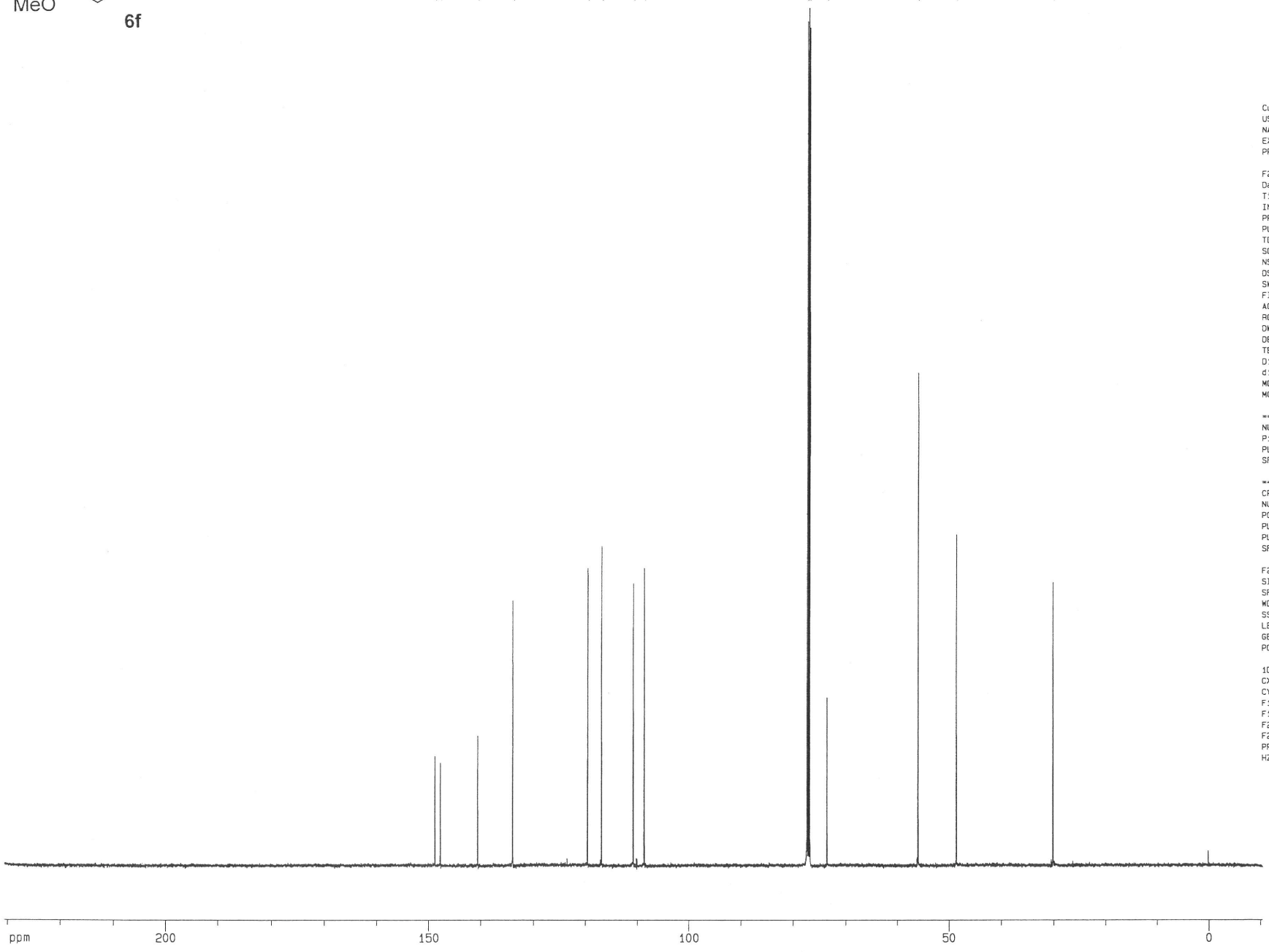
Integral
1.0000
1.0056
1.0136
0.9698
2.0025
6.0970
0.9970
1.0014
0.9851
3.6533

ppm
10
8
6
4
2
0



6f

148.83
147.82
140.65
133.97
119.63
117.02
110.89
108.72
77.48
77.23
76.97
73.65
56.08
48.68
30.19



Current Data Parameters
USER tjbark
NAME 2-TJB-28
EXPNO 5
PROCNO 1

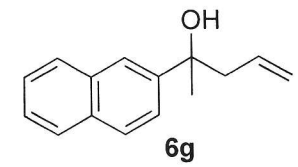
F2 - Acquisition Parameters
Date_ 20080621
Time 11.11
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG zgdc30
TD 65418
SOLVENT CDCl3T
NS 636
DS 4
SWH 30303.031 Hz
FIDRES 0.463222 Hz
AQ 1.0794470 sec
RG 9195.2
DW 16.500 usec
DE 6.00 usec
TE 298.0 K
D1 0.25000000 sec
d11 0.03000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======== CHANNEL f1 ========
NUC1 13C
P1 14.75 usec
PL1 -1.00 dB
SFO1 125.7942548 MHz

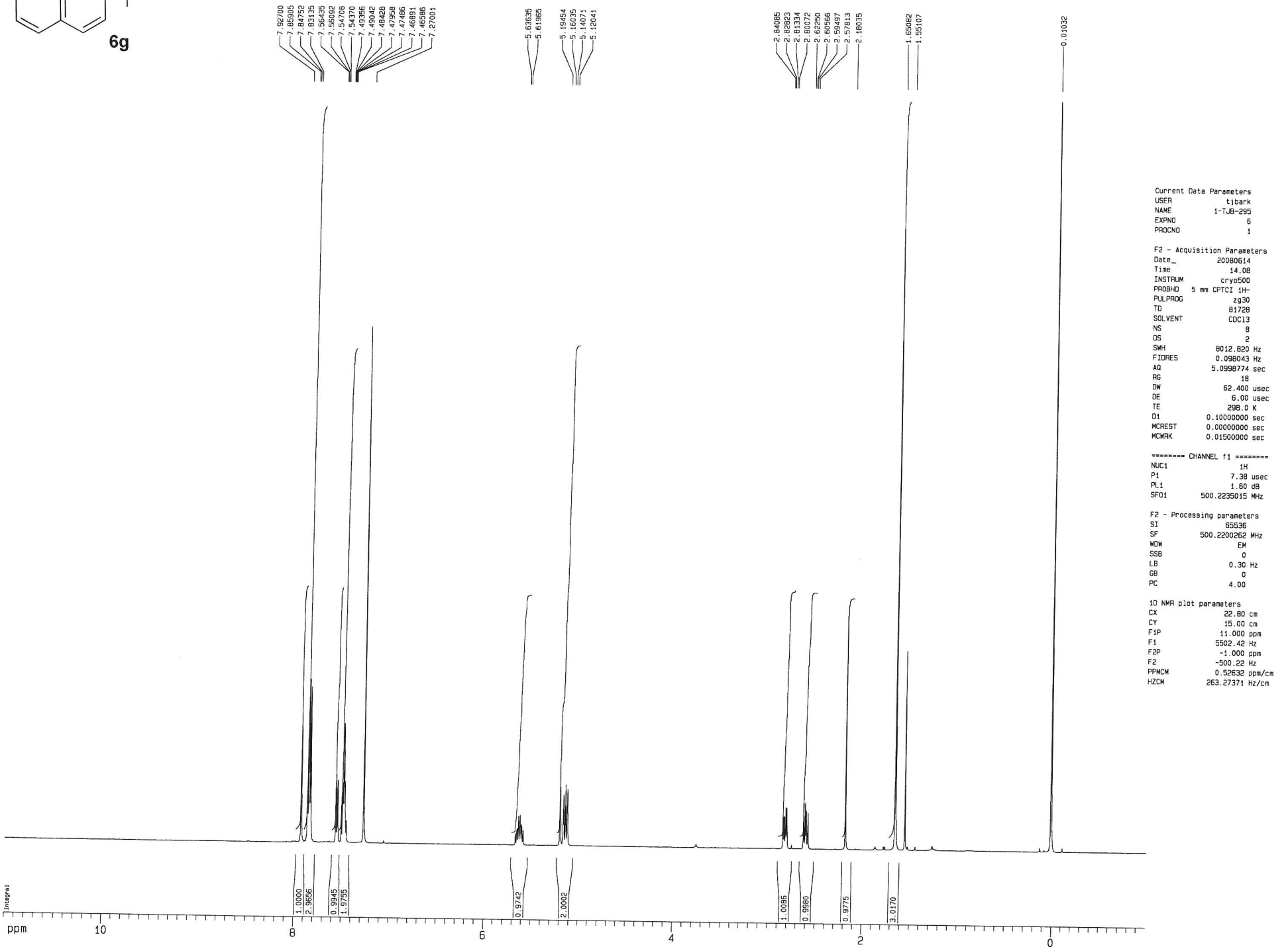
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 1.60 dB
PL12 24.80 dB
SFO2 500.2225011 MHz

F2 - Processing parameters
SI 65536
SF 125.7804011 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 2.00

1D NMR plot parameters
CX 22.80 cm
CY 15.65 cm
F1P 230.603 ppm
F1 29005.29 Hz
F2P -10.318 ppm
F2 -1297.74 Hz
PPMCM 10.56667 ppm/cm
HZCM 1329.08044 Hz/cm



6g



Current Data Parameters

USER	tjbark
NAME	1-TJB-295
EXPNO	5
PROCNO	1

F2 - Acquisition Parameters

Date_	20080614
Time	14.08
INSTRUM	cryo500
PROBHD	5 mm CPTCI 1H-
PULPROG	zg30
TD	81728
SOLVENT	CDCl3
NS	8
DS	2
SWH	8012.820 Hz
FIDRES	0.098043 Hz
AQ	5.0998774 sec
RG	18
DW	62.400 usec
DE	6.00 usec
TE	298.0 K
D1	0.10000000 sec
MCREST	0.00000000 sec
MCWRK	0.01500000 sec

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

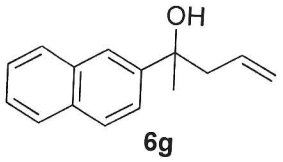
NUC1	1H
P1	7.38 usec
PL1	1.60 dB
SFO1	500.2235015 MHz

F2 - Processing parameters

SI	65536
SF	500.2200262 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	4.00

1D NMR plot parameters

CX	22.80 cm
CY	15.00 cm
F1P	11.000 ppm
F1	5502.42 Hz
F2P	-1.000 ppm
F2	-500.22 Hz
PPMCM	0.52632 ppm/cm
HZCM	263.27371 Hz/cm



6g

145.19
133.80
133.39
132.47
128.36
128.13
127.69
126.29
125.94
123.76
123.42
119.89
77.48
77.23
76.97
74.01
48.50
30.20
0.22

```
Current Data Parameters
USER                tjbark
NAME            1-TJB-295
EXPNO                   5
PROCNO                  1

F2 - Acquisition Parameters
Date_            20080614
Time                13.53
INSTRUM          cryo500
PROBHD   5 mm CPTCI 1H-
PULPROG           zgdc30
TD                 65418
SOLVENT            CDCl3T
NS                   602
DS                     4
SWH            30303.031 Hz
FIDRES          0.463222 Hz
AQ              1.0794470 sec
RG                 13004
DW                 16.500 usec
DE                  6.00 usec
TE                298.0 K
D1             0.25000000 sec
d11            0.03000000 sec
MCREST         0.00000000 sec
MCWRK          0.01500000 sec

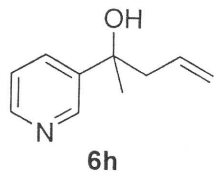
======== CHANNEL f1 ========
NUC1                13C
P1                14.75 usec
PL1               -1.00 dB
SFO1         125.7942548 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                1H
PCPD2            100.00 usec
PL2                1.60 dB
PL12               24.80 dB
SFO2         500.2225011 MHz

F2 - Processing parameters
SI                  65536
SF           125.7803983 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  2.00

1D NMR plot parameters
CX                 22.80 cm
CY                 45.00 cm
F1P               230.625 ppm
F1               29008.05 Hz
F2P               -10.296 ppm
F2               -1294.98 Hz
PPMCM           10.56667 ppm/cm
HZCM          1329.08032 Hz/cm
```

ppm 200 150 100 50 0



6h

8.68150
8.46796
7.79283
7.77681
7.26959
7.26524
5.15803
5.13775
2.55221
2.53611
1.57650

Current Data Parameters
USER tjbark
NAME 1-TJB-298
EXPNO 7
PROCNO 1

F2 - Acquisition Parameters
Date_ 20080827
Time 8.21
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG zg30
TD 81728
SOLVENT CDCl3T
NS 8
DS 2
SWH 8012.820 Hz
FIDRES 0.098043 Hz
AQ 5.0998774 sec
RG 6.3
DW 62.400 usec
DE 6.00 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

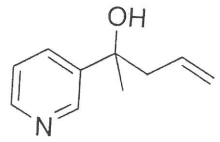
======== CHANNEL f1 ========
NUC1 1H
P1 7.38 usec
PL1 1.60 dB
SFO1 500.2235015 MHz

F2 - Processing parameters
SI 65536
SF 500.2200289 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 4.00

1D NMR plot parameters
CX 22.80 cm
CY 5.00 cm
F1P 11.000 ppm
F1 5502.42 Hz
F2P -1.000 ppm
F2 -500.22 Hz
PPMCM 0.52632 ppm/cm
HZCM 263.27371 Hz/cm

Integral
1.0000
1.0006
0.9860
1.1698
0.9843
2.0095
1.2389
1.5791
0.5584
3.1279

ppm
10
8
6
4
2
0



6h

148.24
147.13
142.99
133.01
132.93
123.24
120.51
77.49
77.23
76.98
72.71
48.56
30.00

Current Data Parameters
USER tjbark
NAME 2-TJB-24
EXPNO 6
PROCNO 1

F2 - Acquisition Parameters
Date_ 20080621
Time 11.01
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG zgdc30
TD 65536
SOLVENT CDCl3T
NS 108
DS 4
SWH 30303.031 Hz
FIDRES 0.462388 Hz
AQ 1.0813940 sec
RG 14596.5
DW 16.500 usec
DE 6.00 usec
TE 298.0 K
D1 0.25000000 sec
d11 0.03000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

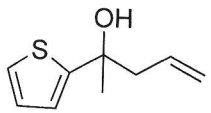
======== CHANNEL f1 ========
NUC1 13C
P1 14.75 usec
PL1 -1.00 dB
SFO1 125.7942548 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 1.50 dB
PL12 24.80 dB
SFO2 500.2225011 MHz

F2 - Processing parameters
SI 65536
SF 125.7803988 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 2.00

1D NMR plot parameters
CX 22.80 cm
CY 15.65 cm
F1P 230.637 ppm
F1 29009.67 Hz
F2P -10.287 ppm
F2 -1293.96 Hz
PPMCM 10.56688 ppm/cm
HZCM 1329.10681 Hz/cm

ppm 200 150 100 50 0



6i

7.26997
7.21531
7.21244
7.20279
7.19989
6.97551
6.96668
6.96316
6.95416
6.93225
6.92940
6.92346
6.92059

5.19861
5.19570
5.18209
5.18986
5.16030
5.15730
5.15398

2.70890
2.69228
2.60542
2.58509

2.27311

1.63511
1.56047

0.01152

Current Data Parameters
USER tjbark
NAME 2-TJB-32
EXPNO 8
PROCNO 1

F2 - Acquisition Parameters
Date_ 20081120
Time 8.52
INSTRUM drx400
PROBHD 5 mm QNP H/F/P
PULPROG zg30
TD 65536
SOLVENT CDCl3T
NS 8
DS 2
SWH 6410.256 Hz
FIDRES 0.097813 Hz
AQ 5.1118579 sec
RG 512
DW 78.000 usec
DE 4.50 usec
TE 298.1 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

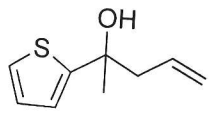
======== CHANNEL f1 ========
NUC1 1H
P1 12.00 usec
PL1 0.00 dB
SFO1 400.1328009 MHz

F2 - Processing parameters
SI 65536
SF 400.1300179 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 2.00

1D NMR plot parameters
CX 22.80 cm
CY 12.00 cm
F1P 11.000 ppm
F1 4401.43 Hz
F2P -1.000 ppm
F2 -400.13 Hz
PPMCM 0.52632 ppm/cm
HZCM 210.59474 Hz/cm

Integral
1.0000
1.0241
1.0418
1.0424
2.1695
1.0952
1.1207
1.0280
3.2453

ppm
10
8
6
4
2
0



6i

153.25
133.50
126.94
124.07
122.52
119.97
77.48
77.23
76.97
73.13
49.35
30.62

ppm 200 150 100 50 0

```
Current Data Parameters
USER              tjbark
NAME           2-TJB-32
EXPNO                 4
PROCNO                1

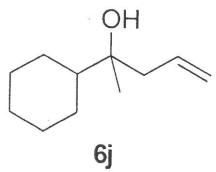
F2 - Acquisition Parameters
Date_          20080621
Time              10.54
INSTRUM         cryo500
PROBHD   5 mm CPTCI 1H-
PULPROG         zgdc30
TD                65536
SOLVENT           CDCl3T
NS                   33
DS                    4
SWH           30303.031 Hz
FIDRES        0.462388 Hz
AQ            1.0813940 sec
RG                13004
DW               16.500 usec
DE                6.00 usec
TE               298.0 K
D1            0.25000000 sec
d11           0.03000000 sec
MCREST        0.00000000 sec
MCWRK         0.01500000 sec

======== CHANNEL f1 ========
NUC1                 13C
P1                14.75 usec
PL1               -1.00 dB
SFO1         125.7942548 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                 1H
PCPD2            100.00 usec
PL2               1.60 dB
PL12              24.80 dB
SFO2         500.2225011 MHz

F2 - Processing parameters
SI                65536
SF           125.7803997 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 2.00

1D NMR plot parameters
CX                22.80 cm
CY                15.65 cm
F1P              230.637 ppm
F1             29009.68 Hz
F2P              -10.287 ppm
F2             -1293.96 Hz
PPMCM          10.56688 ppm/cm
HZCM         1329.10693 Hz/cm
```



6j

7.27009

5.92699
5.90172
5.88414
5.85894

5.16261
5.13730
5.09621

2.25678
2.23204
2.21282
1.88001
1.84941
1.78935
1.76595
1.68925
1.67526
1.65958
1.41989
1.33479
1.32778
1.32051
1.29797
1.24835
1.24061
1.20877
1.17598
1.16789
1.13787
1.10595
1.09579
1.08416
1.06066
1.05291
1.03029
1.02189
1.00887
0.98590
0.97749
0.00342

Current Data Parameters
USER tjbark
NAME 2-TJB-82
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20080802
Time 15.25
INSTRUM drx400
PROBHD 5 mm QNP H/F/P
PULPROG zg30
TD 65536
SOLVENT CDCl3T
NS 8
DS 2
SWH 6410.256 Hz
FIDRES 0.097813 Hz
AQ 5.1118579 sec
RG 90.5
DW 78.000 usec
DE 4.50 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

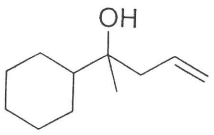
======== CHANNEL f1 ========
NUC1 1H
P1 12.00 usec
PL1 0.00 dB
SFO1 400.1328009 MHz

F2 - Processing parameters
SI 65536
SF 400.1300180 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 2.00

1D NMR plot parameters
CX 22.80 cm
CY 15.00 cm
F1P 11.000 ppm
F1 4401.43 Hz
F2P -1.000 ppm
F2 -400.13 Hz
PPMCM 0.52632 ppm/cm
HZCM 210.59474 Hz/cm

Integral
1.000
2.069
2.160
5.709
1.060
10.190

ppm
10
8
6
4
2
0



6j

Current Data Parameters
USER tjbark
NAME 2-TJB-82
EXPNO 3
PROCNO 1

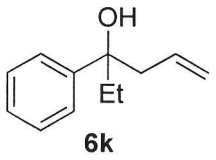
F2 - Acquisition Parameters
Date_ 20080826
Time 19.26
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG zgdc30
TD 65536
SOLVENT CDCl3T
NS 128
DS 4
SWH 30303.031 Hz
FIDRES 0.462388 Hz
AQ 1.0813940 sec
RG 13004
DW 16.500 usec
DE 6.00 usec
TE 298.0 K
D1 0.25000000 sec
d11 0.03000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======== CHANNEL f1 ========
NUC1 13C
P1 14.75 usec
PL1 -1.00 dB
SFO1 125.7942548 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 1.60 dB
PL12 24.80 dB
SFO2 500.2225011 MHz

F2 - Processing parameters
SI 65536
SF 125.7804002 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 2.00

1D NMR plot parameters
CX 22.80 cm
CY 15.65 cm
F1P 230.637 ppm
F1 29009.68 Hz
F2P -10.287 ppm
F2 -1293.96 Hz
PPMCM 10.56688 ppm/cm
HZCM 1329.10693 Hz/cm



6k

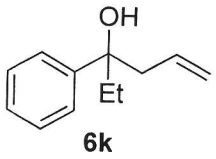
Current Data Parameters
USER tjbark
NAME 2-TJB-37
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20080625
Time 9.26
INSTRUM drx400
PROBHD 5 mm QNP H/F/P
PULPROG zg30
TD 65536
SOLVENT CDCl3T
NS 8
DS 2
SWH 6410.256 Hz
FIDRES 0.097813 Hz
AQ 5.1118579 sec
RG 912.3
DW 78.000 usec
DE 4.50 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======== CHANNEL f1 ========
NUC1 1H
P1 12.00 usec
PL1 0.00 dB
SFO1 400.1328009 MHz

F2 - Processing parameters
SI 65536
SF 400.1300179 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 2.00

1D NMR plot parameters
CX 22.80 cm
CY 10.00 cm
F1P 11.000 ppm
F1 4401.43 Hz
F2P -1.000 ppm
F2 -400.13 Hz
PPMCM 0.52632 ppm/cm
HZCM 210.59474 Hz/cm



6k

Current Data Parameters

USER	tjbark
NAME	2-TJB-37
EXPNO	4
PROCNO	1

F2 - Acquisition Parameters

Date_	20080628
Time	11.27
INSTRUM	cryo500
PROBHD	5 mm CPTCI 1H-
PULPROG	zgdc30
TD	65418
SOLVENT	CDCl3T
NS	128
DS	4
SWH	30303.031 Hz
FIDRES	0.463222 Hz
AQ	1.0794470 sec
RG	14596.5
DW	16.500 usec
DE	6.00 usec
TE	298.0 K
D1	0.25000000 sec
d11	0.03000000 sec
MCREST	0.00000000 sec
MCWRK	0.01500000 sec

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

NUC1	13C
P1	14.75 usec
PL1	-1.00 dB
SFO1	125.7942548 MHz

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

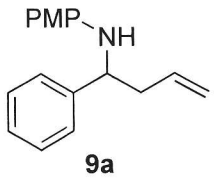
CPDPRG2	waltz16
NUC2	1H
PCPD2	100.00 usec
PL2	1.60 dB
PL12	24.80 dB
SFO2	500.2225011 MHz

F2 - Processing parameters

SI	65536
SF	125.7804006 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	2.00

1D NMR plot parameters

CX	22.80 cm
CY	15.66 cm
F1P	230.606 ppm
F1	29005.75 Hz
F2P	-10.314 ppm
F2	-1297.28 Hz
PPMCM	10.56667 ppm/cm
HZCM	1329.08044 Hz/cm



9a

Current Data Parameters

USER	tjbark
NAME	2-TJB-110
EXPNO	2
PROCNO	1

F2 - Acquisition Parameters

Date_	20080823
Time	11.26
INSTRUM	drx400
PROBHD	5 mm QNP H/F/P
PULPROG	zg30
TD	65536
SOLVENT	CDCl3T
NS	8
DS	2
SWH	6410.256 Hz
FIDRES	0.097813 Hz
AQ	5.1118579 sec
RG	724.1
DW	78.000 usec
DE	4.50 usec
TE	298.0 K
D1	0.10000000 sec
MCREST	0.00000000 sec
MCWRK	0.01500000 sec

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

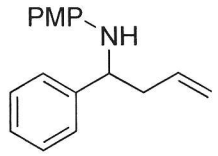
NUC1	1H
P1	12.00 usec
PL1	0.00 dB
SFO1	400.1328009 MHz

F2 - Processing parameters

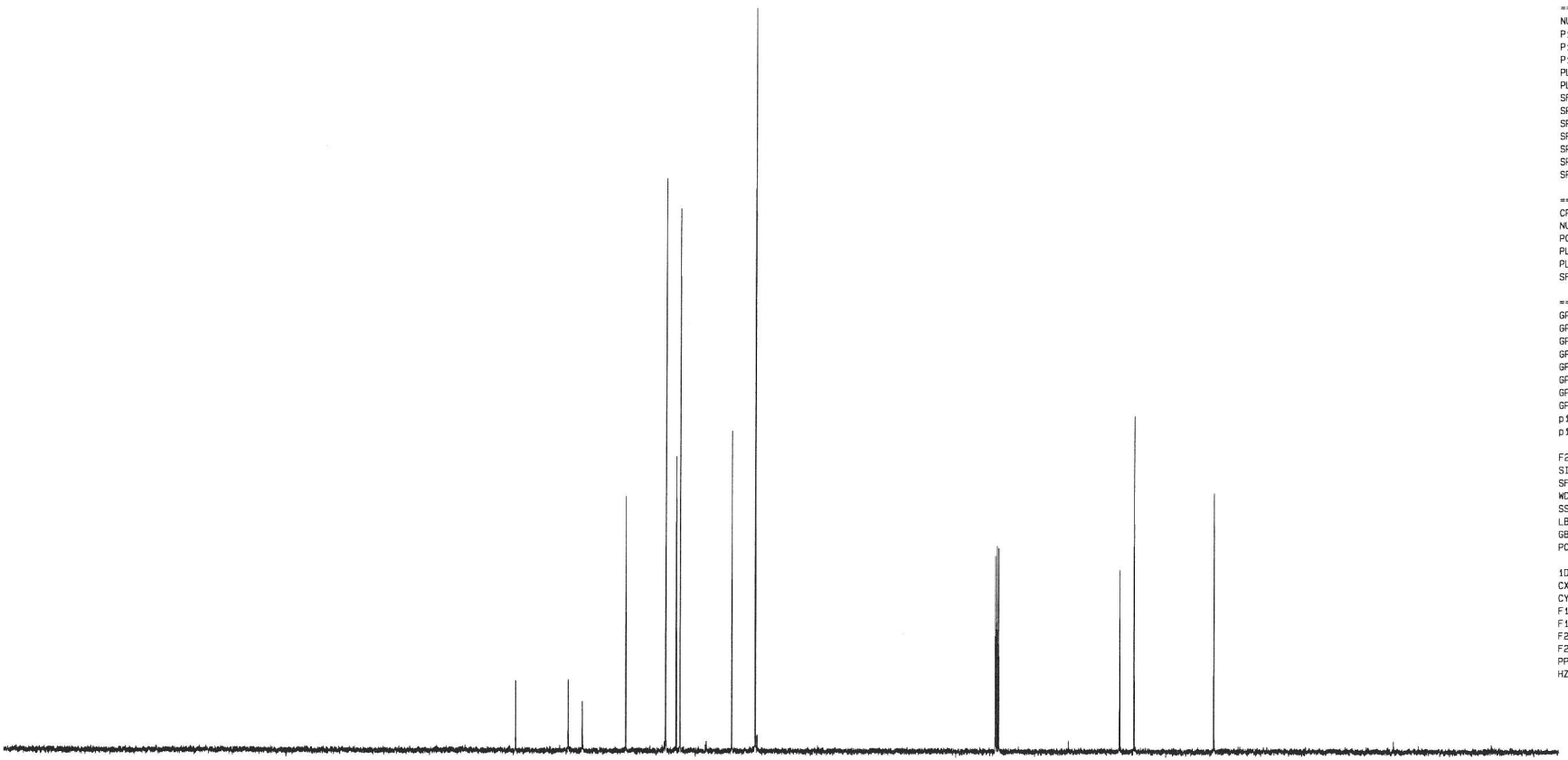
SI	65536
SF	400.1300181 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	2.00

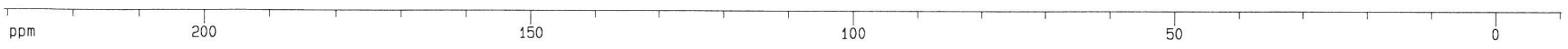
1D NMR plot parameters

CX	22.80 cm
CY	15.00 cm
F1P	11.000 ppm
F1	4401.43 Hz
F2P	-1.000 ppm
F2	-400.13 Hz
PPMCM	0.52632 ppm/cm
HZCM	210.59474 Hz/cm



9a






```
Current Data Parameters
USER                tjbark
NAME            2-TJB-110
EXPNO                   4
PROCNO                  1

F2 - Acquisition Parameters
Date_            20081119
Time                17.58
INSTRUM           cryo500
PROBHD   5 mm CPTCI 1H-
PULPROG  SpinEchopg30gp.prd
TD                  65536
SOLVENT             CDCl3T
NS                     71
DS                     16
SWH           30303.031 Hz
FIDRES        0.462388 Hz
AQ            1.0814105 sec
RG                   3251
DW                16.500 usec
DE                 6.00 usec
TE                298.0 K
D1           0.25000000 sec
d11          0.03000000 sec
D16          0.00020000 sec
d17          0.00019600 sec
MCREST       0.00000000 sec
MCWRK        0.01500000 sec
P2                29.70 usec

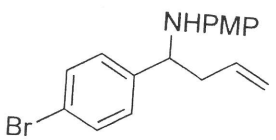
======== CHANNEL f1 ========
NUC1                  13C
P1                 14.85 usec
P11               500.00 usec
P12              2000.00 usec
PL0               120.00 dB
PL1                -1.00 dB
SFO1         125.7942548 MHz
SP1                3.60 dB
SP2                3.60 dB
SPNAM1    Crp60,0.5,20.1
SPNAM2      Crp60comp.4
SPOFF1                0.00 Hz
SPOFF2                0.00 Hz

======== CHANNEL f2 ========
CPDPRG2           waltz16
NUC2                  1H
PCPD2             100.00 usec
PL2                1.60 dB
PL12               24.60 dB
SFO2         500.2225011 MHz

====== GRADIENT CHANNEL =====
GPNAM1          SINE.100
GPNAM2          SINE.100
GPX1                  0.00 %
GPX2                  0.00 %
GPY1                  0.00 %
GPY2                  0.00 %
GPZ1                 30.00 %
GPZ2                 50.00 %
p15                 500.00 usec
p16                1000.00 usec

F2 - Processing parameters
SI                    65536
SF           125.7804048 MHz
WDW                     EM
SSB                      0
LB                    1.00 Hz
GB                       0
PC                    2.00

1D NMR plot parameters
CX                   22.80 cm
CY                   11.00 cm
F1P                230.637 ppm
F1                29009.68 Hz
F2P                -10.287 ppm
F2                -1293.96 Hz
PPMCM             10.56688 ppm/cm
HZCM           1329.10693 Hz/cm
```



9b

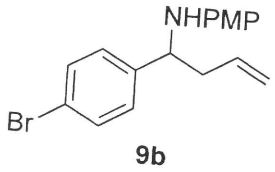

```
Current Data Parameters
USER                tjbark
NAME             2-TJB-110
EXPNO                    2
PROCNO                   1

F2 - Acquisition Parameters
Date_             20080823
Time                 11.26
INSTRUM             drx400
PROBHD   5 mm QNP H/F/P
PULPROG                zg30
TD                    65536
SOLVENT             CDCl3T
NS                       8
DS                       2
SWH               6410.256 Hz
FIDRES            0.097813 Hz
AQ                5.1118579 sec
RG                   724.1
DW                  78.000 usec
DE                   4.50 usec
TE                 298.0 K
D1              0.10000000 sec
MCREST          0.00000000 sec
MCWRK           0.01500000 sec

======== CHANNEL f1 ========
NUC1                    1H
P1                  12.00 usec
PL1                  0.00 dB
SF01          400.1328009 MHz

F2 - Processing parameters
SI                    65536
SF            400.1300181 MHz
WDW                     EM
SSB                      0
LB                    0.30 Hz
GB                       0
PC                    2.00

1D NMR plot parameters
CX                   22.80 cm
CY                   15.00 cm
F1P                 11.000 ppm
F1                4401.43 Hz
F2P                -1.000 ppm
F2                -400.13 Hz
PPMCM             0.52632 ppm/cm
HZCM            210.59474 Hz/cm
```



9b


```
Current Data Parameters
USER                tjbark
NAME            2-TJB-112
EXPNO                  12
PROCNO                  1

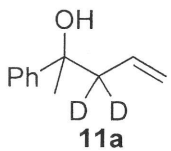
F2 - Acquisition Parameters
Date_            20080830
Time                10.13
INSTRUM          cryo500
PROBHD   5 mm CPTCI 1H-
PULPROG          zgdc30
TD                  65418
SOLVENT           CDCl3T
NS                    300
DS                      4
SWH             30303.031 Hz
FIDRES          0.463222 Hz
AQ              1.0794470 sec
RG                14596.5
DW                16.500 usec
DE                 6.00 usec
TE                298.0 K
D1              0.25000000 sec
d11             0.03000000 sec
MCREST          0.00000000 sec
MCWRK           0.01500000 sec

======== CHANNEL f1 ========
NUC1                  13C
P1                 14.75 usec
PL1                -1.00 dB
SFO1          125.7942548 MHz

======== CHANNEL f2 ========
CPDPRG2           waltz16
NUC2                  1H
PCPD2             100.00 usec
PL2                 1.60 dB
PL12               24.80 dB
SFO2          500.2225011 MHz

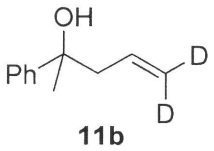
F2 - Processing parameters
SI                    65536
SF            125.7804006 MHz
WDW                     EM
SSB                      0
LB                  1.00 Hz
GB                       0
PC                    2.00

1D NMR plot parameters
CX                 22.80 cm
CY                 22.00 cm
F1P               230.637 ppm
F1                29009.68 Hz
F2P               -10.287 ppm
F2                -1293.96 Hz
PPMCM           10.56688 ppm/cm
HZCM          1329.10693 Hz/cm
```



11a

+



11b

7.46348
7.44490
7.37552
7.37063
7.35711
7.33725
7.27024
7.25258

5.16933
5.14974
5.12637
5.12443

2.69362
2.67750
2.54285
2.52205

2.04603
2.04180

1.56281
1.56033

0.01290

Current Data Parameters
USER tjbark
NAME 2-TJB-72
EXPNO 4
PROCNO 1

F2 - Acquisition Parameters
Date_ 20080721
Time 10.53
INSTRUM drx400
PROBHD 5 mm QNP H/F/P
PULPROG zg30
TD 65536
SOLVENT CDCl3T
NS 1
DS 0
SWH 6410.256 Hz
FIDRES 0.097813 Hz
AQ 5.1118579 sec
RG 456.1
DW 78.000 usec
DE 4.50 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

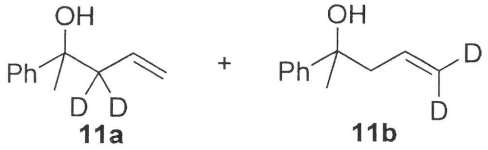
======== CHANNEL f1 ========
NUC1 1H
P1 12.00 usec
PL1 0.00 dB
SF01 400.1328009 MHz

F2 - Processing parameters
SI 65536
SF 400.1300179 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 2.00

1D NMR plot parameters
CX 22.80 cm
CY 8.00 cm
F1P 11.000 ppm
F1 4401.43 Hz
F2P -1.000 ppm
F2 -400.13 Hz
PPMCM 0.52632 ppm/cm
HZCM 210.59474 Hz/cm

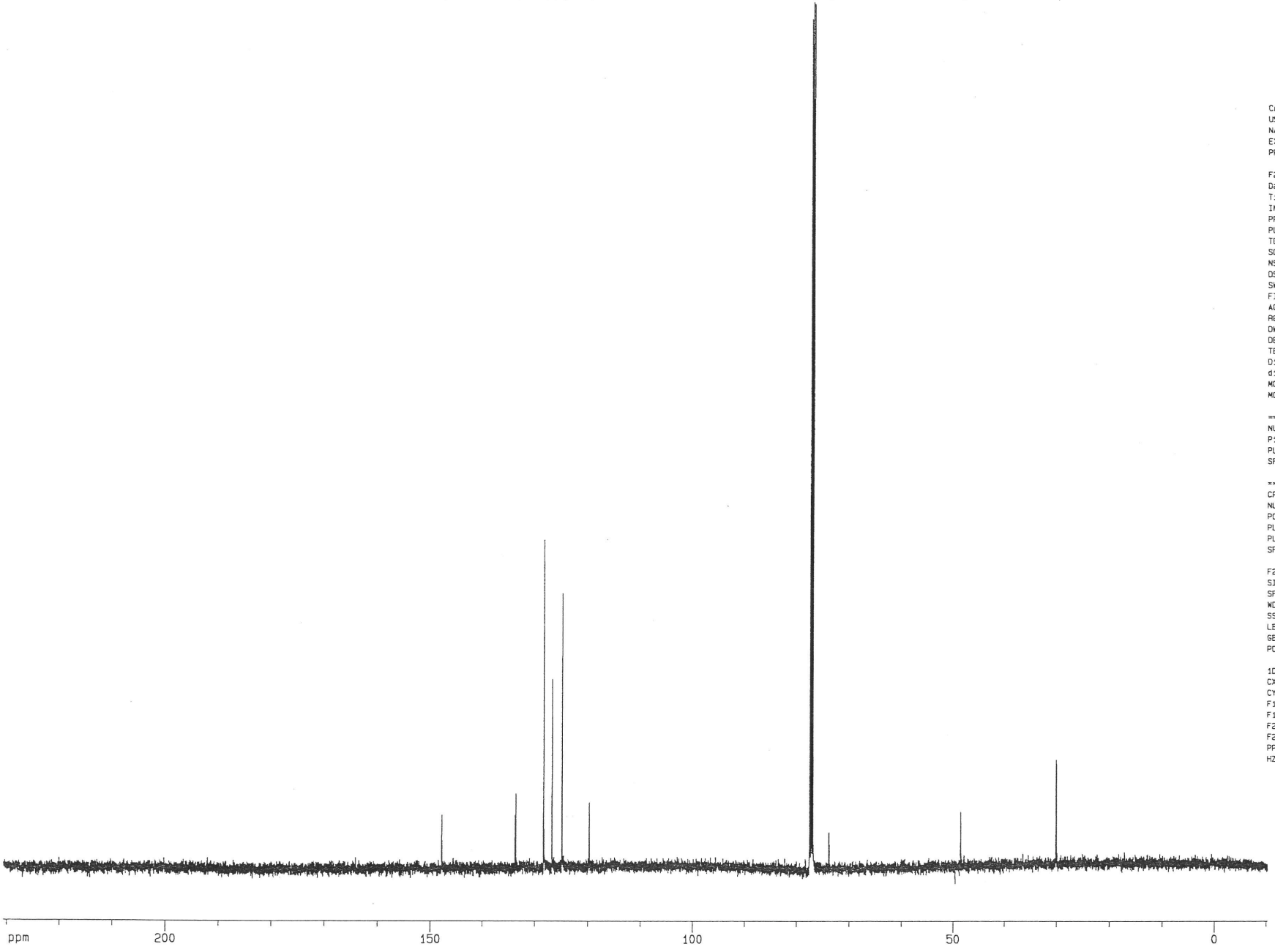
integral
2.0537
2.0153
1.5007
0.9700
1.0030
0.5017
0.4986
0.9841
3.3688

ppm
10
8
6
4
2
0



11a + **11b**

147.83
133.79
133.64
128.39
126.83
124.95
119.76
77.48
77.23
76.98
73.82
48.55
30.13
30.08




```
Current Data Parameters
USER                tjbark
NAME              2-TJB-71
EXPNO                    4
PROCNO                   1

F2 - Acquisition Parameters
Date_            20080827
Time                11.41
INSTRUM          cryo500
PROBHD   5 mm CPTCI 1H-
PULPROG            zgdc30
TD                  65536
SOLVENT             CDCl3T
NS                    129
DS                      4
SWH            30303.031 Hz
FIDRES          0.462388 Hz
AQ              1.0813940 sec
RG                  13004
DW                16.500 usec
DE                 6.00 usec
TE                298.0 K
D1            0.25000000 sec
d11           0.03000000 sec
MCREST        0.00000000 sec
MCWRK         0.01500000 sec

======== CHANNEL f1 ========
NUC1                  13C
P1                 14.75 usec
PL1                -1.00 dB
SFO1         125.7942548 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2             100.00 usec
PL2                1.60 dB
PL12               24.80 dB
SFO2         500.2225011 MHz

F2 - Processing parameters
SI                  65536
SF           125.7804002 MHz
WDW                    EM
SSB                     0
LB                 1.00 Hz
GB                      0
PC                   2.00

1D NMR plot parameters
CX                 22.80 cm
CY                 15.65 cm
F1P              230.610 ppm
F1             29006.22 Hz
F2P              -10.310 ppm
F2             -1296.81 Hz
PPMCM          10.56667 ppm/cm
HZCM         1329.08044 Hz/cm
```