On the nature of the oxidative heterocoupling of lithium enolates

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Supporting Information

Instrumentation

Proton, carbon, and lithium NMR were recorded on a Bruker 500 MHz spectrometer. GC-MS analyses were performed with an HP 5890 Series II Gas Chromatograph with an HP Mass Selector Detector. LC-HRMS data were recorded at the Mass Spectrometry Facility at Notre Dame University. Column chromatography was performed using the automated CombiFlash® Rf system from Teledyne Isco, Inc. Products were separated using prepacked silica gel columns with a gradient elution of either ethyl acetate:hexanes or diethyl ether:hexanes.

Materials for ^{7}Li NMR experiments

THF was purified with a Pure Solv solvent purification system from Innovative Technology, Inc. Toluene was degassed with argon for 1 hour and then stored over activated 3 Å molecular sieves under an inert atmosphere. A stock solution of 2.0M THF/toluene was prepared by diluting 8.1 mL of THF to a final volume of 50 mL with toluene. LiHMDS was purchased from Sigma Aldrich as a white solid and used without further purification. All ketones were purchased from Alfa Aesar or Acros Organics and purified by short-path distillation, recrystallization from *n*-pentane, or column chromatography prior to use. As a reference and locking signal, 0.3M LiCl in CD_3OD was prepared and flame-sealed inside melting point capillaries. All solutions and substrates were stored inside a glovebox filled with argon.

Materials for oxidative heterocoupling reactions

THF was purified with a Pure Solv solvent purification system from Innovative Technology, Inc. LiHMDS was purchased from Sigma Aldrich as a white solid and used without further purification. All ketones were purchased from Alfa Aesar or Acros Organics and purified by short-path distillation, recrystallization from *n*-pentane, or column chromatography

prior to use. Molecular iodine was purchased from Acros Organics and used without further purification. Anhydrous *N*,*N*-dimethylforamide (DMF) was purchased from Acros Organics.

General procedure for the determination of lithium enolate aggregation

All glassware was flame-dried before use. Two portions of LiHMDS (0.304 mmol each) were dissolved in 0.5 mL of 2.0M THF/toluene each in septated vials with magnetic stirrers and cooled to -10 ^{o}C. Ketone A and ketone B (0.3 mmol each) were dissolved separately in 0.5 mL 2.0M THF/toluene and added dropwise to one of the vials of LiHMDS. The two solutions were stirred at -10 ^{o}C for 45 minutes. The enolate solutions were then cooled to -78 ^{o}C, combined via syringe (stirred for 5 minutes), warmed to -10 ^{o}C (stirred for 5 minutes), and recooled to -78 ^{o}C. The solution was transferred to a septated NMR tube containing a sealed insert (0.3M LiCl in CD_3OD). The sample temperature was maintained at -78 ^{o}C until it was placed in the NMR spectrometer (NMR probe temperature was -30 ^{o}C). The sample was locked and shimmed (extensively) using the CD_3OD. A pre-thermal equilibrated ^{7}Li NMR spectrum was obtained for the sample at -30 ^{o}C. The NMR tube was then ejected from the spectrometer, warmed in-hand for 2 minutes, recooled to -30 ^{o}C inside the spectrometer, and the ^{7}Li NMR spectrum was recorded again. The peaks were then integrated with the signal corresponding to the A_4 aggregate set to a value of 1. The shift values are reported relative to the signal for LiCl (0.00 ppm). For all equimolar enolate mixtures reported, the lithium aggregates were ensembles of tetramers (A_4 : A_3B_1 : A_2B_2 : A_1B_3 : B_4) consistent with those reported by Collum *et al.* (*J. Am. Chem. Soc.*, **2008**, *130*, 4859).

General procedure for the oxidative heterocoupling of lithium enolates

All glassware was flame-dried before use. The ketone substrates (0.3 mmol of each) were dissolved together in 1.0 mL of THF in a septated vial with a magnetic stirrer. The vial

was then cooled to -10 ^{o}C. LiHMDS (0.64 mmol) was dissolved in 1.0 mL of THF and added dropwise to the solution of ketones. The solution was stirred at -10 ^{o}C for 45 minutes. The enolate solution was placed in a water bath at room temperature (stirred for 5 minutes) and then cooled to -78 ^{o}C. Molecular iodine (0.3 mmol) was dissolved in 1.0 mL of THF and added to the enolate solution dropwise via syringe with vigorous stirring. The reaction solution was removed from the -78 ^{o}C bath and allowed to slowly warm to room temperature over 30 minutes. The reaction was quenched with an equal volume of brine, separated, and the aqueous layer was extracted three times with ethyl acetate. The organic layers were combined, rotary evaporated to dryness, and the crude reaction mixture was redissolved in $CDCl_3$. DMF (0.3 mmol) was added. Product yields and ratios (heterocoupled product:homocoupled product) were determined by ^{1}H NMR. The heterocoupled products were purified via automated flash chromatography and characterized by ^{1}H NMR, ^{13}C NMR, GC-MS, and LC-HRMS.

^{7}Li NMR spectra of lithium enolate aggregates

5-Methoxy1-indanone (**A**) with pinacolone (**B**)

Procedure 5.2.3.1 was followed. ^{7}Li NMR (2.0M THF/toluene, 194 MHz, shifts relative to 0.3M LiCl in CD_3OD) – δ 1.18 (A_3**B_1**), 1.12 (**A_4**), 0.79 (**A_2**B_2), 0.69 (**A_3**B_1), 0.27 (A_1**B_3**), 0.24 (A_2**B_2**), -0.03 (**A_1**B_3), -0.07 (**B_4**).

5-Bromo-1-indanone (**A**) and pinacolone (**B**)

Procedure 5.2.3.1 was followed. ^{7}Li NMR (2.0M THF/toluene, 194 MHz, shifts relative to 0.3M LiCl in CD_3OD) – δ 0.99 (A_3**B_1**), 0.88 (**A_4**), 0.66 (**A_2**B_2), 0.51 (**A_3**B_1), 0.20 (A_1**B_3**), 0.12 (A_2**B_2**), -0.06 (**B_4**), -0.09 (**A_1**B_3).

1-Indanone (**A**) and pinacolone (**B**)

Procedure 5.2.3.1 was followed. ^{7}Li NMR (2.0M THF/toluene, 194 MHz, shifts relative to 0.3M LiCl in CD_3OD) – δ 1.21 (A_3**B**$_1$), 1.13 (**A**$_4$), 0.80 (**A**$_2$B$_2$), 0.69 (**A**$_3$B$_1$), 0.28 (A_1**B**$_3$), 0.24 (A_2**B**$_2$), -0.04 (**A**$_1$B$_3$), -0.07 (**B**$_4$).

4-Chromanone (**A**) and pinacolone (**B**)

Procedure 5.2.3.1 was followed. ^{7}Li NMR (2.0M THF/toluene, 194 MHz, shifts relative to 0.3M LiCl in CD_3OD) – δ 0.78 (A_3**B**$_1$), 0.73 (**A**$_4$), 0.60 (**A**$_2$B$_2$), 0.44 (**A**$_3$B$_1$), 0.20 (A_1**B**$_3$), 0.06 (A_2**B**$_2$), -0.07 (**B**$_4$), -0.17 (**A**$_1$B$_3$).

1-Tetralone (**A**) and pinacolone (**B**)

Procedure 5.2.3.1 was followed. ^{7}Li NMR (2.0M THF/toluene, 194 MHz, shifts relative to 0.3M LiCl in CD_3OD) – δ 0.97 (A_3**B**$_1$), 0.95 (**A**$_4$), 0.70 (**A**$_2$B$_2$), 0.61 (**A**$_3$B$_1$), 0.27 (A_1**B**$_3$), 0.19 (A_2**B**$_2$), -0.06 (**A**$_1$B$_3$), -0.10 (**B**$_4$).

Synthesis and spectral data for heteocoupled products

2-(3,3-Dimethyl-2-oxobutyl)-5-methoxy-indan-1-one (**12**)

Procedure 5.2.3.2 was followed. Clear, colorless oil. 62% yield. ^{1}H NMR ($CDCl_3$, 500 MHz) – δ 7.68-7.63 (m, 1H), 6.89-6.84 (m, 1H), 6.84-6.81 (m, 1H), 3.84 (s, 3H), 3.38 (dd, 1H, *J* = 7.8 Hz, 17.7 Hz), 3.16 (dd, 1H, *J* = 3.4 Hz, 18.3 Hz), 3.01-2.94 (m, 1H), 2.75 (dd, 1H, 9.5 Hz, 18.3 Hz), 2.56 (dd, 1H, *J* = 4.2 Hz, 17.2 Hz), 1.13 (s, 9H). ^{13}C NMR ($CDCl_3$, 125 MHz) – δ 214.2, 206.3, 165.3, 156.5, 129.8, 125.4, 115.3, 109.5, 55.6, 43.9, 43.0, 38.4, 33.6, 26.4. MS [*m*/*z* (rel int)] 260 (M^+, 9), 203 (22), 175 (100), 161 (10), 147 (36), 131 (9), 115 (13), 103 (11), 91 (12), 77 (9), 57 (31) . LC-HRMS calcd. for $C_{16}H_{21}O_3$ [M+H] 261.1485, found 261.1480.

5-Bromo-2-(3,3-dimethyl-2-oxobutyl)-indan-1-one (**13**)

Procedure 5.2.3.2 was followed. Light yellow oil. 58% yield. ^{1}H NMR ($CDCl_3$, 500 MHz) – δ 7.63-7.57 (m, 2H), 7.51-7.46 (m, 1H), 3.39 (dd, 1H, *J* = 8.0 Hz, 17.3 Hz), 3.18 (dd, 1H, *J* = 2.7 Hz, 17.7 Hz), 2.97-2.92 (m, 1H), 2.88 (dd, 1H, *J* = 8.3 Hz, 18.0 Hz), 2.64 (dd, 1H, *J* = 4.4 Hz, 17.5 Hz) 1.14 (s, 9H). ^{13}C NMR ($CDCl_3$, 125 MHz) – δ 213.9, 206.8, 155.0, 135.5, 131.0, 130.0, 129.8, 125.0, 43.9, 43.0, 38.0, 33.0, 26.5. MS [*m*/*z* (rel int)] 308/310 (M^+, 9), 251/253 (29), 223/225 (76), 145 (22), 115 (88), 89 (21), 57 (100). LC-HRMS calcd. for $C_{15}H_{18}BrO_2$ [M+H] 309.0485, found 309.0462.

2-(3,3-Dimethyl-2-oxobutyl)-indan-1-one (**14**)

Procedure 5.2.3.2 was followed. Clear, colorless oil. 62% yield. ^{1}H NMR ($CDCl_3$, 500 MHz) – δ 7.80-7.75 (m, 1H), 7.62-7.56 (m, 1H), 7.47-7.43 (m, 1H), 7.41-7.35 (m, 1H), 3.46 (dd, 1H, *J* = 8.0 Hz, 17.2 Hz), 3.22 (dd, 1H, *J* = 3.4 Hz, 18.5 Hz), 3.05-2.98 (m, 1H), 2.86 (dd, 1H, *J* = 8.8 Hz, 18.5 Hz), 2.67 (dd, 1H, *J* = 4.6 Hz, 17.6 Hz), 1.18 (s, 9H). ^{13}C NMR ($CDCl_3$, 125 MHz) – δ 214.1, 208.2, 153.5, 136.6, 134.7, 127.4, 126.5, 123.8, 44.0, 43.0, 38.2, 33.5, 26.5. MS [*m*/*z* (rel int)] 230 (M^+, 8), 173 (16), 145 (100), 131 (11), 115 (67), 91 (21), 57 (27). LC-HRMS calcd. for $C_{15}H_{19}O_2$ [M+H] 231.1380, found 231.1383.

3-(3,3-Dimethyl-2-oxobutyl)-chroman-4-one (**15**)

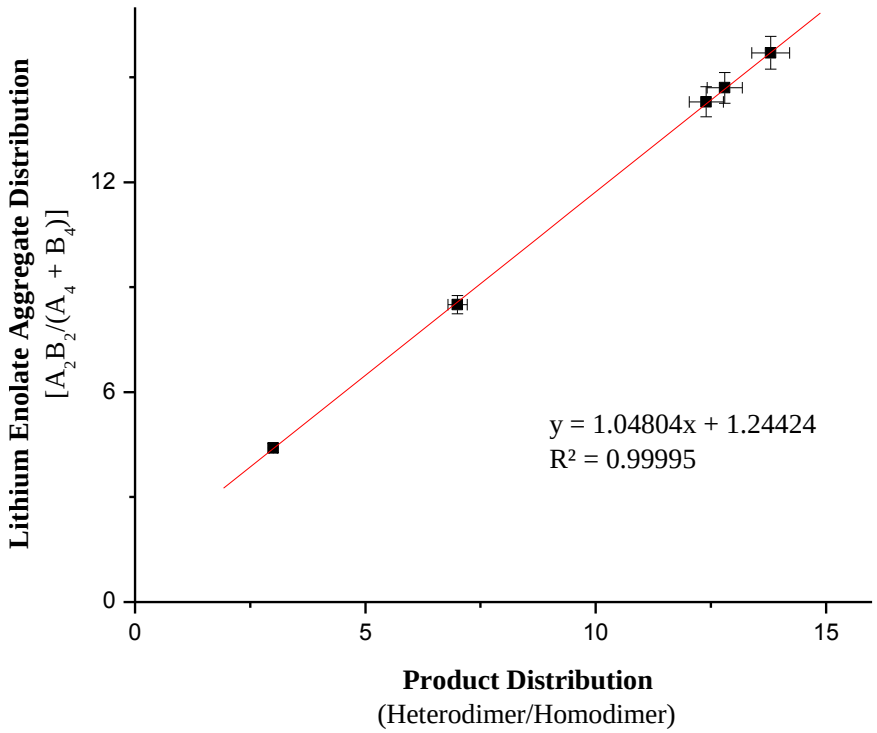
Procedure 5.2.3.2 was followed. Off-white solid. 46% yield. ^{1}H NMR ($CDCl_3$, 500 MHz) – δ 7.88-7.83 (m, 1H), 7.49-7.42 (m, 1H), 7.03-6.97 (m, 1H), 6.97-6.92 (m, 1H), 4.48 (dd, 1H, *J* = 5.2 Hz, 11.1 Hz), 4.20 (t, 1H, *J* = 11.3 Hz), 3.42-3.35 (m, 1H), 3.10 (dd, 1H, *J* = 4.4 Hz, 18.4 Hz), 2.57 (dd, 1H, *J* = 8.0 Hz, 18.1 Hz), 1.18 (s, 9H). ^{13}C NMR ($CDCl_3$, 125 MHz) – δ 212.9, 193.7, 161.8, 135.9, 127.3, 121.4, 120.7, 117.8, 70.3, 44.3, 41.7, 32.6, 26.5. MS [*m*/*z* (rel

int)] 246 (M^+, 3), 189 (100), 171 (9), 161 (24), 147 (40), 133 (6), 121 (16), 92 (14), 57 (36). LC-HRMS calcd. for $C_{15}H_{19}O_3$ [M+H] 247.1329, found 247.1330.

2-(3,3-Dimethyl-2-oxobutyl)-tetral-1-one (**16**)

Procedure 5.2.3.2 was followed. Light yellow oil. 47% yield. ^{1}H NMR ($CDCl_3$, 500 MHz) – δ 8.01-7.96 (m, 1H), 7.47-7.40 (m, 1H), 7.30-7.25 (m, 1H), 7.23-7.19 (m, 1H), 3.26 (dd, 1H, J = 5.1 Hz, 18.0 Hz), 3.17-3.06 (m, 2H), 2.96-2.89 (m, 1H), 2.56 (dd, 1H, J = 6.8 Hz, 18.1 Hz), 2.15-2.08 (m, 1H), 1.89 (dddd, 1H, J = 4.4 Hz, 12.9 Hz, 13.7 Hz, 13.1 Hz) 1.19 (s, 9H). ^{13}C NMR ($CDCl_3$, 125 MHz) – δ 214.2, 199.3, 144.1, 133.2, 132.4, 128.7, 127.4, 126.6, 44.2, 43.8, 37.4, 29.4, 26.5. MS [*m*/*z* (rel int)] 244 (M^+, 1) 187 (100), 169 (16), 159 (5), 145 (11), 131 (27), 115 (11), 91 (16), 57 (21). LC-HRMS calcd. for $C_{16}H_{21}O_2$ [M+H] 245.1536, found 245.1519.

Impact of heteroaggregation on the oxidative heterocoupling of lithium enolates



^{7}Li NMR spetra of lithium enolate aggregates

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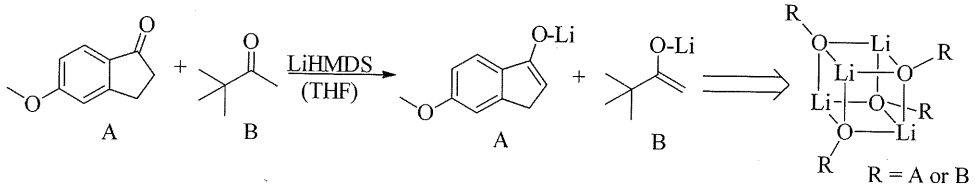

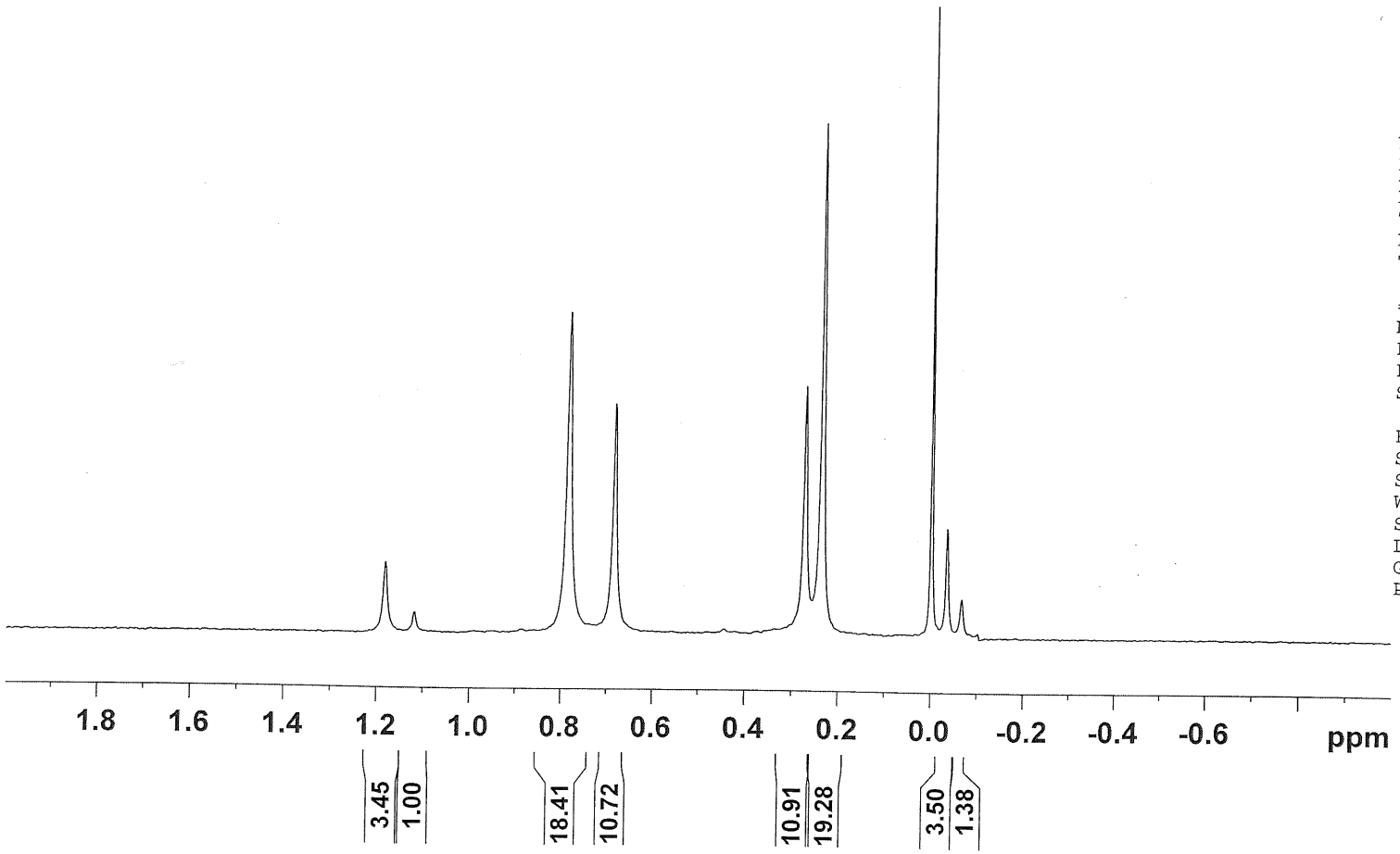

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PL1	-2.00 dB
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F2 - Processing parameters

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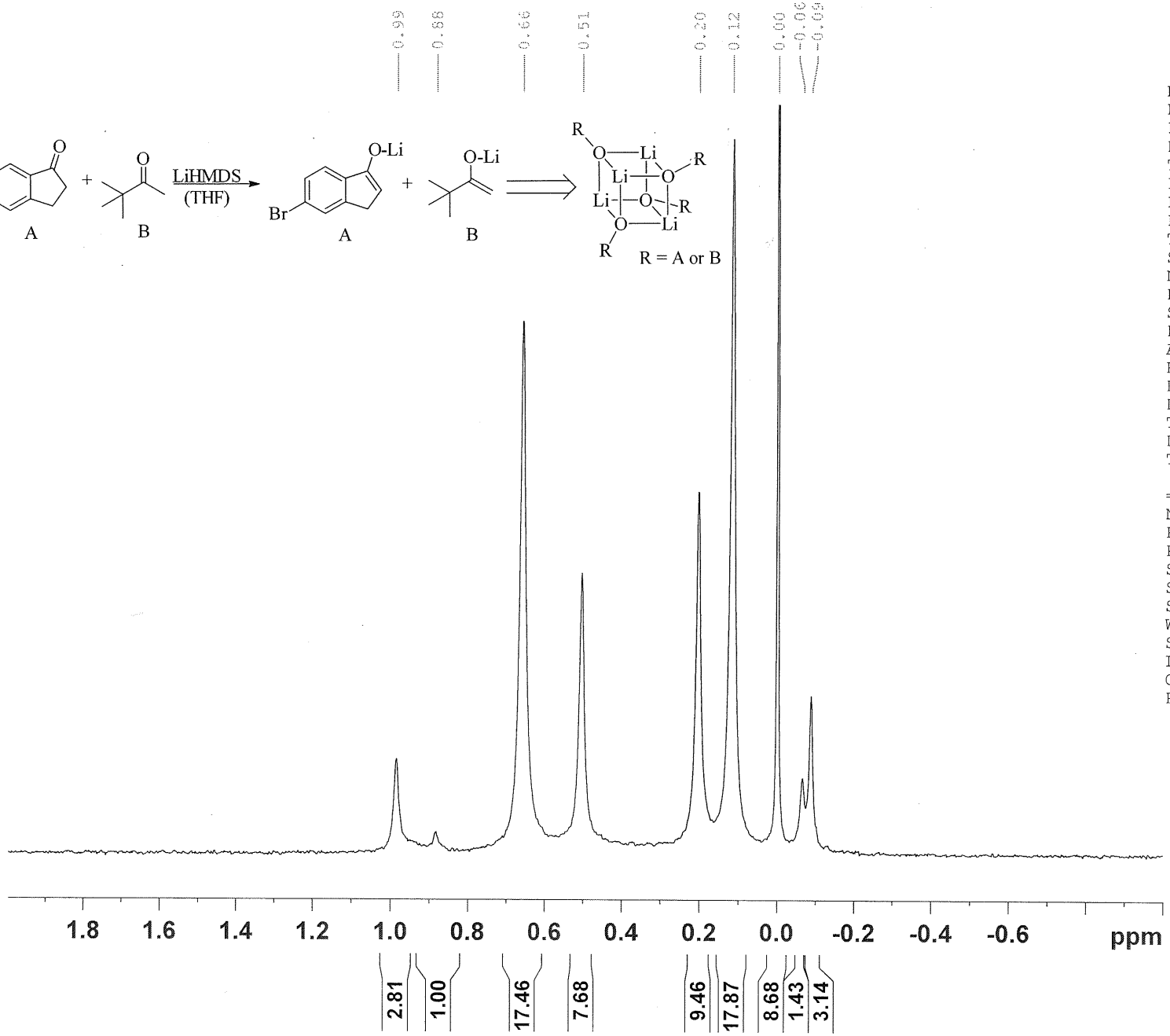
Br-Inda-Pina-0.15M Each-042011
Repeat-warming



BRUKER

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RG	4597.6
DW	50.000 usec
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TE	241.1 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	7Li
P1	9.10 usec
PL1	-2.00 dB
SFO1	194.3695080 MHz
SI	32768
SF	194.3694464 MHz
WDW	EM
SSB	0
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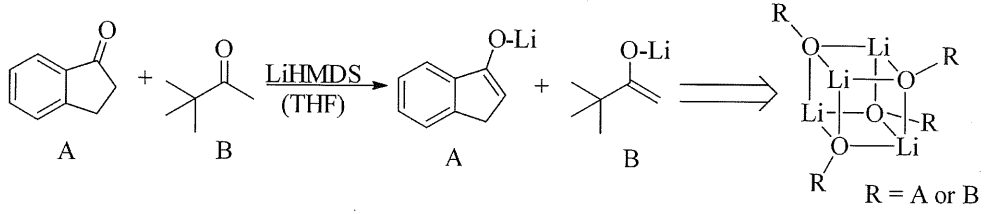
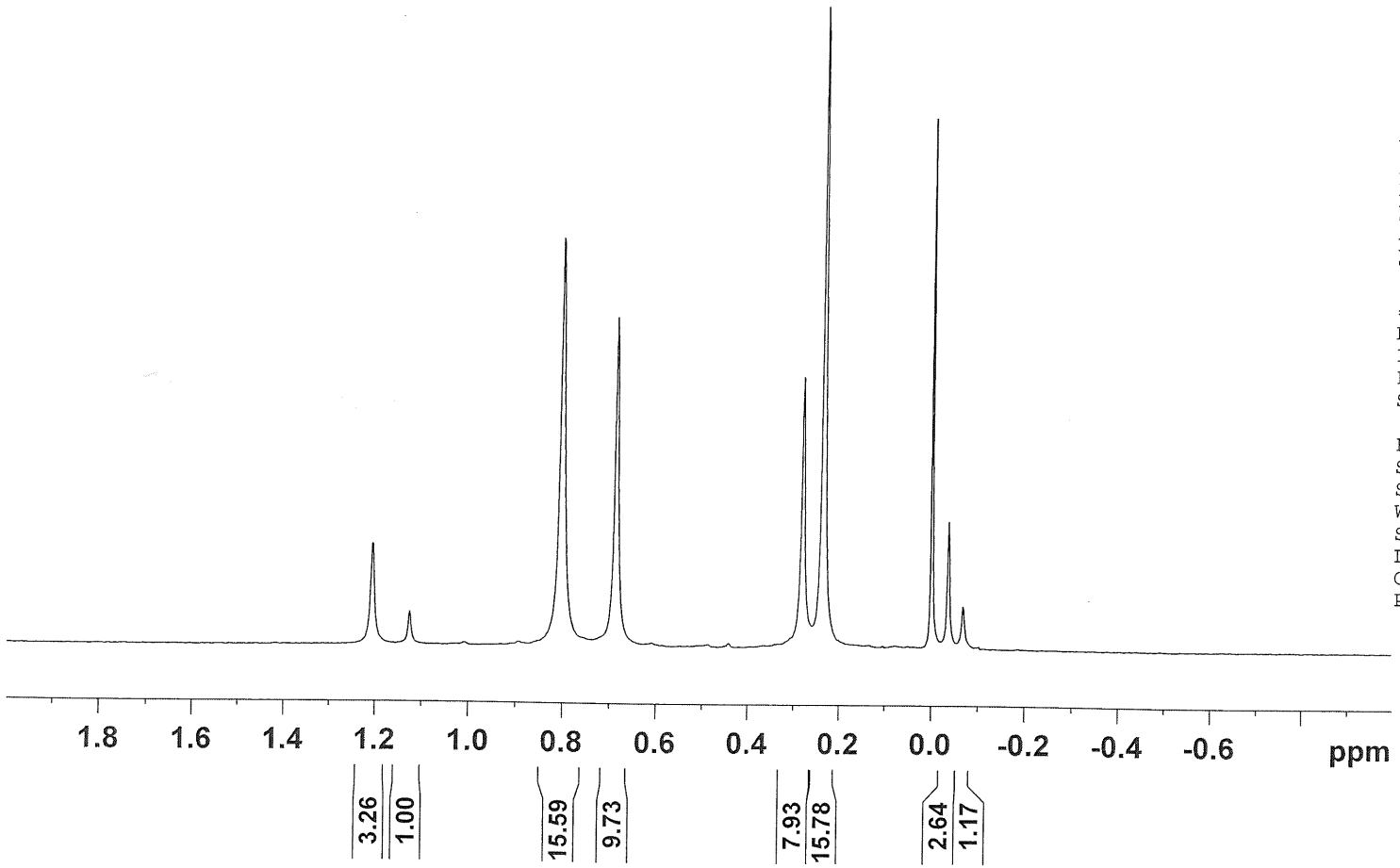
PC	1.40

IndaPina-Enolates-0.15M Each-062310
LiCl in MeOh-d4
Repeat/Warming-2





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

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F2 - Processing parameters

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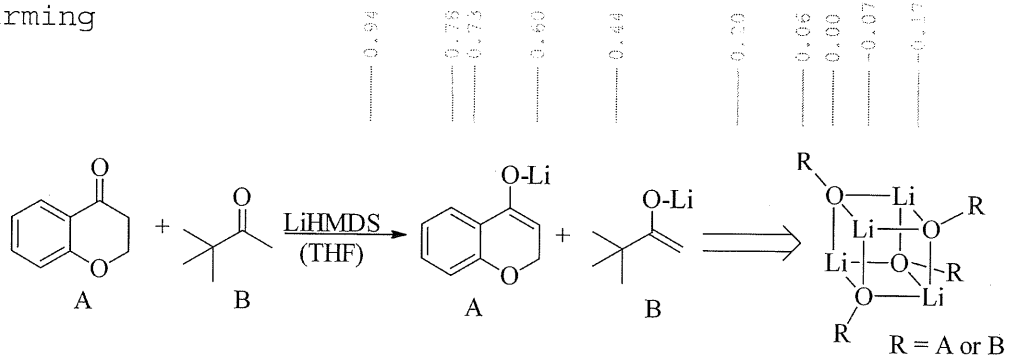
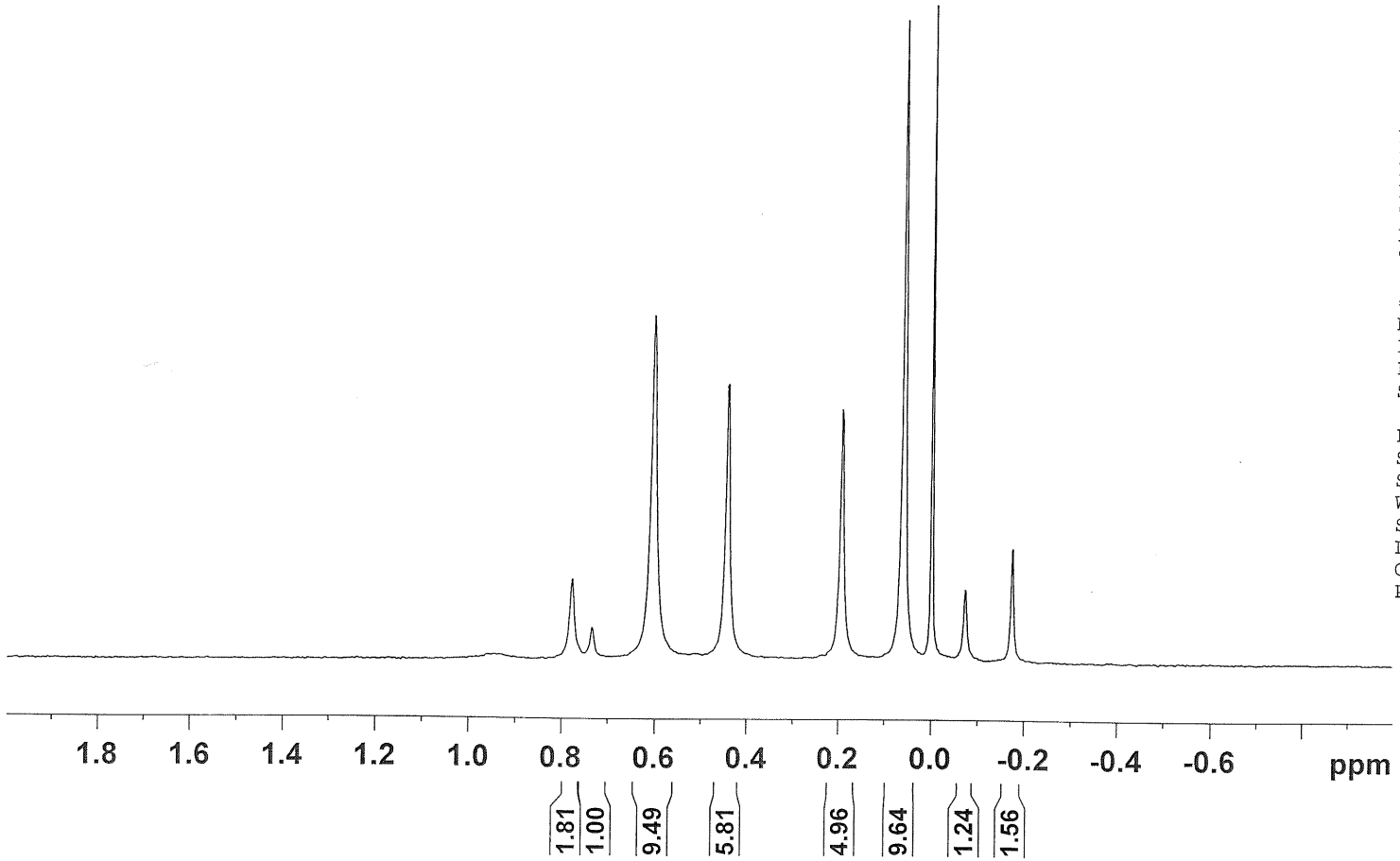
4-Chroma-Pina-Enolates-0.15M Each-063010
LiCl in MeOh-d4
Repeat/Warming





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TE 241.2 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
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F2 - Processing parameters
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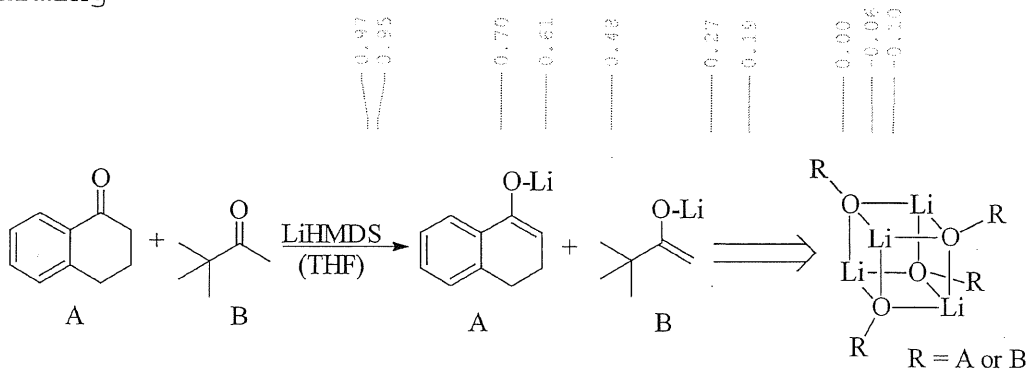
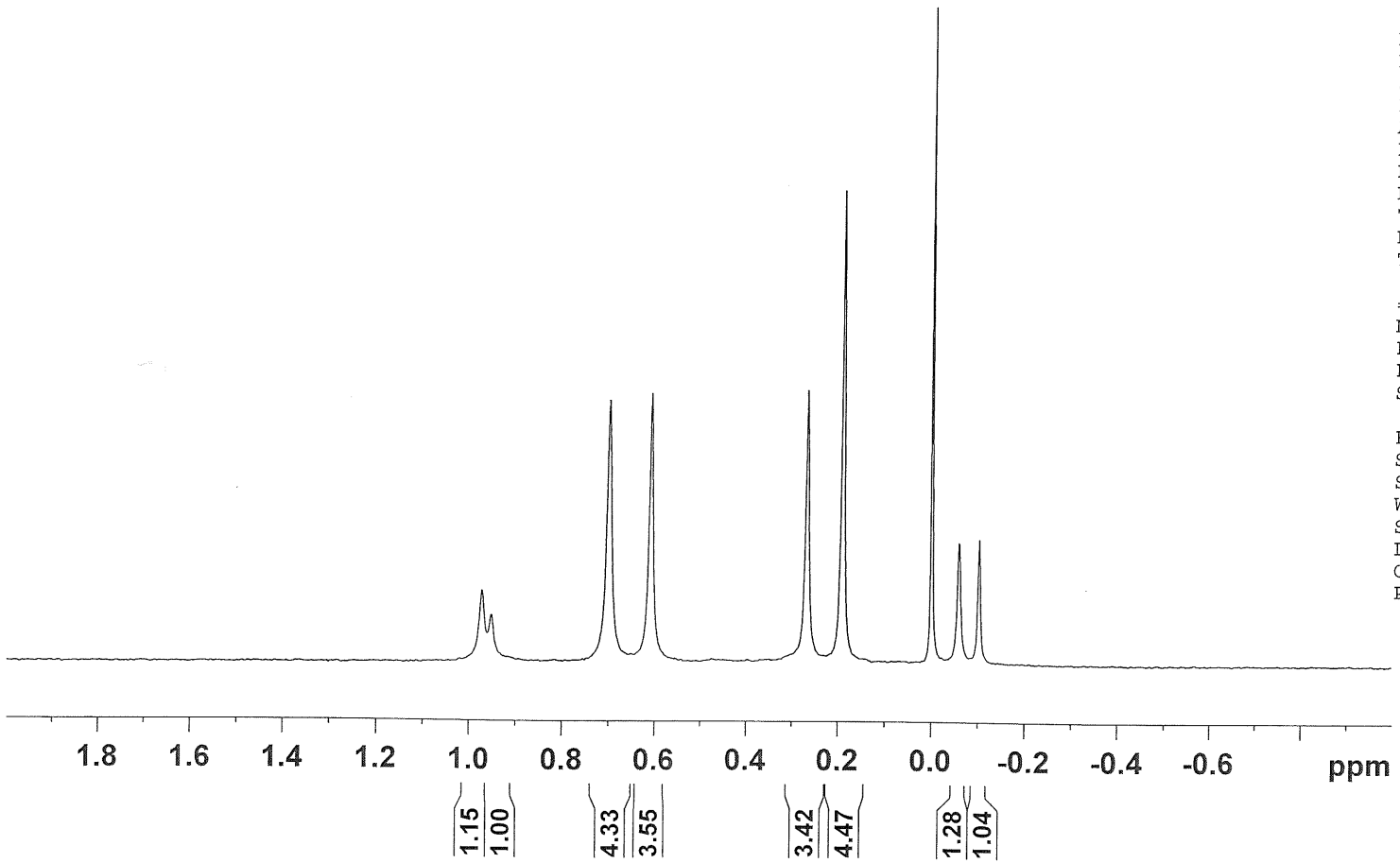
aTet-Pina-0.15M Each-040311
repeat-warming





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PROCNO	1

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RG	4597.6
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D1	1.00000000 sec
TD0	1

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

NUC1	7Li
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PL1	-2.00 dB
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F2 - Processing parameters

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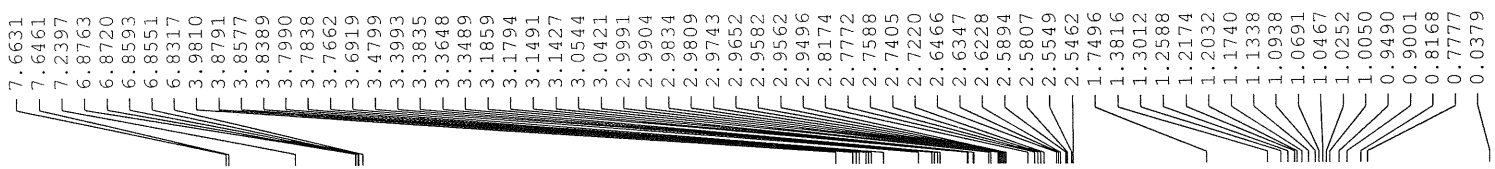
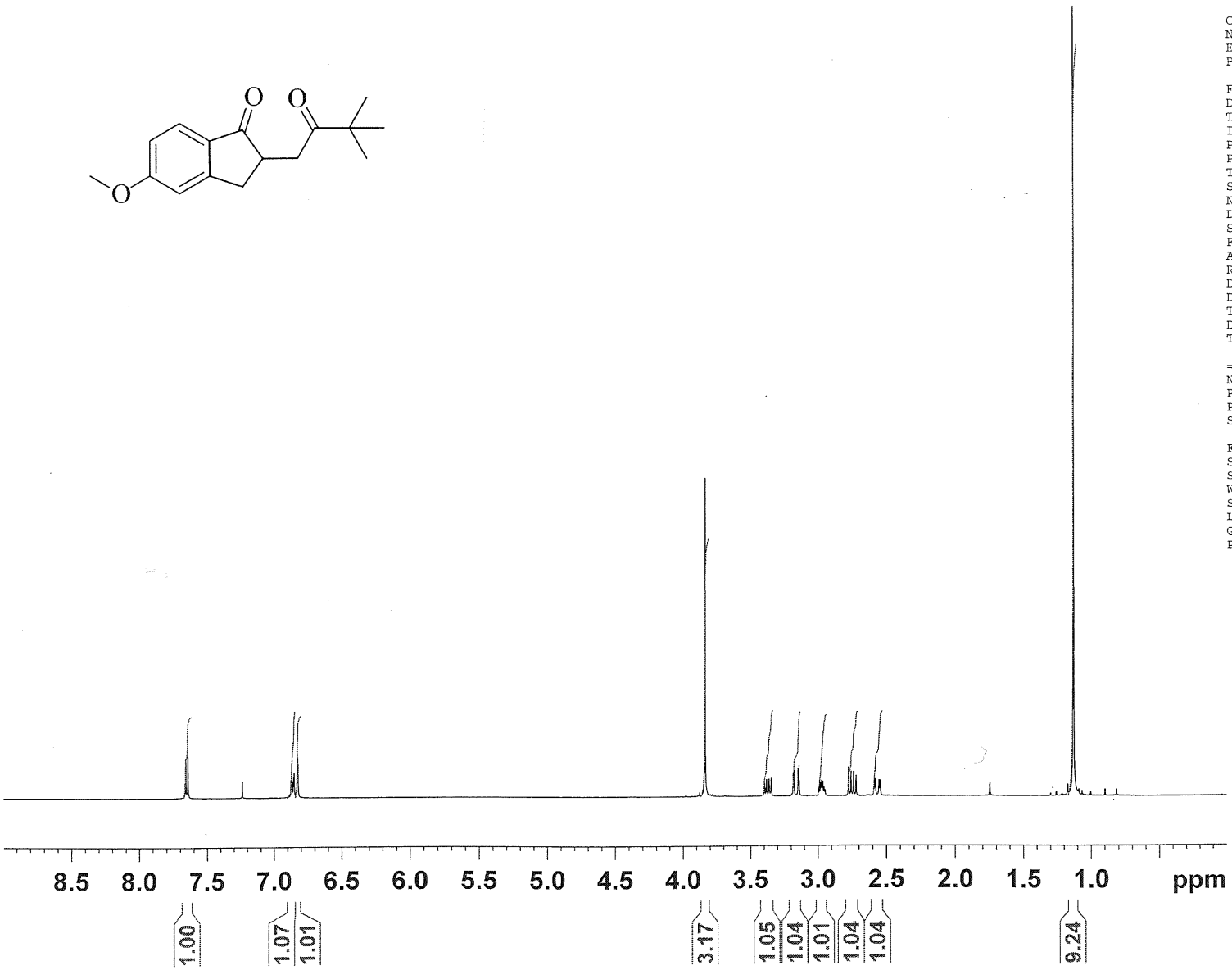
^{1}H NMR and ^{13}C NMR spectra of heterocoupled products

CF Fr8-13





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EXPNO 2
PROCNO 1

F2 - Acquisition Parameters

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

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PL1	1.00 dB
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F2 - Processing parameters

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CF Fr8-13
13C

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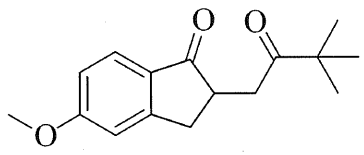
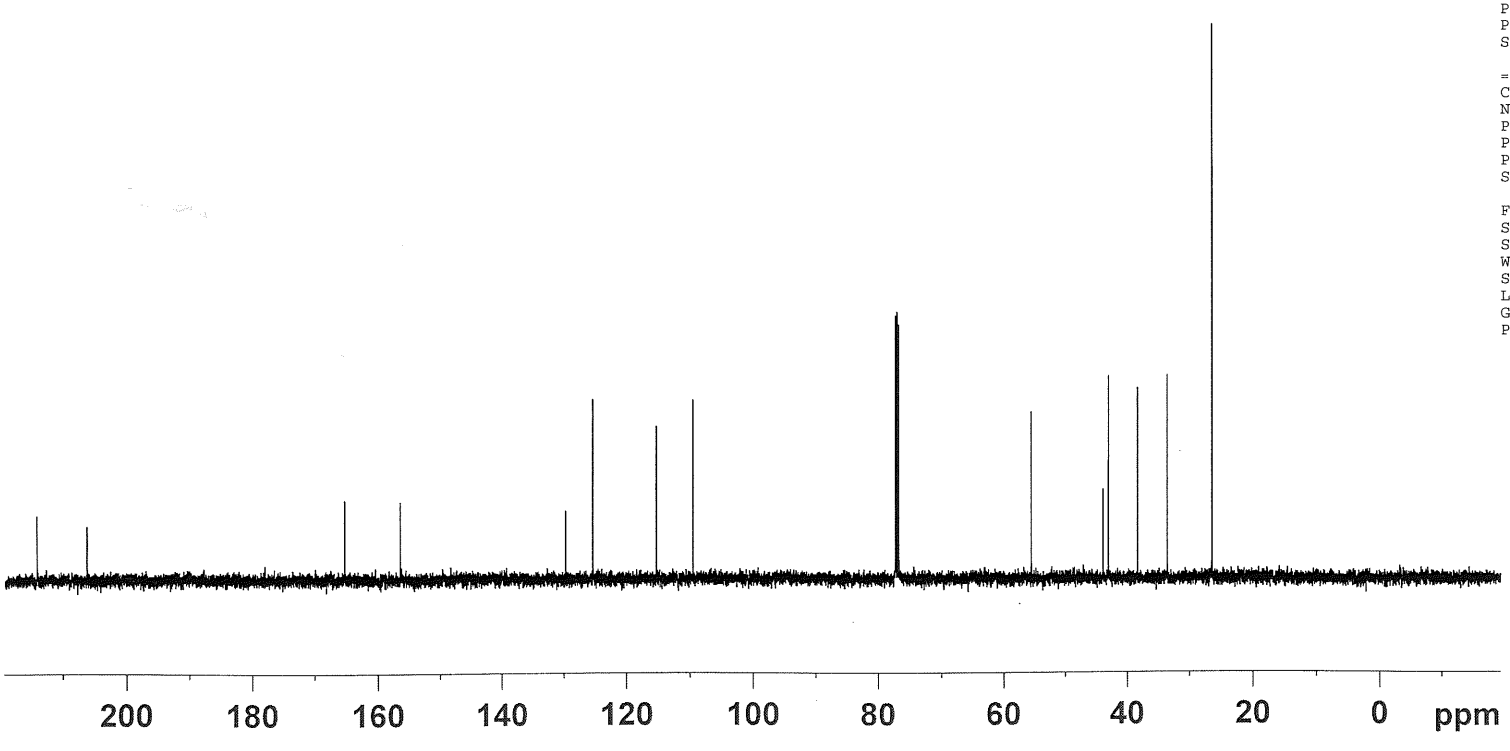
26.41





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PROCNO                1

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RG                 2048
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TE               296.6 K
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d11           0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
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PL1                 1.00 dB
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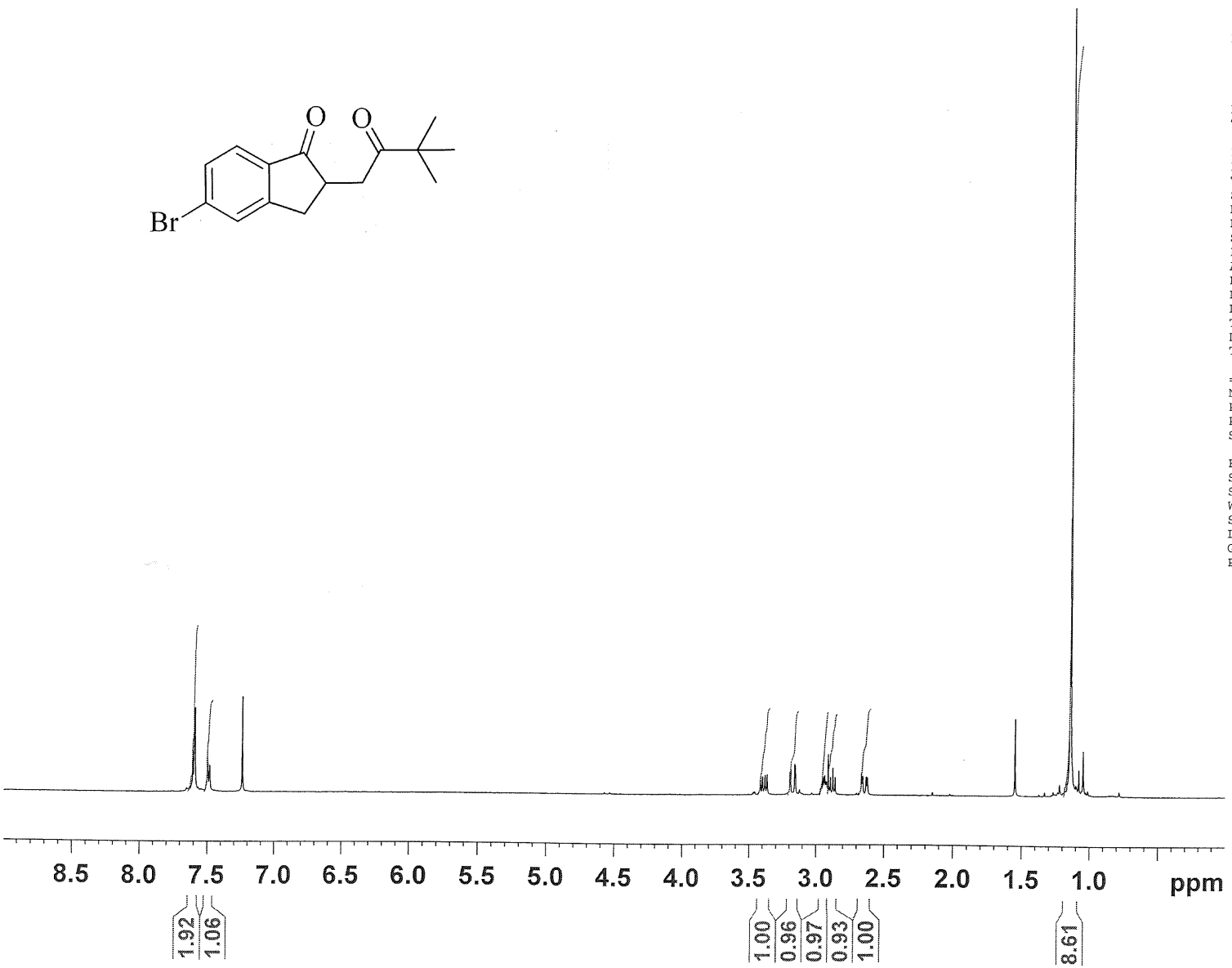
======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                 1H
PCPD2             100.00 usec
PL2                 1.00 dB
PL12               17.50 dB
SFO2        500.1322150 MHz

F2 - Processing parameters
SI                32768
SF          125.7577980 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```

Crude + DMF + 1:1
Tandem Enolization(In)



BRUKER

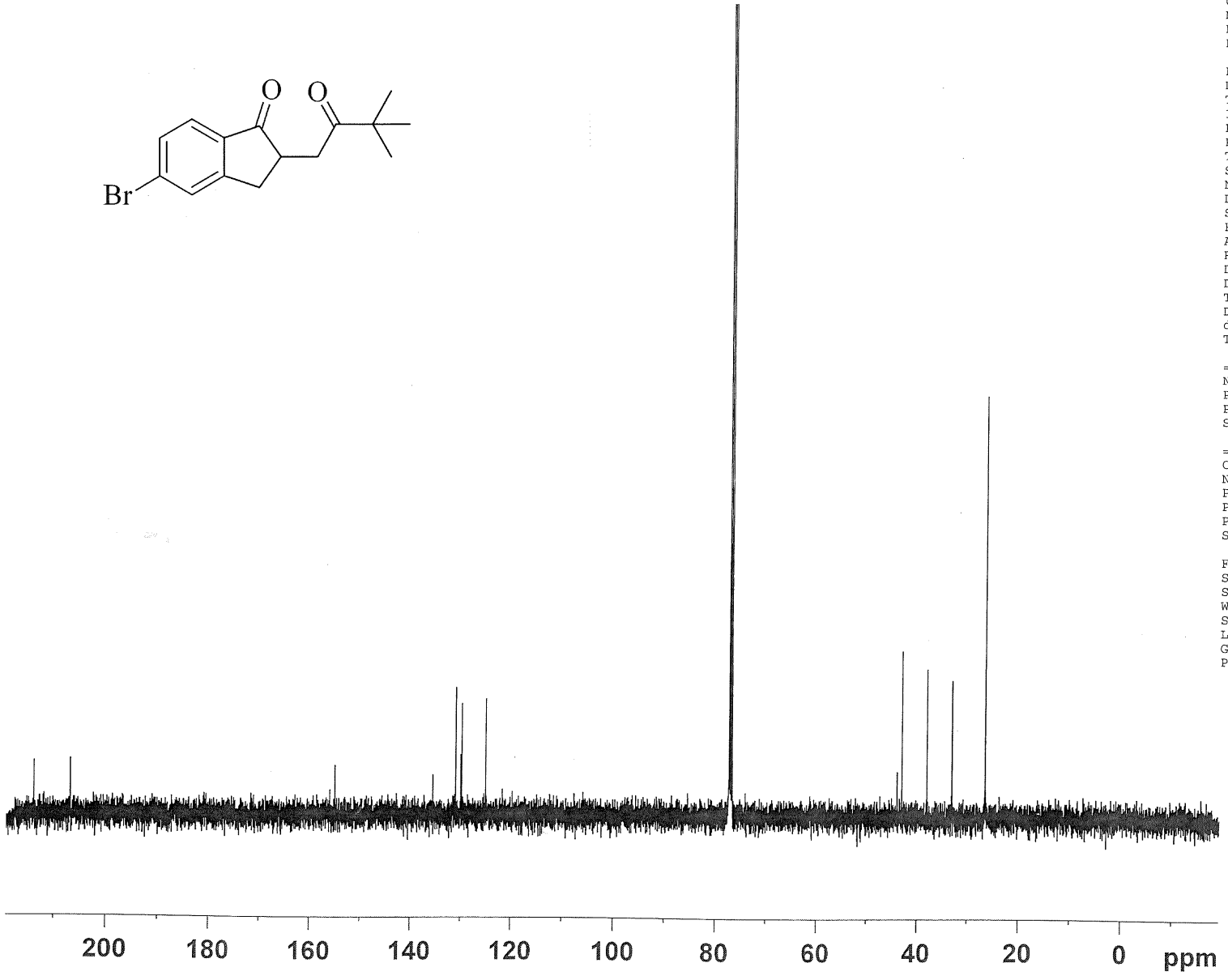
Current Data Parameters
NAME 5Br-IndaPina-LiHMDS-I2-042211
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20110422
Time 13.14
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 4
DS 0
SWH 10000.000 Hz
FIDRES 0.305176 Hz
AQ 1.6385000 sec
RG 406.4
DW 50.000 usec
DE 6.50 usec
TE 296.8 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 14.00 usec
PL1 1.00 dB
SFO1 500.1345012 MHz

F2 - Processing parameters
SI 32768
SF 500.1300232 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

TandemEnolization(Inv)
CF-Fr11
13C



Current Data Parameters
NAME 5Br-IndaPina-LiHMDS-I2-042211
EXPNO 3
PROCNO 1

F2 - Acquisition Parameters
Date_ 20110422
Time 13.20
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zgdc30
TD 65536
SOLVENT CDCl3
NS 210
DS 4
SWH 30030.029 Hz
FIDRES 0.458222 Hz
AQ 1.0912410 sec
RG 2048
DW 16.650 usec
DE 7.00 usec
TE 297.2 K
D1 1.00000000 sec
d11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 13C
P1 8.60 usec
PL1 1.00 dB
SFO1 125.7703640 MHz

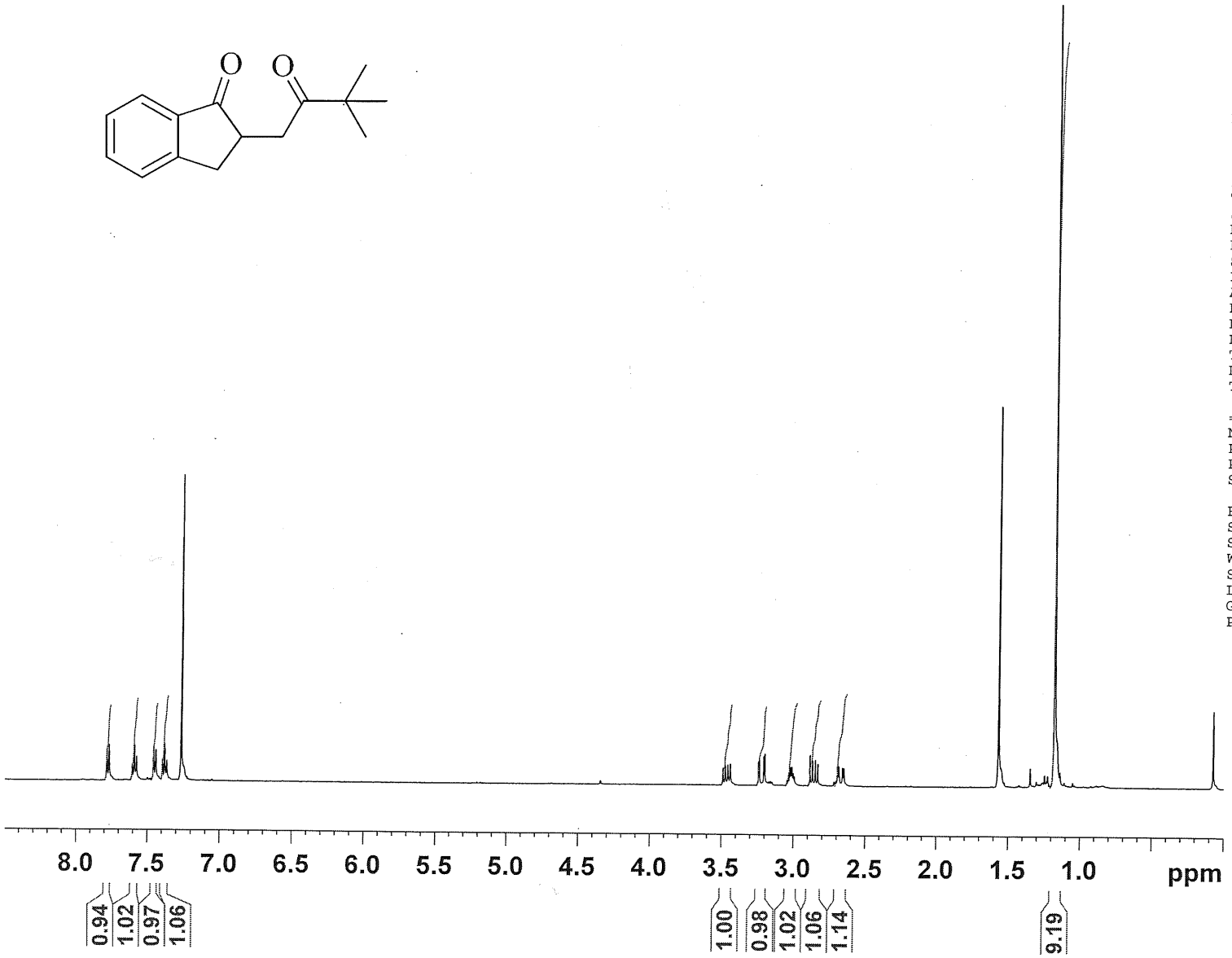
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 1.00 dB
PL12 17.50 dB
SFO2 500.1322150 MHz

F2 - Processing parameters
SI 32768
SF 125.7577921 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

CombiFlash2-Fr12-13

7.7845 7.7691 7.6064 7.5914 7.5766 7.4993 7.4839 7.4572 7.4418 7.3963 7.3813 7.3666 7.2658 7.0541

5.1827 4.3448 3.4911 3.4751 3.4568 3.4408 3.2437 3.2373 3.2070 3.2006 3.1740 3.1617 3.1504 3.0441 3.0370 3.0281 3.0194 3.0119 3.0037 2.9952 2.8869 2.8694 2.8502 2.8327 2.7197 2.7079 2.6945 2.6852 2.6598 2.6508 1.5674 1.5281 1.4269 1.3476 1.3048 1.2597 1.2469 1.2333 1.2264 1.1627 1.1402



Current Data Parameters
NAME Inda-Pina-LDA-CTAN-070209
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters

Date_	20090702
Time	14.09
INSTRUM	spect
PROBHD	5 mm TXI 1H/D-
PULPROG	zg30
TD	32768
SOLVENT	CDCl3
NS	6
DS	0
SWH	7002.801 Hz
FIDRES	0.213709 Hz
AQ	2.3397565 sec
RG	406.4
DW	71.400 usec
DE	6.00 usec
TE	295.8 K
D1	1.00000000 sec
TD0	1

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

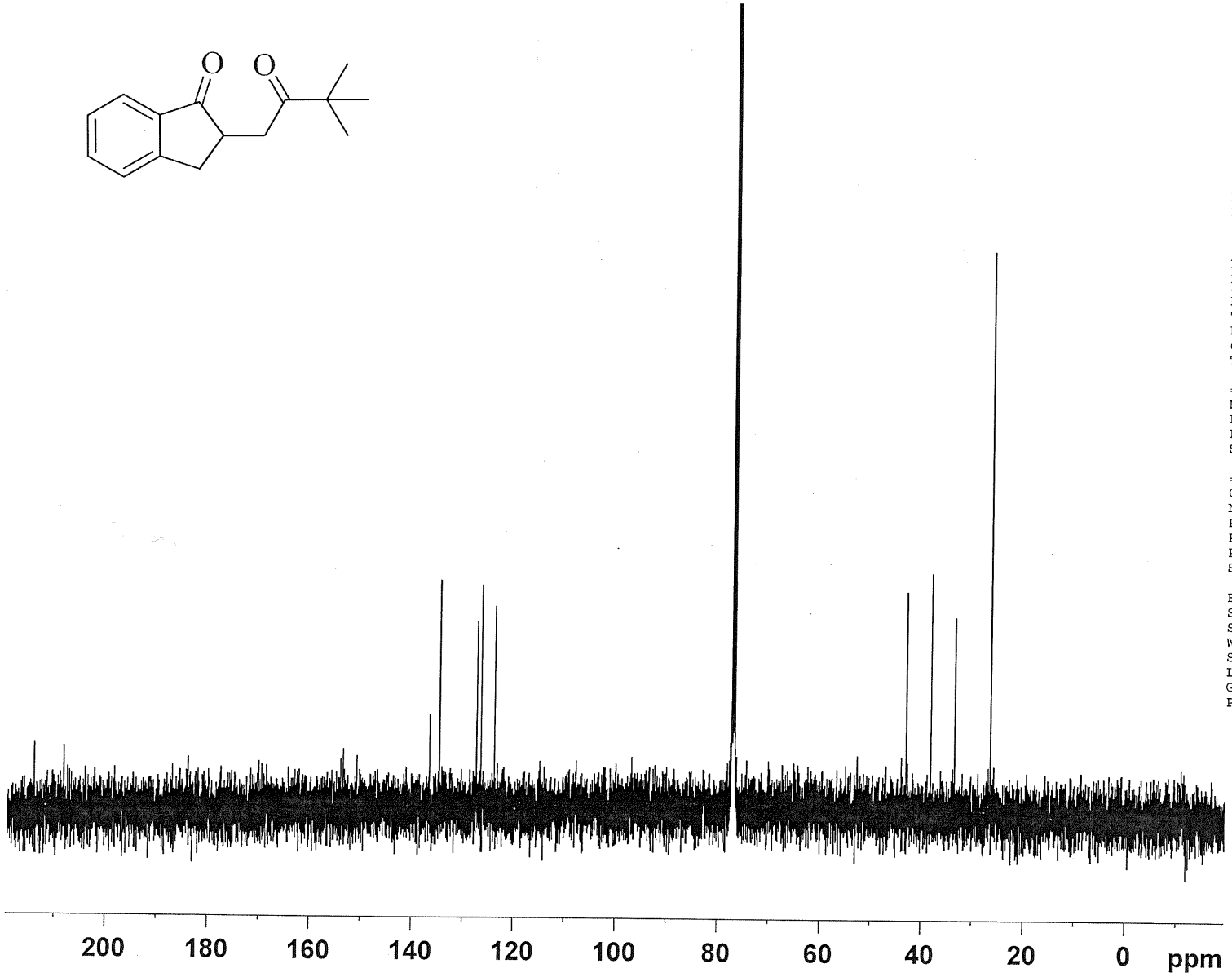
NUC1	1H
P1	7.25 usec
PL1	1.00 dB
SFO1	500.1335009 MHz

F2 - Processing parameters

SI	32768
SF	500.1300102 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

CombiFlash-Fr8-13C NMR

214.07
208.22
153.50
136.59
134.69
127.36
126.49
123.82
43.96
42.99
38.18
33.47
26.49



Current Data Parameters
NAME Inda-Pina-LDA-CTAN-061809-2
EXPNO 3
PROCNO 1

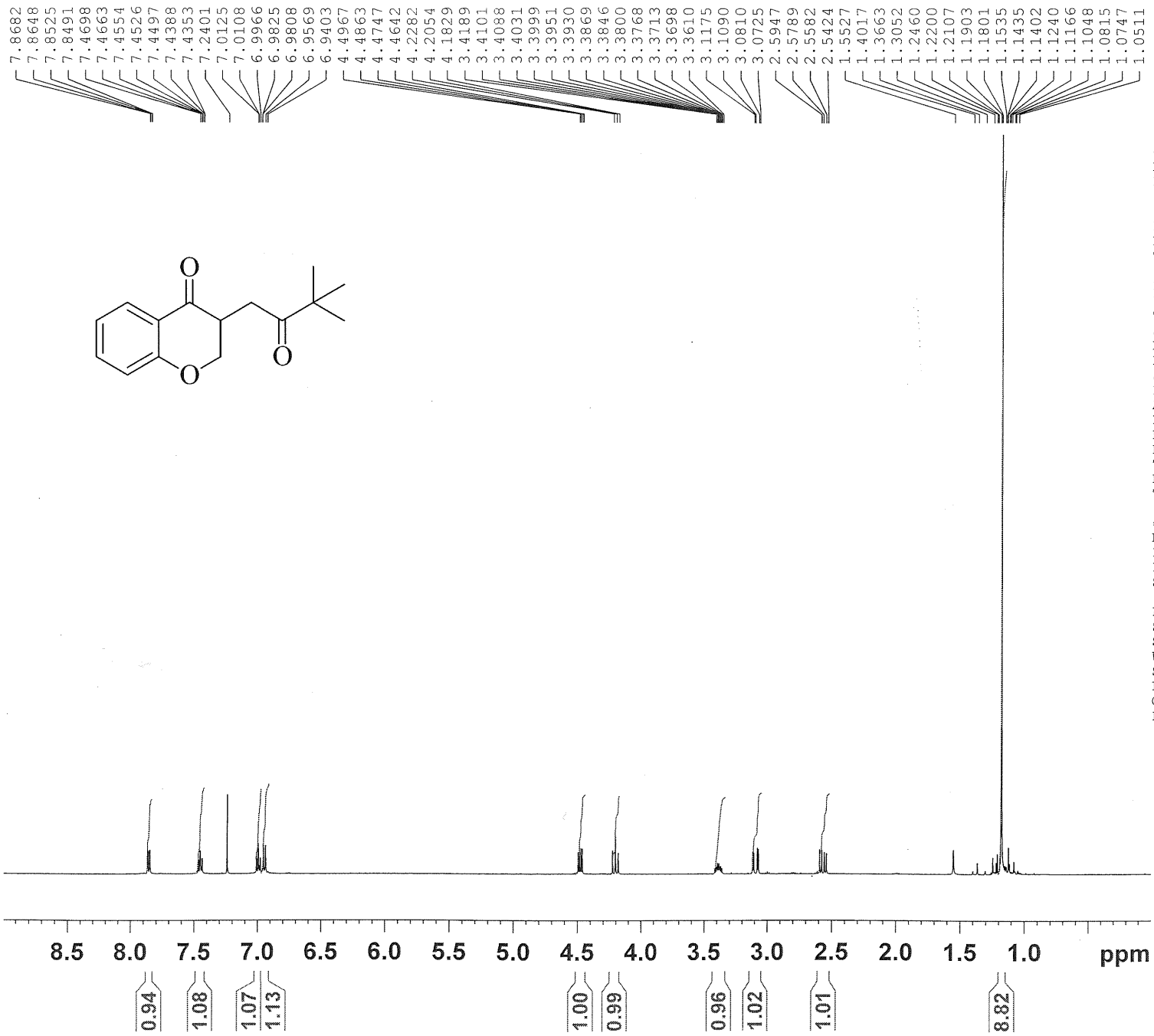
F2 - Acquisition Parameters
Date_ 20090622
Time 10.27
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zgdc30
TD 65536
SOLVENT CDCl3
NS 705
DS 4
SWH 30030.029 Hz
FIDRES 0.458222 Hz
AQ 1.0912410 sec
RG 4096
DW 16.650 usec
DE 7.00 usec
TE 296.9 K
D1 1.00000000 sec
d11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 13C
P1 8.60 usec
PL1 1.00 dB
SFO1 125.7703640 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 1.00 dB
PL12 17.50 dB
SFO2 500.1322150 MHz

F2 - Processing parameters
SI 32768
SF 125.7577923 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

[4-CP]



BRUKER

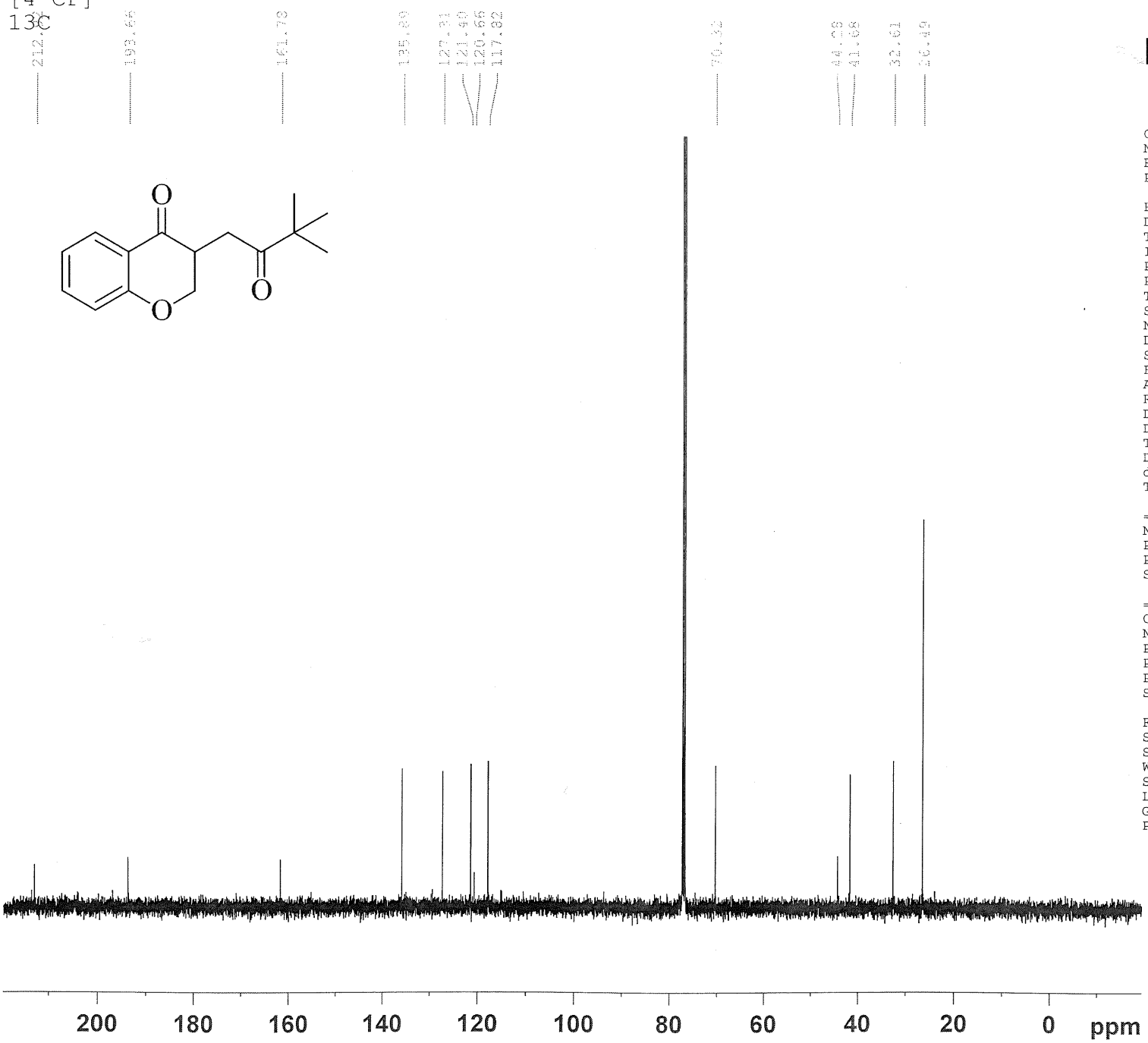
Current Data Parameters
NAME IndaPina-LiHMDS-I2-041411
EXPNO 3
PROCNO 1

F2 - Acquisition Parameters
Date_ 20110418
Time 11.58
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 4
DS 0
SWH 10000.000 Hz
FIDRES 0.305176 Hz
AQ 1.6385000 sec
RG 322.5
DW 50.000 usec
DE 6.50 usec
TE 296.6 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 14.00 usec
PL1 1.00 dB
SFO1 500.1345012 MHz

F2 - Processing parameters
SI 32768
SF 500.1300232 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

[4-CP]
13C



Current Data Parameters
NAME IndaPina-LiHMDS-I2-041411
EXPNO 4
PROCNO 1

F2 - Acquisition Parameters
Date_ 20110418
Time 12.02
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zgdc30
TD 65536
SOLVENT CDCl3
NS 225
DS 4
SWH 30030.029 Hz
FIDRES 0.458222 Hz
AQ 1.0912410 sec
RG 4597.6
DW 16.650 usec
DE 7.00 usec
TE 297.1 K
D1 1.00000000 sec
d11 0.03000000 sec
TD0 1

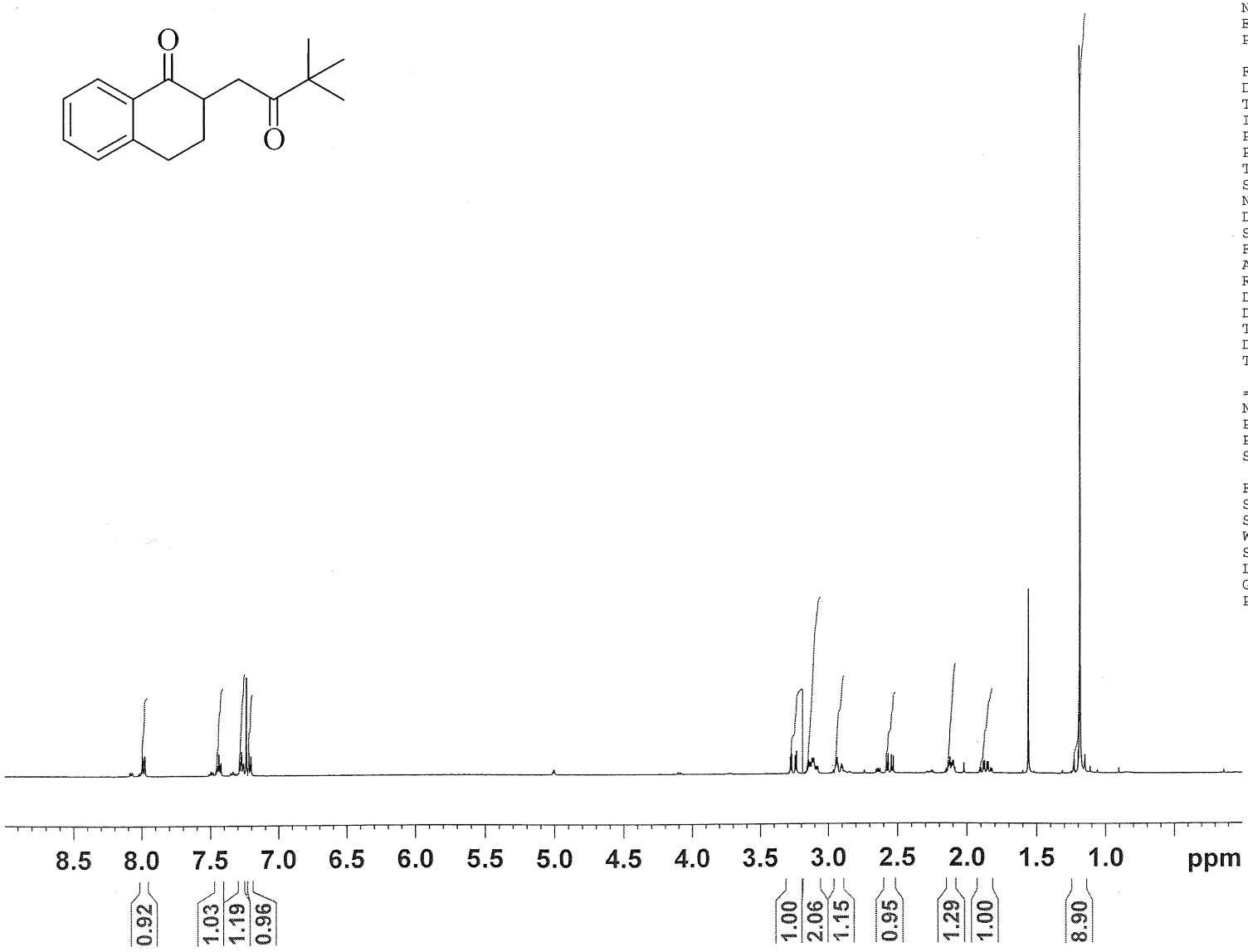
======== CHANNEL f1 ========
NUC1 13C
P1 8.60 usec
PL1 1.00 dB
SFO1 125.7703640 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 1.00 dB
PL12 17.50 dB
SFO2 500.1322150 MHz

F2 - Processing parameters
SI 32768
SF 125.7577919 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

CF-Fr15-16

7.9967 7.9810 7.4557 7.4531 7.4408 7.4383 7.4259 7.4233 7.2894 7.2745 7.2596 7.2401 7.2218 7.2064 3.2818 3.2719 3.2458 3.2360 3.1587 3.1493 3.1455 3.1397 3.1361 3.1319 3.1226 3.1180 3.1137 3.1089 3.0996 3.0897 3.0807 2.9518 2.9457 2.9391 2.9319 2.9122 2.9056 2.8984 2.5820 2.5685 2.5461 2.5326 2.1389 2.1299 2.1242 2.1134 2.1073 2.1041 2.0985 2.0952 2.0895 2.0237 1.8846 1.8761 1.8584 1.8499 1.8322 1.5617 1.2372 1.2303 1.2009 1.1899 1.1535 1.1499 1.1139 0.9055



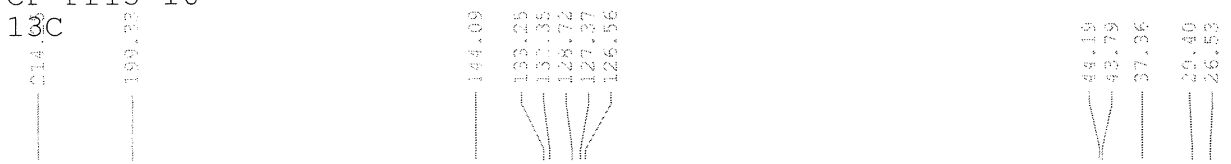
Current Data Parameters
NAME aTetPina-LiHMDS-I2-042711
EXPNO 3
PROCNO 1

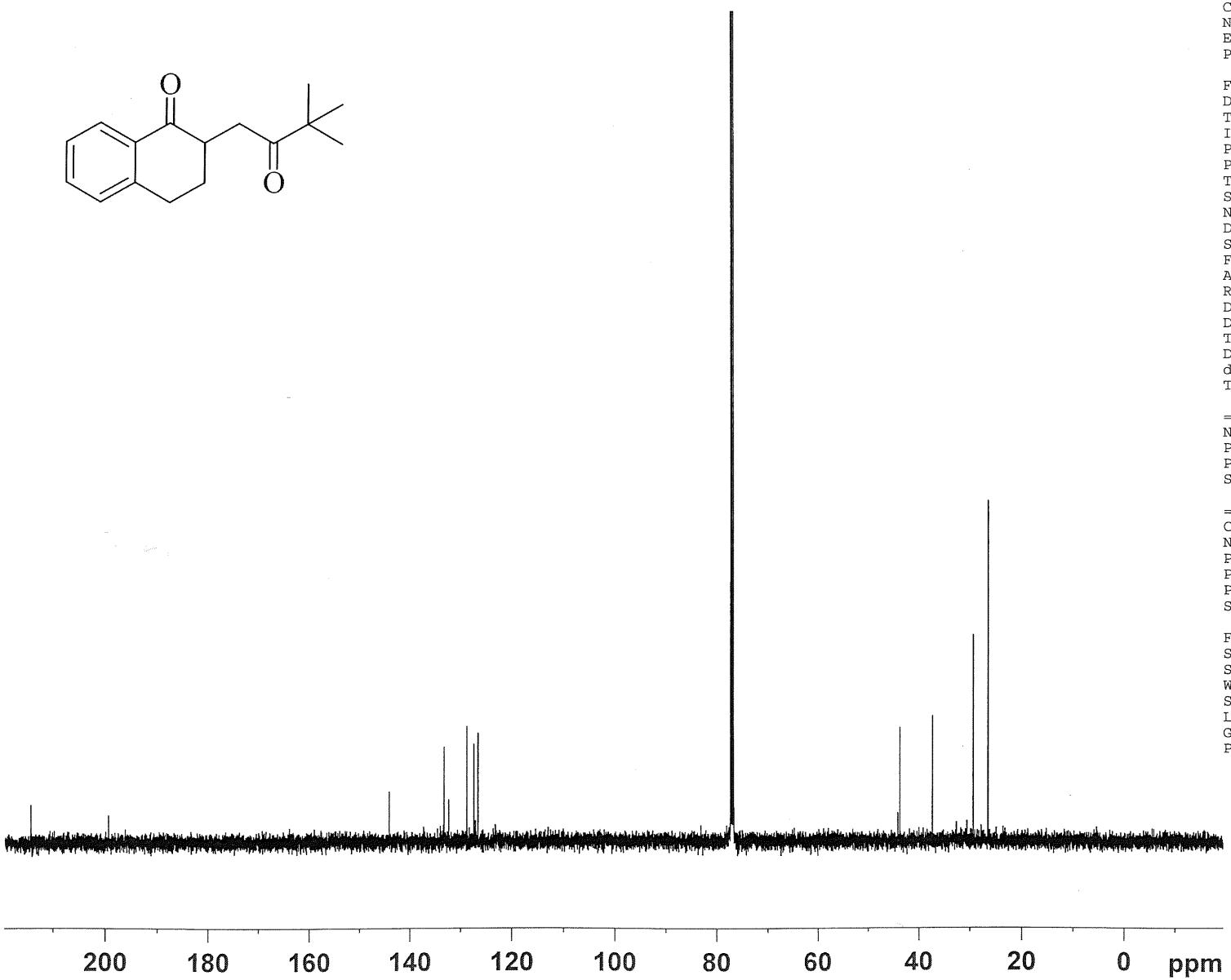
F2 - Acquisition Parameters
Date_ 20110427
Time 16.30
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 4
DS 0
SWH 10000.000 Hz
FIDRES 0.305176 Hz
AQ 1.6385000 sec
RG 406.4
DW 50.000 usec
DE 6.50 usec
TE 296.1 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 14.00 usec
PL1 1.00 dB
SFO1 500.1345012 MHz

F2 - Processing parameters
SI 32768
SF 500.1300232 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

CF-Fr15-16
13C





Current Data Parameters
NAME aTetPina-LiHMDS-I2-042711
EXPNO 4
PROCNO 1

F2 - Acquisition Parameters
Date_ 20110427
Time 16.35
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zgdc30
TD 65536
SOLVENT CDCl3
NS 401
DS 4
SWH 30030.029 Hz
FIDRES 0.458222 Hz
AQ 1.0912410 sec
RG 4096
DW 16.650 usec
DE 7.00 usec
TE 296.5 K
D1 1.00000000 sec
d11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 13C
P1 8.60 usec
PL1 1.00 dB
SFO1 125.7703640 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 1.00 dB
PL12 17.50 dB
SFO2 500.1322150 MHz

F2 - Processing parameters
SI 32768
SF 125.7577924 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40