

**Asymmetric Organocatalytic Desymmetrization of 4,4-Disubstituted  
Cyclohexadienones at High-Pressure: A New Powerful Strategy for the  
Synthesis of Highly Congested Chiral Cyclohexenones**

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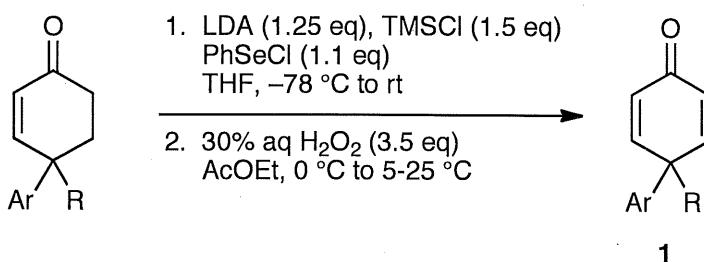
## General.

All reactions were performed in oven-dried glassware under a positive pressure of nitrogen or argon. All melting points were measured on a Yanagimoto MP-S3 micro-melting point apparatus and are uncorrected. The NMR spectra were recorded on a JEOL ECA-500 (500 MHz for <sup>1</sup>H NMR analysis and 125.8 MHz for <sup>13</sup>C NMR analysis) instrument in CDCl<sub>3</sub> unless otherwise stated and are reported in parts per million ( $\delta$ ) downfield from TMS as an internal standard. Mass spectral analyses were performed on a JEOL JMS-700/MStation mass spectrometer. The infrared spectra were measured with a JASCO FTIR-460plus Fourier Transform Infrared Spectrophotometer and are reported in wave-numbers (cm<sup>-1</sup>). Optical rotations were measured on a JASCO DIP-370 polarimeter. HPLC analyses were carried out using a Hitachi L-6200 HPLC system.

Thin-layer chromatography (TLC) was conducted using Merck Kieselgel 60F-254 plates (0.25 mm). Kanto Chemicals silica gel 60N (spherical, neutral 63–210  $\mu$ m) and Merck alumina (90 active, neutral 70–230  $\mu$ m) were used for column chromatography.

Catalysts **A** and **B** are known and prepared following the literature procedure.<sup>1</sup> The starting cyclohexenones were prepared from  $\alpha,\alpha'$ -disubstituted acetaldehydes and methyl vinyl ketone via Robinson-type annulation.<sup>2</sup> 4-Methyl-4-trichloromethylcyclohexadienone **1g** was prepared from *p*-cresol following the literature procedure.<sup>3</sup> Cyclohexenone (*R*)-**8** was prepared following the literature procedure,<sup>2</sup> and used after recrystallization from hexane. The ee of this compound was determined by chiral HPLC analysis (Chiralpak AD column, 0.46  $\times$  25 cm, hexane/*i*-PrOH = 99 : 1, 0.3 cm<sup>3</sup>/min): *R*<sub>t</sub> (*S*) = 25.4 min; *R*<sub>t</sub> (*R*) = 29.2 min (our previous data<sup>2</sup> for the assignment of (*S*)- and (*R*)-isomers should be corrected).

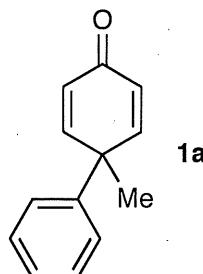
## General Procedure for the Synthesis of Cyclohexadienones (1).<sup>4</sup>



To a solution of *i*-Pr<sub>2</sub>NH (6.25 mmol) in THF (6 mL) was slowly added *n*-BuLi (6.25 mmol) at -78 °C under Ar. The mixture was stirred for 20 min before a solution of cyclohexenone (5.0 mmol) in THF (6 mL) was introduced. After stirring at 0 °C for 30 min, TMSCl (7.5 mmol) was added in one portion and the resulting mixture stirred at rt for 1 h before PhSeCl (5.5 mmol) in THF (5.5 mL) was introduced with stirring at rt. After stirring for 45 min, the mixture was quenched by addition of 10% aq HCl (7.5 mL) followed by stirring for another 1.5 h. The mixture was then extracted with Et<sub>2</sub>O, and the combined extracts were washed with brine, dried (Na<sub>2</sub>SO<sub>4</sub>), and concd. Purification by column chromatography on silica gel (hexane/AcOEt) gave the crude product as a pale yellow oil.

To a solution of this crude product (3.3 mmol) in AcOEt (45 mL) was added dropwise 30% aq H<sub>2</sub>O<sub>2</sub> (3.5 eq) at 0 °C, and the mixture was stirred at 5–25 °C for 60 min. The reaction was quenched by addition of sat. aq NaHCO<sub>3</sub> (22 mL) and sat. aq Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub>, and extracted with AcOEt. The combined extracts were dried (Na<sub>2</sub>SO<sub>4</sub>) and concd. Purification by column chromatography on alumina (eluted with hexane/AcOEt) and recrystallization from hexane gave the cyclohexadienone **1**.<sup>5</sup>

**4-Methyl-4-phenylcyclohexadienone (1a).**



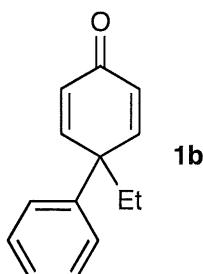
67% yield; colorless solid, mp 53–55 °C (lit.<sup>6</sup> 53–55 °C); R<sub>f</sub> 0.36 (hexane / AcOEt = 5 : 1).

FTIR (KBr) ν 1682, 1666, 1630, 1607, 1450, 1403, 1393, 1364 cm<sup>-1</sup>.

<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>) δ 1.69 (3H, s), 6.28 (2H, d, J = 10.0 Hz), 6.92 (2H, d, J = 10.0 Hz), 7.26–7.36 (5H, m).

<sup>13</sup>C NMR (125.8 MHz, CDCl<sub>3</sub>) δ 23.80, 44.95, 126.28 (×2), 126.90 (×2), 127.53, 128.92 (×2), 139.80, 155.37 (×2), 185.85.

**4-Ethyl-4-phenylcyclohexadienone (1b).**



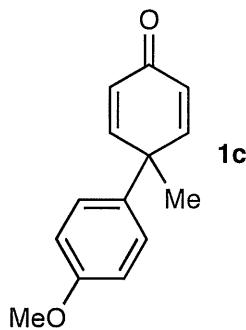
50% yield; colorless solid, mp 43–45 °C; R<sub>f</sub> 0.27 (hexane / AcOEt = 5 : 1).

FTIR (KBr) ν 1665, 1626, 1595, 1489, 1456, 1444, 1398 cm<sup>-1</sup>.

<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>) δ 0.89 (3H, t, J = 7.5 Hz), 2.16 (2H, q, J = 7.5 Hz), 6.36 (2H, d, J = 10.5 Hz), 6.87 (2H, d, J = 10.5 Hz), 7.24–7.35 (5H, m).

<sup>13</sup>C NMR (125.8 MHz, CDCl<sub>3</sub>) δ 8.89, 30.08, 49.44, 126.50 (×2), 127.41, 128.71 (×2), 128.88 (×2), 139.93, 154.10 (×2), 186.13.

**4-Methyl-4-(4-methoxyphenyl)cyclohexadienone (1c).**



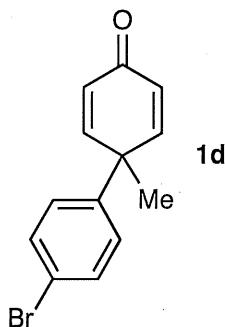
35% yield; colorless solid, mp 55–57 °C;  $R_f$  0.16 (hexane / AcOEt = 5 : 1).

FTIR (KBr)  $\nu$  1666, 1624, 1607, 1509, 1458, 1401 cm<sup>-1</sup>.

<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>) δ 1.65 (3H, s), 3.78 (3H, s), 6.24 (2H, d,  $J$  = 10.0 Hz), 6.85–6.87 (2H, m), 6.88 (2H, d,  $J$  = 10.0 Hz), 7.19–7.21 (2H, m).

<sup>13</sup>C NMR (125.8 MHz, CDCl<sub>3</sub>) δ 23.83, 44.32, 55.22, 114.22 ( $\times$ 2), 126.57 ( $\times$ 2), 127.38 ( $\times$ 2), 131.46, 155.66 ( $\times$ 2), 158.86, 185.85.

#### 4-Methyl-4-(4-bromophenyl)cyclohexadienone (1d).



45% yield; colorless solid, mp 59–60 °C;  $R_f$  0.19 (hexane / AcOEt = 5 : 1).

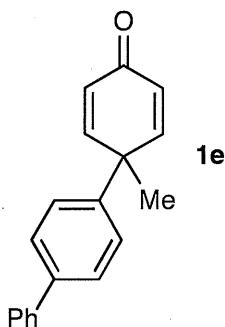
FTIR (KBr)  $\nu$  1661, 1619, 1591, 1508, 1448, 1391 cm<sup>-1</sup>.

<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>) δ 1.67 (3H, s), 6.27 (2H, d,  $J$  = 10.0 Hz), 6.86 (2H, d,  $J$  = 10.0 Hz), 7.15–7.17 (2H, m), 7.46 (2H, d,  $J$  = 8.5 Hz).

<sup>13</sup>C NMR (125.8 MHz, CDCl<sub>3</sub>) δ 23.82, 44.56, 121.66, 127.21 ( $\times$ 2), 128.11 ( $\times$ 2), 132.02 ( $\times$ 2), 139.02, 154.58 ( $\times$ 2), 185.52.

HRMS Calcd for C<sub>13</sub>H<sub>11</sub>BrO + H 263.0072, found 263.0077.

#### 4-Methyl-4-(4,4'-biphenyl)cyclohexadienone (1e).



15% yield; colorless solid, mp 111–113 °C;  $R_f$  0.20 (hexane / AcOEt = 5 : 1).

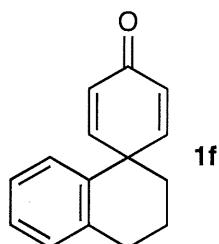
FTIR (KBr)  $\nu$  1665, 1622, 1597, 1484, 1448, 1398  $\text{cm}^{-1}$ .

$^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  1.74 (3H, s), 6.32 (2H, d,  $J = 10.0$  Hz), 6.96 (2H, d,  $J = 10.0$  Hz), 7.35–7.39 (3H, m), 7.45 (2H, t,  $J = 7.5$  Hz), 7.57 (4H, dd,  $J = 7.5, 5.5$  Hz).

$^{13}\text{C}$  NMR (125.8 MHz,  $\text{CDCl}_3$ )  $\delta$  23.90, 44.79, 126.77 ( $\times 2$ ), 127.00 ( $\times 2$ ), 127.06 ( $\times 2$ ), 127.50, 127.65 ( $\times 2$ ), 128.80 ( $\times 2$ ), 138.81, 140.25, 140.56, 155.25 ( $\times 2$ ), 185.85.

HRMS Calcd for  $\text{C}_{19}\text{H}_{16}\text{O} + \text{H}$  261.1279, found 261.1282.

### 3',4'-Dihydro-2'H-spiro[cyclohexa[2,5]diene-1,1'-naphthalen]-4-one.



86% yield; colorless solid; mp 150–151 °C (lit.<sup>7</sup> 144–146 °C);  $R_f$  0.26 (hexane / AcOEt = 5 : 1).

FTIR (KBr)  $\nu$  1662, 1621, 1487, 1445, 1400  $\text{cm}^{-1}$ .

$^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  1.97–2.02 (4H, m), 2.91–2.93 (2H, m), 6.28 (2H, d,  $J = 10.0$  Hz), 6.95 (1H, d,  $J = 8.0$  Hz), 7.02 (2H, d,  $J = 10.0$  Hz), 7.07–7.11 (1H, m), 7.16–7.17 (2H, m).

$^{13}\text{C}$  NMR (125.8 MHz,  $\text{CDCl}_3$ )  $\delta$  19.23, 29.58, 34.21, 44.74, 126.34, 126.79 ( $\times 2$ ), 127.49, 128.79, 130.23, 133.39, 136.46, 155.31 ( $\times 2$ ), 186.16.

### References

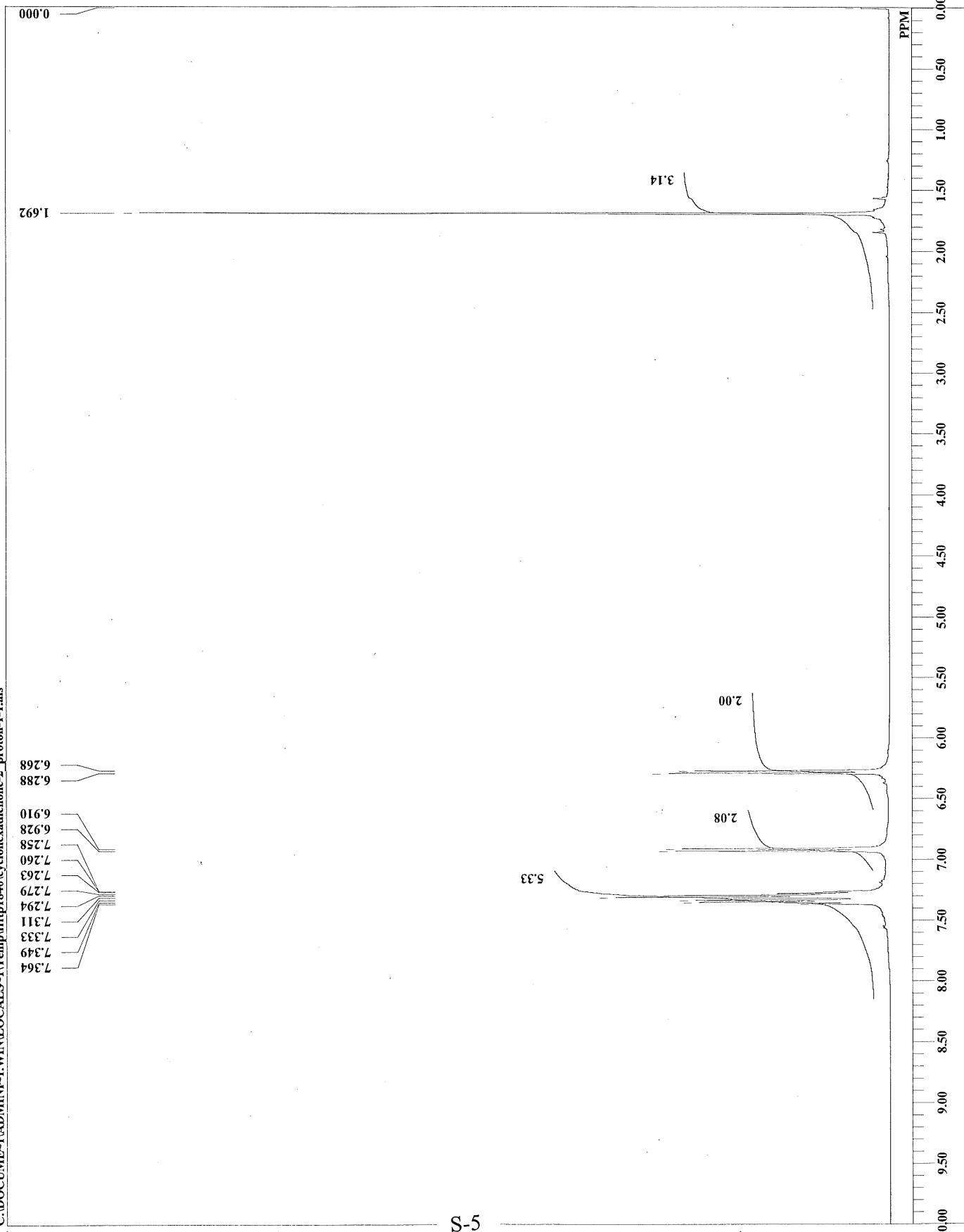
- (1) M. Moritaka, N. Miyamae, K. Nakano, Y. Ichikawa and H. Kotsuki, *Synlett*, 2012, **23**, 2554–2558.
- (2) Y. Inokoishi, N. Sasakura, K. Nakano, Y. Ichikawa and H. Kotsuki, *Org. Lett.*, 2010, **12**, 1616–1619.
- (3) (a) Z. Wang, *Comprehensive Organic Name Reactions and Reagents*, John Wiley & Sons: New York, 2010; Vol. 3, pp 3178–3182; (b) M. S. Newman and A. G. Pinkus, *J. Org. Chem.*, 1954, **19**, 978–984.
- (4) (a) H. Plieninger and W. Gramlich, *Chem. Ber.*, 1978, **111**, 1944–1957; (b) D. Crich, Q. Yao and G. F. Filzen, *J. Am. Chem. Soc.*, 1995, **117**, 11455–11470.
- (5) Yields were not optimized.
- (6) H. E. Zimmerman and G. Jones, II, *J. Am. Chem. Soc.*, 1970, **92**, 2753–2761.
- (7) S. Rousseaux, J. García-Fortanet, M. A. D. A. Sanchez and S. L. Buchwald, *J. Am. Chem. Soc.*, 2011, **133**, 9282–9285.

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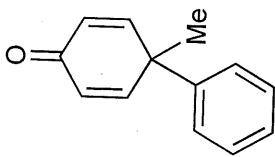
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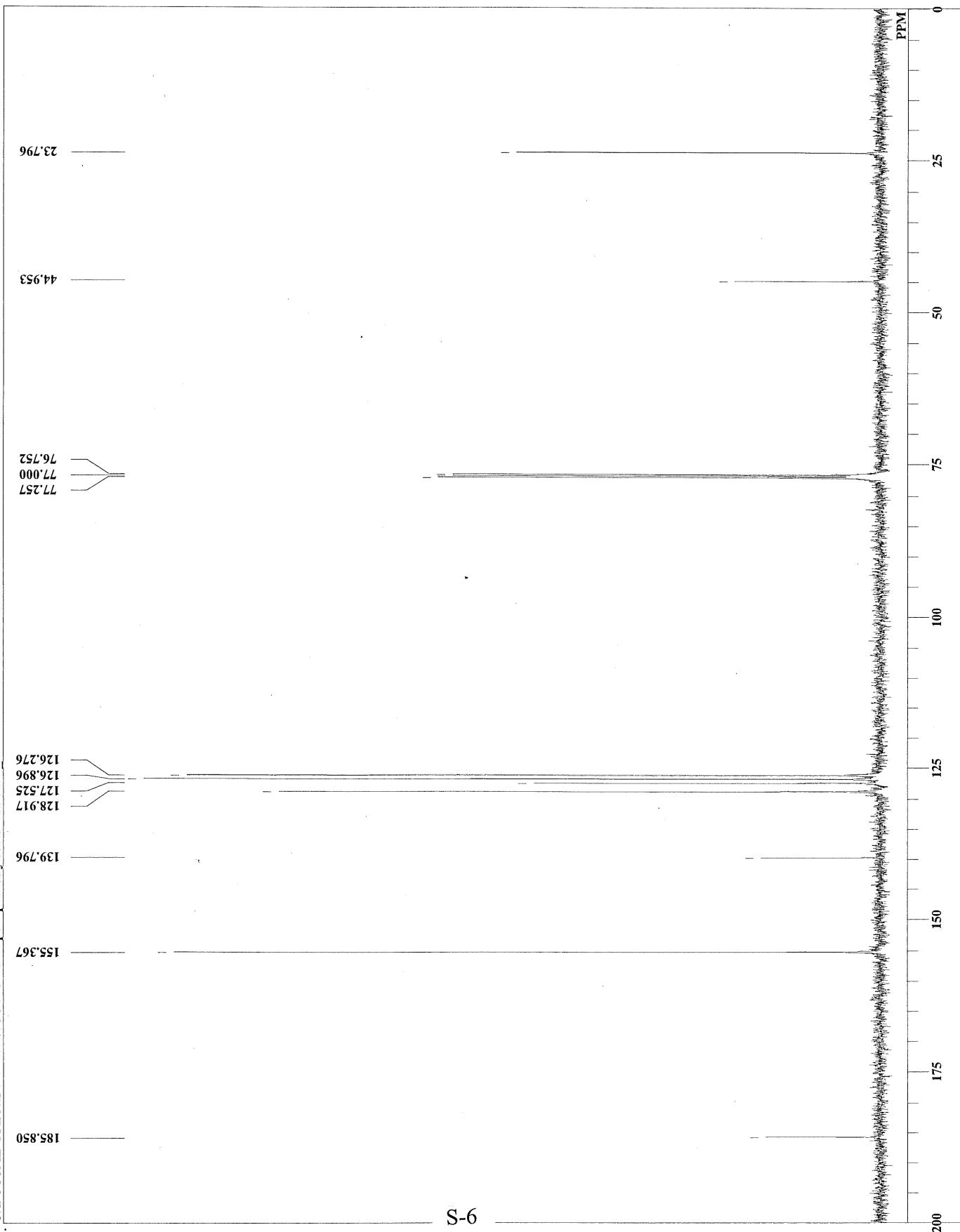
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**1a**  
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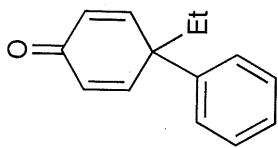
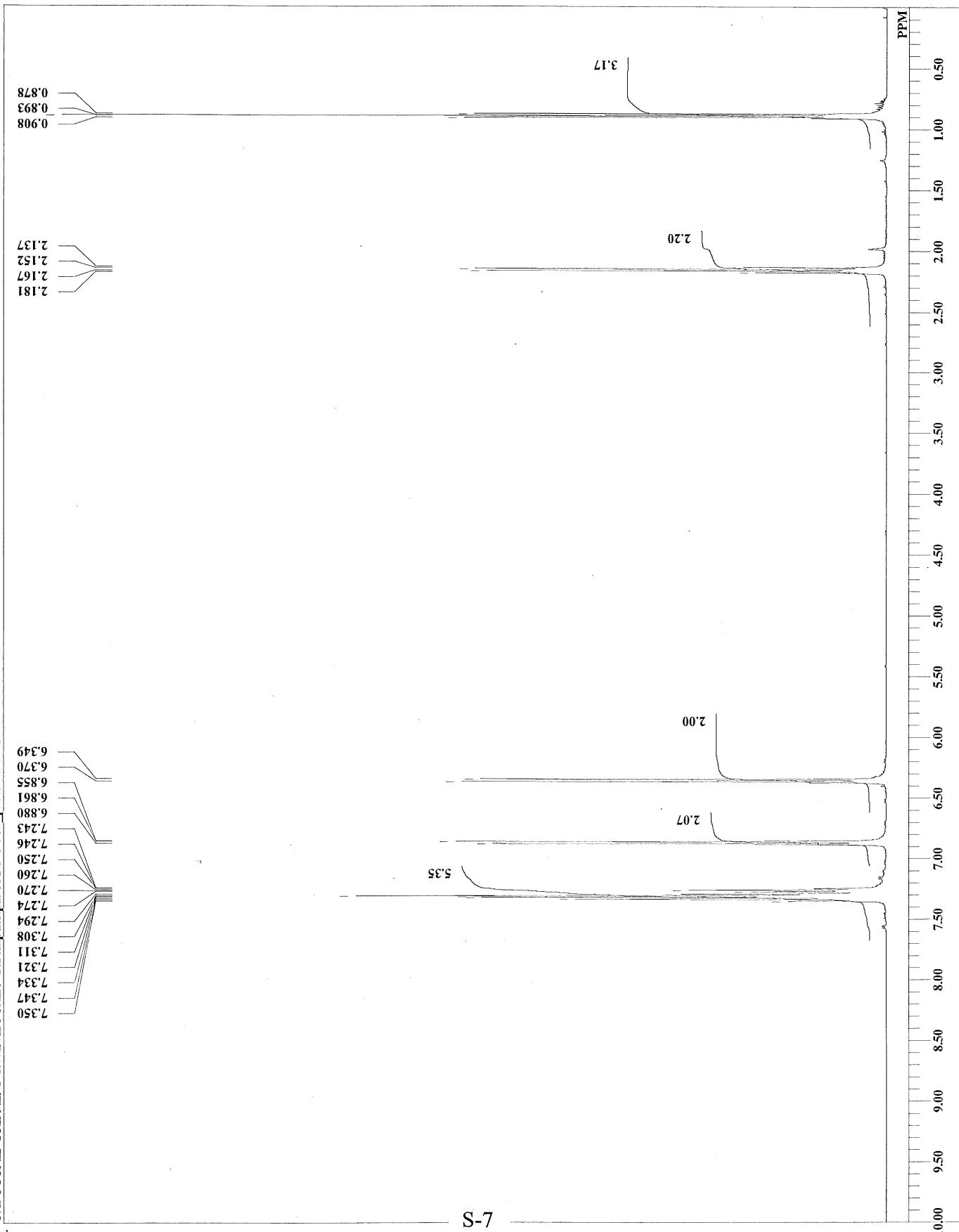


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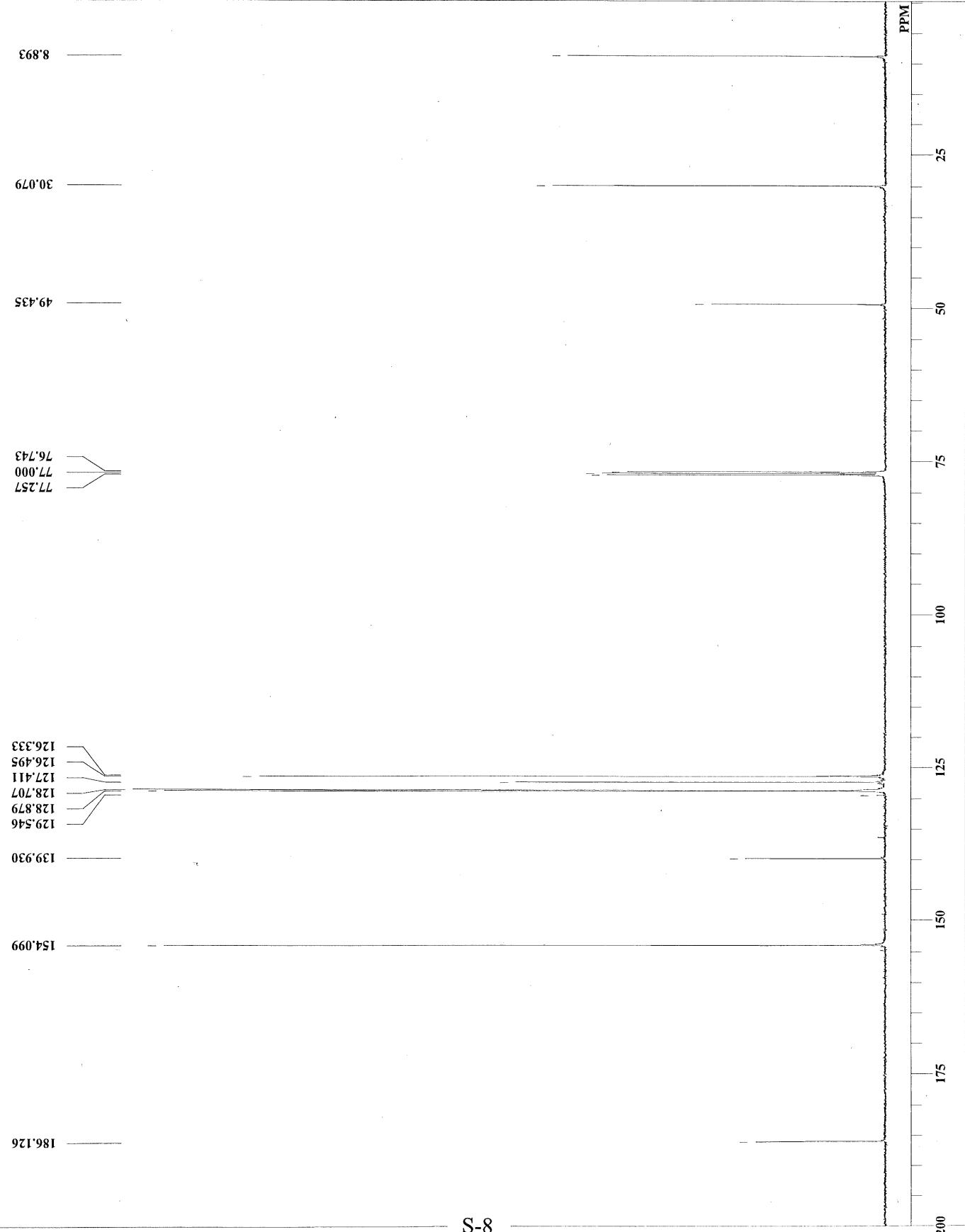


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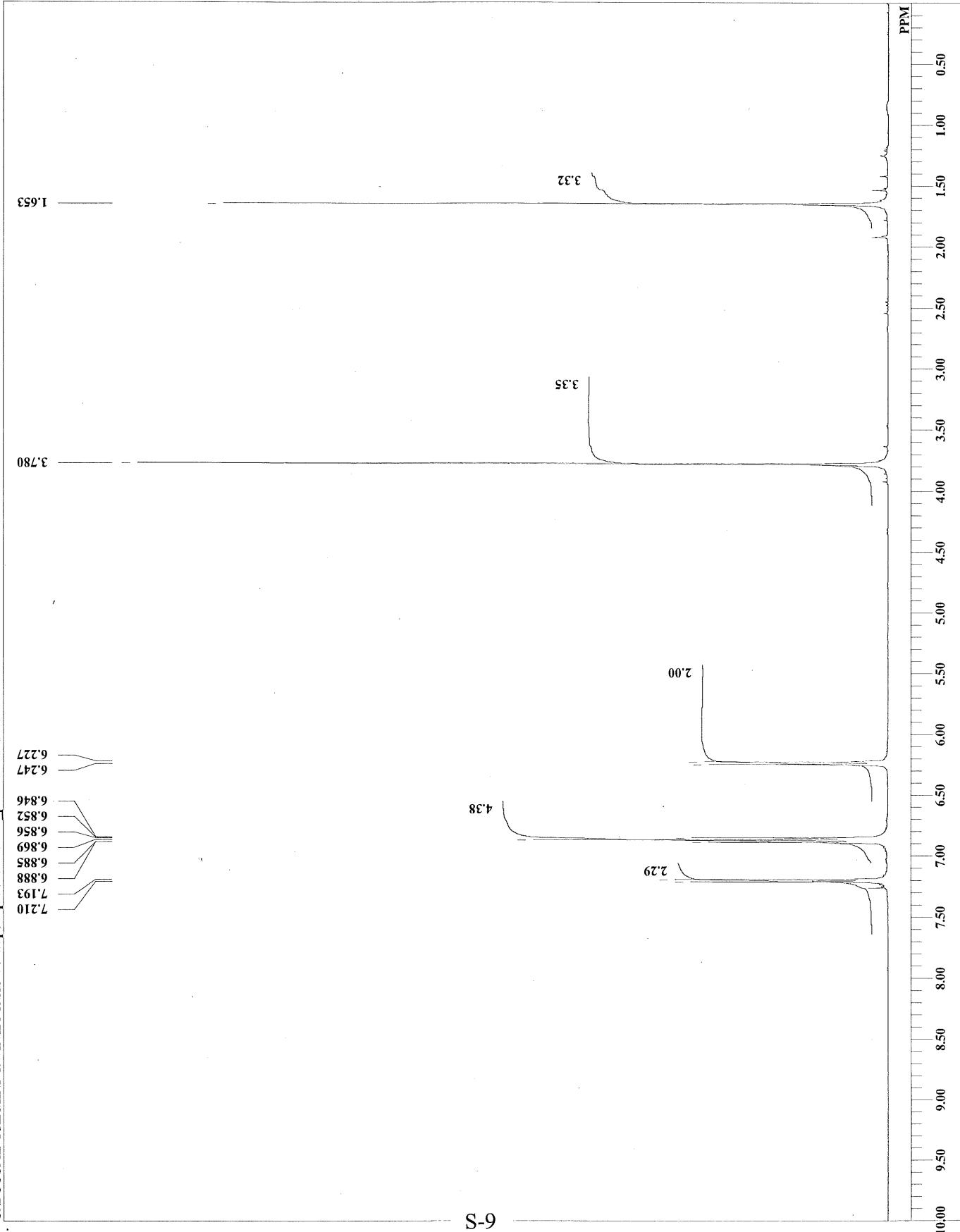
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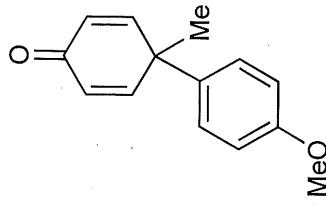
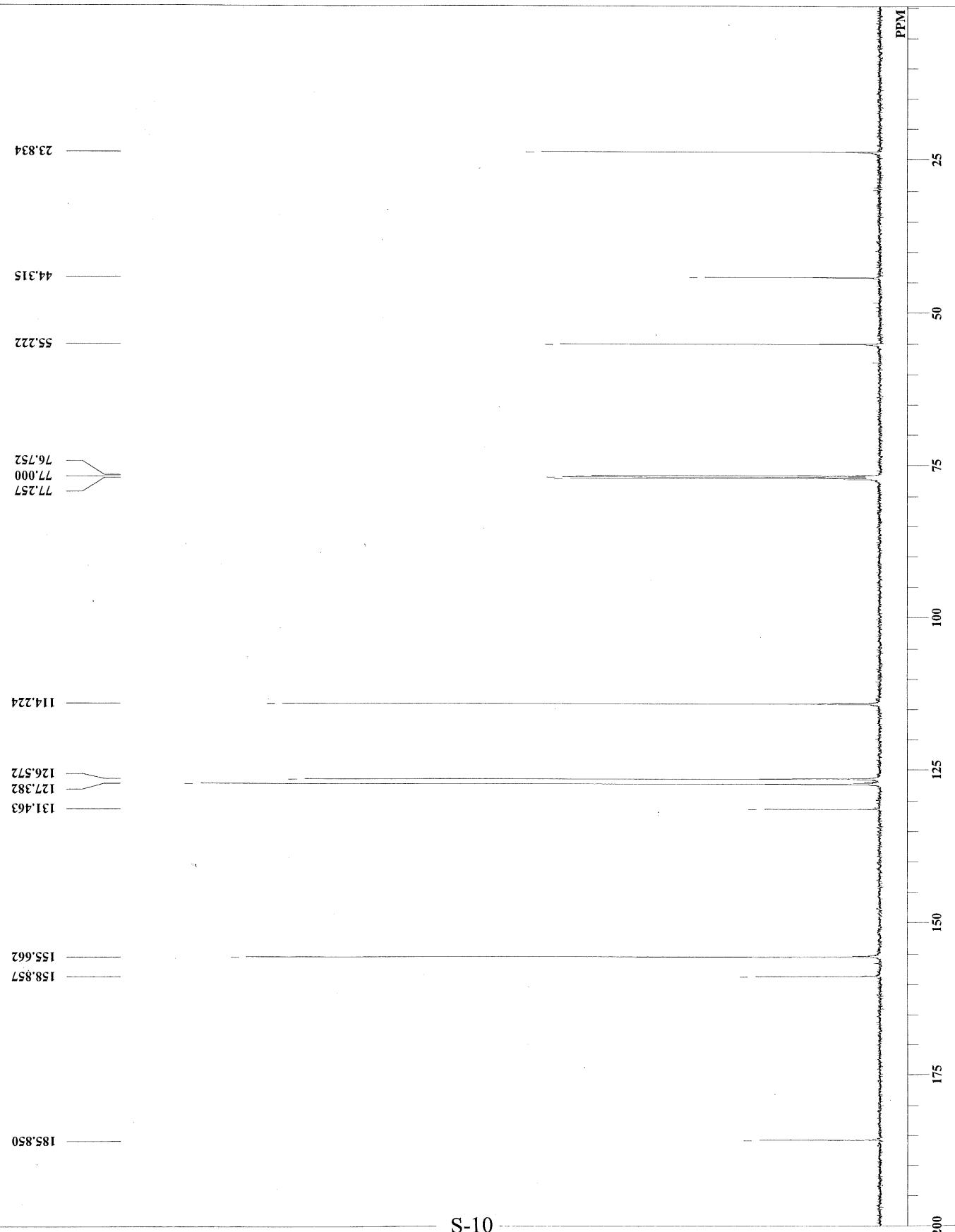


**1b**  
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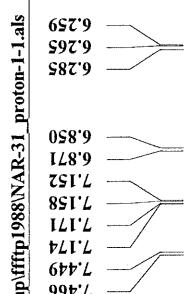
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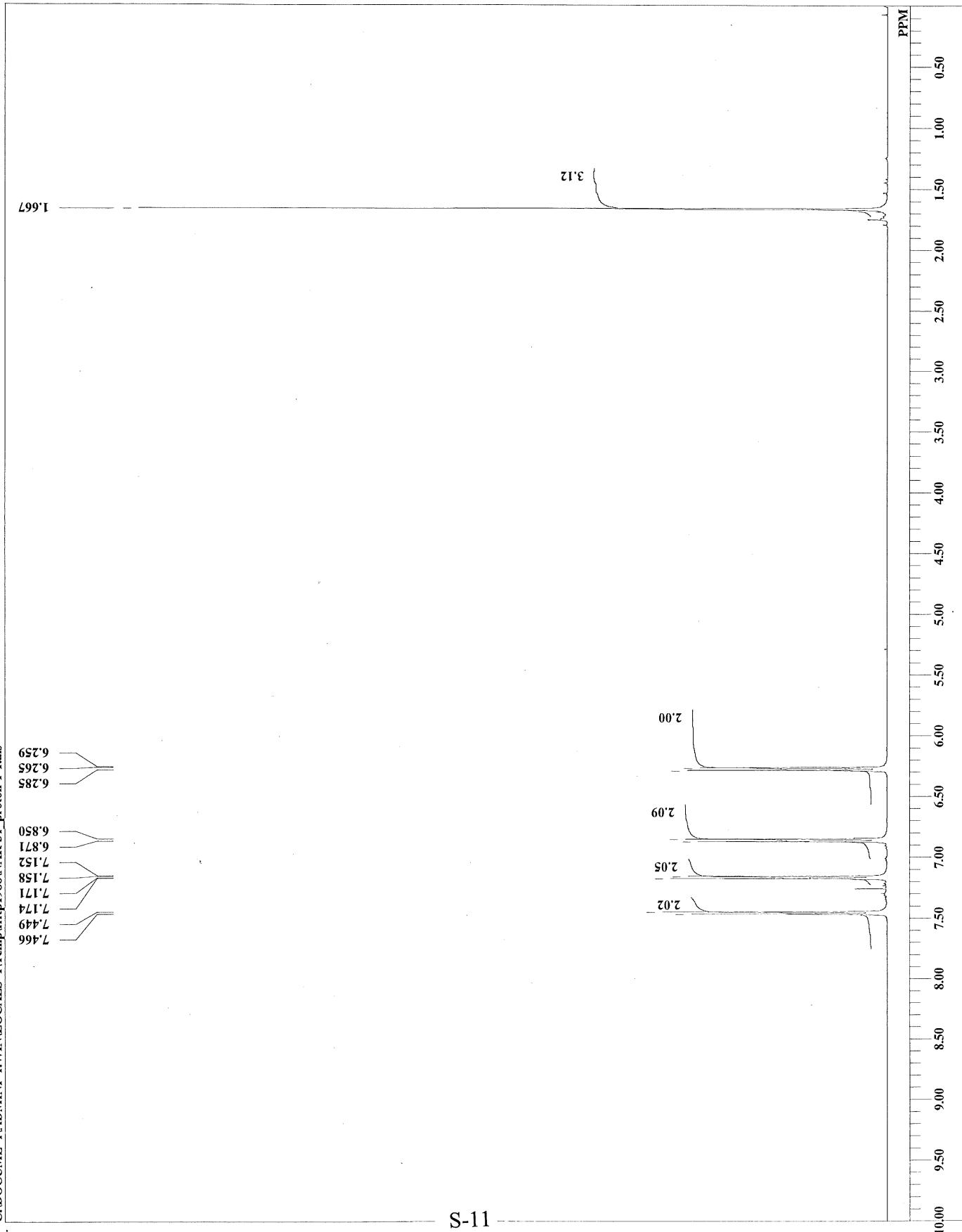
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single\_pulse



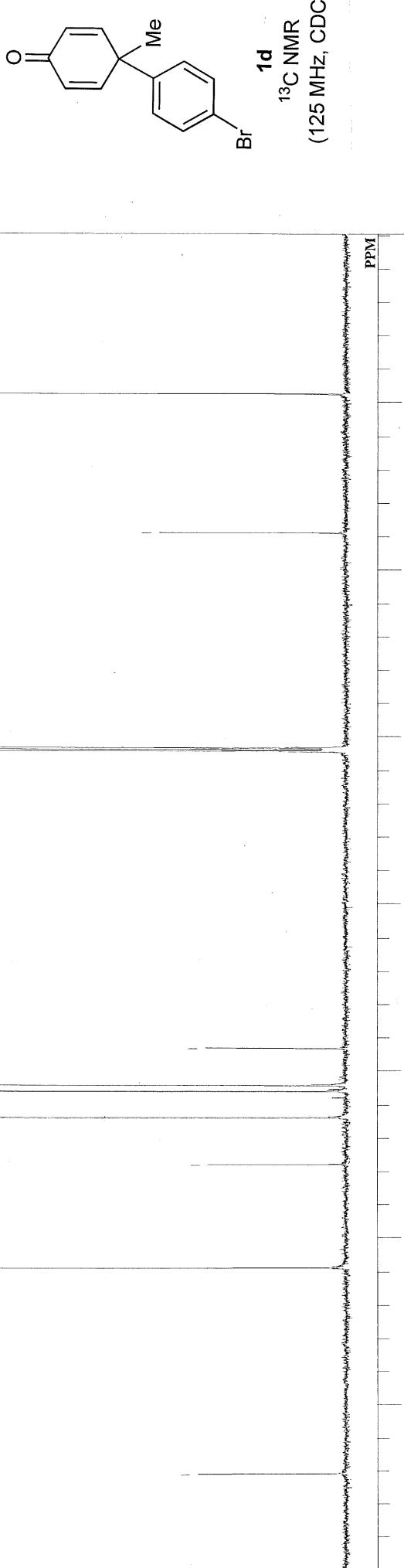
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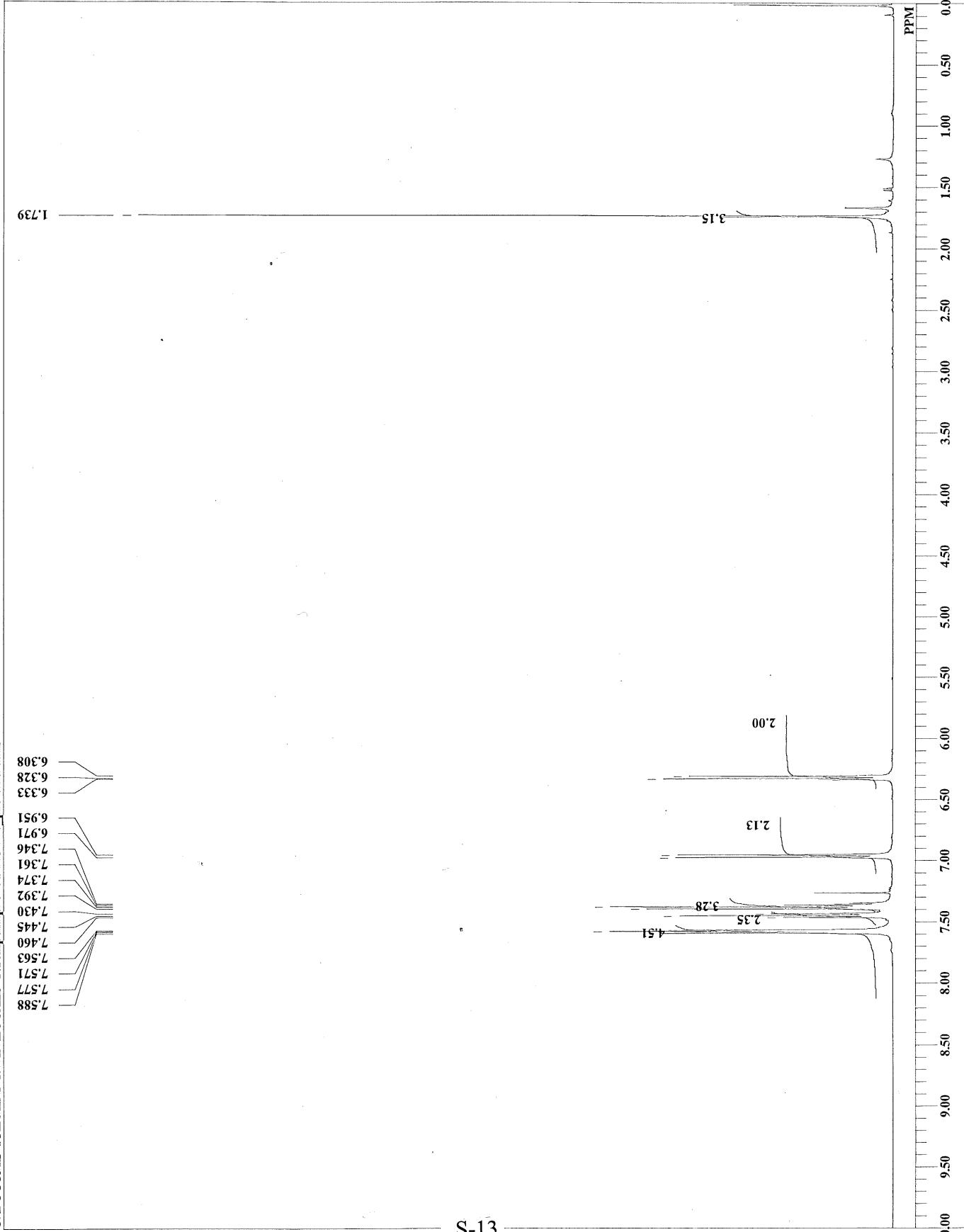
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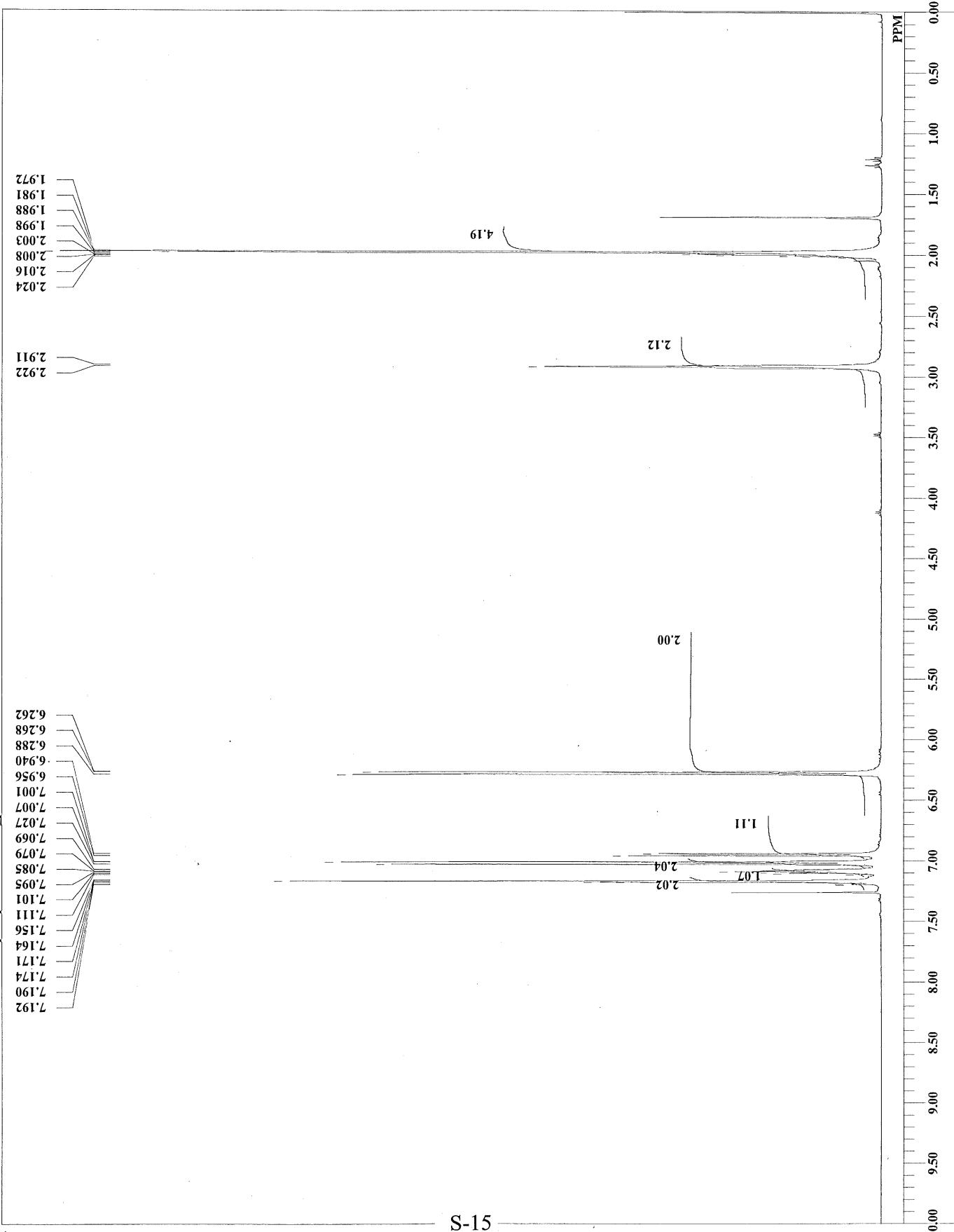
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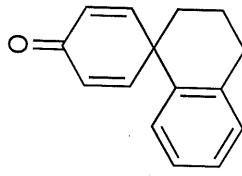
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NAO-237\_Carbon-1-1.als  
single pulse decoupled gated NOE  
13C  
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26224  
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CTEMP  
SLVNT  
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77.00 ppm  
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RGAIN  
23.2 °c  
PPM

DFILE  
COMNT  
DATIM  
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POINT  
FREQU  
SCANS  
ACQTM  
PD  
PWI  
IRNUC  
CTEMP  
SLVNT  
EXREF  
BF  
RGAIN



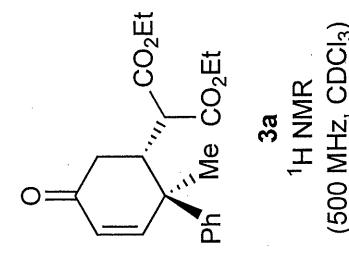
**1f**  
<sup>13</sup>C NMR  
(125 MHz, CDCl<sub>3</sub>)

C:\DOCUMENTS\ADMIN\1.WIN\LOCALS~1\Temp\ffffp1840\MYY-830-up\_proton-1-1.als

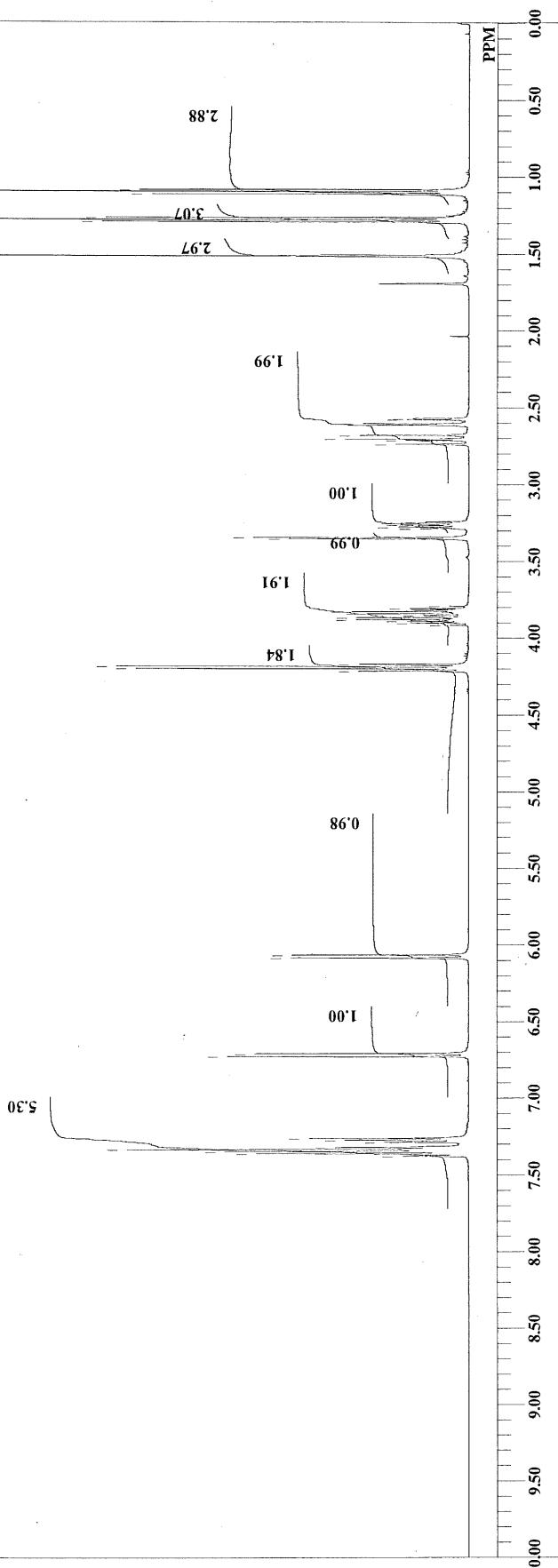
MYT-830-up\_proton-1-1.als

single\_pulse  
2012-07-28 15:55:23  
H

DFILE COMNT DATIM OBNUC EXMOD protonJXP  
OBFRQ 500.16 MHz  
OBSET 2.41 kHz  
OBFIN 6.01 Hz  
POINT 13120  
FREQU 750/7.51 Hz  
SCANS 8  
ACQTM 1.7459 sec  
PD 5.0000 sec  
PW1 4.68 usec  
IRNUC H  
CTEMP 22.4 c  
SLVNT CDCl<sub>3</sub>  
EXREF 7.26 ppm  
BF 0.12 Hz  
RGAIN 34



**3a**  
 $^1\text{H}$  NMR  
 (500 MHz,  $\text{CDCl}_3$ )

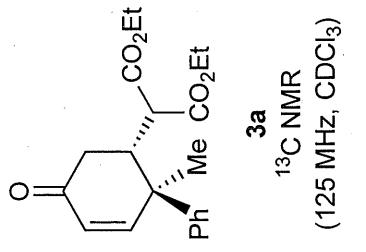


## single pulse decoupled gated NOE

C:\DOCUMENTS~1\ADMINI-1.WINLOCALS~1\Temp\fftp1988\MYY-830-up\_Carbon-1-1.als

MYY-830-up\_Carbon-1-1.als  
 single pulse decoupled gated NOE  
 2012-07-28 16:02:42  
 13C  
 carbon.jdp  
 125.77 MHz  
 OBFRQ  
 OBSET  
 OBFIN 4.21 Hz  
 POINT 26224  
 FREQU 31446.54 Hz  
 SCANS 512  
 ACQTM 0.8336 sec  
 PD 2.0000 sec  
 PW1 2.72 usec  
 IRNUC 1H  
 CTEMP 23.1 c  
 SLYNT CDCL<sub>3</sub>  
 EXREF 77.00 ppm  
 BF 0.112 Hz  
 RGAIN 60

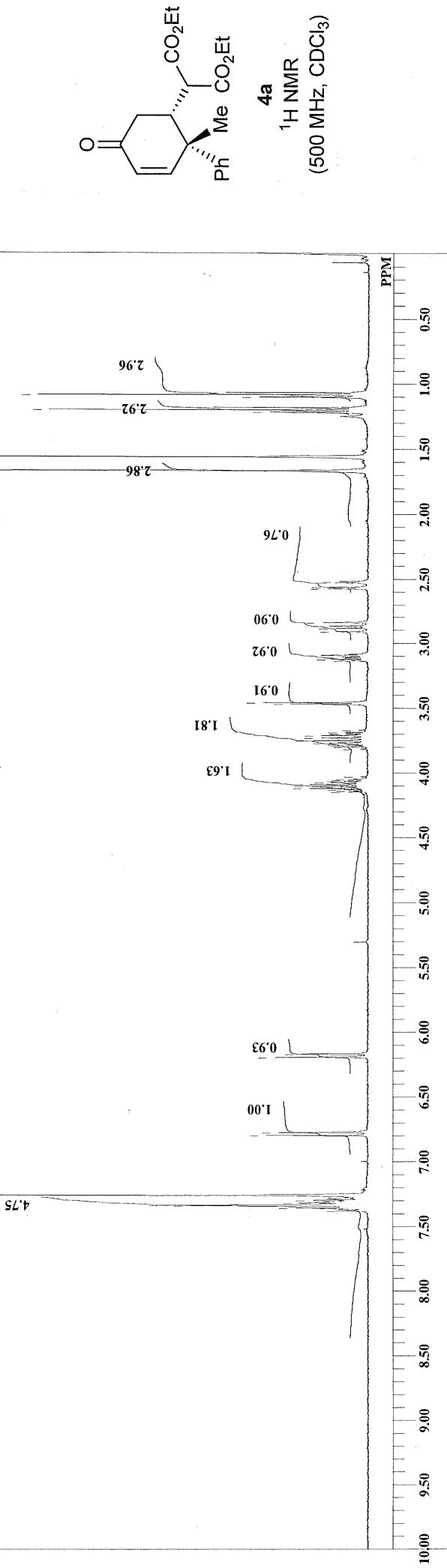
198.111  
 168.372  
 167.800  
 157.998  
 144.306  
 128.622  
 127.306  
 127.039  
 126.963  
 77.248  
 77.000  
 76.743  
 61.601  
 61.535  
 52.114  
 44.515  
 44.229  
 37.640  
 18.037  
 13.956  
 13.765

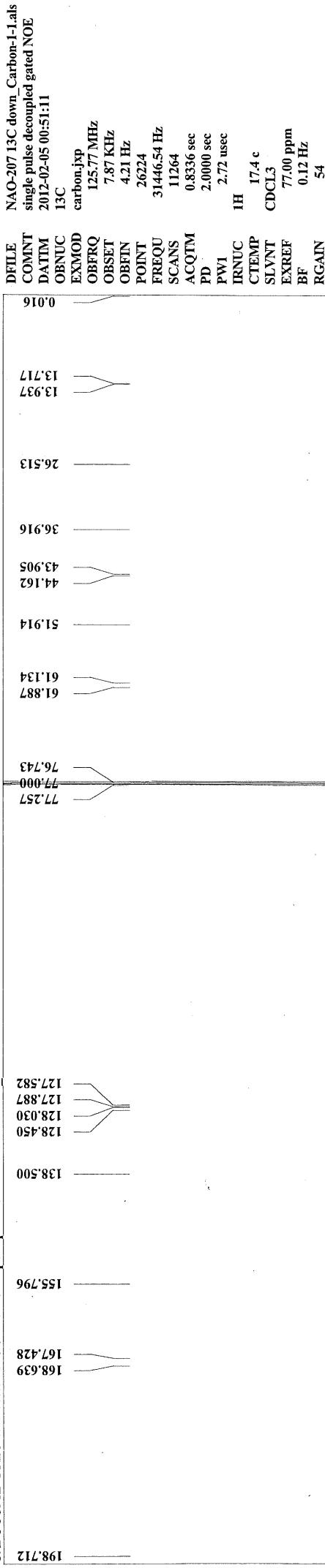


NAO-207 down.xls  
Sat Jan 21 21:45:47 2012

DFILE COMNT  
DATIM OBNUC  
EXMOD  
OBFRQ 399.65 MHz  
OFFSET 0.00 kHz  
OBFIN 134300.00 Hz  
POINT 8192  
FREQU 7993.60 Hz  
SCANS 16  
ACQTM 1.0248 sec  
PD 5.9752 sec  
PW1 4.45 usec  
IRNUC 1H  
CTEMP 18.6 c  
SLVNT CDCL<sub>3</sub>  
EXREF 7.26 ppm  
BF 0.12 Hz  
RGAIN 28

1.070  
1.087  
1.104  
1.1185  
1.1202  
1.1219  
1.1671  
1.2522  
2.535  
2.566  
2.579  
2.800  
2.844  
2.884  
2.911  
3.084  
3.094  
3.106  
3.111  
3.123  
3.133  
3.457  
3.469  
3.667  
3.684  
3.694  
3.704  
3.711  
3.721  
3.731  
3.750  
3.760  
3.770  
3.777  
3.787  
3.797  
3.814  
3.828  
4.028  
4.045  
4.055  
4.065  
4.072  
4.082  
4.092  
4.102  
4.109  
4.119  
4.128  
4.136  
4.146  
6.119  
6.193  
6.772  
6.796  
7.200  
7.227  
7.244  
7.301  
7.311  
7.316  
7.333  
7.341  
7.355  
7.372  
7.377



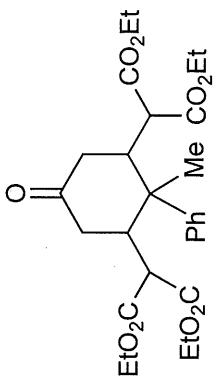


**4a**  
<sup>13</sup>C NMR  
(125 MHz, CDCl<sub>3</sub>)

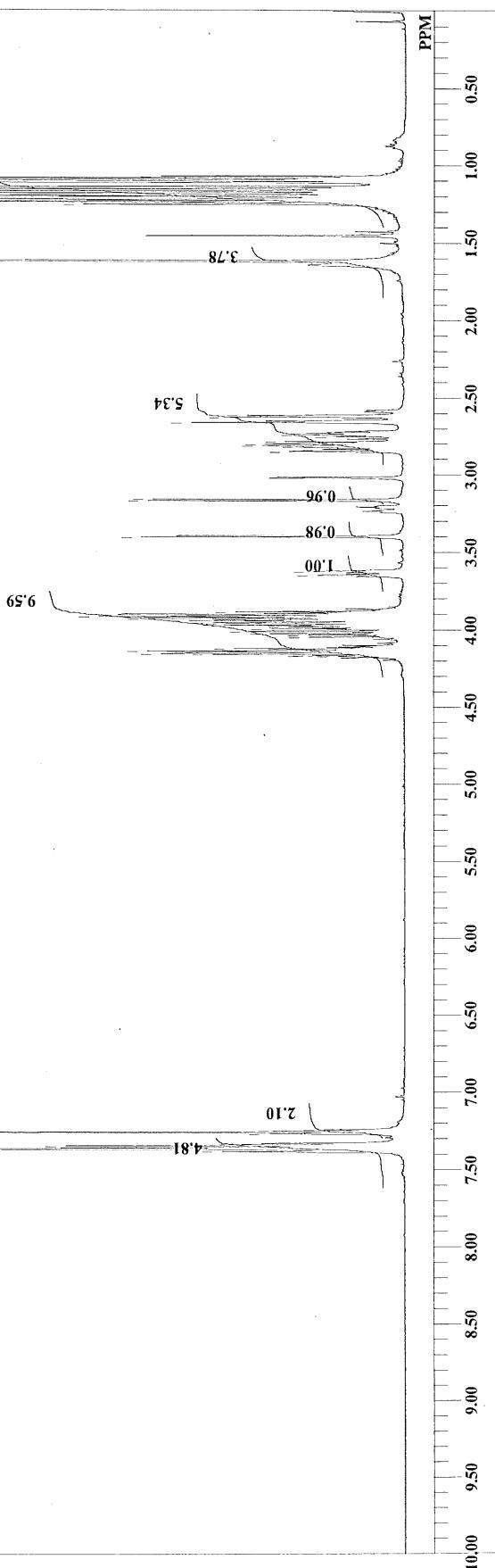
PPM  
200 175 150 125 100 75 50

## single\_pulse

C:\DOCUMENTE\~1\ADMINI~1.WIN\LOCAL.S~1\Temp\fffftp1988\NAO-200-bypro\_proton-1-1.als



**5**  
 $^1\text{H}$  NMR  
 (500 MHz, CDCl<sub>3</sub>)

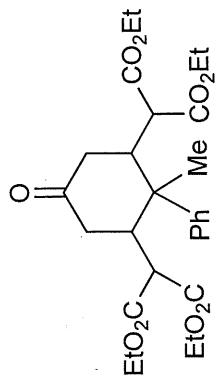


single pulse decoupled gated NOE

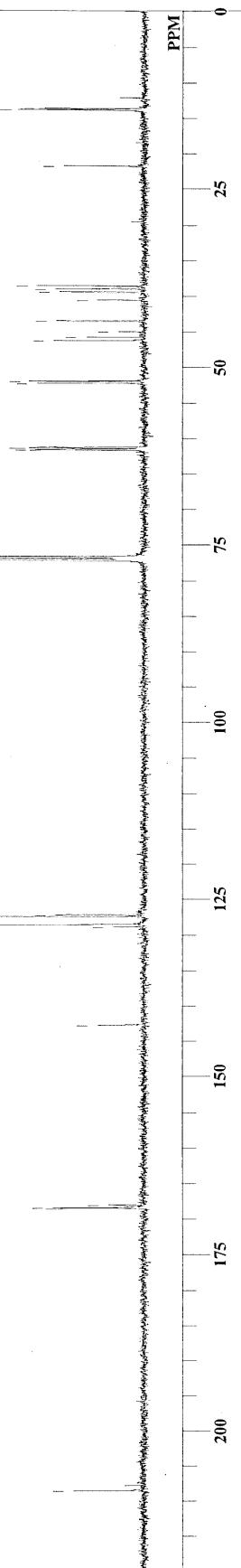
C:\DOCUME~1\ADMINI~1.WIN\LOCALS~1\Temp\ffffp1988\NAO-200-hypro\_Carbon-1-1.xls

208,409	168,553	168,487	168,449	168,391	168,086
77,248	76,743	76,754	76,768	76,773	77,000
61,754	61,678	61,681	61,696	61,701	61,717
61,477	61,595	61,596	61,597	61,598	61,599
52,362	52,363	52,364	52,365	52,366	52,367
46,307	45,821	45,822	45,823	45,824	45,825
46,308	46,309	46,310	46,311	46,312	46,313
40,663	39,566	39,567	39,568	39,569	39,570
38,689	38,690	38,691	38,692	38,693	38,694
13,762	13,763	13,764	13,765	13,766	13,767
13,784	13,785	13,786	13,787	13,788	13,789
13,794	13,795	13,796	13,797	13,798	13,799
13,799	13,800	13,801	13,802	13,803	13,804
13,822	13,823	13,824	13,825	13,826	13,827
13,849	13,850	13,851	13,852	13,853	13,854
13,865	13,866	13,867	13,868	13,869	13,870
13,874	13,875	13,876	13,877	13,878	13,879
127,468	128,593	128,594	128,595	128,596	128,597
127,229	127,230	127,231	127,232	127,233	127,234
142,685	142,686	142,687	142,688	142,689	142,690

NAO-200-hupro\_Carbon-1,1s  
 single pulse decoupled NOE  
 2012-10-22 21:40:45  
 13C  
 carbon.xp  
 125.77 MHz  
 OBTRQ  
 OBSET  
 OBFIN  
 POINT  
 FREQU  
 SCANS  
 ACQTM  
 PD  
 PW1  
 IRNUC  
 CTTEMP  
 SLVNT  
 EXREF  
 BF  
 RGAIN  
 19.9 c  
 CDCl<sub>3</sub>  
 77.00 ppm  
 0.12 Hz  
 60

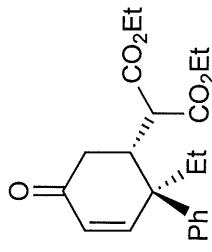


## 5 <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>)

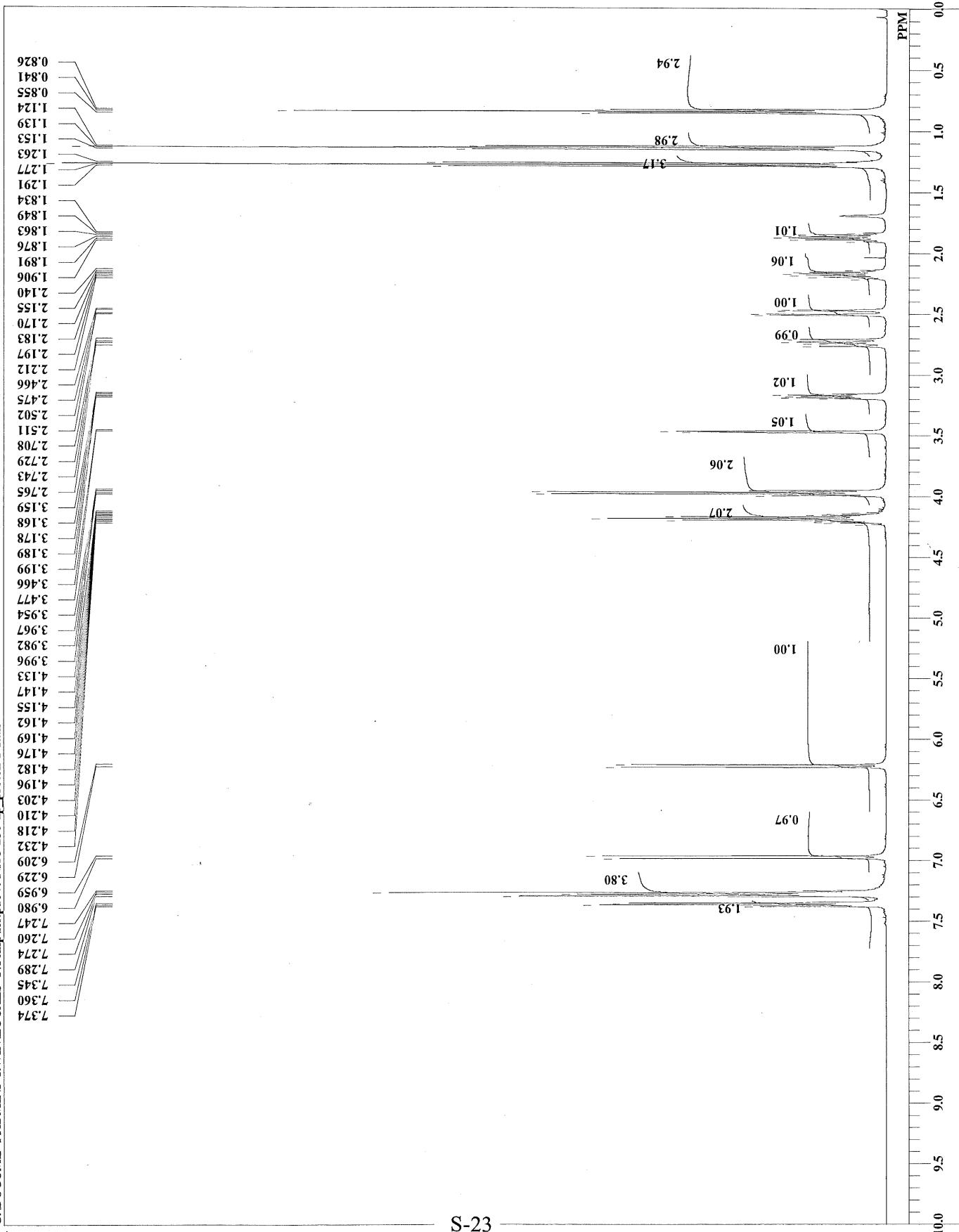


NAO-236-up\_Proton-1-1.als

Parameter	Value
DBFILE	
COMNT	
DATIM	2012-07-19 09:25:55
OBNUC	1H
EXMOD	PROTONJXP
OBFRQ	500.16 MHz
OBSET	2.41 kHz
OBFIN	6.01 Hz
POINT	13120
FREQU	7507.51 Hz
SCANS	8
ACQTM	1.7459 sec
PD	5.0000 sec
PWI	4.68 usec
IRNUC	1H
CTEMP	22.1 c
SLVNT	CDCl <sub>3</sub>
EXREF	12.51 ppm
BF	0.12 Hz
RGAIN	34



**3b**  
 $^1\text{H}$  NMR  
(500 MHz,  $\text{CDCl}_3$ )

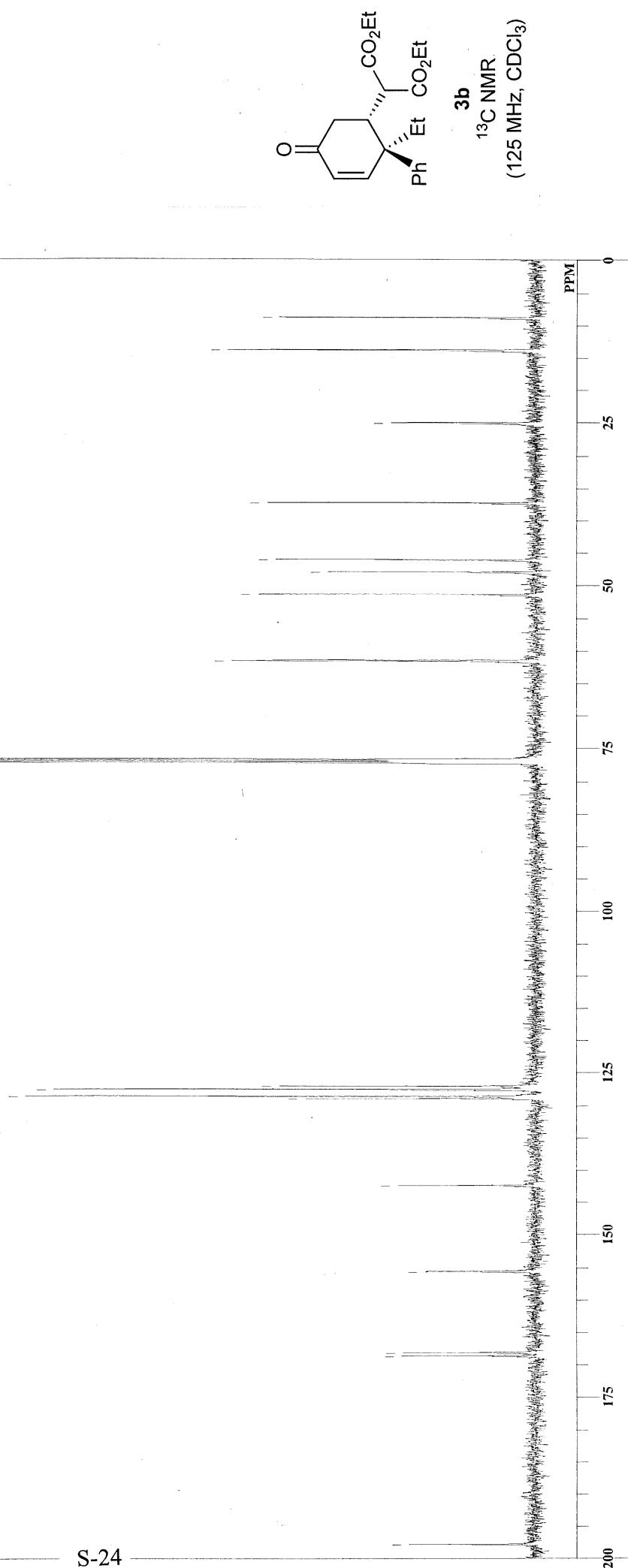


NAO-236-up\_Carbon-1-1.jdf  
single pulse decoupled gated NOE  
2012-07-19 09:29:27

13C carbon.jdp

DFILE COMNT  
DATIM OBNUC  
EXMOD OBFRQ  
OBSET 125.77 MHz  
OBFIN 7.87 kHz  
POINT 4.21 Hz  
32780  
FREQU 39308.18 Hz  
SCANS 512  
ACQTM 0.8336 sec  
PD 2.0000 sec  
PW1 2.72 usec  
IRNUC 1H  
CTEMP 22.9 °C  
SLVNT NONE  
EXREF 77.00 ppm  
BF 0.12 Hz  
RGAIN 60

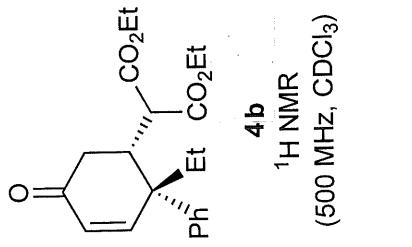
197.835  
168.668  
155.682  
142.390  
128.993  
128.679  
127.592  
127.096  
77.267  
76.762  
61.678  
51.456  
48.005  
46.126  
37.364  
25.121  
13.937  
13.841  
8.912



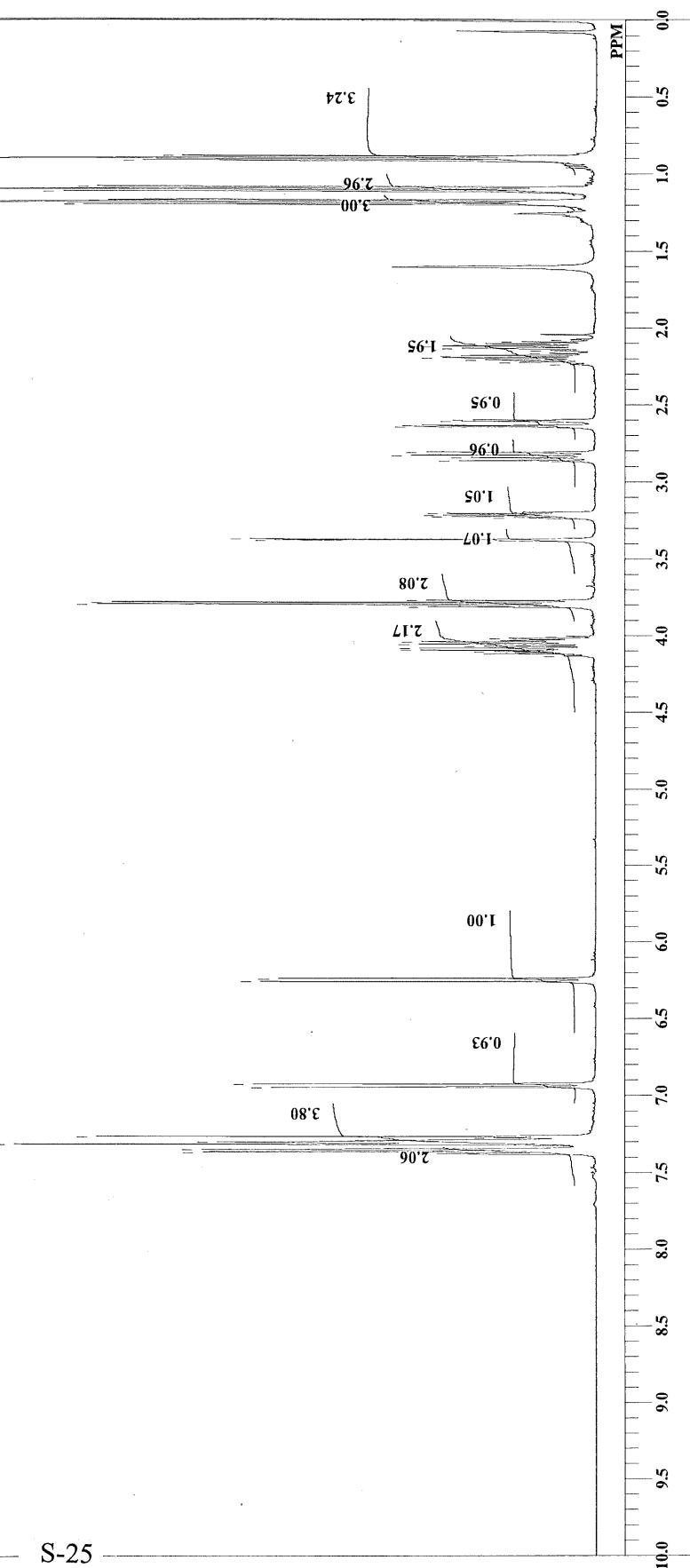
```

NAO-236-down_proton-3-1.ls
NAO-236-syn
2012-08-07 23:31:08
1H
proton.jdp
500.16 MHz
OBFRQ
2.41 KHz
OBFIN
6.01 Hz
POINT
13120
7507.51 Hz
8
SCANS
1.7459 sec
ACQTM
5.0000 sec
PD
4.68 usec
PW1
IRNUC
1H
23.4 c
CTEMP
CDCL3
SLVNT
7.26 ppm
EXREF
BF
0.12 Hz
44
RGAIN

```



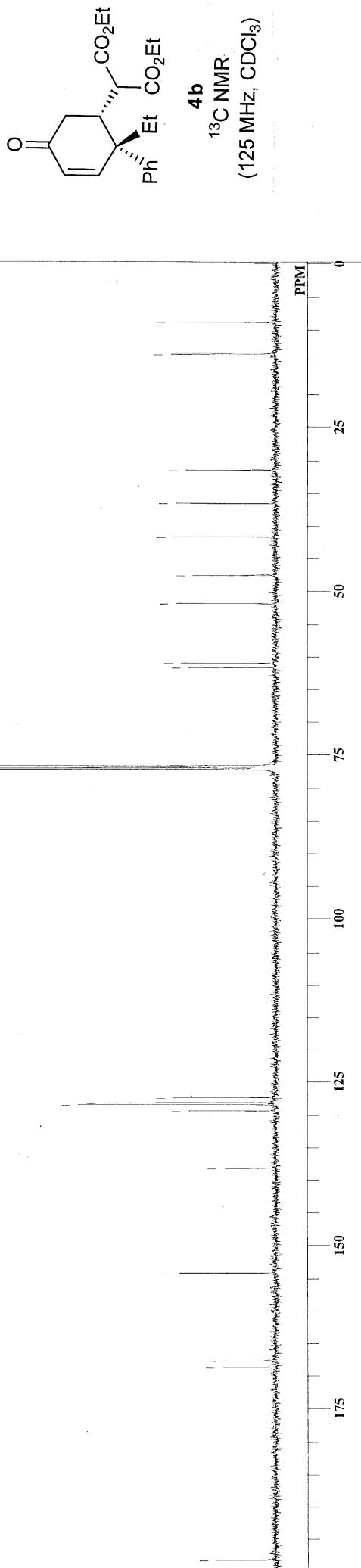
**1H NMR**  
(500 MHz, CDCl<sub>3</sub>)



single pulse decoupled gated NOE

C:\DOCUMENTS\ADMINI-1\WINNOCALS-1\Temp\fffftp1988\NAO-236-down\_Carbon-1-1.jdf

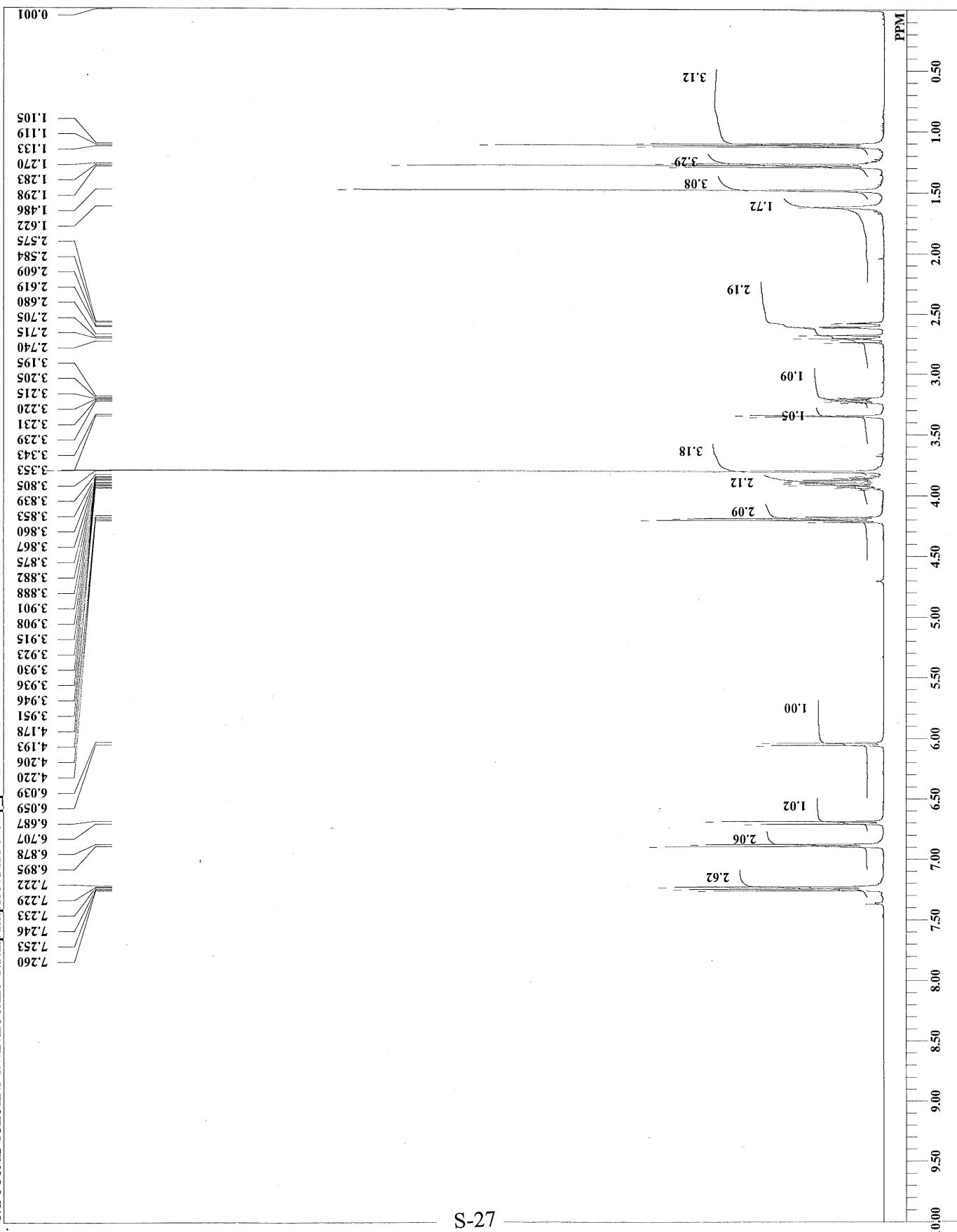
NAO-236-down\_Carbon-1-1.jdf  
single pulse decoupled gated NOE  
2012-08-07 23:38:60  
<sup>13</sup>C  
carbon.jxp  
125.77 MHz  
1.87 kHz  
4.21 Hz  
32780  
39308.18 Hz  
512  
0.8336 sec  
2.0000 sec  
2.72 usec  
1H  
24.0 c  
CDCL<sub>3</sub>  
77.00 ppm  
BF  
RGAIN  
58



```

NAO-245-up_proton-4-1.als
single_pulse
COMNT
DATIM 2012-07-25 00:09:03
1H
OBNUC
EXMOD
proton.jdp
      500.16 MHz
OBFRQ
      2.41 kHz
OBSET
      6.01 Hz
OBFIN
      13120
POINT
      7507.51 Hz
FREQU
      8
SCANS
      1.7459 sec
ACQTM
      5.0000 sec
PD
      4.68 usec
PW1
      1H
CTEMP
      22.8 °c
SLVNT
      CDCL3
EXREF
      12.51 ppm
BF
      0.12 Hz
RGAIN
      42

```



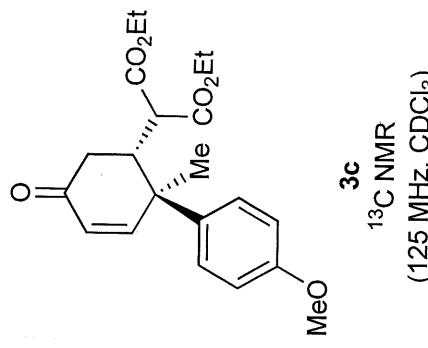
single pulse decoupled gated NOE

C:\DOCUMENTS\ADMINI-1.WINLOCALS\NTemp\ffffp1584\NAO-245-up\_Carbon-3-1.idf

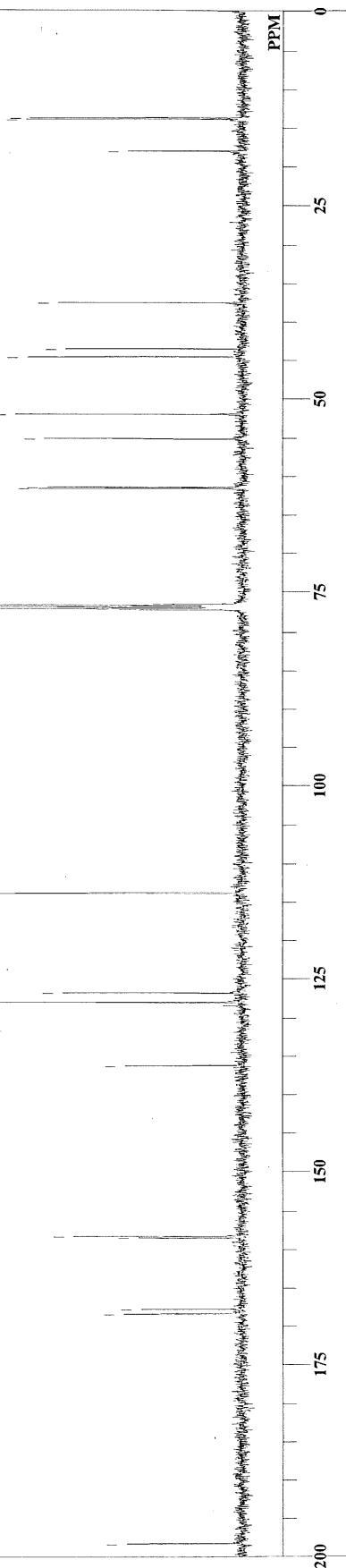
NAO-245-up\_Carbon-3-1.idf  
single pulse decoupled gated NOE  
2012-07-24 22:31:60

13C carbon.jxp  
125.77 MHz  
OBFRQ  
OFFSET 7.87 kHz  
OBFIN 4.21 Hz  
POINT 32780  
FREQU 39308.18 Hz  
SCANS 256  
ACQTM 0.8336 sec  
PD 2.0000 sec  
PW1 2.72 usec  
IRNUC 1H  
CTEMP 22.5 c  
SLVNT CDCL<sub>3</sub>  
EXREF 77.00 ppm  
BF 0.12 Hz  
RGAIN 60

198.312  
168.468  
158.628  
158.447  
167.896  
136.288  
128.088  
126.867  
113.928  
77.257  
77.000  
76.743  
61.649  
55.270  
52.095  
44.677  
43.628  
37.669  
18.065  
13.984  
13.983



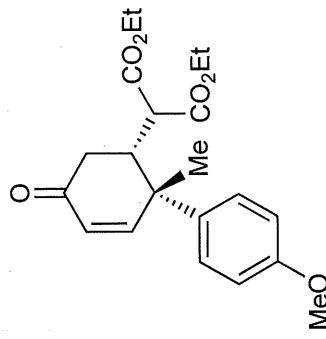
<sup>13</sup>C NMR  
(125 MHz, CDCl<sub>3</sub>)



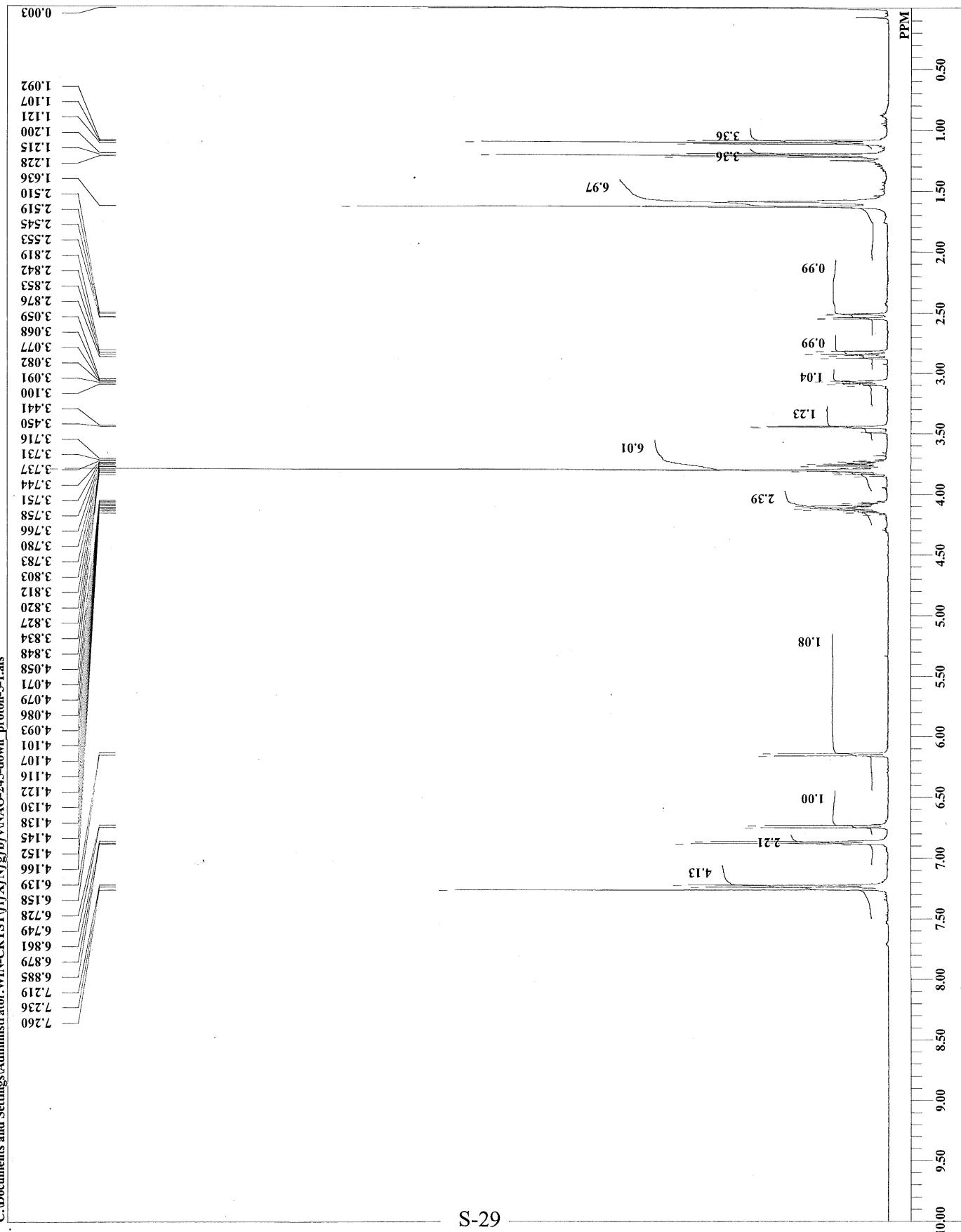
```

DFILE NAO-245-down_proton-5-1.als
COMNT single_pulse
DATIM 2012-08-30 11:55:45
OBNUC 1H
EXMOD proton,jp
OBFRQ 500.16 MHz
OFFSET 2.41 kHz
OBFIN 6.01 Hz
POINT 13120
FREQU 7507.51 Hz
SCANS 8
ACQTM 1.7459 sec
PD 5.0000 sec
PW1 4.68 usec
IRNUC 1H
CTEMP 22.3 c
SLVNT CDCL3
EXREF 12.51 ppm
BF 0.12 Hz
RGAIN 48

```



**4c**  
1H NMR  
(500 MHz, CDCl<sub>3</sub>)

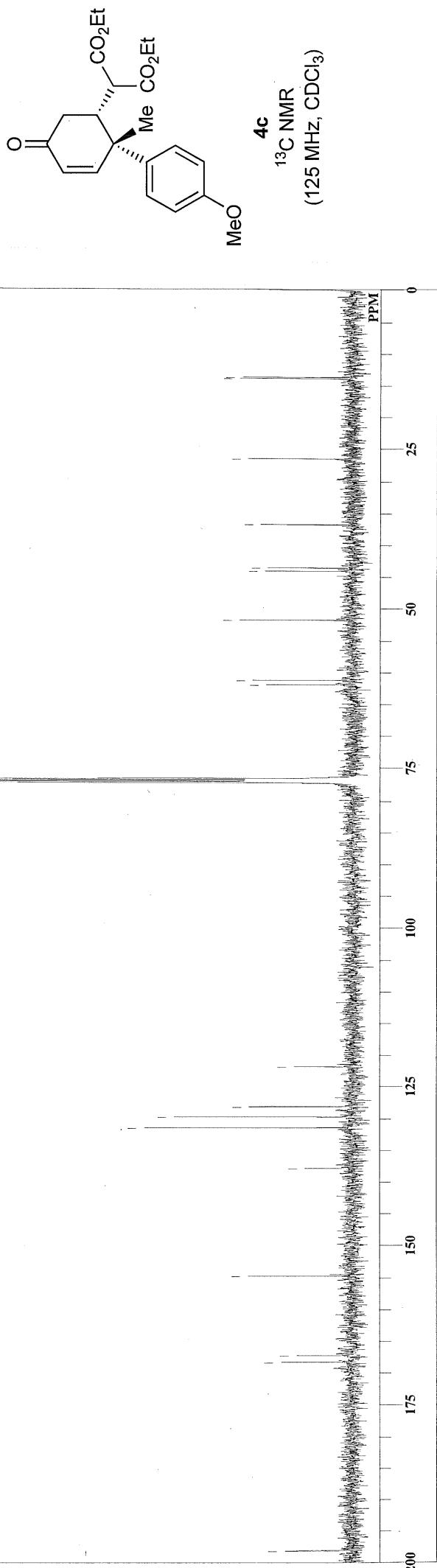


NAR-33-30-down\_Carbon-1-1.idf  
single pulse decoupled gated NOE  
2012-08-22 20:41:05

DFILE  
COMNT  
DATIM  
OBNUC  
EXMOD  
OBFRQ  
OBSET  
OBFIN  
POINT  
FREQU  
SCANS  
ACQTM  
PD  
PW1  
IRNUC  
CTEMP  
SLVNT  
EXREF  
BF  
RGAIN

13C  
carbon.jxp  
125.77 MHz  
7.87 kHz  
4.21 Hz  
32780  
39308.18 Hz  
1024  
0.8336 sec  
2.0000 sec  
2.72 usec  
1H  
23.0 °c  
CDCL<sub>3</sub>  
77.00 ppm  
0.12 Hz  
56

13.927  
13.727  
26.542  
36.830  
44.095  
43.609  
51.866  
61.315  
62.002  
76.743  
77.257  
77.400  
121.909  
128.193  
129.813  
131.520  
137.899  
154.871  
168.439  
167.371  
198.131



C:\DOCUME~1\ADMINI~1.WINN.LOCALS~1\Temp\fffftp1584\NAR-33-up\_Proton-2-1.als

DFILE NAR-33-up\_proton-2-1-als

COMNT single\_pulse

DATIM 2012-09-28 22:44:38

QBNUC 1H

EXMOD Proton-JXP

OBFRQ 500.16 MHz

OBSET 2.41 kHz

OBFIN 6.01 Hz

POINT 13120

FREQU 7507.51 Hz

SCANS 8

ACQTM 1.7459 sec

PD 5.0000 sec

PWI 4.68 usec

IRNUC 1H

CTEMP 20.4 c

SLVNT CDCl<sub>3</sub>

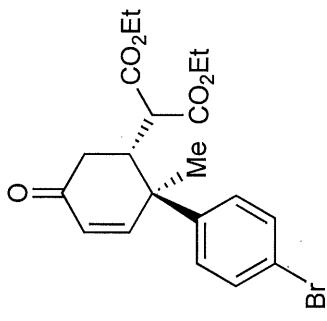
EXREF 12.51 ppm

BF 0.12 Hz

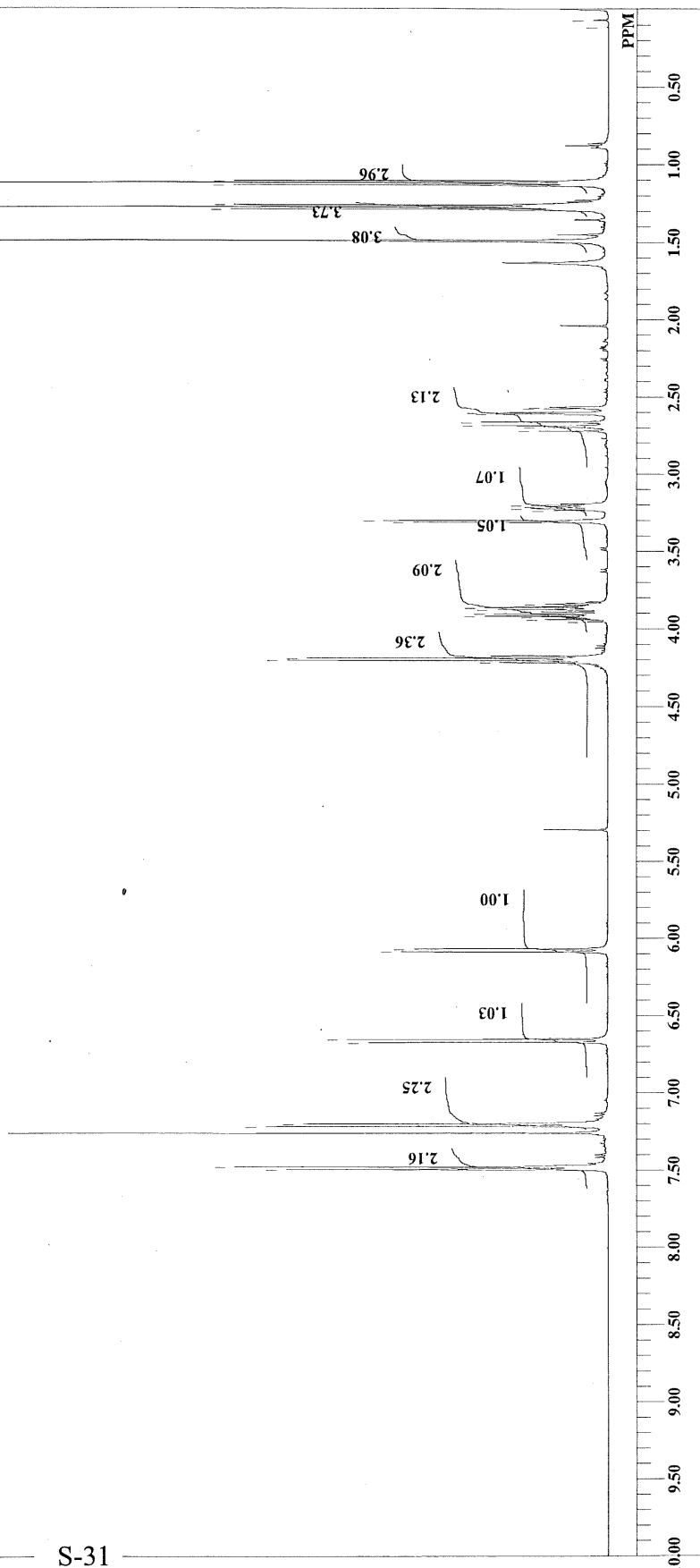
RGAIN 40

Chemical shifts (ppm) listed on the left:

- 0.068
- 0.069
- 0.070
- 0.071
- 0.072
- 0.073
- 0.074
- 0.075
- 0.076
- 0.077
- 0.078
- 0.079
- 0.080
- 0.081
- 0.082
- 0.083
- 0.084
- 0.085
- 0.086
- 0.087
- 0.088
- 0.089
- 0.090
- 0.091
- 0.092
- 0.093
- 0.094
- 0.095
- 0.096
- 0.097
- 0.098
- 0.099
- 0.100
- 0.101
- 0.102
- 0.103
- 0.104
- 0.105
- 0.106
- 0.107
- 0.108
- 0.109
- 0.110
- 0.111
- 0.112
- 0.113
- 0.114
- 0.115
- 0.116
- 0.117
- 0.118
- 0.119
- 0.120
- 0.121
- 0.122
- 0.123
- 0.124
- 0.125
- 0.126
- 0.127
- 0.128
- 0.129
- 0.130
- 0.131
- 0.132
- 0.133
- 0.134
- 0.135
- 0.136
- 0.137
- 0.138
- 0.139
- 0.140
- 0.141
- 0.142
- 0.143
- 0.144
- 0.145
- 0.146
- 0.147
- 0.148
- 0.149
- 0.150
- 0.151
- 0.152
- 0.153
- 0.154
- 0.155
- 0.156
- 0.157
- 0.158
- 0.159
- 0.160
- 0.161
- 0.162
- 0.163
- 0.164
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- 0.166
- 0.167
- 0.168
- 0.169
- 0.170
- 0.171
- 0.172
- 0.173
- 0.174
- 0.175
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- 0.177
- 0.178
- 0.179
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- 0.181
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- 0.190
- 0.191
- 0.192
- 0.193
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- 0.195
- 0.196
- 0.197
- 0.198
- 0.199
- 0.200
- 0.201
- 0.202
- 0.203
- 0.204
- 0.205
- 0.206
- 0.207
- 0.208
- 0.209
- 0.210
- 0.211
- 0.212
- 0.213
- 0.214
- 0.215
- 0.216
- 0.217
- 0.218
- 0.219
- 0.220
- 0.221
- 0.222
- 0.223
- 0.224
- 0.225
- 0.226
- 0.227
- 0.228
- 0.229
- 0.230
- 0.231
- 0.232
- 0.233
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- 0.235
- 0.236
- 0.237
- 0.238
- 0.239
- 0.240
- 0.241
- 0.242
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- 0.245
- 0.246
- 0.247
- 0.248
- 0.249
- 0.250
- 0.251
- 0.252
- 0.253
- 0.254
- 0.255
- 0.256
- 0.257
- 0.258
- 0.259
- 0.260
- 0.261
- 0.262
- 0.263
- 0.264
- 0.265
- 0.266
- 0.267
- 0.268
- 0.269
- 0.270
- 0.271
- 0.272
- 0.273
- 0.274
- 0.275
- 0.276
- 0.277
- 0.278
- 0.279
- 0.280
- 0.281
- 0.282
- 0.283
- 0.284
- 0.285
- 0.286
- 0.287
- 0.288
- 0.289
- 0.290
- 0.291
- 0.292
- 0.293
- 0.294
- 0.295
- 0.296
- 0.297
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- 0.300
- 0.301
- 0.302
- 0.303
- 0.304
- 0.305
- 0.306
- 0.307
- 0.308
- 0.309
- 0.310
- 0.311
- 0.312
- 0.313
- 0.314
- 0.315
- 0.316
- 0.317
- 0.318
- 0.319
- 0.320
- 0.321
- 0.322
- 0.323
- 0.324
- 0.325
- 0.326
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**3d**  
 **$^1\text{H}$  NMR**  
(500 MHz, CDCl<sub>3</sub>)

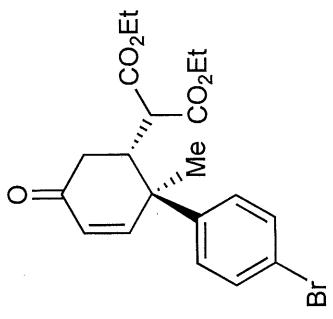


single pulse decoupled gated NOE

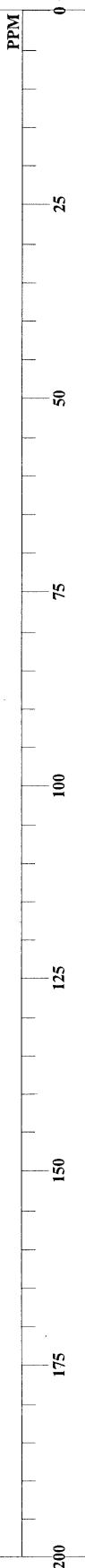
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NAR-33-12-13 Carbon-1-1.jdf  
single pulse decoupled gated NOE  
2012-08-22 20:13:16  
13C  
carbon.jxp  
125.77 MHz  
OBFRQ  
7.87 kHz  
OBSET  
4.21 Hz  
OBFIN  
32780  
POINT  
39308.18 Hz  
FREQU  
256  
SCANS  
0.8336 sec  
ACQTM  
PD  
2.0000 sec  
PW1  
2.72 usec  
IRNUC  
1H  
CTEMP  
23.0 c  
SLVNT  
CDCL<sub>3</sub>  
EXREF  
77.00 ppm  
BF  
0.12 Hz  
RGAIN  
60

197.721  
168.201  
157.083  
143.439  
131.720  
128.803  
127.363  
121.480  
77.248  
77.000  
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61.639  
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37.583  
18.056  
13.965  
13.784

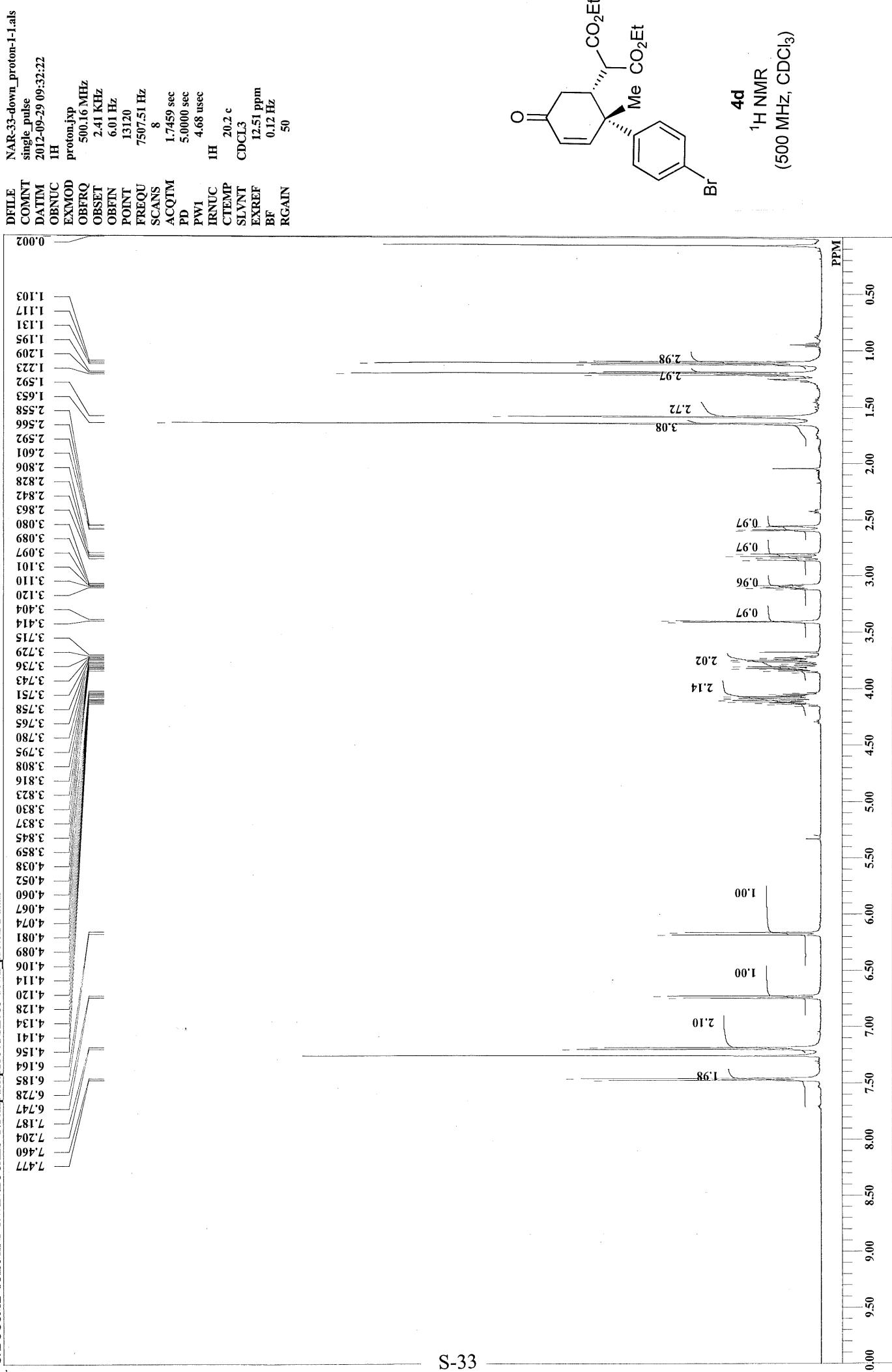


**3d**  
**<sup>13</sup>C NMR**  
(125 MHz, CDCl<sub>3</sub>)



single\_pulse

C:\DOCUMENTS\~1\ADMINI~1.WIN\LOCALS\~1\Temp\fffftp1584\NAR-33-down\_proton-1-1.als



NAO-245-down\_Carbon-1-1.jdf  
single pulse decoupled gated NOE  
2012-07-24 22:59:07

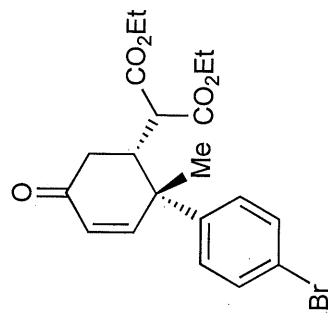
<sup>13</sup>C

carbon.jxp

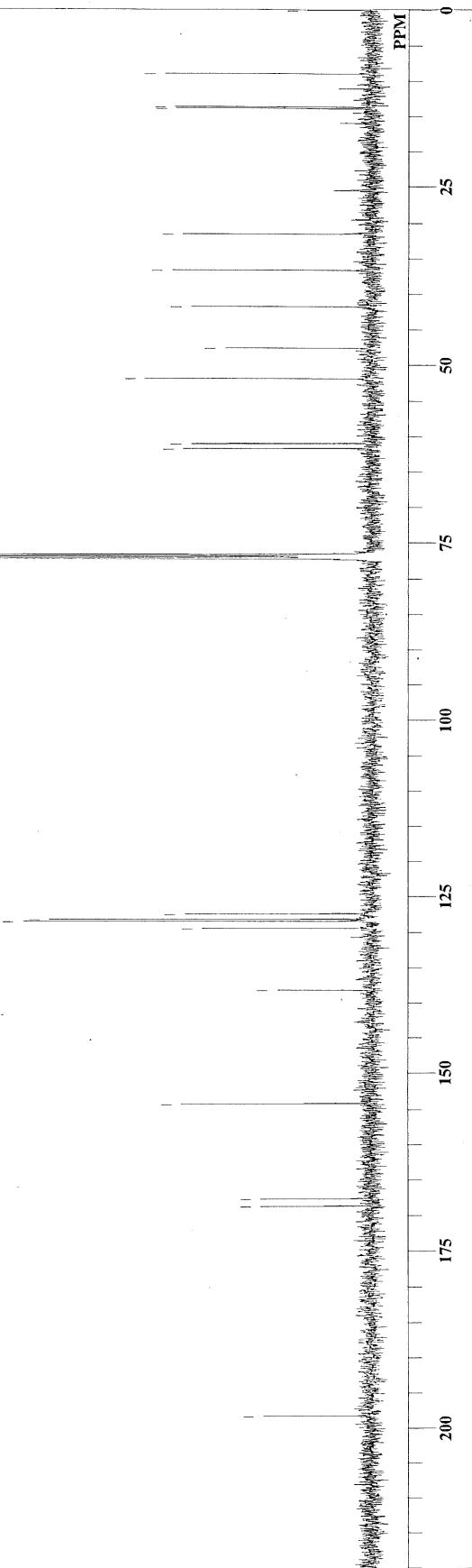
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OBFRQ  
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OBFIN  
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ACQTM  
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PW1  
IRNUC  
CTEMP  
SLVNT  
EXREF  
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RGAIN

-0.003

13.908 8.988 13.698 13.908 31.662 36.763 41.826 47.661 51.962 61.115 61.782 76.743 77.000 77.248 129.489 128.526 128.202 127.439 138.214 154.232 168.687 198.274



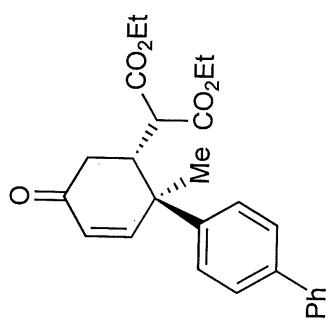
**4d**  
<sup>13</sup>C NMR  
(125 MHz, CDCl<sub>3</sub>)



```

NAO-251-up_proton-2-1.als
single_pulse
2012-08-29 19:59
1H
proton,jsp
500.16 MHz
OBFRQ
2.41 kHz
OBSET
6.01 Hz
OBFIN
13120
POINT
7507.51 Hz
8
SCANS
1.7459 sec
ACQTM
5.0000 sec
PD
4.68 usec
PW1
IRNUC
1H
22.0 c
CTEMP
CDCL3
SLYNT
7.26 ppm
EXREF
BF
0.12 Hz
RGAIN
38

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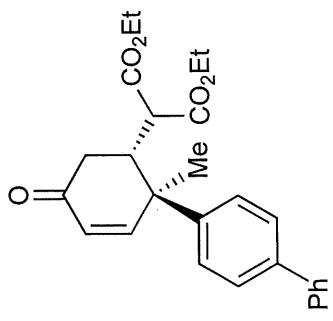
**3e**  
**<sup>1</sup>H NMR**  
(500 MHz, CDCl<sub>3</sub>)

## single pulse decoupled gated NOE

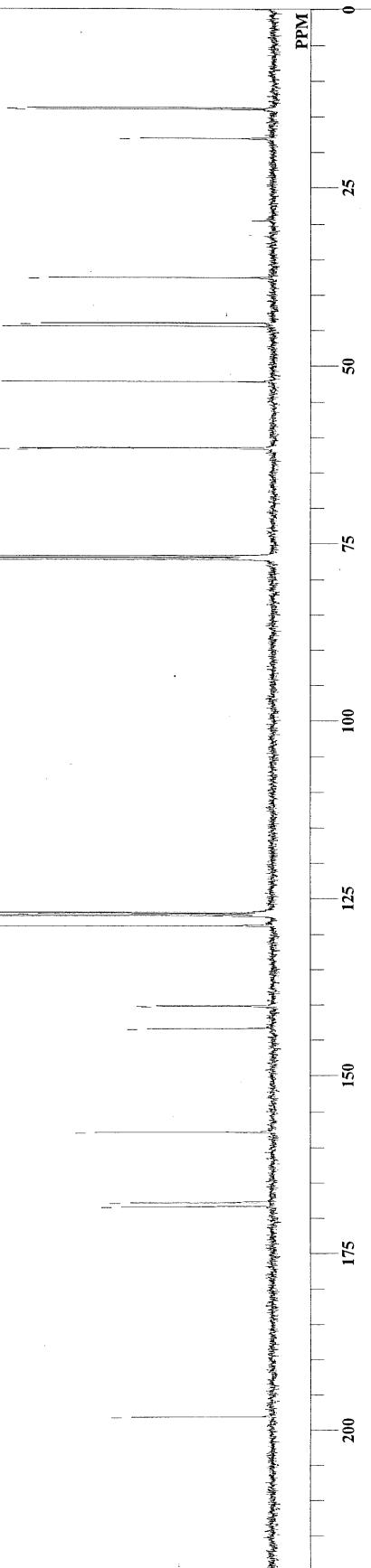
C:\DOCUMENTS\ADMIN-1\WINNLOCAIS-1\Temp\ffffp1584\NAO-251-up\_Carbon-1-1.als

NAO-251-up\_Carbon-1-1.als  
 single pulse decoupled gated NOE  
 2012-08-01 20:05:21  
<sup>13</sup>C  
 carbon.jxp  
 125.77 MHz  
 7.87 kHz  
 4.21 Hz  
 26224  
 31446.54 Hz  
 512  
 0.8336 sec  
 2.0000 sec  
 2.72 usec  
 1H  
 23.7 c  
 CDCL<sub>3</sub>  
 SLYNT  
 EXREF  
 77.00 ppm  
 BF  
 0.12 Hz  
 RGAIN  
 58

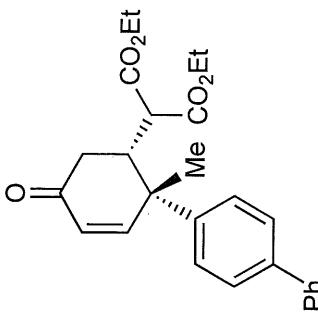
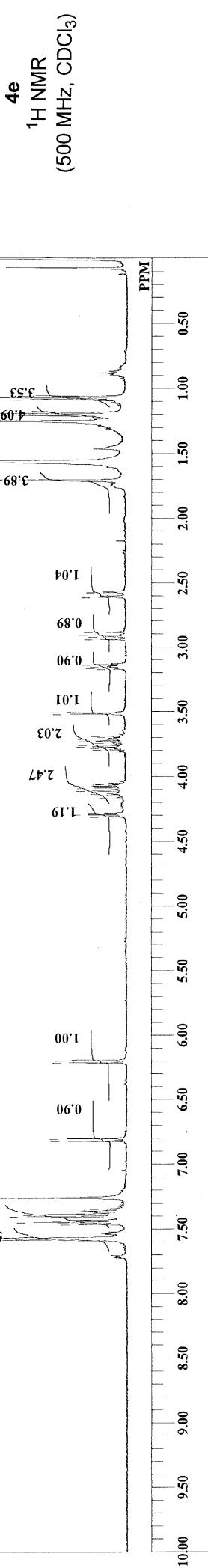
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 127.106  
 126.924  
 77.257  
 77.000  
 76.752  
 61.678  
 61.582  
 52.257  
 44.496  
 44.067  
 37.697  
 18.075  
 13.984  
 13.794



**3e**  
<sup>13</sup>C NMR  
 (125 MHz, CDCl<sub>3</sub>)



NAO-251-down\_proton-4-1.als  
 single\_pulse  
 2012-10-07 18:23:51  
 IH  
 proton.jxp  
 500.16 MHz  
 OBFRQ  
 OBSET 2.41 kHz  
 OBFIN 6.01 Hz  
 POINT 13120  
 FREQU 7507.51 Hz  
 SCANS 8  
 ACQTM 1.7459 sec  
 PD 5.0000 sec  
 PW1 4.68 usec  
 IRNUC 1H  
 CTEMP 20.0 c  
 SLYNT CDCL<sub>3</sub>  
 EXREF 12.51 ppm  
 BF 0.12 Hz  
 RGAIN 54

single pulse decoupled gated NOE

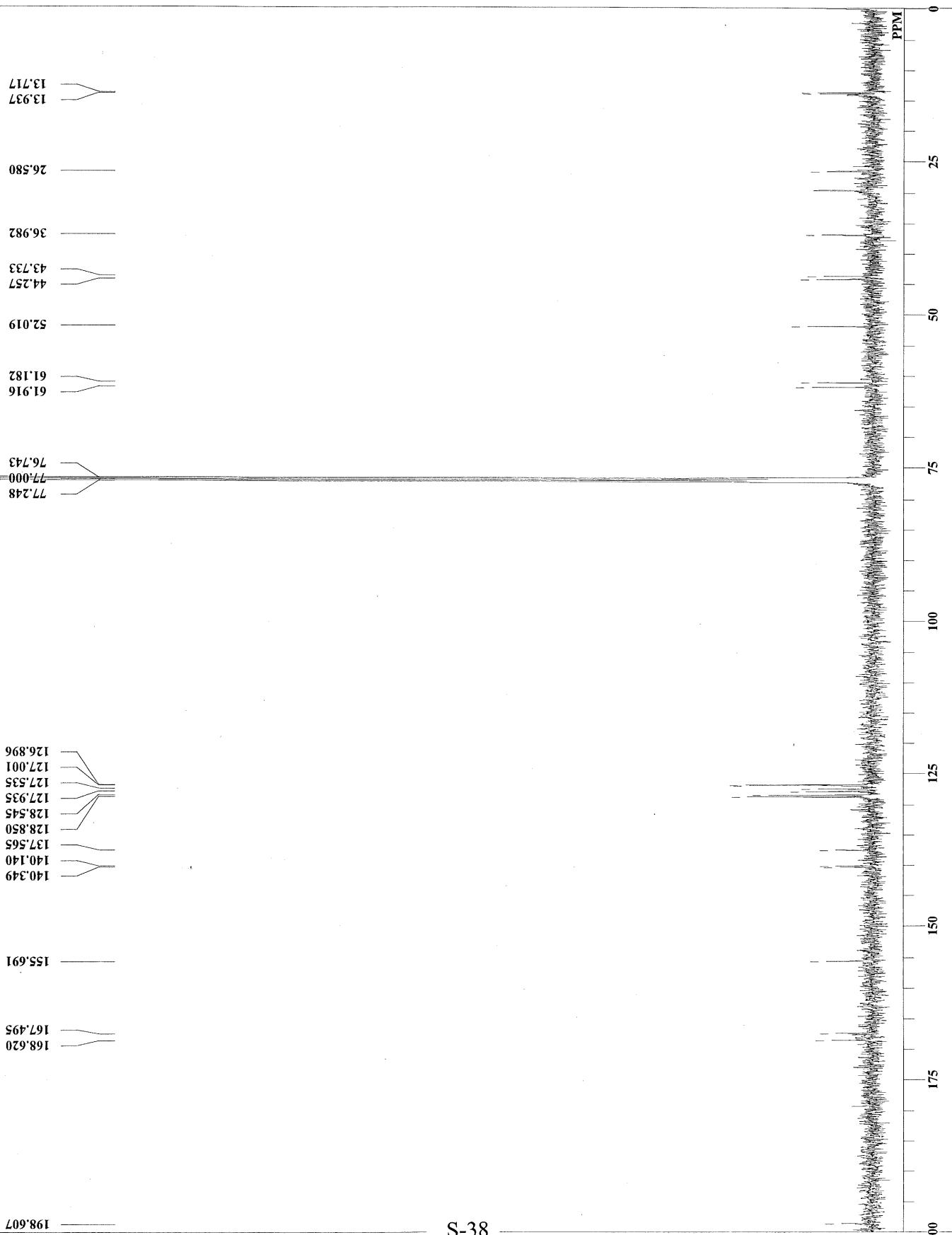
C:\DOCUMENTS\~1\ADMIN\1.WINLOCALS-1\Temp\fffftp1584\NAO-251-down\_Carbon-4.1.idf

```

NAO-251-down_Carbon-4.1.idf
single pulse decoupled gated NOE
2012-10-07 11:24:26
13C
carbon.jdp
DFILE
COMNT
DATIM
OBNUC
EXMOD
OBFRQ 125.77 MHz
OBSET 7.87 kHz
OBFIN 4.21 Hz
POINT 32780
FREQU 39308.18 Hz
SCANS 2048
ACQTM 0.8336 sec
PD 2.0000 sec
PW1 2.72 usec
IRNUC 1H
CTEMP 20.5 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 60

```

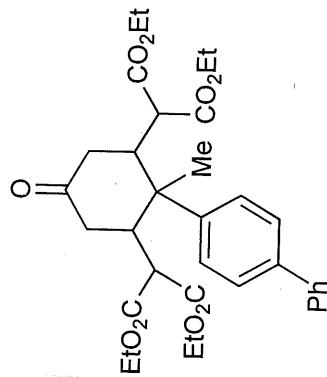
**4e**



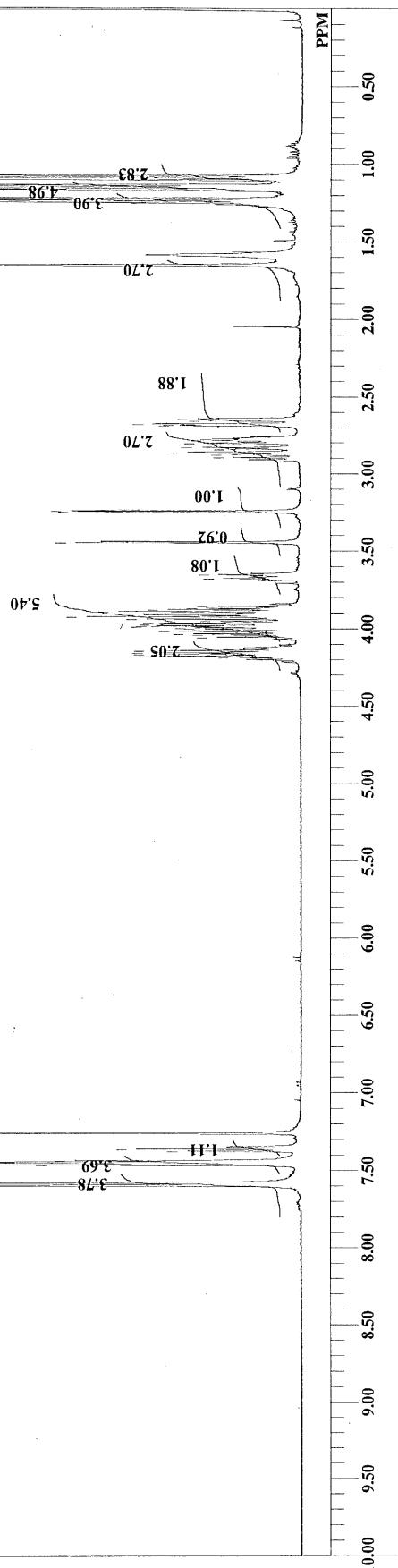
C:\DOCUMENTE\1ADMINI-1\WINLOCALS-S-1\Temp\fftp1584\NAO-251-bpyro proton-2-1.als

NAO-251-bpyro\_proton-2-1.als  
single pulse  
2012-10-24 21:03:04

DEFILE	NAO-251-bpyro_proton-2-1.als
COMNT	
DATIM	2012-10-24 21:03:04
OBNUC	1H
EXMOD	proton J, xP
OBFRQ	500.16 MHz
OBSET	2.41 kHz
OBFIN	6.01 Hz
POINT	13120
FREQU	7507.51 Hz
SCANS	8
ACQTIM	1.7459 sec
PD	5.0000 sec
PWI	4.68 usec
HRNUC	1H
CTEMP	18.5 c
SLVNT	CDCL3
EXREF	12.51 ppm
BF	0.12 Hz
RGAIN	48



<sup>1</sup>H NMR  
(500 MHz, CDCl<sub>3</sub>)



single pulse decoupled gated NOE

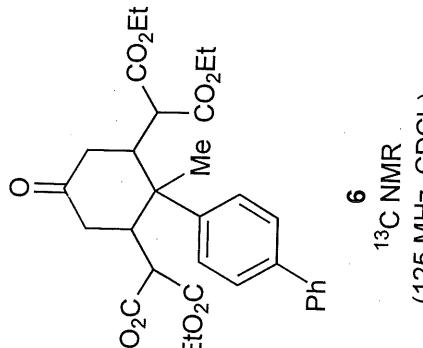
C:\DOCUMENTS~1\ADMINI~1.WINLOCALS~1\Temp\ffffp1584\NAO-251-hpyro\_Carbon-2-1.als

NAO-251-hpyro\_Carbon-2-1.als  
single pulse decoupled gated NOE  
2012-10-24 21:12:02

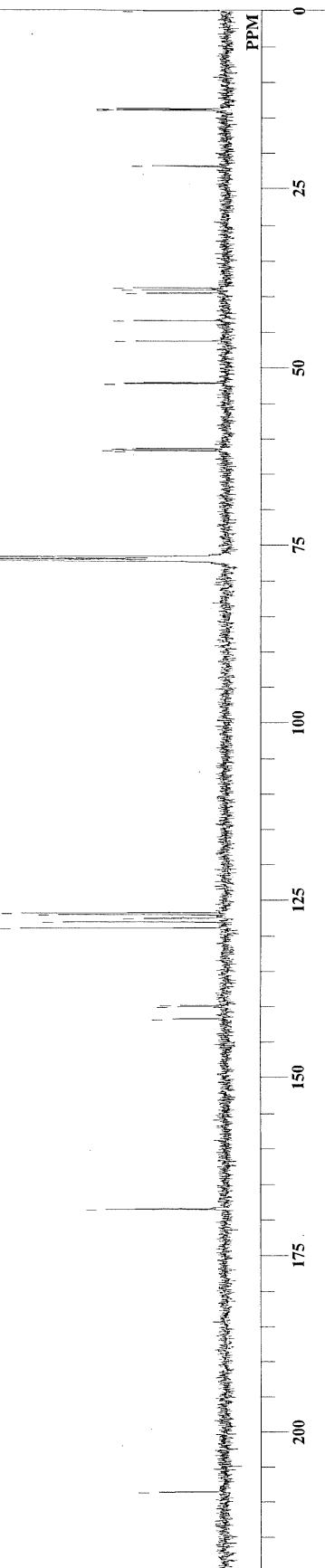
13C  
carbon.jxp  
125.77 MHz  
7.87 kHz  
4.21 Hz  
26224  
31446.54 Hz  
1024  
0.8336 sec  
2.0000 sec  
2.72 usec  
1H  
CTEMP  
SLVNT  
EXREF  
BF  
RGAIN  
0.006

Chemical structure of compound 6: A bicyclic ketone with two ethyl ester groups (EtO<sub>2</sub>C-) at the bridgehead positions and a phenyl group (Ph) attached to one of the carbons.

Peak list (ppm): 126.762, 127.516, 127.020, 128.059, 128.879, 139.787, 139.997, 141.684, 168.487, 168.563, 168.401, 77.000, 76.743, 61.763, 61.515, 61.458, 52.295, 52.181, 46.298, 43.437, 39.576, 39.118, 38.861, 21.851, 13.956, 13.879, 13.813, 13.736



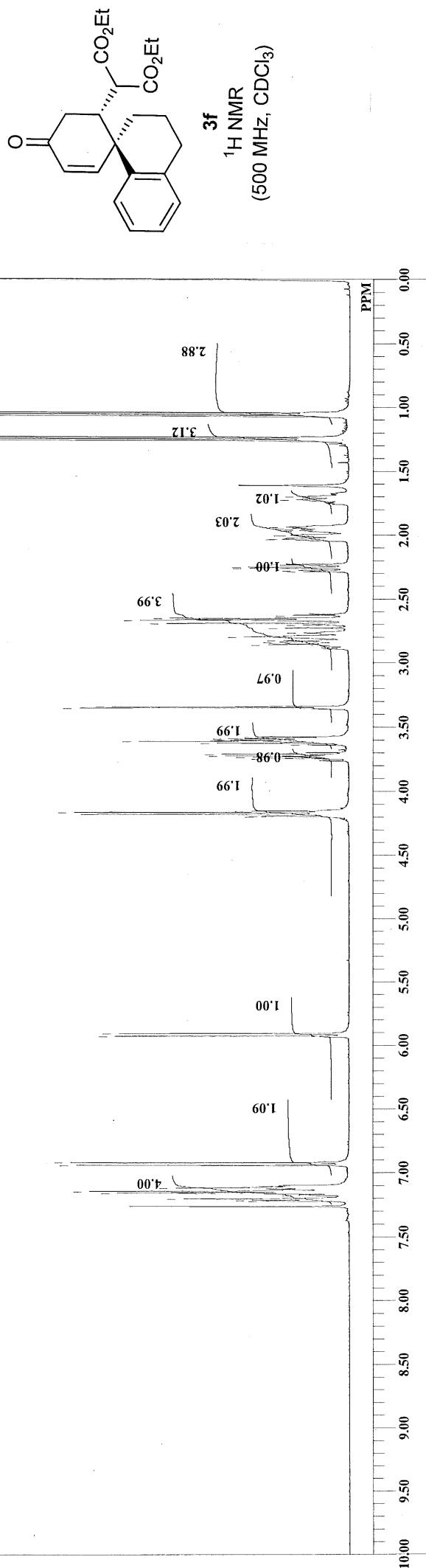
**6**  
<sup>13</sup>C NMR  
(125 MHz, CDCl<sub>3</sub>)



```

NAO-238-up_proton-1-1.als
single_pulse
2012-07-24 23:30:27
IH
proton.jdp
500.16 MHz
OBFRQ
2.41 kHz
OBSET
6.01 Hz
OBFIN
13120
POINT
7507.51 Hz
FREQU
SCANS
8
ACQTM
1.7459 sec
PD
5.0000 sec
PW1
4.68 usec
IRNUC
IH
CTEMP
22.5 °c
CDCL3
EXREF
7.26 ppm
BF
0.12 Hz
RGAIN
40

```



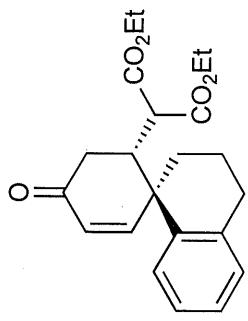
single pulse decoupled gated NOE

C:\DOCUMENTS\J\ADMIN\J.J.WIN\LOCALS-J\TEMP\ffffp1584\NAO-238-up\_Carbon-1-1.jdf

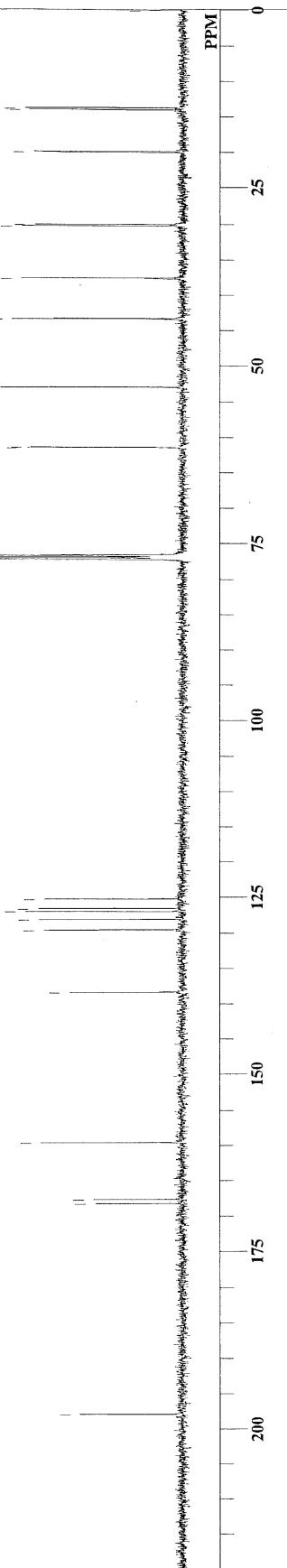
NAO-238-up\_Carbon-1-1.jdf  
single pulse decoupled gated NOE  
2012-07-24 23:37:58

DFILE COMNT  
OBNUC EXMOD  
carbon.jxp  
13C  
OBFRQ 125.77 MHz  
OBSET 7.87 kHz  
OBFIN 4.21 Hz  
POINT 32780  
FREQU 39308.18 Hz  
SCANS 512  
ACQTM 0.8336 sec  
PD 2.0000 sec  
PW1 2.72 usec  
IRNUC 1H  
CTEMP 23.1 c  
SLVNT CDCL<sub>3</sub>  
EXREF 77.00 ppm  
BF 0.12 Hz  
RGAIN 56

197.940  
168.258  
167.676  
159.638  
138.357  
129.632  
128.107  
126.991  
125.562  
125.246  
77.257  
77.000  
76.752  
61.554  
61.477  
53.068  
43.456  
43.361  
37.726  
30.327  
30.146  
19.944  
14.003  
13.736  
-0.003



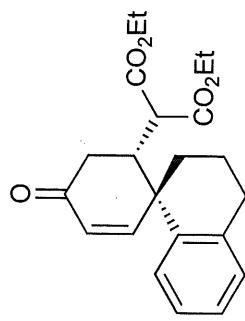
**3f**  
<sup>13</sup>C NMR  
(125 MHz, CDCl<sub>3</sub>)



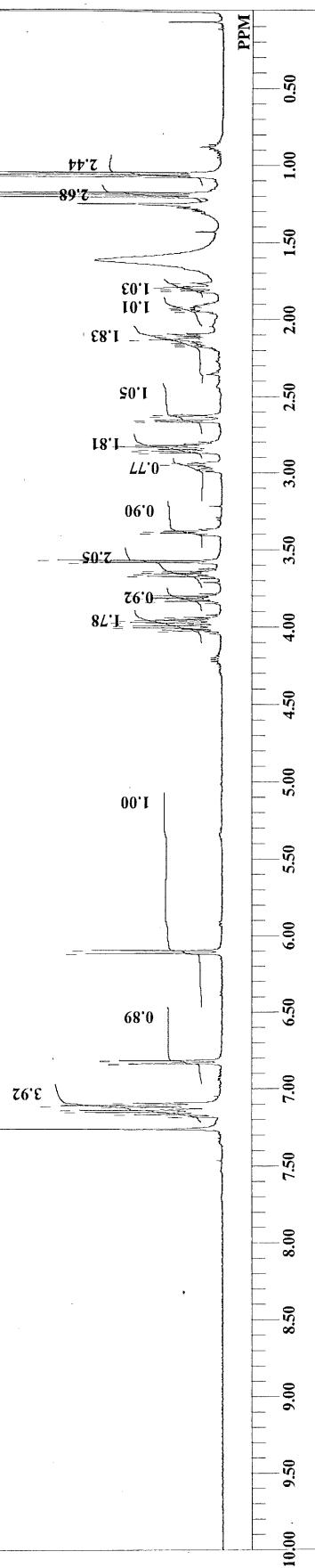
```

DFILE NAO-238-down_proton-2-1.jdf
single_pulse
COMNT
DATIM 2012-10-16 23:08:26
1H
OBNUC
EXMOD proton,jsp
OBFRQ 500.16 MHz
OBSET 2.41 kHz
OBFIN 6.01 Hz
POINT 16400
FREQU 9384.38 Hz
SCANS 8
ACQTM 1.7459 sec
PD 5.0000 sec
PWI 4.68 usec
IRNUC
1H 20.1 c
CTEMP CDCL3
SLYNT 12.51 ppm
EXREF BF 0.12 Hz
RGAIN 50

```



**4f**  
<sup>1</sup>H NMR  
(500 MHz, CDCl<sub>3</sub>)



single pulse decoupled gated NOE

C:\DOCUMENTS\~1\ADMINI-1.WINLOCALES~1\Temp\fffftp1584\NAO-238-down\_Carbon-1-1.als

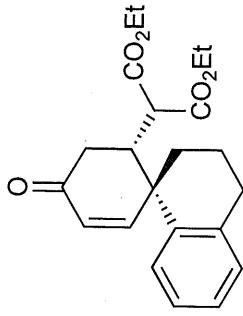
```

NAO-238-down_Carbon-1-1.als
single pulse decoupled gated NOE
2012-10-07 17:22:46
13C
carbon.jdp

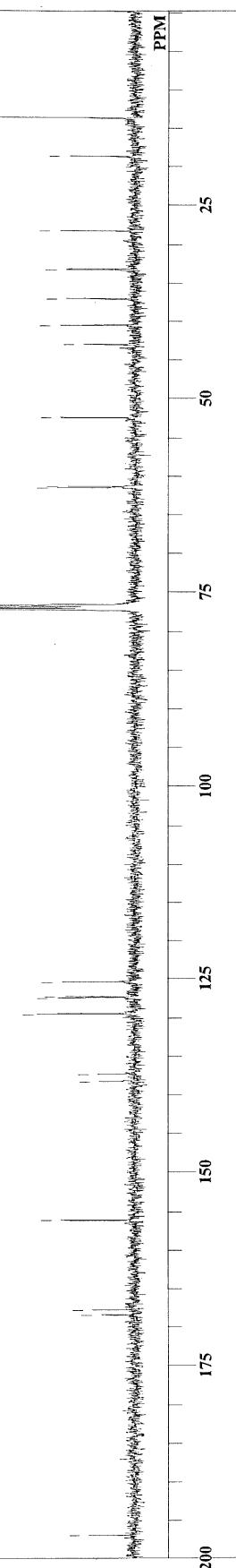
DFILE
COMNT
DATIM
OBNUC
EXMOD
OBFRQ 125.77 MHz
OFFSET 7.87 kHz
OBFIN 4.21 Hz
POINT 26224
FREQU 31446.54 Hz
SCANS 1024
ACQTM 0.8336 sec
PD 2.0000 sec
PWI 2.72 usec
IRNUC 1H
CTEMP 20.3 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 60

```

Chemical structure of compound 4f: A bicyclic system consisting of a trisubstituted cyclohexene fused with a benzene ring. Two ethyl ester groups (CO<sub>2</sub>Et) are attached to one of the ring carbons.



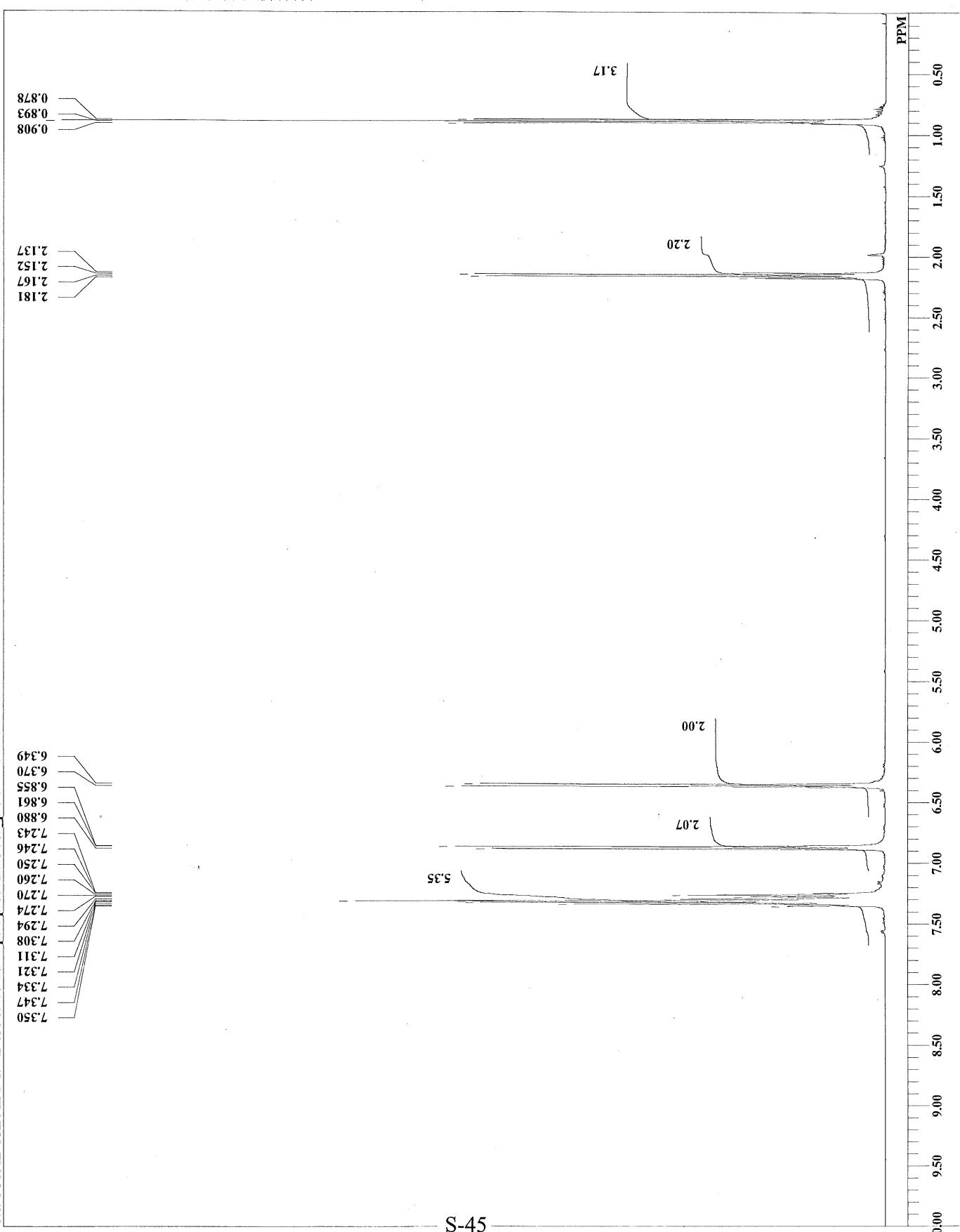
**4f**  
<sup>13</sup>C NMR  
(125 MHz, CDCl<sub>3</sub>)



single\_pulse

C:\DOCUMENTS\1\ADMIN\1.WIN\LOCALS\-\NTemp\ffffpp1584NAO-232\_proton-1-1.als

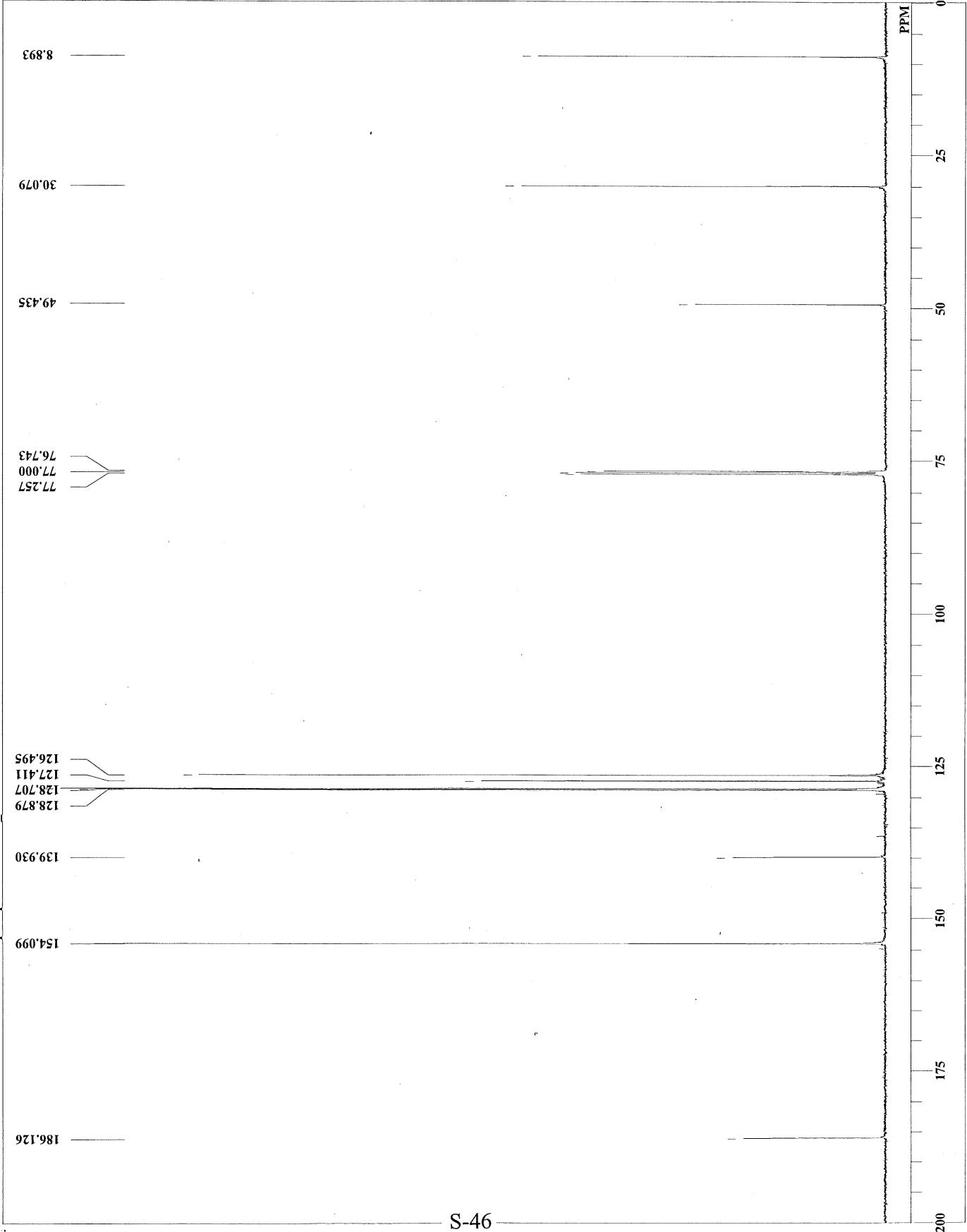
DFILE NAO-232\_proton-1-1.als  
single\_pulse  
COMNT  
DATIM 2012-08-29 14:40:50  
OBNUC 1H  
EXMOD proton,j,p  
OBFRO 500.16 MHz  
OBSET 2.41 kHz  
OBFIN 6.01 Hz  
POINT 13120  
FREQU 7507.51 Hz  
SCANS 8  
ACQTM 1.7459 sec  
PD 5.0000 sec  
PW1 4.68 usec  
IRNUC 1H  
CTEMP 21.7 c  
SLVNT CDCL<sub>3</sub>  
EXREF 7.26 ppm  
BF 0.12 Hz  
RGAIN 30



3g  
<sup>1</sup>H NMR  
(500 MHz, CDCl<sub>3</sub>)

single pulse decoupled gated NOE

C:\DOCUMENTS\ADMIN\WINNOCALS-1\Temp\ffffp1584\NAO-232\_Carbon-1-1.als

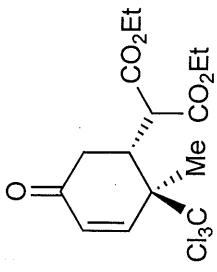


NAO-232\_Carbon-1-1.als  
single pulse decoupled gated NOE

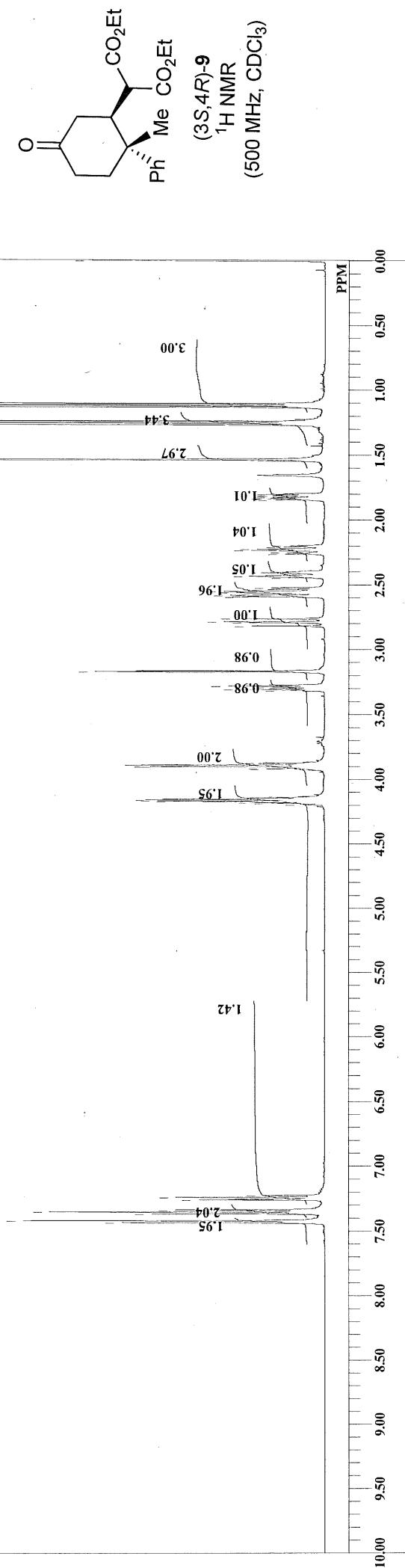
COMNT  
DATIM  
OBNUC  
EXMOD  
OBFRQ  
OBSET  
OBFIN  
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FREQU  
SCANS  
ACQTM  
PD  
PW1  
IRNUC  
CIEMP  
SLYNT  
EXREF  
BF  
RGAIN

125.77 MHz  
7.87 kHz  
4.21 Hz  
26224  
31446.54 Hz  
512  
0.8336 sec  
2.0000 sec  
2.72 usec  
1H  
22.2 c  
 $\text{CDCl}_3$   
77.00 ppm  
0.12 Hz  
60

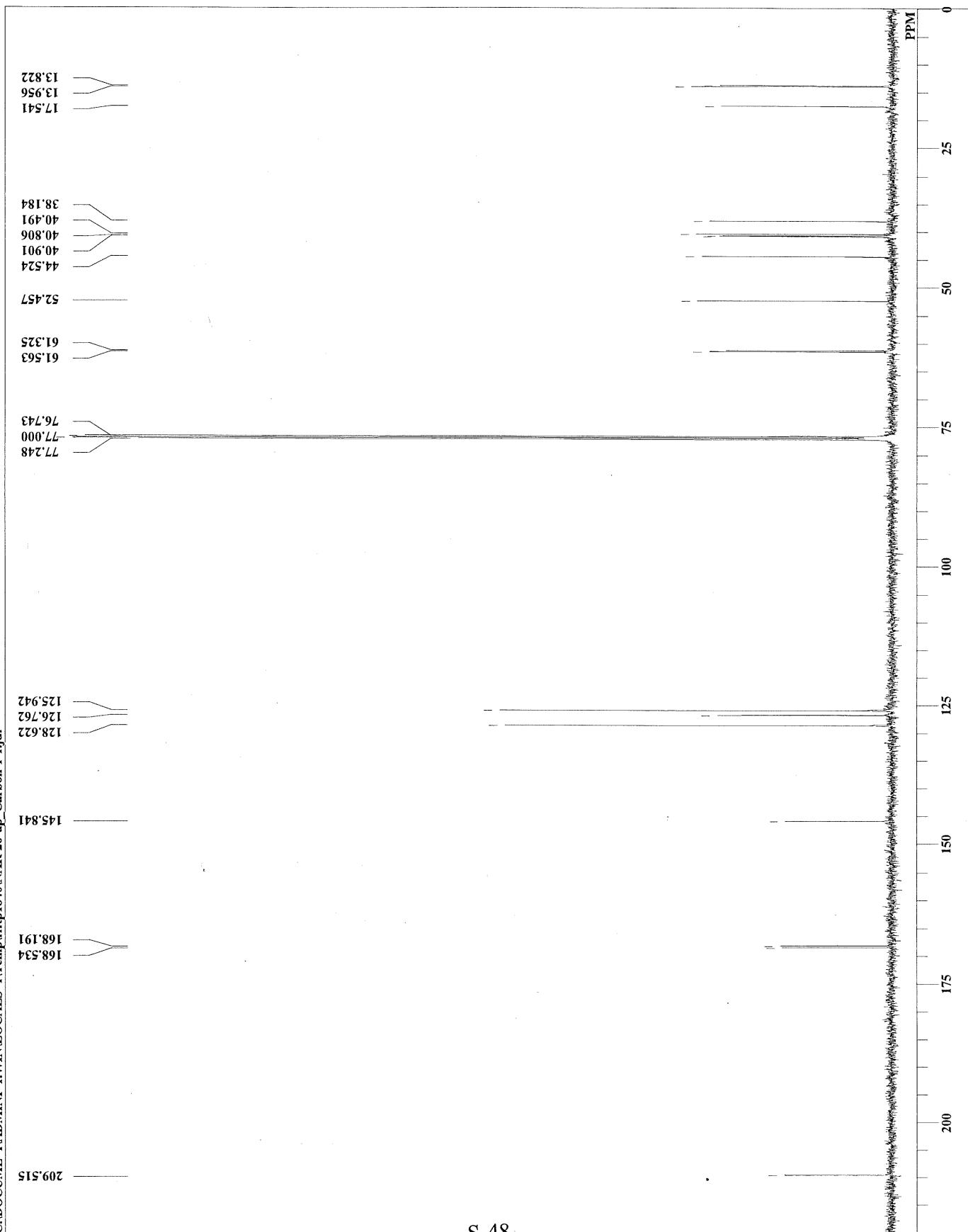
13C



NAR-26-up\_proton-1-1.jdf  
 single\_pulse  
 COMNT  
 DATIM 2012-08-24 14:59:23  
 1H  
 OBNUC proton.jdp  
 EXMOD  
 500.16 MHz  
 OBFRQ 2.41 kHz  
 OBSET 6.01 Hz  
 OBFIN 16400  
 POINT 9384.38 Hz  
 8  
 SCANS 1.7459 sec  
 ACQTM 5.0000 sec  
 PD 4.68 usec  
 PW1  
 IRNUC 1H  
 CTEMP 22.9 °C  
 SLYNT CDCL<sub>3</sub>  
 EXREF 7.26 ppm  
 BF 0.12 Hz  
 RGAIN 38



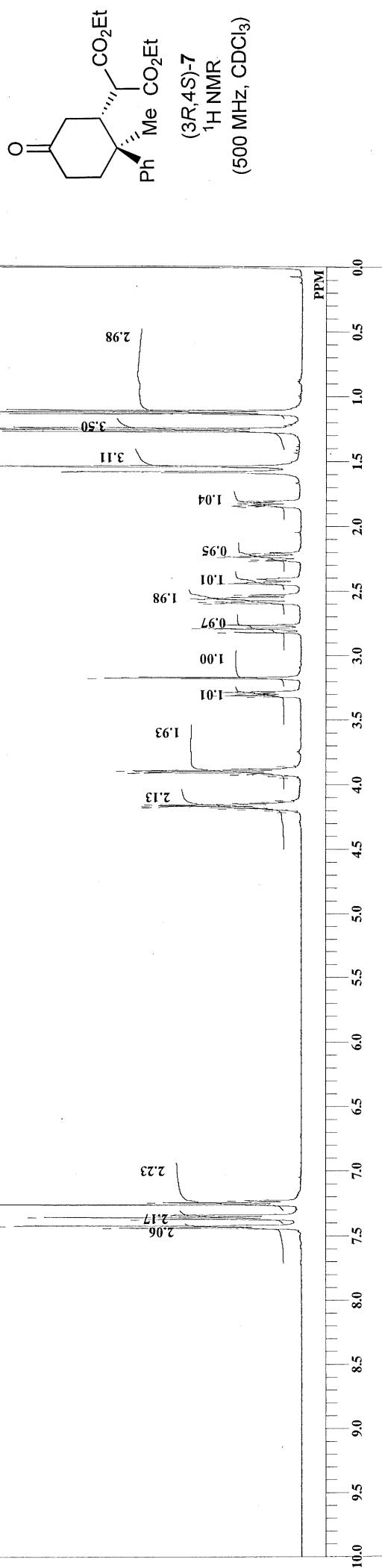
NAR-26-up\_Carbon-1-1.idf  
single pulse decoupled gated NOE  
2012-08-25 (09:29:17)  
13C  
carbon.jdp  
125.77 MHz  
7.87 kHz  
4.21 Hz  
32780  
39308.18 Hz  
256  
0.8336 sec  
2.0000 sec  
2.72 usec  
1H  
22.5 c  
CDCL<sub>3</sub>  
SLVNT  
EXREF  
BF  
RGAIN



(3S,4R)-9  
<sup>13</sup>C NMR  
(125 MHz, CDCl<sub>3</sub>)

NAO-270-up\_proton-2-1.als  
single\_pulse  
2012-07-10 14:56:26  
H  
proton.jxp  
500.16 MHz  
OBFRQ  
OBSET  
OBFIN  
POINT  
EXMOD  
FREQU  
SCANS  
ACQTM  
PD  
PW1  
IRNUC  
CTEMP  
SLVNT  
EXREF  
BF  
RGAIN

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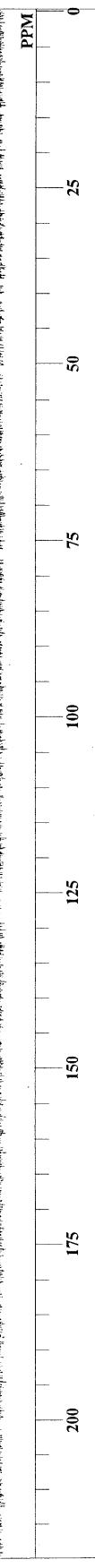
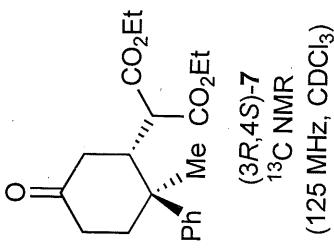


## single pulse decoupled gated NOE

C:\DOCUMENTS\-\ADMIN\WINNOLOCALS-1\Temp\ffffp1584\NAO-254\_Carbon-1-1.jdf



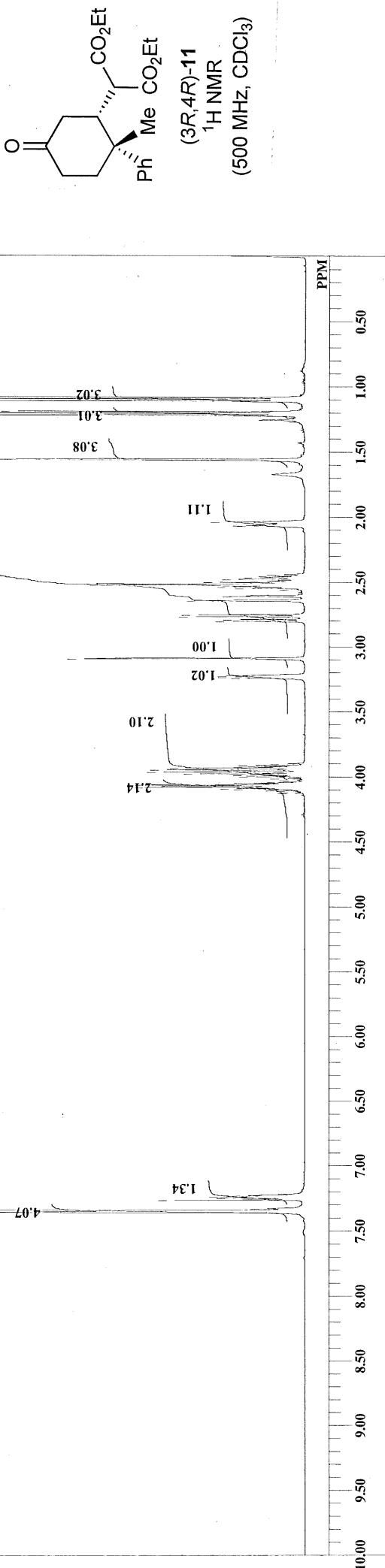
NAO-254\_Carbon-1-1.jdf  
single pulse decoupled gated NOE  
2012-06-02 18:21:03  
13C  
carbon.jxp  
125.77 MHz  
7.87 kHz  
4.21 Hz  
32780  
39308.18 Hz  
512  
0.8336 sec  
2.0000 sec  
2.72 usec  
1H  
19.2 c  
CDCL<sub>3</sub>  
EXREF  
BF  
RGAIN  
58



```

DFILE   NAR-25-down_proton-1.als
COMNT  single_pulse
DATIM  2012-08-24 18:21:20
OBNUC  1H
EXMOD proton-j,xp
      OBFRQ 500.16 MHz
      OBSET 2.41 kHz
      OBFIN 6.01 Hz
      POINT 13120
      FREQU 7507.51 Hz
      SCANS 8
      ACQTM 1.7459 sec
      PD 5.0000 sec
      PW1 4.68 usec
      IRNUC 1H
      CTEMP 23.1 c
      SLVNT CDCL3
      EXREF 7.26 ppm
      BF 0.12 Hz
      RGAIN 36

```

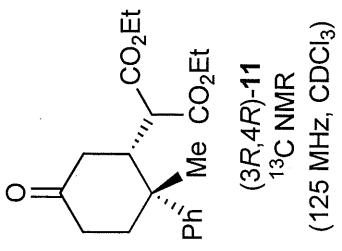


NAR-25-down Carbon-1-1.jdf  
single pulse decoupled gated NOE  
2012-08-25 13:10:12  
13C  
carbon.jxp

DFILE  
COMNT  
DATIM  
OBNUC  
EXMOD  
OBFRQ  
OFFSET  
OBFIN  
POINT  
FREQU  
SCANS  
ACQTM  
PD  
PWI  
IRNUC  
CTEMP  
SLVNT  
EXREF  
BF  
RGAIN

125.77 MHz  
7.87 kHz  
4.21 Hz  
3.2780  
39308.18 Hz  
256  
0.8336 sec  
2.0000 sec  
2.72 usec  
1H  
22.9 c  
CDCL<sub>3</sub>  
77.00 ppm  
0.12 Hz  
58

13.765  
13.832  
27.304  
30.174  
36.696  
39.395  
39.719  
44.181  
52.839  
61.382  
61.525  
76.743  
77.000  
77.257  
123.856  
126.610  
128.593  
146.299  
168.935  
168.983  
208.514



(3R,4R)-11  
<sup>13</sup>C NMR  
(125 MHz, CDCl<sub>3</sub>)

