

Supporting Information

Palladium-Catalyzed sp^3 C-H Activation of Simple Alkyl Groups: Direct Preparation of Indoline Derivatives from *N*-Alkyl-2-bromoanilines

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Experimental Section

2-bromo-*N-tert*-butylanilines (**1**, **3**, **4**, **5a–13a**) were prepared by *N-tert*-butylation of the corresponding 2-bromoanilines as follows, according to the literature procedures.^{1,2}

1. Representative Procedures for Preparation of *N*-Alkyl-2-bromoanilines.

1) Preparation of 2-Bromo-*N-tert*-butylaniline: Method A^{1,2}

To a solution of condensed isobutylene (18 mL) in dioxane (18 mL) were added 2-bromoaniline (17.4 mmol, 3.0 g) and 47% HBr (19.2 mmol, 2.26 mL) at -78 °C in a pressure tube. The tube was sealed and heated for 4 days at 110 °C. After cooling, the mixture was neutralized with 1N NaOH. The mixture was diluted with ethyl acetate, washed with saturated NaHCO₃ and brine, dried over MgSO₄, and concentrated *in vacuo*. The crude material was purified by flash chromatography with hexane/ethyl acetate (30:1) to afford the 2-bromo-*N-tert*-butylaniline (2.18 g, 60 % yield).

1') Preparation of 2-Bromo-*N-tert*-butylaniline: Method B³

To a solution of 2-bromoaniline (23.3 mmol, 4.0 g) in cyclohexane (30 mL) were added *tert*-butyl 2,2,2-trichloroacetimidate (58.2 mmol, 10.4 mL) and BF₃·OEt₂ (25.8 mL /mol, 600 μL) at room temperature under argon. The mixture was stirred at room temperature for 6 h. The mixture was diluted with ethyl acetate, washed with saturated NaHCO₃ and brine, dried over MgSO₄, and concentrated *in vacuo*. The crude material was purified by flash chromatography with hexane/ethyl acetate (30:1) to afford the 2-bromo-*N-tert*-butylaniline (2.68 g, 51 % yield).

2) Preparation of Methyl 2-Bromophenyl(*tert*-butyl)carbamate **1**

A mixture of 2-bromo-*N-tert*-butylaniline (1.0 g, 4.38 mmol) in methyl chloroformate (20 mL) was heated under reflux for 4 h. The mixture was poured into water and extracted with chloroform. The organic extract was, dried over MgSO₄, and concentrated *in vacuo*. The crude material was purified by flash chromatography with hexane/ethyl acetate (20:1) to afford the methyl 2-bromophenyl(*tert*-butyl)carbamate **1** (1.21 g, 97% yield).

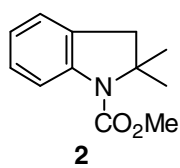
Representative procedure (Table 1, entry 10): Xylene (2.0 mL) was added to a flask containing 2-bromo-*N-tert*-butylaniline **1** (57.2 mg, 0.20 mmol), Pd(OAc)₂ (1.35 mg, 3 mol %), PCy₃·HBF₄ (4.42 mg, 6 mol %), Cs₂CO₃ (98.8 mg, 0.28 mmol) and *t*-BuCO₂H (6.18 mg, 0.06 mmol) under an argon atmosphere. The mixture was stirred at 140 °C for 2 h. After cooling, the reaction mixture was concentrated *in vacuo*. The crude material was purified by flash chromatography with hexane/ethyl acetate (20:1) to afford the indoline **2** (40.3 mg, 98%).

1. Bacque, E.; Qacemi, M. El.; Zard, S. *Org. Lett.* **2005**, *7*, 3817.

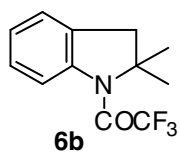
2. Gage, J. R.; Wagner, J. M. *J. Org. Chem.* **1995**, *60*, 2613.

3. Bourgeois, M.-J.; Montaudon, E.; Maillard, B. *Tetrahedron* **1993**, *49*, 2477.

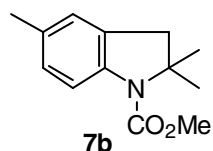
2. Characterization Data for Indolines 2, 6b–17c.



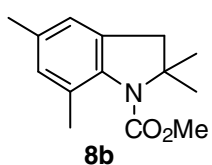
Methyl 2,2-Dimethylindoline-1-carboxylate (2): Colorless oil; IR (ATR) cm^{-1} : 1700 (C=O), 1372 (NAr); ^1H NMR (500 MHz, CDCl_3) δ 1.56 (s, 6H; 2 \times CH_3), 3.02 (s, 2H; CH_2), 3.84 (s, 3H; CO_2Me), 6.95 (t, $J = 7.8, 7.3$ Hz, 1H; Ar), 7.11 (d, $J = 7.3$ Hz, 1H; Ar), 7.16 (dd, $J = 7.8, 7.3$ Hz, 1H; Ar), 7.74 (br, 1H; Ar); ^{13}C NMR (125 MHz, CDCl_3) δ 27.2 (2C), 45.6, 52.0, 64.6, 115.6, 122.5, 124.6, 127.5, 128.7, 142.4, 154.0; HRMS (FAB): calcd for $\text{C}_{12}\text{H}_{15}\text{NO}_2$ (M^+) 205.1103; found: 205.1153.



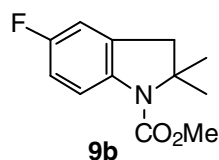
1-(2,2-Dimethylindolin-1-yl)-2,2,2-trifluoroethanone (6b): Brown oil; IR (ATR) cm^{-1} : 1678 (CO), 1410 (NAr), 1220 (CF); ^1H NMR (500 MHz, CDCl_3) δ 1.61 (s, 6H; 2 \times CH_3), 3.01 (s, 2H; CH_2), 7.10 (dd, $J = 7.4, 7.4$ Hz, 1H; Ar), 7.18-7.22 (m, 2H; Ar), 7.30 (br, 1H; Ar); ^{13}C NMR (125 MHz, CDCl_3) δ 25.5 (2C), 45.2, 70.3, 115.7, 116.2 (q), 125.1, 125.5, 127.5, 131.0, 139.6, 154.7 (q); HRMS (ED): calcd for $\text{C}_{12}\text{H}_{12}\text{FNO}$ (M^+) 243.0871; found: 243.0868.



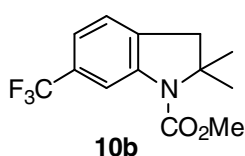
Methyl 2,2,5-Trimethylindoline-1-carboxylate (7b): Colorless oil; IR (ATR) cm^{-1} : 1702 (CO), 1310 (NAr); ^1H NMR (500 MHz, CDCl_3) δ 1.55 (s, 6H; 2 \times CH_3), 2.28 (s, 3H; Ar CH_3), 2.97 (s, 2H; CH_2), 3.83 (s, 3H; CO_2Me), 6.92-6.97 (m, 2H; Ar), 7.63 (br, 1H; Ar); ^{13}C NMR (125 MHz, CDCl_3) δ 20.8 (2C), 27.2, 45.6, 51.9, 64.6, 115.3, 125.3, 127.9, 128.7, 132.0, 140.1, 154.0; *Anal.* calcd for $\text{C}_{13}\text{H}_{17}\text{NO}_2$: C, 71.21; H, 7.81; N, 6.39. Found: C, 71.10; H, 8.00; N, 6.23.



Methyl 2,2,5,7-Tetramethylindoline-1-carboxylate (8b): Colorless oil; IR (ATR) cm^{-1} : 1703 (CO) 1360 (NAr); ^1H NMR (500 MHz, CDCl_3) δ 1.48 (s, 6H; 2 \times CH_3), 2.12 (s, 3H; Ar CH_3), 2.26 (s, 3H; Ar CH_3), 2.89 (s, 2H; CH_2), 3.77 (s, 3H; CO_2Me), 6.80 (br, 2H; Ar); ^{13}C NMR (125 MHz, CDCl_3) δ 20.0 (2C), 20.8, 27.1, 46.1, 51.9, 66.2, 122.6, 126.3, 130.6, 131.5, 133.0, 139.0, 153.7; HRMS (FAB): calcd for $\text{C}_{14}\text{H}_{19}\text{NO}_2$ (M^+) 233.1416; found: 233.1586.

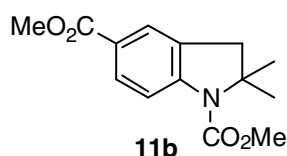


Methyl 5-Fluoro-2,2-dimethylindoline-1-carboxylate (9b): Brown oil; IR (ATR) cm^{-1} : 1697 (CO), 1316 (NAr), 1219 (ArF); ^1H NMR (500 MHz, CDCl_3) δ 1.55 (s, 6H; 2 \times CH_3), 2.99 (s, 2H; CH_2), 3.84 (s, 3H; CO_2Me), 6.81-6.86 (m, 2H; Ar), 7.79 (br, 1H; Ar); ^{13}C NMR (125 MHz, CDCl_3) δ 27.2 (2C), 45.4, 52.1, 65.0, 111.7 (d), 113.6 (d), 116.2 (d), 130.4, 138.5, 153.9, 158.8 (d); HRMS (FAB): calcd for $\text{C}_{12}\text{H}_{14}\text{FNO}_2$ (M^+) 223.0977; found: 223.1451.

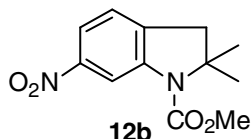


Methyl 2,2-Dimethyl-6-(trifluoromethyl)indoline-1-carboxylate (10b): Brown oil; IR (ATR) cm^{-1} : 1705 (CO), 1318 (NAr), 1234 (CF); ^1H NMR (500 MHz, CDCl_3) δ 1.57 (s, 6H; 2 \times CH_3), 3.06 (s, 2H; CH_2), 3.87 (s, 3H; CO_2Me),

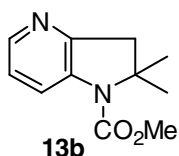
7.20 (m, 2H; Ar), 8.04 (br, 1H; Ar); ^{13}C NMR (125 MHz, CDCl_3) δ 27.2 (2C), 45.3, 52.3, 65.3, 112.4 (d), 120.0 (d), 124.3 (q), 124.7, 130.0 (q), 132.6, 142.8, 153.8; *Anal.* calcd for $\text{C}_{13}\text{H}_{14}\text{F}_3\text{NO}_2$: C, 57.14; H, 5.16; N, 5.13. Found: C, 57.33; H, 5.28; N, 5.05.



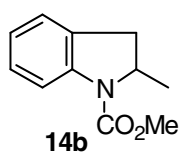
Dimethyl 2,2-Dimethylindoline-1,5-dicarboxylate (11b): Yellow solid: mp 114–115 °C; IR (ATR) cm^{-1} : 1704 (CO), 1613 (CO), 1362 (NAr); ^1H NMR (500 MHz, CDCl_3) δ 1.57 (s, 6H; 2 \times CH_3), 3.04 (s, 2H; CH_2), 3.87 (s, 3H; CO_2Me), 3.88 (s, 3H; CO_2Me), 7.78 (s, br, 2H; Ar), 7.89 (d, $J = 8.6$ Hz, 1H; Ar); ^{13}C NMR (125 MHz, CDCl_3) δ 27.1 (2C), 44.9, 51.8, 52.3, 65.5, 114.8, 124.2, 126.0, 128.8, 130.0, 146.4, 153.8, 166.9; *Anal.* calcd for $\text{C}_{14}\text{H}_{17}\text{NO}_4$: C, 63.87; H, 6.51; N, 5.32. Found: C, 63.57; H, 6.56; N, 5.27.



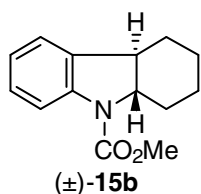
Methyl 2,2-Dimethyl-6-nitroindoline-1-carboxylate (12b): Yellow solid: mp 107–108 °C; IR (ATR) cm^{-1} : 1704 (CO), 1522 (NO), 1344–1389 (NO), 1328 (NAr); ^1H NMR (500 MHz, CDCl_3) δ 1.60 (s, 6H; 2 \times CH_3), 3.10 (s, 2H; CH_2), 3.91 (s, 3H; CO_2Me), 7.22 (d, $J = 8.2$, Hz, 1H; Ar), 7.85 (dd, $J = 8.2$, 2.2 Hz, 1H; Ar) 8.55 (br, 1H; Ar); ^{13}C NMR (125 MHz, CDCl_3) δ 27.2 (2C), 45.2, 52.6, 66.0, 110.6, 118.2, 124.6, 136.2, 143.4, 148.2, 153.7; *Anal.* calcd for $\text{C}_{12}\text{H}_{14}\text{N}_2\text{O}_4$: C, 57.59; H, 5.64; N, 11.19. Found: C, 57.30; H, 5.70; N, 11.0.



Methyl 2,2-Dimethyl-2,3-dihydro-1H-pyrrolo[3,2-b]pyridine-1-carboxylate (13b): Yellow oil; IR (ATR) cm^{-1} : 1701 (CO), 1369 (NAr); ^1H NMR (500 MHz, CDCl_3) δ 1.61 (s, 6H; 2 \times CH_3), 3.16 (s, 2H; CH_2), 3.87 (s, 3H; CO_2Me), 7.07 (dd, $J = 8.2$, 4.4 Hz, 1H; Ar), 7.94 (br, 1H; Ar), 8.12 (d, $J = 4.4$ Hz, 1H; Ar); ^{13}C NMR (125 MHz, CDCl_3) δ 27.5 (2C), 47.5, 52.3, 63.6, 122.0, 122.1, 136.9, 143.1, 151.1, 154.1; HRMS (FAB): calcd for $\text{C}_{11}\text{H}_{14}\text{N}_2\text{O}_2$ (M^+) 206.1055; found: 206.0949.

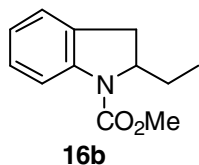


Methyl 2-Methylindoline-1-carboxylate (14b)⁴: Pale yellow oil; IR (ATR) cm^{-1} : 1705 (CO), 1389 (NAr); ^1H NMR (500 MHz, CDCl_3) δ 1.30 (d, $J = 6.4$ Hz, 3H; CH_3), 2.63 (dd, $J = 15.9$, 2.1 Hz, 1H; CHH), 3.36 (dd, $J = 15.9$, 9.5 Hz, 1H; CHH), 3.84 (s, 3H; CO_2Me), 4.50–4.59 (m, 1H; 2-H), 6.96 (t, $J = 7.4$ Hz, 1H; Ar), 7.15 (d, $J = 7.4$ Hz, 1H; Ar), 7.18 (dd, $J = 7.7$, 7.7 Hz, 1H; Ar), 7.80 (br, 1H; Ar); ^{13}C NMR (125 MHz, CDCl_3) δ 21.1, 35.8, 52.3, 55.3, 115.3, 122.6, 125.0, 127.4, 129.8, 141.4, 153.6; *Anal.* calcd for $\text{C}_{11}\text{H}_{13}\text{NO}_2$: C, 69.09; H, 6.85; N, 7.32. Found: C, 69.06; H, 6.93; N, 7.19.

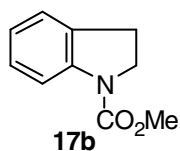


Methyl *trans*-2,3,4,4a-Tetrahydro-1H-carbazole-9(9aH)-carboxylate (15b)⁵: Pale yellow oil; IR (ATR) cm^{-1} : 1705 (CO), 1360 (NAr); ^1H NMR (500 MHz, CDCl_3): $\delta = 1.43$ (m, 3H; 3- CH_2 , 4- CHH), 1.64 (m, 1H; 1- CHH), 1.93 (m, 2H; 2- CH_2), 2.35 (m, 1H; 4- CHH), 2.71 (dd, $J = 11.7$, 9.7, Hz, 1H; 4a-H), 2.90 (dd, $J = 11.7$, 2.9, 2.4, Hz, 1H; 1- CHH), 3.42 (ddd, $J = 11.7$, 11.7, 3.0 Hz, 1H; 9a-H),

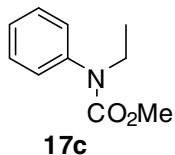
3.83 (s, 3H; CO₂Me), 6.98 (ddd, $J = 7.9, 7.3, 0.9$ Hz, 1H; Ar), 7.10 (d, $J = 7.3$ Hz, 1H; Ar), 7.17 (dd, $J = 8.0, 7.9, 1.3$ Hz, 1H; Ar), 7.74 (d, $J = 8.0$ Hz, 1H; Ar); ¹³C NMR (125 MHz, CDCl₃): $\delta = 25.3, 25.9, 27.2, 31.7, 48.4, 52.2, 69.5, 114.9, 121.4, 122.6, 127.3, 133.5, 143.5, 155.3$; *Anal.* calcd for C₁₄H₁₇NO₂: C, 72.70; H, 7.41; N, 6.06. Found: C, 72.79; H, 7.57; N, 5.80.



Methyl 2-Ethylindoline-1-carboxylate (16b): Colorless oil; IR (ATR) cm⁻¹: 1699 (CO), 1392 (NAr); ¹H NMR (500 MHz, CDCl₃) δ 0.88 (t, $J = 7.4$ Hz, 3H; CH₃), 1.58 (m, 1H; CH₃CH₂H), 1.77 (br, 1H; CH₃CH₂H), 2.76 (dd, $J = 16.0, 2.1$ Hz, 1H; 3-CH₂H), 3.28 (dd, $J = 16.0, 9.6$ Hz, 1H; 3-CH₂H), 3.83 (s, 3H; CO₂Me), 4.38 (s, 1H; 2-H), 6.95 (ddd, $J = 7.7, 7.3, 0.9$ Hz, 1H; Ar), 7.14 (d, $J = 7.3$ Hz, 1H; Ar), 7.17 (dd, $J = 7.7, 7.7$ Hz, 1H; Ar), 7.75 (br, 1H; Ar); ¹³C NMR (125 MHz, CDCl₃) δ 9.0, 27.3, 32.9, 52.4, 60.6, 115.2, 122.7, 124.8, 127.3, 130.4, 142.1, 153.8; *Anal.* calcd for C₁₂H₁₅NO₂: C, 70.22; H, 7.37; N, 6.82. Found: C, 70.32; H, 7.42; N, 6.74.



Methyl Indoline-1-carboxylate (17b)⁴: Pale yellow oil; IR (ATR) cm⁻¹: 1700 (CO), 1317 (NAr); ¹H NMR (500 MHz, CDCl₃) δ 3.07 (t, $J = 8.9$ Hz, 2H; 3-CH₂), 3.84 (br, 3H; CO₂Me), 3.96 (br, 2H; 2-CH₂), 6.93 (ddd, $J = 7.7, 7.2, 1.1$ Hz, 1H; Ar), 7.12 (d, $J = 7.2$ Hz, 1H; Ar), 7.15 (dd, $J = 7.7$ Hz, 1H; Ar), 7.86 (br, 1H; Ar); ¹³C NMR (125 MHz, CDCl₃) δ 27.2, 47.1, 52.2, 114.4, 122.2, 124.4, 127.1, 130.6, 142.4, 153.3; *Anal.* calcd for C₁₀H₁₁NO₂: C, 67.78; H, 6.26; N, 7.90. Found: C, 67.55; H, 6.09; N, 8.01.

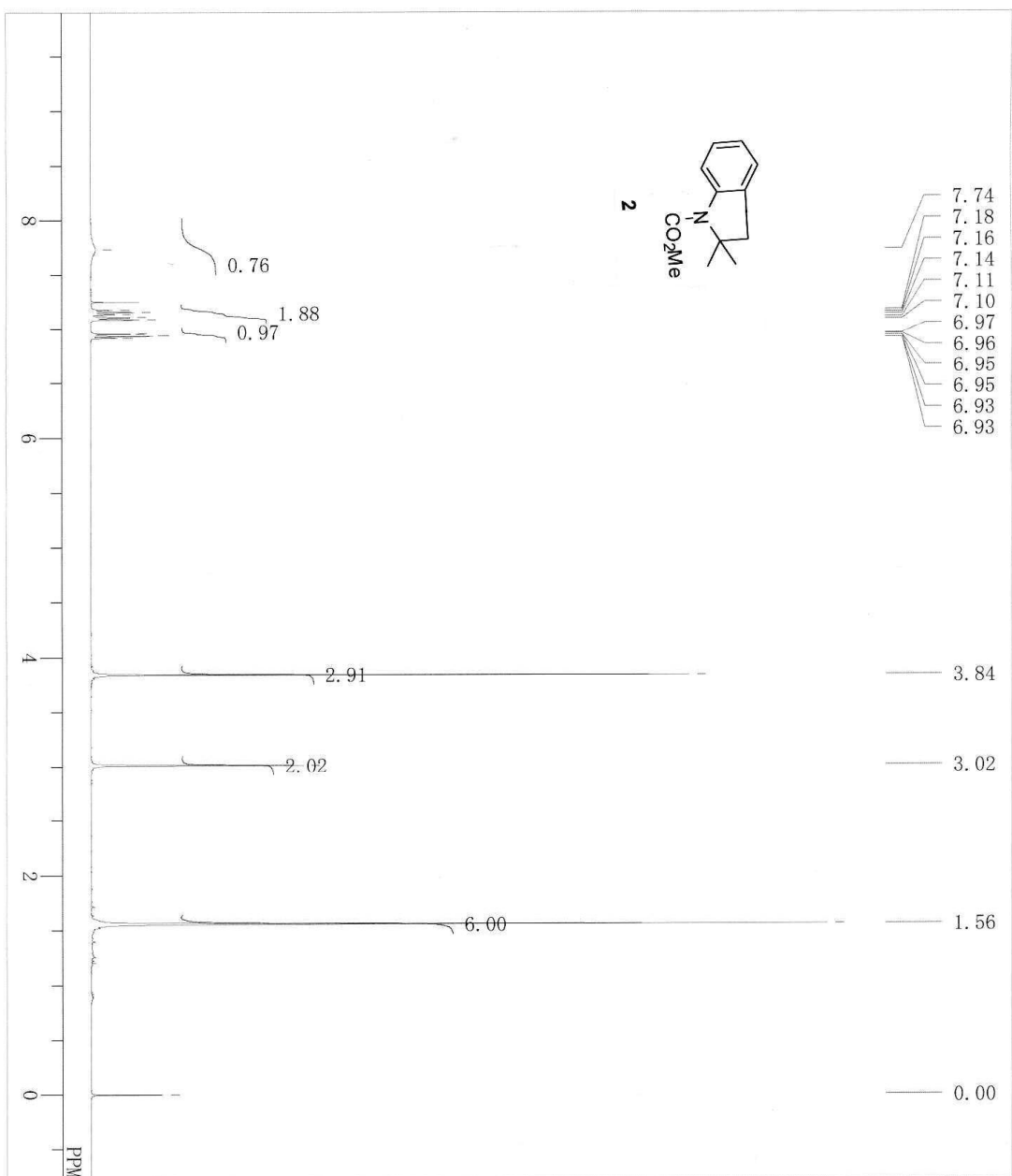


Methyl Ethyl(phenyl)carbamate (17c)⁶: colorless oil; IR (ATR) cm⁻¹: 1695 (CO), 1388 (NAr); ¹H NMR (500 MHz, CDCl₃) δ 1.14 (t, $J = 7.2$ Hz, 3H; CH₃), 3.66 (br, 3H; CO₂Me), 3.71 (q, $J = 7.2$ Hz, 2H; CH₂), 7.18 (d, $J = 7.4$ Hz, 2H; Ar), 7.24 (t, $J = 7.4$ Hz, 1H; Ar), 7.35 (t, $J = 7.4$ Hz, 2H; Ar); ¹³C NMR (125 MHz, CDCl₃) δ 13.5, 45.3, 52.6, 126.5, 127.3 (2C), 128.9 (2C), 141.7, 155.8; HRMS (FAB): calcd for C₁₀H₁₃NO₂ (M⁺) 179.0946; found: 179.0447.

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6. Sawyer, J. S.; Narayanan, B. A. *Synth. Commun.* **1983**, *13*, 135.
7. Guo, Z.; Schultz, A. G. *J. Org. Chem.* **2001**, *66*, 2154.

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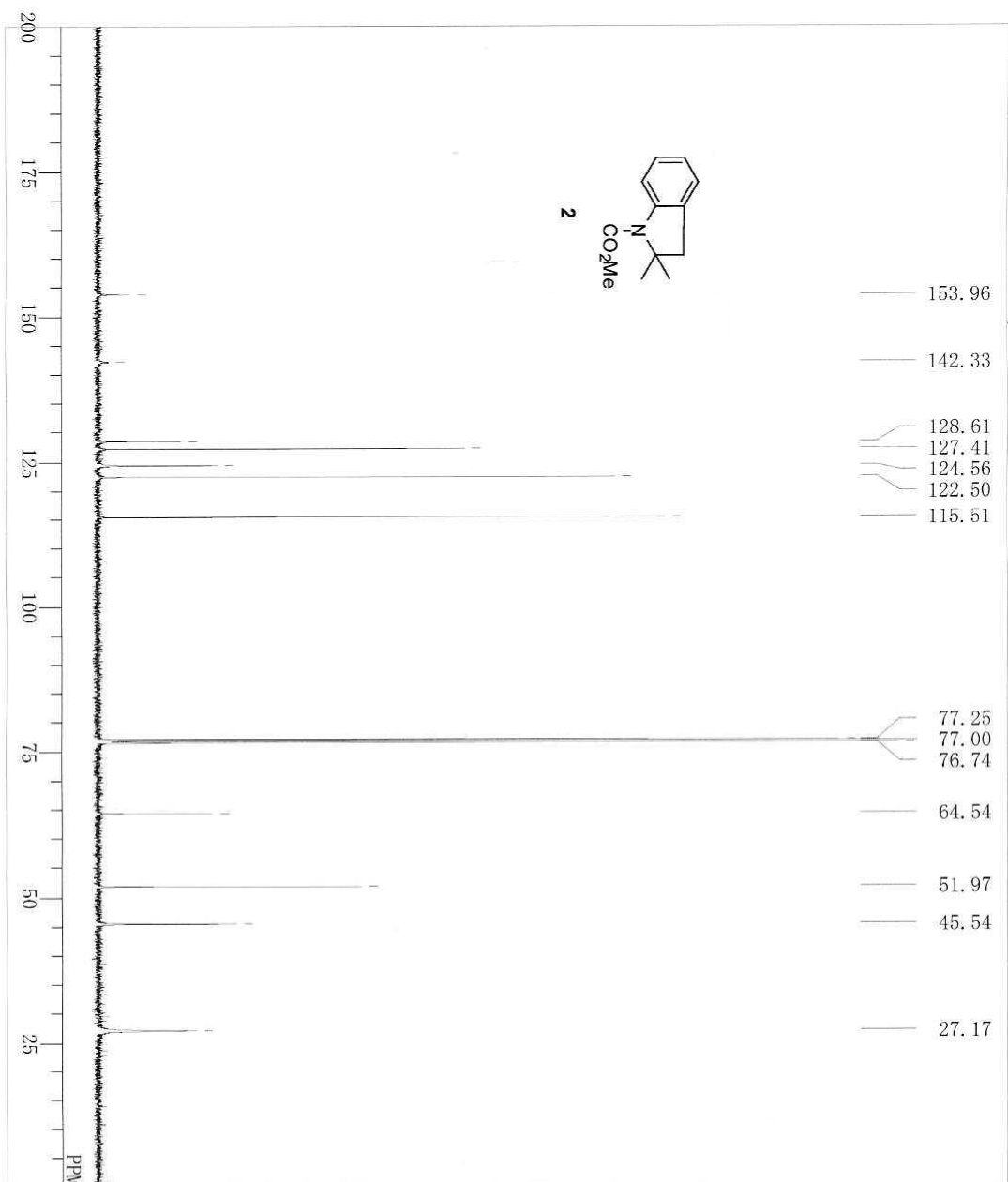
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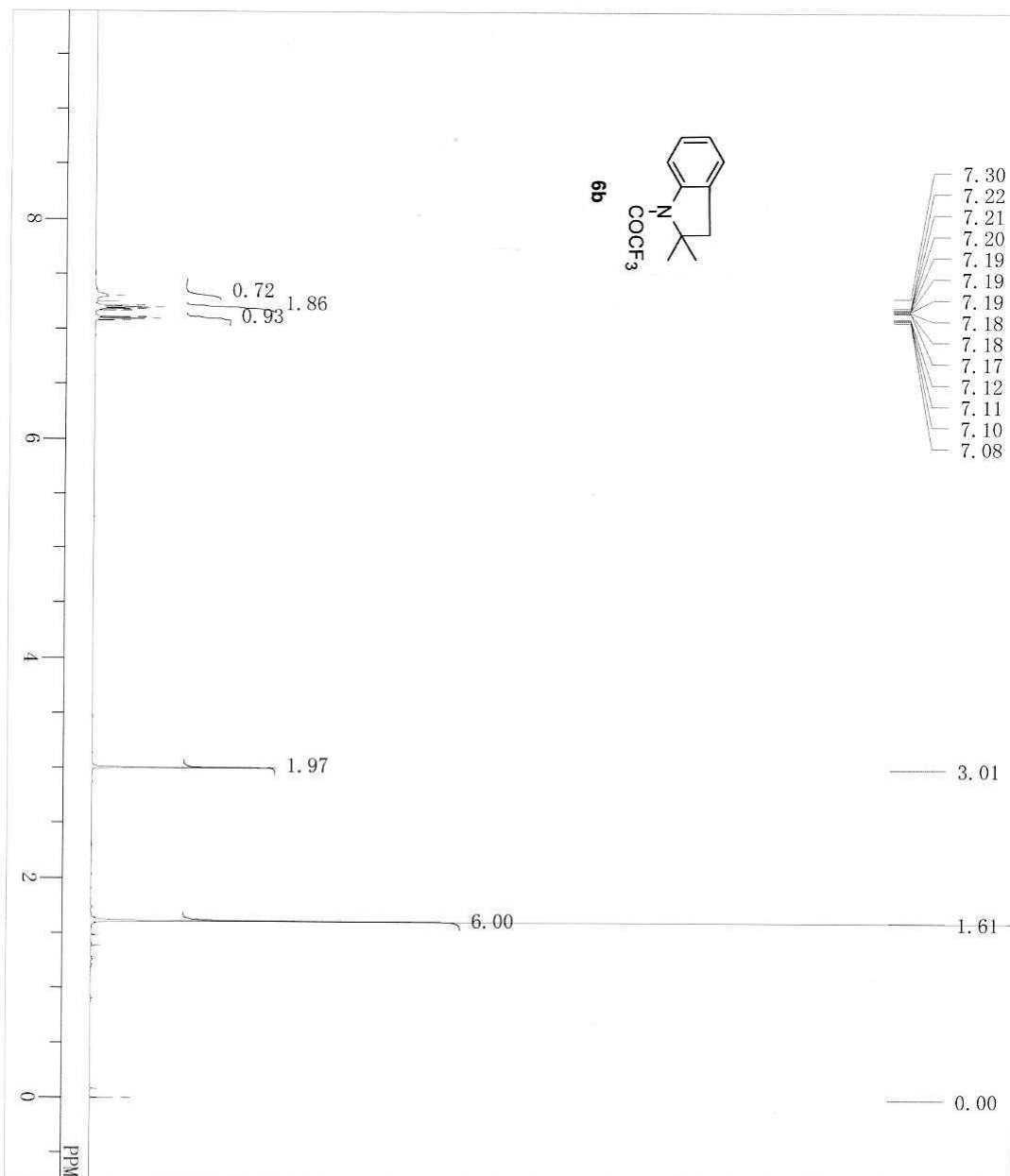
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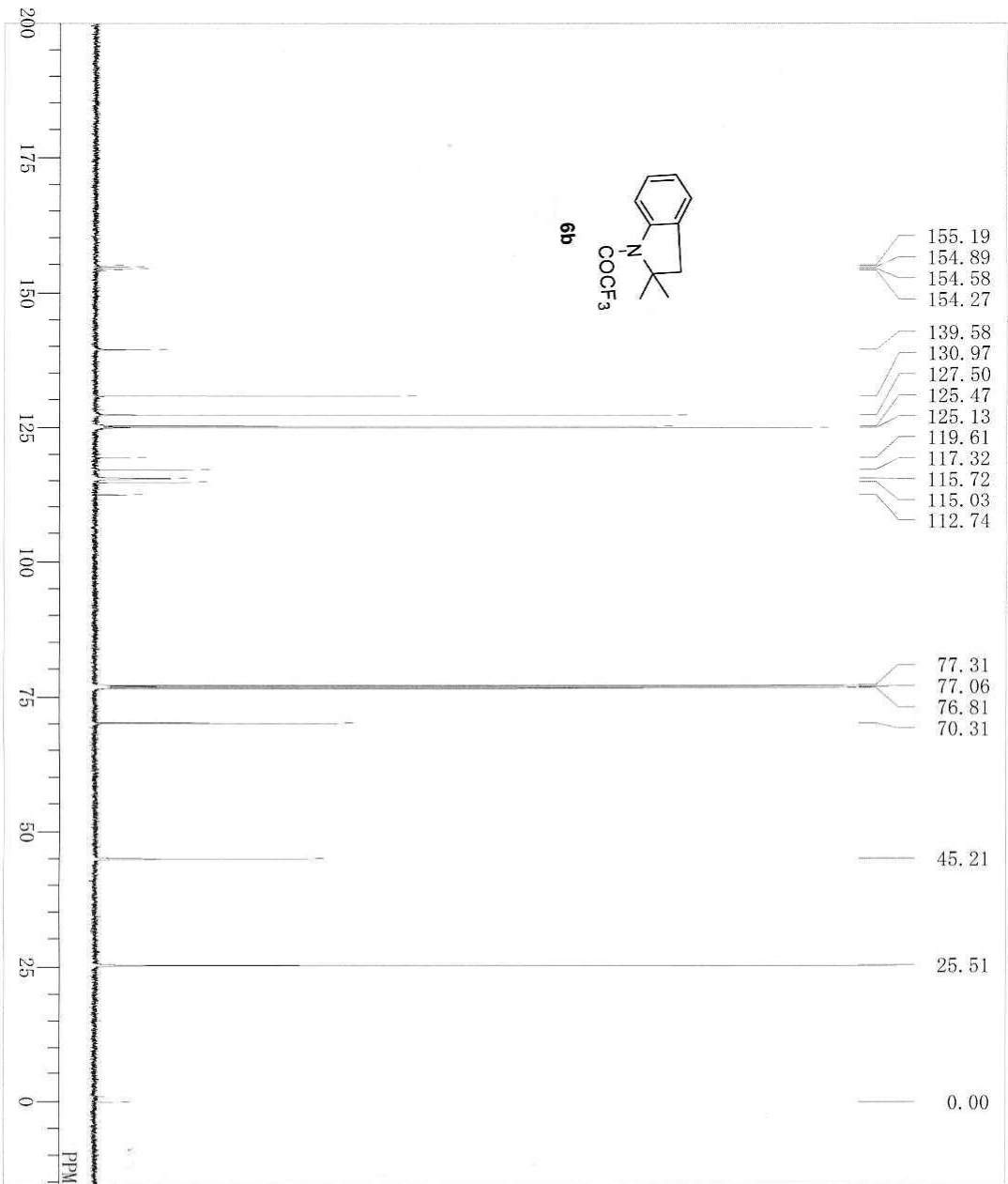
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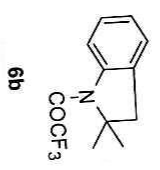
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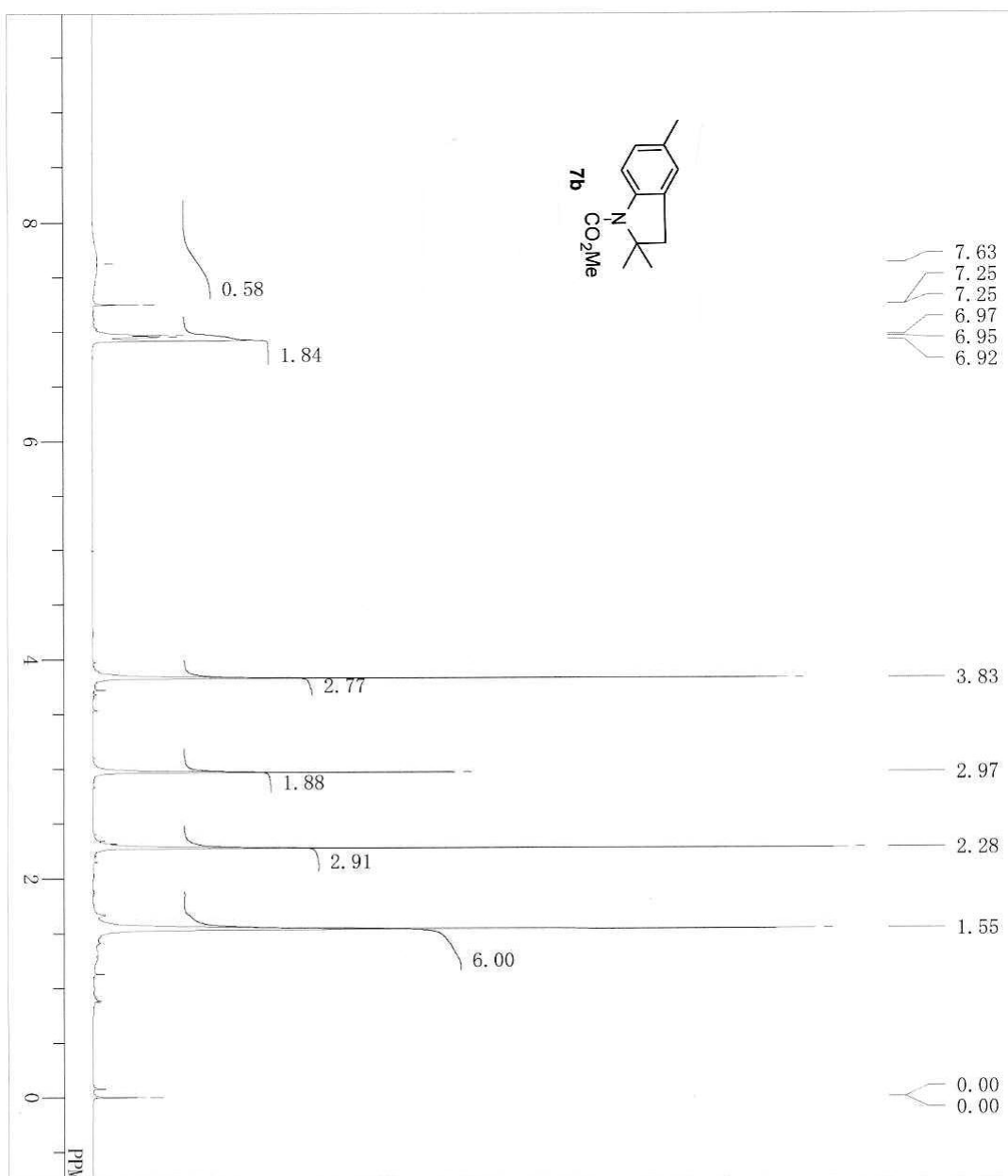


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ORNUC 13C
EXMOD single_pulse_dec
ORPRQ 125.77 MHz
ORSET 7.87 KHz
ORPIN 4.21 Hz
POINT 52428
FREQ 31446.06 Hz
SCANS 1200
ACQTM 1.6672 sec
PD 2.0000 sec
PW1 3.83 usec
IRNUC 1H
CTEMP 26.4 c
SLVNT CDCl3
EXREF 0.00 ppm
BF 1.20 Hz
RGAIN 58
    
```

single_pulse

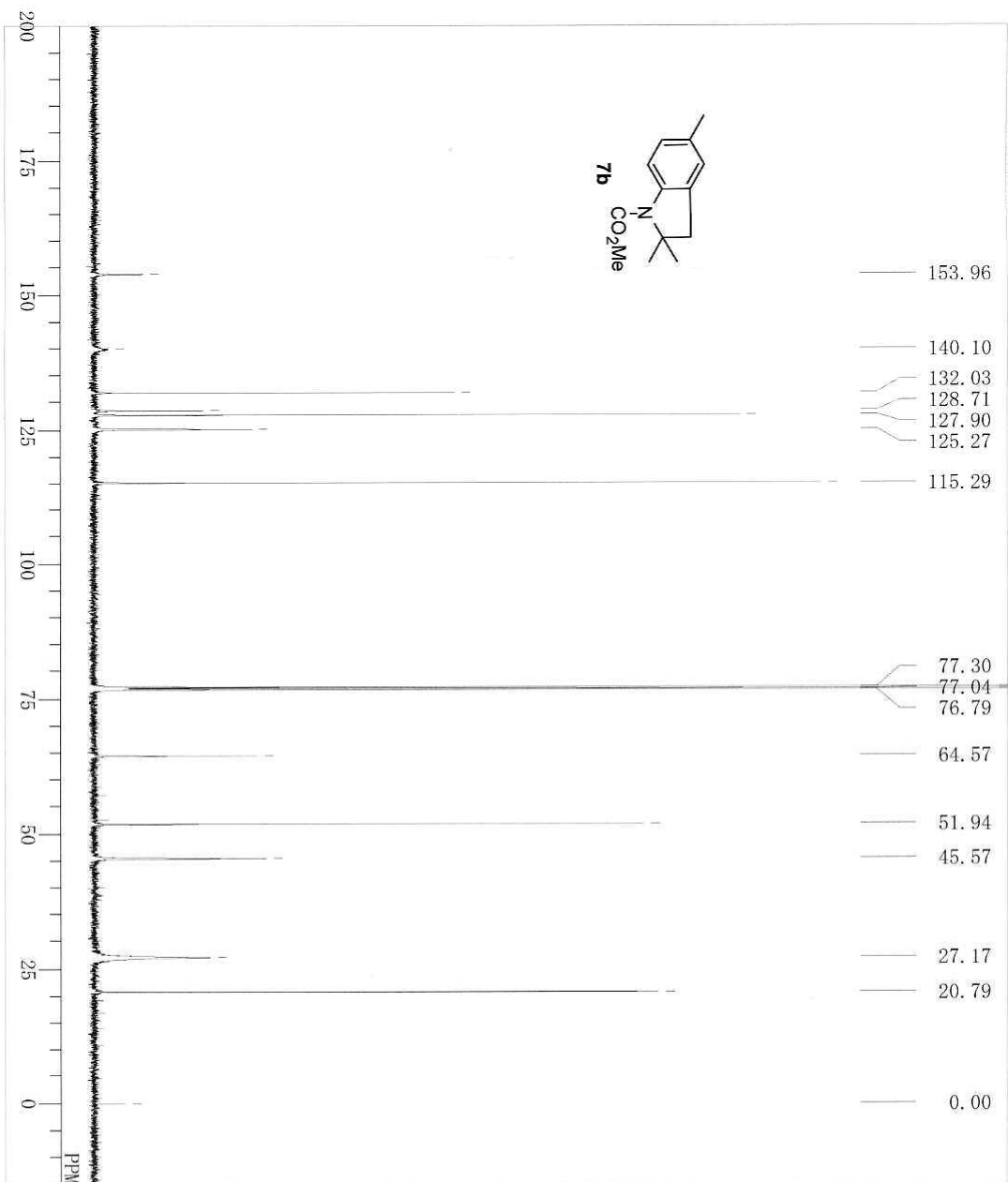
YYSeizo-wi129YSeizoYW NabeYsp3 C-H Br-YD-4 4-Me Pd IH.als



DF11E D-4 4-Me Pd IH.als
COMNT single_pulse
DATIM 07-11-2007 22:11:35
OBNUC IH
EXMOD single_pulse.ex2
OBFREQ 500.16 MHz
OBSSET 2.41 KHz
OBFIN 6.01 Hz
POINT 26214
FREQU 7507.39 Hz
SCANS 8
ACQTM 3.4918 sec
PD 5.0000 sec
PW1 6.25 usec
TRNUC IH
CTEMP 27.3 e
SLVNT CDCl₃
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 30

single pulse decoupled gated NOE

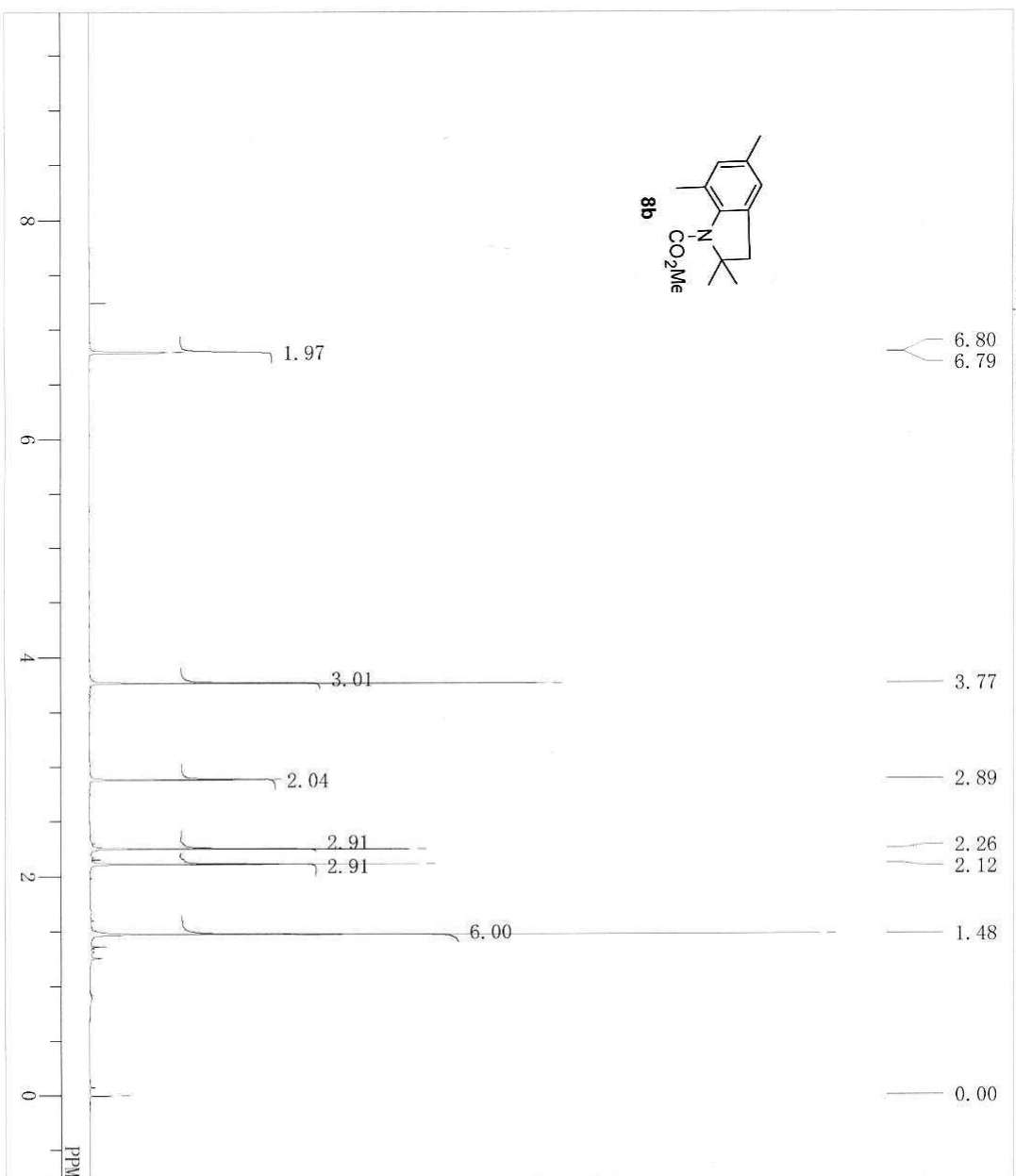
YYSeizo-wi29YseizoYW NabohSp3 C-H BrVD-4 4-Me Pd 13C.als



DF1ILE	D-4 4-Me Pd 13C. als
COMNT	single pulse decoupled gate
DATIM	07-11-2007 23:45:46
ORNUC	13C
EXMOD	single pulse dec
OBFRQ	125.77 MHz
OBSET	7.87 KHz
OBFTN	4.21 Hz
POINT	26214
FREQU	31446.06 Hz
SCANS	1980
ACQTM	0.8336 sec
PD	2.0000 sec
PW1	2.93 usec
IRNUC	1H
CTEMP	27.5 c
SLVNT	CDCL3
EXREF	0.00 ppm
BR	1.20 Hz
RGAIN	60

single_pulse

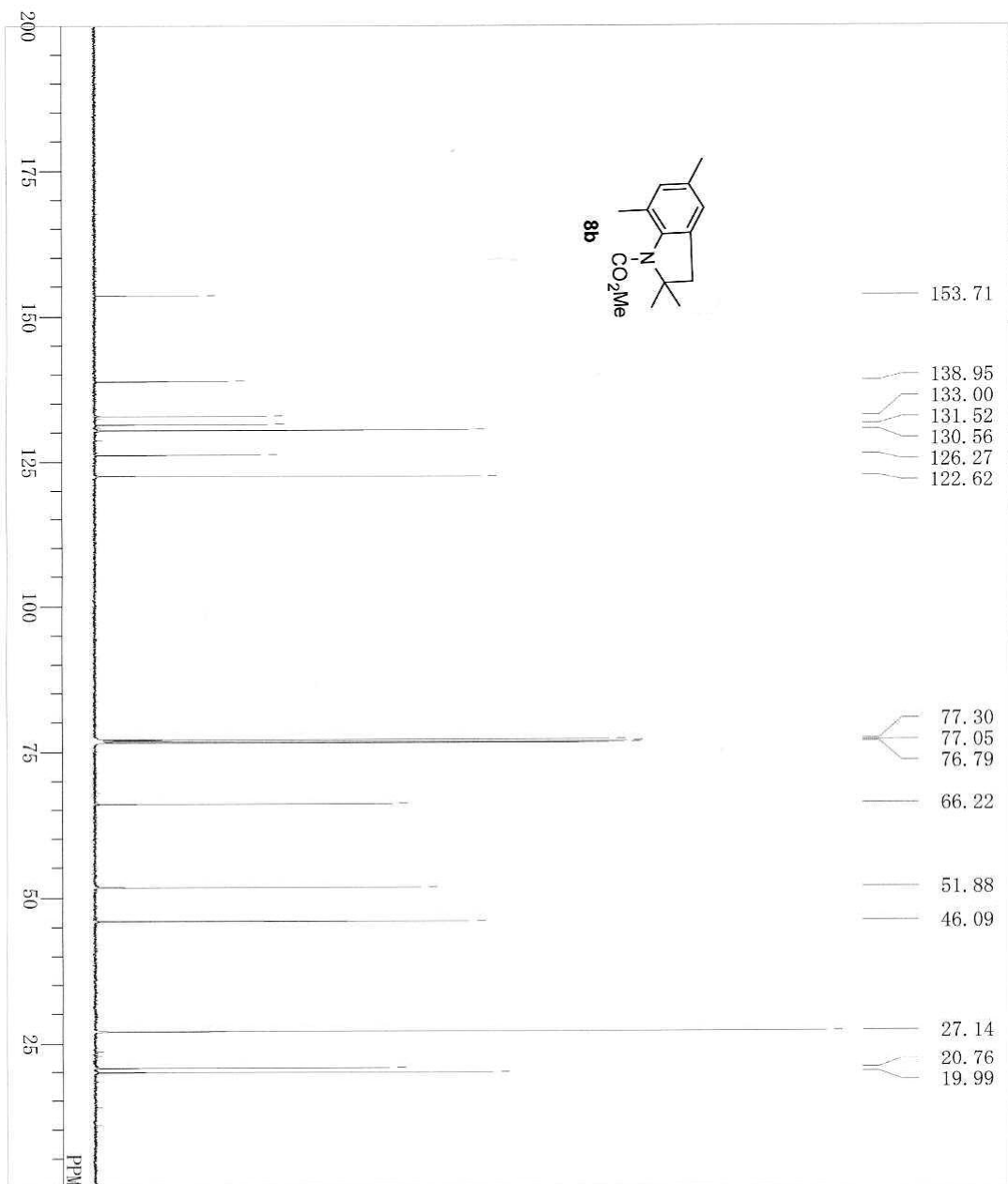
YSeizo-wi29Yseizow Nahofsp3 C-11 BrYc-188 DiMe Pd -1H.als



DF1LE C-188 DiMe Pd -1H.als
COMNT single_pulse
DATIM 31-10-2007 21:15:25
OBNLC IH
EXMOD single_pulse.ex2
OBFRR 500.16 MHz
OBSFT 2.41 KHz
OBFIN 6.01 Hz
POINT 52428
FREQU 7507.39 Hz
SCANS 8
ACQTM 6.9835 sec
PD 5.0000 sec
PW1 6.25 usec
IRNLC IH
CTEMP 26.7 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 30

single pulse decoupled gated NOE

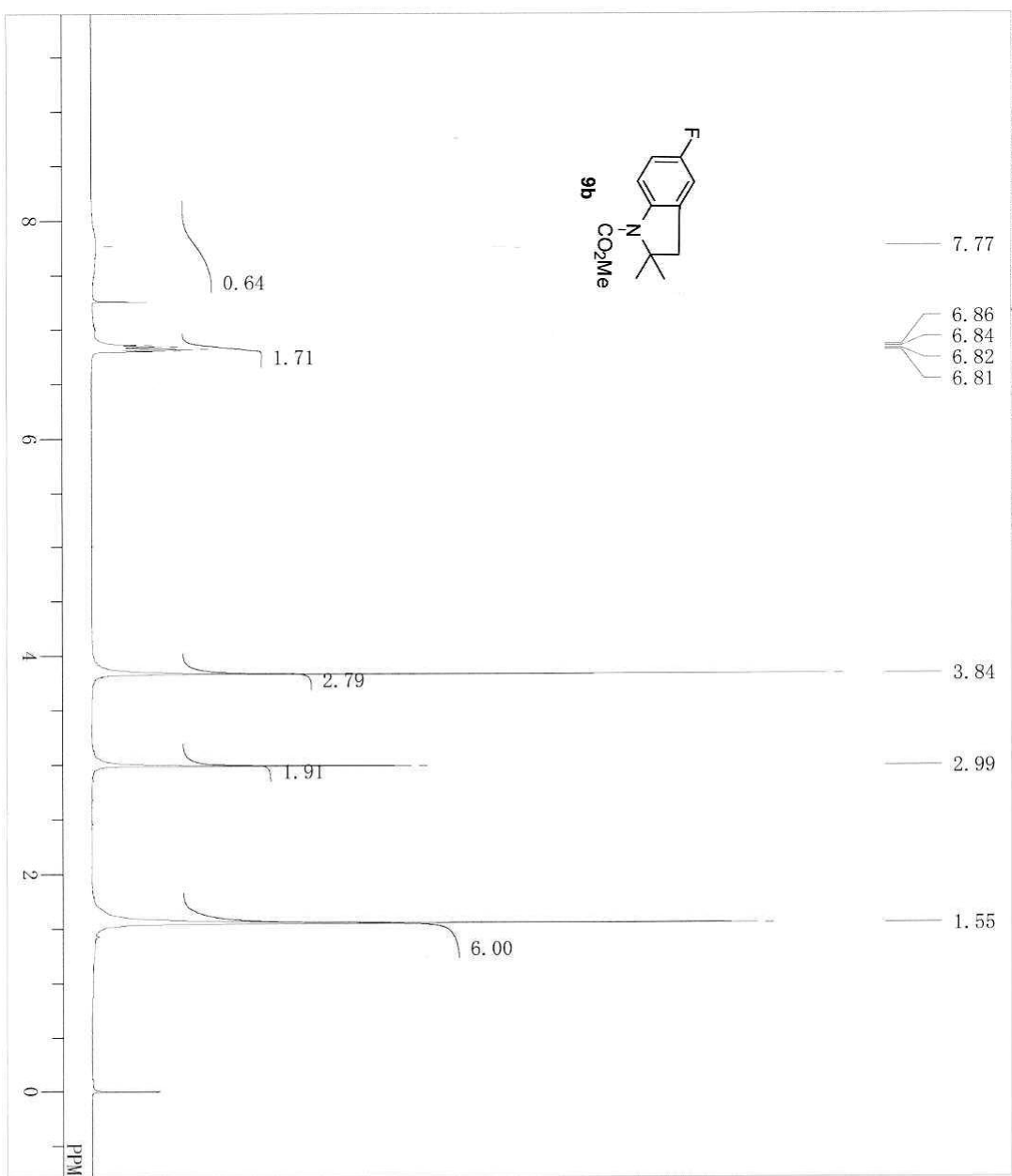
YYSelzo-wir29YselzoYW NaboVsp3 C-H B-YC-188 DiMe 13C Pd.a1s



DF1LE C-188 DiMe 13C Pd.a1s
 COMNT single pulse decoupled gate
 DATTM 31-10-2007 22:17:26
 ORNUC 13C
 EXMOD single pulse dec
 OBFRO 125.77 MHz
 OBSFT 7.87 KHz
 ORFTN 4.21 Hz
 POINT 52428
 FREQU 31446.06 Hz
 SCANS 1000
 ACQTM 1.6672 sec
 PD 2.0000 sec
 PW1 2.93 usec
 IRNUC 1H
 CTMP 26.8 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 1.20 Hz
 RGAIN 60

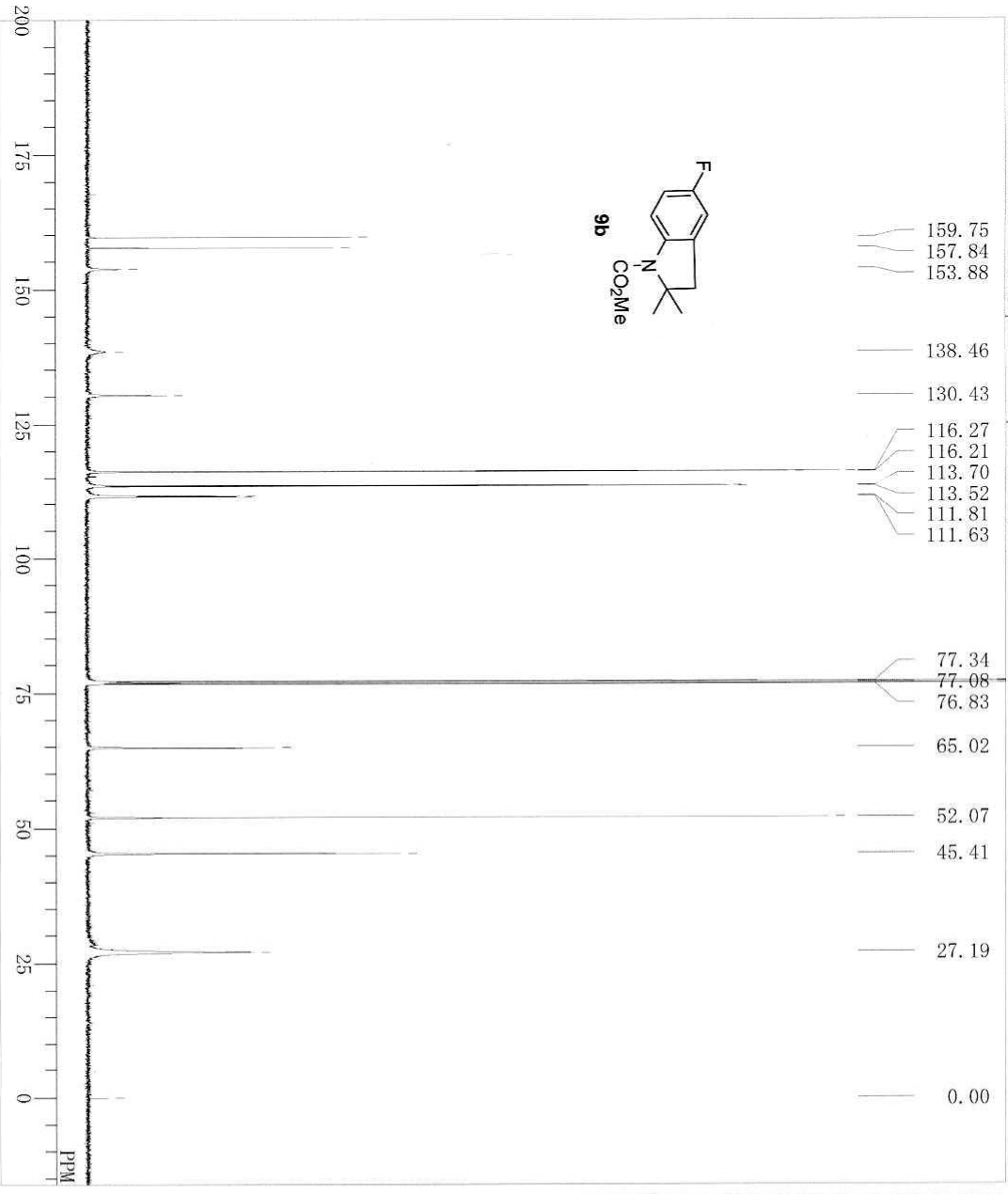
single_pulse

YSeizo-wi129YSeizoYW Nabey3p3 C-II Br-Yp-F Pd III.als



DFILE p-F Pd IH.als
COMT single_pulse
DATIM 01-11-2007 14:20:46
OBNUC IH
EXMOD single_pulse.ex2
OBFREQ 500.16 MHz
OBSET 2.41 KHz
OBFILN 6.01 Hz
POINT 52428
FREQU 7507.39 Hz
SCANS 8
ACQTM 6.9835 sec
PD 5.0000 sec
PWI 6.25 usec
TRNUC IH
CTEMP 25.4 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 32

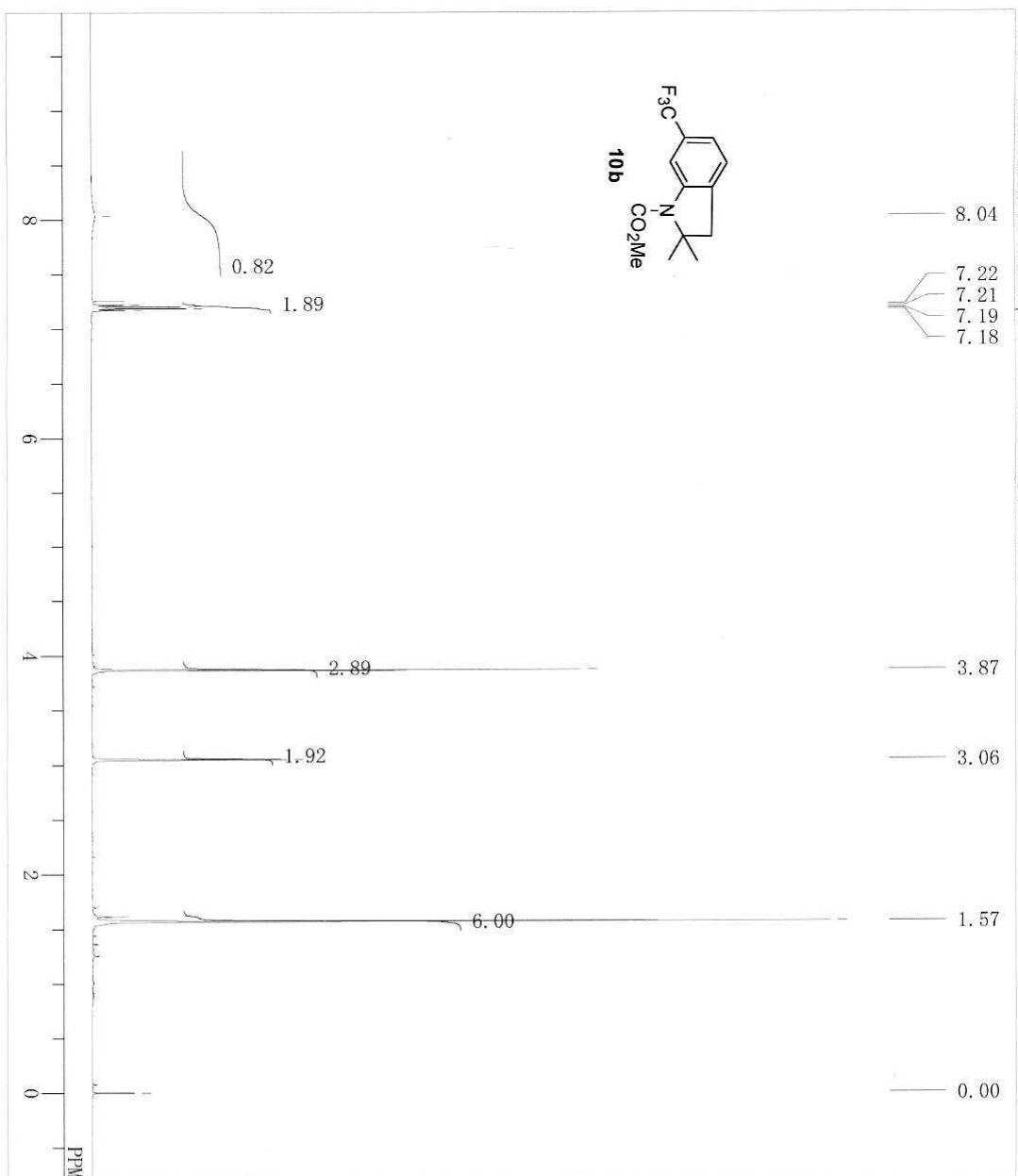
single pulse decoupled gated NOE
 YYSeizo-wi129YseizoYW Nabey'sp3 C-H B-Yp-F 13C Pd.als



DF1LE p-F 13C Pd.als
 COMNT single pulse decoupled gate
 DATIM 22-12-2007 03:49:25
 OBNUC 13C
 EXMOD single pulse dec
 OBFRQ 125.77 MHz
 OBSFT 7.87 KHz
 OBFIN 4.21 Hz
 POINT 52428
 FREQU 31446.06 Hz
 SCANS 5000
 ACQTM 1.6672 sec
 PD 2.0000 sec
 PW1 3.83 usec
 TRNUC 1H
 CTEMP 26.6 c
 SLVNT CDCl3
 EXREF 0.00 ppm
 BF 1.20 Hz
 RGAIN 60

single_pulse

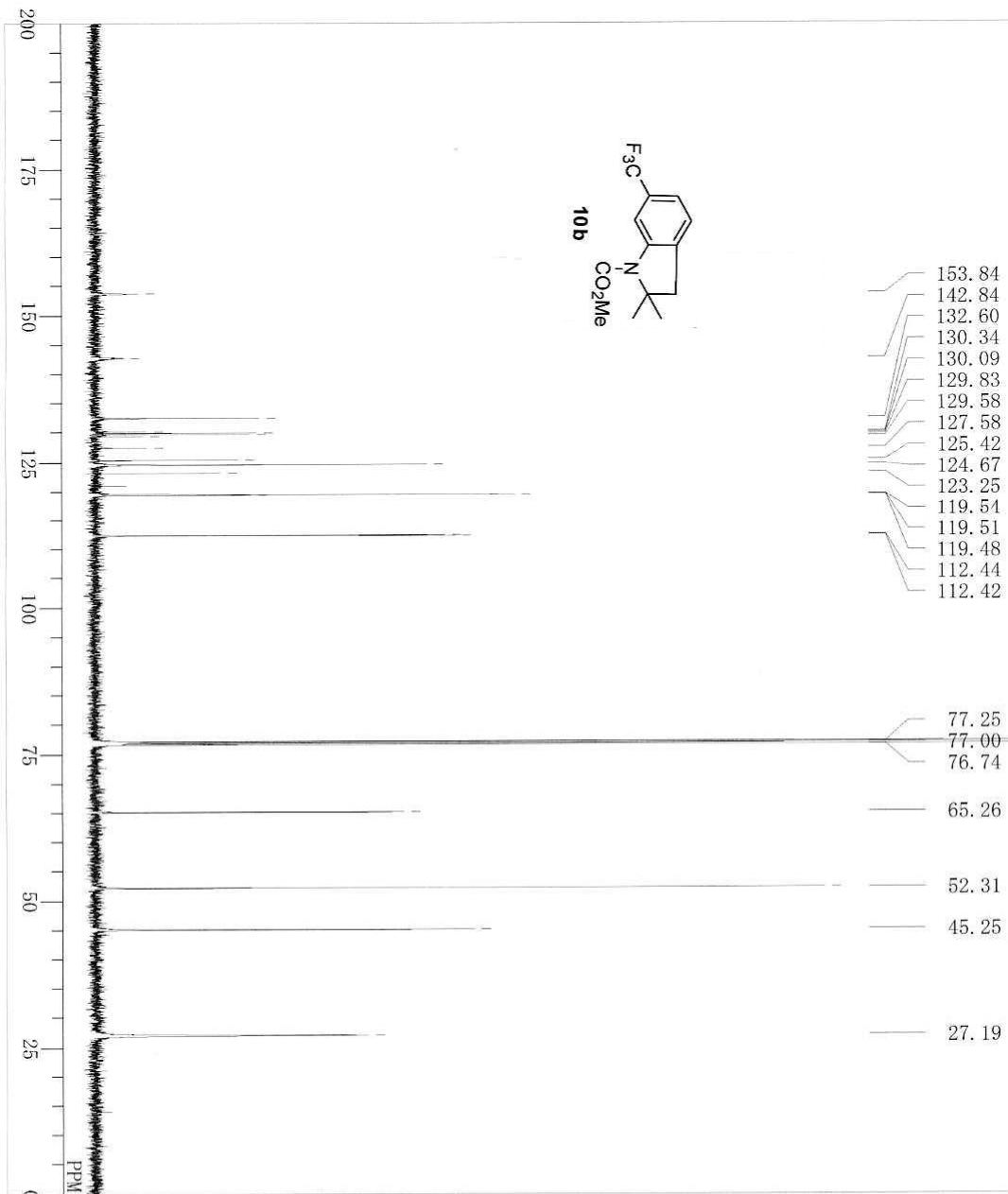
YSeizo-wi29YSeizoYW NabehSp3 C-11 B-YD-1 CF3 Pd IH.als



DPFILE	D-1 CF3 Pd IH.als
COMNT	single_pulse
DATEM	07-11-2007 15:30:28
OBNUC	IH
EXMOD	single_pulse.ex2
OBFRQ	500.16 MHz
OBSET	2.41 KHz
OBFIN	6.01 Hz
POINT	104857
FREQU	7507.46 Hz
SCANS	4
ACQTM	13.9670 sec
PD	5.0000 sec
PW1	6.25 usec
IRNUC	IH
CTEMP	26.0 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.12 Hz
RGAIN	30

single pulse decoupled gated NOE

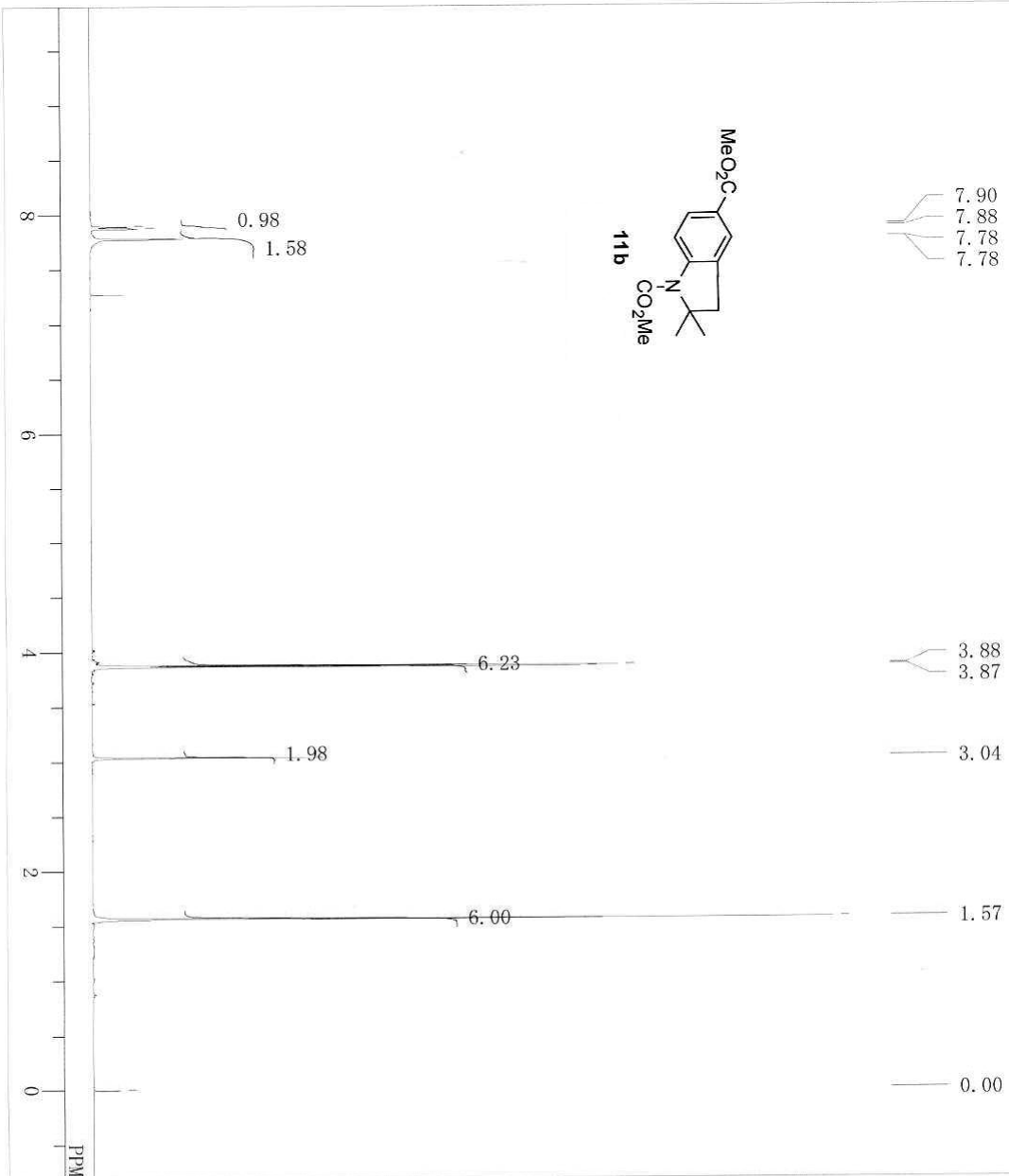
YYSelzo-wir29YselzoYW NabeYsp3 C-H BrYD-1 CF3 Pd 13C. als



DF11E D-1 CF3 Pd 13C. als
 COMMENT single pulse decoupled gate
 DATIM 07-11-2007 16:25:27
 OBNUC 13C
 EXMOD single pulse dec
 OBFREQ 125.77 MHz
 OBSSET 7.87 KHz
 OBFILN 4.21 Hz
 POINT 26214
 FREQUL 31446.06 Hz
 SCANS 1150
 ACQTM 0.8336 sec
 PD 2.0000 sec
 PW1 2.93 usec
 IRNUC 1H
 CTMPP 26.6 c
 SLVNT CDCL3
 EXREF 77.00 ppm
 BR 1.20 Hz
 RGAIN 60

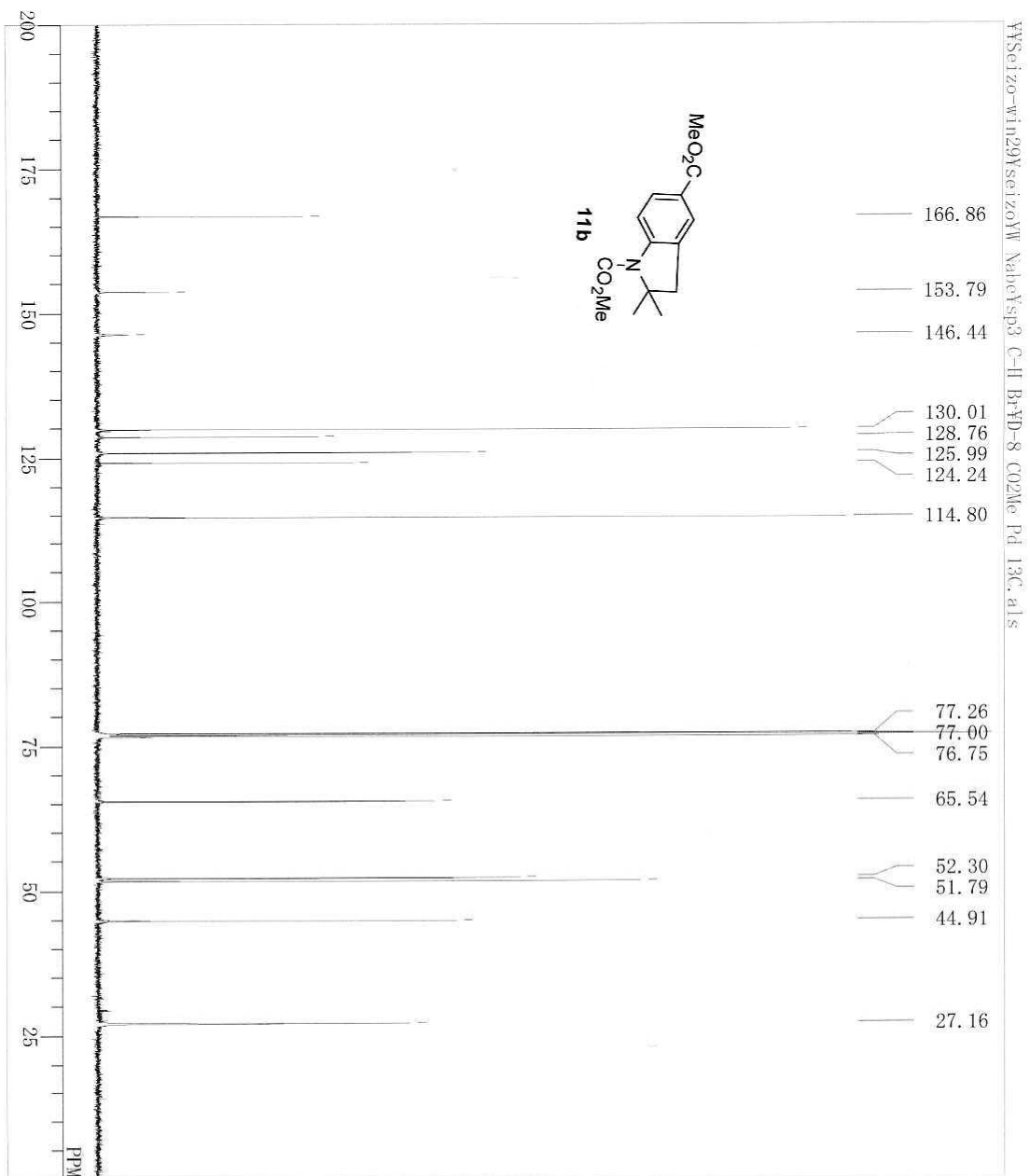
single_pulse

Y:\Seizo-win29\Seizo\W_NabeYsp3 C-H BrYD-8 CO2Me Pd 1H.a1s



DFILE D-8 CO2Me Pd 1H.a1s
COMNT single_pulse
DATIM 09-11-2007 16:03:51
OBNUC 1H
EXMOD single_pulse.ex2
OBFPRQ 500.16 MHz
OBSFET 2.41 KHz
OBF1IN 6.01 Hz
POINT 52428
FREQU 7507.39 Hz
SCANS 8
ACQTM 6.9835 sec
PD 5.0000 sec
PW1 6.25 usec
IRNUC 1H
CTEMP 26.1 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 30

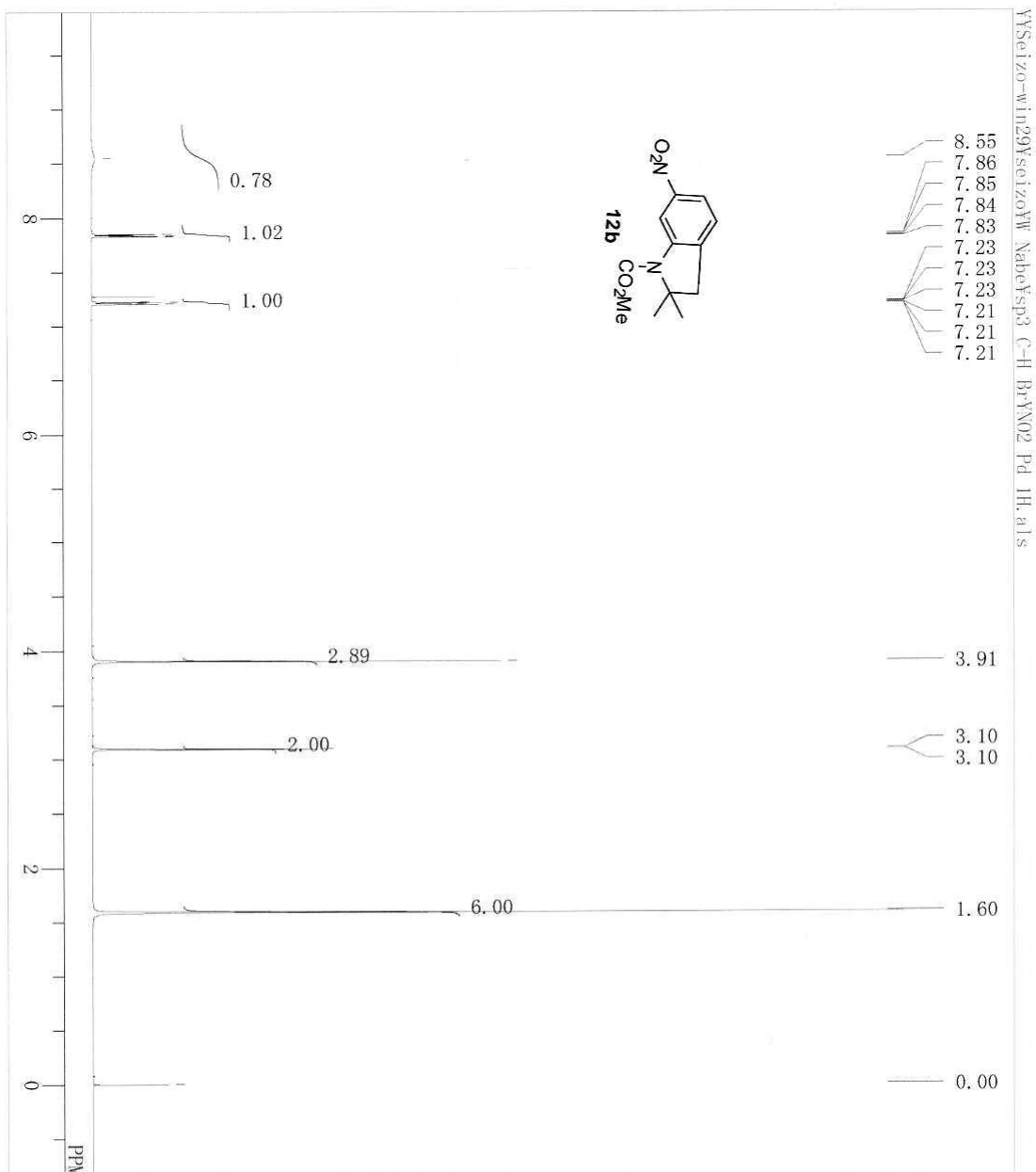
single pulse decoupled gated



```

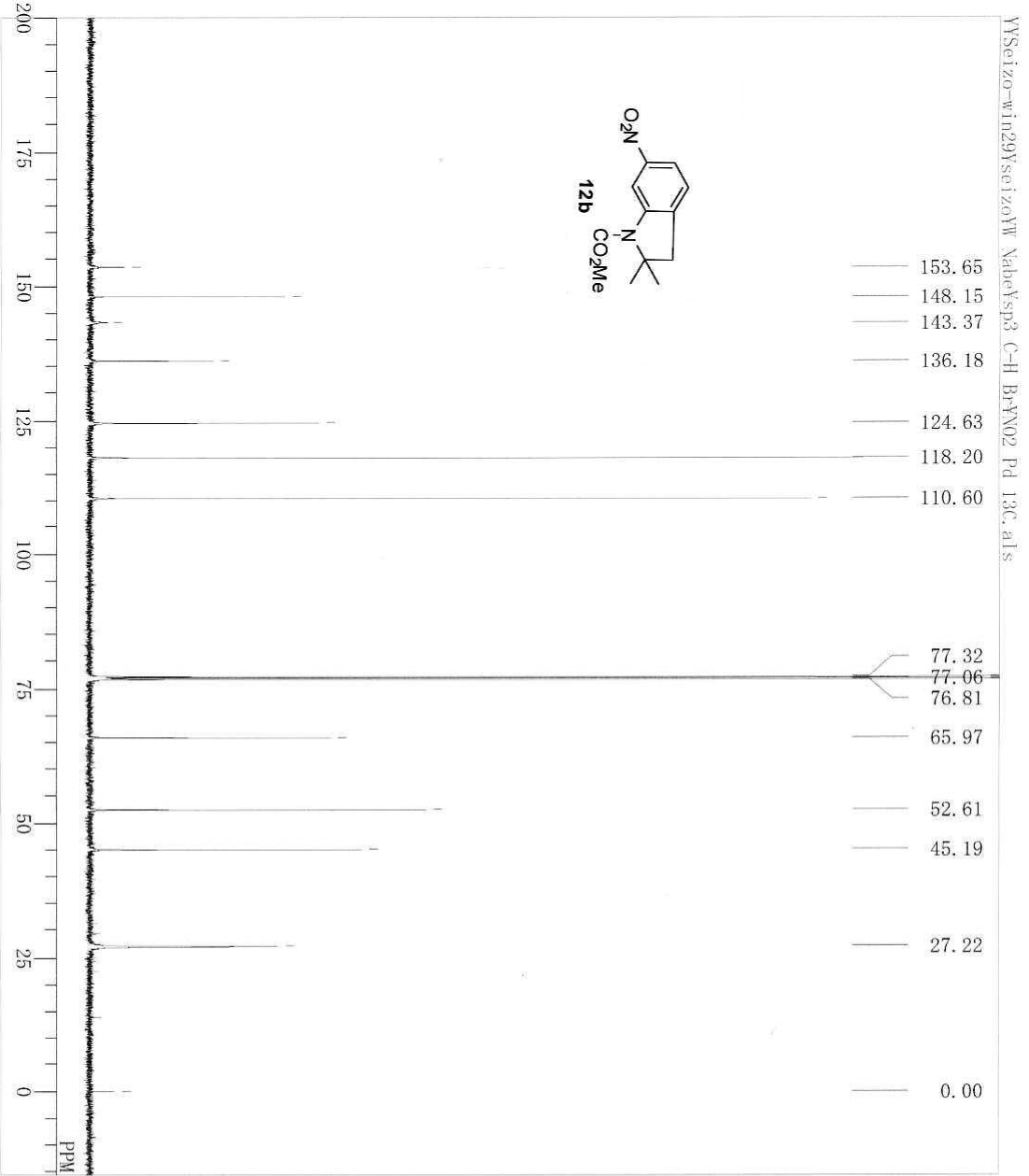
DPF11E D-8 CO2Me Pd 13C.als
COMNT single pulse decoupled gate
DATIM 09-11-2007 16:50:14
OBNUC 13C
EXMOD single_pulse_dec
OBFREQ 125.77 MHz
OBSEFT 7.87 KHz
OBF1IN 4.21 Hz
POINT 26214
FREQU 31446.06 Hz
SCANS 967
ACQTM 0.8336 sec
PD 2.0000 sec
PW1 2.93 usec
IRNDC IH
CTEMP 26.2 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 1.20 Hz
RGAIN 60
  
```

single_pulse



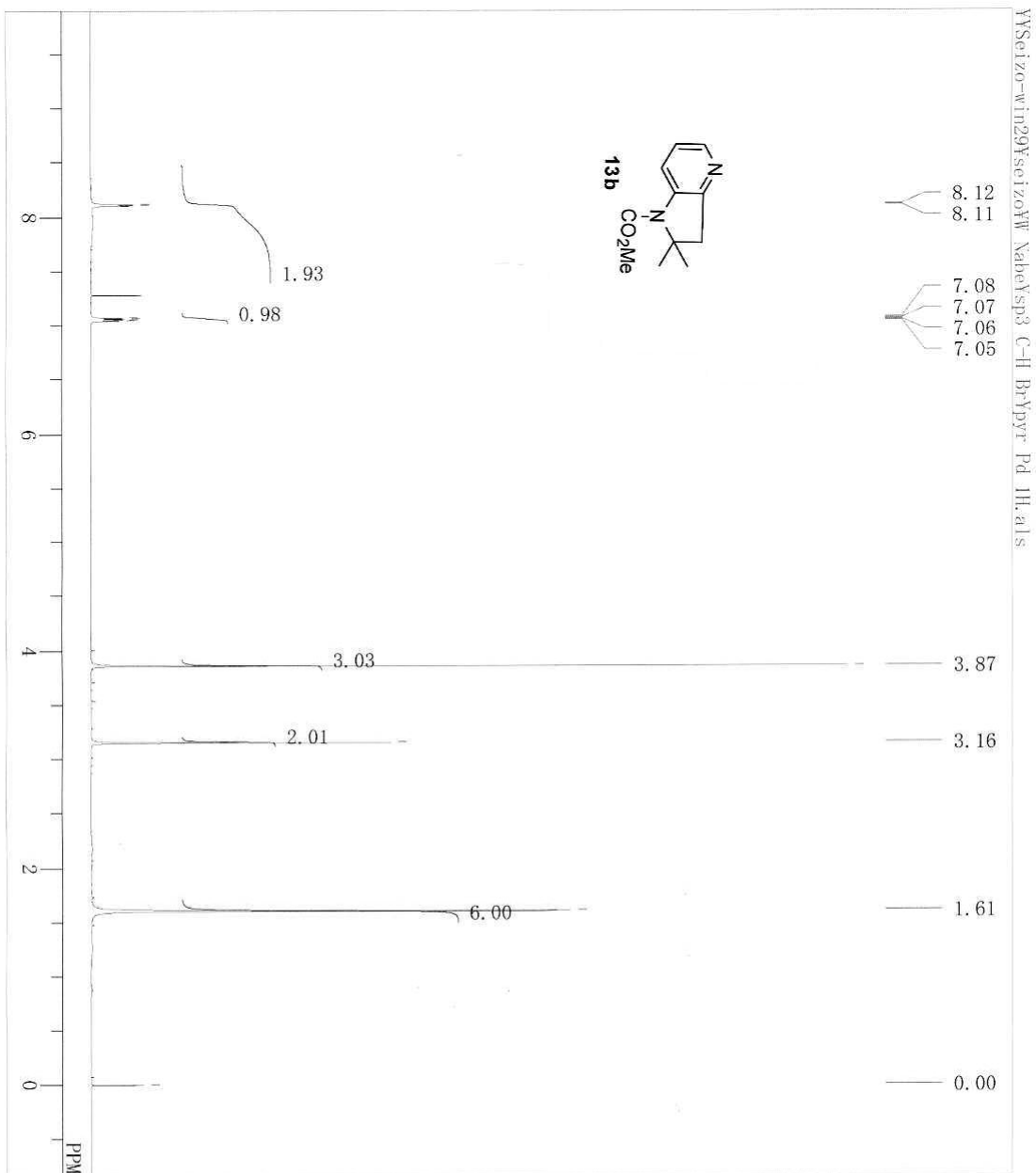
DFILE NO2_Pd_1H.a1.s
COMMENT single_pulse
DATIM 14-11-2007 22:01:44
OBNUC IH
EXMOD single_pulse_ex2
OBRFQ 500.16 MHz
OBSET 2.41 KHz
OBFIN 6.01 Hz
POINT 52428
FREQU 7507.39 Hz
SCANS 8
ACQTM 6.9835 sec
PD 5.0000 sec
PW1 6.05 usec
IRNUC IH
CTEMP 24.3 c
SLVNT CDCl3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 34

single pulse decoupled gated NOE



DF11E NO2 Pd 13C.als
COMNT single pulse decoupled gate
DATIM 14-11-2007 23:43:31
OBNUC 13C
EXMOD single_pulse_dec
OBFREQ 125.77 MHz
OBSSET 7.87 KHz
OBFIN 4.21 Hz
POINT 52428
FREQU 31446.06 Hz
SCANS 1650
ACQTM 1.6672 sec
PD 2.0000 sec
PWI 3.83 usec
TRNUC 1H
CTEMP 24.4 c
SLVNT CDCl3
EXREF 0.00 ppm
BR 1.20 Hz
RGAIN 56

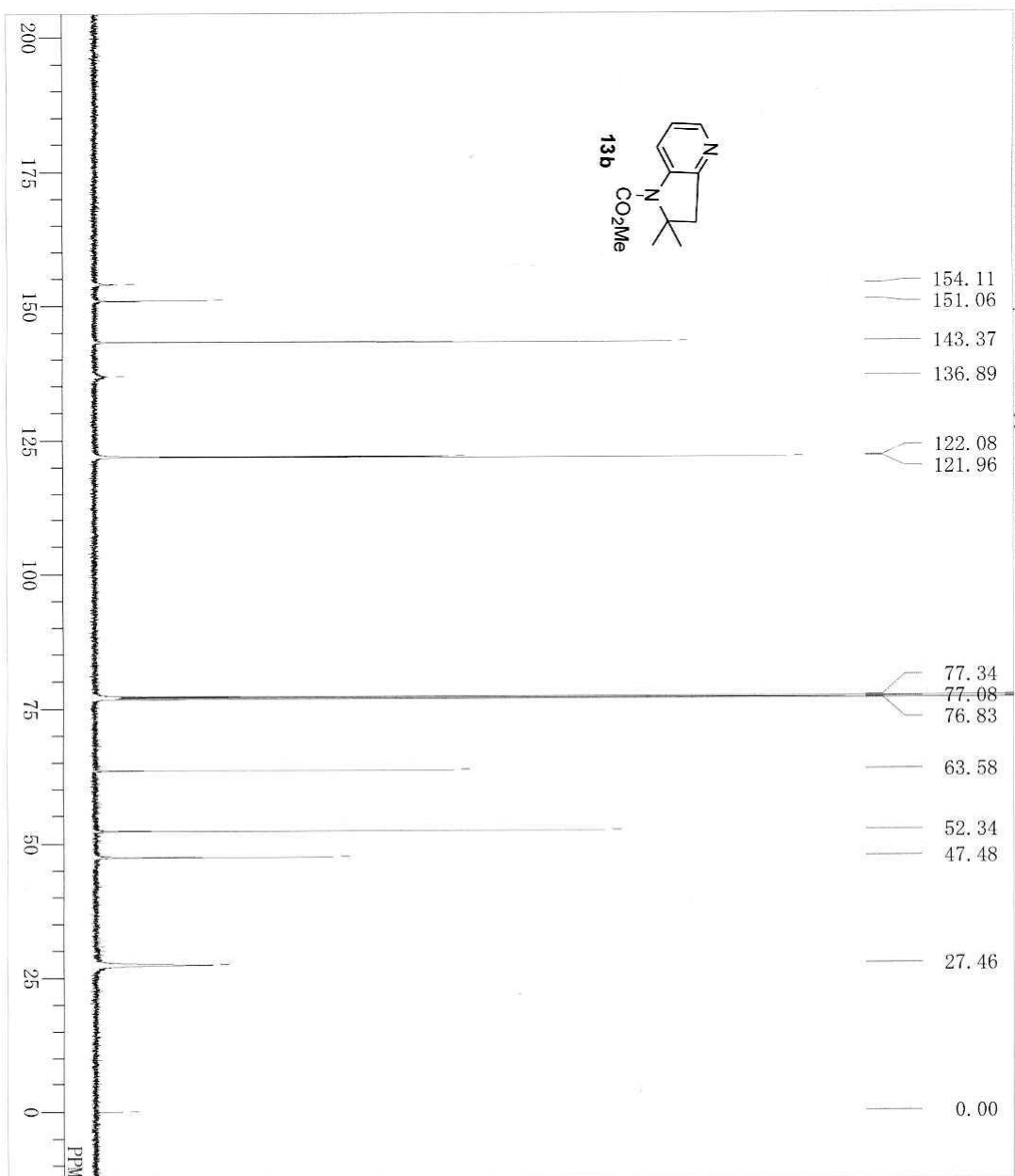
single_pulse



DFILE pyr Pd IH.als
COMNT single_pulse
DATIM 04-12-2007 00:03:41
OBNUC IH
EXMOD single_pulse.ex2
OBFRRQ 500.16 MHz
OBSSET 2.41 KHz
OBFIN 6.01 Hz
POINT 52428
FREQU 7507.39 Hz
SCANS 8
ACQTM 6.9835 sec
PD 5.0000 sec
PW1 6.05 usec
IRNUC IH
CTEMP 28.6 c
SLVNT CDCl3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 34

single pulse decoupled gated NOE

Y:\Seizo-w\1n29Y\seizo\W Nahel\sp3 C-II Br\pyr Pd 13C.als

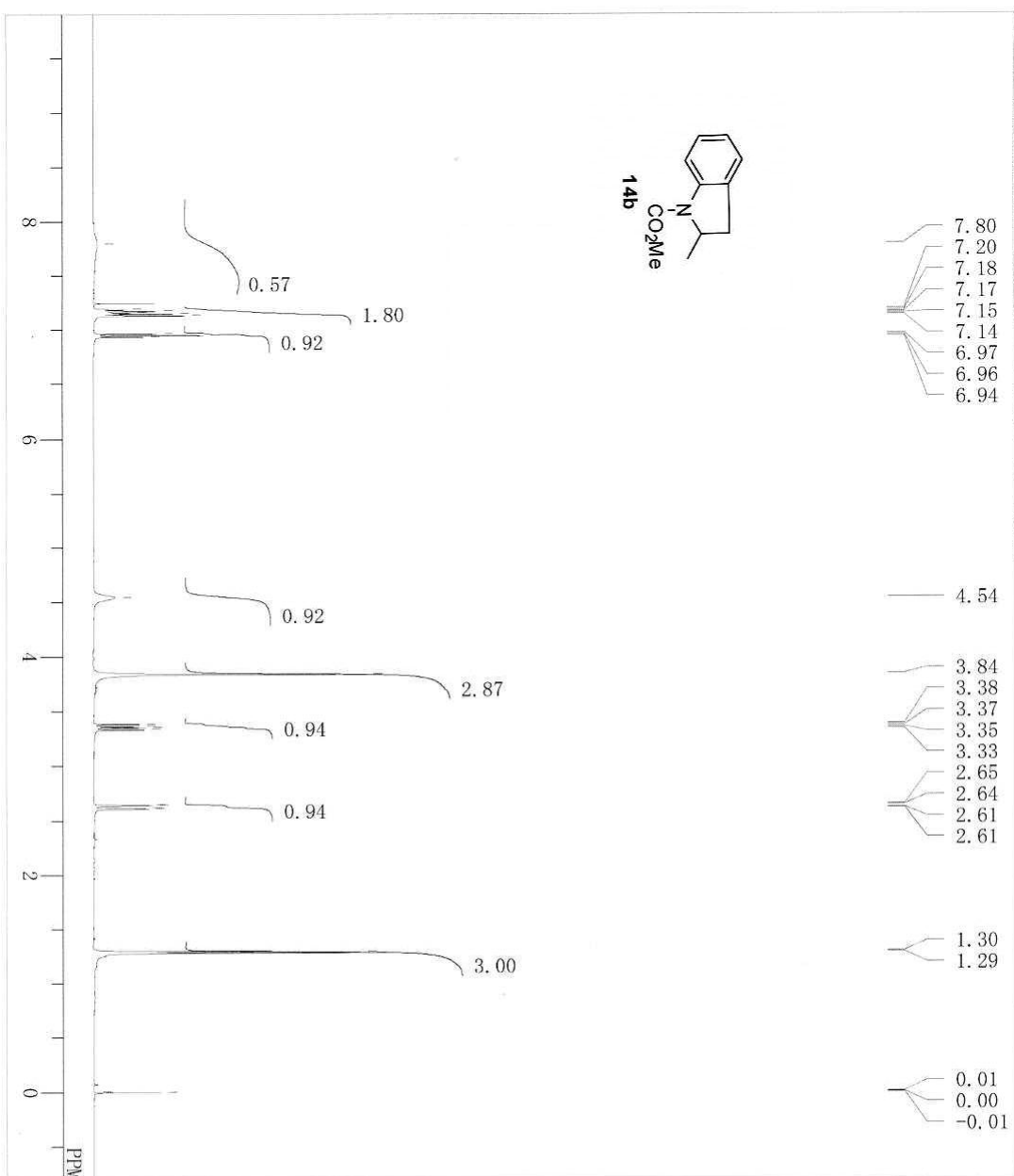


```

DEFILE pyr Pd 13C.als
COMNT single pulse decoupled gate
DATIM 04-12-2007 03:07:57
OBNDUC 13C
EXMOD single_pulse_dec
OBFREQ 125.77 MHz
OBSFET 7.87 KHz
OBFIN 4.21 Hz
POINT 52428
FREQU 31446.06 Hz
SCANS 3000
ACQTM 1.6672 sec
PD 2.0000 sec
PW1 3.83 usec
IRNUC 1H
CTEMP 28.9 c
SLVNT CDCl3
EXREF 0.00 ppm
BF 1.20 Hz
RGAIN 60
  
```

single_pulse

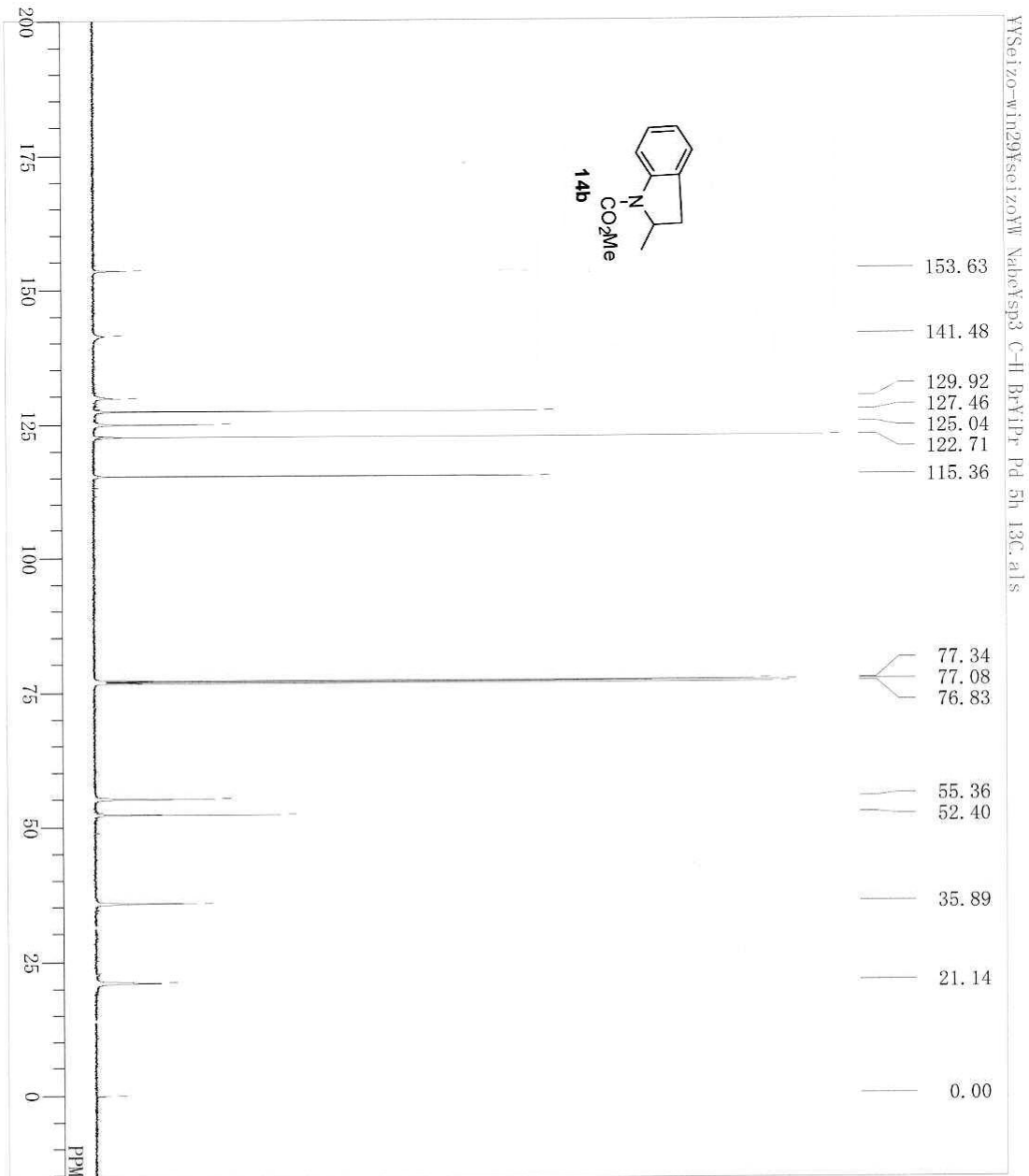
YSeizo-wt129YSeizoYW NabeYsp3 C-H Br-YIPr Pd IH.als



```

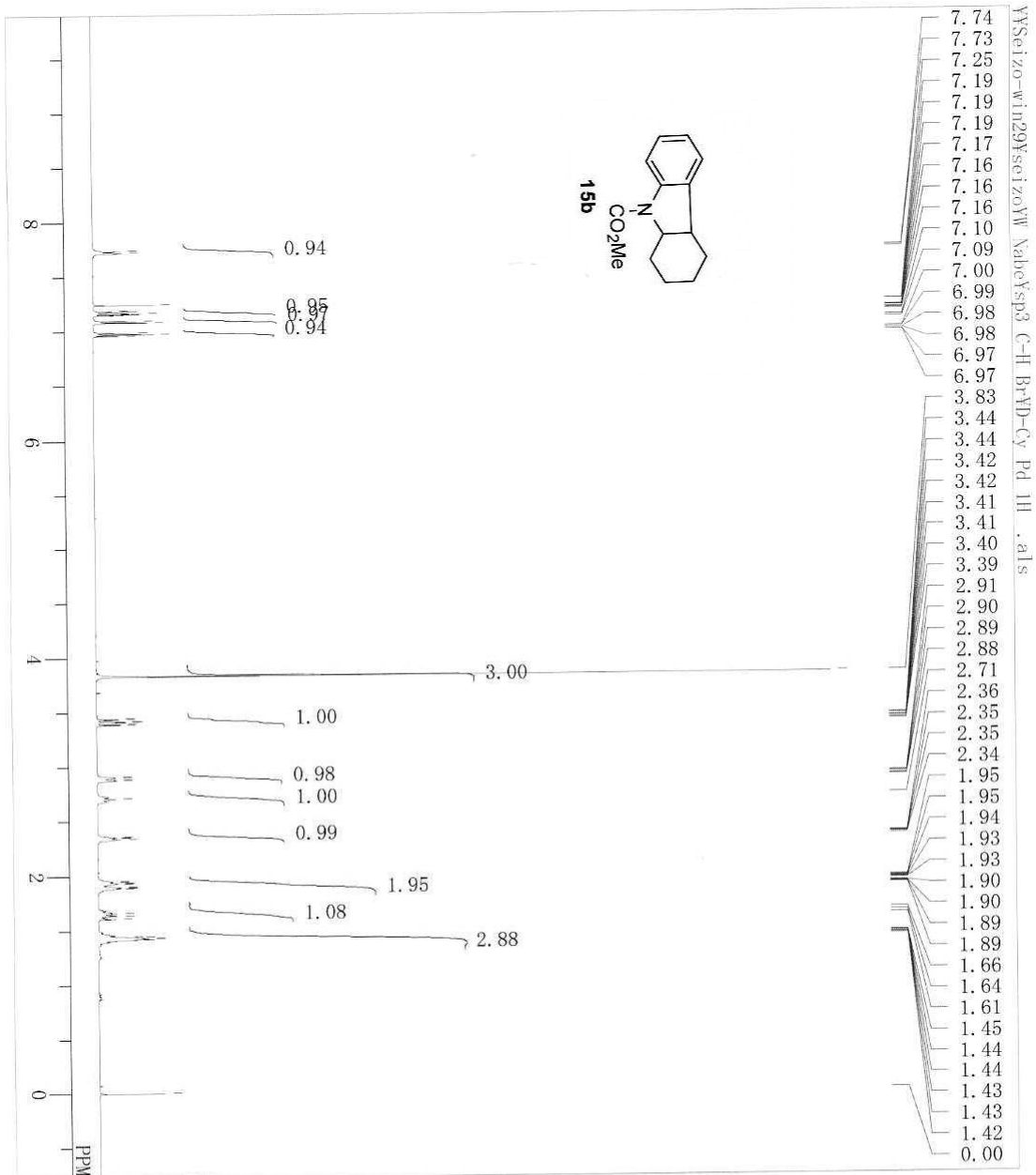
DF1ILE  iPr Pd IH.als
COMNT  single_pulse
DATIM  09-11-2007 12:22:19
OBNDC  IH
EXMOD  single_pulse.ex2
OBFPRQ 500.16 MHz
OBSET  2.41 KHz
OBFIN  6.01 Hz
POINT  52428
FREQU  7507.39 Hz
SCANS  8
ACQTM  6.9835 sec
Pd      5.0000 sec
PW1    6.25 usec
IRNUC  IH
CTEMP  26.7 c
SIVNT  CDCL3
EXREF  0.00 ppm
BF     0.12 Hz
RGAIN  30
  
```

single pulse decoupled gated NOE



DFILE iPr Pd 5h 13C.als
 COMNT single pulse decoupled gate
 DATIM 20-12-2007 02:56:41
 OBNUC 13C
 EXMOD single pulse dec
 OBRFQ 125.77 MHz
 OBSST 7.87 KHz
 OBRFN 4.21 Hz
 POINT 52428
 FREQU 31446.06 Hz
 SCANS 4800
 ACQTM 1.6672 sec
 PD 2.0000 sec
 PW1 3.83 usec
 IRNUC 1H
 CTEMP 26.5 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BR 1.20 Hz
 RGAIN 60

single_pulse

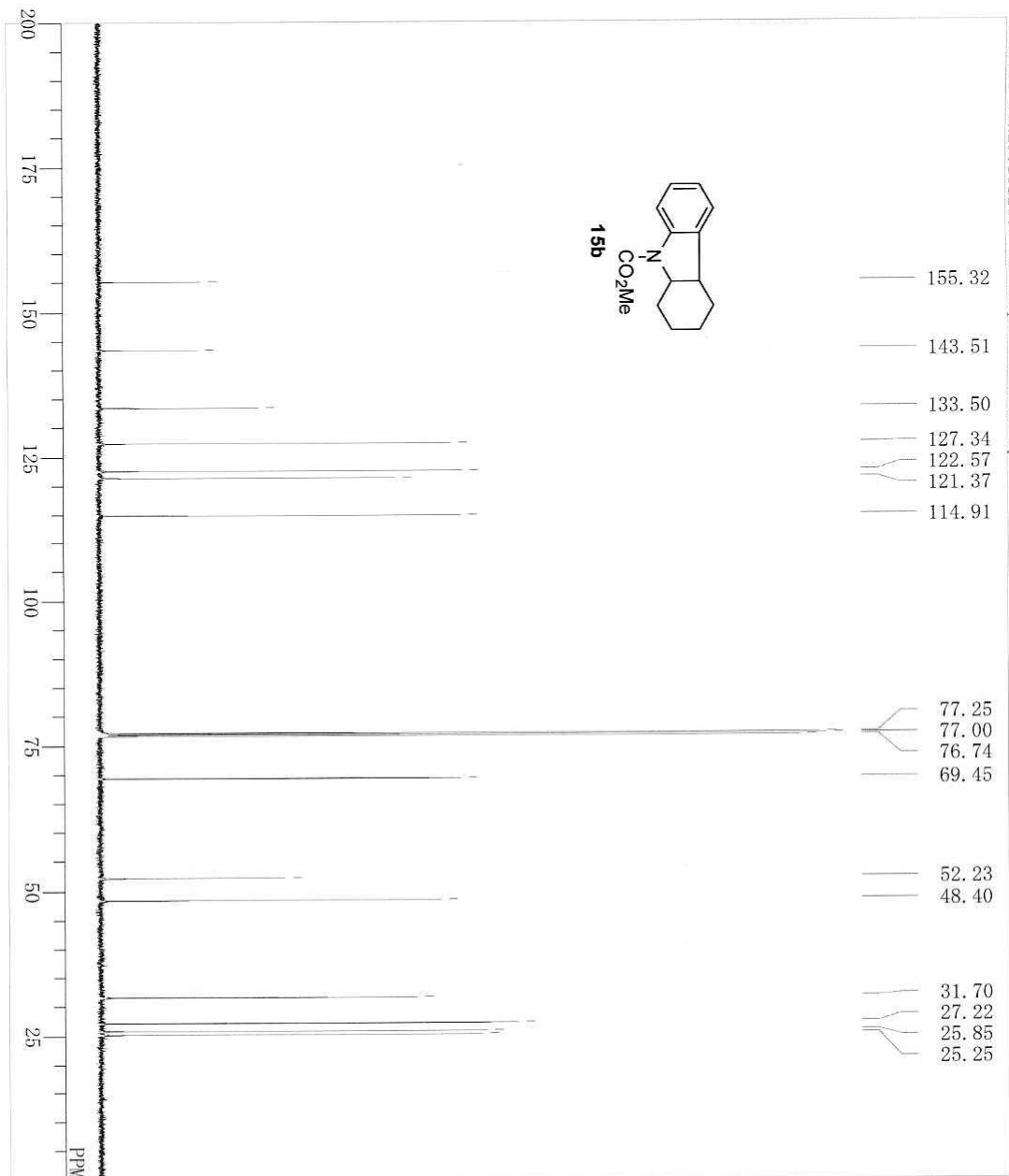


7.74
7.73
7.25
7.19
7.19
7.17
7.16
7.16
7.10
7.09
7.00
6.99
6.98
6.98
6.97
6.97
3.83
3.44
3.44
3.42
3.42
3.41
3.41
3.40
3.39
2.91
2.90
2.89
2.88
2.71
2.36
2.35
2.35
2.34
1.95
1.95
1.94
1.93
1.93
1.90
1.90
1.89
1.89
1.66
1.64
1.61
1.45
1.44
1.44
1.43
1.43
1.42
0.00

DFILE D-Cy Pd 1H .als
 COMMENT single_pulse
 DATIM 05-12-2007 20:16:49
 ORNUC 1H
 EXMOD single_pulse_ex2
 OBFREQ 500.16 MHz
 OBSFT 2.41 KHz
 OBFIN 6.01 Hz
 POINT 52428
 FREQU 7507.39 Hz
 SCANS 8
 ACQTM 6.9835 sec
 PD 5.0000 sec
 PW1 6.05 usec
 TRNUC 1H
 CTMP 27.8 e
 SLVNT CDCl3
 EXREF 0.00 ppm
 BF 0.12 Hz
 RGAIN 36

single pulse decoupled gated NOE

Y:\Seizo-win29\seizo\FW_Nabe\Xsp3 C-H BMYD-Cy Pd 13C .als

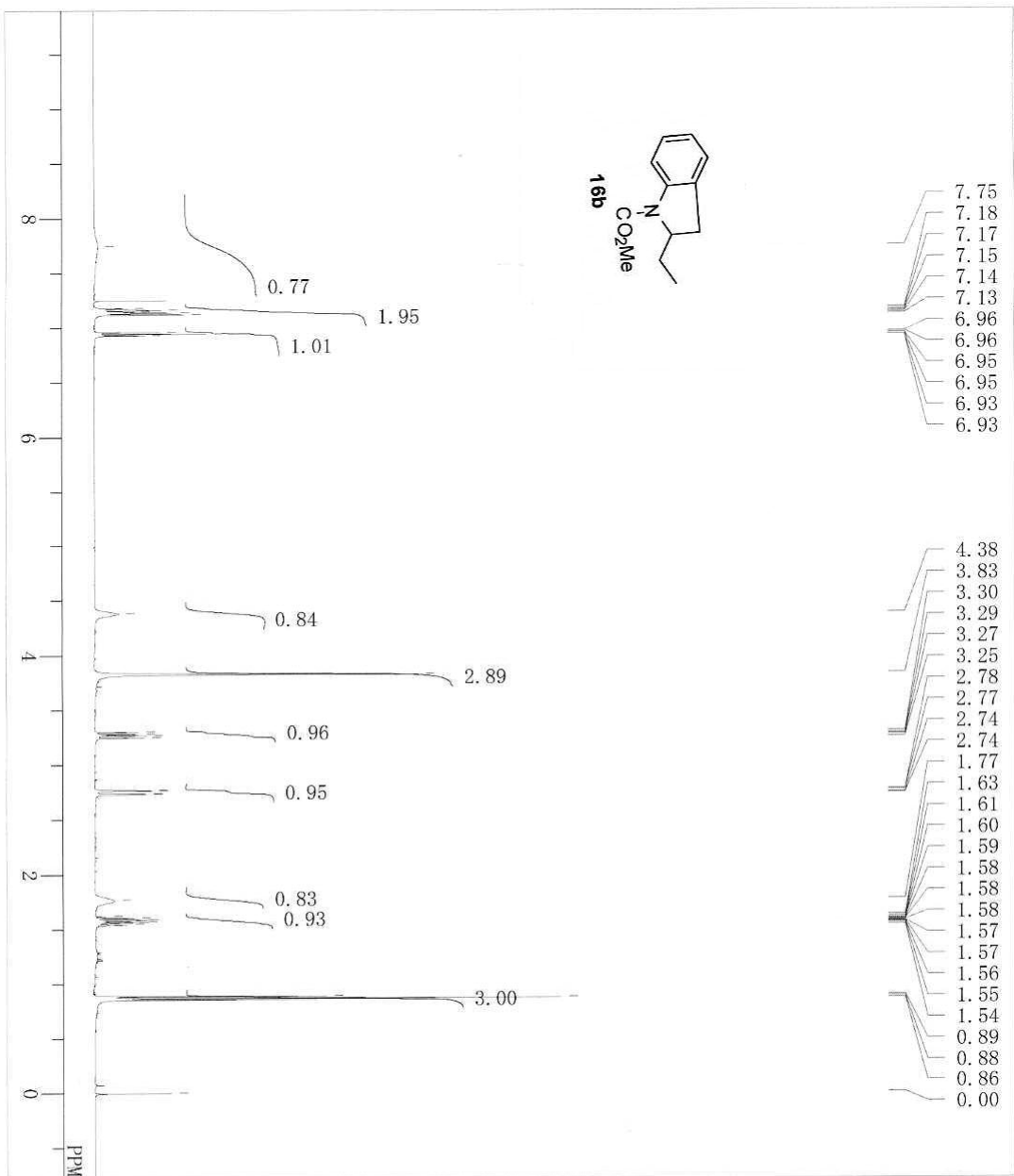


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DPFILE D-Cy Pd 13C .als
COMMENT single pulse decoupled gate
DATUM 05-12-2007 21:39:04
OBNUC 13C
EXMOD single_pulse_dec
OBFREQ 125.77 MHz
OBSET 7.87 kHz
OBFIN 4.21 Hz
POINT 52428
FREQU 31446.06 Hz
SCANS 900
ACQTM 1.6672 sec
PD 2.0000 sec
PWI 3.83 usec
IRNUC 1H
CTEMP 28.3 c
SLVNT CDCl3
EXREF 77.00 ppm
BF 1.20 Hz
RGAIN 58
  
```

single_pulse

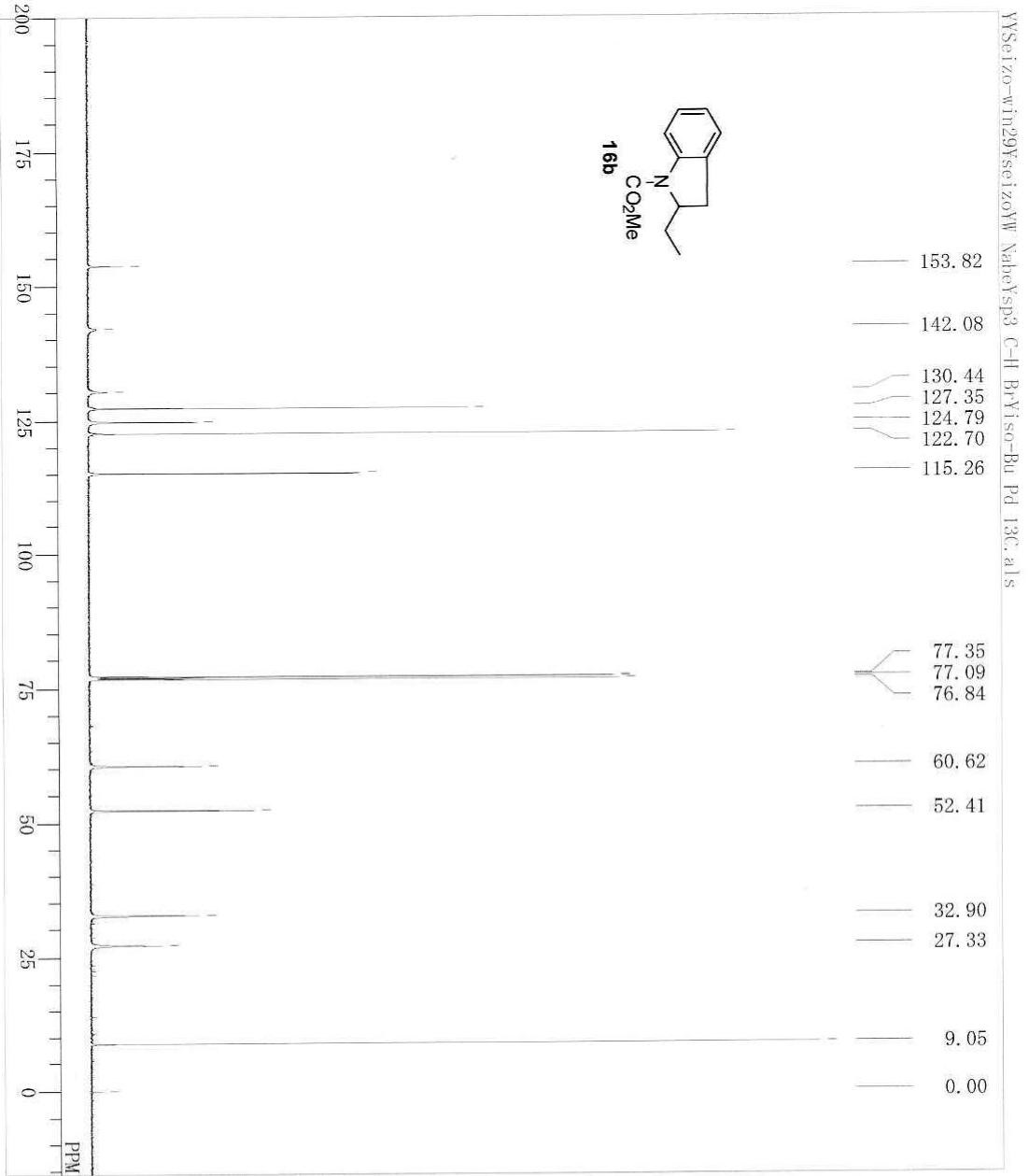
Y:\Seizo-w\129\Seizo\Y\Nabe\ksp3 C-H Br-Yiso-Bu Pd IH.a1s



```

DPFILE iso-Bu Pd IH.a1s
COMNT single_pulse
DATNM 07-12-2007 15:00:10
ORNUC IH
EXMOD single_pulse.ex2
OBFPRQ 500.16 MHz
OBSET 2.41 KHz
OBFIN 6.01 Hz
POINT 52428
FREQU 7507.39 Hz
SCANS 8
AQTM 6.9835 sec
PD 5.0000 sec
PW1 6.05 usec
IRNUC IH
CTEMP 26.7 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 34
  
```

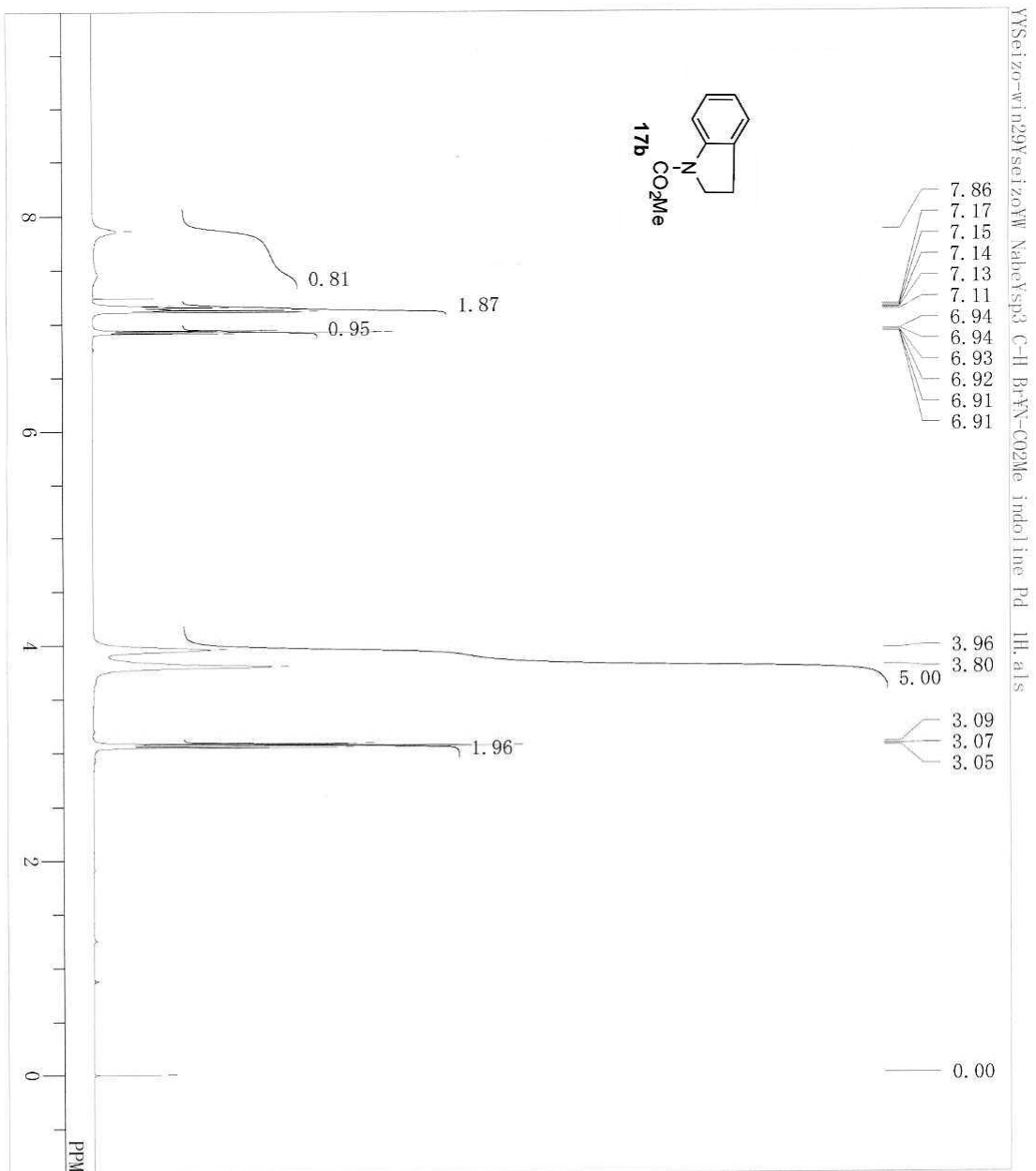
single pulse decoupled NOE



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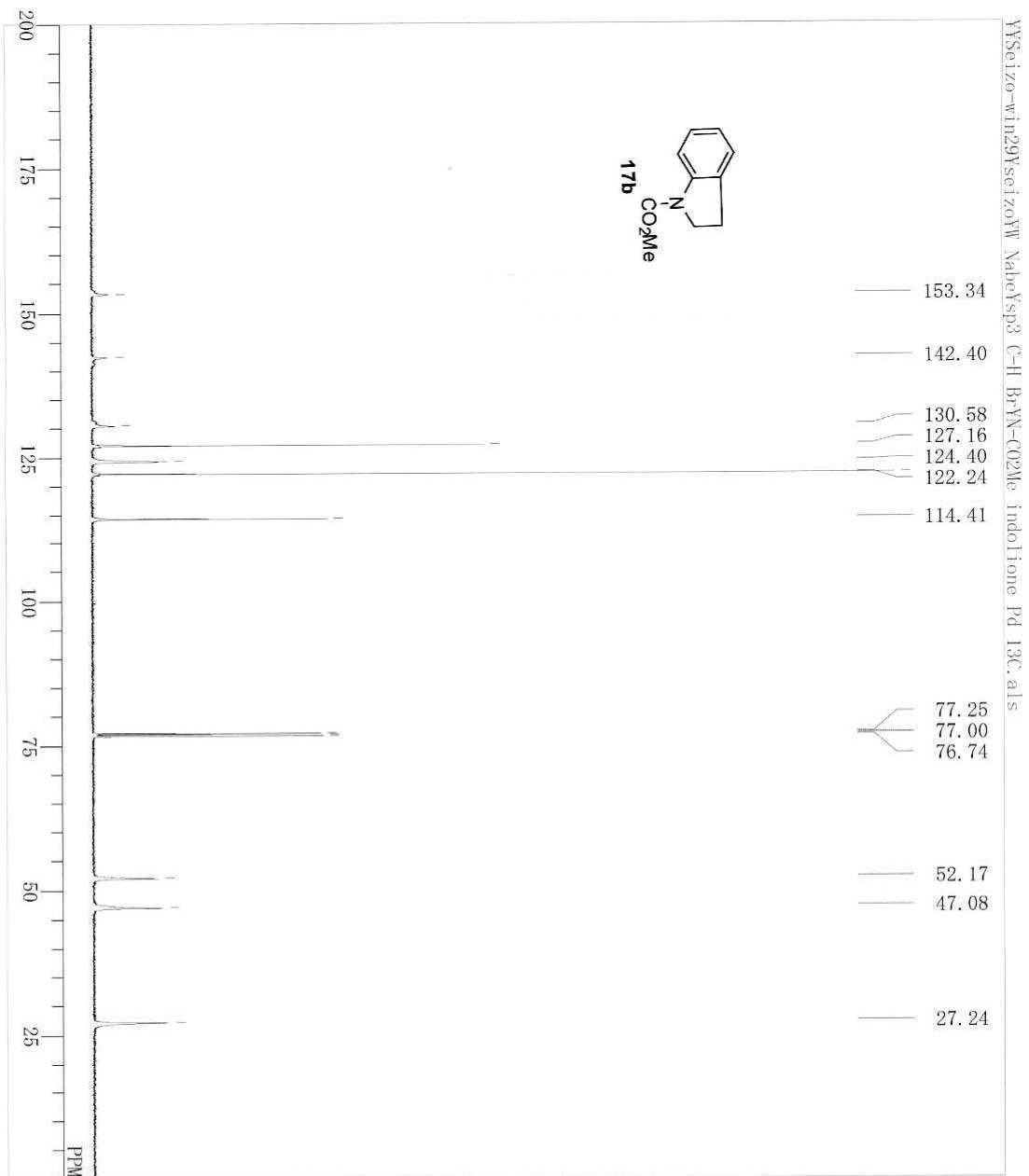
DPFILE iso-Bu Pd 13C. als
COMNT single pulse decoupled gate
DATIM 22-12-2007 08:32:44
13C
EXMOD single_pulse_dec
OBFRQ 125.77 MHz
OBSET 7.87 KHz
OBFIN 4.21 Hz
POINT 52428
FREQU 31446.06 Hz
SCANS 4500
ACQTM 1.6672 sec
PD 2.0000 sec
PW1 3.83 usec
IRNUC 1H
CTEMP 26.1 c
SLVNT CDCl3
EXREF 0.00 ppm
BF 1.20 Hz
RGAIN 58
  
```

single_pulse



DF1LE N-CO2Me indoline Pd 1H.als
 COMNT single_pulse
 DATIM 19-12-2007 16:23:14
 OBNUC 1H
 EXMOD single_pulse.ex2
 OBFPRQ 500.16 MHz
 OBSSET 2.41 KHz
 OBFINT 6.01 Hz
 POINT 26214
 FREQU 7507.39 Hz
 SCANS 8
 ACQTM 3.4918 sec
 PD 5.0000 sec
 PW1 6.05 usec
 TRNUC 1H
 CTEMP 26.8 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.12 Hz
 RGAIN 28

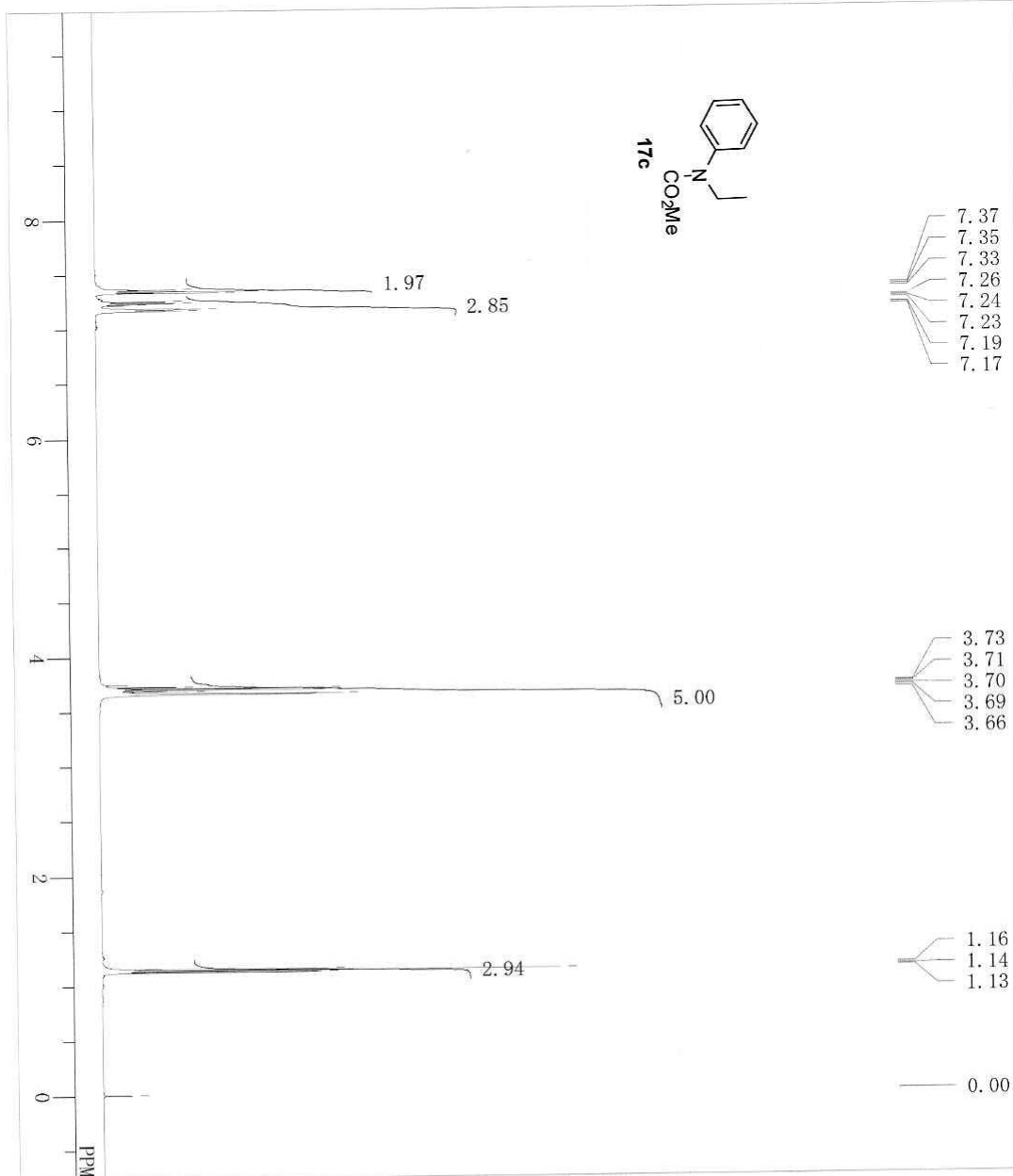
single pulse decoupled gated NOE



DFFILE N-CO2Me indoline Pd 13C.a1
 COMNT single pulse decoupled gate
 DATIM 08-12-2007 16:10:29
 OBNDIC 13C
 EXMOD single pulse dec
 OBFREQ 125.77 MHz
 OBSEFT 7.87 KHz
 OBFINT 4.21 Hz
 POINT 52428
 FREQU 31446.06 Hz
 SCANS 725
 ACQTM 1.6672 sec
 PD 2.0000 sec
 PW1 3.83 usec
 IRNUC 1H
 CTEMP 26.5 c
 SLVNT CDCL3
 EXREF 77.00 ppm
 BF 1.20 Hz
 RGAIN 60

single_pulse

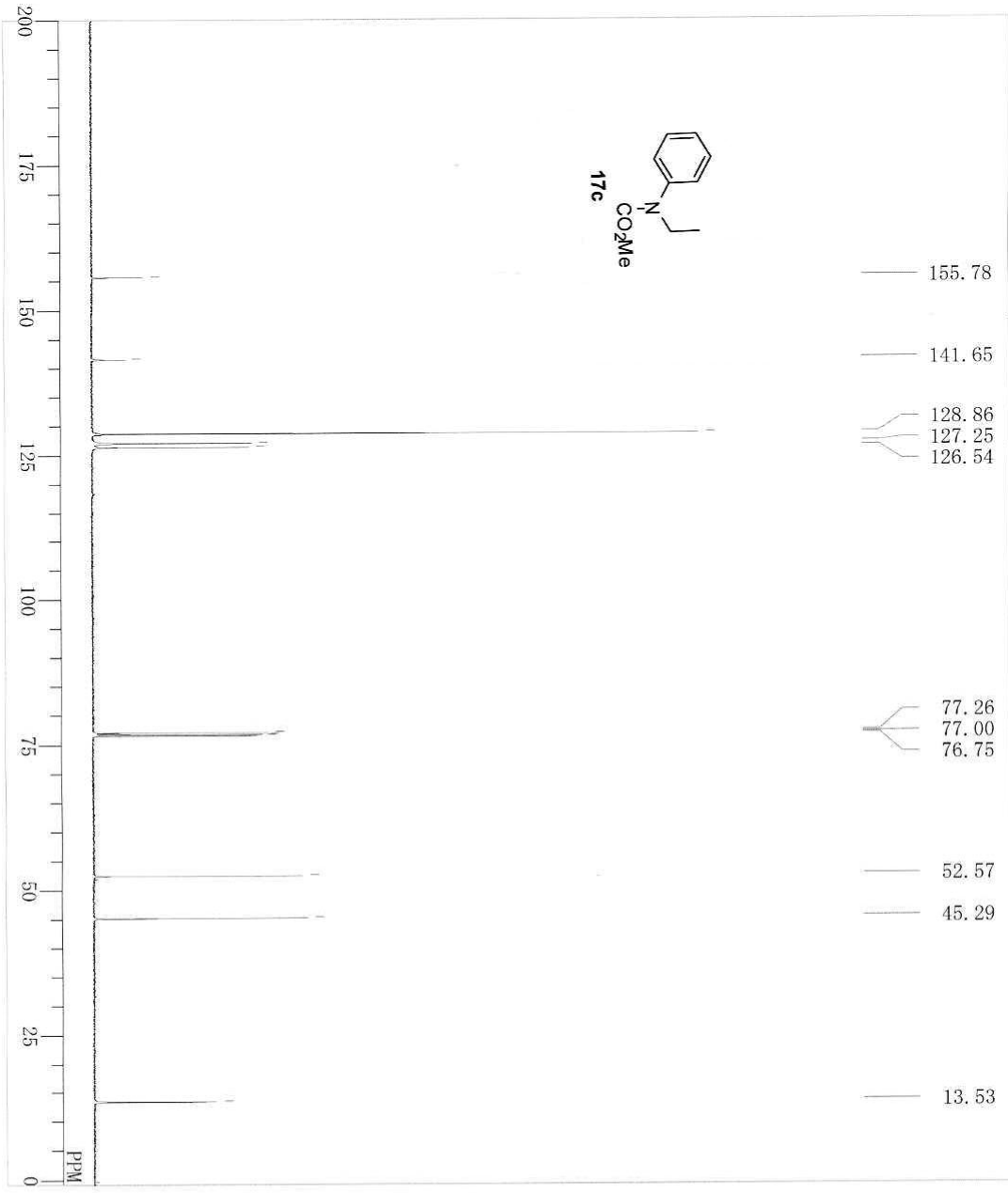
YYSSeizo-win29YSeizoW NabeYsp3 C-H BrYPh N-CO2Me 遷元体Et Pd IH. a1s



DFFILE Ph-N-CO2Me 遷元体Et Pd IH. a
COMNT single_pulse
DATIM 11-12-2007 17:22:34
IH
EXMOD single_pulse.ex2
OBPRQ 500.16 MHz
OBSET 2.41 KHz
OBPTN 6.01 Hz
POINT 52428
FRBQU 7507.39 Hz
SCANS 6
ACQTM 6.9835 sec
PD 5.0000 sec
PW1 6.05 usec
IRNUC IH
CTEMP 26.8 c
SLVNT CDCl3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 30

single pulse decoupled gated NOE

Y:\Seizo-win\29Yseizo\W NahoeYsp3 C-H Br+Ph N-CO2Me 遷元体Et Pd 13C. als



Ph N-CO2Me 遷元体Et Pd 13C.
 single pulse decoupled gate
 11-12-2007 18:12:50
 13C
 single_pulse_dec
 EXMOD 125.77 MHz
 OBFRQ 7.87 KHz
 OBSFT 4.21 Hz
 OBFTN 26214
 POINT 31446.06 Hz
 FREQ 1050
 SCANS 0.8336 sec
 ACQTM 2.0000 sec
 PD 3.83 usec
 PW1
 IRNUC 1H
 CTEMP 27.0 c
 SLVNT CDCL3
 EXREF 77.00 ppm
 BF 1.20 Hz
 RGAIN 60